

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

PROGRESS REPORT No. 9

APRIL - JUNE 1978

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P. COLOMBO AND A.J. WEISS

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NUCLEAR WASTE MANAGEMENT RESEARCH GROUP

DEPARTMENT OF NUCLEAR ENERGY BROOKHAVEN NATIONAL LABORATORY
UPTON, NEW YORK 11973



Prepared for the U.S. Nuclear Regulatory Commission
Office of Nuclear Regulatory Research
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502 284

Table of Contents

	<u>Page</u>
Summary	v
I. Introduction	1
II. Experimental	3
A. Materials	3
B. Equipment	4
C. Procedure	6
III. Results	
A. Effect of Environment on K_d	8
B. Effect of Water/Soil Ratio on K_d	8
C. Variation of K_d with Solution Acidity	9
IV. Conclusion	
References	11
Distribution List	29

List of Tables

1. Physical and Chemical Data for Trench Water WV-37 (Trench 5) Collected from West Valley, New York, Disposal Site November 1977	12
2. Mineral Composition of Unweathered Till from West Valley, New York, Disposal Site 1975	13
3. Chemical Analysis of Unweathered Till from West Valley, New York, Disposal Site 1975	14
4. Operations Sequence to Replace Air in the K_d Manifold with Nitrogen	15
5. Control Positions for Manipulating Trench Water in the Sample Preparation Manifold	16
6. K_d Sorption Coefficient of Trench Water and Soil from West Valley, New York, Disposal Site	17

List of Figures

	<u>Page</u>
1. Trench Water Samples Collected Anoxically at the West Valley, New York, Low-Level Radioactive Waste Disposal Site, November 1977	18
2. Equipment for Anoxic K_d Experiment	18
3. Sketch of West Valley Low-Level Radioactive Waste Disposal Site (from sketch by USGS)	19
4. Schematic Diagram of the Filtration System for K_d Manifold	20
5. Breakdown of the Filtration Unit Used in K_d Experiments	20
6. Schematic Diagram of the Sample Preparation Manifold for Anoxic K_d Experiments	21
7. Sample Preparation Manifold	22
8. Probe Chamber for Measuring pH, Eh, and Temperature	23
9. Glass Tube with Screw Cap Rubber Septum	24
10. Teflon Bottle with Teflon-Lined Flat Rubber Septum and Collimated Counting Shield	24
11. Teflon Bottle with Septum and O-Ring Seals	25
12. Sample Tumbling Chamber with Provision for Maintaining Nitrogen Atmosphere	25
13. Collimated Lead Shield for Counting K_d Samples with Ge(Li) Detector	26
14. K_d Sorption Coefficient Versus Water/Soil Ratio for ^{60}Co	26
15. K_d Sorption Coefficient Versus Water/Soil Ratio for ^{85}Sr	27
16. K_d Sorption Coefficient Versus Water/Soil Ratio for $^{134,137}\text{Cs}$	27
17. K_d Sorption Coefficient Versus Addition of Acid or Base	28

502 286

SUMMARY

This report describes equipment and procedures developed for measuring the sorption distribution coefficient, K_d , of selected radionuclides using site specific soils and trench waters from low-level radioactive waste disposal sites. Batch experiments were conducted under anoxic and oxic conditions with soil and trench water from the West Valley, New York, disposal site. Variations in K_d as a result of changes in pH, aeration, and water to soil ratio are reported.

I. INTRODUCTION

The ultimate container for long-term storage of low-level radioactive wastes buried in shallow land disposal sites is the geologic media of the disposal site. The cardboard boxes, plastic bags, steel drums and concrete casks in which the wastes are presently buried will eventually deteriorate due to weathering and corrosion. Isolation of the buried radionuclides for long periods of time will depend upon several factors, such as the stability of the geologic environment, the hydrological conditions at the site, and interactions of the radionuclides with the geologic media. Retention of radionuclides by geologic media is summarized by the commonly used term "sorption". The mechanisms by which sorption takes place depends upon the chemical and physical interactions between the geologic media and the matrices containing the radionuclides. Materials in the geologic media, such as soil, clay, and rock are complex mixtures of minerals and organic matter. Trench waters at the shallow land disposal sites are complex solutions containing a variety of dissolved components which can influence radionuclide sorption reactions. Evaluation of the interactions of these materials is essential to the understanding of sorption mechanisms and for predicting radionuclide behavior in these environments.

To evaluate the potential for radionuclide migration, one of the parameters that must be quantified is the distribution of the radionuclides between the complex solid and liquid phases. This distribution is expressed as an experimentally determined distribution coefficient, K_d , for each element of interest. The K_d parameter, which is included in all ground-water solute transport models, is generally determined by batch and column methods. These batch and column experiments will be conducted using site specific soils and trench waters from the low-level radioactive waste disposal sites. Column studies will be conducted by passing trench water through representative cores obtained from the burial sites and mapping the resultant radionuclide distribution along the lengths of the cores. These data will be compared to the distribution of radionuclides in cores taken from beneath the burial trenches at the disposal sites.

In this study, the K_d of a radionuclide is defined as the ratio of specific activity in the soil to specific activity in the water after the two phases have been in contact for a given length of time.

$$K_d = \frac{\text{Soil Activity/Weight of Soil}}{\text{Liquid Activity/Volume of Liquid}} \quad (1)$$

Theoretically, K_d is the value of this ratio when a state of equilibrium exists between the two phases. However, if the contact time is long enough to approach equilibrium, the measured K_d will be a good approximation of the theoretical value. K_d is dependent upon the conditions of the system, including temperature, pH, chemical potential, dissolved anions and cations, soil exchange capacity, surface area of the soil, and the ratio of solid to liquid phases.

Sorption studies have generally been performed under oxic conditions. When sorption was studied under both oxic and anoxic conditions, the results for plutonium differed by as much as an order of magnitude.⁽¹⁾

Reducing conditions exist in the trenches at low-level disposal sites due to microbiological degradation of buried organic wastes. As a result, the Eh and dissolved oxygen in trench waters are low. In order to measure K_d under conditions similar to those existing at the disposal sites, batch experiments were performed in an anoxic environment using actual trench water and unweathered soil from the sites.

17. EXPERIMENTAL (G.G. Galdi, R.F. Pietrzak, A.J. Weiss)

The procedure for collecting trench water under anoxic conditions was described in a previous report.⁽²⁾ The trench waters were stored at approximately 4°C in the glass collection bottles shown in Figure 1. Figure 2 shows the manifold that was constructed to perform a series of anoxic operations with trench water. These operations include: (a) filtration, (b) dilution, (c) pH adjustment, (d) addition of specific radionuclides, and (e) transfer to reaction vessel containing soil. The activity remaining in the liquid phase after mixing with the soil is determined by gamma-ray spectroscopy using a Ge(Li) detector.

A. Materials

1. Anoxic Trench Water

The K_d data presented in this report were obtained with trench water (WV-37) collected November 1977 from Trench 5 at the West Valley, New York, disposal site shown in Figure 3.⁽³⁾ Some analytical data for this water sample are given in Table 1. Additional analyses, including dissolved organic compounds, are in progress and will be presented in a subsequent report.

2. Soil

Unweathered till from test hole A2, collected by the U.S. Geological Survey in the summer of 1975 at the West Valley, New York, disposal site, was used in these experiments. Test hole A2 is located adjacent to and east of Trench 2 as shown in Figure 3. Mineralogical and chemical data of representative samples of unweathered till are given in Tables 2 and 3.⁽⁴⁾ A sample of core A2 from a depth of 34.8 to 35.1 feet was ground in a ball mill, and a 100-200 mesh (149 μ -74 μ) fraction was isolated for these experiments. The surface area of this fraction is 10.9 m²/gm based on the BET method using a Quantisorb Surface Area Analyzer.^(5,6)

3. Tracer Solution

A carrier-free solution containing $\sim 50 \mu\text{Ci/ml}$ each of commercially obtained ^{85}Sr , $^{134,137}\text{Cs}$, and ^{60}Co in 0.5M HCl was used to spike the filtered trench water. A 0.1 ml aliquot of tracer solution in 110 ml of trench water contributes $\sim 5 \times 10^{-4} \text{ mmoles of H}^+$ per ml of trench water.

B. Equipment

1. Filtration System

Trench water samples are filtered anoxically as described in a previous report.⁽²⁾ A schematic diagram of the filtration system is shown in Figure 4, and a breakdown of the filter unit is shown in Figure 5. Trench water is transferred from the collection bottle to the filter unit through $3/8"$ i.d. Tygon tubing, and filtered through a 142 mm diameter, 0.1 micron cellulose membrane. All filter body surfaces that come in contact with the trench water are coated with Teflon.

2. Sample Preparation Manifold

The primary function of the sample preparation manifold is to prepare the filtered trench water for the K_d experiment and to transfer measured amounts of the water to reaction vessels containing soil. A schematic diagram of the manifold is shown in Figure 6. The valve operations to perform various manifold manipulations are indicated in the diagram. Three types of valves on the manifold are used to route the trench water: (a) two position/double function valves (PCV, RVV), (b) two position/triple function valves (SV, FV, SIV), and (c) a six position/single function valve (DV). All tubing and valve surfaces of the sample preparation manifold shown in Figure 7 are Teflon.

The manipulations required to prepare a sample for a K_d measurement are described in Tables 4 and 5. Table 4 gives sequential operations for replacing air with nitrogen in various sections of

the apparatus, and Table 5 shows the positions of controls on the manifold for manipulating the filtered trench water. These manipulations include: (a) diverting water to the probe chamber shown in Figure 8 where pH, Eh and temperature are measured, (b) filling the mixing reservoirs where pH adjustments, dilutions, and tracer radionuclide additions are made, and (c) transferring 14.6 ml of water from a mixing reservoir to a reaction vessel containing 0.75 gm of soil.

3. Reaction Vessels

a. Disposable Glass Tubes:

Commercially available 20 ml glass tubes that are sealed with screw cap septa (Figure 9) were used in some K_d experiments. Approximately 2% of the original radioactivity in the solution was adsorbed onto the container wall, and in approximately 10% of the experiments, the tubes failed to maintain an anoxic condition. Failure to maintain an anoxic condition is easily seen by the presence of a brown $Fe(OH)_3$ precipitate.

b. Teflon Bottles:

Teflon bottles were machined from TFE rod and crimp-sealed with Teflon-lined flat rubber septa (Figure 10). These bottles were found to adsorb less than 0.1% of the radioactivity and are ideally suited for K_d experiments, especially where the K_d is large and little of the initial activity remains in solution. The failure rate for maintaining anoxic conditions was high. This problem was resolved by constructing Teflon bottles with both septum and o-ring seals (Figure 11) and working in an inert atmosphere glove box. The single seal Teflon bottles can be used in K_d experiments where anoxic conditions are not required and where the problem of wall adsorption is to be avoided.

4. Sample Mixing Tumbler

The samples are mixed by tumbling the reaction vessels on a ball mill roller. A plastic chamber designed to maintain an inert atmosphere around the sample bottles during the equilibration process is shown in Figure 12. Inert gas is passed through the chamber to insure an anoxic environment in the reaction vessel in the event of an improper seal in the reaction vessel. When anoxic conditions are not required, mixing is effected by packing the reaction vessels in an appropriate container and tumbling on a ball mill roller.

5. Counting Shield

After equilibration, the liquid phase is separated from the solid phase by centrifugation at 3,000 rpm for 20 minutes and analyzed with a Ge(Li) counting system. The reaction vessel is counted in a collimated lead shield shown in Figures 10 and 13, where the soil in the bottom of the vessel is below the circular window facing the detector. Thus, approximately three inches of lead is between the soil and the detector, and as such the detector sees the gamma radiation from the liquid phase with an insignificant contribution from the soil. This counting configuration eliminates opening the reaction vessel to separate the liquid from the soil and enables repeated shaking and counting of the same sample to determine the optimum mixing time to approach equilibrium.

The small fraction of radiation from the soil that penetrates the shield becomes significant when the K_d is very large. In this case, the centrifuged sample is opened in an inert atmosphere glove box to separate the liquid from the soil before counting.

C. Procedure

Soil samples are added to the reaction vessels which are crimp sealed and connected to the K_d manifold. Air in the manifold, filtration unit, probe chamber, and vessels is replaced with inert gas by alternately evacuating and refilling with inert gas at least six times. This

502 292

prevents iron hydroxide from precipitating during the equilibration period. The procedure is summarized in the operation sequence chart shown in Table 4. The chart is read from left to right and from top line down. The filtration apparatus is purged with inert gas prior to purging the manifold. Filtered trench water is passed through the probe chamber where pH, Eh, and temperature are measured. The filtrate is then collected in four of the mixing reservoirs which may contain radionuclide spike, acid, base, or deionized water. Measured volumes of a modified trench water are transferred to an empty control vessel and to each of two reaction vessels containing soil. The transfer lines are then washed with water from the fifth mixing reservoir. Thus, four variations of a single trench water can be studied in one run. The manipulations to accomplish these tasks are shown in Table 5. The reaction vessels containing soil and trench water are removed from the manifold and mixed in the tumbling apparatus for a known length of time. After centrifugation, the samples are analyzed by gamma-ray spectroscopy using a Ge(Li) detector. Samples are counted long enough to accumulate 10,000 counts in each gamma-ray peak, or for a maximum of 1,000 minutes, whichever is reached first. After counting is completed, the final pH and Eh of the water is measured in an inert atmosphere glove box.

502 293

III. RESULTS

Measured K_d values were found to depend upon experimental conditions such as (a) environment (anoxic or oxic), (b) water/soil ratio, and (c) solution acidity. These results were obtained by equilibrating trench water (WV-37) from Trench 5 with unweathered till (WV-A2) for 18 hours. Anoxic experiments were conducted in Teflon reaction vessels; and oxic experiments were conducted in glass reaction vessels.

A. Effect of Environment on K_d

K_d sorption coefficients given in Table 6 were measured with the following water samples:

- (1) Anoxic trench water
- (2) 1:10 dilution of anoxic trench water
- (3) Trench water exposed to air from which the $\text{Fe}(\text{OH})_3$ precipitate was filtered out.
- (4) Deionized water (oxic)

Sorption of carrier free radionuclides from deionized water ($\sim 5 \times 10^{-4}$ M HCl) was very large. The K_d is reported as greater than the measured value due to the low residual activity in the water phase, and because of possible contribution of counts from the soil, as discussed earlier. A more accurate measurement is obtained by counting the separated water phase for a longer time. Radionuclides in anoxic trench water showed the least sorption on soil. The K_d value from a 1:10 dilution of trench water is intermediate between trench water and deionized water. One possible explanation is that the trench water contains dissolved components which are in competition with the soil for the radionuclide. Diluted trench water will have less of these components, which may be complexing agents or other organic species identified in trench waters from the disposal sites. (7)

B. Effect of Water/Soil Ratio on K_d

When varying amounts of soil were equilibrated with a fixed volume of anoxic trench water for 18 hours, the K_d values were not constant. The change in K_d versus water/soil ratio for ^{60}Co , ^{85}Sr , and $^{134,137}\text{Cs}$

are shown in Figures 14, 15, and 16, respectively. The straight line logarithmic relationship between K_d and water/soil ratio resembles the Freundlich adsorption isotherm for sorption of solutes from solution onto solids. No explanation is given at this time for the exceptional behavior of ^{60}Co in deionized water (Figure 14). The K_d 's for ^{60}Co and $^{134,137}\text{Cs}$ from 1:10 diluted anoxic trench water are shown for a water/soil ratio of 20. K_d values for ^{85}Sr as a function of water/soil ratio with anoxic trench water were not obtained in this series.

C. Variation of K_d with Solution Acidity

When small amounts of acid or base were added to a trench water-soil mixture, the pH remained constant at 6.4, indicating a highly buffered system. However, under the same conditions the K_d of ^{60}Co , ^{85}Sr , and $^{134,137}\text{Cs}$ did change as shown in Figure 17. It is evident that 5×10^{-4} mmoles/ml of H^+ in the spike solution is sufficient to significantly affect the K_d . Consequently, spike solutions are now prepared so that a maximum of 1×10^{-4} mmoles/ml of H^+ is added to trench water.

502 295

IV. CONCLUSION

Sorption studies for predicting the migration of radionuclides in the geophysical environment should be performed under anoxic conditions if data that are representative of trench conditions are to be obtained. Unweathered till from the West Valley, New York, disposal site is a strong adsorber of radionuclides. There may be dissolved components in the trench water competing with the soil for radionuclides. Although changes in trench water acidity may not appreciably affect the pH, it can be significant in the sorption of radionuclides.

Finally, our preliminary results do not indicate a limiting value of K_d with decreasing water to soil ratio. This seems to imply that it is necessary to obtain chromatographic K_d values from soil cores and trench water under field conditions. The batch method nevertheless provides a rapid method for determining relative K_d values of a group of radionuclides under the same experimental conditions, and for studying general properties of trench water-soil systems at the disposal sites.

502 296

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TABLE 1

Physical and Chemical Data for Trench Water WV-37 (Trench 5) Collected
from West Valley, New York, Disposal Site November 1977*

<u>Field Measurements</u>		<u>Organic Components</u>	
pH at 10.8°C	7.1	DOC(mg/l)	5560
Eh(mv)**	240		
Specific Conductance (micromhos/cm at 25°C)	7,200		

<u>Cations (mg/l)</u>		<u>Anions (mg/l)</u>	
Barium	45	Chloride	840
Calcium	300	Nitrate	< 0.1
Cesium	0.5	Nitrite	2.9
Cobalt	0.02	Silica	13
Iron	260	Sulfate	< 5
Magnesium	140		
Potassium	340		
Sodium	1600		
Strontium	3.3		

* This is a partial characterization of trench water WV37.
Additional analyses are in progress.

** Eh measured in the field against the Ag/AgCl reference electrode was
corrected to the Normal Hydrogen Electrode at 10.8°C.

502 298

TABLE 2

Mineral Composition of Unweathered Till from
West Valley, New York, Disposal Site 1975⁽⁴⁾

<u>Fraction</u>	<u>%</u>
Sand (>62 μ)	12.7
Silt (2-62 μ)	47.6
Clay (<2 μ)	39.8
	100.1

Cation Exchange Capacity: 10 \pm 4 meq/100g

<u>Clay Fraction (<2μ)*</u>	<u>Silt Fraction (<62μ)*</u>
Illite	Mica
Chlorite	Chlorite
Quartz	K-Feldspar
	Plagioclase
	Calcite
	Dolomite
	Illite

* Qualitative mineral identification from x-ray crystallography of powdered silt and clay fractions.

502 299

TABLE 3

Chemical Analysis of Unweathered Till from West Valley,
New York, Disposal Site 1975(4)

Compound*	%
SiO ₂	58.4
TiO ₂	0.86
Al ₂ O ₃	14.6
Fe ₂ O ₃	2.1
FeO	3.1
MnO	0.07
MgO	2.8
CaO	5.2
Na ₂ O	0.71
K ₂ O	3.3
H ₂ O+	3.1
CO ₂	6.4

* Powdered till was fused with lithium metaborate and dissolved in dilute nitric acid. Metal determinations were made by atomic absorption spectroscopy.

TABLE 4

Operations Sequence to Replace Air in the K_d Manifold with Nitrogen

OBJECTIVE	OPERATIONS SEQUENCE									
A. Preliminary Purge of All Lines Including Filtration Unit, K_d Manifold, and Probe Chamber with Nitrogen	TURN VALVES THROUGH ALL OPERATING MODES									
B.1 Evacuate Reaction Vessels	GFC-1 Close	GFC-2 Close	GFC-3 Close	SV Bypass	SIV Fill	RVV-1 Fill	RVV-2 Fill	RVV-3 Fill	TV-3 W-Cell VAC	Apply Vacuum
B.2 Purge Reaction Vessels with Nitrogen			GFC-3 Open First						TV-3 W-Cell VAC	N_2 Flush
C.1 Evacuate Measured Volume Chamber	GFC-1 Close	GFC-2 Close	GFC-3 Close	SV Bypass	FV Fill	SIV Fill	LFC-2 Open	TV-2 W-Cell VAC		Apply Vacuum
C.2 Purge Measured Volume Chamber with Nitrogen		GFC-2 Open First						TV-2 W-Cell VAC		N_2 Flush
D.1 Evacuate Mixing Reservoirs	GFC-1 Close	GFC-2 Close	GFC-3 Close	SV Bypass	V Fill	DV Any Cell	TV-1 Cell Waste VAC			Apply Vacuum
D.2 Purge Mixing Reservoirs with Nitrogen	GFC-1 Open First						TV-1 Cell Waste VAC			N_2 Flush

TABLE 5

Control Positions for Manipulating Trench Water in the Sample Preparation Manifold

FUNCTION	CONTROL POSITIONS										
	LFC-1	PCV	SV	GFC-1	GFC-2	FV	DV	GFC-3	SIV	LFC-2	RVV
Clear Stream	open		 bypass	open	open		waste	open		open	
Measure pH, Eh, Temperature	open		 bypass	open	open		waste	open		open	
Fill Mixing Reservoir	open		 fill	open	open	 fill	cell	open		open	
Mix Sample in Reservoir	close		 bypass	open	open	 fill	cell	open	 fill	open	
Fill Measured Volume Vessel	close			open	open	 empty	cell	open	 deliver	open	
Transfer Measured Volume to Reaction Vessel	close							open		close	 fill

TABLE 6

K_d Sorption Coefficient of Trench Water and Soil
from West Valley, New York, Disposal Site

Liquid Phase	Container	K_d			
		Sr-85	Cs-134	Cs-137	Co-60
Anoxic Trench Water	Teflon	11 ± 1	30 ± 4	28 ± 4	22 ± 1
	Glass	---	39 ± 2	34 ± 3	9 ± 1
Anoxic Trench Water 1:10 dilution	Teflon	8 ± 1	121 ± 3	120 ± 8	116 ± 12
Air Exposed & Filtered Trench Water	Glass	---	22 ± 1	23 ± 2	29 ± 3
Deionized Water (5×10^{-4} M HNO_3)	Glass	20	> 1800	> 1800	> 900

Trench Water: WV-37, Trench 5

Solid Phase: WV-A2, Unweathered Till

Water/Soil Ratio: 20

Equilibration Time: 18 hours

502 303

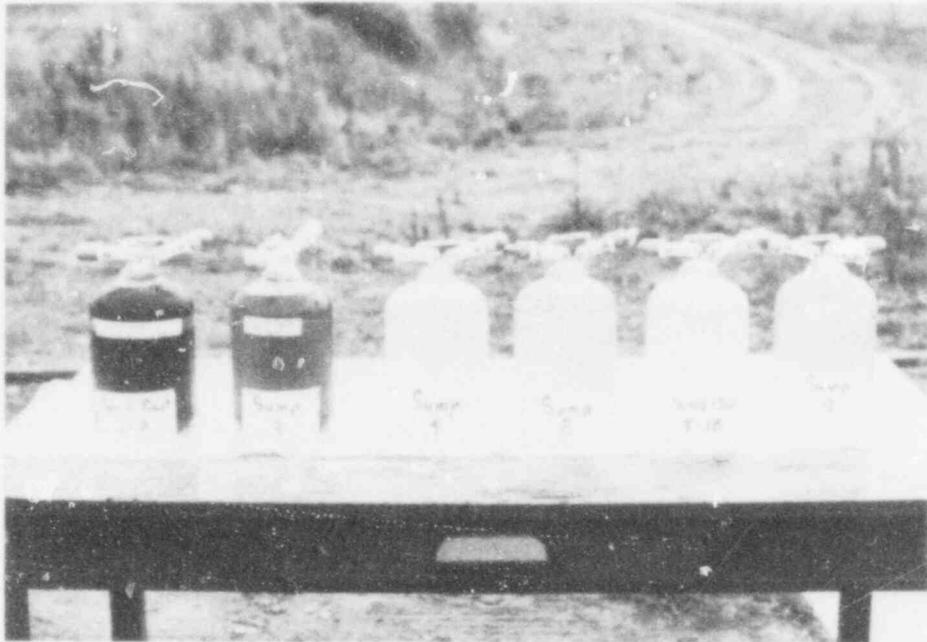


Figure 1. Trench water samples collected anoxically at the West Valley, New York, Low-Level Radioactive Waste Disposal Site, November 1977.

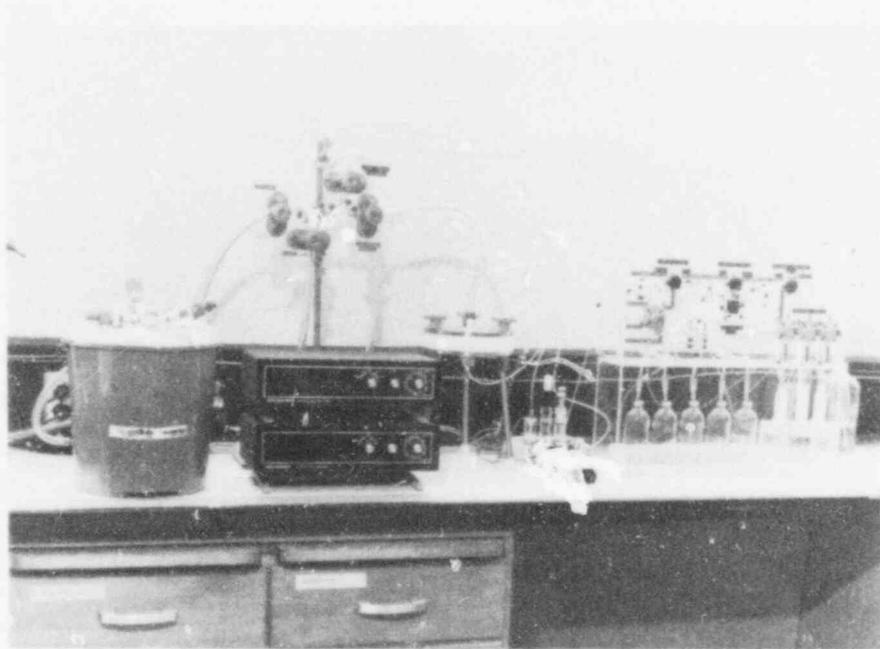


Figure 2. Equipment for anoxic K_d experiment.

502 304

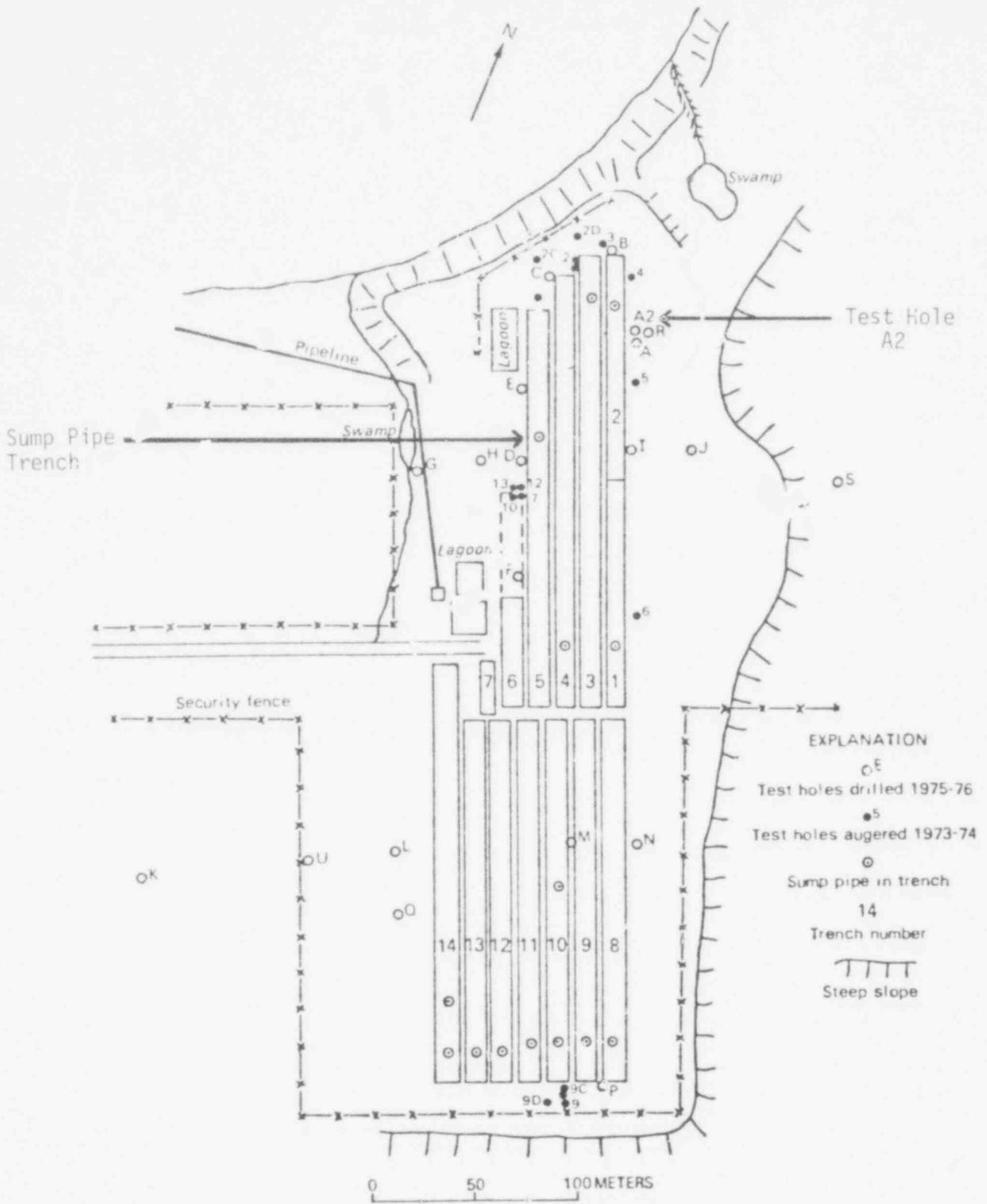


Figure 3. Sketch of West Valley Low-Level Radioactive Waste Disposal Site (from sketch by USGS)(3).

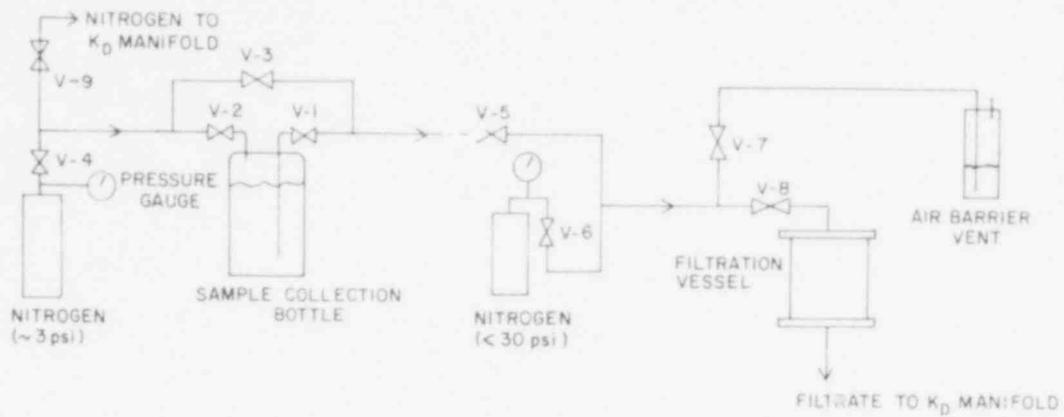


Figure 4. Schematic diagram of the filtration system for K_d manifold.

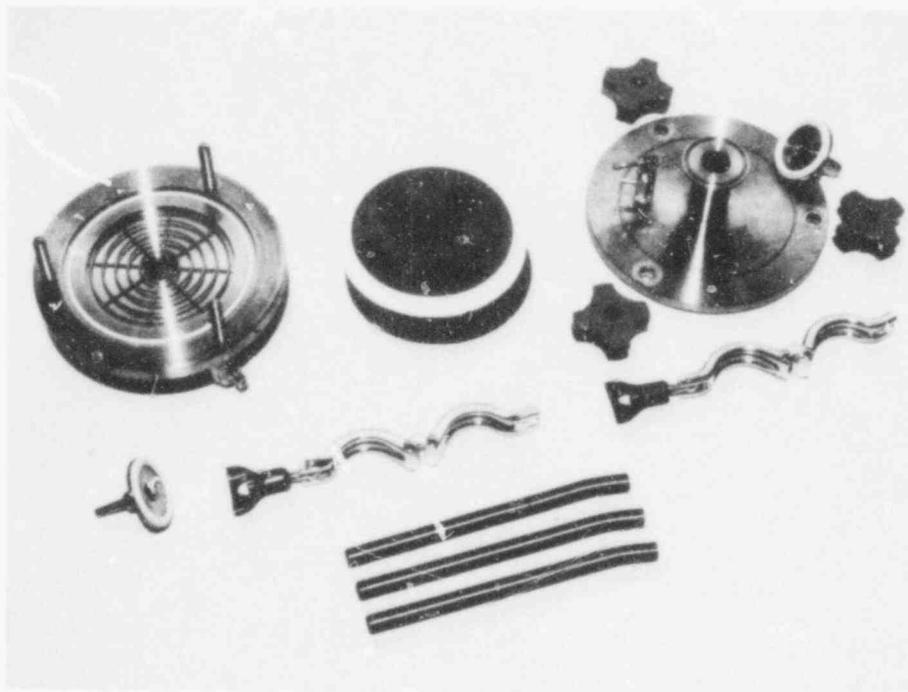
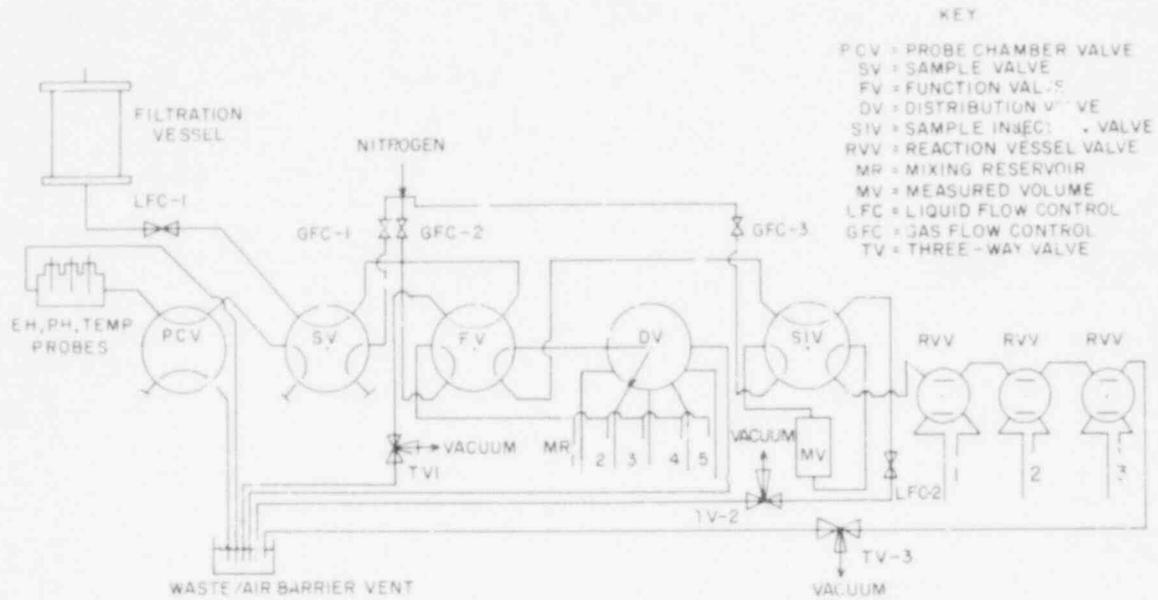


Figure 5. Breakdown of the filtration unit used in K_d experiments.

702-303



- KEY
- PCV = PROBE CHAMBER VALVE
 - SV = SAMPLE VALVE
 - FV = FUNCTION VALVE
 - DV = DISTRIBUTION VALVE
 - SIV = SAMPLE INJECTION VALVE
 - RVV = REACTION VESSEL VALVE
 - MR = MIXING RESERVOIR
 - MV = MEASURED VOLUME
 - LFC = LIQUID FLOW CONTROL
 - GFC = GAS FLOW CONTROL
 - TV = THREE-WAY VALVE

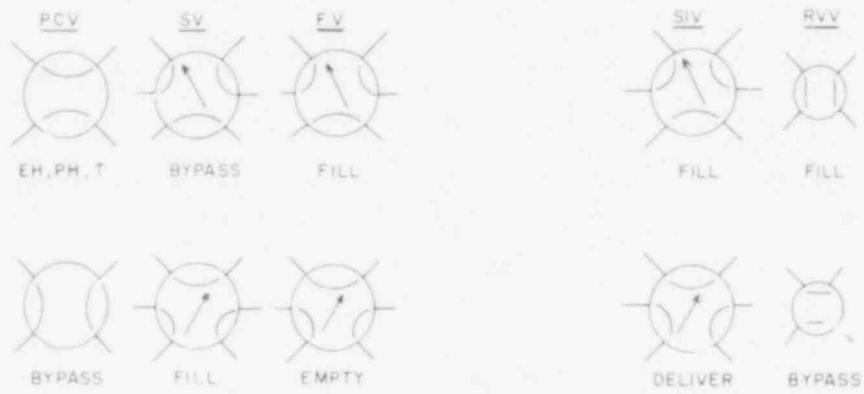


Figure 6. Schematic diagram of the sample preparation manifold for anoxic K_d experiments.

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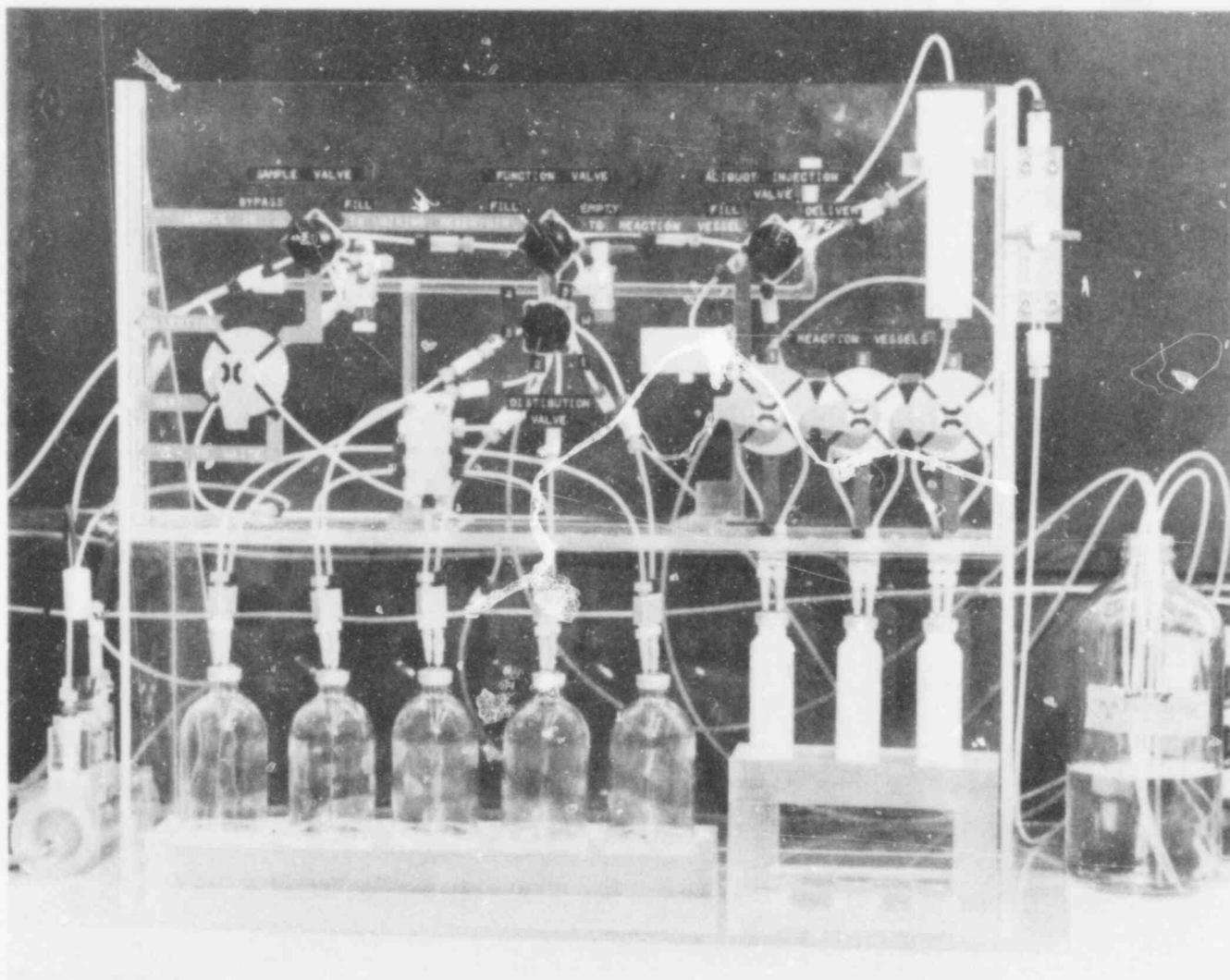


Figure 7. Sample preparation manifold.

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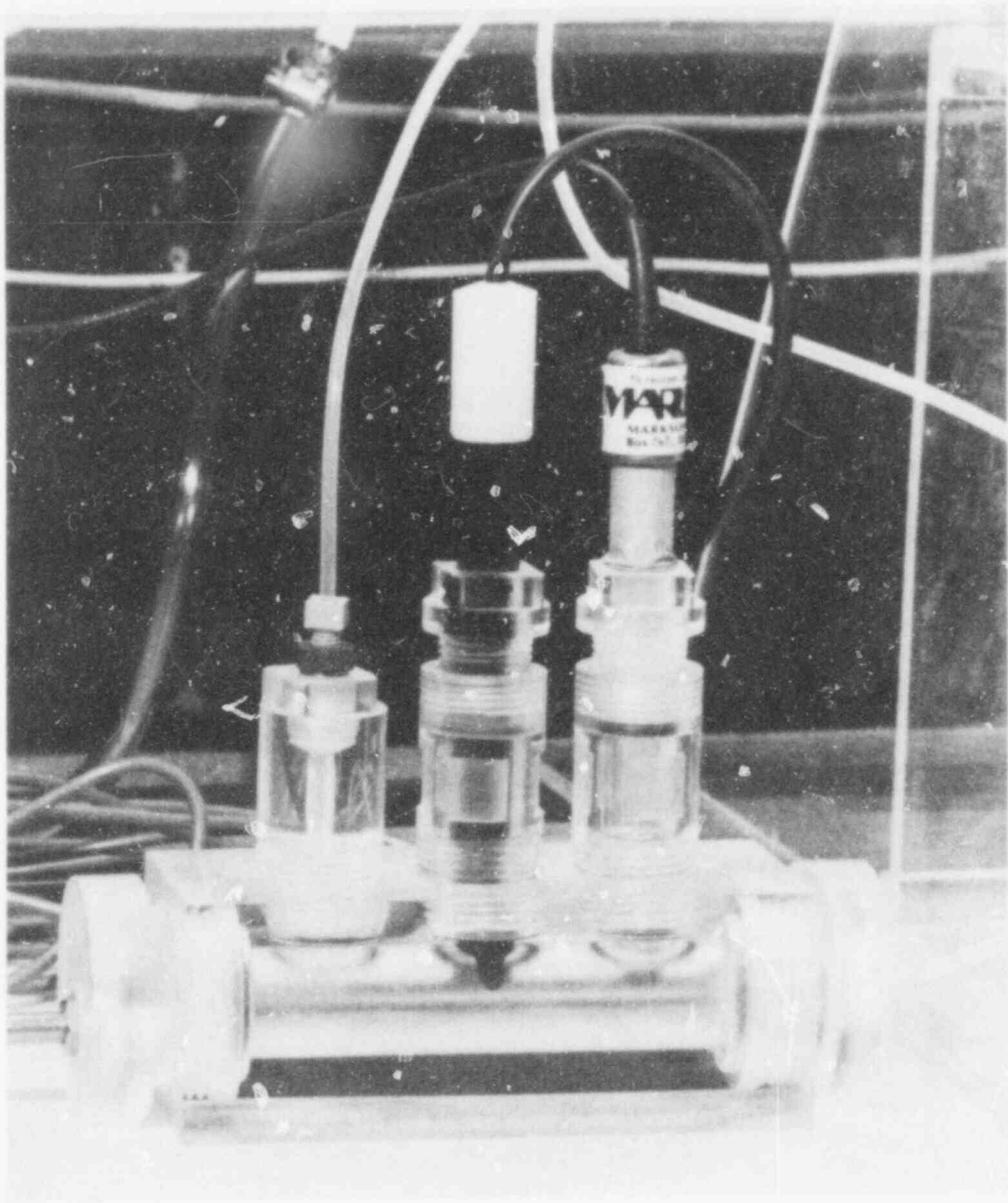


Figure 8. Probe chamber for measuring pH, Eh,
and temperature.

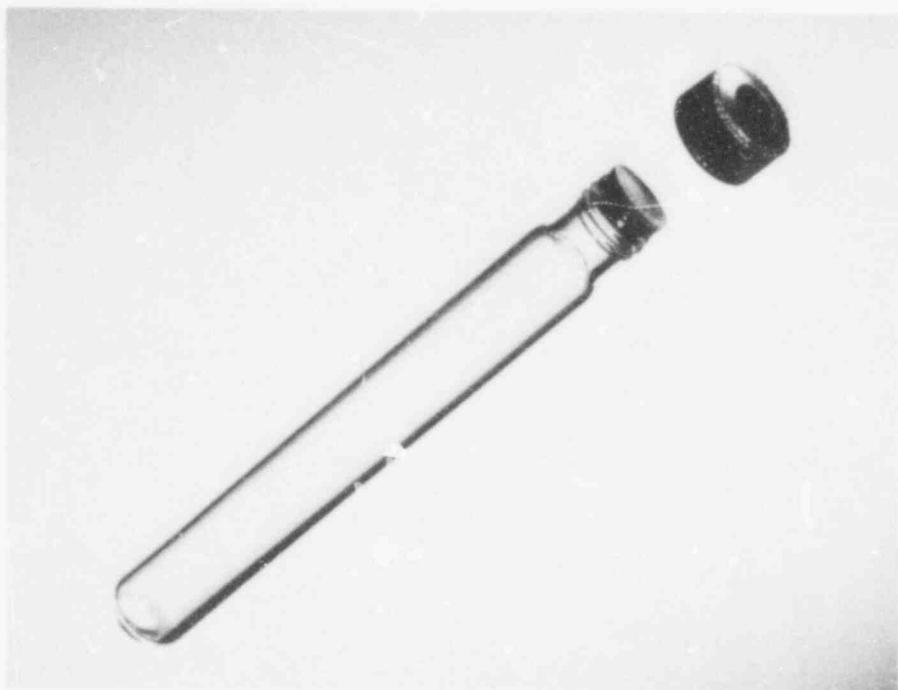


Figure 9. Glass tube with screw cap rubber septum.

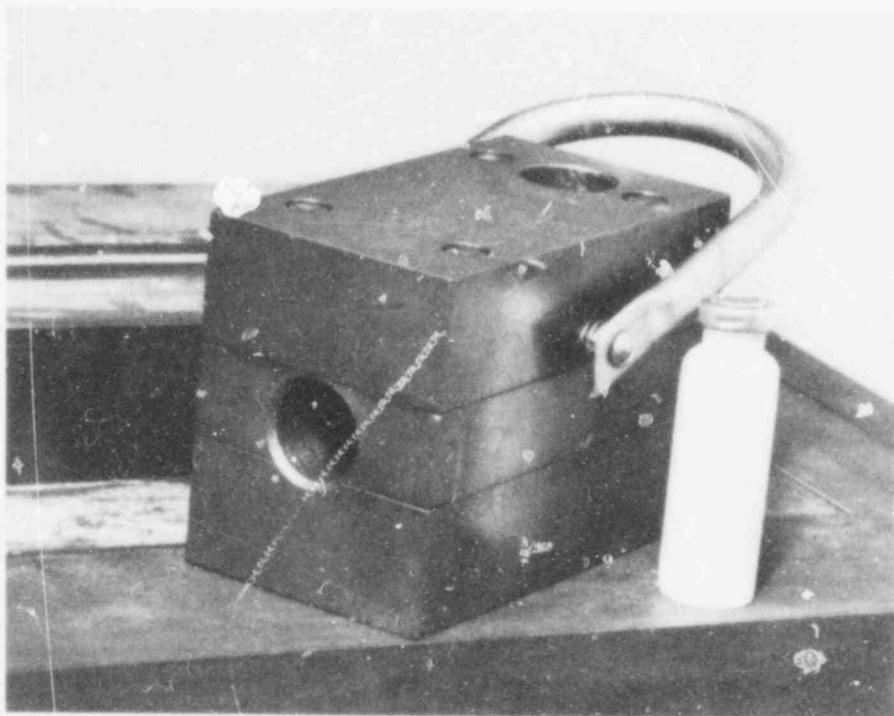


Figure 10. Teflon bottle with teflon-lined flat rubber septum and collimated counting shield.

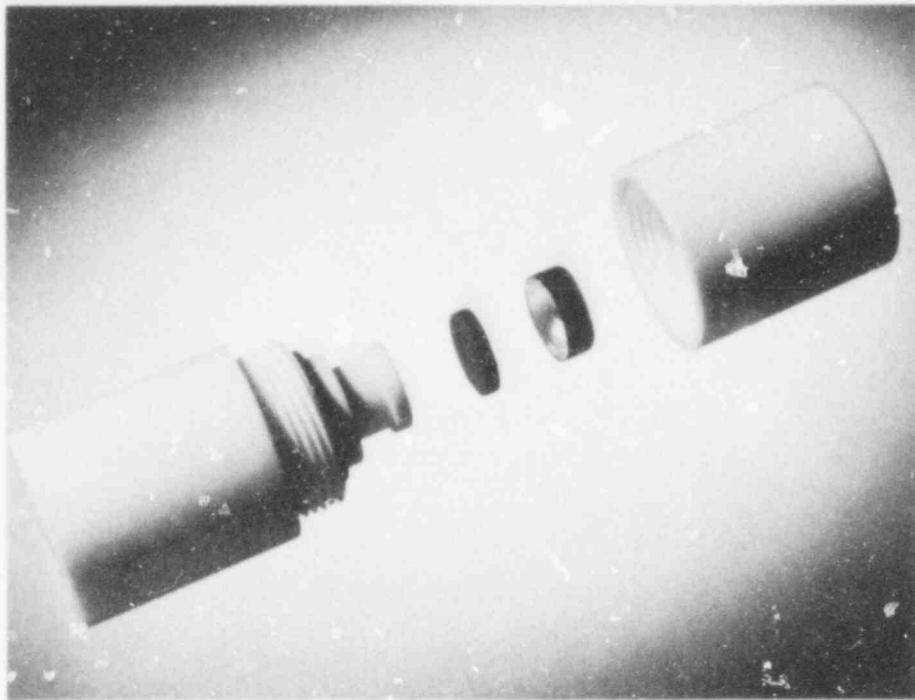


Figure 11. Teflon bottle with septum and O-ring seals.

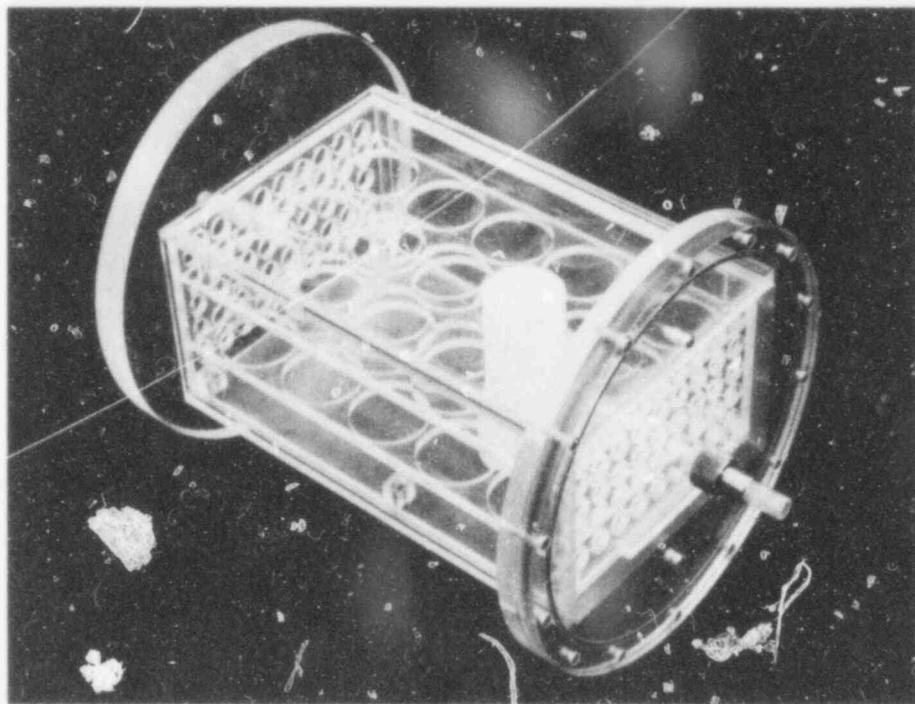


Figure 12. Sample tumbling chamber with provision for maintaining nitrogen atmosphere.

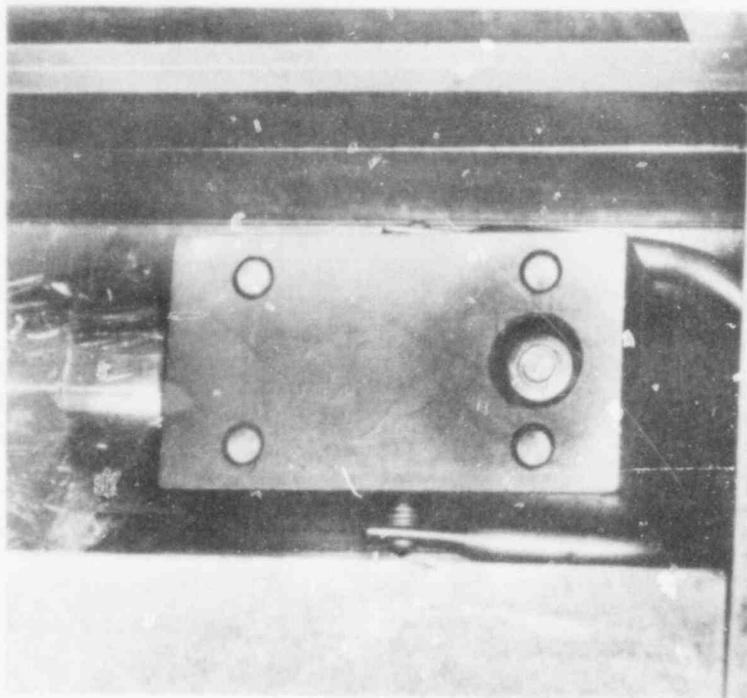


Figure 13. Collimated lead shield for counting K_d samples with Ge(Li) detector.

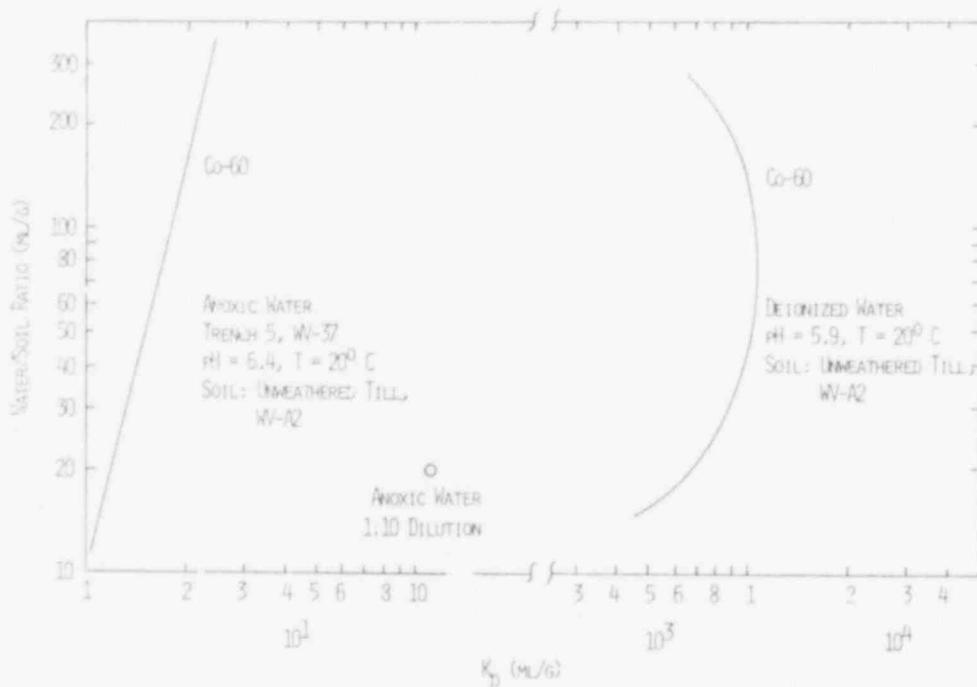


Figure 14. K_d sorption coefficient versus water/soil ratio for ^{60}Co .

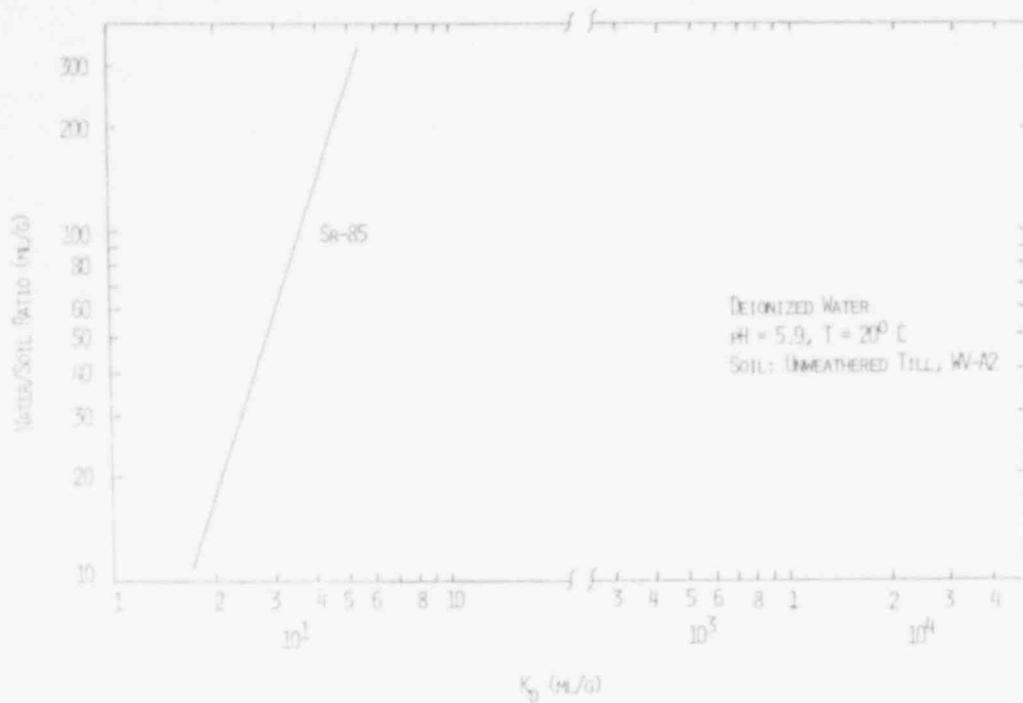


Figure 15. K_d sorption coefficient versus water/soil ratio for ^{85}Sr .

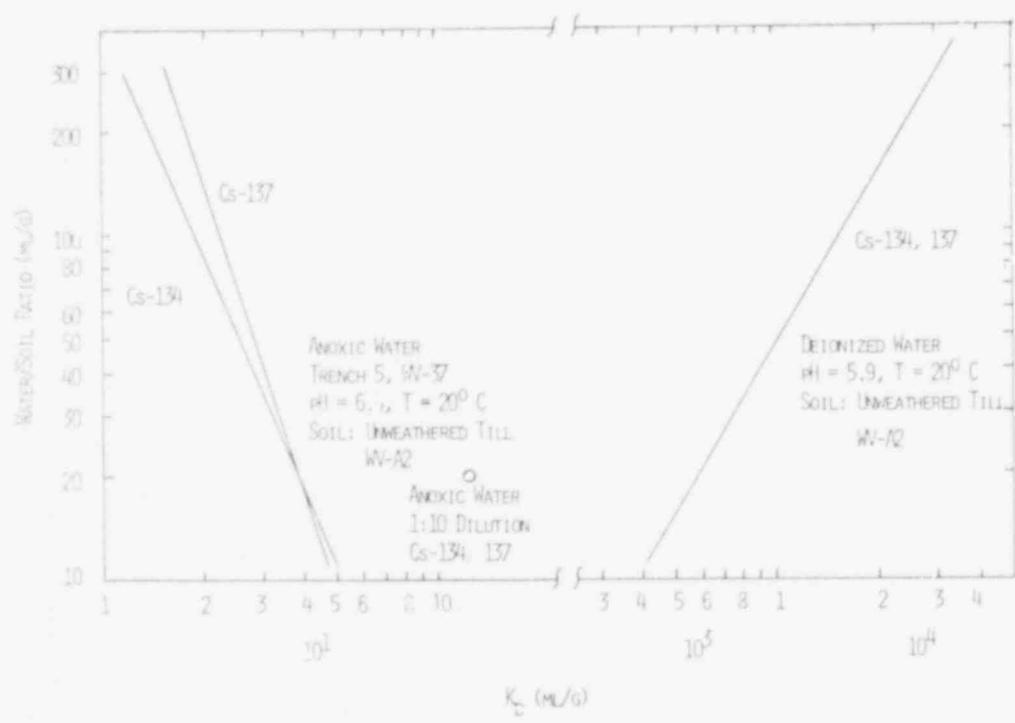


Figure 16. K_d sorption coefficient versus water/soil ratio for $^{134},^{137}\text{Cs}$.

500-13

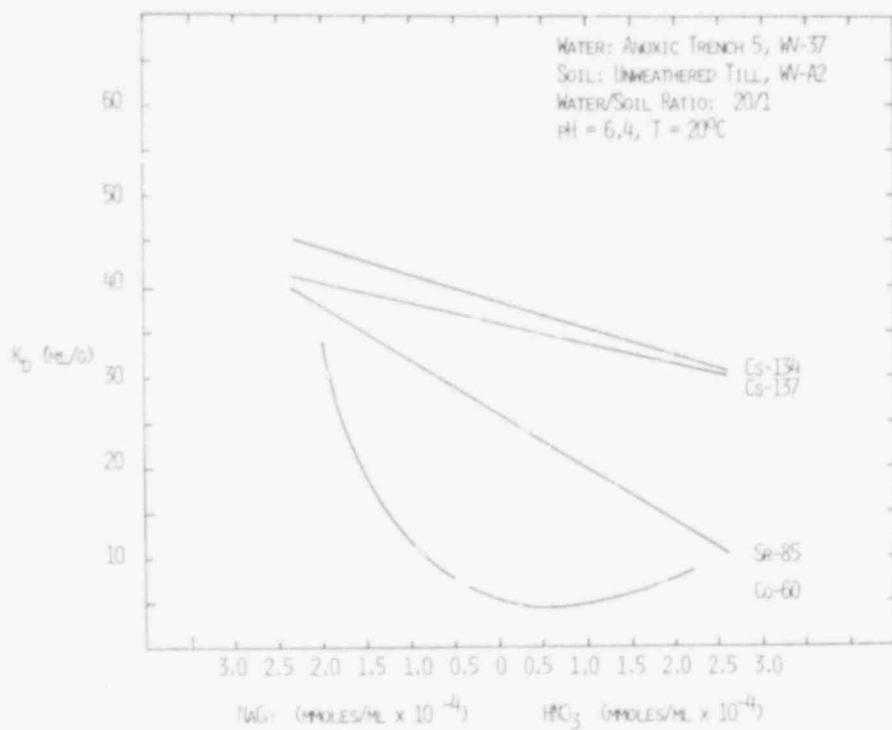


Figure 17. K_d so:ption coefficient versus addition of acid or base.

502 314

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