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NDMA TREATABILITY STUDIES

DIRECTORATE OF ENVIRONICS

DECEMBER 1976

FINAL REPORT : JUNE 1975 - OCTOBER 1975

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AIR FORCE CIVIL ENGINEERING CENTER

(AIR FORCE SYSTEMS COMMAND)

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FLORIDA 32403

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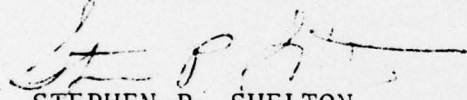
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PREFACE

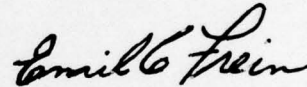
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This report has been reviewed by the Information Office (OI) and is releasable to the National Technical Information Service (NTIS). At NTIS it will be available to the general public, including foreign nations.

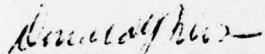
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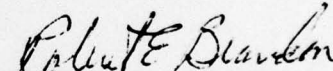
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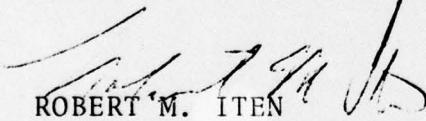
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SECTION I

INTRODUCTION

In the production of the missile fuel unsymmetricaldimethylhydrazine (UDMH), a by-product caustic waste containing an intermediate compound N-Nitrosodimethylamine (NDMA) is produced. Because NDMA has been identified as a suspected carcinogen, it is necessary to treat this waste to a very high degree (the limit of detection is approximately 20 $\mu\text{g}/\text{l}$). The waste of concern, generated in the production of UDMH, contains approximately 200 mg/l of NDMA, 25 percent sodium hydroxide and 5 percent nitrite. It was the purpose of this investigation to ascertain the feasibility of oxidizing the NDMA in this waste by use of the wet air oxidation process.

SECTION II

BACKGROUND ON WET AIR OXIDATION PROCESS

The original development work on wet air oxidation was done more than 20 years ago in the United States, specifically for application to industrial wastes. Figure 1 is a basic flowsheet for wet air oxidation. The waste is pumped into the system by a high pressure pump and mixed with air from the compressor. This mixture passes through a heat exchanger and then into a reactor where the oxygen in the air reacts with the organic matter in the waste. This oxidation is accompanied by an increase in temperature. The gas and liquid phase are separated after they leave the reactor. The liquid phase is then used as the energy source for the heat exchanger on the influent side of the reactor. Subsequently, both the gas and liquid phases are discharged from the process.

The degree of oxidation may be selected to meet waste treatment requirements; ranges from zero to near 100 percent are attainable as required. For example, in a typical domestic wastewater sludge, at a temperature of 150 degrees centigrade ($^{\circ}\text{C}$), approximately 10 percent of the organics will be oxidized with a 5-minute residence time. If the temperature is increased to 320°C , with all other conditions remaining constant, a 99 percent plus oxidation is attained.

It is necessary to maintain some liquid phase water in the system during oxidation. This requirement will determine system pressure. Operation pressures vary from 150 to 3000 pounds per square inch guage (psig).

The flow diagram shown by Figure 1 can be varied greatly. For example, Figures 2 and 3 illustrate variations to maximize energy recovery. In Figure 2, the cycle illustrated generates saturated superheated steam at about 600 psig, which may be combined with the cycle shown in Figure 3 for the production of electrical power.

The cost of a wet air oxidation system is proportional to the volume of the waste system, the required system pressure, the amount of air supplied, the time of retention, and the exhaust energy if used. The pressure and air quantity are related since a larger quantity of air usually indicates a higher degree of oxidation. This requires operation of the reactor at a higher temperature and pressure.

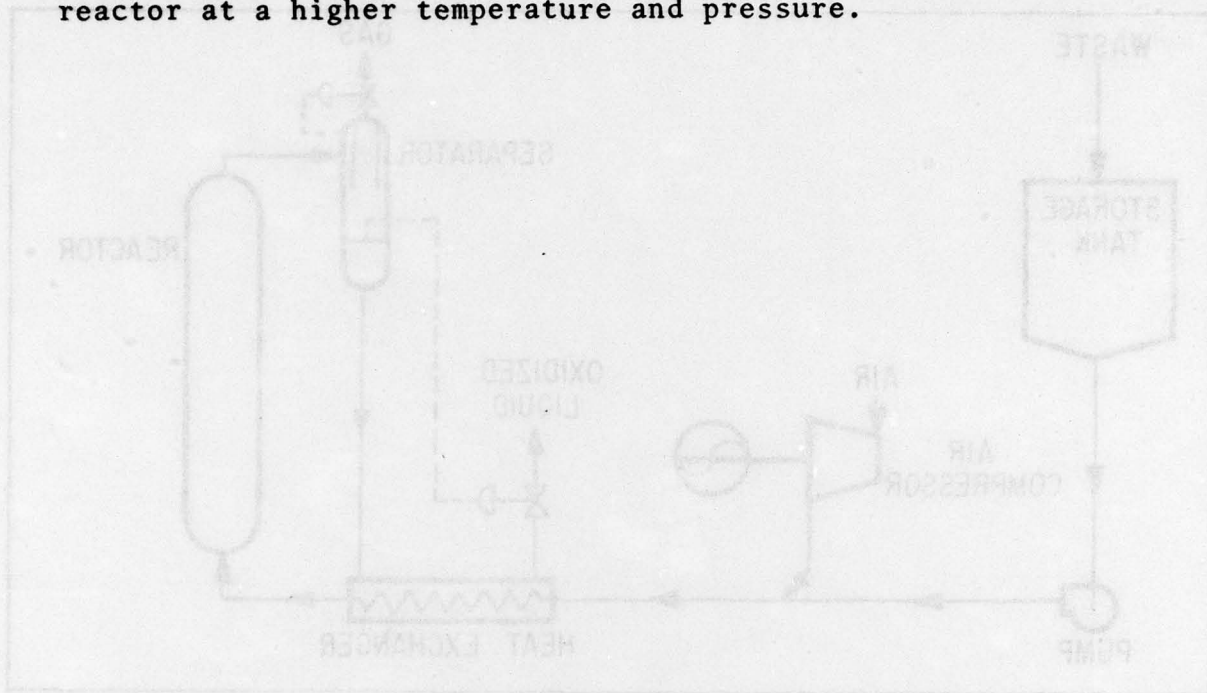


Figure 1. Flowchart of Wet Air Oxidation

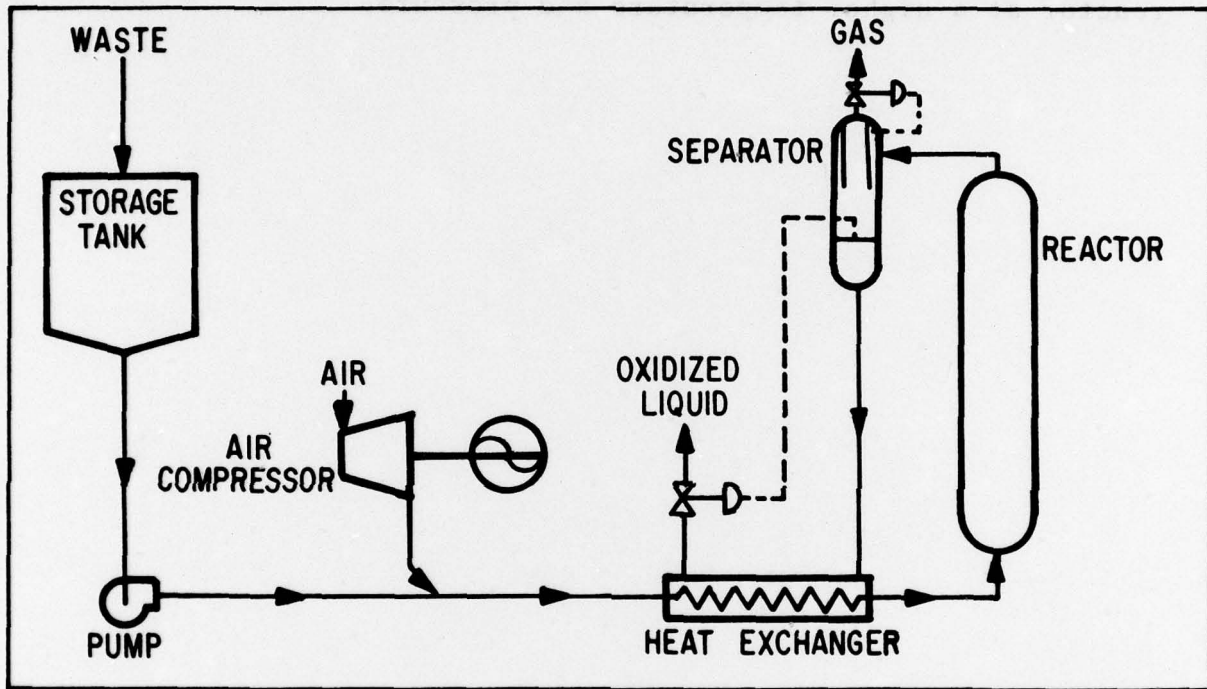


Figure 1. Flowsheet of Wet Air Oxidation

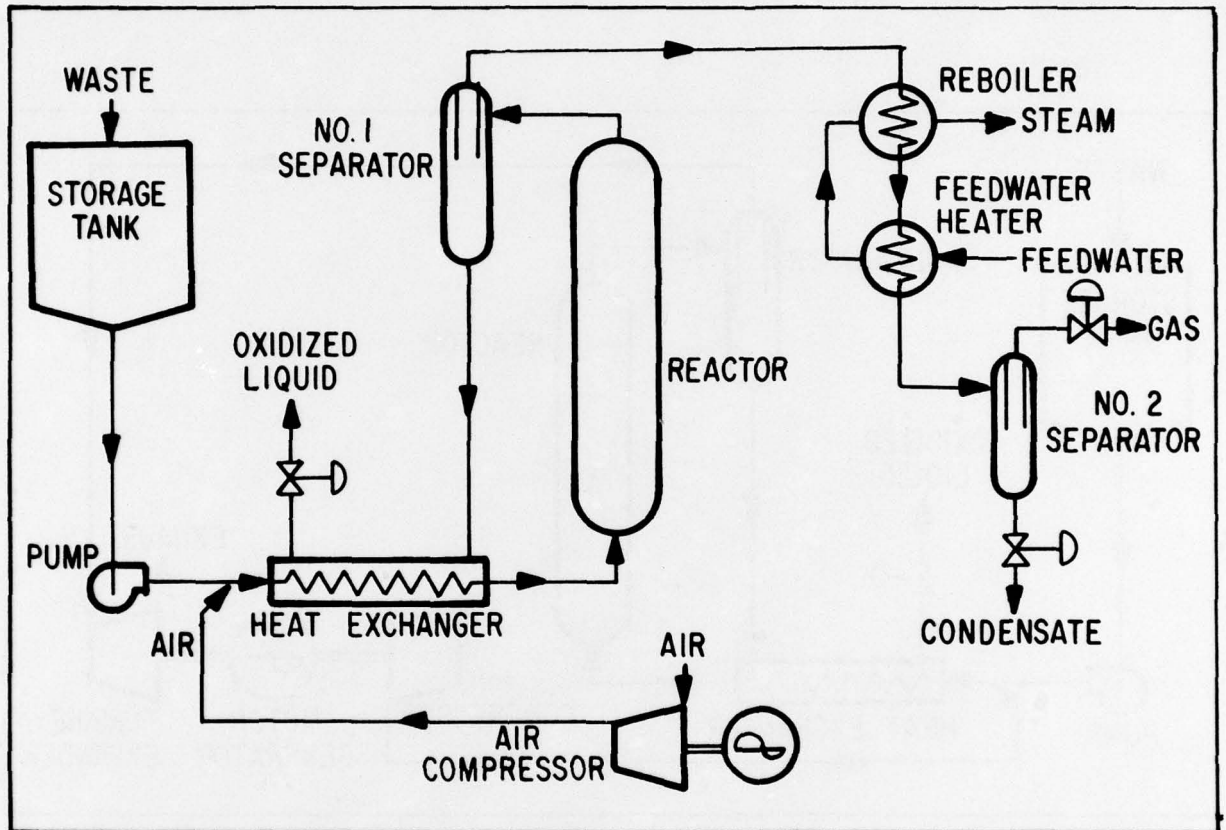


Figure 2. Flowsheet for Steam Generation

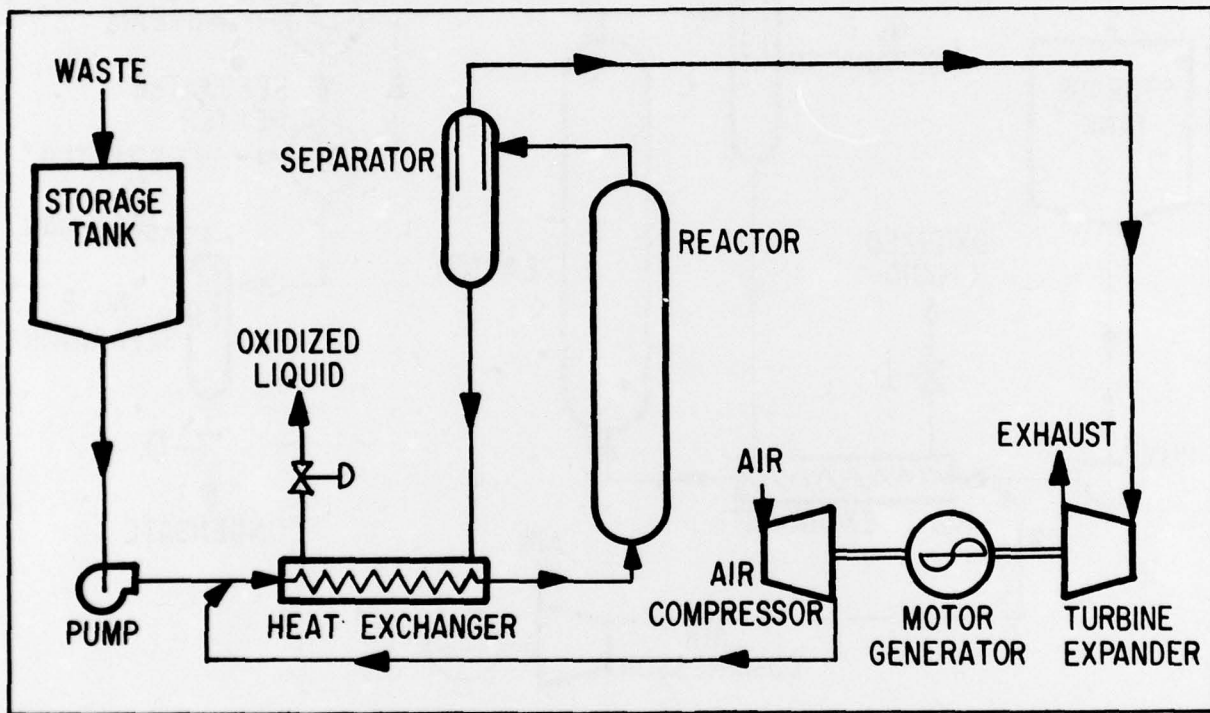


Figure 3. Flowsheet for Power Generation

SECTION III

DISCUSSION OF RESULTS

Ten waste samples were subjected to wet air oxidation by Zimpro, Inc, Rothschild Wisconsin. These oxidations were accomplished under an Air Force (AF) non-personal services contract. Each sample was analyzed independently by both the AF Civil Engineering Center (AFCEC) Analytical Chemistry Division (AFCEC/EVC) and by the contractor. The results of these investigations are shown in Table 1. The first four samples were oxidized to evaluate the effects of temperature and pH at a constant reaction time. These data indicate a significant oxidation of NDMA and can be attained at temperature in the 320°C range. Furthermore, pH modification and catalyst utilization did not significantly enhance the oxidation.

Using the data acquired in the first sequence, a longer reaction time at 320°C was indicated. Two subsequent evaluations were made on separate waste samples as shown in Table 1. From these data two significant observations can be made:

1. Initial sample characteristics are highly significant on oxidation success.
2. Duplication of results, with a high degree of accuracy, is difficult since the raw waste varies greatly.

Table 1. SAMPLE RESULTS

<u>Zimpro Sample No.</u>	<u>Identification</u>	<u>Oxidation Time, Hr.</u>	<u>pH</u>	<u>Temp., °C.</u>	<u>Cat.</u>	<u>N-NDMA, Air Force</u>	<u>Zimpro</u>
694-94-0	Feed	-	4	20	-	480.0 mg/l	510.0 mg/l
694-94-0	Feed	-	10	20	-	350.0 mg/l	370.0 mg/l
694-94-1	Oxidized	1	4	280	No	3.0 mg/l	3.0 mg/l
694-94-4	Oxidized	1	4	280	Yes	8.0 mg/l	7.5 mg/l
694-94-1	Oxidized	1	10	320	No	0.05 mg/l	1.0 mg/l
694-94-4	Oxidized	1	4	320	No	0.05 mg/l	1.0 mg/l
694-104-0	Feed	-	10	20	-		355.0 mg/l
694-105-7	Oxidized	2	10	320	No		0.6 mg/l
694-105-4	Oxidized	4	10	320	No		0.5 mg/l
694-105-1	Oxidized	6	10	320	No		1.4 mg/l
694-139-0	Feed	-	10	20	-	440.0 mg/l	-450.0 mg/l
694-139-1	Oxidized	2	10	320	No	-	- 2.0 mg/l
694-139-1	Oxidized	4	10	320	No	1.0 mg/l	- 1.0 mg/l
694-140-1	Oxidized	6	10	320	No	5.0 mg/l	- 0.5 mg/l

SECTION IV

CONCLUSION

From the data obtained in the preliminary investigation, it appears that the wet air oxidation process shows merit in treatment of NDMA. It is questionable if the process is capable of oxidation of NDMA containing waste to below detectible limits; however, it is important to recognize that oxidation to the 1.0 mg/l level is accomplished with relative ease. Thus, in conclusion, the potential use of wet air oxidation, as a treatment process for NDMA containing waste, would require better definition of the ultimate treatment objectives.

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