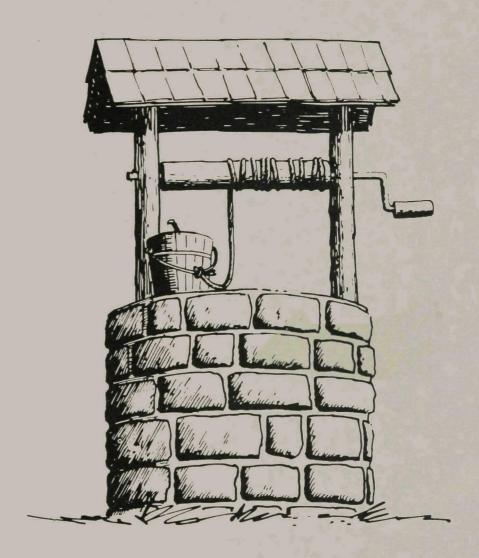


# POTENTIAL POLLUTION OF OGALLALA BY RECHARGING PLAYA LAKE WATER - PESTICIDES -





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## POTENTIAL POLLUTION OF THE OGALLALA BY RECHARGING PLAYA LAKE WATER -pesticides-

bу

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for the

ENVIRONMENTAL PROTECTION AGENCY

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## EPA Review Notice

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#### **ABSTRACT**

Twenty-four playa lakes in Lubbock County were sampled on a routine basis following runoff-producing rainfall for a period of approximately eighteen months to determine whether or not recharging of water collected in these lakes might be a hazard to the quality of water contained in the underlying Ogallala aquifer. In addition, fifteen lakes lying within a triangle bounded by Plainview, Canyon, and Hereford, Texas, were sampled during the summer of 1969 to provide additional data regarding the extent of the potential problem.

Based on results of the detailed analyses of approximately 220 samples of water collected in the lakes and an equal number of sediment samples collected from the lakes, it appears that the quality of water collected in High Plains playa lakes is generally superior to the quality of water contained in the underlying aquifer in terms of the amount of dissolved materials. The amounts of suspended solids, organic material, and microorganisms is subject to wide variation and is highly dependent upon the recent history or treatment of the drainage basin.

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#### CONCLUSIONS AND RECOMMENDATIONS

- 1. It is concluded that playa lake water is of sufficiently good quality that its recharge into the Ogallala formation is not likely to be deleterious to the quality of water in the formation.
  - It is recommended that all levels of government take immediate steps to encourage farmers to recharge playa lake water to the Ogallala by all feasible means.
- 2. It is concluded that playa lake water is generally superior to water contained in the Ogallala in terms of dissolved solids, but that from the standpoints of suspended solids and bacteriological quality, playa lake water quality is generally inferior to groundwater quality.
  - It is therefore recommended that, as recharge practices increase in the future, water quality monitoring programs be instituted to monitor the quality of playa lake water and of water collected both from recharge wells and from observation wells in the vicinity of recharge wells.
- 3. It is concluded that present farming practices in the High Plains area do not pose a significant threat to the quality of water in the Ogallala aquifer or in surface runoff from the area.
  - It is recommended that research into improved methods of recharge of playa lake water be initiated by all levels of government at the earliest possible time.

#### INTRODUCTION

A large quantity of water--estimates vary from one to three million acre-feet--is collected annually in the twenty thousand playa lakes on the High Plains of West Texas as a result of runoff from precipitation. Historically, much of this water has been evaporated to the atmosphere rather than being put to beneficial use. Recently, however, as the water table in the Ogallala formation has declined, increasing interest has been focused on finding economically feasible means of utilizing playa lake water. The direct use of this water for irrigation of the adjacent land has become fairly widespread, but utilizing the water for this purpose is inherently inefficient. When the lakes are fullest, the adjacent farm land is saturated or nearly saturated with moisture, and by the time the land requires additional water, a significant portion of the lake water has been lost to evaporation.

A more efficient method of utilizing playa lake water, and one that has been practiced to a limited extent for a number of years, is the practice of using the water for recharge of the Ogallala aquifer. Increasing concern for conservation of water by farmers and by governmental agencies in recent years has made it likely that increasing quantities of playa lake water will be used for recharge water in coming years.

The Ogallala aquifer serves as a source of supply for municipal, industrial, and domestic use, as well as for irrigation water. It is therefore essential that its usefulness for these purposes be protected.

The quantities of agricultural chemicals--insecticides, herbicides, and fertilizers--used in the High Plains area are increasing as farmers in the area adopt more efficient farming techniques. All of these chemicals are soluble to some extent in water, and any runoff from treated areas can be expected to be contaminated to some extent by them. Since practically all runoff on the High Plains is ultimately collected in playa lakes, it seems to be apparent that the water in playa lakes will generally be contaminated to some extent by these chemicals.

Because playa lake water has always been allowed to evaporate or has been utilized for irrigation of the adjacent land, and because the quality of playa lake water is adequate for use as irrigation water, very few people have been interested in determining the concentration of agricultural chemicals in the playa lake water. Thus, practically no data are available concerning the concentration of agricultural chemicals in these waters.

## Purpose

This research program was undertaken for the purpose of determining whether or not the concentrations of insecticides and herbicides in playa lake waters are sufficiently high to adversely affect the quality of water in the Ogallala aquifer if the playa lake water were recharged. Additional funds were made available from the Texas Water Quality Board to allow supplemental analyses for nitrates and phosphates.

#### Scope

Lakes selected for this study were chosen to represent the widest possible variation of test conditions. These lakes include some that receive only urban runoff, some that receive both urban and agricultural runoff and are routinely treated for mosquito control, some that receive only runoff from agricultural lands, and some that have been recently modified and that had not been inundated prior to the start of the study. Additional lakes designed for recharge and equipped with observation wells which permit samples to be taken from the aquifer at various distances from the recharge well were also included in this study.

The chlorinated hydrocarbon insecticides, especially DDT, Toxaphene, Endrin, Dieldrin, and Aldrin, were given first priority in this study because of their toxicity and proven long residual time. Conversely, the organophosphate insecticides, such as Parathion, Malathion, and Di-Syston, were not given immediate attention because of their relatively rapid decomposition.

In the herbicide field, Treflan, because of its recently greatly expanded market and stable nature, was included in analytical studies. The second most commonly used herbicide, Propazine, was given the same priority as the organophosphate pesticides. A critical problem and one that greatly complicated the research was the identification of the decomposition products of the pesticide compounds. These products are not well known and they may be as hazardous as the parent compounds.

#### MATERIALS AND METHODS

#### Lake Selection

Playa lakes sampled in this study were selected to represent the major land use patterns on playa watersheds. Categories selected were: (1) cropland; (2) pasture land; (3) urban areas; and (4) combinations of these. In addition, two lakes which had been extensively modified were selected because a new basin had been created which had never been inundated prior to the initiation of this study. Two additional lakes located in Hale County, Texas, and used for studies of recharge of the Ogallala formation were included in the sampling scheme.

Twenty-four lakes located in Lubbock County and two in Hale County were selected for intensive study which required sampling after each major rainfall period. An additional fifteen lakes were sampled once during the summer of 1969. These lakes were located in a triangle between Plainview, Hereford, and Amarillo, Texas, and they were primarily cropland watersheds.

A stratified selection technique was used to assure that the intensive study sites would be well distributed throughout Lubbock County, Figure 1. Detailed data on land use on the watershed, size of the watershed, and cropping practices are given in Table 1.

## Sampling Methods

A one-gallon sample of water and a one-gallon sample of sediment were taken from each lake on each sampling date. One-gallon, brown glass jars which had been thoroughly cleaned in a chromic acid solution and thoroughly rinsed were used for sample containers. These containers were closed with a bakelite screw cap lined with sheet aluminum foil. Samples collected for analysis were retained in a refrigerator at approximately 2° C until analyzed. Water samples were taken by compositing one-half pint samples from each of sixteen locations within a lake. A one-half pint dipper was used to obtain the sub-samples which were composited directly into the sample jar. Mud (sediment) samples penetrated approximately the top one inch of sediment below the water-sediment interface, and they were taken with the same dipper technique.

#### Pesticide Use on Watershed

Although exact quantities of pesticide and fertilizer used on each watershed could not be obtained, certain generalizations can be given. Almost all cropland is fertilized with both nitrogen and phosphorus in one of several forms. The total amount of each element applied is fairly uniform from watershed to watershed. Average application rates are eighty pounds of nitrogen and sixty pounds of

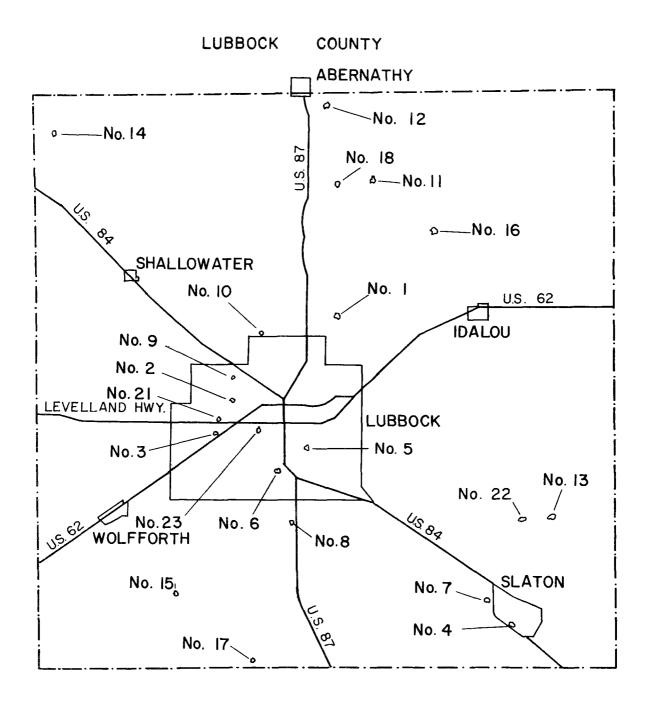


Figure I. Location of Lakes Sampled.

TABLE 1. LAKE CLASSIFICATION, WATERSHED ACREAGE AND PESTICIDE USE ON WATERSHEDS OF LAKES INCLUDED IN STUDY.

		Acres in		Wate	ershed /	Acreag	e in V	arious C	rops		Acreage Tr	eated**	Pesticide Lb/Waters	
Lake No.	Type*	Lake Bed	Cotton			Soy-		Pasture		Total	Insecti- cide	Herbi- cide		Herbi- cide
1	P-U	6	350	0	0	0	0	0	500	850	35	280	17.5	140
2	C-U	8	0	0	360	0	0	0	400	760	0	0	0	0
3	U	15	0	0	0	0	0	0	700	700	0	0	0	0
4	C-U	8	800	320	0	0	0	0	600	1720	240	896	80	448
6	U	4	0	0	0	0	0	0	800	800	0	0	0	0
7	С	4	900	900	0	0	0	0	300	2100	540	1440	157	720
8	C-U	2	100	0	320	0	0	0	1000	1420	10	80	5	40
9	C-U	6	160	160		400	0	0	800	1520	96	576	18	288
10	С	2	400	400	0	0	0	0	0	800	240	640	70	320
11	С	10	320	320	80	640	80	0	0	1340	192	1024	56	512
12	С	8	320	640	320	320	0	0	0	1600	352	1024	96	512
13	Р	6	0	0	0	0	0	1280	0	1280	0	0	0	0
14	С	15	640	640	200	400	0	0	0	1980	384	1344	112	672
15	C-P	8	800	600	0	0	0	400	0	1800	380	1120	115	560
19	С	35	200	240	0	100	0	0	0		140	432	40	216
20	С	35	500	600	100	40	0	40	0		350	912	100	456
21	Ŭ	10	0	0	0	0	0	0	1000	1000	0	0	0	0
22	С	6	320	400	200	0	0	0	300	1220	232	576	66	288
23	Ř	15	0	0	0	0	0	0	1200	1200	0	0	0	0
24	C	10	320	640	320	160	0	0	0	1440	352	896	96	448

<sup>\*</sup> P-Pasture Land, U-Urban Land, C-Crop Land, R-Urban-Residential

<sup>\*\*</sup> Acreages reported based on the assumptions that 10% of cotton, 50% of grain sorghum treated with insecticides, 80% of cotton, grain sorghum, and soybeans treated with herbicides.

<sup>\*\*\*</sup> Quantities based on application rates of 0.5 pounds of insecticide per acre of cotton, 0.25 pounds per acre of grain sorghum, 0.5 pounds of herbicide per acre of cotton, grain sorghum, and soybeans.

phosphorus per acre on cotton, and 120 pounds of nitrogen and sixty pounds of phosphorus per acre on grain sorghum. On grain sorghum, however, only about fifty percent of the acreage receives phosphorus fertilizer. Almost all urban lawns are also fertilized, mainly with nitrogen, but occasionally with some phosphorus.

Herbicides are used on seventy-five to eighty percent of the cropland in Lubbock County. Approximately three-fourths of the cotton and soybean acreage is treated with a pre-plant application of Treflan (Trifluralin). Other herbicides used on cotton not treated with Treflan are Planavin, Karmex, and Caparol. Amiben and Tenoran are used on soybean. Atrazine and Propazine are the main herbicides used on the seventy-five to eighty percent of the acreage of grain sorghum that is treated.

Insecticides are normally used on cotton and grain sorghum only in response to specific insect problems that arise. In 1969, little of the cotton acreage was treated. Some farmers treated the seed with low rates of an organophosphate systemic insecticide, either Di-Syston or Thimet. Grain sorghum, on the other hand, was subjected to severe infestations of aphids. As a result, almost all of the acres in this crop were treated at least once. Parathion or Di-Syston were the primary insecticides used, with about seventy-five percent of the crop being treated with Parathion.

#### Rainfall, Runoff and Lake Levels

When the first sampling run was made on February 11, 1969, most of the lakes selected for sampling were dry. Samples were obtained from Lakes 4, 8, and 9 only. A heavy snow of approximately ten inches in March produced a considerable amount of runoff, but Lakes 1, 7, 11, 12, 14, and 16 remained dry. The water level in Lakes 4, 8, and 9 rose considerably. A one-inch rain in April raised the levels in most of the lakes which had been sampled previously and allowed an initial sample to be taken from Lake 14.

General rains of almost four inches in the first half of May, 1969 produced runoff to all lakes except Lake No. 17. All lakes except No. 17 caught additional water following a rainfall of about one and one-half inches in the middle of June.

Three lakes had gone dry when a sampling run was made early in July, but a two and one-half inch rain on July 21 produced additional run-off to all lakes except No. 17.

Heavy general rains in September produced runoff to all lakes except Lakes 11, 15, 16, 17, and 24. These lakes lost water quickly and stayed dry most of the time.

Samples were taken again in November, 1969 following a moderate rainfall in the area. Several of the lakes were dry at this time and they remained dry throughout the winter.

Samples were obtained from five of the ten lakes that contained water following a moderate rainfall in March, 1970. The other five lakes containing water were sampled in April.

Two sampling runs were made in June, and two additional runs in July, 1970. No more than ten to twelve lakes contained water at the same time in either June or July, and the water contained in some of the lakes was believed to be derived from irrigation tailwater rather than from runoff from precipitation.

All lakes not sampled more than once either dried up after the first sample was taken or were included in the random samples taken before and during the period in which specific lakes were regularly sampled. Figure 2 indicates the period during which each lake contained water.

Lake I		1969 JFMAMJJASONDJFMAMJJASOND
2		
2 3 4		
4 5	(1)	
6	(1)	*
6 7		
8 9	(2)	
10	(-)	
  2		
13		
14		
15 16		
17	(3)	
18 19	(2) (- (2)	4)
20		
21 22	(2) (2)	
23	(2)	
24	(2)	

Figure 2. Inundation Periods for Lakes Included in the Study

- (1) Sampled only one time because not incorporated into regular sampling program.
- (2) Condition not known as of February, 1969 because not included in the study until March, 1969.
- (3) Never caught water.
- (4) Sampled only once because owner requested sampling be stopped.

## PESTICIDE ANALYTICAL PROCEDURES

Approximately 220 samples each of water and sediments were analyzed in performing this research. In general, very low concentrations of pesticides were found in sediments, and water samples were found to be free of concentrations detectable with the equipment used.

The analytical procedures used generally consisted of three separate parts:

- 1. The extraction of pesticides from the water or soil, both of which may contain or have previously contained plants.
- 2. Cleanup or separation of the pesticide residues from the extract.
- 3. Identification and quantitative determination of the concentration of pesticides.

The extraction and cleanup procedures are usually the limiting and time-consuming factors when there is organic contamination of the samples. Most of the contamination found is thought to be caused by living or decomposing plant material contained in the water and sediments.

After extraction and cleanup as required, all samples were analyzed on a Varian Aerograph Model 600 C Gas Chromatograph equipped with a Tritium Electron Capture Detector and a Leeds and Northrup Speedomax H l millivolt recorder. A schematic diagram of the system is shown in Figure 3.

Detection of a particular pesticide in an unknown sample requires that the response of the instrument to that sample be compared to the response of the instrument to a known standard pesticide. Provided all variables such as gas flow rate, temperature, electronic variables, type of absorbent column, etc. remain constant, a particular compound passes through the chromatograph at a particular time.

Since all variables involved could not be held absolutely constant from day to day, a procedure was adopted to minimize errors resulting from uncontrolled variables. The procedure adopted was based on the fact that, while the absolute retention time of different compounds may vary from day to dáy as test conditions vary slightly, the relative retention time of all compounds will remain constant under any particular set of test conditions. The instrument was therefore calibrated each day with hexane solution containing a standard concentration of Aldrin, and all other peaks observed were recorded in terms of their retention times relative to Aldrin. Thus,

 $RTA = \frac{Time \ unknown \ peak \ comes \ off}{Time \ Aldrin \ peak \ comes \ off}$ 

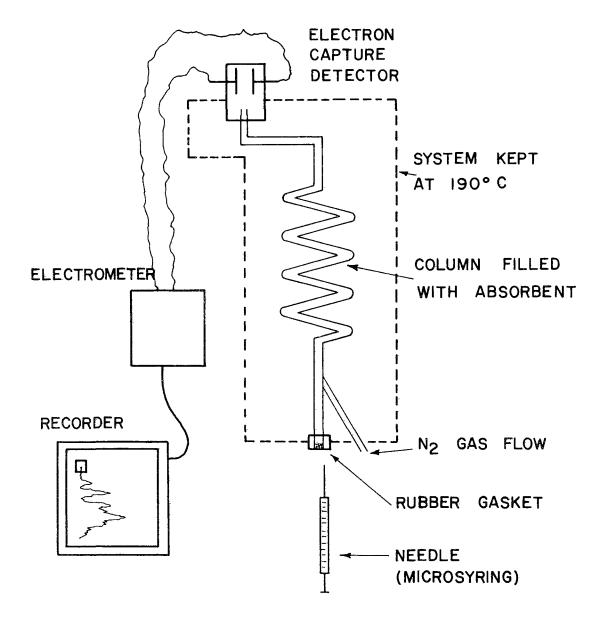


Figure 3. Schematic Diagram of Gas Chromatograph.

Hence, the relative retention time for Aldrin is 1.00. Substances with longer retention times than Aldrin have RTA's greater than 1.00, and substances with shorter stays in the chromatographic column have RTA's less than 1.00. Relative retention times of several pesticides and of some unidentified compounds are shown in Table 2 along with standard deviations as determined over a period of approximately five months.

A typical chromatogram of a sediment extract is shown in Figure 4. Figure 5 is a chromatogram obtained from a sample containing pure compounds as indicated.

Compounds were occasionally detected with an RTA very close to a known standard. In order to determine whether or not the compound was, in fact, the standard compound, a mixture of the standard and the unknown was introduced into the chromatograph. A larger, smooth curve across the unknown then indicated that the unknown and the standard were the same compound, while a slight difference in RTA's was indicated by a shoulder on the curves as suggested by the values of 1.00 to 1.07 in Figure 4.

It is possible that a compound could have an RTA exactly equal to the standards used, yet be a different compound. The RTA's used were dependent on the specific absorbents and other conditions in the separation and in the system. While all reasonable precautions were taken to avoid the possibility of such an error, instrumentation available did not permit absolute confirmatory tests to be made.

## Analytical Procedure for Water

The determination of pesticide concentrations in water was a fairly straightforward process usually involving no cleanup procedure. The extraction procedure used required that 600 ml of water and 50 ml of n-Hexane be agitated by magnetic stirring for twenty minutes in a 1000 ml Erlenmeyer flask at a rate that resulted in the formation of n-Hexane droplets in the water, but not in emulsification of the mixture. The mixture was then poured into a 1000 ml separatory funnel and allowed to separate into two layers. After separation, the lower water layer was discarded. Tests showed that a single extraction by this method removed 99%+ of the pesticides present in the water, thus eliminating further time-consuming extractions of the sample. The hexane layer was separated and reduced in volume by evaporation at room temperature, after being passed through a small column containing powdered anhydrous sodium sulfate on a glass wool plug. The column had been rinsed previously with n-Hexane. The elutant was collected in a 50 ml amber bottle with a foil-lined screw cap.

Most samples were then ready for immediate injection into the gas chromatograph. However, if organic contamination was found to be present, the sample was put through a Florisil cleanup procedure which will be described later.

TABLE 2. RETENTION TIMES FOR THE NON-POLAR COLUMN

Pesticide Standards	Retention Times Minutes	Relative Retention Times (To Aldrin)	Relative Retention Times Samples (To Aldrin)	Accepted Retention Times 5% Dow II on Chromosorb W
Lindane	1.3 + 0.2	0.44 ± .01		.44
Heptachlor	2.4 <sup>±</sup> 0.4	0.78 + .01		.80
Aldrin	3.0 - 0.4	1.00 ± .00	1.00	1.00
			1.07* 1.50*	_
Dieldrin	6.2 + 0.3	2.00 ± .03	2.00	1.94
			2.15*	_
Endrin	6.6 + 1.1	2.19 ± .05		2.18
			2.50*	
pp' DDT	10.0 + 1.8	3.28 + 0.11	3.29	

<sup>\*</sup>Unidentified peaks.

Experimental values reported were calculated from chromatograms which were run over a  $\underline{\mathbf{5}}$  month period.

## Column Conditions

Support: Chromosorb W 60/80 mesh

Coating: 5% Dow II
Length: 6 ft.
Diameter: 1/8" I.D.

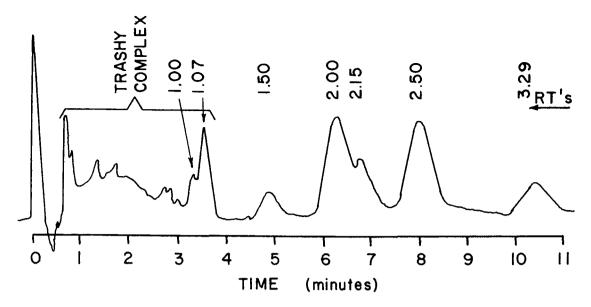


Figure 4. Typical Chromatogram Obtained from Soil Extract.

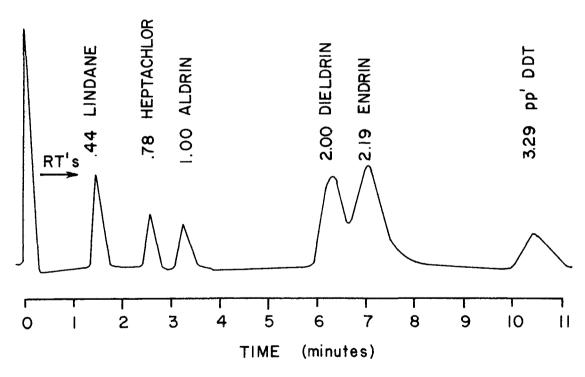


Figure 5. Chromatogram Obtained from Analysis of Pure Compounds as Indicated.

In the early testing stages of the program, the hexane solutions were transferred and diluted to 25~ml. Later, the analytical procedures for the lake waters were changed so that more hexane was recovered in the extraction and cleanup procedure and made up to smaller final volumes. The detectable range for each pesticide was established by the introduction of known quantities of the pesticide in a series of duplicated water samples, so that the range of linear response of the chromatograph could be established, the lower detectable limit shown, and the effectiveness of the extraction process demonstrated. The introduction of 1  $\mu g$  of pesticide into the 600 ml water sample was approximately the least quantity detectable with certainty above the various high trash backgrounds and corresponds to a pesticide concentration of 1.6 ppb. Non-trashy water samples allowed this detection limit to be reduced ten fold to the 0.1 ppb limit.

Recoveries of 95% - 100% ± 3% have been obtained using the method described above. Pesticides were usually detected in the water as trace quantities only at the beginning of the mosquito season when spraying for insects was most intensive. Even then, pesticides were normally found in trace concentrations only if the rainfall had been light enough that extensive dilution had not occurred.

The main problem encountered in extraction of water samples was the problem of emulsification during the extraction process. This problem was most pronounced during the spring and early summer months when there was a profuse growth of plants in the watersheds because of rainfall. This problem was less severe in samples taken during dry weather, and it disappeared completely during the winter months. It was therefore concluded that the "trashy" or contaminated chromatograms obtained when cleanup was omitted resulted from organic byproducts of plants. The Florisil procedure described later did not always remove this contamination. A solution to this problem is discussed under the analytical procedure for sediment.

#### Analytical Procedure for Sediment

The extraction of soil samples was more difficult than was the extraction of water samples. It was also more productive in terms of number and amounts of pesticides detected.

Three methods of extraction were tested as follows:

- A 25 gm sample of sediment saturated with water was stirred in a 125 ml Erlenmeyer flask for twenty minutes with 50 ml n-Hexane.
- 2. A 15 gm sample of sediment that had been air-dried and ground to a fine powder was extracted by allowing 25 ml of n-Hexane to gravity filter through the sample.

3. A 25 gm sample of sediment that had been air-dried and ground to a fine powder was extracted by magnetic stirring for twenty minutes with 50 ml of n-Hexane in a 125 ml Erlenmeyer flask.

In all cases, the n-Hexane was collected and passed through a small column containing powdered anhydrous sodium sulfate. The extract was then ready for analysis on the gas chromatograph if it was free of interfering organic contaminants.

When the sample was found to contain organic interferences, it was run through a Florisil cleanup process. In this process, the sample extract was concentrated to approximately 10 ml by evaporation over a 70° C water bath equipped with an aspirated air stream to pull off vapors and speed up evaporation. A 15 gm charge of activated Florisil was placed in a 5/16 I.D. column over a glass wool plug topped by 1/2 in. of anhydrous sodium sulfate. After the Florisil was tapped in place in the column, an additional 1/2 in. of anhydrous sodium sulfate was added to the top of the column. After cooling, the column was pre-eluted with 30 ml n-Hexane and the pre-elutant was The sample extract was transferred to the column just before the top layer of anhydrous sodium sulfate was exposed to air. The extract was followed with 50 ml n-Hexane and the total volume of elutant was collected and evaporated over a 70° C water bath to a volume of approximately 5 ml. The concentrated extract was then diluted back to the pre-Florisil treatment volume and injected into the gas chromatograph.

In extraction method (1), the presence of water tended to produce emulsions which presented a barrier to the passage of pesticides from the soil to the Hexane. The emulsions did not break up upon standing nor could they be destroyed by centrifuging. Recoveries by this method were therefore very low, and it was rejected as an unacceptable procedure.

Of the last two methods, the former gave slightly better recoveries of pesticides. However, since method (2) required more time for gravity filtration and precise collection of the first 25 ml of n-Hexane passing through the filter, method (3) was used for most of the work. Recoveries by method (3) are comparable to those obtained using method (2). Recoveries of  $84\% \pm 7\%$  to  $35\% \pm 4\%$  were obtained, depending upon the type of pesticide and the texture of the soil.

An additional problem was encountered when the Florisil cleanup procedure failed to remove all of the interferences present in some of the soil samples. In these cases, extracts produced chromatograms with large contamination peaks that were able to mask pesticides present. A recent publication in the <u>Journal Analytical Chemistry</u> 42-2, p. 282, 1970, described a procedure which was being tested. Preliminary results indicated it would be useful in removing many of the organic contaminants before the extract is put through the Florisil cleanup.

Concentrations of pesticides in sediment samples which were interference-free generally exceeded the concentrations found in water samples by a factor of 100 to 1000. Most pesticide concentrations detected in sediment samples range from .01 to 1 ppm. Three sediment samples taken from two lakes in the City of Lubbock contained Dieldrin in concentrations of 1.06, 1.87, and 2.82 ppm.

## Analytical Difficulties

From the beginning, extraction of muds and soils was a problem because of the appearance of trashy substances in the Hexane phase. The typical range of most of the trouble encountered is illustrated in Figure 4. Often the trashy complex was much more intense, producing a record such as that shown in Figure 6.

Various cleanup procedures were tried using such absorbents as Florisil, Attaclay, and Norit. These procedures were frequently unsuccessful, since the absorbents removed pesticides as well as the trashy substances. A cleanup method that removed the trashy complex without also removing pesticides was finally found. This method is as follows:

- 1. Pipet a 10 ml aliquot of trashy Hexane into a 125 ml separatory funnel.
- 2. Add 10 ml saturated KOH solution of absolute ethanol to the separatory funnel; shake for two minutes.
- 3. Leach the ethanol-KOH out of the Hexane with 20 ml distilled water, shaking for about one minute. Allow phases to separate.
- 4. Remove and discard water phase from separatory funnel. Repeat procedure in step 3 above three times, or until Hexane layer is optically clear.
- 5. Add a small quantity of powdered anhydrous Na<sub>2</sub>SO<sub>4</sub> to remove any water in contact with Hexane.
- 6. The Hexane solution is now ready for analysis on the gas chromatograph.

This procedure made possible quantitative detection of Aldrin with an RTA within the typical trashy range shown in Figure 6 (note dotted line result of cleanup where Aldrin is present). This procedure was found to remove the Lindane and p, p-DDT, and to reduce the concentrations of Treflan. However, since it did not affect Aldrin, Heptachlor, Dieldrin, or Endrin, it was used in conjunction with these pesticides. Other trashy complexes appeared occasionally at higher retention times, and certain water samples also contained these same interfering substances.

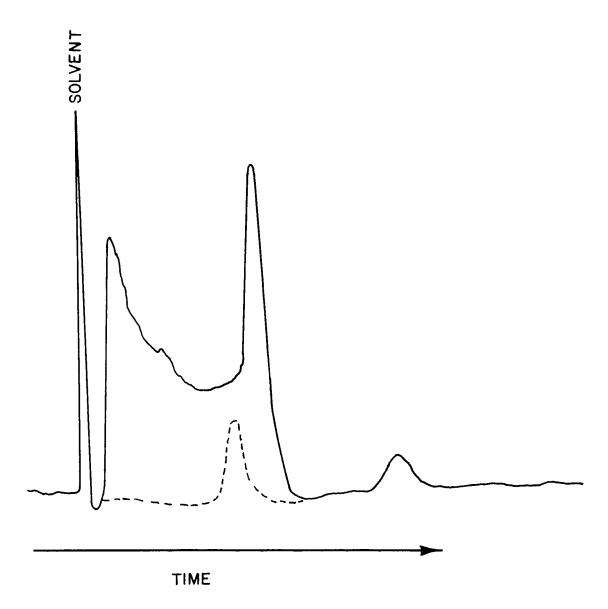


Figure 6. Chromatogram of Sample Contaminated by High Concentration of "Trashy Complex".

One procedure was found to be successful in confirmation of apparent pesticides. This procedure was used in conjunction with the gas chromatograph. It involves extraction p-values (Analytical Chemistry, 37, 2, Feb. 1965), where p-values are defined as follows:

p-value = Amount of pesticide in upper phase (2nd analysis)

Total amount of pesticide (1st analysis)

This method is based on the distribution of the pesticide between two immiscible phases. A given pesticide will have its own specific p-value for the same phase system, i.e., its own distribution ratio. Thus a suspected pesticide can be checked by determining whether its p-value is the same as that of a known standard pesticide.

#### RESULTS AND INTERPRETATIONS

None of the water samples analyzed contained measurable concentrations of any of the herbicides or insecticides commonly used in the area.

Aldrin, Dieldrin, and DDT were the only insecticides found in sediment samples in the lakes, and no measurable concentrations of herbicides were found in any sediment samples. Measurable concentrations of Dieldrin were found in the sediments in about eighty percent of the lakes. Aldrin was found to be detectable in sediments in less than ten percent of the lakes, and, surprisingly, DDT was present in detectable concentrations in only three of the samples analyzed. These results are shown in detail in Table 3.

As noted earlier, all sediment samples were obtained from the top one inch of sediment in the lakes. Because of the generally negative results obtained for analyses of sediment samples taken in this manner, it was decided to obtain core samples from a few lakes to determine whether or not pesticides had been carried deeper into lake sediments by percolation of water over a period of several years.

Core samples were therefore taken from the bottoms of four lakes that were included in the study, with two of the cores being taken from lakes at which mosquito control programs had been in operation for several years, and the other two being in farming areas not subject to mosquito control programs. Core samples were taken at one-inch increments from the sediment surface to a depth of twelve inches. Each sample obtained was analyzed in the same manner as were other sediment samples.

A substantial difference was found in the four lakes cored. Although Dieldrin was found in all four lakes and DDT was found in the sediments in one of them, the two rural lakes contained low concentrations of Dieldrin that remained fairly constant in the twelve samples analyzed. The two urban lakes, however, contained higher concentrations of Dieldrin that apparently varied with the depth of the samples. Although the concentration of Dieldrin varied in Lakes 2 and 3 for the first four to five inches, a definite trend was indicated later, Figures 7 and 8. Starting with the five inch sample on both lakes, there was a steady decrease in pesticide concentration until the seven to eight inch level was reached. After the lowest concentration was reached at the eight inch level, a sharp increase in concentration values throughout the remaining four inches was indicated. This increase may be an indication of heavier treatments for mosquito control in past years.

The Lubbock City-County Health Department has indicated that, from 1956 through 1962, Dieldrin was sprayed on lakes within the City for mosquito control during the summer with concentrations of one-half

TABLE 3. INSECTICIDE CONCENTRATION IN MUD SAMPLES

N.D. = 1	Not detectable				<del></del>	
Lake Number	Location	Number of Sample	Date	Apparent Aldrin PPM	Apparent Dieldrin PPM	Apparent DDT PPM
1	Airport	20 145 213	3/14/69 6/20/69 7/22/69	N.D. N.D. Trace	.11 .25 .33	N.D. N.D. .49
2	4th & Quaker	23 43 49 77 251 331 351	3/14/69 3/25/69 4/12/69 5/ 6/69 9/19/69 5/14/70 6/ 2/70	.11 N.D. .022 .087 .031 .085 N.D.	1.87 .23 .11 .76 .42 .034	N.D. N.D. N.D. N.D. N.D. N.D.
3	24th & Vicksburg	24 27 45 51 79 105 117 249 289	3/14/69 3/18/69 3/25/69 4/12/69 5/ 6/69 5/29/69 6/17/69 9/19/69 11/28/69	.034 .0092 .029 .072 .038 .053 N.D. N.D.	.27 .27 1.06 2.82 .21 .19 .27 .07	N.D. N.D. .11 .38 N.D. N.D. N.D. N.D.
4	Old Slaton	12A 18A 33 53 67 99 137 179 225 239 265 315 335 363	12/23/68 2/11/69 3/18/69 4/12/69 5/ 3/69 5/28/69 6/18/69 7/10/69 7/23/69 9/15/69 11/22/69 4/23/70 5/15/70 6/ 5/70	N.D19 N.D. N.D. N.D. N.D. N.D. N.D. N.D. N.D	.08 .11 .06 .01 .04 .05 .16 .065 .067 .065 .008 Trace Trace	N.D. N.D. N.D. N.D. N.D. N.D. N.D. N.D.
5	50th & Avenue A	12	12/23/68	N.D.	.034	N.D.
6	66th & University	8 47 55	12/23/68 12/23/68 4/12/69	N.D. N.D. .015	.047 .27 .27	N.D. N.D. N.D.

TABLE 3. INSECTICIDE CONCENTRATION IN MUD SAMPLES - Continued
N.D. = Not Detectable

Lake Number	Location	Number of Sample	Date	Apparent Aldrin PPM	Apparent Dieldrin PPM	Apparent DDT PPM
		81 109 125 195 217 245 293 301 323 359	5/ 6/69 5/30/69 6/17/69 7/12/69 7/23/69 9/19/69 11/28/69 3/17/70 5/13/70 6/ 2/70	N.D. N.D. N.D. N.D. N.D. N.D. N.D.	.039 .32 .10 .11 .010 .016 .029 .016 .032 .038	N.D. N.D. N.D. N.D. N.D. N.D. N.D. N.D.
7	New Slaton	65 95 135 177 227 237 263 297 333	5/ 3/69 5/28/69 6/18/69 7/10/69 7/23/69 9/15/69 11/22/69 3/17/70 5/15/70	N.D. N.D. N.D. N.D. N.D. N.D. N.D.	N.D. .054 N.D. .030 .067 .061 .007 N.D.	N.D. N.D. N.D. N.D. N.D. N.D. N.D.
8	Strip Lake	16 31 57 83 107 119 219 255 271 299 337 361	2/11/69 3/18/69 4/12/69 5/ 6/69 5/30/69 6/17/69 7/22/69 9/19/69 11/26/69 3/17/70 5/15/70 6/ 5/70	N.D. N.D. N.D. N.D. N.D. N.D. N.D. N.D.	.088 .33 .39 .098 .050 .16 .053 .024 .050 .055 .053	N.D. N.D. N.D. N.D. N.D. N.D. N.D. N.D.
9	Loop 289 & N. Quaker	29 73 123 201 209 241 291	3/18/69 5/ 6/69 6/17/69 7/16/70 7/22/69 9/19/69 11/28/69	N.D. N.D. N.D. N.D. N.D. N.D.	.083 .15 .29 .14 .30 .11	N.D. N.D. N.D. N.D. N.D. N.D.

TABLE 3. INSECTICIDE CONCENTRATION IN MUD SAMPLES - Continued

N.D. = Not Detectable

Lake Number	Location	Number of Sample	Date	Apparent Aldrin PPM	Apparent Dieldrin PPM	Apparent DDT PPM
		305 339 <b>3</b> 49	4/ 4/70 5/15/70 6/ 2/70	N.D. N.D. N.D.	.065 .121 .062	N.D. N.D. N.D.
10	North University	2 14 35 61 75 91 115 203 211 253 279 309 325 347	12/23/69 2/11/69 3/18/69 4/12/69 5/ 6/69 5/28/69 6/17/69 7/16/69 7/22/69 9/19/69 11/17/69 4/23/70 5/14/70 6/ 2/70	N.D. N.D. N.D. N.D. N.D. N.D. N.D. N.D.	.27 .16 .11 .14 .21 .15 .052 .082 .058 .12 .030 .056 .032	N.D. N.D. N.D. N.D. N.D. N.D. N.D. N.D.
11	Huddleston	21 101 121 191 235A 283 311 327 343	3/14/69 5/30/69 6/18/69 7/11/69 7/24/69 11/28/69 4/23/70 5/14/70 6/ 2/70	N.D. N.D. N.D. N.D. N.D. N.D. N.D.	.53 .15 .034 .061 .26 .075 .15 .027 Trace	N.D. N.D. N.D. N.D. N.D. N.D. N.D.
12	Abernathy #2	22 103 129 183 233 257 277	3/14/69 5/29/69 6/18/69 7/11/69 7/24/69 9/20/69 11/17/69	N.D. N.D. N.D. N.D. N.D. N.D.	.022 .081 .11 .063 .060 .073	N.D. N.D. N.D. N.D. N.D. N.D.
13	Yellowhouse	37 63 69 93 133	3/20/69 4/12/69 5/ 3/69 5/28/69 6/18/69	N.D. N.D. N.D. N.D.	N.D. N.D. N.D. N.D.	N.D. N.D. N.D. N.D.

TABLE 3. INSECTICIDE CONCENTRATION IN MUD SAMPLES - Continued
N.D. = Not Detectable

Lake Number	Location	Number of Sample	Date	Apparent Aldrin PPM	Apparent Dieldrin PPM	Apparent DDT PPM
		181 223 235B 267 295 341	7/10/69 7/23/69 9/15/69 11/22/69 3/17/70 6/ 2/70	N.D. N.D. N.D. N.D. N.D.	N.D. N.D. .46 N.D. N.D. N.D.	N.D. N.D. N.D. N.D. N.D.
14	Shallowater	6 38 85 87 143 193 231A 258 275	12/23/68 3/20/69 5/ 6/69 5/28/69 6/20/69 7/12/69 7/24/69 9/20/69 11/17/69	.038 N.D. N.D. N.D. N.D. N.D. N.D.	Trace .067 .090 .067 .12 N.D. .084 .067	N.D. N.D. N.D. N.D. N.D. N.D. N.D.
15	Woodrow #2	41S 97 139 268	3/20/69 5/28/69 6/16/69 11/26/69	N.D. N.D. N.D.	.011 .064 .052 .015	N.D. N.D. N.D.
16	Culpepper	40	3/20/69	N.D.	.060	N.D.
17	Woodrow #1	39\$	3/20/69	N.D.	Trace	N.D.
18	Hufstedler	89	5/28/69	N.D.	.12	N.D.
19	Halfway-North Halfway-North Observ. Well N-1 North Well Lake	205 204 204 204	7/ 4/69 8/20/69 8/20/69 8/20/69	N.D. N.D. N.D. N.D.	N.D. N.D. N.D. N.D.	N.D. N.D. N.D. N.D.
	North Well after Recharge	204	10/ 2/69	N.D.	N.D.	N.D.
	North Well Pumping	204	4/ 6/70	N.D.	N.D.	N.D.
20	Halfway-South Well South Well Lake South Well	205 205	7/ 4/69 7/ 4/69	N.D. N.D.	N.D. N.D.	N.D. N.D.
	after Recharge	205	10/10/69	N.D.	N.D.	N.D.

TABLE 3. INSECTICIDE CONCENTRATION IN MUD SAMPLES - Continued

N.D. = Not Detectable

Lake Number	Location	Number of Sample	Date	Apparent Aldrin PPM	Apparent Dieldrin PPM	Apparent DDT PPM
	South Well Lake	205	10/31/69	N.D.	N.D.	N.D.
	South Well In- take Ditch	205	4/ 6/70	N.D.	N.D.	N.D.
21	19th & Vicksburg	71 141 197 207 243 287 307 319 353	5/ 5/69 6/20/69 7/16/69 7/22/69 9/19/69 11/28/69 4/23/70 5/13/70 6/ 2/70	N.D. N.D. N.D. N.D. N.D. N.D. N.D.	.25 .016 .030 .091 .063 .049 .057 .035	N.D. N.D. N.D. N.D. N.D. N.D. N.D.
22	Petroleum Engr.	113 187 221 261 273	6/11/69 7/11/69 7/23/69 9/20/69 11/26/69	N.D. N.D. N.D. N.D. N.D.	.18 .059 .051 .13 .080	N.D. N.D. N.D. N.D. N.D.
23	K. N. Clapp	185 215 247 281 303 321	7/11/69 7/23/69 9/19/69 11/28/69 3/17/70 5/13/70	N.D. N.D. N.D. N.D. N.D.	N.D. Trace N.D. N.D. .006 .036	N.D. N.D. N.D. N.D. N.D.
24	Biology	127 189 229 285 313 329 345	6/18/69 7/11/69 7/23/69 11/28/69 4/23/70 5/14/70 6/ 2/70	N.D. N.D. N.D. N.D. N.D. N.D.	Trace .13 N.D. .019 .050 .053	N.D. N.D. N.D. N.D. N.D. N.D.
25	Experiment Station	10	12/23/68	N.D.	.079	N.D.
26	South of Idalou	4	12/23/68	N.D.	.16	N.D.
27	Hereford (3 mi SW)	149	7/ 7/69	N.D.	.17	N.D.

TABLE 3. INSECTICIDE CONCENTRATION IN MUD SAMPLES - Continued N.D. = Not Detectable

Lake Number	Location	Number of Sample	Date	Apparent Aldrin PPM	Apparent Dieldrin PPM	Apparent DDT PPM
28	Summerfield (1/2 mi)	151	7/ 7/69	N.D.	Trace	N.D.
29	Summerfield	153	7/ 7/69	N.D.	.13	N.D.
30	Summerfield	155	7/ 7/69	N.D.	N.D.	N.D.
31	Happy (6 mi)	157	7/ 8/69	N.D.	N.D.	N.D.
32	Canyon (3 mi)	159	7/ 8/69	N.D.	N.D.	N.D.
33	66th & Sunset	161	7/ 8/69	N.D.	.095	N.D.
34	Wildorado	163	7/ 8/69	N.D.	Trace	N.D.
35	F.M. 1912 & 287	165	7/ 8/69	N.D.	N.D.	N.D.
36	Amarillo (5 mi & 1541)	167	7/ 8/69	N.D.	N.D.	N.D.
37	Amarillo (24 mi)	169	7/ 8/69	N.D.	N.D.	N.D.
38	Tulia on 87 (3 mi)	171	7/ 8/69	N.D.	.12	N.D.
40	Plainview (3 mi)	175	7/ 8/69	N.D.	.027	N.D.
41	Post Lake	231	8/ 1/69	N.D.	.094	N.D.
42	Tulia on 87 (8 mi)	173	7/ 8/69	N.D.	N.D.	N.D.

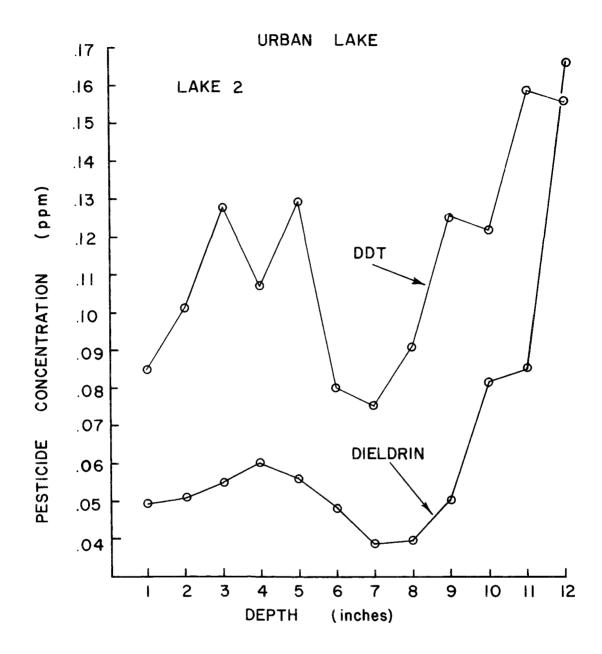


Figure 7. Concentrations of DDT and Dieldrin in Sediment Layers of an Urban Lake.

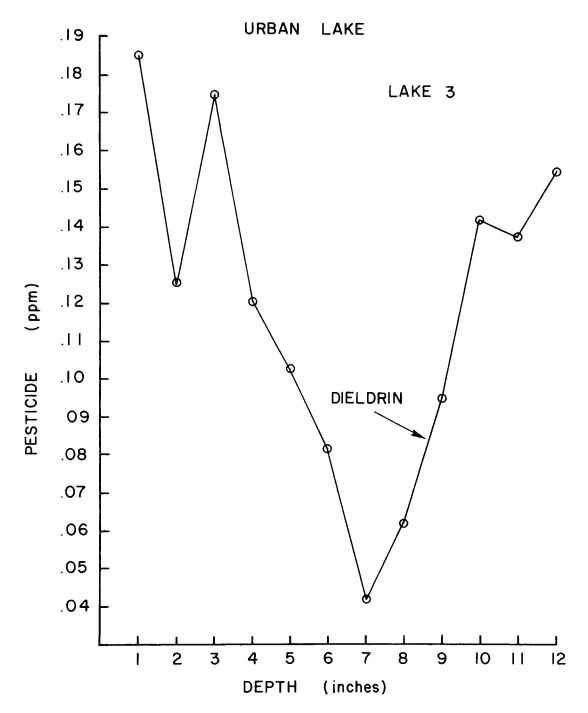


Figure 8. Concentration of Dieldrin in Sediment Layers in an Urban Lake.

pound per acre used for each application. Also, in 1962 and 1963, three pounds of DDT per acre were sprayed on the lakes. Since 1956, DDT dust and Malathion have been used as larvicides and insecticides in conjunction with the other spray programs.

The concentrations of DDT and Dieldrin follow roughly the same patterns from a depth of eight to eleven inches. It therefore appears that in the seven or eight years since Dieldrin was used extensively on urban lakes, very little of it has concentrated in the upper sediment layers of the lakes. The runoff into the lakes since about 1962 has likely carried with it enough silt to deposit the seven or eight inches of sediment found over the levels containing high concentrations of Dieldrin.

The concentrations of DDT in sediments in Lake 2 are of the same magnitude as are the concentrations of Dieldrin. The erratic values obtained for concentrations of DDT from the one to six inch depth levels might be attributed to uneven distribution of the pesticide resulting from wave action or to water level fluctuations resulting in different concentrations being applied to the water in different years.

It is interesting to note that in Lake No. 3, although Dieldrin concentrations were found to be consistently higher than the concentrations found in Lake No. 2, no DDT was present in a measurable concentration, Figure 9.

The concentrations of Dieldrin in rural lakes were found to be considerably lower than those found in urban lakes, Figure 9. Again, it is interesting to note that Dieldrin was the only pesticide found at any depth in the two rural lakes.

Under terms of a parallel contract with the Texas Water Quality Board, all samples collected for this project were analyzed to determine the concentrations of nitrates and phosphates in playa lake water. Nitrates were generally found to be present in concentrations ranging from one to six mg/l, and phosphate concentrations were generally found to range from about 0.01 to a maximum of about 1.0 mg/l.

Lakes 19 and 20 were selected for the specific reason that waters from these lakes are currently being recharged into the Ogallala. Although no tables have been given in this report, it appears that the inorganic elements calcium, phosphate, chloride, and ammonia are present in lake waters in higher concentrations than in Ogallala water. The concentration of nitrate in lake water was in all cases lower than the concentration existing in the groundwater.

After periods of recharge, the concentration of phosphate in well water tended to be less than that found in playa lake water but

phosphate concentrations in well water tended to change with sampling time.

A similar phenomenon was observed regarding the concentration of nitrate. In one series of tests, water containing 3.4 mg/l of nitrate ion was used for recharge. After recharging was complete, a sample from the well indicated a nitrate concentration of 3.2 mg/l. Two months later, a sample from the same well showed a nitrate concentration of 0.1 mg/l. This same nitrate concentration was found at an observation well approximately 200 feet from the recharge well both while recharge was taking place and also two months after recharge had ceased. These findings suggest that recharged water does not necessarily stay in the immediate vicinity of the recharge well, but, under the relatively steep water table gradients induced by normal recharge operations, recharged water tends to move away from the point of recharge at a fairly rapid rate.

# RURAL LAKES

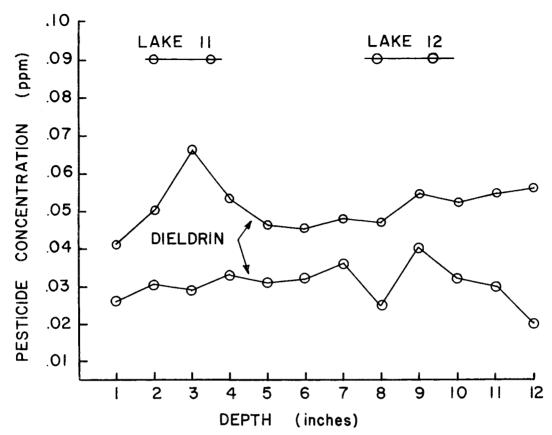


Figure 9. Variation of Dieldrin Concentration with Depth of Sediment, Lakes II and I2.

#### **ACKNOWLEDGMENTS**

The study on which this report is based was supported by the Environmental Protection Agency (formerly the Federal Water Quality Administration). The primary purpose of this project was to determine whether or not the utilization of playa lake water for recharge of the Ogallala aquifer is likely to result in permanent damage to water quality in the form of herbicide and insecticide contamination.

Professional members of the research team were Ellis W. Huddleston, Robert G. Rekers, and Dan M. Wells. In addition, several graduate and undergraduate students contributed to and benefited from this research. Many landowners in the study area cooperated by furnishing information on their farming practices and in permitting samples to be taken from their playas.

A parallel study involving the same research team was financed by the Texas Water Quality Board. The primary purpose of this latter project is to determine the concentrations of nitrates and phosphates in playa lake waters. Some of the data presented in this report were obtained from the TWQB financed project.

# GLOSSARY OF TERMS - Herbicides

AlanapN-l-naphthylphthalamic acid
AMIDENacid
Atrazine2-chloro-4-ethylamino-6-isopropylamino-1,3,
5-triazino
Bandanepolychlorodicyclopentadiene isomers
Danvel D
Barban4-chloro-2-butynyl-N-(3-chlorophenyl)-
carbamato
CIPCisopropyl n-(3-chloro-phenyl) carbamate
Dactid I
2,4 D2,4-dichlorophenoxyacetic acid
2,4, DBdimethylamine salt of 4-(2,4-dichlorophe-
noxy)-butric acid
Dichlobenil2,6-dichlorobenzonitrile
2,4 D (iso-Octylester)iso-octyl 2,4-dichlorophenoxyacetate
Diuron3-(3,4-dichlorophenyl)-1, 1-dimethylurea
DNBP2-(1-methyl-n-propyl)-4,6-dinitrophenyl
2.4 UP acid
Eptamethyl n, n-di-n-propyl thiocarbamate Erbon2-(2,4,5-trichlorophenoxy) ethyl 2,
Erbon2- $(2,4,5-trichlorophenoxy)$ ethyl 2,
2,2-dichloropropionate
Falonetris, $\beta$ -(2,4-dichlorophenoxy) ethyl phos-
phite
IPCn-phenyl isopropyl carbamate
MCPAachloro-2-methylphenoxyacetic acid Prometone2-chloro-4,6-bis(ethylamino)-s-triazine
Propazine2-chloro-4,6-bis(isopropylamino)-1,3,
5-triazine
Rogue3,4 - dichloropropionanilide
Silvex2-(2,4,5-trichlorophenoxy) propionic acid
2,4,5 T2,4,5-trichlorophenoxyacetic acid
Tillamn-propyl N-ethyl-N-(n-butyl) thiocarbamate
2,4,5 T (iso-Octylester)iso-Octyl 2,4,5-trichlorophenoxyacetate
Treflan (Trifluralin) $\alpha$ - $\alpha$ - $\alpha$ -trifluoro-2,6-dinitro-n,n-dipropyl-
p-toluidine
Wallopparathion and 2-chloro-N-isopiopylacetani-
lide combination
Zytrono-(2,4-dichlorophenyl) o-methylisopropyl-
phosphoramidothioate

# GLOSSARY OF TERMS - Insecticides

Aldrin1,2,3,4,10,10-hexachloro-1,4,4,5,8,8 $\alpha$ -hexahydro-1,
4-endoexo-5,8-dimethanonaphthalene
BHC1,2,3,4,5,6-hexachlorocyclohexane (mixed isomers)
Chlordane1,2,4,6,7,8,8-octochloro-3 $\alpha$ ,4,7,7 $\alpha$ -tetrahydro-4,
7-methanoindane
DDT1,1,1-trichloro-2,2-bis(p-chlorophenyl) ethane
Dieldrin1,2,3,4,10,10-hexachloro-6,7-epoxy-1,4,4,5,6,7,8,
8a-octahydro-1,4-endoexo-5,8-dimethanonaphtha-
lene
Endosulfan6,7,8,9,10,10-hexachloro-1,5,5 $\alpha$ ,6,9,9 $\alpha$ -hexahydro-6,
9-methano-2,4,3-benzodioxathiepin-3-oxide
Endrin1,2,3,4,10,10-hexachloro-6,7-epoxy-1,4,4 $\alpha$ ,5,6,7,8,
8α-octahydro-1,4-endo,endo-5,6-dimethanonaphtha
lene
Heptachlor1,4,5,6,7,8,8-heptachloro- $3\alpha$ ,4,7,7-tetrahydro-4,
7-methanoindene
Lindanegamma-1,2,3,4,5,6-hexachlorocyclohexane
Methoxychlor1,1,1-trichloro-2,2-bis(p-methoxyphenyl)-ethane
Mirexdodecachloroctahydro-1,3,4-metheno-2H-cyclobuta
[cd] pentaline
Parathiono,o-diethyl-o,p,nitrophenyl-phosphorothiolate
Perthane1,1-dichloro-2,2-bis-(p-ethylphenyl) ethane
Strobaneterpene polychlorinates
TDE2,2-bis-(p-chlorophenyl)-1-chloroethylene
Toxaphenechlorinated camphene isomers

	Accession Number	2 Subject Field &	& Group	SELECTED WATER RESOURCES ABSTRACTS		
	_	05A		INPUT TRANSACTION FORM		
5	Organization Texas Tech Ui	niversity Water	r Resou	rces Center, Lubbock, Texas		
6	Potential Po	llution of the	Ogalla	la by Recharging Playa Lake WaterPesticides		
10	Author(s)	16	Project	Designation		
	Wells, Dan M.			Project No. 16060 DCO		
	Huddleston, Ellis Rekers, Robert G.	W. 21	Note			
22	Citation					
23	Descriptors (Starred First)					
	*Herbicides, *Insecticides, *Runoff, *Playa Lakes, *Ogallala					
25	Identifiers (Starred First)					
27	Abstract					
	The purpose of this study was to determine the concentrations of herbicides and insecticides in playa lake water in the High Plains of West Texas. Twenty-four					

The purpose of this study was to determine the concentrations of herbicides and insecticides in playa lake water in the High Plains of West Texas. Twenty-four urban and rural lakes were sampled routinely for the period of eighteen months following runoff-producing precipitation events. Samples of water and sediment were analyzed by means of a gas chromatograph to determine concentrations of all herbicides and insecticides commonly used in the area. Findings of the research are that runoff water does not contain any measurable concentrations of any of the commonly used herbicides or insecticides, and that sediment samples contain very low concentrations of some of the compounds. The compound most commonly found in sediment samples was Dieldrin, with Aldrin being next most common, and DDT found in only a few sediment samples.

Abstractor Dan M. Wells Texas Tech University

WR:102 (REV JULY 1969) WRSIC