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Volker Starrock and Alfred Krippendorf

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Edited by
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Alfred Krippendorf, Hazard Control Ltd., Fassberg

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PREFACE

The work described in this report was presented at the "International Symposium on the Destruction of Chemical Weapons – Technologies and Practical Aspects" in Munster, Germany in March 1998, nearly one year after the Chemical Weapons Convention entered into force, and right at the place, where German chemical warfare activities started during World War I.

This symposium was arranged by the Munster EXPO 2000 Committee (ARGE EXPO 2000) in close co-operation with the German Federal Ministries of Defence and for Foreign Affairs and was generously supported by the U.S. Army European Research Office as well. The symposium was chaired by Dr. Bernd Staginnus, Director, Federal Armed Forces NBC Defence Institute Munster (WIS), and was co-chaired by Dr. Wolfgang Spyra, Full Professor, Brandenburg Technical University at Cottbus (BTU). The Federal Armed Forces NBC Defence Institute (WIS) was in charge of putting together the scientific and technical programme of this symposium, while the Munster EXPO 2000 Committee (ARGE EXPO 2000) took care of the entire symposium organization, kindly supported by all Munster Armed Forces installations.

The purpose of the symposium was to create an interdisciplinary forum for the mutual exchange of information between international and government agencies, industry, and the scientific community with special emphasis to the technological and practical aspects of the destruction of chemical weapons, and to foster further progress towards the accomplishment of this challenging task.

There was quite an overwhelming international and national response to this symposium clearly indicating the importance of such a particular meeting; approx. 180 attendees from 14 different countries gathered in Munster on this occasion. The editors would like to thank both all the contributors for their presentations and the participating industry for the exhibition kindly arranged in connection with the symposium.

It is hoped that this report will be widely accepted and distributed within the international community dealing with the technological and practical aspects of the destruction of chemical weapons to promote further fruitful developments in this field.

Volker Starrock and Alfred Krippendorf, Editors

Munster, June 1998

ACKNOWLEDEMENT

The Munster EXPO 2000 Committee gratefully acknowledges the support granted by both the German Federal Ministry of Defence and the U. S. Army European Research Office (ERO) to hold this symposium.

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IUPAC WORKING PARTY ON CHEMICAL WEAPONS DESTRUCTION TECHNOLOGIES

Graham S. Pearson

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Introduction

The International Union of Pure and Applied Chemistry (IUPAC) Working Party on Chemical Weapons Destruction Technologies has been set up to carry out an independent review of the chemical weapon destruction technologies which have been demonstrated to be effective for the destruction of chemical warfare agents. The Working Party is affiliated with the IUPAC Committee on Chemical Weapons Destruction Technologies. The membership of the Working Party is Professor Irina Beletskaya, Department of Chemistry, Moscow State University, Russia, Professor Herbert de Bisschop, Head, Department of Chemistry, Royal Military Academy, Brussels, Belgium, Professor Richard Magee, Executive Director, Center for Environmental Engineering and Science, New Jersey Institute of Technology, Newark, NJ, USA and Professor Graham Pearson, Department of Peace Studies, University of Bradford, UK as Chairman.

Approach

The aim of the Working Party is to carry out a comprehensive review of technologies for destruction of chemical weapons. Whilst there is much published information about the technologies that some countries use or intend to use, there is a paucity of information about the proven effectiveness of the destruction technology especially in respect of the freedom from environmental pollution of the process. Some of the available information emphasises the technology or technologies preferred by a nation. The Working Party is carrying out an objective comparison of proven destruction technologies.

Our approach is to utilize and build upon all available information. The final product will be an authoritative and informative independent appraisal of the technologies available aimed at the officials and citizens of CWC States Parties who have to destroy any chemical weapons (CW) in their possession or that are found on their territory. The appraisal will provide a primer of the state of the art which will focus on technological maturity, the scale of proven chemical agent destruction and the potential impacts of the destruction technology on health and the environment.

Report Structure

The planned structure for the report of the Working Party is as indicated:

I	**Mandate for destruction**
II	**Historical perspective**
III	**Nature of the problem**
IV	**Transport of CW munitions and agent**
V	**Removal of CW agents**
VI	**Agent destruction - high temperature**
VII	**Agent destruction - low temperature**
VIII	**Effluent treatment**
IX	**Old recovered munitions**
X	**Conclusions**

Mandate for Destruction

This section will start by providing a historical perspective about the nature of chemical warfare and what agents were developed, used or stockpiled in World War I and II and subsequently thereby setting the scene in respect of the types of chemical warfare agents that need to be destroyed. It then goes on to show that there are hazards associated both with destroying chemical weapons and with not destroying chemical weapons. The requirements of the Chemical Weapons Convention (CWC) are then outlined in respect both of the total prohibition of the development, production, stockpiling and acquisition of chemical weapons and of the destruction of chemical weapons within a specified time from the entry into force of 29 April 1997 of the

Convention -- in 10 years for Schedule 1 chemicals and in 5 years for all other chemicals in chemical weapons. It is clear that there are two types of chemical weapon/chemical warfare agents -- those that are held in a stockpile, either as a weapon or in bulk agent storage and are thus generally in reasonable condition and of a known nature, and those that are non-stockpile, which are frequently buried in unknown locations, of uncertain condition and unknown nature.

Historical Perspective of CW Destruction

Previous methods of disposal, such as burial, dumping at sea and open-pit burning, are no longer acceptable and, indeed, are specifically prohibited in the CWC. Nevertheless, very large quantities of CW and of chemical warfare agents have already been safely destroyed using two principal techniques -- incineration and neutralization.

Nature of the Problem

The next section recognises that there are the two principal categories -- stockpile and non-stockpile -- which have different CWC destruction requirements. It notes that there are several components that have to be destroyed or decontaminated. First, there is the CW agent itself, second is the munition/container in which it had been stored, third are the energetic components of the munition (fuses, primers, explosives and propellants, and fourth are the packaging materials such as the pallets on which the munitions were stored. Each of these may require a different technique for its destruction or decontamination recognising that the levels of contamination with agent can be very different.

The criteria as to what is an environmentally acceptable process varies from country to country, as well as with time, as more demanding standards are set for what is permissible to be released into the environment. Whatever destruction method is used for CW and CW agents, the aim must be to achieve an appropriately high standard of safety and of environmental responsibility and acceptability. There is a trade off to be achieved between these: environmental emission standards for process residuals must be set at realistic and scientifically justifiable levels so that the destruction can be carried out safely with minimum health risk both in the short and the long term to those carrying out the destruction and to those living in the vicinity of the destruction facility and with the minimal impact on the environment. If environmental emission levels are set unrealistically low, the processes may be very slow and may be potentially more hazardous to plant personnel because of their extended duration.

There are three principal categories of chemical warfare agent in the stockpiled munitions and bulk agents: Mustard gas, Lewisite and the nerve agents (GA, GB and VX). Although incineration can be used for all three types, the incineration of lewisite, which contains arsenic, requires that the arsenic products be collected prior to release to the environment. There are a wide range of alternative technologies for the destruction of chemical warfare agents; however, these technologies have to be selected for the nature of the agent to be destroyed. Not every agent can be readily destroyed by every technique. A further factor relates to the quantity of agent to be destroyed and the process to be used. For large quantities of agent, there are advantages in using a destruction process that can be run continuously, whereas for small quantities of agent, a batch process will suffice and often be more convenient as it lends itself to being used on a campaign basis. Further, agent contaminated munitions or containers, energetics and/or packaging materials may require a different technological solution than the neat agent alone. Sections of the report will address the transportation of CW and chemical warfare agents as well as processes for the removal of agents from the chemical weapon.

Destruction technologies are conveniently divided into high and low temperature processes together with effluent treatment. The following processes are regarded by the Working Party as having been sufficiently demonstrated with actual chemical warfare agent and thus are considered as credible candidates for chemical agent destruction within the time frame of the CWC requirement.

High Temperature

Incineration
Pyrolysis
Catalytic Extraction Processing

Hydrogenolysis

Low Temperature

Hydrolysis with water
Reactions with aqueous sodium hydroxide
Reactions with amines
Electrochemical oxidation with Silver II
Sodium in liquid ammonia
Arsenicals

Effluent treatment is addressed according to its nature under the three headings: gas, liquid and solid.

Selected Technologies

The high temperature technologies being addressed are:

a. Incineration, regarded in many countries as the baseline process for CW destruction

b. Pyrolysis

c. Catalytic Extraction Processing using molten iron or nickel metal

d. Hydrogenolysis using hydrogen and steam to reduce nerve agents and mustard

The low temperature technologies being addressed are:

a. Hydrolysis of mustard agent with hot water.

b. Hydrolysis of nerve agents and mustard with aqueous sodium hydroxide

c. Reaction of nerve agents with monoethanolamine

d. Electrochemical oxidation using a silver (II) to oxidize nerve agent or mustard

e. Sodium in liquid ammonia

f. Processes for arsenical agents

For each of the technologies selected, available information will be provided on the following

- Process description
- Scientific principles
- Technology status
- Safety considerations
- Environmental impact

Effluent Treatment

The status of various technologies for treatment of gas, liquid and solid effluent will be discussed. These will include the following:

- Gas - acid scrubber, reactive carbon, particulate removal, sulphur recovery...
- Liquid - super critical water oxidation, biodegradation, UV H_2O_2, ...
- Solid - Solidification, stabilization, biomass, salts to landfill, ...

Old Recovered Munitions

Many countries have old chemical weapons which are discovered from time to time usually during construction work which is frequently at a location in which it is not realized that chemical weapons have been buried. These present particular problems for their safe destruction and these will be addressed in a separate section of the report.

Conclusions

The IUPAC Working Party is working on bringing together all available information and is taking care to avoid duplication of other ongoing efforts. The Working Party recognises, however, that many of the destruction technologies thus far selected or used have been chosen in order to meet particular national or local requirements and concerns. The technology chosen for one location may well not be the most appropriate for another location and the aim of this IUPAC Working Party will be to set out the information available about the various proven technologies and the options that need to be selected by the States faced with destroying the CW within their State.

Request for Information

The IUPAC Working Party would be very grateful for any information that can be provided to them about the technologies that have been demonstrated to be effective in destroying CW and chemical warfare agent. As the timescale for destruction of CW is very tight -- 10 years for CW containing Schedule 1 agents and 5 years for CW containing all other chemicals, the IUPAC Working Party has limited its consideration to proven technologies which have demonstrated the destruction of at least 1 kg of CW agent. Information should be sent to the Chairman, Professor Graham S Pearson at the following address:

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ACTIVITIES OF NATO IN THE FIELD OF CW DEMILITARIZATION TECHNOLOGIES

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Abstract

This paper deals with the activities of the NATO Advisory Panel on Disarmament Technologies in organizing so-called Advanced Research Workshops (ARW) in the field of Chemical Weapons. The most important results of these workshops which have been published up to now are described.

Introduction

Chemical Weapons (CW) have been around since the First World War and methods for CW destruction have evolved over the past half-century.

The problem of old and abandoned chemical weapons poses an additional challenge.

Over the course of several decades, a broad and complex agreement known as the Chemical Weapons Convention (CWC) was negotiated. Since 1993, CWC has been signed by 165 countries and ratified by 89 countries.[1)]

The CWC defines the destruction of chemical weapons as „a process by which chemicals are converted in an essentially irreversible way to a form unsuitable for production of chemical weapons, and which, in an irreversible manner, renders munitions and other devices unusable as such". The method of destruction is to be determined by each country, but the manner of destruction must ensure public safety and protect the environment. [1)]

We differentiate between stockpiled and non-stockpiled weapons. Stockpiled weapons are identified and characterized and stored in controlled environment, e.g. ammunition depots. All other items are non-stockpiled and include old munitions discovered during excavation, abandoned and lost munitions etc. Only the United States and the Russian Federation have officially acknowledged their stockpile of chemical weapons.

Two basic types of chemical agents comprise the CW stockpile: neurotoxic (nerve) agents and mustard (blister) agents. Both types are frequently , and erroneously, referres to as „gases", even though they are liquids at normal temperature and pressure. [1)]

The nerve agents include the organic phosphorous compounds designated as VX, GB (Sarin), and GA (Tabun). These chemicals present a significant toxic hazard because of their action on the nervous system of humans and animals through inhibition of acetylcholinesterase enzyme. They are considered extremely toxic and, even though VX is more acutely toxic than GB, the latter represents a greater potential hazard because of its higher volatibility and thus, the greater likelihood of being inhaled.

The mustards (designated H [non-distilled mustard], HD [distilled mustard], and HT [thickened mustard]) and lewisite do not present significant acute lethal hazards. Their principal effect is severe blistering of the skin and mucous membranes. They have been implicated as being carcinogenic, however, and may present a cancer hazard to individuals exposed acutely.[1)]

Mustard gas has been used by Saddam Hussein against Kurdish inhabitants in Halabja, March 1988.

The Chemical Weapons Convention (CWC) makes a distinction between various classes of chemical weapons. They are [2)]:

a) Chemical weapons produced before 1925,
b) Chemical Weapons produced between 1925 and 1946 which are determined to be „unusable",
c) Chemical Weapons produced between 1925 and 1946 which are determined to be „usable",
d) Chemical Weapons produced after 1946, irrespective of their condition,
e) Chemical Weapons buried before January 1977 (and which remain buried),
f) Chemical Weapons dumped at sea before January 1985, and
g) Abandoned Chemical Weapons.

Chemical weapons of these classes are perceived to pose different level of risk to the object and purpose of the Convention. Chemical weapons which were produced either before 1925 or produced between 1925 and 1946 and which are determined to be „unusable" are both classified as Old Chemical Weapons. Old Chemical Weapons must be declared and, after their status has been verified by the Technical Secretariat, must be destroyed as toxic waste.

Abandoned Chemical Weapons are defined by the Convention as all chemical weapons, including Old Chemical Weapons, which were abandoned by a State after 1 January 1925 on the territory of another State without the latter's consent.

Destruction must be undertaken at a declared facility and in a safe manner.

In 1969 the US Army began developing industrial scale processes to destroy large quantities of chemical weapons under controlled conditions. Incineration and chemical neutralization with alkali were both used. A prototype incinerator was constructed and tested in Tooele, Utah starting in 1979 and compared with chemical neutralization [3].

Based on those tests, in 1983 the Army chose incineration and built a full scale incinerator on Johnston Island in the Pacific. This system began tests in 1990 and, by early 1996, had destroyed more than one thousand tons of chemical agent. Some of the materials destroyed at Johnston Island were weapons previously stored in Germany by the United States and removed in 1990.

At Tooele, Utah, a second generation incinerator was built and began operation in the summer of 1996.

In Munster, Germany, a munition disposal facility is used for weapons manufactured during World War I and II [4]. Old chemical weapons found on this site include: arsenicals (Lewisite, Adamsite, Clark I and II), sulfur and nitrogen mustards, phosgene and tabun. Often sulfur mustard is mixed with arsenicals such as phenyldichlorarsine, and aging has created a complex mixture called „viscous mustard" - a major disposal problem.

Agents are drained into polyethylene barrels and incinerated at 1000°C. Phosgene is not incinerated, but is hydrolyzed.

Arsenicals are precipitated out of the incinerator scrubber as $FeAsO_4$. The arsenic sludge is buried in drums in a waste dump, a former salt mine. Since operation began in 1980, 73 tons of concentrated chemical weapons agents and 600 tons of contaminated material have been destroyed.

Recently, a special incinerator is used for arsenic contaminated soil. After a flotation soil wash (most of the arsenic goes with 10% of the soil), the enriched fraction goes to a plasma arc reactor where agents, carbon etc. Are combusted and the soil is melted to a glassy slag containing some arsenic.

Activities of the NATO Advisory Panel on Disarmament Technologies

The Disarmament Technologies Panel has supported several Advanced Research Workshops (ARW), which are activities with cooperation partners. The most important workshops in connection with chemical weapons are the following:

1) Destruction of Military Toxic Materials
Dr. R.W. Shaw, US Army Research Office, North Carolina, USA
Prof. O. Korobeinichev, Russian Academy of Sciences, Novosibirsk, Russia
22-27 May 1994, Naaldwijk, Netherlands

2) Sea Dumped Chemical Munitions
Mr. K. Olson, Chemical and Biological Arms Control Institute, Virginia, USA
Dr. A. Kaffka, Russian Academy of Sciences, Moscow
12-14 January 1995, Kaliningrad / Moscow

3) Scientific Advances in Alternative Demilitarization Technologies
Dr. F.W. Holm, Science Applications International Corporation, MD, USA
Dr. M. Koch, Military Institute of Chemistry and Radiometry, Warsaw, Poland
24-25 April 1995, Warsaw, Poland

4) Chemical Problems associated with Old Arsenical and Mustard Munitions
Prof. J.E. Bunnett, University of California, USA
Prof. M. Mikolajeczyk, Polish Academy of Sciences, Lodz, Poland
17-19 March 1996. Lodz. Poland

5) Chemical and Biological Technologies for the Detection, Destruction and Decontamination of Chemical Warfare Agents
Prof. J.R. Wild, Dept. Biochemistry, Texas University, USA
Dr. A. Boronin, Environmental Science and Biotechnology, Pushchino State University, Russia
11-15 May 1996, Moscow, Russia

6) Analytical Chemistry associated with the Destruction of Chemical Weapons
Ms. M. Heyl, AMC Treaty Laboratory, USA
Dr. V. Stein, Military Technical Institute of Protection, Brno, C.R.
12-15 May 1996, Brno, Czech Republic

7) Assessment of Mobile Technologies for Remediating Sites Contaminated with Chemical Warfare
Dr. F.W. Holm, USA
Prof. F. Kastanek, Czech Republic
1-2 July 1996, Prague, Czech Republic

8) Destroying Chemical Weapons: Technical Responses to Safety, Health, and Environmental Concerns
Dr. T. Wander, The Potomac Foundation, USA
Mr. V. Orlov, Policy Studies in Russia Center, Moscow, Russia
18-20 November 1996, Moscow, Russia

Of these mentioned eight NATO Advanced Research Workshops, there have been published only the Proceedings of four workshops. This is the reason that we can describe in more detail only four workshops.

Robert W. Shaw
Destruction of Toxic Military Materials
Naaldwijk, The Netherlands, 22-27 May 1994

At this workshop, disposal problems were described for the US, Germany, Russia, Indonesia and Iraq. The technologies discussed were high temperature processes such as incineration, plasma torch and molten metal. Also medium temperature processes were treated: pyrolysis, supercritical and wet air oxidation, hydrogenation. Under the low temperature processes, metal oxide catalysis, silver (II) oxidative electrolysis reactions with dry HCl were discussed.

Participants developed a set of disposal problem scenarios and made recommendations for best technologies for each scenario.

Although much information about the chemistry of chemical warfare agents is not readily available, several recent reports about destruction of chemical warfare agents have been published. For example, effective chemical recipes exist for detoxifying chemical agents [5) 6)] .

Alexander V. Kaffka
Sea-Dumped Chemical Weapons: Aspects, Problems and Solutions
Kaliningrad (Moscow Region), Russia, January 12-15, 1995

The problems of the ecological threat produced by chemical weapons (CW) dumped in the seas after the Second World War are described [7)] .

After World War II CW were disposed of in the shallow depths of Baltic Sea and North Sea area of active fishing - in close proximity to densely populated coastlines, with no consideration of the long-term consequences.

The conference in Kaliningrad was the first attempt to address this sensitive issue of sea-dumped CW on a comprehensive scientific basis. One of the achievements of the conference was that it was able to raise virtually all the questions in its agenda and to lay a basis for further comprehensive applied research.

The conclusion of the conference in Kaliningrad was the following:

1 The old chemical munitions dumped in the North European seas pose a potential environmental danger;
2 The scale of this environmental danger is not qualifiable at present. Steps should be undertaken by the international community to provide a solid scientific assessment of the situation.

Francis W. Holm
Scientific Advances in Alternative Demilitarization Technologies
Warsaw, Poland, April 24-15, 1995

In the conference, scientists and engineers of different countries delivered presentations of their findings [8] :

Countries describing their technologies included:

- Poland: pre-processing, thermal oxidation and instrumentation
- Russia: molten salt oxidation, plasma, catalytic oxidation, molten metal, fluid bed reactions and hydrogenation
- Germany: supercritical water oxidation and detoxification
- United Kingdom: electrochemical oxidation
- United States: wet air oxidation, detoxification and biodegradation
- Czech Republic: biodegradation

Only a few low-temperature technologies are capable of providing for complete agent destruction in a single-step process. In most cases, the technologies require multi-step processes, e.e., combining technologies in series, to achieve the level of destruction required. High temperature and low-pressure oxidation technology alternatives, like incineration demonstrate the capability to destroy agents in a single-step process and provide braod versatility in dealing with other chemical weapons components.

Francis W. Holm
Mobile Alternative Demilitarization Technologies
Prague, Czech Republic, July 1-2, 1996

The mobile, or transportable technologies are alternatives to incineration technology for destruction of munitions, chemical warfare agents and associated materials and debris [9] .

These technologies are grouped into three categories based on bulk operation temperature:
- low temperature: 0 - 200°C
- medium temperature: 200 - 600°C
- high temperature: 600 - 3500°C

Reaction types considered include:
- hydrolysis
- biodegradation
- electrochemical oxidation
- gas-phase high-temperature reduction
- steam reforming
- gasification
- sulfur reactions
- solvated electron chemistry
- sodium reactions
- supercritical water oxidation
- wet air oxidation, and
- plasma torch technology

These procedures are being described in more detail in the book of the Advanced Research Workshop (ARW). It contains a broad spectrum of processes, some of which have been studied only in the laboratory and some of which are in commercial use for destruction of hazardous and toxic wastes.

A few low-temperature technolgies have the capability to provide for destruction in a single-step process.

Monica Heyl and Raymond Mc Guire
Analytical Chemistry associated with the Destruction of Chemical Weapons
Brno, Czech Republic, May 12-15, 1996

One of the major technical problems is that the stockpile of chemical agents have degraded with time, some quite seriously. Detecting and identifying the products of this decomposition and ageing process is important [10].

In addition it is very important to analyze chemical agents in the surrounding area of the destruction process.

A new sample preparation method based on solid phase micro-extraction (SPME) is presented.

With regard to mass spectrometry techniques, Chemical Ionization (CJ) and Ion Trap instruments are effective for the analysis of chemical agents.

There also seems to be growing interest in Liquid Chromatography / Mass Spectrometry (LC / MS) techniques, where very good results can be obtained on non-volatile compounds without having first to derivatize them.

The final major discussion topic is Air Monitoring and Screening techniques, primarily for use in measuring existing hazards.

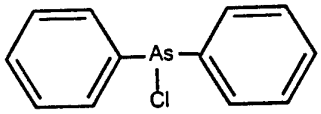
Database for Components of Propellants, Explosives, Pyrotechnics and Chemical Weapons

For the calculation of the performance of energetic materials such as gun propellants, rocket propellants, gas generators, high explosives, pyrotechnics, chemical agents and other ingredients which have been used together with energetic materials, thermochemical data are necessary.

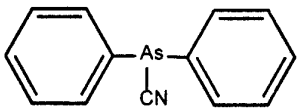
These data include the following properties of substances:
Sum formula, state of aggregation, molecular weight, oxygen balance, density, melting point, boiling point, enthalpy of formation, heat of combustion, structure formula.

In this connection, a database was established which contains data of about 4000 substances. The database is updated regularly and is available with English or German names of the substances and as MS-DOS or Windows - version.

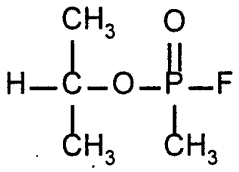
The database allows to search for
- names
- sum formulas
- part of names
- part of sum formulas
- substances consisting of certain elements
- substances with certain properties
- substances belonging to certain classes, e.g. oxidators, liquid propellants, chemical warfare agents, ...



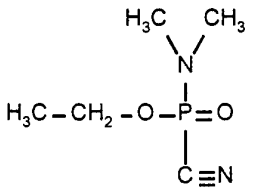
C 12 H 10 AS 1 CL 1		Solid
DA CLARK I	DIPHENYLCHLOROARSINE	CHLORODIPHENYLARSINE
Molecular Weight:	264.586 [g]	Oxygen Balance: -181.41 [%]
Density [g/cm3]:	1.422 at 20°C	Source: 232
Melting Point [°C]:	41-44.5	Source: 234
Boiling Point [°C]:	333 D	Source: 234
		1-1499



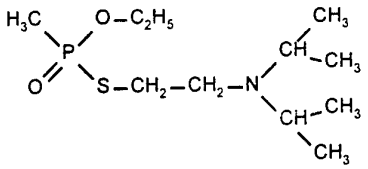
C 13 H 10 AS 1 N 1		Solid
DC DIPHENYLARSINECARBONITRILE DIPHENYLCYANOARSINE CLARK II		
Molecular Weight:	255.151 [g]	Oxygen Balance: -203.79 [%]
Density [g/cm3]:	1.45 at 20°C	Source: 232
Melting Point [°C]:	33	Source: 231
Boiling Point [°C]:	346 D	Source: 231
		1-1500



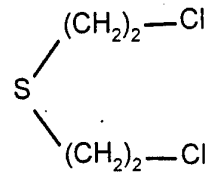
C 4 H 10 F 1 O 2 P 1		Liquid
SARIN	METHYLPHOSPHONOFLUORIDIC ACID ISOPROPYLESTER ISOPROPYLMETHANE FLUOROPHOSPHONATE GB	
Molecular Weight:	140.094 [g]	Oxygen Balance: -148.47 [%]
Density [g/cm3]:	1.089 at 25°C	Source: RP
Melting Point [°C]:	-57	Source: 231
Boiling Point [°C]:	147	Source: 231
		1-1488



C 5 H 11 N 2 O 2 P 1		Liquid
TABOON A	ETHYL-DIMETHYLAMIDOCYANOPHOSPHATE DIMETHYLPHOSPHORAMIDOCYANIDIC ACID ETHYLESTER	
Molecular Weight:	162.128 [g]	Oxygen Balance: -157.89 [%]
Density [g/cm3]:	1.073 at 25°C	Source: RP
Melting Point [°C]:	-48	Source: 231
Boiling Point [°C]:	246 D	Source: 231
		1-1480



C 11 H 26 N 1 O 2 P 1 S 1		Liquid
O-ETHYL-S-2-NN-DIISOPROPYLAMINOETHYL-METHYLPHOSPHONO-FLUORIDATE VX		
Molecular Weight:	267.367 [g]	Oxygen Balance: -224.40 [%]
Density [g/cm3]:	1.008 at 25°C	Source: RP
Melting Point [°C]:	-30	Source: 232
Boiling Point [°C]:	298	Source: RP
		1-1474



C 4 H 8 CL 2 S 1		Liquid
1,1'-THIOBIS-(2-CHLOROETHANE) BIS-(2-CHLOROETHYL)-SULFIDE		2,2'-DICHLORODIETHYLSULFIDE MUSTARD GAS
Molecular Weight:	159.074 [g]	Oxygen Balance: -130.75 [%]
Density [g/cm3]:	1.274 at 20°C	Source: H
Melting Point [°C]:	14.45	Source: B013
Boiling Point [°C]:	216.8	Source: B013
		1-1486

Conclusion

This paper deals with the activities of the NATO Advisory Panel on Disarmament Technologies in organizing and sponsoring so-called Advanced Research Workshops (ARW) in the area of Chemical Weapons.

The aim was to characterize the different chemical weapons and agents which are released from World War I and II and from later periods and which are stored in a variety of containment systems, also to find out the problem of the ecological threat posed by chemical weapons dumped in the seas after Second World War. In addition it was of great interest to put together all the knowledge and the experience we have in eastern and western countries with the disposal, destruction and decontamination of chemical warfare agents.

The same holds for the processes of recovery, cleaning, detection, identification and analysis of old and abandoned chemical weapons.

The results of the Advanced Research Workshops are contained in the NATO ASI Series published by Kluwer Academic Publishers in cooperation with the NATO Scientific Affairs Division. Our report exhibits the references and contents of all proceedings volumes which were published up to now in the field of chemical weapons.

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OVERVIEW OF THE UNITED STATES CHEMICAL DEMILITARIZATION PROGRAM

Dr. Theodore Prociv
Deputy Assistant to the Secretary of the Army

The United States Chemical Demilitarization Program has been in operation for approximately eight years. The original U.S. stockpile of chemical weapons (CW) totaled some 31,000 agent tons and is situated at eight locations in the continental United States with an additional site at Johnston Island in the Pacific Ocean. To date, 11.49 percent of that stockpile has been destroyed. With the entry into force of the Chemical Weapons Convention (CWC) on 29 April 1997, a 10 year deadline was established for the complete elimination of all CW stockpiles. The United States is preparing to meet this CW disposal deadline using the method of baseline incineration in addition to alternative technologies which are presently under study. The most paramount factor guiding the U.S. program is that of safety and protection to the public, personnel involved in destruction efforts, and the environment.

The Army has obtained a significant amount of experience over several years in evaluating varying methods for safely destroying chemical weapons. As a result of this extensive research, the Army developed the baseline incineration process for disposing of the stockpile. The National Research Council of the National Academy of Sciences has repeatedly endorsed the baseline technology as one that is technically sound and environmentally safe. It is the only demonstrated technology currently available that safely and effectively destroys all components of fully uploaded weapons – agent, explosive components, metal parts and associated dunnage.

Presently baseline incinerators are operating at Johnston Island and at the Tooele Chemical Agent Disposal Facility (TOCDF) at the Deseret Army Depot, Utah. Together, these two sites account for nearly 49 percent of the total U.S. chemical weapon stockpile. The facility at Johnston Island has been in operation since 1990 and has built on its successes there and used lessons learned with the baseline process to develop more efficient and safe destruction methods and facilities for Tooele. In addition to having destroyed 1,494 tons of agent (which translates to 73.6 percent of the stockpile at Johnston Island) and several different types of munitions, over 1.5 million pounds of explosives have also been destroyed there.

Although TOCDF has been operational only since August 1996, it has already surpassed the amount of agent successfully destroyed at Johnston Island. As of April 22, 1998, TOCDF disposed of 1,565 agent tons (approximately 9.8 percent of the Tooele stockpile) as well as various munitions and bulk containers.

Alternatives to the baseline incineration technology are currently under development for potential use at the two bulk-only storage sites: Newport, IN and Aberdeen, MD. They are well-suited to incorporate alternative technologies given that the agents are stored in steel ton containers which do not have explosive components or complex internal structures typically associated with chemical munitions.

A second program is underway to find alternative solutions for safely disposing of assembled CW munitions. Known as the Assembled Chemical Weapons Assessment (ACWA), the program was initiated in 1996 on legislative directives from Congress (PL 104-201 and PL 104-208). It requires the demonstration of at least two alternative technologies to baseline incineration for assembled chemical weapons. $40 million was appropriated for the effort and plans for construction of destruction facilities at two sites, Pueblo, CO and Blue Grass, KY, were suspended until six months after the final report is submitted to Congress. So far, seven companies have met the technical criteria that is required to proceed with further development of the technologies and they are each expected to be tested in the second half of 1998 . The final results are expected in December, 1998.

Two of the three remaining sites, Umatilla Chemical Agent Disposal Facility and Anniston Chemical Agent Disposal Facility, are under construction. Destruction operations are scheduled to begin there in November 2001 and January 2002, respectively. The announcement of a contract award for the destruction site in Pine Bluff, Arkansas is expected by April 30, 1998 with construction scheduled to begin by the fall of the same year.

In addition to the disposal of CW munitions at the stockpile sites, the United States is also working on a campaign to dispose of non-stockpile materiel. This consists of chemical warfare materiel (CWM) that is not included in the unitary chemical stockpile such as: binary chemical weapons, former chemical weapon production facilities, miscellaneous CWM, and buried and recovered CWM. Of these, the buried chemical weapon materiel represents the greatest challenge for recovery and disposal. The CWC does not obligate countries to recover chemical weapons buried prior to January 1, 1977. However, if they are recovered, they

must be declared to the Organization for the Prohibition of Chemical Weapons (OPCW) and eventually destroyed according to specified time limits within the treaty. Presently, the U.S. Army estimates that there are suspected burial sites at approximately 63 locations. In order to be able to respond in a safe and efficient manner in the event of a recovery of CWM, technology solutions have been or are currently being developed. These include the Rapid Response System, the Munitions Management Device (2 versions), and the Emergency Destruction System.

As the U.S. chemical demilitarization program continues to advance across the country, an increase in public awareness and involvement becomes more critical. Several initiatives have been launched to educate citizens living in affected communities, special environmental interest groups and other concerned individuals about the program. For example, Citizen's Advisory Commissions have been established at each of the sites to act as a clearinghouse of information and to involve the local public in reviewing environmental and technical data throughout the life of the program. At every site, outreach office staff manage booths at state and local fairs, business expos and other events to discuss the program and encourage people to ask questions and voice concerns. One of the most critical lessons learned during the last eight years is that maximizing stakeholder involvement has been a necessary ingredient for program success.

* * *

The United States is continuing its efforts to bring disposal operations at the other sites on-line in the near future in order to meet the CWC disposal deadline of 2007. Chemical demilitarization is a multi-faceted and complex program that requires close coordination among several key players. The program also assigns maximum safety to the public, environment, and workers as the first and foremost priority in its ongoing operations and during its continuing search for additional safe and effective destruction technologies.

Overview of the United States Chemical Demilitarization Program

International Symposium
Destruction of Chemical Weapons
- Technologies and Practical Aspects -

Munster, Germany

22-24 March 1998

Dr. Theodore Prociv

Deputy to the Assistant Secretary of Defense

for Chemical Demilitarization

Proceedings of the International CW Destruction Symposium
Munster, Germany, March 22-25, 1998

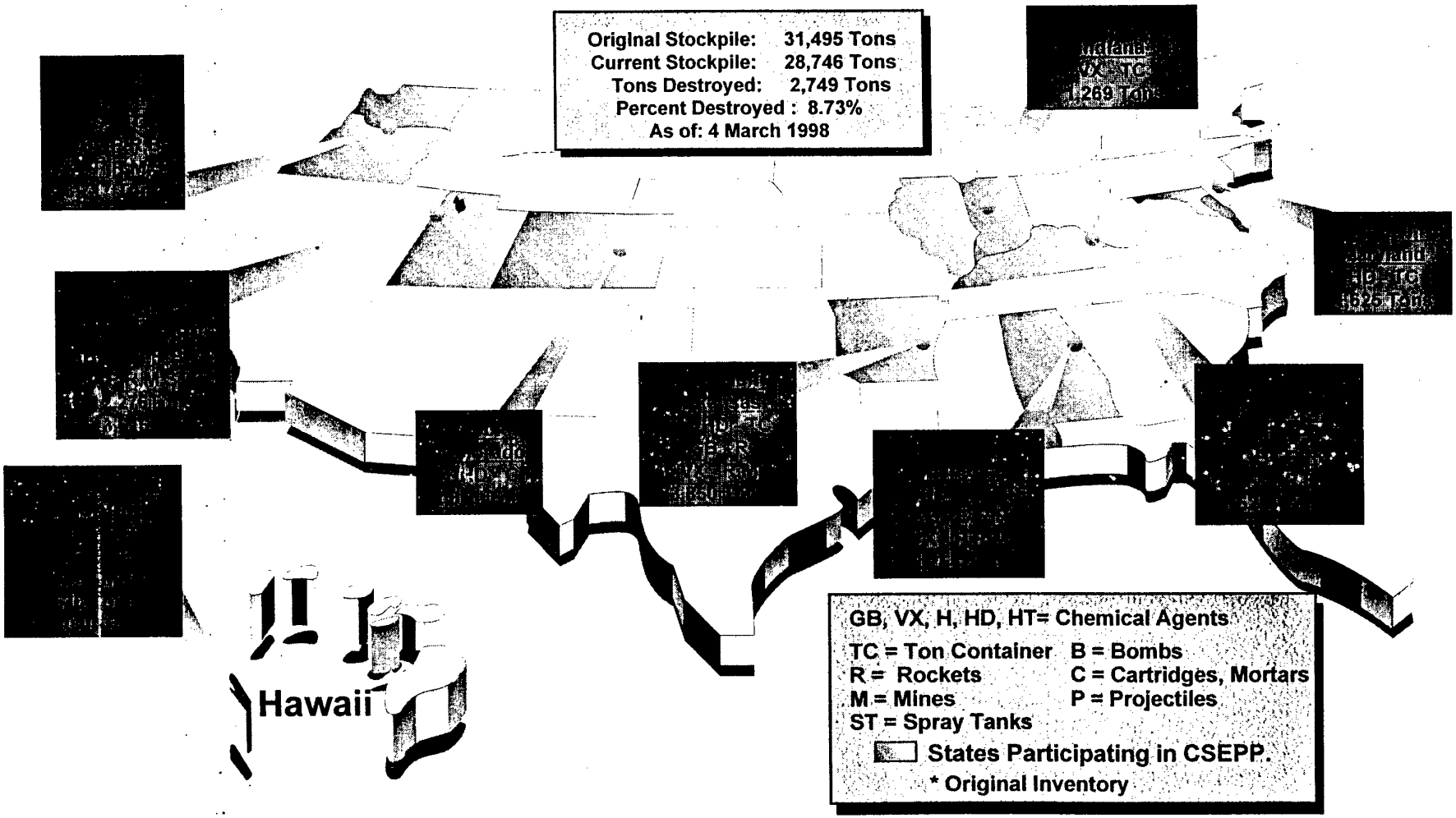
Mission

- **Destroy all U.S. Chemical Warfare Related Materiel while ensuring Maximum Protection of the Public and Personnel involved in the Destruction Efforts, and the Environment**

- **Chemical Weapons Destruction will be Completed by the CWC Deadline of 2007**
  - **baseline incineration**
  - **alternative technologies**

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Locations of the U.S. ChemicalWeapon Stockpile



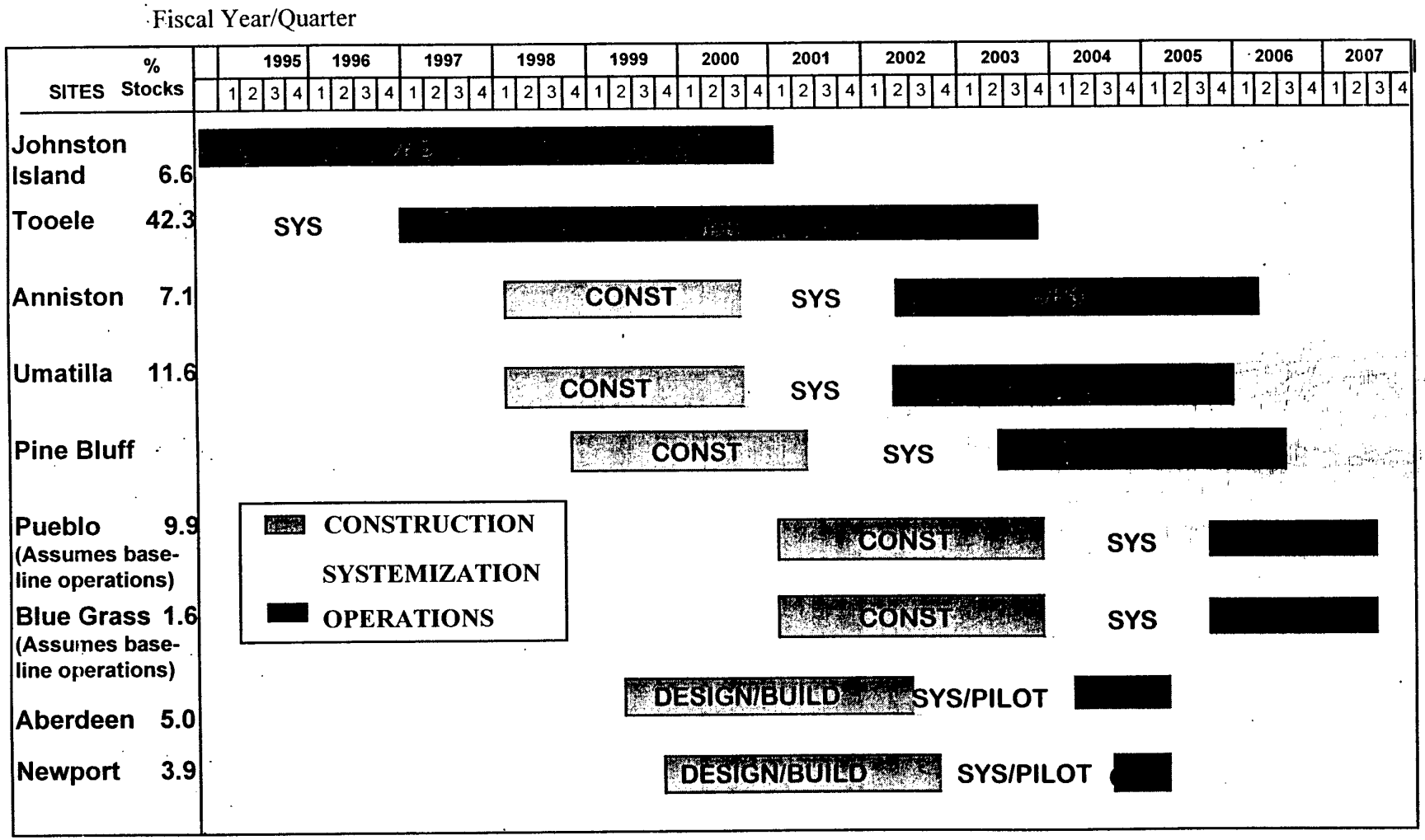
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Congressional Interests and Impacts

- P.L. 99-145 and 101-510: Requirements to report annual status of chemical stockpile disposal program
- P.L. 104-201 and 104-208: Requirements to conduct assessments of alternative technologies
- P.L. 104-206: Routine requirements to identify cost-saving measures
- SASC S.Rpt. 105-29: Requirements to report compliance with destruction obligations of the CWC

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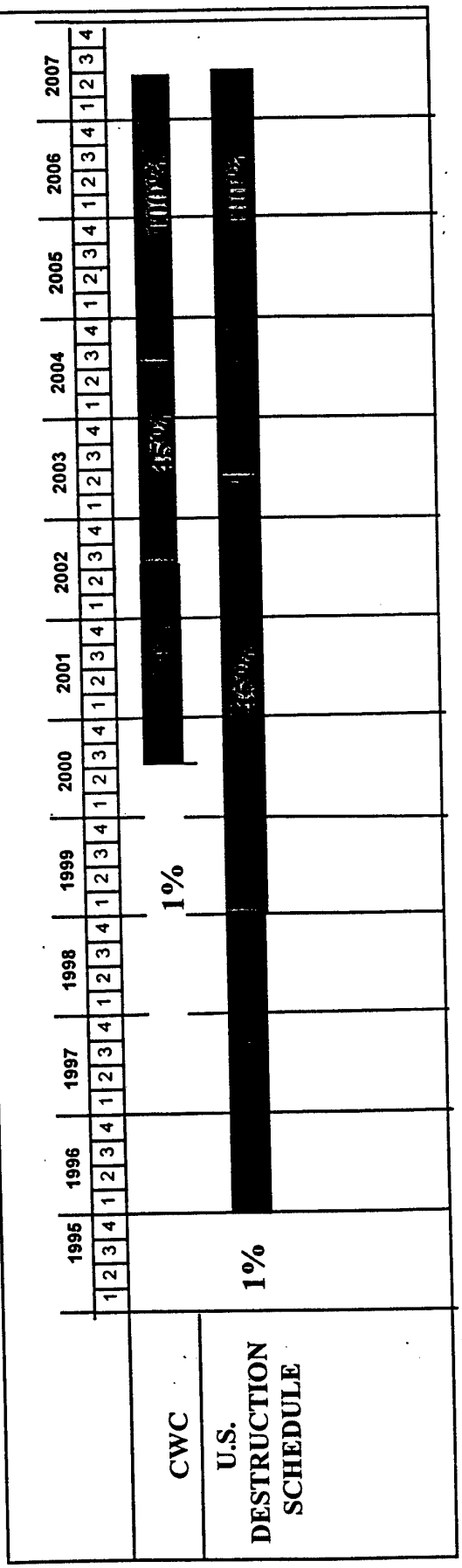
Chemical Stockpile Disposal Schedule



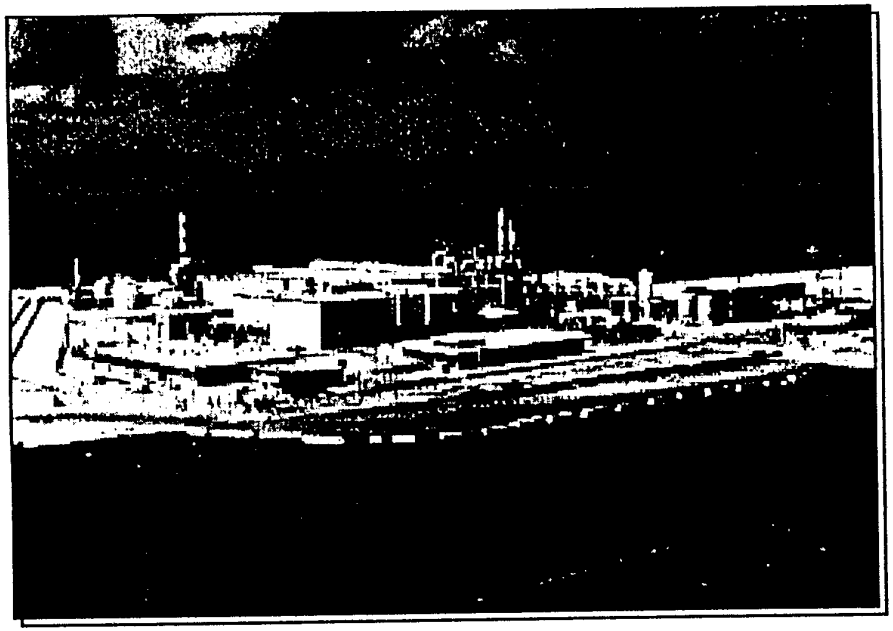
As of February 1998

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CWC Category I Destruction Requirements

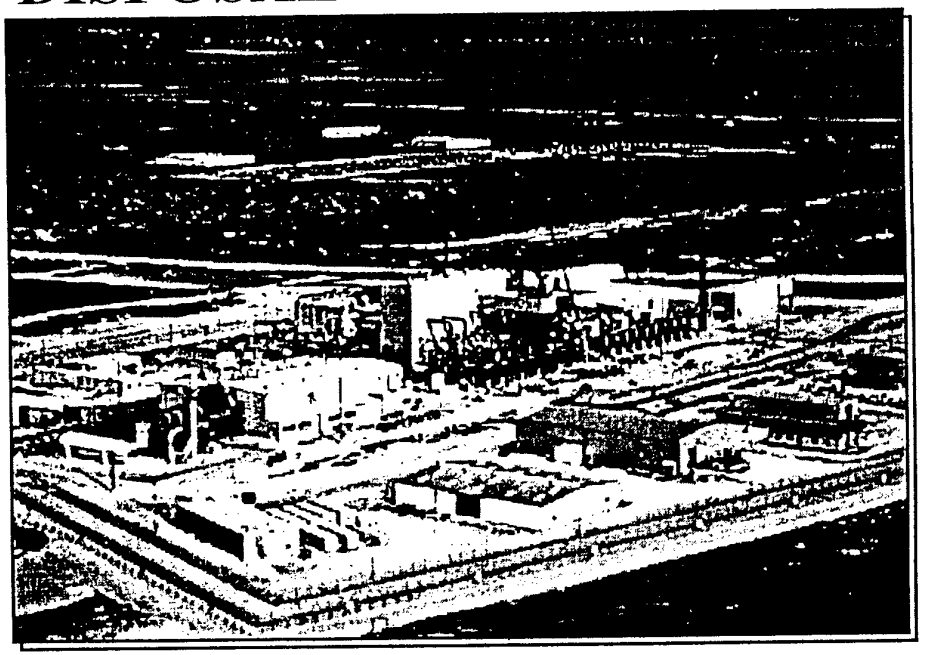


Stockpile Destruction Facilities (currently in operation)



JOHNSTON ATOLL CHEMICAL AGENT DISPOSAL SYSTEM

TOOELE CHEMICAL AGENT DISPOSAL FACILITY



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Accomplishments
Johnston Island

- ✓ **Operational Since 1990**
- ✓ **The First Full-Scale Chemical Munitions Incineration Facility**
- ✓ **Extensive Shake Out Effort has been Required**
- ✓ **Successfully Destroyed 1,462 Tons of Agent (72% of JI Stockpile)**
  - All M55 Rockets
  - All MC-1 Bombs (Air Force)
  - All MK-94 Bombs (Navy)
- ✓ **Over 1.5 Million Pounds of Explosives Destroyed**
- ✓ **Currently destroying GB 8-inch Projectiles**
- ✓ **Contractor: Raytheon Engineers & Constructors**

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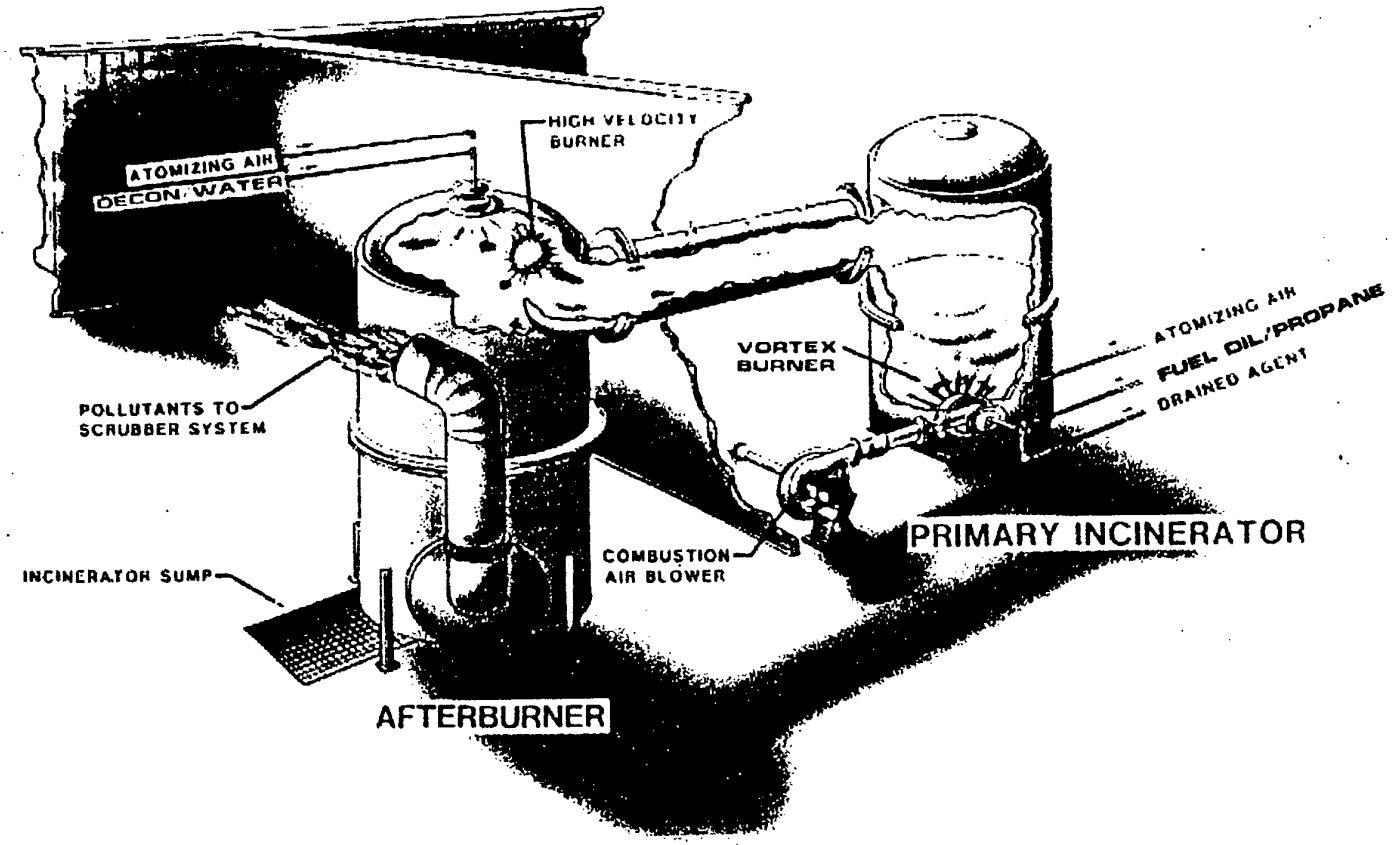
Accomplishments
Tooele

- ✓ Facility Construction Certification Completed July 1993
- ✓ Systemization Status - Completed All Systems Acceptance Tests
- ✓ Emergency Preparedness
- ✓ Began Destruction of U.S. Stockpile - August 1996
- ✓ Successfully Destroyed 1,340 Tons of Agent (Approx. 9.8% of Tooele Stockpile)
- ✓ Contractor: EG&G

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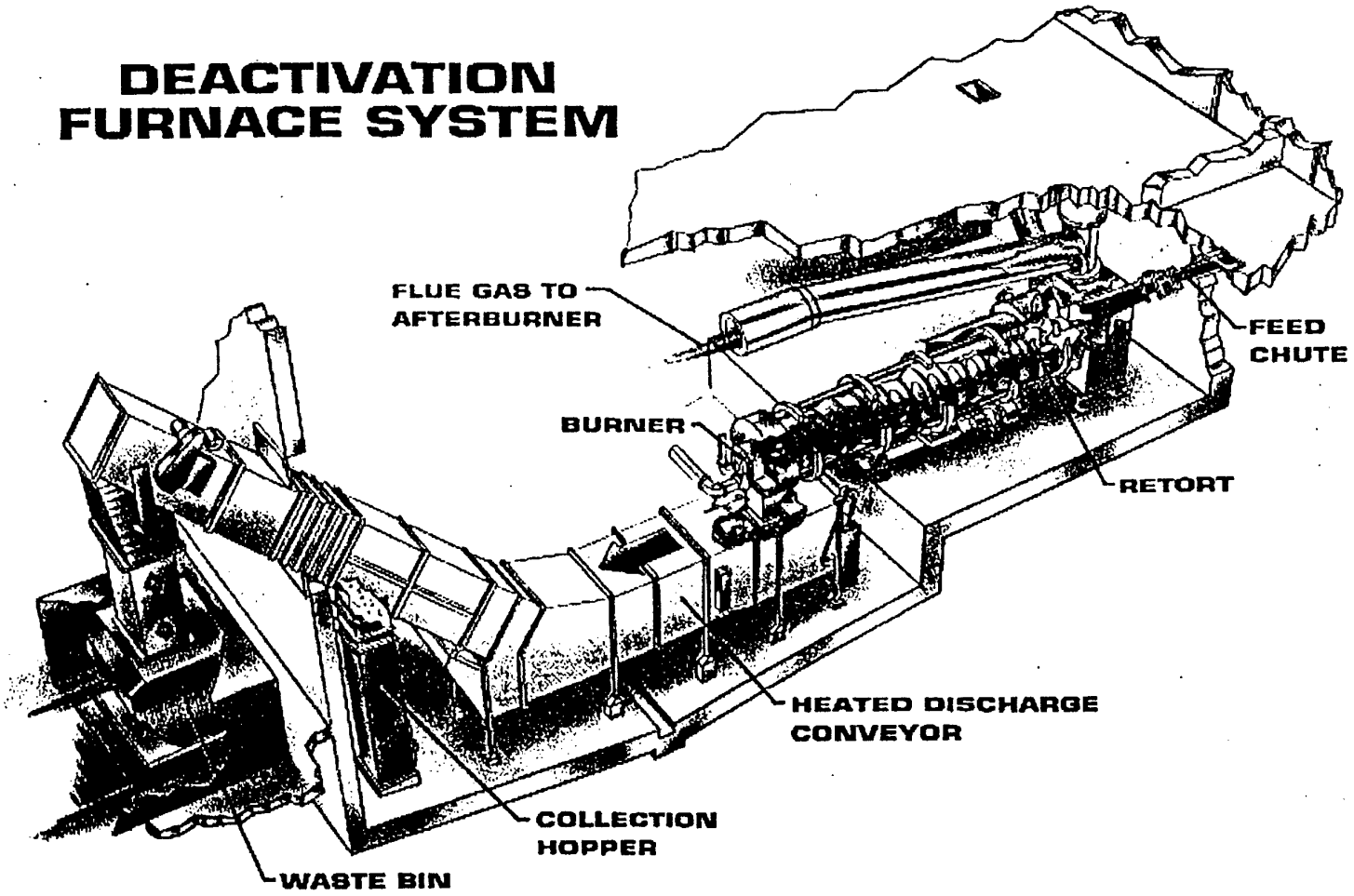
Baseline Incineration Process

LIQUID INCINERATOR



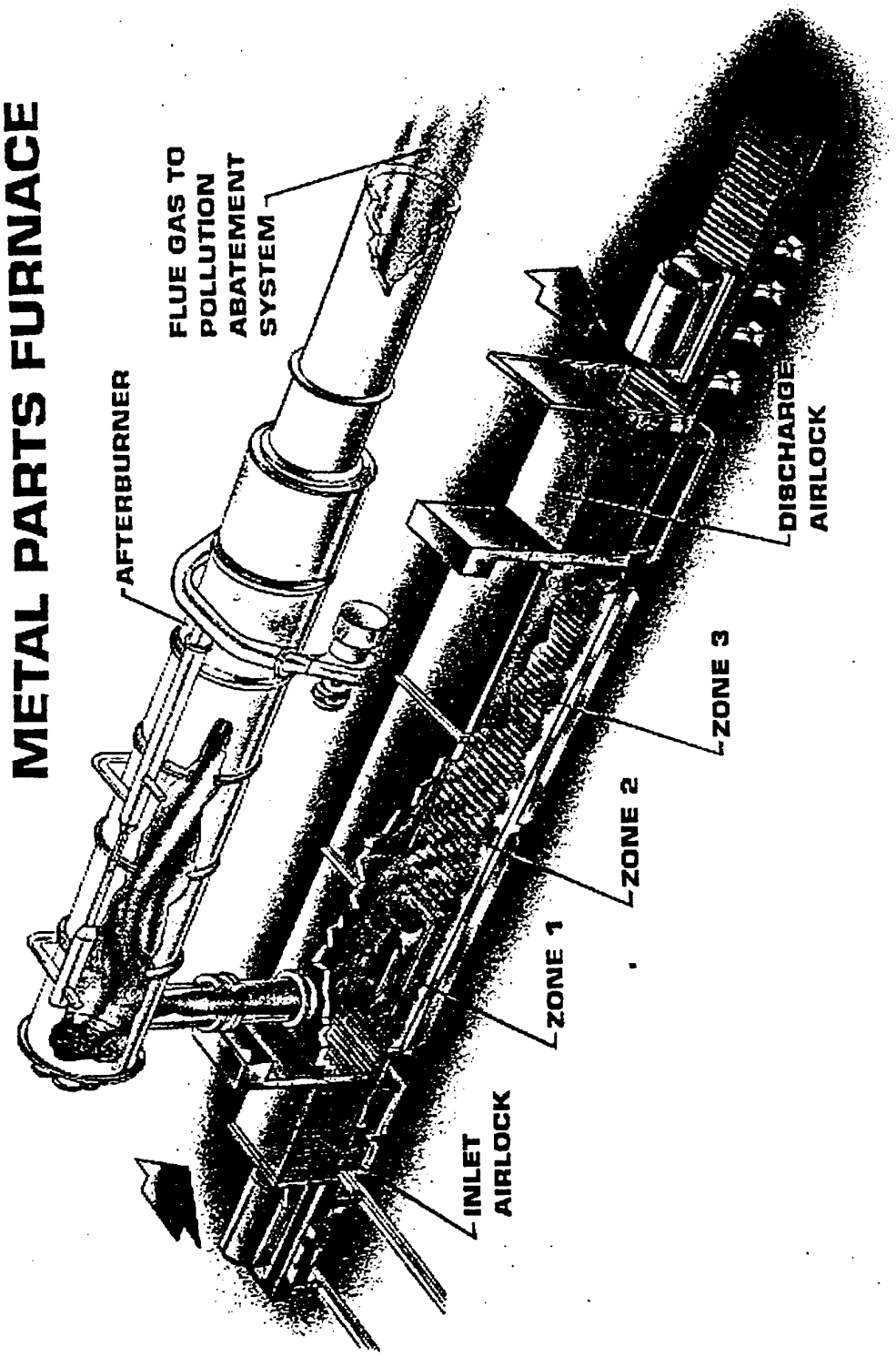
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Baseline Incineration Process (cont'd)

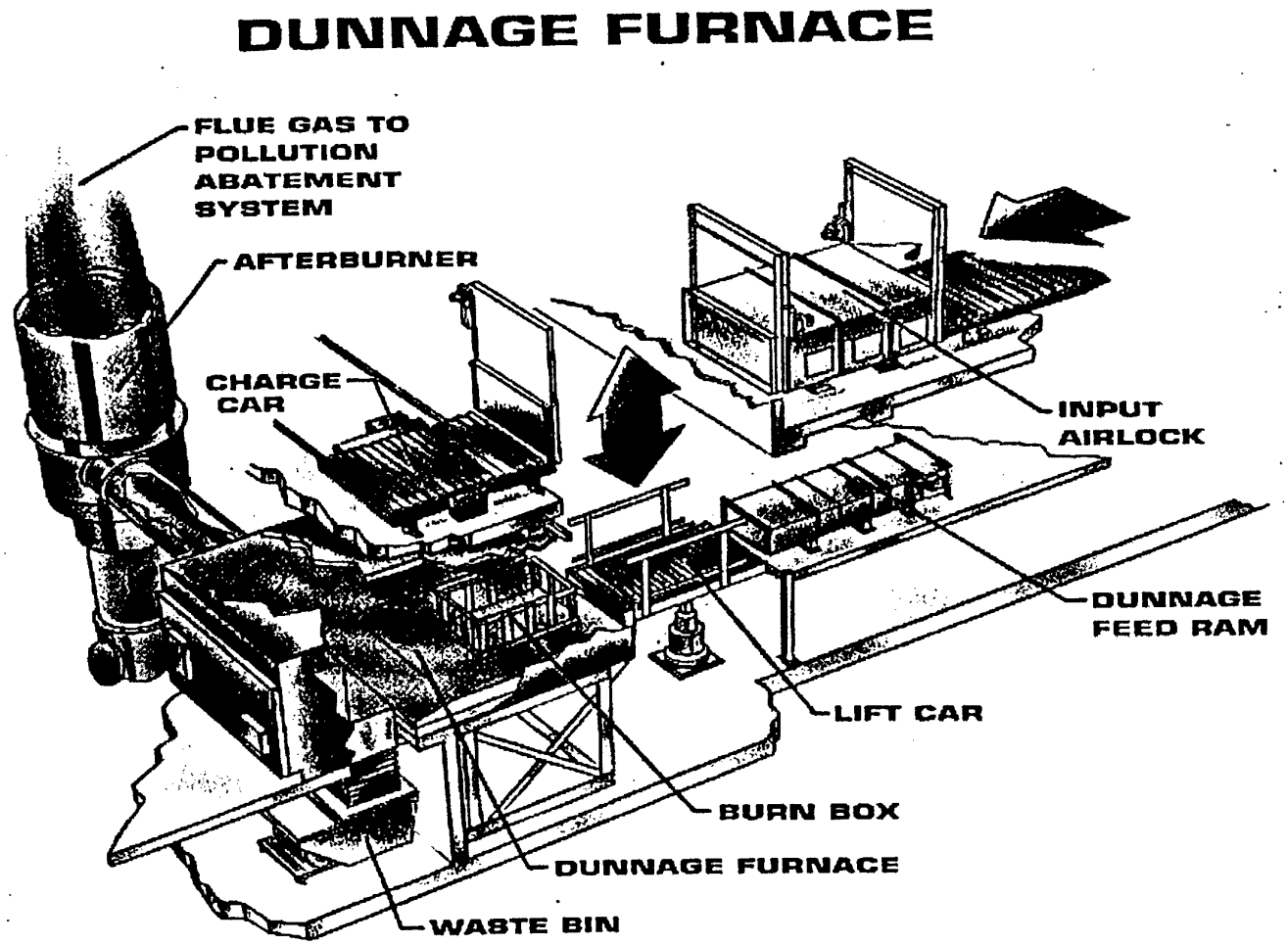


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Baseline Incineration Process (cont'd)



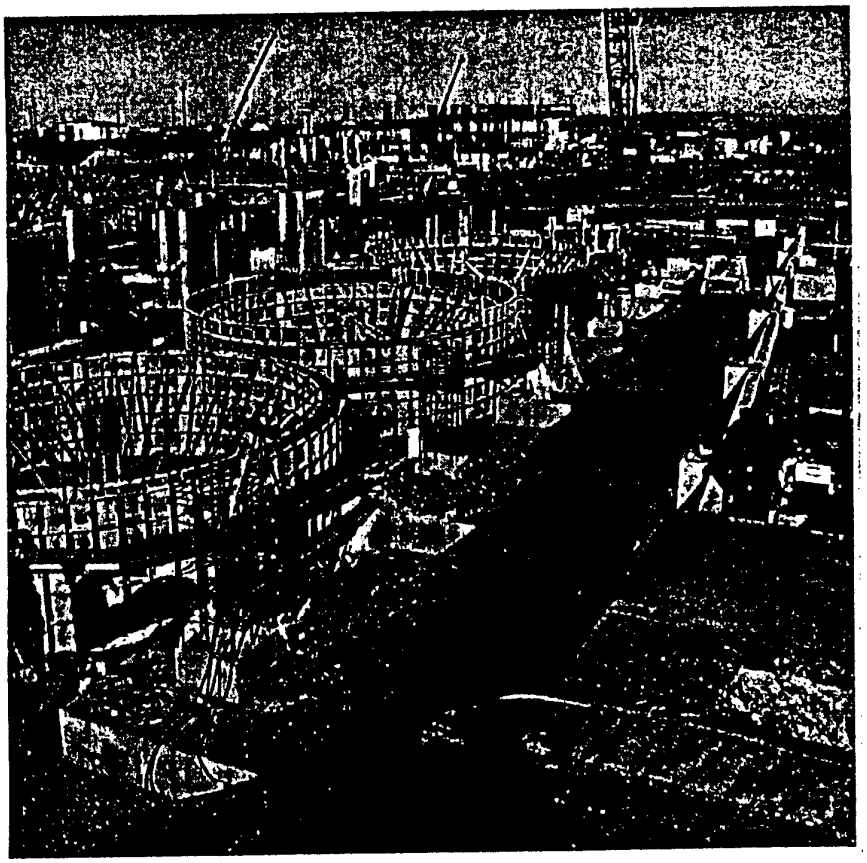
Baseline Incineration Process (cont'd)



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Stockpile Facilities Under Construction

UMATILLA CHEMICAL AGENT DISPOSAL FACILITY



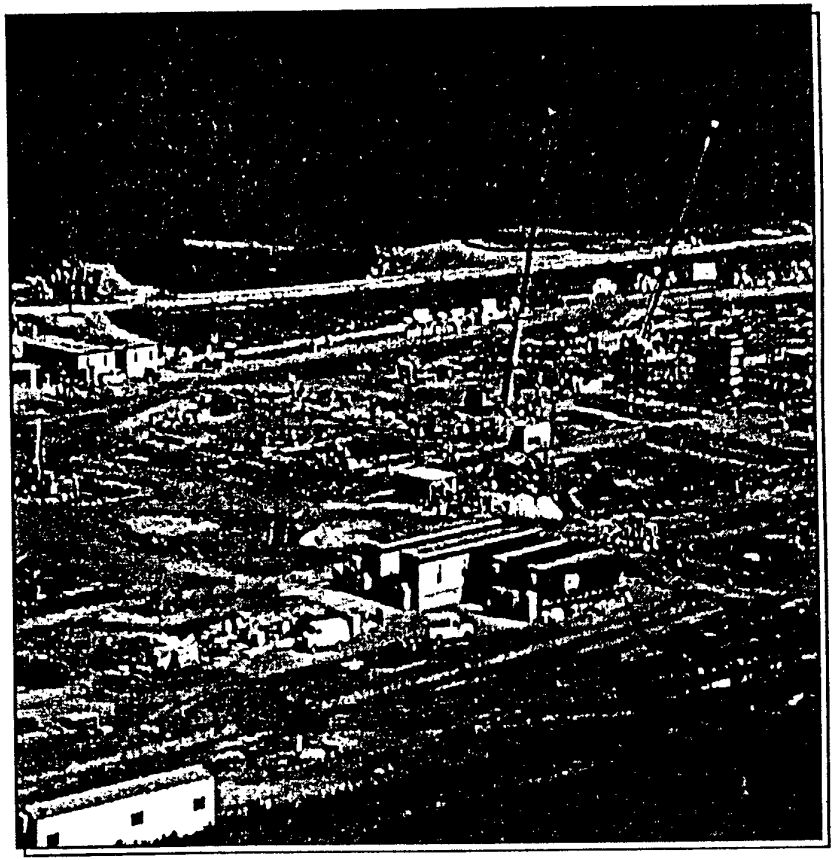
- **Construction began in July 1997.**
- **As of March 1998, approximately 13% of the facility has been constructed.**
- **Destruction operations will begin November 2001.**
- **Contractor: Raytheon**

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Stockpile Facilities Under Construction

ANNISTON CHEMICAL AGENT DISPOSAL FACILITY

- **Construction began in February 1996.**
- **As of January 1998, 14% of the facility had been constructed.**
- **Destruction operations will begin January 2002.**
- **Contractor: Westinghouse**



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Alternatives to Baseline Incineration

- ***Bulk CW agent*: PMCD to manage (Alt Tech I)**
- **Neutralization**
  - Aberdeen Proving Ground, MD
  - Newport Chemical Activity, IN
- ***Assembled CW munitions*: PM for Assembled Chemical Munitions (Alt Tech II)**
- **Minimum of two technologies to be demonstrated**
  - Blue Grass Depot, KY
  - Pueblo Depot, CO
- **Final results expected December 1998**
- **Congressional hold on construction**

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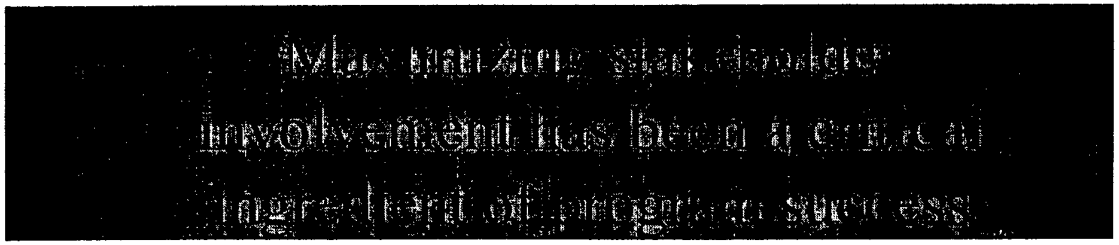
ACWA Proposals

OFFEROR	AGENT/ENERGETIC TREATMENT
AEA Technology	Electrochemical oxidation using silver ions in nitric acid at 90 C (Silver II)
ARCTECH	Hydroloysis with a-HAX (humic acid and strong base, KOH) at 90°C
Burns and Roe	Plasma arc in inert argon
General Atomics	Hydrolysis with caustic, supercritical water oxidation (SCWO)
Lockheed Martin	Hydroloysis with caustic at 90°C, SCWO, Eco Logic gas phase chemical reduction (GPCR)
Parsons and Allied Signal	Hydrolysis with caustic at 90° C followed by biotreatment
Teledyne Commodore	Solvated electron treatment (SET) at -30°C

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Accomplishments
Public Outreach

- Citizen's Advisory Committees at every site
- Public Education
  - town meetings
  - poster sessions
  - visits to schools
- ACWA Dialogue



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Non-Stockpile Chemical Materiel Program

- Disposal of all chemical warfare materiel (CWM) not included in the unitary chemical stockpile
  - binary chemical weapons
  - former chemical weapon production facilities
  - miscellaneous CWM
  - recovered CWM
  - buried chemical weapons
    - suspected burial sites at 63 locations
    - technology solutions:
      - Rapid Response System
      - Munitions Management Device (2 versions)
      - Emergency Destruction System

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On Schedule and Moving Forward

* **Maximum protection applies to all aspects of safety, health and the environment**

* **The program is on track to meet the 2007 CWC destruction deadline**

* **Maximizing stakeholder involvement has been a key proven success factor for the program**

Proceedings of the International CW Destruction Symposium
Munster, Germany, March 22-25, 1998

AN OVERVIEW ON THE NATIONAL IMPLEMENTATION OF THE CHEMICAL WEAPONS CONVENTION

Ronald Sutherland (Canada), Thomas Kurzidem (Germany) and Thomas Stock (Germany)

I. Introduction

The Chemical Weapons Convention (CWC) has entered into force 29 April 1997. As of February 1998 107 countries have ratified the treaty, among them the USA and since November last year, Russia too.

There is no doubt, the participation by the Russian Federation and the United States in the CWC is more than an essential step for it's primary goals: the total elimination of chemical weapons. Looking back into the history of the negotiations on the CWC there is clear evidence that both countries played an important role and took a special responsibility.

This treaty does not only focus on the one disarmament goal: the destruction of CW. The treaty will establish a regime which allows the verification of compliance, and this in the context of universality. This compliance verification is related to many different activities, including the non-production of certain chemicals in industry, a regime which will cope with old and abandoned CW etc.

The CWC was opened for signature in Paris in January 1993 with entry into force (EIF) being contingent on 65 ratifications. The formal EIF taking place 180 days later after the sixty fifth is received. This process was anticipated to take two years, but at the final end it was more than 4 years.

The obvious question is why has the ratification process taken so long relative to the expectations of the Geneva negotiators? The answer lies mostly in the fact that the complex process of developing legislation and a National Authority together with the poorly understood process of collecting the required data from the civilian chemical industry and National Departments of Defence as well as its transmission to a novel international authority was underestimated. In most developed countries the chemical industry (production, processing and consumption of chemicals) is heavily regulated by many acts and government departments (health, safety, environment, imports, exports etc.) and development of the Legislative Act and regulations necessary to national implementation of the CWC as required by Article VI turned out to be a slow process involving input from many government departments and the industries involved.[1]

This paper attempts to analyze the tasks which have to be undertaken by a State Party to the CWC in the process of national implementation and the first experience available.

II. The Tasks of a National Authority under the CWC

Each State Party to the Chemical Weapons Convention has agreed to a definite set of fundamental obligations under that Convention. A State Party accepts the basic provisions of the Convention in Article I[2] and will implement the obligations by taking the necessary domestic legal and administrative measures required by Article VII (National Implementation Measures). It will provide the declarations and information required under Articles III (General Declarations), IV (Declarations on Chemical Weapons), V (Declarations on Chemical Weapons Production Facilities) and VI (Activities not prohibited under the Convention) and this information will be subjected to the verification provisions of these articles using the machinery of Article VIII (The Organization). The State Party also agrees to accept challenge inspection under Article IX if the need arises. In order to do this, each State Party must:

- appoint a National Authority;
- produce the required Declarations;
- communicate and co-operate with the Organisation for the Prohibition of Chemical Weapons (OPCW);
- assist international inspection of its facilities;
- co-operate with other States Parties;
- treat all communications with the OPCW and other States Parties as confidential; and
- enact penal legislation to prevent any activity prohibited to a State Party.

[1] For a detailed overview see Effective Implementation of the Chemical Weapons Convention, edited by T. Kurzidem, Y. von Lersner, P. Radler, T. Stock and R.G. Sutherland, 272 pages, Bad Homburg, Germany 1995.

[2] This article oulines among others the following: Every State Party should „... never under any circumstances: ....to develop, produce, otherwise acquire, stockpile or retain chemical weapons, or transfer, directly or indirectly, chemical weapons to anyone; ...to use chemical weapons; ...to engage in any military preparations to use chemical weapons;...to assist, encourage or induce, in any way, anyone to engage in any activity prohibited to a State Party under this Convention"; in addition every State Party is obliged to destroy existing CW stockpiles and CW production facilities.

National implementation regimes will differ from State Party to State Party according to the impact of the CWC on its activities and its chemical industry.
The nature of its National Authority will be a function of:

- possession or non-possession of chemical weapons;
- possession or non-possession of chemical weapons production facilities (CWPF);
- the potential for a State Party's chemical industry to produce chemical weapons;
- the nature of a State Party's chemical industry; and
- the existing and size of old or abandoned CW stockpiles.

If we examine the responsibilities of a National Authority in some detail, it will have some or all of the following tasks in its interactions with the OPCW:

- initial declarations under Articles III, IV, V and VI;
- annual declarations under Articles IV, V and VI;
- escorting OPCW inspections under Articles IV, V, VI, IX and X;
- accrediting OPCW Inspectors;
- developing Facility Agreements;
- overseeing closure and destruction activities relating to CW and CWPF;
- co-ordinating the provision of national assistance under Article X;
- reviewing national regulations in international trade in chemicals; and
- old and abandoned chemical weapons.

In addition to these tasks, the National Authority will have to be involved in international activities relating to representation to the Conference of States Parties, the Executive Council and other activities as required. In general terms a State Party's responsibilities which will likely be delegated to its National Authority will have two basic elements: an *internal* one that involves providing information of regulatory nature to an international body, and an *international* one where it provides technical support to its delegation to the OPCW.

III. Demonstration of Compliance with Article VI of the CWC

It is the task of each State Party to demonstrate its compliance through the provision of information to the OPCW via its National Authority. The major initial burden then falls on the National Authority to collect the data and transmit it to the OPCW. This process has to be organized in such a way that either state security will be not affected or losses of confidential business information (CBI) will not occur. It goes without saying, the international verification process, conducted by the OPCW, will fail if the national process of data collection and transmission is not previously in place.

The CWC deals with the destruction of existing weapons (disarmament) and the control of potential production activities which might be of relevance for CW (arms control or non-proliferation aspects).

This extends the reach of the CWC into civilian owned chemical facilities, world-wide and requires State Parties to collect data from industry in order to develop their declarations to the OPCW. For example, this means that the National Authority has to be able to deliver declarations 30 days after entry into force not only of government activities so far as chemical weapons are concerned, but also for all industrial facilities covered by Article VI. Thus, each State Party to the CWC will have to do the following:

- identify all companies affected by the CWC including producers, processors, consumers, importers and exporters;
- ensure that its chemical and related industries are aware of their obligations both to the National Authority and the OPCW;
- develop procedures to assist industries that may be subjected to routine inspection by the OPCW; and
- meet with trade associations and companies affected by the OPCW.

It should be noted that the constraints of Article VI fall on the industrial sector that produces or uses chemicals that appear on the schedules of the CWC. These are measures designed to show that chemicals of concern to the CWC are not misused or diverted to purposes prohibited by the CWC. The chemicals on schedules 2 and 3 have many legitimate uses in the manufacture of pesticides, pharmaceuticals, ink and the like and this is why the CWC opts for the routine monitoring of 'dual use' industrial chemicals, their production and processing facilities. Verification will be difficult since:

(*1*) many of the scheduled chemicals have legitimate commercial uses, and
(*2*) many of the known chemical weapons could be made in commercial chemical facilities.

Industry and Declarations under Article VI of the CWC

After the appropriate implementing legislation in an individual State Party, declarations (initial and annual data reporting) will be mandatory for companies that produce, import or export chemicals listed in schedules 1, 2 and 3 in quantities above the thresholds specified in the CWC. This also applies to companies that process or consume quantities of schedule 2 chemicals above the thresholds stated. Besides the producers of industrial chemicals, the following industry branches could be involved: pharmaceuticals, mining, paper, textiles, plastics, printing inks, semi-conductors, electronics, etc. Also, companies that produce discrete organic chemicals in quantities greater than 200 metric tonnes must declare these facilities or, if their products contain phosphorus, sulphur or fluorine, over 30 metric tonnes. Facilities that produce pure hydrocarbons or explosives are excluded. This information can only be obtained from industry in the first instance. The CWC provides specific definitions for industrial activities in Article II: of particular importance for industry are those for production, processing, consumption, facility, unit, plant, plant-site and facility agreement.

As soon as practical, the National Authority has to:

- ascertain national trade in schedules 2, 3, and 'other discrete organic chemicals';
- ascertain imports and exports in Schedule 2 and Schedule 3 chemicals;
- ascertain the number of plant sites and plants that will be liable to inspection by the OPCW.

In summary the National Authority will be responsible for three different types of data reporting:
(*a*) data concerning the first declaration after entry into force of the Convention for the State Party;
(*b*) annual data reporting; and
(*c*) reporting of changes on annually submitted data.

The data should be retrievable, authorised and assessable on the National Authority level. Examination of the timetable for the declarations makes it clear that the National Authority must ensure that declarations from industry will need to be obtained far in advance of the CWC deadline so that aggregate national data can be developed and possible errors checked and corrected.

Industry and Inspections under Article VI of the CWC

Once a State Party provides its declarations to the OPCW, these will be verified by on-site inspections (routine and challenge inspections) by the Technical Secretariat. The CWC provides for various levels of access but industry has rights as to the extent of the inspection process. A major task of the National Authority will be to assist the facility operators in this regard. Facilities that produce

Schedule 1 chemicals can be inspected at anytime, and there is no limit on the actual number of inspections per annum. Individual Schedule 2 or 3 plants can receive a maximum of two inspections per year. Plants that produce 'other discrete organic chemicals' will not be inspected during the first three years, but the total annual number of inspections for Schedule 3 and these other facilities is 20 or 3 plus 5% of the total number of the plant sites declared whichever is smaller. There is also a time limit as to how long inspectors from the OPCW can spend at an individual site unless the National Authority agrees to an extension. The time limit is 96 hours for schedule 2 and 24 hours for Schedule 3 or other relevant facilities.

Arrangements must be made for practical matters such as the arrival of an inspection team at the point of entry of the inspected State Party (e.g., travel arrangements and the provision of escort service). This will be the responsibility of the National Authority.

Schedule 2 chemicals/facilities
During the first three years after entry into force, all commercial facilities that produce, process or consume Schedule 2 chemicals above the threshold should undergo an initial inspection during which negotiations will commence on a Facility Agreement. This will be negotiated between representatives of the OPCW and the National Authority with assistance from the plant management. The document should be agreed within 90 days or further inspections will involve 'managed access'. The Facility Agreement is for the plant site which contains the plant. The document specifies which parts of the site apart from the plant are of concern to the Inspectorate and thus, may form part of the routine inspection pathway. It limits the activities of the OPCW inspectors at the plant and plant site as well as the obligations of the management to provide information and samples to the OPCW team through the National Authority.

Schedule 3 chemicals /facilities
Schedule 3 facilities are also subject to routine inspections after entry into force, but there is an initial priority for Schedule 2. There is no requirement for a formal initial inspection nor for a Facility Agreement, although one can be requested by the facility management.
Other chemical production facilities
The inspection regime for 'other chemical production facilities' will not begin until four years after entry into force and then only after a decision by the Conference of States Parties.

Challenge inspections
There is also a provision for short notice inspections at any site, declared or undeclared, and at government or privately owned facilities. These are challenge inspections under Article IX. There has to be suspicion that activities prohibited by the CWC are being undertaken at the site, and such suspicion has to be documented by the challenging State. The National Authority has the major responsibility to ensure that the inspection results are unambiguous and that the challenged State is in compliance with its CWC obligations. There are complex procedures to balance necessary intrusiveness with legitimate concerns over either national security or trade secrets (CBI). These inspections should be rare at commercial locations.

Additional tasks for the National Authority

Requirements under Article IX
Additionally are the requirements under Article IX—Consultations, Co-operation and Fact-finding—which are vital to the proper functioning of the CWC. Article IX calls upon each State Party to 'consult and co-operate, directly among themselves, or through the Organisation or other appropriate international procedures . . . on any matter which may be raised relating to the object and purpose, or the implementation of the provisions, of this Convention' and obliges each State Party to 'whenever possible, first make every effort to clarify and resolve . . . any matter which may cause doubt about compliance with this Convention, or which gives rise to concerns about a related matter which may be considered ambiguous'. In this respect the National Authority will play an important role if requests are made concerning specific matters of compliance. Quick response will be required and the National Authority will have to be able to provide the OPCW with supplementary data and information to resolve such ambiguities.

Requirements under Article X
Article X concerns assistance and protection against CW. The CWC defines assistance as: 'the coordination and delivery to States Parties of protection against chemical weapons'. Three types of assistance are defined as regards:
(*a*) use of CW;
(*b*) threat of CW use; and
(*c*) the results of an on-site investigation after an alleged use of CW.

The different types of assistance envisaged are: emergency, supplementary and humanitarian assistance. Means of assistance are defined *inter alia* as detection equipment, alarm systems, protective equipment, medical antidotes and treatments, decontamination equipment and decontaminants and advice on protective measures.

Taking the context of Article X into account, a State Party: (*a*) has to provide assistance to the OPCW; (*b*) may provide assistance to other States Parties through the OPCW; and (*c*) can provide directly assistance to another State Party.

In preparing for national implementation, States Parties have to consider how they can fulfil their obligations related to assistance under Article X. Article X calls for two different types of contributions and information: (*a*) mandatory, and (*b*) voluntary. A State Party with national programmes related to protective purposes is obliged to provide annual information on such programmes. Greater openness and more transparency will serve to increase confidence among States Parties in the early days of the CWC.

Requirements for old and abandoned CW
Within 30 days after entry into force of the CWC, every State Party with old or abandoned CW on its territory has to provide data on these weapons to the Technical Secretariat. If a State Party has abandoned CW on the territory of another party it also has to submit information. This information shall include to the extent possible information on the location, type, quantity and the current condition of the CW and will be verified by the Technical Secretariat through initial inspection later. The purpose of this inspection is to: (*a*) verify the information submitted, (*b*) determine whether the CW meet the definition of old CW, (*c*) verify the origin of the abandoned CW, and (*d*) establish evidence concerning the abandonment and identity of the abandoning state.

After the initial inspection the 'usability' of the old or abandoned CW has to be determined in accordance with the definition of old CW under Article II, paragraph 5. If the CW meets the definition under Article II, paragraph 5 (b) (CW produced between 1925 and 1946 and that have deteriorated to such extent that they can no longer be used as CW), destruction is required in accordance with Article IV and Part IV (A) of the Verification Annex.

If abandoned CW are present, the territorial state has to consult with the abandoning state with the goal of establishing a mutually agreed plan for destruction, which has to be approved by the Technical Secretariat. If the abandoning state cannot be identified or is not a party to the CWC, the territorial state may request assistance from the OPCW or another State Party in destruction.

The National Authority of a State Party with old or abandoned CW will be involved in or responsible for:
(*a*) preparing the initial declarations;
(*b*) preparing the initial inspection and escorting the inspectors to the site where old or

abandoned CW are located;
(*c*) destroying or disposing of the declared old CW, if they meet the definition of toxic wastes (as specified under Article II, paragraph 5 (a));
(*d*) destroying old CW, if they meet they definition under Article II, paragraph 5 (b), based upon an approved destruction plan which includes systematic verification by the OPCW inspectors (prior to Verification Annex, Part IV (A), paragraphs 41 to 43);
(*e*) negotiating the plan for destruction with the abandoning state in the event of abandoned CW; and
(*f*) requesting assistance from the OPCW or other States Parties, if the abandoning state cannot be identified.

IV. Models for National Authorities

The National Authority is characterized under the CWC in Article VII as the liaison point between the states and the OPCW and with other states on CWC matters. The national decision is to what extent the National Authority should fulfil the CWC national activities (declarations, preparation for inspections etc.) or to what extent other government agencies should be involved. The main elements of CWC related functions are *(1)* foreign affairs *(2)* defence concerns *(3)* economic or industrial concerns.

Theoretically two approaches are common in setting up the National Authority. If a State Party decides after reviewing its chemical industry and any past CW history that the impact of the implementation requirements is very limited, a structure for the National Authority may be chosen which is based upon the appointment of a branch or only one official in an already existing department. Such a nominated department could be the Department of Foreign Affairs, the Department of Chemical Industry or the Department of the Environment.

If, on the other hand, a State Party has evidence that implementation will have a major impact and place a heavy burden on its chemical industry and that many activities will be related to Articles III (Declarations), IV (Chemical Weapons) and V (Chemical Weapons Production Facilities (CWPFs)), the nomination of an existing organisation or the establishment of a new organ acting as the National Authority seems necessary.

Thus, from a very pragmatic point of view two models for the National Authority have won general acceptance: (*1*) the designation of an existing agency, or (*2*) the establishment of a new agency.

In the light of the responsibilities of a National Authority two options are pertinent: (*a*) a centralised National Authority with responsibility for all CWC-related issues, or (*b*) a decentralised approach, a National Authority which serves only as a liaison point[3] and delegates the real functional aspects to already existing agencies, offices or ministerial bodies.

In analyzing the available experience from the past few years on national implementation the preferred location of National Authorities is either in Departments of Foreign Affairs or Economic Affairs with no examples involving Departments of Defence directly. The National Authority is rarely a stand-alone agency but usually a unit within an existing agency. It is still uncertain as to how well they will be able to discharge their functions since the level of required resources is still uncertain since the full extent of the workload will largely remain undermined until EIF.

V. National Implementation Legislation

The implementation of the CWC requires enacting special legislation at the State Party's level. Article VII of the CWC outlines:

1. Each State Party shall, in accordance with its constitutional processes, adopt the necessary measures to implement its obligations under this Convention. In particular, it shall:

(a) Prohibit natural and legal persons anywhere on its territory or in any other place under its jurisdiction as recognized by international law from undertaking any activity prohibited to a State Party under this Convention, including enacting penal legislation with respect to such activity;

(b) Not permit in any place under its control any activity prohibited to a State Party under this Convention; and

(c) Extend its penal legislation enacted under subparagraph (a) to any activity prohibited to a State Party under this Convention undertaken anywhere by natural persons, possessing its nationality, in conformity with international law.

In reviewing the approaches how countries have set up their national implementation legislation it is quite obvious that the individual countries have chosen their country specific approach to national implementation.

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[3] Tanzman, E., Zeuli, A. R. and Kellman, B., 'Legal aspects of National Implementation of the Chemical Weapons Convention', paper presented to the Regional Seminar on the National Implementation of the Chemical Weapons Convention, Jakarta, Indonesia, 28-30 Nov. 1994.

This is due to several factors:

(a) how international law has to be incorporated into national law;
(b) the size of chemical industry;
(c) the already existing regulatory framework with respect to industry, especially chemical industry, with respect to data reporting, environmental regulations, trade regulations and work, safety and health regulations;
(d) the magnitude of trade with chemicals;
(e) the organizational set-up of industry, such as Chemical Manufacturers Associations;
(f) the size of the problem related to old chemical weapons or abandoned chemical weapons;
(h) the existing or not existing of activities under Schedule 1, including single small-scale facility; and
(i) the experience the respective Ministry of Foreign Affairs might have with already existing agreements with respect to reporting of declarable data.

Different strategies can be identified. Setting up a new legislation act or amending already existing acts seems to be the two general possibilities. However, it can be identified, based upon the situation that most countries do not possess CW, that activities related to the destruction of CW or CWPFs are not of concern in the implementation legislation.

VI. Experience with national implementation

Based upon the publicly available information from countries already undertaken the national implementation of CWC it is possible to identify certain key measures which seem to be essential for effective implementation. The following is a short analysis of 3 different countries (Sweden, Germany and Canada) with respect to their national implementation undertakings. These countries have ratified the CWC during the last 4 years, but have chosen different approaches (Sweden had ratified in 1993, Germany in 1994 and Canada in 1995). In addition, these countries have different magnitude in chemical industry and an involvement or not involvement in the issue of old CW.

Canada

The Canadian Legislation[4] passed through all the relevant processes and received Royal assent in July 1995 with formal ratification taking place in September 1995. The development of the legislation was in the hands of the Department of Foreign Affairs and International Trade (DFAIT) and involved many government departments as well as several divisions within DFAIT. The Act's purpose was to implement Canada's obligations under the CWC. It gives the Minister of Foreign Affairs the authority to implement the Act and designate a National Authority (in DFAIT) to execute the Minister's responsibilities under that Act. It contains provisions for obtaining the required information and protecting its confidentiality. It develops provisions for the conduct of international inspections in Canada. It extends existing controls on chemicals by specifying that all CWC chemicals be subject to the authority of the Export and Imports Permit Act. It establishes penal provisions while making clear that domestic enforcement of the Act will be conducted under the Criminal Code. The legislation also provides for the formulation of the appropriate regulations concerning the collection of required information and procedures under which the National Authority will carry out its assigned responsibilities.

The overall object of the legislation was to achieve maximum effectiveness with minimum interference in legitimate activities of Canadian industry. Industry was closely consulted through its various trade associations throughout the formal phase of the CWC negotiations and preparation for implementation in Canada.

It can be seen from the timelines that it took over two years to develop the implementing legislation after signing the CWC and a great deal of creativity to harmonize the legislation with existing laws since so many other department's activities impacted on the requirements of the CWC. The National Authority was set up in DFAIT in late 1993. It is staffed by 3 professional members and two support staff.

Its overall mandate can be summarized as follows:

International

- communicate and co-operate with the OPCW;
- communicate and co-operate with other States Parties;
- prepare and escort all international inspection;
- arrange provision of direct/indirect assistance to States Parties threatened by CW;
- monitor compliance by other States Parties; and
- provide advice/support to Canadian representatives at the OPCW.

National

- regulation and operating procedures (Bill C87);

[4] Bill C87 An Act to implement the Convention on the Prohibition of the Development, Production, Stockpiling and Use of Chemical Weapons and on their Destruction, First reading 1 May 1995 and royal assent 13 July 1995.

- define clientele (private and public sectors) and assist them with CWC responsibilities;
- Database creation and management to ensure declaration/responses to international monitoring;
- industrial outreach;
- single-small-scale facility (SSSF);
- disposal of old and abandoned CW; and
- co-ordination with other government departments and the Provinces.

As part of these activities[5], it has developed an Industry Advisory Group (IAG) to assist it in reaching the chemical industry and in developing regulations.

Germany

Legislation

Germany signed the CWC during the Paris Conference on 13 January 1993 and determined to ratify the Convention at the earliest possible date. In Germany the ratification of an international treaty requires the prior approval by Parliament and the Federal Chamber, the representative organ of the states. Since most of the regulations of the CWC are not self executing, additional implementing legislation is required to implement the obligations of the Convention. Owing to the very short time lines (i.e., for submitting declarations to the OPCW after entry into force of the Convention), the Federal Government decided to submit both the Chemical Weapons Convention Act,[6] which empowers the Federal Government to ratify the CWC, as well as the respective Act for Implementing the CWC[7] to the legislative organs at the same time. The first Act passed both Parliament and the Federal Chamber in 1994 without any problems, whereas the Implementation Act was criticized during discussions in the Federal Chamber. Especially some of the East German *Länder* (states) raised the question of whether the CWC would have financial implications at the state level. The respective formulation in the reasoning to the Draft Act was found to be inadequate, and the Draft Act was referred to the Mediation Committee. In the Committee, the Federal Government and the *Länder* agreed on a formulation that the Federal Government will be responsible for all costs following ratification and implementation of the CWC. Following the agreement, the Act was approved by the Federal Chamber in early July 1994. The Implementation Act entered into force on 10 August 1994, and Germany ratified the CWC on 12 August 1994.[8] Owing to the fact that the CWC had not yet entered into force in that time, not all the regulations of the Implementation Act were in force. The basic provisions related to prohibited activities, powers and functions of the National Authority as well as those relating to declarations and some of the penal sanctions entered into force on 10 August 1994. All other provisions, especially those related to inspections, have entered into force with the date of entry into force of the CWC, 29 April 1997.[9]

The structure of the German National Authority

The National Authority in Germany consists of three elements: *(a)* the Head of the National Authority, assigned to the Ministry of Foreign Affairs; *(b)* a 'military' component under the responsibility of the Minister of Defence; and *(c)* a 'civilian' component under the responsibility of the Minister of Economics.

A special section within the Ministry of Foreign Affairs is acting as the supervising authority. In addition, it serves as the national focal point for communication and co-operation between the National Authority and the OPCW and does represent the National Authority externally. Declarations are to be submitted by the Head of the National Authority to the OPCW based on the information submitted by the competent national agency. The Ministry of Foreign Affairs is also responsible for transfer of the information it receives from the OPCW to the subsidiary agencies (e.g., the request for an inspection).

The military component will be the responsibility of the Ministry of Defence. The tasks with regard to military installations will be assigned to the Verification Centre of the *Bundeswehr* in Geilenkirchen which has already acquired experience in the verification of other arms control treaties (e.g., the CFE Treaty). However, the activities of the Verification Centre are restricted to the escorting of international inspectors at military facilities only if not provided otherwise by law.

The major responsibility for effectively implementing the CWC at the national level, especially all activities related to Article VI and the relevant inspection activities, is assigned to the Export Control Office *(Bundesausfuhramt)* at Eschborn, a subsidiary agency of the Ministry of Economics within that agency. The National Authority has been established as an individual section. The Export Control Office is primary responsible for collection, processing and evaluation of the data required by the CWC; it further has the right to

[5] The Chemical Weapons Convention: How will it affect Canadian Industry, R.G. Sutherland and J.P. Tracey, Canadian Chemical News, 17 May 1995

[6] Gesetz zum Chemiewaffenabkommen, Bundesgesetzblatt (Federal Gazette) II 1994, p. 806.

[7] Ausführungsgesetz zum Chemiewaffenabkommen, Bundesgesetzblatt (Federal Gazete) I 1994, p. 1954.

[8] See "Ratifikationsurkunde bei UN in New York hinterlegt', *Frankfurter Rundschau*, 15 August 1994, p.4.

[9] Article 21.

request any information from a facility. In addition, national inspectors may request access to business documentation or to a facility site during normal working hours. The Federal Government has enacted detailed regulations which regulate the frequency, periods to be reported upon, contents and form of data reporting as well as the means and time limits for the submission of such information.

The Export Control Office negotiates Facility Agreements with the OPCW and is entitled to conclude, such arrangements with the OPCW in co-ordination with the ministries of Foreign Affairs and Economics.

Inspectors of the OPCW conducting an inspection in a private facility will as a rule be escorted by a team of national inspectors from the Export Control Office. The leader of the escort team is responsible for the conduct of an inspection and will give instructions as necessary to ensure that the inspection is conducted properly

Sweden[10]

Legislation[11]

Sweden was amongst the earliest ratifiers of the Chemical Weapons Convention, the ratification bill being approved on 7th June 1993. This bill contained only the legislative proposals necessary to ratify the CWC. The CWC obligations mainly refer to Schedule VI for industry and a research laboratory of the Swedish National Defence Research Establishment (FOA) which will house its Single Small Scale Facility (SSSF). The CWC was incorporated into Swedish national law by amendments of existing acts; this avoids various repetitions and it is easier for industry to apply certain rules since the provisions already apply to them while new provisions required to implement the CWC are found in known acts. In some cases existing legislation covered Convention obligations while in others changes and amendments were required. Amongst these were:

- Act on items which could be used for mass destruction (Dual-Use Act);
- Act on war materials;
- Penal code;
- Act on limitations and privileges;
- Ordinance on Dual-Use Products; and
- Ordinance on war materials.

a totally new act, the Act on Inspections, was promulgated at the same time. The Acts, and amendments, were promulgated on 24 March 1994. The entry into force of these acts await the actual EIF of the CWC. However the Ordinances on Dual-Use Products and War Materials entered into force on 1 July 1994.

Other Acts involved were:

- Act on Chemical Products;
- Act on Protection of International Business Information;
- Customs Administration Act;
- Act on Transporting of Dangerous Goods; and
- Act on Penalties for Run Goods.

These did not have to be presented to Parliament since the provisions already covered CWC obligations. The government's bill for national legislation required by the CWC gives guidelines as to how to analyze and interpret the various acts.

National Authority[12]

The preparatory work in Sweden was carried out by the Ministry of Foreign Affairs, the Ministry of Defence (Military Headquarters and the National Defence Research Establishment) and the National Chemicals Inspectorate (Kem I). In principle, the Ministry of Foreign Affairs will be responsible for contact with the Organisation for the Prohibition of Chemical Weapons (OPCW).

Last the newly formed National Inspectorate for Strategic Goods (ISP) from 1996 is housing the National Authority; this office is small and exercises a coordinating function in the implementation of the convention assignments at the national level which involves the War Materials Inspectorate for chemicals regarded as war material e.g., Schedule 1 and the Chemicals Inspectorate for chemicals normally in civilian use, e.g., Schedule 2, 3 and other chemicals and also National Defence. Kem I is responsible for declarations.

As noted before, Sweden has not created a permanent body for which to form escort teams and so early identification of the type of inspection and hence notification of the relevant activities is essential. The people involved in inspections will have to be seconded from their normal tasks. The smooth conduct of inspection in Sweden is based on written documentation provided by its National Authority.

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[10] see T. Svensson p. 52-56 in *Effective Implementation of the Chemical Weapons Convention*, edited by T. Kurzidem, Y. von Lersner, P. Radler, T. Stock and R.G. Sutherland, 272 pages, Bad Homburg, Germany 1995 (see reference 1).

[11] Country Profile regarding the National Implementation of Chemical Weapons Convention: Sweden, P. Radler, 17 pages, SIPRI-Saskatchewan-Frankfurt Project, *Effective National Implementation of the Chemical Weapons Convention*, September 1995.

[12] Sweden: Preparation for Inspections under the CWC - Development of Inspection Manuals, PC-XIV/B.WP8 July 1996.

VII. Conclusions

For the first time in arms control and disarmament the CWC requires detailed national implementation. Besides the penalization of any prohibited activities under the CWC, States Parties participating in this treaty securing its proper functioning by providing information about activities in industry and former or current CW related activities to the international organisation, the OPCW. These activities are the target for monitoring and inspections through the OPCW. This monitoring process will last in some form or another for the lifetime of the CWC since all existing chemical weapons and CWPFs will be destroyed within 10 to 15 years after entry into force.

The system of declarations/inspections is the real heart of the confidence building structure which demonstrates compliance with CWC obligations. To ensure that this information is available to the National Authority, a State Party has to establish the country-specific legal framework necessary to collect the requested data and information.

The proper functioning of the National Authority will be essential for the successful participation of every State Party in the CWC.

It is still too early to develop final conclusions about the successful implementation of the CWC. However, what is already clear from the existing experience: the tasks involved in national implementation are much more complex than the negotiators at Geneva ever expected. Each State Party has to develop its own legislation, regulations and approach to its National Authority. Each State Party has to give careful thought as to the structure and location of its National Authority within government and to possible assistance from other ministerial bodies and/or institutes. Its relationship with those industries that produce, process or consume chemicals that appear on the CWC's schedules, as well as those who import or export these chemicals is crucial to the State's ability to demonstrate compliance with the obligations under Article VI of the Convention.

In the process of national implementation the magnitude of tasks for the National Authority will be directly related to the nature of the chemical industry in each specific country and will be complicated by the fact that the chemical industry is a dynamic one driven by the need for new products and new markets.

From the successful story of national implementation future arms control and disarmament treaties will gain a lot. The individual rights and obligations under the CWC for every State Party are assured through its national implementation undertaking. The concept of State Party's primary responsibility to show compliance with a treaty via providing information on certain issues and their verification through the international organisation contains a dimension which will secure in the future the universality of such treaties. Individual States Parties act independently in the framework of such a treaty. However, the legal framework has to be established at the national level which will secure that the State Party is able and capable to provide the date which are necessary to show compliance.

Table 1: Summary of Reporting and Inspection Requirements under the Chemical Weapons Convention for the Chemical Industry

Chemical	Reporting Threshold	Inspection Threshold	What Firms are Affected?
Schedule 2 A	100 kg	1 tonne	Producers, processors, consumers, importers, exporters
Schedule 2 A*	1 kg	10 kg	Producers, processors, consumers, importers, exporters
Schedule 2 B	1 tonne	10 tonnes	Producers, processors, consumers, importers,
Schedule 3	30 tonnes	200 tonnes	Producers, importers, exporters
Unscheduled DOCs	200 tonnes	200 tonnes**	Producers
PSF Chemicals	30 tonnes	200 tonnes**	Producers

Table 2. Declaration requirements and deadlines of declarations

	Schedule 2	Schedule 3	DOC and PSF
Initial declaration	Aggregate national data EIF(SP) + 30 days Declarations for plant sites EIF(SP) + 30 days	Aggregate national data EIF(SP) + 30 days Declarations for plant sites EIF(SP) + 30 days	Declarations for plant sites EIF(SP) + 30 days
Annual declarations (past activities)	Aggregate national data Year end + 90 days Declarations for plant sites Year end + 90 days	Aggregate national data Year end + 90 days Declarations for plant sites Year end + 90 days	Annual update Year end + 90 days
Annual declarations (anticipatory activities)	Declarations for plant sites Beginning of year – 60 days	Declarations for plant sites Beginning of year – 60 days	
Additionally planned activities	Change after annual anticipatory declarations Change – 5 days	Change after annual anticipatory declarations Change – 5 days	
Declarations on past production of scheduled chemicals for CW purposes	Declarations for plant sites EIF(SP) + 30 days	Declarations for plant sites EIF(SP) + 30 days	

GERMAN APPROACH TO THE DISMANTLING OF OLD CHEMICAL WEAPONS (OCW) IN MUNSTER

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1. Introduction

As a belligerent nation of World War I and II Germany is afflicted with the heritage of "Old Chemical Weapons" (OCW) which can be dated from 1915 up to 1945. OCW are still being found throughout in Germany, particularly in the ground of former production sites, filling stations, testing areas and storage facilities. A large amount of OCW was dumped in the sea, for instance in the Baltic Sea. Due to external corrosion, unpredictable ballistic properties and fuze mechanisms and the extent of decomposition of chemical fillings such OCW are no longer of military use as chemical weapons (CW). However, since they represent a burden to the environment and potential hazard, Germany decided to destroy those items found on her territory. The undertaking in Germany related rather to a "collection" of various OCW items than an "OCW stockpile". Most of these items, including captured OCW from other countries, were sporadically recovered in the past four decades. Each of these items requires individual assessment by explosives ordnance disposal (EOD) personnel of its condition prior to destruction. Therefore, a high degree of automation aimed at speeding up certain steps in the disposal process, which would also reduce costs and improve the working conditions of the personnel, does not appear to be feasible at the present stage. Whenever possible, high-risk operations must be carried out under remote control. A completion date of the disposal program is difficult to predict given the considerable uncertainty of the total amount of OCW that might still be hidden in the ground. Only some of the experience gained in terms of the technology used, time frames and costs of the overall effort may apply to the destruction of large current CW stockpiles. However, Germany is ready to share its experience with other countries also faced with similar problems. Particular experiences exist with the handling of arsenic compounds.

2. Types of OCW munitions and chemical fillings

A large variety of OCW munitions, and even storage tanks, produced within the 1915 - 1945 time frame, may be found. These include:

- artillery shells - mortar ammunition
- hand and rifle grenades
- land mines
- bombs
- spray canisters.

The wide spectrum of toxic chemicals then used as fillings in OCW munitions includes various preparations of sulphur mustard, nitrogen mustard, lewisite, other arsenicals (e. g. Adamsite, Clark I, Clark II), phosgene and tabun. Sulphur mustard fillings in many cases contain arsenicals such as phenyl dichloroarsine or "arsine oil". Only a small number of tabun shells or bombs have been recovered in Germany and were destroyed. The recovered OCW items originate from fielded or experimental types of chemical munitions, which are increased in number and variety by items captured from various adversaries of the World Wars. Hence, OCW munitions are dispersed over large parts of Central Europe, due to both World War I operations and battles (e.g. Flanders), and due to the dislocation of chemical weapons to different depots during World War II. Dismantled former live agent test sites and destroyed former agent production or filling stations like Munster, are believed to have OCW still buried in the ground nowadays; most common until recently were e. g. failure batches of spray canisters filled with viscous sulphur mustard.

3. Procedural steps of OCW disposal

3.1 Reconnaissance and locating

Prior to the scouring of the terrain for the presence of OCW a comprehensive search of the archives and all other available sources, including evident facts and notes of eye witnesses is required. Thorough evaluation of aerial infrared photographs has proven to be a suitable means. Irregularities of the surface in areas surrounding former CW production or filling facilities would indicate underground artifacts, such as hidden munitions. Reconnaissance missions on the ground are carried out by EOD experts using magnetic probes or other metal detectors. These instruments are capable of locating metal parts up to a depth of 6 meters. Those spots giving a positive detector response are marked with flags and safeguarded to prevent unauthorized access.

3.2 Unearthing and identification

In order to recover and identify the artifact hidden in the ground, EOD experts cautiously clear away the soil until the item or the number of items can be recognized visually. At a later stage mobile X-ray equipment is used to further evaluate the internal structure of the munition. Following the preliminary assessment of the item as to whether or not it relates to OCW, its state of preservation and possible leakage, a decision is made on its hands-portability. Some OCW munitions have to be disassembled and have to be decontaminated on-site. Unidentified items are transported to the nearest demilitarization facility and opened for sample-taking and chemical analysis of the contents. During the whole process of unearthing and identification, appropriate safety areas around the discovery points must be established and standardized operational procedures (SOPs) of individual protection are applied and enforced.

3.3 Removal and transportation

Since OCW items are often found in places quite distant from each other, they have to be collected and transported to an intermediate storage site. For transportation of OCW items rugged containers are needed which remain gas-tight under pressure and which are coated in a way as to allow the use of aggressive decontaminants. The transport containers must be approved by the federal authority for transportation of dangerous goods and explosives. The planning of transportation comprises negotiations with federal state police departments and environmental agencies. Detailed agreement is required on the routes to be taken. In case of significant quantities, OCW transports must be secured by police and accompanied by trained personnel for detection, decontamination and medical support.

3.4 Intermediate storage

In order to facilitate further treatment and final destruction and for reasons of safety and clarity, the storage of OCW is organized in a way that identical munition items and heterogeneous ones are collocated separately. This, of course, results in a need for facilities for the safe intermediate storage of OCW, which have been removed from different areas of the region or other federal states. The storage bunkers must meet a number of stringent standards with regard to over-pressure stability, fragment protection of walls and doors, gas-tightness in case of accidental release of toxic chemicals, chemical agent detection and alarm systems. Filtered air ventilation and automated fire-fighting installations including sprinklers are also needed to ensure occupational safety and the protection of the environment. In addition, a basin for the collection of waste water in eventual decontamination or fire fighting missions is required. The bunker, with its separate compartments, is structured in a way which keeps the various types of munitions segregated. Walls of adequate height are installed in order to prevent the propagation of eventual explosions to other munition staples. The storage facility is subject to frequent inspections in accordance with safety and security regulations issued for its operation. Plans for early warning and eventual evacuation of the population living in surrounding areas have to be established. These plans must be continuously updated taking into account changing results of risk analyses. Although very unlikely, the release of toxic chemicals as a result of explosions, fires or direct impact of an airplane cannot totally be ruled out.

3.5 Preparation of OCW for demilitarization ("demil")

The munitions excavated from the ground are mostly covered with dirt and rough layers due to corrosion. For this reason, prior to further treatment, they have to be cleaned using a high-pressure water jet. Since the internal structure of recovered OCW differs considerably from item to item, X-raying of each of them individually is absolutely crucial, followed by a thorough evaluation of the negative film produced, which includes as accurate measurements as possible of all relevant features shown on the film. Only then are the EOD experts in the position to determine the detailed demil route, which is pertinent to the specific OCW item. In order to reduce the internal pressure of volatile liquid or gaseous chemical fillings, the munition items are frozen in a final step prior to demil. Thus, agent spillings out of the shell can be avoided or at least limited when being opened.

3.6 Demilitarization

Demil of OCW is the mechanical disassembling of munition items, which includes the elimination of the fuzes, explosive charges (bursters) and the draining if chemical fillings. In most cases, for the execution of these particularly critical and hazardous steps, remote-controlled special demil tools and devices are required. these include defuzing tools, machines for drilling, milling and sawing and chopping benches. In the event of explosions or release of toxic chemicals, the personnel operating these devices from a demil control bunker are protected against fragments, vapours and aerosols by thick concrete walls and an air filtration system of high efficiency. The demil process is constantly monitored and video-recorded, which provides thorough

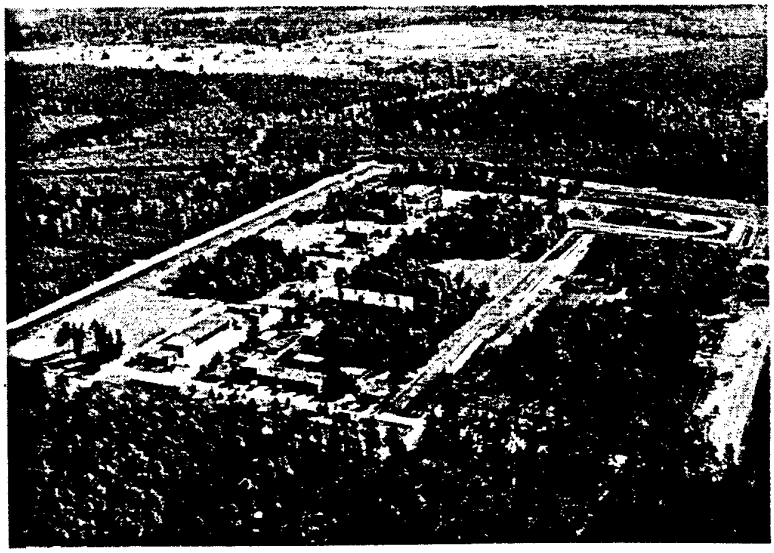
documentation and retrospective evaluation of the entire operation. The techniques that have been developed for the draining of OCW include

- the draining of liquids after automatic piercing, drilling or milling through the wall of the compartment;
- expansion of pressurized volatiles (e.g. phosgene) through a pipe system connected with an absorption column;
- removal of viscous fillings using special tools, such as spatulas;
- removal of solid fillings from opened munitions by melting using hot steam injection or by punching out using a special pestle device

Draining of chemical munitions bears a high risk of contamination not only of the demil devices themselves, but to all surfaces inside the demil chamber. Therefore, encapsulation of all parts not related to actual mechanical operation is required. Impermeable protective suits and respirators with filter canisters must be worn by personnel while working in the demil chamber on the preparation, maintenance or repair of demil devices. At present, none of the aforementioned draining techniques is used in automated continuous operation. (In the case of large numbers of sufficiently uniform munitions, typical of current CW stocks a highly automated demil line would be required and has been shown by the United States to be feasible.) For thorough decontamination of all exposed surfaces inside the demil chamber a highly effective decontaminant, such as a decont emulsion introduced by the Federal Armed Forces, must be kept in readiness. After any mission that could involve exposure to toxic chemicals, the protective equipment used has to be decontaminated and carefully inspected. Furthermore, special first-aid medical support and transportation with ambulances must be ensured at any time while demil operations are being carried out.

4. Final destruction of chemical agents recovered from OCW

The German OCW disposal concept includes as a final and decisive step the incineration of the chemical agent fillings at high temperatures, which results in the complete mineralization of the chemical compounds. The flue gases arising from the combustion are scrubbed with sodium hydroxide in aqueous solution. Emissions leaving the stack are continuously monitored in a way that meeting the stringent German environmental standards is ensured. Prior to incineration, chemical agents and demilitarization wastes as well as empty shells are intermediately stored in polyethylene (PE) containers which can be burned up together with its combustible contents. Likewise used protective suits, gloves, boots and exhausted active carbon filter elements constitute a multiple volume of solid and liquid waste that has to be disposed of in the same way. Poor solubility of the large quantities of viscous mustard, discovered particularly in the region of Munster, in any type of solvent was the main reason for selecting a batch-type incineration technology. In the case of phosgene as filling of munitions easy disposal is possible by neutralization with sodium hydroxide. The incineration plant in Munster is laid out for a destruction capacity of approximately 70 tons per year including all the contaminated waste and metal munitions scrap.
An additional incineration plant is currently under construction for the disposal of soil contaminated with both arsenicals and other toxic chemicals. Moreover crumbs of explosives (TNT) with adherent arsenicals, originating from old pit burning places, will be processed in a very special way in this new plant also.



Aerial view of Munster EOD Facility

German chemical agents of World War I

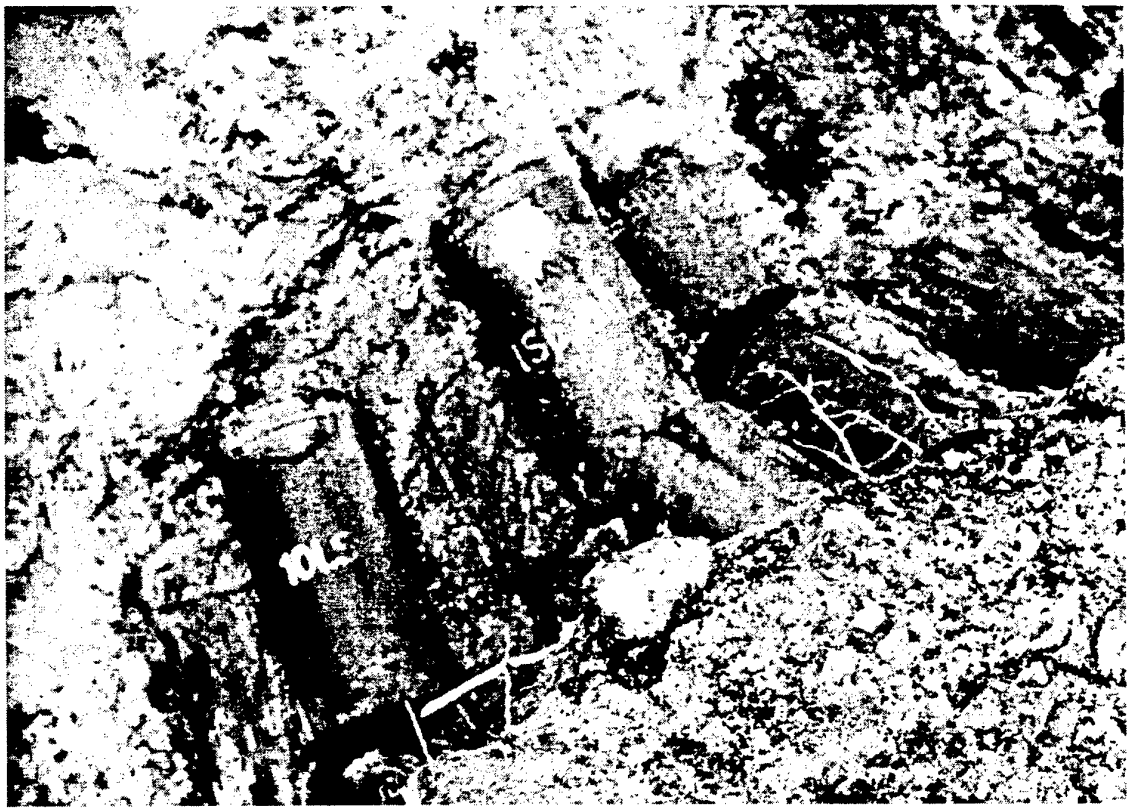
CWA group name:	Chemical agents:
Grünkreuz (Green cross)	Chlorine Phosgene Diphosgene (Perstoff)
Blaukreuz (Blue cross)	Clark I (Diphenyl-chloroarsine) Clark II (Diphenyl-cyanoarsine)
Gelbkreuz (Yellow cross)	Sulphur mustard (Yperite, S-Lost)
Weißkreuz (White cross)	Tear gases Bromo-aceton Bromo-methylethylketon Chloropicrin (Klop) Cyanogen chloride

German chemical agents of World War II

Group of chemical agents	Chemical agent types (Code name)
Nerve agents	Tabun (Trilon 83/GA) Sarin (Trilon 46/GB) Soman (GD) (only lab. scale)
Blister agents	Sulphur mustard (HD) Nitrogen mustard (HN) "Winterlost", HD for winter use "Zähllost", viscous mustard "Oxol-Lost", Oxygen mustard
Blood agents	Hydrocyanic acid (AC)
Choking agents	Phosgene (OLF) Diphosgene (Perstoff)
Organoarsenic agents	Adamsite (Azin/DM) Clark I and II Arsine-oil (OL A)
Tear gases	Omega salt Chloro-acetophenone (CN)

Chemical agent mixtures with explosives:

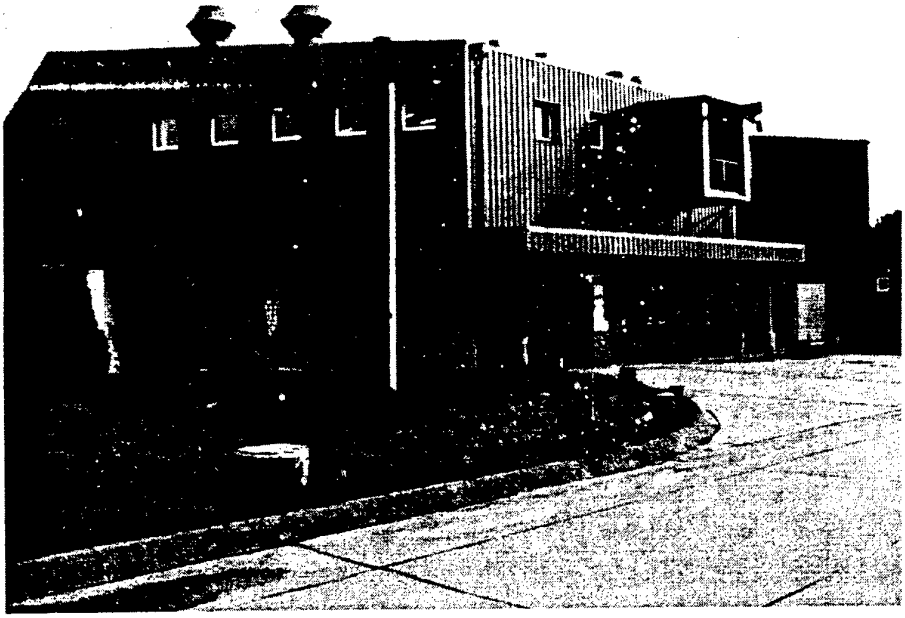
PETN / CN
Nitrocellulose/DM



Buried German WW II Mustard Spray Cans



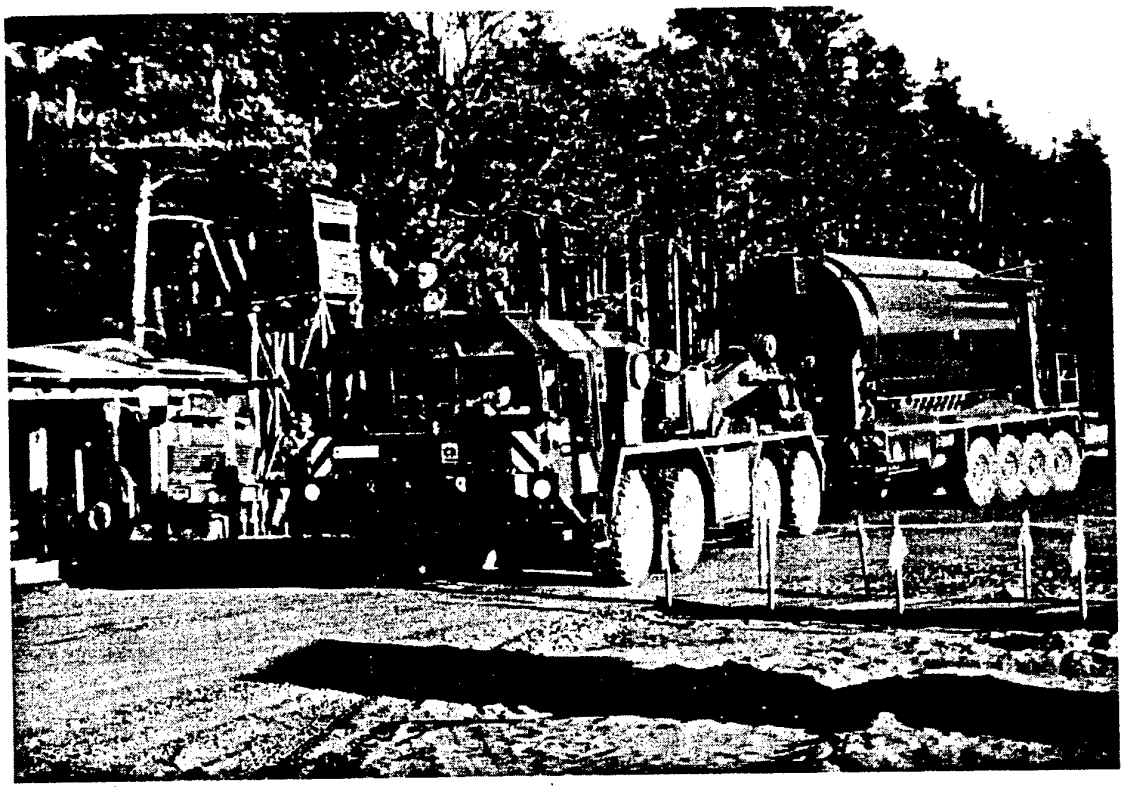
Dismantling of recovered German WW II Mustard Spray Cans



Munster EOD Facility – OCW Dismantling Station



Automatic Milling Device for OCW Dismantling



Mobile Explosion Containment Vessel for recovered OCW



Handling of German OCW prior to Dismantling

THE TOOELE CHEMICAL DEMILITARIZATION FACILITY - DESTROYING MORE THAN 40 PERCENT OF THE U.S. CHEMICAL WEAPONS STOCKPILE

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Former Deputy Assistant Secretary of Defense (Environment)

PROGRAM HISTORY

Chemical weapons have been in existence since World War I. In 1975, the United States signed the Geneva Protocol, as a first step toward banning the use of chemical weapons, except for use in retaliation to chemical attack. In 1985, PL 99-145 was enacted. This law directed the Department of Defense (DoD) to destroy our stockpile of unitary weapons. These weapons, some stored for over 40 years, contain pre-mixed, live chemical agents. No agents or munitions have been manufactured since 1968. The U.S. Army Office of Program Management for Chemical Demilitarization (PMCD) is responsible for this program.

PL 99-145 dictates that the facilities be dismantled after destruction of the stockpile. Partially because of cost (current program estimates are $13 billion) and partially because of other possible uses, Congress has directed a follow-on study for practical uses of these facilities after destruction of the stockpile.

Currently, munitions are stored at nine sites, eight of which are in the continental U.S., in varying types and quantities: Johnston Island, HI (6.6%); Tooele Army Depot, UT (42.3%); Anniston Army Depot, AL (7.1%); Umatilla Army Depot Activity, OR (11.6%); Pine Bluff Arsenal, AR (12%); Pueblo Army Depot Activity, CO (9.9%); Aberdeen Proving Ground, MD (5.0%); Newport Army Ammunition Plant, IN (3.9%); and Blue Grass Army Depot, KY (1.6%). The Johnston Island Chemical Demilitarization facility became operational in June, 1990, and the Tooele facility was started up in August, 1996.

The Army, supported by several independent advisors decided that the best approach to destruction of the stockpile, from a programmatic viewpoint, was by on-site incineration at each individual facility. This approach eliminates the risk of transporting the weapons to more centralized facilities. Safety, health and environmental concern for the public and the workers dictated the design and operation of the incineration facilities. This process was first tested on chemical weapons in 1979 at the Army's Chemical Agent Disposal System (CAMDS) in Tooele, UT, and it has successfully operated for almost 20 years.

On April 23, 1997, the United States Senate ratified the Chemical Weapons Convention (CWC) Treaty. The CWC was negotiated over a period of 24 years (1968-1992). It has been signed by 160 nations and ratified by 45. The CWC has a 10-year mandatory destruction deadline (2006) for all signatories.

Although the CWC will affect the program schedule, planning, and testing full scale operation is already underway (see Insert A). The largest stockpile and biggest variety of weapons (see Insert B) is at the Tooele Army Depot, Utah. The two major considerations in meeting program schedules are the technology used in weapons destruction and the related environmental, safety and health requirements for safe operation. The Tooele facility is the first to be constructed in the continental United States. There have been many unique challenges related to project startup.

Programmatic Objectives

From the beginning, the government has strived to keep the local communities, citizens and interest groups informed and involved in the destruction of the stockpile. In addition to state and local government, who establish special permits and emergency preparedness requirements, the program has oversight from the Congress, GAO, the National Research Council of the National Academy of Sciences, U.S. Department of Labor, Federal Emergency Management Agency, U.S. Department of Health and Human Services, Department of Defensive Explosives Safety Board, OSHA, Council on Environmental Quality and the U.S. Environmental Protection Agency.

Under the National Environmental Protection Act (NEPA), the Army was required to perform both a programmatic (umbrella) Environmental Impact Statement (EIS) and a site-specific EIS.
Both processes required lengthy, complicated public participation processes.

TOOELE PROGRAM

Since the Tooele, Utah facility is the first to be constructed in the continental U.S., it is the model for those that follow. As part of this process, a Record of Decision (ROD) was executed in 1988. In September, 1989 the Army awarded a contract to the EG&G Team for the construction, operation and closure of the facility. Team members

included Morrison Knudsen and Battelle Memorial Institute.

The Army's philosophy for design is E^3s (Efficiency, Economy, Environment and Safety).

The Tooele facility was designed using information gained at the isolated Johnston Island facility which was built, as a full scale plant, to test and ensure public and worker safety before facilities would become operational in the U.S. One of the main safety criteria was minimal contact between human operators and the chemical munitions.
The chemical storage at Tooele is four times larger than any other facility, and has the largest mixture of weapons - mortars, projectiles, ton containers, rockets, bombs, spray tanks and mines. The facility has been designed to destroy more than one type weapon at a time providing only a single agent is involved.

The weapons are moved from the storage yard in specially designed containers, inspected and moved into a total containment area where they are unpacked. At this point, the weapons is legally declared a RCRA waste. Then the agent is separated from the explosives. This is done in one of five mechanical processes: rocket shear machine, mine machine, bulk drainage, projectile/mortar disassembly machine or the multi-purpose demil machine. This operation is followed by thermal destruction in one of four incinerators - Deactivation Furnace, Metal Parts Furnace, Liquid Incinerator (2), and Dunnage Furnace. The end products are residue, scrap metal and liquid brine (or salts). Each incinerator has its own pollution abatement system that cools and scrubs the exhaust gases, chemically neutralizes acidic components, and removes particle matter.

CHEMICAL STOCKPILE WEAPONS

The unitary weapons stockpile consists of two types. The first is a blister agent, mustard (H/HD/HT) that attacks the eyes, lungs, and skin. The agent is toxic and works by attaching and destroying cells. Exposure is generally only fatal if inhaled. The second is nerve agent, GB and VX. Nerve agents attack the central nervous system. Both are derived from pesticide-like compounds. GB is a volatile liquid with a specific gravity similar to water, while VX is a more viscous material similar in consistency to motor oil. Both have similar molecular organo-phosphate, structure. While both agents are considered lethal, GB is a quick reacting material normally ingested or respired. VX, on the other hand, is a skin penetrate, slower acting primarily due to the absorption process. The agents are stored in various ways with approximately 60 percent in bulk, or ton containers. The remaining munitions consists of bombs, rockets, mortar rounds, spray tanks, mines and artillery projectiles.

The M-55 rockets are considered the most challenging weapon in the stockpile. They are fully assembled weapon containing agent, propellant, fuse, and an explosive device. The rockets are the primarily source of leaking munitions. These munitions are located at five of the eight continental U.S. storage sites. With the exception of the rockets, there are few problems from leaking munitions. However, continued storage does constitute an increasing risk to the surrounding area, and munitions deterioration continues with age.

Environmentally, two major permits were required: Clean Air and the Resource Conservation and Recovery Act (RCRA). Both presented challenges. The State of Utah Department of Environmental Quality (DEQ) is responsible for issuing both permits. Because of the nature of the material and obvious political sensitivity, the State of Utah, in conjunction with the Army, set destruction rate efficiency for all surrogate trial burns at 99.9999 percent destruction (so called "six-nines" standards). Additionally, the same standard was applied to the two Liquid Incinerators for Agent Trial Burns prefacing each campaign. A Toxic Substance Control Act (TSCA) Demonstration Burn was also conducted as required by the U.S. EPA and the enhanced standard of 99.9999% was applied. This was necessary because of the M55 rocket tubes containing small quantities of PCBs used as stabilizers.
The issuance of the RCRA permit had several unique features because both the State and EPA consider the Tooele Chemical Disposal Facility (TOCDF) a RCRA Treatment Storage and Disposal (TSD) facility.

As such, it is governed by all RCRA requirements including:

- All agents, once received from storage by TOCDF, is defined as a "waste" and must be stored and disposed of accordingly. Unique waste codes have been developed for this purpose.
- All residue is to be considered hazardous for handling and disposal.
- All Engineering Change Proposals (ECPs) are considered permit changes and require permit modifications from the state. There are currently over 2000 ECPs that have been processed.
- Real time monitoring for agent release is required in all areas of the facility and continuous monitoring surrounds the site.
- Peak production processing rates are established in the permit and valid through Agent Trial Burns.

During the systemization phase of the program, all mechanical systems were tested using inert munitions. Instrumentation systems, safety systems, system controls, and alarm systems were all evaluated to ensure full adherence to design requirements. The workforce was trained, qualified and certified to ensure that they were prepared to handle live agent. At the completion of this phase of work, EG&G certified it's readiness to the Army through a series of Operational Readiness Evaluations (OREs). The procedures for testing, pre-operations phase, with live agent are governed by Federal and Army Regulations and requirements of the State of Utah. Once the systemization was completed, it was necessary to conduct trial burns with toxic agent and incinerator feed rates will be established during trial burns.

The testing is conducted on the two liquid incinerators, metal parts furnace, deactivation furnace and dunnage furnace before each of the three campaigns (GB, VX and mustard), and for each representative munition.

The stack emissions are governed by a permit issued under the Clean Air Act. Carbon dioxide and water vapor are discharged, along with minute quantities of So_3,, No_x, CO and particles. The system is continuously monitored for agent release to level which far exceeds legal requirements (0.0003 Mg/M^3).

Testing for the GB campaign has just been successfully completed, with the exception of testing the second liquid incinerator and that is scheduled for testing later this year. The incineration processes passed the test. For some tests, test results achieved removals that greatly exceeded requirements. Similar tests will be conducted for each munition and each agent campaign and will last throughout the life of the project. The current GB campaign is scheduled to be completed by November, 1998.

During the conduct of systemization and testing, there has been a need to make many engineering changes. (It should be noted that through February, 1998, there have been about 200 Class I ECPs and a total of over 2000 ECPs). This has affected operations and required revision to engineering drawings, procedures and training. Although Tooele has an extremely comprehensive safety training program, concerns have been expressed that all the upgrading that has occurred could, in the short-term, create unsafe conditions. For example, there is a normal lag time to revise all of the documents once a change is made. The plant has developed a "work-around" system that addresses this problem. However, because of the high visibility of the program, EG&G and PMCD concluded that more stringent systems need to be put in place. EG&G, PMCD, IOC and major subcontractors have developed and initiated a Safety Culture Plan. The Plan is scheduled to be fully implemented by the end of 1998. The goal is to attain "World Class Safety." The foundation of the Plan is DuPont's STOP program and OSHA's comprehensive Voluntary Protection Program (VPP) Star status. When fully implemented, it is expected that there will be a culture driven by individual attitudes and behaviors, reinforced through professional training and maintained and measured through usable metrics. In addition to this initiative, both EG&G and PMCD conduct oversight audits. This effort has resulted in a Record of Injury Rate (RIR) of 3.68, in over 950,000 man-hours. There has been zero lost time due to injury and about one-half million pounds of GB agent have already been destroyed.

The program goal of PMCD is to complete destruction of the entire stockpile by 2004, prior to the dates stipulated in the Chemical Weapons Treaty.

The work being done at Tooele and the programmatic lessons learned (PLL) will be the cornerstone for success of the program. Both PMCD and EG&G are fully committed to developing a safe, environmentally sound program and simultaneously meeting production objectives, public expectations, and all Treaty commitments.

Proceedings of the International CW Destruction Symposium
Munster, Germany, March 22-25, 1998

POLISH EXPERIENCE IN SAFE DISPOSAL OF ARSENIC CONTAINING CHEMICAL WARFARE AGENTS

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Abstract

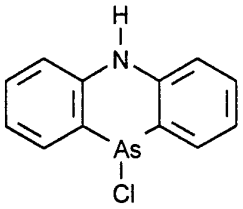
Approximately 9 tons of adamsite have been discovered on the territory of Poland after the World War II. This dangerous chemical agent was stored in steal barrels and special preventive measures were undertaken in order to protect it against spreading. This agent is considered as the old and abandoned chemical weapons under the rules of The Chemical Weapons Convention (CWC). The Polish Government has decided to destroy the abandoned adamsite and different suitable technologies were considered. The first laboratory experiments have started in 1996 and elimination of the adamsite on semi-technical scale will begin by the June 1998. In this paper methods of neutralisation of adamsite, based on its hydrolysis with hydrochloric acid, reduction with phosphorous acid and fusion with sulphur are discussed. These methods were found to be useful at the laboratory scale. Advantages and disadvantages of considered methods of destruction of organic arsenical agents have been discussed. The most promising method seems to be the reduction of adamsite with phosphorous acid. The products of this reaction are: metallic arsenic, diphenylamine (DPA) and hydrogen chloride. These products can be separated and reused or neutralised.

1. Introduction

There are 9.326 tons of adamsite in Poland, which is considered, under the rules of The Chemical Weapons Convention [1], as "the abandoned and old chemical warfare agent". The adamsite was discovered immediately after the end of the World War II, and its origin is still unknown. In the late 1940s the adamsite was transported from a storage area for items from the World War II in Lodz to the town of Modlin, and in the mid-1950s it was moved to the town of Ostrow Mazowiecka-Komorowo, where it is stored now. The agent is stored in a safe facility in 219 steel barrels, each of which contains 50 litres of adamsite sealed in a polythene bag [2]. Taking into account The Chemical Weapon Convention, which was ratified by the Polish Parliment in 1995 the Polish authorities decided to destroy the abandoned adamsite.

2. Properties of adamsite

Adamsite (10-chloro-5,10-dihydrophenarsazine) is a nasal and throat-irritating powder. It was produced just after 1918. It is a dark-green solid with melting point at 193-195°C. Chemical structure of adamsite is shown below.



The chemistry and reactivity of adamsite is well described in by Lohs [3], Franke [4] and Compton [5]. The excellent revue of physico–chemical properties and toxicity of adamsite is elaborated by Kopecz [6]. Recently different methods technologies neutralisation of arsenic containing chemical warfare agents (CWAs) have been described by the Russian authors [7, 8].

3. Selection of technologies

The project on destruction of the adamsite is under developing in Institute of Chemistry and Protection Against Chemical Warfare Agents, at Military University of Technology [9, 10].
We considered different methods and technologies for destroying the adamsite.

Physical methods:

High temperature thermal treatment;
Fusing with sulphur;
Dispersion in concrete.

Chemical methods:

Wet chemical neutralisation;
Wet chemical oxidation;
Electrochemical oxidation;
Wet chemical reduction.

Physical methods, based on the thermal treatment of arsenicals have some disadvantages. High temperature thermal treatment methods are publicly undesirable because they produce a couple of problems like emissions, toxic liquid and solid wastes from the burned CWA. Incineration is also very expensive, especially when small amounts of agents have to be destroyed. Incineration products from arsenicals also generate some environmental problems with respect to dischargement and deposition, which have not been solved yet. However, such installations are only economic when they are located in the vicinity of very large amounts of contaminated material to be incinerated. We came to the conclusion that for small quantities of adamsite the chemical neutralisation method in principle would have the best advantages.

It is evident that the neutralisation of arsenic-containing CWA needs technologies based on the elimination of the arsenic compound and aresnic as an element from the environment. The elimination of arsenicals, however, is associated with many serious basic problems.

Extremely long-term hydrolysis of adamsite ends up with phenylarsine oxide which still has irritant properties. Further steps leading to total mineralisation have not been observed with adamsite.

After detailed considerations we selected three technologies, which seen to be the most suitable for solving the problem of stockpiled adamsite:

Fusing the adamsite with sulphur
Chemical neutralisation with hydrochloric acid (HCl)
Chemical reduction the adamsite with phosphorous acid.

The acceptable technology has to ensure maximum protection of the personnel and public , as well as the environment. The safety precautions were considered as the greatest importance.

4. Fusion of adamsite with sulphur

When fusing the adamsite with sulphur the brown solid substance is obtained. The reaction between sulphur and adamsite is shown in Figure 2. The product does not contain the free adamsite, but it still contains arsenic as well, and is considered as toxic waste.

The flow chart of the fusion technology is shown in Figure 1.

The adamsite and powdered sulphur are mixed and heated up to 180°C. The product of reaction is neutral and its solubility in water is very low. Low installation and processing costs, irreversibility of reaction between adamsite and sulphur and simplicity of technology are the main advantages of this method. As disadvantages of the method the following aspects can be pointed out: (i) the volume of waste products is c.a. 4 times greater than the volume of disposed adamsite; (ii) long-term storage of arsenic containing solid wastes is necessary.

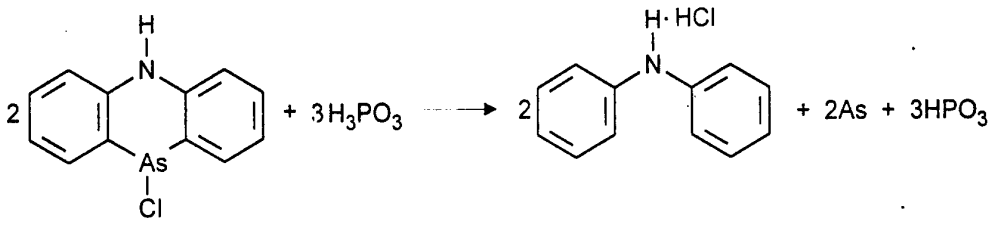
5. Neutralisation with hydrochloric acid

Adamsite reacts with highly concentrated hydrochloric acid to form diphenylamine (DPA) and arsenic chloride. Both products can be reused in chemical industry as by-products. Arsenic chloride can be hydrolysed and oxidised to arsenic oxide. Diphenylamine can be burned in typical industrial incineration facility. The flow chart of hydrolysis of adamsite with hydrochloric acid is shown in Figure 2.

The main advantages of this method are: (i) relatively low costs of technical facility and low processing costs; (ii) products of neutralisation (As and DPA) can be reused, and (iii) low volume of neutralisation products. Extremely corrosive reaction environment (HCl_{aq}), low reaction kinetic and necessity of disposal of arsenic containing wastes are the main disadvantages of this neutralisation technology.

6. Wet reduction with phosphorous acid

Adamsite is very resistant for low temperature oxidation. It is relatively easy to reduce organometalic compounds with chemical reductants. It is worth to notice that microbiological destruction of organoarsenicals proceeds via reduction stage [11, 12]. This idea was the basis for developing the technology based on reduction of adamsite with phosphorous acid. The process is fully irreversible and leads to the high purity of metallic arsenic and DPA. This reaction is shown below.



In fact the reducting agent is phosphoric hydride (PH_3), which is formed during the disproportionation reaction of phosphorous acid:

$$4\,H_3PO_3 \;=\; PH_3 \;+\; 3\,H_3PO_4$$

The flow chart of the proposed technology is presented in Figure 3.

This reaction is fully irreversible. Reaction products (As and DPA) can be reused and only small amount of solid toxic wastes must be stored or disposed. The technology with phosphorous acid is more complicated and expensive than technologies with hydrochloric acid and fusing the adamsite with powdered sulphur. Detailed conditions of kinetics of the reduction reaction must be established.

7. Conclusions

The acceptable technology of adamsite neutralisation has to ensure the maximum protection of public and personnel, as well as the environment.
The main factors, which influence the selection the most suitable technology for neutralisation of adamsite are as follow:

Risk level to the personnel, residents and the environment,
Irreversibility of neutralisation reaction;
Amounts of toxic solid wastes, and
Total costs of destruction.

References

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[11] P. JOHN, JR. PELLEGRINI, T. O'HARA, *Controlling soildwelling fungi with phenarsazine compounds.* Patent N 3,629,476, Dec. 21, 1971.

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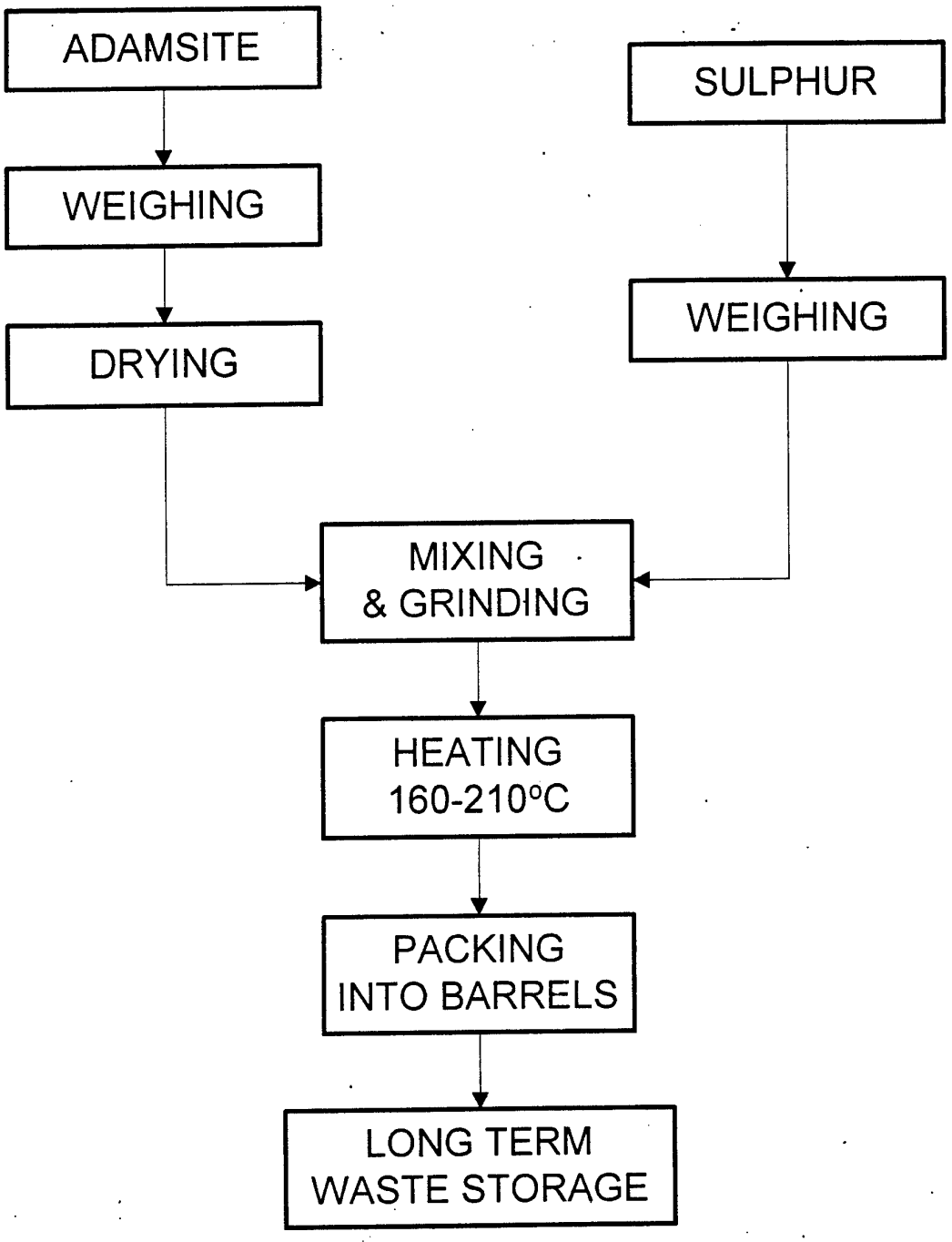


Figure 1. The flow chart of fusion of adamsite with sulphur

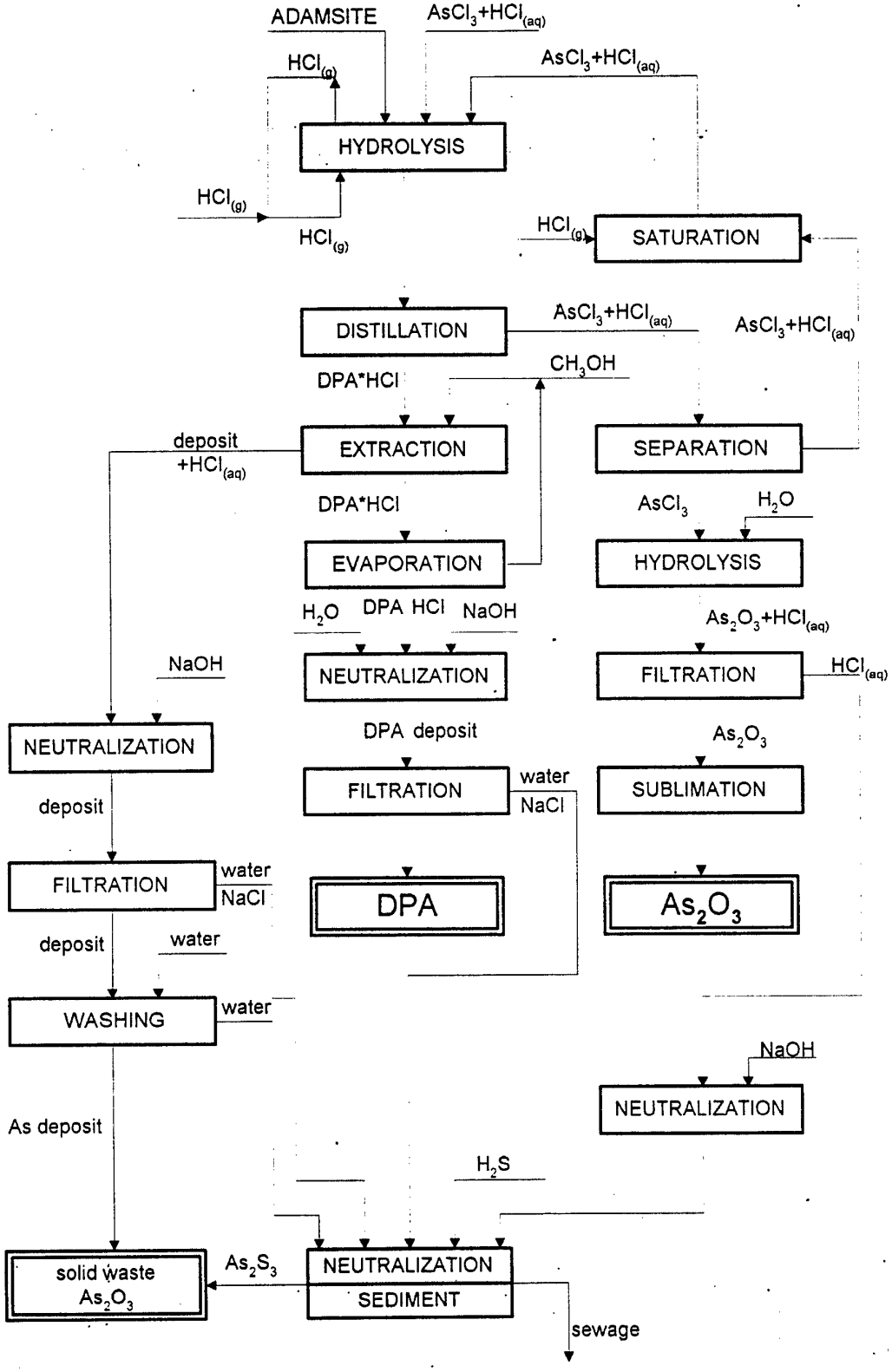


Figure 2. The flow chart of hydrolysis of adamsite with hydrochloric acid

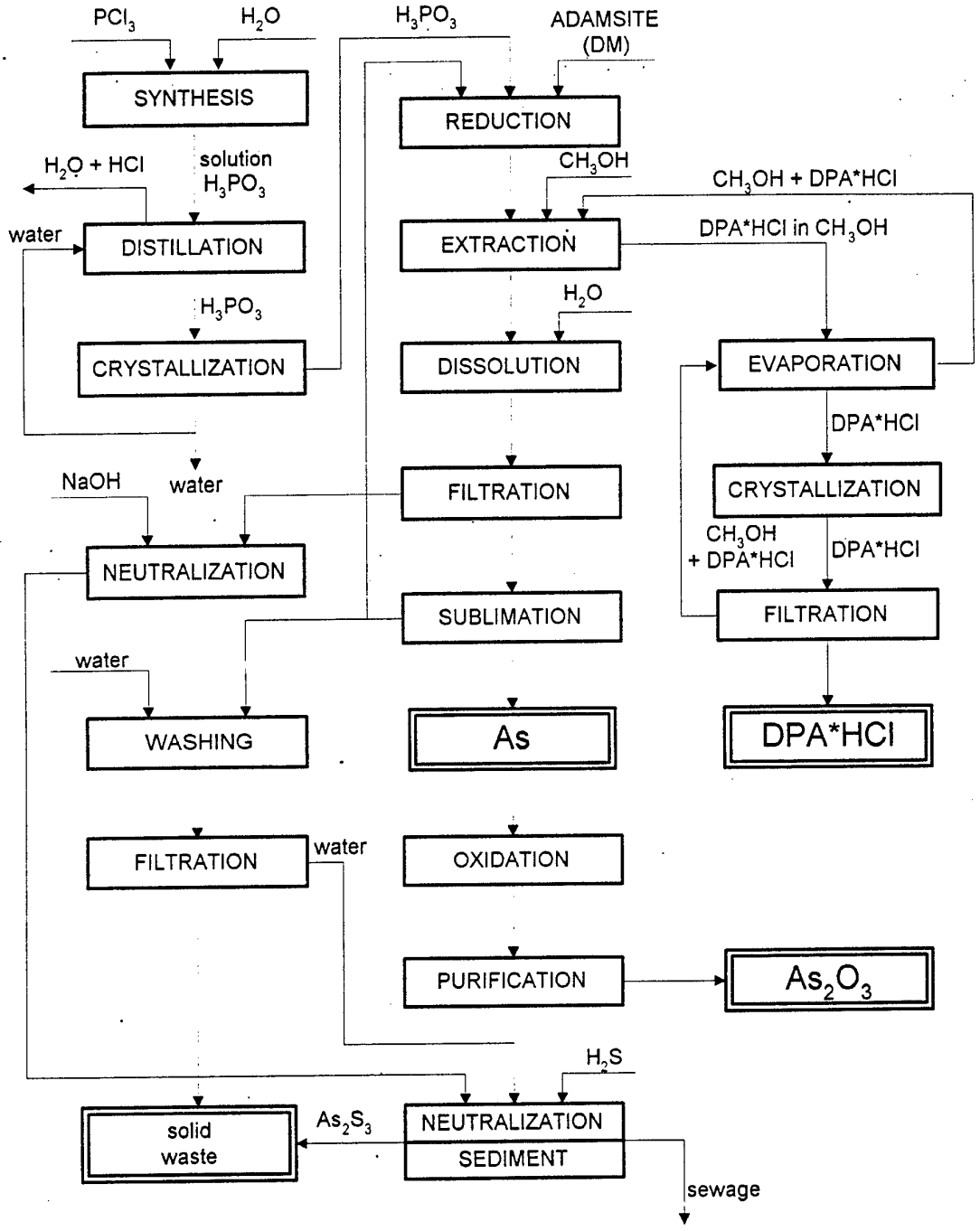


Figure 3. The flow chart of reduction of adamsite with phosphorous acid (H_3PO_3)

PROJECT ERASER: DISPOSAL OF OLD CHEMICAL AGENT WASTE BURIED IN CONCRETE IN A RADIOACTIVE WASTE SITE

John M. McAndless

DRES Canada

Executive Summary

Project ERASER (Entrained Residual Agent Stocks for Extraction and Remediation) describes a co-operative project undertaken by Atomic Energy of Canada Ltd (AECL) and the Canadian Department of National Defence (DND) to safely dispose of obsolete chemical warfare agent material and arsenic powder located at AECL, Chalk River, Ontario. This waste, estimated to contain less than 250 kg of chemical warfare agents, was transferred in 1960 from the Defence Research Establishment Ottawa to AECL, encased in an eight cubic metre block of concrete, and buried within an AECL Waste Management Area. The limited documentation associated with this disposal indicated the waste contained only mustard chemical agent and white arsenic powder.

AECL contacted DND in 1994 concerning potential hazards associated with the buried agent waste and requested assistance in removing the waste from their Waste Management Area, as this Area was licenced only for low level radioactive waste disposal. DND tasked the Defence Research Establishment (DRES) to carry out the technical aspects of dismantling the concrete block, extracting all waste within it, safely destroying the waste, monitoring operations to ensure maximum personnel safety and mitigating any environmental impacts. DRES project staff had considerable previous experience in the destruction of chemical warfare agents; the most notable examples included destruction of Canada s agent stockpiles at DRES in 1991 (Project Swiftsure) and participation with the United Nations in destroying Iraqi chemical weapons following the Gulf War (1992 - 1994). AECL had specific responsibility for excavating the concrete block, for providing radiation protection and monitoring, for disposing of any waste not related to chemical warfare agents and for providing site services, security and other infrastructure support as necessary. Other agencies involved in the project included the Canadian Forces Base (CFB) Petawawa (logistics and medical support) and Golder Associates Ltd. This latter company, through a contract with DRES, conducted an independent risk assessment of disposal options, a ground penetrating radar survey to precisely locate objects embedded in the concrete block and soil toxicity tests at the excavation site to determine the extent of soil contamination, if any.

A public consultation program was carried out from November 1996 - January 1997 during development of project plans. This program included discussions with mayors and town officials of the local communities in the vicinity of AECL, separate briefings for AECL management and employees, and an Open House meeting for local citizens and the media. The public consultation process was very successful and local area press coverage was positive. No significant issues were raised to cause project plans to be modified, and project operations within AECL Waste Management Area B commenced on 10 February 1997. All operations were completed over a three week period. The ambient temperatures during this time (< 10 °C) ensured that any mustard agent was in a frozen form, thus ensuring a high degree of safety and environmental protection.

The block was located precisely by using ground penetrating radar and by reference to early site maps. Once excavated and raised, the block was found to be slightly contaminated with low level radioactivity as a result of leaking Thorium-232 waste found at the same location. A containment building was placed over the block and the surfaces of the block were scarified to remove the residual radioactive contamination. AECL transferred control of site operations to DRES on 18 February 1997.

The ensuing extraction and destruction processes included the following steps:

- a ground penetrating radar survey of the block was conducted to located embedded objects and to guide the container extraction process;
- the concrete was mined by drilling boreholes, detonating small explosive charges to create fractures, then using manual chiselling to remove containers;
- in-container chemical neutralization of agent fills was completed using potassium hydroxide/methanol decontaminant solution;
- controlled thermal decontamination of the containers and thermal destruction of neutralization by-products was carried out ;
- arsenic and other inorganic materials were packaged for commercial disposal.

DRES and AECL staff monitored all activities to ensure there were no harmful emissions. Monitoring equipment included specialized radiological survey meters, chemical agent monitors (CAMs) and commercial

monitors for regulated substances such as sulfur dioxide. A Chemical Agent Detection System (CADS) was established on-site to continuously monitor the emissions from the thermal destruction unit and inside the containment building. All activities were recorded on video tape and photographed.

During the container extraction process, several unexpected items were found, in addition to the expected containers of mustard and arsenic powder. Unexpected items included ordnance such as 25-pounder artillery shells, Milk Can chemical mines, containers of agent precursor chemicals, as well as containers of zinc chloride and chlorosulphonic acid (smoke generating chemicals). Two non-fused 25-pounder artillery shells recovered from the block were found to contain nerve agent GB (Sarin), as determined using Chemical Agent Monitors, 3-Way Detector Paper and visual inspection of liquid samples removed from the shells. The shells were immediately immersed in decontaminant, neutralized and subsequently thermally decontaminated. A media conference was held immediately thereafter to inform the public living in communities adjacent to AECL property. This conference described and emphasized the safety procedures in place and alleviated any concerns about project operations.

Container extraction and agent waste disposal operations were completed by 22 February 1997. The total amount of agent destroyed was approximately 33 kg of mustard and 1.5 kg of nerve agent. Inorganics such as zinc chloride and arsenic powder were packaged for commercial disposal. The empty, decontaminated agent containers were taken to a training range at CFB Petawawa on 25 February 1997 and subsequently destroyed by explosive demolition. On the same day, with AECL and DND consent, the concrete residues and containment building at the burial site were burned to the ground to destroy any potential residual contamination. Site soil samples were subjected to a battery of toxicity tests and were found to be contamination-free. The site was re-visited during warmer weather in May 1997 to certify that no chemical or radioactive contamination remained.

Throughout the project operations, there was no immediate threat to workers and to public health and safety. Normal daily work within Waste Management Area B was able to continue at all times, except within the 50 m safety template imposed around the block, where access was restricted to members of the Project Team.

Project ERASER operations were viewed by an officer from the Verification Division of the Organization for the Prohibition of Chemical Weapons (OPCW), as well as the Chairman of the Biological-Chemical Defence Review Committee (BCDRC), an independent arms-length organization made up of individuals from outside DND.

All agencies cooperated well throughout this project. Project goals were achieved, on time, within budget, without environmental impact and most importantly, without threat to human health and safety. The pro-active public communication process was effective in ensuring public concerns and media interest were addressed. AECL provided a safe, controlled work site that proved to be an ideal location to conduct extraction and disposal operations. Finally, the Project Team employed proven technologies and procedures that were simple and effective. This provided the Team with the flexibility to adapt to unexpected developments without causing significant delays. These technologies and procedures are suitable for other small scale chemical demilitarization projects.

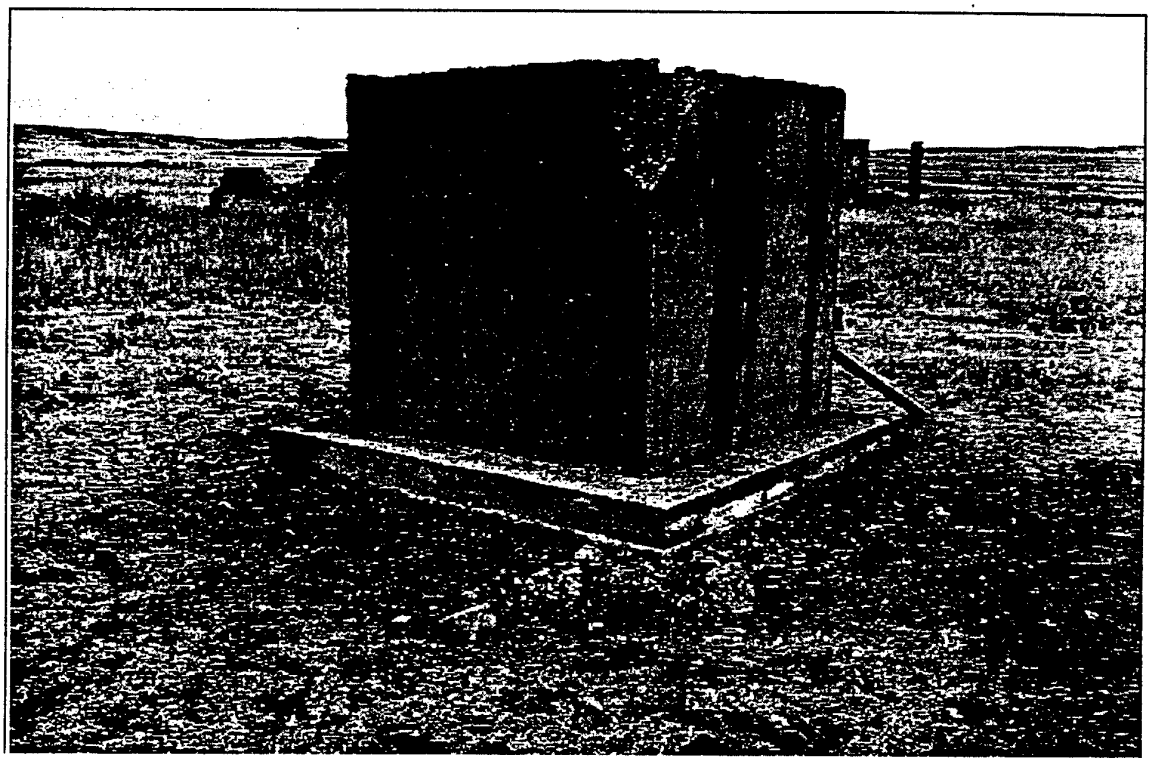
AECL WASTE MANAGEMENT AREA B



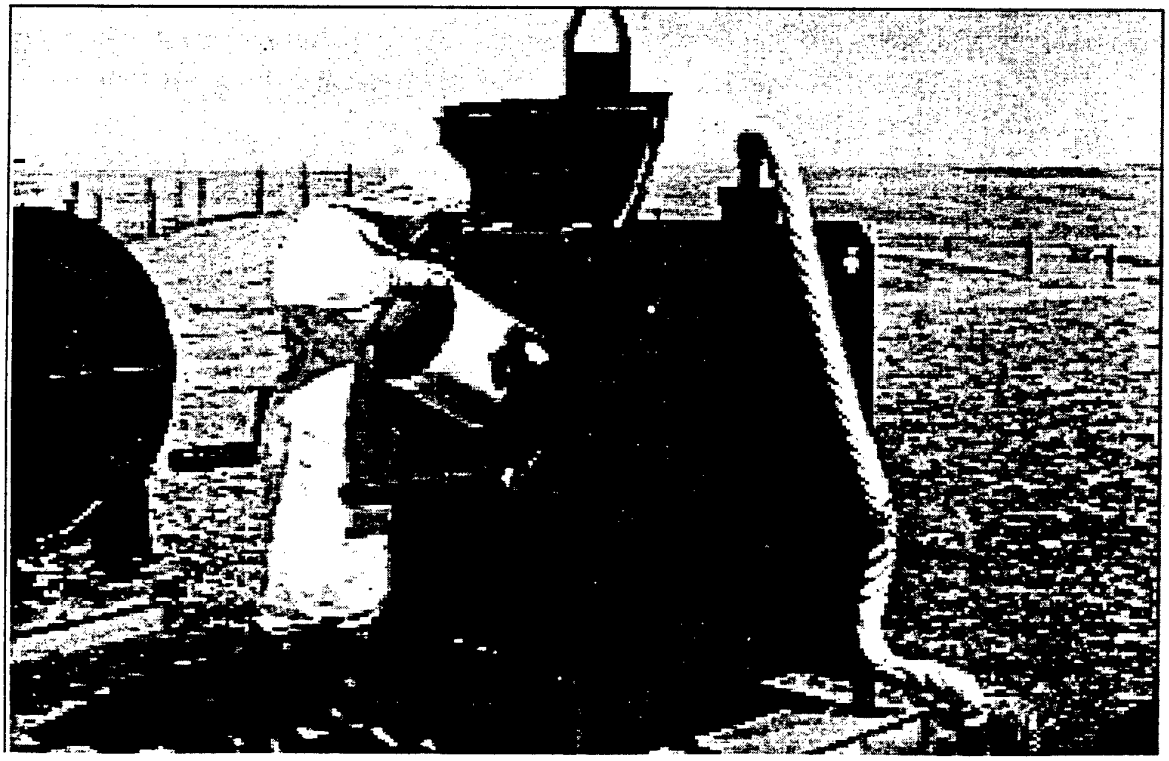
MG&A SITE



TEST BLOCK



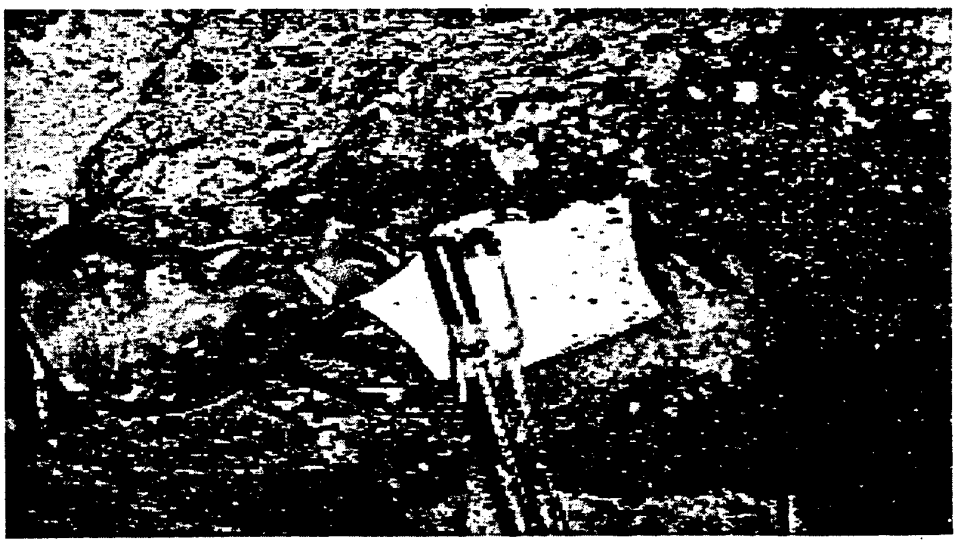
KIMBELL COOKER



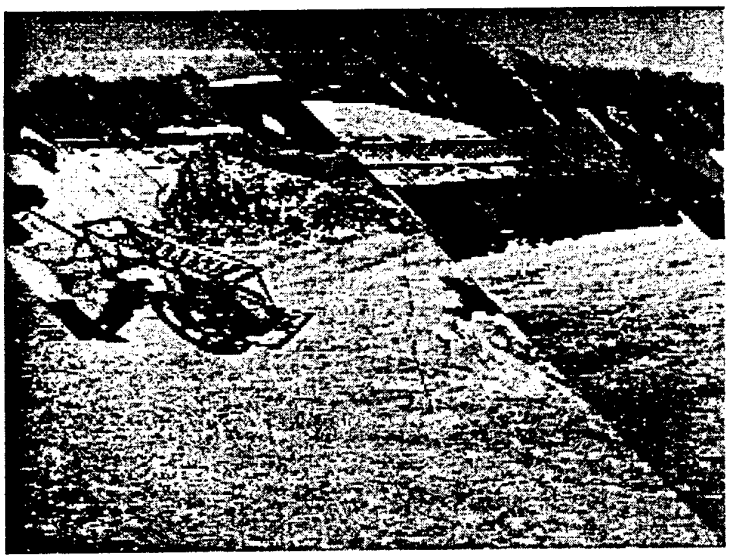
CONTAMINATION CONTROL

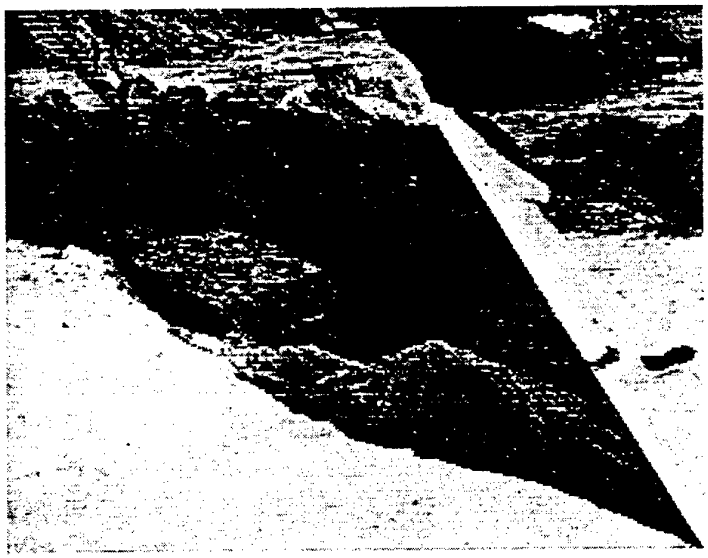


AGENT NEUTRALIZATION

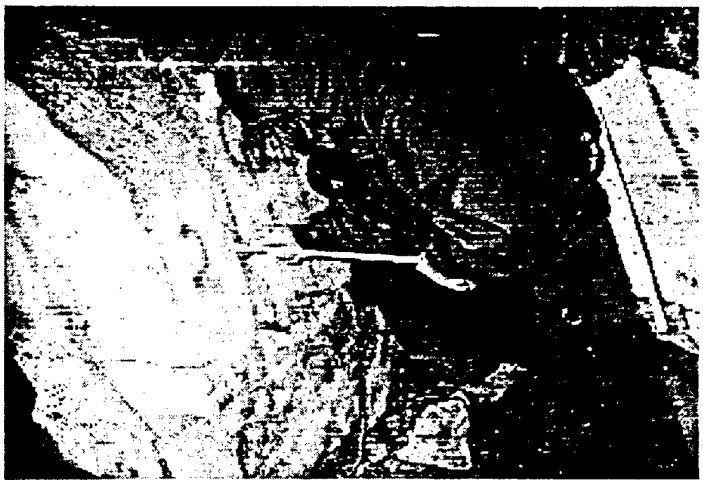


EXCAVATION OPERATION

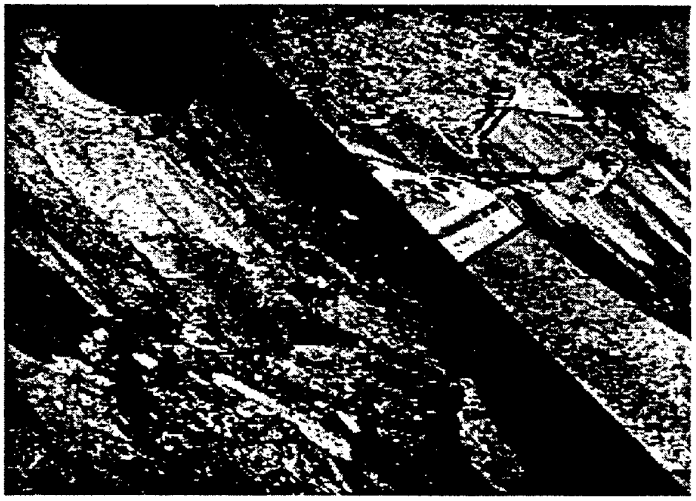


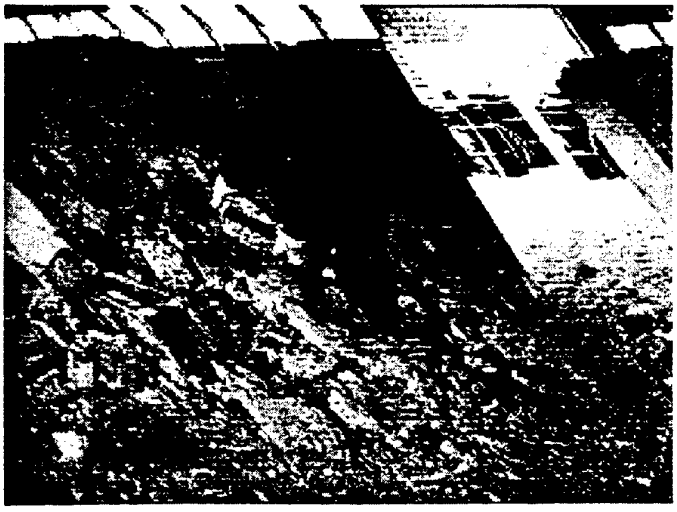






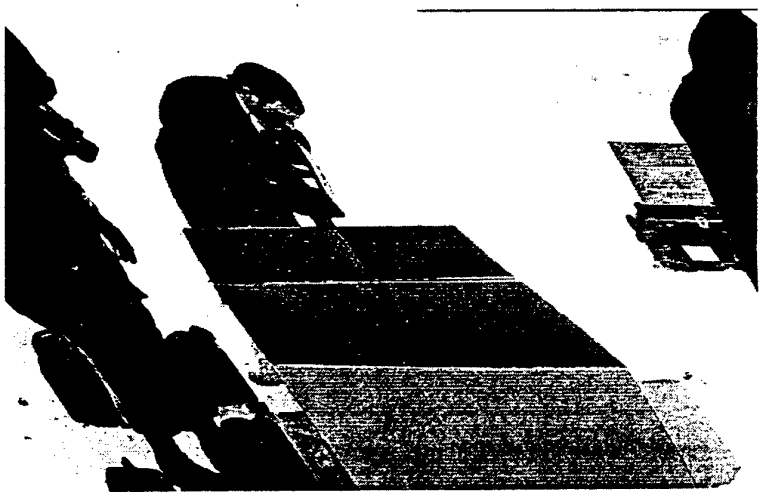
EXTRACTION PROCESS







CONTAINER DEMOLITION





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ENTRAINED RESIDUAL AGENT STOCKS (for) EXTRACTION (and) REMEDIATION



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Project ERASER

PROBLEM

- MUSTARD GAS (MG) AND ARSENIC (A) WASTE BURIED IN CONCRETE AT AECL CHALK RIVER SINCE 1960
- MG&A SITE LOCATED IN WASTE MANAGEMENT AREA LICENCED FOR RADIOACTIVE WASTE ONLY
- WASTE MANAGEMENT AREA REQUIRES PROGRAM TO REMEDIATE RADIOACTIVE WASTE CONTAMINATION PROBLEMS

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CB DEMILITARIZATION PROJECTS

MUSTARD DESTRUCTION (1972 - 1973)

700 TONNES MUSTARD DESTROYED BY NEUTRALIZATION/INCINERATION/BIODEGRADATION

EPG RANGE CLEARANCE (1977 - 1979)

THOUSANDS OF CW MUNITIONS RECOVERED

PROJECT SWIFTSURE (1989 - 1991)

400 TONNES CW AGENTS, MUNITIONS AND CONTAMINATED SCRAP DESTROYED BY NEUTRALIZATION/INCINERATION

CFB BORDEN (1991)

UNCOVERED CW MUNITION LANDFILL REMEDIATED

IRAQI CHEMICAL WEAPONS DESTRUCTION (1992 - 1994)

AGENTS, PRECURSORS, MUNITIONS, CHEMICAL WEAPONS PRODUCTION FACILITIES DESTROYED

EPG CONTAMINATED SITES (1994 - ongoing)

RISK ASSESSMENT/REMEDIATION OF 80 SITES

PROJECT ERASER (1996 - 1997)

DISPOSAL OF OBSOLETE CW AGENT MATERIALS BURIED IN CONCRETE AT AECL, CHALK RIVER

PROJECT ORACLE (1997 -)

RISK ASSESSMENT/REMEDIATION OF ABANDONED CHEMICAL WASTE DISPOSAL PITS AT DREO

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Project ERASER

SCOPE

CONCRETE BLOCK

DIMENSIONS:	2m x 2m x 2 m
VOLUME:	8 m^3
AGE:	37 YEARS
LOCATION:	AECL WASTE MANAGEMENT AREA B
STATE:	BURIED 2 m

CONTENTS

- **LIVENS MORTARS**

AMOUNT:	9 or 10
TYPE:	NON-EXPLOSIVE STEEL CONTAINER
FILL:	MUSTARD CHEMICAL AGENT
TOTAL FILL:	~ 120 kg

- **ARSENIC**

AMOUNT:	18 kg
FORM:	ARSENIC TRIOXIDE POWDER
CONTAINER:	UNKNOWN

- **LABORATORY WASTE**

AMOUNT:	UNKNOWN
FORM:	BOTTLES, STEEL DRUMS
FILL:	MUSTARD CHEMICAL AGENT

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Project ERASER

CONCEPT OF OPERATIONS

MAXIMIZE SAFETY

- CONDUCT OPERATIONS AT TEMPERATURES <10°C
- GPR SURVEY
- PROVEN TECHNOLOGY, SOPs AND IPE
- HEALTH AND SAFETY PLAN
- AVAILABLE INFRASTRUCTURE

MAXIMIZE ENVIRONMENTAL PROTECTION

- CONDUCT OPERATIONS AT TEMPERATURES <10°C
- CONTAINMENT STRUCTURE

PUBLIC CONSULTATION

- INDEPENDENT RISK ASSESSMENT
- REGULATORY COMPLIANCE
- FORMALIZED ENVIRONMENTAL ASSESSMENT
- OPEN HOUSE

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Project ERASER
PUBLIC CONSULTATION

OBJECTIVES

- STAKEHOLDERS BECOME KNOWLEDGABLE ABOUT PROJECT
- PROVIDE OPPORTUNITIES TO RAISE ISSUES
- STAKEHOLDERS GAIN CONFIDENCE IN PROJECT PERSONNEL

MESSAGES

- WASTE SAFELY STORED
- PUBLIC HEALTH/SAFETY NOT COMPROMISED
- DRES PROJECT TEAM HAS CONSIDERABLE EXPERIENCE
- SAFE AND PROVEN TECHNOLOGY/PROCEDURES USED
- ALL SAFETY AND ENVIRONMENTAL REQUIREMENTS MET
- PUBLIC HAS OPPORTUNITY FOR INPUT
- WASTE WILL BE DESTROYED NOW RATHER THAN IN FUTURE

APPROACH

- ENVIRONMENTAL ASSESSMENT IN PUBLIC REGISTRY
- BRIEF KEY GROUPS, LOCAL OFFICIALS AND AECL STAFF
- DEVELOP BRIEFING NOTES, Q and A's, MEDIA INTERVIEWS
- OPEN PUBLIC MEETING
- ADDRESS ISSUES BEFORE OPERATIONS COMMENCE

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Project ERASER
OUTLINE

PHASE	DESCRIPTION
I **PLANNING**	PROJECT TEAMS AGENCY RESPONSIBILITIES INFORMATION REVIEW ENVIRONMENTAL ASSESSMENT HEALTH and SAFETY PLAN OPERATIONAL PLAN COMMUNICATIONS PLAN
II **ASSESSMENT**	OPTIONS RISK ASSESSMENT DISPOSAL OPTION SELECTION PRE-OPERATIONAL TRIALS PUBLIC CONSULTATION EXCAVATE MG&A BLOCK GPR SURVEY VEC SAMPLING
III **DISPOSAL**	CWA EXTRACTION CWA DESTRUCTION ARSENIC DISPOSAL CONTAINER DEMOLITION SITE DECONTAMINATION

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CONCLUSIONS

OPERATIONS

- AGENT WASTE SUCCESSFULLY DESTROYED
- AGENT WASTE: 33 kg HD, 1.5 kg GB, 20 kg ARSENIC
- NO HEALTH OR ENVIRONMENTAL IMPACTS
- WITHIN BUDGET
- EXCELLENT INTER-AGENCY CO-OPERATION
- UNEXPECTED WASTE INVENTORY

PUBLIC CONSULTATION

- COMMUNICATIONS GOALS ACHIEVED
- NO SIGNIFICANT ISSUES
- POSITIVE MEDIA RESPONSE

BENEFITS

- RECOGNIZED CANADIAN CAPABILITY
- CWC COMPLIANCE
- DRES RESOURCE TEAM
- DND SUCCESS STORY

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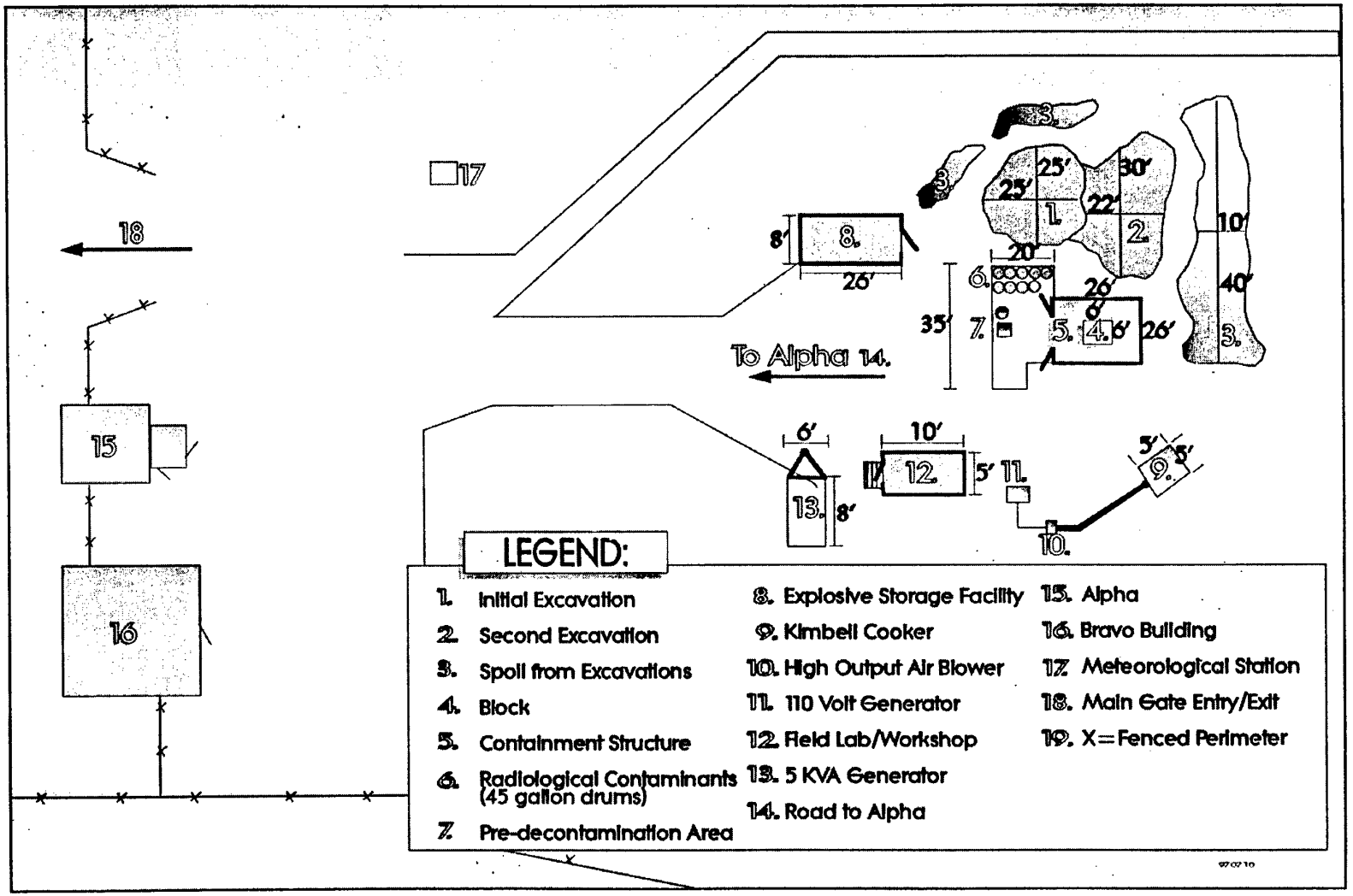
Project ERASER

PROJECT TEAM

NAME	FUNCTION
JM McANDLESS	TEAM LEADER SCIENTIFIC AUTHORITY
K PIRIE	FIELD TECHNICAL MANAGER EXPLOSIVE APPLICATIONS
B KOURNIKAKIS	BIOWARFARE EXPERT
CP ADIE	TECH SUPPORT CHEMICAL SAFETY
WG SOUCEY	TECH SUPPORT RADIATION SAFETY
R TREMBLAY	TECH SUPPORT MILITARY LIAISON

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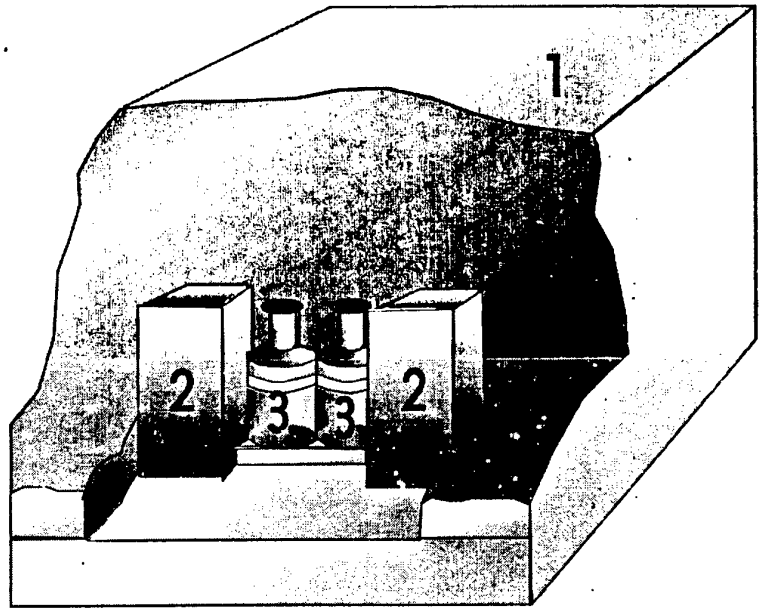
National Defence / Défense nationale

DRDB
DRDD

1. Concrete Block

2. Chlorosulphonic Acid in glass container. Packed in wood.

3. Mustard "H" in "Mine Chemical No. 1" Mk. 1, (1939-45)



DRES
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Canada

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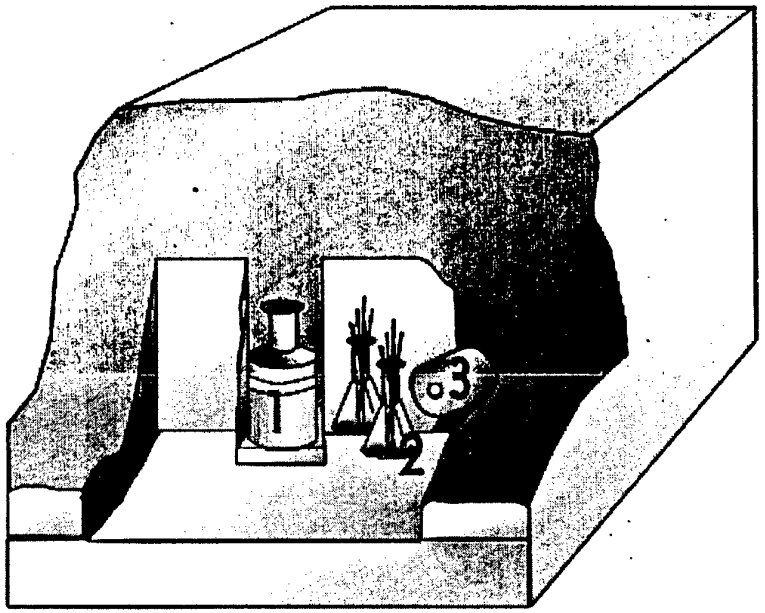
National Defence / Défense nationale

DRDB
DRDD

1. Mine Chemical No. 1. Mk. 1. "H" Mustard

2. Erlenmeyer Flasks (14 vials/13 vials) "H" Mustard

3. Livens Bomb/Projector "H" Mustard



DRES
CRDS

Canada

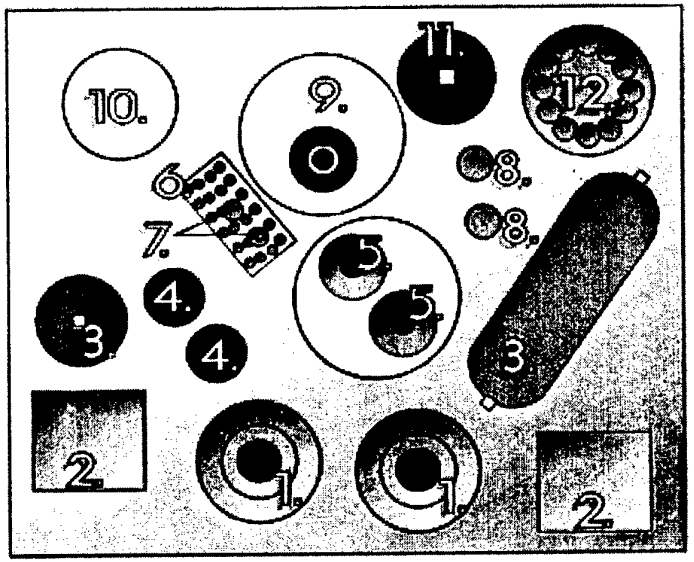
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National Defence / Défense nationale

DRDB
DRDD

1. Mine Chemical No1. MK. 1 " H" Mustard
2. Chlorosulphonic Acid (2)
3. Livens Bomb/Projectors "H" Mustard (2)
4. Zinc Chloride in Metal Cans (2)
5. Erlenmeyer Flasks (2 Flasks with 14 vials 13 vials x 100 ml) "H" Mustard
6. Partitioned Box (24x100 ml Vials) "H" Mustard
7. 250 ml Glass Jars (2) "H" Mustard
8. Projectile, 25 pounder, Base Ejection, Chemical Carrier (GB x 2)
9. Agent Precursor 2 L Reinforced Glass Container (1/2 Winchester)
10. Arsenic Trioxide (5 Gallon Heavy Duty Cardboard Container)
11. Glass Lined Reaction Vessel 1.5 Gallon (US Ordnance)
12. 5 gallon Can with 10x500 ml Glass Jars "H" Mustard



DRES
CRDS

Canada

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DEVELOPMENT OF CW DESTRUCTION TECHNOLOGIES IN THE RUSSIAN FEDERATION

COL Igor N. Krotovich

Ministry of Defence, Moscow, Russian Federation

In the former Soviet Union (FSU) discussions with regard to the destruction of chemical weapons started in the 1970s. The complexity of this problem was caused by the multiplicity of the chemical agents comprising organo-phosphorous compounds, and sulphur- and arsenic-containing substances as well, which all differ in terms of physical, chemical, and toxicological properties, and with regard to related assembled chemical ammunition.
Due to this multiplicity in terms of chemical agents and chemical ammunition as well, rather universal destruction methods had to be developed preferably, e. g. they have to be applicable to all types of agents and ammunitions. In addition, observance of safe working conditions and environmental protection laws are major prerequisites with respect to the implementation of a particular destruction method. Approved methods and procedures with regard to the handling of highly toxic chemical agents are to be taken into account.

Methods for the destruction of organo-phosphorous chemical ammunition

The first destruction activities in Russia were related to chemical weapons based on organo-phosphorous compounds, e. g. nerve agents. Following respective research activities, a mobile destruction facility for chemical weapons had been developed, applying a two-stage chemical agent detoxification process.
During a first step the organo-phosphorous compounds are detoxified in a chemical reactor, the reaction products are incinerated during a follow-on second step. Test results have shown, that this chosen method is suitable to destroy this kind of chemical weapon; in all cases, the agents were completely destroyed and no environmental pollution was encountered.
In 1980 this mobile destruction facility for chemical agents was put into service by the Soviet Army. Between 1980 and 1987 small quantities of chemical ammunition were destroyed with the help of this particular facility. During the course of operation all destruction process parameters were optimized and measures to ensure the protection of the environment had been improved further. Special emphasis was put on both improved disassembly methods for chemical ammunition and on complete decontamination of the resulting metal parts. Approximately 4.000 ammunition pieces had been destroyed; this corresponds to 280 metric tons of chemical agents. This mobile chemical agent destruction facility was presented in Shikhany Chemical Depot to delegates of the Geneva Disarmament Conference in 1987 demonstrating the destruction of an aerial bomb filled with sarin (GB).
As usual, test facilities are operated prior to the construction of large-scale facilities. According to a decision of the FSU government taken in 1986, an industrial test facility for the destruction of chemical weapons should be set up close to the city of Chapayevsk. The facility, comprising of four disassembly lines for chemical ammunition (medium and large caliber artillery shells, rocket launcher projectiles), was put into operation in 1989. The planned average destruction capacity of the facility amounted to 300 to 350 metric tons of chemical agents for continuous operation during a 100 days period (during summer). The disassembly of small caliber ammunition had a severe limiting effect on the throughput of the facility. On the other hand the chemical reactor could cope with 1.000 to 1.500 metric tons of chemical agents, and the capacity of the disassembly lines for medium and large size chemical ammunition (aerial bombs, spray tanks, chemical war heads) would have matched these figures.
The main purpose of this industrial test facility aimed at the evaluation of all chemical agent destruction technologies including environmental monitoring, decontamination procedures, burn-out kilns for munition casings, testing of different measures to ensure workplace safety and protection of the environment. It should be pointed out, that the facility could serve as a large-scale universal testing bench for different decontamination procedures, which could be attributed still to basic research at that time.
In 1989 operational tests were performed using dummy chemical ammunition filled with agent simulants. These tests have proven both the reliability and the effectiveness of the chemical ammunition disassembly lines, of the chemical reactor, and of the control and auxiliary equipments. In spite of these positive results, the facility could not start with the destruction of real chemical weapons due to changes of the overall political situation. The Chapayevsk facility was transformed into a training centre for personnel earmarked for later deployment to future large-scale chemical destruction facilities.
Experiences made during the construction of the Chapayevsk plant have shown, that practicable solutions with regard to all the problems encountered during industrial destruction of chemical weapons will only be possible, unless prior exact evaluation of the technologies involved, taking into account national and international experiences, and unless an agreed national CW destruction programme has been implemented.
Such a programme has been prepared for many years. A first draft of a national programme with regard to the destruction of chemical weapons was submitted to the Supreme Soviet of the FSU in March 1990. The

programme was screened by the FSU Environmental Protection Agency and other state bodies resulting in several recommendations aiming at the improvement of this draft. A revised draft was submitted in 1991. Due to the disintegration of the FSU taking place at that stage the programme was not pursued. Between 1992 and 1995 several newly revised drafts were evaluated, and finally in March 1996 the "Federal Programme for the Destruction of the Russian Federation Chemical Weapons Stockpile" entered into force by governmental decree; in April 1996 the programme was raised to Presidential Programme status.

Creating the technological base to execute this programme includes the following issues:

- Selection and localization of chemical weapons destruction sites
- Selection of environmentally and economically acceptable, safe destruction and waste disposal technologies
- Design and construction of chemical weapons destruction facilities
- Testing of chemical weapons destruction facilities and evaluation of their performance

The right selection of the most suitable CW destruction technology is of fundamental importance. Quite a number of different proposals have got to the Russian Ministry of Defence; roughly speaking, they can be divided into two groups with respect to the technologies involved:

- (1) Thermal treatment of the chemical agents
- (2) Chemical destruction of the chemical agents

The first group comprises:

- Direct incineration of the chemical agents drained from the respective ammunition and thermal treatment of contaminated ammunition casings
- Cryofracture of the ammunition followed by thermal detoxification of the metal parts, evaporation and incineration of the chemical agents
- Incineration of agent vapours emanating from drilled ammunition bodies
- Controlled detonation of the chemical ammunition inside a closed containment
- Different low- and high-temperature plasma technologies combined with the destruction of the ammunition casings at the same time
- Destruction of chemical ammunition in furnaces
- Adiabatic compression
- Destruction of liquid chemical agents by using liquid fuel rocket motors
- Thermal destruction of the chemical agent inside the ammunition
- Destruction of chemical ammunition with the help of nuclear detonation

A separate issue is related to the use of high-energy radiation technologies, e .g. UHF-radiation, for the destruction of chemical agents.

The second group comprises:

- Chemical neutralization followed by special treatment of the reaction products
- Oxydation of the chemical agents using different oxidizers
- Reduction of the chemical agents using different reducing agents
- Micro-biological detoxification methods
- Chemical detoxification of the chemical agents followed by treatment of the reaction products with bitumen (the modern Russian two-stage chemical agent destruction method)

Due to the large number of destruction technologies proposed, a stepwise evaluation was required in order to determine to which extent the following prerequisites were met:

- Has the complexity of the problems related to the destruction of chemical weapons been taken into account?
- Which stage of development has been reached with regard to the different destruction methods proposed? (e. g. test facility for the destruction of agent simulants or of live agents, design data for a destruction facility, feasibility study, pilot plant, complete documentation)
- To which extent has the particular destruction technology been tested with live agents?

Of the known destruction technologies two approaches, (1) the direct incineration, and (2) the two-stage method comprising of chemical detoxification followed by treatment with bitumen, meet the nowadays requirements These two methods have been evaluated and tested quite extensively; the first one is applied in the U. S. and gets developed further there, while the second one is earmarked to be used in Russia.
Direct incineration is a proven technology and has been chosen therefore for implementation in Germany, in the U. S., in Canada, and in the U. K. as far as the destruction of old and stockpiled chemical weapons is concerned. The continuously running incineration process requires appropriate control and monitoring systems with regard to the process itself, the delivery systems for the chemical agents, the fuel and air/oxygen supply including a sophisticated but reliable feed-back system which ensures the proper function of the chosen incineration process

at all times. To enable this continuously running incineration process mentioned above, rather large amounts of munition-free chemical agents have to be available at any time.
On the other hand, chemical detoxification usually requires less energy and probably less sophisticated equipment; this might facilitate the design and the construction of the destruction facility concerned. Of special importance is the decrease in temperature which goes along with chemical detoxification. High temperatures accelerate corrosion; e. g. fluorine compounds resulting from the destruction of organo-phosphorous nerve agents will cause problems at temperatures higher than 200 °C with regard to the choice of material for reaction vessels, pipe systems, and other appliances. Lower temperatures in the range of 100 to 150 °C allow for much more flexibility in terms of material and industrial equipment. The "soft" chemical detoxification process renders possible the use of known proven technology and equipment, which also contributes to a high safety standard.
In 1985, following governmental orders, the Russian Ministry of Defence invited tenders for suitable technologies to destroy organo-phosphorous nerve agents. A group of experts coming from state and respective regional government agencies, universities, and from the industry concerned was tasked with the evaluation of the offers submitted. Main priorities were given to safety, environmental protection issues, technical maturity, consumption of energy and other resources, economical feasibility, and available test data with regard to chemical agents. According to the evaluation, the best choice resulting was the two-stage chemical detoxification process developed by specialists from the GOSNIIOCHT research institute.
At the same time joint Russian - U. S. laboratory tests were conducted to evaluate this two-stage chemical detoxification approach also, applying different modern analytical methods by which one could demonstrate, that no chemical agents were present after the final treatment step with bitumen. The test results were checked by an independent group of scientists under the auspices of IUPAC. According to the outcome this two-stage process was declared suitable for the destruction of organo-phosphorous nerve agents.

The Russian two-stage chemical agent destruction method.

This approach comprises of the separate destruction of chemical weapons components, i. e. the chemical agents and the munition bodies or casings.
The essential issues are (1) a two-stage destruction of the agent (chemical detoxification followed by bitumen treatment of the reaction products), and (2) a two-stage treatment of the munition bodies (chemical decontamination followed by thermal treatment of metal parts).
During the first step, the agent is drained from the munition body and is subjected to a soft and strictly controlled chemical treatment subsequently, causing the formation of reaction products which are not any longer suitable for military purposes. In case of G-agents monoethanolamine is used, in terms of V-agents an alcoholate solution is applied.
In a second step, bitumen is added to the reaction products resulting from the first step, leading to the formation of bitumen-salt conglomerates which can be safely stored for good at waste disposal sites.
The munition bodies and casings will be subjected to a two-stage destruction process also. First, simultaneously with the agent draining system the emptied munition bodies will be treated with a decontamination solution, while in a second step, remaining agent residues are destroyed by thermal treatment.
The final products resulting from this destruction process are safely disposable bitumen-salt conglomerates, and clean metal scrap, which can be used as secondary raw material for the metal industry.

Technologies for the destruction of blister agents.

Chemical reaction features of blister agents are well known, but not all of the blister agent decontamination reactions are suitable with respect to an industrial application. The development of industrial methods for the destruction of blister agents (Lewisite, Mustard, and mixtures of the two) started in 1980s, resulting in proven technologies which can be used today.

Methods for the destruction of Lewisite

a) Melting with sulphur
Lewisite reacts with molten sulphur (present in excess) at high temperatures forming a solid polymerisate suitable for final disposal at a waste site. The most favourable process conditions are: 1 : 3 mass ratio of Lewisite to sulphur, reaction time 30 to 40 min, temperature 160 to 180 °C. After cooling down, the polymerized reaction product is cut into blocks for final disposal. Wash out of arsenic is less than $6 \cdot 10^{-6}$ g/cm² per day.

b) Polymerization
The approach is based on the reaction of Lewisite with 2-Hydroyethyl-methacrylate in the presence of acceptors for hydrogen chloride, followed by polymerization of the reaction products. The process is similar to the molten sulphur approach, it requires lower temperatures (60 to 90 °C) but a prolonged reaction time (60 to 80 min).

c) Chlorination
Lewisite is subjected to chlorine vapour at 110 to 130 °C leading to the formation of both arsenic trichloride and a rather complex mixture of chlorinated hydrocarbons containing 1.2-Dichloroethene, 1.1.1-Trichloroethene, 1.1.2-Trichloroethane, and Tetra-, Penta-, and Hexachloroethane as well.

d) High temperature oxidation
Lewisite can be reduced to metallic arsenic in one step in the presence of excessive hydrogen at 900 to 1000 °C. A complex mixture containing saturated and unsaturated hydrocarbons, hydrogen chloride and soot is formed as by-product.

e) Treatment with alcoholates
Reaction of Lewisite with sodium alcoholates causes the formation of the following products: trialkylarsenites, acetylene, and sodium chloride. Reaction parameters are: 50 to 80 °C for 2 to 3 hours, 1 : 3.6 mass ratio of Lewisite and sodium alcoholate. The trialkylarsenites formed will be reduced to metallic arsenic.

d) Reduction with ammonia
This is a one-step Lewisite destruction reaction leading to the formation of metallic arsenic. Lewisite and ammonia are fed into a flow reactor and are heated to 650 to 700 ° C. The created vapour-aerosol mixture gets cooled down with water, the metallic arsenic yielded is filtered and washed with a solution of ammonium chloride. By means of evaporation, the ammonium chloride is recycled and can be reused again.

e) Hydrolysis in an alkaline solution followed by electrolysis
This Lewisite destruction process can be divided into three steps: (1) the alkaline hydrolysis, (2) the follow-on electrochemical reduction of the reaction mixture yielded, and (3) purification of the crude metallic arsenic to yield high quality arsenic.
In the first step Lewisite is subjected to an alkaline hydrolysis at approx. 100 °C; this is followed by an electrolysis yielding technically pure metallic arsenic and sodium hypochlorite as well. The reaction solutions can be recycled if required. This type of destruction process seems to be most favourable in terms of ecological safety issues. The follow-on purification of the yielded crude metallic arsenic comprises of a chlorination step of the sublimated pulverized arsenic leading to the formation of arsenic trichloride, which gets reduced with hydrogen to yield very pure metallic arsenic after another sublimation step.

In June 1994 the different Lewisite destruction technologies were evaluated by the Russian MOD. A team of experts decided the alkaline hydrolysis followed by electrolysis to be the first choice amongst the different destruction methods proposed; reduction with ammonia was rated second choice, while treatment with alcoholates was rated third choice.
The first and second choice Lewisite destruction methods as mentioned here are considered for possible implementation at the earmarked Gorny (Saratov Oblast) Lewisite destruction facility.

The Destruction of Mustard

The chemical agent which has been investigated most is sulphur mustard definitely; well proven methods for its destruction are available.
High temperature oxidation of mustard by means of incineration is a highly effective destruction method leading to the complete disintegration of the mustard molecule. But, this process gets a negative touch with regard to the possible release of dioxines and benzofuranes involved. High temperature pyrolysis (or subjection to plasma treatment) eliminates this particular disadvantage. The reaction of mustard with a hydrogen peroxide solution yielding bis(2-chloroethyl)-sulfoxide seems to be quite tempting, because this reaction product is used in flotation processes as means to increase the flotation effectivity when extracting metals from flotates of sulfide ores containing copper and nickel. In addition, very effective sorption agents for gold, platinum, palladium, and mercury can be created on the basis of bis(2-chloroethyl)-sulfoxide and sodium polysulfides, which can extract these precious metals from weakly concentrated solutions.

The following Mustard destruction methods have been developed in Russia:

a) Thermo-chemical destruction
This process is based on the reaction of Mustard with a mixture of monoethanol-amine and ethylene glycol (ratio 9 : 1) at 100 to 140 °C under permanent stirring for 2 hours; the residual Mustard concentration in the

reaction mixture is less than $6.4 \cdot 10^{-3}$ %. To destroy 1 metric ton of Mustard, 1.1 metric tons of this reaction mixture are required. The use of ethylene glycol allows both better control of the exothermic reaction and faster dosing of the reactants. In a second step, the final reaction mixture is incinerated. The very low amount of resulting remnants (which would have to be delivered to a waste site for final disposal) is of particular advantage.

b) Combined chemical detoxification and bitumen treatment
Opposite to the previously mentioned Mustard destruction approach, the follow-on incineration step is avoided here and is replaced by treating the reaction mixture with bitumen, yielding a bitumen-salt mixture which is suitable for final disposal at a waste site. This technology has been tested before already. To increase the stability of the bitumen-salt mixture, and to recycle the monoethanolamine used in the process, potassium hydroxide is added during the bitumen treatment, which runs at 110 to 120 °C. The mass ratio of the reactants is as follows: reaction mixture : bitumen : potassium hydroxid = 1 : 1.5 : 0.2. Finally the reaction temperature is raised to 180 °C causing the formation of a monolithic polymeric bitumen-salt mixture, which is quite stable with regard to water (solubility in water at 100 °C amounts to 1.5 to 1.7 %). The process described here has been recommended for implementation at Gorny (Saratov Oblast) chemical weapons destruction facility.

Destruction of Mustard - Lewisite - Mixtures

Gorny chemical agent storage site in Saratov Oblast contains Mustard, Lewisite and Mustard-Lewisite - mixtures as well. Therefore, for practical reasons, it deemed appropriate to develop suitable destruction procedures which could accommodate both single chemical agents and mixtures of these chemical agents.

a) Treatment with molten sulphur
Mustard-Lewisite - mixtures are subjected for two hours to a melting bath of sulphur at 140 to 150 °C in the presence of N-methylpyrolidon which binds the hydrogen chloride formed. The final reaction product is a polymeric compound of rather unknown structure which solidifies between 90 and 100 °C. In spite of simple technical equipment and availability of starting products, the process does not meet environmental protection requirements.

b) Two-stage polymerization process
In a first step the Mustard-Lewisite - mixture is treated for 2 to 2.5 hours with 2-Hydroxyethyl-methacrylate at 50 to 70 °C in the presence of tertiary amines to bind the hydrogen chloride formed. Afterwards the temperature is raised to 90 to 100 °C and monoethanolamine is added to ensure a residual Mustard concentration in the range of $5 \cdot 10^{-3}$ %. In the follow-on second step the reaction mixture is kept for three to four hours at 80 to 100 °C in the presence of a radical forming catalyst (2,2'-Azo-bis-isobutyronitril) to initiate polymerization, resulting in a solid polymer which can be disposed of safely at a waste site. Of particular advantage of this process is (1) simple technical equipment, and (2) most of the blister agents stored in bulk containers there can be destroyed. Unfortunately, this process is not adaptable to the destruction of Mustard-Lewisite - mixtures kept in dichloroethane in Gorny (Saratov Oblast) as well, as dichloroethane would inhibit the required follow-on polymerization reaction.

c) Reduction with ammonia
This destruction process is suitable for both pure Mustard-Lewisite -mixtures and for mixtures of these agents in dichloroethane. Compared to the destruction of pure Lewisite using this method (as described earlier), similar technical equipment and process parameters apply for the destruction of these Mustard-Lewisite - mixtures.

d) Treatment with bitumen
The destruction method is identical to that described earlier with regard to Mustard.

Ensuring the safe destruction of chemical weapons

One of the most important requirements with respect to running chemical weapons destruction facilities is to ensure the safety for both the plant personnel involved and the population living close to these installations.
The finally implemented chemical weapons destruction technology, hence the appropriate technical equipment including a reliable system of measures to prevent failures and accidents, has to guarantee the necessary safety standards per se.
In addition, safety is ensured by different organizational and technical measures, continuous agent monitoring and regular analytical checks inside the destruction facility, strict adherence to workplace safety rules and an overall environmental monitoring regime.

DESTRUCTION OF OLD CHEMICAL WEAPONS IN AUSTRIA

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Task

In the last 20 years, in particular as a result of improved detection devices, there were discovered in total 123 shells with presumable filling of chemical agent, in the closer vicinity of a former ammunition factory. These shells are known as L.F.H. Grenade 38 Nb, caliber 105 mm designated with an impact fuse (kl.A.Z. 23 Nb). The military name is "Grünring 1", usually containing nitrogen mustard.

This scattered ammunition was in a bad condition caused by the long storage down in the ground. The igniters were corroded strongly and not removable anymore. Since the shells were no more secure for transportation, the destruction had to be carried out in the immediate vicinity of the stock location. The intention was to built a plant in the field, with low expenditure and minimal preparatory works. After the project, the plant should be dismantled in complete again, without permanent influence to the environment.

Organization

A field method was planned to open the shells, and to remove the chemical agent with subsequent decontamination. The ammunition parts with explosive charge and fuse should be decontaminated and disposed in a separated manner.

Disposal was guided by the ministry of interior in cooperation with the ministry of defense. The tasks were divided up between 3 teams of specialists:

Team 1 was responsible for the transportation of the shells from the bunker to the container, and for opening them. At the end this team had to blast the decontaminated central body containing the explosive charge.
Team 2 decontaminated the ammunition parts and guaranteed the decontamination of the chemical agent. Moreover, they had to take samples and analyze them to determine the filling and also to observe the decontamination process.
Team 3 had to check the contamination of persons and devices and to carry out decontamination measures as well as they had logistic tasks and to establish and maintain security measures.

The realization was scheduled for the cold season (fall/winter).

Preparatory works

Some preparatory works were necessary in the beginning. Each shell was screened by X-rays to determine the filling height. Analysis of the contents according to known means was not required for most of the shells.

As it turned out later, the main part of the shells contained in fact nitrogen mustard, partial in very pure form (> 95%), often with a lot of deposits too. The contents of the 123 shells divides up as follows:

nitrogen mustard (tris (2-chloroethyl) amine or HN-3)	102
empty or covered with a layer of rust inside	15
smoke ammunition (SO_3)	4
unknown solid material	2

No sample could be taken, from the first of these two unknown shells, The content was a pale brown substance which was hard as a rock. From the second unknown shell, the main-content of the hardened white substance, could be determined as phosphite, phosphate and pyrophosphate. Chemical warfare agent was detected at none of these, therefore they could be blasted without further measures.

A container was especially arranged for opening the shells under remote control and the work with warfare agent. An air suction and filter system should guarantee the rapid removal of smoke and warfare agent vapor. A video system was installed to supervise the course of events from a central office and if required to communicate with the persons inside the container.

Methods for conversion of nitrogen mustard with chemicals into less harmful products are well-known by literature. The aim of investigations was to find out a suitable decontamination process. This field method should also work out as simple as possible, also at low temperatures with only a few aids needed.
Among the possible chemicals the fixing salt (Sodium thiosulphate) was selected since it is simple to handle and also easy available. Thiosulphate substitutes the atoms of chlorine of the nitrogen mustard in quantitative amounts. An attachment product is formed. In order to assure a better conversion the influence of solvents on this reaction was examined. Good results were achieved by adding isopropyl alcohol. Since the combustion of the detoxicated warfare agent is expensive, it was attempted to work with concentrations as high as possible.

Fig.1 shows the degree of decontamination after 2 days with different ratios of water and alcohol and equimolar amounts of thiosulphate. Since the tests should be close to real conditions, a temperature of 10 °C was chosen, and the chemicals were mixed and stirred only a short time each day. The influence of the higher content of alcohol at low concentrations of mustard, can be obviously seen. In these cases, a nearly homogenous solution is formed. At higher concentrations, nitrogen mustard separates down to a second phase.

Fig. 2 shows the result after 7 days at the same conditions with a various doses of alcohol. A complete substitution of chlorine was obtained with a content of 25 % alcohol.

The addition of sodium hydrogen carbonate for buffering the pH-value showed no influence on the reaction-rate under this conditions.

Safety measures

The opening of the shells was only performed above a wind speed of 2 m/s to achieve a sufficient distribution and a dilution of the warfare agent cloud in the case of a possible uncontrolled blast. However, this led to longer delays since often no wind came up.
NBC protection was prescribed for all persons working at the area. A heavy, rubber protection suit was worn of all those, in direct contact with the warfare agent. Additional a light protection suit, was put on over it only for single use. It turned out as more economical to dispose them after use since the rubber protection suit has not to be decontaminated separately.
A medical team was available during the entire operation for immediate actions.
The ground-area in which was worked on the chemical agent, was always covered with films of polyethylene to reduce the danger of ground contamination. It could be confirmed that this measure was sufficient enough. In the case of later measurements, no warfare agent could be detected in the endangered area.
For safety reasons inflammable solvents, like alcohol were added only outside of the container.

Realization

The work flow is topic of a short movie.

A time frame of 3 weeks was scheduled for the completion of our work.
Already after opening the first shell, it turned out that this schedule was chosen too optimistically. During opening, the fluid warfare agent still escaped in addition to strong formation of smoke. The following cleaning works took a lot of time.
About 10% of the filled shells were under an internal pressure, which caused liquid to splash uncontrolled while opening the shells. The chemical agent escaped almost completely and contaminated the machine. The reason for this strong internal pressure is unknown. In some of these shells, the warfare agent was already in a decomposing state, in other cases pure nitrogen mustard without detectable conversion products was found.
The remaining residues on the ammunition parts were removed with simple tools and non-flammable solvents like dichloromethane. However, the conversion slowed down very much in the presence of this solvent. It was attempted to handle dichloromethane as economically as possible and to volatilize it as far as possible.
A cleaning with organic solvent alternating with diluted decontamination solution turned out to be effective. The aqueous solution was especially favorably for cleaning the central cylinder on which the metal was coated with a thin layer of polyvinyl alcohol, that restrained the warfare agent very well from removal.

Only concentrated nitric acid and chromic sulphuric acid are proved effective agents for rapid decontamination of small lots of nitrogen mustard. Chromic sulphuric acid was used for the final cleaning of the shell cases on which hardly removable crusts had often formed.
The main quantity of the nitrogen mustard was manually poured into barrels of polyethylene (200 l) for further decontamination. After the conversion, the barrels with the reaction products were supplied into a civilian combustion plant for toxic wastes. This plant had in addition to the suitable incineration technology and the necessary systems of emission control also the continuous air surveillance needed for the accurate disposal.

After bursting the ammunition parts, soil samples from the blasting area were checked for possible residues of nitrogen mustard in the laboratory. With residues on the ammunition parts above 3 bars (chemical agent monitor) we were able to detect traces of nitrogen mustard in the soil after the bursting. Below this value no traces of the warfare agent remained.

Conclusion

About 20 persons were constantly employed in this process. During our work, we could dispose two shells in the morning and further two in the afternoon. The initially scheduled eight piece a day could not be kept. For safety reasons only four shells were simultaneously taken to decomposition site. Also in order to have enough time for the corresponding cleaning during the already mentioned contamination, caused by escaped warfare agent.

Although the mission could be completed successfully and without accidents, however some steps in this procedure are supposed to be improved. The restriction of only one working-container for opening and cleaning the shells, should localize the possible contamination to a small area. The lack of an own container for decontamination tasks was disadvantageous for the work flow and for the decontamination procedure. Especially the removal of remaining quantities of warfare agent in form of solid deposits was very time-consuming.
By the connection of a further container for decontamination tasks via a floodgate, the working procedure would have been able to be sped up. The recovery of solvents, used for cleaning, is possible to reduce the material-flow.

The annihilation action took 13 weeks of work and ended in March 1997.

NEW CONCEPTS FOR THE DESTRUCTION OF OLD CHEMICAL WEAPONS (OCW)

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1. Introduction

This presentation is a joint work between the companies Goepfert, Reimer & Partner Consulting Engineers Ltd. and Hazard Control Ltd.

Goepfert, Reimer & Partner has experience in the field of chemical warfare destruction for more than 20 years now as we did the general planning for the chemical warfare destruction plants Munster one and two.

Hazard Control has longterm experience in the field of dismantling of old chemical weapons.

The chemical weapons convention (CWC) was opened for signature in January 1993. It entered into force at 29th of April 1997. Until now 168 states signed and already 107 states ratified the contract.
The CWC bans production, stockpiling, development, use, retention and the transfer of chemical weapons. It requires the destruction of all chemical weapons. This includes the destruction of old and abandoned chemical weapons. The CWC prohibits in former times commonly used destruction techniques as sea dumping, open-pit burning and land burial.
The time schedule shows that all chemical weapons must be destroyed 10 years after the chemical weapons convention enters into force. This means until the year 2007. This deadline maybe extended by 5 years under special circumstances upon approval of the conference of State Parties.
Old chemical weapons are defined by CWC as weapons which were produced before 1925 or chemical weapons produced in the period between 1925 and 1946 which are in such a bad condition that they can not be used as weapons any more. Abandoned chemical weapons are defined as chemical weapons including old chemical weapons abandoned by a State after 1st of January 1925 on the territory of another State without consent of the letter.

The destruction of old and abandoned chemical weapons is a challenging task as a lot of countries are concerned as for example Belgium, China/Japan, France, Germany, Italy, Russia and probably many others.

2. Destruction of Old Chemical Weapons

There are a lot of special problems in the destruction of old and abandoned chemical weapons which can not be compared with the destruction of properly stockpiled weapons.

At first unknown production conditions have to be considered, this means that unknow production technologies have been performed. The mixture of chemical warfare agents is for many weapons not exactly known. A major problem are changes during the production due to war restrictions. This means that even chemical weapons which look the same may contain different warfare agents or different explosives. Especially savings in the production of bursters and fuses during the war lead to special dangers in the handling of these weapons.

A further problem is the unknown storage conditions as the chemical weapons may have been burried for many years. This may lead to cracks in the shells. Many shells show deformation and corrosion. The leackage of agents makes the handling of these weapons very dangerous. Water penetrated into the shell during the years. Even the thermal storage conditions may vary in a wide range between -40 °C upto +50 °C.
For destruction technologies this means that there will be unknown types of ammunitions. The state of shells, explosives and chemical warfare agents is mostly unknown and can differ from shell to shell.

Considering all these points it means for a destruction technology, that all manual handling must be minimized as far as possible.

3. Overview on Technologies

3.1 Disassembly Technologies

The advantages and disadvantages of different technologies for disassembly of chemical weapons can be seen in figure.

The manual disassembly which includes drilling, sawing and milling has the advantage that all types of ammunition can be accessed. By the years the personnel gets a lot of experience. The main disadvantages are the high risk for the personnel and the very low throughput of this technology. A lot of highly qualified personnel is required.

Cryofracture is the deep-cooling of shells in fluid nitrogen or oxigen and the hydraulic fracture of the shell. This technology is not specific to ammunition types and reduces handling steps. Disadvantages are that the explosives are after the treatment still active and that some explosives can even get more sensible by a treatment with deep temperatures. Furthermore cryofracture is technically demanding.

Waterjet cutting means that burster and fuse are cut off by abrasive materials in one operation. This technology also reduces handling steps and it even leads to an inactivation of the explosives. As disadvantages we see that there is still a certain risk of explosion and that water and abrasives are contaminated by chemical agents. A lot of maintenance may be needed due to the abrasive materials.

While the previously described technologies lead to a separation of explosives and chemical agents, the technologies of detonation chambers and rotary kiln follow the idea of a one-step detonation.

In a cold detonation chamber the explosives are forced to detonation in an armoured kiln by external ignition. The advantages of such a technology are the low energy consumption and the flexibility to different types of ammunitions. Due to the fact that there are no rotating parts such a chamber is easy to seal. The disadvantage of this technology is that the remaining scrap is still contaminated with chemical agents. The external ignition may require manual handling with the ammunitions. The throughput of this technology is expected to be quite low due to the fact that only 1 shell at a type can be treated.

In a rotary kiln the explosives are forced to detonation in an armoured kiln by heat. The technology is also not specific to ammunition and allows higher throughput. It is a disadvantage that the rotating parts must be sealed specially. The residence time in a rotary kiln is quite short. The scrap may still be toxic and the energy consumption is quite high.

With the hot detonation chamber the explosives are forced to detonation in armoured kiln by heat as well. Due to the fact that there are no rotating parts such a chamber is easy to seal. The high residence time up to 20 hours and the high temperature between 400 and 500 °C lead at least to a surface decontamination of the material according to the 3X definition of the US-Army. This technology is also not specific to certain types of ammunitions.

As disadvantages the high energy consumption can be named and the fact that the scrap may still be toxic concerning arsenicals. But considering the high temperature and high residence times the handling of the residues will be certainly less dangerous.

3.2 Destruction Technologies

The following overhead shows the suitability of different destruction technoligies to the treatment of metal parts, explosives, chemical agents, dunnage and finally the applicability to whole munitions.

The green symbol means that the technology is suitable, grey stands for conditionally suitable and the red minus indicates that a technology is not suitable for a certain material.

In a hearth-van system as it can be seen here in Munster at plant 1, no explosives can be treated and consequently no whole munitions can be destructed.

A rotary kiln is a very flexible treatment unit, as metal parts, chemical agents and dunnage can be treated. The presence of explosives is possible in armoured kilns, even the destruction of whole munitions may be possible, although two rotary kilns might be needed.

Due to the nature of fluidized bed combustors metal parts and whole munitions can not be treated.

A plasma furnace seems to be suitable or conditionally suitable for all types. Although whole munitions can not be treated in any case.

It can be seen that most of the Alternative Systems as neutralization, super critical water and wet air oxidation are spezified for the treatment of explosives and the chemical agent. Just the molten metal pyrolysis leads to the same range as the classical incineration systems.

The different destruction technolgies were analyzed regarding the operation conditions as for example temperature level, throughput, experiences. Furthermore it was investigated which technologies require either a dilution of agents or a flue gas treatment as these treatment steps are very costly. The description of the symbols is given here, red colour means a negative effect as a high required energy input for all incineration systems or a low throughput for the hearth-van.

A major disadvantage of many alternative technologies is the quality of the residues, which mostly are still toxic or hazardous and require special disposal or further treamtent. Some technologies even increase the mass that has to be disposed.

4. Description of Concepts

In a flow-chart the two different destruction strategies are compared. In the top the problem of the old chemical weapons can be seen. At the bottom is the goal, the environment-friendly disposal of the residues. The two colours on the left and the right side stand for two different ways to achieve that goal.

On the right side in blue colour the proposed way can be seen. A one-step detonation technologie leads to contaminated scrap and maybe dunnage. These residues have to be treated with a thermal destruction technology as for example a rotary kiln or a plasma furnace, which leads to non-hazardous residues.

The left side in grey-colours shows an alternative way. Separation technologies lead to different flows of chemical agents, explosives, contaminated scrap and dunnage. For chemical agents and explosives a chemical treatment is possible, while contaminated scrap and dunnage must be treated by thermal techologies.

Furthermore it must be asked, if the residues of a chemical treatment can be disposed environment-friendly or if it is still necessary to go to further treatment.

Considering that a thermal destruction step might be needed even if alternative technologies are preferred it should be eaysier and in the end less costly to go the way of one-step detonation for disassembly combined with thermal destruction.

4.1 Semi-Mobile Plant

Consequently following these considerations the idea of a semi-mobile plant is presented. This plant is designed to operate independently on sites where the operation of a stationary plant would not make sense and on the other hand there are to many shells and the site is to remote to allow transportation.

Such a plant, shown by the simpified scheme, should consist of the following units:
excavation unit, inititial inspection unit and a small intermediat storage unit. A thermal disassembly will be carried out by a one-step detonation chamber. A secondary combustion chamber will be needed to achieve a total destruction of gaseous chemical agents. The further flue gas treatment depends on the regulations of the country and should consist at least of a scrubber or a dry adsorbens unit with a filtering system.

The residues of this process are the still toxic scrap. However it is for sure that further transportation of the residues to a final destruction plant poses not a problem or risk to the population.

4.2 Stationary Plant

Finally the idea of a stationary plant for the destruction of old chemical weapons is sketched. This concept consists of an excavation unit, an initial inspection unit and an intermediate storage unit at the burial site.

A special transportation unit brings the chemical weapons to the plant site, where a final identification is performed. A main intermediate storage unit allows the storage of different types of ammunitions.

A thermal disassembly with one-step detonation makes sure that no explosives remain before the residues, mainly contaminated scrap is conveyed to the thermal destruction unit. A manual disassembly unit should be installed to make sure that unknown types of ammunitions can be dismanteld and examined.

The thermal destruction unit allows futhermore treatment of residues from a remote plant and the treatment of contaminated waste and other residues like dunnage. After the destruction unit a secondary combustion chamber and a further fluegas treatment have to be installed.

As residues of the entire process we just get slag, which may have a high content of arsenicals, but can be disposed environment-friendly as this slag is not hazardous. In the case of a plasma-furnace the quality of the slag is expected to be that high that it might even be used as building material for example for road constructions.

5. Summary

The special problem in the destruction of old and abandoned chemical weapons is the unknown condition of each single shell. This requires technologies with a minimum of manual handling.

An overview on technologies for disassembly and destruction showed that thermal treatment is most flexible and may reduce treatment steps.

The shown concept of a semi-mobile plant consists mainly of a thermal one-step detonation chamber and a flue gas treatment. This leads to non-explosive hazardous waste, which is suitable for tranportation and further treatment.

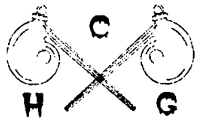
The main components in the concept for a stationary plant are a thermal one-step detonation, followed by a high-temperature treatment as for example plasma-furnace or rotary kiln and a flue gas treatment. This concept minimizes the overall amount of residues and the still remaining residues are environment-friendly disposable.

New Concepts for the Destruction of Old Chemical Weapons

GRP

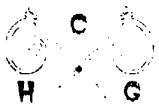
Goepfert, Reimer & Partner Consulting Engineers Ltd.

Dr. Karsten Wilbrand
Peter Hillebrand



Hazard Control Ltd.

Alfred Krippendorf
Dieter Miersch



Structure of Presentation

GRP

1. Introduction

2. Problems of Old Chemical Weapons

3. Overview on Technologies

   - Disassembly Technologies
   - Destruction Technologies

4. Description of Concepts

   - Semi-Mobile Plant
   - Stationary Plant

5. Summary



Chemical Weapons Convention (CWC)

GRP

Chemical Weapons Convention (CWC)

History and Current state of CWC
- opening for signature in January 1993
- entered into force 29 April 1997
- 168 states signed (18 February 1998)
- 107 states ratified (18 February 1998)

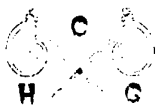
Bans
- production
- stockpiling
- development
- use
- retention
- transfer

of chemical weapons.

Requires
- destruction of all chemical weapons
- destruction of old and abandoned chemical weapons

Prohibits common "destruction techniques" as
- sea dumping
- open-pit burning
- land burial

Time Schedule
- destruction of all CW 10 years after CWC enters into force
- under certain circumstances extension of 5 years (approved by State Parties of CWC)



Definition

GRP

Old Chemical Weapons

- CW produced before 1925
- CW produced between 1925 and 1946 which are in such a bad condition that they can not be used as weapons any more

Abandoned Chemical Weapons

- "Chemical weapons, including old chemical weapons, abandoned by a State after 1. January 1925 on the territory of another State without consent"

Concerned Countries (i.a.)

- Belgium
- China / Japan
- France
- Germany
- Italy
- Russia
- others



Problems of Old Chemical Weapons

GRP

Special Problems in the Destruction of Old and Abandoned Chemical Weapons

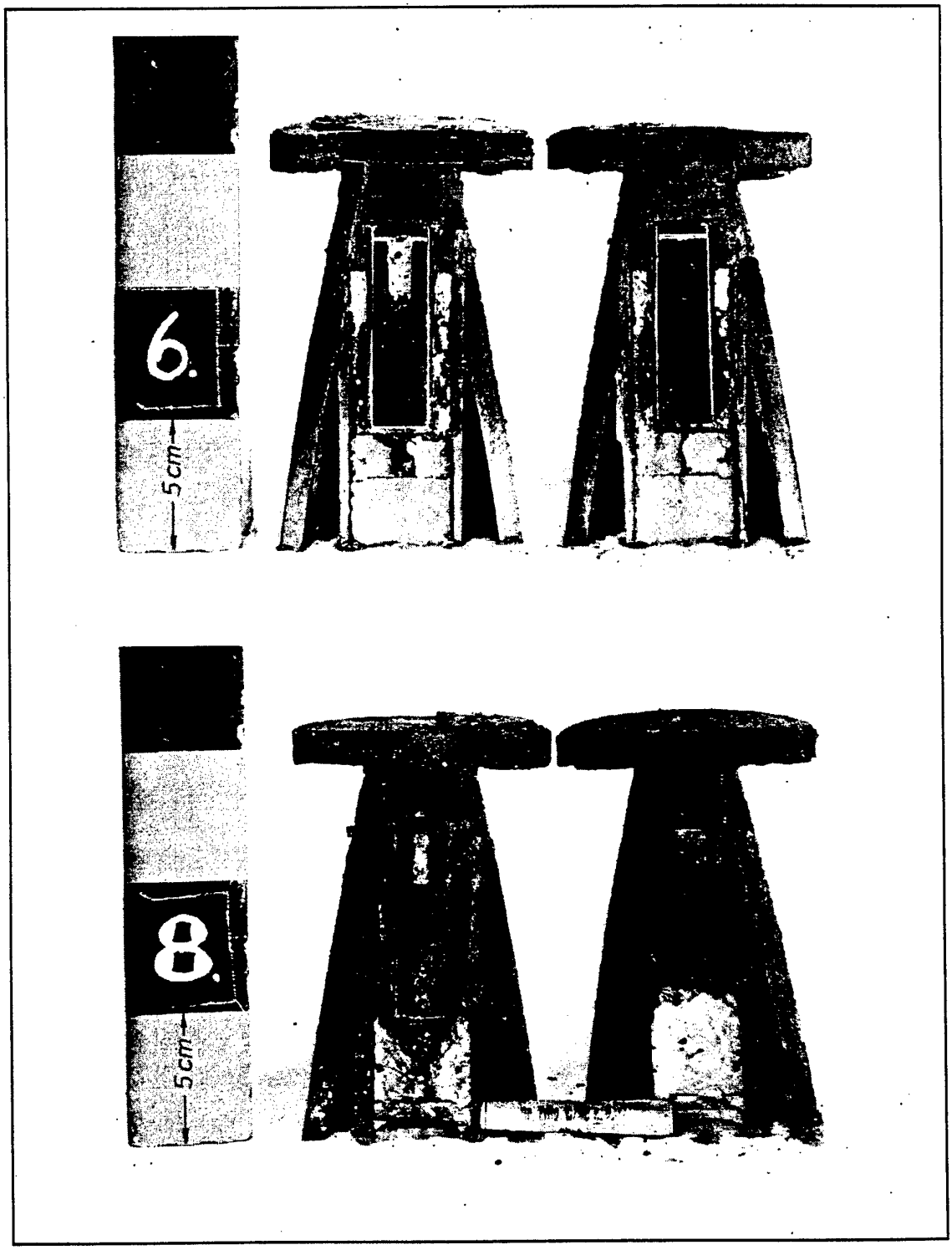
Unknown Production Conditions
- unknown production technology
- unknown mixture of chemical warfare agents
- changes during production due to war restrictions

Unknown Storage Conditions
- cracks in shells
- deformation of shells
- corrosion of shells
- leakage of agent
- water penetration
- thermal storage conditions (-40°C to +50 °C)

Meaning for Destruction Technologie
- unknown munitions types
- unknown state of shell
- unknown state of explosives
- unknown state of chemical warfare agent

⇨ **minimize manual handling !**

Ammunitions GRP





Suitability of Destruction Technologies

GRP

Technology	Suitability				
	Metal Parts	Explosives	Chemical Agents	Dunnage	Whole Munitions
Incineration Systems					
hearth-van	☑	-	☑	☑	-
rotary kiln	☑	☑	☑	☑	☑
fluidized bed	-	☑	☑	☑	-
plasma furnace	☑	☑	☑	☑	☑
Alternative Systems					
chemical neutralization	-	-	☑	-	-
supercritical water	-	☑	☑	-	-
wet air oxidation	-	☑	☑	-	-
steam gasification	-	-	☑	-	-
molten metal pyrolysis	☑	☑	☑	☑	☑
molten salt oxidation	-	☑	☑	-	-

Description of symbols		
	☑	Suitable technology
	☑	Conditionally suitable
	-	Not suitable

Operation Conditions of Destruction Technologies

GRP

Technology	Temperature	Pressure	Throughput	Quality of Residues	Energy Input	Personel	General Experience	Experience with CW	Dilution of Agents Required	Gas Treatment Required
Incineration Systems										
hearth-van	O	O	O	O	O		O	O		☑
rotary kiln	O	O		O	O	O	O	O		☑
fluidized bed	O	O	O		O	O	O	O		☑
plasma furnace	O	O	O	O	O	O	O			☑
Alternative Systems										
chemical neutralization	O	O		O	O	?	O	O	☑	
supercritical water		O		O		?	O	O	☑	
wet air oxidation		O		O		?	O		☑	☑
steam gasification				O	O	?	O	O		
molten metal pyrolysis	O	O	O	O	O	O	O			☑
molten salt oxidation	O	O	O	O	O	O				☑

Description of symbols		
	O	negative effect
		medium
	O	

Summary

GRP

Problems of Old Chemical Weapons

- unknown production conditions
- unknown storage conditions

□ unknown state of ammunitions
□ choose technologie with minimum manual handling

Overview on Technologies

- disassembly technologies
- destruction technologies

□ thermal treatment is most flexible
□ thermal treatment minimizes steps

Description of Concepts

Semi-Mobile Plant
- one-step detonation technology
- flue gas treatment

□ non-explosive hazardous waste suitable for transportation and further treatment

Stationary Plant
- one-step detonation technology
- high-temperature treatment (plasma furnace, rotary kiln, others)
- flue gas treatment

□ minimize amount of residues
□ residues are easyly disposable

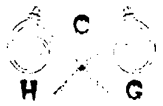
Advantages and Disadvantages of Disassembling Technologies

GRP

Technology and Process Description	Advantages	Disadvantages
manual disassembling - drilling, sawing and milling	- all types of ammunition - personnel gets experience - personnel gets experience	- high risk for personnel - very low throughput - highly qualified personnel required
cryofracture - deep-cooling of shells in fluid nitrogen - hydraulic fracture	- not specific to ammunition type - reduction of handling steps	- explosives are still active - some explosives can get more sensible by treatment - technically demanding
waterjet cutting - cutting of burster and fuse in one operation	- reduction of handling steps - inactivation of explosives	- still certain risk of explosion - water and abrasives are contaminated by chem. agents - maintenance due to abrasives

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Advantages and Disadvantages of Disassembling Technologies



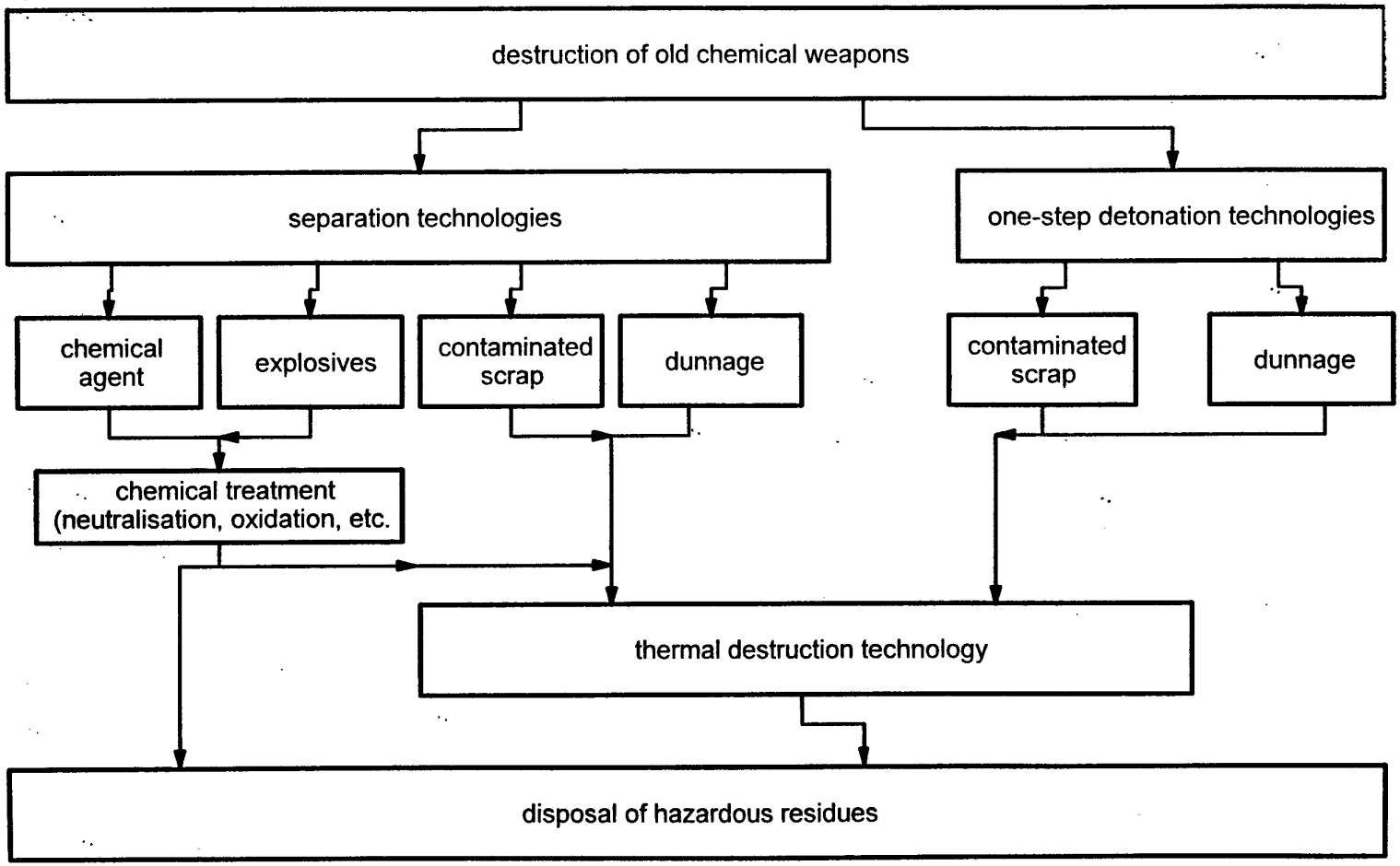
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Technology and Process Description	Advantages	Disadvantages
cold detonation chamber - explosives are forced to detonation in armoured kiln by external ignition	- low energy consumption - not specific to ammunitions - no rotating parts; easy to seal	- scrap is still contaminated - external ignition required - lower throughput
rotary kiln - explosives are forced to detonation in armoured kiln by heat	- not specific to ammunitions	- rotating parts must be sealed - short residence time in kiln (< 1 h) - risk that not all explosives are removed - scrap may still be toxic
hot detonation chamber - explosives are forced to detonation in armoured kiln by heat	- no rotating parts; easy to seal - high residence time (20 h) - high temperatures (500 °C) lead to partly detoxification of agents - not specific to ammunitions	- energy consumption - scrap may still be toxic (arsenicals), but is less dangerous

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Destruction Strategies

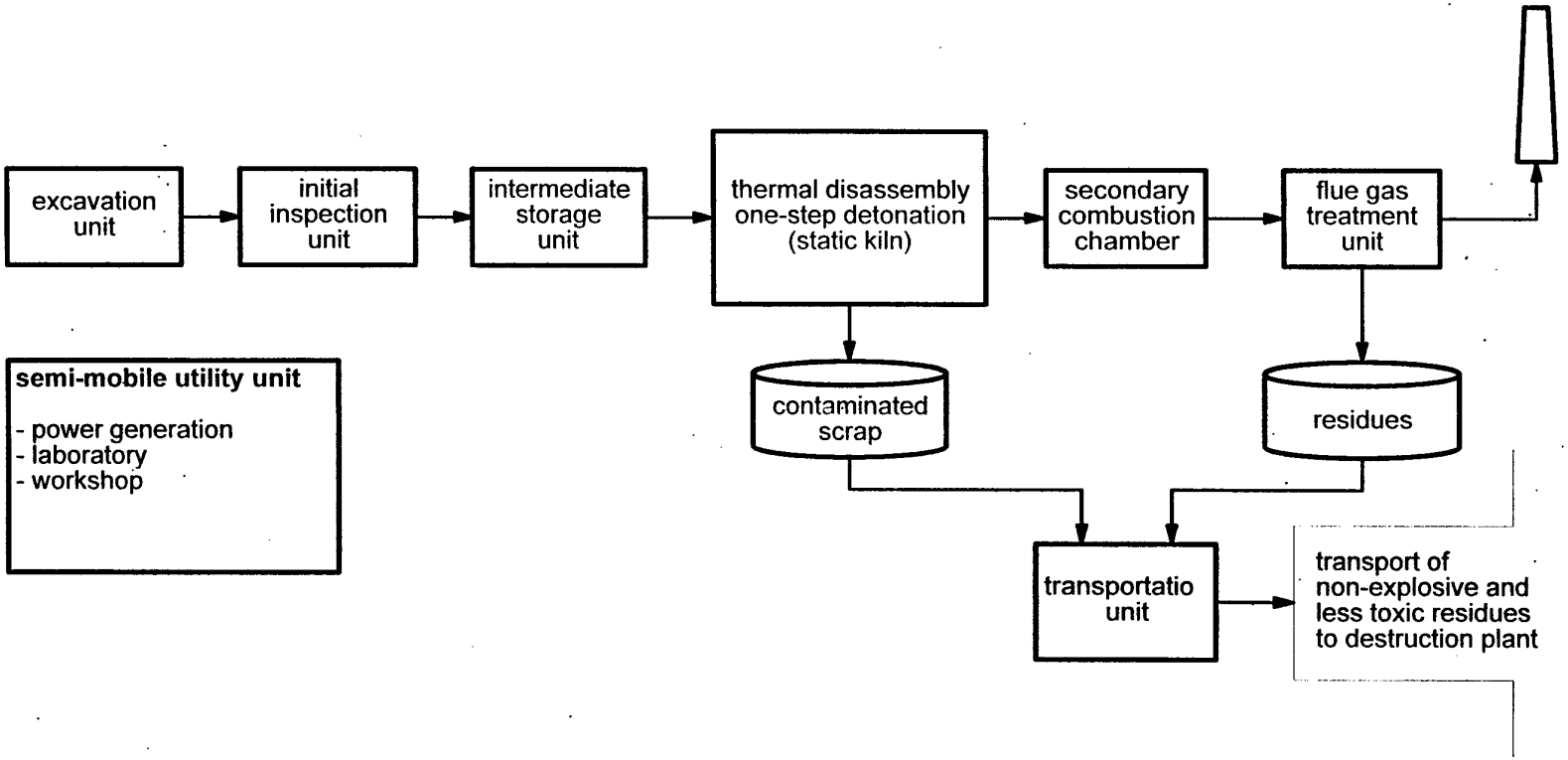
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Concept for Semi-Mobile Plant

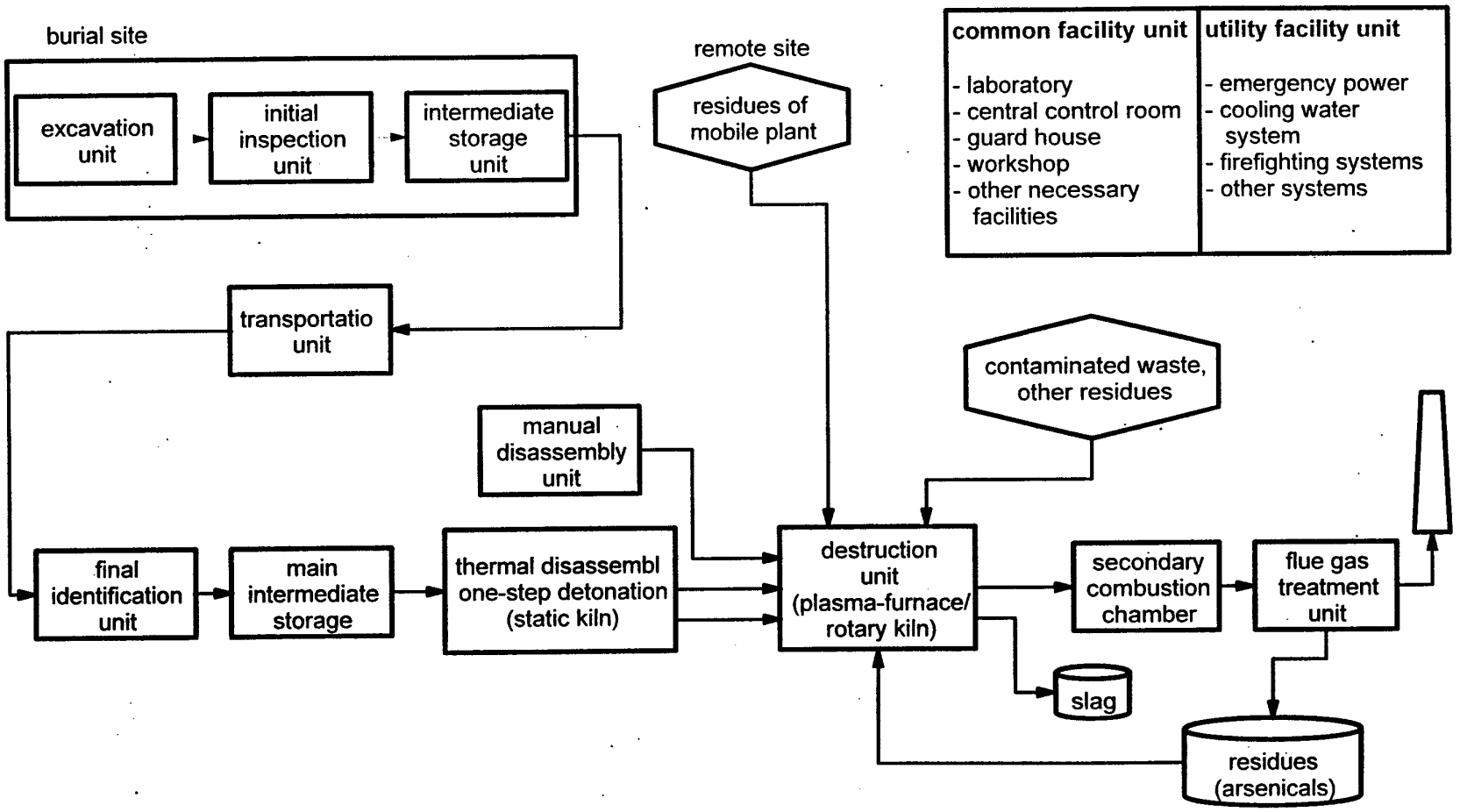
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Concept for Stationary Plant

GRP



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Munster, Germany; March 22-25, 1998

Proceedings of the International CW Destruction Symposium
Munster, Germany, March 22-25, 1998

THERMAL DESTRUCTION OF CHEMICAL WARFARE AGENTS

Klaus Maczek
Lurgi Umwelt GmbH, Frankfurt, Germany

The first plant LURGI built for the destruction of chemical weapons is the incineration plant for the Deutsche Bundeswehr in Munster. The second plant is for Gorny in Russia, which is under construction at the moment.

Munster Incineration plant

Process requirements

Cleaning of the Munster site was and still is a very difficult task, because although the total amount of agent was comparably small (lower than 200 tons), a great variety of different agents in various shells and other containers had to be destroyed:

Mustards (HD), phosgene (CG), Clark I and II, Adamsit, Tabun (GA) and Sarin (GB)

However, the main task was the destruction of several thousands of spray containers, each containing 10 litres of viscous mustard. This mixture contained different mustards, arsenical compounds (for example Dichlorophenylarsin) to make it usable in winter and wax or chlorinated rubber as thickener for a grease-like consistency. Due to the fact that the containers were stored or hidden in the ground for more than 40 years, most of the shells were highly corroded and the surrounding soil was already contaminated.

Because of the poor solubility in almost every solvent the decision was made to demilitarize the containers first and then to destroy the agent and all contaminated materials in an incineration process.

Incineration was chosen, because it offered some important advantages:

- Incineration was proven technology (plant was comissioned in 1980)
- Incineration has high reliability
- Incineration at high temperatures has high destruction efficiency, regarding chemical agents

The process LURGI developed for Munster offered high flexibility concerning the different types of materials to be treated:

- mustard spray containers
- artillery shells
- used protective clothing
- contaminated laboratory material
- spent decontamination solution

Very low impact on the environment could be acchieved by efficient flue gas cleaning and waste water treatment.

Process design

Loading area
All materials to be incinerated are stored in 30 l polyethylene drums, for each batch several of these drums are placed on a small rail car. A flat, brick lined and therefore heat resistant tub is mounted on top of the steel construction of the car. The area in the tub, where the drums are placed is about 2 m². To protect the brick lining from beeing damaged by chemical reaction, the material is put into extra tubs made of temperature resistant steel.

In the loading/unloading area the lids of the drums are removed and for destruction of viscous mustard the car is driven automatically into the evaporation chamber through a sluice.

The complete handling area is kept under negative pressure, the vent air is cleaned by an activated carbon filter. The workers have to wear protective clothing.

Evaporation chamber
In the evaporation chamber the mustard is treated with inert gas at a temperature of 300°C for about 12 hours. The inert gas is produced by stoichiometric combustion of fuel oil. The agent vapourizes and the vapours are sucked into the post combustion chamber, where complete burnout is reached at a temperature of about 1000°C, excess oxygen and a residence time of more than two seconds. The mustard forms CO_2, H_2O, SO_2 and HCl. Reaction temperature is maintained by a fuel oil burner.

Burnout chamber
After evaporation the car is moved over automatically by a shunting station into the incineration chamber, where the remaining substances are burnt out at 1000°C for another 8 hours. The off gas is again routed to the post combustion chamber.

Also small amounts of liquid agents were found in Munster. These substances were sprayed directly into the post combustion chamber via atomization lances.

Flue gas cleaning
The flue gas from the post combustion chamber is quenched and saturated with water in a pipe quenche: The hot flue gas enters a vertical bundle of thin pipes from the top together with the quench water. At the bottom, excess water and saturated flue gas are separated in a separation vessel. The water is collected and pumped to the flue gas entry, the flue gas enters the first scrubber. A pipe quench has the advantage of very reliable cooling effect during part load operation.

SO_2, HCl and other acidic compounds are removed in two packed column scrubbers, the first with an additional spraying zone and very effective droplet separators. The wash water is neutralized with caustic soda.

The flue gas then passes the induced draught fan and leaves the plant via the stack.

The emission limits are still kept most of the time, although the plant was designed 12 years before the values of the now relevant 17. BImSchV became valid.

Waste water treatment
The purge water from quench and scrubbers has to be treated in order to remove the arsenic components. Therefore the purge is first oxidised with $KMnO_4$. Remaining SO_2 then forms Na_2SO_4, Chloride doesen't react and the arsenic components form Arsenate salts, for example Na_3AsO_4. The arsenate is precipitated as ferric arsenate by addition of ferric chloride solution and maintaining a pH-value of about 8. The sludge is separated from the waste water in a thickener and dewatered in a filter press.

The dewatered sludge is filled in 200 l drums and dumped in an underground landfill.

Plant performance

Process data Munster Incineration Plant

	Data	unit
throughput mustard	25 - 35	kg/h
waste throughput	50	t/a
fuel oil consumption	100	kg/h
inert gas (from fuel oil)	400	scm/h
electrical energy	190	kW
plant water	2	cm/h
plant air / instrument air	60	scm/h
flue gas	2.800	scm/h

Emission parameters Munster Incineration Plant
Compared with the permitted limits, the actual emission data are remarkably low:

Parameter	average data1992	Permission 10.1980	TA Luft 1986	17. BImSchV	Unit
SO_2	14,5	200	100	50	mg/scm
HCl	7,5	100	50	10	mg/scm
As	0,09	-	1	0,5	mg/scm
Dust	< 10	20	30	10	mg/scm
total Carbon	< 10	50 1980	20	10 1998	mg/scm
As in water	0,05	1		0,1	mg/l

Problems in operating the plant
The only emission parameter which caused trouble sometimes was Arsenic: At high temperatures Arsenic forms arsenictrioxide, which is an aerosol in the flue gas and is difficult to separate in scrubbers. Therefore the amount of arsenic in each batch had to be calculated and measured. In 1997 the problem was solved by installation of a wet electrostatic precipitator (WESP), located after the flue gas scrubbers:

Remaining dust particles are ionized by high voltage (50 kV) and separated on collecting electrodes. The vertical collecting electrodes are flushed permanently by a thin film of water, this prevents the collected dust from reentering the flue gas.

One heavy accident happenend during operation of the plant: A fuse or burster which wasn't removed exploded in the incinerator, which was severely damaged and had to be replaced. The new incinerator has a round steel shell and can stand a pressure of about 10 bar.

Gorny residues incineration plant
The task for the residues incineration plant for Gorny in Russia seemed to be completely different: Lewisite which is stored in the area of Saratow at the river Wolga, will be hydrolysed with caustic soda first, then the obtained solution of sodium arsenate will be electrolysed for recovery af arsenic. Only the residues from the different process steps are treated in the incineration plant. But these waste streams may also be contaminated with agent.

Design basis (excerpt)

Liquid residues

- Organic solvent containing
  - ethylenglycolester 44 wt%
  - tri-butylamine 23 wt%
  - tri-butylamine-hydrochloride 33 wt%
- Aqueous solution containing
  - tri-butylamin, ethylenglycolester
  - arsenic oxides
  - sodium hydroxide
- laboratory waste water containing
  - reaction products of lewisite 2 wt%

organic solvents	5 wt%
- decon solution containing	
dimethylformamide	3 wt%
sodium hydroxide	20 wt%

Solid residues

- handling materials
  - cotton cloth
  - filter materials
  - activated carbon
- filter sludge
- metal containers, metal drums
- personal protection equipment
  - rubber boots and gloves
  - rubber suits
  - filter masks

Conclusions

- Most of the materials may be contaminated
- Incineration had to be highly flexible concerning the waste
- Flue gas cleaning had to have great separation efficiency regarding acidic components and aerosols

Process design

Concept

Munster concept is also suitable for Gorny, changes are necessary, but only in detail and not in general:

- Due to the small amounts of agent (no bulk material) an evaporation step is not necessary
- In order to separate the possibly contaminated loading and preparation area from the unloading area for burnt out ashes and scrap metal, the new solids incinerator has a front entry and a rear exit for the rail car, each equipped with a sluice
- For the different liquid waste streams a large post combustion chamber with multi fuel burner and lances for aqueous waste is foreseen, the combustion will be two-stage to minimize formation of NO_x.
- The last scrubber stage will be water cooled for recovery of the evaporated quench water
- For removal of aerosols, especially arsenic oxide, a wet electrostatic precipitator will be installed
- For removal of arsenic from the waste water, a two stage precipitation with ferric chloride is planned

Incineration process

The car is loaded, the containers have to be opened and the car is driven automatically into the entry sluice. The external diameter of the incinerator is 3 m, overall length about 7 m. The doors are each equipped with a hydraulic opening system and a bayonet socket for mechanical bolting.

During operation, three cars are in the solids incinerator. The gap between the cars and the brick lining of the incinerator is narrow, secondary air enters the incinerator from the bottom, so the rails and the steel construction of the cars are not in the radiation area of the flame and will always be cooled.

For changing the cars, the rear door is opened, the last car is pulled out into the exit sluice and the rear door is closed again. Cooling air is sucked into the exit sluice and then into the post combustion chamber. Then the front door opens and the new car is pushed in, pushing the other two to the rear. The front door is closed and the

gas burners start. If necessary, a temperature of 1200°C can be maintained continously. The actual residence time in the solids incinerator will depend very much on the type of waste and can't be predicted. But it is very important that residence time at high temperature is not limited by the equipment.

Post combustion
Flue gas from the solids incinerator is routed into the second stage of the post combustion chamber. In the vertical post combustion chamber the organic liquids are fed to a multi fuel burner mounted on top of the incinerator. The aqueous waste stream is injected with atomizing lances. To acchieve low NO_x formation rate, the amount of primary air will be less than stoichiometric, complete burnout is acchieved by excess air in the secondary stage, 1200°C reaction temperature and a residence time of at least two seconds.

Reaction temperature will be maintained with natural gas if the heating value of the waste is not high enough.

Flue gas cleaning
The hot flue gas is immediately quenched by leaving the post combustion chamber through a venturi type quench at the bottom of the furnace. The flue gas cools down to about 85°C and is now saturated with water. The advantage of a direct quench is, that any chemical reactions, such as the formation of chlorinated dioxins, are immediately stopped. The disadvantage is the higher formation rate for aerosols, which have to be removed in the flue gas cleaning.

Most of the dust load and part of the acidic gases, mainly Hydrogenchloride, is removed in the first scrubber, a venturi type scrubber.

The remaining acidic gases are removed in a two stage packed column scrubber, where the wash water is neutralized with caustic soda. When a high amount of hydrogenchloride in the incinerator leads to formation of free chlorine, Cl_2, it will be removed by addition of hydrogensulfite as reducing agent. The wash water of the second stage will be cooled indirectly to decrease the flue gas exit temperature down to 60°C and to recover the evaporated water from the quench.

Remaining dust particles and aerosols will be removed in the wet electrostatic precipitator: Ionization of the particles with high voltage of about 50 kV leads to precipitation of the particles on the vertical collecting electrodes. The collecting electrodes are permanently flushed by thin film of water, which prevents the particles from reentering the flue gas. After passing a doplet separator and the induced draught fan the flue gas leaves the plant via the stack.

Waste water treatment
The waste water purge from the scrubbers will be treated in two stage precipitation process: In the first stage the water is neutralized with caustic soda, Arsenic is oxidized to Arsenate by $KMnO_4$, precipitated with ferric chloride and separated in a thickener. This is repeated in a second stage, then the water is finally cleaned in a microfiltration.

The sludge from the different stages will be collected and dewatered in a filter press.

Process data Gorny Residues Incineration Plant

Parameter	Data (expected)	unit
liquid waste throughput	250	kg/h
	290	t/a
solid waste throughput	165	t/a
natural gas consumption	max. 300	scm/h
flue gas at stack	3.800	scm/h
SO_2	(< 20)	mg/scm
HCl	(< 10)	mg/scm
Dust	(< 5)	mg/scm
Arsenic in flue gas	(< 0,5)	mg/scm
Arsenic in water	0,05	mg/l

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DISPOSAL OF CHEMICAL WARFARE AGENTS RESIDUES AND MUNITIONS - A NEW APPROACH BY MANNESMANN DEMAG AND MGC-PLASMA

Holger Weigel*, Dr. Thomas Stock*, Achim Stumpfe*, Dr. Wolfgang Hoffelner**, Dr. René Burkhard** and Alex Zeman**

I. Introduction - About Mannesmann Demag and MGC Plasma

Mannesmann Demag is one of 10 divisions of the Mannesmann Holding. Mannesmann is a group of technology companies which builds and supplies machines and plants, produces systems and components for technologies used in automobile industry. The company is active in measuring, automation and information technology, offers telecommunications services, manufactures steel tubes and is engaged in world-wide trade activities. In Mannesmann the work of 120,000 employees results in an annual turn over of 30 billion DM.

Mannesmann Demag Energy and Environmental Technology (MDEU) acts as general contractor in Europe and world-wide for major orders in pipeline and plant construction, in oil and gas industry and in environmental technology. MDEU is one of the leading companies for piping systems construction in power stations, in building installations technology and industrial plants and for water and sewage treatment processes.

In April 1989 MGC Plasma AG was founded as a joint venture between Moser-Glaser & Co Ltd., Muttenz and the Sandoz Group, Basel. The business objective of the company is the layout and construction of plasma plants such as:

- PLASMOX®, a process for treatment of toxic wastes;
- PLASMARC®, a process for treatment of nuclear wastes; and
- PLASMAREC®, a process in which metals from waste materials are recovered.

MDEU and MGC are working together on an exclusive base to combine the strengths of both companies for the customer's benefit.

II. Armament wastes - environmental and public concern

Since the beginning of the 1980's a growing awareness of the public about the negative impact human wastes might have on the environment can be recognized. The declining willingness to accept damages by wastes as a „normal disease" of the modern society has resulted, among other things, in a remarkable reduction of environmental emissions.

Furthermore, the changed political climate in Europe at the end of the Cold War and the increasing interest of most European states in environmental policy has led to the realization that there is a need for greater co-operation on environmental matters. There is an increased openness and acceptance to discuss the problems related to the destruction of weapons and armaments.

However, these developments had much less impact on the awareness and acceptance of already existing environmental contaminations - for example, abandoned munitions, munitions polluted areas and highly toxic military residues, such as chemical warfare agents.

The environmental and technical problems which have occurred since the end of World War II have demonstrated the risks that old munitions, especially chemical ones, pose to people and the environment. Many countries, not only in Europe, must cope constantly with discovered munitions, chemical and conventional ones. In addition, former military facilities, munition production sites and testing grounds do pose an additional burden.

Many years of experience in the treatment of highly toxic substances and the remediation of contaminated soils have placed MDEU into a position of being able to find the best suitable solutions for any kind of above

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mentioned contamination problems. Such a solution is always based on a combination of technologies, individually optimized according to the requirements of the particular situation, taking our long-term experience into account.

For a few years, MDEU has been working together with his Swiss partner, MGC Plasma AG and the German authorities to realize a project, which is unique in Europe: *The construction of a plant for the destruction of chemical warfare agents and contaminated soils.*

III. Destruction of chemical warfare agents and contaminated soils - the Munster Project

The destruction of toxic military waste materials is one of the most demanding disposal tasks our industrial society is facing. There are numerous locations suspected of being contaminated by warfare and munition wastes, products from armament production and wastes from military activities. These contaminated sites do pose a considerable potential danger for both the population and the environment.

In many cases there is an immediate need for actions to be taken, particularly in such cases where chemical warfare agents or munitions have been produced, tested or stored in past years.

In Germany there is a great variety of old chemical weapons from the 1915-45 period including artillery shells, mortar ammunitions, hand and rifle grenades, land mines, bombs and spray canisters. The history of the German armament programme for chemical warfare is closely connected to Munster in both World War I and World II. When chemical weapons began to be used in World War I Munster was chosen as the proving ground for chemical warfare agents and munitions. Besides the construction of the Breloh-Camp in 1916, this site became the place for the production of chemical warfare agents and munition filling stations. Furthermore, this site was chosen to be a place for conducting ballistic tests on munitions. After World War I the Breloh site was the place where large quantities of chemical warfare agents and munitions were assembled for destruction. In 1919 an accidental explosion destroyed many building, including production facilities, approximately 1000 tonnes of chemical warfare agents, more than one million shells, a quarter of a million land-mines and railway tank-wagons, all with chemical warfare agents. This explosion and the test and production activities from the past contaminated the place Munster North, as it is called in nowadays, with chemical warfare agents from filled shells and with arsenic compounds (contained in many munition fillings from that time).

During World War II the Munster site was used as research and testing facility for chemical warfare agents, a new army proving ground was established, facilities for the production of new agents, a munition depot and a filling station were set up. In addition, there was a 10 000 hectare area for field trials, artillery field tests and bomb tests. During all the years from the 1919 explosion on attempts were made to clear the debris and left behinds by blastering and burning. However, until now the area is still contaminated. The blastering and burning led to an additional contamination of the soil with arsenic compounds.

Owing to the specific situation at the Munster area a decision was taken to construct an incineration plant, which is based upon a two-stage, batch-type furnace. This facility began operation in 1980 on the premises of the German Armed Forces Scientific Institute for Protection Technologies: NBC Protection at Munster, in the federal state of Lower Saxony. The plant was tailor made to solve the problem of destroying viscous mustard. The throughput for sulphur mustard at the incineration facility is at a rate of 20-35 kg/hour.

Due to the complex situation at Munster with regard to contaminated materials, the large amount of arsenic contaminated wastes and the various different chemical warfare agents in the late eighties a decision was taken to go for a second plant, which will be more flexible with respect to the furnace and which will realize via a continuos process a much larger throughput.

A consortium comprising the MGC-Plasma AG and Mannesmann Demag Energy- and Environmental Technology, Duesseldorf, was awarded the contract for the project of the plant engineering at the end of 1991 by the Public Building Office in Munster as the primary contractor. The required licensing documents were compiled in co-operation with the consulting engineers in accordance with the German Law on Immission Protection (BImSchG) for constructing a "Pilot plant for the destruction of warfare agents".

The treatment concept

For the treatment of the materials contaminated with chemical warfare agents, chemical weapons material and so-called dunnage material the above mentioned consortium selected a solution, based upon a combined process. The solution consists of:

(1) a thermal inertizing process for the highly contaminated material - the PLASMOX®-process - with a maximum capacity of 1 tonne per hour; and
(2) a physical-chemical soil washing process, with a maximum throughput of 2.5 tonnes of earth material per hour.

The demands required with the placement of this order to the consortium can be described in simplified terms as the following:

- The plant must be able to handle a large amount of different chemical warfare agents as defined by the German armed forces.
- The plant must be suitable for destruction or inertizing contaminated wastes from the armed forces, such as contaminated protective clothing, wastes resulting from the overall decontamination processes, storage and filter material, etc.
- The plant must be able to clean the contaminated soil from toxic material, in particular arsenic compounds must be handled.
- The cleaned soil must be suitable for refilling, based upon the German legislation for refilling.
- The concentrated contaminants, as a result of the washing process, must be inertized or bound into a non-leachable slag.

The planning of the plant was based on the following demands:
- The plant should have a throughput of about 3000 tonnes of highly toxic waste material per year. In addition, approximately 9000 tonnes of contaminated soil should be processed through the plant.
- The plant has to be flexible with respect to fluctuation in the constitution of the source materials, the content of toxic material.
- Any contact between the plant operating personnel and the contaminated materials in the plant must be prevented via a complex control system and extensive protective measures.

At the beginning of April 1994 the Federal Republic of Germany, represented by the Hannover Regional Finance Office and the Public Building Office in Munster, was granted approval by the Lueneburg District Authority to erect the first combined plant - PLASMOX® plant and soil remediation - for the disposal of chemical warfare wastes in Germany. This plant is supposed to be supplied by the consortium of MDEU and MGC Plasma AG.

Project management

In an extremely complex and highly sensitive project of this nature, particularly where a new thermal treatment technology (PLASMOX®) is also to be applied, the expert investigations as well as the supervision of the tendering companies by consulting engineers from the client have to be carried out to extraordinarily strict standards.

The Public Building Office in Munster, as contractor and applicant, commissioned the consulting engineering company named Göpfert, Reimer & Partner (GPR), Hamburg, as the responsible consulting engineers to carry out the preliminary planning, to issue the tender document and to co-ordinate the elaboration of the licensing application. The Lüneburg District Authority, as the licensing body, has called in for its part the expert authorities and administrative offices provided for in such cases. Furthermore, the responsible authority has placed great emphasis on the expert evaluations of the process engineering and the protection against harmful emissions as well as on the investigation of the plant safety on the basis of the submitted safety analysis in accordance with paragraph 5 German BImSchG (Immission Protection Law) and paragraph 7 of the regulations on abnormal occurrences in industrial plants (12. BImSchV).

The thermal treatment process - PLASMOX®-process

The input of chemical warfare agents and contaminated material into the plasma chamber is facilitated through various lines:
(1) with a feeding system for solid feed materials;
(2) with a liquid pump for liquid residues and auxiliary fuel; and
(3) with a slurry pump for the contaminated slurry residues from the soil washing stage.

These three different feed lines as well as the slag removal are all equipped with purification systems in order to prevent any form of backward contamination.

The thermal treatment of the feed materials is carried out in two stages:

(a) the plasma high-temperature pyrolysis stage; and
(b) the subsequent oxidation of the pyrolysis gases in a post-combustion chamber.
For the pyrolysis process the plasma chamber is equipped with a high-performance plasma burner, which is operated with direct current. The high arc temperatures (up to 20 000 °C) and the high energy densities of the plasma torch will permit an effective destruction of the waste materials. As the melt is being formed it will act as a resistor and it also heats up, even more due to the Joule effect.

Because of the specific applied construction the energy input for the plasma chamber can be concentrated and applied at high density. The required reaction chamber is kept so small, because in this case even in the event of a failure in the plant management during operation only small volumes can continue to react after the plasma burner has been switched off.

The solid materials smelted in the plasma stage will be retained so long until reaction has been completed and all material has been converted and smelted. The gaseous components pre-treated by the plasma torch are transported into the post-combustion stage for controlled and complete oxidation. Only vitrified slag and dry evaporation residues will result as waste materials after a complex waste gas scrubbing process, using waste-water-free technology.

The smelt lies initially in a cooled centrifuge rotating at approximately 40 revolutions per minute around its vertical axis. Here the surface of the molten slag is formed as a paraboloid of revolution. The capacity of the centrifuge is approx. 1 m^3 i.e. 2 to 5 tonnes depending on the specific density of the smelt. The capacity is therefore sufficiently large to permit the pouring of the molten slag in a time-frame of several hours, for example at the change of shift. The quality of the smelt and of the solidified slag respectively can be widely influenced using additives so that an inert, non-leaching product can be achieved. A later treatment of the residue is therefore not longer required.

The pouring of the smelt is initiated by reducing the centrifugal speed. The melted slag then flows through the central opening in the bottom of the centrifuge into the waiting slag molds, which are transported out via a bulkhead-type chamber system.

Waste-gas scrubbing

The gaseous components resulting from the plasma pyrolysis process have to be treated in such a way that they follow the German 17. BImSchV Immission Protection Law. Therefore, the gases are fed into a post-combustion system, where they are quickly oxidized in a controlled way with the addition of an air/oxygen mixture.

These waste gases are kept at temperatures above 1200 °C for a residence time of more than 2 seconds in the post-combustion chamber, designed in the form of a swirl chamber.

Afterwards the gases are discharged through a waste gas pipe into the waste heat boiler, where the waste gases at a temperature of approximately 1250 °C deliver in a direct heat exchanger so much thermal energy to the cooling cycle that a gas exit temperature of approximately 90 °C results after passing through the waste heat boiler and the quench. The recovered amount of heat is used for re-heating the waste gases for the $DeNO_x$ plant and also as a source of heat for the waste water evaporation plant.

The state-of-the-art waste gas purification plant cleans the waste gas of harmful substances such as HCl, SO_2, NO_x, HF, NH_3, heavy metals, dioxins, furanes and dust to levels below the limit values set down in the above mentioned German Immission Protection Law. The essential components of the waste gas purification stage are quench, aerosol separator, multi-stage ionizing wet scrubber as well as a catalytic $DeNO_x$ plant. Waste gas fans in parallel operation ensure low pressure in the complete plant so that under no circumstances can a leak cause hazardous gases to escape to the atmosphere.

In addition, the pressure-tight plasma chamber is connected with an evacuated surge chamber system, which prevents a contamination of the outside chamber even in the event of failures due to overpressure. The design and the layout of the waste gas purification plant also ensure that no impurities in the air can cause environmentally hazardous effects for the immediate neighbourhood and the population in general.

The waste water arising in the scrubbing stages as well as the wash waters from the quench and from the aerosol separation plant are treated in a waste water evaporation plant after appropriate conditioning.

Soil cleaning process

Originally it was intended to treat the contaminated materials by pyrolysis and incineration in a rotary kiln. In such a set up the whole amount of feed materials would have had to be subjected to thermal treatment. The treatment of every 100 kg of contaminated soil would result in 85 kg of rotary kiln slag, which later would have to be disposed of on a dump site for waste materials requiring special supervision. In addition, around 25 kg of mixed salts and dusts from the waste gas scrubber system would result from the process, which could only be buried in underground salt caverns.

With the currently approved process the flow of materials, which will be subject to the thermal treatment (PLASMOX®), is reduced by a ratio of 1:10 by an upstream soil washing process with fine-grain separation and cleaning of the remaining soil according to the principles of flotation.

As pilot tests on site with a mobile plant already confirmed, a suitable soil washing process with flotation can purify approximately 90 per cent of the treated soil to such an extent that it can be re-filled and used again. The toxic materials flushed out in this process are concentrated and remain in the residual 10 per cent of the feed materials. From a total input of 100 kg therefore approximately 90 kg of soil is recovered for re-filling and re-use. Only approximately 10 kg of highly contaminated wastes result for final disposal in an above the ground dump (slag material) or in an underground expository (for residues from the waste gas scrubbing) after the thermal treatment in the PLASMOX®-process.

Environmental and working protection in the Munster Plant

Besides the protection of the specialist workers involved in the future operating of the plant, the effective protection of the environment from accidental releases of the highly toxic chemical warfare material was one of the basic demands in the overall planning for the facility. The whole area for the handling of the contaminated materials in the new Munster plant is kept under a slight low pressure and is accessible only via locks. All conveyors and apparatus in this hall that handle contaminated material are encapsulated and are themselves kept at a low pressure. The low pressure is generated by an exhaust system equipped with ultra-fine mesh fabric filters and special activated carbon filters mounted on the roof. The graduated low pressure inside the hall ensures that no emissions escape into the hall. The filters of the exhaust system prevent the escape of emissions into the atmosphere.

There are three feeding lines for chemical warfare contaminated material:
(1) Special materials and highly concentrated contaminants which cannot be cleansed and are thus charged directly into the plasma plant, very often summarized as dunnage materials;
(2) Thickened substances and liquids; and
(3) Soil.

The toxic materials can be charged either in big bags or in polyethylene drums which are automatically emptied by a robot handling system. There are storage drums for the soil to be cleansed, for the special materials which are fed into the plasma plant and also for the light materials, e.g. the polyethylene containers in which the material is delivered. There are also various facilities for the preparation of materials, e.g. shredders and crushers, and also a special washer for dissolving agglomerates.

A highly sophisticated control system, including various sensors for measurements of individual toxic components and the exhaust gases, will assure that no residues from the chemical warfare agents are leaving the facility.

IV. Destruction of old chemical weapons - a new concept

The technical and practical matters which must be taken into account when coping with destruction of old chemical weapons differ from those which need to be considered when dealing with chemical weapons in stockpile. It may be difficult to determine whether a munition is chemical or conventional, it may be difficult to identify the origin of the old chemical weapon. In some instances the explosive charges will be active and unable to be removed. Furthermore, old chemical weapons often contain a complex mixture of various chemical warfare agents that have already undergone decomposition. Therefore, planning, dismantling and destruction of old chemical weapons are complicated.

In general, the following steps can be identified in the process of handling old chemical weapons:
(a) reconnaissance and location;
(b) unearthing and identification;
(c) removal and transport;
(d) intermediate storage;
(e) preparation for demilitarization; and
(f) demilitarization.
Up to now the destruction of old chemical weapons does pose a major technical challenge on very high expenses. The major time and manpower consuming step was always the so-called conventional delaboration of the old munitions, after unearthing and transport. The main disadvantages of the conventional delaboration are:

- need for a number of highly experienced fire-workers;
- need for a large number of personnel in general;
- high risk for personnel and environment;
- low throughput;
- a significant amount of secondary material, contaminated with chemical warfare agents, will be created;
- cost extensive operation; and
- major investment into very special hardware.

Taking the above disadvantages into account MDEU and MGC Plasma AG are working together on a new concept for treating old chemical munitions.

This concept is based upon the following principles:
(a) The old chemical munition will be automatically transferred into a detonation chamber. Within this chamber the munition will be brought to detonation. As a result, all explosive materials will be transferred into a status, which does not any longer pose a risk for the follow-on treatment process with respect to possible explosions.
(b) Via different lock systems the residues, after the treatment in the detonation chamber, are fed directly into the PLASMOX® plant.

The advantages of this concept will be:
(1) minimizing the risk for the personnel involved in dismantling and handling operations;
(2) no separation of explosive charges and chemical warfare agent filling is necessary;
(3) large throughput rates; and
(4) high degree of automation is possible.

V. Future applications of the Munster Plant type process

The approval of the PLASMOX®-process for the Munster project has confirmed that the high-temperature plasma technology is a potent new technology for the treatment of problematic waste materials. The process parameters necessary for the permanent operation of such a plant will be systematically determined during the pilot operation phase, which will start in the first half of the year 1998.

With the optimal matching of the plant design to the waste materials stored in Munster and the knowledge which will be acquired from operating the facility, already today it can be assumed that this process will be an important and promising solution for future waste disposal problems we are facing.

On the basis of the plasma smelting technology originating from the field of metallurgy the PLASMOX®-process has been developed into a tool which will have a market potential as a thermal method for the environmentally acceptable disposal of hazardous waste materials.

The benefits of the PLASMOX®-process combined with soil cleansing, as it is the case for Munster, can be described as follows:

- inertization of the residues, even with widely fluctuating charge materials;
- heavy metals are reliably bound into a glass matrix;
- possibility of charging a very wide range of different, even damp, materials;
- possibility of further use of the slag;
- the plant operates completely self-sufficient without producing waste water;
- the waste gas volume is very low thanks to the plasma pyrolysis;
- no dioxins or furanes are produced during the process;
- decontamination of soils from highly problematical polluted sites;
- processing of filter dusts, highly contaminated sludge residues and flotates; and
- great potential to use the treated material for possible refilling of former contaminated sites.

VI. Potential use of the PLASMOX®-process in civilian applications

Highly contaminated toxic waste materials also occur in the civilian sector of our industrial society, although only in comparatively small amounts. The PLASMOX®-process, which is characterised by its excellent destruction efficiency for hazardous organic materials as well as for the vitrification of inorganic materials in the same process step, also represents a forward-looking supplement to other thermal treatment processes known today.

For example, the extremely energy-intensive subsequent treatment of slag materials is no longer necessary at all. The various hazardous materials can be handled specifically with the flexible process management. Even waste materials with low caloric values can be treated by a smelting operation. Parameters in the PLASMOX®-process that can be freely varied are:

- the degree of pyrolysis or partial oxidation;
- the choice of air or oxygen as oxidising agent in the plasma chamber (normally oxygen) and the post-combustion chamber (normally air); and
- the location where liquid materials are fed into the plasma chamber (highly problematic material is charged into the plasma chamber whereas less problematic materials are fed directly into the highly turbulent mixing zone in the post combustion chamber).

In summarizing future potential applications for the PLASMOX®-process the following waste material areas can be identified:

- Processing of problematical and toxic special wastes, including those from military sites;
- Recovering of valuable metals as copper, chromium, nickel, aluminium, molybdenum, titanium from waste metals.

VII. Conclusions

The destruction of chemical warfare agents, contaminated materials and weapons is one of the major technical challenges our society is facing. After the Chemical Weapons Convention has entered into force in April 1997 the destruction of old chemical weapons is time-limited for States Parties having such old weapons in stockpile. After 10 years the destruction will have to be finished.

The available experience with respect to the destruction of old chemical weapons has proven that such undertaking is time-consuming, expensive and requires well experienced experts and a reliable technology. Hereby, the delaboration of the old munition shells (unearthing, identification, cleaning, transportation and dismantling) is the most time-consuming step. Experienced fire-workers are required.

The PLASMOX®process, as applied in the Munster facility, will prove the excellent capability of this process to be used for highly toxic materials. In combination with a detonation chamber for pre-treatment of the chemical munition, a concept will be established which allows to minimize the risk of the dismantling process. Mannesmann Demag and MGC Plasma together working on this concept and having established the Munster plant will be in a position to play a major role in the destruction of chemical weapons. Experience, knowledge and expertise are available with both companies.

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NEW TECHNOLOGIES FOR THE DESTRUCTION OF CONVENTIONAL AND CHEMICAL MUNITIONS

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Sur Met Impulstechnologien GmbH & Co. KG

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Brandenburg Technical University Cottbus

The destruction of munitions has always been an issue in every age:

- munitions that have been in storage beyond their use by date and whose performance parameters are no longer guaranteed,
- calibres that are no longer in use (e.g. munitions of the National Peoples Army of the GDR),
- munitions that are no longer required in the light of disarmament and détente,
- munitions to be destroyed in accordance with international agreements, such as chemical weapons
- conventional found munitions
- chemical found munition
- leaked munition

Whilst the first categories of munitions are correctly stored and documented and their properties can be estimated, this does not apply to the found munitions. The behaviour of found munitions cannot be defined at all before investigated, and they thus harbour considerable dangers if they are moved at all. As a result, corroded old explosives (e.g. picric acid) may explode when jarred even slightly. Found chemical munitions and incorrectly stored chemical munitions also contain undefined products of decomposition and resin products so that opening and boring is not enough to remove the chemicals. Some of the munitions are no longer sealed due to corrosion and this can lead to poisoning of the ground. It is particularly difficult to dispose of chemical munitions from the First and Second World Wars as these often contain arsenic and mercury compounds.

Standard current technologies require disassembly of the munitions. Conventional found munitions are cautiously sawn into several pieces. The individual pieces are then burned in special installations. From time to time there are accidents by explosions during the sawing process, as was the case in North Rhine-Westphalia recently.

How should we saw open Chemical Munitions?

The warfare agent escapes and poisons the appliances and the rooms. The capture of the warfare agents from the munitions is one of the key problems during disassembly.
This process of disassembly is a very risky procedure. All disposal procedures which don't need any handling are the better processes in the sense of safety disposal.

What are the requirements of an environmental friendly and save disposal?

- no disassembly
- simultaneous explosion, pyrolysis and burning
- maximum level of automation, no personnel required on site
- reutilisation of scrap metal
- small quantities of washed waste gases
- small quantities of dust and residues
- Processing of unkown warfare agents (waxes, polymers, viscous materials)

Sur Met Technology meets all of these requirements.

„Sur Met GmbH Impulstechnologien" has many years' experience in the construction and operation of detonation chambers that function under vacuum conditions. This work started with explosive welding and explosive deformation under vacuum conditions.
Building on this, the company then built and operated several **"cold detonation chambers"** that are capable of causing fuses, hand grenades and small mines to explode directly in the detonation chamber. Sur Met Impulstechnologien is well experienced in destroying munition in one piece up to calibre 105 mm. Higher calibres are possible. The extreme dangers associated with the disassembly of these munitions are no longer an

issue. In addition, only a minimum of waste gas is produced. The plants have a high capacity in a range from 20 kg/h to 100 kg/h. Some of the munitions of the National People's Army of the GDR were destroyed in the new federal states of Germany in plants built by Sur Met.

The destruction process in the "cold vacuum detonation chamber" has since been further developed. Sur Met now offers a **"high-temperature vacuum detonation chamber"**. This is particularly suitable for the destruction of found munitions and even more so for the destruction of chemical munitions. This process is probably the most cost-effective method of destroying all kind of munitions but especially **found munitions and chemical munitions** to date, and I would even go as far as to say that it is unrivalled by any other process (***Fig. 1: detonation chamber***).

The **principle** of the newly developed detonation chamber is as follows:
The munitions are fed into the chamber in an explosion-pressure-proof container measuring ca.10-15 m in height with an internal diameter of ca. 2 m. They then explode following heating to ca. 600 to 800 °C. In the case of chemical weapons the warfare agents are simultaneously pyrolysed to a high degree by the heat and pressure. The energy contained in the explosive promotes the pyrolysis of the warfare agents. Chemicals are added to the detonation chamber so that the pyrolysis gases - in particular the halogens - are chemically bound.

The detonation chamber **is constructed** in a number of layers and materials in order to deaden the detonation waves during propagation in the material. (key word: earthquakeproof construction). The blast waves of the explosion gases are weakened by the vacuum. Shell splinters accumulate in the detonation chamber and are burned out at fixed intervals and then removed. The energy of the explosives helps to maintain the desired temperature inside the chamber. Oxygen and propane gas are used for the support firing. The oxygen burns the excess carbon of the explosives in addition to the warfare agents released and their products of pyrolysis. The use of oxygen instead of air means that a very small quantity of waste gas is generated, and as a result, the subsequent process stages involving waste gas cleaning can be kept to a minimum. The quantity of waste gas resulting from this process is ca. 30 times smaller than in the case of conventional processes.

The destructive capacity of the plant is 200 t/a up to 3000 t/a of net TNT-equivalent. **(Per Loading 2 to 15 kg TNT-equivalent)**

The **Chemical Munitions** to be destroyed are packed in **special containers** ***(Fig. 2)*** as soon as they are recovered. Said containers are specially designed to prevent leaks of the warfare agents. The special container is fed into the detonation chamber automatically without being opened. No personnel is present during this stage.

In the case of **chemical weapons, 3 calibre 150** shells can be fed into the chamber EC-1000 as a single package. In the case of **calibre 75, 12 shells** can be fed in as a single package. No special packaging is required for **conventional munitions**. Due to the high content of explosives, **1 calibre 150 shell or 7 calibre 75 shells** can be fed in at any one time. Shells with higher calibres still have to be disassembled.

Sur Met's system is also capable of **burning material containing explosives as well as highly toxic substances.** These substances can be fed into the chamber in drums with a capacity of ca. 80 l to 200 l. The combustion technology in this case involves two stages. The detonation chamber is also capable of burning out metals with an unknown degree of contamination (***Fig. 3: Flow diagram of entire plant***)

After leaving the detonation chamber, the **waste gases** are subjected to secondary treatment, similar to the process in incinerating plants for special waste. The waste gases pass through the known process stages:

- afterburning at a temperature of up to 1450 °C with a sojourn time of 3 secs.
- spray drier to separate the salts
- shock cooling to prevent the formation of dioxines
- dust filter and gas cooler
- basic washing
- acidic washing
- neutral washing
- nitrogen removal
- activated carbon filter

The plant can also be supplied as a **mobile system**. The detonation chambers are dismantled into several parts during transport. Suitable foundations must exist at the new location.

Sur Met also offers technologies to deal with the
- **packing,**
- **transport and**
- **storage of chemical warfare agents** following location of the munitions.

Status of development of the destruction plant for the conventional and chemical munition.
- The detonation chamber is certified.
- The waste cleaning system is certified.
- The project for a plant in North Rhine-Westfalia was finished in March 1997.

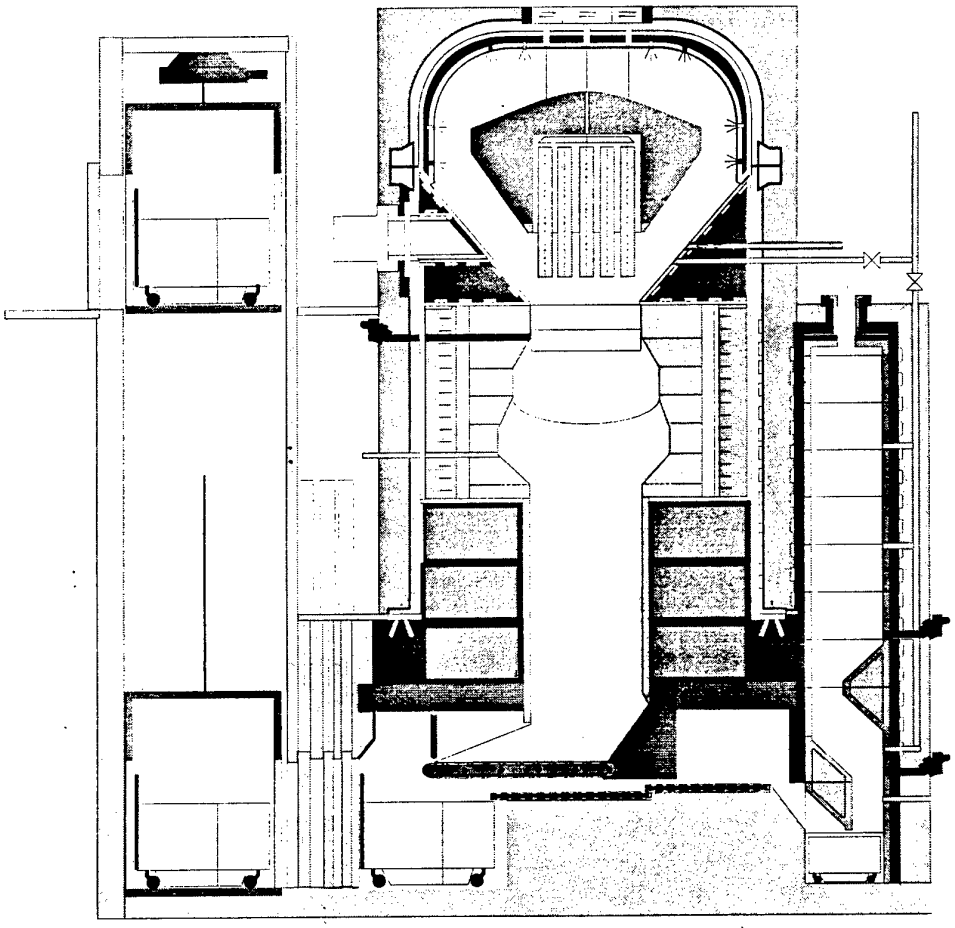


Fig. 1 Explosion and Pyrolysis Chamber

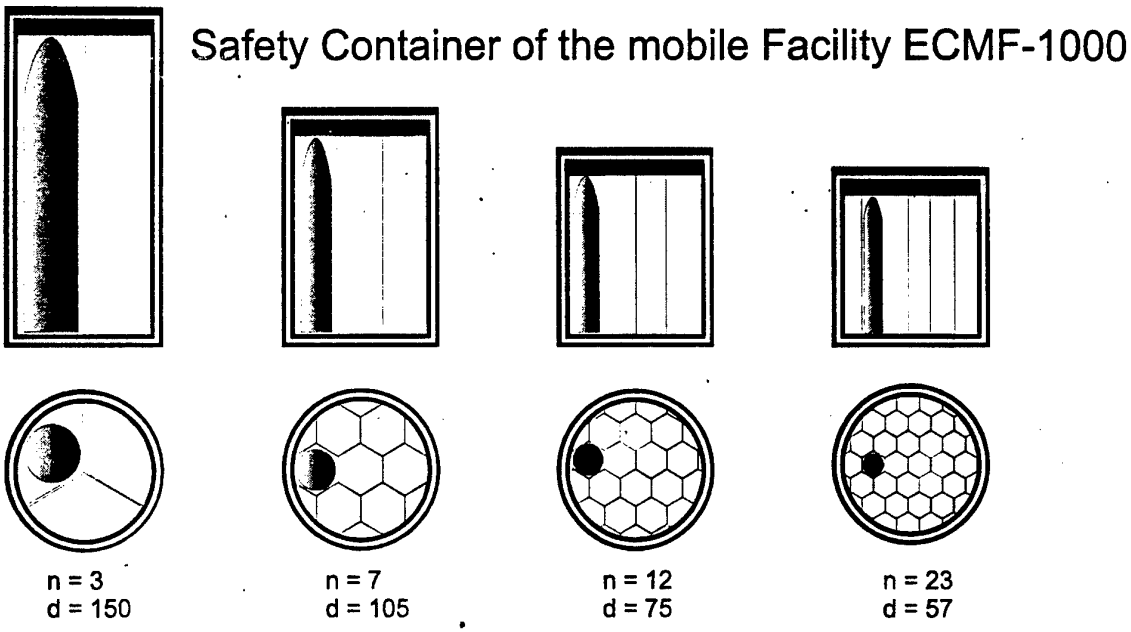

Safety Container of the mobile Facility ECMF-1000
n = 3
d = 150
n = 7
d = 105
n = 12
d = 75
n = 23
d = 57

Fig. 2

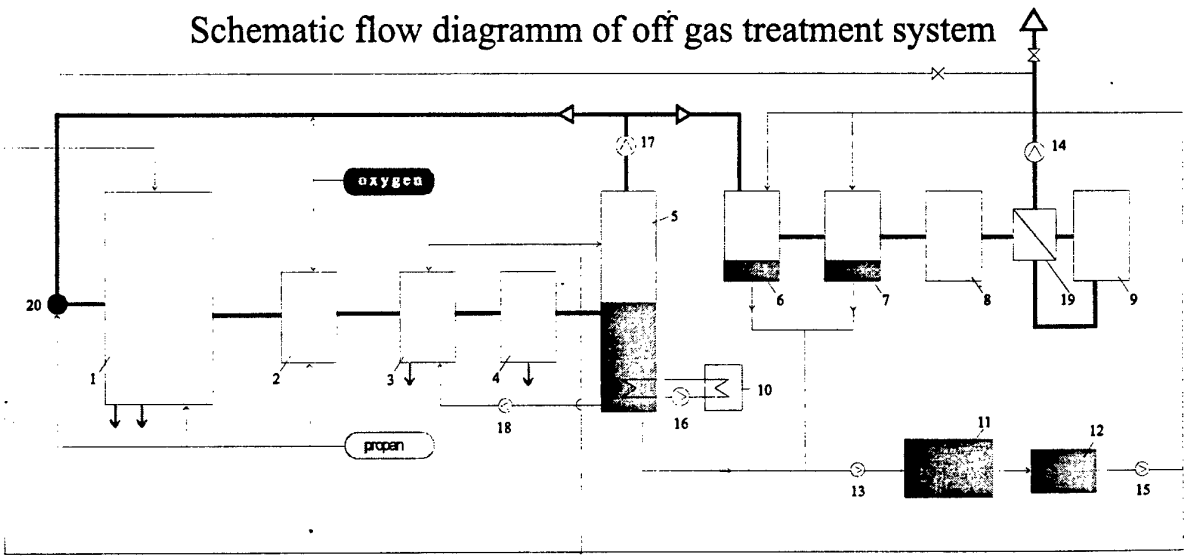

Schematic flow diagramm of off gas treatment system
oxygen
propan
1. EC-chamber
2. thermal incinerator
3. spray dryer
4. filter
5. off gas quench
6. scrubber
7. scrubber
8. fixed bed adsorber
9. SCR
10. air cooler
11. wash water system
12. clean water tank
13. waste water pump
14. off gas blower
15. clean water pump
16. cool water pump
17. recirculation gas blower
18. spray pump
19. heat changer
20. burner
scrap metal
waste material

Fig. 3

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STATIC KILN FOR DESTRUCTION OF CW MUNITIONS

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1. Introduction

Dismantling of found ammunition is often a dangerous and time consuming process due to corrosion of the ammunition parts. This is especially true for chemical munition That is the reason we started the developement of equipments for destruction of munitions without prior dismantling of the different components. The Static Kiln is a gastight equipment where disposal of explosives material take place through a burning/deflagration/detonation process caused by heat. The destruction process takes place in a chamber designed to withstand the effects of detonating munition. The chamber is heated by indirect heat which means that heating gases/heating elements are never in contact with the explosive material or chemical agents. The gases from the destruction process flows to a waste gas treatment plant via a pipe system. The metal scrap is emptyied into sealed drums which are to be transported to a disposal site or a plant for aftertreatment of scrap material.

2. Process Description

The munition to be disposed is transported to the loading room. Loose munition details have to be packed in paper or plastic tubes. The material to be disposed is placed in the loading box which feeds the destruction unit batchwise. The feeding cycle time depends of the munition to be destroyed, its content of explosives and weight of inert material. The loading box is equipped with a valve. The first inlet valve opens and the munition is moved into the loading sluice. The first inlet valve is then closed. The muniton is moved to a position close to the second inlet valve. The second inlet valve is opened and the munition falls into the destruction chamber and lands on the scrap bed. The second inlet valve is closed. Between each feeding cycle the inlet sluice is cleaned by using compressed air. The cleaning air is then used as secondary air in the destruction process in order to have an efficient burning and heat transfer process.
The temperatur in the inner destruction chamber can be varied between 400 - 600°. The heat is transferred to the munition by the scrap bed material and surrounding air and cause a destruction of the explosive material by burning/deflagration/detonation and in most cases a decomposition of the chemical agents. The inner chamber is designed to withstand a large number of detonations of various kinds of munition. Additional air (secondary air) is pressed into the destruction chamber at the bottom through the scrap bed. The metal scrap from the destruction process is collected in the chamber and adds to the scrap bed.
At certain intervals, normally once per shift, excess scrap is emptyied from the chamber through the bottom emptying system. This system is designed to prevent stacking of scrap material. A certain amount of scrap must always remain in the destruction chamber to form the new scrap bed for next destruction cycle. After emptying the outlet valves are closed and the space between the valves is cleanblown by compressed air which also is used as secondary air.The scrap containers can then be disconnected from the destruction chamber and transported to a disposal site or to a plant for treatment of the scrap. The scrap containers are also gastight.
Waste gases are flowing by overpressure in the destruction chamber to a waste gas treatment unit. The waste gas treatment units can be of different kinds, chemical reverser process, plasma process or conventional afterburner and filtration systems. Due to the indirect heating system the quantities of waste gas are very low, normally less then 200 m^3N per hour.

3. Safety aspects

The inner chamber is designed to withstand effects from exoplosions such as blast wave and fragments. This is the first containment level. If a partial collapse should occur by any unforeseen circumstance there is a second containment level which is the outer chamber wall which is also capable to withstand a full detonation of the same size as the inner chamber. The third containment level should be the room or the space around the kiln.

4. Experience from operation of static kiln.

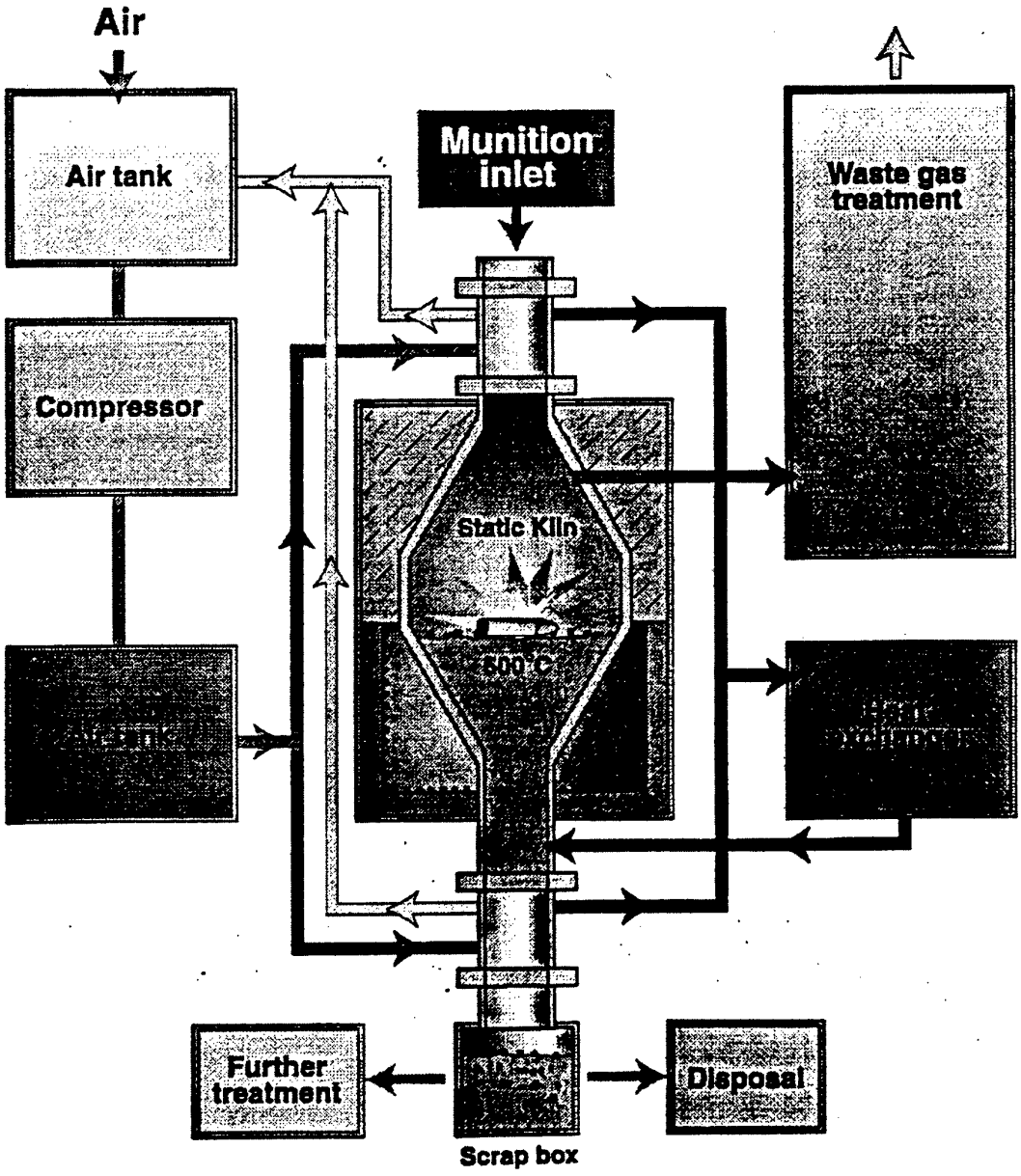
There are two Bofors Dynasafe static kilns in operation for destruction of conventional munitions, one in Spain and one in Sweden. Another unit is under manufacture and scheduled for delivery to the Swedish Defense

Material Administration in June 1998. The unit in Spain is connected to a Chemical Reverser waste gas treatment system (MODEX) and has been in operation for approx. one year.

Testing of destruction of chemical munitions has successfully been carried out in Sweden in an Olcon pilot static kiln with munitions ranging from 20 mm up to 155 mm with simulated liquid chemical agents. Further tests will probably take place with other munition types in order to study the time from loading to reaction of the explosive part of different munitions, fragmentation of the shell body etc.

5. Conclusion.

Operation of Bofors Dynasafe static kiln and tests with Olcon pilot static kiln has shown that the equipment is capable to demil chemical munition without prior disassembly of the munition. Using a hot atmosphere and a long exposure time guarantees the destruction of the explosives part in the munition. Chemical agents will decompose or be transformed into gaseous form for further treatment in the waste gas treatment system. Olcon static kiln can easily be connected to different suitable waste gas treatment systems and can hopefully be a contribution to chemical weapons demilitarization programs.



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WHOLE SHELL DISSOLUTION FOR WASTE MUNITIONS TREATMENT

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Abstract

Testing was conducted to assess the viability of using acid solutions to dissolve chemical munitions in the interest of effecting their treatment. Steel coupon testing was used to select the type of acid and the temperature range to efficiently dissolve full scale muntions. Full scale inert chemical munitions were dissolved using concentrated nitric acid at 50 to 70 °C. Complete digestion was possible in 2 to 4 hours. Laboratory tests were conducted to determine whether explosives would be sensitized in the digestion process. Results indicated that the explosives tested were not sensitized in the presence of the digestion liquors. Laboratory tests were conducted to determine the reaction extent between the digestion liquors and the chemical warfare agents HD, GB, and VX. Substantial reaction was observed between spent digestion liquors and these agents. Indications are that the nitric acid digestion liquors can provide sufficient destruction of these chemical agents. Concepts for the use of acid digestion for treatment of both chemical and conventional munitions are presented.

Keywords

Acid Digestion, Chemical Weapon, Explosives Treatment, Mustard, HD, Sarin, GB, VX, Non-Stockpile, UXO/EOD, Demilitarization, Assembled Chemical Weapons

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Background

Treatment of waste munitions is receiving greater attention as the Chemical Weapons Stockpile is destroyed, as recovery operations at Formerly Used Defense Sites (FUDS) and at Base Realignment and Closure (BRAC) sites occur, and as foreign stockpiles and recovery sites are declared. In the past, treatment of unserviceable or recovered munitions was simple: put the munitions in a pit and burn or detonate them in the open air. This practice has received some measure of public concern and its practice has been severely curtailed. This is especially true for the case of chemical weapons. It is becoming desirable to have the means to access the filling of recovered munitions to effect a safe and publicly acceptable treatment process such as neutralization or other treatment. Examples are the reverse assembly process used in the Chemical Stockpile Disposal Program (CSDP) and in the Munitions Management Device (MMD) family of treatment processes being developed for the Non-Stockpile Chemical Materiel Program (NSCM). In the stockpile program, leaking munitions are difficult to process as are munitions which are resistant to disassembly. In addition, the CSDP is faced with investigating alternative technologies to incineration which may require other than reverse assembly techniques currently being used. For conventional munitions, safe and reliable means other than open burning and open detonation are in the developmental phases and processes which concurrently access and allow for treatment of the explosive contents of munitions are not readily available. Munitions which pose particularly difficult treatment and disposal problems are those which contain depleted uranium (DU). Also, munitions which, due to exposure to the elements, are no longer easily handled, are difficult to treat.

Various methods have been proposed or attempted for accessing the fill from waste munitions. These methods fall into two broad categories, mechanical approaches and explosive approaches. Most waste munitions consist of a steel casing, filled with a solid explosive or liquid chemical agent. They may be fused or not fused and the chemical munitions may contain bursters. Their structural condition, especially those recovered from the ground, is usually unknown. This makes the use of mechanical systems for access difficult. Unless warranted by safety conditions, the use of explosive means of treatment has met resistance from the public due to potential uncontrolled release of chemical agent vapors.

This project succeeded in developing a simple means of accessing the fills of waste munitions within a contained system. Feasibility of chemically reacting the munition casing (i.e. the steel munition body) to eliminate it as a container was demonstrated. This involves dissolving the steel munition body with a suitable solvent/reactant. Once the munition body is breached, the contents are made accessible for removal or continued treatment. It was possible to dissolve the entire munition casing leaving the contents completely exposed. In the case of conventional munitions, the explosive fill may or may not be dissolved and could potentially be recovered for alternative uses. For liquid chemical fills, such as chemical warfare agents, once the munitions casing is dissolved, the remaining liquid can be treated as necessary to render it non-hazardous For recovered old munitions and for those munitions in the larger stockpile sites which are leaking, this process could provide a safe and effective means of treatment.

Sensitivity of the waste munition explosive contents was an early concern[1] as was the ability of the acid solutions to treat chemical agent fills. The rate of dissolution vs. time as concentration of Fe increases in the bath had to be addressed as it applies to the sensitivity question as well as to the agent neutralization.

Determination of explosives sensitivity as well as chemical agent treatment was investigated using standard U.S. Army methods of analysis.

Approach

The approach to this project was to demonstrate the potential for the waste munitions dissolution process using actual munitions bodies. Short initial tests on steel coupons were conducted to determine optimum dissolution rates. These tests included varying the reagent, temperature, and sparging of the dissolution baths.

Concurrent with the laboratory testing, a dissolution cell capable of processing an actual (inert) 155 mm projectile, was assembled. Once the optimum dissolution conditions were determined in the laboratory testing, the dissolution cell was operated using 2 inert 105 mm projectiles and 2 inert 155 mm projectiles. The test projectiles were filled with ethylene glycol. Measurement of temperature and dissolution rate were made as well as qualitative observations on the dissolution process. It was determined that dissolution rate is an important factor in full scale shell dissolution operations.

Following initial digestion testing, it was determined that the process exceeded earlier digestion concepts as far as rates of dissolution were concerned.[2] At this point, additional testing on the energetics and on chemical agents was necessary to establish whether the process could be conducted safely. Testing was conducted at the U.S. Army Research Laboratory at Aberdeen Proving Ground, Maryland, USA to determine the sensitivity of energetic compounds undergoing the digestion process. Chemical agent destruction in the digestion liquors was tested at the Army Material Command Treaty Laboratory located at the Edgewood Area, Aberdeen Proving Ground, Maryland, USA.

Experimental Design

Experiments on direct dissolution were conducted as screening experiments. As this technology was considered conceptually simple, initial experiments were conducted solely to determine rate and ability to process. Following the dissolution tests, more detailed experiments were conducted on the explosive contents of the munitions and on the chemical warfare agent fills.

Coupon Testing

Testing was conducted with small rectangular steel specimens (coupons) fabricated from A-516 carbon steel (a mild steel alloy similar to that used for projectile bodies). A variety of dissolving solutions were tested; most involved nitric acid. Other solutions, such as hydrochloric acid and hydrochloric/nitric acid mixtures, were tested, but were found to be less effective on steel. Testing was conducted over a wide range of temperatures. Nitric acid at temperatures between 50-70 °C was ultimately selected for the full scale test process to dissolve steel-bodied SETH munitions.

The coupon testing was conducted in jacketed 250-mL beakers on a hot plate. Cooling water was pumped through the jackets for temperature control of the exothermic reactions. The hot plate was used initially to preheat the solutions. Coupon testing typically used about 150 mL of acid solution with a 10-gram coupon. Some testing was done with additional steel to assess the effects of higher iron concentration on reaction rate.

Simulated Equipment Test Hardware (SETH) Testing

Testing was conducted using SETH 105-mm and 155-mm projectiles provided by the U.S. Army Program Manager for Chemical Demilitarization. The shells, as delivered, were assembled with inert fuses, inert detonators, inert bursters, and agent simulant (ethylene glycol). The 105-mm rounds weighed approximately 35 pounds; the 155-mm rounds weighed approximately 100 pounds. Testing was done with both fully assembled rounds and rounds that had been partially disassembled (detonator and burster removed). The as-received shells were fully painted. The paint was left on the shell for 3 of the tests. The paint was removed from one of the shells to determine the effect of de-painting on dissolution rate.

Testing with the SETH rounds was conducted in a Teflon-lined 30-gallon vessel, about 1 foot deeper than the tallest shells (155-mm) to allow total submergence of the shells (see Figure 1). These tests were conducted in a flow-through configuration. Acid was pumped from a pre-heated supply tank into the reactor after the shell was sealed inside (see Figure 2). Thermocouples in the reactor monitored temperature throughout the process. A bottom drain in the reactor returned partially reacted hot acid to the supply tank via a water-cooled heat exchanger that cooled the acid.

The SETH projectiles were dissolved to various degrees of completion in heated nitric acid solutions, each time at least penetrating the rounds and releasing the simulated agent (glycol solution), while causing significant overall corrosion of the body and other components.

Explosives Sensitivity

Laboratory tests were conducted to investigate the effects of the nitric acid solution on the energetic materials contained in the bursting charge and fuses of the chemical weapons. The tests were performed to determine if byproducts were formed during the acid dissolution process of the munition body which were sensitive to electrical discharge, impact, or subject to violent chemical reaction.

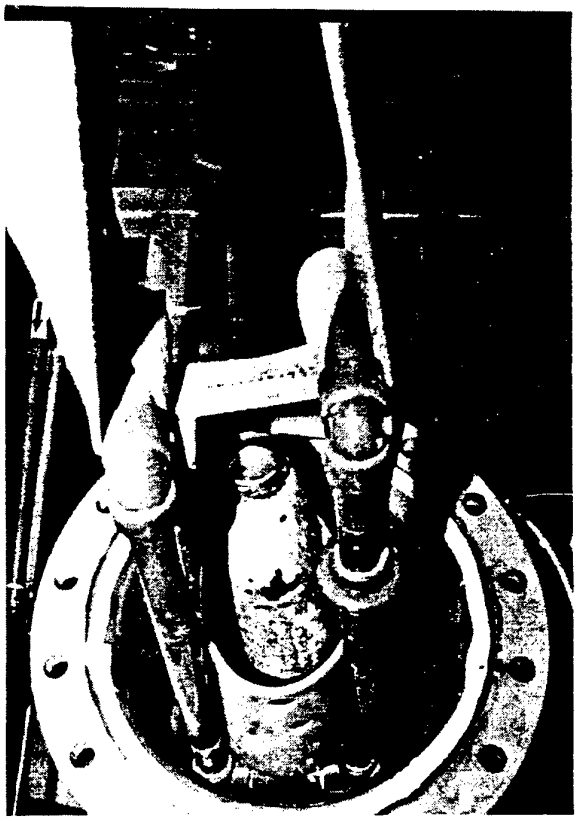


Figure 1: 30 Gallon Teflon Lined Reactor Used in Seth Munition Testing

A series of batch tests were performed on two explosives, RDX and TNT, to determine if sensitive byproducts are formed by the reaction of nitric acid and dissolved metals.

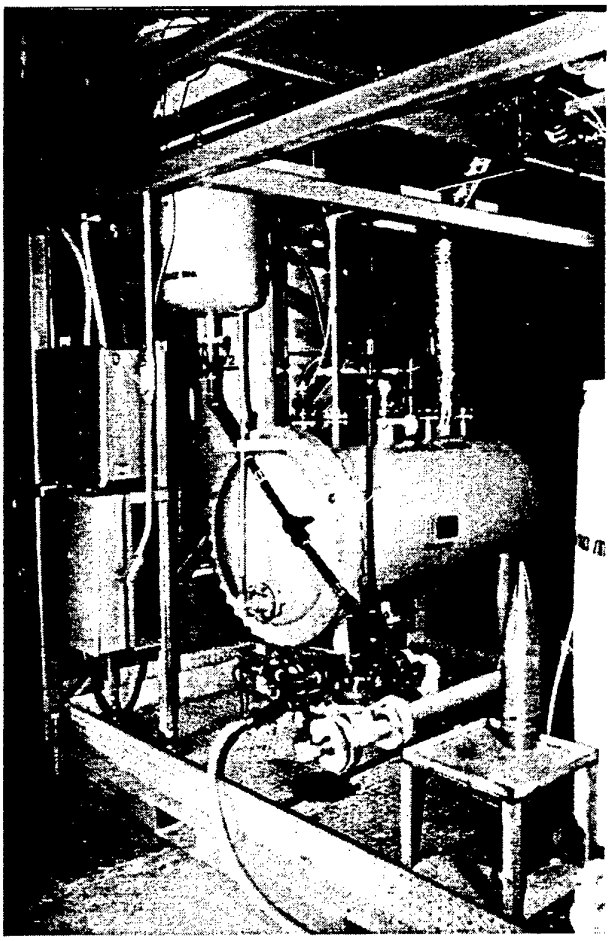


Figure 2: Picture of SETH Munition Test Skid Showing Large Acid Reservoir (bottom) and 30 Gallon Reactor (top left)

These two explosives are components of the bursting charge of many chemical munitions. A third explosive, Tetryl (N-Methyl-N,2,4,6,-Tetranitrobenzenamine) is also found in chemical munitions. However, tetryl was unavailable for evaluation during these tests.

Figure 3 is a flow diagram of the energetics evaluation program. The figure shows the steps in preparing the samples and the analyses that were performed on the soluble and insoluble residues.

First, the test solutions were prepared by adding metal shavings from the casing of a 105 mm howitzer round to a beaker and dissolving the material in 7M HNO_3. Two different acid solutions with different concentrations of dissolved metal and unreacted acid were prepared. The two solutions reflected the different conditions that exist in the reaction vessel during the initial and late dissolution stage of the munition.

Next, small quantities of each of the explosives were added to the acid solutions. The solutions, with explosive, were heated to 60°C. This is the reaction temperature for the proposed process. After one hour, the solutions were allowed to cool then filtered to separate the insoluble material from the material that remained in solution with the acid.

The insoluble material was oven dried. The soluble fraction was evaporated to remove most of the HNO_3 then oven dried under vacuum to obtain a dry material for evaluation.

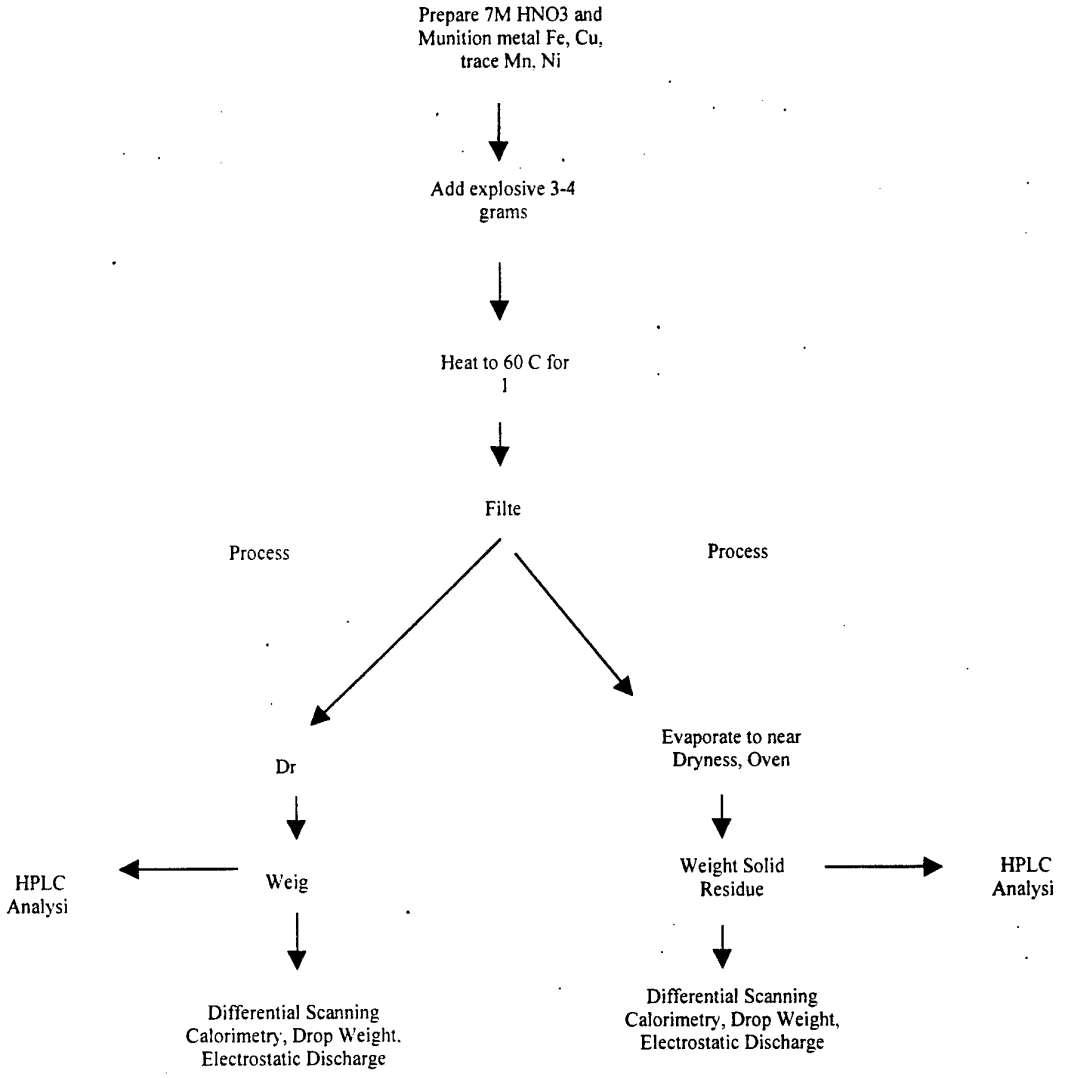


Figure 3: Energetics Sensitivity Test Program Carried Out at Army Research Laboratory

To determine the amount of each explosive present in the soluble and insoluble fractions, the dried samples of soluble and insoluble material were analyzed by High Pressure Liquid Chromatography (HPLC) and Differential Scanning Calorimetry (DSC). These methods were also used, along with an analysis of an unreacted sample of the same lot of explosive as that was used for the tests to determine if the explosives had reacted to produce other organic impurities.

The samples of soluble and insoluble materials were also tested for ignition sensitivity using Drop Weight (DW) and Electrostatic Discharge (ED) testing. These tests determined if the residues were more sensitive to initiation than the original explosive.

Finally, batch tests were also conducted in which Potassium Chlorate ($KClO_3$) and Potassium Perchlorate ($KClO_4$) were added to the acid and explosive at 60°C. The batch tests were intended to check for gross reactivity of the explosive as evidenced by detonation, violent reaction, color changes or gas evolution. Both $KClO_3$ and $KClO_4$ are components of the igniter or fuse element of some chemical munitions.

Chemical Agent Destruction

Testing was performed using weapons grade HD, GB, and VX of the following lots:

GB: Sarin Lot No. GB-S-6030-CTF-N
HD: Mustard Lot No. HD-S-5164-CTF-N-4-S
VX (U.S. version); Lot No. VX-S-5312-CTF-N-4

Ten mL of GB was used in each of 3 tests. The concentration of GB in the neutralization solution was 9 % by volume. Only six mL of HD and VX were used in each of 3 tests due to agent availability. The concentration of HD and VX in the neutralization solution was 9.1 % by volume. The neutralization solution was spent acid digestion solution from SETH munition testing conducted earlier. The same batch of solution was used for each of the tests.

Tests were conducted in 250 mL round bottom flasks heated with a hot plate, and mineral oil bath. The spent digestion fluid was placed into the flask and brought to 60°C. The chemical agent was added over a 2-3 min interval and the reaction mass allowed to react at 60°C for a total of 30 minutes. The reaction mass was then cooled to 30°C and two samples were taken for analysis.

The analytical test methods used were as follows:

- HD – U.S. Army Alternative Technologies Program (ATP) Method HN-02
- VX - U.S. Army Alternative Technologies Program (ATP) Method VN-02A
- GB - Method ROBAT 0080 (Battelle method developed for GB in neutralization solutions; PMCD Class One P&A (spike and recovery) performed for this method. A copy of this method is on file at the AMC Treaty Laboratory.

The Quality Assurance/Quality Control for the operations, including the chemical analysis, was in accordance with the AMC Treaty Laboratory ISO 9001 Certified QA/QC program. All chemical methods included QA/QC validations such as 5 point calibration curves, check standards; and matrix spike recoveries as specified in the individual analytical methods.

In addition to direct chemical agent neutralization testing, testing was conducted to determine the degree of decontamination of munition casings following contact with the digestion fluid to determine the degree of decontamination achievable without complete digestion.

Metal coupon testing was conducted by dropping agent onto steel coupons, immersing them into the digestion liquor, waiting 10 minutes, removing the coupons (without rinsing), bagging the coupons, and testing for agent using sorbent (DAAMS) tubes after 4 hours.

Results of Experiments

The results of the four separate experimental activities from this project are provided below. Complete reports provided under Testing and Evaluation at Army laboratories for the explosives sensitivity testing and the chemical agent destruction testing were prepared.[3,4]

Coupon Testing (steel coupon dissolution)

As described above, the initial testing was performed with small steel corrosion specimens. The steel alloy was ASTM A-516, a mild steel similar to the steel used to fabricate projectile bodies such as Howitzer shells. The coupons were rectangular (approximately ½ inch x 1 inch x 1/8 inch), each weighing approximately 10 grams. These coupons were suspended in beakers of the acid solutions to be tested using inert Teflon thread.

Temperature has a large effect on the dissolution rate of steel. Dissolution rates at ambient temperatures (20° to 25°C) are quite low (inches per year), while at slightly elevated temperatures (50° to 70°C), rates increase to inches per day. The reactions of steel dissolving in nitric acid are quite exothermic and require cooling to moderate the temperature, even though preheating may be used to initiate the process at a suitable rate.

Nitric acid was determined to be the most effective solution tested for steel dissolution. Hydrochloric acid and hydrochloric/nitric acid mixtures were also tested, but gave slower dissolution rates. A wide range of nitric acid concentrations was evaluated (3 M to 10 M) to select an optimum acid strength for shell dissolution. Acid

concentration of about 8 M produced the highest rates of steel dissolution. A starting concentration of ~8 M nitric acid was then used for subsequent full-scale SETH munition tests.

Concentrated nitric acid is a very strongly oxidizing acid and will passivate the surface of steel, thereby reducing its dissolution rate. Dilute nitric acid is too weak to dissolve steel at an appreciable rate. However, nitric acid at a concentration of about 8 M dissolves steel at very high rates.

For the coupon tests, approximately 150 mL of acid solution were used per test, with steel coupon mass ranging from 10 to 20 grams. Sampling and analysis were based on iron going into solution. This was determined by analyzing samples of the acid for iron and free acid, and by periodically weighing the coupons during the test to determine weight loss.

Steel dissolution rates for the coupon tests at the optimum temperature and acid concentrations were determined, by weight loss, to be about ½ inch per hour. A solution consisting of ~8 M nitric acid preheated to ~50°C gave acceptable dissolution rates, while still being a controllable process for subsequent full-scale testing.

Approximately 100 separate tests were conducted with steel coupons in various combinations of solution type, concentration, and temperature. Figure 4 shows results of selected coupon tests, illustrating the effects of temperature and acid concentration on steel dissolution rates. In this figure, the steeper curves represent faster dissolution rates. Temperatures of 50°C were the highest tested with the coupons. The exothermic reactions made temperature control difficult above 50°C in the beaker tests due to the limitations of the jacket for removing heat. Acid concentrations of about 8 M gave high reaction rates without passivating the steel surfaces.

The coupon testing was conducted in jacketed 250-mL beakers on a hot plate. Cooling water was pumped through the jackets for temperature control of the exothermic reactions. The hot plate was used initially to preheat the solutions. Coupon testing typically used about 150 mL of acid solution with a 10-gram coupon. Figure 5 shows a time series of photographs of coupon testing.

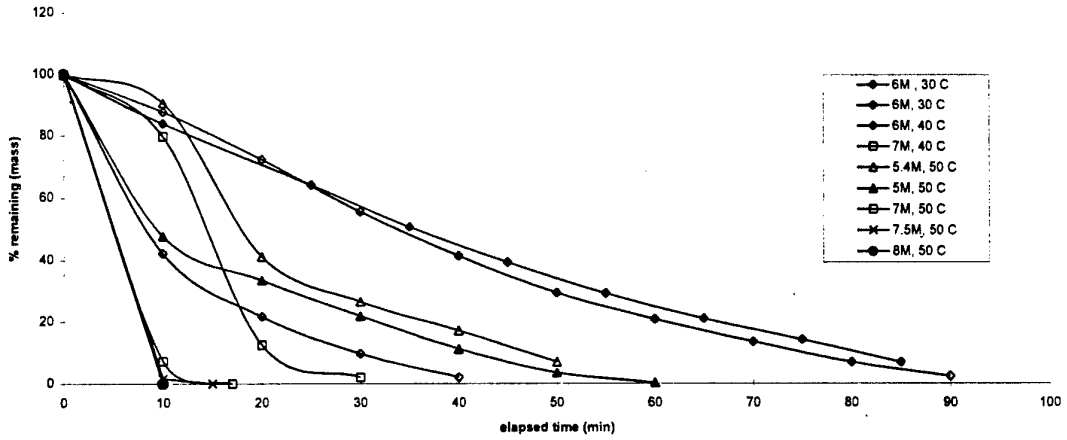


Figure 4: Steel Coupons in Nitric Acid

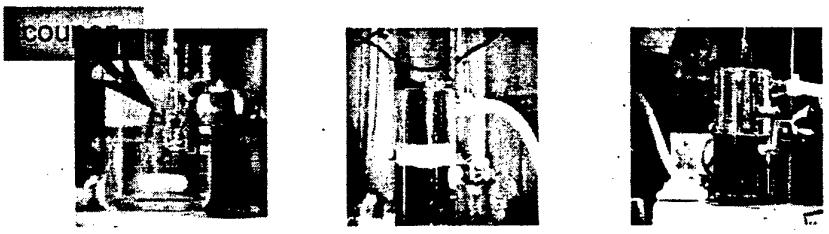


Figure 5: Coupon Testing Sequence

SETH Testing

Four full-scale tests were conducted with 105-mm and 155-mm SETH munitions. These tests were conducted with starting acid concentrations of about 8 M nitric acid. Complete dissolution of the munitions was effected after a typical duration of 2 to 4 hours at 50° to 70°C depending on the type of munition (105-mm or 155-mm). The time required for dissolution increased with lower temperatures. However, it is clear that rates would increase at higher temperatures.

The effluents from this dissolution step were solid, liquid, and gaseous. The solid effluents were composed of potentially unreacted steel (depending on degree of reaction desired), unreacted non-steel components (most likely aluminum, since brass components react even faster than steel), and some unreacted paint flakes. Liquid effluent from the digester was a spent nitric acid solution containing some free acid and dissolved metals. Steel dissolution in nitric acid generates a substantial quantity of NO_x gas. Gaseous effluents from the reactor consisted primarily of NO_x, acid, and water vapor.

Figure 6 shows the results of a test conducted with a 105-mm SETH munition. Following approximately 2 hours at a temperature of 50° to 70°C, the acid was drained and the contents of the reactor were removed. The steel was almost totally dissolved. Only small pieces remained on the shell body, and they would likely have dissolved in a few additional minutes of reaction. The burster casing retained its shape, but had undergone significant corrosion. The brass components were totally dissolved. The aluminum detonator cup and fuse well had been uniformly corroded, but not completely dissolved. Other tests with the SETH rounds were taken to varying (but similarly nearly complete) degrees of complete dissolution of the steel bodies.

The results shown in Figure 7 indicated that removing the paint allowed a more uniform attack, but had little overall effect on the rate of penetration of the steel. In general, attack began at the detonators and brass rotating rings and quickly breached the shell bodies, allowing acid access to the interior of the shells. Attack also initiated at any flaws (e.g., pinholes and scratches) in the paint, quickly dissolving large holes through the steel under the flaws.

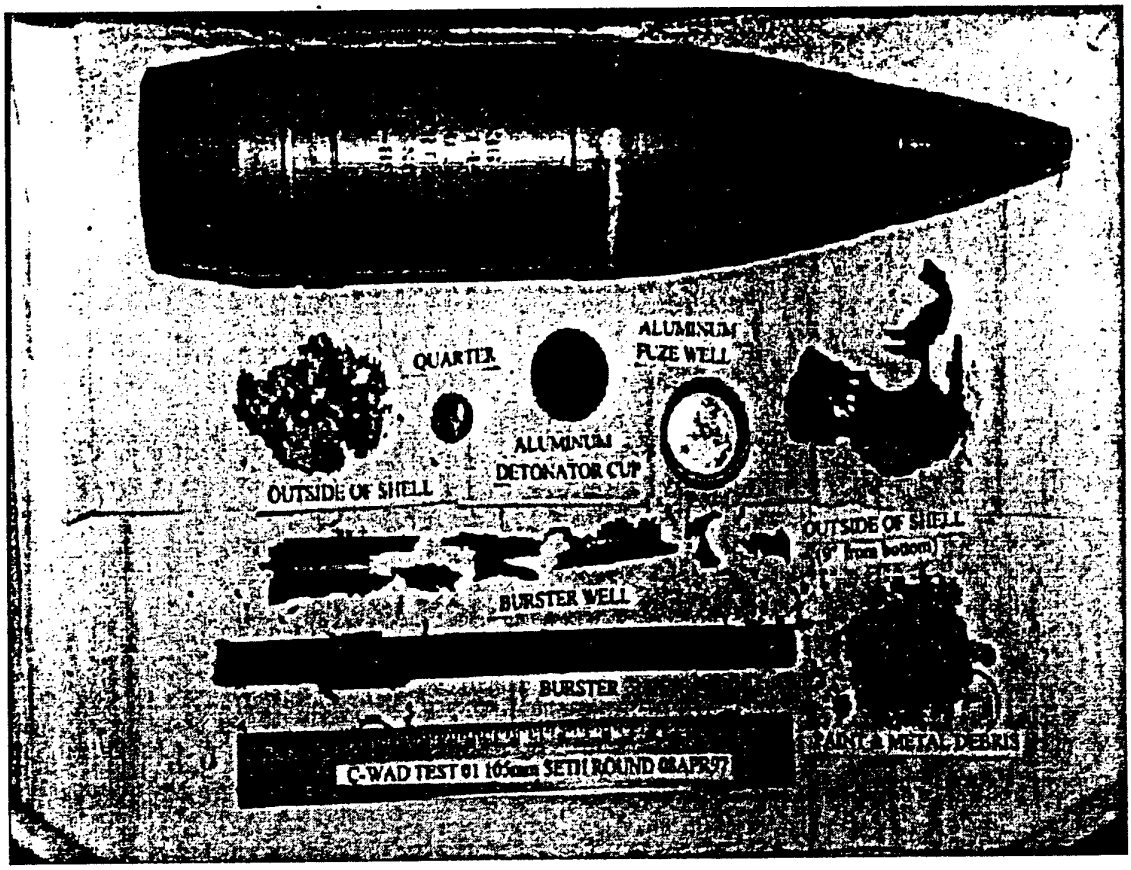


Figure 6: Acid Digestion of Complete 105mm Munition

Explosives Sensitivity

The tests showed that the portion of the explosive that dissolves in the nitric acid and the insoluble residues that remain in the reaction vessel are not sensitized by the formation of reactive byproducts. The explosives, TNT and RDX, do not react with the acid solution and are not sensitized by exposure to the acid or the dissolution residues.

The insoluble residues were determined to be primarily undissolved and unreacted explosive. The soluble fraction contained the dissolved and digested base metal of the shell casing and some dissolved TNT. The separated and dried soluble and insoluble

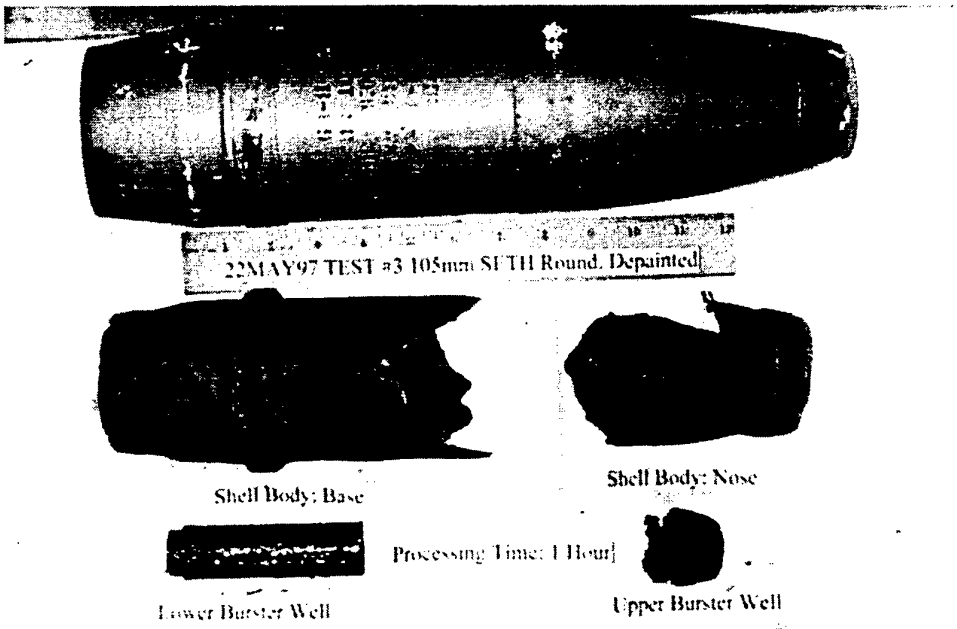


Figure 7: Acid Digestion of De-Painted 105mm Munition without Fuse or Burster

residues were found to be no more sensitive to electrostatic discharge or impact than the untreated military grade explosives.

The HPLC and DSC measurements did not detect any organic impurity formed during the acid treatment. The only organic impurities that were measured were those already present in the unpurified military grade explosive.

An additional result of the energetic sensitivity testing related to process efficacy was that the iron nitrate formed through the acid reaction with the steel munitions body was found to decompose at temperatures above 125 °C to iron oxide. This result is significant in that the product stream from dissolution can be easily converted at low temperatures to an innocuous product (iron oxide) with the the subsequent formation of NO_x which can be recovered as nitric acid and reused in the process.

Chemical Agent Destruction

Conversion of the chemical agents is described here as destruction efficiency. Agent destruction efficiencies will depend largely on the time the agent is contacted with the nitric acid, the concentration of the acid, and the temperature. The longer the time of contact, the higher the concentration of acid and the higher the temperature, the greater the destruction efficiency. Estimates of destruction efficiency for HD, GB, and VX are provided in Table 1.

Table 1: Results of Chemical Agent Neutralization in Acid Digestion Liquors

Agent	Estimated Destruction Efficiency [1,2]	Reaction Time	Temperature
HD	>99.9992%[3]	30 min	60 °C
GB	>99.978%[4]	30 min	60 °C
VX	87.437%[5]	30 min	60 °C

1 Destruction efficiency calculated as [(mass of agent in – mass of agent out)/mass of agent in] x 100%. Mass of agent in is taken as volume of agent in times the density of agent. No adjustment was made for the purity of the weapons grade agent.
2 These destruction efficiencies are considered overly conservative. Greater than worst case agent to acid solution concentrations were used. Acid solution was spent solution from SETH munitions testing.
3 Average of three tests
4 All tests resulted in below quantifiable limits of GB.
5 VX is more resistant to chemical neutralization and will require longer reactions times, greater concentration of acid, higher temperatures or a combination of these factors. Assuming pseudo first order kinetics, a 99.99% DRE could be reached in 2.5 hours.

The results of the chemical agent neutralization tests are that the nitric acid digestion fluid is an effective means of destroying the chemical agents HD, GB, and VX. Demonstration of the technology can proceed with a high degree of confidence that chemical agents HD and GB will be effectively destroyed without modification of the current design basis. VX may be effectively destroyed with additional time, temperature or concentration of digestion liquor.

The summary results for metal parts decontamination are that the acid digestion process is able to rapidly decontaminate metal parts to a XXX (3X) condition. Of the three agents tested, the HD contaminated metal parts were the most consistently decontaminated to below the 3X threshold. Both GB and VX coupons were not as decisively decontaminated at the end of 10 minutes but the data showed that the method was capable of attaining a 3X level of decontamination. It was concluded that a longer period of contact would be required for both GB and VX contaminated metal parts.

The scale of the testing on surface decontamination was 1 inch by ½ inch metal coupons. The time allowed for decontamination was 10 minutes.

Complete results of agent testing were docuemnted by the AMCTL in the report Evaluation of Chemical Agent Neutralization During Whole Shell Dissolution.[2]

Process Concepts

The use of acid digestion for processing various munitions or waste containers may prove both viable and cost effective. While initially thought of as a technology for treating leaking chemical munitions or munitions which are difficult to disassemble, the acid digestion process may find application in other areas.

During the course of this research project, one application came into focus due to a program instituted in the Army to investigate alternatives to the baseline incineration process for the chemical agent stockpile. Other applications are still in the concept mode and will be discussed briefly.

Alternative Technology for Assembled Chemical Weapons

In the early 1980s, the U.S. Army sought alternatives for demilitarizing chemical munitions to replace chemical neutralization methods employed at that time. One alternative concept which was considered was titled Acid Roasting[2], a process in which the munitions would be placed in large tanks and immersed in acid. The acid would dissolve the steel munitions body and expose the chemical agent and explosives for treatment. Treatment consisted of heating the spent acid solution containing agent, explosives and iron salts to 1600 °F to destroy the agent and explosives while oxidizing the iron salts to iron oxide allowing for the recovery and reuse of the acid. Thus, the roasting process provided for destruction of both the agent and the explosives.
The Army commissioned an independent review of the technology. The findings, based principally on lack of data and to the particular processing requirements was that the concept did "...not warrant further development because of the lack of any cost savings and potential safety problems."[1]

To address the first concern, coupon tests and full scale SETH munitions testing was conducted. This testing program indicated that the munitions could be completely (>99%) dissolved using nitric acid in much less time than the originally estimated 20 to 28 hours. This advancement came from using nitric acid based solutions rather than hydrochloric acid.

Having dispelled the first fault of this process, answering the second finding required the use of sophisticated methods of analysis for explosives and chemical agent destruction. Testing was conducted at the Army Research Laboratory at Aberdeen Proving Ground to assess the sensitivity of the explosives during the dissolution process. Using the military's proscribed methods of analysis for sensitivity and chemical analysis, the U.S. Army Research Laboratory found that the explosives were not sensitized. This testing partially dispelled the issue of explosives safety for the digestion process.

The effectiveness of nitric acid as a digestion solution provided a secondary benefit for chemical munitions destruction. Nitric acid is a recognized decontaminant for chemical agents. The oxidizing power of nitric acid is sufficient to effect reactions with the chemical agents and thus render them neutralized. As the nitric acid is aqueous, hydrolysis reactions also occur. While the use of nitric acid as a standard decontaminant is not recommended due to its corrosive properties, it is this corrosiveness which makes it the acid of choice for the digestion process. The agent is both accessed and destroyed by the nitric acid solution in a contained environment. Vapors of agent are not released to a room but are contained in the process and destroyed. Munition bodies are no longer a problem for decontamination due to the aggressive action of the acid. If desired, the entire munition can be dissolved into solution.

The resulting digestion liquor has to undergo further treatment to destroy the products of agent destruction and the explosives. Review of available processes led to selection of a Hydrothermal Treatment Process (HTP) for the treatment of the digestion liquors. These processes have been used, since the late 1950s, as treatment processes for various wastes. Commercial vendors of HTP have performed tests on explosives and compounds similar to those expected from the neutralization of chemical agents.

Other features of a full scale CWM acid digestion process are tanks designed to collect gaseous emissions from the process prior to release to the atmosphere. In this way, no effluent from the plant will be released prior to analysis for chemical agent. Recent analysis of the residues from the munition digestion indicate that the iron nitrates produced from the digestion process are oxidized to iron oxide at temperatures less than 300 °F. This allows recovery of the acid without the high temperature roasting operation described in the early 1980s, which also partially dispells safety concerns of the digestion process.
A simplified block diagram of the process is shown in Figure 8.

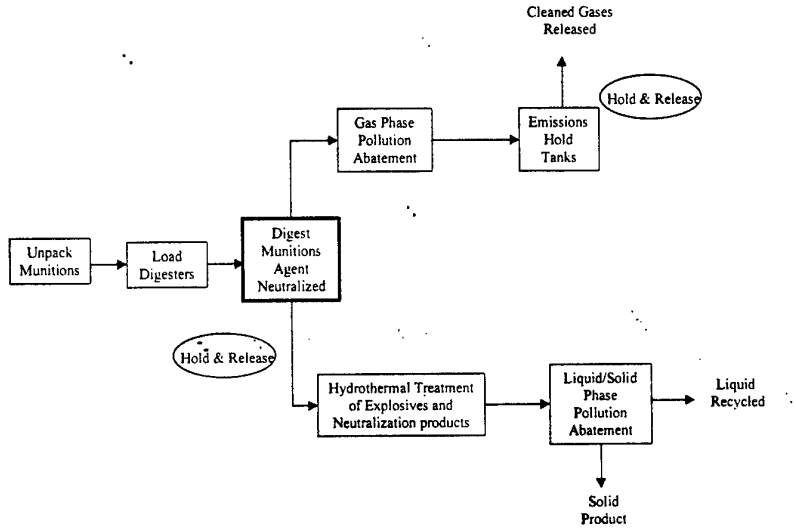


Figure 8: Simplified Block Diagram of Acid Digestion Process for Assembled Chemical Weapons

Chemical Stockpile Disposal Program Leaking Chemical Munitions

One of the originally conceived uses of Acid Digestion was to treat munitions in the Chemical Stockpile Disposal Program which were not amenable to the reverse disassembly process used in the baseline incineration process. During processing of chemical munitions using the baseline CSDP process, munitions are sometimes encountered which pose problems in disassembly. Some munitions have had their burster wells welded into the munition instead of the standard press-fit. Others are leaking and pose a safety issue during unpacking and handling. These munitions could potentially be treated using the acid digestion process in concert with the baseline incineration technology. A simple flow diagram for such a process is shown in Figure 9.

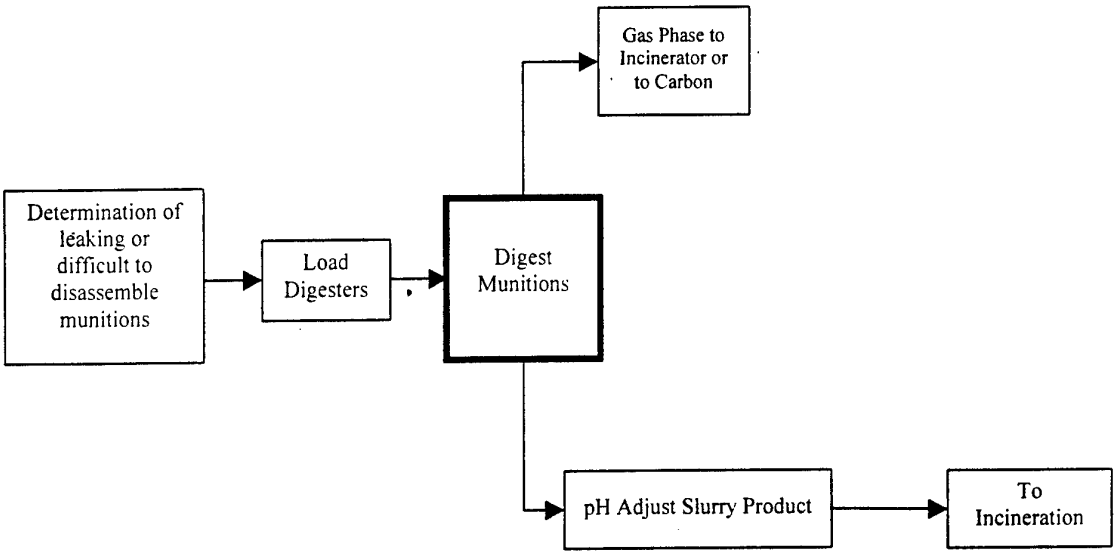


Figure 9: Concept Diagram for Leaking or Hard to Disassemble Munitions Using Baseline Incineration

Non-Stockpile Chemical Materiel

"House Appropriations Report 101-822, accompanying the fiscal year 1991 Defense Appropriations Act, stated that,

> The Program Manager for Chemical Demilitarization (PMCD) currently has responsibility for demilitarization only those items which were originally identified in 1986 as part of the CSDP (Chemical Stockpile Disposal Program). Not included are a host of lethal wastes from past disposal efforts, unserviceable munitions, chemically contaminated containers, chemical production facilities, subsequently located chemical munitions, sites known to contain significant concentrations of buried chemical weapons and waste, and binary weapons and components. The Committee therefore directs the Secretary of Defense to organize an overall program so that operational responsibility for all Defense Department chemical warfare activities rests within a single office which shall be fully accountable for total program execution." [5]

On October 1, 1992, the Army established the Program Manager for Non-Stockpile Chemical Materiel (PMNSCM) to manage the 5 non-stockpile categories:

- Former Chemical Weapon Production Facilities
- Buried Chemical Warfare Materiel
- Recovered Chemical Warfare Materiel
- Binary Chemical Weapons
- Miscellaneous Chemical Warfare Materiel

The PMNSCM is investigating means of safely managing these 5 categories. The use of Acid Digestion technology may find uses in 3 of the above categories, Recovered CWM, Buried CWM and Former Chemical Weapon Production Facilities.

Recovered and buried chemical warfare materiel consists principally of unserviceable chemical munitions, typically in over-packs. This category is amenable to acid digestion treatment. A concept for recovered munition treatment is shown in Figure 10.

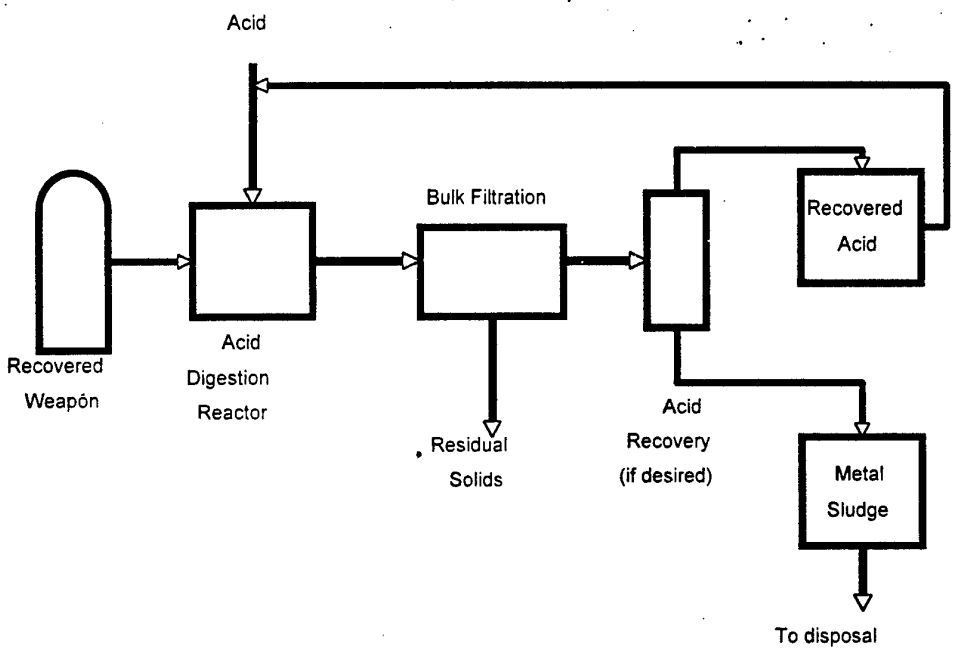


Figure 10: Concept for Recovered Munition Treatment Using Acid Digestion

This process would apply to buried munitions upon recovery. If possible, it is desirable to place the munition itself directly into the digester. However, non-stockpile munitions are often over-packed in separate containers due to safety concerns. This is true even on a burial site where munitions are being recovered. It is possible that the Acid Digestion process can be used on munitions without removal from the over-pack. This would greatly enhance the safety of a non-stockpile treatment process. The steps for treatment of an over-packed munition are as follows:

Process Steps	Purpose
1) Acid+Overpack	Dissolve Overpack/Flush Vermiculite
2) Acetone + Plastic Wrap	Dissolve Plastic Wrap
3) Acid + Plaster Seal	Dissolve Plaster Seal
4) Acid + Munition	Dissolve Munition and Neutralize Agent
5) Complete Treatment as Non-Agent Hazardous Waste	

Various solutions (e.g.- acetone for plastic bags) might be necessary to effect the complete treatment of an over-packed muntion.

Another Non-Stockpile materiel managed area which might find use for Acid Digestion technology is the destruction of former production facilities. These facilities typically contain equipment which might be contaminated with chemical agent or its precursors. For some equipment items, an acid digestion bath could be employed to completely destroy both the metal equipment part (e.g. pipe, reactor, heat exchanger, etc.) and the chemicals potentially contained therein.

Conventional Ammunition Demilitarization

"The demilitarization of the U. S. armed forces has led to an increase in the stockpile of [conventional] warfare materials. The current inventory is estimated at 400,000 tons and growing rapidly at a rate of 40,000 tons per year. The stockpile is distributed throughout the country at more than 200 Department of Defense (DOD) and Department of Energy (DOE) installations. Many of the materials in the stockpile are old, unstable and unsafe. The most common disposal method in use today is open burning (OB) and open detonation (OD). OB/OD activities are a relatively simple and cost effective means for stockpile reduction. However, these activities can generate air pollutants."[6]

OB/OD operations continue to face opposition from a concerned public and alternatives are currently being sought for safe, reliable, cost effective systems to treat these materials. The Acid Digestion technology could be used as part of a larger system for gaining access to the explosive contents of some of these munitions.

A particular area of interest for the Acid Digestion technology is in the treatment of munitions containing depleted uranium (DU). Safe disposal of DU munitions can result in substantial quantities of low-level radioactive waste. If explosives are present, these wastes will likely require treatment as mixed wastes. The Acid Digestion process has the potential to safely access the DU in these munitions. Once in solution, ion exchange processes similar to those used in nuclear material production could be used to separate the DU.

UXO/EOD

As with the recovered PMNSCM materiel, conventional ammunition which has been recovered after firing or after disposal may be treated with the Acid Digestion technology. The process would be identical to that for the chemical munition, only the explosives would require larger treatment systems or an explosives recovery system for disposal off-site.

Another possible use in the EOD field is as a bomb suppressing technology. Bombs could be placed into a digester, the appropriate corrosives added, and the bomb would be rendered safe. Of course appropriate explosion proof enclosures would still be required.

Conclusions

The use of acid digestion for the treatment of chemical munitions was determined to be a viable means for accessing chemical munitions and treating the contained chemical agent. Factors which would make the technology undesireable, (i.e.- lengthy rates of dissolution and sensitization of explosive components), were shown to be surmountable through appropriate selection of the acid used for digestion and use of appropriate process parameters

Energetics do not appear to be sensitized during the process. Chemical agents tested are effectively neutralized through the process providing a significant reduction in risk. The process is operable at low temperatures and pressures. Iron salts produced in the process can be processed again at low temperature and pressure to recover nitric acid and produce an innocuous solid product, iron oxide.

Concepts were presented for the use of Acid Digestion technology as an alternative technology for assembled chemical weapons, Non-Stockpile munitions treatment and former production facility treatment, Chemical Stockpile Disposal Program leaking munitions and hard to access munitions, conventional munitions demilitarization, and use in UXO/EOD operations. Demonstration testing will be required to field the Acid Digestion technology in any of these areas.

References

1 Evaluation of Acid Roaster Demilitarization Technology Concept, Working Paper, MITRE Corporation, Contract # 68-03-3159, USATHAMA, April 1985.

2 Evaluation of Alternative Thermal Processes for the Disposal of Obsolete Chemical Weapons, Phase 1 Final Technical Report, Rockwell International, Report # DRXTH-TE-CR-83213, USATHAMA, April 1983.

3 CP034783 Quick-Look Data Report on Acid Dissolution of Chemical Agent Munitions Soluble and Insoluble Residue Senstivities, Michael P. Failey, Battelle, September 11, 1997.

4 Final Report, Test and Evaluation Contract Task 97-003-C, Evaluation of Chemical Agent Neutralization During Whole Shell Dissolution, AMC Treaty Laboratory, September 3, 1997.

5 Non-Stockpile Chemical Materiel Program Survey and Analysis Report, US Army Chemical Materiel Destruction Agency, November 1993.

6 US EPA, Internet Reference, http://www.epa.gov/asmdnerl/obod.html, 15 February 1996.

Whole Shell Dissolution for Waste Munitions Treatment

Presented at:

International Symposium on Destruction of Chemical Weapons

Munster, Germany

24 March 1998

Craig A. Myler

Battelle

Defense Technology

Proceedings of the International CW Destruction Symposium
Munster, Germany, March 22-25, 1998

Concept

- Whole munition is dissolved using acid
- Chemical Warfare Agent contents are neutralized
- Explosives are post treated and acid recovered

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Munster, Germany, March 22-25, 1998

Background

- 1983 Battelle Study Suggests Acid Roasting
- 1985 MITRE Evaluation
- 1996 Battelle Develops Acid Digestion

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1996 Battelle Study

- Independent Development Project
- Demonstration Using SETH Munitions
- Agent Destruction Conducted Using Approved Methods (AMCTL)
- Explosives Sensitivity Evaluated Using Standard Methods (ARL)

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Battelle Study Activities

- Demonstration Using SETH Munitions
- Agent Destruction Conducted Using Approved Methods (AMCTL)
- Explosives Sensitivity Evaluated Using Standard Methods (ARL)

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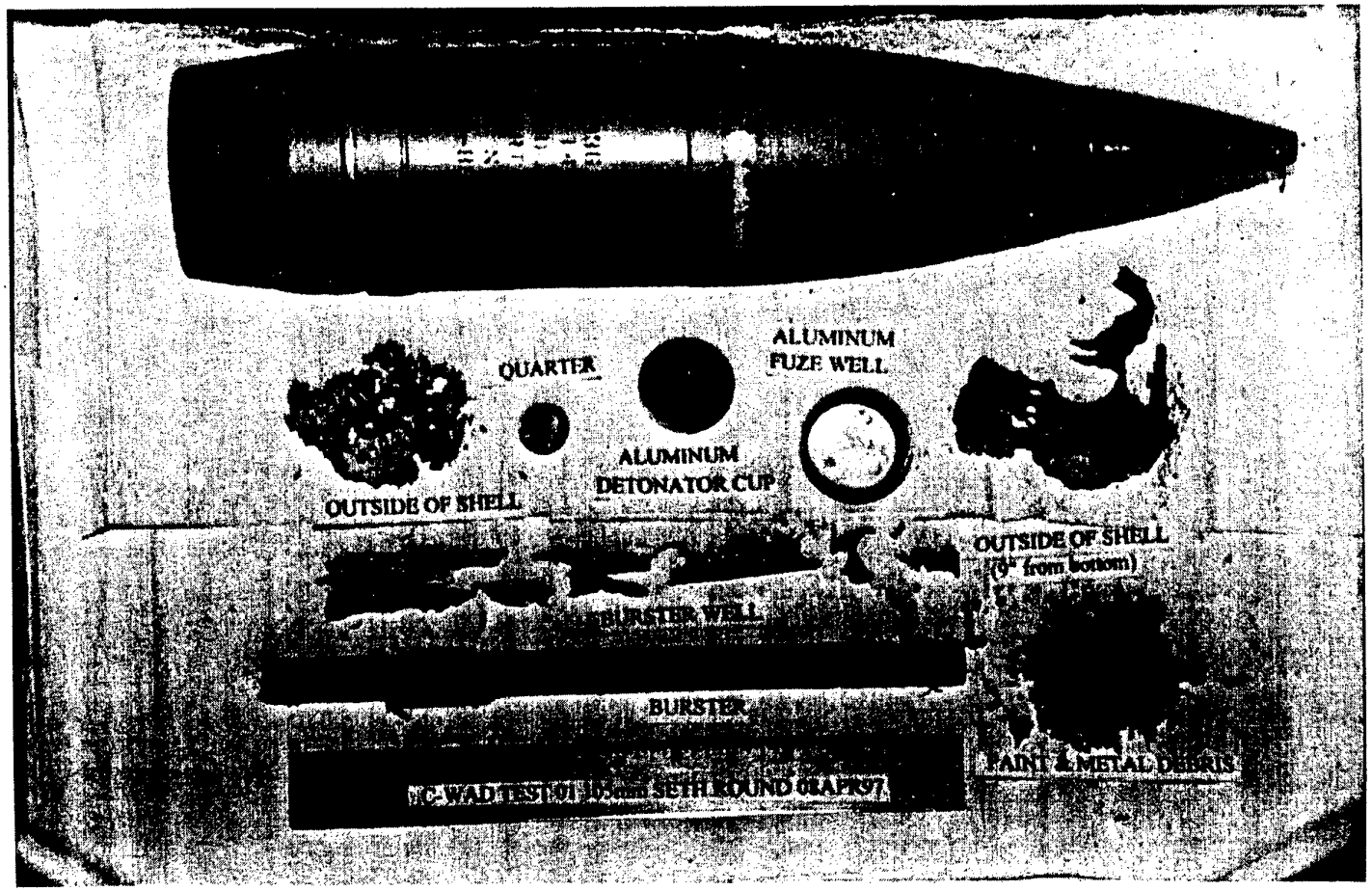
Results

- Rates for complete digestion (~ 2 hrs possible)
- Energetics not sensitized
- Agent degradation (GB,HD,&VX) demonstrated
- No high-temperature processing step
- Decomposition for acid recovery at ~125 °C

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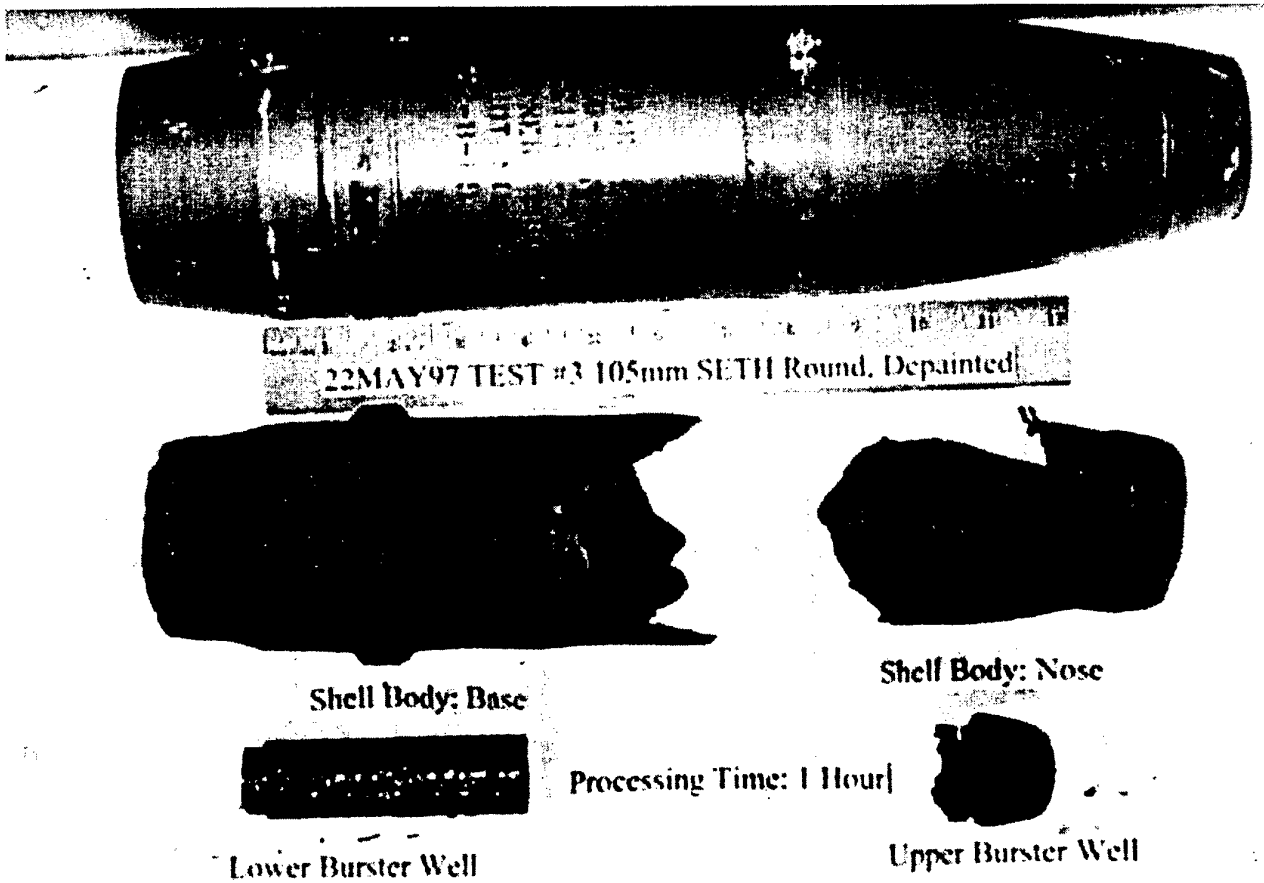
Example Performance - Complete Digestion



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Example Performance - Partial Digestion



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Solid FeO_2 from "Roasting" Reaction



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Advantages

- Comprehensive Treatment Possible
- Safety aspects
  - minimize handling
  - render safe
  - reduced agent emissions
- Chemical agent neutralized by digestion fluid
- Batch Process
- Low Temperature (<200 °F) Neutralization
- No exotic chemicals or operations
- Sample and Release
- Process is easily controlled
- No exotic chemicals or operations

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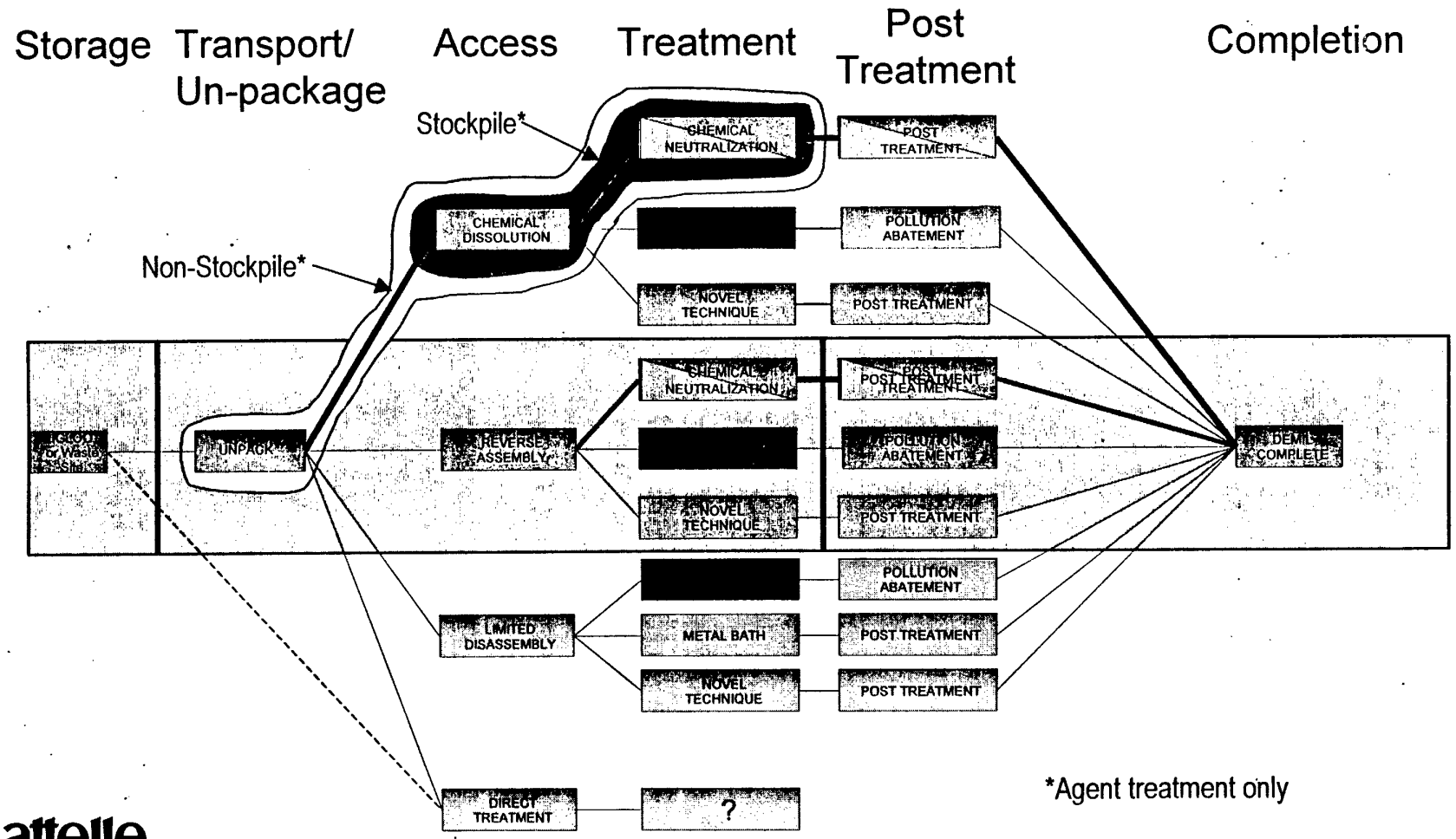
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Applications

- Stockpile
- Non-Stockpile
- Conventional

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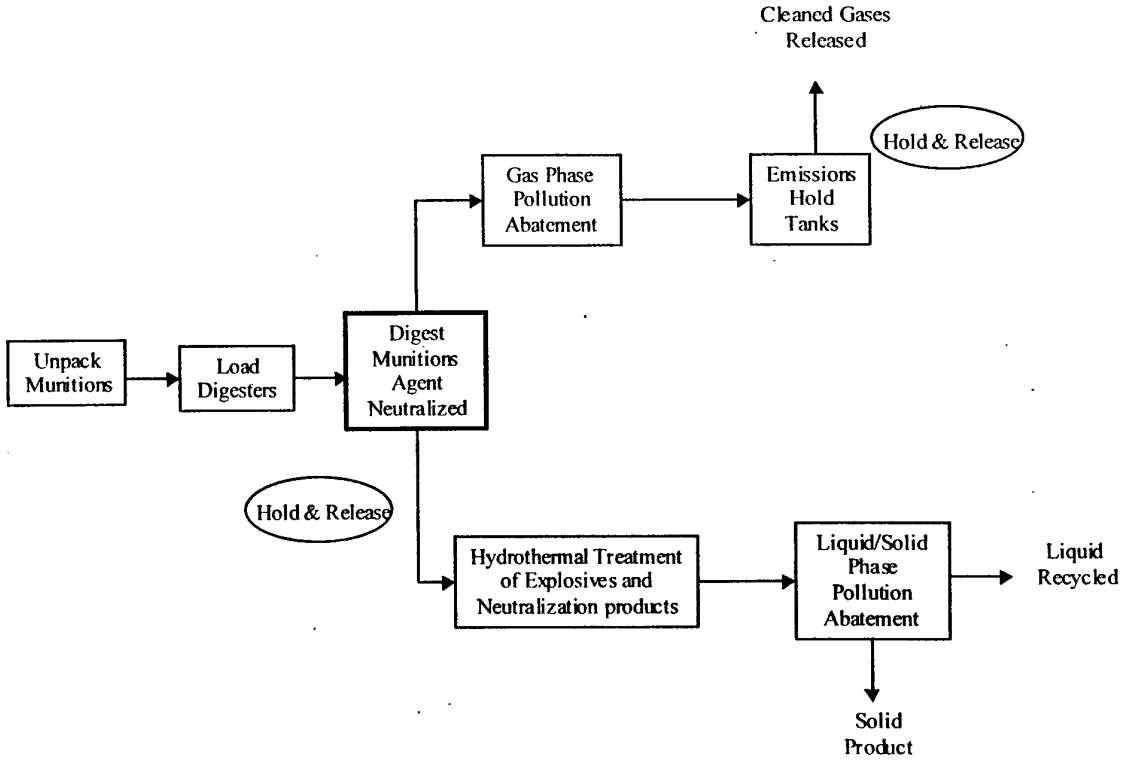
Chemical Demilitarization Systems Analysis



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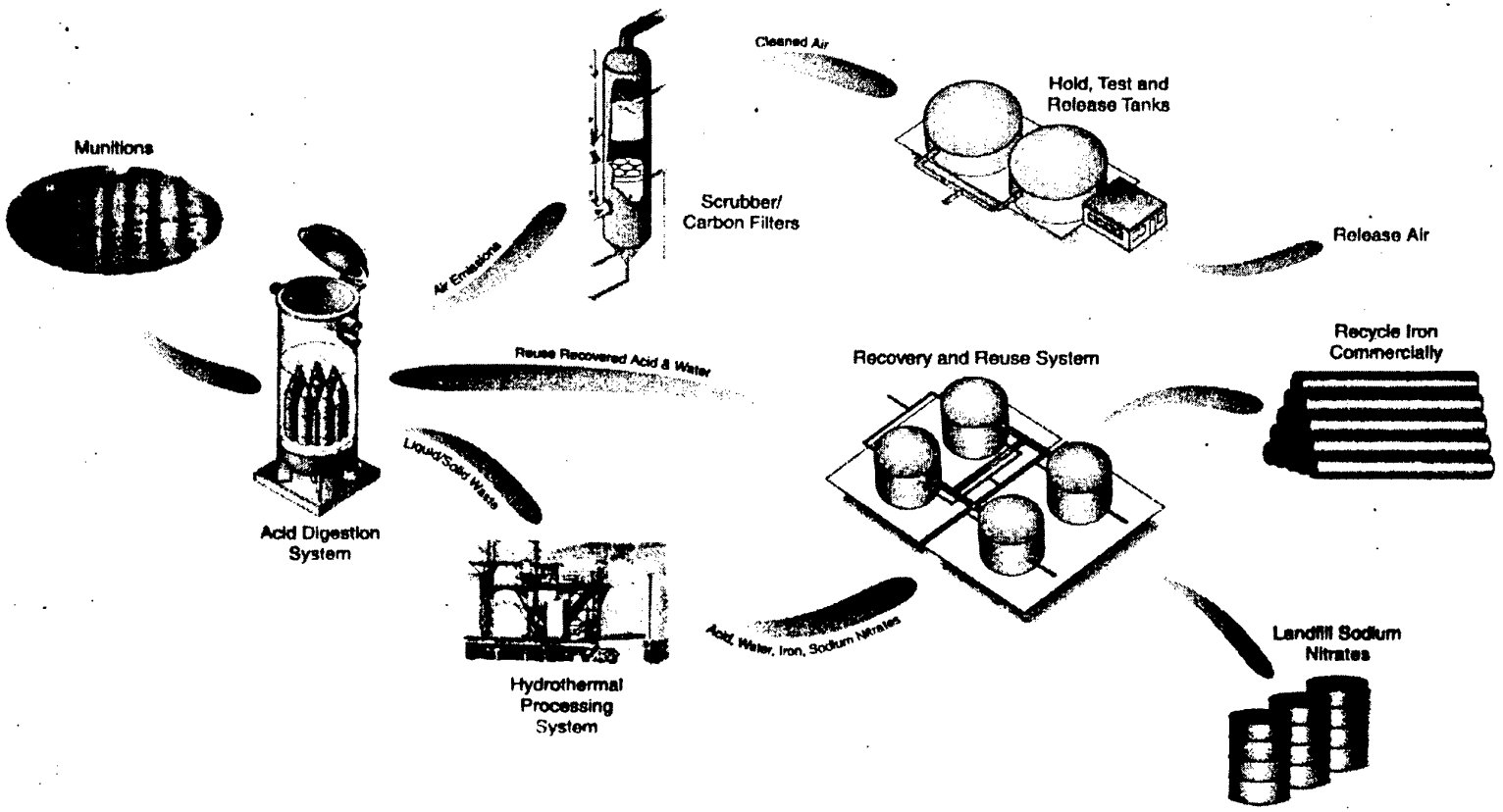
Stockpile Flow Diagram



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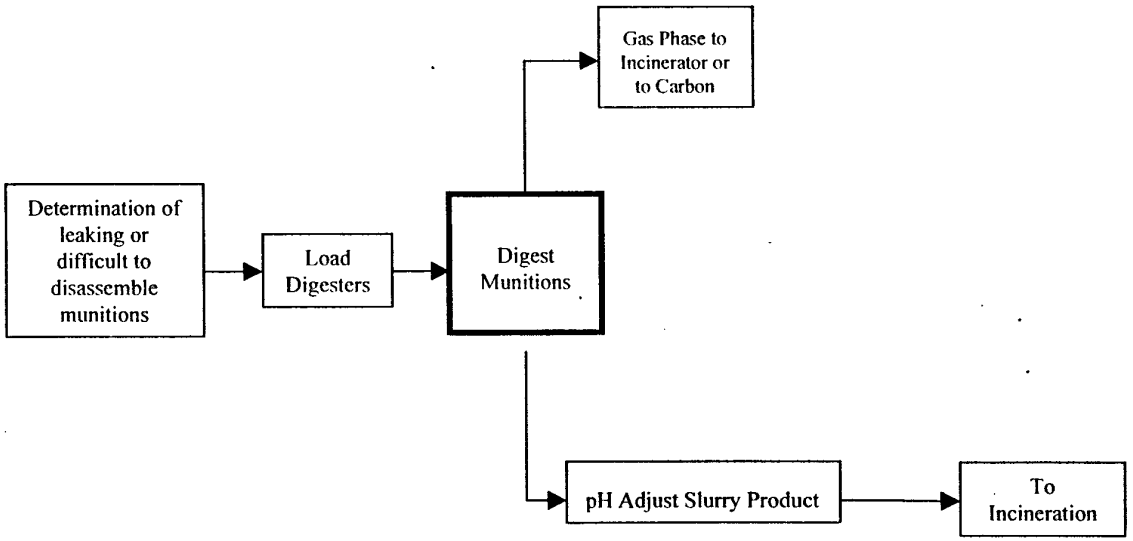
Approach to Stockpile Munitions Total System



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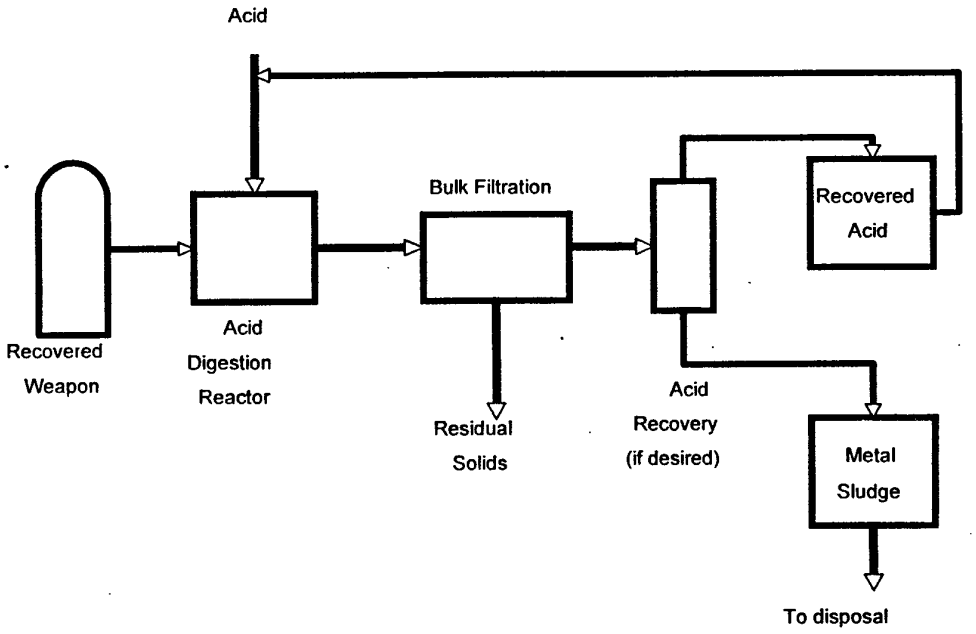
Leakers Flow Diagram



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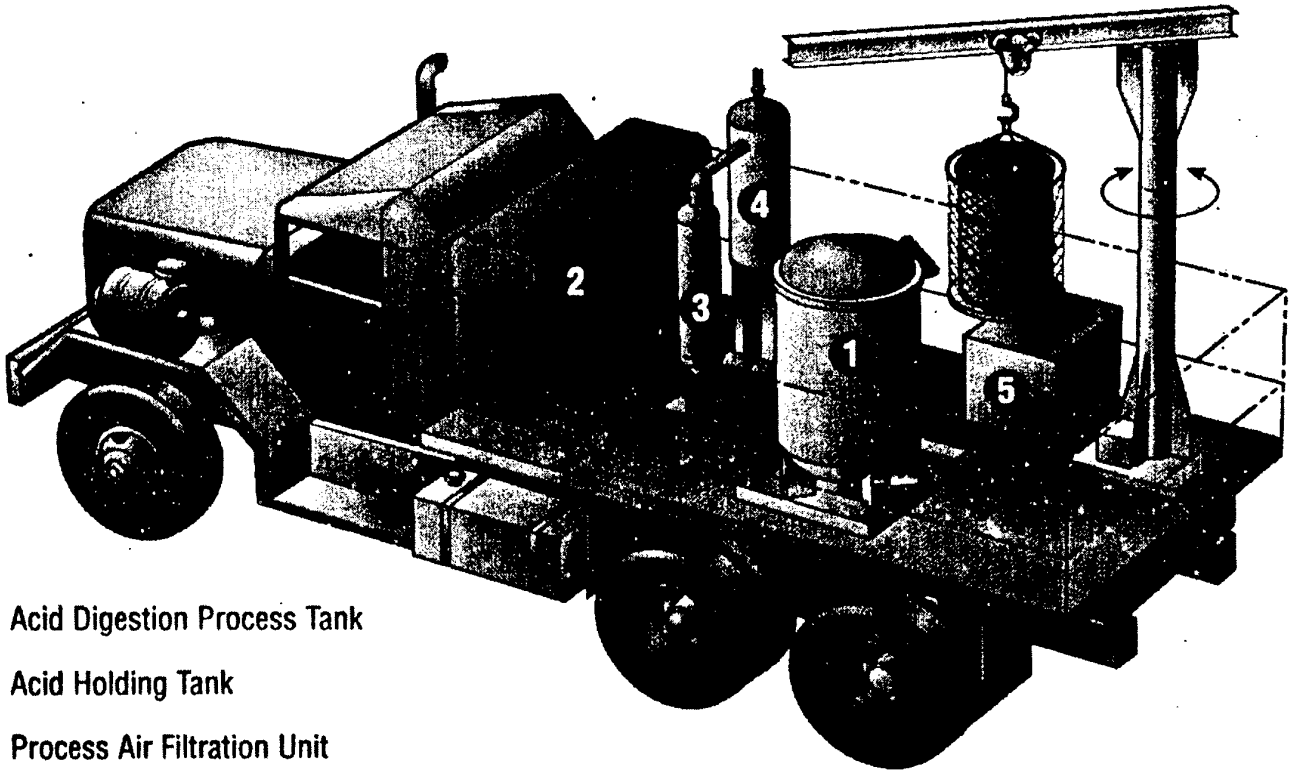
Non-Stockpile Flow Diagram



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Mobile Acid Digestion Concept



1. Acid Digestion Process Tank
2. Acid Holding Tank
3. Process Air Filtration Unit
4. Process Air Holding Tank
5. Power Supply

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Additional Applications

- Domestic Preparedness
- Biological Weapons
- Mixed Waste

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Closing

- Battelle has developed a process for dissolving chemical munitions which eliminates disassembly, chemically neutralizes contained chemical warfare agents, and provides a suitable process stream for energetics treatment

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Battelle

PYROKAT - TECHNOLOGY FOR THE DESTRUCTION OF ARSENIC AND SULFUR CONTAINING CHEMICAL WARFARE AGENTS

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Our company is engaged in both fields of destruction of chemical weapons:

(1) World War I + II agents mostly buried in the ground (non stock pile), and
(2) in the destruction of modern phosphororganic agents being produced after World War II (stock pile).

Under the first headline we have been cleaning the Munster exercise field from chemical ammunition in the years 1990 up to 1996 and actually we started for one year the cleaning of this famous place. Also we have accompanied British Telecom in 1993 through the dumping areas of the Baltic sea. British Telecom placed the telefoncable through the maindumps. Also we cleaned a smaller ground near Magdeburg from chemical weapons.

Unter the first and second we are engaged since 4 years in Russia by Contract of Foreign Office in Bonn and technical advise by the Ministry of Defence in Moscow. For both purpusoses, the destruction of old and new chemical agents, we developed a mobile and self supporting process, meeting all environmental requests. That means our process is able to destroy high toxic organic pollutants. The system is called PYROCAT and has already been used in Germany by order of the German Ministry of Defence state by NATO. In July and August last year we destroyed 16.808 Adamsite grenades formally produced in United States, used by the British Army and buried by the British Army in Wulfen. That's a place close to Münster, Nordrhein-Westfalien.

Our Subject means the introduction of PYROCAT, a mobile and self-supporting process which meets all environmental requests especially the decomposition of high toxic organic pollutants.

The processing is an technical reaction coupling the thermo-pyrolysis under inert gas atmosphere and Thermo-Catalytic-Separation in the gas phase with the gas cleaning following. The application of the PYROCAT-processing has been extended during the tests and approval period for problematic organic waste and high toxic organic pollutants as well as the mixtures of those. This means especially metallorganic compounds, organic halogen, sulfur and nitro compounds for example pesticides, herbicides, polyciklic aromates as well as waste of the carbon and oil processing.

What you should keep in your mind is that PYROCAT is able to treat solid and liquid chemical agents.

The basic of this introduced technology are two processes which according to the problems can be used together or independent

- as a pyrolysis in the stream of inert gas under exclusion of oxygen
- and as a catalytic separation and hydrogenating.

Both processes have been combined for the destruction of chemical agents and other high toxic materials to adapt the process to the problems of the day.

The possibilities for diversification of the design of the decomposition plant are itemised in the following 5 points:

1. pre-treatment for processing of contaminated materials
   - universal shredder cooled down to -170 °C
   - exhaustor for pollutants in air together with dust and carbon-filter

2. thermal-treatment for example severing of pollutants from the matrix and destroying of pollutants
   - hermetic rotary kiln with inert gas and working temperature between 200 - 1.000 °C within three zones for the decomposition of pollutants
   - cooling of gas and linked up gas-washer with proper solvents

3. catalytic decomposition of pollutants
   - thermal-catalytic-separation of condensate and pollutants insolvent processed to hydrogen and carbonoxide

4. off-gas treatment and exploitation
   - afterburning of off-gases ex rotary kiln in a furnace with backfire and hot gas catalytic, gasquenche and zyclon and absorber in off-gas stream

5. Insert of carbon-filter
   - we used carbon-filter for the absorption of the pollutants to be free of residues like off-gases, water, washing-water and off-air, the loaded active carbon is self regenerating five times during the process. No transportation and no expensive regeneration outside the plant is necessary.

There are some more internal circulation's which take care that no pollutants leave the process. Reasons for this chosen system are:

- the combustion of the pollutants themselves should be avoided, to avoid especially the handling and cleaning of the huge quantity of gases. Another advantage is that if arsenic material is inserted we obtain an treatable arsenic or material without any toxic oxides.
- a plant following this principle can be designed as well as mobile plant or stationary plant. We need 5 20'ft container to move and operate at any place in the world.
- mostly the chemical agents are not found in a pure clean condition but they are mixed with soil, metal pieces of ammunition or former containers, decomposition products or polymerisation's products. Even a mixture of explosives and chemical agents can be treated.
- The costly hydrogenating stage is minimised by scales and is only feeded with suitable material.

Basics

Pyrolysis

The base for both important steps of processing may be seen in the pyrolysis and the thermal-catalytic-separation. Take the example of the thermal-decomposition of Adamsite.

You understand which proceedings lead to the destruction of chemical agents. The pyrolysis of Adamsite happens with all probability first at the arsenic carbon compounds. It comes to existencs, beside the metallic arsenic and chlorohydrogen, a radical fragment which changes during the process together with hydrogen radicals first to diphenylamine, carbazol and benzol.

You will find diphenylamine, carbazol and benzol in vestiges in the off-gas.

Polymerisation- and crackprocesses are going on and, as usual in Pyrolysis, lower molecule gases, tar and carbon arise. Air and contamination's will be brought in and this oxygen appears as carbonmono- and carbondioxid as well as water. The better this will be kept out of the process, the lesser appears in the pyrolysis gas. According to our experiences there is no tendency to oxidation of arsenic. If the air input is kept around 1 - 2 %, inserted water is not able to result in oxidising metallic arsenic under hydrogen generation. Resulting chlorhydrogene will be bound by the moderator as solid chloride.

The utmost complete conversion of carbon-compounds in the gas and coke and carbon will be destined by the factors of temperature and dwell-time as well as partial pressure of components. The increasing of temperature is limited because the resulting arsenic is vaporising from 700 °C out of the mixture of reactions and is gone with the gas. The time of standing will be substantial parameter to guaranty the complete treatment of the carbon-compounds out of Adamsite. We have to be aware that the inert gas stream will be low and that the input of water will be low as well as the current velocity. The relation of throughput and volume must be optimised.

Thermo-Catalytic-Decomposition

The Thermo-Catalytic-Decomposition of pollutants in mixture with carbon-hydrogene and watersteam in presence of a basic catalytic works at temperatures between 600 - 800 °C and results in hydrogen, methane, carbonmonoxide and carbondioxide.

Final stabil products result and without any further transposition they can used as burning gas.

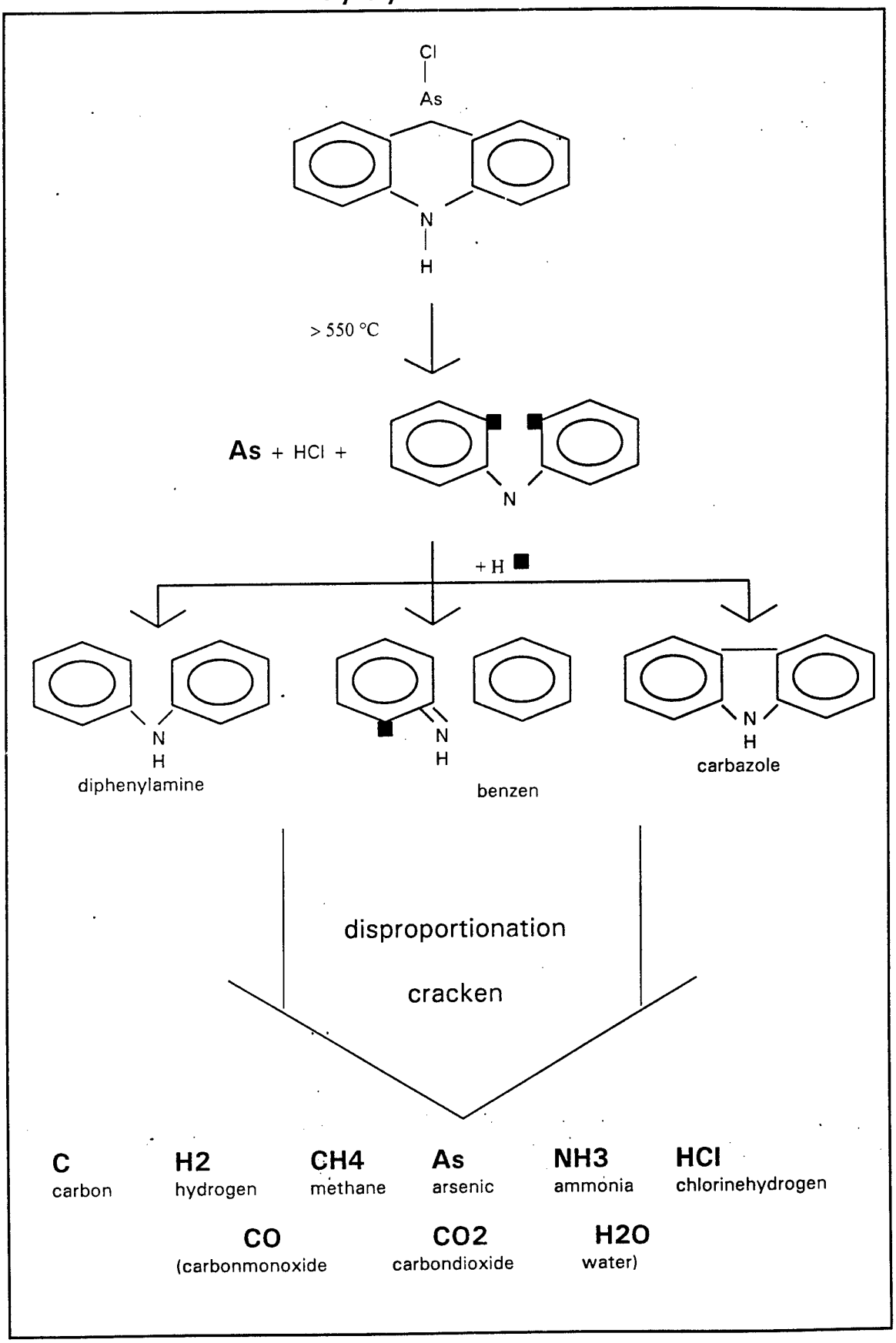
A self developed catalytic helps to use the water-gas-reaction and water-shift-reaction. We get hydrogen and hydrogen-radicals, which hydrogenates the structure of pollutants.

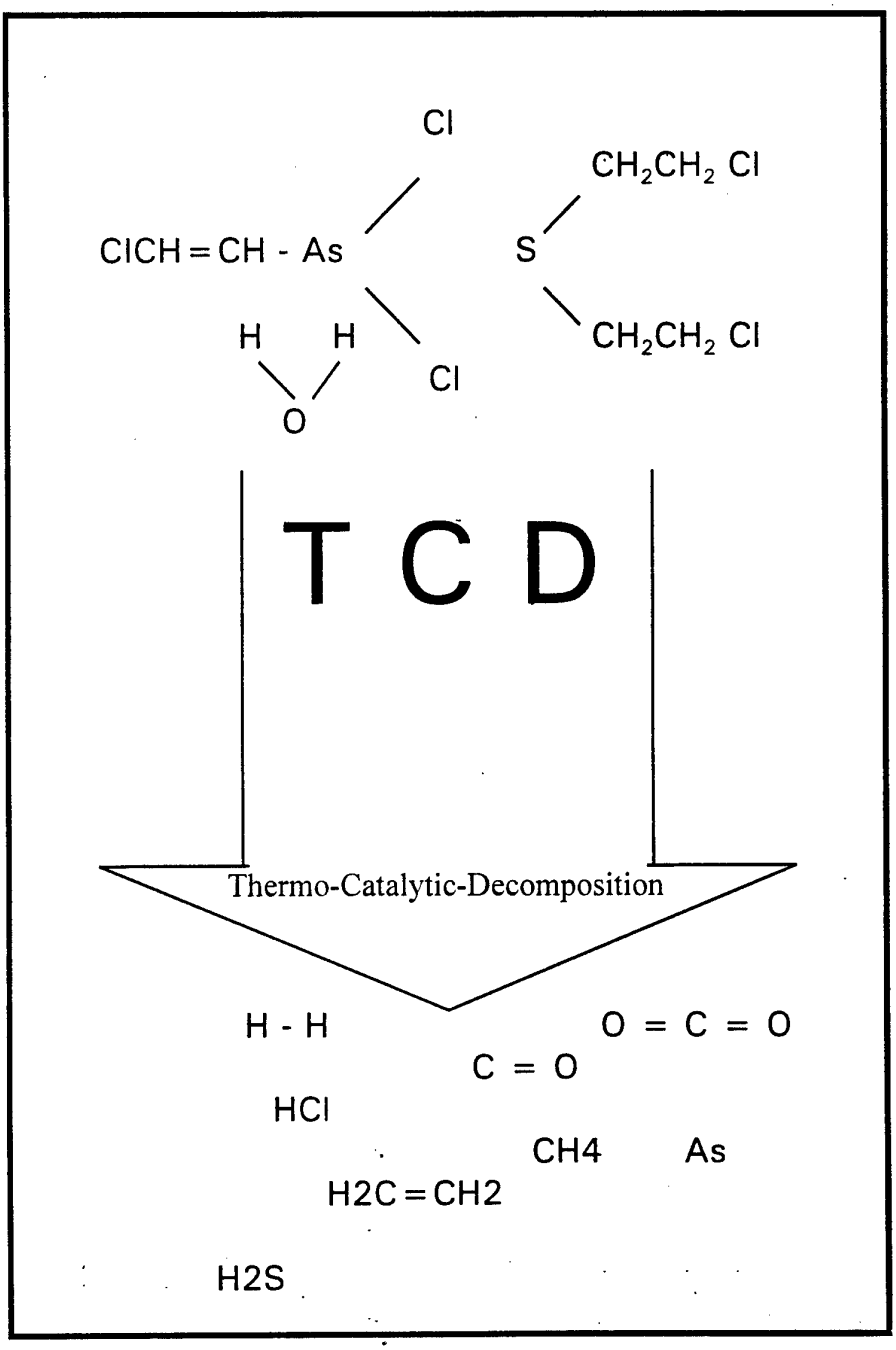
Following this reaction the C-C-, C-H-, C-O-, C-N-, C-S and C-Hal-compounds are splitted to carbonhydrogene by hydrogen and the conversion into hydrogen-products like H_2S, NH_3, H_2O, H-Hal.

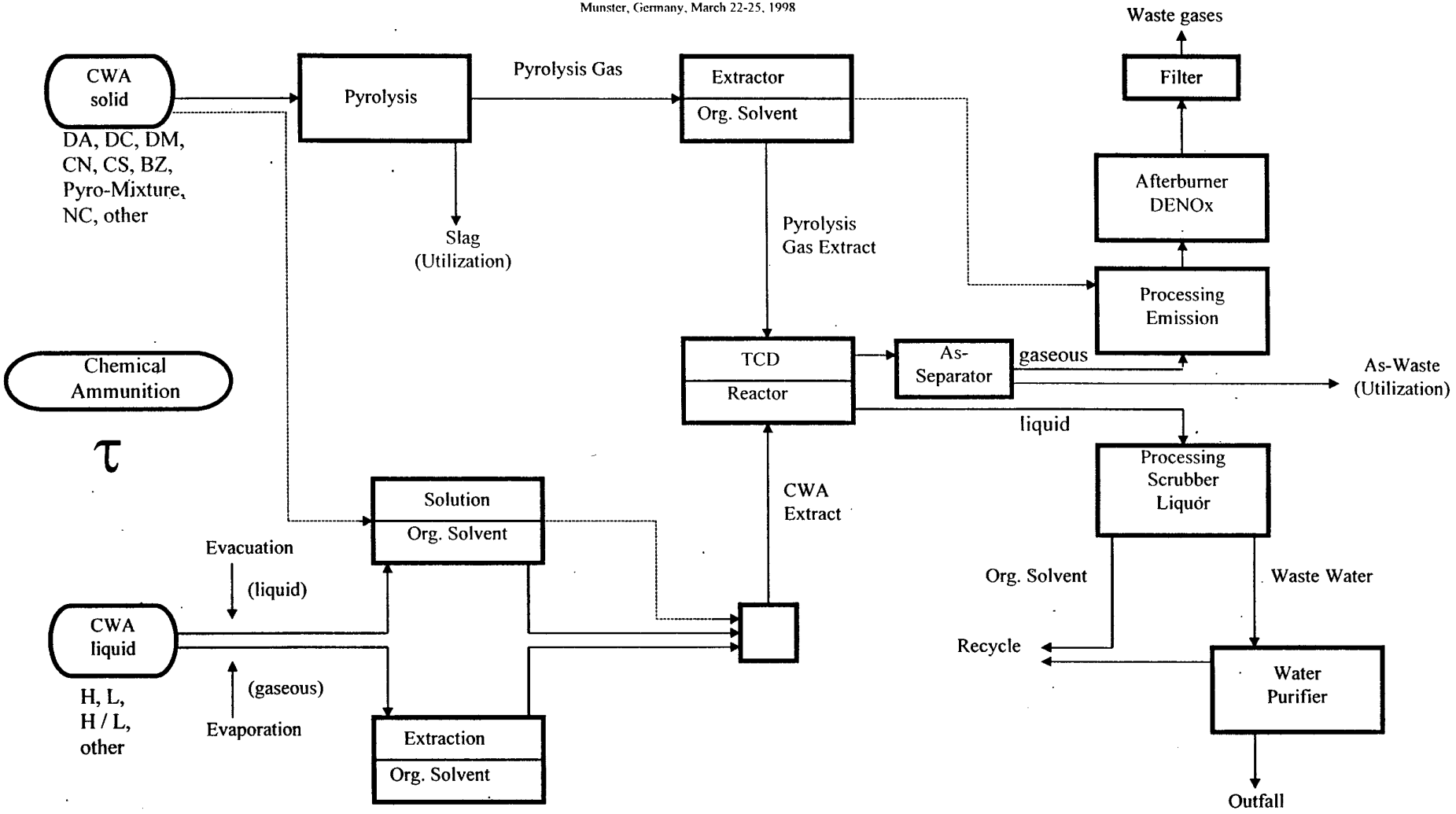
In this stage the extract of the gas washing is inserted. As extractionsmedium will normally be used alcohol's, they produce hydrogene according to the reactions of water-gas and this hydrogene will be able to hydrogenate chemical agents and other dangerous toxic compounds.

Arsenic will be refined. A hydrogenating degradation becomes possible without supply of hydrogen-gas and without highering pressure to force the hydrogenating process.

Pyrolysis of adamsite

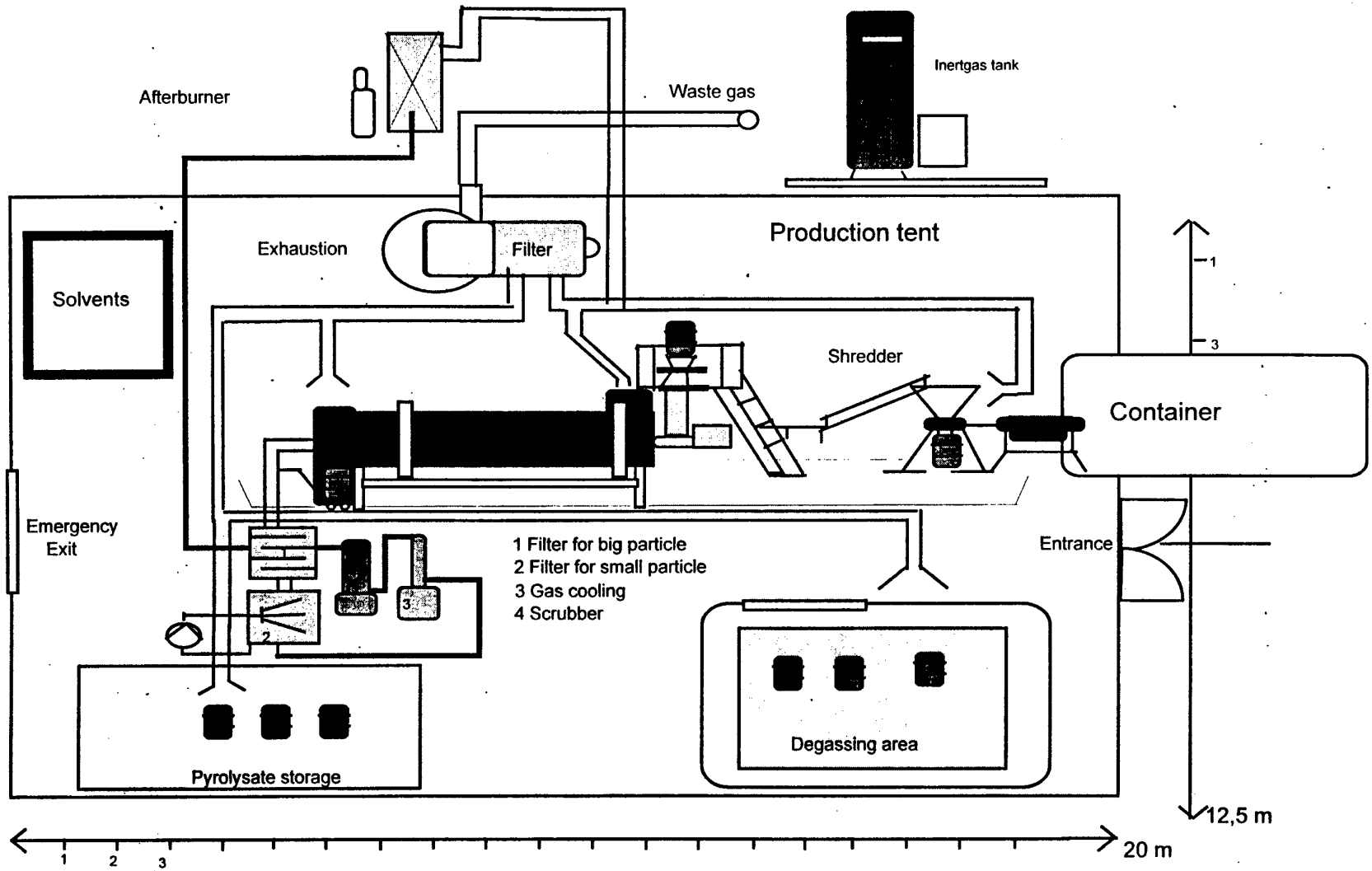



Cl
CH2CH2 Cl
ClCH = CH - As
S
H H
Cl
CH2CH2 Cl
O
T C D
Thermo-Catalytic-Decomposition
H - H
O = C = O
C = O
HCl
CH4
As
H2C = CH2
H2S



PYROCAT

Thermo-Catalytic Decomposition (TCD)
Principal Procedure for solid / liquid CW Agents



DR. KOEHLER GMBH M6 test site at Ammunition Depot Wulfen

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CHEMICAL DETOXIFICATION OF CHEMICAL WEAPONS APPLYING THE SODIUM-TECHNOLOGY

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Abstract

The Sodium-Technology is a chemical method to completely destroy chemical warefare agents in a smooth reaction at moderate temperatures with very high efficiency and safety. The same technology can be applied to a large series of ozone relevant gases deriving from different industry sectors (i.e. aluminium production, semiconductor etching processes).

1. Introduction

It is my personal wish to thank the Honorary Chairman, the Federal Ministry of Defence, Germany and the Symposium Chairman Dr. Bernd Staginnus for the kind invitation to give a short presentation of a modern approach to alternative technologies for the chemical destruction of chemical weapons. Owing to a financial support by the NATO scientific programme, we were able to verify the feasability of the Sodium-Technology in the specialized laboratories of the Polish army in Warsaw.

Since the technology we developed within the last four years is applicable to solids, liquids and gases it can be used for the solution of many environmental problems in soil, liquids and gases. For this reason I am sure that not only those participants enganged in the destruction of chemical weapons but also collegues dealing with ozone related subjects will find valuable points for the discussions within the next days.

Dr. Bilger Umweltconsulting GmbH is a privately owned company located at Freigericht, approximately 50 km east of Frankfurt/Main. The company consists of a team of chemists, chemical engineers, microbiologists and lab staff working in close cooperation with engineering specialists and constructing companies to develop and make available modern complete solutions for environmental problems up to turnkey mobile and stationary units.
Major targets are the product development on base of alkalimetals such as sodium and potassium, as well as the consulting of small and medium size companies in the safe handling and application of sodium and other chemicals in general. This covers the full range from the purchase of chemicals to optimized handling of residual amounts of chemicals.

The main objectives in the last four years have been the detoxification of contaminated mineral oil such as transformer oil (destruction of PCB`s and chlorinated dioxins in oil with sodium), the degradation of organic pollutants such as COD and AOX in water using the UV-oxidation technology and the deodorization of gases applying hydrogen peroxide.

2. Experience in the Handling of Sodium in Chemical Industry

We have gained broad experience in the application of sodium, its derivatives and alloys in 1) the chemical industry: synthesis of vitamin A; synthesis of indigo; 2) metallurgy: dearsenation of zinc; lead raffination; 3) environmental technique: destruction of PCB`s; desulfurization of naphtha; dehalogenation of process oil.

Since we have substantial knowledge and experience in the handling and application of sodium it was a most important and attractive target to develop a cost-efficient chemical method for destruction of chemical weapons in a mobile unit.

Our resons for the choice of sodium may be summarized as follows :

- Sodium is one of the most electronegative elements.
- Sodium is very reactive towards oxygen and halogen in inorganic and organic compounds.
- Sodium is a well-known and described technical chemical.
- Sodium can be applied as fine particles in oil, or as a thin layer on inorganic supporter.

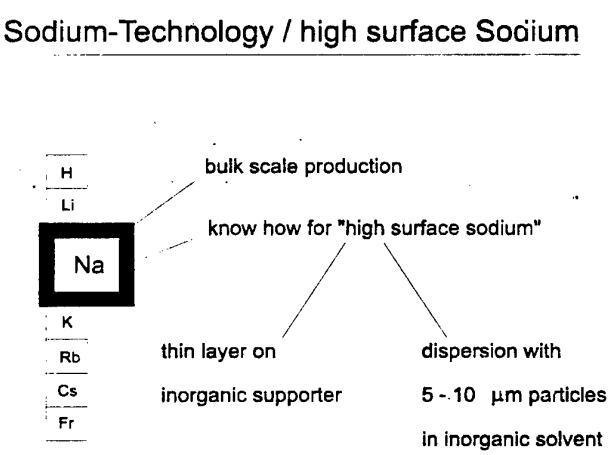


Figure 1

Sodium is a commercially available bulk chemical at a rather low price. The handling of sodium is subject to proper training of personal staff but basically not complicated. Our technical service offers application assistance to all customers.
Sodium can be applied in form of molten metal, solution in ammonia, on inorganic supporter or as a dispersion in an inert organic liquid such as paraffinic mineral oil. For several reasons, including economic and legislative, we prefer the dispersion in oil since this form is technically accessible with low investment costs and fewer administrative problems as compared with toxic ammonia.

3. Construction of Static and Mobile Detoxification Plants

With our technical and scientific staff we are able to construct and operate mobile and stationary units from lab scale up to stationary units with a capacity of 50,000 tons of oil.
With respect to chemical weapons being existent in different compartments such as small cylinders or large drums the mobile units must be flexible enough to treat filled drums without and refilling procedure prior to detoxification. A model for an online treatment unit is depicted below.

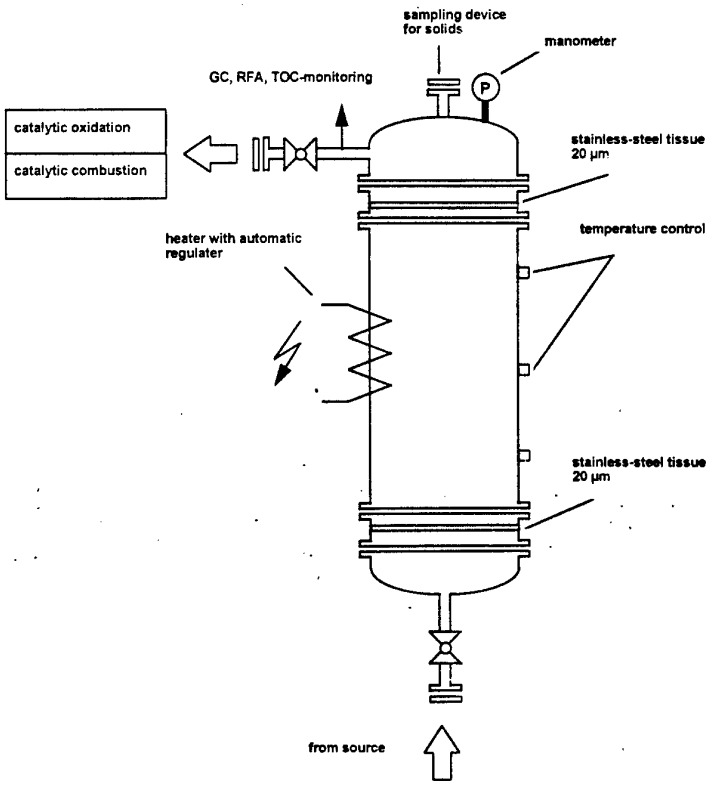


Figure 2

4. Sodium in the environmental field

It has been found that the chemistry of extremely fine sodium particles differs remarkably from that of molten fused sodium. Many reactions take place under very mild conditions if the sodium has a very large surface, as achieved in a dispersion in oil with particles of 1-10 µm or as a thin layer on an inert inorganic supporter.

The aim of the Sodium-Technology is to react chemicals in liquid, dissolved or gaseous stat to yield inorganic salts of sodium and small organic molecules of low or no toxicity.

The idea for the chemical destruction of chemical weapons has been derived from the application of the Sodium-Technology for the destruction of toxic chemicals such as chlorinated dioxins, furans, chlorinated phenols and benzenes in oil and effluents from local landfills.

The activity of sodium is very high due to ist extreme surface.

1 kg of sodium as a cube of 10 x 10 x 10 cm has a surface area of only 0.06 m².
In the form of very fine particles of 5 µm in diameter the same quantity of sodium offers an active surface of 1200 m².
This is a factor of 20,000 responsible for complete reactions under mild conditions.

4.1 Hamburg-Georgswerder

As an example for the application of a mobile plant we would like to make some remarks on the detoxification of effluent from a local landfill in Hamburg-Georgswerder.

In a commercial project our goal was to detoxify effluent from the well-known local landfill Hamburg-Georgswerder. In an area of 440,000 m² , chemical waste was dumped together with conventional waste resulting in a „mountain" 40 m high. Due to hydrostatic pressure, large amounts of effluent have been released. Within the organic part of the effluent the full range of toxic chlorinated organic compounds including benzenes, phenols, cyclohexanes, furanes, dioxins and others have been found. The total chlorine content was between 2 and 4 %.

The contract conditions have been simple and clear. The objective was the chemical destruction of all chloro-compounds in the organic effluent down to below detection limit.
The KEY contract item was the condition that if destruction is positive, City of Hamburg pays for all analytical work (DM 100.000,--), if destruction is negative, we pay. The results: no detectable trace of halogenated compound has been found after treatment with sodium dispersion.
As an example, the analytical data for the dioxin series is given below.

TABLE 1

toxic compound	concentration in ppb	tox equivalent factor acc. to NATO/CCMS	TE	final concentration in ppb
2,3,7,8-Tetra CDD	42	1.0	46	n. d.
other Tetra CDD	46	(0.01)	0.46	n. d.
1,2,3,7,8-Penta CDD	13	0.05	6.5	n. d.
other Penta CDD	243	(0.01)	2.43	n. d.
1,2,3,4,7,8-Hexa CDD	82	0.1	8.2	n. d.
1,2,3,6,7,8-Hexa CDD	375	0.1	37.5	n. d.
1,2,3,7,8,9-Hexa CDD	154	0.1	15.4	n. d.
other Hexa CDD	1397	(0.01)	13.97	n. d.
1,2,3,4,6,7,8-Hepta CDD	1890	(0.001)	1.89	n. d.
1,2,3,4,6,7,8-Hepta CDD	3765	0.01	37.65	n. d.
Octa-CDD	11139	0.001	11.14	n. d.
sum			177.14	n. d.

n.d. : not detectable
TE : toxicity equivalent

Treatment of Solids

For quantitative reaction with sodium, solids must be transferred into fine particles. This can be achieved either after homogenization and grinding followed by dissolution in an organic solvent or very economically using liquid nitrogen and a specialized shredder system.

As an example for the treatment of solids we constructed the following project : A thick rubber lining from a steel pipe of a chlorine line with an age of 3 years was removed out as a mixture of very soft and medium hard particles with about 1 to 5 cm diameter.

The total organic chlorine content was found to be 52.4 %! Large amounts of hexachlorobenzene and chlorinated dioxines were found in the ppm – region.
This material was brought into liquid nitrogen and then ground up.

The finely powdered material was picked up in paraffinic oil and treated with a freshly prepared sodium dispersion. It was possible to reduce the total chlorine from 524.000 ppm in one step to below 5 ppm.

Treatment of Gases

From reactions of CFC's such as CCl_2F_2 (Frigen R12) with sodium on inorganic supporter we know that these molecules are completey destroyed when reacting them with sodium. For example CCl_2F_2 reacts with sodium starting at room temperature again. Only solid products have been observed (control: halogens > 99 % of weight was found on the column).

example for effectivness of sodium : CF_2Cl_2 (Freon, R12)

reaction starts at room temperature

$$CF_2Cl_2 + 4\,Na \longrightarrow 2\,NaF + 2\,NaCl + C$$

ΔH_F 1538 822 kJ/mol + 1148 kJ/mol $\Delta\Delta H_F$ = - 432 kJ/mol

reaches 1000 °C within short time (3 Min.)

➡ **F and Cl found quantitative as NaF/NaCl on the column**

Figure 3

In the semiconductor industry processes for etching the silicon wafers are used involving tetrafluorocarbon (CF_4) or BCl_3 or other „difficult" gases are used.
In the aluminium industry we have to think about fluorinated gases released into the atmosphere.

All these gases can be chemically converted to inorganic sodium fluoride with sodium.
A different example for the effectiveness of sodium is the unexpected reaction with SF_6. This molecule is of very high importance when we regard the potential for the global worming effect. SF_6 is the „most effective chemical" regarding the greenhouse effect. Therefore we must be very carefull to avoid any release of SF_6 and other halogenated ozone attacking gases into atmosphere.

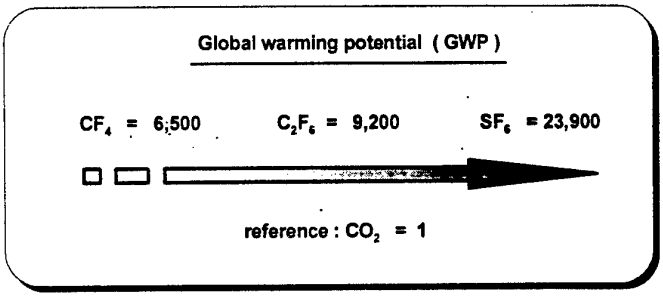


Figure 4

Sulfur hexafluoride, SF_6, is said to be a chemically unreactive material. It is published that SF_6 does not react appreciably with fused sodium metal at temperatures of 250 - 300 °C . On the other hand it is well known that sodium will melt in SF_6 - atmosphere without any reaction taking place. In our internal study in contrast we found that SF_6 can be decomposed very rapidly by direct contact with sodium starting at room temperature.

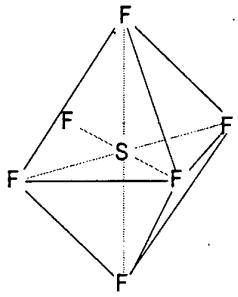


Figure 5

A good technology for destruction of chemical warefare agents and other toxic compounds must be able to treat all chemical weapons independent of their chemical nature, being an organic or inorganic compound in solid or pasteous or liquid state. Gases must be accessible likewise.

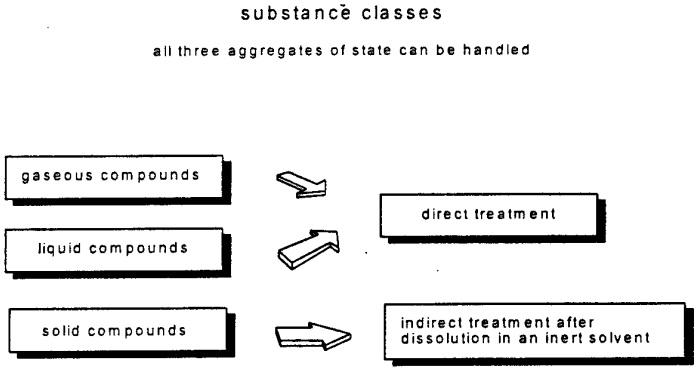


Figure 6

Most of the chemical weapons have chemical structures basically accessible for cleavage of the bonding systems by sodium. Knowing that C-Br, C-Cl, C-F and S-Cl and
As-Cl-bonds are attacked by sodium, we can deduce that analogue structures found in chemical weapons will also be attacked by sodium. The driving force is the formation of solid ionic inorganic compounds with high bonding enthalpies. As is well known from literature the resulting organosodium compounds are exceedingly reactive, sensitive to air and easily hydrolysed by water.

For the destruction of chemical weapons in general it is essential to find a chemical method applicable not only for one chemical weapon but for at least several chemical weapons.
As an example arsenicals and mustards in general require only ONE method for the chemical destruction.

Mustard, $Cl - CH_2 - CH_2 - S - CH_2 - CH_2 - Cl$

can easily be destroyed by sodium at the marked positions to form NaCl, $Na_2C_2H_4$ and Na_2S as intermediate products and a saturated hydrocarbon as further final product.

Sarin, (methylfluorophosphonic acid isopropylether), a liquid and almost odourless water soluble compound has a bonding system accessible for sodium attack. The P-F as well as the P-O-bond will be broken followed by cleavage of the P=O-bond.

Other chemical weapons such as Tabun, Agent White, Clark I and Clark II react likewise.

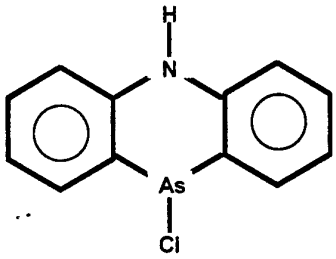
For pollution control it is envisaged to use gas chromatographic verification of complete destruction of the specific compounds.

For the process control as well as environmental protection we prefer to install GC-MS analytical systems due to their high selectivity and low detection limits. The high speed of these analysis techniques make them most useful for direct process control.
The on-line analysis offers complete results within 5 minutes.
Good results have been obtained by a research team of the University of Hamburg using a MS-devive MM 1 (Brucker Franzen GmbH, Bremen).

5. Experience in destruction of Adamsite

According to extended experience with the Sodium-Technology for destruction of highly toxic chlorinated dioxins and furanes, the existing chemical weapons such as arsenicals (example: Adamsite) or mustards can be treated in mobile detoxification units on site as well as in large scale plants depending on the actual quantity to be treated.

In the experimental part carried out as main subject with financial support of the NATO scientific programme (DISRM.EV 951479) at the Military Institute of Chemistry and Radiometry in Warsaw under the leadership of Col. Dr. Dominas and Dr. Sokolowski it was possible to demonstrate and confirm that the chemical weapon *Adamsite* can be destroyed completely by the reaction with sodium at low and even ambient temperatures with short reaction time. As a second target we could confirm that the originally organic bounded arsenic is transferred to inorganic (ionic) arsenic and therefore can be precipitated as insoluble sulfide.



ADAMSITE

Figure 7

Path : ⇒ reaction with Na-dispersion followed by addition of water and precipitation of As as As_2S_3

⇒ application of UV-oxidation for possible water soluble compounds with the target to
- ⇒ oxidize organic compounds completely
- ⇒ oxidize As^{3+} to As^{5+}
- ⇒ precipitate As compounds

results :

1. After keeping the reaction mixture under argon over night no *Adamsite* was detected.
2. In addition, the liquid chromatograph equipped with UV-detector gave no signal for any aromatic structure left.
3. GC/MS analysis gives no hint for volatile organic compound.
4. No organic bounded arsenic could be found in the xylene layer!
5. After precipitation with thioacetic acid As-sulfide was found in large quantities.

6. Mobile unit for chemical detoxification of chemical weapons

A full scale mobile plant for destruction of Adamsite in Poland is readily designed and could be set up within 3 - 4 month after agreement on political and financial questions.

mobile unit for Adamsite destruction

- readily prospected -

not yet realized due to lack of money

request for only 2 - 3 Mio DM (1 Mio US$)

possible result after commissioning of said mobile plant:

➡ **all Adamsite in Polish stock destroyed**

within one year operation

general layout for a mobile unit :

Adamsite detoxification plant applying Sodium-Technology and Sodium-Dispersion

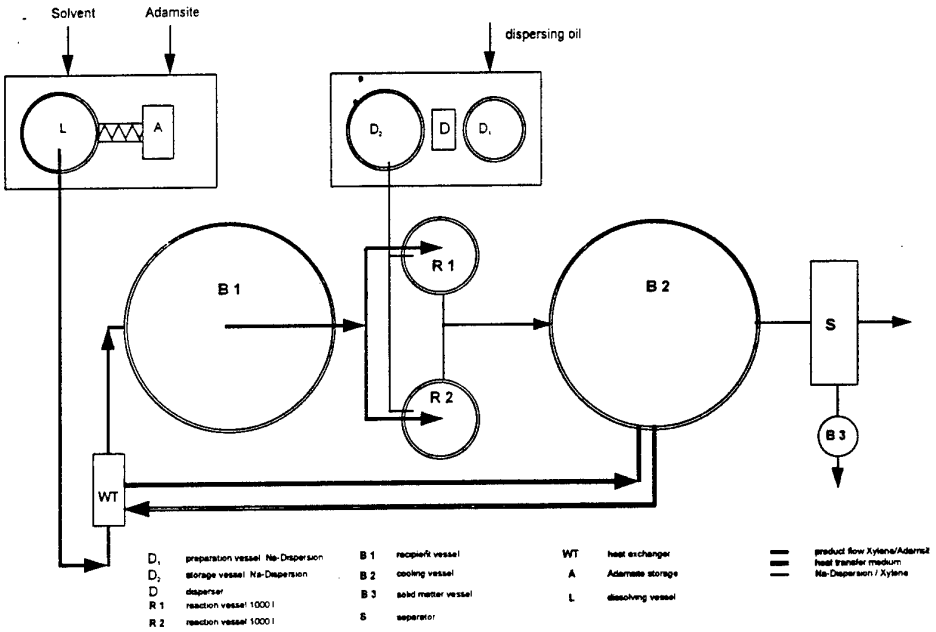


Figure 8

Major advantages of the Sodium Technology

- low temperature technology
- mineralization of chemical weapon
- low investment costs
- inexpensive agents
- no fear of formation of dioxins and furanes in contrast to incineration
- production of solid or dissolved reaction products
- no hazardons gaseous products released
- mobile detoxification units possible

7. Conclusions

The Sodium-Technology is an effective chemical path and general method for disarmament of chemical weapons as well as ozone sensitive gases and toxic halogenated liquids.

Currently, chemical weapons can be rendered inoffensive by thermal treatment. The products of combustion have to be treated downstream of the incinerator in an efficient treatment plant.

By application of the Sodium-Technology we can control the chemistry, and analyse at any stage of treatment because we keep all substances in closed compartments until the products are released after thorough final analysis.

This is in contrast to incineration, where there is no chance to correct a possible malfunction. If a toxic compound has passed the chimney, it will become and remain a danger for the environment.

Detoxification units can be set up in non-expensive mobile units as weil as large scale stationary plants.

It should be our common understanding to utilize for example a low amount of the military budget of each country for the cleanup of contaminated sites and environmental problems in general.
It is our task to give back to nature what we have taken from our planet : clean air, clean water and clean soil.
Following generations have the same right as we to live in a world with distinct natural living conditions.

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MOBILE CHEMICAL MUNITIONS TREATMENT TECHNOLOGY

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Abstract

The United States developed a variety of chemical munitions in response to foreign threats dating back to World War I. Production fell dramatically in the late 1960's and was completely banned by the 1970's. With the passing of the cold war, the United States (U.S.) and many other countries have jointly signed treaties to destroy these munitions. The U. S. has organized the implementation of chemical warfare destruction into stockpile and non-stockpile programs. The former effort addresses the large storage inventory of offensive chemical weapons. The latter program, and perhaps the more logistically difficult one, is finding, characterizing, treating, and disposing of chemical warfare materiel at training sites, ranges, and other facilities nationwide that are not covered by the stockpile program.

This paper focuses on the United States Army's development of mobile treatment systems for the non-stockpile program including a mobile system developed to interrogate and treat the contents of chemical agent identification sets previously utilized for military training.

Non-Stockpile Program Overview

Research, development, testing, and production of chemical weapons over a 70-year time span has created a unique recovery and cleanup challenge for the U.S. Army. To manage and direct this effort, the Non-Stockpile Chemical Materiel Program (NSCMP) was created to oversee the disposal of all non-stockpile chemical materiel in a safe, environmentally sound, and cost-effective manner. Five categories of Non-Stockpile Chemical Warfare Materiel (CWM) have been identified by the Army for disposal: recovered CWM, buried CWM, former chemical weapons production facilities, miscellaneous CWM, and binary chemical munitions. This paper only addresses the categories of existing recovered CWM and CWM that may be recovered from burial sites. Under the direction of the Project Manager, Non-Stockpile Chemical Materiel (PMNSCM), the Army is designing, building, and testing mobile treatment systems for these categories with its lead Non-Stockpile Systems contractor Team Teledyne. These mobile systems are primarily designed to treat the family of nerve, blister, and choking agents.

Materiel to be addressed in this program often was buried, and available records indicate that some land burials may still contain chemical agent. While most of the potential burial sites remain on military property, some are known to be located on land that is no longer under Government control. The sites are scattered across 65 locations in 31 states and the U.S. Virgin Islands.

Munitions Assessment System

The Mobile Munitions Assessment System (MMAS) is a transportable system developed by the Army that has the capability to non-intrusively interrogate the contents of munitions. Once recovered, munitions are assessed for condition and content using non-intrusive techniques such as x-ray and Portable Isotopic Neutron Spectroscopy (PINS). The MMAS has two functions, interrogation and communication. It can assess the contents of a recovered munition with minimal movement of the materiel. Munitions are analyzed by a PINS and associated on-board computer. The PINS uses gamma rays to identify spectral elements of the contents that are in turn interpreted by computer finger printing. Also housed in the MMAS is a portable x-ray photography device that can be used to examine the internal explosive qualities of a munition with virtually no disturbance.

Munitions Destruction

A family of mobile Munition Management Devices (MMDs) designed to access and treat non-explosive CWM, explosively configured munitions, and bulk chemical agent containers is in development. The MMD-1, designed for non-explosive items, is now in field test and evaluation. The main processing equipment is housed in two trailers, one for chemical processing and one for systems control. In addition, the process trailer is supported by skid-mounted equipment such as the chiller system, carbon filter system, compressed air system, etc. The MMD-1 is designed to receive, handle, and treat non-explosive munitions - up to 500-lb bombs. To accommodate larger items, such as ton containers and explosive munitions, a second MMD is in design. The

primary difference is the size and orientation of equipment necessary to handle the different sizes of CWM and the need for explosion containment and handling capability.

General Process Train

MMD-1 is the first completed unit and is now undergoing testing in the state of Utah. This unit, as stated earlier, accesses and treats only non-explosively configured CWM. From the point that materiel is brought onto the site, a seven-step process is applied to treat the CWM.

Unpacking. Processing begins by bringing the CWM on a cart from the storage and service magazine racks into the unpacking area. The next step is to manually remove the CWM from the overpack containers and prepare it for processing. Personnel performing this operation are in positive air safety gear. The CWM is then transported via cart to the process trailer where it is placed into the munitions treatment vessel (MTV).

Treatment. Operations in the process trailer are controlled remotely. A rotary position transducer and home switch are used for an accurate indication of the CWM's position in the MTV. The CWM is accessed with tooling mounted in towers in the MTV. After accessing the CWM, a vapor sample is collected and analyzed by a gas chromatograph/mass spectrometer detector (GC/MSD) to verify the contents. This process requires approximately 20 minutes to complete.

Based on the analytical results, appropriate reagents are prepared or drawn from pre-mixed supplies. Reagents used are monoethanolamine (MEA) and water for Mustard, GB (Sarin), and VX. Sodium hydroxide (NaOH) and water are used for Phosgene. Extensive treatability studies have determined that reagent-to-agent ratios of 10:1 volumetric and 3:1 molar ratio are effective. The MTV and a liquid reactor vessel (LRV) are used for the treatment processes. Mixing is accomplished by circulating the reactant mixture through a static mixer element. This circulation process is continued until the fluid meets the treatment goals. Reaction times will vary by the type of agent being treated and its concentration. The system also has the capability for heating or cooling the reactant mixture as required. Treatment goals for the system are 1 mg/l of agent in the waste stream. Reagent mixtures are tested after each run to determine if there is sufficient available reagent remaining for the next run.

Vapor treatment occurs after liquid treatment has been completed. Residual vapors in the reactor vessels are treated with a separate processing system. The vapor treatment goals are as follows; HD, 0.003 mg/m^3; Sarin, 0.0001 mg/m^3; Phosgene, 0.4 mg/m^3; and VX, 0.00001 mg/m^3.

Vapors in the MTV are removed by a gas vacuum pump and flow through the gas reactor cooler, gas reactor knockout drum, and gas reactor, prior to storage in the waste gas knockout drum. If final sampling reveals an unacceptable concentration of agent or industrial chemical, the vapor is recirculated through the MTV, LRV sparger, gas reactor cooler, knockout drum, and gas reactor. Treatment is accomplished by sparging the gas through the reagent mixture in the LRV. Final treatment involves passing the vapor through a waste gas chiller to remove moisture, followed by a heater to reduce relative humidity, and then adsorption on three carbon filters in series. Following successful systems tests of the MMD-1 at Dugway Proving Ground, Utah, Team Teledyne anticipates tasking to conduct field operations.

Future Units. MMD-2 is currently in the final design phase. It will have the capability to assess and treat the contents of explosively configured CWM up to the size of an 8-inch round, and bulk containers up to a ton container. The MMD-2 will use the same chemical treatment technology as is being used for MMD-1.

Treatment of CAIS with the Rapid Response System

Another product of chemical warfare was the development of Chemical Agent Identification Sets (CAIS) used for training personnel in chemical defense measures. Produced from 1928 to 1969, CAIS consist of chemical agents sealed in glass ampoules and bottles, then packed in metal shipping containers or wooden boxes. Most of the CAIS items have been recovered from storage locations or from burial sites.

Some ampoules contain the agents Lewisite, Sulfur Mustard, and Nitrogen Mustard, some of which is in solution with chloroform. Other ampoules contain industrial chemicals such as Phosgene, GA-simulant, Cyanogen Chloride, and Chloropicrin.

A mobile treatment system known as the Rapid Response System (RRS) has been designed and constructed by the U.S. Army to access and treat agents contained in the CAIS. The RRS, with a multi-trailer configuration, is capable of receiving, containing, interrogating, monitoring, and treating CAIS. Chemical agents will be treated on-site; industrial chemicals will be repackaged.

The primary process equipment is housed in two trailers, an operations trailer and a utility trailer. Accessing and treatment of CAIS is accomplished in the process trailer using a glove box operating at a negative pressure. Overpacked CAIS is passed into the airlock station of the glovebox through an access door. The access door is closed and the glovebox negative pressure restored. The overpacked CAIS is then moved into the unpack station where the overpack is opened and the CAIS removed. The CAIS is then interrogated by RAMAN spectroscopy and segregated for treatment by type of item, e.g., agent in chloroform, agent on charcoal, neat agent. Agent is treated in a batch process reactor at the treatment station. The resultant waste is

sampled and analyzed to ensure that treatment goals have been met. The waste and the repackaged industrial chemicals are shipped to an approved waste treatment facility for ultimate disposal.

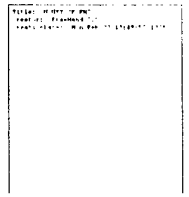
Research conducted by the Army has indicated that the agents, industrial chemicals, and treatment by-products are effectively removed from the air stream by carbon adsorption. Air exiting the glovebox is processed through redundant carbon filters linked in series. The process trailer also incorporates a real-time air monitoring system.

Team Teledyne is conducting RRS final systems tests at Deseret Chemical Depot in Utah. The RRS is C-130 transportable and Team Teledyne anticipates deployment to an initial site in Alaska after Utah testing is finished. State of Alaska approval has been obtained.

Summary

The U. S. Army in association with its Non-Stockpile Systems contractor, Team Teledyne, is developing a family of mobile systems for accessing and treating the contents of chemical warfare materiel. Following testing in Utah, the MMD-1 and RRS will be ready for field deployment. The MMD-2 system is in design and is anticipated to go to the field after fabrication and testing. By utilizing traditional, well established chemical treatment methods on these projects, the Army and Team Teledyne are pursuing a path to minimize development times, decrease risks, and reduce costs.

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U.S. Army Non-Stockpile Systems Program

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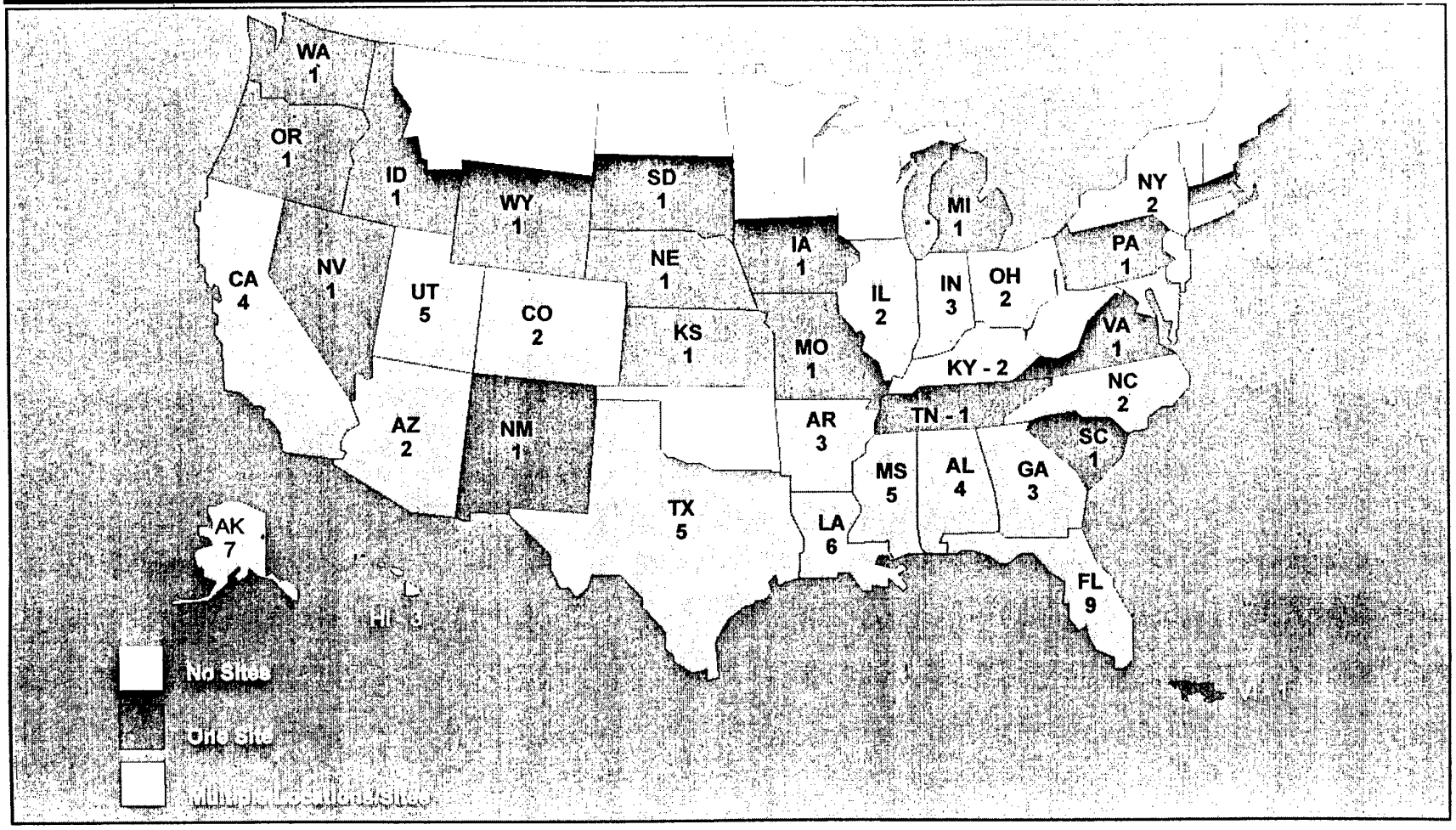
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POTENTIAL CHEMICAL WARFARE MATERIEL BURIAL LOCATIONS

U.S. Army Non-Stockpile Systems Program

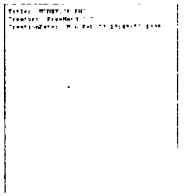


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THE FIVE CATEGORIES OF NON-STOCKPILE CHEMICAL WARFARE MATERIEL (CWM)



U.S. Army Non-Stockpile Systems Program

- Recovered chemical weapons
  - Previously recovered from ranges or burial sites
  - Stored at chemical installations
- Buried chemical warfare materiel
  - 63+ locations
- Binary chemical weapons
- Former production facilities
- Miscellaneous chemical warfare materiel

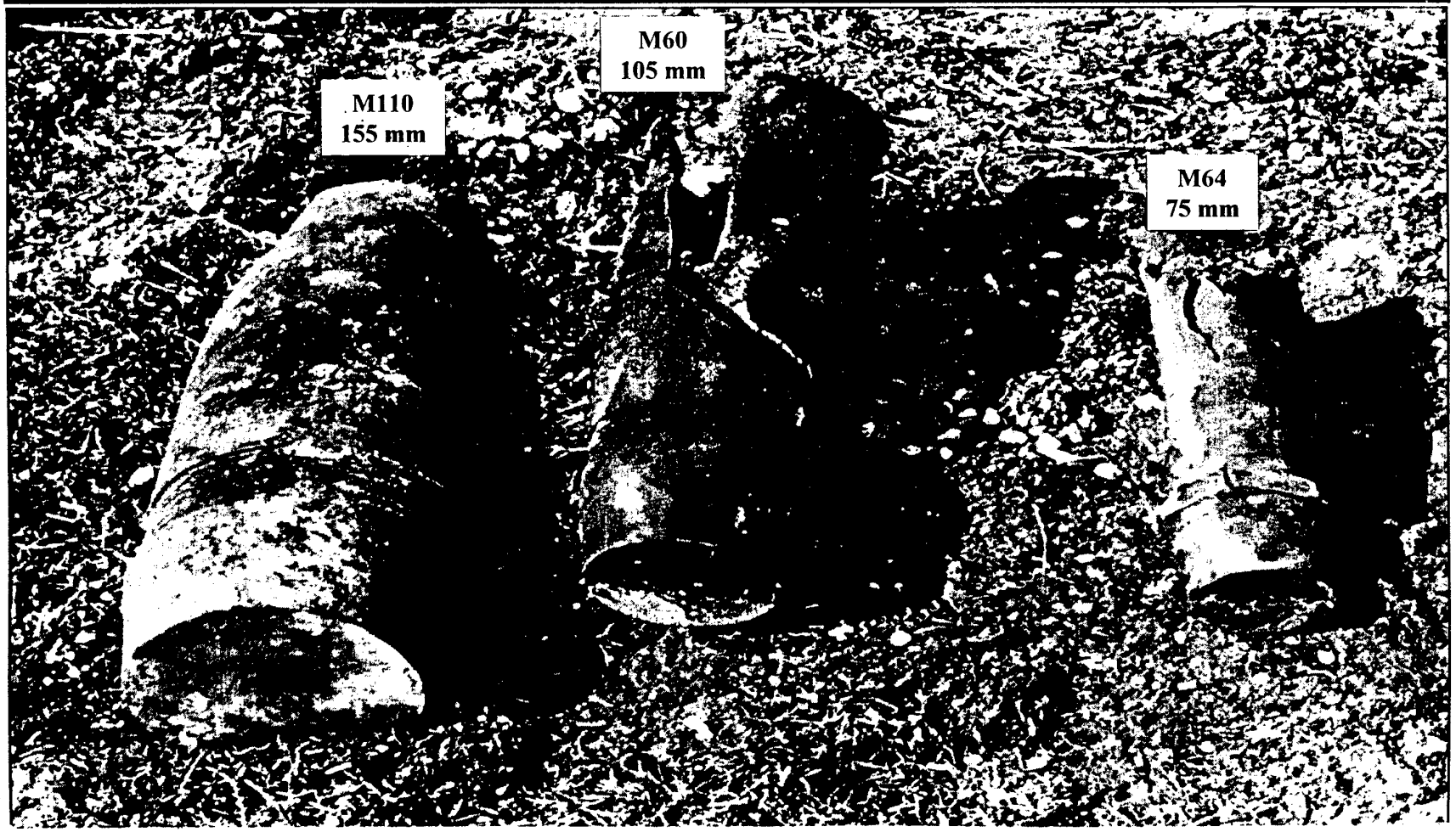
980318-002

Proceedings of the International CW Destruction Symposium
Munster, Germany, March 22-25, 1998

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RANGE RECOVERED MUNITIONS

U.S. Army Non-Stockpile Systems Program



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WEAPONS RECOVERED FROM SPRING VALLEY SITE

U.S. Army Non-Stockpile Systems Program



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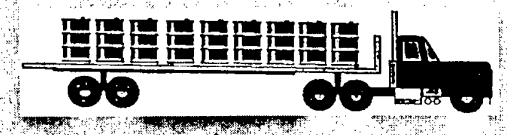
Proceedings of the International CW Destruction Symposium
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RECOVERED CWM OPERATIONAL SCHEME

U.S. Army Non-Stockpile Systems Program

TEAM TELEDYNE'S MISSION

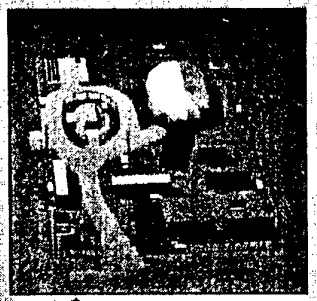


Recover - COE

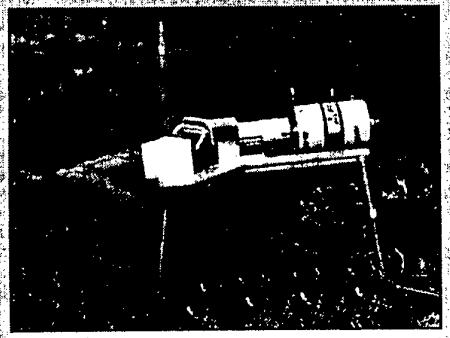


Dispose To Commercial Treatment, Storage, and Disposal Facility (TSDF)

Process



COE/MARB

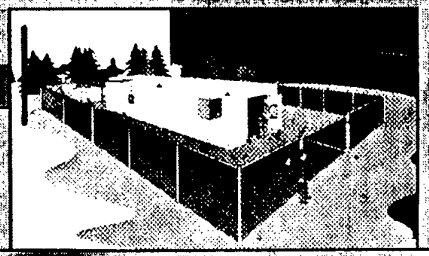


Assess (PINS/X-Ray/ RAMAN)

Army



Store



Ammo Bunker - *or* - Interim Facility

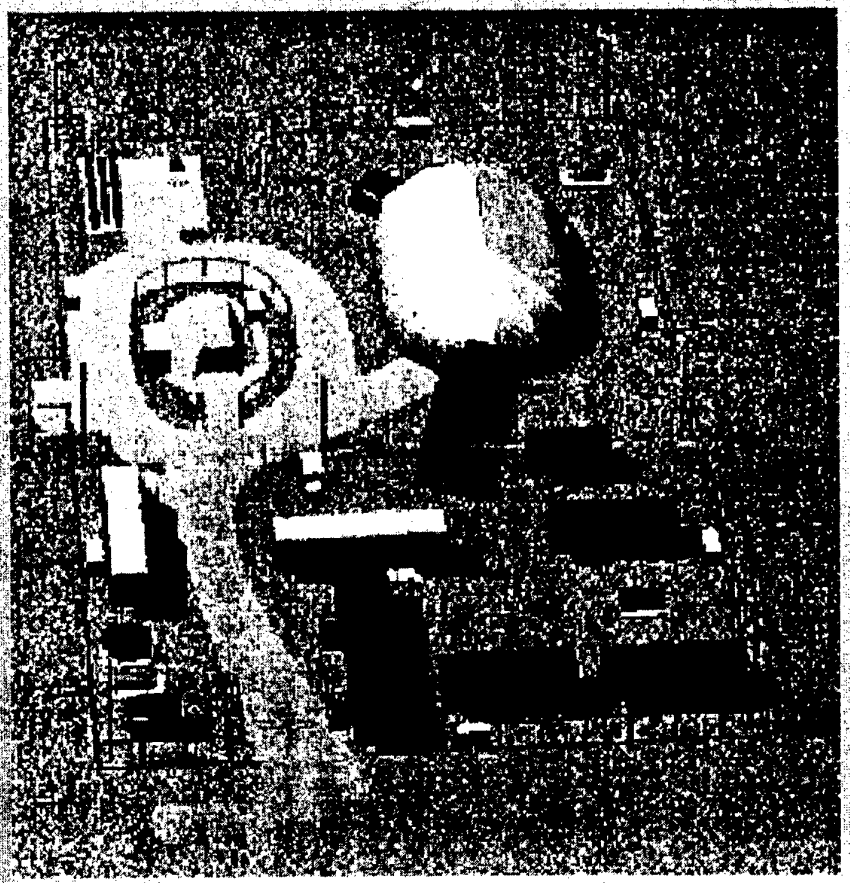
980225-LM-018

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MMD SITE OPERATIONS

U.S. Army Non-Stockpile Systems Program



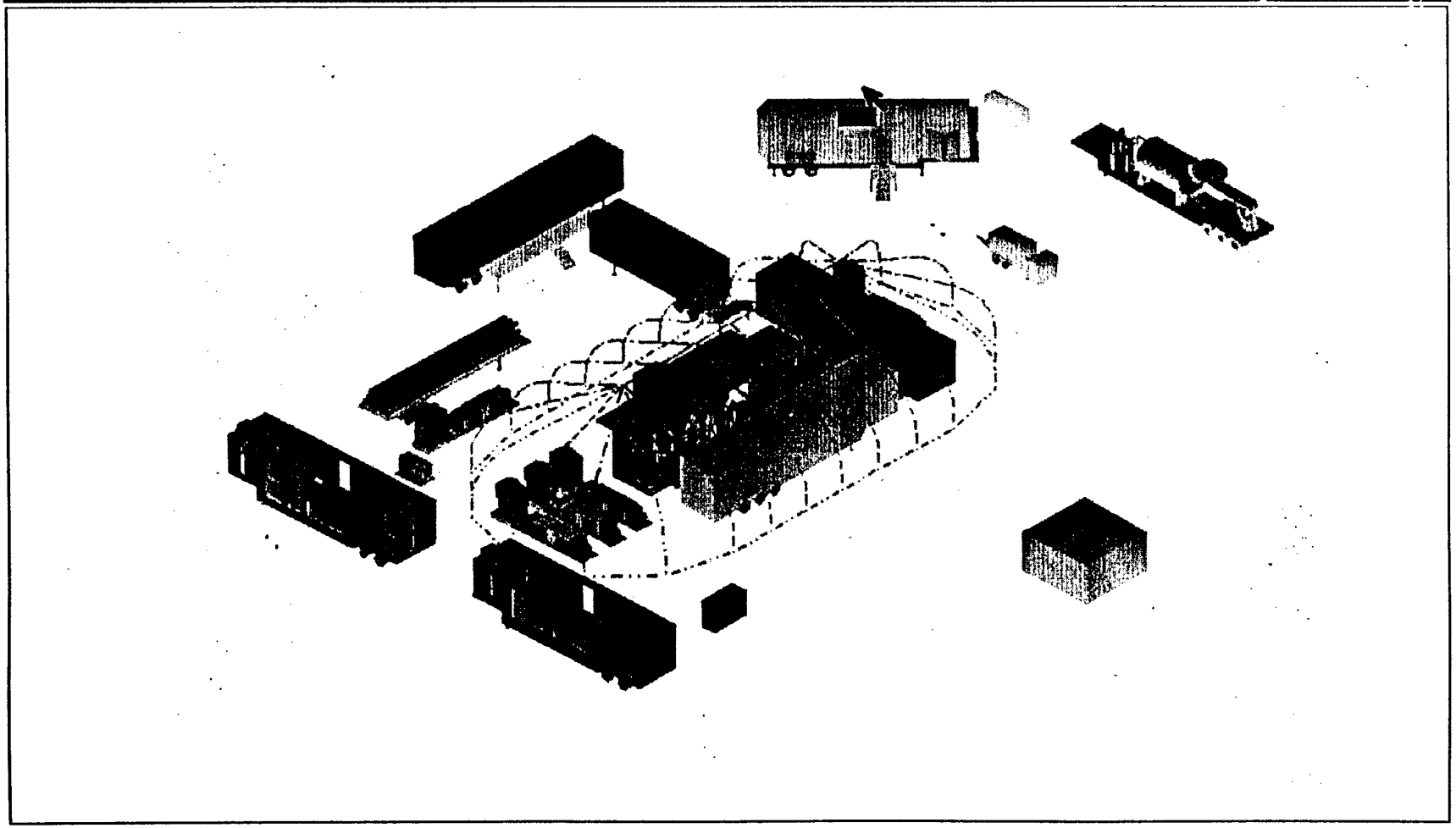
980307-014

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MMD OPERATIONS SITE

U.S. Army Non-Stockpile Systems Program



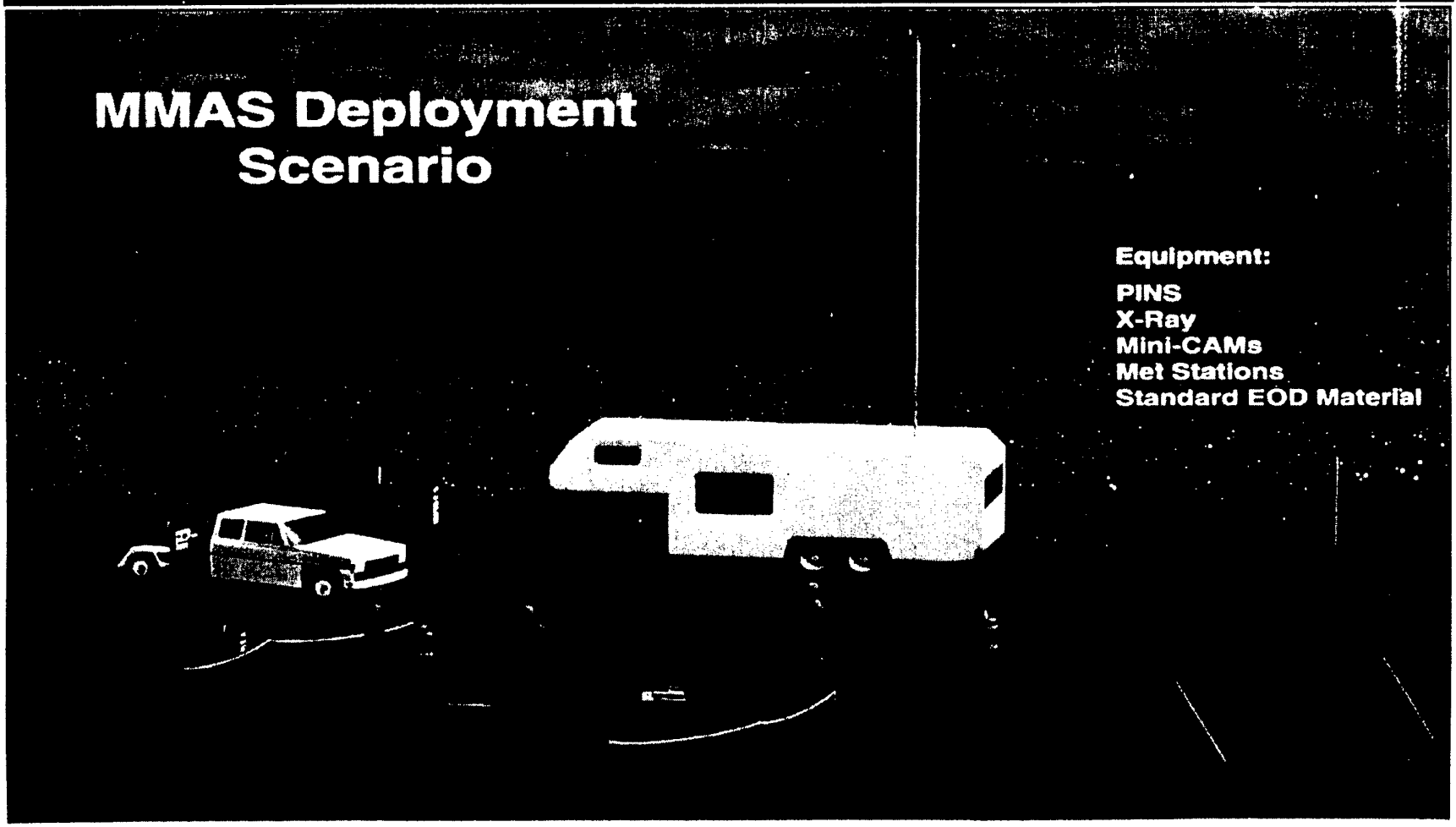
980313-006

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MMAS DEPLOYMENT SCENARIO

U.S. Army Non-Stockpile Systems Program



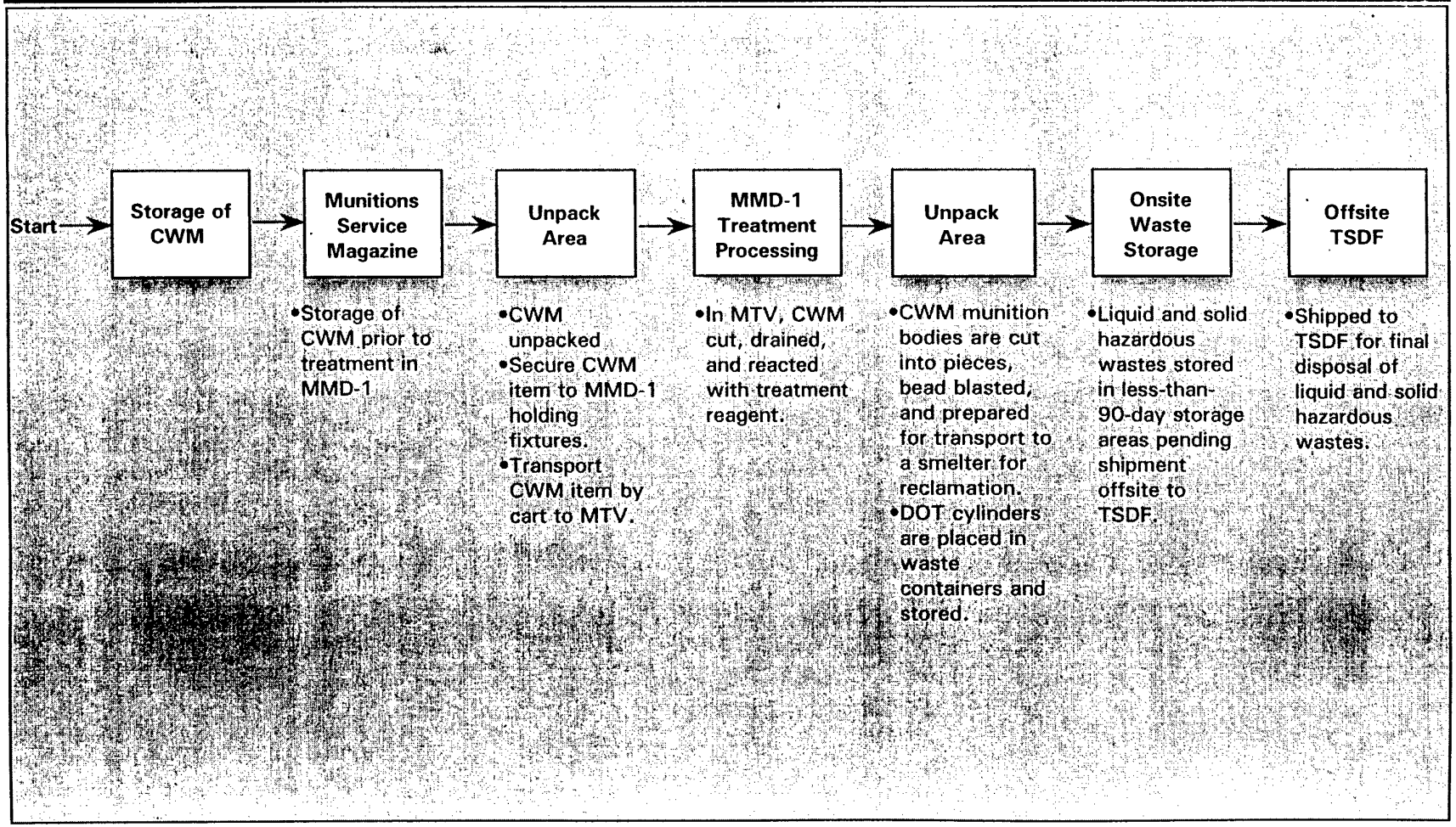
980319-001

Proceedings of the International CW Destruction Symposium
Munster, Germany, March 22-25, 1998

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MMD-1 GENERAL TREATMENT PROCESS FLOW FOR TREATING CWM

U.S. Army Non-Stockpile Systems Program



980318-001e

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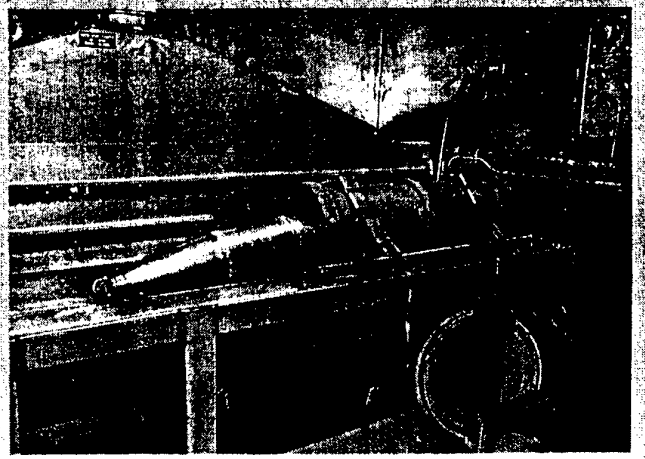
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MMD-1 UNPACK AREA

U.S. Army Non-Stockpile Systems Program







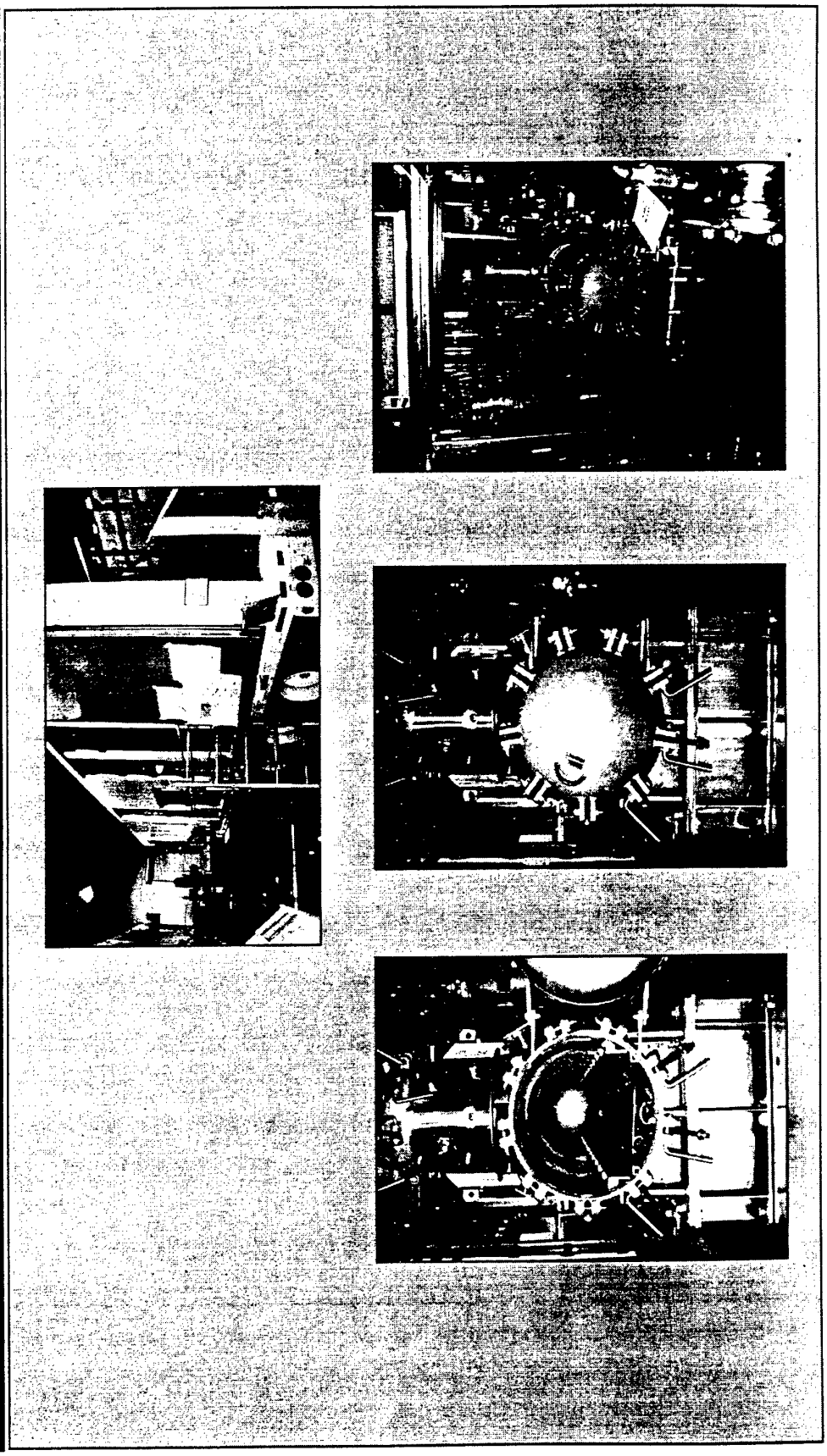
980317-003

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U.S. Army Non-Stockpile Systems Program

MMD-1

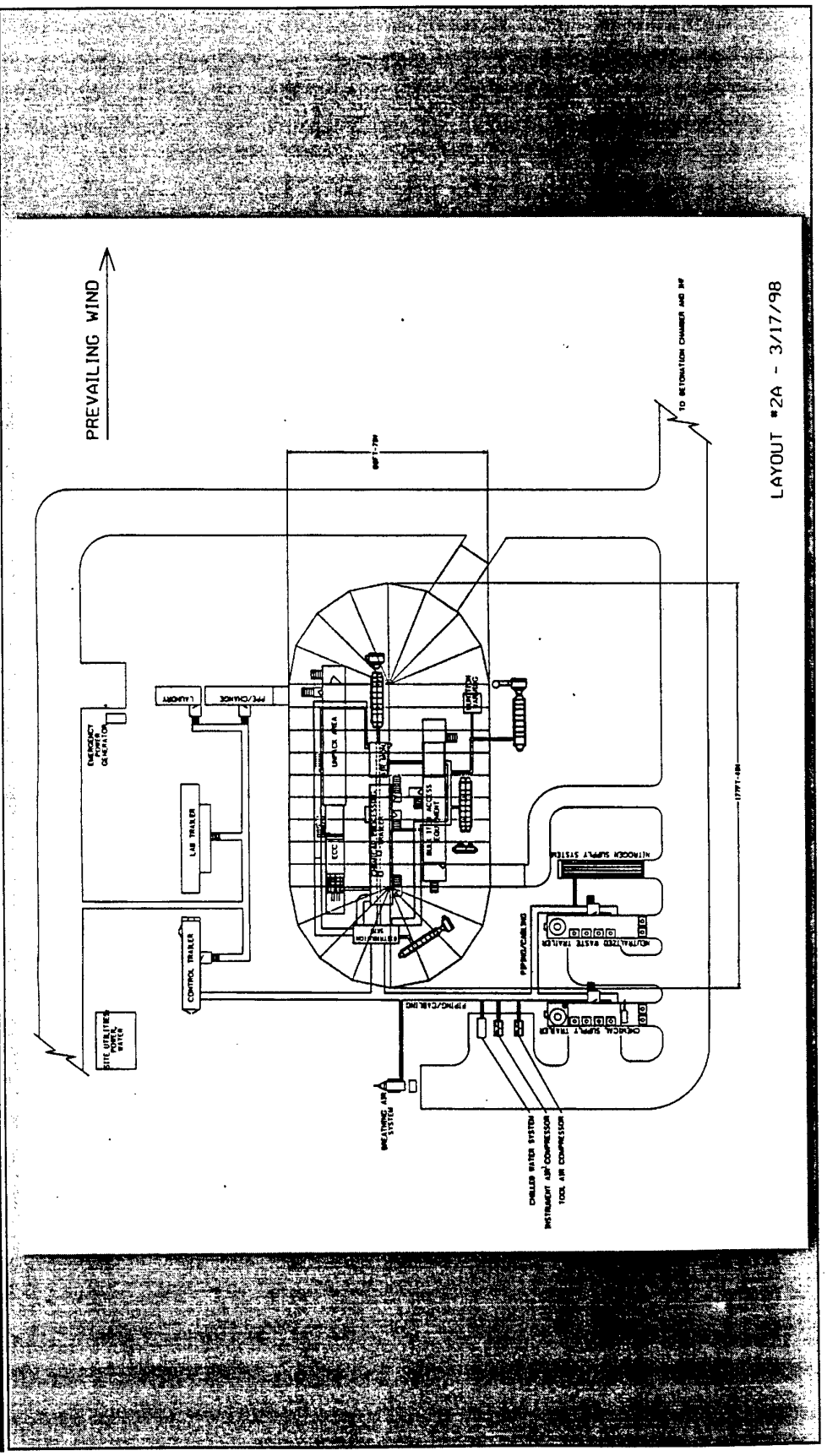


980313-003

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U.S. Army Non-Stockpile Systems Program

MMD-2
PRESENT SITE LAYOUT

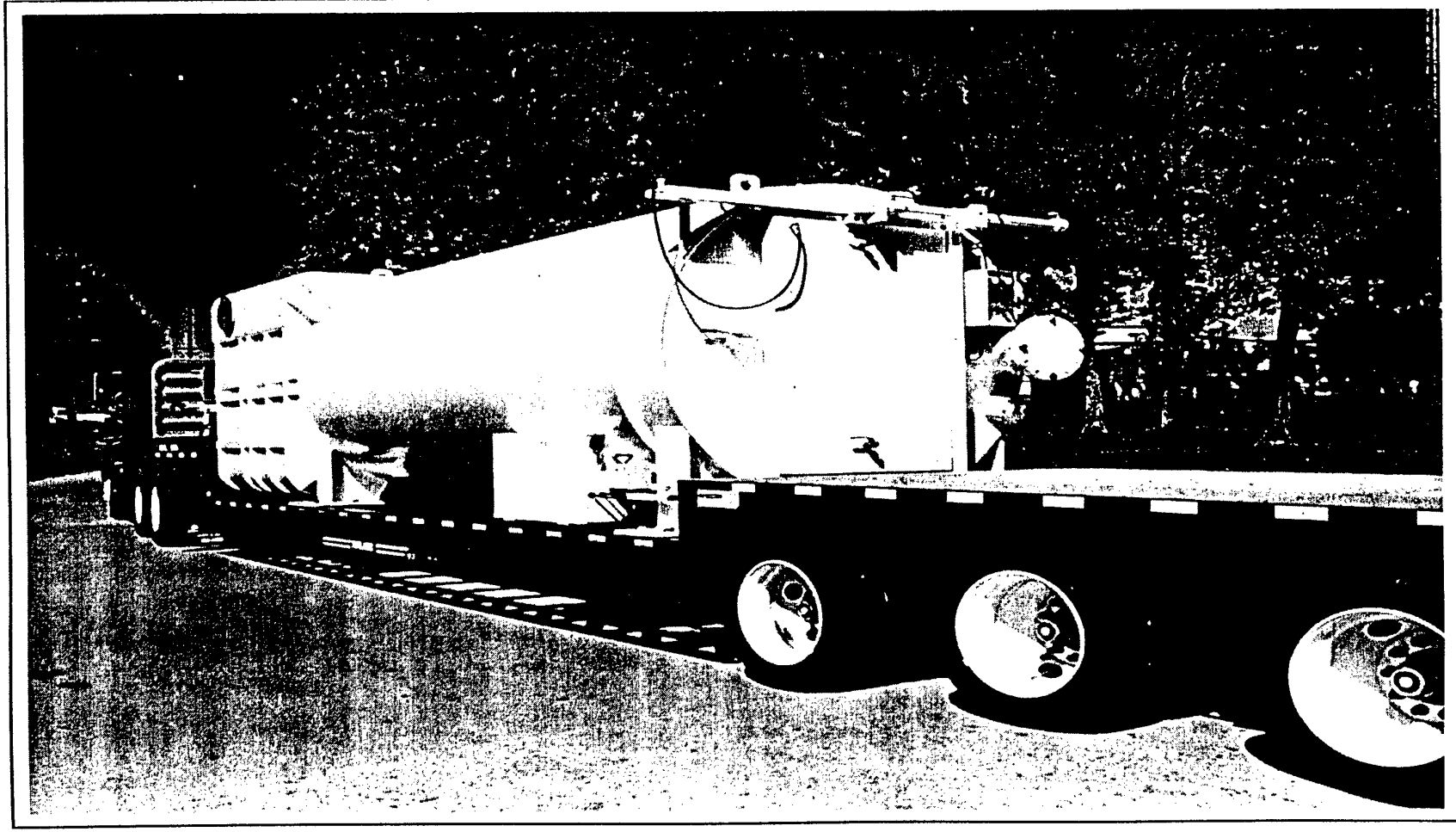


980318-004

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MMD-2 EXPLOSIVE CONTAINMENT CHAMBER

U.S. Army Non-Stockpile Systems Program



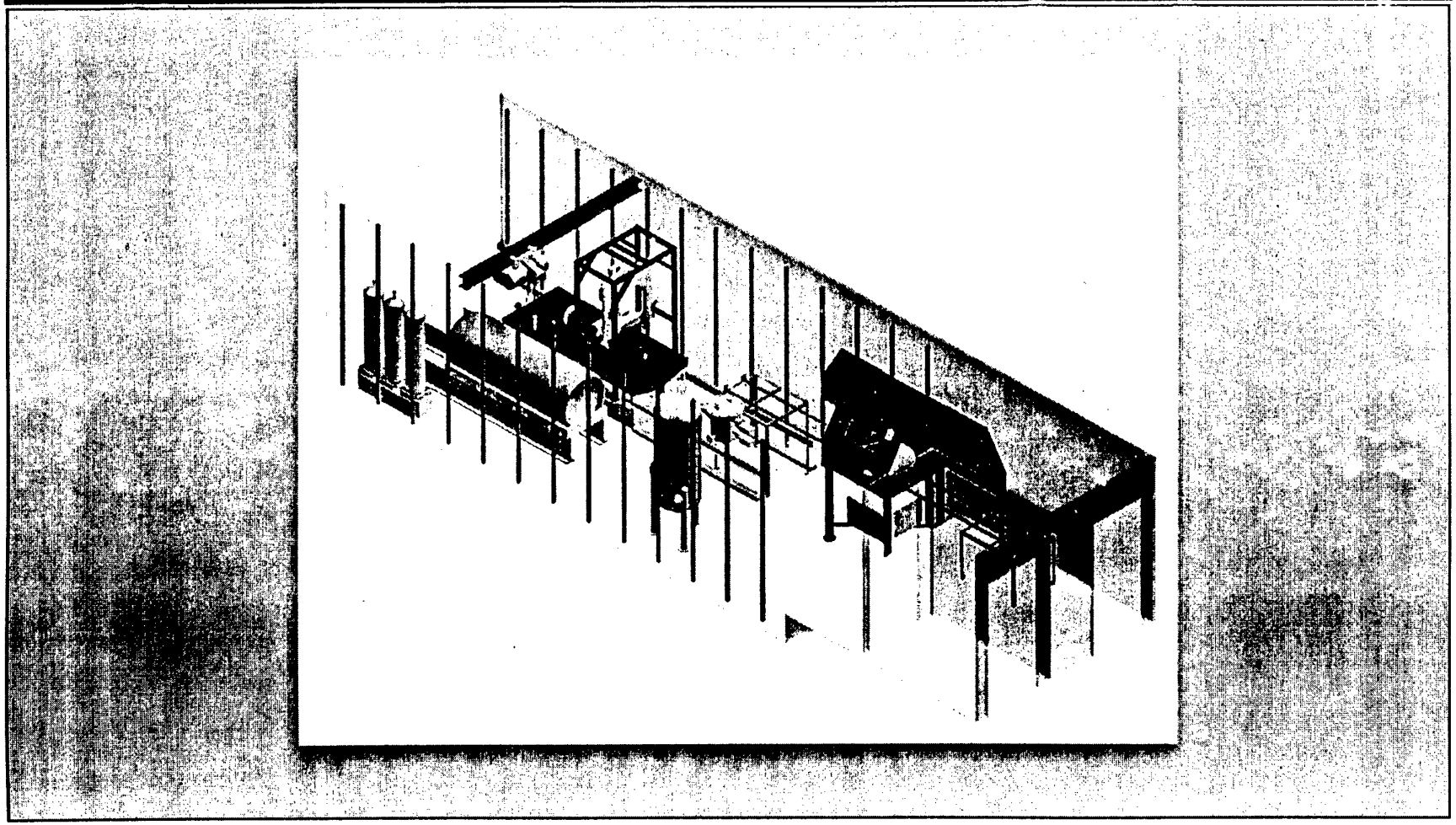
980119-LM-006

Proceedings of the International CW Destruction Symposium
Munster, Germany, March 22-25, 1998

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MMD-2 BULK ITEM TRAILER

U.S. Army Non-Stockpile Systems Program



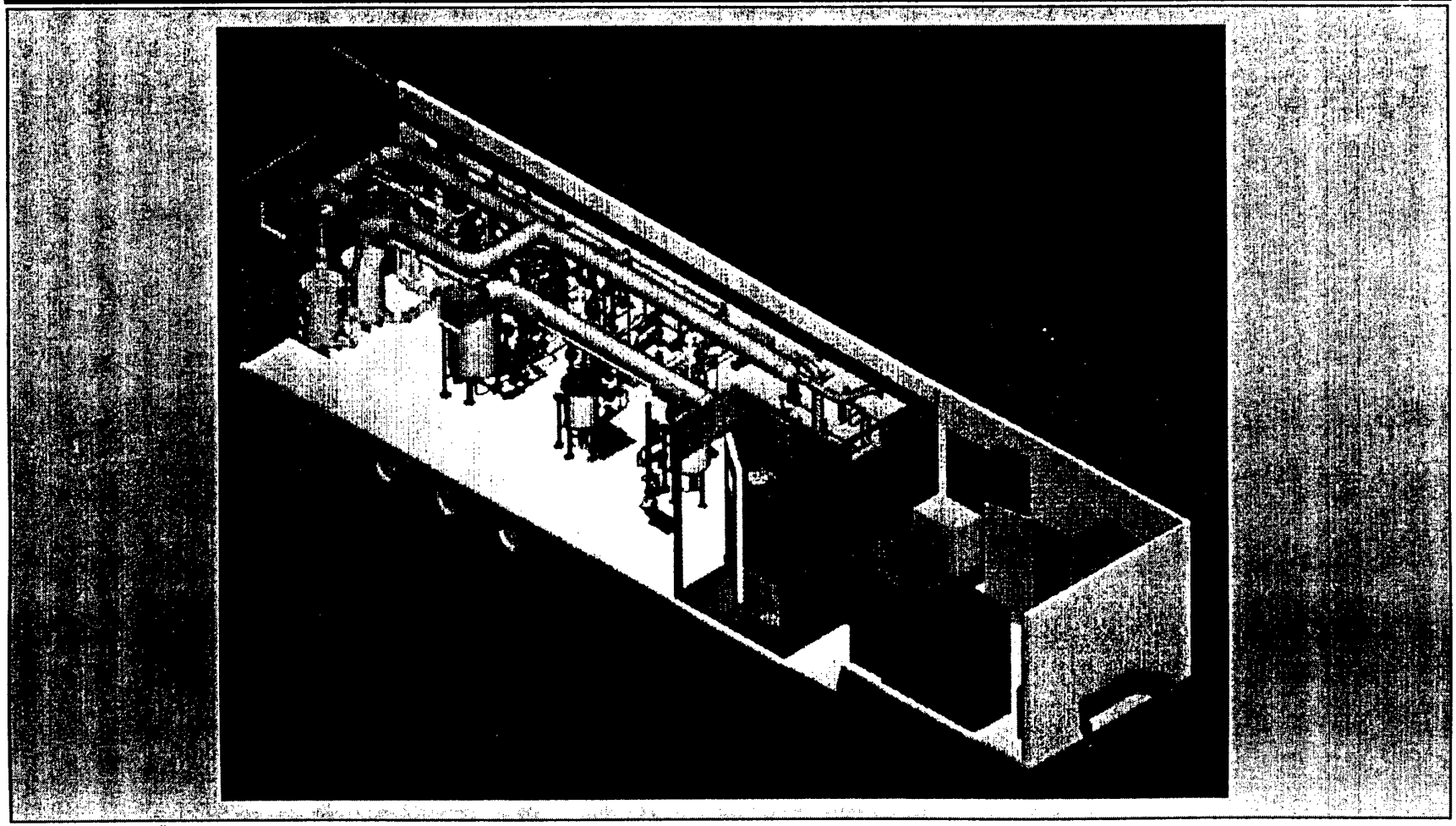
980313-011

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MMD-2 CHEMICAL PROCESSING TRAILER

U.S. Army Non-Stockpile Systems Program



980313-004

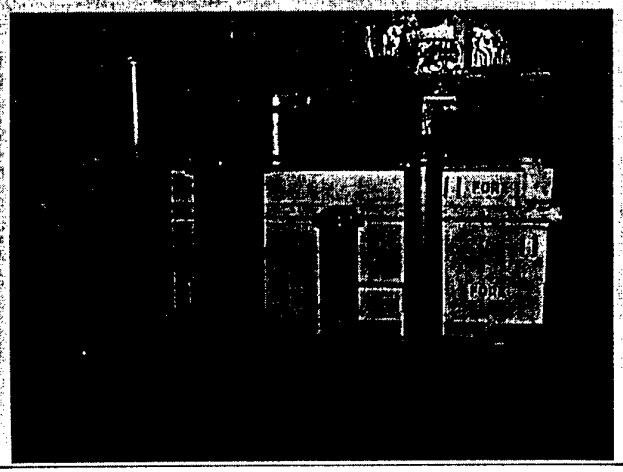
Proceedings of the International CW Destruction Symposium
Munster, Germany, March 22-25, 1998

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CAIS OVERPACKS AND TRANSPORT CONTAINERS

U.S. Army Non-Stockpile Systems Program





- CAIS may be packed in pigs, SRCs, ammo cans, small drums, or wood boxes.
- CAIS items are delivered to the RRS in a Single Pallet-Only Rocket Transporter (S.P.O.R.T).

980307-RRS-017

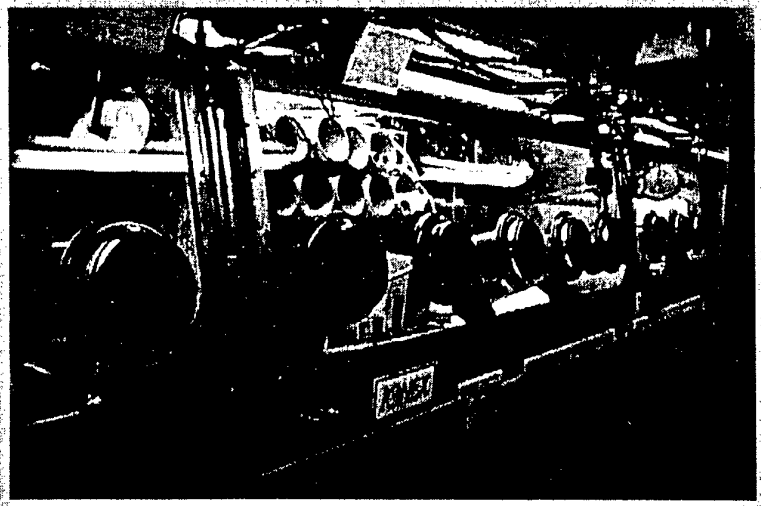
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GLOVEBOX

U.S. Army Non-Stockpile Systems Program

- 3 Station glovebox provides engineering controls for operations involving CAIS.
- Provides for storage of CAIS items awaiting processing.



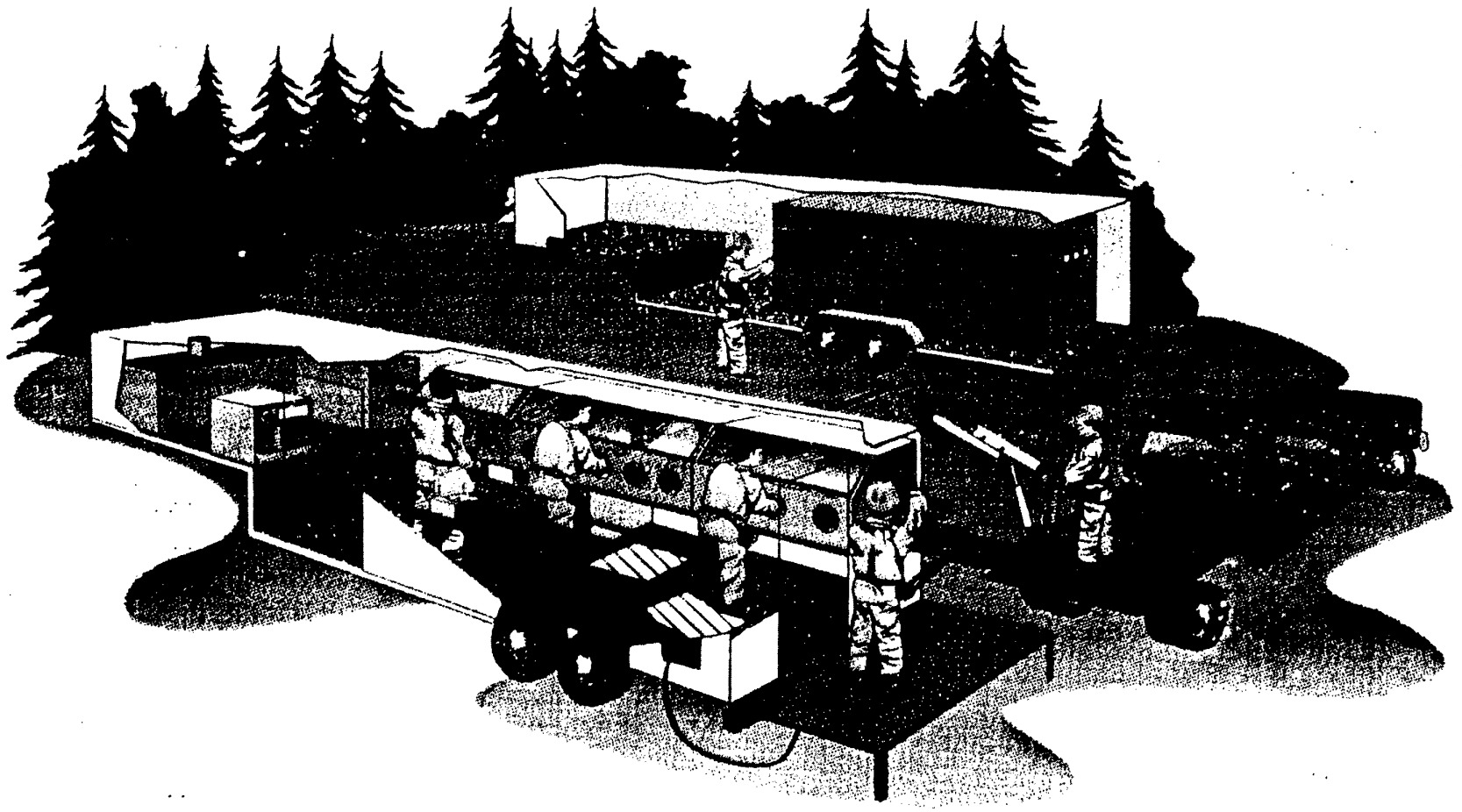
980307-RRS-021

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Munster, Germany, March 22-25, 1998

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RAPID RESPONSE SYSTEM

U.S. Army Non-Stockpile Systems Program



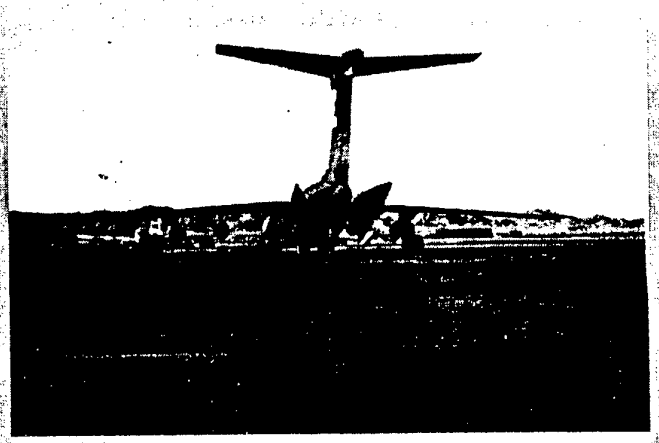
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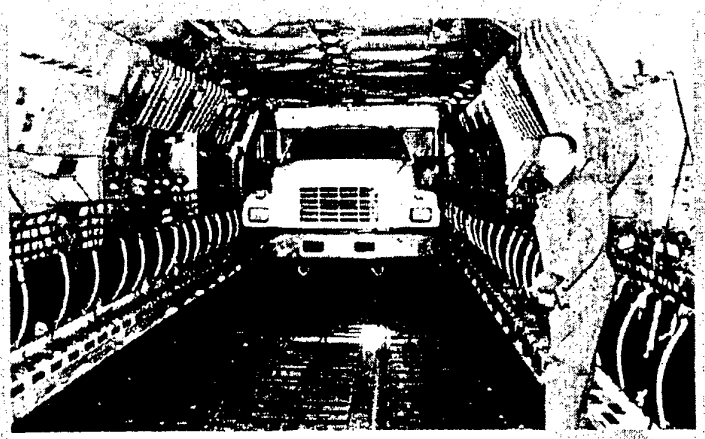
Proceedings of the International CW Destruction Symposium
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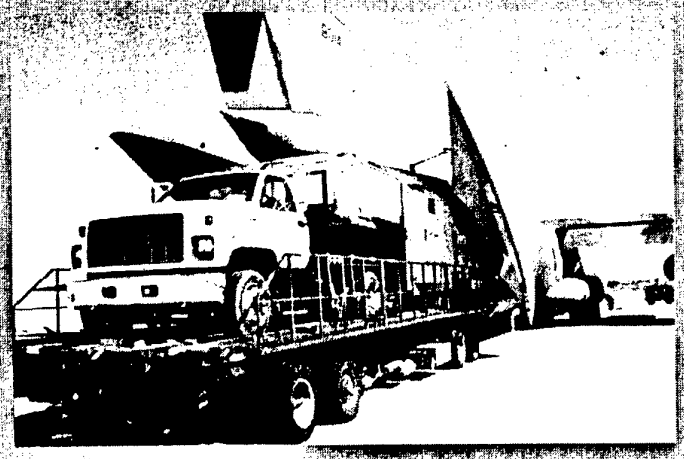
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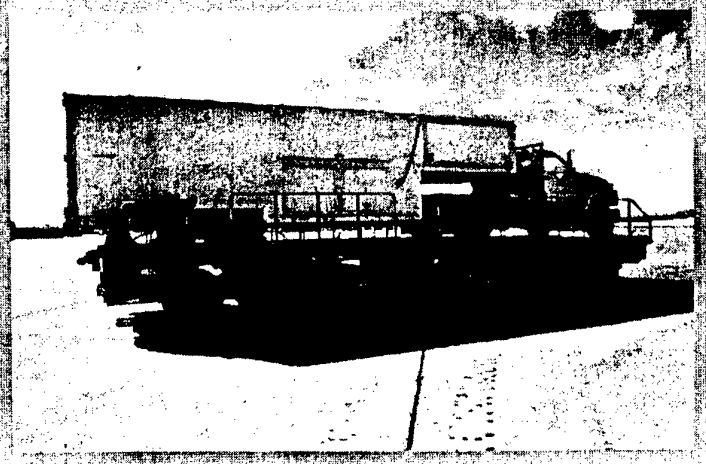
RRS - AIR TRANSPORT

U.S. Army Non-Stockpile Systems Program









980319-002

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Munster, Germany, March 22-25, 1998

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Munster, Germany, March 22-25, 1998

REVIEWING OF THE RUSSIAN CW DISPOSAL TECHNOLOGIES

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The text of the Chemical Weapons Convention does not limit the choice of technologies of the CW disposal as long as it irreversibly converts the chemical warfare agents into a form unsuitable for the production of chemical weapons, and renders the munition and other equipment unusable.

There are, however, a number of factors that need to be taken into account when considering the alternative technologies of disposal.

Destruction is available for virtually all toxic agents significant for military purposes. Specific difficulties may arise however if the CW agents are loaded into munitions, particularly when the latter become deteriorated or if the CW agents have been polymerized in bulk. Another aspect that should be taken into consideration concerning any CW disposal technology is the final form of remnants after detoxification.

The disposal of the Russian CW stockpiles is the task difficult from the scientific, technical and political point of view. Its solution is closely connected to the fulfillment of the international obligations accepted by the Russian Federation, and also with the safety of the population and the environment.

The declared stocks of this weapon are enormous. They include the following toxic substances: V-gases, sarin (GB), soman (GD), the mustard gas, lewisite, mustard gas/levisite mixtures and phosgene.The total amount of the Russian CW stockpile consists of more than 40 thousand tons (on substance), of :

- Organophosphorus supertoxic agents -about 30 thousand tons;
- Mustard gas, lewisite and their mixtures – about 8 thousand tons;
- Phosgene -about 5.0 thousand tons.

The Russian CW stockpiles for many years are stored at seven depot facilities on the territory of the Russian Federation.

The decision on the partial disposal of CW stocks was adopted by the Governement of the Soviet Union long before the CW production was stopped, as a result of the multilateral negotiation on the development of the CWC. It was caused by the necessity to destroy small parties of the obsolete or damaged chemical munition.

It is known, the CW agent that appeared in the Russian chemical munition is stored separately from an explosive component, that eliminates some of the danger of dismantle, which arises with the destruction of an American chemical arsenal. One of the first steps to solve the problem of the CW disposal was the construction of a Mobile Complex for Destruction of Organophosphorus Supertoxic Agents that make up to 80 % of the Russian chemical arsenal.Constructed at the enterprises of the Ministry of Chemical Industry, the Complex was applied by the Soviet Army in 1980.

The technology for the disposal of the chemical munition on this Complex is based on the two-stage process: chemical destruction of the CW agent with triethanolamine (for sarin or soman) or with orthophosphorous acid solution in ethylene glycole (for V-gases). The resulting low-toxic masses are subsequently incinerated in furnaces. When this technology was used, the double barrier of safety was achieved, guaranteeing both the completeness and the safety of the CW disposal operations. The process of destruction of chemical munition on a Mobile Complex is carried out in a periodic mode, that makes possible the location of practically any emergency with the help of the constant analytic control. From 1980 to 1987, the disposal of small parties of the obsolete chemical munitions was succesively carried out with the use of the Mobile Complex.

In the years that followed, much had been done at the State Research Institute GosNIIOKhT on the perfection of the CW disposal technology for the chemical munition and detoxification of their internal surfaces, on the optimization of the technological parameters of CW disposal processes and improvement of measures to preserve the environment.

For this period with the use of a Mobile Complex more than 4 thousand pieces of chemical munition were effectively destroyed with the total organophosphorus supertoxic agent mass about 280 tons. In 1987 on the Shikhany site the work of a standard Mobile Complex for the CW disposal was demonstrated to the representatives of the members-countries on disarmament negotiations in Geneva.

Decision to build a stationary the CW disposal facility designated for the development of technologies of destruction of toxic agents was accepted a little bit later. Such facility was constructed close to Chapaevsk near the Volga River. Here the same modified two-stage technology of chemical munition disposal was used which secured the safety of the personnel and ecological status of the installation. Its operation was planned for a 100-day summer period of every year. Chapaevsk disposal facility was quite ready to use the long-term successful experience of Mobile Complexes operation. The principal scheme of Chapaevsk the CW disposal facility consists of two-stage consequence of demilitarization.

Opening of munition --- Evacuation of agents --- Chemical disposal (detoxification) of agents --- Chemical detoxification of empty munition

Chapaevsk disposal facility was constructed very quickly, and without delays, but was not put into operation, as the political, economic and social changes in Russia have forced to " freeze" it untill present time.The negative experience of Chapaevsk facility has shown, that the destruction of the chemical weapons stockpiles requires another approach to solution of a number of questions concerning this problem.

Political and legal readiness of Russian Federation to begin the implementation of its international obligations, is reflected in the Federal Programme, which was authorized by the Government on March 21, 1996.

The main objectives of this Programme are: the destruction of stockpiles of the chemical weapons, the improvement of the ecological situation in areas of the CW storage and destruction, the removal of the socio-psychological tension of the population in these areas.The Programme stipulates to start the CW disposal operations from the stockpiles of blister CW agents stored in capacities, that makes about 19 % from the total amount of the Russian CW arsenal, the chemical munition containing organophosphorus CW supertoxic agents and phosgen would be destroyed in the second turn.

For an estimation of technologies of CW disposal four groups of basic criteria were chosen which reflect main requirements to such technologies: technological and ecological safety, efficiency and profitability. Besides each group of criteria should include individual criteria estimating separate properties both of a method of the CW destruction, and a technical system, which applies the method.

According to the Federal Programme some original CW destruction technologies have been created for the basic types of the chemical weapons. Here we consider some of the most probable technological solutions.Each one deserves a separate presentation.

Destruction of lewisite

There are a few possible ways of lewisite detoxification. The declared Russian stocks of this poisoning agent are stored at the sites in Gorny (293 tons), ambarka (6349 tons) and Kizner(129 tons). 98 % of Russian lewisite is stored in containers, and only 2 % - inside the chemical munition. So, it is a serious problem.

For destruction of lewisite stockpiles the most advantageous technology is based on the alkaline hydrolysis of this CW, with subsequent electrochemical hydrogenation of thus formed reaction mass to a metallic arsenic according to the scheme:

$2ClCH = CH - AsCl_2 + 6NaOH + 2H_2O \rightarrow As + 2CH = CH$ + other products

The finely dispersed metallic arsenic contains some inorganic impurity. In the technical product which has been dried up under a moderate temperature, up to 10 % of moisture remains. After subsequent sublimation in a flow of an inert gas a technical grade arsenic can be used as a starting raw material in electronic industry.

Destruction of mustard gas

One of the most advantageous way of destruction of the mustard gas stockpiles, stored at the site in Gorny is the combination of a two-stage character and periodicity of the process.The first stage consists of chemical detoxification of mustard gas to the low-toxic reaction masses.It is essential to note that the process would be carried out in a periodic mode, and with the limited amount of CW agent in a running cycle, which allows to locate any possible emergency situation. The main reactive basic agent for the first stage of disposal is a mixture of 90% of technical-grade monoethanolamine and 10% of ethyleneglycole, it allows to carry out the process of detoxification under mild conditions. It is essential to note here, that the chemical destruction of mustard gas with the above mentioned basic reactive mixture proceeds very quickly, thus diminishing the thermal effect of CW destruction. Cleavage of mustard gas molecules proceeds according to following scheme:

$$(ClCH_2CH_2)_2S+2HOCH_2CH_2NH_2 \rightarrow S(CH_2CH_2)_2N^+H(CH_2CH_2OH)Cl^- + (H_3N^+CH_2CH_2OH)Cl^-$$

$$3\ (CH_2CH_2Cl)_2S + 10HOCH_2CH_2OH \rightarrow 6ClCH_2CH_2OH + 6H_2O + (C_2H_4OC_2H_4OC_2H_4OC_2H_4OH)_2S + 6ClCH_2CH_2OH + 6H_2O$$

The second stage of the process assumes a thermal disposal of the low-toxic reaction masses: after chemical detoxification the final products of reaction may be packed into the bituminized blocks and then incinerated. On the basis of this study a pilot disposal plant for mustard gas destruction was constructed and more than 500 kg of mustard gas have been destroyed with the help of it.

Destruction of organophosphorus CW Agents

A modern Russian two-stage technology provides a separate destruction of the components of the chemical weapon - poisoning substances and shells of a chemical munition. This technology allows to safely dispose of

all kinds of chemical munition with organophosphorus toxic agents. The basic principles of the technology are the follows:The first stage consists of the agent removal from munitions and its detoxification under mild conditions up to the toxicity level of the household pesticides. The temperature of the disposal process is less than 100°C, and the pressure is less than atmospheric.

From a large variety of basic destructive reagents for demilitarization of organophosphorus supertoxic agents the most effective turned out to be a solution of monoethanolamine (for sarin and soman) and the alcoholate demilitarising mixture RD-4, containing as the basic destrictive component - potassium isobutylate and isobutyl alcohol - for V-gases.

For example:

$$2(CH_3)_2CHOP(O)F(CH_3) + 3HOCH_2CH_2NH_2 + H_2O \rightarrow (CH_3)_2CHOP(O)(CH_3)OH + (CH_3)_2CHOP(O)(CH_3)OCH_2CH_2NH_2 + 2HOCH_2CH_2N^+H_3F^-$$

The second stage of disposal is the full detoxification of the resulted products by means of the bitumenization process. At this stage the ecologically harmless substances resulted.

For the empty munition units the same detoxification two-stage procedure was applied. Its first stage consists of the chemical neutralization of the empty munition shells. This procedure is effectively carried out by washing the shells with the same reagent, which is used for neutrlalization of poisonous CW agents. It allows to perform the cleaning of the system of CW evacuation and excludes accumulation of poisonous substances in all pipelines. And the second stage of chemical munition disposal is the heat treatment. This procedure should detoxify the traces of CW which can penetrate into micropores and cracks of the metal and into the painted surface of chemical munition. The final products of this process are non-toxic bitumenous masses ready for a burial and the metal, used as the secondary raw material in an iron and steel industry.

The basic operations of the technological cycle are the following:

- Opening of munition ...
- Evacuation of poisonous substances ...
- Chemical neutralization of CW ...
- Bitumenization of products of chemical destruction ...
- Chemical neutralization (detoxification)of munition shells ...
- Thermal processing of the neutralized munition shells ...

In summary we would like to mention the general steps of the Russian - American program of an estimation of two-stages organophosphorus CW agents destruction technology.

The cooperation between the governments of Russian Federation and USA is carried out on the basis of a number of the documents and within the framework of the Program " For joint reduction of danger". In the frameworks of these plans is the construction of an industrial site for the CW destruction.

In January 1995 the group of Russian technical experts took part in the meeting at the Edgewood research centre in the USA, where they have made a report on the Russian approach to destruction of organophosphorus toxic agents During this meeting the decision for a joint estimation of Russian two-stage technology was accepted.

In May 25,1995 the Russian experts from the State Institute GosNIIOKhT came to Edgewood to start the Russian-US Programme of a joint estimation of the method. The Programme provided the experiment on destruction of three American CW agents - sarin, soman and American VX - using Russian two-stages CW disposal technology.

The second evaluation stage took place in a Russian town Saratov. Here as the object of destruction experiments the Russian battle-grade CW: sarin,soman and Russian V-gas were used.

The experiments were carried out under the scheme similar to the previous one in Edgewood. In the course of this work the newest analytical methods to control the concentration of CW agents in reaction products and bitumenized masses at a necessary level were applied.

The Russian-American joint estimation experiments have demonstrated: Russian two-stage technology is reliable and effective. It showed that the completeness of detoxification is better than 99.99999 % for sarin, better than 99.9999 % for soman and more than 99.99997 % for VX (both for Russian, and for American CW).

The above two-stage technology was found sutable by independent international expert Committee PRC (Peer Review Committee) in the frame of IUPAC.

With great interest I have studied the experience of German chemists and engineers with regard to the disposal of the "chemical heritage " of the last two world wars.

Nevertheless in this case we have a good example of disposal of rather small amounts of toxic agents. However many decisions of technological character and the experience of work at the Munster facilities can be with success used as additions to the Russial technologies of CW destruction.

Some lessons could be taken also from the unsuccessful experience of JACADS.
In summary I would like to emphasize that immutable fact, that the above mentioned Russian Disposal technologies have been created by first-class chemists and technologists with long-term experience.

The hostile criticism has a plane purpose to spoil the beginning of performance by Russia of International obligations on destruction of the Chemical Weapons.

Proceedings of the International CW Destruction Symposium
Munster, Germany, March 22-25, 1998

HANDLING SYSTEMS OF CHEMICAL WARFARE AGENTS - A GERMAN CONTRIBUTION TO THE DESTRUCTION OF CHEMICAL WEAPONS IN THE RUSSIAN FEDERATION

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Dipl.-Ing. Valentin Ratuschenko
Government Scientific Institute for organic Chemistry and Technology (GOSNIIOChT), Moscow

Introduction

- The destruction of chemical warfare agents pursuant to the UN Convention requires the transfer of the toxic chemicals to be destroyed, together with the ammunition or the devices and equipment devised for their application, as well as from other storage containers, into the objects of the destruction facilities.
- The handling of the chemical warfare agents on the way to the destruction facilities represents an important aspect of the preparation and realization of the ultimate destruction of these toxic chemicals.
- The execution of the handling process depends on political decisions (financial, material, industrial, sociological as well as safety aspects, and others) with respect to the location of the destruction facility and the resulting distance between the present storage location of the chemical warfare agents and the site of the destruction facility, the type of the destruction process and the capacities of the destruction installation as well as the type of storage of the chemical warfare agents.
- Handling systems reduce, on one hand, the risks in the range of the storage site of the chemical warfare agents by decreasing the storage risks, but they possess, on the other hand, an inherent risk potential on account of the immanent processing conditions. The handling process requires, as a consequence, an extremely safety conscious technological design on the basis of the physico-chemical properties and the total risk potential of the toxic chemicals, also under consideration of the storage process.
- The further elaboration is restricted to toxic chemicals and relevant preliminary products of the types of chemical warfare agents S-Yperit, Lewisit and their mixtures in the sovereignty of the Russian Federation.

1. The physico-chemical, toxicological and technological requirements of the chemical warfare agents regarding technical facilities and systems

Major physico-chemical and toxicological data of the agents present are listed in Enclosure.

When considering the requirements on the part of the chemical warfare agents regarding the technical facilities and systems, the effects of the storage length and storage conditions on these agents or mixtures of agents must be taken into consideration, besides the basic data of the chemical warfare agents in general.

When planning the handling processes, it must be taken into consideration that mixtures of agents with different properties must be regarded in a common process.

The following items are of particular importance for the handling process:

- Density
- Temperature
- Vapor pressure
- Boiling point
- Viscosity (dynamic and kinematic)
- Explosive properties (ignition energy, ignition temperature, flash point, minimum oxygen content, chlorine explosion ability and similar)
- Reaction with and effect on other agents (corrosion, resorption, swell effect)
- Initiating exothermic reactions in the course of conveying processes (mixing, turbulences) in semi-stable systems
- External influences by means of outside temperature, solar radiation, air humidity and similar influences

A major aspect when considering the physical properties, is posed by processes during storage, as detailed in the following:

- The storage of the above-mentioned chemical warfare agents was effected in the Russian Federation also without ammunition in the form of cisterns and tanks (capacity approx. 50 m^3), as well as drums (100 / 250 ltr), of

  - chemical warfare agents with different solution agents or mixtures of Yperit and Lewisit (mixtures of Yperit carbon tetrachloride, Yperit-Aniline Yperit/Lewisit mixtures with and without solution agents (dichloro ethane).

- Production period of time of the chemical warfare agents dates up to fifty years ago, and the resulting technical particularities during manufacture (different manufacturing processes, relatively low purity of the primary products - the military use did not require extreme purity of the products),
- Problems of product stability, during manufacture and the beginning of the storage, the aspect of the long-term storage was taken into consideration either not at all or only to a limited extent,
- Effects of the long-term storage and technical problems on account of the time due to leaks at coupling spots (gaskets, fittings and similar devices) as well as the influence of undesired effects, such extreme temperature changes, penetration of oxygen of the air and air humidity into the storage containers, counter-reactions of the chemical agents among each other, with Fe^{n+} and in the presence of metallic complexes, emissions of highly volatile substances from the containers,
- Effects of the forces of gravity (separation of solutions, formation of layers corresponding to the different densities, settling of solid reaction mixtures or dissolved corrosion products),
- Vulcanization effects in the case of Yperit mixtures (formation of long-chain thioether compounds as well as elastic marginal layers),
- Effects of aggressive components on the storage containers (corrosion) including connected or built-in technical systems and safety installations with the reduction of the safety of the container as well as the total storage sector,-
- Presence of chemical warfare agents in different compositions per storage container with solving agents in different concentrations, various preliminary products, by-products and decomposition products as well as different types and proportions of solid agents.
  It is possible, as a consequence, that within one cistern substances may be present with densities from 0.8 to 2.1 kg/dm^3, vapor pressures from 0.5 to 242 mbar, saturation concentrations from 0.61 to 985 g/m^3, flash points from -10 to +160 °C, ignition temperatures from 270 to 982 °C as well as PDK values (threshold limit values required by the Russian Federation) from 0.0002 to 92 mg/m^3.
- The chemical analysis and the assessment of physical actual conditions of the contents is of special importance for the handling and subsequent destruction processes.

Another aspect of the coverage of the physical properties is the determination of the type of decontamination strategy within the processing range and the agents to be applied in the course of these processes.

- Selection of a solvating process with suitable solvent agents by dissolving the toxic chemicals, absorption of the solvent mixture, subsequent separation of the mixtures in the range of the site of the destruction facility, destruction of the toxic agents in the destruction installation and renewed utilization of the solvents suitable to be employed again.
- Selection of a decontamination process by the use of mixtures suitable to react on site and subsequent treatment by means of rinsing agents, the absorption of all reaction agents and their destruction in the destruction facility.

In addition to the well-known toxic properties of the chemical warfare agents, also those of the decomposition products (arsine carbon disulfide and others) must be taken into consideration. The presence of different toxic substances requires high demands regarding the safety installations of the system, the measuring processes to be applied and the resulting measuring technique. It is to be taken into consideration that the permissible concentrations of noxious substances in the air of the environment for the production sites and regions in the vicinity must be observed and maintained, and it must be safeguarded that these can be verified. When selecting the measuring techniques to be employed, it must be taken into consideration that the handling processes require relatively brief usable assessment periods of time inline (problems on account of processing pressure and processing temperature) and that cross sensitivity must be excluded.

2. Inclusion of a handling system for chemical warfare agents in a destruction object for chemical weapons

This elaboration so far shows that the physico-chemical properties of the chemical warfare agents stored in the object GORNY, in the Saratow region, pose extremely high requirements for the mobile installation modules to be designed and supplied by our firm, for the handling of the chemical warfare agents. Simultaneously, the "Standards for the designing and utilization of production installations for the destruction of chemical weapons", 1995 status, as well as the environmental protection demands of the Russian Federation must be taken into consideration.

The Russian party submitted the following basic requirements to the German enterprise for the development of a semi-mobile handling system:

- Transportation of the chemical warfare agents of the above-mentioned types and their mixtures must be effected exclusively in vacuum.
- A Technological Transportation Container of Russian production must be used as a means of transportation for the chemical warfare agents. This design is "Container in Container", capacity approx. 0.8 m^3, filling of the interior container is effected via an integrated suction/filling head after opening/removal of the container cover of the exterior container.
- Feeding of detoxication liquids through mobile supply systems
- Application of firmly established detoxication methods, detoxication liquids and fire extinguishing agents.

Furthermore, the following Russian demands were made:

According to the probability of a possible release of the chemical warfare agent in the course of the handling process, the mobile installation modules are subdivided into three separate risk groups.

The sections under the Risk Group I of the mobile installation modules, that means the rooms where the chemical warfare agent is located for intermediary storage or is refilled, or where fumes or exhaust gases or exhaust air are purified, are to be separated from the sections of the Risk Group II by means of locks.

A vacuum of the required magnitude must be created in the lock modules for the entering of the maintenance staff, and the special treatment of personal protective equipment as well as materials employed must be warranted by the installation of a "Black Zone".

The doors arranged in the mobile installation modules must have dimensions which allow the evacuation of the staff in the case of an accident without any problems. They must open automatically, they must be provided with optical signal devices which display a function of the exhaust installation and the presence of the vacuum demanded in the section, and they are allowed to permit an access only on the condition that the operation parameters demanded are warranted.

All surfaces of the mobile installation modules and the incorporated assembly groups and units must be provided with protective coats which exclude the absorption of chemical warfare agents of the above-mentioned types in the form of vapor or drops to a maximum and which must, in addition, be resistant to chemically aggressive detoxication recipes. All sections where quantities of chemical warfare agents and detoxication recipes may escape must be equipped with collection basins to collect any such quantities of chemical warfare agents and detoxication recipes having possibly escaped. The size of these basins is to be designed in such a dimension that the total noxious substance present in the section in question (from the containers and the tubes) as well as the required detoxication, sorption, neutralization or fire extinguishing agents can safely be collected.

In all sections of the mobile installation modules, a mechanical forced aeration system must be installed, and in the sections with the Risk Groups I and II, a natural aeration must be excluded. The exhaust air from all mobile installation modules must be fed via a separate exhaust air system to a purification system, which belongs to the installation complex, for the exhaust air. The noxious substances concentrations contained in the exhaust gas flow /exhaust air flow are checked in the complete exhaust air system by means of automatic gas measuring devices and by taking samples for the analysis in the laboratory. The only currently available automatic measuring system that meets the demands of assessing the fixed PDK values (threshold limit values required by the Russian Federation) and the process conditions (pressure, temperature, cross sensitivity) is a Ion Mobility Spectrometer of a German manufacturer. This measuring system was selected and adapted to the specific requirements (simultaneous measuring and evaluation of six separate substances). It works within a reasonable evalutation time.

The exhaust air, cleaned by chemo-sorption and adsorption filters, is released into the atmosphere (0,5 PDK-value) only after having been reduced to the maximum permissible concentration.

In terms of organization of the technological process, the following is to be taken into consideration:

- Utilization of equipment, the quantity of which must be as small as possible, in which chemical warfare agents of the type mentioned are contained;
- Reduction of the quantity of chemical warfare agents that are present simultaneously in the mobile installation to the minimum required;
- Exclusion of the direct contact of the operators with the chemical warfare agent;
- Equipment of the workplaces with means which safeguard the sealing of leaks and the decontamination and detoxication of contaminated surfaces;
- Maximum automation of the multifarious technological processes;
- Checking and surveying of especially endangered sections both from the safety control operation stand and from a central surveying control board by means of video installations and wired connections.

Extremely high demands are made, as has been explained before, regarding the equipment and materials to be used. The tube lines, fixtures, special assemblies and collecting basins that come into contact with chemical warfare agents of the above-mentioned types in the form of vapor or drops must be manufactured of corrosion resistant types of steel. The materials used for sealing various connections must be resistant both to chemical warfare agents and to detoxication recipes.

Protective coatings are to be applied to the protection of surfaces of the constructions and equipment incorporated in the sections with the Risk Groups I and II, which are made of carbon steel. These protective coatings are to absorb, on one hand, the chemical warfare agent of the type mentioned to an extent as small as possible and they must, on the other hand, withstand several special treatments with various detoxication recipes.

For tube lines through which chemical warfare agents are conveyed, seamless tubes must be used. Any welding seams which become necessary must be checked one hundred percent by non-destructive testing methods. Flange connections between tube lines and fittings or equipment which contain chemical warfare agents are to be executed with a sealing surface "Key and Feather".

For the special protection in the case of all lines conveying noxious gases outside of the containers, double tube or double hose systems were selected, the spacing in between being filled permanently with a vacuum, and which can be checked by means of special measuring methods whether the inside tubes or hoses are still leakproof.

In the case of fittings, the demand for special requirements for leak protection has been made, in particular the prohibition of stuffing box packings for valve spindle sealing. For this reason, products were selected for the ball valves which are provided with special seals of the ball proper and the drive shaft, which equal the bellows of a valve and have been tested by competent survey associations for a permissible leakage rate of a maximum of 10^{-6} mbar * ltr/sec.

The requirements for the automated systems utilized are as follows:

- They must safeguard the automatic and semi-automatic guiding of the handling process in the operation, transportation and accident regime with a maximum of safety.
- They must display the condition of the actuating systems of the fittings and devices with remote control.
- They must safeguard the tightness of the technological systems used.
- They must signal by acoustic and optical means trouble and defects in the course of the technological process.
- They must interrupt the process of the technological procedure in such a way that dangers by means of the chemical warfare agents is excluded.
- They must signal by acoustic and optical means when the maximum of the concentrations permitted of the noxious substances are exceeded in the sections.
- They must trip alarm devices in the case of fire and switch on the fire fighting devices.

The explanation of these normative requirements makes it evident that - notwithstanding the special technical knowledge of the staff employed in our enterprise - it was necessary in the interest of achieving optimum technical solutions to work together with a competent institution. Thus it was possible, on the basis of a contract

concluded with a government-approved research institute for organic chemistry and technology, to test a large number of materials which are employed in the mobile modules of the installation, regarding their stability to chemical warfare agents of the above-mentioned types and to the detoxication recipes.

The examinations show how difficult it is to find materials generally offered in the trade which are resistant both to the factors of effect of Yperit, of Lewisit, of the detoxication recipes and of the solvents in question.

The results of the examinations permit us to select suitable protective coats of paint, sealing materials and components and thus to realize the high requirements in terms of reliability of the technological equipment to observe environmental protection and the disposal of used materials.

For the execution of the process, storage programmable controls have been selected with a high degree of availability (redundant systems) which meet the requirements of Safety Class VI for process control technology. The storage programmable controls are coupled with a process control system via two separate bus connections (wire and radio) and connected as slave. The storage programmable controls are in a position to maintain, in the case of a failure of the process control system, a safety operation in communication with other storage programmable controls.

3. Arrangement and performance principles of a handling system for chemical warfare agents

On the basis of the Russian requirements, a semi-mobile handling system for chemical warfare agents was developed by the German specialists in cooperation with the Russian partners which is designed in a modular way and which can be adapted at modest expenditures to other performance requirements.

3.1 Purpose of application

- Removal of highly toxic, explosion prone and aggressive warfare and dangerous substances from

  - tank containers, cisterns
  - containers

  optional from warfare ammunition via delaboratory systems

- Refilling possibilities of above-mentioned chemical warfare agents into:

  - TTCs of Russian production

  optional into
  - Double-wall tube-line systems
  - 1,000 ltr safety containers with (UN License) or
  - 10m³ safety transport containers with (UN License)

The handling complex consists of

- Emptying container, with average and sanitary module,
- Detoxication/solvent agent container
- Preparation container (dry process)
- Preparation container (wet process) with storage container
- Lock container
- Process guiding container
- Container for the production and preparation of compressed air

Optional systems

- Tube line / Go-devil system for the feeding of highly viscous substances
- Metering container for the production of detoxication liquid from three different components and one detoxication liquid from one liquid component and one component in powder form
- Preparation container for mixtures for the separation of liquid and solid components
- High-pressure detoxication container with intense jet adapter for the high-pressure cleansing for the cisterns / containers

- Cooling water preparation container with return cooling of heated process cooling water and the measuring checking regarding the presence of dangerous substances due to the penetration of the gas cooling component.

Tasks for emptying container:

- Vacuum conveying of chemical warfare / dangerous agents and substances from containers
- Refilling in safety containers with:

- Maximum performance:
  - When using TTC of Russian production approx. 3,200 ltr or 3.2 m^3 / work shift (8 hours)
- optional
  - When using 1,000 ltr safety containers, approx. 4,800 ltr or 4.8 m^3 / work shift (8 hours)
  - When using 10 m^3 - safety transportation containers approx. 9,600 ltr or 9.6 m^3 / 3 hours or approx. 25 m^3 / work shift (8 hours)

- Process under storage programmable controls for automatic and manual operation

3.2 Tasks for detoxication / solvent agent container:

- Conveying of detoxication agents / solvent agents to the location required
- Storage of detoxication agents / solvent agents
- Refilling of detoxication agents / solvent agents
- Performance possible:
  - independent suction and conveying of different types of liquids, including explosion prone substances
  - storage capacity one container at 3.5 m^3, one container at 2.5 ^{3}
  - possible refilling between the two containers,
  - release at four different user locations,
  - process under storage programmable controls for automatic and manual operation
  - construction of the container pursuant to the Water Resources Act

3.3 Tasks for preparation container (dry process) in the 20' ISO container

- Removal of warfare agent gases / dangerous substances gases from the exhaust air or from ground-air exhaust systems with medium concentration (approx. ten times the permissible threshold limit value),
- Checking of the concentration of the exhaust air,
- Automatic recirculation of the warfare agents / dangerous substances gases at concentrations > ten times the threshold limit value to the preparation container (wet process),
- Process under storage programmable controls for automatic and manual operation

3.4 Task of the preparation complex wet process (10'/20' ISO container)

- Chemo-sorptive preparation of:
  - warfare gases,
  - dangerous substances gases (acid, alcaline) from different emission sources (space / container inside air, floor-air exhaustion an similar sources)
- collection of liquid warfare agents or other dangerous substances in case of accidents from containers, from container inside areas or other sources
- Gas/air suction performance with the built-in densifier in the container of 600 m^3/h,
- Storage of 1 m^3 of an alkaline/solvating, one acid chemo-sorption liquid as well as a regeneration liquid
- Filling and refilling in connection with the container into the containers or tube lines respectively of the chemo-sorption container,
- Measuring controls and checking of the chemo-sorption including exhaust air,
- Process under storage programmable controls for automatic operation

3.5 Task of the safety lock container (10' ISO container

- Entry and exit without danger via a hermetically closing connection system to containers where dangerous substances are emitted or in which risks are existing on account of liquids or solid substances,
- Surveying of staff working in dangerous areas,
- Survey by means of measuring devices of the working atmosphere,
- Utilization of the 'Block Lock' with shower system, air preparation via active carbon filter, additional exhaust with flexible suction hose, air condition installation for extreme conditions,
- Realization of measures of personal detoxication / decontamination
- Process under storage programmable controls for automatic and manual operation

3.6 Task of the process guiding system (20' ISO container)

The process guiding container serves for the central control and guiding of the functionally connected special containers of the emptying complex, including the safety surveying activities, of the communication with the storage programmable controls of the containers arranged as slave, the recording of the automatic functions of the components of the installation and the manual operation.

The process guiding container consists of:
- Thermo-insulated container shell (20') with air-condition system and access safety lock,
- Process guiding system, Messrs. Fisher-Rosemount, Type RS-3 on the basis of MS-Win NT 4.0"
- Electric current supply unit with independent, self-contained current energy supply, measurement technique (electric energy supply, temperature measuring and similar devices)
- Fire fighting center (License granted by the Association of Insurance Companies), of the complex, connected with the sensors installed in the containers, as well as fire fighting agents
- Video center with control and long-term storage modules, color and black and white monitors, where up to ten cameras can be connected.

3.7 Task of the container for the generation and preparation of compressed air

The container for the generation and preparation of compressed air serves to generate and prepare highly pure compressed air for controlling, fittings, pumps and other users.

- Parameters:
  - Capacity of 620 Nm^3/h at 8 bar
  - Residual oil content < 0.005 mg/m^3 at 20 °C / 7 bar (absolute)
  - Solid particle residual size < 0.01 µm / m^3
  - Differential pressure (new condition) < 0.2 bar

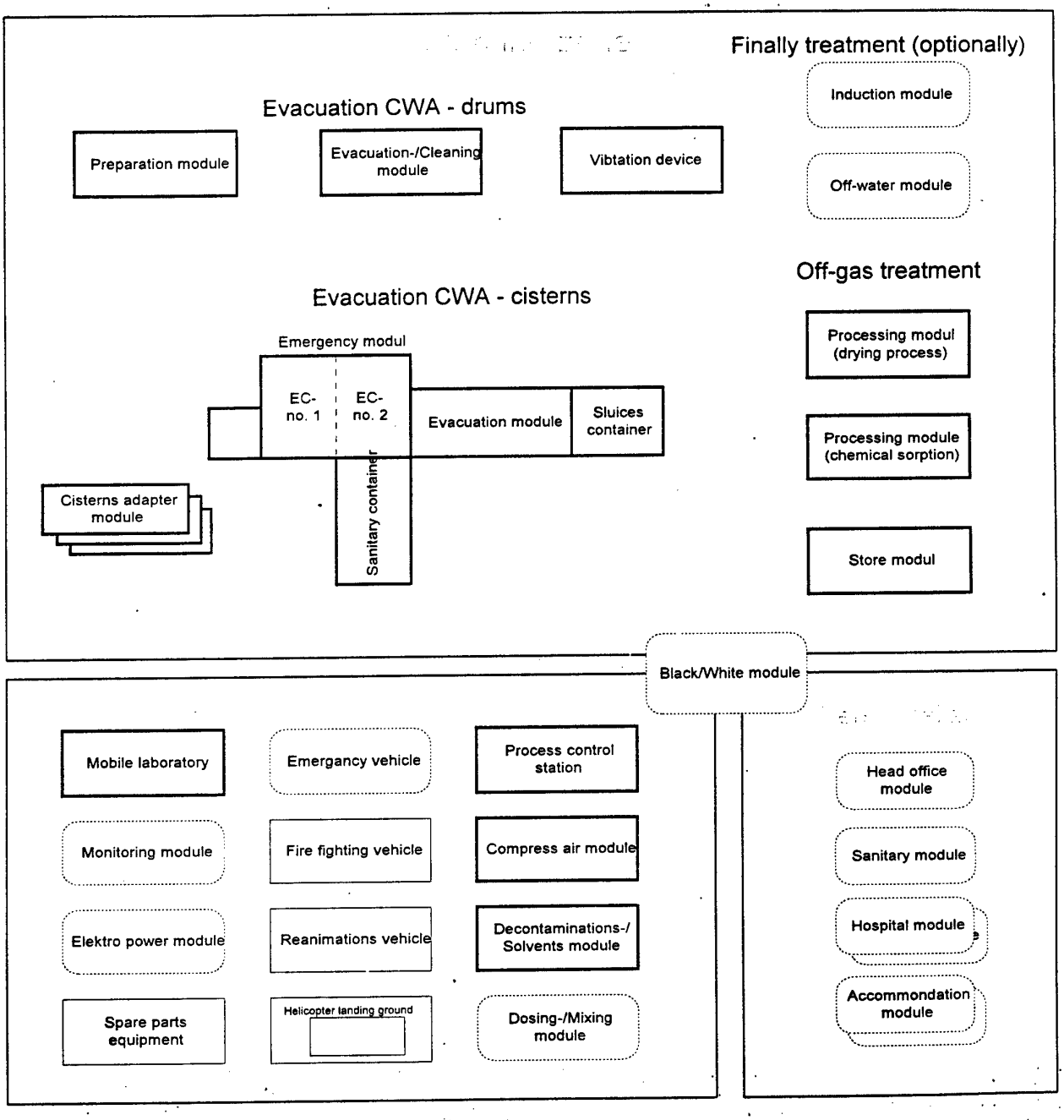
- Unrestricted ability to work at ambient temperatures from -5 .... +45 °C at 100 % air humidity; limited ability to work still from -25 °C to 6 °C as well as +46 ... +50 °C at a maximum of 80 % air humidity.
- Up to 6 exit lines which can be controlled separately for large-scale users (can be split up according to demand, at option), additionally one reserve or working line respectively.
- Safety cut-off of the output lines by means of a pressure control measuring process which was developed by our firm (called "smart") in the case of trouble in connection with outputs or the flexible lines respectively.
- Compressed air output lines with safety coupling, can be varied from DN 40 to DN 15, existing lengths of lines of compressed air safety hoses at 40 m each,
- Internal compressed air storage tank 2 m^3 with a permissible maximum internal pressure of 10 bar (absolute),
- Control via storage programmable controls built into the containers, with status and warning messages, with incorporation possible into existing Process Guiding Systems / Storage Programmable Control Systems via potential-free contacts or special bus connection, optional via radio guided remote control (as far as 1 km, as far as 10 km or via satellite).

Please feel free to contact us for any questions, queries, explanations and specifications you may have. Our specialists at the DR. KOEHLER GMBH Burg and the Stoffverwertungs- und Entsorgungsgesellschaft mbH Burg will be at your disposal.

Enclosures
Overview of the chemical-/physical dates from Lewisit, Yperit, there mixtures incl. pre- and crack products
mobile handling equipment in a CWA-Destruction Object (variant)
Container of Evacuation Complex

DR. KOEHLER GMBH

Mobile handling equipment in a CWA-Destruction Object (variant)



Explanation :

Special modules of the delivery 1994 - 97 into the RF

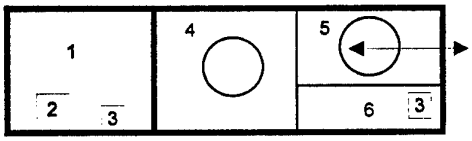
Special modules with optionally delivery

Equipment of the CWA-Destruction Object

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Container of Evacuation Complex

1. Evacuation - container (20'-ISO) with adapted channel system (10'-ISO)



1 Channel section
2 Shower section
3 Filter ventilation equipment
4 Filling section, 1,2 m^3
5 Transfilling section, with automatally transport system and scales equipment
6 Control section

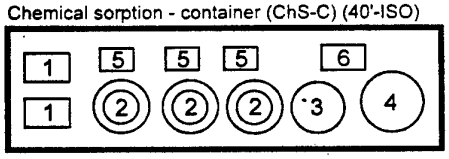
2. Processing - container (drying process) (30'-ISO)



1 Channel section
2 Shower section
3 Filter ventilation equipment
4 Active carbon filter section
2 x 6 Filter rows á 300 m^3/h
5 Compressor section, Gas compressor
2 x 300 m^3/h á Δ P + 650 mbar
6 Drying, cooling and control equipment

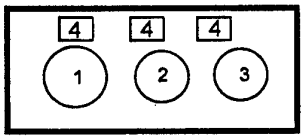
3. Processing complex (chemical sorbtion processing)

Chemical sorption - container (ChS-C) (40'-ISO)



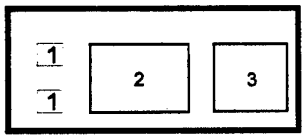
1 Roots-Compressor, 2 x 300 m^3/h á Δ P + 650 mbar
2 Chemical sorption unit, 3 x
3 Gas store, 1,5 m^3
4 Collecting store for hazardous liquids, 2 m^3
5 Air compress special pump, DN 25, 6 m^3/h
6 Air compress special pump, DN 50, 12 m^3/h

Store - container (St-C) (20'-ISO)



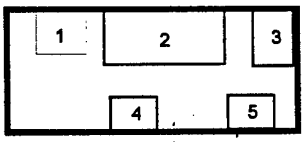
1 Store for sorption and regeneration liquids, 2 m^3
2 Store (bas./solv. liquids), 1 m^3
3 Store (acid liquids), 1 m^3
4 Air compress special pump, DN 25, 6 m^3/h

4. Decontamination/Solvents (DS) - container (20'-ISO)



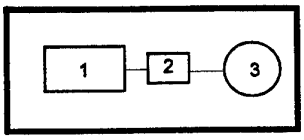
1 Compress air special pump, 6 m^3/h
2 DS store, 3,5 m^3
3 DS store, 2,5 m^3

5. Process control system - container (20-ISO)



1 Video centre
2 Process control system
3 Fire alert centre
4 Air conditioning system
5 Filter ventilation equipment

6. Compress air -container (20'-ISO)



1 Air compressor, effective power 625 Nm^3/h by 8 bar
2 compress air processing, -25 °C - +50 °C and up to 95 % relative air moisture
3 compress air store, 2 m3, 10 bar

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Execution: DR. KOEHLER GMBH Bereich CW-Destruktion In der alten Kaserne 10 39288 BURG Tel. 03921-919-60 Fax 03921-984516	Projekt: CW-Handling
	Datum: 25.09.96
	Projektbearbeiter: Ing. Jens Petersen

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STUDIES RELATED TO THE SMALL SCALE DEMILITARIZATION OF CWA

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Abstract

There is a need in the US to provide a capability for the rapid disassembly and decontamination of chemical munitions at locations other than those with permanent disposal processing facilities. The proposed reactor for chemical detoxification of the agents in these munitions would utilize a water/monoethanolamine reaction medium for G-agents and HD and a water/NaOH/monoethanolamine system for VX. A series of detoxification reactions were conducted in a 5 L glass reactor to provide thermal data on these reactions, optimize analytical procedures for product identification and to prepare sufficient reaction product to conduct of thorough toxicity tests.

Introduction

An significant concern in the chemical weapons disposal program has been the disposal of small quantities of chemical weapons and materials which may be located outside of the normal storage depots or which may, as yet, lie undiscovered in testing sites. Transport of these materials to a large reactor facility would be dangerous and inefficient. These materials must be safely destroyed on site and it is proposed that this be accomplished by using a truck transportable reactor system. Such a completely self-contained system would consist of a chemical reactor and accompanying equipment for handling the munitions, monitoring the process and testing the final product for completeness of agent destruction.

An important consideration in the design of this system is the chemical process to be used in the agent destruction. Several factors are important. The reactions must be sufficiently rapid to allow the processing of each batch to be completed in a reasonable working day. The process must be able to be safely run in a simple metal reactor with temperature control and stirring. The process should not require temperatures in excess of 60 °C and should be done at atmospheric pressure. Finally, it would be a significant advantage if the processes for all target CW agents utilized the same basic chemicals and similar reaction conditions.

It has been known for many years at ERDEC that monoethanolamine (MEA) reacts with G-agents and HD and to some extent with VX. Studies were thus undertaken to determine the conditions by which this reagent could be used as the basis of a small-scale detoxification process. It was determined that VX could be destroyed in a water/MEA mixture if NaOH was added. Thus, a basis was found to operate a detoxification process with only two reactants having to be carried to the site (MEA and NaOH).

In the studies reported here, the reactions were scaled up to approximately 4-liters. The purpose was threefold: to verify that the processes could be readily controlled in larger scale reactors, to evaluate the effects of varying rates of agent addition and heating to the overall process and to prepare sufficient reaction product for the battery of toxicological testing which will be required to proceed to the construction of a prototype system.

Experimental

Equipment: A Kontes 5 L cylindrical jacketed glass reactor with baffle cage was fitted with reflux water condenser, inlet tube, type-J thermocouple probe and Lightnin' Labmaster electronically controlled mechanical stirrer driving a Lightnin' A-320 three-blade impeller (figure 1). The thermocouple was connected to a Cole-Palmer digital thermometer which was, in turn, connected via an RS-232 port to an Apple Macintosh PC. The output of the thermocouple was calibrated to 0.1 °C against an NIST-traceable mercury thermometer. Agent delivery was effected via a PTFE diaphragm pump plumbed with 1/8" PTFE tubing. The pump had a variable speed control but delivery flow rates were not calibrated. All reported weights were measured on a calibrated Mettler top loading electronic balance readable to 0.001 g.

Materials: All agents were supplied by the Chemical Transfer Facility, ERDEC and were munitions-grade. MEA was obtained from Aldrich Chem. Co., stated purity of 99+%. NaOH: 50% w/w aqueous solution obtained from Aldrich Chemical Co. Methylene chloride and hexane were chromatography grade.

General procedure: The reagents required for each run were prepared by weight immediately prior to the run. The reaction mixture was added to the reactor, the stirrer started and, were necessary, the temperature allowed to equilibrate. The temperature record was initiated at this point. At the appropriate time, the agent delivery was

begun with time = 0 taken at the point when agent was seen to emerge from the end of the delivery tube. In all cases, after agent addition, the pump and tubing were rinsed with a small portion of reactant or solvent to clear the agent from the system.

All samples were taken from the bottom drain port of the reactor, collected in tared containers and the sample weights recorded. Extractions were performed by transferring a known volume of the sample to a measured amount of solvent in an extraction vial. Hexane was used to extract HD and methylene chloride was used for the G-agents and VX. The extraction vials were agitated in a ultrasonic bath for a minimum of 15 min.

Once the reaction was complete, the product was drained into tared containers and stored at ambient temperature for later toxicity testing.

Analysis: Reaction mixture extracts were analyzed by gc/ms for residual agent by the Analytical Chemistry Team, ERDEC. Reactor samples from G-agent and VX runs were tested for ChE inhibition using standard methods. Reactor samples from HD runs were tested for the presence of residual alkylating agent using the DB-3 method.

Results

G-agents: GA, GB, GD and GF were reacted with water/MEA in the ratios of 1640 g water and 2000 g MEA reacting with 365 g of agent. As the temperature profiles (figures 2-5) illustrate, the reactions were essentially identical. The agent dissolved readily in the water/MEA and an exothermic reaction commenced immediately. Once the initial exothermic reactions had ceased, heat was applied to destroy any residual agent. ChE and chromatographic analyses confirmed that the level of agent after the heating was sufficiently low to allow removal of the product from agent containment facilities. In all cases, the G-agents were >99.999% destroyed prior to the heating of the reaction mass. The heating is used only to provide extra assurance that the process has gone to completion.

VX: The lower reactivity of VX toward nucleophilic displacement made it necessary to add sodium hydroxide to the water/MEA reactant. The optimal composition was 600 g of 50% w/w aqueous NaOH, 3600 g of MEA and 400 g of VX. Even with the incorporation of NaOH, the reaction of VX was slower than the G-agents (figure 6). The VX is not miscible in this mixture so the early stages of the reaction occur in a two-phase system where effective stirring is essential. Approximately 95% of the VX was destroyed before the heating phase but, unlike the G-agents, heating was essential to destroy the last traces of agent in this system. The final product was a single phase liquid. No cleavage of the phosphorus-oxygen bond of VX was detected.

HD: The reaction of HD with MEA has been studied extensively at ERDEC and is mush more complex that those of the organophosphorus agents. The principal step in the HD/MEA reaction is alkylation of the MEA amine group by HD. This is followed by a second alkylation reaction which may involve a second MEA molecule to produce a linear product or may involve alkylation of the same MEA to produce a 1,4-thiomorpholine ring (figure 7). This latter pathway was by far the dominant route, accounting for 85-95% of the total HD added. The reactions were conducted using 300 g water and 3000 g MEA for 360 g HD. Although the rate of the HD reaction at ambient temperature is quite slow, sufficient heat is produced to increase the temperature in the reactor with no bath fluid circulating (figure 8). When heat is applied via the reactor jacket after two hours, the reaction rate and accompanying heat produced result in a temperature spike which slightly exceeds the maximum desired for the reactor. If, however, the reaction is allowed to proceed for an additional 30 min, the temperature spike is much reduced. If the reaction is allowed to proceed for about three hours, no temperature spike is observed at all.

Conclusions

MEA based reaction systems can be successfully used to destroy the common CW agents relatively quickly and at moderate temperatures. Thus, their use in a small-scale detoxification system is quite attractive. The studies performed here indicate that in actual practice, controlled agent addition, stirring and reactor heat loss will substantially influence reactor performance and may also have safety implications. These considerations must be addressed in the next phase of this program which will involve larger all metal reactors. They do not, however, represent any unusual engineering challenges and it appears that this chemistry can be readily adapted to the proposed purpose.

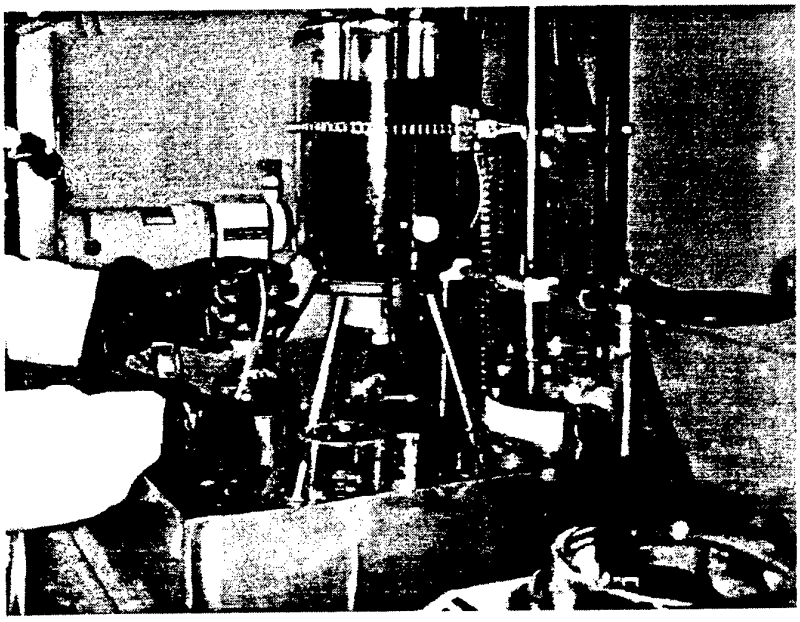


Figure 1. The 5 L jacketed glass reactor and agent delivery pump used in these studies.

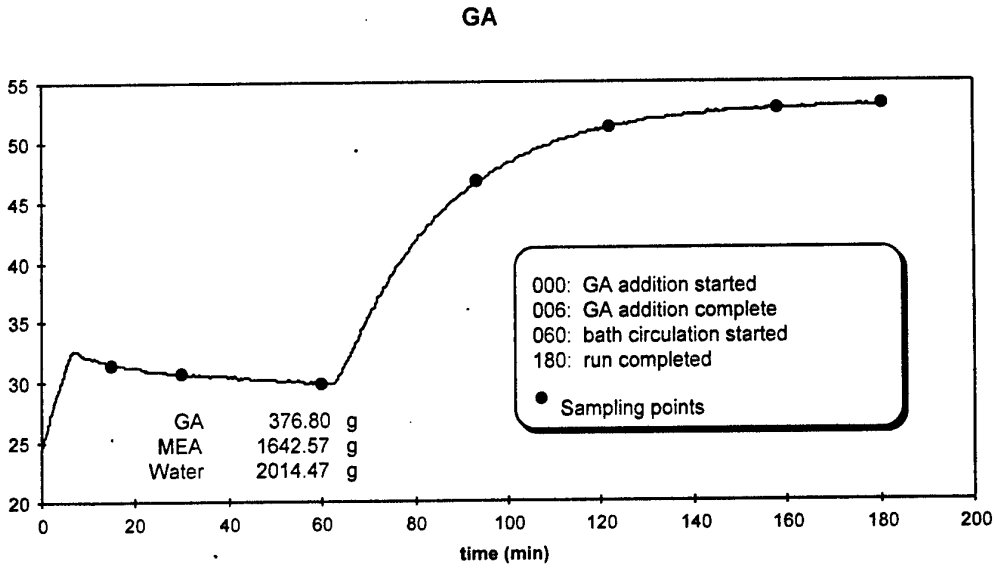


Figure 2. Typical temperature profile for the reaction of GA with aqueous MEA.

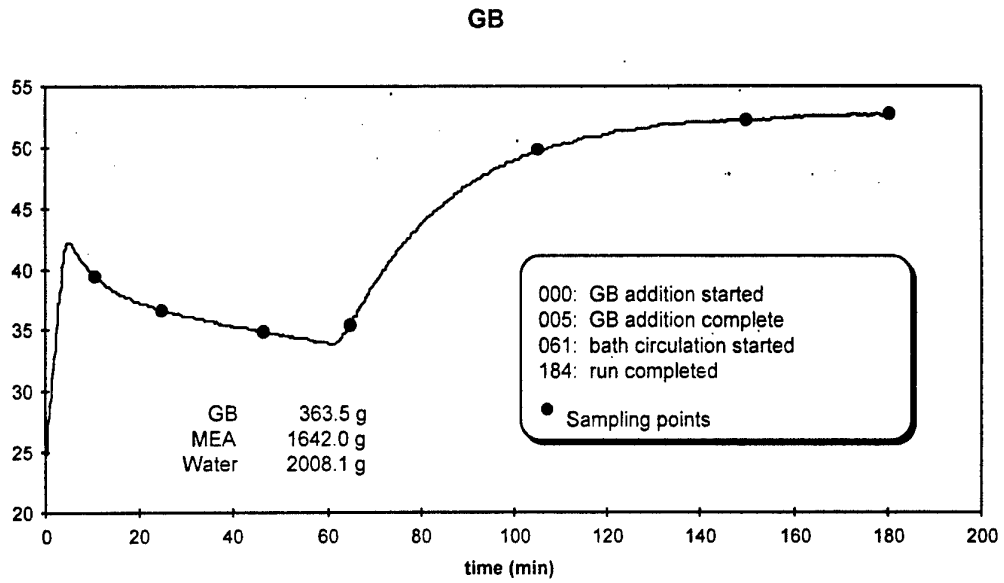


Figure 3. Typical temperature profile for the reaction of GB with aqueous MEA.

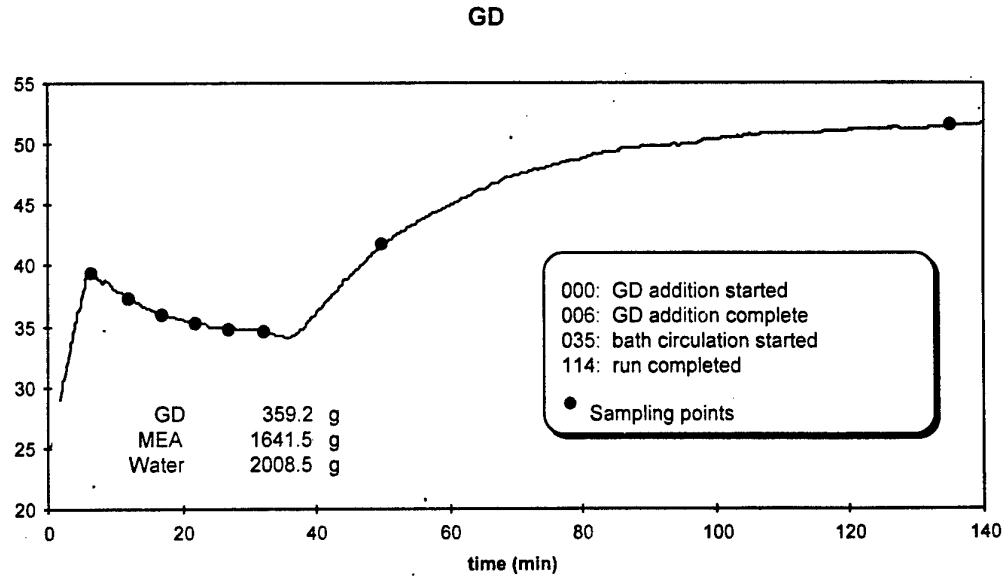


Figure 4. Typical temperature profile for the reaction of GD with aqueous MEA.

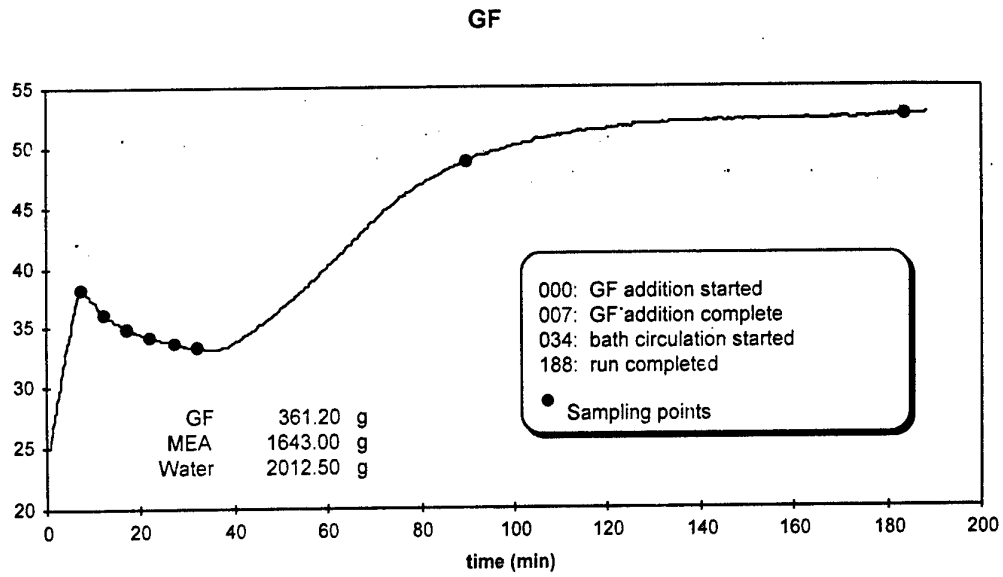


Figure 5. Typical temperature profile for the reaction of GF with aqueous MEA.

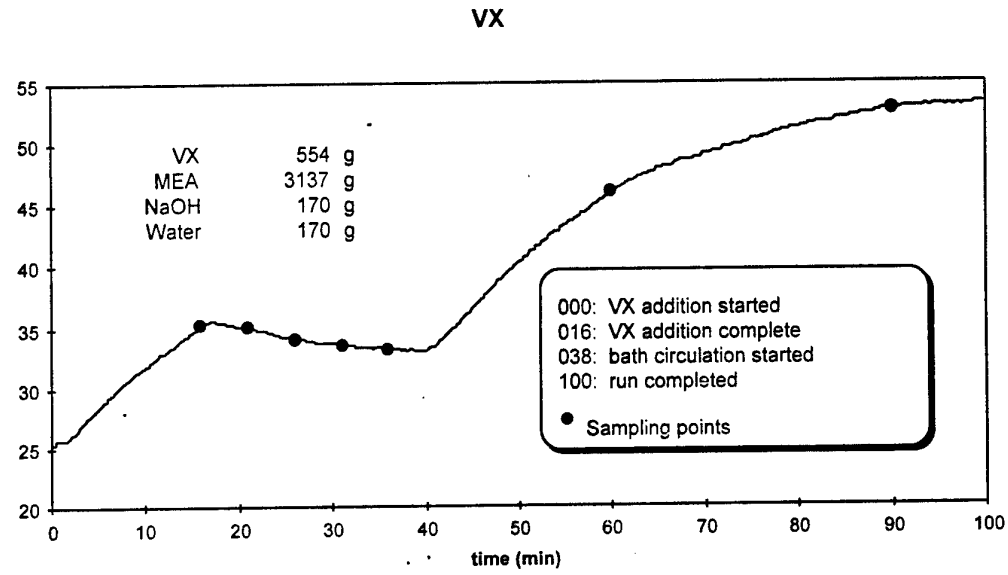


Figure 6. Typical temperature profile for the reaction of VX with aqueous NaOH/MEA.

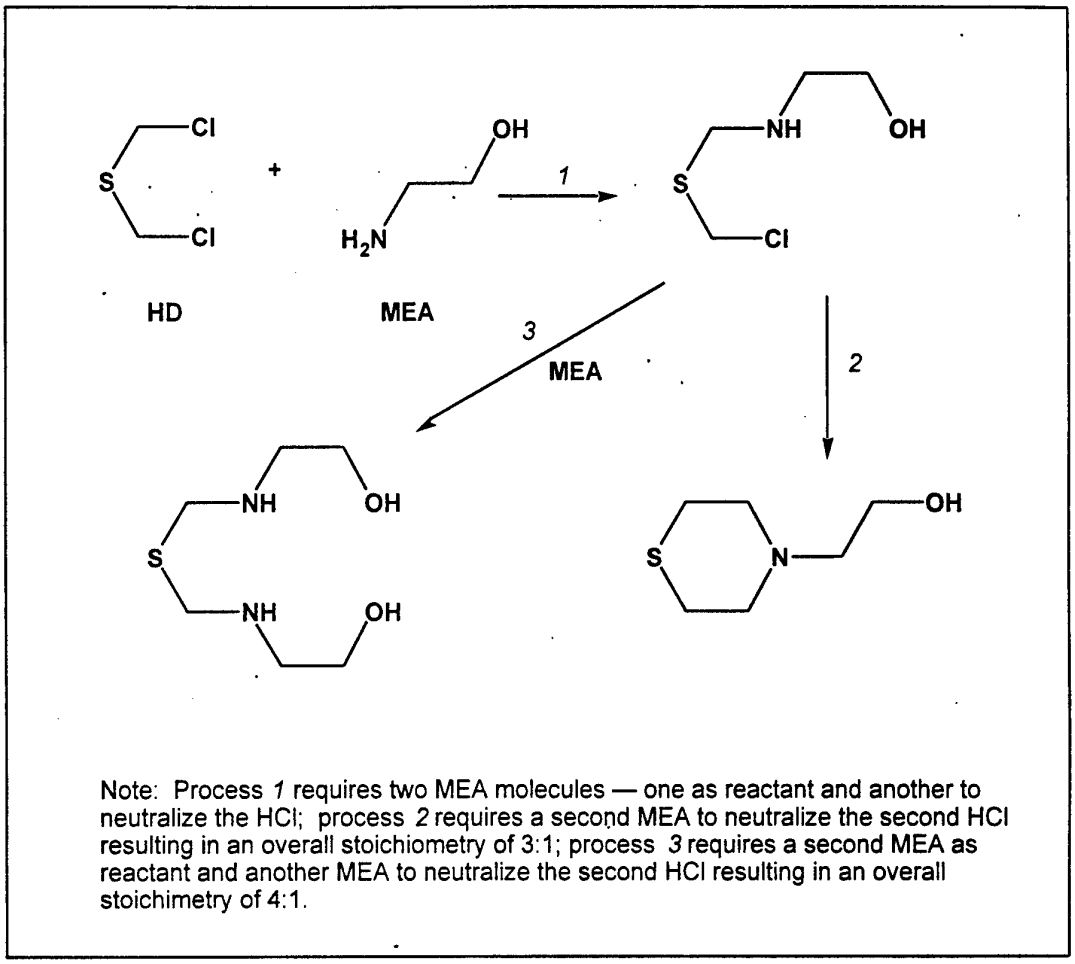


Figure 7. Reaction pathways for the reaction of HD with MEA.

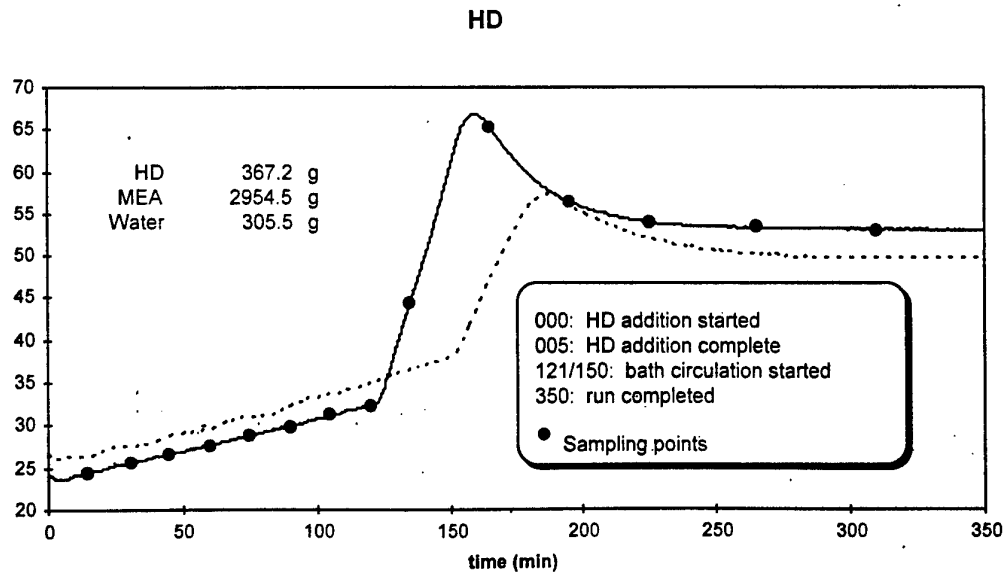


Figure 8. Temperature profile for the reaction of HD with aqueous MEA; heating applied after 120 min. (solid line) and 150 min (broken line).

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DESTRUCTION OF CHEMICAL WARFARE AGENT SIMULANTS IN CORONA DISCHARGE

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Introduction

The program of destruction and disposal of chemical warfare agents encounters growing public resistance. The main concern is the safety of the destruction/disposal technologies. Public opposition is based on a variety of reasons, among which are concerns about preparedness of workers and surrounding populations to accidental releases of chemical agents, release of unburned agent into the environment under normal operations, release of products of incomplete combustion or neutralization and highly toxic byproducts, and uncertainties on the long-term effects of trace amounts of chemical agents on human populations.

Chemical agent destruction processes such as incineration or neutralization, produce gaseous emissions containing trace amounts of residual agent or non-agent materials. These non-agent materials include the intermediates and byproducts of the process (such as the products of incomplete combustion, PICs, in incineration) and other hazardous substances like dioxins and furans [1]. Both the residual agent and non-agent hazardous substances have the potential of causing chronic health effects and are therefore causes of concern to the public. These concerns, while justified, can delay the implementation of disarmament programs.

By eliminating trace amounts of agent and byproducts in discharges from treatment processes, public concerns can be reduced and the process of destruction of chemical warfare can proceed more rapidly and efficiently. Similar problems exist in storage of aging chemical munitions, where leaking agents are disposed into atmosphere through ventilation. A low capital and operational cost technology capable of deep agent destruction and efficient operation at low agent concentration is therefore highly desirable as a technology which is "polishing", complementary to the baseline technologies of incineration and neutralization.

Recently, corona discharge emerged as a promising technology for treatment of low concentration stack emissions. Initial research on the decontamination of organophosphorus compounds was performed in the mid-80s [2-5]. During the last years significant progress was achieved in understanding of corona discharge plasma in application for conversion of volatile organic compounds (VOCs) and oxides of nitrogen and sulfur (NOx and SOx) [6-10]. However, the research on the kinetics, products and the mechanism of the destruction of actual Chemical Warfare Agents as well as their surrogates is still very limited [2-5].

In [5], the decomposition of dimethyl methyl-phosphonate (DMMP) diluted in helium at atmospheric pressure in alternating current capacitance coupled discharge was studied as a function of flowrate and concentration. Trace oxygen was found to improve the destruction. No quantitative data on the degree of the destruction as well as on the energy requirements were obtained in these works.

In this paper, an experimental study on the destruction of DIMP in dielectric barrier corona discharge was performed. The efficiency of the destruction was determined as a function of the specific discharge energy deposition.

Experimental

A tubular dielectric barrier AC corona discharge reactor coupled to a mass-spectrometer was used to study the efficiency and the kinetics of DIMP destruction in air at concentrations of 150-300 ppm.

The experimental setup is shown in Fig.1.

The flow reactors used are glass tubes (length 105 cm, ID = 1 cm) with a thin Ni-Cr coaxial wire (0.43 mm in diameter)used as a high-voltage electrode. The second (grounded) electrode is aluminum foil wrapped outside the tube. A corona discharge is excited by application of high AC voltage (up to 20 kV, 50 Hz) to the central wire. This arrangement results in an uniform discharge along the length of the reactor. The discharge power is varied by varying the input voltage of a high voltage transformer over the range 0 - 90 W. The reactant (DIMP) and the products of the destruction are monitored by a molecular beam sampling quadrupole mass spectrometer (MBMS).

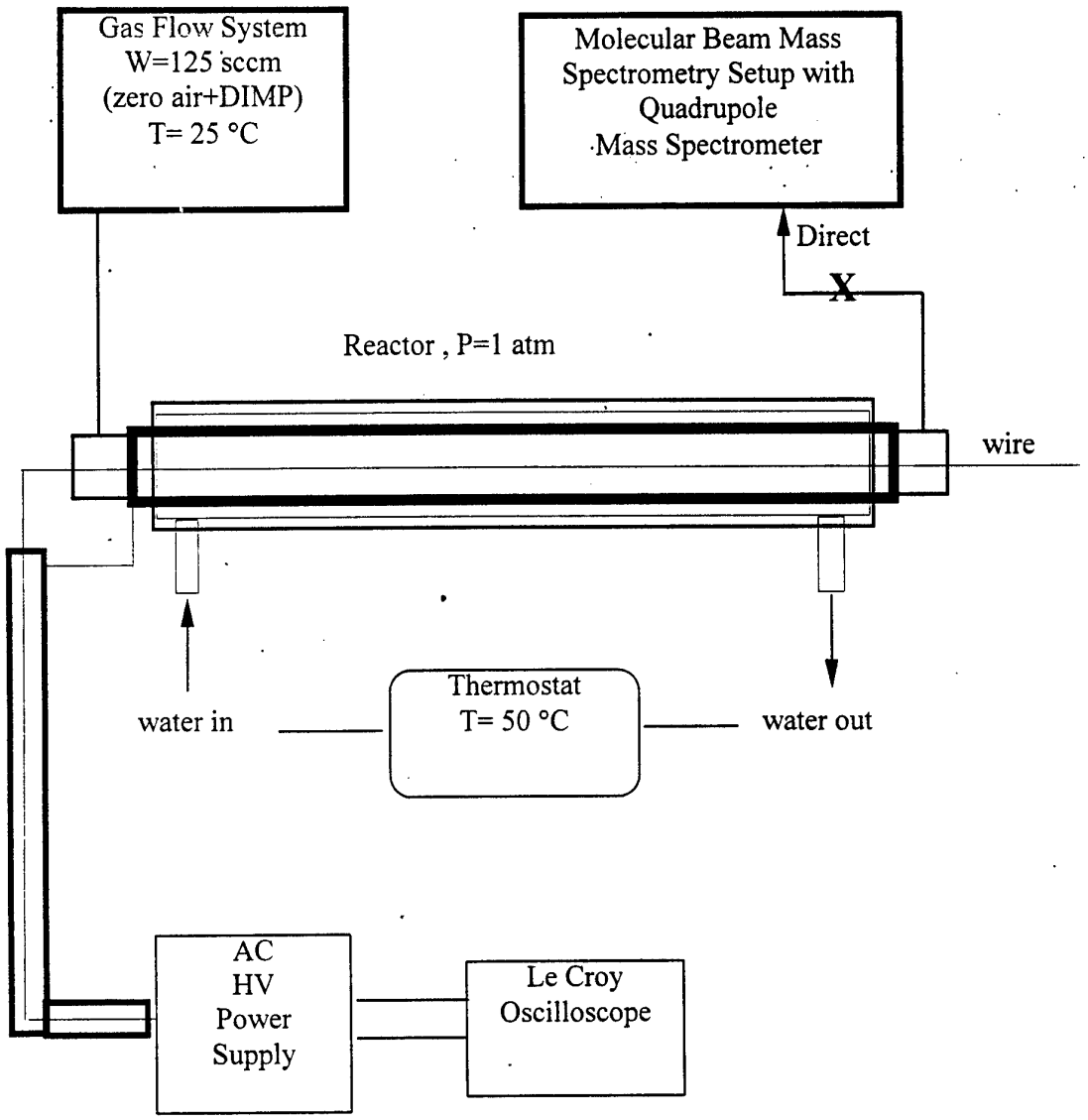


Fig.1 Experimental setup

The efficiency of the destruction was studied as a function of the active power dissipated in the reactor, the contact time, the flow rate and the concentration of organophosphorous compounds.

Active power determinations were based on the current-voltage measurements. The output voltage of the high voltage transformer is measured using a two-resistor voltage divider The discharge current is monitored by measuring the voltage across a resistor connected in series with the output grounded wire. Voltage and current oscillograms are recorded using a digital oscilloscope (Le Croy 9310AL), multiplied and averaged to produce the average value of the active power. The parasitic losses in the high-voltage transformer were determined with disconnected reactor. These losses were subtracted from the active power measured with connected reactor.

Mixtures of diisopropil methilphosphonate (DIMP) with oxygen were fed into the reactor at pressure 1 atm. DIMP concentration in the mixture was kept low (150-300 ppm). The typical mixture flow rate was 0.6 sccs (standard cubic centimeters per second). The mixtures were prepared in flow by bubbling oxygen through an thermostated impinger containing DIMP at 25 C. The reactor temperature was sustained at 75 C to prevent condensation of DIMP (vapor pressure is 3 Torr at 66 C). No thermal decomposition of DIMP is anticipated at this temperature. After passing the reactor the mixture is analyzed using molecular beam sampling with a quadrupole filter analyzer.

Results

To determine the extent of the destruction, the mass-spectra peaks of DIMP were recorded with and without the discharge. Neither CO and CO2 nor other peaks of the main destruction products observed previously when studying DIMP destruction in flames were not found in the current study. Production of ozone is observed.

The extent of the destruction as a function of the discharge power is shown in Fig. 2. The destruction efficiency increases with absorbed power. The destruction follows the first-order kinetics with respect to the discharge power. The absence of the other detected peaks with measurable intensity indicates formation of non-volatile phosphorous containing compounds (such as orthophosphoric acid).

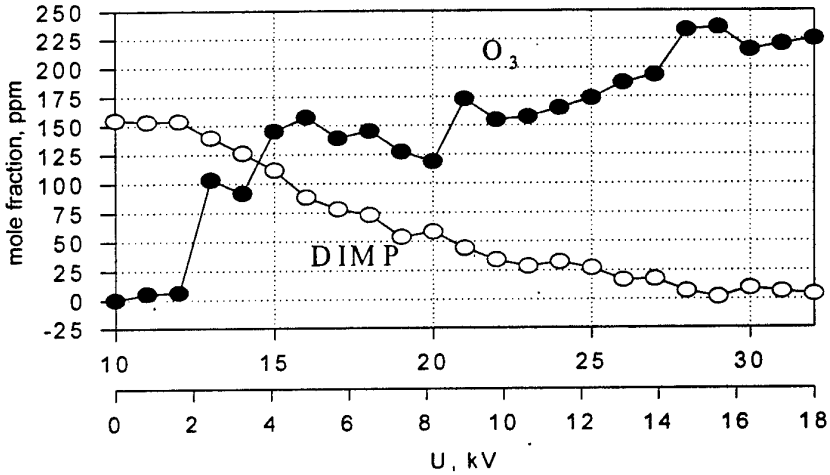


Fig.2 Profile DIMP and O_3 of the discharge voltage

The degree of DIMP destruction observed is ca. 98% at the power dissipated in the discharge of 5-7 W and the t flow rate of 0.6 sccs. This destruction efficiency correlates well with these established for a number of Volatile Organic Compounds and Nitrogen Oxides in the previous studies [10]. This indicates an universal mechanism of the destruction in corona discharge. This important result allows to expect similar energy consumption for the destruction of real CW agents. Also, it implies an (expected) increase of the destruction efficiency with the decrease of CW agent concentration, as was observed for hydrocarbons and nitrogen oxides.

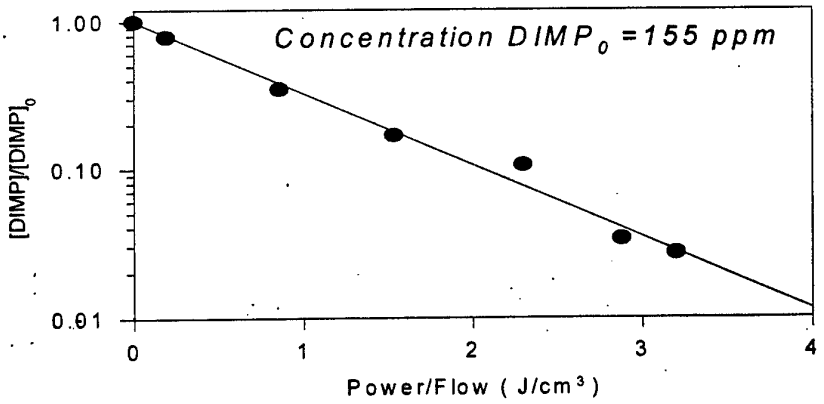


Fig. 3 DIMP destruction at the power dissipated

Acknowledgement

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PUBLIC OUTREACH: A KEY FACTOR IN CHEMICAL WEAPONS DESTRUCTION

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Public involvement does not ensure public acceptance, but it does give the community a participatory role and a sense of ownership in the decision-making process, even if the final decisions do not fully reflect community sentiments. Equally important, public involvement provides the army the opportunity to understand and address the concerns of the affected communities. ***(National Research Council, Washington, 1996)***

The origin of public opposition to national programmes of chemical weapons destruction

Opposition has successfully stalled or impeded chemical weapons destruction programmes both in the U.S. and Russia. In analysing the reasons, several factors and errors can be singled out, which lead to citizens opposing the destruction of their local chemical weapons stockpile, despite the obvious benefit that the weapons would stop to be a threat to the community:

- Credibility of the army is low.
- Citizens have not been informed on or involved in the technology selection process, despite the fact that citizens have displayed repeatedly high technical competence.
- Concerns of communities are treated inadequately. Risk, emergency preparedness, state of health, and infrastructure development are primary concerns which have to be addressed. Input by the public should be taken into consideration, feedback to the public must be assured.
- As the army is perceived as a single entity, work results and communicated content must be closely co-ordinated between the different departments. Contradicting information leads to confusion.
- Often, representatives of the army are unprepared and untrained in public outreach. Senior army managers must be involved to provide credibility.
- Sufficient resources must be allocated for public affairs programmes.
- Advocacy groups opposed to the national chemical weapons destruction programme tend to be well organised and vocal and to be able to engage local citizens in their activities.

Despite the distrust of people in the army, people want to be involved in decisions regarding destruction technology, monitoring operations, decommissioning. Many groups recognise that the ultimate decisions and responsibility are with the army, however they still believe there should be a substantial role for their input on matters that affect their health, safety and future well-being.

The way out of confrontation

An active approach is essential for increasing public understanding of the issues, identifying and clarifying areas of confusion and misinformation, and providing information necessary for citizens to make informed, independent assessments. In work with the public, three levels must be identified which reflect a continuum of increasing information exchange, mutual understanding, and public participation:

- **Public relation** is used to provide information to the public at large and to groups with specific interests about the scope, risks, and impact of the disposal programme. Public relations are largely passive efforts. Examples include brochures, posters, toll-free numbers, and videos.
- **Public outreach** is a proactive approach for determining specific concerns in the communities at each stockpile location and for providing information in direct response to the concerns of individuals and groups. Examples are information meetings, public forums, workshops, surveys, site tours, citizens advisory commissions, public outreach offices.
- **Public involvement** is the process by which public concerns are directly incorporated into governmental decision-making and by which people who have a stake in the outcome get a chance to influence its content before it is made. Public involvement is two-way communication with the goal of better decisions supported by the public.

Green Cross activities in public outreach and public relation

Green Cross is active since 1995 in public outreach activities in Russia and the U.S. It acts as a non-partisan mediator between army, ministries and population, similar to the Red Cross in armed conflicts. Green Cross has organised so far five public hearings:

- Saratov (Russia), October 1995.
- Izhevsk (Russia), May 1996.
- Washington (U.S.), July 1996.
- Indianapolis (U.S.), December 1996.
- Shchuch'ye (Russia), July 1997.

Two public outreach offices have been established in Kurgan and Shchuch'ye. The offices display information material on chemical weapons and the planned destruction facility in Shchuch'ye. Personnel at the office provide answers to questions of citizens. Two more outreach offices will be opened in April 98 in Penza (stockpile Leonidovka) and Izhevsk (stockpiles Kizner and Kambarka).

Green Cross published in Russia also several popular information booklets on chemical weapons, destruction technologies, risk, legal issues and medical aspects.

Green Cross projects complementing public outreach activities

Besides its core outreach activities, Green Cross facilitates chemical weapons destruction by running projects benefiting stockpile communities. The projects target the improvement of public health, emergency preparedness, and infrastructure. Examples in 1997 are:

- **In Russia:** Training programme for hospital personnel, authorities and population in stockpile areas in order to increase emergency preparedness; risk assessment of continued storage vs. destruction of the Shchuch'ye chemical weapons stockpile; health assessment in the Shchuch'ye region; investigation of an old chemical weapons dump site in Penza; NGO meeting in support of CWC ratification.
- **In the U.S.:** Co-ordination of a coalition of NGOs to support CWC ratification; workshops and roundtables on Capitol Hill to evaluate chemical weapons destruction technologies, conversion, arms control and non-proliferation policies, and environmental security; co-organiser of the Shchuch'ye hearing; Fourth Legacy Forum on Military Base Cleanup and Conversion; National Dialogue on Assembled Chemical Weapons Assessment.

The Green Cross Legacy Programme

- **Overall goal:** to overcome and prevent the long-term consequences of armed conflicts and related industrial catastrophes.
- **Programme strategy:** hands-on projects yield tangible improvements; strengthening of democratic structures and processes in order to secure results.
- **Subprogrammes:**
  - **Radleg:** collection and compilation of data on the environmental impact of the military use of nuclear materials in Russia.
  - **Chemtrust:** promotion of the responsible destruction of chemical weapons.
  - **Conweap:** military base clean-up and conversion.
  - **Socmed:** improvement of public health and local infrastructure of communities affected by chemical and nuclear problems.
- **Start of the Legacy Programme:** July 1994
- **Steering Committee:**
  - Roland Wiederkehr (MP, Programme Director, Executive Director Green Cross Switzerland)
  - Prof. Sergey Baranovsky (Vice-President Green Cross Russia)
  - Dr. Paul Walker (Director Legacy Programme Global Green USA)
  - Dr. Stephan Robinson (International Programme Co-ordinator)
- **Member organisations:** GC Estonia, GC Russia, GC Switzerland, Global Green USA.
- **Main network partners:** Swiss, U.S., Russian and Estonian governments; regional administrations in Kurgan, Izhvesk, Penza (Russia); State of Indiana (USA); Organisation for the Prohibition of Chemical Weapons (OPCW); Swiss and Swedish NC Defence Centre; Institutes for Social and Preventive Medicine in Bern and Basel (Switzerland).
- **Financial scope 1994-1997:** USD 3.09 million
- **Financial sources:** Swiss Ministry of Foreign Affairs, US Department of Defence, private foundations, and public donations.
- **More information:** Dr. Stephan Robinson, tel. +41-61-382 91 97, greencross@ubaclu.unibas.ch

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MODULAR DISPOSAL UNIT FOR EXPLOSIVES - MODEX

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1 Introduction

Explosives only have a restricted live time and must be disposed by time. Therefore disposal of explosives is a need for anyone who owns ammunition on stock or is faced with the problem of found ammunition. Open burning and open detonation of explosives from dismantled munitions and even complete munitions are two alternatives to solve the problem.
It is well know, that detonating and burning of ammunition causes severe damages to the environment especially to the air. A more financial aspect of this type of disposal is, that reuse of inert parts (aluminum, brass ...) is impossible.
Environment friendly disposal of explosives, adherence of German Clean Air Act regulations, easy operation, a wide range of operational limits and reuse of inert ammunition parts are the main advantages to be found in ***MODEX***.

MODEX (Modular Disposal Unit for Explosives) is a containerized disposal plant, developed by *E.S.T. - Entsorgungs- und Sanierungstechnik GmbH*, a subsidiary of *Dasa - Daimler-Benz Aerospace* in the *Daimler-Benz* group.
The plant can be designed in different ways, with different disposal capacities and for different customers needs. Modularity gives the possibility to solve customers problems by just choosing the adequate facility components as there are thermal inertization modules (rotary kiln -and/or batch incinerator), feeding systems and fluegas treatment.

E.S.T. itself, designed an built a stationary plant in Germany to dispose mainly the old ammunition of the former NVA (Nationale Volks Armee). The dismantled ammunition, the explosives and even complete small caliber ammunition are burned in the *E.S.T.* facility in Steinbach / Saxonia.
Furthermore *E.S.T.* has delivered a ***MODEX*** installation to a Spanish customer. This plant is in operation for disposal of residues from a demilitarization process.
This experience and also the experience from developing and assembling various types of ammunition, that Dasa owns for decades, gives the customer the certainty to receive a technology, well proven and at the highest standard available.

2 Preface

A modular facility for the demilitarization of ammunition, high explosives (H.E.), H.E. filled objects and hazardous items by making use of thermal inertization technology brings up the following aspects:

- Environmentally compatible disposal
- Use of state-of-the-art technologies
- High safety standards
- Use of local resources
- Use of existing infrastructure

The disposal is done under observation of the German clean-air-act according to BImSchV 17. Most of the actual values of the facility are well below the limits required in these regulations. This is guaranteed by the use of up to date technologies as described below.
Safe operation and protection of the environment are the most important criteria for the plant. For our understanding, this concerns safety of the personnel as well as protection of the facility.
To operate the demilitarization facility, a suitable infrastructure is required in which water, energy, fuel and compressed air supply is available as well as auxiliary buildings like offices, bunkers etc. Appropriate traffic requirements are necessary. The modular design of the facility permits provision of independent supply units to assure completely independent operation of the plant.

3 Operational

4 Plant Site Requirements

Besides the infrastructure requirements, the safety distances defined in the relevant national regulations for

plants in which storage, handling and/or work with hazardous materials (explosives, toxic materials) are accomplished, must be observed within the plant boundaries as well as in its vicinity. For this purpose, remote sites are preferred for the installation of such a plant. Furthermore, the transportation distances between the various modules of the plant should be as short as possible. The following four typical units of operation are defined:

- Operating unit 1 storage area, plant internal transportation
- Operating unit 2 disassembly (disintegration) / dismantling
- *Operating unit 3 incineration / disposal / fluegas treatment*
- Operation unit 4 auxiliary facilities

5 Operational Characteristics of UNIT 3

Capacity:	320 kg/hour material for disposal (GW)
Thereof:	30 kg explosives of max. energy content/hour (TNT_{equiv})
Personnel required :	3 employees/shift
Operation in partial workload:	feasible to a rate of > 20% capacity

6 Technical

Modular equipment

Unit 3 (***MODEX***) consists of several modules to accomplish different tasks:

1: Static Kiln (SK)
2: Chemical Reverser (CR)
3: Feeding and handling system (FS, HS)
4: Processwater-treatment (PWT)
5: Control center (CC)
6: Emission measurement

Static Kiln (SK)
The explosives and/or the munitions are heated indirectly in the batch incinerator. A detonative reaction is accepted if the amount of unconfined explosive is lower than 1,5 kg. The inner wall of the oven is designed to protect the walls from fragment impact. It can be renewed after longer use.
Removal of the particulated residues is done by rotating the oven. The fluegases are led to the high-temperature decomposition unit with fluegas purification (CR).

Chemical Reverser (CR)

The fluegases from the batch incinerator contain noxious substances which are treated in the high temperature decomposition unit. Total destruction of all noxious substances into thermodynamically stable small molecules results from the high temperature decomposition. This is assured by minimum temperatures of 2.000°C in the whole reaction chamber and a good mixture of the fluegases through appropriate construction. Oxidizing as well as reducing conditions of the reaction are adjustable by the wide control-range of the burners. The unit consists of identically built modules which can be connected or disconnected as required.
A multi-stage fluegas purification system is installed to minimize the burden for the environment by the fluegases created by the incineration of explosives, propellants, solid propellants, fuses, chemical agents and other materials. The main components are:

- Quencher to abruptly cool the fluegases (no de novo synthesis)
- Multiple stage washer to remove heavy metals and to neutralize the acid fluegas components
- Tank for the precipitation and removal of the resulting salts
- Catalytic oxidation unit to avoid emission of CO

The fluegases are abruptly cooled to about 70°C in a quencher which is directly connected to the reaction chamber. First neutralization of acid components is done using a caustic liquid (i.e. sodium hydroxide solution). The acid components are mainly halogenous substances like hydrochloric acid (HCl) and hydrofluoric acid (HF). The fluegas treatment system extracts heavy metals and the acid compounds like sulfur dioxide (SO_2), hydrochloric acid (HCl) and hydrofluoric acid (HF). After quenching, the fluegases enter the scrubbers. The

evolved wash water is to the largest amount lead in a circuit. A part of the spent water is removed with the precipitated salts and replaced by fresh water. The subsequent demisting process extracts fine solid and liquid particles from the fluegas.
The preferentially used reducing reaction conditions in the high temperature combustion system prevent the production of NO_X. But a catalytic post oxidation of CO to CO_2 is necessary. The activated charcoal filter mounted immediately before the stack serves as a safety filter.

Feeding and handling system (FS, HS)

The batch incinerator is used to demilitarize munitions which only can be disintegrated or for incineration of HE with high efforts (e.g. hand-grenades). Through a central obturation equipment with holding fixture, the material is filled remotely into the sphere shaped oven.
The feeding system is tailored for the respective task and represents the feeding interface for the various components of the facility. Interim store and batch material for disposal (high explosives, fuses, pyrotechnic substances, propellant powder, partially dismantled munitions etc.) are fed into the incinerator. Depending on the type of material under disposal, the feeding system determines whether operation is accomplished semi-automatically or in processes automated by dosing and weighing systems.

Processwater-treatment (PWT)

An auxiliary system for processwater recycling is installed to guarantee a safe, efficient and environmentally compatible operation of the demilitarization plant. The minimization of fresh water requirement is most important. The heat resulting from the process is converted into steam and used for heating. An utilization of the created energy e.g. for the supply of electricity makes no sense for the modular plant because of the additional cost. The waste-water system for further purification neutralizes all noxious substances in a five-step-process so that afterwards the waste-water can directly be led into the main-drain. The maximum amount of fresh water needed for the whole plant is less than 1 m^3/h.

Control center (CC)

The high-temperature combustion and the fluegas treatment are a fully automated and continuously running process. The facility is operated from a control center. Recorded measurement data are processed by a control system aided by a process computer. The central system also regulates and controls operation of the facility. The total process flow is documented continuously (measurement records) to confirm completion of the disposal.
Most different materials are handled in the disposal facility. Material residues and reaction products resulting from demilitarization are transferred to waste depots in accordance with the relevant declaration analysis and observing applicable acceptance criteria.

Emission measurement

The emission measurement and control offered is in accordance with the standards for incineration installations.
The following substances are measured and protocolled according to the German BImSchV17:

- Dust, NO_x, SO_2, HCl, CO, C_{total}, O_2

Set-up and operation

Due to the mobility and modularity of ***MODEX*** only little preparatory work is needed for infrastructure. E.g. only 250 m^2 of load-bearing surface is needed for the set-up of the plant. After the infrastructure being prepared, ***MODEX*** is delivered to the site via low-bed trailer. The modules can easily be lifted in their final position with a mobile crane. ***MODEX*** can be put into operation after all connections for the various supplies have been made and the installation has been tested in a first run.
MODEX is designed for a 3-shift operation with 2 employees per shift. ***MODEX*** can also be operated in 1 or 2-shift operation with holding the installation in a so called 'warm status'. Temperatures of SK and CR are automatically reduced resp. raised during the stand-by periods.
The personnel needed have to perform the following tasks:

- feeding and emptying of SK
- control center
- PWT operation
- packaging of scrap and other residues

Interested parties should address their questions to:

E.S.T. - Entsorgungs- und Sanierungstechnik GmbH
Mr. Schenkel
P.O. 1340
D - 86523 Schrobenhausen / GERMANY

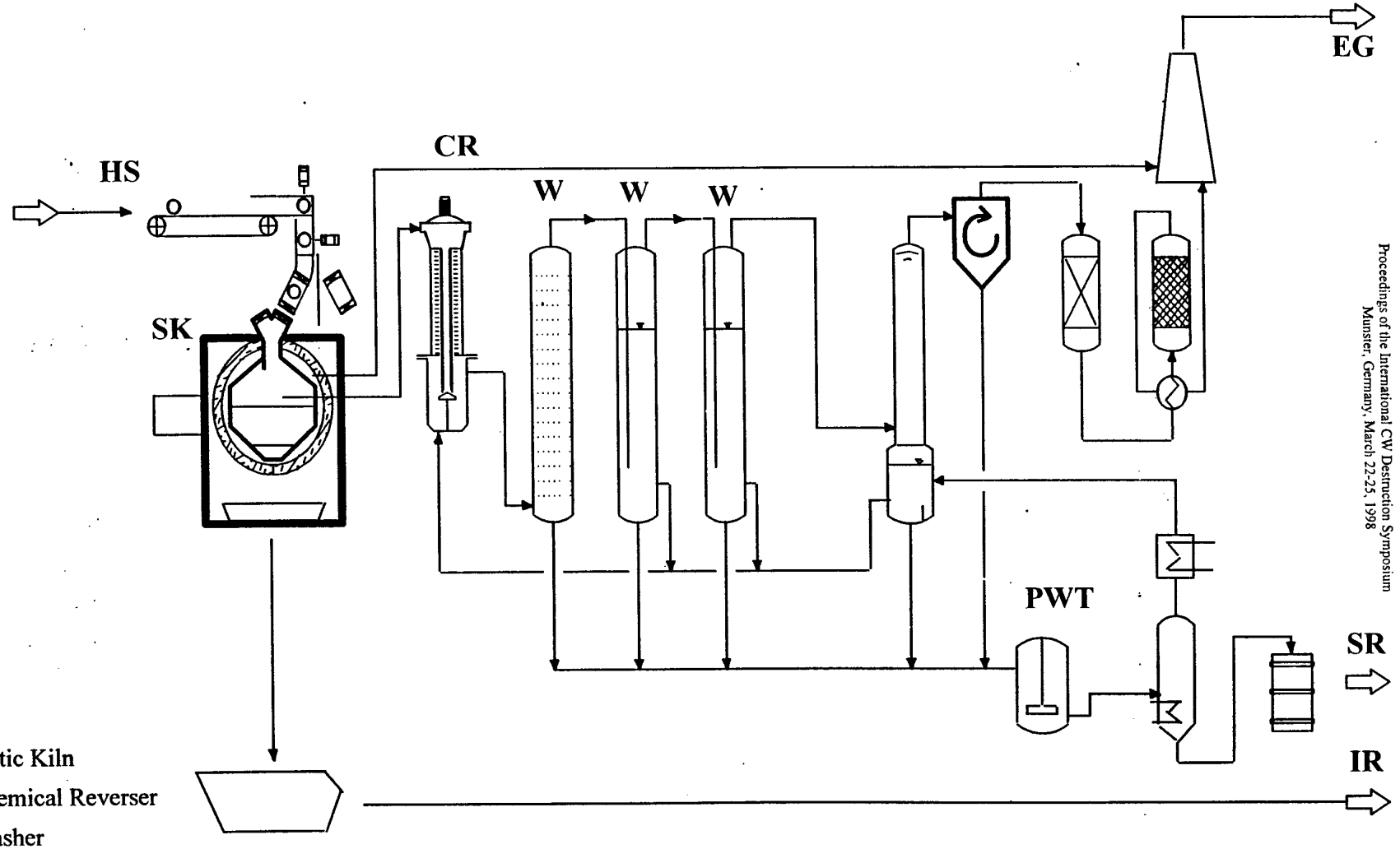
phone +49 (8252) 996 - 411
Fax +49 (8252) 996 - 827
email EST.MEKa@t-online.de

ANNEX

Slide 1 shows	the MODEX process in general
Slide 2 shows	the plant transportation and set-up
Slide 3 shows	the infrastructural needs and the erected plant
Slide 4 shows	the kiln
Slide 5 shows	some more pictures of the installed equipment
Slide 6 shows	the remnants after cleanburning

MODEX plant

Daimler-Benz Aerospace
E.S.T. Entsorgungs- und Sanierungstechnik GmbH



SK:. Static Kiln
CR:. Chemical Reverser
W:. Washer
PWT: Process Water Treatment
HS:. Handling System

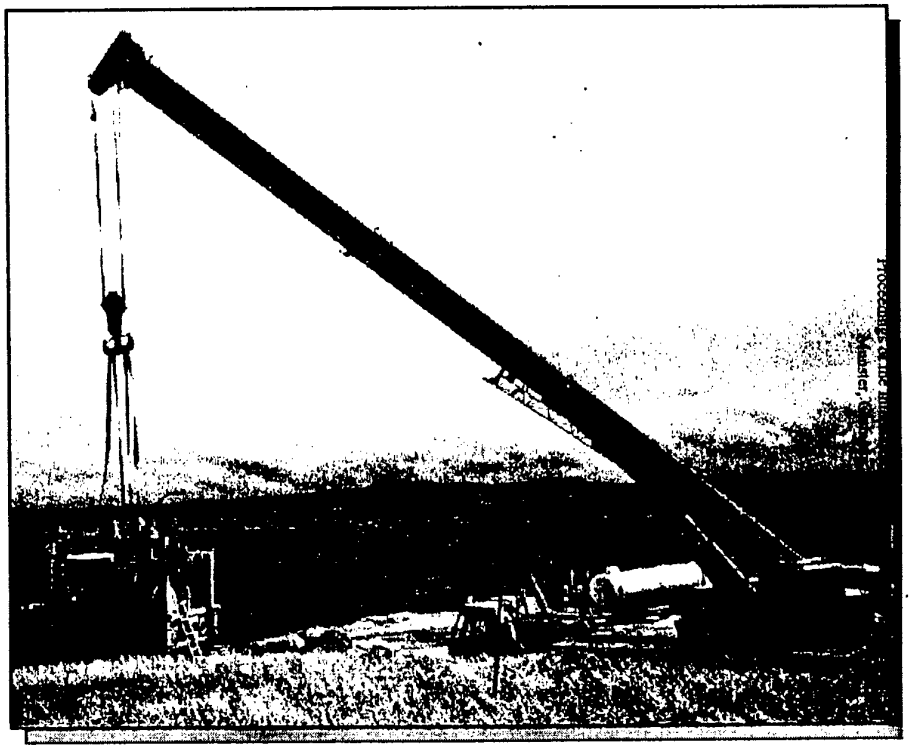
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MODEX plant

Daimler-Benz Aerospace
E.S.T. Entsorgungs- und Sanierungstechnik GmbH



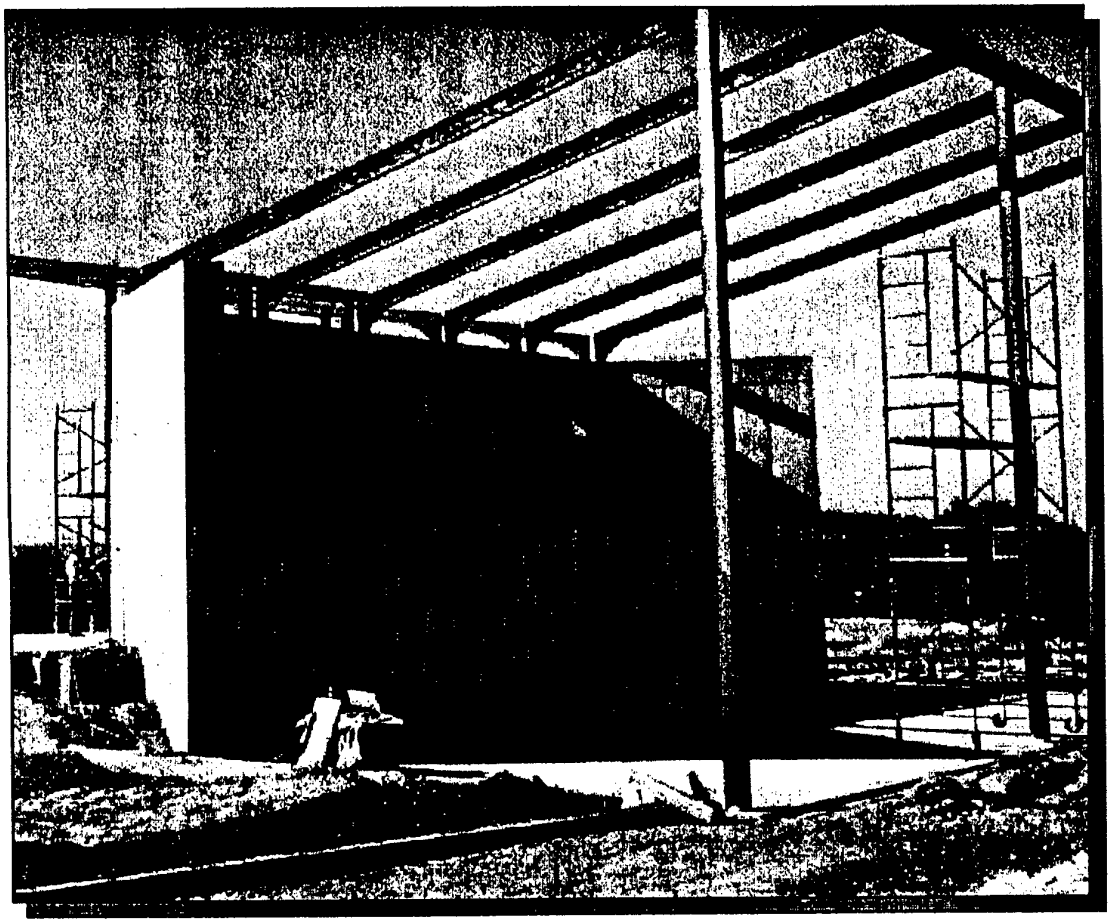
MODEX transportation after integration



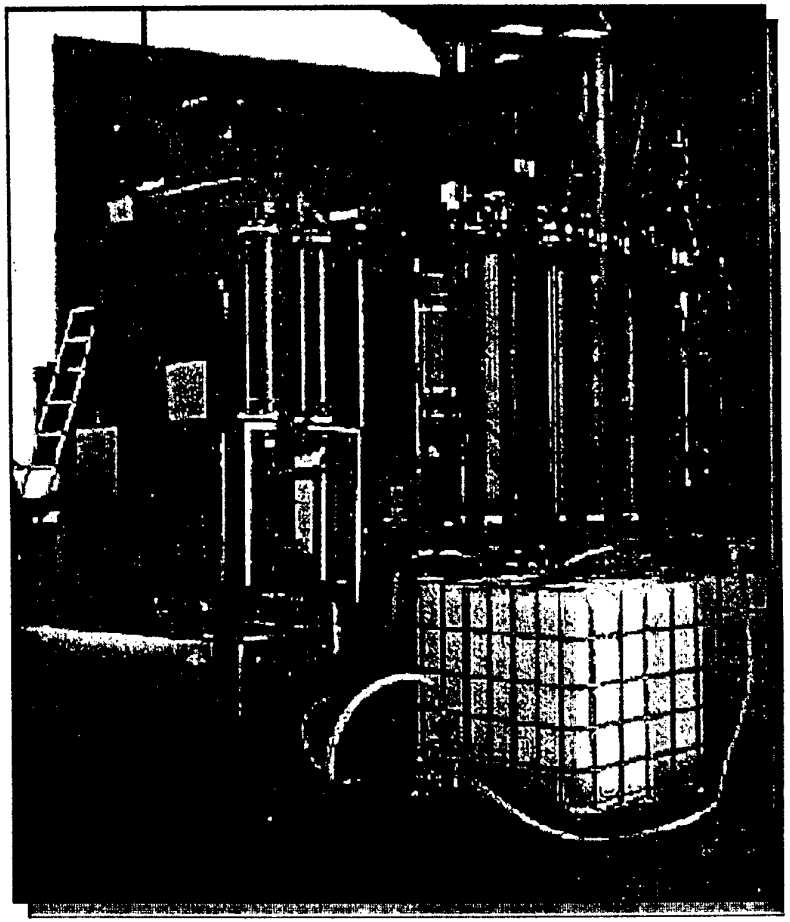
Static Kiln during unloading and positioning

MODEX plant

Daimler-Benz Aerospace
E.S.T. Entsorgungs- und Sanierungstechnik GmbH



Minimum preparatory construction work for MODEX



MODEX set-up after positioning

MODEX plant

Daimler-Benz Aerospace
E.S.T. Entsorgungs- und Sanierungstechnik GmbH



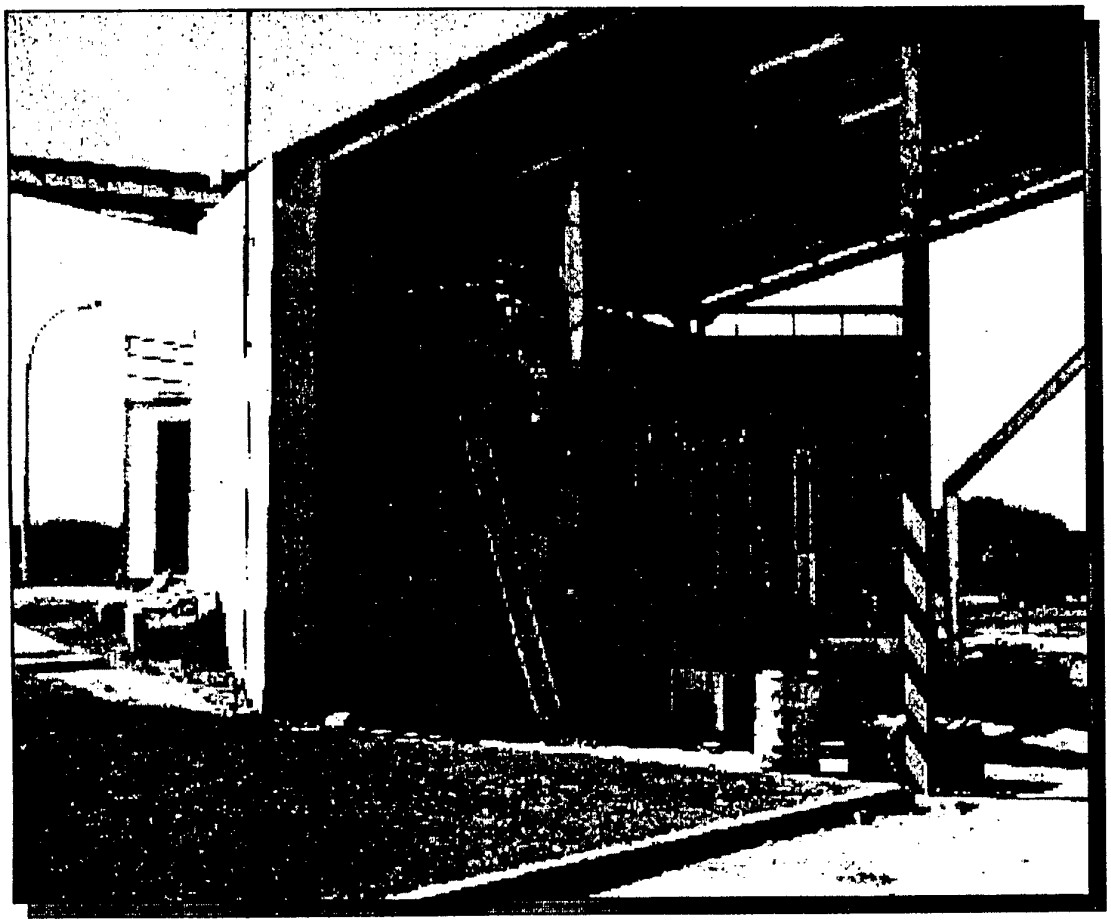
Static Kiln
and
feeding system



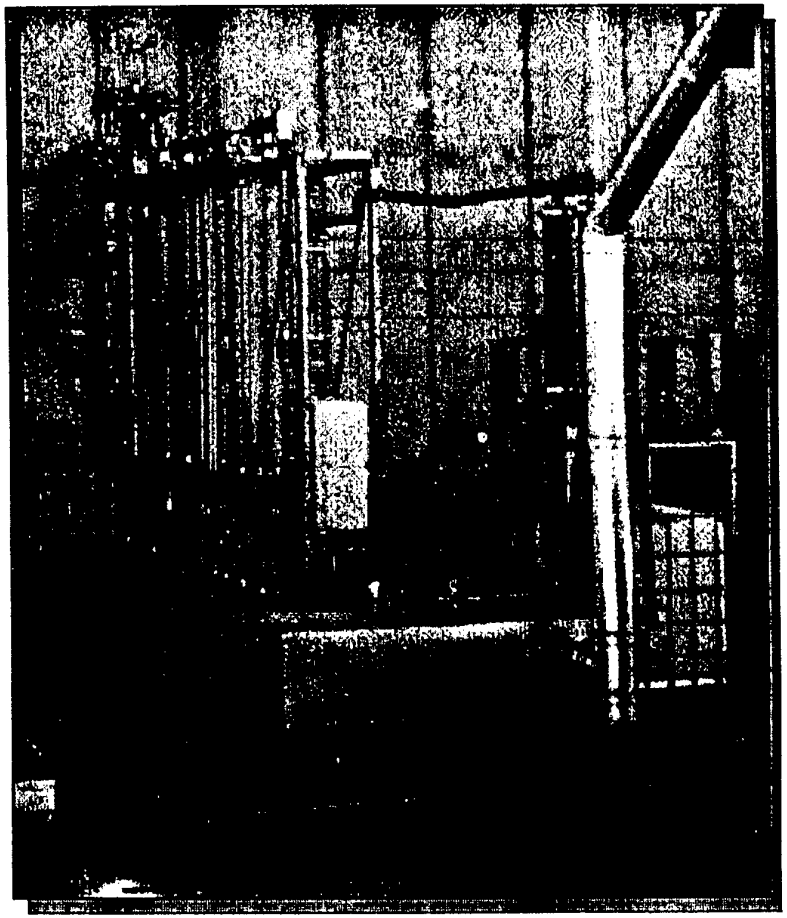
Static Kiln
and
scrap box
during turning for emptying

MODEX plant

Daimler-Benz Aerospace
E.S.T. Entsorgungs- und Sanierungstechnik GmbH



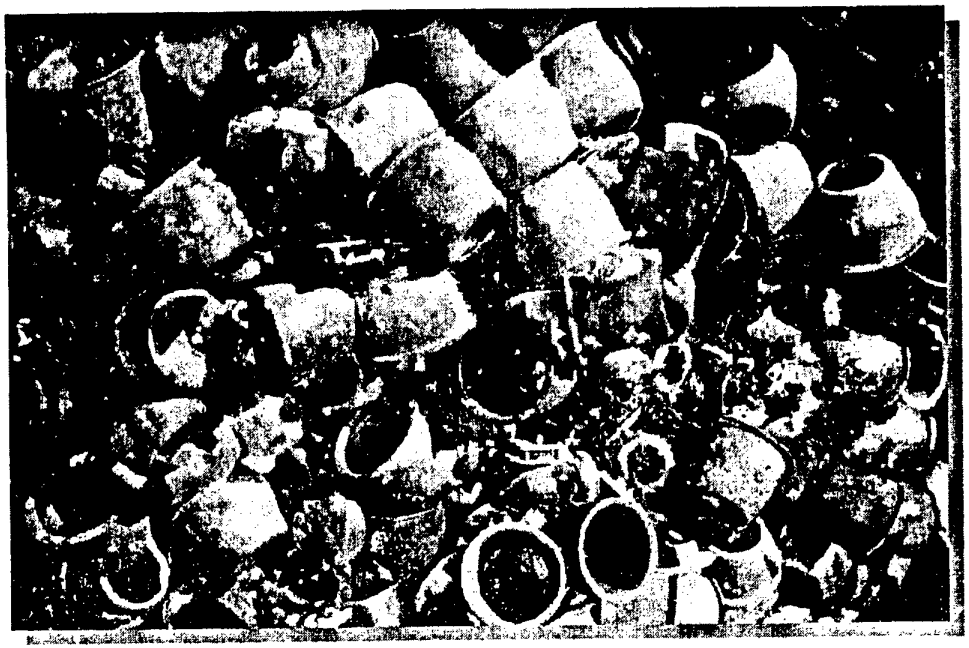
MODEX installed

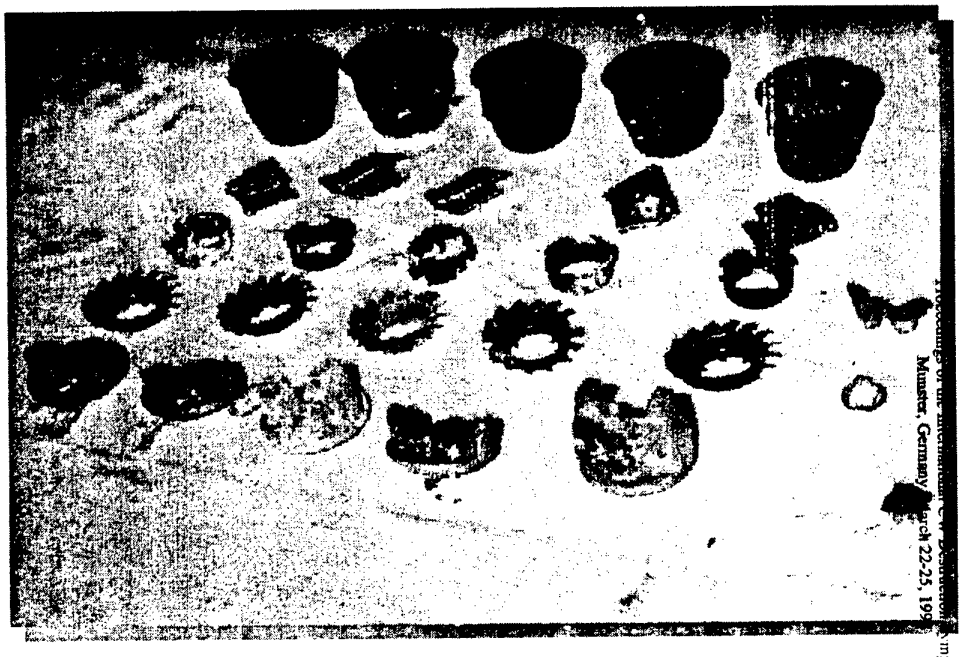


Chemical Reverser with catalytic converter, offgas pipe and processwater handling

MODEX plant

Daimler-Benz Aerospace
E.S.T. Entsorgungs- und Sanierungstechnik GmbH





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Scrap as it comes out of the Static Kiln after
- incineration
- clean burning
- emptying

Recovered ammuniton parts
- evidence of complete destruction
- evidence of complete incineration
- evidence of clean burning

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COLD COMBUSTION - A HOT TECHNOLOGY (OXIDATION OF CWA BY ACTIVATED OZONE STRUCTURES)

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Introduction

Beside the declarations of the CWC (Chemical Weapon Convention) the chemical weapons are representing a big problem for the people worldwide. The official and not official existence of such weapons and its chemical components is an extreme potential of danger not only as an agent itself but also in the contamination of the environment (seas, lakes, soil etc.).

Subject of this report should not be the judgement of other disposal engineering. The one and only task is to find solutions to solve the problems of CW.

In such a way, the first ideas of evolution were based on the demands:

- high efficacy
- plant mobility
- closed performing circuit
- energetically small consumption
- economical plant and costs

Some short words to the plant mobility. This demand results from the transportation problems. Transportation of such high-risk chemical warfare agents can not often be carried out and needs special conditions (very expensive!). The best solution is the local disposal.
As in the most things, the readiness and the price to be paid for it is not often together.

According to information of the International Conversion Center (BICC) in Bonn:
Up to now spent the USA more than three billions of dollars for the destruction of their C-weapons in this way, but nevertheless four percent of the inventory could be destroyedup to now.

The readiness of destruction of chemical arms also insists on the side of Russia but it is a question of the money there, too. A lot of helps from outside e.g. from the USA or Germany - even 500 million dollar and more - achieve not an end.

In Russia, considerations are made to retrieve specific components and arsenic from the chemical weapons.

Arsenic is extremely expensive (US$ ~ 2.000 - 3.000 per kilogram) on the international market and is required mostly in the fields of micro chip (microprocessor) and semiconductor technology. In times of economical crisis in Russia, the thought of earnings in hard currency makes a procedure of detachment and not destroying of arsenic components very attractive.

Out of our developments in 1996 and 1997 we tried to solve the above mentioned problem with success. It was possible to reach a plasma-supported precipitation to non-water-crystals without warfare character. This e.g. arsenic material is storable and is able to be manufactured for industrial purpose.

2. Theoretical Introduction into Activated Ozone Structures

The principle of ozone generation is to generate in a first step atomic oxyggen (O) from molecular oxygen (O_2) by dissociation which, in a second step, leads to the formation of ozone (O_3) by subsequent addition reaction. Allowing for the energies required for reaction, the chemical reactions in the simplest case by may described as follows:

$$O_2 \longrightarrow 2\,O - 5.116\ eV$$

$$O + O_2 \longrightarrow O_3 + 1.084\ eV$$

Hence follows the overall reaction:

$$^3/_2 O3 \longrightarrow O_3 - 1.474\ eV$$

From this a specific minimum demand for energy of 0.82 kWh/kg is calculated, leading to a maximum of theoretical ozone yield of 1,220 g/kWh. Commercially used ozone plants, however, under most favorable conditions achieve only an energy recovery of 14% compared to the theoretically possible ozone yield.
The ozone generators applied so far primarily aimed at the utilization of the ozone although it was known that ozone is highly toxic and has an half-life of more than 5 hours. Many applications utilize its toxic phase (for the killing of germs and viruses etc.) other applications, the oxidative phase (bleaching processes, industrial oxidation, etc.). Thus, technical improvements mainly aimed at increasing the ozone yield. The underlying problem is that the amount of energy needed is relatively high due to the fact that the major portion of the energy fed into discharge is lost for the ozone synthesis, and finally is converted to heat. Since the use of ozone in a wide range of applications turned out to be uneconomical - due to these losses, the long half-time-values required for obtaining O-radicals which are necessary for the oxidation processes and the subsequent destruction of residual ozone - it is rarely used these days compared to its manifold possible applications.
The major objective of this procedure and the test arrangement is to apply most recent scientific findings and perform the generation of ozone and ozone in its excited state. In the following, the various ozone structures are referred to as "activated ozone structures".

Since 1987 the generation of ozone has been described in detail as 2 basic reactions:

- the decay of the molecular oxygen in the basic state $0_2(^3\Sigma^-g)$
- the activation of molecular oxygen $[O_2(1\Delta g), O_2(^1\Sigma^+g), O_2(^3\Sigma^-g,v)]$

Thereby, the following activated ozone structures may develop:

- basic state
- excited state
- ionized state O_3^+ and O_3^-

Schematically, the above described process may be summarized as below proceeding from the basic state:

$$^3/_2 O_2 \longrightarrow O_2 + O \longrightarrow O_3 \text{ or } O_3^*$$

$$^3/_2 O_2 \longrightarrow {}^3/_2 O_2 \longrightarrow O_3 \text{ or } O_3^*$$

whereby O_3^- represents the activated ozone structures $[O_3(^3B_2). O_3(^1A_1,v), O_3(^1B_2)]$, O_3^+ and O_3^-.

The half-time-values of these activated ozone structures is said to range between 70 msec and 70 sec, which means that in a shorter period of time the number of radicals available for oxidation is considerable larger.

Activated ozone structures are produced by withdrawing the ozone from the discharge gap as quickly as possible following the dissociation of molecular oxygen (O_2) to atomic oxygen (O) and the subsequent formation of ozone. This impedes the repeated decay resulting from an electron collision in the discharge gap.
The ozone synthesis during an electric discharge basically needs a plasma which is characterized by a high temperature of electrons and a low temperature of gas. With the passage through the gas-permeable electrode, the essential physical conditions for the plasma ensuring optimal reaction phases are met, namely that the electron temperature is higher than the temperature of the ions, atoms, and molecules present in the gas.
The formation of the activated state $O_3(^3B_2)$, $0_3(^1A_1,v)$ $O_3(^1B_2)$, O_3^+ and O_3^- is also much favored by electrons colliding with the electrode sheathing or the conductive filling material of the electrode, causing an exchange of electrons.

Among the favorable physical conditions for the plasma also is the pressure in the discharge space. It influences the time required for maintaining conditions in a non-thermal plasma in such manner as to keep the electron temperature above that of the ions, atoms and molecules present in the gas.

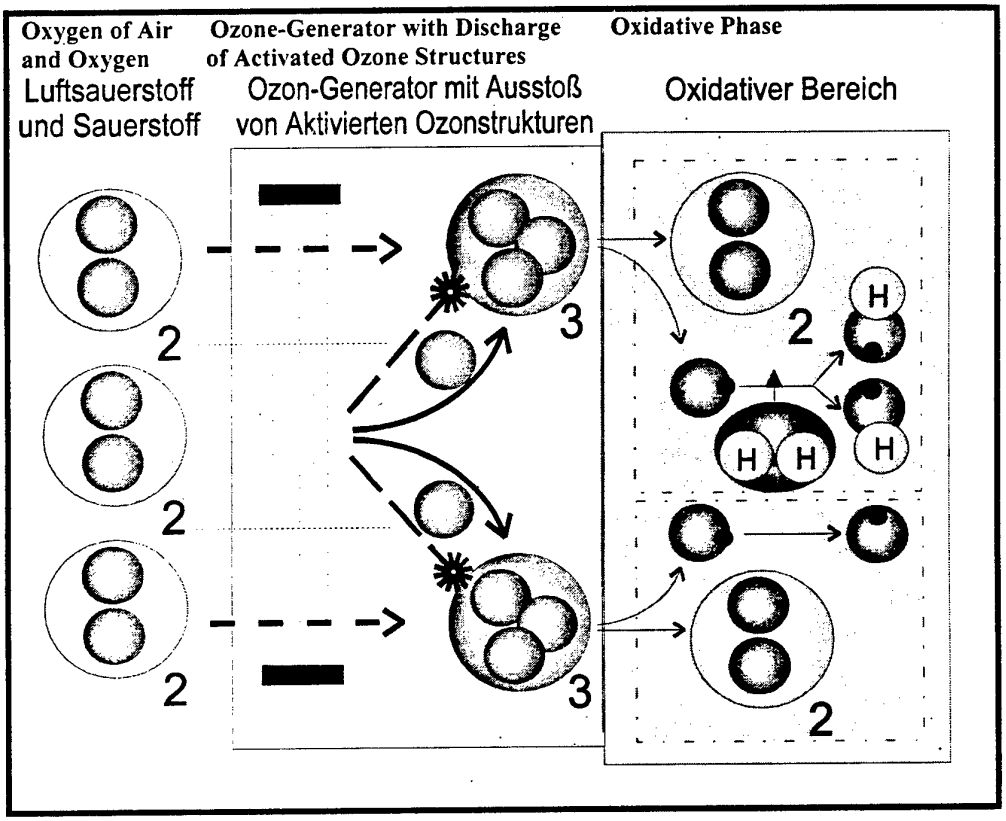
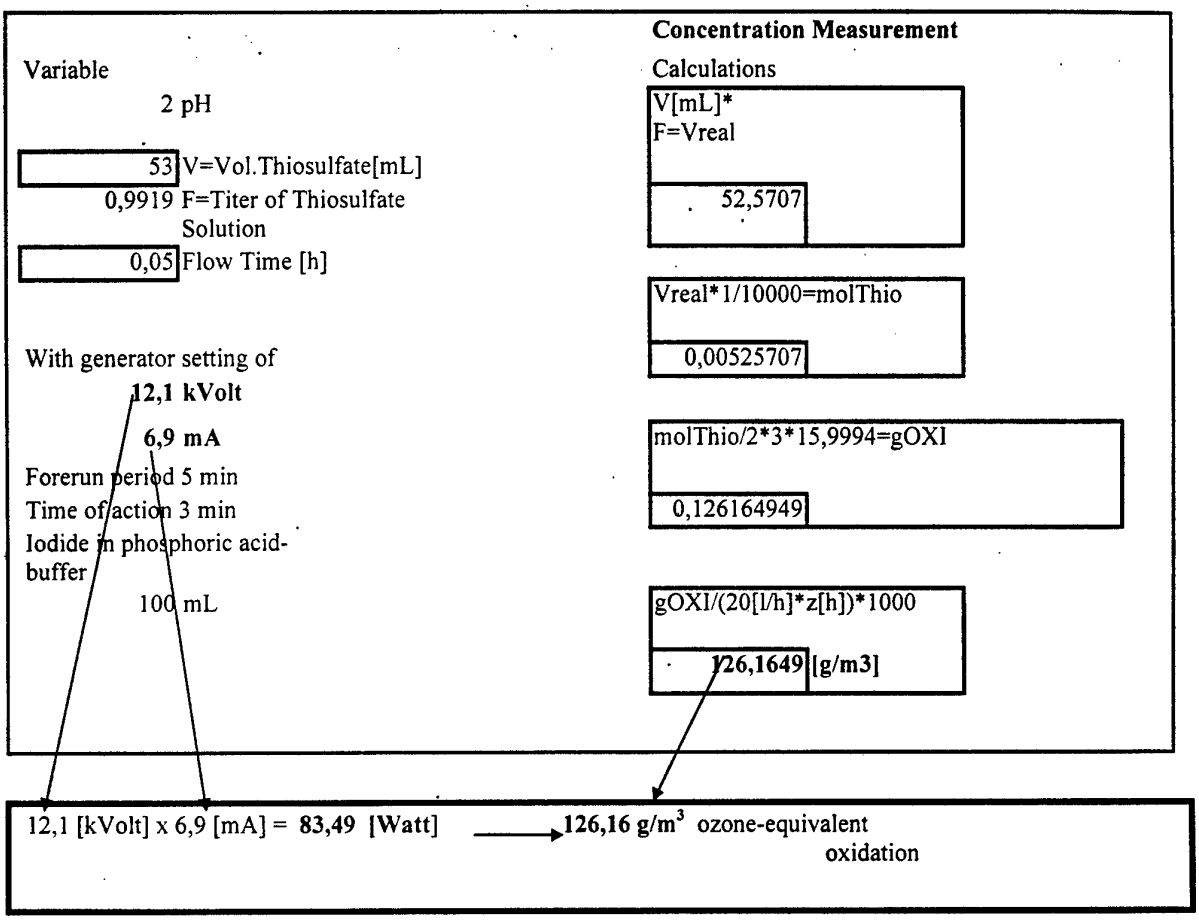


Figure 1

Following the formation of activated ozone structures, the decay occurs after $t_{1/2}$ = 70 sec.
During this period the transition from the toxic phase to the oxidative phase takes place. The intended applications must be precisely adjusted with respect to time as the transition from the activated ozone structures to the oxidative radicals is reflected by a great number of transition reactions. Depending on the influence, two types of radicals applicable for the industrial use of oxidation are usually formed:
1. In the dry phase, O-radicals
2. In the moist or liquid phase, OH-radicals whose oxidative reactions are just as radical as those of O-radicals (see Fig. 1)

Ozone-equivalent Oxidability of Activated Ozone Structures

The oxidability of the activate ozone structures is readily determinable by means of the tritration procedures frequently cited in the literature, whose parameters and equation results can be seen from the table below:



This means an ozone-equivalent oxidability of > 1.500 g / kW-h

As far as the above statistical model is concerned the number of radicals available after only $t_{1/2}$= 2 min. is equivalent to that produced after more than 5 hours with "normal ozone".

By the way of titration the oxidability proved to be approx. **1000-fold** compared to that of the "normal ozone".

Summarization for an industrial production:

The successful tests of oxidation of Clark I (CAS-Nr. 712-48-1) have shown that the treatment of Lewisit or other chemical or biological warfare agents could be the method of choice. The detoxification-reaction of Lewisit can be uprated by the Plasmazone-technology to an advanced mineralization.

In a careful calculation it seems to be possible to handle with one conversion-unit round about 2 tons of agents per day. Too, it seems to be possible to upscale a unit up to 10 tons per day.

3. Survey of Civil Application Spectrum

Processing of drinking water	Sterilization and disinfection Oxidation of odoriphores and food flavorings	Oxidation of iron and manganese Oxidation of dissolved hydrogen sulfide
Processing of swimming/whirlpool water	Disinfection Oxidation of urea	Elimination of chloramine formation
Processing and treatment of cooling, sewage, and industrial waters	Decomposition of phenol compounds Oxidation of dyestuffs (indigo) and cyanides Elimination of algae growth COD-reduction	Acting as a precipitant Stimulation of decomposition process in biological sewage plants; Oxygen enrichment of activated sludge by means of peroxides; Sewage containing feces Oxidation of organic contaminants
Purification of exhaust air resulting from: **a. odors and** **b. chemical pollution** **Use in the chemical industry**	Purification of exhaust air , such as formaldehyde, H_20-Oxidation, SO_2-Oxidation, NO-Oxidation Conversion of dioxin and furans to CO_2 Production of pure oxidation agents for chemical processes Super-clear water production Waste air and sewage treatment	Removal of odor elements Oxidation of solvent vapors Production of basic materials for the pharmaceutics and cosmetics
Use in food production	Bleaching of food Sterilization of food Sterilization of storage space Sterilization of packaging machinery Sterilization of production machinery Disinfection of spraying water and room air	Sterilization during the packing process, Disinfection of seawater for shell and fish breeding, Disinfection of used drinking water, Disinfection and increased durability of fruits Elimination of salmonellae growth Soil pest control
Optimizing of combustion process	Increased performance of vehicle motors Reduced discharge of exhaust gas Combustion compensator for vehicles with regenerative fuel system	Uniform combustion of the central heating systems
Technology of the environmental protection	Processing of contaminated ground water for irrigation fields Oxidation of dioxin and other organic agents e.g. in sewage of special waste deposits, in flue gas of combustion plants, in solid dusts Treatment of contaminated soil (f. i. with benzene etc.) Sterilization of children's playing grounds Sewage water treatment applied in microelectronics	Splitting of organic double-bonds for a better biological decomposition, Treatment of cooling tower water Oxidation of plant spraying agents (pesticides fungicides, herbicides), Treatment of mining waste water for recycling as for instance titanium Use in bio-gas procedures Removal of formaldehyde flue gas resulting from industrial wooden products

Use in hospital, industrial and domestic waste disposal	Sterilization of infectious disposal, Eliminating spores in biological disposal, Oxidation of special waste	
Use in glass, wood and textile industry	Oxidation of agents, such as formaldehyde Cleaning of surfaces in the galvanic and aluminum industry	
Use as a filter in protection shelters		
Use in the preservation of monuments and national heritage	Elimination of algae and microorganisms Preservation of monuments Preservation of art objects in museums Preservation of wood exposed to pollutants	
Medical application	Killing and inactivating of viruses Skin treatment Disinfection of surfaces	Injection of ozon-ions Oxygen enrichment Injection of pharmaceutical products

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APPLICATION OF A TRANSPORTABLE GC/MS FOR THE ANALYSIS OF CW-RELEVANT AND ENVIRONMENTAL SAMPLES

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Bruker-Franzen Analytik GmbH, Fahrenheitstr. 4, D-28359 Bremen

Mobile and transportable GC/MS systems have been applied for more than 15 years for on-site analysis of samples containing chemical warfare agents. The latest generation of mobile mass spectrometers is more compact, and offer the analytical performance of laboratory systems in the field [1].

Bruker has developed two types of *E*nvironmental *M*ass spectrometers with different vacuum and coupling systems:

- EM 640 - with *membrane inlet system* and an *ion getter pump*
- EM 640S - with *direct GC/MS coupling* and a *turbomolecular drag pump.*

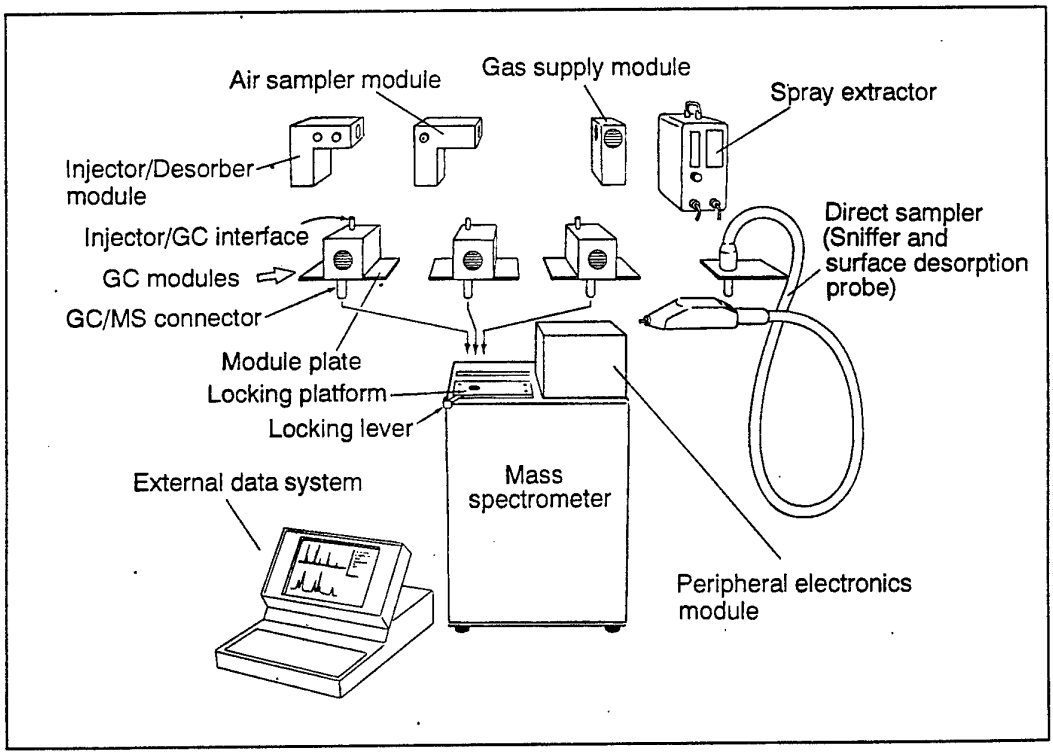


Fig. 1: Modular layout of the EM 640 and EM 640S hardware: the system consists of the central mass spectrometer coupled to an external data system and the periphery modules. The GC module is directly coupled to the MS, the injector modules are fixed on the GC modules.

The mobile GC/MS system EM 640 has a very rugged vacuum system and therefore is capable of measuring while underway. The EM 640 is used, e.g. by fire brigades or the German Coast Guard, for on-site analysis during chemical accidents and fires.

The transportable GC/MS system EM 640S is used for on-site ultra-trace analysis e.g. of water or ambient air and for on-site inspections by the Organisation for the Prohibition of Chemical Weapons (OPCW). Apart from the modification of some hardware features, analytical application procedures, analytical check procedures for QA/QC and "blinded" instrument operation procedures have been developed.

Both systems consist of the central mass spectrometer and the GC peripheral modules. The different modules can easily be interchanged with another. This allows fast changes e.g. from the Split/Splitless-Injector module to the Thermal Desorber module or between GC modules with different column specifications [1].

The mass spectrometer of the EM 640S consists of a rugged vacuum system, including ion source, quadrupole analyser, dual channeltron detector and the electronic system for controling the instrument and data acquisition. The special feature of this spectrometer arrangement is the coupling of a turbomolecular drag pump to a membrane roughing pump, which are both mounted inside the instrument. The mass spectrometer is ready for analytical operation in less than 10 minutes, including the analytical QA/QC procedure within less than 1 hour.

The analytical performance of the EM 640S can be demonstrated by chromatogram obtained from the OPCW QA/QC Check Mixture displayed in Fig.2. It contains a series of alkanes for retention index calculation and some typical compounds, which are also contained in the Grob Mixture, for checking the GC and MS performance. The GC/MS run in Fig. 3 shows a similiar example.

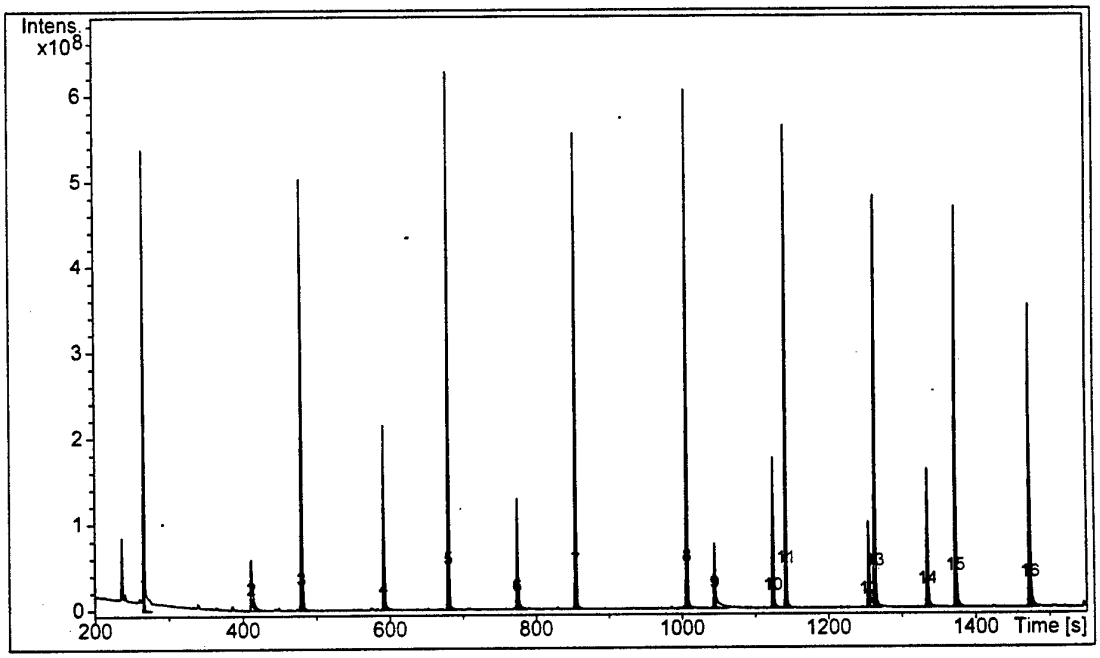


Fig. 2: OPCW Performance Check mixture at 10 ng/ l each, analysed on EM 640S: 1: Octane; 2: Phosphoric acid, trimethyl ester; 3: Decane; 4: Phenol, 2,6-dimethyl-; 5: Dodecane; 6: Benzenamine, 5-chloro-2-methyl-; 7: Tetradecane; 8: Hexadecane; 9: Phosphoric acid tributyl ester; 10: Dibenzothiophene; 11: Octadecane; 12: Malathion; 13: Eicosane; 14: Octadecanoic acid, methyl ester; 15: Docosane; 16: Tetracosane

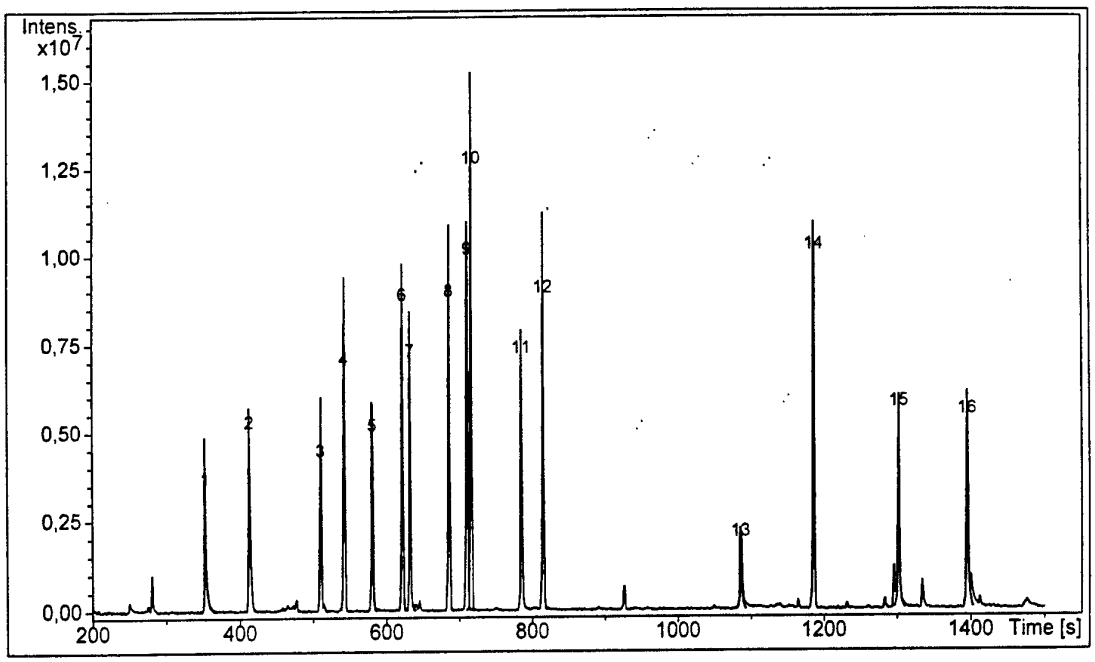
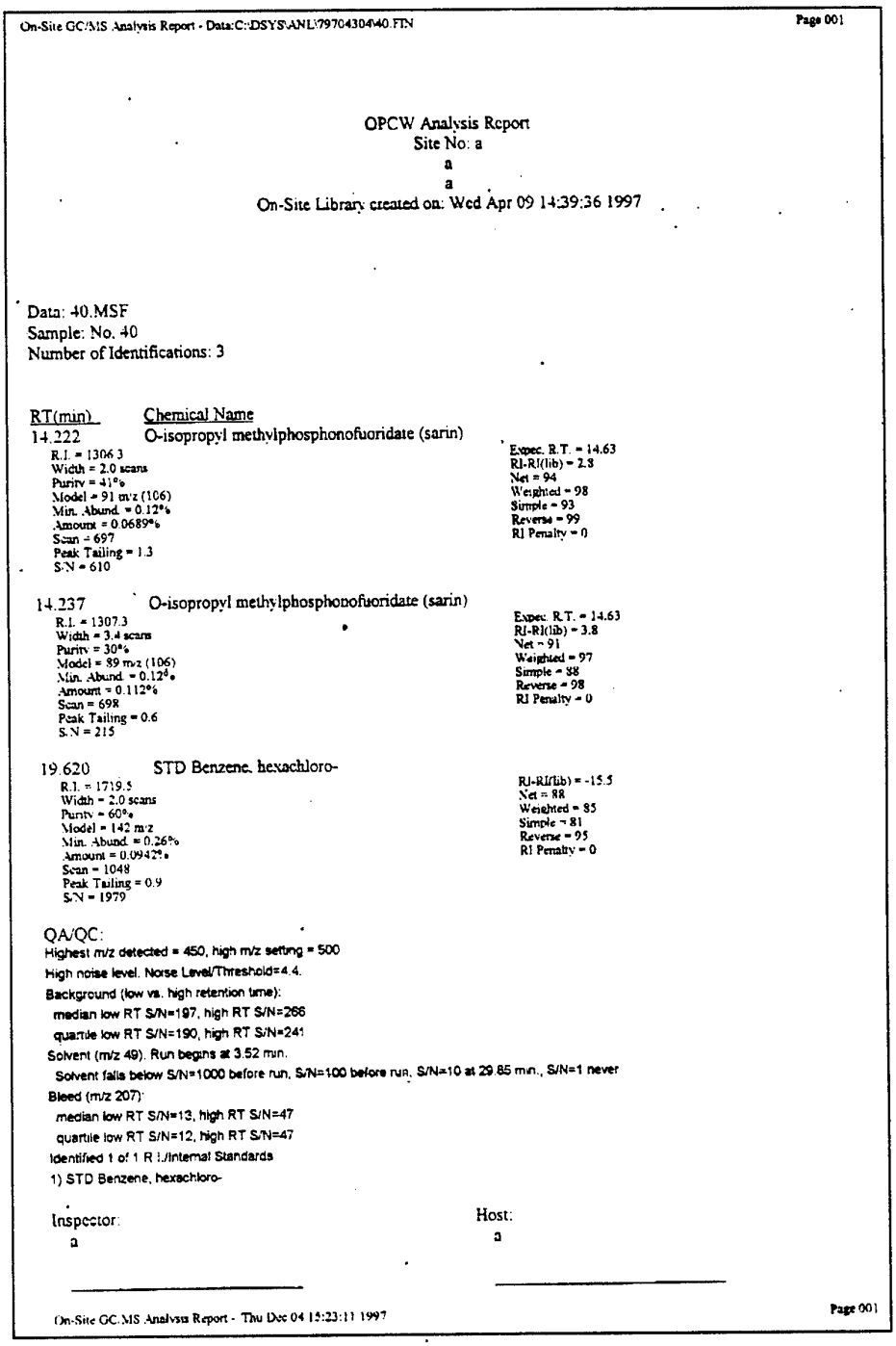


Fig. 3: Typical result of a CW performance check mixture 5 ng/ 1 each, analysed on EM 640S: 1: Dimethyl methylphosphonate (DMMP); 2: Phosphoric acid, trimethyl ester; 3: Phenol, 4-fluoro-; 4: Benzene, 1,2-Dichloro-; 5: 1-Octanol; 6: Phenol, 2,6-dimethyl-; 7: Triethyl phosphate; 8: Benzenamine, 2,6-dimethyl-; 9: Naphthalene; 10: Dodecane; 11: 1-Decanol; 12: Benzenamine, 4-chloro-2-methyl-; 13: Phosphoric acid tributylester; 14: Dibenzothiophene; 15: Malathion; 16: Octadecanoic acid, methyl ester

For the use during on-site inspections under the terms of the Chemical Warfare Convention a special "blinded" data system has been developed, that neither displays analytical information nor allows access to the data file system. This software only controls the instrument and the analytical performance is checked by a standard QA/QC SOPs. NIST developed a special GC/MS postprocessing software AMDIS for OPCW, that reports the analytical results only as paper printouts. AMDIS characterizes the analytical results by a set of numbers, e.g. retention index, identification purity and S/N ratio of the GC run. A typical OPCW analysis report is shown in Fig.4.

Fig. 5 shows an example for the GC/MS analysis of a realistic sample taken from a production process performed on the EM 640 (membrane inlet system). Regarding the identification of the compound, especially the solvents and the series of low-volatile alkanes present, the sample seemed to be an real-life mixture of a phosphor-organic pesticide. It was highly concentrated and additionally spiked with DIMP (100 ppm). Methyl Parathion could easily be identified although it was covered by alkane peaks. The small peak No. 10 could not clearly be identified. The main masses 99 and 125 u together with the relative retention time give an indication for a methyl phosponofluoridate derivative.

The above discribed examples demonstrate well the power and usefullness of transportable GC/MS systems for the on-site analysis of CW related samples. These instruments fullfill standard specifications for laboratory GC/MS in the field. They are designed for field operation and demonstrated to work reliably. In the meantime these instruments have been operated worldwide. They are packed in transport cases and sent by regular air freight to the location of operation. There they are installed and ready for work in approx. 1 hour even when applying a standard QA/QC procedure as performance check.



On-Site GC/MS Analysis Report - Data:C:\DSYS\ANL\79704304\40.FIN Page 001

OPCW Analysis Report
Site No: a
a
a
On-Site Library created on: Wed Apr 09 14:39:36 1997

Data: 40.MSF
Sample: No. 40
Number of Identifications: 3

RT(min)	Chemical Name	
14.222	O-isopropyl methylphosphonofluoridate (sarin)	
	R.I. = 1306.3	Expec. R.T. = 14.63
	Width = 2.0 scans	RI-RI(lib) = 2.8
	Purity = 41%	Net = 94
	Model = 91 m/z (106)	Weighted = 98
	Min. Abund. = 0.12%	Simple = 93
	Amount = 0.0689%	Reverse = 99
	Scan = 697	RI Penalty = 0
	Peak Tailing = 1.3	
	S/N = 610	
14.237	O-isopropyl methylphosphonofluoridate (sarin)	
	R.I. = 1307.3	Expec. R.T. = 14.63
	Width = 3.4 scans	RI-RI(lib) = 3.8
	Purity = 30%	Net = 91
	Model = 89 m/z (106)	Weighted = 97
	Min. Abund. = 0.12%	Simple = 88
	Amount = 0.112%	Reverse = 98
	Scan = 698	RI Penalty = 0
	Peak Tailing = 0.6	
	S/N = 215	
19.620	STD Benzene, hexachloro-	
	R.I. = 1719.5	RI-RI(lib) = -15.5
	Width = 2.0 scans	Net = 88
	Purity = 60%	Weighted = 85
	Model = 142 m/z	Simple = 81
	Min. Abund. = 0.26%	Reverse = 95
	Amount = 0.0942%	RI Penalty = 0
	Scan = 1048	
	Peak Tailing = 0.9	
	S/N = 1979	

QA/QC:
Highest m/z detected = 450, high m/z setting = 500
High noise level. Noise Level/Threshold=4.4.
Background (low vs. high retention time):
median low RT S/N=197, high RT S/N=266
quartile low RT S/N=190, high RT S/N=241
Solvent (m/z 49). Run begins at 3.52 min.
Solvent falls below S/N=1000 before run, S/N=100 before run, S/N=10 at 29.85 min., S/N=1 never
Bleed (m/z 207):
median low RT S/N=13, high RT S/N=47
quartile low RT S/N=12, high RT S/N=47
Identified 1 of 1 R.I./Internal Standards
1) STD Benzene, hexachloro-

Inspector: Host:
a a

On-Site GC/MS Analysis Report - Thu Dec 04 15:23:11 1997 Page 001

Fig. 4: Print out of a OPCW Analysis Report as produced by this AMDIS software. Only the QA/QC standards and substances identified by MS database comparison are listed. In this case a training database was used. In case of an inspection the MS database will be negotiated between the OPCW inspectors and the inspected side party (ISP).

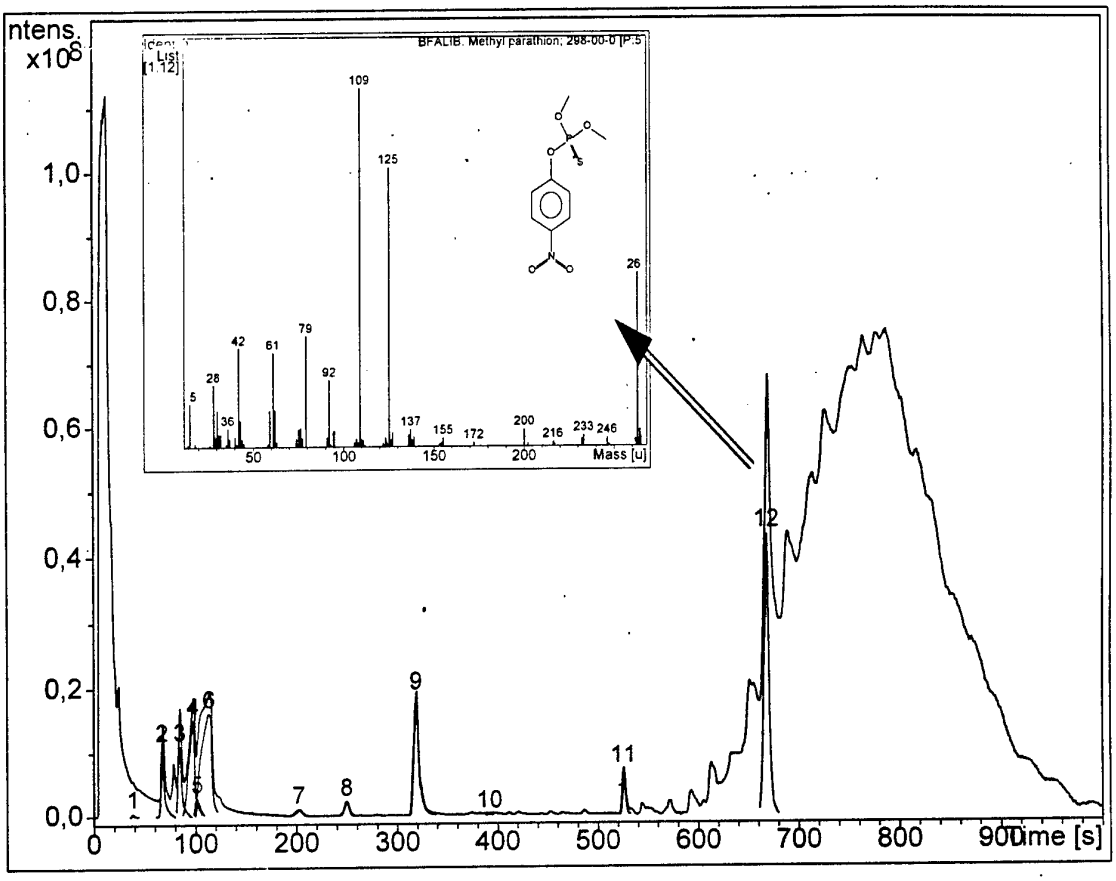


Fig. 5: A realistic sample from a production process, analysed on EM 640 (membrane inlet system). It contains some typical solvents (1-5: acetone, n-butanol, substituted benzenes and cyclohexanone), 6: DMMP, 7: TMP, 8: DIP, 9: spiked DIMP (100 ppm) and 12: Methylparathion, covered under a broad peak of overlapping alkanes.

Lit.: [1] G.Baykut, J.Franzen; TRAC 13 (1994) 267; 14 (1995) 10.

Proceedings of the International CW Destruction Symposium
Munster, Germany, March 22-25, 1998

NON-DESTRUCTIVE EVALUATION OF CWA CONTAINERS USING NEUTRON ACTIVATION ANALYSIS

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Bruker Saxonia Analytik GmbH, Leipzig, Germany

1. Introduction

One of the most powerful methods for analyzing contents of closed containers is the Neutron Activation Analysis [1]. The analysis system NIGAS uses one specific kind of these method, the Prompt Gamma Neutron Activation Analysis, for the identification of chemical warfare agents.
A pulsable neutron generator, which is based on the deuteron-deuteron reaction, serves as neutron source. The system is portable and can be applied in ammunition depots, chemical demilitarization facilities or under field conditions.

2. Method of Analysis

The NIGAS system uses prompt gamma neutron activation to distinguish between different explosives and chemical warfare agents on the basis of their element compositions.
A neutron source supply neutrons, which penetrate through the containment wall into the analyte and interact with its atomic nuclei. These nuclear reactions cause the emissions of gamma quanta of discrete energies that are specific for the element atoms involved in the nuclear reactions [2,3]

The energies of the neutrons strongly determine in which way they interact with the atomic nuclei to be analyzed.
The main interaction of neutrons with an energy of 2 to 5 MeV is the inelastic scattering ((n,n'γ) reaction). The atomic nuclei of the object to be analyzed are excited by the neutrons. The transition from the excited state to the original state emits characteristic γ-radiation [4,5].
The energy of neutrons decreases by their interactions and the so-called thermal energy range is reached. Within this thermal energy range, the interaction of the neutron capture ((n,γ) reaction) is used for analysis by measuring the energy which is released during the nuclear reaction in the form of gamma quanta [6].
Therefore, since the γ-radiation correspond to discrete energies, the nuclei involved in the interactions can be determined.

For distinguishing between explosives and chemical warfare agents on the basis of their elemental compositions, the detection of the elements given in Table 1 is especially important:

CWA	MATRIX ELEMENTS				KEY ELEMENTS				
	H	C	N	O	F	P	S	Cl	As
Clark I	3.8	54.5						13.4	28.3
Clark II	4.0	61.2	5.5						29.4
Lewisite	1.0	11.6						51.3	36.1
Adamsit	3.3	51.9	5.0					12.8	27.0
S-Lost (HD)	5.1	30.2					20.2	44.6	
Phosgene		12.1		16.2				71.7	
Tabun	7.4	32.0	18.7	21.3		20.6			
Sarin	7.2	34.3		22.8	13.6	22.1			
Soman	8.9	46.2		17.8	10.4	17.0			
VX	9.8	49.4	5.2	12.0		11.6	12.0		
TNT	2.2	37.0	18.5	42.3					

Tab.1: Elemental composition of chemical warfare agents and explosives (examples)

As a rule, it is not necessary to determine the absolute elemental composition. In many cases, unambiguous identification of the relevant elements and determination of the ratio of the elemental composition is sufficient to identify compounds or substance groups.
In many cases, the elements listed on the right side of Tab. 1 can be used as key elements for identification purposes. The identification of chemical warfare agents is possible by detecting the presence of one or two key elements.

3. Set-up of the analytical system

The analytical systems consists of 4 units:

- pulsable neutron source
- γ-radiation detection system with shielding equipment
- ammunition holder
- computer

The whole system is portable. Fig. 1 shows the set-up.

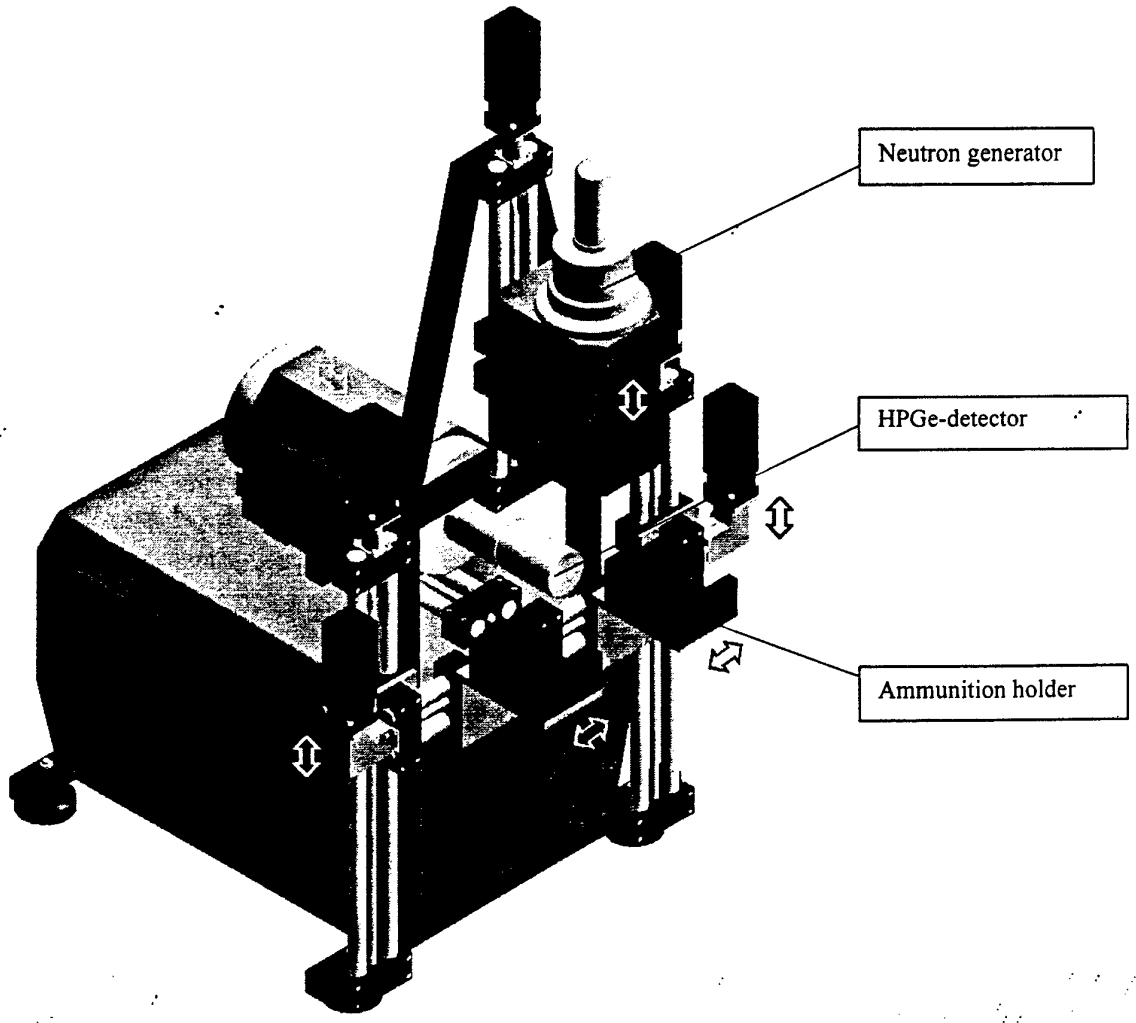


Fig.: 1 Instrument set-up

3.1 Neutron Source

Due to the use of a neutron generator as a neutron source, mutual interferences caused by different interactions can be avoided by pulsed operations of the generator and recording of the gamma spectra in time intervals.
The inelastic scattering reactions are observed during the neutron pulse. The time-delayed nuclear reactions taking place during the pauses between neutron pulses are recorded in separate spectra.

The neutron generator, unlike an isotope neutron source, has the advantage of emitting neutrons only during its operation. During pauses and transport ionizing radiation is not generated. By using a neutron generator on the basis of the deuteron-deuteron-reaction, the generator contains no radioactive material.

3.2 Gamma Radiation Detector with Evaluation Software

The energy of the gamma quanta emitted by the nuclear reactions is in the range of 100 keV to about 10 MeV. On the one hand, this relatively high energy is favorable for the capability to penetrate through the container wall, on the other hand it necessitates large volume of the detectors. For energy dispersive measurements with high energy resolution an HPGe detector is used. The detector has to be cooled with liquid nitrogen or via a cooling unit.
Principally, the detectors are damaged by the neutrons. Therefore, and also because of the interfering γ-radiation, they have to be shielded from the neutron source.
The evaluation electronics are able to record several gamma spectra during the cycle time of 0.1 ms. Using fast amplifiers and ADC minimizes counting losses.
Every 10 s the accumulated spectra are analyzed during the measuring process. A special software calculates peak areas and carries out a statistical valuation. The recognized elements are displayed and in dependence on the element composition the substance name is given out.

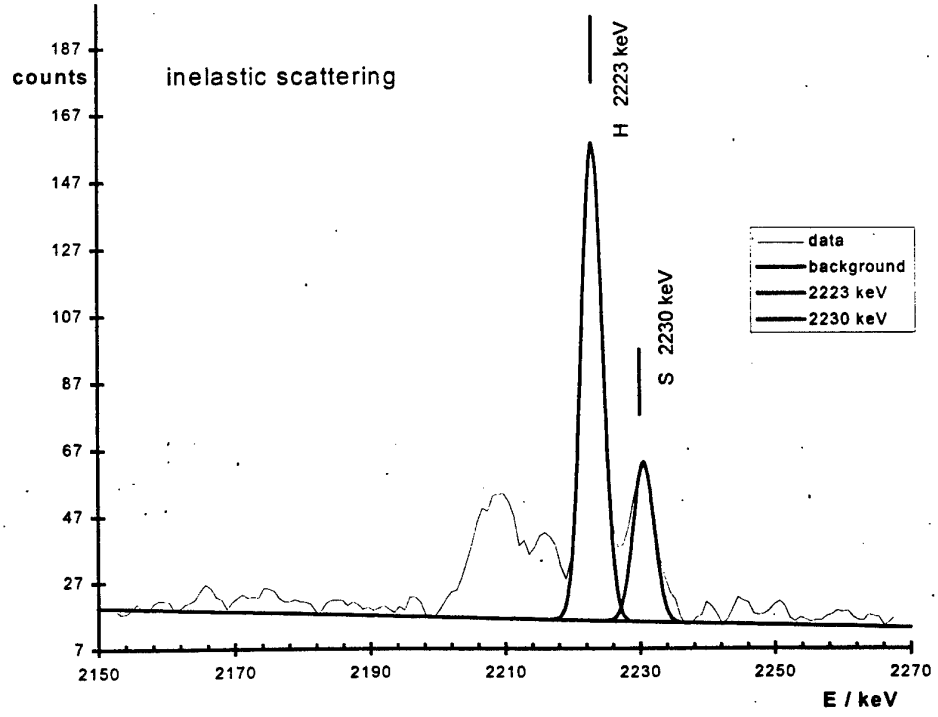
4. Results of CWA measurements

To date, several test substances have been analyzed using the system of analysis.

In Fig 2 an example of a chemical warfare agent measuring result is shown.

Thickness of container wall: 15 mm of steel
Content: simulation substance of HD

The two key elements chlorine and sulfur were identified.



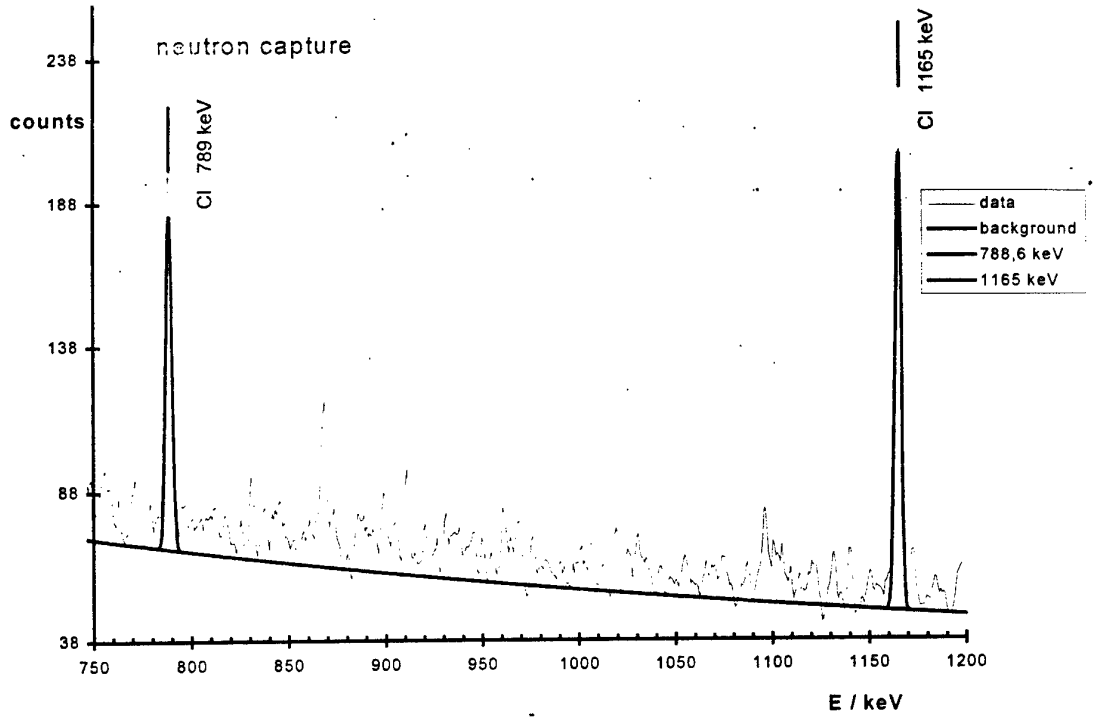


Fig. 2 : Gamma spectra of HD simulation substance (15 mm wall thickness)

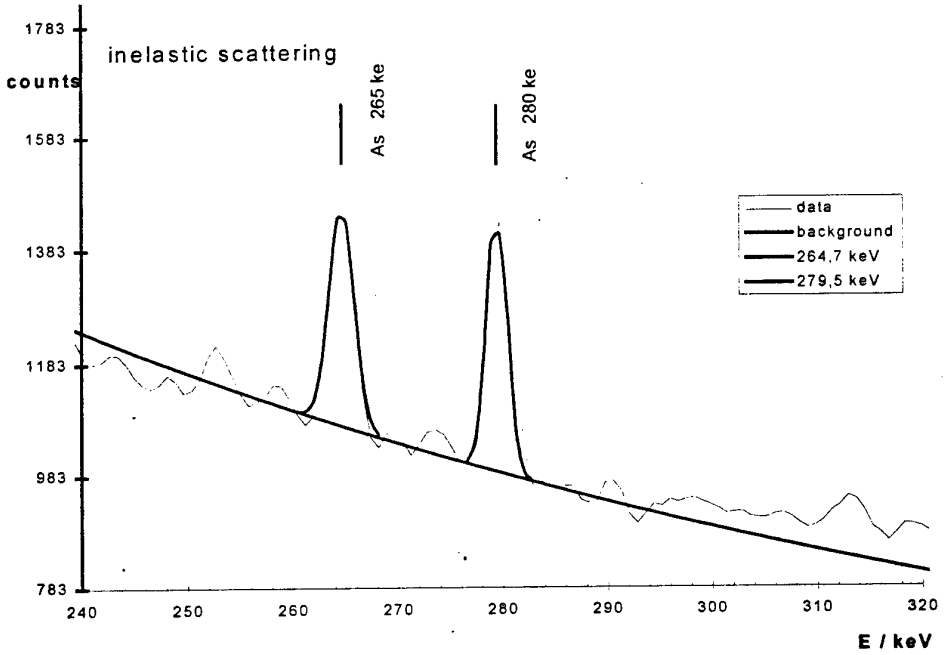


Fig. 3 : The Arsenic part of a of Lewisite Gamma spectrum (10 mm wall thickness)

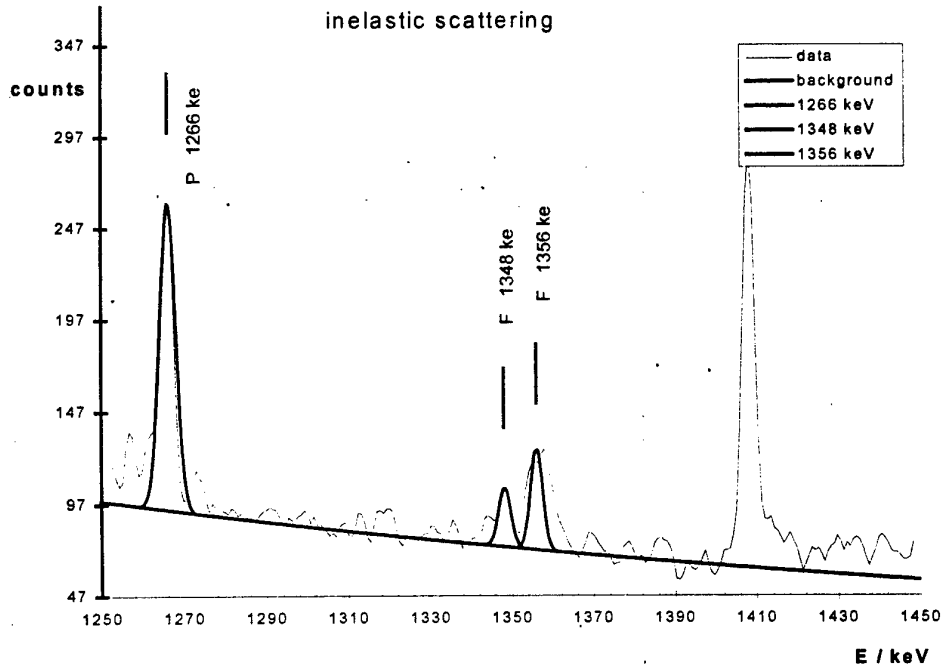


Fig. 4 : The Fluorine and Phosphorus part of a Sarin Gamma spectrum (10 mm wall thickness)

In Fig 3 and 4 the other examples of chemical warfare agent measuring results are shown.

Thickness of container wall: 10 mm of steel
Content: simulation substance of the skin agent Lewisite (Fig. 3)
simulation substance of the nerve agent Sarin (Fig. 4)

These examples show how CWAs or explosives can be identified.
The presence or absence of key elements leads to a typical pattern of the gamma spectra. By means of a check at certain energy regions the software can decide which substance is in the container.
On the PC display a graphic shows the results of the measurement and the analytical calculation (Fig. 5). Every key element is evaluated statistically. Elements which are detected by the software and the identified substance are displayed in the upper side of the graphic. A special chart on the right side warns if a chemical warfare agent is present.

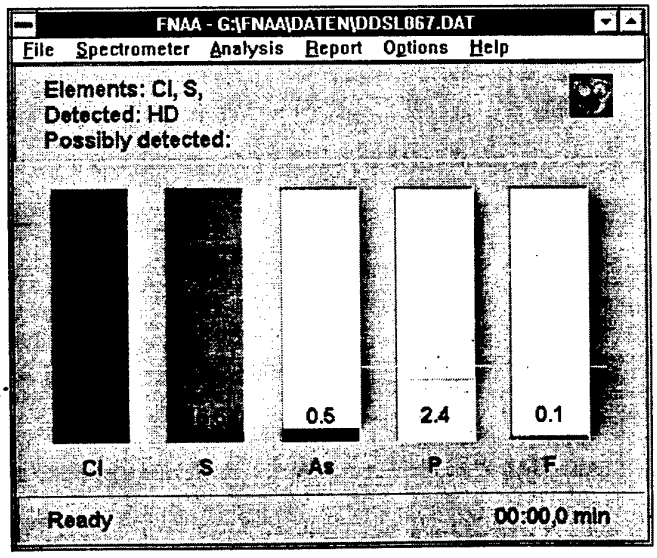


Fig. 5 Display of spectrometer control and analytical results output

5. Summary

Neutron activation analysis is a powerful method for the non-destructive evaluation and analysis of the content of containers. By using neutron generators which only emit radiation during the measuring process and compact detection systems it is possible to produce portable analytical systems which can be operated at the site of the substance to be analyzed.
The measuring result which are obtained within a few minutes provides an element analysis allowing an assignment to the substances of interest with given substance groups. This enables the distinction between different explosives and chemical warfare agents on the basis of their element compositions.

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[4] C.M Lederer, V.S. Shirley, "Table of Isotopes", 7th ed., Wiley & Sons, New York 1978

[5] D. Duffey, A. El-Kady, F.E. Senftle, Nucl. Instr. and Meth. 80 (1970) 149

[6] A.M. Demidov, M.R. Ahmed et al., Atlas of Gamma Ray Spectra from the Inelastic Scattering of Reactor Fast Neutrons, Moscow, 1978

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APPLICATION OF ION MOBILITY SPECTROMETRY DURING DISPOSAL OF CHEMICAL WARFARE AGENTS

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Ion mobility spectrometry (IMS) has become one of the most important on-site methods for the detection of chemical warfare agents during the last years. This trend is mainly caused by typical advantages of this technique that offers high sensitivity, instrumental simplicity, low cost, analytical flexibility, and real time monitoring capability [1,2]. During disposal of chemical warfare agents, the method is deployed for various tasks [3]:

a) Protection of personnel,

b) Field screening for detection of contaminated sites,

c) Hazardous site inspections for detection of leakages etc.,

d) Identification of chemical warfare agents during dismantling of ammunition,

e) Monitoring of chemical warfare agents in facilities used for disposal of warfare agents,

f) Breakthrough surveillance of filters for environmental protection.

In the chemical warfare agent disposal facility of the German Armed Forces, stationary installed ion mobility spectrometers (RAID-S, Bruker-Saxonia Analytik GmbH) are used for the control of bunkers in which chemical warfare agents are stored. The stationary warning device RAID-S is a gas trace detector, which can be used for the detection of chemical warfare agents and other toxic substances. The sensor of this device is an ion mobility spectrometer based on the design of the RAID-1 [1,2], which is modified to achieve longer maintenance periods (approximately one year). The RAID-S can be used as a single instrument or in monitoring networks of up to four instruments in the standard RAID-S version or up to 255 instruments using a special module.

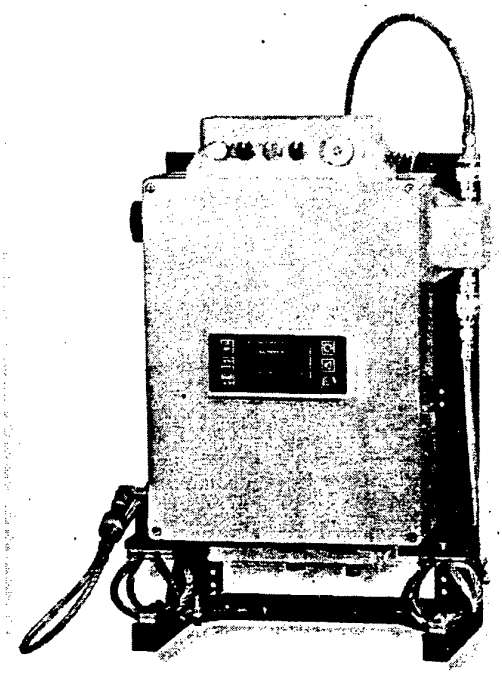


Fig. 1: Stationary ion mobility spectrometer RAID-S

In the disposal facility, filter monitoring for environmental protection is also realized by stationary ion mobility spectrometers (RAID-S). However, an ammonia ion chemistry insight the measuring cell has to be taken into account for this type of application. Due to the presence of ammonia in activated carbon filters, additional product ions have to be considered for the identification and quantification of simulants and chemical warfare agents in the positive ion mode. Corresponding library entries and calibration curves are programmed in the RAID-1 and RAID-S.

Mobile ion mobility spectrometers (RAID-1, Bruker-Saxonia Analytik GmbH) are applied for the protection of personnel as well as for a fast identification of the warfare agents found in the disposed munition, which is important for the decontamination procedure of the metal parts of the shells. Due to an automatic polarity switching, the RAID-1 detects continuously all relevant chemical warfare agents (GB, GA, GD, VX, HD, HN, L, AC, CK, CG) in the ppb concentration range present in the ambient air.

[1] Ionenmobilitätsspektrometrie - Grundlagen und Applikationen, J. Stach, Analytiker Taschenbuch, 16 (1997) 119.

[2] G.A. Eiceman und Z. Karpas, Ion Mobility Spectrometry, CRC Press, Boca Raton,1994.

[3] J. Stach, K. Miersch, D. Schaper und H.-J. Döring, 5th Int. Symp. on Protection Against Chemical and Biological Warfare Agent, Stockholm, 11.-16.6.1995.

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Munster, Germany, March 22-25, 1998

EMISSION MONITORING BY MEANS OF AN IMS DURING M6 CN/DM-HANDGRENADES DESTRUCTION

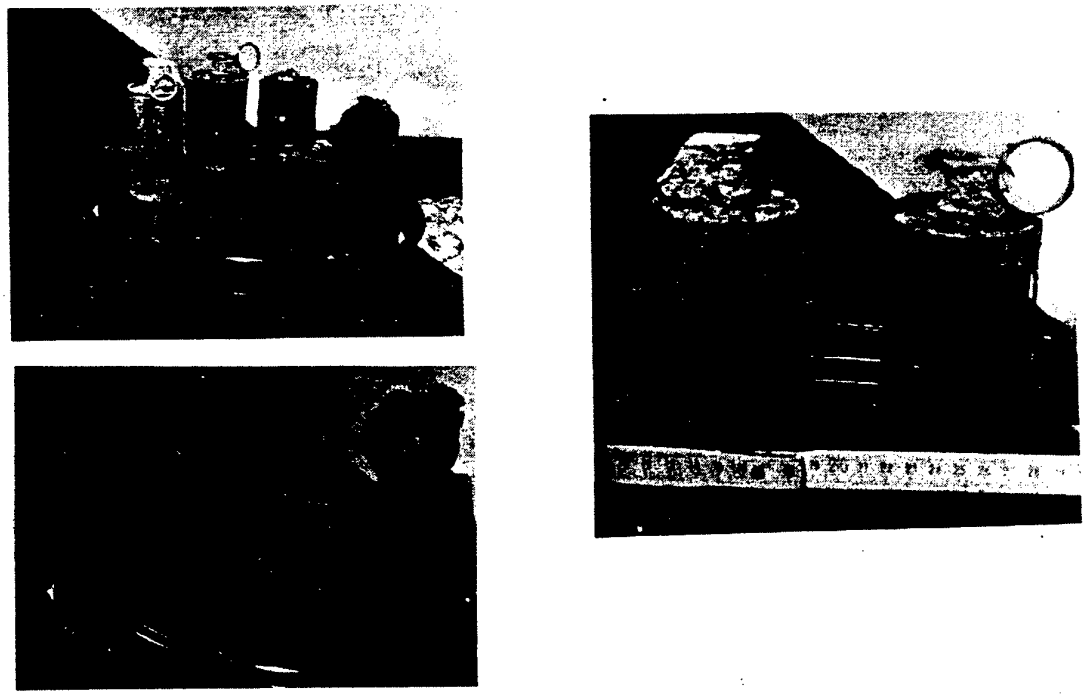
Bensch, H.[1)], Katzung, W. [2,3)], Leonhardt, J.[1)], Standtke, K.[1)]

[1)] IUT Institut für Umwelttechnologien GmbH, Rudower Chaussee 5, D-12489 Berlin
[2)] Toxikologe, Postfach 321, D-39288 Burg
[3)] Dr. Koehler Gesellschaft für Kampfmittelbeseitigung mbH, D-39288 Burg,
In der Alten Kaserne 10

Problem

Corroded hand grenades M6 were found in the ground. The disposal was done by means of PYROCAT technology [1)]. Exhaust air and process gases had to be controlled before discharging to the atmosphere. German authorities of the district of Munster determined the concentration of Chloroacetophenon as lead concentration. Alarm threshold was 50 ppb as the maximal working place concentration.

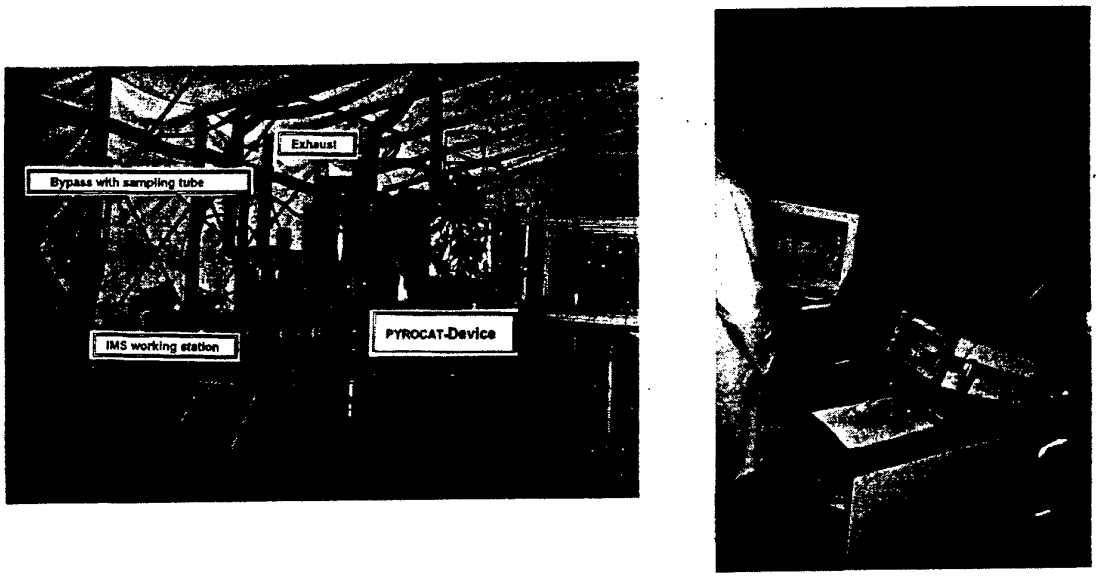
fig. 1: **Samples of old ammunition**



Analytical Situation

The exhaust air of the delaboration plant contained about 10 ppm methanol vapour behind the gas washer unit and also a relative humidity of about 80 %. So a bypass was installed (in accordance with BImSchG) which fed the IMS entrance port.

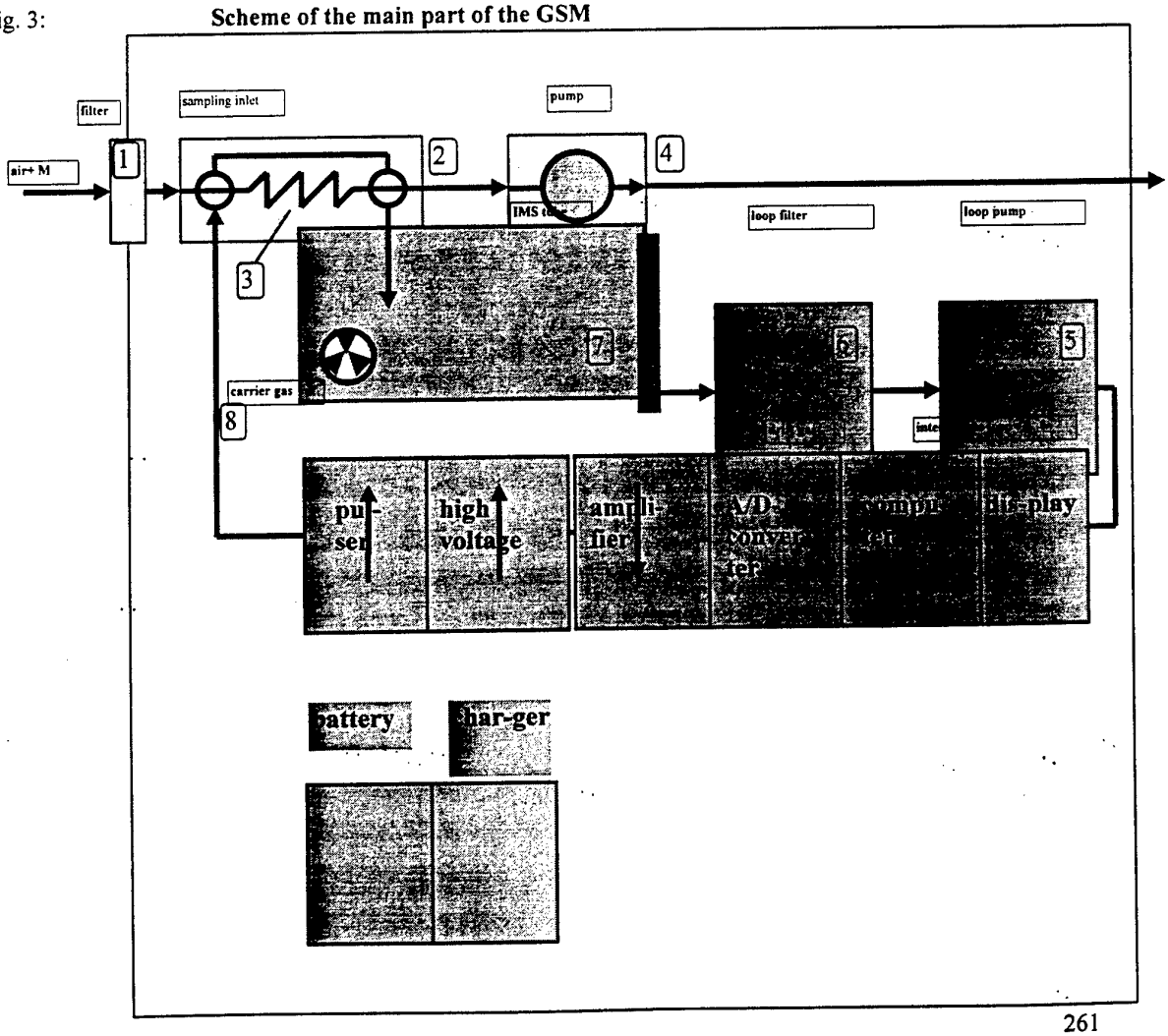
fig. 2: **Equipment for the destruction**



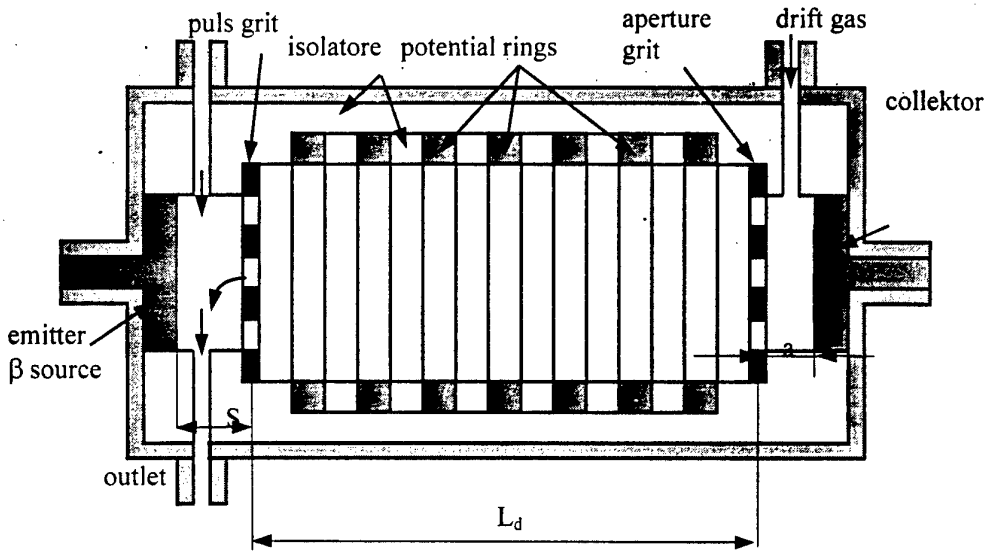
Method

The IUT IMS GSM [2] is equipped with an automatic sampling unit, which provides about 0,5 cm^3 exhaust air from the bypass to the ion source every 30 seconds.

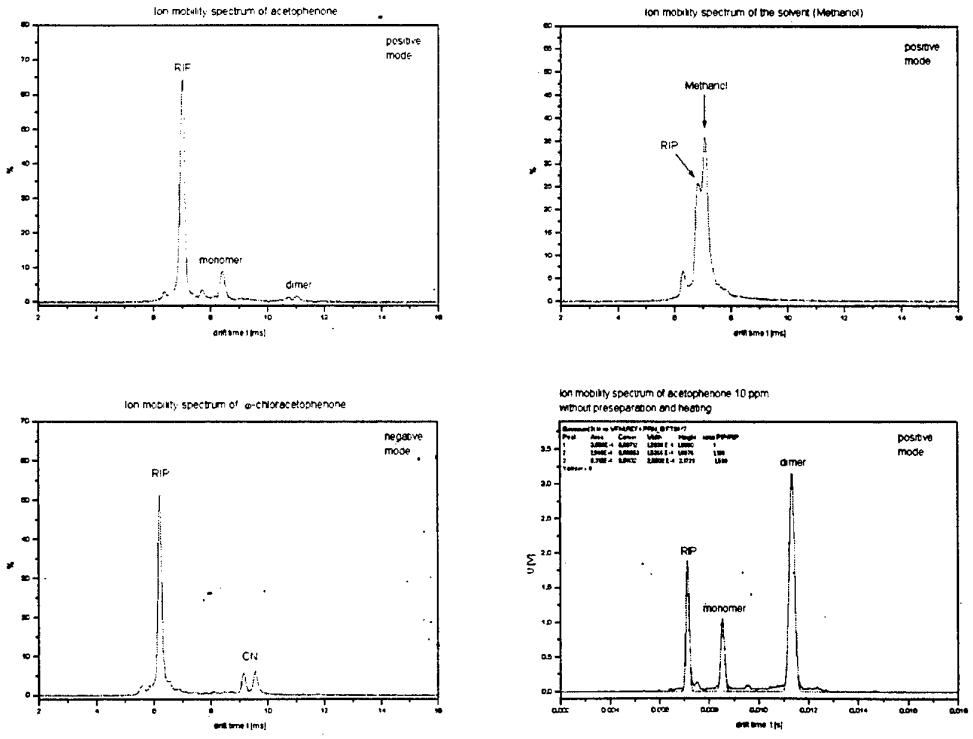
fig. 3: **Scheme of the main part of the GSM**



Scheme of IMS drift tube design



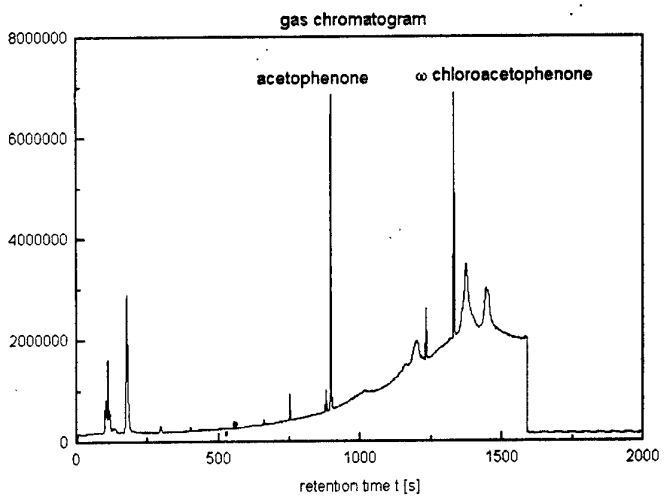
The realistic spectra of the IMS are as follows:
fig. 4:

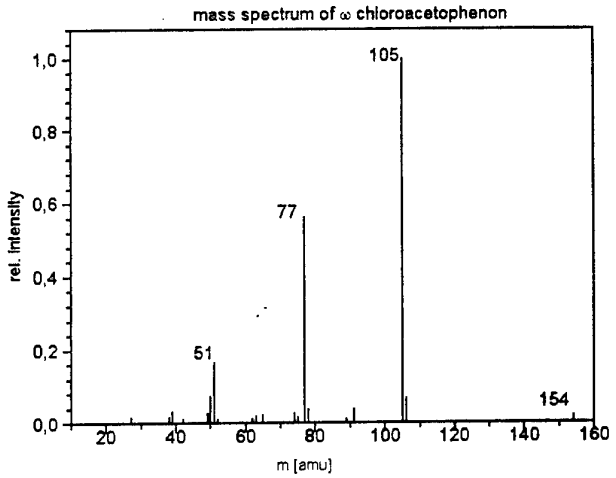


Calibration

The calibration of the system is sophisticated due to the CN-hydrolysis. So the calibration samples had to be controlled by GC-MS.

fig. 5:





So in the positive mode there may be some irritation of the acetophenon cross sensitivity.

Results

- The IMS was used for 3 months continuously.
- The minimal detection concentration (MDC) achieved was about 5 ppb, i.e. 1/10 of the demanded alarm threshold.
- Data were recorded by means of an internal computer and transmitted to a PC.

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SNOOPER 1.1 : A PLANNING AND OPTIMIZING TOOL FOR FOX NBC RECONNAISSANCE SYSTEMS

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The programme SNOOPER has been developed as a joint product of Henschel Wehrtechnik, Kassel, and IABG, Ottobrunn. This software is representing a system with which the deployment of FOX NBC reconnaissance vehicles can be planned and co-ordinated, and an overview of the NBC situation within the theatre of operation can be achieved more quickly.

The programme analyses the concentration values recorded by the recce vehicles in order to estimate the size of the contaminated area. The programme provides two different calculation methods for estimating the contamination area: The Gaussian model, which is well suited as "Quick look" method. However, since topography effects cannot be taken into consideration and constant wind conditions must be assumed, the calculation results in a rather crude estimation. The Lagrangian model, with which the effects of complex topography as well as temporal changes of the weather conditions can be taken into account.

Besides the dispersion calculations, the programme additionally features the possibility to estimate the location of different toxic sources on the basis of concentration measurements gathered by the FOX NBC recce vehicles.

The programme was designed as a Windows application for the operating systems Windows 3.1x, Windows 95, and Windows NT, whereby a user-friendly application interface can be made available. It provides different types of result view to the user. A map window displays a topographic map in which positions of all relevant objects (recce vehicle, toxic material sources, meteorological stations, measurement points) are marked by corresponding symbols, respectively. A second window is available in which the measurement points are listed in tabular form and the characteristics of the individual objects can be monitored.

The input and the output of the programme meet the requirements of NATO ATP 45. It provides the possibility to receive and to transmit NBC messages, which are held in logs for later use, respectively. Messages to be sent are created automatically by the programme and can be edited by the user before transmitting. Received NBC messages are analyzed automatically by the programme as well. The programme is intended to be used at platoon level as part of a mobile operating unit consisting of a laptop and communication hardware, which are placed in a portable metal box.

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THE DESTRUCTION OF CHEMICAL WEAPONS UNDER THE PROVISIONS OF THE CHEMICAL WEAPONS CONVENTION

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The Chemical Weapons Convention (CWC) places a duty on its Member States to both declare and destroy any chemical weapons they own or possess or that are located in any place under their jurisdiction or control or that they abandoned on the territory of another States Party. This destruction must be undertaken in accordance with special timelines, in an approved manner and verified. The CWC separates chemical weapons into different classes according to the risk they pose to the object and purpose of the Convention and the level of verification is related to the class of chemical weapons being destroyed. This paper examines the potential impact of these requirements on the design and operation of facilities for the destruction of chemical weapons.

THE DESTRUCTION OF CHEMICAL WEAPONS UNDER THE CWC

by
Ron G Manley
Special Adviser to the Director-General
Organisation for the Prohibition of
Chemical Weapons

OPCW

Proceedings of the International CW Destruction Symposium
Munster, Germany: March 22-25, 1998

SOME STATISTICS

■ Member States	107
■ Signatory States	61
■ Months since EIF	10
■ Inspections completed	160
■ Member States Inspected	25

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Munster, Germany, March 22-25, 1998

DEFINITION OF A CHEMICAL WEAPON

"Chemical Weapon" means the following, together or separately:

(a) Chemical Agents and their precursors;

(b) The munitions and devices for the delivery of Chemical Agents; and

(c) Equipment specifically designed for use with the above.

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CLASSES OF CHEMICAL WEAPON

- Chemical Weapons produced before 1925
- CW produced between 1925 and 1946 and determined to be "unusable"
- CW produced after 1946 and CW produced between 1925 and 1946 which are determined to be "usable"
- Abandoned Chemical Weapons
- CW buried before January 1977
- CW dumped at sea before January 1985

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VERIFICATION OBJECTIVES

- To confirm the identity and quantity of the chemical weapons stocks to be destroyed; and
- To confirm that these stocks have been destroyed

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VERIFICATION AT CW STORAGE SITES

Confirm:

- Inventory
- Identification
- Status

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DESTRUCTION OF CHEMICAL WEAPONS

Means a process by which chemicals are converted in an **essentially irreversible** way to a form unsuitable for the production of chemical weapons and which in an irreversible manner renders munitions and other devices unusable as such.

OPCW

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PROHIBITED DESTRUCTION OPTIONS

- LAND BURIAL
- OPEN PIT BURNING
- SEA OR INLAND WATER DUMPING

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CHEMICAL WEAPON CATEGORIES

- **Category 1**

  Chemical weapons on the basis of Schedule 1 chemicals and their parts and components

- **Category 2**

  Chemical weapons on the basis of all other chemicals and their parts and components

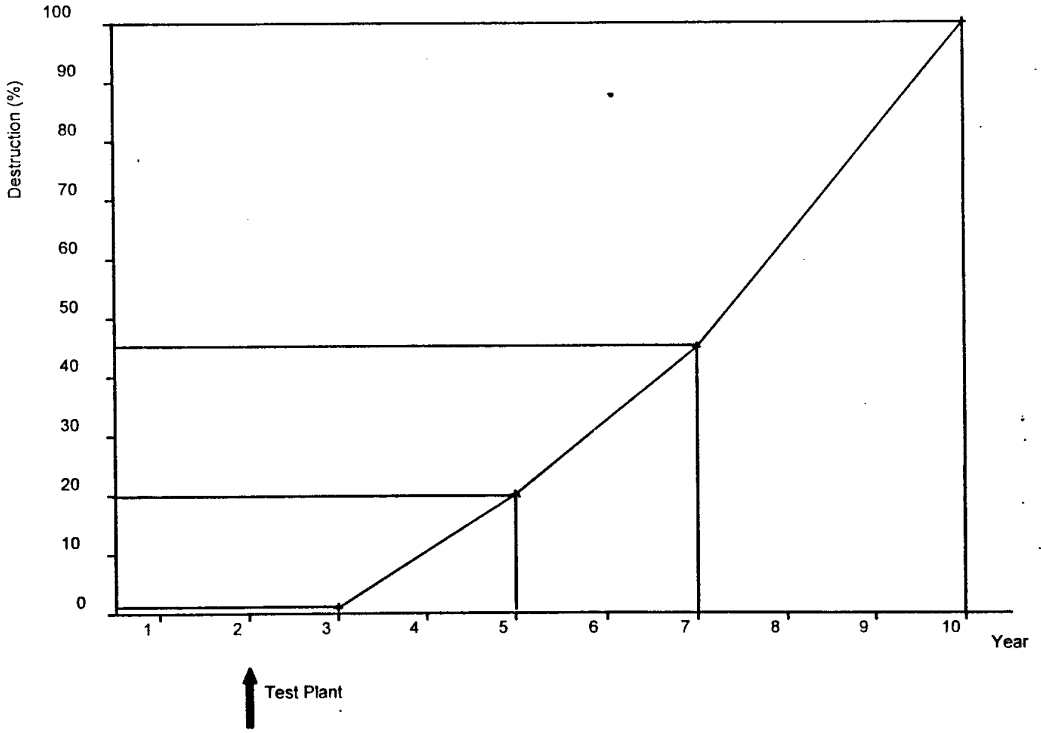
- **Category 3**

  Unfilled munitions and devices and equipment

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REQUIRED DESTRUCTION RATE



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STEPS IN THE VERIFICATION PROCESS

- State Party to submit detailed destruction plans
- Secretariat to draw up verification plan for each site
- Continuous on-site presence by inspectors
- Facility agreements
- Annual destruction plans
- Bilateral destruction agreements

OPCW

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VERIFICATION AT CW DESTRUCTION SITES

- The receipt of CW at the facility
- Inventory of the temporary holding area
- The specific type and quantity of CW being destroyed
- The process of destruction
- The end-product of the destruction process
- The mutilation of metal parts
- The integrity of the destruction process

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DESIGNING DESTRUCTION FACILITIES

Some factors to be considered:

- Means to verify the CW input to the system
- Provision for sample collection
- Installed monitoring systems
- Means to demonstrate system integrity
- Means to verify the products from the process
- Means to demonstrate completeness of destruction

OPCW

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DISMANTLING INSTALLATION FOR WORLD WAR I TOXIC CHEMICAL AMMUNITION IN BELGIUM

Senior CPT Alfons Vander Mast

Belgian EOD Squad, 8920 Langemark-Poelkapelle, Belgium

In the western part of Belgium, and more especially near the Flemish town of Ypres (the World War I front line city and the source of inspiration for the well-known synonym for the sulphur mustard "Yperite") farmers and construction workers are daily discovering artillery and mortar shells as well as grenades dating from The Great War. Several teams of the Belgian EOD Squad of Poelkapelle daily collect these discovered unexploded ordnance (UXO) in order to treat them at the Steenstraat Quarters, an army depot at approximately 15 km north of Ypres.

In the first part the following subjects will be presented:

- The organisation of the unit
- The main tasks of the unit

In the second part a historical overview of that particular Belgian "problem" is given.

The third part gives a more conceptual information about the dismantling facility itself:

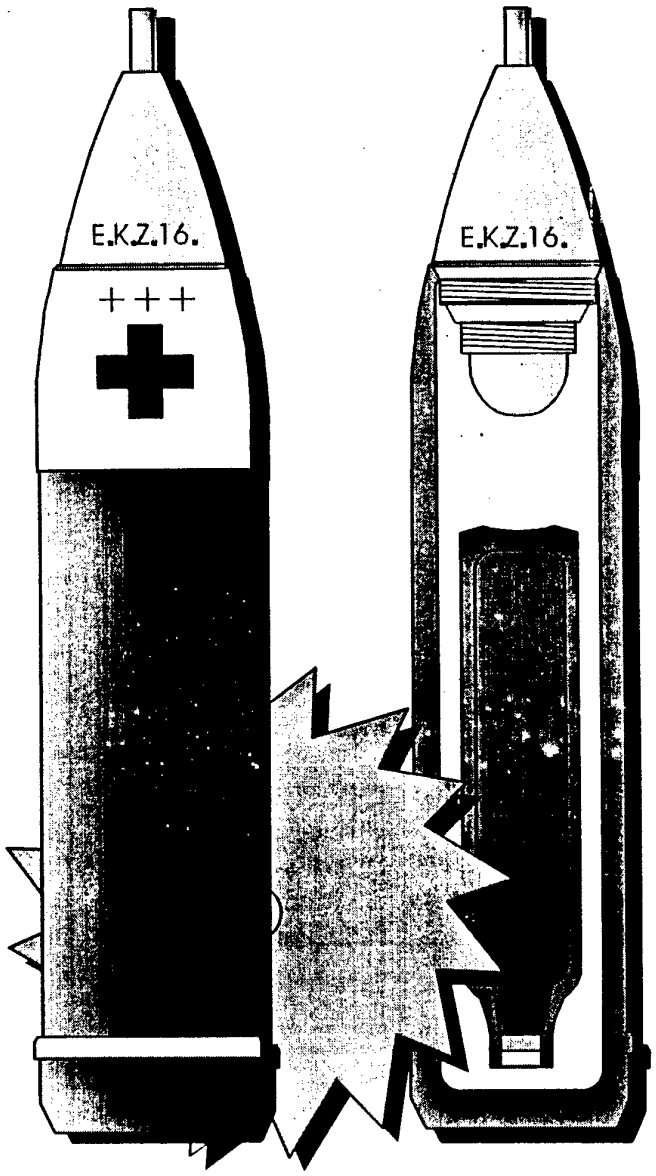
- The identification process
- The dismantling process

In the fourth and last part the different safety aspects with regard to the infrastructure and to the procedures will be presented for the following risk scenarios:

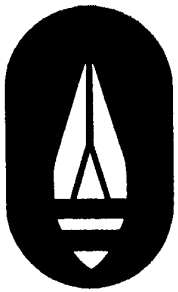
- The risk of explosion
- The risk of fire
- The risk of contamination

THE DISMANTLING OF CHEMICAL AMMUNITION WW1 AT POELKAPELLE

March, 23th 1998



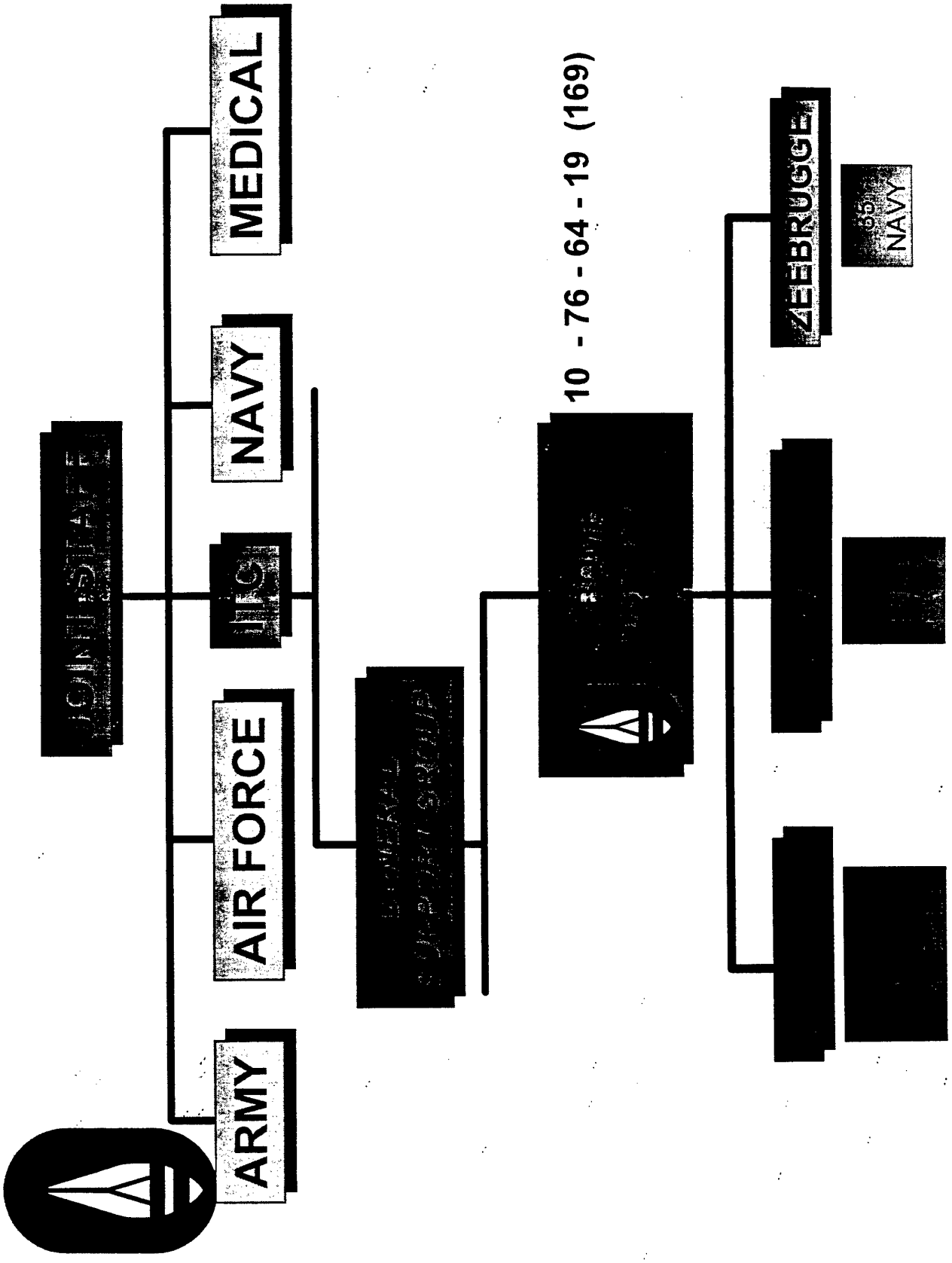
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CONTENT

- Dependence, organisation and main tasks
- Toxic munitions : history
- The dismantling installation
  - The identification process
  - The dismantling process
- Safety

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MEDICAL
NAVY
AIR FORCE
ARMY
10 - 76 - 64 - 19 (169)
ZEEBRUGGE
NAVY

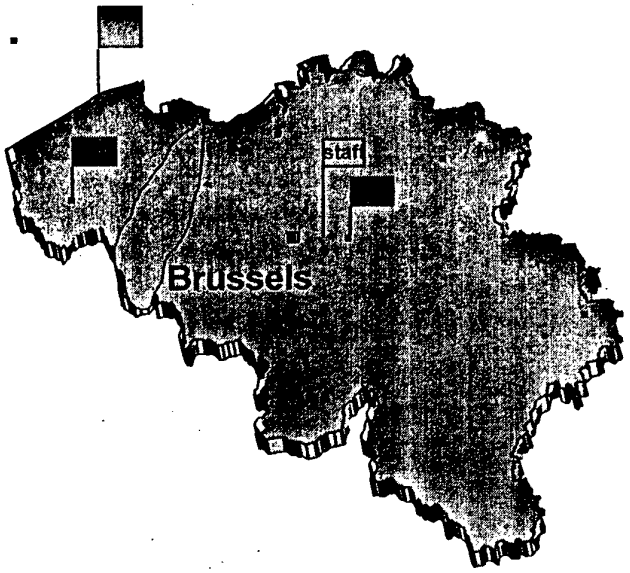


II

JMB DISPOSAL

10-76-64-19 = 169

- Staf — 7-22-11-5=45
- Det Meerdaal — 1-26-16-0=43
- Det Poelkapelle — 2-28-37-14=81
  - Intervention
  - [illegible]
- Det Navy — 3-24-8-0=35
  - NAVCLEARMIN
  - Diving School



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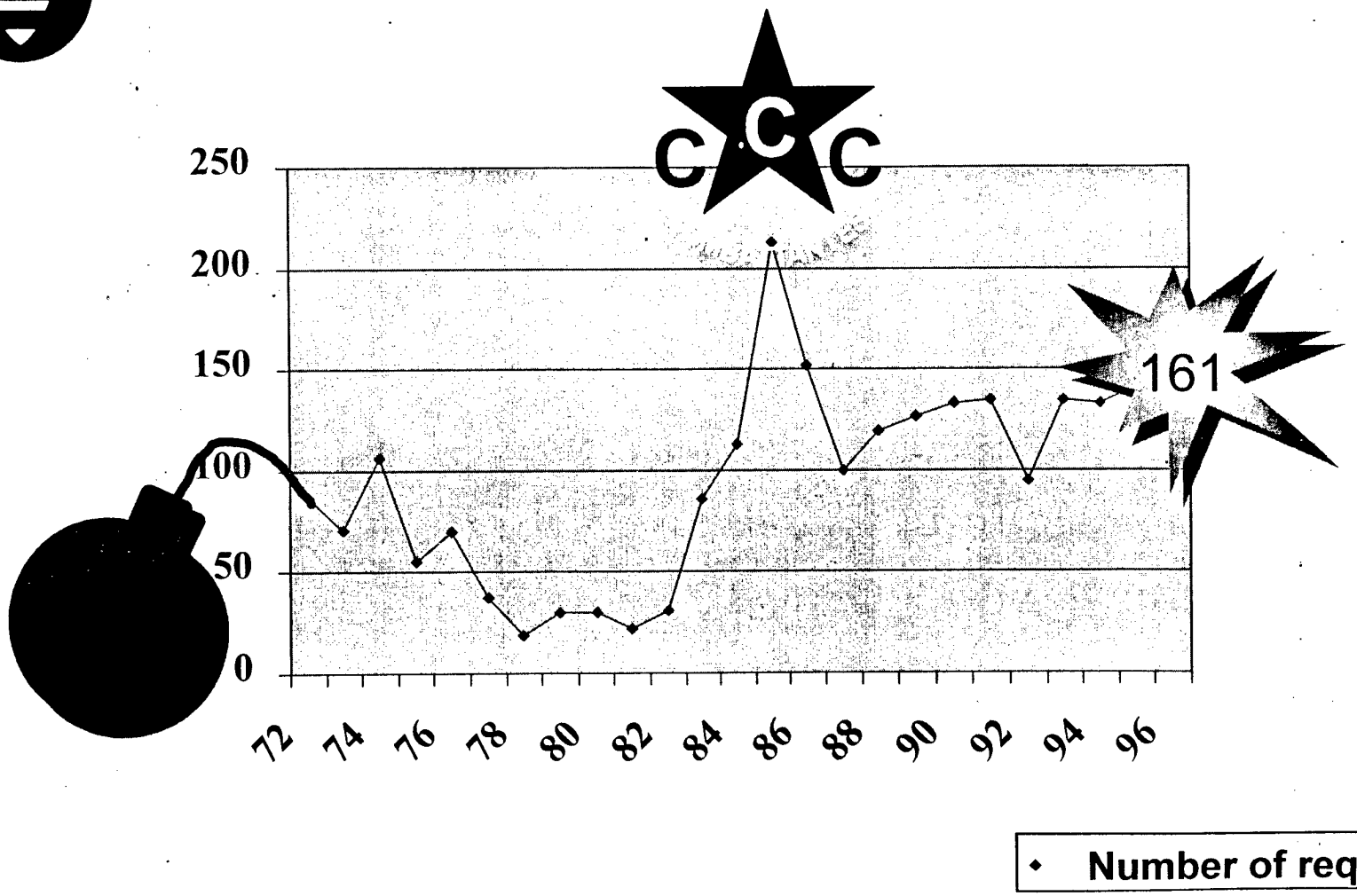
MAIN TASKS

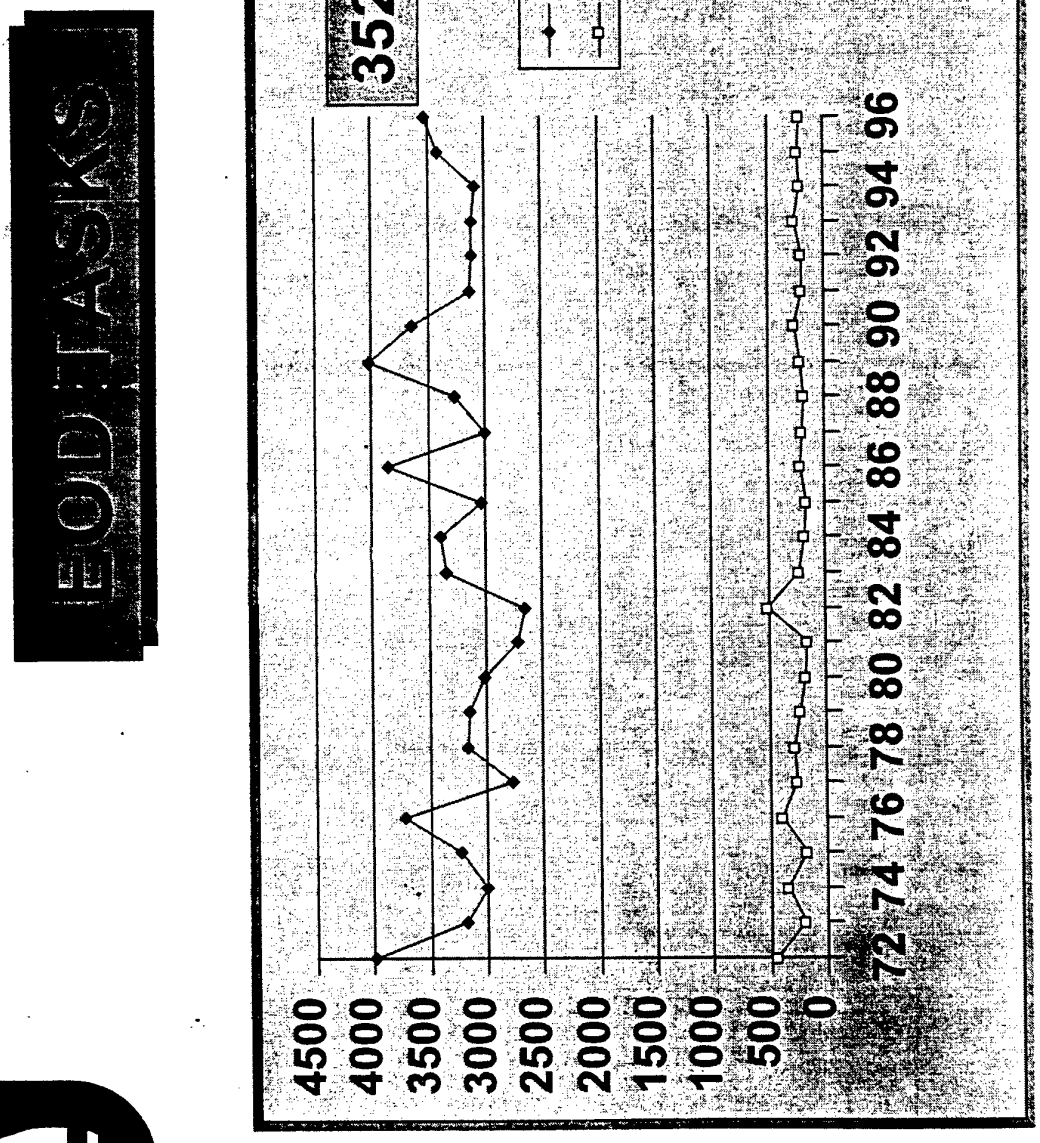
- TECHNICAL ASSISTANCE TO THE JURIDICAL AUTHORITIES (IEDD)
- TREATMENT OF MUNITIONS FROM BOTH WW DISPOSAL OF OBSOLETE MUNITIONS (EOD)
- THE DISMANTLING OF THE TOXIC CHEMICAL AMMUNITION OF WW 1
- INSTRUCTION & TRAINING
- HUMANITARIAN OPERATIONS

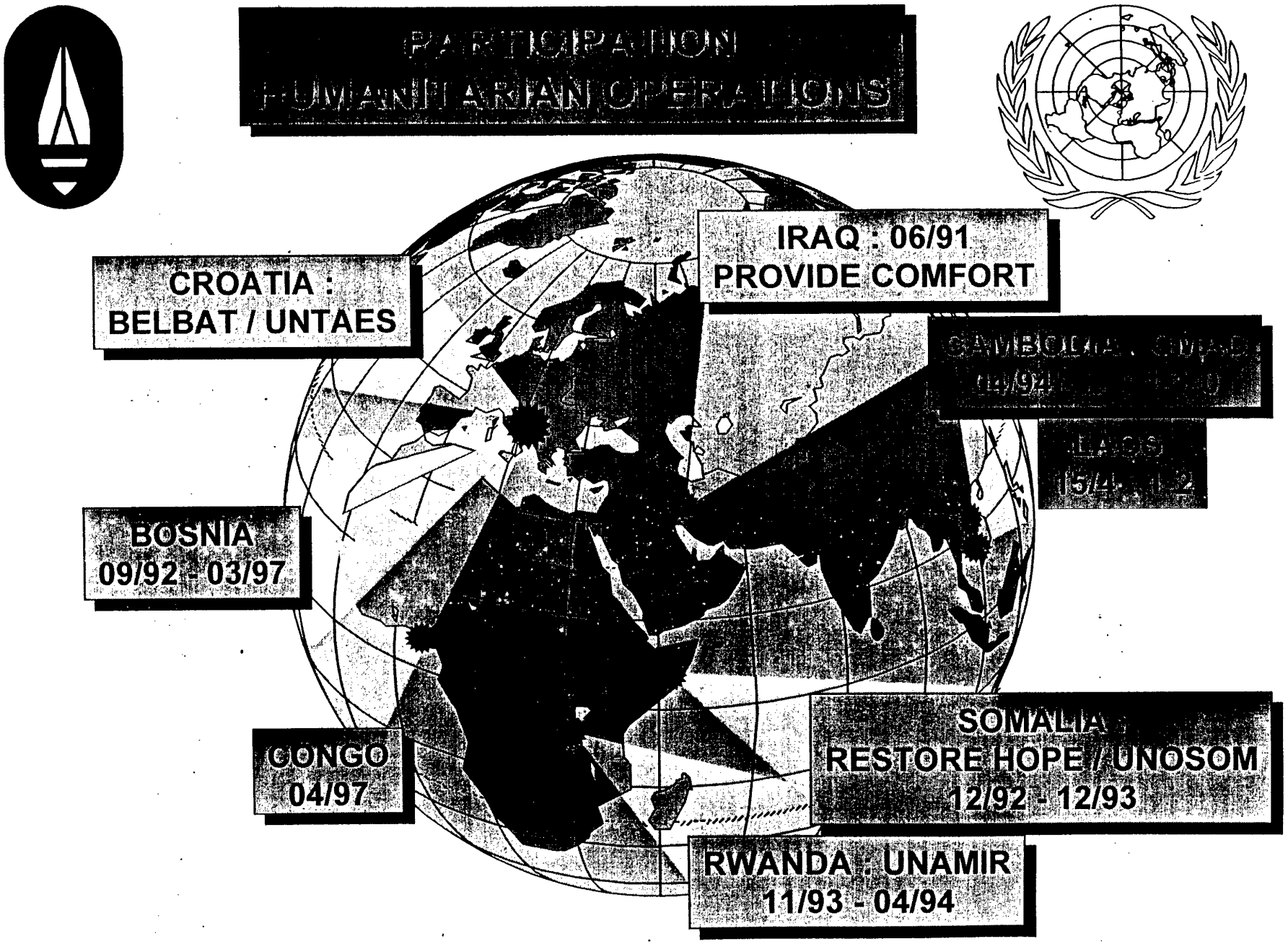
IEDD : IMPROVISED EXPLOSIVE DEVICE DISPOSAL
EOD : EXPLOSIVE ORDNANCE DISPOSAL

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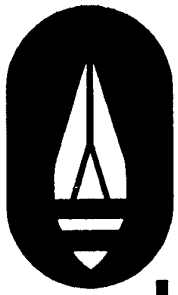
HDD TASKS


C C C
250
200
150
100
50
0
161
72
74
76
78
80
82
84
86
88
90
92
94
96
Number of requests


EOD TASKS
3525
Number of requests
Tonnage
4500
4000
3500
3000
2500
2000
1500
1000
500
0
72 74 76 78 80 82 84 86 88 90 92 94 96



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HISTORY

- **History**
  - 72 : Dumping in the Gulf of GASCOGNE
  - 80 : Emergency situation (last dumping)
  - 89 : Governmental decision - Dismantling plant
  - 92 : Protocol MOD - Ministry of Environment
  - 93 : Construction start at POELKAPELLE
  - 96 : Completion of the building
  - 97 : Start up identification
  - 98 : Start up dismantling
- **WHERE ? principally in the WESTHOEK**
- **Yearly amount : ± 17 TONS**

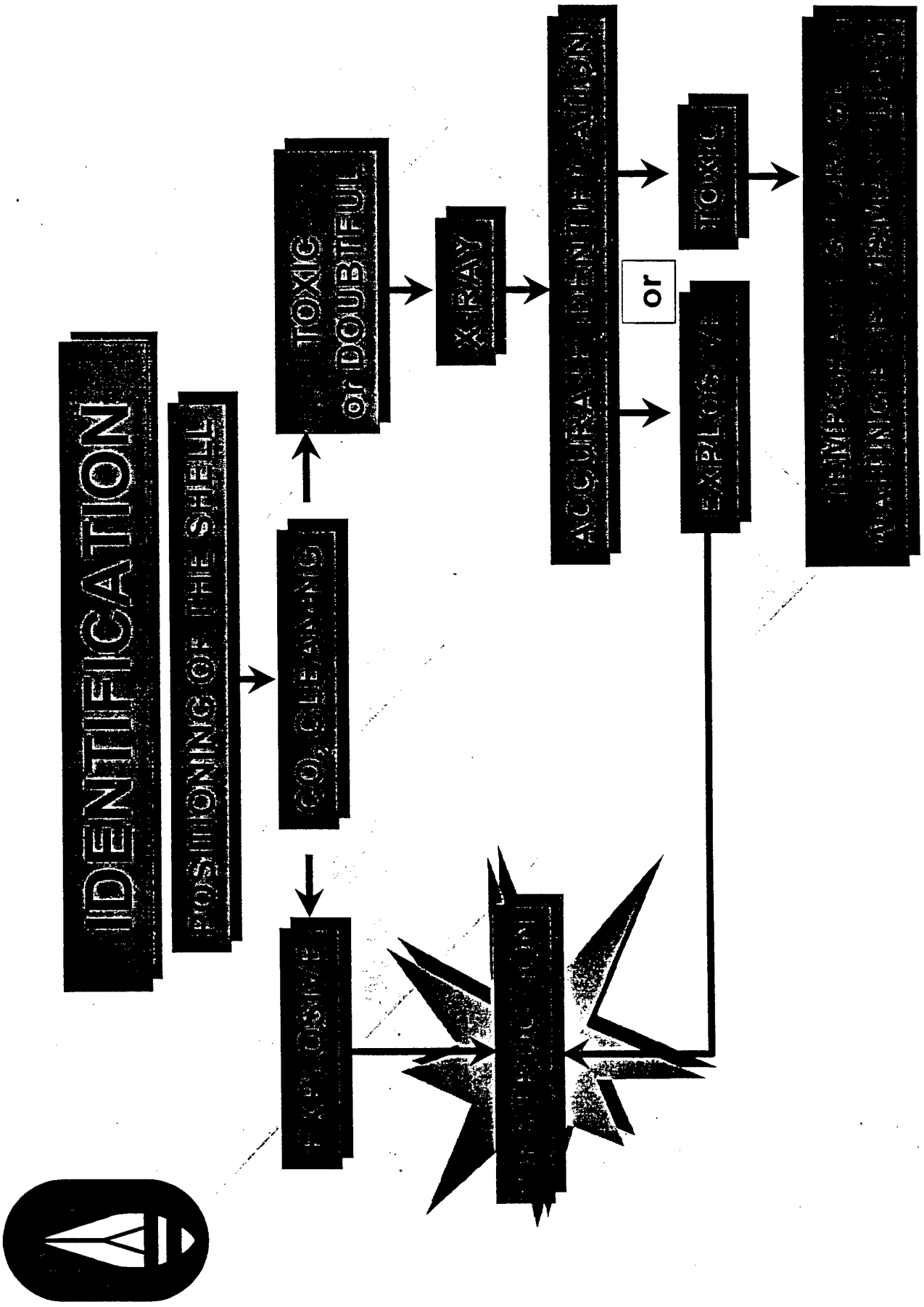
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STATISTICS
Fired rounds in 1914-18

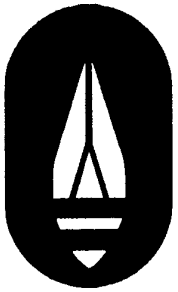
LAND	TOX MUN		OTHER MUN		TOTAL	
	Number (millions)	%	Number (millions)	%	Number (millions)	%
GERMANY	33	6,4	485	93,6	518	35,6
FRANCE	16	4,6	334	95,4	350	24,1
GREAT-BRITAIN	4	2,2	178	97,8	182	12,5
US	1	12,5	7	87,5	8	0,5
RUSSIA	3	4,2	69	95,8	72	4,9
AUSTRIA	5	2,9	170	97,1	175	12,0
ITALY	4	2,7	146	97,3	150	10,3
TOTAL	66	4,5	1.389	95,5	1.455	100,0

Reference : "Chemical Warfare", January 1935 of A.M. PRENTISS

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IDENTIFICATION
POSITIONING OF THE SHELL
TOXIC or DOUBTFUL
X-RAY
or

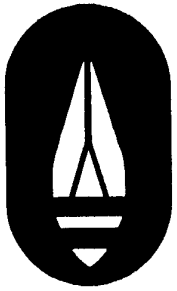
LIFE DISMANTLING PROCESS
SOLID
TREPANNING
EXPLOSIVE
TOXIC
LIQUID
DRILLING
TOXIC
EXPLOSIVE
LAB
TEMPORARY STORAGE
TRANSPORT
DECONTAMINATION
DESTRUCTION
DECONTAMINATION



THE SAFETY

- Explosion risks
- Fire risks
- Contamination risks
- Protection of the personnel

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THE SAFETY
Explosion and fire risks

- Anti-blast or blow-away walls
- Protective wall
- Explosion detectors and watercurtain
- One round at the time
- Fire detection & fire fighting systems
- Fireproof wall

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THE SAFETY : Contamination risks

- Chemical detection
- Audible alarm system
- Filtering 24Hr a day
- Underpressure of specific rooms
- Choice of materials
- Periodic decontamination
- Waste administration
- Weather & prediction station

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THE SAFETY : Protection of the personnel

- Minimum of personnel in risk area
- Protective clothing
- Buddy in protective clothing
- Surveillance camera
- Interlocks & automated process
- Emergency plan (Plan Yperite)

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ELIMINATION OF OLD CHEMICAL WEAPONS IN FRANCE

Bruno Despagne

Ministère de la Défense
DGA/DSP/SPNuc/SDAN
00460 Paris-Armées, France

Eighty years after World War I one still finds Old Chemical Weapons (OCW) in France. These weapons are collected by farmers during working in the fields, or are discovered during civilian construction activities, e. g. during construction of new roads or tracks for the high speed railway system. The majority of these ammunitions had been fired, but had not exploded (the reason why they are always present). The average quantity ranges between 20 and 50 tons (of the total collected quantity about 10% are chemical).
Evolution of environmental regulations prohibits dumping of ammunition into the sea (since 1990). With regard to this French national problem, there is a work sharing between the Ministry of the Interior (collection, identification, and transportation) and the Ministry of Defence (destruction). We need to solve two main problems:

- the first one is to treat on site a limited number of very corroded ammunition which cannot be transported,
- and the second one is to build a specific plant to eliminate the totality of chemical weapons containing different chemical compounds (smokes, obscurants, incendiary mixtures, vesicants, etc.).

The "Délégation Générale pour l'Armement" (DGA) of the Ministry of Defence is in charge to manage preliminary conceptual studies. After an industrial consultation phase we have selected three main competitors: Aérospatiale, Thomson, and SNPE. The purpose of this preliminary phase is to identify and to evaluate the process and to select available technologies to elaborate a technical and financial proposal for the construction of a plant and which includes a statement of the operating costs.

It seems that thermal treatment is the only process available to eliminate pyrotechnic materials, toxic compounds, and contaminated metal parts. Dismantling of recovered ammunition is a difficult, time-consuming, and hazardous process. The majority of OCW containing less than 100 g of explosive material can be detonated directly. The three competing industrial companies have got 11 to 12 months to provide the DGA with an optimized estimate of the overall costs taking into account the necessary safety requirements.

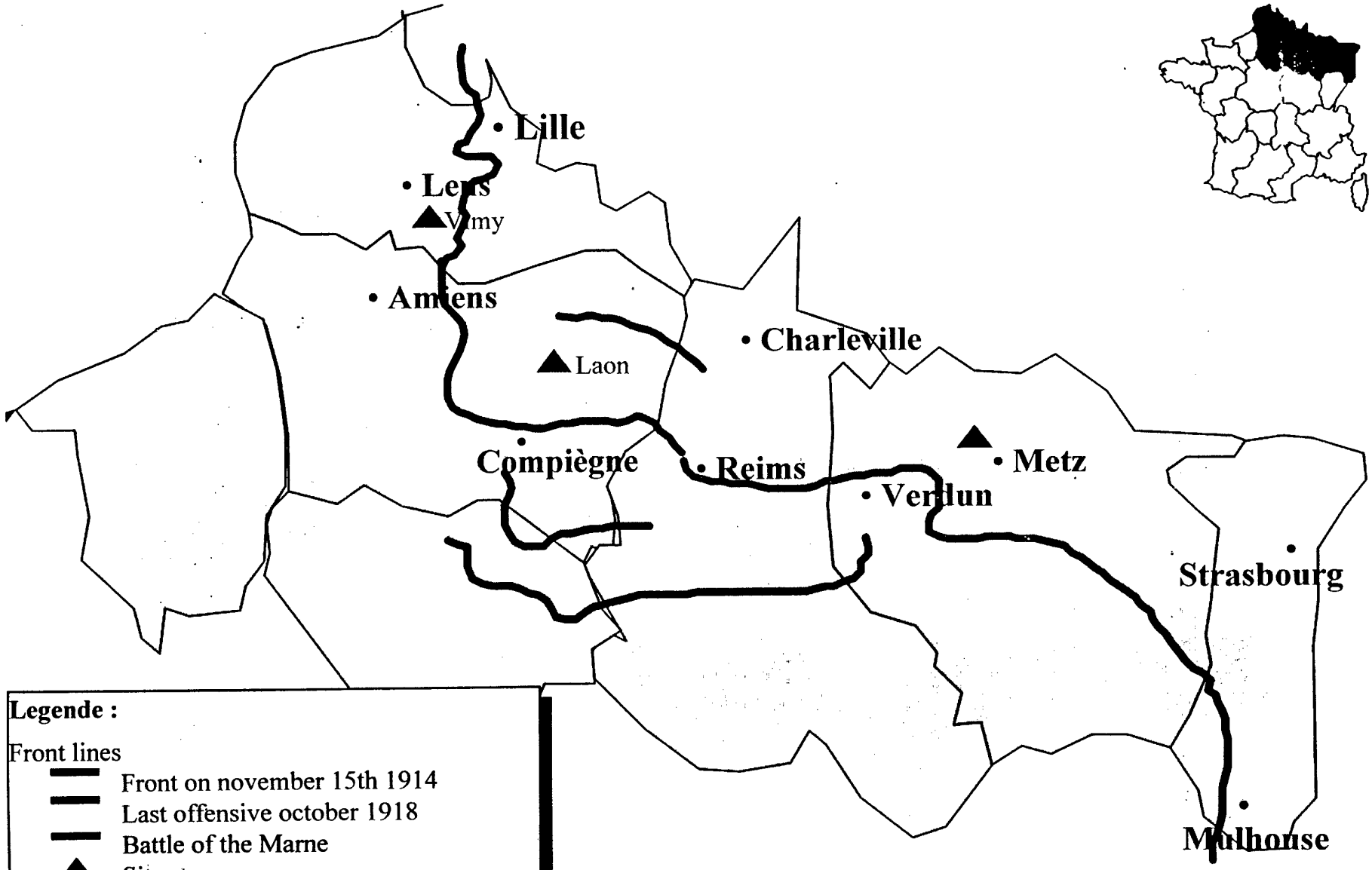
Elimination of old chemical weapons

(French program : SECOIA*)

* Site pour l'Elimination des Chargements d'Objets Identifiés Anciens
* Site for Elimination of Eldest Identified Loaded Objects ; SEEILO

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BATTLEFIELD DURING 1914 - 1918



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GENERAL CONSIDERATIONS 1/2

- **World war 1 reminder**
  - Theather of operations NORTHERN and EASTERN PARTS OF FRANCE
  - About 60 millions of chemical toxic munition fired*
  - Out of wich 10 to 15% are not exploded

* PRENTISS

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GENERAL CONSIDERATIONS 2/2

- **EXISTING STORAGE : 146 T (about14 800 munitions)**
- **ANNUAL DISCOVERY : 50 T**

  (10% of total discovery)
- **TO THESE MUNITIONS IT IS TO BE ADDED 3 or 5%**
  - Smoke
  - Flare
  - Incendiary

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DISTRIBUTION OF MUNITION COLLECTED IN FRANCE

■ BY ORIGIN

– France	30%
– Great britain	10%
– Germany	60%

■ REPARTITION IN WEIGHT

– Artillery shells	90% (7 to 120 kg)
– Trenches munitions and mortar bombs)	10% (20 to 60 kg)

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STATUS

- **France must respect international agreement (CIAC) and national arrangement (according to the french law the state is responsable for it)**
- **Worksharing between :**
  - ministry for inner affairs (collect, identification, transport)
  - ministry of defence (destruction)

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ORIENTATIONS

- MOD has decided to operate in complet "transparency"
- Steering committee created
- Public inquiry : authorization to operate (European standard for environmental regulation
- Délégation générale pour l'armement(DGA) manage the programm for MOD
- Operating phase conducted by civil manufacturers

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DGA SECOIA PROGRAM

- **3 main phases**
  - preliminary conception
  - complete conception and realization
  - operating phase
- **Time table**
  - 04/1998 Notification contracts (with 3 competitors)
  - 07/1999 Beginning of complete conception (with 1 competitor)
  - 12/2000 Starting of construction phase
  - 04/2002 Reception test
  - 10/2002 Operating phase

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PRILIMINARY CONCEPTION STUDY 1/2

GOAL

- Inventory of process, selection of one (or more) process and elaborate a technical and financial proposal for a construction and operating costs

CONSTRAINTS

- Do not dissociate the responsability of conception and realization, we search a prime contractor capable of guaranteeing the operating phase

- National operator is preferable to adjust french reglementation and assume the delegation of state responsability

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PRILIMINARY CONCEPTION STUDY 2/2

MAINS SPECIFICATIONS

- Munitions fired and non exploded
- List and repartition of weapons to eliminate
- List of toxic compounds (more than 40 products)
- Capability about 100 tons a years (50-60 ammunitions a day)
- Elimination of stockpile after 3 or 4 operating years

MOST IMPORTANT FUNCTIONS

- Reception - identification
- Dismantling and destruction
- Treatment of destruction products

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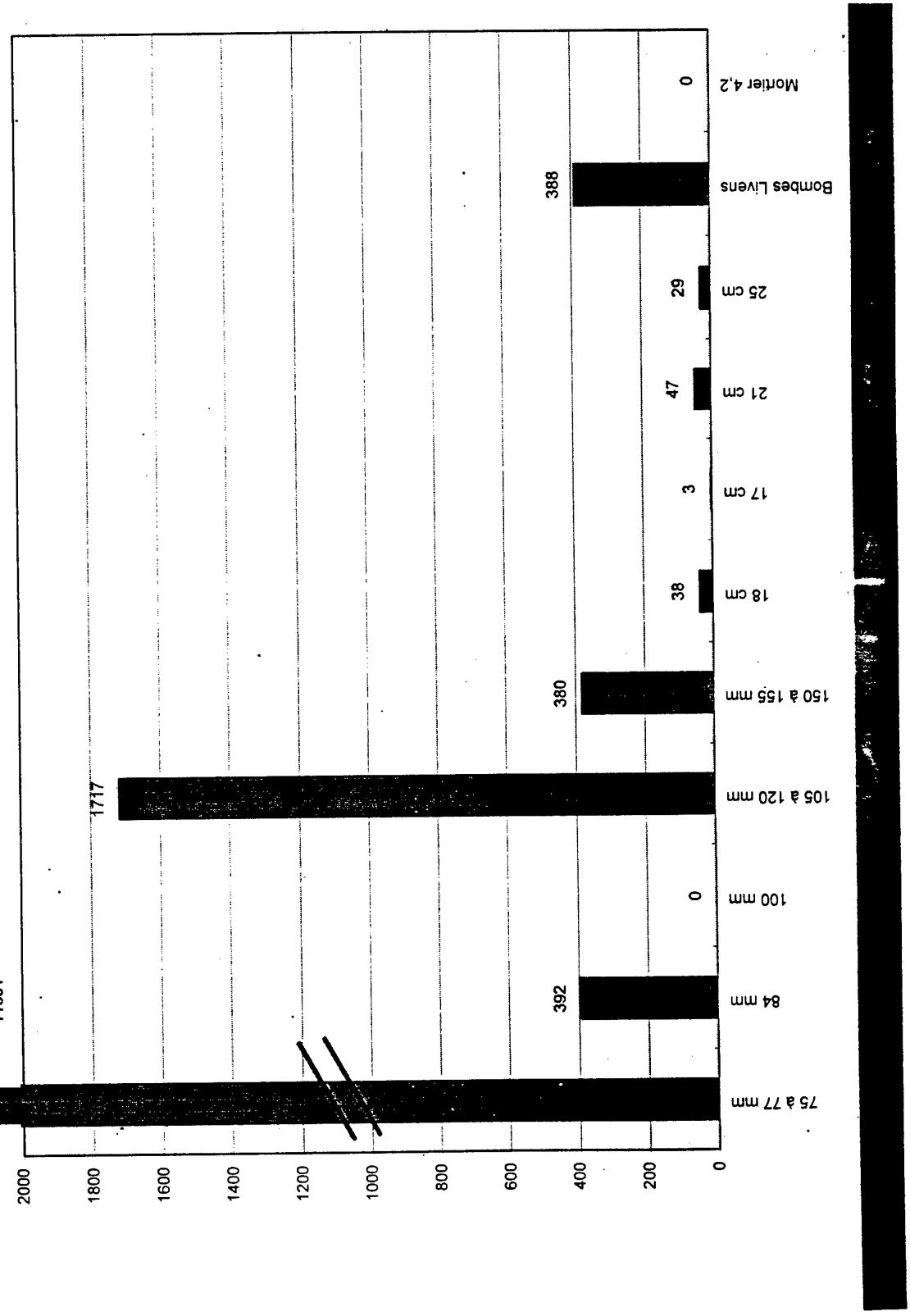
DISTRIBUTION OF MAIN CHEMICAL AGENTS*

Phosgène	[$COCl_2$]	32%
Disphogène	[$CCl_3\ CO_2Cl$]	20%
Ypérite	[$S(CH_2\text{-}CH_2\text{-}Cl)_2$]	18%
Chloropicrine	[$CCl_3\ NO_2$]	12%
Vincenite	[HCN]	7%
Diphenylarsine	[$(C_6H_5)2$ - As - X]	6%
Others		5%

(* 48 chemical compounds)

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REPARTITION IN NUMBER VERSUS DIAMETER



CONSULTATION PHASE

06/1997 publication "asking for candidature" (10 answers)

07/1997 6 compagnies selected for consultation

10/1997 5 answers

11/1997 3 consortiums selected

- Aérospatiale + Tredi
- Thomson + Teris
- SNPE + SNPE Ingenierie

01/1998 End of negociation (technical specification/price)

04/1998 Contracts expected

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MAIN ORIENTATIONS TAKEN IN ACCOUNT

- Pyrotechnics hazards require a sure identification
- Automatisation is required for safety conditions
- It seems that thermal treatment is the only process available to eliminate : pyrotechnic, toxic compounds and poluted metals
- Alternative technologies must be analysed to justify the selected process
- 60% to 70% of "OCW" containing less than 100 g of explosive material do not necessite a dismantling
- Ammunitions with high explosive material (8 kg) need a specific line for treatment

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CONCLUSIONS

The industrialists have 11 - 12 months to provide at DGA the best "possession cost" regarding the safety conditions

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DESTRUCTION OF CHEMICAL WARFARE AGENTS WITH SOLVATED ELECTRON TECHNOLOGY

MG(ret.) Gerald G. Watson and COL(ret.) Richard W. Smith
Presented by Larry S. Maupin

Teledyne-Commodore, MS-68, Huntsville, AL 35807-7007, U.S.A.

Solvated electron, or reducing metal, solutions are powerful reducing agents, capable of reacting with chemical weapons agents, explosives, and propellants, to produce less hazardous organic and inorganic products. These solutions are produced by dissolving sodium in liquid anhydrous ammonia. Existing stewardship programmes for these very common industrial chemicals assure operations safety and reliability. This solvated electron technology has been patented as the SETTM process. A pilot plant is currently in operation for the destruction of nitrate explosives and propellants, and a production-scale munitions-disposal facility is in develop-ment.
The reaction is controlled by the rate of addition of metal reactant or agent. In laboratory tests with chemical weapons agents and with energetic materials used in chemical weapons, treatment has resulted in destruction of at least 99.9999% of the chemical agents HD, HT, L, HL, GA, GB, GD, and VX; and at least 99.9995% of the explosives TNT, hexogen (RDX), nitroglycerine (NG), tetryl (CE), and picric acid (PA), individually and in conventional military mixed explosive compositions. Further treatment of the SET residues by chemical oxidation in water solution results in products which contain con CWC Schedule 1 or 2 materials and which pass U.S. regulatory requirements for safe disposal. Stabilization of the arsenic product from Lewisite in a form safe for disposal has been demonstrated.



Use of Solvated Electrons for the Reduction of Chemical Agents and Explosives

Larry S. Maupin
Teledyne-Commodore
300 Sparkman Drive, Mail Stop 30
Huntsville, Alabama 35805

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PROPRIETARY



What is Solvated Electron Technology?

- **Solvated Electron Solutions**
  - Na, Ca, K dissolved in ammonia
  - Valence electron dissociates
- **Highly Effective Reducing Environment**
  - Destroys labile covalent bonds
  - All halogen bonds
  - Many S, N, P, As bonds
- **Conventional Wet Chemistry**
  - 0 - 140 psi liquid, -33 C to room temperature

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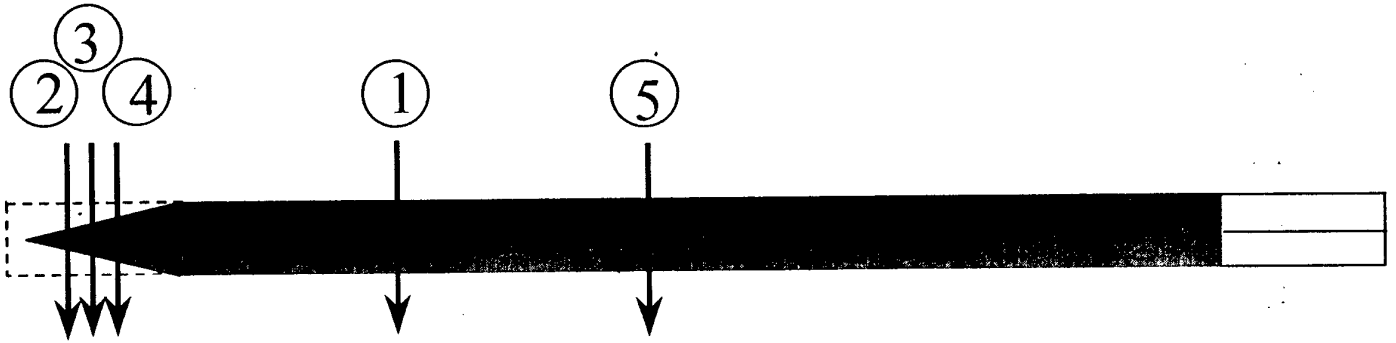
Access Subsystem Concept Overview

- High pressure fluid jet is used to cut components and washout chemical agent and explosives.
- Utilizes up to 50 ksi fluid to safety cut through steel and aluminum with speed.
  - Cuts through interrupted cuts and mixed materials with ease. Cuts M55 rockets while nested in their shipping and firing tubes.

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Access Subsystem Concept Overview



M55 Rocket Demilitarization Process

1. Pierce warhead and drain agent.
2. Section M417 fuze and washout booster.
3. Cut off M417 fuze and washout M34/M36 bursters.
4. Cut off burster well tubesheet and washout warhead.
5. Cut off warhead aft of bulkhead and washout rocket motor.

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Access Subsystem Concept Overview

- **Why fluid jets?**
  - Safest known system for sectioning HE projectiles.
  - Substantial safety margin for all secondary explosives.
  - In use for over six years in commercial and government installation conventional weapons demilitarization without event.

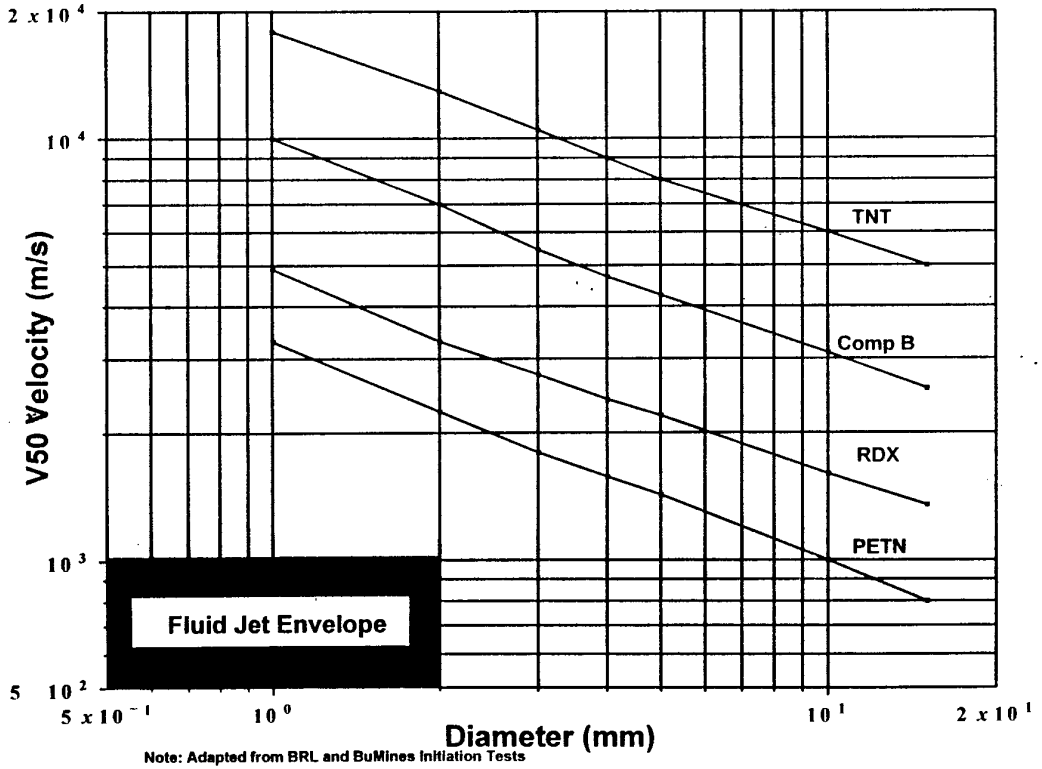
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Access Subsystem Concept Overview

Explosive Impact Initiation Probabilities

Impact Diameter vs. V50



Note: Adapted from BRL and BuMines Initiation Tests

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Status of Technology

- Commercial PCB, CFC Destruction
- EPA National Authorizations
  - PCB - contaminated soils
  - PCB - contaminated oils
- Rapid Commercialization Initiative
  - White House Approved
  - DoD Sponsored

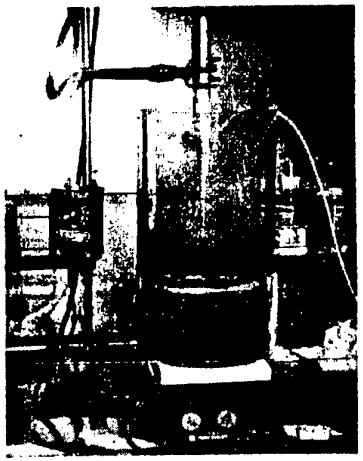
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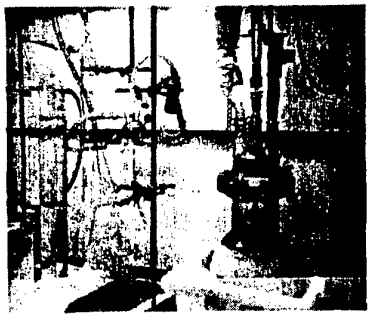
Experiments Carried Out In Multiple Reaction Systems



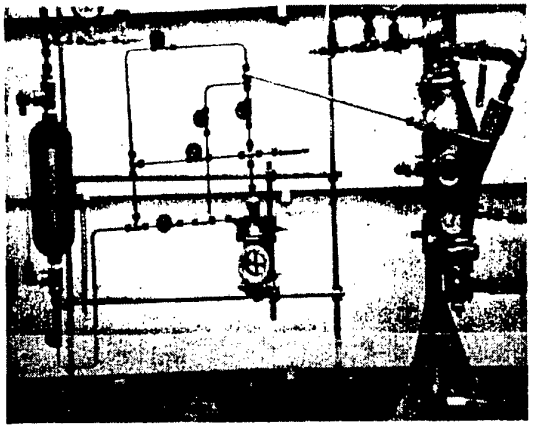
500 ml Flask



6 L Reactor



5 L Closed Reaction Flask



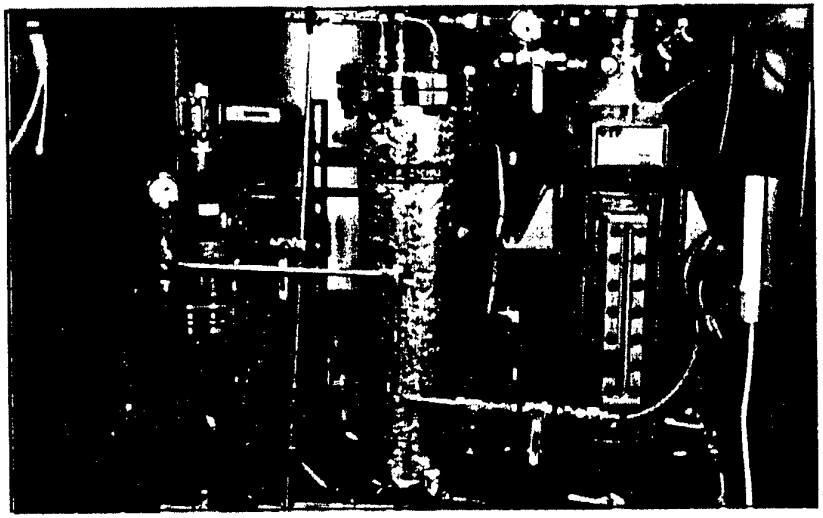
2.2 L Pressure Reactor

24.06.98Teledyne-Commodore

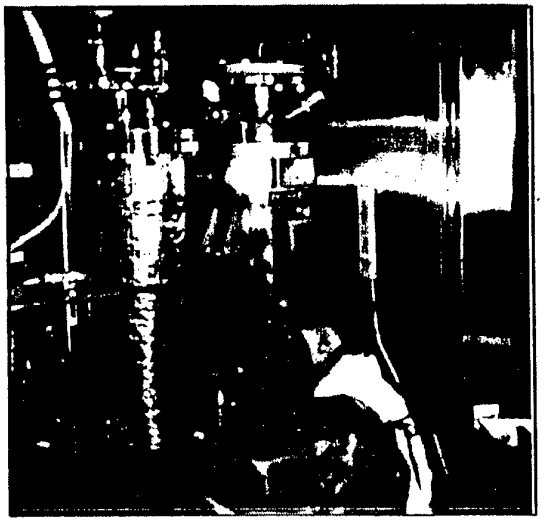
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One Pound Apparatus



Agent Destruction System



Operation

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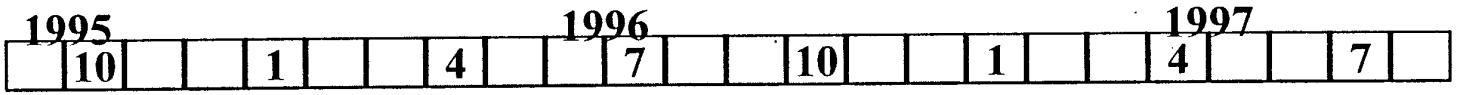
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Agent Testing

Initial Screening
- HD, GB, VX, L
- ~ 0.1 gm
- Verified Kills

Concept Refinement
- HD, GB, VX, L +
- ~ 10 - 100 gm
- Reproducability

Oxidative Treatment
- HD, GB, VX, L
- ~ 10-25 gm
- No Schedule 2 Mat'ls



Scaling Demo
- HD, VX,
- ~ 20 gm
- Verified Kills

Scale-Up
- HD, GB, VX, L
- ~ 1 lb (400 - 650 gm)
- Verified Kills
- Batched Operation
- 100 lb Malathion

Process Definition
- HD, HT, GB, VX, L
- ~ 10 - 40 gms
- No agent in offgas

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Total Quantity of Chemical Agents Destroyed

Agent	Number of Tests	Largest Single Quantity (gm)	Total Quantity of Agent Tested (gm)
HD	22	636.1	987
T	2	23.0	31
HT	7	491.0	510
H (heel)	12	254	370
VX	14	480	908
GB	11	571	886
GA	2	6.9	7.0
GD	3	10.0	20.0
GF	1	20.0	20.0
L	25	314.0	602.3
HL	4	7.0	29
HN-1	1	6.0	6.0
HN-3	1	6.0	6.0
CG	2	1.8	3.5
CK	2	13	16
Malathion	2	45 Kg	90 Kg

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SET Destruction Efficiency

Agent	Quantity (Largest Test) (lb)	Residual Agents	SET Destruction Efficiency (%)
HD	1.4	ND	> 99.9999
HT	1.1	ND	> 99.9999
H (heel)	0.6	ND	> 99.9999
VX	1.1	ND	> 99.99999
GB	1.3	ND	> 99.99999
Malathion	100.0	ND	> 99.9999
L	0.7	ND	> 99.9999

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Reaction Product Toxicity*

Agent	Toxicity	
HD	Class	0
VX	Class	0
GB	Class	1
L	Class	1

* Results are for SET Reaction followed by Oxidation

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Material Balances for Agent* (Basis 1 lb Agent)

Starting Material / Products	Agent HD	Agent H	Agent HT	Agent GB	Agent VX
Feed Material					
Agent	1.000	1.000	1.000	1.000	1.000
Sodium Metal	0.665	0.665	0.665	0.411	0.198
Sodium Hydroxide, 50% sol	0.677	0.677	0.677	3.770	5.558
Sodium Persulfate, 50% sol	5.798	5.798	5.798	8.670	12.122
Solid and / or Liquid Products					
Sodium Chloride	0.763	0.763	0.763		
Sodium Sulfate	3.474	3.474	3.474	5.173	7.765
Sulfur (S8)	0.205	0.205	0.205		
Sodium Fluoride				0.300	
Sodium Phosphate				1.171	1.075
Isopropanol				0.046	
Ethanol					0.017
Organic Compounds					0.448
Gaseous Products					
Hydrogen	0.002	0.002	0.002	0.018	
Methane				0.065	0.060
Ethylene	0.321	0.321	0.321		
Ethane	0.034	0.034	0.034		0.101
Propane	0.001	0.001	0.001	0.280	

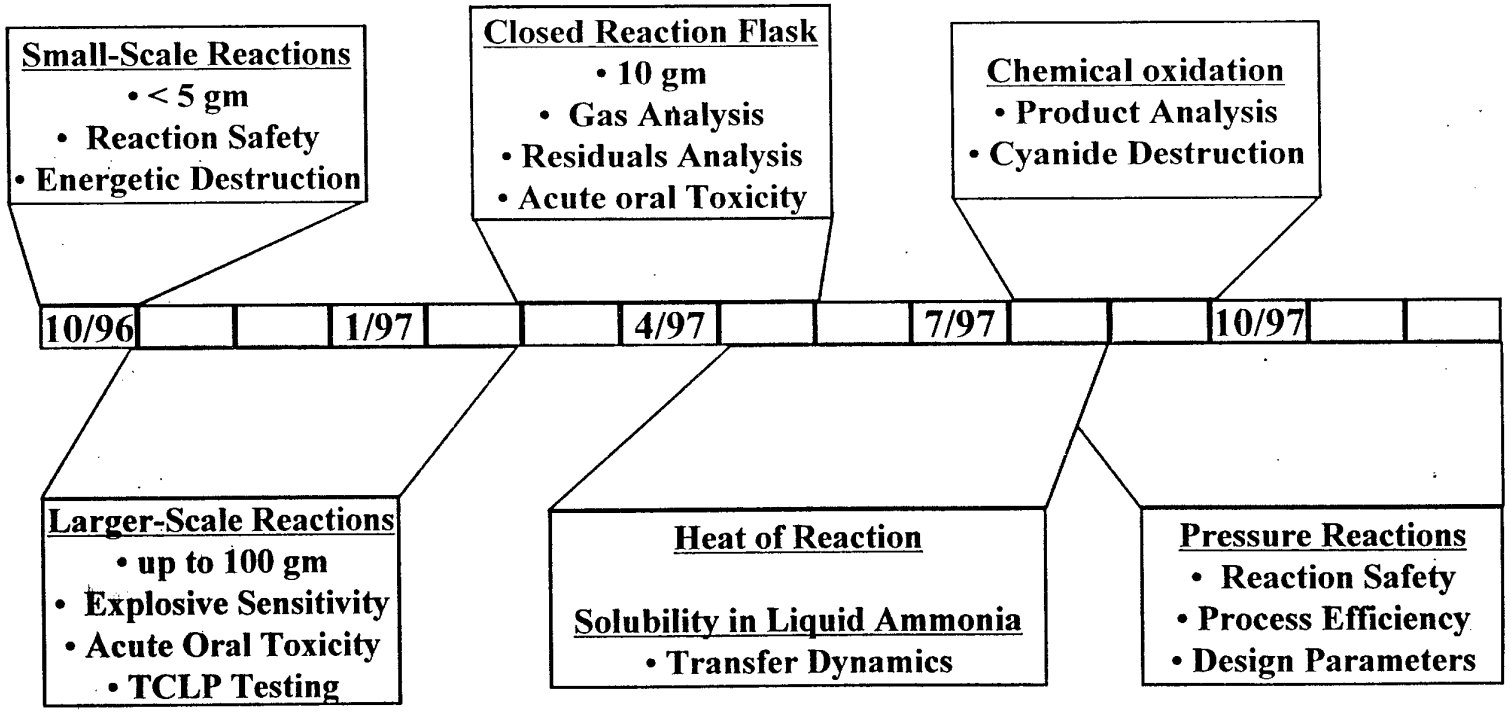
* Masses are exclusive of Process and Reaction Water

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Energetics Testing



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Explosive Sensitivity

- Impact Sensitivity
- Friction Sensitivity
- Thermal Stability
- Electrostatic Sensitivity
- Ignition and Deflagration

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Destruction Efficiency

Energetic	Energetic Concentration (%)	Sodium Concentration (%)	Final Energetic Concentration (mg/kg)	Destruction Efficiency (%)
TNT	1.5	0.83	< 2	99.999
M-28	1.5	0.86	< 6.25 (NG)	99.991
RDX	1.5	0.77	< 5	99.999
Tetryl	1.5	0.39	< 5	99.999
Comp B	1.5	1.42	< 8.9 (TNT)	99.994

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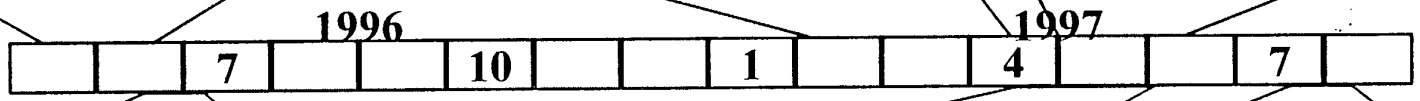
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Metal Parts and Dunnage Testing

Metal Parts Screening
- -33°C, open beaker
- HD, VX, GB
- Metals decontaminated

Charcoal Process Definition
- -33°C, open beaker
- HD, VX, GB
- Defined Parameters

PPE Decontamination
- 20°C, pressure rxn
- HD, VX, GB
- Successful with plastics dissolved in DMF



Charcoal Screening
- -33°C, open beaker
- HD, VX, GB
- Charcoal decontaminated

ACWA Mat'ls Screening
- -33°C, open beaker
- HD, VX, GB
- Metals decontaminated
- Non-metals deconed
- Issues: HD, Rubber

Waste Oils Screening
- 20°C, pressure rxn
- HD, VX, GB (100 ppm)
- Successful decontamination

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Agent Destruction Efficiency

Material	Treatment (All tests at -33°F unless noted)	DE		
		HD(%)	GB(%)	VX(%)
Wood (Pine)	Shred and treat as slurry	>99.999	> 99.99999	99.9999
Butyl Rubber	Shred and treat as slurry	>99.999	99.99999	99.99999
Silicon Rubber	Shred and treat as slurry	>99.999	99.99999	TBD
Fiberglass	Shred and treat as slurry	>99.999	99.99999	99.9997
Particle Board	Shred and treat as slurry	> 99.9998	> 99.99999	99.99999
Charcoal	Shred and treat as slurry	> 99.997	> 99.99997	> 99.99997
PPE (CPE/PVC)	Demonstrated: Dissolve in DMF and treat at 70°F Developmental: Treat shredded PPE	> 99.96	> 99.9999	> 99.9999
Hydraulic Fluid	DECON 100 PPM of Agent at 70°F	> 99	> 99.99	> 99.99
Waste Oils	DECON 100 PPM of Agent at 70°F	> 99	> 99.99	> 99.99
Carbon Steel	Treat pieces in sizes remaining after cutting oeration	99.99992%	>99.99999998%	99.9999999%
Stainless Steel	Treat pieces in sizes remaining after cutting oeration	99.99992%	99.9999999%	99.9999999%
Copper	Treat pieces in sizes remaining after cutting oeration	99.99997%	>99.99999998%	99.9999999%
Aluminum	Treat pieces in sizes remaining after cutting oeration	99.99994%	>99.99999998%	99.9999999%

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Process System Design Considerations

- Components Available from Commercial Sources
- Adaptations of "Off-the-shelf" Technology
  - Fuze Detonation From Iowa Army Ammunition Plant, Alliant Techsystems
  - Ammonia Fractionation Towers Similar to LPG Separation
  - Static Mixers used in Alt. Tech. Program Designs
  - Stirred Tank Reactors "Standard" for Storage or Reactions
- No Concerns For Material of Construction
  - Stainless Steel for Reactors/Fluid Jet
  - Carbon Steel for Ammonia Recovery

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SES Process Chemicals

- Ammonia
  - Standard Industrial Chemical and Refrigerant
  - Use Industry Procedures for Handling, Storage, and Worker Protection
  - Scrubbers Installed on HVAC Streams for Complete Removal of Ammonia
  - Standard Industrial Detection and Monitoring Equipment
  - Ammonia Operations Isolated and Entry Controlled

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Sodium

- Standard Industrial Chemical (Molten State)
- Reaction Hazard In Contact With Water
- Use DuPont Standard Procedures for Storage & Transfer, Worker Protection and Emergency Procedures
- Closed Feed System From Storage To Process
- Hot Oil For Heating

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Solvated Electron System (SES)

Summary

- SES is effective for agent and energetic destruction; metal parts and dunnage decontamination
- Reaction products and waste streams meet toxicity standards suitable for landfill
- Process is low temperature - non-thermal
- Process is mature, non-reversible with both production and scale design concept available
- Safety and environmental standards met
- Cost competitive

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SUPERCRITICAL WATER OXIDATION FOR CHEMICAL WARFARE AGENT DESTRUCTION

Louis D. Johnson

General Atomics, San Diego, CA 92186-9784, U.S.A.

Destruction of chemical agents has been successfully accomplished for many years using incineration. Environmental requirements and public perception in recent years have made incineration of chemical agents more difficult, and alternative methods of destruction have been investigated. Supercritical Water Oxidation (SCWO) technology has been recognized as an effective means of chemical agent destruction.

SCWO is a process that utilizes the unique properties of water at supercritical conditions (>374 °C and 22.1 MPa) to rapidly oxidize organic materials. Development of the process started in 1982, and significant effort has been applied in recent years to mature the process. Typical products from the process are carbon dioxide, water, and inorganic salts or acids. SCWO is now a compact, safe, and well-defined process that allows effluents to be stored for analysis and verification prior to release.

General Atomics has developed the SCWO process over the past six years and has demonstrated its successful application for the destruction of numerous hazardous materials in laboratory and pilot plant scale systems. Destruction of chemical agents was first demonstrated at bench scale in 1994. Destruction of a hydrolysate from a VX neutralization process was subsequently demonstrated at pilot plant scale in 1996. As a result, the U.S. Army has selected the SCWO process for treatment of VX hydrolysate at the Newport (Indiana) Chemical Agent Disposal Facility.

The characteristics of the SCWO process and the results of agent destruction tests are presented.

USE OF SUPERCRITICAL WATER OXIDATION FOR DISPOSAL OF CHEMICAL MUNITIONS

Chemical Weapons Destruction Symposium
Munster, Germany
March 1998

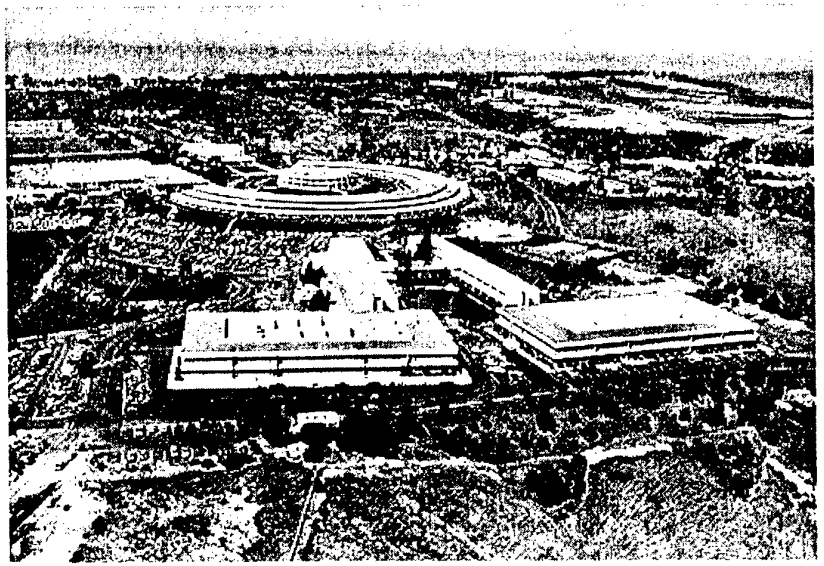
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GENERAL ATOMICS

LOCATION:	**San Diego, California**
FOUNDED:	**1955 by General Dynamics**
STATUS:	**Privately held corporation**
OWNERS:	**Neal and Linden Blue**
BUSINESS:	**High technology research, design, manufacturing, and production for industry and Government in the U.S. and overseas**



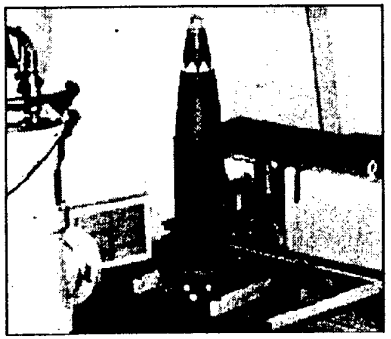
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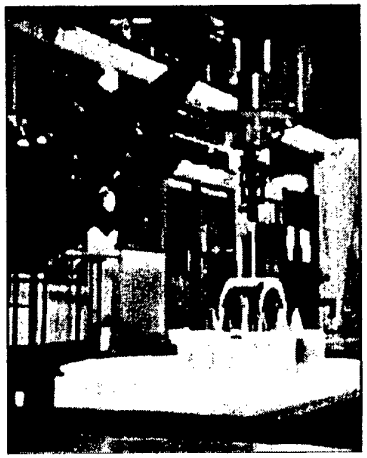
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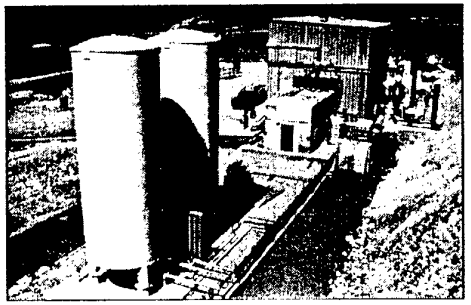
GA DEMILITARIZATION SYSTEM EXPERIENCE

Munitions Inspection Systems



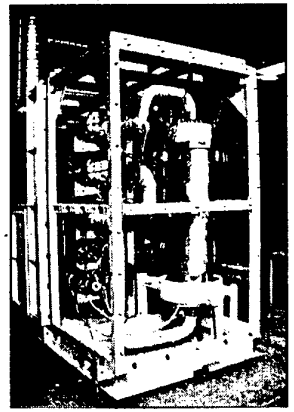
Cryofracture

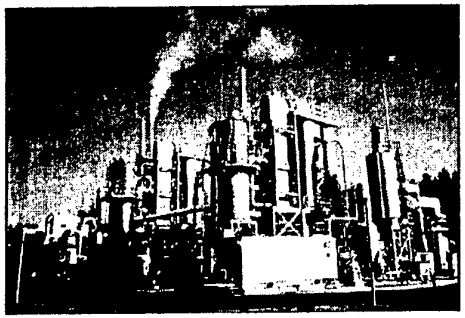




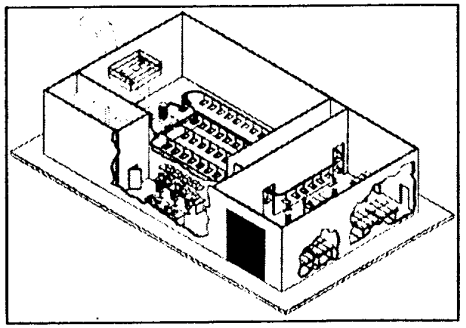
Solid Rocket Motor Demil

Supercritical Water Oxidation of Hydrolyzed Agent





Energetics Incineration



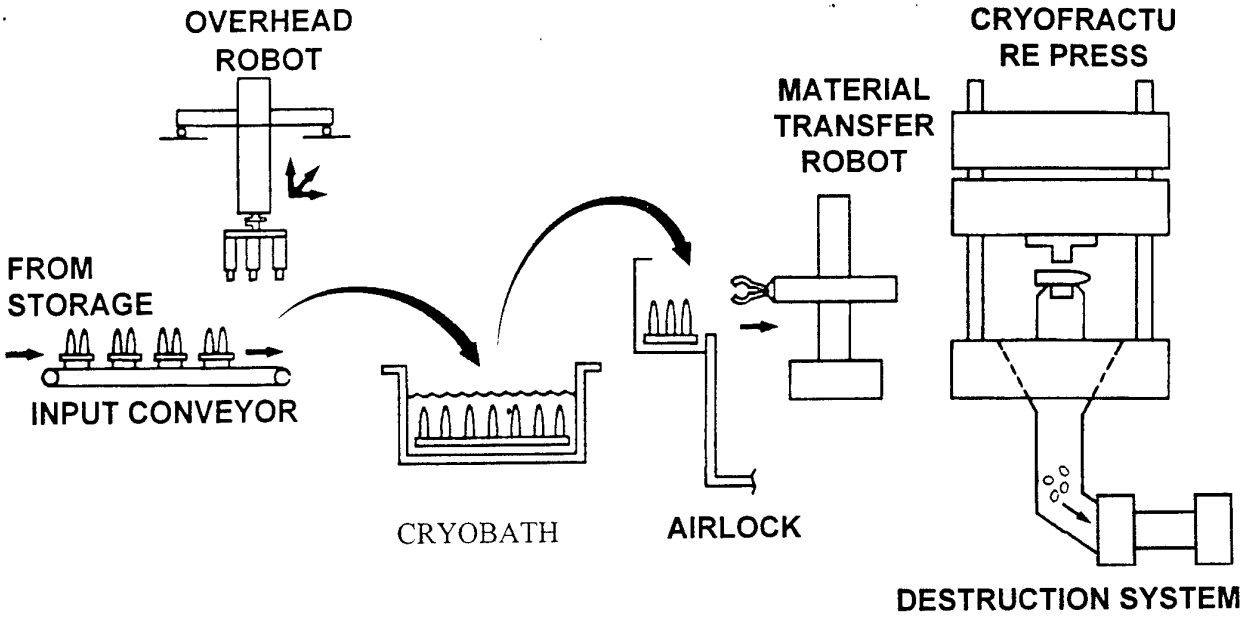
Munition Cryofracture Facility

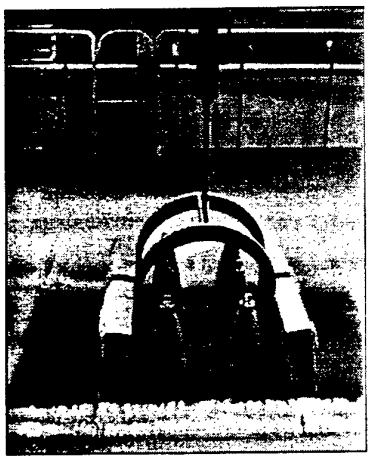
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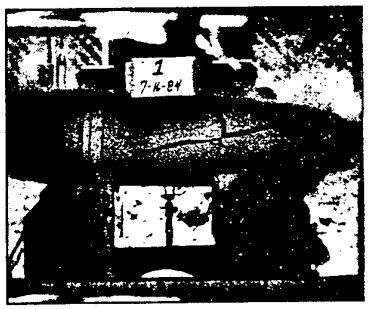
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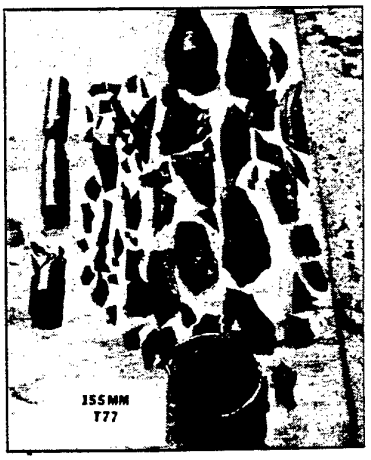
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MUNITION CRYOFRACTURE PROCESS







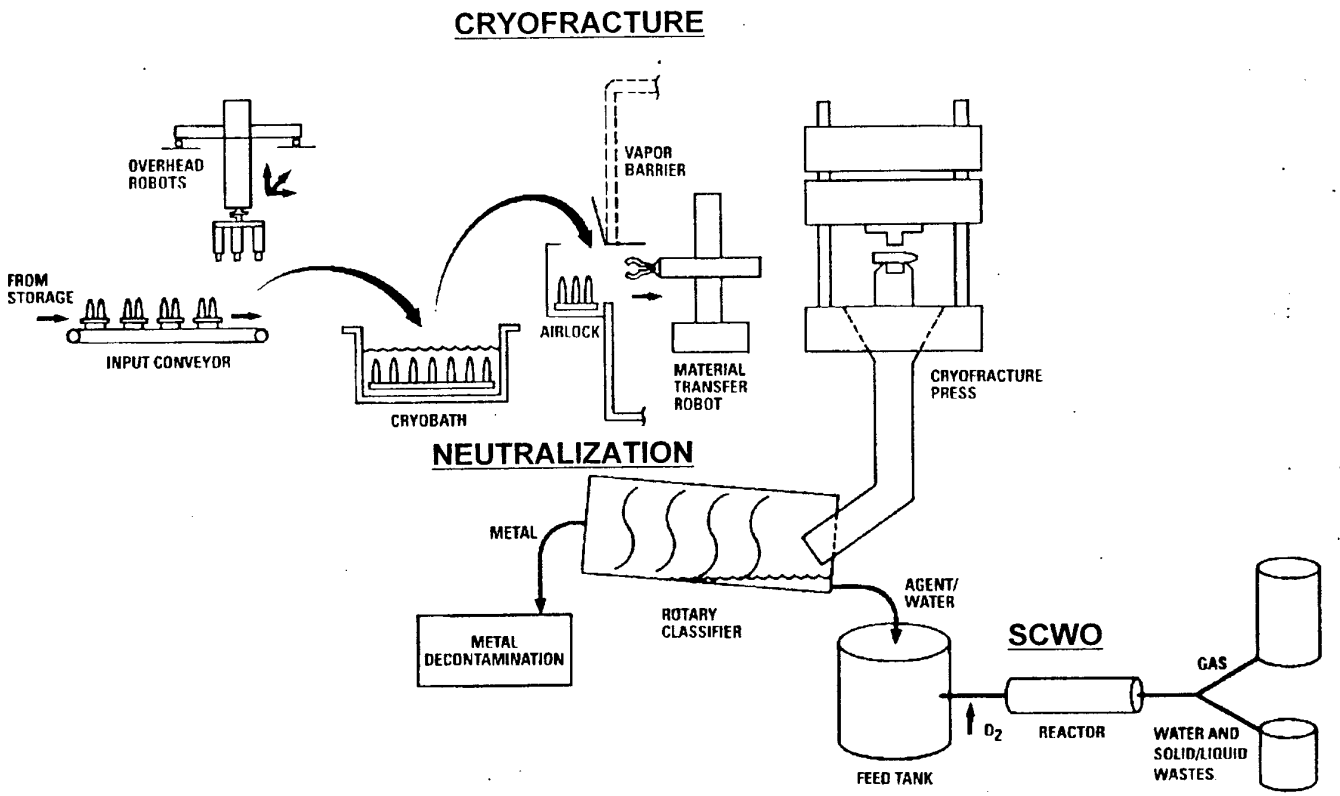


DESTROYS MUNITIONS EFFICIENTLY AND THOROUGHLY

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8-6-96

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ALTERNATE CRYOFRACTURE/ NEUTRALIZATION/SCWO CONCEPT



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8-6-96

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ADVANTAGES OF SCWO

- Excellent kinetics/DREs
- No airborne particulates
- Low NO_x, SOx, and TOC
- excellent process stability/control
- Simple safety measures/process upset recovery
- Capability for complete containment of effluents
- Compact equipment

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L-819(1)(a)
12-10-96

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MAJOR MILESTONES IN SCWO DEVELOPMENT AT GA

1992

- ARPA/ONR program focused on chemical warfare agents, energetics and other DoD wastes
- Joint Ordinance Commanders Group/U.S. Air Force (Armstrong Lab) program directed at solid rocket propellants

1995

- DARPA/ONR program aimed at shipboard treatment of Navy excess hazardous material
- Related DARPA/ONR program directed at heavy metals removal from SCWO effluent

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10-22-97

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MAJOR MILESTONES IN SCWO DEVELOPMENT AT GA

1995	• CRADA with Los Alamos National Lab for SCWO corrosion testing
1996	• Acquisition of MODAR, Inc. assets
1997	• Customer-funded treatment of municipal sludge ("biosolids") and industrial wastes
	• Gasification of biomass for U.S. DOE
	• Extended tests of hydrolyzed VX chemical agent for U.S. Army
	• Test SCWO system for Air Force
	• Follow-on Air Force program related to solid rocket propellants

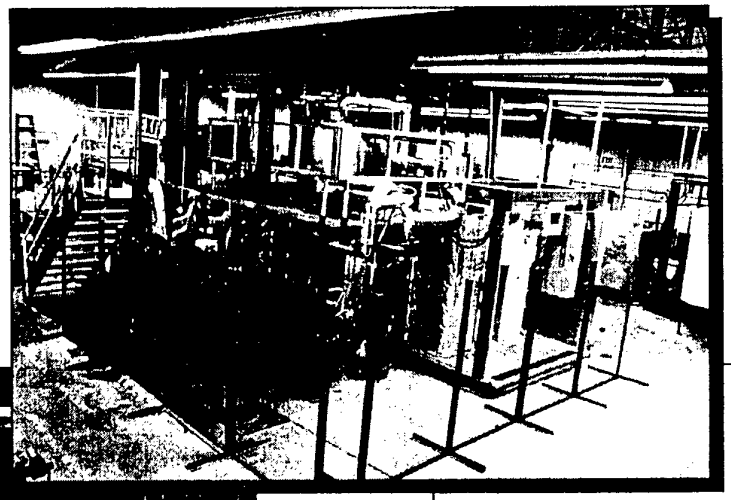
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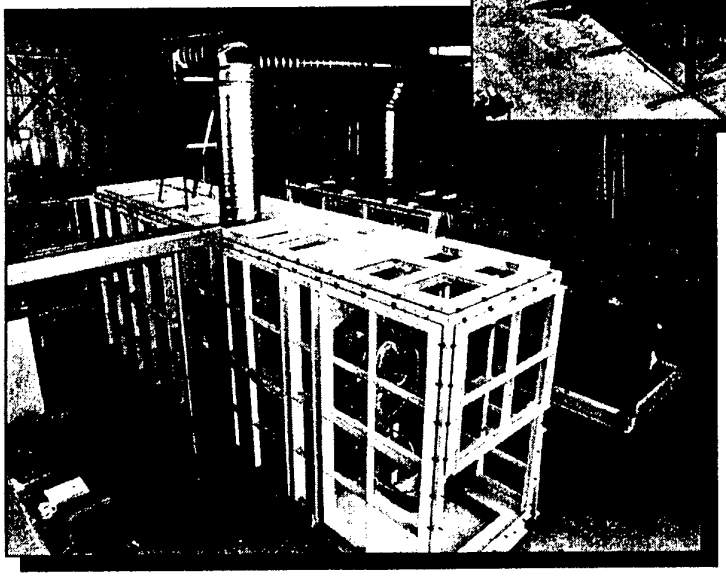
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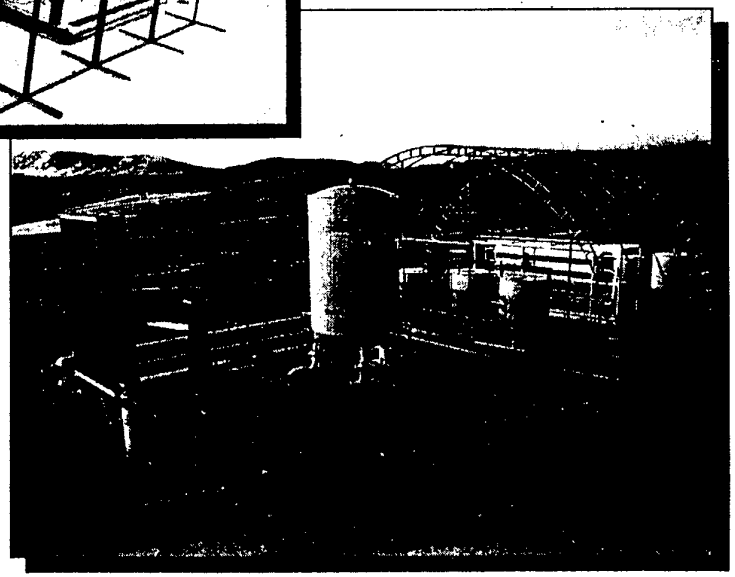
GENERAL ATOMICS SCWO PILOT PLANTS

MODAR Pilot Plant





DARPA/ONR Pilot Plant



Air Force Pilot Plant

L-712(37)
10-22-97

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PILOT-SCALE TEST EXPERIENCE

Test Programs

- U.S. Army Alternative Technology, Defense Advanced Research Project Agency (DARPA), U.S. Air Force Solid Rocket Motor Demil, U.S. Navy, Department of Energy, and Private Customers

Wastes Treated

- VX hydrolysate and simulants, chemical agents (VX, GB, HD), rocket propellant, thiodyglycol (mustard simulant), fuels, chlorinated and non-chlorinated solvents, phosphate and sulfate salts, waste oils, hydraulic fluid, film forming foam, adhesives, contaminated Trimsol, greases, paint, biological waste, sludges, and PCB-contaminated oils

Total Processing Time

- 3625 hours (3 pilot plants)

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2-18-98

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SCWO EXPERIENCE WITH CHEMICAL AGENT DEMILITARIZATION

- Laboratory scale tests with GB, VX and mustard (DARPA sponsored)
  - Demonstrated DREs >99.99999% for GB and VX
  - Demonstrated DREs >99.9999% for mustard

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VX-HYDROLYSATE

L-966(4)
2-18-98

QTYUIOP

VX HYDROLYSATE TESTING SUMMARY

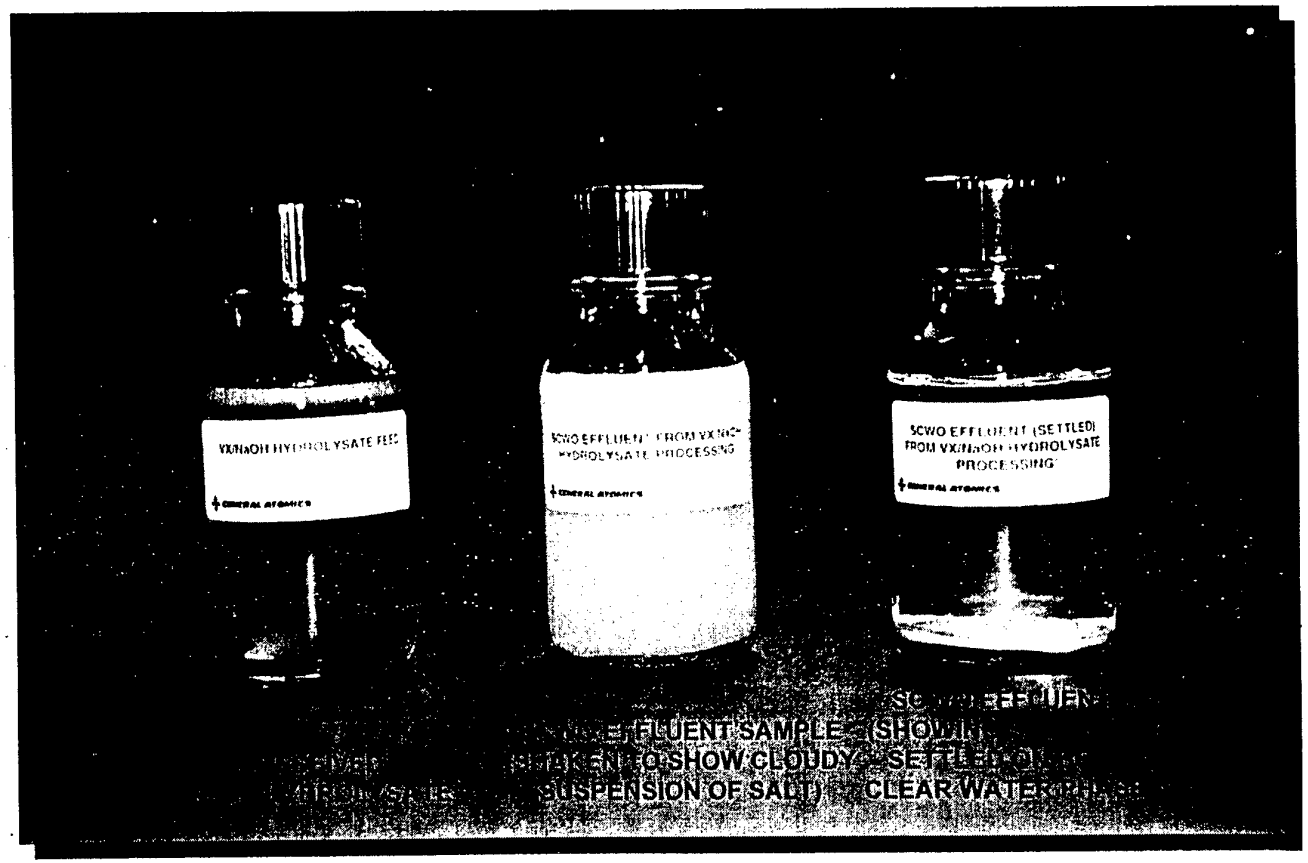
- Processed over 274 liters of VX hydrolysate
- Achieved >99.99% destruction of TOC and VX-THIOL
- Demonstrated continuous processing of 20% salt solution for 30 hours
- Obtained data required for scaleup

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2-18-98

QTYUIOP

EFFLUENT FROM VX HYDROLYSATE TEST



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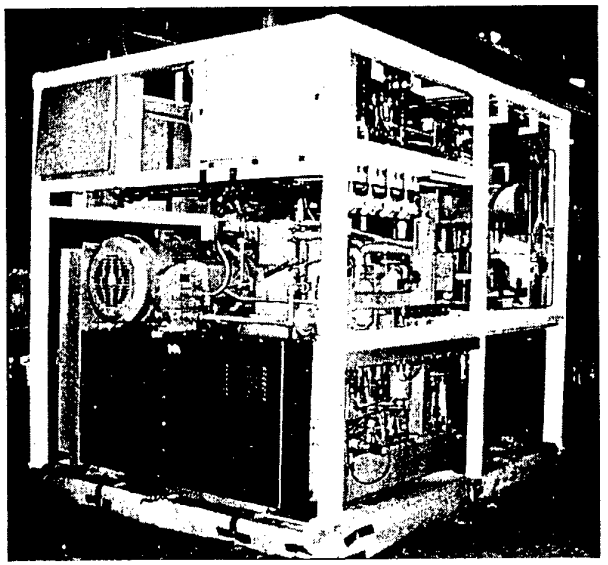
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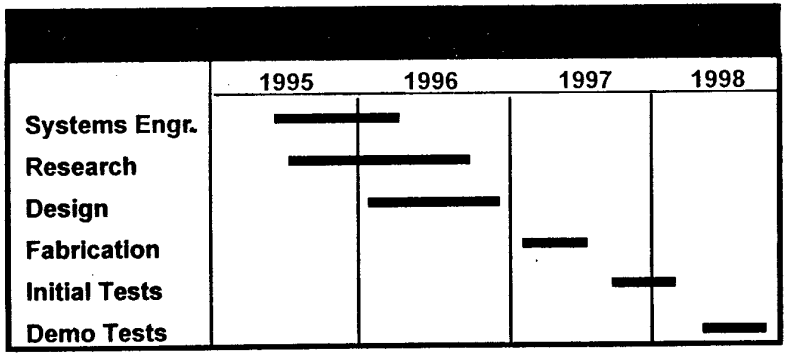
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DARPA NAVY SCWO PROGRAM

Design, develop, and demonstrate a full-scale 45 kg/hr SCWO unit for treatment of Navy shipboard excess hazardous materials that:

- Meets International, Federal, State, and Local environmental requirements
- Has dimensions less than 2.5 m x 3 m x 2.75 m
- Is self-contained and simple to operate and maintain
- Is compatible with eventual testing at sea





L-526(7)(b)
2-18-98

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SHIPBOARD SCWO UNIT DESIGNED TO THE FOLLOWING STANDARDS

Item	Standard	Comment
Noise	MIL-STD-740	Enclosure to be insulated to reduce noise to Grade E level. (High noise area where intelligible speech communication is necessary.)
Shock	MIL-STD-901	Designed for Grade B shock. SCWO equipment need not remain operational but must not come adrift or pose hazard to personnel or Grade A equipment.
Vibration	MIL-STD-167	Vibration isolation mounts used with compressor
Ships Motion	DOD-STD-1399 Section 301A	Design allows bracing for 20° roll angle = 1.7 g
EMI	MIL-STD-461	SCWO EMI believed comparable to other EMI sources on ship.
IR Signature	—	Hot SCWO components to be insulated and enclosed in an insulated skid. Effluent flow is cooled to <52°C (125°F) prior to discharge below waterline.

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Munster, Germany, March 22-25, 1998

L-712(16)
2-11-98

QTYUIOP

SAFETY STRESSED IN GA DESIGN

- Enclosure provides protection against pipe and reactor failures
- Reactor designed and tested to ASME Boiler & Pressure Vessel Code, Section VIII, Div. I
- Piping designed to ANSI Chemical Plant and Petroleum Refinery Piping Code, B31.3
- Control system provides automatic shutdown in the event of component failure

L-712(21)a
10-22-97

QTYUIOP

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OPERATOR REQUIREMENTS MINIMIZED

- Operator interfaces are simple:
  - Touch screen for Start and Stop
  - LCD display for alarms and diagnostics
- Operator actions limited to the following:
  - Load feed drums/remove collected solids
  - Perform routine maintenance (off shift)
- PLC-based control system provides automated control of startup, operation, and shutdown

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L-712(19)a
10-22-97

QTYUIOP

ADVANTAGES OF SCWO

- Well developed with good data base
- Excellent destruction efficiencies
- No airborne particulates
- No dioxins or furans
- Simple, safe, compact equipment
- No smokestack (completely enclosed)
- Energy efficient
- Low cost

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L-818(3)
5-5-97

QTYUIOP

OTHER BENEFITS OF SCWO

- Yields ~ 1 kg of dry solid per kg of agent
- Recovers water required for processing
- Recovers energy required for heating
- Provides complete holdup and monitoring of effluents prior to discharge

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L-818(7)
2-18-98

QTYUIOP

CONCLUSION

- SCWO is a well-developed technology with a broad data base
- SCWO provides a safe, compact, contained alternative to incineration for destruction of chemical agents or their neutralization products

Proceedings of the International CW Destruction Symposium
Munster, Germany, March 22-25, 1998

L-966(6)
2-18-98

QTYUIOP

Proceedings of the International CW Destruction Symposium
Munster, Germany, March 22-25, 1998

BIOLOGICAL DESTRUCTION OF CHEMICAL WARFARE AGENTS INCLUDING ARSENICALS

John A. Scott

Parsons
Pasadena, CA 91124, U.S.A.

Arsenical compounds such as mustard/lewisite mixtures can be readily neutralized in hot water. The mustard goes to thiodiglycol (TDG) while the lewisite hydrolyzes rapidly to acetylene and soluble arsenic salts. The arsenic salts con be removed by precipitation with $FeCl_3$. Explosives are also readily hydrolyzed with aqueous caustic. The two resulting hydrolysates can be combined for effective synergism in providing the nutrients required for bio-treatment.
Arsenic tolerant bugs have been isolated from arsenic contaminated soils that can tolerate dilute concentrations of arsenic left in the hydrolysates.

Water jet cutting is a safe and effective method to get access to the explosives and agent in the chemical munitions. We have data that substantiates its safety and effectiveness for both primary and secondary explosives. There is no penalty in the system for the contaminated water generated from water jet cutting since the aqueous systems we are using require water in excess of that generated by the water jet cutting. The grit is removed with the precipitates prior to bio-treatment.
We are also developing a method for complete decontamination of munitions metal parts using superheated steam. The steam is subsequently condensed as serves as water source to the project.

Technology for Neutralization of Assembled Chemical Weapons

Parsons - AlliedSignal



Proceedings of the International CW Destruction Symposium
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Parsons-AlliedSignal: (WHEAT)*
Water Hydrolysis of Explosives and Agent Technology

WHEAT (Water Hydrolysis of Explosives and Agents Technology) is a closed loop water based system as shown in the illustration on the following page. Water is introduced to the system through the steam boiler. The resulting steam, at approximately 1,200F and less than ambient pressure, is used to totally decontaminate the metal parts from the disassembled munitions. The steam is then condensed to water and subsequently used as make-up to the hydrolysis reactors and the equipment decontamination system.

The agent from the munitions is reacted with the water, including appropriate pH adjustment using sodium hydroxide, to totally destroy all agent. In a similar manner, the energetic material (explosives) is reacted with pH adjusted water to hydrolyze and destroy energetics. The resulting hydrolysates from the agent and the energetics are combined for biological treatment so that the organic compounds and minerals produced by the energetics hydrolysis can provide nutrition to the micro-organisms in the biotreatment process. The biotreatment process provides the mechanism for mineralizing the combined agent and explosives hydrolysates and oxidizing the organic compounds. Upon completion of biotreatment, the solids are separated from the biotreated liquids, and the water recycled to the steam boilers. The cycle is complete, and the process begins again.

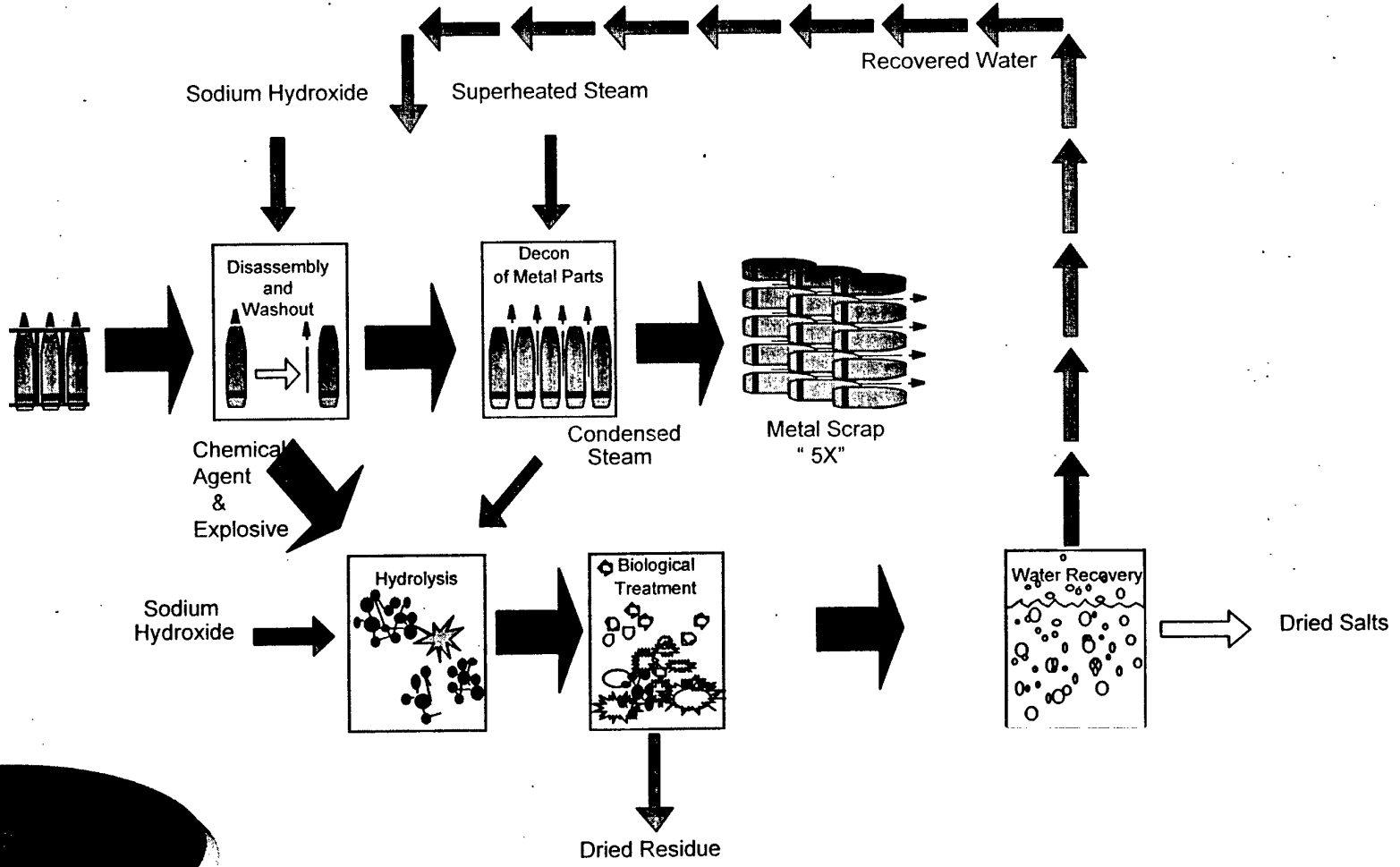


*Patents Pending

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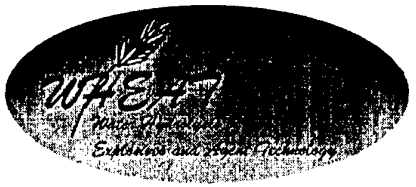
Proceedings of the International CW Destruction Symposium
Munster, Germany: March 22-25, 1998

WHEAT: Total Solution



Proceedings of the International CW Destruction Symposium
Munster, Germany, March 22-25, 1998

WHEAT: How it works

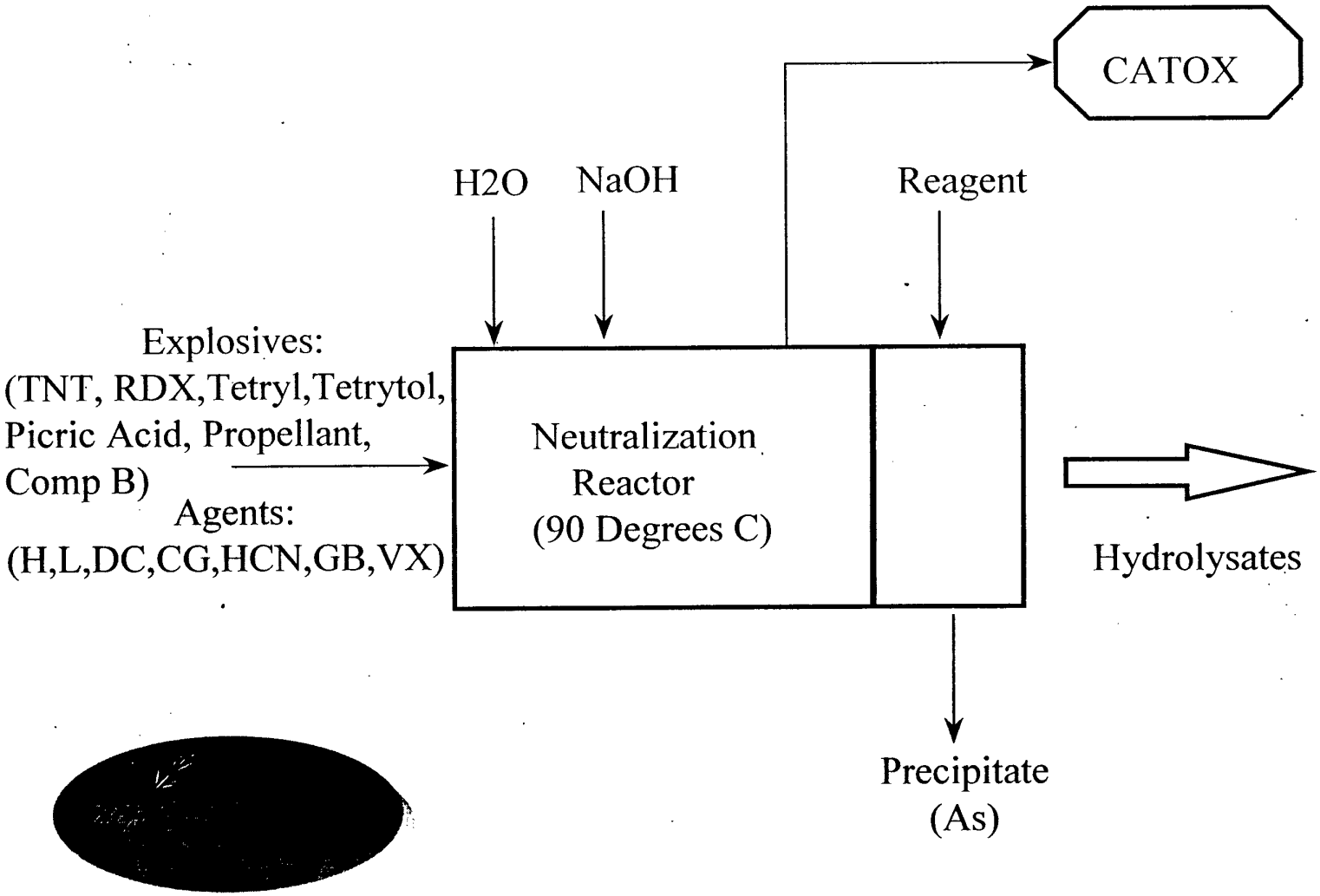


Hydrolysis is the naturally occurring decomposition of both agents and explosives in the presence of water. The speed at which this happens is determined by the solubility and rate of reaction; both of these can be increased by increasing the temperature of the water. The agent hydrolyzer is designed to totally destroy chemical warfare agents by hydrolysis, including the addition of sodium hydroxide, under atmospheric pressure and temperatures in the vicinity of 90 degrees C (194 degrees F). The result is a hydrolysate containing intermediate organic and inorganic compounds of very low toxicity. This hydrolysate can be biologically treated to further reduce toxicity. The Immobilized Cell Bioreactor™ uses aerobic digestion by selected specialty microorganisms to decompose the intermediate products in the hydrolysate to biomass and harmless basic minerals and carbon dioxide, nitrogen and water.

Energetics (explosives and propellants) are hydrolyzed and biodegraded in a similar manner to chemical agents. The resulting energetics hydrolysate contains a mixture of small molecular weight organic compounds and nitrogen species capable of supporting microbial growth. These actually serve as a source of nutrient essential to the metabolic health of the microbes. The combination of agent and explosive neutralization products produces an ideal solution for biological treatment. This mixture minimizes the need to add other nutrients, thereby enhancing the digestion of the organics that occur in lower concentrations

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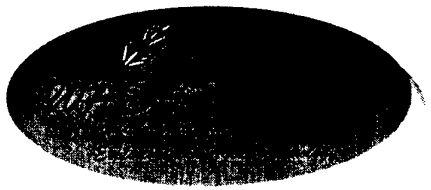
Neutralization of Agents and Explosives



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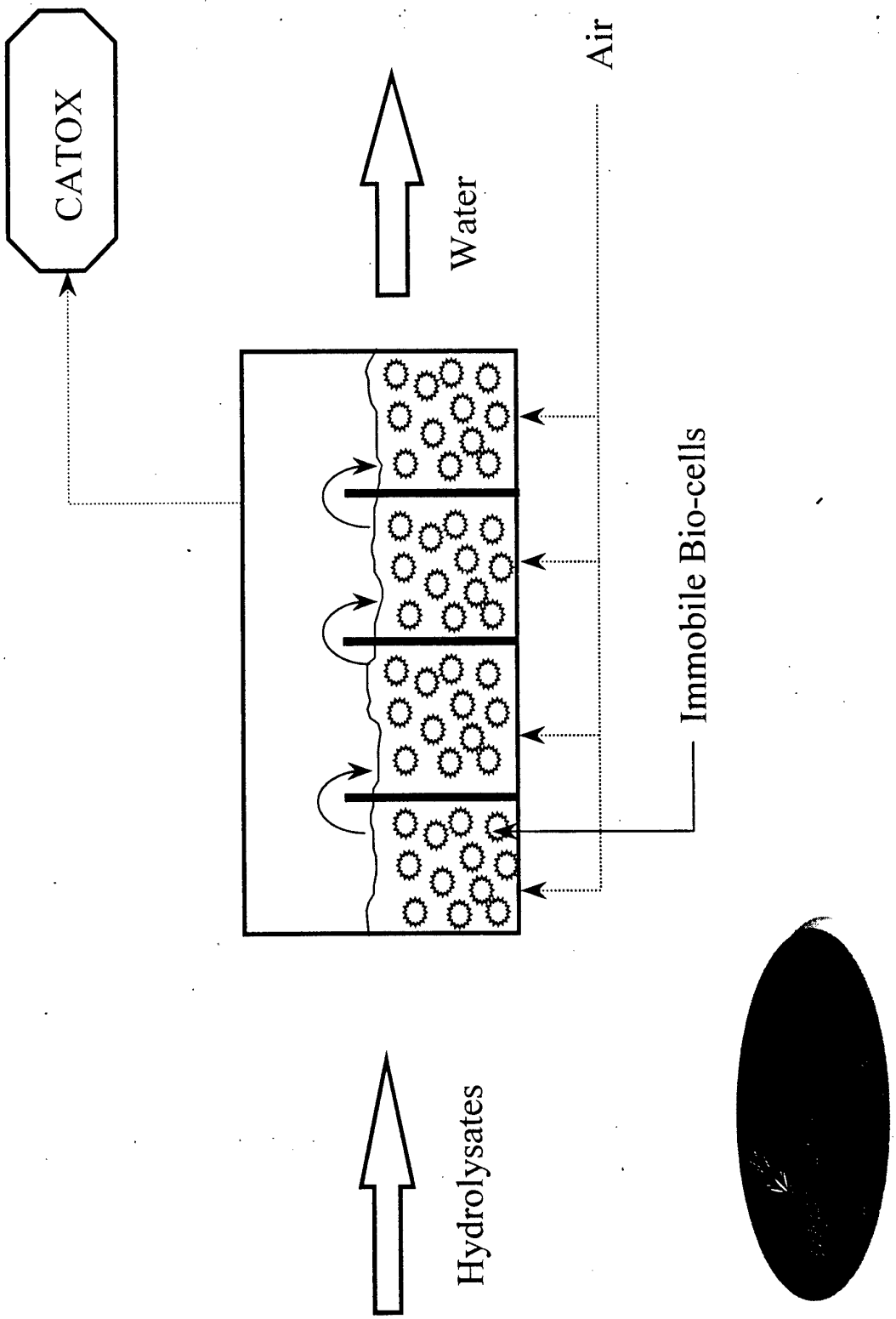
Neutralization Hydrolysates

Agent	Hydrolysate	Bio-treatment
H, HD,HT	TDG	→
Lewisite	As salt	→
Nerve Agents	IMPA, MPA	→
DC, DA	Phenyls	
Phosgene	NaCl	
HCN	NaCN	→
Explosives	Nitro-organics	



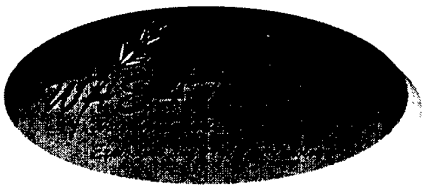
Proceedings of the International CW Destruction Symposium
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Bio-treatment of Hydrolysates



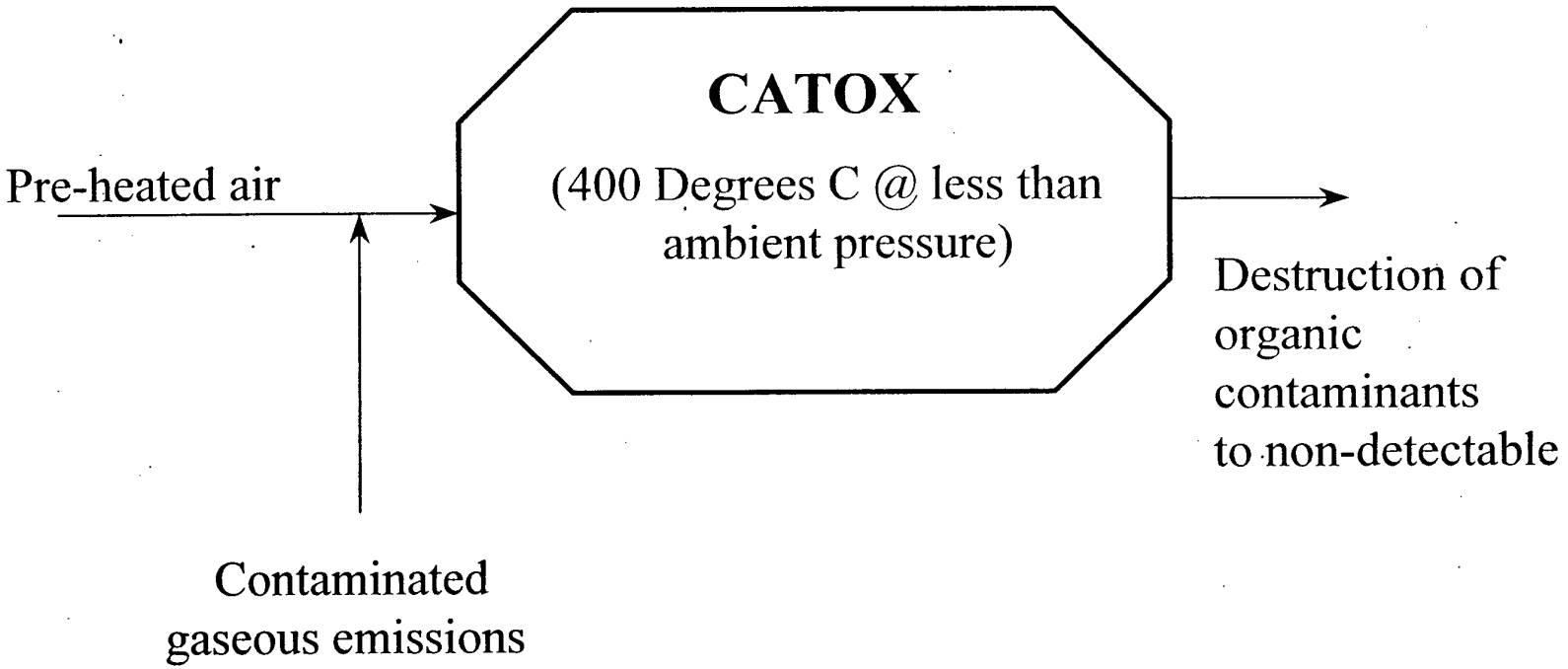
ICB Bio-treatment

- Immobilized microbes result in high residence time
- Plugged flow between cells allow cells to specialize to mineralize hard to treat organics
- Air-stripping to CATOX of trace volatiles
- Synergistic effect between agent and explosive hydrolysates



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Detoxification of Gaseous Emissions





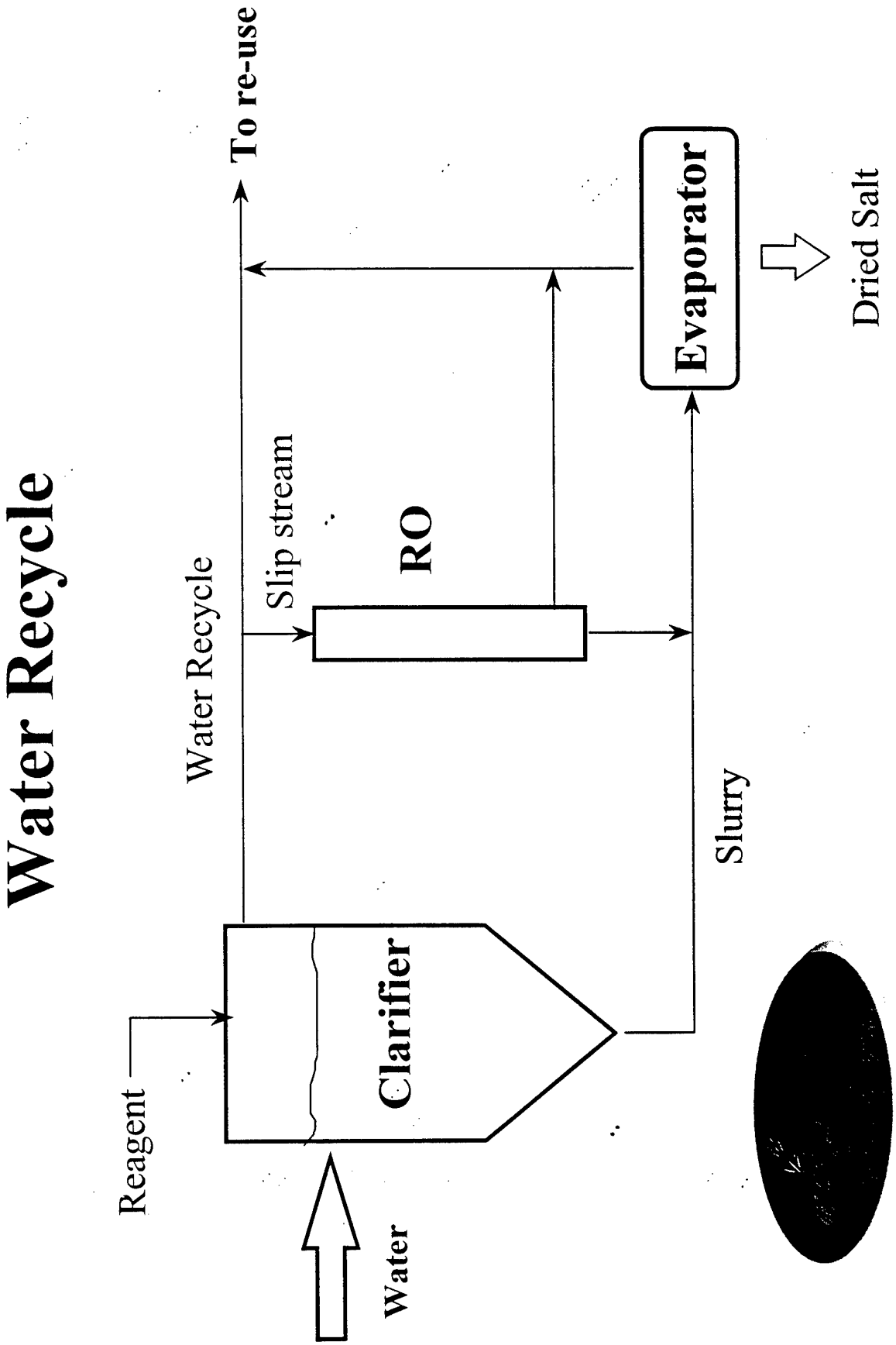
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Munster, Germany, March 22-25, 1998

CATOX Destruction of Organics

- Catalytic conversion is flameless
- Halogen resistant
- Accommodates high humidity
- No moving parts, high reliability
- Destroys PCB's and furons
- Oxidizes all organics

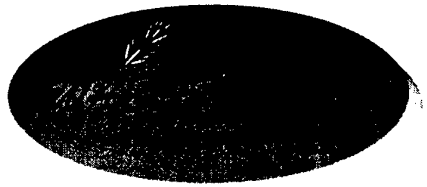


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Water Recycle
To re-use
Evaporator
Dried Salt
Slip stream
RO
Water Recycle
Slurry
Clarifier
Reagent
Water

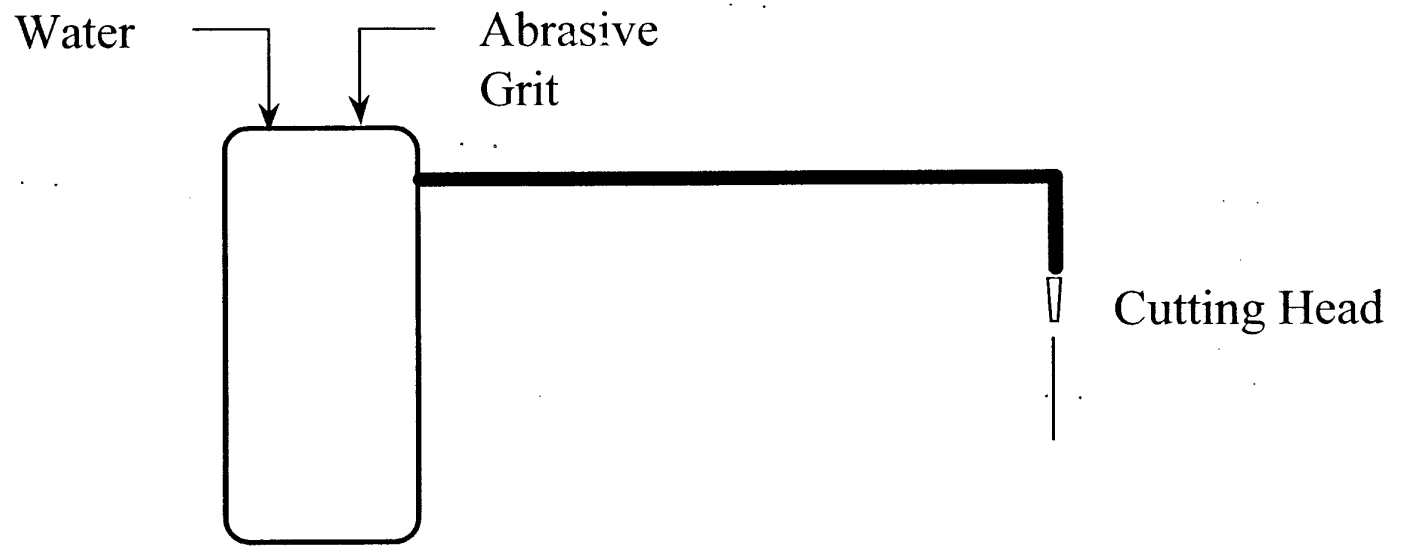
Water Recycle

- No liquid discharge
- Amount of slip stream through RO determined by salt mass balance: to less than 4% NaCl
- Water returned to process



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Water-jet Cutting *



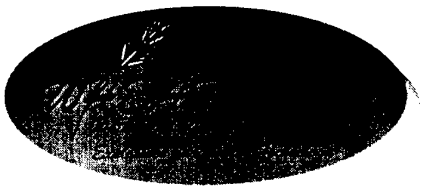


* ALVIS

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Advantages of Water-jet cutting

- No contact between munition and cutting head
- Equipment located outside of agent area
- High reliability, easy maintenance
- WHEAT accommodates water as input
- Easily accommodates deformed and corroded munitions



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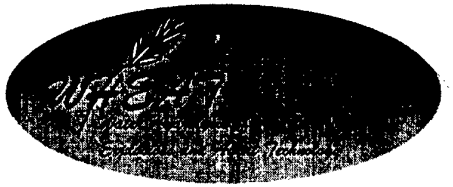
Safety First

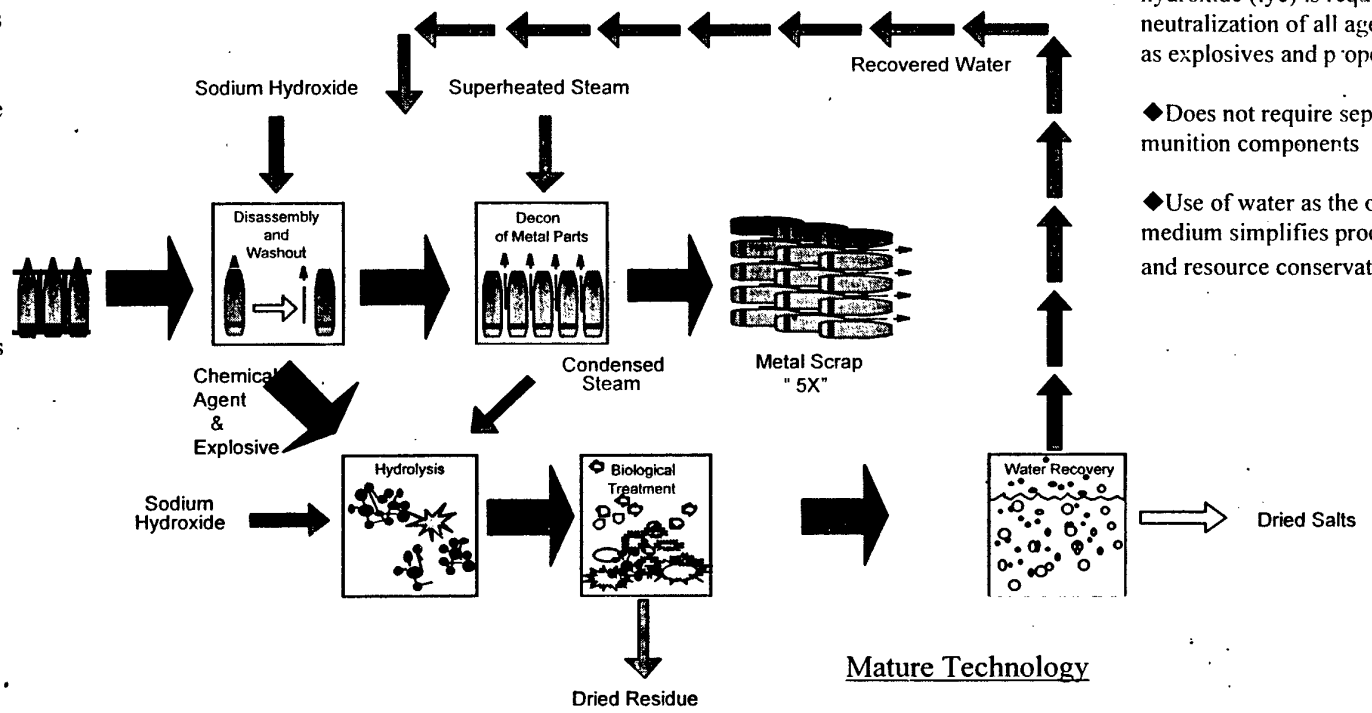
◆Uses water and sodium hydroxide

◆Low Temperature / low pressure agent and explosives neutralization

◆Batch processes that can be monitored to assure agent/explosives destruction prior to release

◆Selection of technology maximizes the opportunity to incorporate lessons learned from US Alt Tech I as well as the US Baseline Demilitarization Process.





Simple Process

◆No exotic chemicals or materials of construction

◆Just one chemical, sodium hydroxide (lye) is required for neutralization of all agents as well as explosives and p opellants

◆Does not require separation of munition components

◆Use of water as the only process medium simplifies process balance and resource conservation.

Environmentally Friendly

◆Treatment of secondary agent and explosive wastes accomplished by biodegradation

◆Closed loop system with no liquid discharge

◆Low temperature processes preclude the potential for formation of dioxins and furans

◆Metal parts can be totally decontaminated to "5X" on-site

Mature Technology

◆Use of commercially available biotreatment technology

◆Total system approach maximizes the use of US Baseline Chemical Demilitarization systems (other than incineration) to assure technical maturity

◆Sodium hydroxide has been used effectively and safely in demil operations for more than 20 years

◆US Alt Tech I selection of base hydrolysis will continue to mature the technology.

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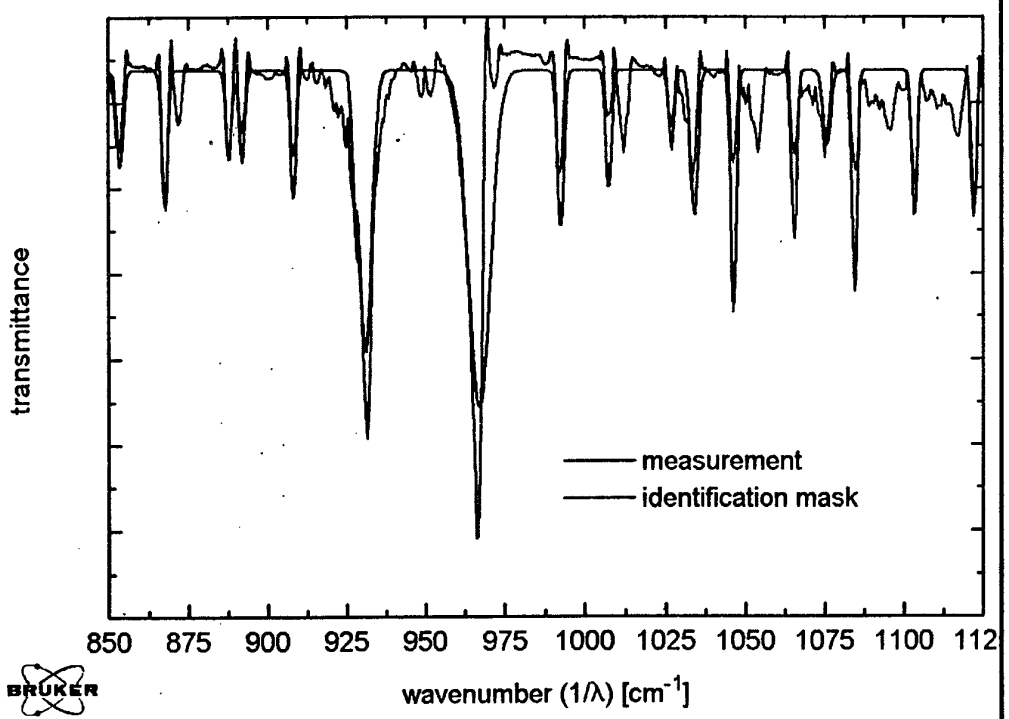
Proceedings of the International CW Destruction Symposium
Munster, Germany, March 22-25, 1998

FT-IR Remote Sensing at Recovery, Transport, Storage, and Destruction of Chemical Warfare Agents

S. Morley, Bruker Saxonia Analytik GmbH, Permoserstraße 15, D-04318 Leipzig

One major task during recovery, transport, storage, and destruction of chemical warfare agents (CWA) is to ensure that there is no unacceptable safety hazard neither for the personal concerned with this duties work nor for civilians in the immediate neighbourhood or for the environment. In order to master this highly delicate task, Bruker developed the **R**emote **A**ir **P**ollution **I**nfrared **D**etector (RAPID) system. The idea behind the RAPID is strongly connected to the fact, that any substance has its own specific signature (fingerprint) in the infrared region (IR) of the electromagnetic spectrum that surrounds us. Thus the RAPID is based on the well proven Bruker RockSolid™ flex-pivot interferometer and measures the strength of these signatures and correlates them to product of concentration × pathlength × temperature difference of target species and background. There are two modes in which the RAPID is able to work, thereby covering the whole scope of IR remote sensing. In the passive mode the natural IR emission of the environment and possible hazardous gases is used. This mode provides a maximum of flexibility, as the instrument only has to be focussed at the scenery. The measured spectra will show specific signatures of the gases within the optical path as long as there is a significant temperature difference between the gases and the background. An increase in the system sensitivity can easily be achieved by the second operational mode: the active open path measurement. This technique employs an IR light source at elevated temperatures and thereby bears some inflexibility. Nevertheless, as the temperature difference between background (IR source) and pollutant is enhanced up to three magnitudes this mode is very advantageous with reference to a higher sensitivity and a wider spectral range.

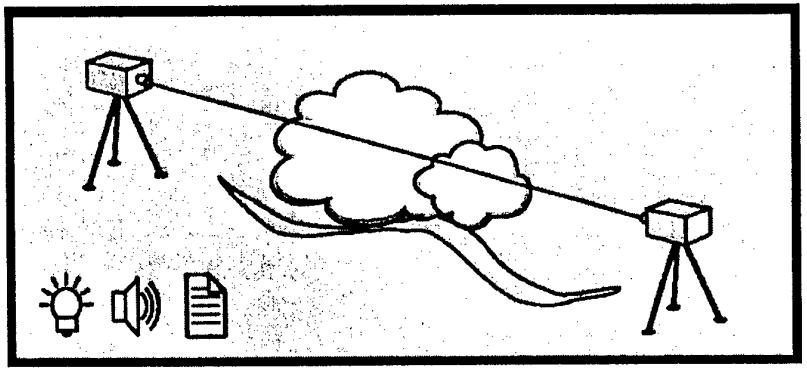
INFRARED SPECTROSCOPY



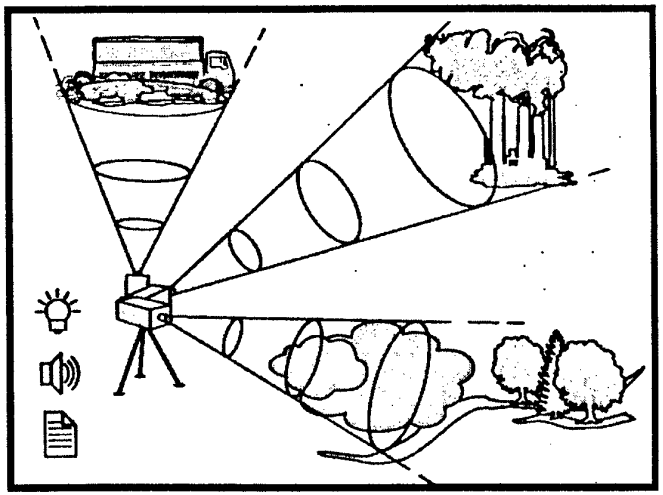
Spectral analysis of electromagnetic radiation in the wavelength range from 1 mm to 800 nm (i.e. frequencies 1 cm^{-1} to 12500 cm^{-1})

ACTIVE / PASSIVE EX SITU

active (in situ) open path measurements



passive remote IR - spectroscopy



PRINCIPLE OF DETECTION

Due to the principle of measurement the probability of detecting a target species P_d in a passive scenario does not only depent on its concentration but also on:

- the optical thickness of the target cloud
- the temperature difference between target cloud and background
- the measurement time (noise)
- the absorption coefficient of the species
- instrumental properties (e.g. throughput)

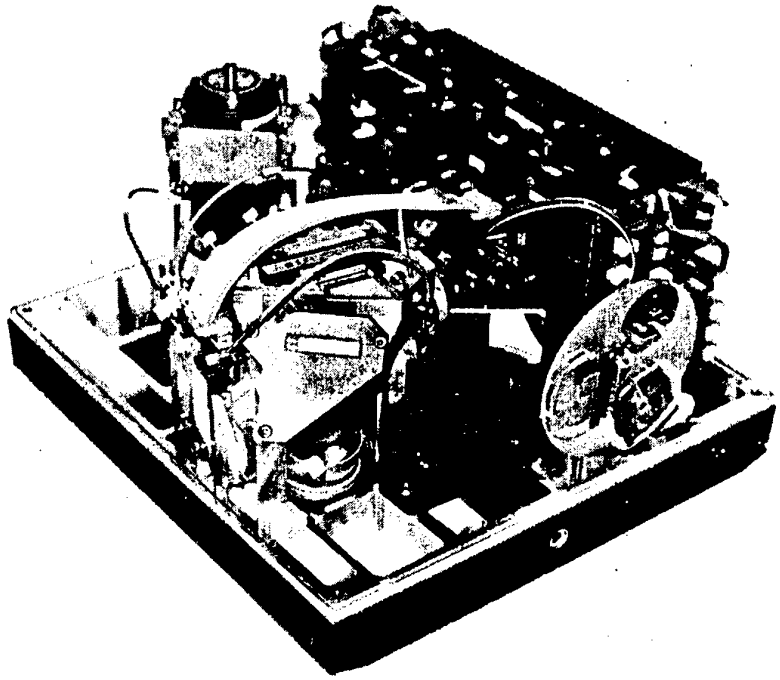
DETECTION LIMITS

Passive Configuration ($\Delta T>1°C$, L>100m, 60 s)	
Sarin 700 ppb	Mustard Gas 1.5 ppm
Carbonyl dichloride 500 ppb	Ammonia 800 ppb

Active Configuration (Globar 1500K, L>100m, 60 s)	
Sarin 1.4 ppb	Mustard Gas 3 ppb
Carbonyl dichloride 0.9 ppb	Ammonia 1.5 ppb

ADVANTAGES

- remote,
- non destructive,
- fast (i.e. online-useable)
- mobile
- highly sensitive even as a screening method
- low interference



TECHNICAL DETAILS

patented Bruker Rock Solid™ interferometer

optical resolution up to 0.5cm^{-1}

field of view 30mrad

LN_2 cooled MCT detector
(closed cycle version optional)

ZnSe entrance window

tripod mounting

stationary or notebook PC

independant power supply 12V-36V

ANNEXES

Abstract of:

Déminest

a french quarterly technical and friendship review for the E.O.D. fireworkers

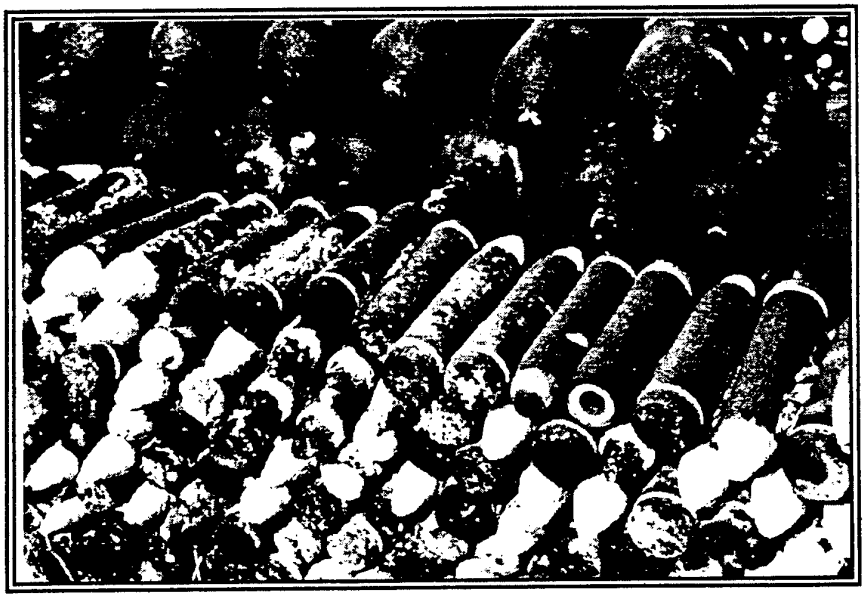
by Henry Belot

Dans les plaines de Picardie, des piles de projectiles chimiques attendent leur élimination définitive.

* * *

In the plains of Picardie, piles of chemical projectiles are still waiting for their final disposal.





Dans les plaines de Picardie, des piles de projectiles toxiques attendent encore leur élimination définitive.

In the plains of Picardie, piles of toxic projectiles are still waiting for their final disposal.

Proceedings of the International CW Destruction Symposium
Munster, Germany, March 22-25, 1998

THE SITUATION OF THE FRENCH *SERVICE DU DEMINAGE* IN THE APPLICATION OF THE INTERNATIONAL AGREEMENTS ON CHEMICAL AMMUNITION DISPOSAL

LA SITUATION DU DEMINAGE FRANCAIS DANS L'APPLICATION DES ACCORDS INTERNATIONAUX SUR LE TRAITEMENT DES MUNITIONS CHIMIQUES

French E. O. D. services regularly discover old chemical ammunition on first world War sites.
Most of the time this is discovered in civilian areas and the responsibility of collection rests with the *Service du Déminage*, Mine Clearance Corps of Interior Ministry, that includes the head of Defence and Civilian Safety.

These discoveries are frequent because of the wide battle area, the duration and the bitterness of the fighting, (so that means a great quantity of buried ammunition), a high population density that have various activities (agriculture and forestry work, hunting...).
Till 1994 the E. O. D. service of Interior Ministry destroyed the chemical shells by detonation in the bay of Somme not far away from Le Crotoy, where the sea withdraws by several kilometres at each low tide.
The ammunition was exploded under several meters water using a very high explosive charge. The water hydrolysed the non-persistent agents and the very high temperature of the explosion broke the persistent one such as yperit or trichloronitromethan.

Little by little this solution of subaquatic explosion has been questioned on environmental grounds although nothing has prooved that those activities regarding especially explosive ammunition has ever damaged either the local flora or fauna. The destruction operations in the sea have therefore been more and more curtailed.

In December 1996 an accidental explosion of several tons of ammunition in Le Crotoy dump brought the existence of this site to an end. The explosion was caused by the growing old and the oxydation of an incendiary shell, the destruction of which was postponed several times.

Régulièrement, le Service du Déminage français est confronté à la découverte de munitions chimiques anciennes sur les sites de combat de la première guerre mondiale. Ces munitions sont le plus souvent trouvées en domaine civil, et leur ramassage incombe aux démineurs de la Direction de la Défense et de la Sécurité Civiles (Ministère de l'Intérieur).
Ces découvertes sont fréquentes en raison de l'étendue des zones de batailles, de la durée et de l'âpreté des combats (donc du nombre de munitions enterrées) et aussi de la densité de la population qui occupe les zones et y exerce diverses activités (travaux agricoles ou forestiers, chasses, etc.).

Jusqu'en 1994, le Service du Déminage du Ministère de l'Intérieur pouvait détruire les munitions chimiques par explosion en baie de Somme, non loin du CROTOY, en un site où la mer se retire sur plusieurs kilomètres à chaque marée. Il s'agissait de pétarder les munitions anciennes sous plusieurs mètres d'eau à l'aide d'une très forte charge explosive. L'eau de mer hydrolysait les agents fugaces et la très haute température de l'explosion brisait les agents persistants comme l'ypérite ou le trichloronitrométhane.
Petit à petit, la solution du pétardement subaquatique en mer fut remise en cause dans un souci de protection de l'environnement, bien qu'il n'ait jamais été démontré que ces activités portant surtout sur des engins explosifs aient nui à la faune et à la flore locales. Les opérations de destruction en mer ont ainsi été de plus en plus limitées.
En Décembre 1996, l'explosion accidentelle de plusieurs tonnes de munitions au dépôt du CROTOY a mis un point final à l'existence de ce site. Le vieillissement et l'oxydation d'un obus incendiaire dont la destruction avait été repoussée à plusieurs reprises sont à l'origine de l'explosion.

Aging and oxydation have ever damaging consequences for chemical ammunition, especially the thin walled one. As it cannot be destroyed anymore - *the means of destruction dont't legally yet exist in France* - this ammunition has to be stored with all the attendant preservation problems.

The decree N°96-1081 of December 5 th, 1996 modified the decree of March 4th, 1976. The later fixed the mutual attributions of Interior Ministry and Ministry of Defence regarding research for neutralization, collection and destruction of the explosive ammunition.
It stipulates that Interior Ministry is in charge of collecting the chemical ammunition, determines the category they belong to, and organizes transport to the dismantling place.

Ministry of Defence is responsible for the storage, the dismantling of this ammunition and the elimination of residual toxic waste.

Implementing this decree means achievment of a special organized depot to store projectiles which are mostly fired and damaged. A plant will have to be coupled with this site.

These plants won't be operational for several years. Meanwhile the fireworkers of the Service du Déminage will go on storing old chemical ammunition, in mostly precarious conditions.

Three storage sites are used at present time in France (Vimy, Laon, Metz).

The storage of old chemical ammunition which should have been destroyed several years ago will represent more and more risks as the time flies.
This concerns german, british and french missiles, artillery shells or projectiles for mortar trenches or saturation systems.
The collected chemical ammunition is for the most part badly corroded, meaning a significant reduction of the protective wall thickness, that could lead it being pierced or broken.

This corrosion depends on the type of the projectile wall. Drop steel often ages worst than cast steel or cast iron.
Outer ground environment is also important, as leafmould eats more away as limestone clay. Frequently emissions of explosive or toxic substances intensify corrosivity of the surrounding environment.
Attacks on equipment before being buried, partial explosions of dumps, the repeated unseccessfull attempts to explode (« duds »), the distorting impacts, the recovering of copper or brass by scrap merchants are also to be taken in consideration.

Ces vieillissements et oxydation sont encore plus lourds de conséquences en ce qui concerne les munitions chimiques, notamment celles à paroi mince. Comme elles ne peuvent plus être détruites (*les moyens légaux d'élimination n'existent pas encore en France*), ces munitions sont stockées, avec tous les problèmes découlant de leur état de conservation
Le décret n°96-1081 du 5 Décembre 1996, modifiant le décret du 4 Mars 1976 fixant les attributions respectives des ministère de l'Intérieur et de la Défense en matière de recherche, de neutralisation, d'enlèvement et de destruction des munitions explosives précise qu'en ce qui concerne les munitions chimiques, le Ministère de l'Intérieur procède aux opérations de collecte, détermine leur appartenance à cette catégorie et assure leur transport jusqu'au lieu de démantèlement.
Le Ministère de la Défense est responsable du stockage et du démantèlement de ces munitions, ainsi que de l'élimination des déchets toxiques résiduels.
L'exécution des dispositions de cet arrêté implique la réalisation d'un dépôt spécialement organisé en vue d'abrité des projectiles pour la plupart tirés et endommagés. Une installation de démantèlement devra être liée à ce site.
Ces installations ne seront pas opérationnelles avant plusieurs années. En attendant, les artificiers de la Direction de la Défense et de la Sécurité Civiles continueront à stocker dans des conditions souvent précaires les munitions chimiques anciennes. Trois sites de stockage sont actuellement utilisés en France (VIMY, LAON, METZ).
Le stockage des munitions chimiques anciennes dont certaines attendent depuis plusieurs années leur élimination présentera de plus en plus de risques au fil des ans. Il s'agit d'engins allemands britanniques ou Français, obus de l'artillerie ou projectiles pour mortiers de tranchée ou matériels de saturation.

Les munitions chimiques récupérées sont pour la plupart extrêmement corrodées, avec une réduction conséquente de l'épaisseur de l'enveloppe pouvant aller jusqu'au perçage ou à la rupture.
Cette corrosion dépend de la nature de l'enveloppe, l'acier vieillissant généralement plus mal que la fonte aciérée. Elle dépend également du milieu externe, l'humus par exemple étant plus agressif que l'argile calcaire. Fréquemment, des émissions d'explosifs ou de toxiques viennent intensifier la corrosivité du milieu ambiant.
Il faut également tenir compte des agressions qui ont pu précéder l'enfouissement des matériels : tentatives de pétardement, explosions partielles de dépôts, tirs suivis de non-fonctionnement, impacts déformants, récupération d'éléments en cuivre ou laiton par des ferrailleurs...

Every old ammunition shows various degrees of external or internal corrosion as well as contact corrosion induced by an electrochemical difference between two metals acting as an anode and a cathode.

This has to be especially noticed on 7,7 cm for field gun and 10,5 cm for light howitzer W. W. I german shells headed with direct action fuzes of casted aluminium-zinc alloy.

Aging fuzes heads of this type (particularly the KZ 14 nA, the EKZ 17 bew. and the EHZ 17 bew.) get yet disintegrating and falling off.

W. W. I toxic agents fillings of these shells, systematically including a halogen (bromide, chloride or iodine) may add the internal corrosion to the external one, the latter being often increased by picric acid of the exploder.

Often leakages occur between seals on the steel socket of the exploder and the zinc collar of the fuze, or between this collar and the shell mouth. More than 50 % of the toxic leakages proceed from such a corrosion, especially phosgene.

Some german projectiles contained a lead tank, filled with lachrymatory or toxic agents, which get corroded in contact with steel.

After such a long time, contact on this very large surface weakened both the steel projectile wall and the lead container, more especially as the long term missile tightness had not been the main regard of their designers.

Corrosion might have been intensified by the leakage of a small toxic substance coming into contact of the steel. Corrosion on both surfaces of chemical Minenwerfer projectiles might have weaken an already extremly thin steel wall, which led to serious safety problems for the diging up, transport and storage. Such ammunition sometimes happen to be pierced when hit with a pickaxe or to get fragmented during handlings.

Other leackages occur at the junction between the metal parts of the protective wall, even if of the same kind.
So threated or flanged sectors are weakened or can ever break for instance at the junction between the screwed head and the cylindrical body of the longs 7,7 cm and 10,5 cm W. W. I german shells.

Another significant example is noticeable on the 194 mm projectile for the Livens projector for saturation firing, most of the time filled with phosgene.

Sur toutes les munitions anciennes, nous constatons à des degrés divers des corrosions érosives, externes ou internes, ainsi que des corrosions par contact, ces dernières par l'effet d'une différence électrochimique de potentiel entre deux métaux agissant comme anode et cathode.

A titre d'exemple, les corrosions par contact sont fréquemment observées sur les obus allemands de 7,7 cm et de 10,5 cm pour obusiers, armés de fusées percutantes sensibles en alliage à base de zinc. Avec le temps les ogives de ces fusées (notamment les types Kz 14 nA, EKZ 17 et EHZ 17) finissent, lorsqu'elles existaient encore, par se désagréger et tomber.
Lorsque ces obus de la première guerre mondiale sont chargés en agents toxiques de combat comportant un halogène (chlore, brome, iode), la corrosion interne peut compléter l'externe, cette dernière étant souvent accentuée par l'acide picrique du relais de détonation. On observe alors fréquemment des fuites aux joints entre la douille d'acier du relais de détonation et l'embase en zinc de la fusée, ou entre cette embase et l'oeil de l'obus. Ce type de corrosion est cause de plus de la moitié des évasions de toxiques de combat chargés sous forme liquide.
Certains projectiles allemands (et quelques rares projectiles français) renfermaient un récipient de plomb rempli d'un agent lacrymogène et toxique qui se dégradait au contact de l'acier. Avec le temps, le contact entre les deux métaux sur une surface très étendue a souvent fragilisé à la fois l'enveloppe d'acier et le récipient de plomb, d'autant que l'étanchéité à très longue échéance du corps de projectile n'avait pas été le principal souci de leurs concepteurs. La corrosion a également pu être accentuée par le passage d'une petite partie du chargement toxique au contact de l'acier. Sur des projectiles de tranchée de ce type la corrosion sur les deux faces a pu ainsi affaiblir une paroi déjà fort mince, ce qui pose de sérieux problèmes de sécurité pour le dégagement hors du sol, le transport et le stockage. Il arrive que la munition soit percée d'un coup de pioche, ou qu'elle se fragmente en cours de manipulations.

D'autres fuites se produisent aux lignes de séparation des pièces métalliques constituant l'enveloppe, même si elles sont de même nature.
On peut ainsi constater la fragilisation et même la rupture de secteurs filetés ou emboutis, comme à la jonction entre l'ogive vissée et le corps cylindrique des obus allongés allemands de 10,5 cm et de 7,7 cm.
Un autre exemple caractéristique est donné par le projectile britannique de 194 mm du *projector Livens* pour tirs de saturation, chargé le plus souvent en phosgène.

At 20°c (68°f) this choking agent develops high steam pressure (1,55 atm), that fast grows depending on the temperature. It reaches 2,2 atm. by 30°c (86°f).
The sun radiation on one side, the opposite remaining rather cold, frequently causes leakages, especialiy around the detonator taking edge, where surfaces fittings are inanother pressed in.
When phosgene escapes, its evaporation causes a high cold point at the end of an overheated surface, weakening the tightness and intensifying the seal corrosion.
So a lot of ammunition has to be destroyed before the military plants for storage and dismanteling of these missiles are efficients.
With a care for safety of the personal and environmental protection, means to legally eliminate the chemical ammunition which represents the greatest danger have to be found as soon as possible. First priority :

- Britih Livens drums,
- German green-cross shells for 18 cm Gaswerfer, and for the 7,6 cm, the 17 cm and the 25 cm Minenwerfer.
- British projectiles for 4,2 inches Stokes mortar.

Most of these light cased missiles are filled with choking agents.

The solution currently under study at the Service du Déminage concerns the creation of a mobile transportable plan (or on a trailer), enabling the projectiles to get empty of their toxic agents, to identify these agents and then to decompose them at once whenever possible, especially the high steam-pressure ones.
That will mean phosgene and mixtures of phosgene and chloropicrine, as these agents cause almost 80% of leakages of chemical warfare agents.

Considerably lowering temperature and therefore the pressure, another variant of this solution might consists in transfering the phosgene from the projectile into special containers. The chemical industry would then take responsability for the elimination of the toxic agents. This has limitations because intervention is restricted and depends on the containers and goodwill of the private partner.

Extracting other toxic substances with low steam pressure (especially mustard !) might be significant. These agents could be separated from untransportable projectiles, in damaged condition or with a too much dangerous exploder or fuze.

The inside of the mostly empty missile will have then to get decontaminated and finally burned or exploded.

A la température de 20° ce toxique suffocant présente une tension de vapeur élevée (1,55 bar) et la pression interne augmente rapidement avec la température, atteignant 2,2 bars à 30°.
L'action directe du soleil sur une face du récipient, le côté opposé restant relativement froid, aboutit fréquemment à des fuites, surtout du côté de l'amorçage où les surfaces sont emboîtées en force.

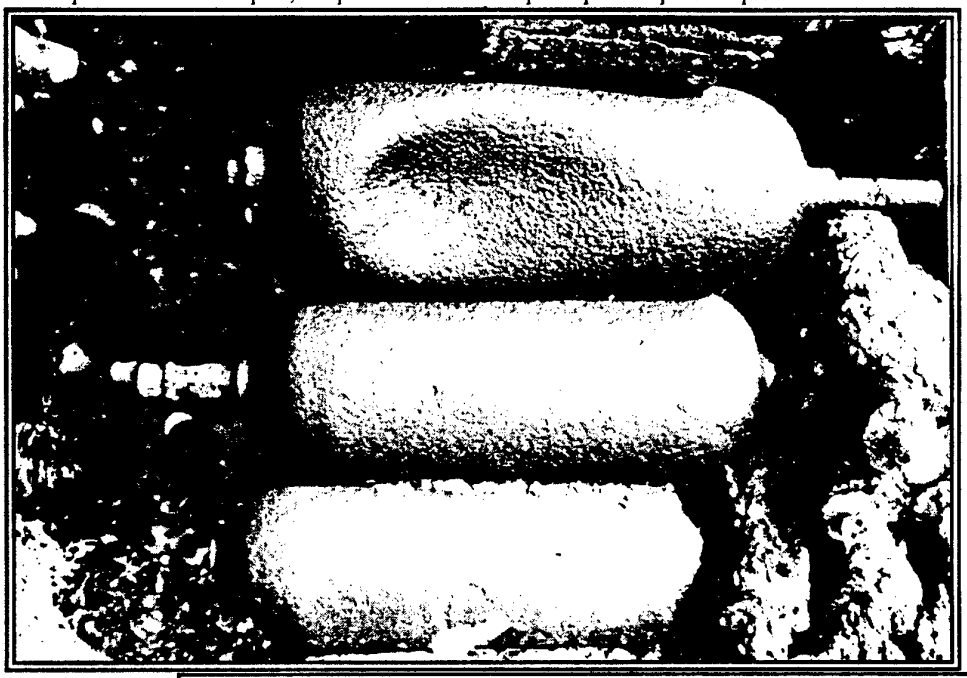
Lorsque le phosgène s'évade, son évaporation génère un point très froid à l'extrémité d'une surface surchauffée, intensifiant la perte d'étanchéité et la corrosion du joint par condensation d'acide.
Tout cela pour dire que de nombreuses munitions chimiques devront être détruites avant que les structures militaires d'accueil et de démantèlement ne soient opérationnelles.
Dans un souci de sécurité des personnels et de protection de l'environnement, la Direction de la Défense et de la Sécurité Civiles devra disposer au plus vite d'un moyen d'éliminer légalement les munitions chimiques présentant le plus de risques, et donc en priorité les projectiles Livens, les mines allemandes à croix verte pour le Gaswerfer lisse de 18 cm, les projectiles des Minenwerfer de 25 cm, 17 cm et 7,6 cm et ceux des mortiers britanniques Stokes de 4,2 pouces. Tous sont construits avec des parois minces, et la plupart sont chargés en agents suffocants.
La solution faisant actuellement l'objet d'études réside dans la création d'une unité mobile transportable ou remorquable, permettant de vider les projectiles de leur agent toxique, d'identifier ce dernier et de le décomposer sur le champ chaque fois que possible, et surtout s'il est à tension de vapeur élevée.
Ce sera le cas du phosgène ou du mélange phosgène et chloropicrine, agents qui sont en cause dans près de 80% des fuites de toxiques de combat.

Une variante de cette solution consisterait, après un fort abaissement de la température et donc de la pression, à transférer le phosgène des projectiles dans des récipients spéciaux. Le toxique serait ensuite confié pour destruction à l'industrie chimique. Cette procédure entraîne une limitation de la capacité d'intervention, car tributaire des récipients et du bon vouloir du partenaire privé.

Il serait profitable de pouvoir extraire d'autres toxiques liquides à faible tension de vapeur, et notamment l'ypérite laquelle pourrait être séparée de projectiles devenus intransportables du fait de leur dégradation ou de la situation des composants pyrotechniques.
L'intérieur de l'engin vidé de l'essentiel de son chargement sera ensuite décontaminé pour être finalement incinéré ou détruit par pétardement.

This way of mobile destruction capacity will still be kept useful, even when the defence-destruction plant is operational, fot it will be enable an occasional treatment of discovered chemical ammunition, that is considered to be untransportable without risk, which will be more and more frequent as the time flies.

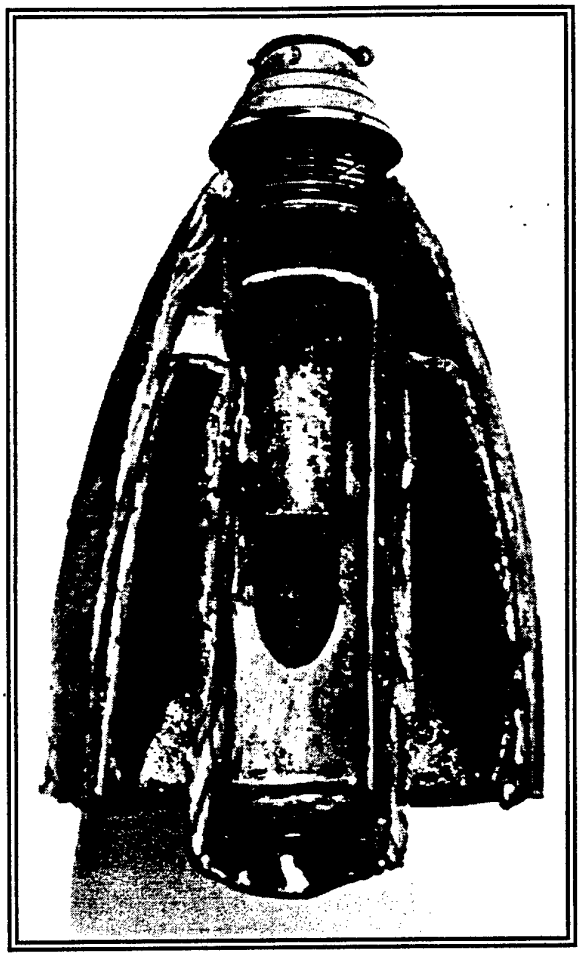
Ce moyen de destruction mobile aura encore toute son utilité lorsque l'installation lourde de destruction sera opérationnelle, car elle permettra le traitement ponctuel de munitions chimiques découvertes et se révélant intransportables sans risques, ce qui sera avec le temps de plus en plus fréquent.

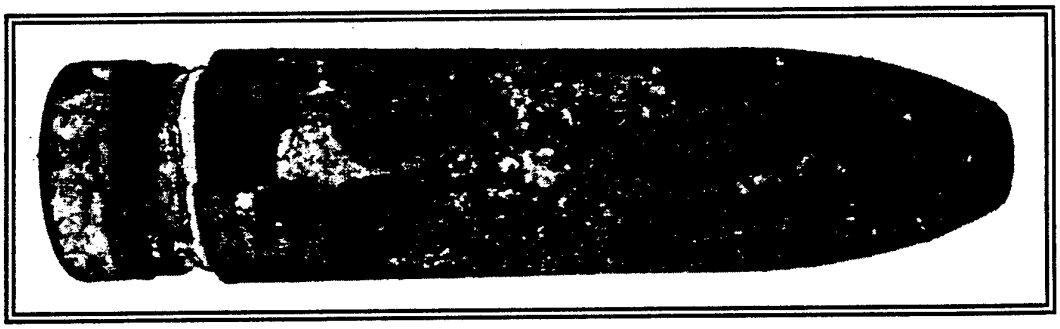




La munition du *Projector Livens* britannique était un engin de saturation fréquemment chargé en phosgène.
Aujourd'hui, ce dernier s'évade souvent (➔) de l'enveloppe mince et mal conservée, détruisant parfois la gangue de calcaire et de rouille.

The british ammunition of the projector Livens was a saturation device frequently loaded with phosgene.
Today, the latter often escapes (➔) from the thin and badly preserved case, sometimes destroying the limestone and rust gangue.





En haut : coupe de l'ogive d'un projectile chimique allemand de 17 cm pour Minenwerfer moyen.
En bas, un autre qui a accidentellement fonctionné.

Le projectile toxique pouvait comme ici renfermer un vase de plomb lorsque le chargement ne supportait pas le contact de l'acier. Avec le temps, la réaction électrolytique a souvent corrodé toute la construction.

On top : section of a-German chemical projectile of 17 cm for medium Minenwerfer.
Below : another one that has worked accidentally.

The toxic projectile could as here contain a lead vessel when the filling did not bear contact with steel. With time, the electrolytic reaction has often corroded the construction.

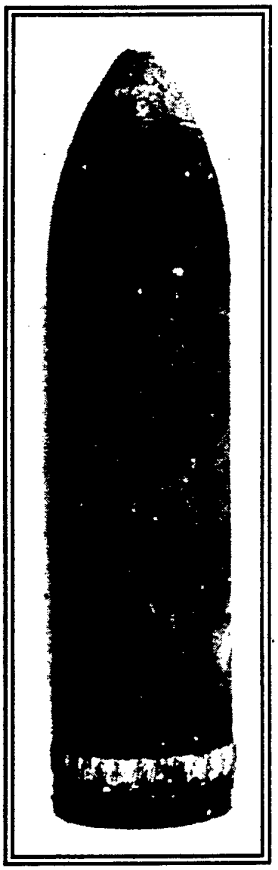


Le projectile lisse du Gaswerfer allemand de 18 cm était chargé en liquide suffocant ou en explosif additionné de diphénylchlorarsine.
L'enveloppe d'acier peut se percer par corrosion, ou fuir au niveau du joint de la gaine d'éclatement ou de la plaque de culot.

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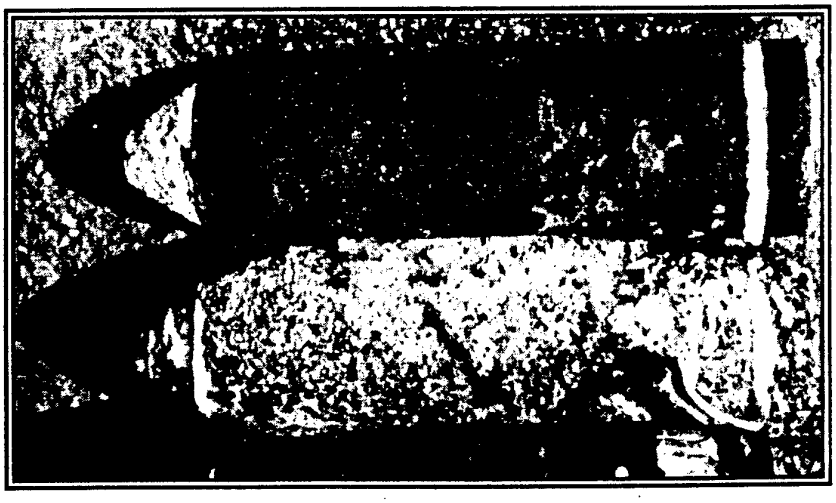
The German 18 cm Gaswerfer's smooth projectile was filled with a liquid choking agent, or a diphenyl-chlorarsine added explosive.
The steel case can get perforated by corrosion or leak by the exploder gain joint or the base plate.

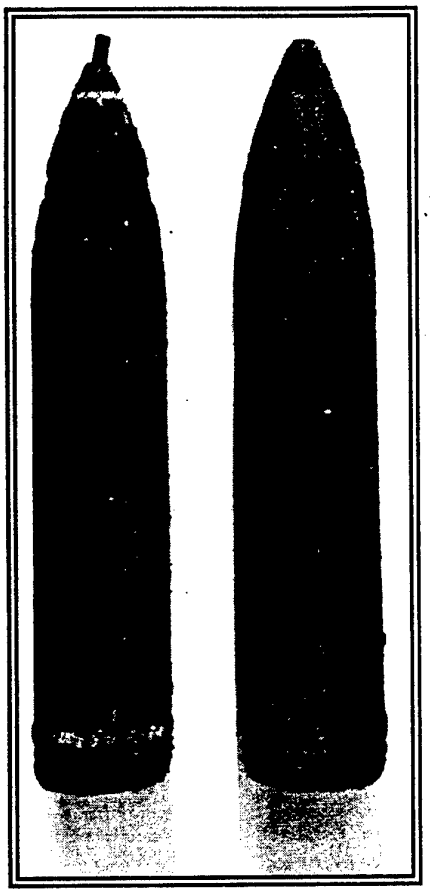




L'obus allongé allemand poùr obusier léger de campagne a renfermé pratiquement tous les types de chargements toxiques alors en usage. La fusée sensible EHZ 17, réalisée à partir d'un alliage de zinc, se rencontrera dans toutes les situations de corrosion, parfois intacte, très souvent dégradée, et même fréquemment dépourvue de la tête de fusée, tombée, avec les conséquences que l'on imagine pour l'étanchéité et la sécurité des personnels.

The long German shell for light field howitzer has contained nearly all types of explosives and toxics in use at the time. The sensible EHZ 17 fuze, made of a zinc alloy, will be encourted in every corrosion situation, sometimes intact, very often damaged, and even frequently devoided of the fuze head, fallen down, with the consequences that one can imagine for the imperviousness of the shell and the safety of the personnels.



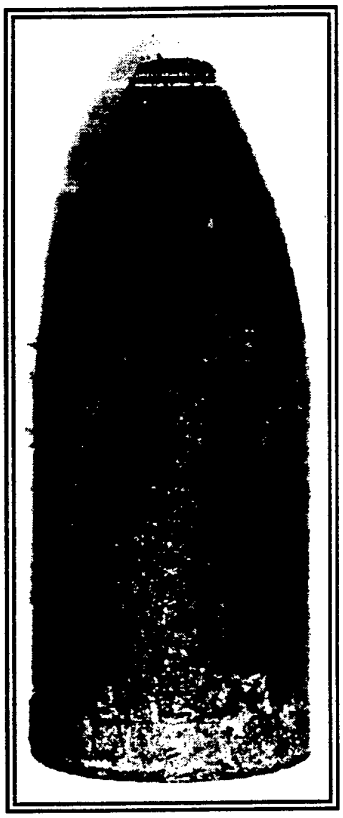






Les fusées en alliage de zinc armant les obus longs de 7,7 cm allemand sont très souvent corrodées au point de s'effriter, favorisant la fuite des chargements chimiques.

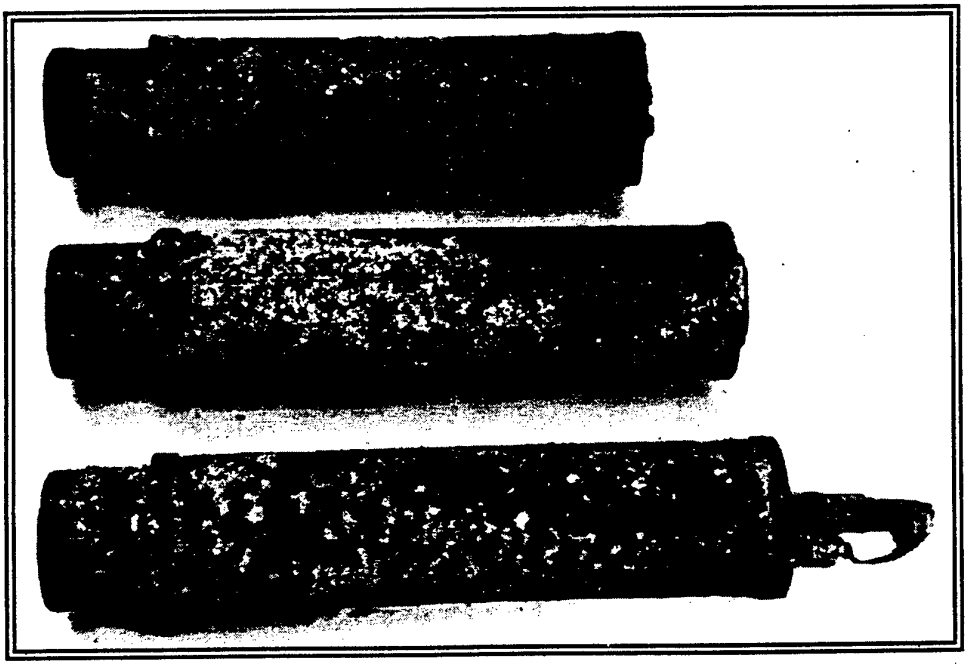
The zinc-alloy fuzes heading the 7,7 cm long German shells are very often corroded, so much so that they fall, helping the leak of their chemical loading.



Ce projectile chimique allemand de 25 cm pour Minenwerfer lourd était chargé en phosgène, dans une très mince enveloppe d'acier moulé puis usiné. La plupart des fuites se produisent au niveau du joint de la gaine d'éclatement, trop large pour la pression du chargement.

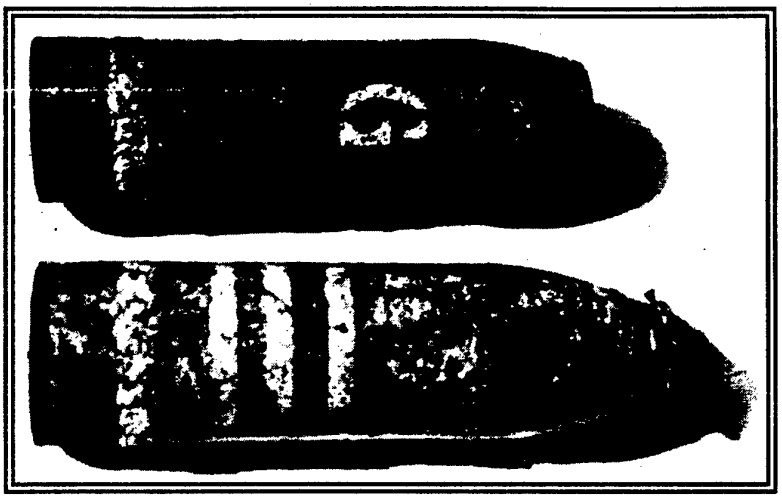
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This German cast steel chemical 25 cm projectile was filled with phosgene in a very thin walled tooled case. Most leaks happen by the exploder gain joint, too wide for the agent's pressure.



Les projectiles britanniques de 4 pouces pour mortiers Stokes peuvent tout autant laisser fuir du phosgène, de l'acide cyanhydrique décomposé, du lacrymogène ou du phosphore blanc.

The 4-inch British projectiles for Stokes mortars can just as well let phosgene, decomposed hydrocyanic acid, tear gas or white phosphor escape.



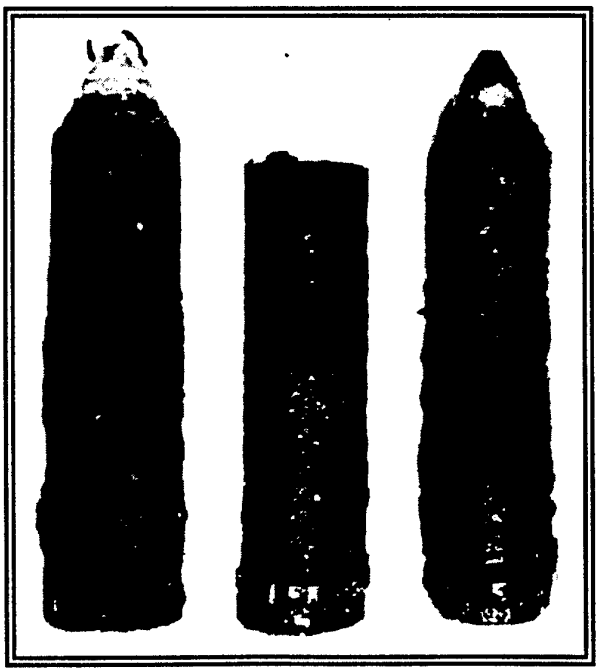
Les projectiles suffocants allemands de 7,6 cm pour Minenwerfer léger sont parfois retrouvés avec leurs marques encore lisibles. Ceci est toutefois exceptionnel, et la corrosion de l'enveloppe épaisse de seulement trois millimètres entraîne très souvent la fuite du chargement.

The german choking 7,6 cm. projectiles for light Minenwerfer are sometimes found with their marking still legible. This is however an exception and the corrosion of the only 3 mm thick sheath very often hads to loading leak.

* * *

Dans le cas des projectiles cylindriques à vase de plomb ci-dessous, la réaction électrolytique entre les deux métaux a favorisé le processus de destruction de l'enveloppe.

In the matter of lead vessel cylindrical projectiles, the electrolytic reaction between the two metals has helped the destruction process of the steel case.



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