EVALUATION OF ISOTOPE MIGRATION - LAND BURIAL Water Chemistry at Commercially Operated Low-Level Radioactive Waste Disposal Sites

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JULY - SEPTEMBER 1976

P. COLOMBO, A.J. WEISS, AND A.J. FRANCIS

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SUMMARY

This is the second quarterly progress report of water chemistry at commercially operated low-level radioactive waste disposal sites. The program is a joint investigation undertaken by the United States Nuclear Regulatory Commission and the United States Geological Survey as part of a comprehensive plan to study the hydrogeological and geochemical behavior of existing commercially operated low-level radioactive waste disposal sites.

The analytical methods that were used to concentrate, separate and identify organic compounds found in filtered trench water samples taken from the Maxey Flats disposal site April 1976 are described. A variety of organic compounds were isolated and identified in all of the trenches sampled.

I. BACKGROUND

The disposal of low-level radioactive wastes at commercially operated disposal sites began in 1962 at Beatty, Nevada. Since that time, the industry has expanded to include three private companies operating six sites. The sites are located in Maxey Flats (Morehead), Kentucky; Beatty, Nevada; Sheffield, Illinois; Barnwell, South Carolina; West Valley, New York; and Richland, Washington. The three companies operating these sites are the Nuclear Engineering Company (Illinois, Kentucky, Nevada, and Washington), Chem Nuclear Systems (South Carolina), and Nuclear Fuel Services (New York). Although these facilities are managed by private industry, they are located on Federal or State owned land. In general, the disposal sites are regulated by the state in which they are located, according to provisions of the agreements between the individual states and the United States Nuclear Regulatory Commission (NRC).⁽¹⁾ The one exception is the site located in Sheffield, Illinois, which is regulated by both the State of Illinois and the NRC.

The disposal sites are located in remote areas of sparse population selected for their hydrogeological environment and were considered suitable at the time of selection for the purpose of shallow land burial of low-level radioactive wastes. In addition, these locations are in proximity to centers of nuclear industry to minimize long distance transportation of wastes.

Projections have been made of the available capacity of the commercial sites and the results indicate that the current burial capacity of all six existing sites may be exceeded by 1998 when existing sites may have to be closed or enlarged or new sites will have to be established.⁽²⁾

Recently, questions have been raised regarding the adequacy of these disposal sites.^(3,4) With the increasing growth of the nuclear energy industry, important considerations must be given to the selection of any new sites for disposal of radioactive wastes.

The United States Geological Survey (USGS) funded by the United States Congress and with concurrence of the individual state is conducting hydrogeological and geochemical investigations at all commercially operated low-level radioactive waste disposal sites except Richland, Washington.

The United States Nuclear Regulatory Commission (NRC) has the responsibility for establishing criteria for selection and operation of low-level radioactive waste disposal sites.

In support of USGS investigations, NRC funds Brookhaven National Laboratory (BNL) to perform analyses for USGS of natural waters and of waste leachate that have accumulated in waste trenches at the disposal sites.

II. SCOPE

The low-level radioactive waste disposal sites in use today throughout the United States are located in different hydrogeological environments. Until the various physical and geochemical processes governing transport are understood, the continued monitoring of these sites to detect movement, if any, of radionuclides from the disposal trenches is needed.

One of the goals of the USGS program is to collect hydrogeological and geochemical data at each site and to correlate these data with theoretical and laboratory results to develop a transport model for each disposal site. The ultimate goal is to establish criteria for selection, operation, and monitoring of low-level radioactive waste disposal sites.

In support of the USGS program, samples of materials from the disposal sites will be taken at selected intervals and the analysis of the chemical and radioactive species will be performed at Brookhaven National Laboratory. Methods for collecting and analyzing samples from each of the burial sites will be developed. The study will include the measurement of inorganic, organic, and radioactive constituents in water samples.

Soil column tests will be performed in the laboratory to simulate the hydrogeological conditions at each of the disposal sites and to measure the values of a number of factors involved in the migration rates of the various wastes.

III. ORGANIC ANALYSIS

A preliminary report of the organic analyses of water samples collected from Maxey Flats in April 1976 was presented in a previous report.⁽⁵⁾ This report presents additional data of the organic compounds identified in the trench water samples. The organic compounds present in the trench water samples were extracted by using purified amberlite XAD-2 resin* and by liquid-liquid extraction with methylene chloride.**

A. Amberlite XAD-2 Extraction

The purified resin supplied by the manufacturer was further purified by washing several times with distilled water, glass distilled diethyl ether acetonitrile and methanol.** The methanol was drained off and the resin was washed with distilled water prior to passage of the trench water sample. An aliquot (100-200 ml) of the trench water sample was acidified with HCl to pH 2.0 and passed through the XAD-2 resin column (0.6 cm I.D. x 10 cm; resin bed approximately 7.0 cm) at a flow rate of 9-12 ml/min. The organics that were adsorbed by the XAD-2 resin were desorbed with diethyl ether, dried over anhydrous sodium sulfate and concentrated to 1.0 ml with the aid of Kuderna-Danish and Micro-Snyder column evaporators. The concentrated ether extract was divided into two equal portions. Bis (trimethyl silyl) trifluoroacetamide (BSTFA) was added to one portion to form the trimethyl silyl derivatives.

The DOC content of the water sample before and after passage through XAD-2 resin was measured in order to determine the amount of organics retained by XAD-2 resin. These results are shown in Table 1. Considerable amounts of organics were found in the effluent sample after XAD-2 extraction.

B. Methylene Chloride Extraction

The organics that were not retained by the XAD-2 resin but still present in the water sample were extracted three times with 30 ml portions of methylene chloride. The methylene chloride extracts were combined,

* Applied Science Laboratories, State College, Pennsylvania

** Burdick and Jackson Laboratories, Inc., Muskegon, Michigan

TABLE 1

Dissolved Organic Carbon Retained by XAD-2 Resin
in Water Samples Taken from Maxey Flats, April 1976

<u>Sample</u>	<u>DOC (mg/liter)</u>	
	<u>Total</u>	<u>Retained by XAD-2 Resin</u>
Trench 1	52	17 (32.7%)
Trench 5S	153	17 (11.1%)
Trench 7	175	28 (16.0%)
Trench 18	466	92 (19.7%)
Trench 19S	381	66 (17.3%)
Trench 33L4	1900	300 (15.8%)
Trench 33L9	4613	727 (15.8%)
Trench 35A	421	85 (20.2%)

dried over anhydrous Na_2SO_4 , concentrated to 1.0 ml, and the trimethyl silyl derivatives were prepared as described earlier.

C. Gas Chromatography

The ether and methylene chloride concentrated extracts and their trimethyl silyl derivatives were analyzed by gas chromatography (GC).

Typical gas chromatographic conditions were:

GC:	Perkin Elmer 900
Column:	6 ft x 1/8 in i.d. stainless steel column
Packing:	OV-1 5% or 10% on 80/100 mesh chromosorb-W (HP)
Program:	60° for 4 min, then to 250° at 4°/min
Carrier Gas:	Helium at 35 cc/min
Sample Size:	2 μl
Detector:	Flame Ionization

Appropriate blanks were run for each method of sample concentration used in this study.

D. Identification of Organic Compounds

After preliminary GC analyses the extracts were analyzed by combined gas chromatography - mass spectrometry (GC/MS) for identification of the various components. For this purpose, a Hewlett Packard (HP) gas chromatograph 5700/HP 5983A EI/CI Dodecapole mass spectrometer and an HP 5933 data system were used. Identification of the various components present in the trench water extracts were achieved by comparison with known standard spectra from HP contributed libraries (about 2000 spectra), and EPA-NIH library (about 10,000 spectra). In addition, manual searches of the Aldermaston Eight Peak Index of Mass Spectra⁽⁶⁾ were employed. There were several peaks with spectral data that were not identified by the above search methods. It is likely that some of the peaks were not well resolved and probably contain more than one compound. However, some of the unidentified compounds will be identified using the Mass Spectral Search System, handled by the Cyphernetics Corporation, Ann Arbor, Michigan. This system contains about 40,000 reference mass spectra.⁽⁷⁾

The kinds of organics present in the trench water samples are depicted in Tables 2-9. These results should be regarded as preliminary data which illustrate the kinds of organic compounds found in the trench waters. The list is far from complete as it is subject to the limitations of the extraction techniques and the analytical method employed in this study. Furthermore, authentic standard compounds were not run to confirm the identity of these compounds.

TABLE 2

Organic Compounds Identified in Trench 1 Water Sample Taken from Maxey Flats, April 1976

<u>Compound</u>	<u>Major Component*</u>	<u>Minor Component*</u>	<u>Found In</u>	
			<u>XAD-2 Ether Extract</u>	<u>CH₂Cl₂ Extract</u>
Bis (2-ethyl hexyl) adipate	+		+	
Cyclohexanol	+		+	+
1,1-Diethoxy ethane	+		+	
Diethylphthalate		+	+	
Dioctyl adipate	+		+	
Di-(iso-octyl) adipate		+	+	
Fenchone	+		+	
Heptanoic Acid		+	+	
3 Hexanol	+		+	
2-methyl-2-butanol	+		+	
Toluene	+		+	

* Major Component: peak height >5 cm; Minor Component: peak height <5 cm of gas chromatograph attenuation setting 4×10^{-12} amps/mv.

TABLE 3

Organic Compounds Identified in Trench 5S Water Sample Taken from Maxey Flats, April 1976

<u>Compound</u>	<u>Major Component*</u>	<u>Minor Component*</u>	<u>Found In</u>	
			<u>XAD-2 Ether Extract</u>	<u>CH₂Cl₂ Extract</u>
Di-N-Butylphthalate	+			+
Dioctyl adipate	+			+
Dioctyl phthalate		+	+	
Methyl isobutyl ketone	+		+	
Octanoic Acid	+			+
Palmitic Acid		+		+
Stearic Acid		+		+
3,3,5-Trimethylcyclohexanol**	+			+
Unknown phthalates (2)	+			+

* Major Component: peak height >5 cm; Minor Component: peak height <5 cm of gas chromatograph attenuation setting 4×10^{-12} amps/mv.

** Tentative Identification.

TABLE 4

Organic Compounds Identified in Trench 7 Water Sample Taken from Maxey Flats, April 1976

Compound	Major Component*	Minor Component*	Found In	
			XAD-2 Ether Extract	CH ₂ Cl ₂ Extract
Biphenyl	+		+	
Bis (2-chloroethyl) ether	+		+	
2-Butanol	+		+	
O-Cresol	+		+	+
Dibutylphthalate	+			+
1,1-diethoxy-2-chloroethane**	+		+	
2,3-dimethyl-2-hexene	+			+
Dioctyl adipate	+		+	
p-dioxane	+		+	+
2-ethyl hexanoic acid**	+		+	
iso-octyl phthalate		+	+	
Oleic Acid	+			+
Palmitic Acid	+			+
Paraldehyde	+		+	+
2-phenyl cyclohexanol	+		+	
Stearic Acid		+		+
Toluene	+		+	+

* Major Component: peak height >5 cm; Minor Component: peak height <5 cm of gas chromatograph attenuation setting 4×10^{-12} amps/mv.

** Tentative Identification.

TABLE 5

Organic Compounds Identified in Trench 18 Water Sample Taken from Maxey Flats, April 1976

<u>Compound</u>	<u>Major Component*</u>	<u>Minor Component*</u>	<u>Found In</u>	
			<u>XAD-2 Ether Extract</u>	<u>CH₂Cl₂ Extract</u>
Arachidic Acid**	+		+	+
Diethyl phthalate		+	+	
Diethyl phthalate		+	+	
p-dioxane	+		+	
3-ethyl hexanol		+	+	
Tetrahydrofuran		+		+
Tributyl phosphate	+		+	
Triethyl phosphate		+	+	

* Major Component: peak height >5 cm; Minor Component: peak height < 5 cm of gas chromatograph attenuation setting 4×10^{-12} amps/mv.

** Tentative Identification.

TABLE 6

Organic Compounds Identified in Trench 19S Water Sample Taken from Maxey Flats, April 1976

Compound	Major Component*	Minor Component*	Found in	
			XAD-2 Ether Extract	CH ₂ Cl ₂ Extract
Acetic Acid		+		+
Butanoic Acid		+	+	+
p-cresol	+		+	
Diethyl phthalate		+	+	
Di-2-ethyl hexylphthalate	+		+	+
Dioctyl adipate	+		+	
p-dioxane	+			+
Ethyl acetate		+	+	
Formic Acid		+		+
Oxalic Acid		+		+
2-methyl butanoic acid	+		+	

* Major Component: peak height >5 cm; Minor Component: peak height <5 cm of gas chromatograph attenuation setting 4×10^{-12} amps/mv.

TABLE 7

Organic Compounds Identified in Trench 33L4 Water Sample Taken from Maxey Flats, April 1976

Compound	Major Component*	Minor Component*	Found In	
			XAD-2 Ether Extract	CH ₂ Cl ₂ Extract
Aceto vanillon		+		+
Benzoic Acid	+			+
Borneol**		+	+	
Butyric Acid		+		+
Cyclohexanol		+		+
O-cresol	+			+
1,1-Diethoxyethane		+	+	
Dioctyl phthalate		+		+
p-dioxane	+		+	+
2-ethyl hexanol	+		+	+
2 ethyl heptanoic acid**	+		+	+
Hexanoic Acid	+			+
4-hydroxy-3-methoxy benzoic acid		+		+
iso butyric acid**		+		+
Methylcyclohexanol**	+		+	+
2-methoxy phenylacetic acid		+		+
Octanol	+		+	+
Phenylacetic Acid	+			+
Phenylpropionic Acid	+			+
Tetrahydrofuran		+		+
Toluene		+	+	
Vanillin	+		+	+

* Major Component: peak height >5 cm; Minor Component: peak height <5 cm of gas chromatograph attenuation setting 4×10^{-12} amps/mv.

** Tentative Identification.

TABLE 8

Organic Compounds Identified in Trench 33L9 Water Sample Taken from Maxey Flats, April 1976

<u>Compound</u>	<u>Major Component*</u>	<u>Minor Component*</u>	<u>Found In</u>	
			<u>XAD-2 Ether Extract</u>	<u>CH₂Cl₂ Extract</u>
p-dioxane	+			+
2 ethyl-1-hexanol	+		+	+
Tributyl phosphate	+		+	+
1,1,1-Trichloroethane		+		+

* Major Component: peak height >5 cm; Minor Component: peak height < 5 cm of gas.
chromatograph attenuation setting 4×10^{-12} amps/mv.

TABLE 9

Organic Compounds Identified in Trench 35A Water Sample Taken from Maxey Flats, April 1976

<u>Compound</u>	<u>Major Component*</u>	<u>Minor Component*</u>	<u>Found in</u>	
			<u>XAD-2 Ether Extract</u>	<u>CH₂Cl₂ Extract</u>
Benzoic Acid.	+			+
O-cresol		+	+	+
p-cresol		+	+	+
Cyclohexane	+			+
Diacetone alcohol**		+		+
Diethyl phthalate		+	+	
Dioctyl phthalate	+			+
p-dioxane	+		+	+
3-ethyl-1-hexanol	+		+	
1-iso-butyl-4-ethyl octyl bromide**		+		+
Iso valeric acid	+		+	+
2-mercapto benzoic acid**	+		+	
Octanol		+	+	
Octanoic Acid		+	+	
Phenylacetic Acid	+			+
Phenylpropionic Acid	+			+
Toluene	+		+	
O-Toluic Acid		+		+

* Major Component; peak height >5 cm; Minor Component: peak height < 5 cm of gas chromatograph attenuation setting 4×10^{-12} amps/mv.

** Tentative Identification.

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