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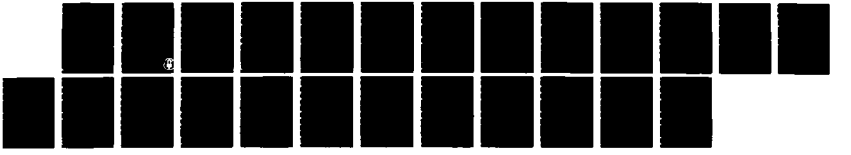

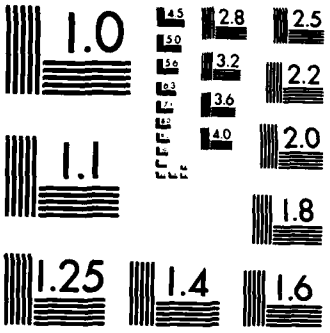

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TECHNICAL REPORT 8604

LIQUID CHROMATOGRAPHIC DETERMINATION OF EXPLOSIVES AND POLYNUCLEAR AROMATIC HYDROCARBONS (PAHs) IN DEACTIVATION FURNACE ASH

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AUGUST 1986

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Some liquid chromatographic (LC) methods have been developed for the determination of explosives and polynuclear aromatic hydrocarbons (PAH) in deactivation furnace ash. The explosives are extracted with acetonitrile and filtered through a 0.45 micron filter prior to analysis. The PAHs are extracted with methylene chloride in a soxhlet extraction apparatus and purified with a silica gel/solid phase extraction column.

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20. Abstract (continued)

Two different sets of chromatographic conditions were used to achieve separation of all the PAHs from background interferences. Separation of the explosives was achieved by using a C18 column and a water/methanol mobile phase employing a linear gradient elution. A programmable UV spectrophotometric detector was used to monitor the LC effluent for explosives and PAHs.

The recovery of explosives ranged from 67.87 percent for 1-acetylhexahydro-3,5-dinitro-1,3,5-triazine (TAX) to 97.86 percent for 2,4-dinitrotoluene (2,4-DNT). The recovery of PAHs ranged from 95.38 percent for indeno(1,2,3-cd)pyrene to 116.04 percent for benzo(k)fluoranthene. Detection limits for all explosive and PAH compounds are included in the report.

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INTRODUCTION

The U.S. Army uses a specially designed rotary kiln deactivation furnace to safely destroy a large variety of obsolete explosives. The residues, collected as baghouse and cyclone dusts, could possibly contain undestroyed explosive compounds as well as other hazardous materials.

Preliminary screening of untreated ash samples for organic compounds by gas chromatography/mass spectroscopy (GC/MS) indicates the presence of polynuclear aromatic hydrocarbon-type compounds (PAHs).[1,2] A number of these compounds are potential human carcinogens.[3] In addition, environmental concern for military explosives has increased recently due to mounting evidence that many aromatic nitro derivatives possess biological properties detrimental to animals and humans.[4-7]

Several procedures have been developed for the separation and measurement of PAHs in environmental samples.[8-11] However, these methods require extensive sample purification procedures to isolate the PAHs as a purified fraction before determination by gas chromatography or high performance liquid chromatography (HPLC).

Gas chromatography (GC), because of its speed and sensitivity, has received much attention as a method for the analysis of explosives. This technique is limited, however, because many explosive compounds are thermally unstable.[12-14]

HPLC appears to be a viable method for the analysis of explosives because it can be conducted at ambient temperatures without loss in efficiency of separation or speed of analysis. Also, improvements in reversed-phase column technologies have made it possible to achieve near baseline separation of many PAHs in HPLC.

The work herein describes rapid, reliable, and sensitive HPLC methods for determining representative PAHs and explosives in deactivation ash samples. In most cases, UV spectra were acquired with the use of a photodiode array spectrophotometer to aid in confirming the identity of the respective explosive and PAH-type compounds.[15] The PAHs include phenanthrene (Phen), fluoranthene (F), pyrene (Py), benz(a)anthracene (BaA), benzo(b)fluoranthene (BbF), benzo(k)fluoranthene (BkF), benzo(a)pyrene (BaP), benzo(ghi)perylene (BghiP), and Indeno(1,2,3-cd)pyrene (IP). The explosives include 1-acetyloctahyro-3,5,7-trinitro-1,3,5,7 tetrazocine (SEX), 1,3,5,7-tetranitro-1,3,5,7-tetraocine (HMX), 1-acetylhexahydro-3,5-dinitro-1,3,5-triazine (TAX), 1,3,5-trinitro-1,3,5-triazine (RDX), N,2,4,6-tetranitro-N-methylaniline (TETRYL), 2,4,6-trinitrotoluene (TNT), 2,6-dinitrotoluene (2,6-DNT), and 2,4-dinitrotoluene (2,4-DNT).

EQUIPMENT AND MATERIALS

EQUIPMENT*

A Waters liquid chromatographic system (Waters Chromatography Division, Milford, MA) was used throughout the study. The system consisted of the following components: two model 6000A solvent delivery systems, a model 721 programmable system controller, a model 730 data module, and a model 710B WISP autosampler. The UV detector was a Spectroflow 783 programmable absorbance detector (Kratos Analytical Instruments, Ramsey, NJ). The photodiode array spectrophotometer was an HP 1040A HPLC Detection System (Hewlett Packard Company, Avondale, PA).

A soxhlet extraction apparatus (Kontes Glass Co., Vineland, NJ) and heating mantle (Lab Line Instruments, Inc., Melrose Park, IL) were used in the extraction of the PAHs. A rotor evaporator was utilized to evaporate the soxhlet extract (Brinkman Instruments, Westbury, NY). Silica gel solid phase extraction columns (J.T. Baker Chemical Co., Phillipsburg, NJ) were employed in the sample cleanup of the PAHs. HPLC grade methanol, acetonitrile, methylene chloride and pentane were obtained (Burdick and Jackson Laboratories, Inc., Muskegon, MI) and used without further purification. Reagent grade water was obtained with a Milli-Q ^{R} water purification system (Millipore Corp., Bedford, MA).

MATERIALS

Phenanthrene, fluoranthene, pyrene, benz(a)anthracene, benzo(a)pyrene, indeno(1,2,3-cd)pyrene and benzo(ghi)perylene were obtained from Supelco Inc. (Bellefonte, PA). Benzo(b)fluoranthene and benzo(k)fluoranthene were obtained from CHEM Service, Inc., (West Chester, PA). All PAH standards were used without further purification.

2,6-Dinitrotoluene (2,6-DNT) and 2,4-dinitrotoluene (2,4-DNT) were obtained (Aldrich Chemical Co., Milwaukee, WI) and recrystallized twice with methanol prior to use.

SEX and TAX were obtained (SRI International, Menlo Park, CA) and their purity were found to be 98^{+} percent and 99.9 percent, respectively.[16,17] HMX and RDX were acquired (Holston Army Ammunition Plant, Kingsport, TN) and purified in our laboratory to greater than 98 percent (as determined by HPLC) by recrystallization from acetone.

2,4,6-Trinitrotoluene was obtained (Volunteer Army Ammunition Plant, Tyner, TN), recrystallized twice with methanol and assayed by GC to be 99^{+} percent pure. 2,6-Dinitrotoluene and 2,4-dinitrotoluene were acquired (Aldrich Chemical Co., Milwaukee, WI), recrystallized twice with methanol and assayed for purity by GC. The purity of 2,4-DNT and 2,4-DNT were both 99^{+} percent.

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* Use of trademarked name does not imply endorsement by the US Army, but is used only to assist in identification of a specific product.

The purity of tetryl (source unknown) was determined by injecting a 100 ppm tetryl solution (in acetonitrile) on the HPLC under the conditions stated in the methods sections. Only one peak was observed in the HPLC chromatogram.

METHODS

SAMPLE NAMES

(85-129) - Baghouse Ash (Tooele Army Depot, UT) Deactivation Furnace, 20 mm high-explosive incendiary (HEI), cartridges (M56A3) Project #42-21-0443-84 (25 Sept. 84); (85-130) - Baghouse Ash (Tooele Army Depot, UT), Deactivation Furnace, 7.62 mm, ball/tracer cartridges (4-M80, 1-M62) Project #42-21-0443-84 (25 Sept. 84); (85-133) - Baghouse Ash (Tooele Army Depot, UT), 0.30 CAL armor piercing incendiary (API), cartridges (M14) Project #42-21-0443-84 (27 Sept. 84).

SAMPLE PREPARATION AND HANDLING

Explosives

Three 5-gram portions of each ash sample were weighed into 50-mL Erlenmeyer flasks on an analytical balance. Next, 10 mL of acetonitrile was added to each sample portion. The containers were then shaken for 30 minutes on a wrist arm shaker, clarified by centrifugation (2,000 RPM for 10 min) and filtered (0.45 micron filter) prior to analysis by HPLC. Each sample extract that contained an explosive with a concentration in excess of 10 mg/L was diluted with acetonitrile to obtain a sample concentration within the upper and lower limits of the appropriate standard curve. Because some of the explosives are light-sensitive, amber-colored glassware was used throughout the procedure.

PAH

Three 10-gram portions of each ash sample were extracted with 100 mL of methylene chloride for 4 hours in a soxhlet extraction apparatus. Each extract was collected in a 125-mL round bottom flask and evaporated to dryness by rotary evaporation. The residue remaining after evaporation was reconstituted with 4 mL of pentane and ultrasonicated for 5 minutes in a sonic bath.

Three 4-mL aliquots of each standard solution were evaporated to dryness, reconstituted with 4 mL of pentane and ultrasonicated for 5 minutes in a sonic bath.

Each 4-mL standard and sample pentane extract was eluted through a silica gel solid phase extraction column (6 mL, high capacity) and collected in a glass scintillation vial. Following this, 2 mL of pentane was passed through each extraction column to elute any PAHs trapped on the column. This brought the extraction volume to a total of 6 mL. The pentane extracts (containing the PAHs) were evaporated to dryness under streams of dry nitrogen, and each reconstituted with 4 mL of acetonitrile and ultrasonicated for 5 minutes prior to HPLC analysis.

Each sample extract that contained a PAH with a concentration in excess of 10 mg/L was diluted with acetonitrile to obtain a sample concentration within the upper and lower limits of the appropriate standard curve.

PREPARATION OF STOCK AND STANDARDS

A stock solution containing the eight explosive compounds of interest was prepared by dissolving 20 mg of each compound in 100 mL of acetonitrile to yield a concentration of approximately 200 mg/L. Stock solution aliquots of 1, 2, 5, and 10 mL were diluted to 100 mL with acetonitrile to prepare standards with concentrations of 2, 4, 10, and 20 mg/L, respectively.

A stock solution containing the four PAH compounds of interest was prepared by dissolving 5 mg of each compound in 100 mL of methylene chloride to yield a concentration of 50 mg/L. Stock solution aliquots of 2, 5, 10, and 20 mL were diluted to 100 mL with methylene chloride to yield PAH standards with concentrations of 1, 2.5, 5, and 10 mg/L.

HPLC CONDITIONS

Explosives - Separation of the explosives was achieved with a Zorbax ODS column (25 cm x 4.6 mm I.D., DuPont Instruments Co., Wilmington, DE). A linear gradient was employed in which the eluent was changed from 100 percent pump A (20 percent methanol/water) to 50 percent pump B (80 percent methanol/water) in 30 min at a flow rate of 1.2 mL/min.

The column effluent was monitored at 254 nm, 0.04 absorbance units full scale (AUFS). The injection volume was 25 microliters.

PAHs - Unfortunately, not all the PAHs could be chromatographed under one set of operating parameters. Therefore each standard and sample extract was chromatographed under two different sets of conditions, as listed below:

(System 1 conditions) Column - Zorbax ODS; Mobile Phase - pump A (water), pump B (50 percent methanol/acetonitrile); Linear Gradient - 80-100 percent B in 20 minutes; Flow Rate - 1 mL/minute; UV - 254 nm (0.1 AUFS) for 12.2 minutes, then 289 nm (0.04 AUFS) for the remainder of the chromatographic run; Injection Volume - 20 microliters.

(System 2 conditions) Column - Supelcosil-LC-PAH (Supelco Inc., Bellefonte, PA); Mobile Phase - pump A (35 percent acetonitrile/water), pump B (90 percent acetonitrile/water); Linear Gradient - hold at 0 percent B for 2 minutes, then 0-100 percent B in 25 minutes; Flow Rate - 1.5 mL/minute; UV - 254 nm (0.1 AUFS) for 22 minutes, then 289 nm (0.04 AUFS) for the remainder of the chromatographic run; Injection Volume - 20 microliters.

The initial UV absorbance of the eluent from each chromatographic system was monitored at 254 nm because of phenanthrene's strong absorbance at this wavelength. The remainder of the chromatographic run was monitored at 289 nm because the PAHs provide a strong signal relative to background interferences.

CALCULATIONS

Peak areas for all working standards are plotted against their concentrations to obtain a standard curve. The peak area of the sample unknown is

compared to the appropriate standard curve to obtain a concentration in mg/L. Next, the sample's concentration (mg/L) obtained from the standard curve is converted to µg/10 mL (explosive) or µg/4 mL (PAH) and divided by the sample's weight (in grams) to obtain a concentration in µg (explosive or PAH)/g (ash).

The detection limit for each explosive and PAH compound was determined on the basis of the chromatographic conditions used. The detection limit was defined here as the lowest concentration that could be reproduced three times with relative standard deviation of not more than 10 percent. Tables 1 and 2 show these detection limits.

The accuracy of the explosive and PAH extraction methods was tested by conducting recovery studies on ash samples spiked with the appropriate explosive or PAH standards (Tables 3 & 4).

TABLE 1. DETECTION LIMITS (µg/g) FOR EXPLOSIVES IN ASH MATRIX

Compound	Detection Limit
SEX	1.00
HMX	1.00
TAX	1.00
RDX	1.00
Tetryl	1.00
TNT	1.00
2,6-DNT	1.00
2,4-DNT	0.50

TABLE 2. DETECTION LIMITS (µg/g) FOR PAHs IN ASH MATRIX

Compound	Detection Limit
Phenanthrene	0.25
Fluoranthene	0.20
Pyrene	0.50
Benz(a)anthracene	0.10
Benzo(b)fluoranthene	0.30
Benzo(k)fluoranthene	0.20
Benzo(a)pyrene	0.10
Benzo(ghi)perylene	0.30
Indeno(1,2,3-cd)pyrene	0.30

TABLE 3. ACCURACY OF EXPLOSIVE DETERMINATIONS, ASH MATRIX

Compound	Amount Added (µg/g)	Amount[a] Recovered (µg/g)	S.D.	RSD (%)	Recovery (%)
SEX	20.81	17.97	1.89	10.52	86.35
HMX	24.98	22.94	1.66	7.24	91.83
TAX	20.79	14.11	1.68	11.91	67.87
RDX	23.21	20.97	1.11	5.29	90.35
TETRYL	20.99	19.99	3.52	17.61	95.24
TNT	20.86	19.73	0.32	1.62	94.58
2,6-DNT	21.78	19.84	1.48	7.46	91.09
2,4-DNT	21.98	21.51	1.60	7.44	97.86

a. Mean of three determinations.

TABLE 4. ACCURACY OF PAH DETERMINATIONS, ASH MATRIX

Compound	Amount Added (µg/g)	Amount[a] Recovered (µg/g)	S.D.	RSD (%)	Recovery (%)
Phenanthrene	1.20	1.22	0.05	4.10	101.67
Fluoranthene	1.34	1.36	0.05	3.68	101.49
Pyrene	1.43	1.47	0.08	5.44	102.80
Benz(a)anthracene	1.00	1.07	0.05	4.67	107.00
Benzo(b)fluoranthene	1.19	1.26	0.07	5.56	105.88
Benzo(k)fluoranthene	1.06	1.23	0.07	5.69	116.04
Benzo(a)pyrene	1.04	1.10	0.10	9.09	105.77
Benzo(ghi)perylene	1.23	1.23	0.13	10.57	100.00
Indeno(1,2,3-cd)pyrene	1.30	1.24	0.08	6.45	95.38

a. Mean of three determinations.

RESULTS AND DISCUSSION

Figure 1a shows a typical chromatogram of the eight explosive standards investigated. All eight compounds were completely resolved from one another in addition to showing good peak symmetry.

Figure 1b shows a chromatogram of a deactivation ash sample. Preliminary investigations in our laboratory had shown that all eight explosive standards could be separated using isocratic (water/methanol) mobile phase conditions. However, isocratic mobile phase conditions were insufficient in providing optimal resolution from interferences in the sample matrix. A water/methanol gradient was employed to achieve adequate resolution from background interferences.

Also shown in Figure 1b, peaks 5 and 6 appear to be tetryl and 2,4,6-TNT because their retention times are within ± 0.5 minutes of their respective standards. Although their retention times are not identical to their corresponding standards, complex sample matrices can cause shifts of the magnitude seen here in retention times. Spectral analysis (Figures 2 and 3) shows a complete mismatch in spectra between standard and sample peaks. This suggests that peaks 5 and 6 are not tetryl and 2,4,6-TNT, respectively.

Figures 4a and 4b show a chromatogram of PAH standards and a deactivation ash sample, respectively, under system 1 conditions. All the PAHs were adequately separated from background interferences in the sample and standard extracts. However, benzo(b)fluoranthene and benzo(k)fluoranthene are partially resolved from one another, making their quantitative analysis by electronic integration difficult.

Complete resolution of all PAH standards is accomplished with the HPLC conditions listed in system 2 (Fig. 5a). However, pyrene cannot be separated from interfering substances from the deactivation ash (Fig. 5b). Pyrene is resolved from sample interferences under the conditions of chromatographic system 1 (Fig. 4b).

The explosives and PAHs separated by HPLC were tentatively identified by comparing the retention times of peaks in the sample chromatograms with those in the standard chromatograms. Additional confirmation of peak identity was obtained by comparing the UV spectra (230-330 nm for explosives, 254-318 nm for PAHs) of peaks with similar retention times in standard and sample chromatographic runs.

The amount recovered, standard deviation, relative standard deviation, and percent recovery for ash samples spiked with the appropriate explosive or PAH standards are given in Tables 3 and 4. The recovery of explosives ranged from 67.87 percent for TAX to 97.86 percent for 2,4-DNT. The recovery of PAHs ranged from 95.38 percent for indeno(1,2,3-cd)pyrene to 116.04 percent for benzo(k)fluoranthene. Calibration curves constructed for the explosive and PAH type compounds showed a linear relationship over the concentration range of 2 mg/L to 20 mg/L and 1 mg/L to 10 mg/L, respectively.

Tables 5 and 6 summarize the data obtained from the analysis of deactivation ash samples for their explosive and PAH contents, respectively. The

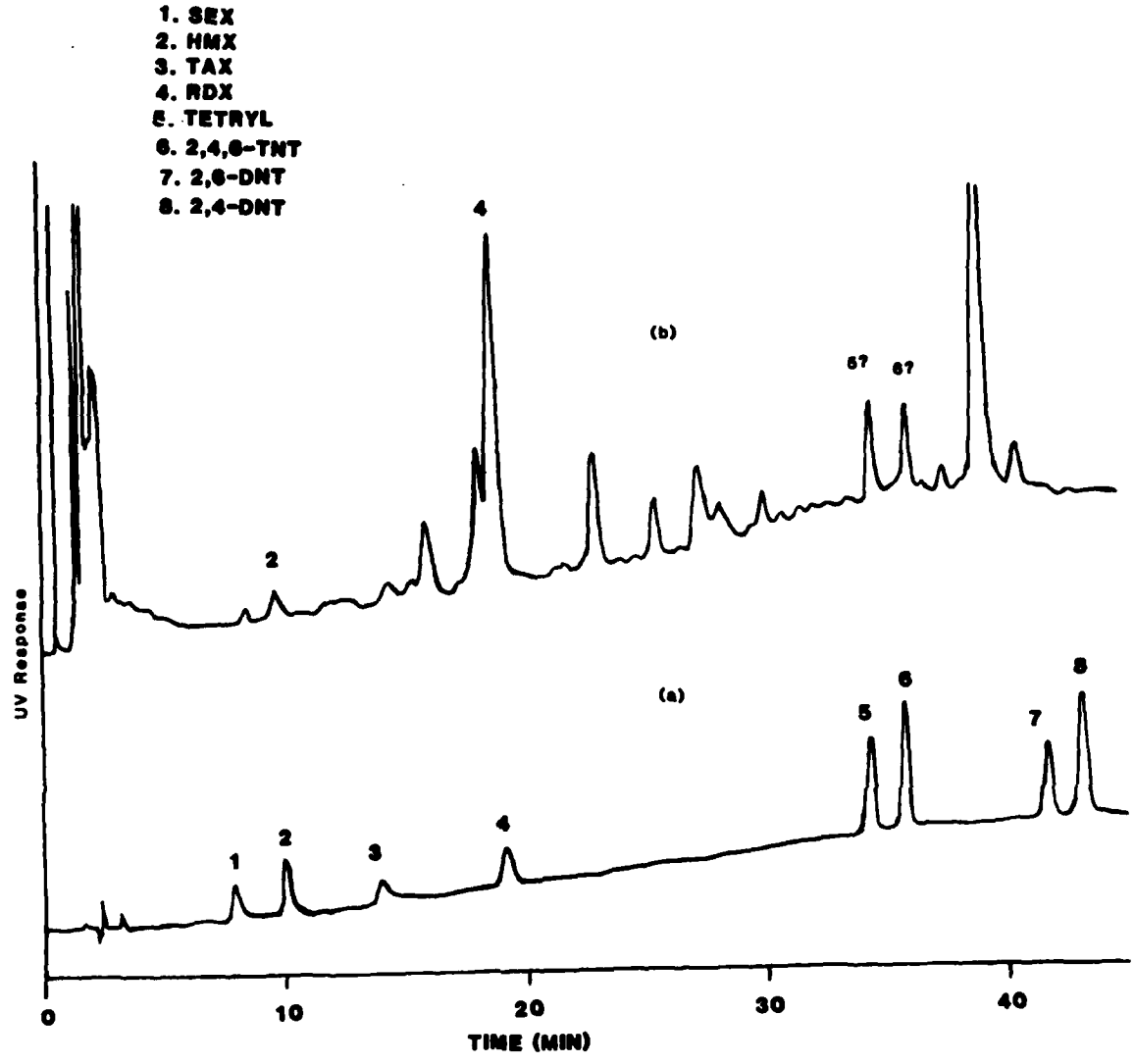

Figure 1. HPLC Chromatogram of (a) Explosive Standards and (b) Deactivation Ash Sample (85-129).

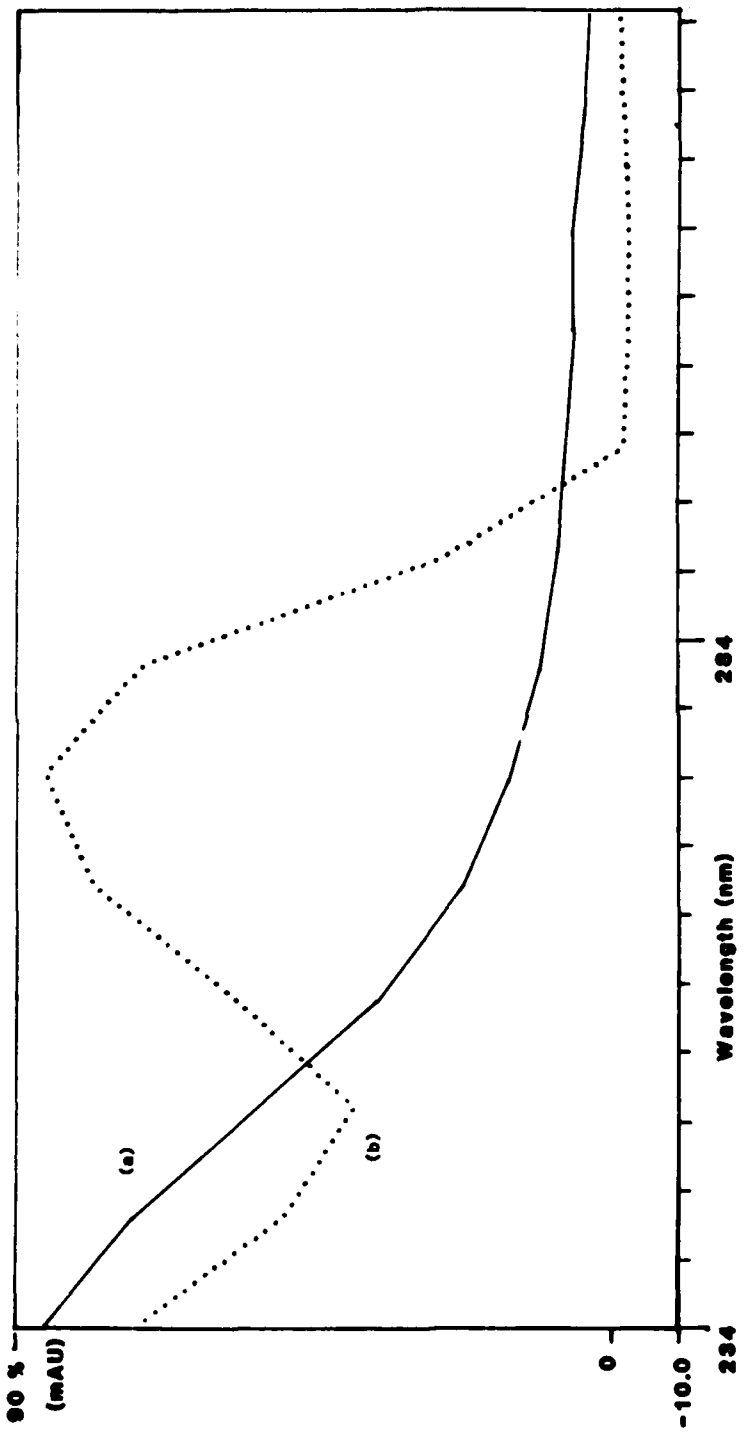

Figure 2. Peak Apex Spectra of (a) Tetryl Standard (RT = 34.12 min), and (b) Sample Peak (RT = 34.67 min).

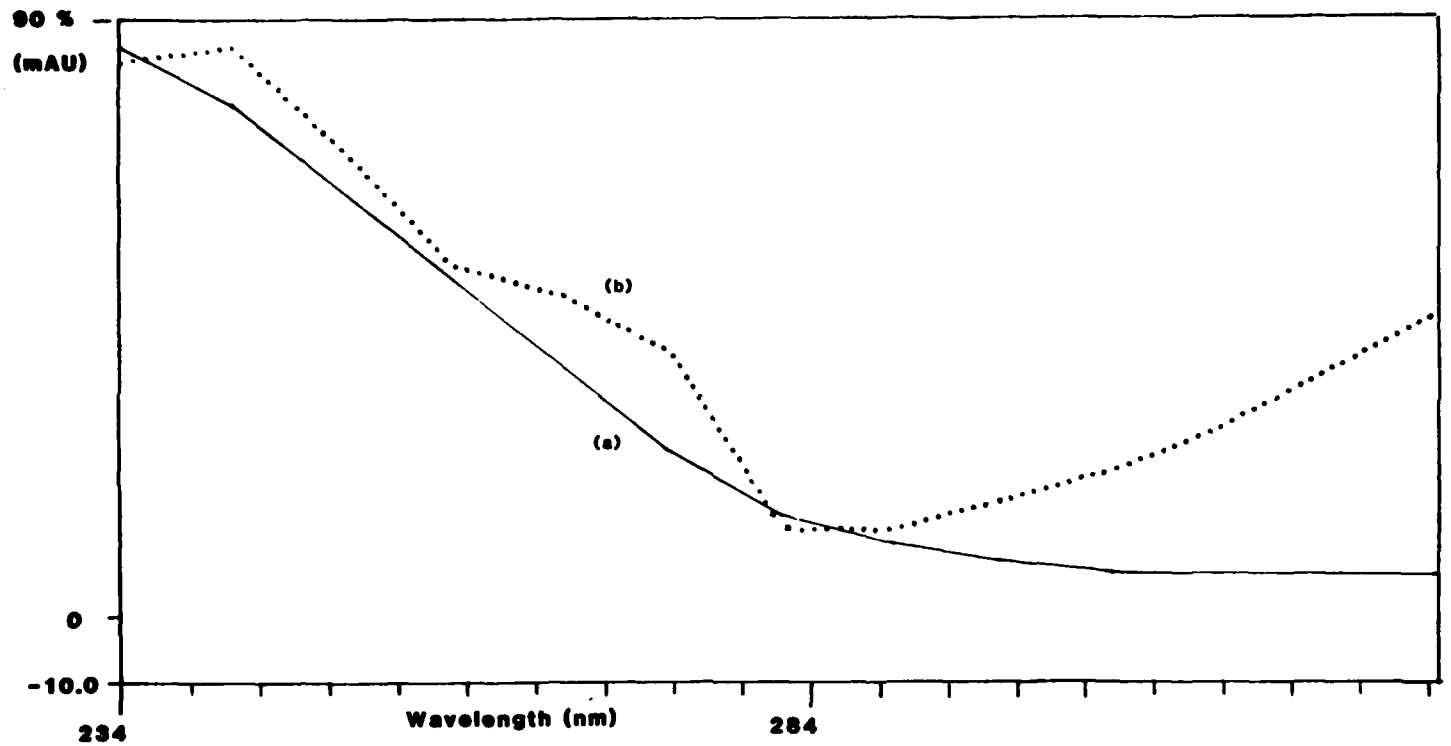

Figure 3. Peak Apex Spectra of (a) TNT Standard (RT = 36.00 min), and (b) Sample Peak (RT = 36.25 min).

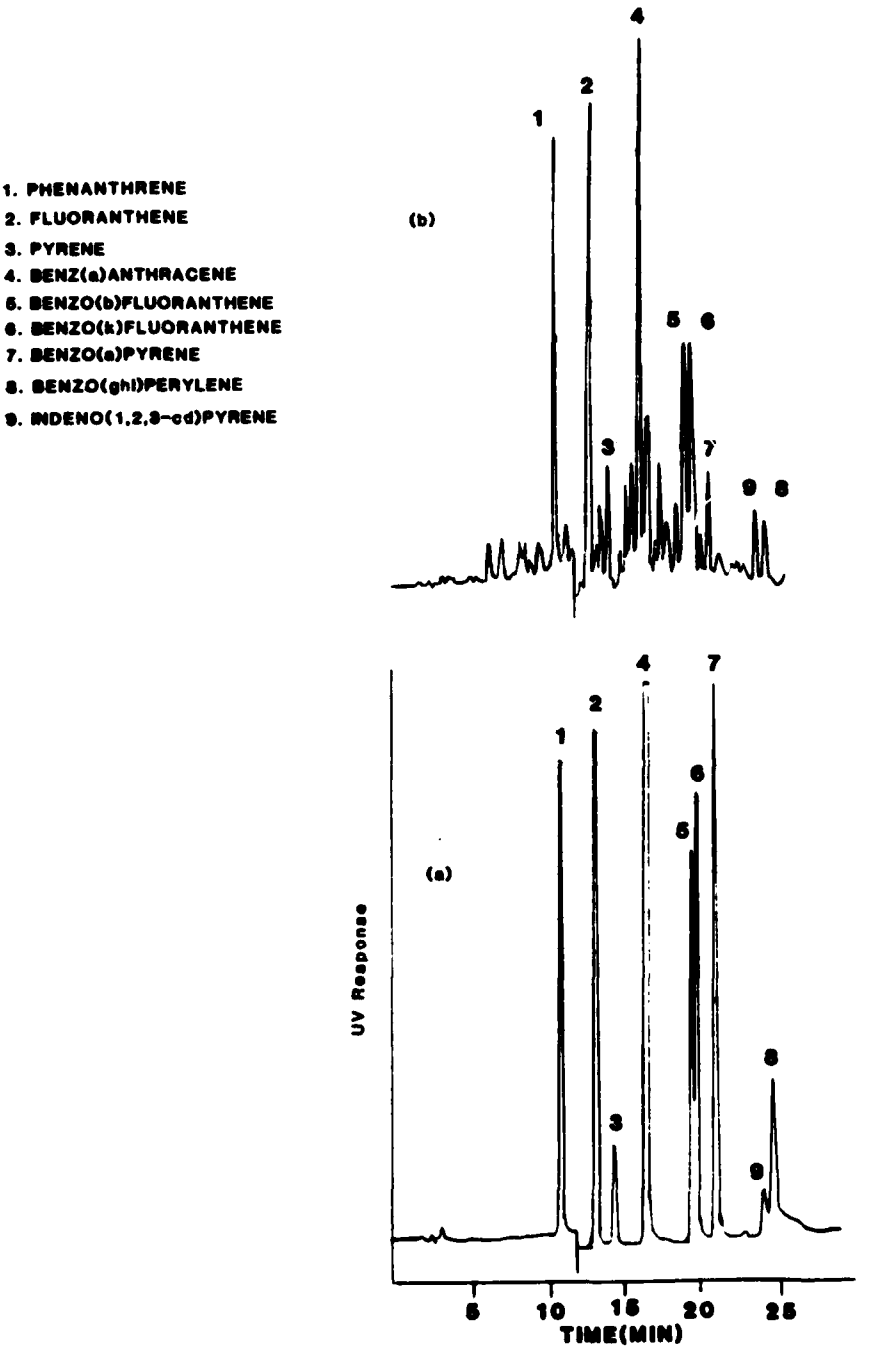

Figure 4. HPLC Chromatogram of (a) PAH Standards, and (b) Deactivation Ash Sample (System 1 Operating Conditions).

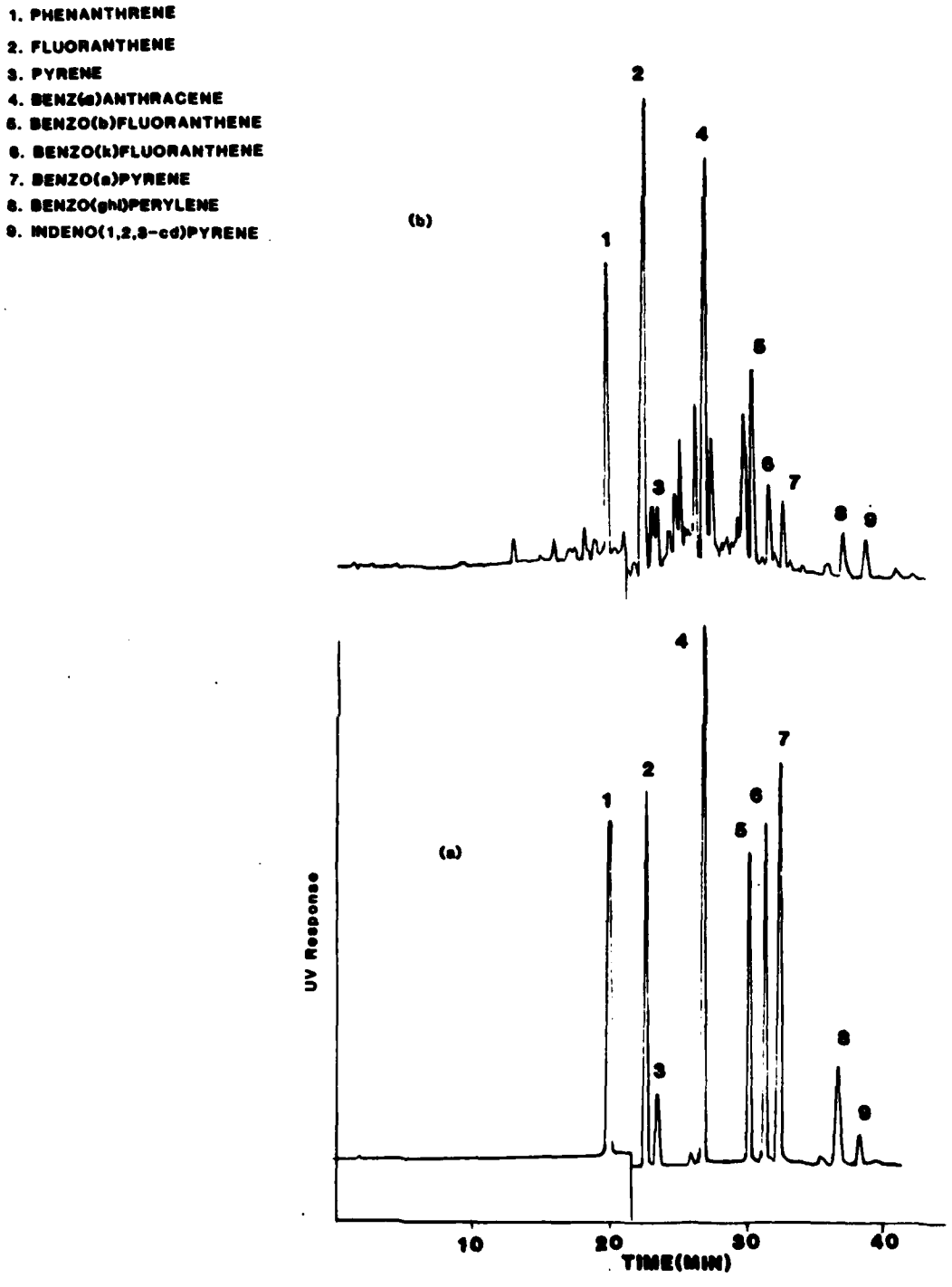

Figure 5. HPLC Chromatogram of (a) PAH Standards, and (b) Deactivation Ash Sample (System 2 Operating Conditions).

TABLE 5. EXPLOSIVES FOUND IN ASH SAMPLES (μg/g)[a]

Compound	Sample 85-129	85-130	85-133
SEX	<1.00	<1.00	<1.00
HMX	5.78 (0.20)	3.44 (0.19)	1.39 (0.06)
TAX	<1.00	<1.00	<1.00
RDX	50.93 (2.18)	28.25 (0.53)	12.08 (0.05)
TETRYL	<1.00	<1.00	<1.00
TNT	<1.00	<1.00	<1.00
2,6-DNT	<1.00	<1.00	<1.00
2,4-DNT	<0.05	1.08 (0.09)	11.62 (0.09)

a. Mean of three determinations (standard deviations).

TABLE 6. PAHs FOUND IN ASH SAMPLES (μg/g)[a]

Compound	Sample 85-129	85-130	85-133
Phenanthrene	4.10 (0.36)	1.63 (0.20)	0.93 (0.09)
Fluoranthene	5.45 (0.35)	2.81 (0.19)	1.06 (0.19)
Pyrene	4.11 (0.26)	2.38 (0.17)	0.86 (0.14)
Benz(a)anthracene	1.06 (0.08)	0.69 (0.09)	0.26 (0.05)
Benzo(b)fluoranthene	1.35 (0.17)	1.13 (0.07)	0.63 (0.15)
Benzo(k)fluoranthene	0.50 (0.06)	0.42 (0.04)	0.20 (0.05)
Benzo(a)pyrene	0.71 (0.09)	0.37 (0.05)	0.11 (0.02)
Benzo(ghi)perylene	0.76 (0.13)	0.55 (0.06)	0.32 (0.06)
Indeno(1,2,3-cd)pyrene	0.76 (0.12)	0.64 (0.07)	0.33 (0.08)

a. Mean of three determination (standard deviations).

values shown are averages of triplicate analysis. It is apparent from these data that trace levels of various explosives and PAHs were present in all the deactivation ash samples analyzed.

CONCLUSION

Relatively rapid and reliable HPLC methods have been developed for the determination of explosives and PAHs in deactivation furnace ash.

In order to obtain desirable accuracy in PAH determinations, it was necessary to chromatograph each standard and sample extract under two different sets of chromatographic conditions. The HPLC conditions listed in system 1 could adequately separate all the PAHs from background interferences in the sample and standard extracts. However, accurate quantitative measurements were difficult to obtain for benzo(b)fluoranthene and benzo(k)fluoranthene because of their incomplete separation from one another. Although the chromatographic conditions listed in system 2 were unable to separate pyrene from background interferences, it allowed for the complete separation of benzo(b)fluoranthene from benzo(k)fluoranthene. Therefore, system 1 was employed to separate and analyze pyrene and system 2 was used to separate and analyze benzo(b)fluoranthene and benzo(k)fluoranthene.

The detection limit of the explosives (with the exception of 2,4-DNT) was 1.00 µg/g. The detection limit for 2,4-DNT was 0.50 µg/g. The detection limit of the PAH compounds ranged from 0.10 µg/g for benzo(a)pyrene and benz(a)anthracene to 0.50 µg/g for pyrene.

The use of a photodiode array spectrophotometer aided in confirming the identity of the explosives and PAHs found.

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