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CHEMICAL CHARACTERIZATION OF DIMETHYLSULFOXIDE (DMSO) MUNITIONS RECRYSTALLIZATION PROCESS SAMPLES

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We thank MAJ David L. Parmer for providing information on the origin of the samples.

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INTRODUCTION

A pilot plant for the operation of a new munitions recrystallization process was set up at Holston Army Ammunition Plant (HSAAP) in 1979 and was operated for approximately 5 months. The new process involves recovery and recycling of dimethylsulfoxide (DMSO), in contrast to an older batch process utilizing acetone and cyclohexanone as recrystallization solvents. Though more efficient and cost effective, the DMSO process provoked concern over possible health hazards to workers due to the known rapid absorption of the solvent (along with any nitramine munition compounds, breakdown products or other trace organics in the solvent) into body tissues. Chemical characterization of the new process samples at HSAAP was limited to analyses of the nitramines hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX), octahydro-1,3,5,7tetranitro-1,3,5,7-tetrazocine (HMX), 1-acety1hexahydro-3,5-dinitro-1,3,5triazine (TAX), and 1-acetyloctahydro-3,5,7-trinitro-1,3,5,7-tetrazocine (SEX) by high pressure liquid chromatography (HPLC).¹ The samples were tested for acute toxicity and mutagenicity by extramural laboratories and, when two of the process samples showed mutagenicity, US Army Medical Bioengineering Research & Development Laboratory (USAMBRDL) was tasked with chemical characterization and identification of any trace organics present in addition to the nitramines. Portions of the two samples, designated as evaporator sludge and recycle solvent, and a sample of unused DMSO were first received in June 1983, and the results of the analyses were described briefly in a Memorandum Report.² Additional portions of evaporator sludge and recycle solvent were received and analyzed in January 1984. An evaporator sludge sample which had not yet been tested for mutagenicity and a sample designated as recovered DMSO, as well as two river water samples, were received and analyzed in April 1984. The results are compared and contrasted with those of four samples from the older acetone/cyclohexanone process which were analyzed in June 1984.

EQUIPMENT AND METHODS

HIGH PRESSURE LIQUID CHROMATOGRAPHIC (HPLC) ANALYSES

A Waters liquid chromatographic system (Waters Associates, Milford, MA) consisted of the following components: two Model 6000A solvent delivery systems, a Model 721 programmable systems controller, a Model 730 data module, a Lambda-max Model 480 LC spectrophotometer, and a Model 710B Waters intelligent sample processor (WISP). A Zorbax C_8 reverse phase stainless steel column (25 cm x 4.6 mm ID, particle size 6 µm, DuPont Instruments, Wilmington, DE) was used. The nitramines were eluted using a linear gradient program in which pump A contained 1:4 methanol/water and pump B contained 4:1 methanol/ water, and the methanol/water composition was changed from 95:5 A/B to 50:50 A/B in 25 min at a flow rate of 1.2 mL/min. The effluent was monitored at 254 nm, 0.05 absorbance units full scale (AUFS). Standard solutions were prepared as described previously.³

GAS CHROMATOGRAPHIC/MASS SPECTROMETRIC (GS/MS) AMALYSES

GC/MS analyses were performed with a Hewlett Packard 5985B system equipped with a 25 m x 0.2 mm ID fused silica capillary column (cross-linked OV-1, 0.11 µm thick) interfaced directly to the source (source temperature 200° C). Injection temperature was 150° or 250° C, and the GC oven was programmed from 60° to 250° at 20° C per minute with an initial hold of 1 minute. The data system was upgraded to RTE-VI and included a data base of 70,000 spectra.

PREPARATION OF SAMPLES

For HPLC analyses, 1 mL of liquid was made up to 100 mL in distilled deionized water; further dilutions were made when necessary. A 4 mL portion of each solution was passed through a 0.45 μ m Millex-SR filter into a WISP sample vial. Weighed amounts of solid samples were dissolved in acetonitrile (5 mL) and made up to 100 mL in distilled deionized water and treated as above.

For GC/MS analyses, liquid samples containing DMSO and munitions compounds (5 mL) were diluted with water (10-50 mL), filtered when necessary to remove any precipitated munitions compounds, and extracted with methylene chloride (CH_2Ci_2 , 5-10 mL). The CH_2Cl_2 extracts were washed with three 25 to 50 mL portions of water, dried, and evaporated, and the residues were dissolved in acetone for injection. The DMSO pilot plant water samples and the RDX and HMX system filtrates from the earlier acetone/cyclohexanone recrystallization process (100-200 mL) were extracted with CH_2Cl_2 and the extracts were dried and treated as above. The used acetone sample was partially evaporated and the resulting aqueous suspension was extracted with CH_2Cl_2 and treated as above. A small portion (0.2 mL) of the used cyclohexanone was taken to dryness under N₂ and redissolved in acetone for analysis.

RESULTS AND DISCUSSION

Chemical characterization of evaporator sludge and recycle solvent samples included qualitative analysis for trace organics by GC/MS and quantitative analysis for nitramine munitions by HPLC.³ Results of sample analyses in June 1983 and January 1984 are summarized in Tables 1 and 2.

Compound	Retention Time (min)	Evaporator Sludge Jan 1984 Amt (ppm) ^a	Recycle Solvent Jun 1983 Amt (ppm)	Recycle Solvent Jan 1984 Amt (ppm)
SEX	7.3	64	150	412
нмх	9.1	1,750	3,500	41,900
TAX	11.2	2,400	580	648
RDX	15.7	403	5,500	39,900

TABLE 1. HPLC AMALYSES OF MUNITIONS FROM DMSO RECRYSTALLIZATION PROCESS SAMPLES

a. Relative amounts in June 1983 sample were TAX > RDX > HMX >> SEX.

The observed discrepancies in nitramine content between the two sets of analyses (Table 1) may be attributed to inconsistent sampling or sampling errors, due to inhomogeniety of the mixtures and/or temperature differences at the time of sampling.

No impurities were found in the unused DMSO. Of the trace organics found in the recrystallization process samples (Table 2), all except DDBH and <u>trans-</u> 4-chlorocyclohexanol were commercially available compounds. Diacetone alcohol is formed by aldol condensation of acetone, and dimethylsulfone is an obvious oxidation product of DMSO. The genesis of DDBH, found in three of four samples, is obscure. A literature survey revealed few citations: the compound was first reported as the end product of a synthesis requiring six steps from commercially available chemicals,⁴ but was recently found as one of many trace organics in highly polluted rivers in Japan⁵ and in volatile constituents of certain fungi.⁶

This finding of a variety of trace organics unrelated to the munitions manufacture process or to possible decomposition or transformation of the nitramines prompted an investigation of the organic constituents of samples of water from the Holston River, which had been used in the pilot plant process. In addition, another sample of evaporator sludge which had not yet been tested for mutagenicity and a sample designated as "recovered DNSO" were analyzed for trace organics and munitions compounds; the results are summarized in Tables 3 and 4.

letention Time (min)	Compound	Evaporator Sludge	Recycle Solvent
	June 1983	<u>****</u> *	
2.4	Diacetone alcohol	+	_ ^b
4.1	Dimethylsulfone	-	۲
4.9	Benzothiazole	+	+
6.7	1,5-Di-t-buty1-3,3-dimethy1- bicyclo(3.1.0)hexan-2-one (DDBH)	-	۲
	January 1984		
2.5	Diacetone alcohol	+	+
2.6	1,4-Dithiane	+	-
3.4	trans-4-Chlorocyclohexanol	۲	+
4.9	Benzothiazole	+	+
5.8	l,5-Di-t-butyl-3,3-dimethyl- bicyclo(3.1.0)hexan-2-one (DDBH)	ŀ	۲
7.0	2,6-Di- <u>t</u> -buty1-4-methylphenol (BHT)	+	+

TABLE 2. GC/MS IDENTIFICATION OF TRACE ORGANICS PRESENT IN DMSO RECRYSTALLIZATION PROCESS SAMPLES^a

a. +, present; -, not detected.

b. Not detected because of DMSO interference, but believed to be present.

TABLE 3. HPLC ANALYSES OF MUNITIONS IN
NEW DMSO RECRYSTALLIZATION
PROCESS SAMPLES, APRIL 1984

.

	Retention	Europeanten Cludus ³
Compound	Time (min)	Evaporator SLudge ^a Amt (ppm)
SEX	7.6	66
HITX	10.2	1,940
TAX	12.5	2,540
RDX	18.5	410

a. Munitions in "recovered DMSO" were below detection limit (<0.3 ppm).

TABLE 4.	TRACE ORGAN	IC CONTEN	IT OF RIVEI	R WATER	R AND DMS	50
RECRY	STALLIZATION	PROCESS	SAMPLES,	APRIL	1984 ^a	

Sample	Retention Time (min)	Compound
River water, single	2.5	Diacetone alcohol
, 0	6.4	p-Hydroxyacetanilide
	7.6	<u>p-</u> Hydroxyacetanilide Unidentified, MW 213 ^b
River water,	2.5	Diacetone alcohol
composite ^Ć	6.4	p-HydroxyacetanįLide
•	7.3	Diphenyloxazole ^d
	7.6	Unidentified, NW 213 ^b
Evaporator sludge	10.9	Acetyl tri-n-butyl citrate
		(Citroflex A, citrate plasticizer)
Recovered DNSO	2.5	Diacetone alcohol
	7.0	2,6-di-t-Butyl-4-methylphenol (BHT)
	7.7	3,4'-Dimethyldiphenylmethane
	8.9	N-Butylbenzenesulfonamide
	12.1	Octadecanamide

a. In addition to phthalates.

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b. Mass spectrum below m/z 135 identical to that of <u>p</u>-methoxystyrene.

c. Composite consisted of 4 aiiquots taken at 6 hr intervals.

d. Mass spectrum indicated 4,5-isomer rather than the commercially available 2,5-isomer.

Comparison of the results for the April 1984 evaporator sludge with those of the January 1984 sample (Tables 1 and 2) showed good consistency for the nitramines, but the trace organic content of the two samples was quite different. Furthermore, except for the consistent presence of diacetone alcohol (found in six of eight samples), the trace organic content in the river water samples was different from the process sludge and solvent samples. Significantly, benzothiazole, a suspect mutagen present in the four samples of sludge and solvent analyzed in June 1983 and January 1984, was not found in any of the samples analyzed later. The difference in organic content between "recycle solvent" DNSO and "recovered" DNSO may be attributable to the fact that the samples were taken at different stages of the process. The former was a composite taken from a storage tank and decanted from precipitated munitions; the latter was taken after separation of process DNSO from munitions and water.

Trace organic content of the four samples from the acetone/cyclohexanone process was low. Diacetone alcohol was found in the RDX filtrate and the used acetone samples, but not in the HMX filtrate. It was not possible to detect trace organics in the presence of the enormous quantity of cyclohexanone, and its relative insolubility in water precluded concentration of possible trace organics by liquid/liquid extraction procedures. No trace organics were Identified in the residue after evaporation of the cyclohexanone to dryness, but volatile compounds such as diacetone alcohol would also have been removed by the evaporation.

CONCLUSION

It must be emphasized that none of the trace organics identified can be attributed either to the munitions manufacture process or to breakdown or transformation of the munitions compounds. Only dimethylsulfone clearly originated from reaction of the solvent, DNSO. Diacetone alcohol could have been a solvent-derived product in the acetone/cyclohexanone recrystallization process, but not in the DMSO process. The wide variety and lack of consistency (except for the presence of diacetone alcohol) of trace organics found in the latter process samples suggest river water used in the process as the source. While trace organic contents of the single and 24 hr composite river water samples taken in 1984 were similar, and different from that of any of the 1979 pilot plant samples (again, except for the presence of diacetone alcohol), variation of the nature of organic pollutants in the river over extended time periods is not unexpected. The observed variation in organic content of the evaporator sludge samples (Tables 2 and 4), however, is not readily explained.

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