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CHARACTERIZATION OF PYROTECHNICALLY DISSEMINATED TEREPHTHALIC ACID AS RELEASED FROM THE M8 SMOKE POT

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M8 training smoke pots were detonated in the field to characterize the major by-products contained in the generated smoke. Previous studies on the M83 grenades had shown certain organic compounds to be present, specifically benzene and formaldehyde. With the dissemination of a larger amount of material from the pot (25 lb as compared to 360 g), organic vapor concentrations are also going to be higher. At the 8-ft sampling line, the concentrations of formaldehyde, benzene, toluene, carbon monoxide, and carbon dioxide were 34, 63, 1.2, 791, and 190 ppm respectively. However, using molecular modeling, concentrations were calculated to 50 ft. This distance is specified in the operational Standard Operating Procedure as the minimal distance that must be maintained from the pot when performing routine work. All concentrations of organic combustion products at this 50-ft distance were below their respective threshold limit values, therefore supporting the idea that the M8 is currently the safest training smoke pot used by the Army.

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PREFACE

The work described in this report was authorized under Project No. 1O162622A553, CB Defense/General Investigation. This work was started in May 1994 and completed in September 1994. The experimental data are contained in laboratory notebooks no. 93-0057 and 93-0070. All smoke pots were obtained from Pine Bluff Arsenal and are from lot no. PB-93G000E013. All safety requirements were followed for detonation of the smoke pots as described in Standard Operating Procedure No. CR9-1NP030-95J.

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CHARACTERIZATION OF PYROTECHNICALLY DISSEMINATED TEREPHTHALIC ACID AS RELEASED FROM THE M8 SMOKE POT

1. INTRODUCTION

The terephthalic acid mix (Table 1) contained in the M8 training smoke pot (Figure 1) was developed to replace the highly toxic HC mix in the M4A2 smoke pot.[1] The purpose of this study was to characterize the major by-products contained in the generated M8 smoke and to use the toxicological data from the M83 grenade to assess the toxic potential of the smoke.[2,3] They are both comprised of the same materials, with terephthalic acid being the major component; however, the smoke pot involves the dissemination of about 25 pounds of material while the smoke grenade disseminates about 360 grams of material. The major difference between the pot and the grenade is the 250 g of microcrystalline wax that is used to line the pot, but not the grenade. Previous studies on microcrystalline wax have shown that formaldehyde was a major combustion product of these waxes[4,5], and therefore could also appear in higher concentrations in the pot. This study will help characterize whether dissemination of a larger amount of material creates organic concentration levels above their respective TLV's (Threshold Limit Values), and whether trace components that could not be detected in the smoke grenades could now be found in the pots.

Although the Standard Operating Procedure (SOP) for the smoke pot describes that work must be performed past the 50 ft. radius, our samples were taken approximately 8 feet from the pot. This was done to assure that an adequate sample size would be attained to identify and quantitate any trace components. From this data, modeling was performed to assess how the components would theoretically disseminate over time, and what their concentrations would be at the 50 ft. mark.

Table 1- Components of the TA smoke mixture

Ingredient	Parts by weight
Terephthalic Acid	57
Magnesium Carbonate	3
Stearic Acid	3
Potassium Chlorate	23
Sugar	14
Polyvinyl Alcohol Binder	*

* Dissolved in water to form a 4.0% nominal solution. PVA content of completed mixture is approximately 1.0% on a dry weight basis.

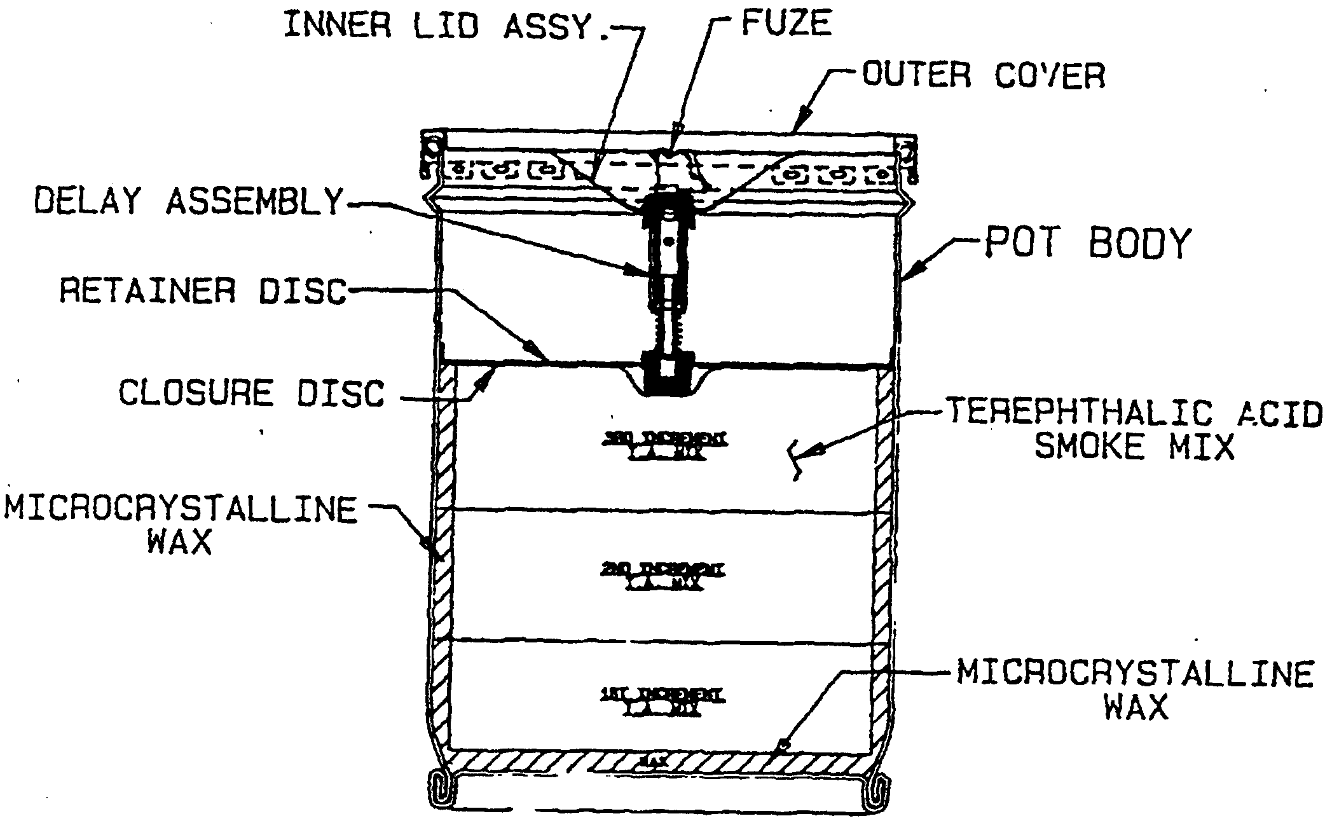

Figure 1 - Diagram Specifications for M-8 Smoke pot

2. MATERIALS AND METHODS:

2.1 Experimental Design:

On four separate occasions, the design for detonation and sample collection was identical for the four smoke pots. A small wooden house approximately 10 feet high was used to concentrate and collect the smoke from the pot. The front face of the house was open and fitted with a plywood tunnel extending outward from the front face (Figure 2). The pot was placed on the concrete slab at the front of the tunnel and detonated by trained Munitions personnel. The amount of smoke produced was in such high quantity that it was forced to travel down the 8 foot tunnel and into the wooden house. Before sampling, enough time was allowed to fill the house with smoke. Samples were collected at a distance of 8 ft. from the pot and at 4 ft. above ground level. Sampling dates, times and meteorological conditions are shown in Table 2. The total collection time for all samples was two minutes.

2.2 Collection / analysis of benzene and other volatile organic compounds (higher molecular weight)

A volume calibrated vacuum pump (Anderson) was used to draw and trap airborne components onto standard tenax collection tubes (SKC Inc. Cat. No. 226-35-03). Tenax sorbent tubes were used to collect a wide range of volatile organic materials (boiling points < 230 C). In order to collect different volumes of air, varying sized orifices were placed in front of the tubes (Figure 23). Higher collection volumes were necessary to analyze for trace components, while smaller collection volumes were necessary to analyze for more concentrated analytes (e.g. benzene). The orifices ranged in size so as to deliver air sampling volumes from 46 ml/min to 400 ml/min.

Table 2 - Meteorological conditions

Sampling date	Detonation time (24 hr clock)	Sampling start time after detonation (seconds)	Meteorological conditions
5/2/94	8:00	15 seconds	clear, sunny, 70°F, slight breeze
5/5/94	8:00	30 seconds	clear, sunny, 70°F, slight breeze
5/18/94	9:15	45 seconds	clear, sunny, 65°F, breezy
5/20/94	8:00	45 seconds	overcast, drizzle, 60°F, no breeze

Following sample collection, the tenax tubes were analyzed by thermal desorption Gas chromatography / Mass spectrometry (GC/MS). Instrument conditions are shown in Appendix A. Compounds present in the chromatograph were identified by mass spectrometry. Quantitative analysis was performed on the more concentrated components (benzene and toluene), while qualitative analysis was performed on the trace components.

2.3 Collection / analysis of aerosol concentrations

Aerosol concentrations of terephthalic acid (TA) Smoke Mix were determined from atmosphere air samples collected on 25 mm glass fiber filter pads. Aerosol samples were taken at four different locations over a one foot horizontal plane at flow rates of 683 ml/min, 1078 ml/min, 1225 ml/min, and 1300 ml/min. Samples were taken for two minutes using a constant flow vacuum pump. Filters were then weighed to determine aerosol concentrations.

2.4 Collection / analysis of formaldehyde, carbon monoxide and carbon dioxide (lower molecular weight organic compounds)

2.4.1 Collection

Vapor concentrations of the TA smoke combustion products were collected with glass cylindrical funnels (vol. 376-380 ml) at a flow rate of 1000 ml/min (Figure 3). The funnels were equipped with a stopcock at each end. Vapor samples were trapped in the glass cylinders by using electric solenoid valves to close the intake and exhaust ports of the

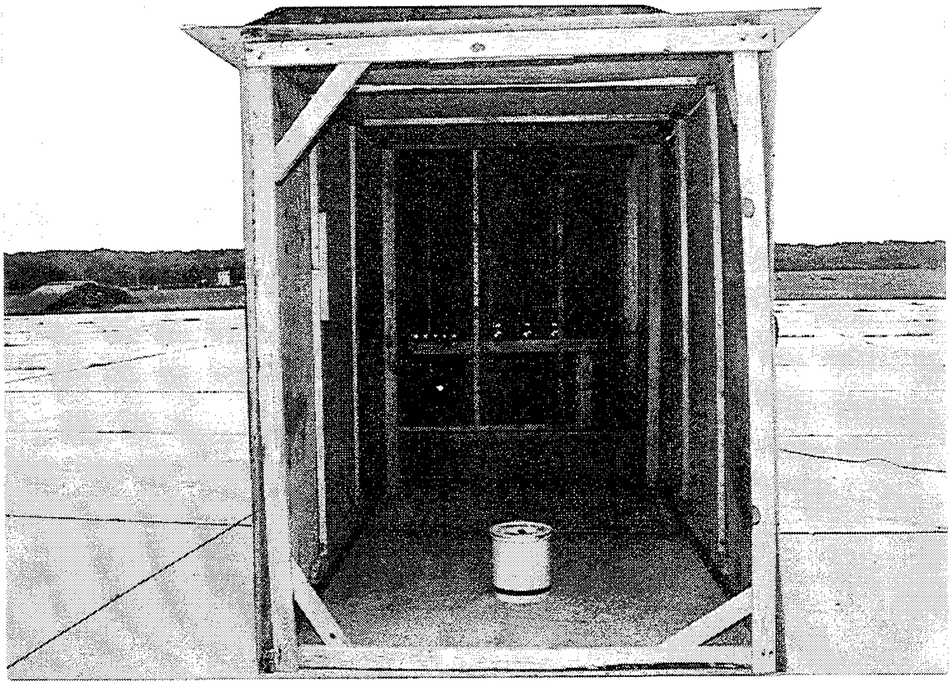

Figure 2 - Field setup for detonation of M8 Smoke pot

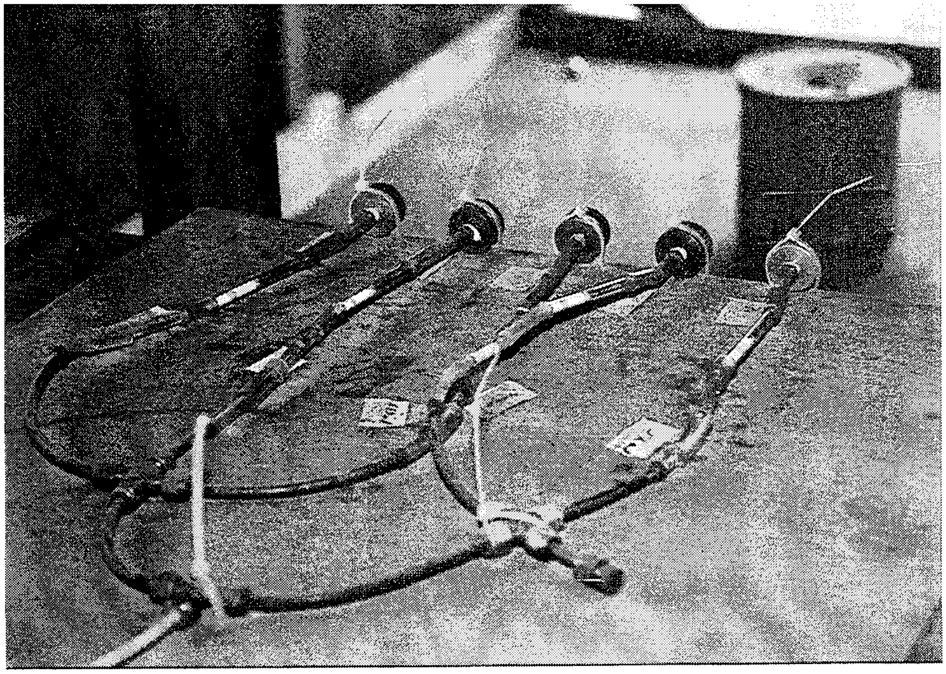

Figure 3 - Sampling apparatus for benzene and other volatile organics

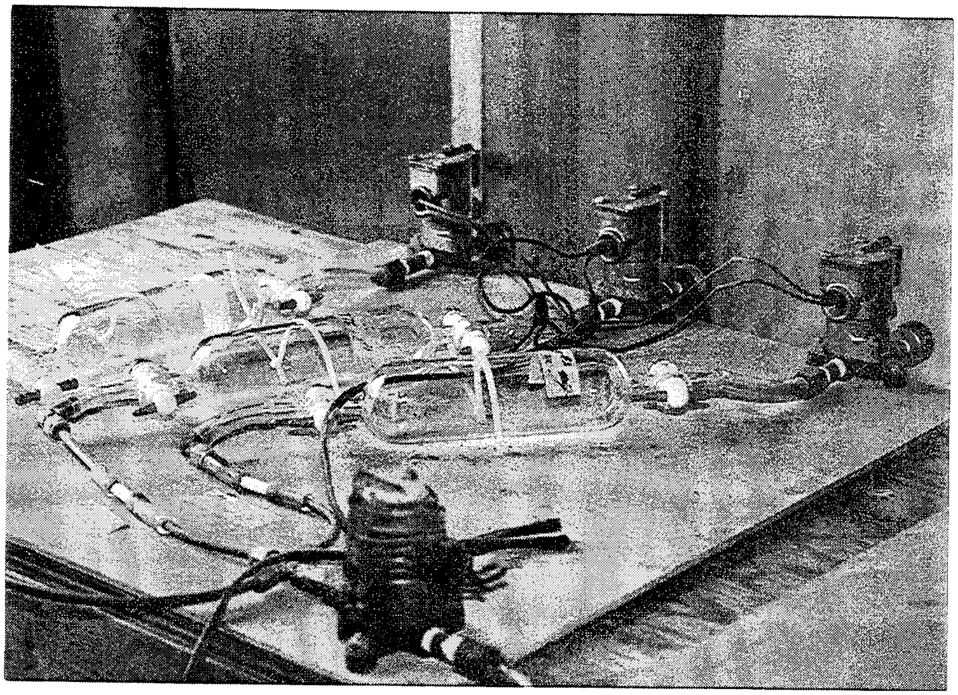

Figure 4 - Glass cylinder traps and filter pad assembly

funnels after sampling was complete. Samples were taken for two minutes using a constant flow vacuum pump. The gas contents from the glass cylinders were transferred and diluted into a 4.5 L Teflon® gas sampling bag (Alltech) with a 2 L gastight syringe (Hamilton®).

2.4.2 Analysis

Gas detector tubes were used to obtain concentration estimates for the three compounds. Since formaldehyde and carbon monoxide exhibited concentrations above their TLV's at the 8 ft. line, other analytical techniques were used to obtain more accurate concentration measurements. Carbon dioxide concentrations were much lower than it's TLV at the 8 ft. line, and did not need to be complemented with other analytical techniques.

2.4.2a Gas detector tubes for formaldehyde, carbon monoxide and carbon dioxide.

Gas sampler detector tubes (Kittagwa®) were connected via a small piece of tygon tubing to the gas sampler bag. The opposite end of the tube was connected to a 100 ml volume calibrated hand pump. A predetermined volume specified for each compound by Matheson was drawn into the detector tubes for analysis. The concentration of the desired compound was shown on the side of the detector tube through a colorimetric change. The concentration of each constituent was determined from the detector tube readout multiplied by the air volume dilution factor. For carbon dioxide concentrations, a blank was performed and subtracted from the detector tube concentration. Since there is a small concentration of carbon dioxide in air, this subtraction was necessary to obtain the actual concentration from the pot.

2.4.2b Gas Chromatography for formaldehyde

A calibrated vacuum pump was used to draw air samples from the gas sampling bags onto ORBO-22 (Supelco®) sorbent tubes. The flow rate was calibrated before and after sampling to deliver 50 ml/min for 20 min. The formaldehyde reacted with the sorbent material to form the derivative 3-benzyloxazolidine (BOZ). After collection, the sorbent material was transferred to a scintillation vial containing 1 ml of isooctane. The vial was sonicated for 45 minutes to assure complete solvent desorption. The extracts were then analyzed using Gas chromatography/FID (Appendix B for method conditions).

2.4.2c Determination of carbon monoxide with the AIM® Gas Detector

Carbon monoxide levels were also determined by an electro-chemical gas sensor detector. The detector sampled the smoke from the gas sampling bag for approximately 30 seconds to measure the concentration of carbon monoxide.

3. RESULTS

On observation, dissemination of the first three smoke pots each produced a dense white smoke which lasted for approximately 5 minutes. The burning appeared normal and dissemination of the material seemed fairly uniform. The fourth smoke pot, however, produced an intense 6-8 ft high orange flame 90 seconds into the run which lasted for 2 minutes. The duration of the burn was about 5 minutes, but after flaming, the smoke appeared black instead of white. Flaming caused the formation of soot (6"-12") in the tunnel after dissemination of the fourth pot.

3.1 Benzene and other volatile organic compounds (higher molecular weight)

3.1.a Quantitation of benzene and toluene

The chromatograms from the GC/MS analyses showed numerous organic combustion products present in the four smoke pots. Many of the various alkanes and alkenes that appear, are indicative of normal smoke processes.[6] At times, the background in the chromatograms rose throughout the run due to the presence of these common compounds. The computer software eliminated this problem by subtracting the background before quantitating the isolated components. Individual mass fragments specific for a certain compound were used for data processing of the peaks of interest. Background and unimportant alkanes/alkenes were therefore not processed, making data analysis much easier.

Of the multiple peaks present, the largest peaks found were identified by MS to be benzene and toluene. Tables 3 and 4 summarize the sampling and quantitative data. The first column describes the sample collection date and flow rate. For example, the file 50292ml means that the sample was collected on May 2 at a flow rate of 92 ml/min. The total volume drawn refers to the liters of gas sample collected directly from the pot. From the experimental data, the concentrations of benzene and toluene were calculated into ppm via Equation 1.

Equation 1: $$\text{ppm} = \frac{(\text{ug collected/total volume drawn})(24.46\ \text{L/mole})}{\text{Molecular wt. analyte}}$$

Table 3 - Benzene concentrations

Filename	Samp. time After deton.	Time sam. (minutes)	Total vol. drawn (liters)	Area	ug collected (ug)	Calc. conc. (ppm)
50292ml	15 sec.	2	0.184	53,200,000	23.4	39.9
502235ml	15 sec.	2	0.47	171,000,000	75.1	50.1
502400ml	15 sec.	2	0.8	408,000,000	179	70.2
502483ml	15 sec.	2	0.966	416,000,000	183	59.4
50452ml	30 sec.	2	0.104	32,100,000	14.1	42.5
50492ml	30 sec.	2	0.184	101,000,000	44.4	75.7
504156ml	30 sec.	2	0.312	153,000,000	67.2	67.5
51846ml	45 sec.	2	0.092	32,400,000	14.3	48.7
51852ml	45 sec.	2	0.104	40,800,000	17.9	54
51892ml	45 sec.	2	0.184	109,000,000	47.9	81.6
518400ml	45 sec.	2	0.8	428,000,000	188	73.7
52046ml	45 sec.	2	0.092			
52052ml	45 sec.	2	0.104	54,200,000	23.8	71.8
52092ml	45 sec.	2	0.184	99,200,000	43.6	74.3
520400ml	45 sec.	2	0.8	448,000,000	197	77.2

Table 4 - Toluene concentrations

Filename	Samp. time After deton.	Time sam. (minutes)	Total vol. drawn (liters)	Area	ug collected (ug)	Calc. conc. (ppm)
50292ml	15 sec.	2	0.184	563,000	0.664	0.97
502235ml	15 sec.	2	0.47	2,400,000	1.51	0.86
502400ml	15 sec.	2	0.8	peak tailing		
502483ml	15 sec.	2	0.966	16,700,000	8.08	2.25
50452ml	30 sec.	2	0.104	76,100	0.44	1.14
50492ml	30 sec.	2	0.184	507,000	0.638	0.93
504156ml	30 sec.	2	0.312	654,000	0.705	0.607
51846ml	45 sec.	2	0.092	284,000	0.535	1.56
51852ml	45 sec.	2	0.104	294,000	0.54	1.4
51892ml	45 sec.	2	0.184	600,000	0.681	0.995
518400ml	45 sec.	2	0.8	peak tailing		
52046ml	45 sec.	2	0.092	peak tailing		
52052ml	45 sec.	2	0.104	323,000	0.553	1.43
52092ml	45 sec.	2	0.184	564,000	0.664	0.97
520400ml	45 sec.	2	0.8	peak tailing		

Benzene concentrations ranged from 40-80 ppm and the toluene concentrations ranged from 0.5-2.3 ppm. Variations during field testing from wind direction /strength, temperature, and % Relative Humidity (RH) account for the variation in benzene and toluene levels. For toluene, since the concentration was well below its TLV (50 ppm) at the sampling site (8 feet), it is obvious that the concentration of toluene at the 50 foot line would also not be significant. However, benzene did exhibit a concentration value above its TLV (10 ppm) at the 8 foot mark, 1-3 minutes following dissemination. From mathematical models, the theoretical concentration range at the 50 foot line was approximately $8.63 \times 10^{-8} - 8.63 \times 10^{-7}$ ppm. Obviously, this concentration is far below the TLV for benzene.

3.1.b Qualitative determination for other volatile organic compounds (higher molecular weight)

In the four runs, there were other organic compounds identified in the smoke, but their concentrations were far below their TLV's to be considered harmful. Many of these, such as ethylbenzene and styrene, are also very volatile and are derivatives of benzene and toluene. No higher boiling organic compounds were detected.

3.2 Aerosol concentrations

The concentration of smoke produced in the house for the first three runs was calculated via Equation 2 and ranged from 5000- 9000 ug/L (Table 5). Due to flaming in the fourth smoke pot and subsequent ash formation, the aerosol concentration range (8000-13000 ug/L) was higher than for pots 1-3.

Equation 2:

$$\text{Conc. (ug/L)} = \frac{\text{wt. material (mg)} \times 1000\text{ug/ mg}}{\text{vol material (L)}}$$

3.3 Formaldehyde, carbon monoxide and carbon dioxide results

3.3a Matheson gas detector tubes

Tables 6a-6d depict the data obtained from the Matheson gas detector tubes. For carbon dioxide, each day's blank yielded a concentration of 450 ppm. This was subtracted from the detector tube concentrations to obtain the actual carbon dioxide concentrations from the pot. Carbon monoxide levels ranged from 553-950 ug/L, carbon dioxide levels ranged from 50-550 ug/L, and formaldehyde levels ranged from 55-95 ug/L.

3.3b Formaldehyde results from gas chromatography analysis

The concentration of formaldeyhde at the 8 foot line (Table 6) ranged from 26-42 ppm (32-51 ug/L). Equations 3 and 4 were used to calculate the amount of formaldehyde in ug/L and ppm respectively.

Equation 3:

$$\frac{\text{(ug/ml of BOZ)(1ml)}\dfrac{\text{(30g/mol formaldehyde)}}{\text{163 g/mol BOZ}}\dfrac{\text{(3000 ml)}}{\text{380 ml}}}{\text{Liters air sampled}} = \text{ug/L}$$

Equation 4:

$$\frac{\text{(Formaldehyde conc. ug/L)(24.46 L/mol)}}{\text{30 g/mol}} = \text{ppm}$$

As previously described in 2.4.2a, BOZ (3-benzyloxazolidine) is the derivative formed on the sorbent material when formaldehyde is collected from the air. The linear regression curve for BOZ, generated by injecting known standards into the GC, was used directly for the quantitation of formaldehyde. The 1 ml term in Equation 3 refers to the amount of isooctane necessary for solvent extraction of the derivative from the sorbent.

Table 5 - Aerosol concentrations

Samp date	Filter pad #	Wt material (mg)	Flow rate (ml/min)	Sam time (min)	Vol material (ml)	Conc (ug./L)
5/2/94	1	11.225	1038	2	2076	5407
5/2/94	2	15.111	1225	2	2450	6168
5/2/94	3	16.644	1300	2	2600	6402
5/2/94	4	20.904	1150	2	2300	9089
5/4/94	1	16.911	1038	2	2076	8146
5/4/94	2	19.61	1225	2	2450	8004
5/4/94	3	19.242	1300	2	2600	7401
5/4/94	4	9.864	683	2	1370	7200
5/18/94	1	12.711	1038	2	2076	6123
5/18/94	2	15.61	1225	2	2450	6371
5/18/94	3	15.242	1300	2	2600	5862
5/18/94	4	9.787	683	2	1370	7144
5/20/94	1	22.569	1038	2	2076	10871
5/20/94	2	30.052	1225	2	2450	12266
5/20/94	3	34.704	1300	2	2600	13348
5/20/94	4	11.53	683	2	1370	8416

Table 6a - Concentrations obtained on 5/2/94

Gas analyzed	Sam time (minutes)	Volume collected (# strokes)	Sample 1	Sample 3
Carbon monoxide	9	300 ml(3)	789 ppm	987 ppm
Carbon dioxide	5	100 ml(1)	50 ppm	250 ppm
Formaldehyde	3	300 ml(3)	67 ppm	67 ppm

Sample 2 was lost

Table 6b - Concentrations obtained on 5/5/94

Gas analyzed	Time sampled (minutes)	Volume (# strokes)	Sample 1	Sample 2	Sample 3
Carbon monoxide	9	300 ml(3)	789 ppm	789 ppm	789 ppm
Carbon dioxide	5	100 ml(1)	250 ppm	150 ppm	150 ppm
Formaldehyde	3	300 ml(3)	94 ppm	79 ppm	71 ppm

Table 6c - Concentrations obtained on 5/18/94

Gas analyzed	Time sampled (minutes)	Volume (# strokes)	Sample 1	Sample 2	Sample 3
Carbon monoxide	9	300 ml(3)	553 ppm	553 ppm	553 ppm
Carbon dioxide	5	100 ml(1)	50 ppm	50 ppm	50 ppm
Formaldehyde	3	300 ml(3)	55 ppm	79 ppm	71 ppm

Table 6d - Concentrations obtained on 5/20/94

Gas analyzed	Time sampled (minutes)	Volume (# strokes)	Sample 1	Sample 2	Sample 3
Carbon monoxide	9	300 ml(3)	871 ppm	871 ppm	950 ppm
Carbon dioxide	5	100 ml(1)	450 ppm	550 ppm	550 ppm
Formaldehyde	3	300 ml(3)	79 ppm	95 ppm	87 ppm

Table 7 - Formaldehyde concentrations using gas chromatography

Sam date	Total vol. drawn (liters) (from sam. bag)	Area	Raw conc. (ug/L) (Equation 3)	Calc. conc. (ppm) (Equation 4)
5/2/94	0.73 L	74000	50	41
5/4/94	1.46 L	185000	42	34
5/18/94	1.46 L	329000	32	26
5/18/94	2.94 L	137000	34	28
5/20/94	3.06 L	619000	51	42

The formaldehyde concentrations from the GC method (NIOSH # 2502) were used in the modeling program to predict formaldehyde concentrations at the 50 ft. line. Results show that approximately 4.10 x 10^{-8} - 2.93 x 10^{-6} ppm of formaldehyde is present at the 50 ft line, 1-3 minutes following smoke dissemination. This program predicts a formaldehyde concentration well below the TLV (0.3 ppm).

3.3c. Carbon monoxide results with the AIM Detector

Carbon monoxide levels ranged from 813-987 ppm. These results favorably compare with the concentrations obtained using the detector tubes. After averaging all carbon monoxide readings from both methods, the mean concentration of carbon monoxide present at the 8 foot line is 791 ppm. Modeling experiments show at the 50 foot line, 1-3 minutes following dissemination, approximately 4.39 x 10^{-8} - 3.14 x 10^{-6} ppm of carbon monoxide is present.

4. DISCUSSION

Over the past few years, there has been an extensive amount of research dealing with creating a safer training smoke for troops.[1,2] Terephthalic acid (TA) has emerged as the best possible material to produce good obscurant effects and to produce less toxic combustion products when compared to HC.[5] Our research group has published a technical report which details an animal inhalation study on the M83 (TA based) grenade.[2] The goal of this study was to determine whether dissemination of a larger amount of Terephthalic acid material (M8 smoke pot), would create hazardous chemical levels above the TLV's for those respective products, and whether other products not previously seen in the grenades appeared due to the dissemination of a larger material. The Threshold Limit Values (TLV's) for the combustion products identified in this study are shown in Table 8.

Table 9 summarizes the experimental concentrations at the 8 ft mark and the theoretical concentrations at the 50 ft mark of the identified combustion products. At the 8 ft. sampling site, the concentrations of benzene, formaldehyde, and carbon monoxide did exceed their respective TLV's . The Multiple Source Integrated Puff Model (MSIP) was used to estimate the concentrations of these combustion products 50 ft. from the dissemination source. Certain assumptions were made to run the model such as the dissemination rate of the compunds emitted from the pot. A best estimate of 1 g/sec emission rate was used. At 1-3 minutes following dissemination, concentrations for all of the identified combustion products were well below their respective TLV's at the 50 foot line. Over time, these concentrations shown in Table 8 would eventually become 0 as the materials were infinitely diluted in ambient air. The time to accomplish this out to a distance of 100 ft. was 17-18 minutes. Since the concentrations of carbon dioxide and toluene did not exceed their TLV's at the 8 foot sampling site, it was unnecessary to predict through the modeling program what their concentrations would be at the 50 ft. distance.

Table 8 - TLV's for studied organic compounds[7]

Organic compound	Time Weighted Average (TWA) (ppm)
Formaldehyde	*0.3
Benzene	10
Toluene	50
Carbon monoxide	25
Carbon dioxide	5000

*Threshold Limit Value - Ceiling

Note: For formaldehyde, since a time weighted average was not available, the threshold limit value - ceiling is shown.

Table 9 - Summary of experimental and theoretical concentrations

Organic compound	Experimental Concentrations (Average) (ppm)	
	8 ft line	**50 ft line (modeling)**
Formaldehyde	34	4.10×10^{-8} - 2.93×10^{-6}
Benzene	63	8.63×10^{-8} - 8.63×10^{-7}
Toluene	1.2	not calculated
Carbon monoxide	791	4.39×10^{-8} - 3.14×10^{-6}
Carbon dioxide	190	not calculated

The concentration range of Terephthalic acid at the 50 ft. line using this model was calculated to be 7.41×10^{-9} - 5.30×10^{-8} ppm. Again, this range is far below the TLV for Terephthalic acid(1.5 ppm). Obviously, there is no concentration reported at the 8 ft. line because it was not analyzed.

5. CONCLUSIONS AND RECOMMENDATIONS

After conducting the field tests, and subsequently analyzing the data, we were able to identify benzene, toluene, formaldehyde, carbon monoxide, styrene, ethylbenzene and carbon dioxide in the combustion products. At the 8 foot mark, benzene, formaldehyde, and carbon monoxide exceeded their TLV's, but toluene, styrene, ethylbenzene, and carbon dioxide did not. Through modeling exercises, all of the identified combustion products were predicted to be well below their TLV's at the 50 ft. mark. However, since TA is considered to be an irritant smoke, respiratory protection is still recommended at this mark.

At this time, terephthalic acid is the safest material for use as a training smoke in the M83 grenade and the M8 smokepot. We have found that dissemination of a larger amount of terephthalic acid in the pot does not create hazardous concentration levels at the 50 foot distance. Other organic compounds (i.e. styrene and ethylbenzene) were identified in the combustion products of the M8 pot, but their concentrations were negligible to be considered hazardous.

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6. Robbat, A., Jr., Liu, T-Y, and Abraham, B.M., "Development of an On-Site Gas Chromatography /Mass Spectrometer for Rapid Detection of Polycyclic Aromatic Hydrocarbons and Polychlorinated Biphenyls at Hazardous Waste Sites" Anal Chem. Feb. 1992.

7. American Conference of Government Industrial Hygienists, 1994-1995 Threshold Limit Values for Chemical Substances and Physical Agents and Biological Exposure Indices, Cincinnati Oh.

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APPENDIX A

GC/DETECTOR CONDITIONS:

Column: HP-5MS
20m x 0.25mm x 0.25um

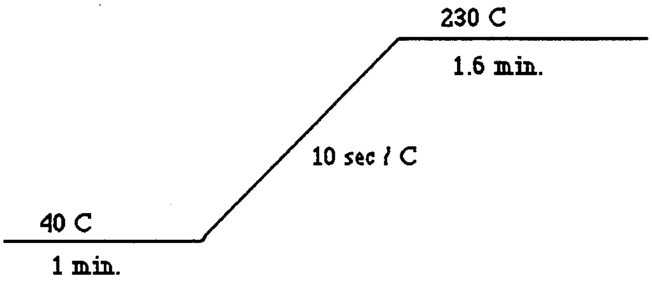
Carrier Charcoal filtered air

Split ratio: 100:1

Gas flow: 1 liter/min

Desorption temp. (for injection) 240 C

Temp. program:



DETECTOR CONDITIONS:

Type: Mass Spectrometer (manufactured by Bruker Instruments Inc.)

Source: Electron ionization

Temperature: 180 C

Scan Range: 45-400 amu

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APPENDIX B

GAS CHROMATOGRAPH CONDITIONS

Instrumentation:	HP5890 with 3396A Integrator
Column:	Restek 30m x 0.25mm x 0.25um Stabilwax, Capillary
Gas Flows:	
Helium Carrier:	20 psi.
Column Head Pressure:	16 psi.
Total flow:	126 ml./ min.
Septum flow:	3-5 ml./ min.
Column flow:	90 ml./ min.
Operation Mode:	Splitless
Detection:	Flame ionization (FID)
Detector flows:	
Hydrogen:	16 psi.
Air:	40 psi.
Injector temperature:	180 C
Detector temperature:	280 C
Purge off:	0.0 min.
Purge on:	1.0 min.
Temperature program: