EVALUATION OF ISOTOPE MIGRATION - LAND BURIAL
WATER CHEMISTRY AT COMMERCIALY OPERATED LOW-LEVEL
RADIOACTIVE WASTE DISPOSAL SITES

PROGRESS REPORT No. 5

APRIL - JUNE 1977

MASTER

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

DATE PUBLISHED - DECEMBER 1977

NUCLEAR WASTE MANAGEMENT RESEARCH GROUP

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Prepared for the U.S. Nuclear Regulatory Commission
Office of Nuclear Regulatory Research
Contract No. EY-76-C-02-0016

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Printed in the United States of America
Available from

National Technical Information Service
U.S. Department of Commerce
5285 Port Royal Road
Springfield, VA 22161

Price: Printed Copy \$4 50; Microfiche \$3 00

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SUMMARY

Water samples were collected from ten trenches at the Maxey Flats (Morehead) Kentucky, low-level radioactive waste disposal site and analyzed for the presence of organic compounds. Identification of the organics was achieved by combined gas chromatography - mass spectrometry. Alcohols, aliphatic and aromatic acids, phthalates, adipates, and tributyl phosphate are the predominant kinds of compounds found in most of the trench water samples. These organic compounds may react with the radio-nuclides buried in the trenches and possibly affect their mobility in the environment.

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 with the prediction that the current burial capacity of all six existing sites may be exceeded between 1988⁽²⁾ and 1998⁽³⁾ when existing sites may have to be closed or enlarged or new sites will have to be established. At the present time, the West Valley, New York, site has discontinued disposal operations and the Maxey Flats, Kentucky, site has reduced its disposal volume.

Recently, questions have been raised regarding the adequacy of these disposal sites.^(4,5) 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 states 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 parameters 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. INTRODUCTION

Although minimal information is available on the specific nature or form of organic wastes buried at the Maxey Flats, Kentucky, low-level radioactive waste disposal site, it is obvious that they consist of both solids and liquids such as protective clothing, plastics, decontamination solutions, animal carcasses, etc.⁽⁶⁾ Since wastes coming into the burial site were not segregated, it can be assumed that practically every trench at Maxey Flats contains varying concentrations of organic type wastes. These organics can react chemically with other constituents in the trenches or they can be acted upon by bacteria to produce organic compounds which are capable of solubilizing or forming complexes with radionuclides and possibly affect the mobility of the radionuclides in the ground.

A preliminary analysis of the trench water samples taken at Maxey Flats site early in 1976 indicated the presence of a variety of organic compounds.⁽⁷⁾ In the fall of that year, trench water samples were collected again for a more detailed analysis of radiochemical, inorganic and organic constituents. The results of the radiochemical and inorganic analyses are presented in the 4th Quarterly Progress Report.⁽⁸⁾ This report describes the extractable and chromatographable organic compounds identified from the trench water samples.

IV. MATERIALS AND METHODS

A second set of trench water samples were collected from Maxey Flats in September 1976. Due to the lack of accumulated water in several of the trenches that were previously sampled, only trenches 7, 18, 19S, 33L4, and 33L9 that had water were sampled. In addition, water samples from trenches 2, 26, 27, 32, and 37 were collected for analysis for the first time. The location of the trenches sampled are shown in Figure 1.

All the samples were collected under anoxic conditions as described by Colombo et al.⁽⁹⁾ Samples were kept cold in ice and shipped to the laboratory. Upon receipt at the laboratory, they were refrigerated at $\sim 4^{\circ}\text{C}$.

For organic analysis, the trench water sample was filtered through a $0.45\ \mu$ silver membrane filter under anoxic conditions.⁽⁸⁾ The filtrate was collected in a clean, baked, one liter amber bottle fitted with Teflon-lined screw cap and was stored at $\sim 4^{\circ}\text{C}$ until further analysis.

Dissolved Organic Carbon Analysis:

The total and inorganic carbon content of the filtered trench water samples were determined by using the Beckman Total Carbon Analyzer. The difference between the total and the inorganic carbon yielded the dissolved organic carbon (DOC) content of the water samples.

Organic Analysis:

The scheme used for the extraction and separation of the various organic constituents present in the trench water sample is shown in Figure 2. An aliquot (200-500 ml) of the filtered trench water sample was acidified with HCl to pH 2.0 and was extracted three times with 30 ml portions of methylene chloride.* The combined methylene chloride extracts containing the acid and neutral compounds was dried over anhydrous Na_2SO_4 and concentrated

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

NOTE: SCALE APPLIES ONLY TO FENCELINE. POSITION AND DIMENSIONS OF TRENCHES ARE APPROXIMATE.

FIGURE 1 Sketch of Maxey Flats Radioactive Waste Disposal Site
(updated from sketch by EMCON Associates). (8)

FIGURE 2 Extraction and Separation Scheme used in the Organic Analysis of Trench Water Samples.

to 1.0 ml with the aid of Kuderna-Danish and Micro-snyder column evaporators. The concentrated methylene chloride extract was divided into two equal portions. Bis(trimethyl silyl)trifluoroacetamide (BSTFA) was added to one portion to form the trimethyl silyl (TMS) derivatives.

The aqueous layer containing the basic compounds was adjusted to pH 11 with NaOH and extracted three times with 30 ml portions of the methylene chloride. The combined methylene chloride extracts containing the basic organic compounds was dried over anhydrous Na_2SO_4 and concentrated to 1.0 ml.

Appropriate control blanks with the same volume of the solvent and reagents were included for each set of analysis.

Gas Chromatography:

The methylene chloride concentrated extracts and the trimethyl silyl derivatives were analyzed by gas chromatography. Typical gas chromatographic conditions were:

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

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 was achieved by comparison with known standard spectra from HP contributed libraries (about 2,000

spectra), and EPA-NIH library (about 10,000 spectra). In addition, manual searches of the Aldermaston Eight Peak Index of Mass Spectra, and a computer search using the Mass Spectral Search System (MSSS) handled by the Cyphernetics Corporation, Ann Arbor, Michigan⁽¹⁰⁾ were used to identify some of the compounds. There were several peaks with spectral data that were not identified by the above methods. It is likely that some of the peaks were not well resolved and probably contain more than one compound.

V. RESULTS AND DISCUSSION

The dissolved organic carbon content of the several trench water samples are listed in Table 1. The concentration of DOC varied from trench to trench and was generally in the range of 200 to 4,500 mg/liter. In certain trench water samples, for example, trenches 2, 7, 18, and 32 both inorganic and organic carbon concentrations were more or less the same.

The kinds of organic compounds identified from the trench water samples are shown in Figures 3-12. In general the organic compounds found in the samples are organic solvents, decontamination liquids such as tributylphosphate, several low molecular weight aliphatic and aromatic acids and several unidentified adipates and phthalates. In addition, volatile amines such as cyclohexylamine and dicyclohexylamine were also identified.

The organics listed in Figures 3-12 are far from complete due to the limitations of the extraction techniques and analytical methods used in this study. Since no special column was used in the GC analysis for the separation of the basic fraction, it is possible that some of the basic compound(s) may have been left undetected. Furthermore, no effort has been made thus far to quantify the organic compounds.

The trench water samples are rich in organic carbon and nitrogen compounds, and thus provide a favorable environment for the proliferation of microorganisms. The organic compounds can be degraded to yield CO_2 , low molecular weight organic acids, alcohols, aldehydes, ketones, and esters in soils under anaerobic conditions.^(11,12) The presence of electron acceptors such as NO_3^- and $\text{SO}_4^{=}$ will support growth of certain aerobic bacteria under anoxic conditions. As a strict anaerobic environment develops, CO_2 and low molecular weight organic compounds, such as acetate, will be converted to methane by methanogenic bacteria. The presence of several low molecular weight organic acids and

CO₂ in several trench waters suggests the possible involvement of microbial activity.

The organic compounds may play an important role in the formation of organo-metallic complexes, leachability, solubility, and mobility of the radiochemicals that are buried in the trenches. Future study will focus on detailed qualitative and quantitative analysis of a few selected trench water samples for various organic constituents, such as hydrophobic and hydrophilic components, that are known or likely to influence the transport of radionuclides in the ground.

Table 1

Concentration of Dissolved Carbon in Trench Water

Samples Taken from Maxey Flats, September 1976

Sample	Total Carbon mg/liter	Inorganic Carbon mg/liter	Organic Carbon mg/liter
Trench 2	480	270	210
Trench 7	460	210	250
Trench 18	940	440	500
Trench 19S	700	78	620
Trench 26	1100	150	950
Trench 27	730	<2	730
Trench 32	1300	510	790
Trench 33L4	1900	<2	1900
Trench 33L9	4700	180	4500
Trench 37	3300	23	3300

FIGURE 3 GAS CHROMATOGRAM OF METHYLENE CHLORIDE EXTRACT OF TRENCH 2 WATER SAMPLE

Key to Figure 3

Acidic Fraction

<u>Peak #</u>	<u>Compound</u>
1	Toluene
2	Bis(2-chloroethyl) ether
3	2-Ethylhexanol
4	Bis(2-chloroethoxy) methane
5	Butoxyethane (?)
6	Tributylphosphate
7	(Propylene glycol) n
8	Unknown Phthalate
9	(Propylene glycol) n
10	(Propylene glycol) n
11	Unknown Phthalate
12	(Propylene glycol) n

Acidic Fraction - Tms derivative

<u>Peak #</u>	<u>Compound</u>
1	2-Ethylhexanol - Tms
2	Bis(2-chloroethoxy) methane
3	C ₅ acid - Tms
4	C ₈ acid - Tms
5	C ₈ acid - Tms
6	C ₈ acid - Tms
7	Tributylphosphate
8	Unknown Phthalate

Basic Fraction

<u>Peak #</u>	<u>Compound</u>
1	Unknown Phthalate
2	Diethylphthalate
3	Unknown Phthalate
4	Unknown Phthalate
5	Unknown Phthalate

Unnumbered peaks are unidentified compounds.

FIGURE 4 GAS CHROMATOGRAM OF METHYLENE CHLORIDE EXTRACT OF TRENCH 7 WATER SAMPLE

Key to Figure 4

Acidic Fraction

<u>Peak #</u>	<u>Compound</u>
1	Bis(2-chloroethyl)ether*
2	Cresol isomer*
3	Cresol isomer
4	1,2-Bis(2-chloroethoxy)ethane
5	4-tert-Butyl phenol
6	2-Phenylcyclohexanol*

Acidic Fraction - Tms derivative

<u>Peak #</u>	<u>Compound</u>
1	C ₆ acid - Tms
2	Cresol isomer - Tms
3	Cresol isomer - Tms
4	C ₈ acid - Tms
5	C ₈ acid - Tms
6	C ₈ acid - Tms
7	C ₈ acid - Tms
8	C ₈ acid - Tms
9	Phenylacetic acid - Tms

Basic Fraction

<u>Peak #</u>	<u>Compound</u>
1	p-Dioxane
2	Aniline
3	Unknown Phthalate
4	Dioctyl phthalate
5	Unknown Phthalate
6	Unknown Phthalate
7	Unknown Phthalate
8	Unknown Phthalate

Unnumbered peaks are unidentified compounds.

*Compounds were found in sample collected in April, 1976.

FIGURE 5 GAS CHROMATOGRAM OF METHYLENE CHLORIDE EXTRACT OF TRENCH 18A WATER SAMPLE

Key to Figure 5

Acidic Fraction

<u>Peak #</u>	<u>Compound</u>
1	Triethyl phosphate*
2	4-tert-Butyl phenol
3	Tripropylene glycol methyl ether
4	Diethyl phthalate*
5	2,2,4-trimethyl pentane-1,3-diol diisobutyrate
6	N-ethyl-tol-?-yl sulfonamide (?)
7	Tributyl phosphate*
8	N-ethyl-tol-?-yl sulfonamide (?)
9	Dimethyl naphthalene (?)
10	Di-n-heptyl adipate (?)
11	Unknown Adipate
12	Unknown Phthalate
13	Unknown Phthalate
14	Unknown Phthalate
15	Unknown Phthalate
16	Unknown Phthalate
17	Unknown Phthalate

Acidic Fraction - Tms derivative

<u>Peak #</u>	<u>Compound</u>
1	C8 acid - Tms
2	C8 acid - Tms
3	C8 acid - Tms
4	Tributyl phosphate
5	Diocetyl phthalate*
6	Unknown Phthalate

Basic Fraction

<u>Peak #</u>	<u>Compound</u>
1	Cyclohexylamine
2	Dicyclohexylamine
3	Unknown Phthalate
4	Diocetyl phthalate

Unnumbered peaks are unidentified compounds.

*Compounds were found in sample collected in April, 1976.

FIGURE 6 GAS CHROMATOGRAM OF METHYLENE CHLORIDE EXTRACT OF TRENCH 19 WATER SAMPLE

Key to Figure 6

Acidic Fraction

<u>Peak #</u>	<u>Compound</u>
1	Butanoic acid*
2	Pentanoic acid
3	Hexanoic acid
4	Cresol*
5	Phenylpropionic acid
6	Tributyl phosphate

Acidic Fraction - Tms derivative

<u>Peak #</u>	<u>Compound</u>
1	2-Methyl butanoic acid - Tms*
2	Pentanoic acid - Tms
3	C ₆ acid - Tms
4	C ₆ acid - Tms
5	Hexanoic acid - Tms
6	C ₇ acid - Tms
7	Cresol - Tms
8	2-Ethyl hexanoic acid - Tms
9	Benzoic acid - Tms
10	Octanoic acid - Tms
11	Phenylacetic acid - Tms
12	Toluic acid - Tms
13	C ₉ acid - Tms
14	Phenylpropionic acid - TMS
15	C ₁₀ acid - Tms

Unnumbered peaks are unidentified compounds.

* Compounds were found in sample collected in April 1976.

FIGURE 7 GAS CHROMATOGRAM OF METHYLENE CHLORIDE EXTRACT OF TRENCH 26A WATER SAMPLE

Key to Figure 7

Acidic Fraction

<u>Peak #</u>	<u>Compound</u>
1	p-Dioxane
2	Toluene

Acidic Fraction Tms derivative

<u>Peak #</u>	<u>Compound</u>
1	2-Methyl butanoic acid - Tms
2	Pentanoic acid - Tms
3	C ₆ acid - Tms
4	Hexanoic acid - Tms
5	C ₇ acid - Tms
6	C ₈ acid - Tms
7	Cresol - Tms
8	C ₈ acid - Tms
9	C ₈ acid - Tms
10	C ₈ acid - Tms
11	Benzoic acid - Tms
12	C ₈ acid - Tms
13	Phenylacetic acid - Tms
14	C ₉ acid - Tms
15	Phenyl propionic acid - Tms
16	C ₁₀ acid - Tms

Basic Fraction

<u>Peak #</u>	<u>Compound</u>
1	p-Dioxane
2	Unknown Adipate
3	Unknown Phthalate
4	Unknown Phthalate

Unnumbered peaks are unidentified compounds.

FIGURE 8. GAS CHROMATOGRAM OF METHYLENE CHLORIDE EXTRACT OF TRENCH 27 WATER SAMPLE

Key to Figure 8

Acidic Fraction

<u>Peak #</u>	<u>Compound</u>
1	2-Methyl pentanoic acid
2	2-Ethyl hexanol
3	Hexanoic acid
4	Cresol
5	C ₈ acid
6	C ₉ acid

Acidic Fraction - Tms derivative

<u>Peak #</u>	<u>Compound</u>
1	2-Methyl propionic acid - Tms
2	2-Methyl butanoic acid - Tms
3	Pentanoic acid - Tms
4	C ₆ acid - Tms
5	Phenol - Tms
6	Hexanoic acid - Tms
7	C ₇ acid - Tms
8	Octanol - Tms
9	C ₈ acid - Tms
10	Benzoic acid - Tms
11	Octanoic acid - Tms
12	C ₉ acid - Tms
13	Phenylpropionic acid - Tms
14	Decanoic acid - Tms

Basic Fraction

<u>Peak #</u>	<u>Compound</u>
1	Unknown Adipate
2	Unknown Adipate
3	Unknown phthalate
4	Unknown Adipate
5	Diethylphthalate
6	Unknown Phthalate
7	Unknown Phthalate
8	Unknown Phthalate
9	Unknown Phthalate

Unnumbered peaks are unidentified compounds.

FIGURE 9 GAS CHROMATOGRAM OF METHYLENE CHLORIDE EXTRACT OF TRENCH 32 WATER SAMPLE

Key to Figure 9

Acidic Fraction

<u>Peak #</u>	<u>Compound</u>
1	Toluene
	Butanoic acid
2	Xylene
	Methyl-n-pentanoic acid
	Pentanoic acid
3	Phenol
4	Hexanoic acid
5	Cresol
6	Naphthalene
7	α -Terpineol
8	Phenylpropionic acid
9	4-(2,5-xylyl) butanoic acid
10	Tetramethyl butyl phenol
11	Tributyl phosphate
12	?-(?, ?-xylyl) butanoic acid

Acidic Fraction - Tms derivative

<u>Peak #</u>	<u>Compound</u>
1	2-Methyl butanoic acid - Tms
2	Pentanoic acid - Tms
3	C ₆ acid - Tms
4	C ₆ acid - Tms
5	Phenol - Tms
6	Hexanoic acid - Tms
7	C ₇ acid - Tms
8	Cresol - Tms
9	C ₈ acid - Tms
10	Benzoic acid - Tms
11	Phenylacetic acid - Tms
12	Toluic acid - Tms
13	Phenylpropionic acid - Tms

Basic Fractions

<u>Peak #</u>	<u>Compound</u>
1	p-Dioxane
2	Unknown Adipate
3	Unknown Adipate

Key to Figure 9 (Cont'd)

Basic Fractions (Cont'd)

<u>Peak #</u>	<u>Compound</u>
4	Unknown Phthalate
5	Unknown Phthalate
6	Dioctyl Phthalate
7	Unknown Phthalate
8	Unknown Phthalate
9	Unknown Phthalate
10	Unknown Phthalate

Unnumbered peaks are unidentified compounds.

FIGURE 1C GAS CHROMATOGRAM OF METHYLENE CHLORIDE EXTRACT OF TRENCH 33L4 WATER SAMPLE

Key to Figure 10

Acidic Fraction

<u>Peak #</u>	<u>Compound</u>
1	Butanoic acid*
2	Cyclohexanol*
3	2-Ethyl hexanol
4	Cresol*
5	2-Ethyl hexanoic acid
6	Vanillin*
7	Acetovanillon*
8	4-Hydroxy-3-methoxy phenylacetic acid

Acidic Fraction - Tms derivative

<u>Peak #</u>	<u>Compound</u>
1	2-Methyl butanoic acid - Tms
2	Pentanoic acid - Tms
3	Phenol - Tms
4	C ₆ acid - Tms
5	2-Ethyl hexanol - Tms*
6	Cresol - Tms*
7	C ₆ acid - Tms
8	Benzoic acid - Tms*
9	Octanoic acid - Tms
10	Phenylacetic acid - Tms
11	p-Hydroxybenzaldehyde - Tms
12	Phenylpropionic acid - Tms
13	Vanillin - Tms
14	3-Methoxy-4-hydroxy-benzoic acid* - DITMS

Basic Fraction

<u>Peak #</u>	<u>Compound</u>
1	p-Dioxane

Unnumbered peaks are unidentified compounds.

*Compounds were found in sample collected in April, 1976.

FIGURE 11 GAS CHROMATOGRAM OF METHYLENE CHLORIDE EXTRACT OF TRENCH 33L9 WATER SAMPLE

Key to Figure 11

Acidic Fraction

<u>Peak #</u>	<u>Compound</u>
1	2-Ethyl hexanol*
2	Tributyl phosphate*

Acidic Fraction - Tms derivative

<u>Peak #</u>	<u>Compound</u>
1	2-Ethyl hexanol - Tms*
2	Tributyl phosphate*

Basic Fraction

<u>Peak #</u>	<u>Compound</u>
1	p-Dioxane*

Unnumbered peaks are unidentified compounds.

*Compounds were found in sample collected in April, 1976.

FIGURE 12 GAS CHROMATOGRAM OF METHYLENE CHLORIDE EXTRACT OF TRENCH 37 WATER SAMPLE

Key to Figure 12

Acidic Fraction

<u>Peak #</u>	<u>Compound</u>
1	p-Dioxane
2	Toluene
3	Xylene
4	Naphthalene
5	Diethyl phthalate

Acidic Fraction - Tms derivative

<u>Peak #</u>	<u>Compound</u>
1	Phenol - Tms
2	C ₆ acid - Tms
3	2-Ethylhexanol - Tms
4	Cresol - Tms
5	C ₈ acid - Tms
6	Benzoic acid - Tms
7	Octanoic acid - Tms
8	Phenylacetic acid - Tms
9	Phenylpropionic acid - Tms

Unnumbered peaks are unidentified compounds.

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