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TECHNICAL REPORT 9213

CHEMICAL AND PHYSICAL CHARACTERIZATION OF AN EXPERIMENTAL WHITE SMOKE FORMED FROM COMBUSTION OF AMMONIUM PERCHLORATE, AMMONIUM CHLORIDE, AND CARBOXY-TERMINATED POLYBUTADIENE

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Smoke was produced from a candidate smoke material, composition C28, in a laboratory glove box; and the chemical and physical characteristics of the smoke were determined in order to evaluate its potential for health hazards to exposed soldiers. The C28 smoke system, developed by the U.S. Army Chemical Research, Development and Engineering Center (USACRDEC) as a candidate to replace hexachloroethane smoke systems, consists of ammonium perchlorate (62 percent by weight) and ammonium chloride (20 percent) in a matrix of carboxy-terminated polybutadiene (18 percent). Miniature grenades provided by the USACRDEC contained 10 g of the C28 mixture plus a 1-g starter mix and an electric match. These canisters were electrically ignited in a Class III glove box. Samples were taken for off-line analysis for anions, cations, and organic compounds and for determination of particle size distribution; hydrogen chloride and carbon monoxide were measured using real-time instruments. The smoke produced by the 10-g C28 canister consisted mainly of ammonium chloride particles with a mass median diameter of 1 µm. Ammonium chloride is regarded as a mild respiratory irritant.

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However, 15 percent of the chloride in the smoke was present as hydrogen chloride. This is sufficient to make the smoke an irritant to the eyes, skin, and respiratory tract. The experimental results differed markedly from the predictions of a commonly-used model, which predicted no ammonium chloride in the smoke. A number of polycyclic aromatic hydrocarbons (PAHs) were produced by the 10-g canisters, and the total predicted mass of PAHs from a full-size smoke grenade would be comparable to the mass of PAHs produced by burning 1.0 kg of wood under starved air conditions in a residential wood stove. Mass and configuration of the smoke munition influence the temperature profile during the burn, which is a major factor determining the products of combustion. It is possible, therefore, that smoke produced by full-scale grenades could be substantially different from the smoke produced in these laboratory-scale experiments.

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INTRODUCTION

SMOKE COMPOSITION C28

The smoke produced by an experimental smoke composition, designated C28, which was a candidate to replace the hexachloroethane (HC) fill in the M8 smoke grenade and the M4A2 smoke pot, was characterized at the U.S. Army Biomedical Research and Development Laboratory (USABRDL). The C28 fill mixture, developed by the U.S. Army Chemical Research, Development and Engineering Center (USACRDEC), consists of ammonium perchlorate (62 percent by weight), ammonium chloride (20 percent), and carboxy-terminated polybutadiene (18 percent). There has been no toxicologic evaluation of the smoke produced by the C28 composition, and prior attempts to chemically characterize the smoke were not successful[1].

NASA-LEWIS MODEL

A computer model (NASA-Lewis algorithm[2]) developed for use with rocket engines was used by Picatinny Arsenal[3] to predict the exhaust products of the C28 mixture with the results shown in Table 1. It should be noted that ammonium chloride, which was projected to be the major obscurant ingredient in the smoke, was not among the compounds predicted by this model. Because the conditions and assumptions of the NASA-Lewis model are not necessarily those that would apply when the C28 mixture is burned in a smoke grenade or pot, experimental determination of the chemical composition and physical nature of the smoke was considered necessary as a first step in the evaluation of potential health hazards presented by the smoke.

TABLE 1. C28 Smoke Components and Concentrations Predicted by the NASA-Lewis Algorithm[3]

Component/ Compound	Mole Fraction ppm	Mass Fraction	Mass (mg) in Smoke 10-g Test	Undiluted Concentration mg/L
Hydrogen	313560	.01707	170.71	14.11
Carbon monoxide	229890	.33540	3354.04	277.20
Hydrogen chloride	183310	.36102	3610.25	298.38
Water	144560	.14067	1406.73	116.26
Nitrogen	90990	.06884	688.42	56.90
Carbon dioxide	29190	.06939	693.92	57.35
Methane	7110	.00616	61.61	5.09
Ammonia	1230	.00120	11.98	0.99
Hydrogen cyanide	100	.00015	1.46	0.12
Formaldehyde	40	.00006	0.65	0.05
Cyanic acid	10	.00002	0.24	0.02
TOTALS	999990	1.00000	10000.00	826.48

PLANNED EXPERIMENTS

Two experimental phases were planned in order to obtain data on the composition of the smoke produced by the C28 composition. The first phase was a laboratory-scale experiment in which smoke produced by burning 10-g samples of the C28 mixture in a Class III glove box was analyzed. The second phase of experimentation, in which full-size grenades with the C28 fill were to be functioned outdoors, using camping tents as environmental enclosures, was not carried out because further development of the C28 composition was abandoned in favor of a smoke composition based upon terephthalic acid. The first phase was completed and is reported here.

MATERIALS AND METHODS

GLOVE BOX

The C28 canister smoke characterization experiments were conducted in a glove box (Lab-Guard Class III, Model NU-701, NuAire, Inc., Plymouth, MN). The 10-g canisters were supplied by the USACRDEC and were numbered starting with No. 3580 GT 8911301. These canisters were equipped with an electric match which served as a means of functioning them remotely. Samples taken during each of the experiments with the canisters were identified by the last three numbers in the USACRDEC canister number. The volume of the glove box was 500 liters.

SAMPLING TRAINS

The following sampling trains, shown schematically in Figure 1, were used to acquire samples for later analysis:

1. Glass fiber filters followed by low-flow sampling pumps

Two low-flow sampling pumps were placed inside the glove box, each equipped with glass fiber filters followed by charcoal adsorption tubes (Orbo-32 "large").

2. Glass fiber filters followed by midget impingers

Three low-flow sampling pumps were placed inside the glove box with midget impingers filled with either distilled water, a cation absorbing solution (12 mM HCl/0.5 mM diaminopropionic acid monohydrochloride), or an anion absorbing solution (3.9 mM $NaHCO_3$/3.1 mM Na_2O_3), preceded in each case with a glass fiber filter.

3. Miniature impinger without pre-filter

One low-flow sampling pump was placed inside the glove box with a midget impinger filled with distilled water and no pre-filter. All attempts to take a sample in this manner resulted in pump failure, probably due to pump damage caused by aerosol particles which bypassed the impinger and entered the pump.

4. Cascade Impactors

Two In-Tox Model No. 02-140, seven-stage cascade impactors (In-Tox Products, Albuquerque, NM) were placed inside the glove box connected to high-flow sampling pumps outside the glove box.

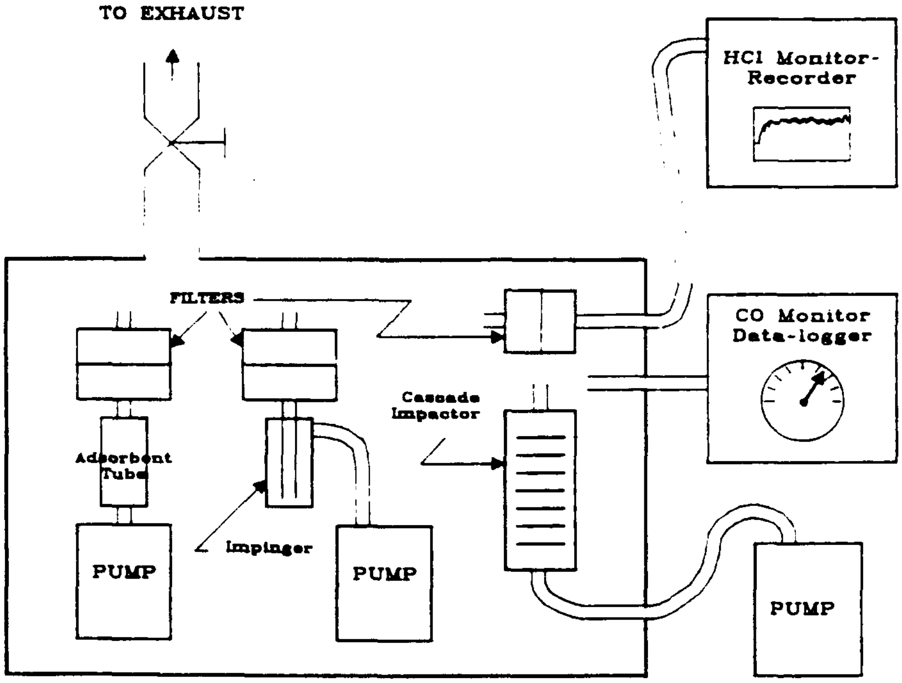

Figure 1. Schematic Diagram of Air Sampling Trains and On-line Instrumentation

ON-LINE INSTRUMENTATION

The following on-line instrumentation, also shown schematically in Figure 1, was used to monitor hydrogen chloride (HCl) and carbon monoxide (CO).

1. HCl Monitor

The intake probe and pre-filter of a USABRDL HCl monitor[4] were placed inside the glove box. The monitor, its internal sampling pump, and a strip-chart recorder were located outside the glove box.

2. CO Monitor

The intake probe of a CO101 (Neotronics, Gainesville, GA) CO monitor was placed inside the glove box. The monitor (with internal pump and in-line particle filter) and a data-logger were located outside the glove box.

EXPERIMENTAL PROCEDURE

All sampling pumps were calibrated using a Gilibrator primary flow calibrator (Gilian Instruments Corp., Wayne, NJ) with the sampling trains in place. The canisters were held by clamps on a ring stand inside the glove box. Leads attached to the terminals of the electric match were passed through the door of the box. The pumps for the filters, adsorption tubes and impingers were turned on and these sampling trains were placed in the box; all doors and openings were sealed; and ignition was brought about by touching the leads to the terminals of a 9-V dry cell. The pumps for the cascade impactors were turned on one minute after ignition for 10-15 sec. The CO and HCl monitors were operated from the time of ignition. Temperature and humidity in the box were not measured or controlled. After a predetermined time (usually 10 min) the exhaust valve for the glove box was opened, the hood blower to which it was connected was turned on, and the door of the glove box was opened to allow the glove box atmosphere to be replaced with fresh ambient air. The pumps with sampling trains attached were removed and re-calibrated.

SAMPLES

Filter samples were divided among those for inorganic ion analysis and those for organic compound determination. Usually, the filters from the trains with adsorbent tubes were used for analysis of organic compounds and those from the trains with impingers were taken for specific ion chromatography. The filter samples were cut in half and one half was sonicated for 10-15 minutes in 5 mL of anion eluant and the other in cation eluant. The two halves were analyzed for chloride or ammonium, respectively, using the ion chromatograph. The impinger trapping solutions were made up of distilled water, cation eluant solution, or anion eluant solution. The distilled water impinger samples were used to determine pH, and the other solutions were analyzed by ion chromatography for the cations or anions. The mass deposition on each stage of the cascade impactors was assumed to be proportional to the chloride ion on the stage. The glass slides from the cascade impactor stages were sonicated for 10-15 minutes in 2 mL of anion eluant, and the resulting solutions were analyzed for chloride using the ion chromatograph.

A calibration curve for the HCl monitor was constructed in the range of the expected HCl concentrations, and the strip chart recording in mV was converted to HCl concentration. The CO monitor is direct reading, and the CO concentration was recorded on a data-logger and then read into a personal computer which reduced the data and produced a report of CO concentration during each second of the run.

The filter samples to be analyzed for organic compounds were transferred to 5-mL screw-cap vials. Four mL of methylene chloride was added to each vial and the contents were vortexed vigorously for 2 min. The solvent extracts were filtered through 0.45 μm glass fiber filters, evaporated to dryness, and reconstituted in 1.0 mL of acetonitrile. The acetonitrile solutions were analyzed for polycyclic aromatic hydrocarbons (PAHs) in a Waters liquid chromatographic system (Waters Chromatography Division, Milford, MA), consisting of two Model 600A solvent delivery systems and a Model 710B Wisp autosampler. The fluorescence detector was a Hewlett-Packard Model 1046 Programmable Fluorescence Detector (Hewlett-Packard, Avondale, PA) with filters set at 240 nm excitation and 370 nm emission. A Supelcosil LC-PAH column (25 cm x 4.6 mm i.d., 5 μm particle size, Supelco, Inc., Bellefonte, PA) was used to separate the PAHs. A gradient elution program was used in which the eluent was changed from 34 percent acetonitrile/water after a 2-minute hold to 100 percent acetonitrile in 25 minutes at 2 mL/min. The injection volume was 5 μL. HPLC grade acetonitrile (Baxter Healthcare Corp., Burdick and Jackson Division, Muskegon, MI) was used without further purification. Water for the HPLC was purified with a Mill-Q™ water purification system (Millipore Corp., Bedford, MA). The standard mixture for the PAHs was purchased from Supelco, Inc., Bellefonte, PA.

The carbon adsorption tubes from two experimental runs were desorbed and subjected to mass spectroscopy using a Hewlett-Packard model 5970 mass selective detector equipped with a Hewlett-Packard Model 5890A Series II gas chromatograph. Compounds were separated on a Hewlett-Packard Ultra 2 (cross-linked 5 percent phenyl methyl silicone gum phase) 25 m x 0.2 mm i.d. x 0.33 μm film thickness capillary column.

RESULTS

FILTER SAMPLES

The results were reported as total mass of analyte per filter, impinger, or adsorbent tube, as appropriate. The volume of chamber air sampled was calculated by taking the average of the pumping rates (liters/min) before and after sampling and multiplying by the number of minutes between ignition and the start of the emptying cycle. The analytical data was then converted to concentration of the analyte in the chamber air; and finally, by multiplying the mass of analyte per liter by the 500-L volume of the glove box, the mass of analyte produced by the 10-g smoke canister was calculated. These results are summarized in Table 2, which also shows the projected mass of each major constituent that would be produced by a 500-g grenade.

GAS PHASE ORGANICS

Compounds that were identified by gas chromatography/mass spectroscopy included ethynyl benzene, isocyanobenzene, and chlorobenzene.

Table 2. Summary of Analytical Data for C28 Experiments and Projected Mass Produced by 500-g C28 Grenade

Constituent	Amount (g) Produced by 10 g C28	Projected Mass (g) in 500-g Grenade
Ammonium Chloride	1.763	88.2
HCl	0.375	18.8
CO	1.264	63.2
Carbonaceous residue	0.650	32.5
PAHs	0.0006	0.03

POLYCYCLIC AROMATIC HYDROCARBONS (PAHs)

Several PAHs were identified in the samples of particulate matter collected on filters in the glove box. The total PAH content varied from experiment to experiment, but the relative masses of the PAHs stayed about the same. The masses of identified PAHs produced by several of the 10-g canisters are summarized in Table 3.

ION ANALYSIS

Table 4 summarizes the mean concentrations of the two major ions.

Table 4. Ammonium and Chloride Ion Concentrations from Filter Samples

Run No.	mg Cl^-/L air	Std Dev	mg NH_4^+/L air	Std Dev	Molar Ratio NH_4^+:Cl^-
701	2.62	.17	1.17	.05	.88
702	1.57	.02	.68	.04	.85
703	1.10	.04	.55	.04	.98
801	8.39	1.62	3.67	.49	.96
704	.46	.02	.23	.02	.97
802	.46	.04	.24	.03	1.00
AVG	2.44		1.09		.92
Std Dev	3.03		1.31		.10

Table 3. Polycyclic Aromatic Hydrocarbons Extracted from Particulate Samples Taken from C28 Smoke
Mass of PAHs (mg) Produced by One 10-g Canister

SAMPLE	305F mg/can	701F5 mg/can	702F1 mg/can	703F1 mg/can	703F2 mg/can	704F1 mg/can	704F2 mg/can	MEAN mg/can	IARC Evaluation[a]
Fluorene		.1014	.0906	.3478	.3623	.1442	.1268	.1955	3
Fluoranthene	.08	.1268	.1159	.4022	.3696	.0580	.0580	.1731	3
Pyrene	.09	.0543	.0507	.1558	.1667	.0826	.0721	.0962	3
Phenanthrene	.16	.0188	.0159	.0120	.0134	.0359	.0366	.0419	3
Dibenzo(a,h)anthracene		.0130	.0116	.0214	.0221	.0264	.0236	.0197	2A
Indeno(1,2,3-cd)pyrene	.01	.0065	.0058	.0181	.0210	.0120	.0109	.0127	2B
Benzo(a)pyrene	.02	.0065	.0047	.0130	.0214	.0083	.0072	.0116	2A
Benzo(b)fluoranthene	.01	.0040	.0040	.0163	.0159	.0120	.0120	.0107	2B
Anthracene	.01	.0058	.0054	.0170	.0203	.0072	.0058	.0106	3
Benzo(g,h,i)perylene		.0080	.0069	.0058	.0112	.0141	.0130	.0098	3
Chrysene		.0043	.0043	.0134	.0149	.0152	.0054	.0096	3
Benzo(a)anthracene		.0018	.0018	.0047	.0062	.0141	.0149	.0072	2A
Benzo(k)fluoranthene	.01	.0018	.0018	.0069	.0065	.0062	.0054	.0054	2B
Total Identified PAHs	0.39	0.3530	0.3194	1.0344	1.0515	0.4362	0.3917	0.6040	

[a] International Agency for Research on Cancer (IARC) evaluations of carcinogenic risk[5,6]:
3 = The agent is not classifiable as to its carcinogenicity in humans.
2B = The agent is possibly carcinogenic to humans.
2A = The agent is probably carcinogenic to humans.

PARTICLE SIZE DISTRIBUTION

Complete particle size distribution information was obtained for two experiments, those using canisters 3850GT8911704 and 3850GT8911801. The respective mass median aerodynamic diameters (MMAD) and geometric standard deviations (in parentheses following the MMAD) were 1.08 μm (1.68) and 0.90 μm (1.39). The particle size distributions are shown in Figures 2 and 3. These results are typical of aerosols produced by condensation in the atmosphere of gaseous products of combustion, and they indicate that respirable particles accounted for greater than 90 percent of the mass of particles in the C28 smoke.

CARBON MONOXIDE

Figure 4 shows the time profile of CO concentration in the glove box following ignition of the canister. The initial spike, which shows the CO concentration rapidly rising above 1200 ppm and then falling just as rapidly, can be attributed to the starter mixture. The CO concentration rose again to 2800 ppm and then leveled off at 2400 ppm until the glove box was emptied. The peak concentration may reflect incomplete mixing at the point where the CO probe was placed, and the plateau concentration is probably a more accurate representation of the CO produced from burning the C28 mixture.

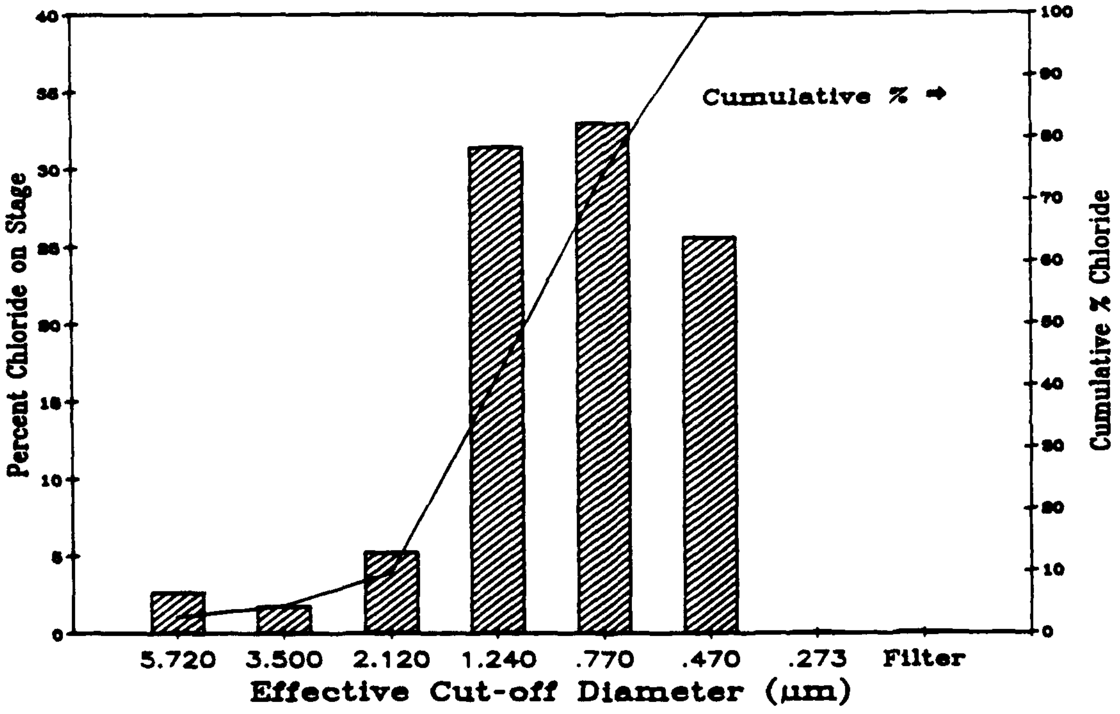

Figure 2. Particle Size Distribution Histogram for Canister No. 704

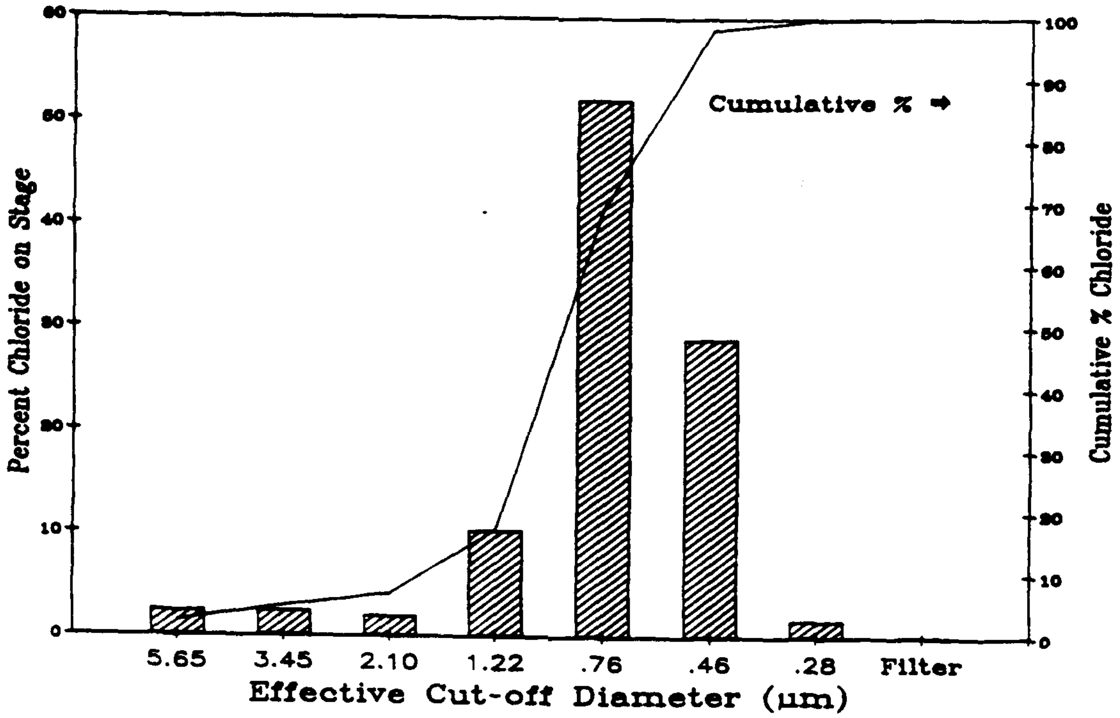

Figure 3. Particle Size Distribution Histogram for Canister No. 801

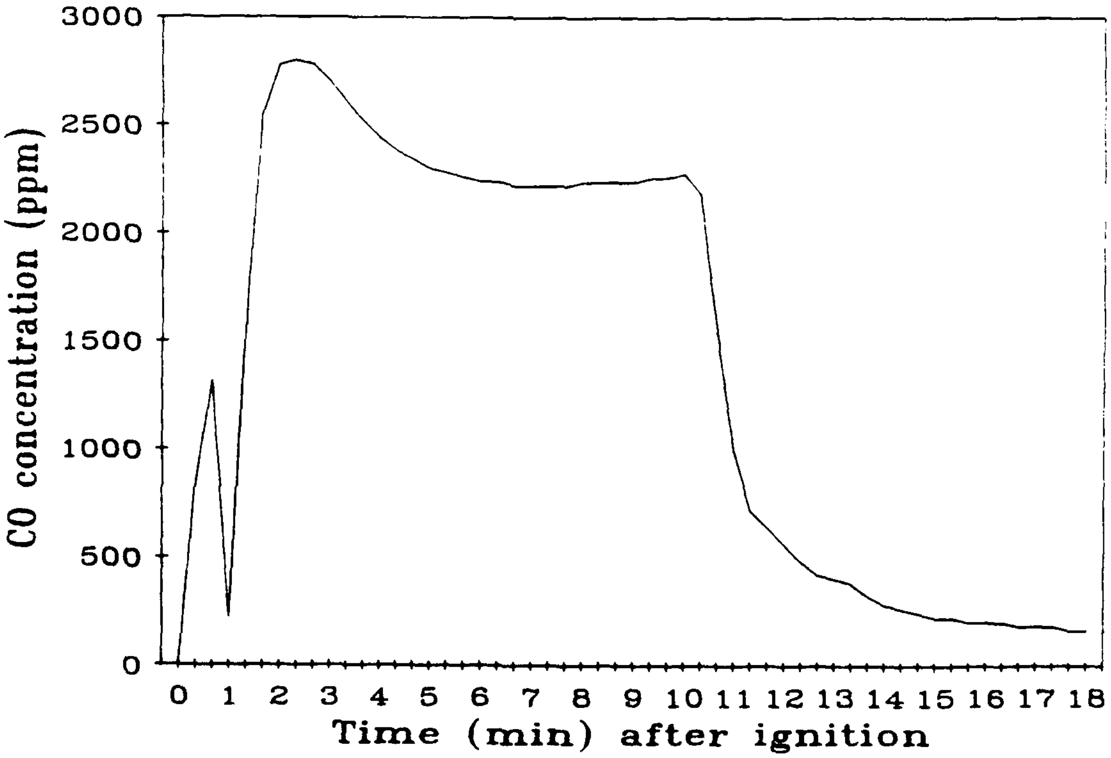

Figure 4. Profile of Carbon Monoxide Concentrations During One Glove-box Experiment - Canister No. 703

DISCUSSION

AMMONIUM CHLORIDE: THE MAJOR CONSTITUENT OF C28 SMOKE

Results of these laboratory-scale experiments indicate that the major ingredient of the smoke produced by the 10-g C28 canisters is ammonium chloride (NH_4Cl). Hydrogen chloride vapor (HCl) accounts for about 15 percent of the chloride in the smoke. Although the individual particles were not examined, it is likely that additional HCl was adsorbed on the surface of the NH_4Cl particles. This supposition is supported by the facts that (1) the ratio of ammonium to chloride in the filter samples was never more than and usually less than one, and (2) by the observation that the solid particles adhering to the stainless steel surfaces of the glove box deliquesced upon standing. The liquid droplets so produced etched into the surface of the stainless steel in a period of 24 hours.

COMPARISON OF EXPERIMENTAL RESULTS WITH NASA-LEWIS MODEL

Comparison of Tables 1 and 2 shows that the NASA-Lewis algorithm was not a good predictor of the combustion products of the C28 formulation under the conditions of this experiment. Under the conditions modeled by the NASA-Lewis algorithm, all of the ammonium moiety, from the ammonium chloride in the starting mixture and from the breakdown of ammonium perchlorate, would appear to be converted to its elements or to simple oxidation products of nitrogen and hydrogen, with a small quantity of gaseous ammonia. Our experiments showed that ammonium was the major cation in the collected particles. These results should act as a caution against reliance upon such modeling efforts to assess the potential health hazards of similar pyrotechnically deployed smoke munitions in the absence of chemical analysis.

PAHs

As noted in Table 3, many of the PAHs found in the smoke are considered carcinogens in man[5,6]. The mean individual and total PAHs produced by the 10-g sample can be multiplied by 50 to project the mass of PAHs that would be produced by a grenade with a 500-g fill of C28. The total PAH mass (30.2 mg) and the mass of benzo(a)pyrene (0.58 mg) are in the same range as the total PAH (32.3 mg) and benzo(a)pyrene (0.62 mg) emissions reported by T. Ramdahl et al.[7] per kg of dry wood in a small residential stove when the stove was operated in a "starved air" condition with spruce wood as the fuel. Because the production of PAHs is a temperature-dependent reaction and the temperatures attained in a full-sized grenade may be different from those attained in the 10-g canisters, the composition and concentrations of these materials in the 500-g grenade may differ from those predicted.

MASS BALANCE AND EXPERIMENTAL ERROR

The analytical results show that there was a great variance in the amounts of chloride and ammonium found on the filters from different experimental runs using different C28 canisters. The variance was not great for individual runs, and the ratio of chloride to ammonium was generally consistent. The source of the variance between runs is unknown.

The total amount of chloride accounted for by the filter analysis and the HCl monitor is 50 percent of the mass that would be present in 10 g of the starting mixture. Some material obviously settled on the surfaces of the glove box during the run, so it can be assumed that the air concentration of aerosol particles declined during the 10-min run time. Since the standard deviation of the chloride concentrations from the different runs was greater than the mean, this variation from run to run also could account for the difference between the chloride concentration found and the theoretical amount. The total of the carbonaceous residue and the CO accounted for 77 percent of the carbon in 10 grams of the C28 mixture. Carbon dioxide was not measured and could easily account for the remainder.

SUMMARY AND CONCLUSIONS

In summary, the smoke produced by the 10-g C28 canister was found, contrary to the prediction of the NASA-Lewis model, to consist mainly of ammonium chloride particles. These are respirable particles with a mean diameter of 1 μm. Ammonium chloride is regarded as only a mild respiratory irritant with an 8-hr time-weighted-average threshold limit value of 10 mg/m^3 and a short-term exposure limit[8] of 20 mg/m^3. However, the smoke also contained sufficient hydrogen chloride to make it an irritant to the eyes, skin, and respiratory tract. Several PAHs were produced by the 10-g canisters, and the projected total of these compounds resulting from burning a 500-g charge in a full-size smoke grenade would be comparable to the amount produced by burning 1.0 kg of wood under worst case conditions in a residential wood stove. Such emissions are a recognized public health problem[9].

The organic components of the smoke produced by an ammonium perchlorate-based munition will vary in identity and quantity with different formulations, and the geometry of the munition will affect temperature and burn time, which will affect the mixture of combustion products. But HCl--with its property of being an irritant to the eyes and the respiratory system--is most likely to be a component of any smoke produced from an ammonium perchlorate mixture.

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