

Occurrence of Cotton Herbicides and Insecticides in Playa Lakes of the High Plains of West Texas

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ABSTRACT

During the summer of 1997, water samples were collected and analyzed for pesticides from 32 playa lakes of the High Plains that receive drainage from both cotton and corn agriculture in West Texas. The major cotton herbicides detected in the water samples were diuron, fluometuron, metolachlor, norflurazon, and prometryn. Atrazine and propazine, corn and sorghum herbicides, also were routinely detected in samples from the playa lakes. Furthermore, the metabolites of all the herbicides studied were found in the playa-lake samples. In some cases, the concentration of metabolites was equal to or exceeded the concentration of the parent compound. The types of metabolites detected suggested that the parent compounds had been transported to and had undergone degradation in the playa lakes. The types of metabolites and the ratio of metabolites to parent compounds may be useful in indicating the time that the herbicides were transported to the playa lakes. The median concentration of total herbicides was 7.2 micrograms per liter, with the largest total concentrations exceeding 30 micrograms per liter. Organophosphate insecticides were detected in only one water sample. Further work will improve the understanding of the fate of these compounds in the playa lakes area.

INTRODUCTION

Cotton farming may require as much as 7 kg/ha (kilograms per hectare) of herbicides and 5 kg/ha of insecticides annually (Coupe and others, 1998). The intensive application of pesticides often is necessary, especially in the Southern United States, where weed and insect pressures are great. Applications of pesticides to cotton are three to five times greater per hectare than applications to corn and occur more frequently (average of 4.7 annual applications compared to 1.2) (Coupe and others, 1998), yet there have been few regional studies of

water quality and pesticide fate in cotton-producing areas of the country. For this reason, the U.S. Geological Survey's (USGS) Toxics Substances Hydrology Program initiated research on the occurrence of cotton pesticides in the aquatic environment with the purpose of determining the extent and magnitude of herbicide and insecticide concentrations in both surface and ground water of the Southern United States.

Since the inception of this water-quality research in 1995, two publications have described the extent of occurrence of cotton pesticides in surface water (Coupe and others, 1998; Thurman and others, 1998). The first publication (Thurman and others, 1998) dealt with the occurrence of cotton pesticides in surface water of the Mississippi Embayment. The work described in this fact sheet indicates that cotton herbicides occur frequently in surface water, with the major compounds, in order of percent detections, being: fluometuron > cyanazine > metolachlor > norflurazon > prometryn. Unfortunately, the phenylurea herbicide, diuron, was not examined in this earlier work. Insecticides were also reported in this study, with the important detections being: dicotophos > profenofos > methyl parathion > malathion.

A second publication by Coupe and others (1998) dealt with the usage of herbicides and their occurrence in surface water of the Mississippi Delta. A major finding described in the report was that the distribution and duration of total herbicide concentrations were much different from that found in regional studies of herbicides in the Midwestern United States (Coupe and others, 1998). In the Midwest, the total herbicide concentration in surface water showed a sharp peak during the spring immediately after application of herbicides to crops, followed by a gradual decrease in concentration (Thurman and others, 1991; Thurman and others, 1992). In the Mississippi Delta, the total herbicide concentration in surface water was more sustained, with multiple peaks due to different application times and post-emergent applications of

herbicides on cotton and rice (Coupe and others, 1998).

In this paper, the concentrations of herbicides and insecticides in water samples from playa lakes of the West Texas High Plains are reported because playa lakes form the major surface drainage in the area. Playa lakes are small lakes formed from precipitation and runoff in arid regions and are temporary lakes that either evaporate or infiltrate into the soil. Land-surface drainage is to playa lakes after rainfall. Farmers in the High Plains of West Texas grow both dryland (nonirrigated) cotton and irrigated cotton. Water samples were collected from each of 32 playa lakes (fig. 1) on July 22–23, 1997, after pesticide application to cotton in the region.

SAMPLE COLLECTION AND ANALYSIS

Surface-water grab samples were collected at each of the 32 playa lakes. Samples were filtered through 0.70- μm (micrometer) glass-fiber filters and stored on ice until analyzed at the USGS laboratory in Lawrence, KS. Water samples were analyzed for the following parent compounds: atrazine, cyanazine, diuron, fluometuron, metolachlor, norflurazon, prometryn, and propazine.

Materials used in analysis consisted of C-18 cartridges (Waters, Milford, MA) with 350 mg (milligrams) of 40- μm , C-18 bonded silica. Analytical standards were obtained from various sources. Atrazine, cyanazine, 3,4-dichloroaniline (3,4-DCA), deethylatrazine (DEA), deisopropylatrazine (DIA), deisopropylprometryn,



Figure 1. Location of study area where playa lakes were sampled in West Texas, July 22–23, 1997.

dicrotophos, diuron, fluometuron, hydroxyatrazine, metolachlor, norflurazon, prometryn, and propazine were obtained from Supleco (West Chester, PA). Dichloromethylphenylurea (DCMPU) and dichlorophenylurea (DCPU) were obtained from Jennifer Field, Oregon State University (Corvallis, Oregon). Demethylfluometuron (DMFM), trifluoromethylphenylurea (TFMPU), and trifluoromethylaniline (TFMA) were obtained from the U.S. Department of Agriculture, Agriculture Research Service (Stoneville, MS). Demethylnorflurazon was obtained from Sandoz Agro, Inc. (Des Plaines, IL). Metolachlor oxanilic acid was obtained from Novartis (Greensboro, NC). Metolachlor ethane sulfonic acid was synthesized in the USGS laboratory in Lawrence, KS, by Aga and others (1996).

The following gas chromatograph/mass spectrometry (GC/MS) method is also described in Thurman and others (1990). Solid-phase extraction (SPE) was automated on a Waters Millilab workstation (Milford, MA). C-18 Sep-Pak cartridges were conditioned sequentially with 2 mL (milliliters) methanol, 2 mL ethyl acetate, 2 mL methanol, and 3 mL distilled water. Each 123-mL water sample was spiked with a surrogate standard, terbuthylazine (1.23 ng/ μ L, nanograms per microliter; 100 μ L, microliters), and pumped through the cartridge at a rate of 20 mL/min (milliliters per minute) by a robotic probe. Analytes were eluted with ethyl acetate and spiked automatically with phenanthrene d-10 (0.2 ng/ μ L, 500 μ L). The extract was evaporated by a TurboVap (Zymark, Palo Alto, CA) at 45 °C (degrees Celsius) under nitrogen gas to 100 μ L.

GC/MS analysis of the eluates was carried out using a Hewlett-Packard model 5890A GC interfaced to a 5970A mass selective detector (MSD) (Palo Alto, CA). One microliter (1 μ L) of sample was injected automatically. Separation of the herbicides and insecticides was accomplished with a fused-silica capillary column of 5-percent phenyl methyl silicone (Ultra 2) with a film thickness of 0.33 μ m, 12 m (meter) x 0.2 mm (millimeter) inside diameter (Hewlett Packard, Palo Alto, CA). Helium was used as the carrier gas at a flow rate of 1 mL/min and a head pressure of 35 kPa (kilopascals). The column temperature was held at 60 °C for 1 minute ramped at 6 °C per minute to 200 °C, and then ramped at 30 °C per minute to 250 °C, where the temperature was held for 4 minutes. The samples were injected in the splitless

mode using an autoinjector at an injector temperature of 180 °C.

The source of the mass spectrometer was held at 280 °C. The emission current was 70 eV (electronvolts). The electron multiplier was set at 400 V (volts) above autotune. The filament and multiplier were turned on after 4 minutes into the analysis. An autotune using perfluorotributylamine was performed daily prior to analysis of samples. The calibration curve was prepared on the basis of the area ratio of the base peaks relative to the response of the 188 (amu) ion of phenanthrene d-10, the internal standard. Confirmation of the compounds was based on the presence of the molecular ion and two confirming ions, a retention-time match within 0.2 percent relative to phenanthrene d-10, and correct area ratios of the confirming ions.

Metolachlor ethane sulfonic acid (ESA) and metolachlor oxanilic acid were analyzed according to the method of Hostetler and Thurman (1999). Briefly, high-performance liquid chromatography (HPLC) is carried out on two in-line analytical columns. First is an octadecyl silica (ODS) 5- μ (micron), 250- x 3-mm column coupled to an ODS 3- μ , 250- x 4.6-mm column. The mobile phase was 60:35:5 of pH 7.0, 25-mM (millimole) phosphate buffer/methanol/acetonitrile with a flow rate of 0.6 mL/min. The instrument consisted of a high-performance liquid chromatograph, Hewlett-Packard 1090 (Hewlett-Packard, Palo Alto, CA), equipped with a diode array detector.

Using the previously described automated SPE procedure, diuron and its metabolites, DCMPU and DCPU, were extracted from water. The ethyl acetate extract was solvent exchanged to methanol by evaporating to approximately 100 μ g/L, adding 2 mL methanol, and then evaporated to 40 μ L. The final volume of the extract was adjusted to 75 μ L by the addition of 35 μ L of pH 7.0 phosphate buffer.

The extract then was analyzed by HPLC. The analytical columns used were identical to the previously described columns. The mobile phase consisted of 50 percent methanol and 50 percent, pH 7.0 phosphate buffer, with a gradient ramping to 75 percent methanol. The flow rate was 0.5 mL/min.

The detection and quantitation limits were 0.05 μ g/L for all compounds analyzed by GC/MS and 0.2 μ g/L for compounds analyzed by HPLC. All laboratory blanks were free of pesticides or metabolites. The variation of the duplicate samples

was within ± 5 percent at one standard deviation. The correlation coefficients of the standard curves were 0.998 ± 0.002 . Any samples with concentrations greater than $10 \mu\text{g/L}$ were diluted and re-analyzed.

OCCURRENCE OF HERBICIDES IN PLAYA LAKES

Cotton and (or) corn herbicides were detected in 97 percent of one-time samples from 32 playa lakes in the High Plains of West Texas. The playa-lake samples contained nine herbicides and

their metabolites. The herbicides were, in order of percent detections: diuron > prometryn > atrazine > propazine > metolachlor > fluometuron > norflurazon > simazine > cyanazine (fig. 2). Only atrazine and propazine are not used on cotton; however, corn and sorghum are also grown adjacent to the cotton and are the most probable sources of atrazine and propazine in the playa lakes. All of the playa-lake samples contained herbicides, with a median concentration of total herbicide of (parent herbicides and metabolites) of $7.2 \mu\text{g/L}$ and a range from 0.57 to $33 \mu\text{g/L}$.

Figure 3 shows the total concentration of all herbicides and metabolites detected in samples



Figure 2. Frequency of detections of herbicides and metabolites in 32 playa-lake samples from High Plains of West Texas.

from each playa lake. Metabolites make up a significant proportion of the total herbicide concentration in the lake samples (fig. 3). Only one playa-lake sample (playa lake 19) did not contain metabolites. The median metabolite percentage was 27 percent of the total herbicide present. The highest metabolite percentage was 70.5 percent. The metabolites that were detected (fig. 2) included three metabolites of atrazine (deisopropylatrazine or DIA, deethylatrazine or DEA, and hydroxyatrazine), three metabolites of diuron (dichloromethylphenylurea or DCMPU, dichlorophenylurea or DCPU, and 3,4-dichloroaniline or 3, 4-DCA), two metabolites of fluometuron (demethylflu-

ometuron and trifluoromethylaniline or TFMA), two metabolites of metolachlor (metolachlor ethane sulfonic acid or metolachlor ESA and metolachlor oxanilic acid), one metabolite of norflurazon (demethylnorflurazon), and one metabolite of prometryn (deisopropylprometryn). Of these metabolites, the most frequently detected (fig. 2) were DEA (atrazine), at more than 60 percent detections, followed by DCMPU (diuron) > deisopropylprometryn (prometryn) > 3,4-DCA (diuron) > hydroxyatrazine (atrazine) > DCPU (diuron) > DIA (atrazine) > metolachlor oxanilic acid (metolachlor) > metolachlor ESA (metolachlor) > demethylnorflo-

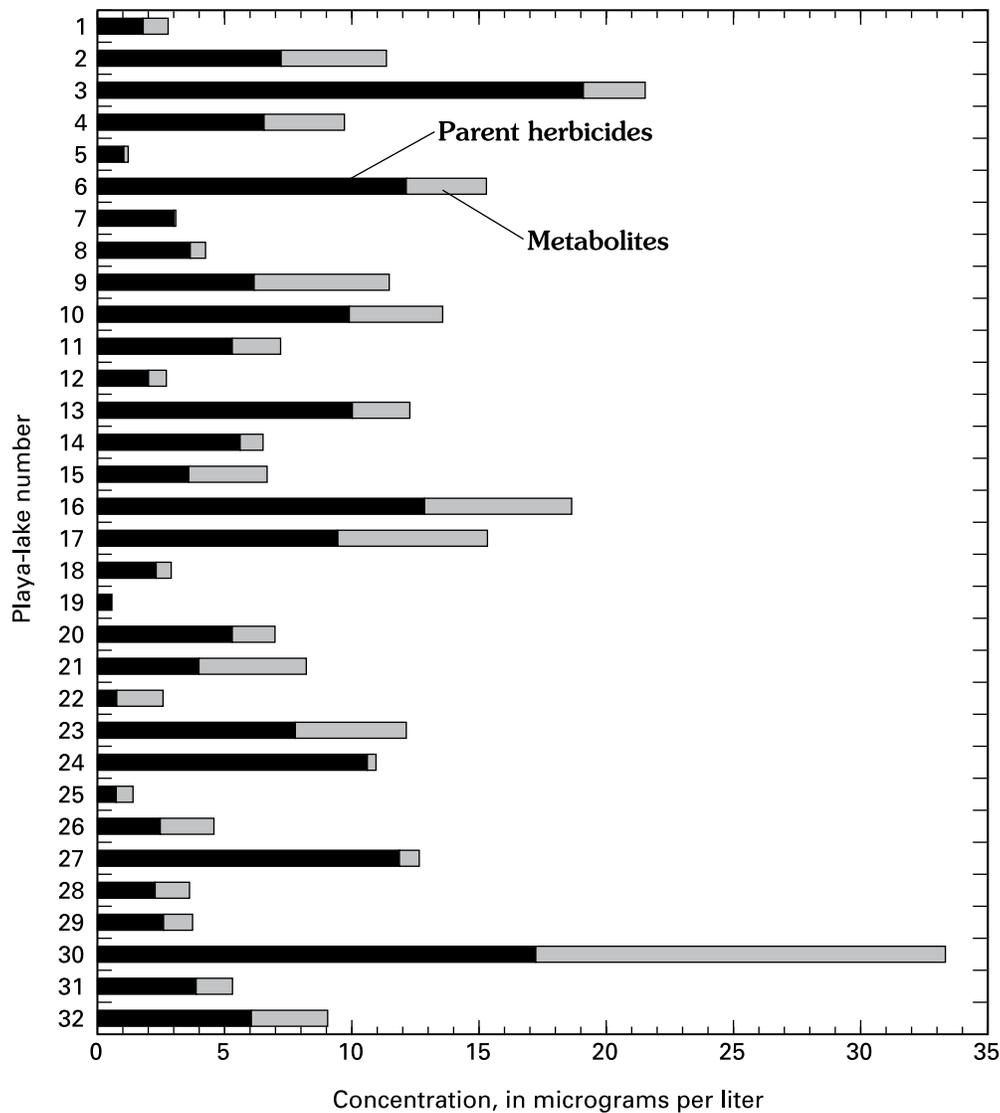


Figure 3. Concentrations of parent herbicides and metabolites in playa-lake samples from High Plains of West Texas.

razon (norflurazon) > TFMA > demethylfluometuron (fluometuron).

Figure 4 shows mean concentrations of herbicides and metabolites detected in the playa-lake samples. Compounds with relatively high mean concentrations are diuron (2.7 µg/L), prometryn (1.3 µg/L), and metolachlor (1.0 µg/L). Nondetections were averaged as 0.0 in mean calculations. The frequency of detection (fig. 2) and the mean concentrations (fig. 4) of these three compounds indicate that they are probably the ones most used and readily transported into the playa lakes. All three compounds are used on cotton, and metolachlor also may be used on corn and sorghum. The other cotton herbicide that was detected frequently was fluometuron, which had a mean concentration of 0.5 µg/L. Maps of fluometuron use in 1992 show that it was not used in West Texas (Thurman and others, 1998). However, these maps may be incorrect on the basis of detections of fluometuron in the playa-lake samples. That information is not consistent with the results of the 1997 sampling.

Metabolites of four cotton herbicides (diuron, fluometuron, metolachlor, and prometryn) also were detected in samples from many of these same

playa lakes. For example, the metabolites of diuron (DCMPU, DCPU, and 3,4-DCA) were found in 71 percent of the samples with positive detections of diuron. DCMPU was the most frequently detected and was present at the highest concentrations of the three metabolites. The mean concentrations of the three metabolites were 0.45 µg/L for DCMPU, 0.31 µg/L for 3,4-DCA, and 0.2 µg/L for DCPU (fig. 4). Demethylation of the phenylurea herbicides is apparently a common degradation pathway (Mueller and Moorman, 1991), and this pathway also occurs in fluometuron. For example, the detected metabolites of fluometuron were demethylfluometuron and TFMA; one is a demethylated metabolite (DMFM), and the other is trifluomethylaniline and is analogous to the 3,4-DCA metabolite of diuron. Norflurazon also degrades to a demethylated metabolite, demethylnorflurazon. That metabolite was detected in 16 percent of the samples with a mean concentration of 0.17 µg/L. Its concentration and frequency of detection are similar to the parent compound, reflecting the fact that the half-life of norflurazon is relatively short, approximately 45 days in soil (Ahrens, 1994).

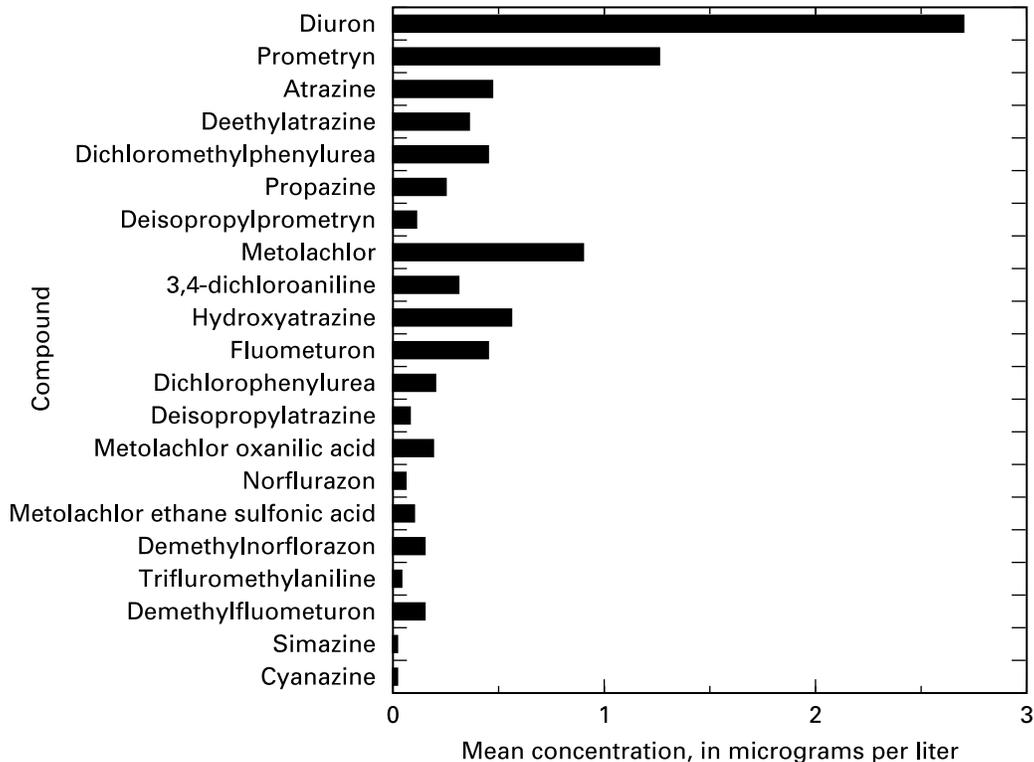


Figure 4. Mean concentrations of herbicides and metabolites in 32 playa-lake samples from High Plains of West Texas.

Metolachlor metabolites included both the ESA and the oxanilic acid of metolachlor. The mean concentration of the oxanilic acid metabolite was 0.19 $\mu\text{g/L}$ as compared to 0.1 $\mu\text{g/L}$ for the ESA metabolite. The fact that oxanilic acid concentration exceeded ESA concentration (true for all samples) suggests that the degradation of the parent metolachlor occurred in the playa lakes rather than in the soil. When metolachlor degrades chiefly in the soil, the major metabolite is the ESA (Field and Thurman, 1996; Thurman and others, 1996; Kalkhoff and others, 1998). Further work is underway to validate this hypothesis.

Another important cotton herbicide frequently detected in the playa-lake samples was prometryn, with a frequency of detections of 72 percent and a mean concentration of 1.3 $\mu\text{g/L}$. The metabolite of prometryn, deisopropylprometryn, was in nearly every sample that contained prometryn. The metabolite comes from the de-alkylation of the herbicide on the amino side of the molecule, which is a common degradation pathway for triazine herbicides such as prometryn. The relative abundance of the metabolite to the parent compound was 0.1 or about 10 percent of the mass of the parent compound. Prometryn is a resistant triazine with a long half-life of about 60 days or more in the soil (Ahrens, 1994).

The other two triazine herbicides that were detected routinely in the playa-lake samples were atrazine and propazine. Atrazine was detected in 72 percent of the samples with a mean concentration of 0.47 $\mu\text{g/L}$. Propazine was detected in 59 percent of the samples with a mean concentration of 0.25 $\mu\text{g/L}$. Atrazine and propazine have one common metabolite, DEA, which was found in 63 percent of the samples with a mean concentration of 0.36 $\mu\text{g/L}$. Because propazine has a longer soil half-life than atrazine (atrazine is 45–60 days and propazine is at least 60 days), it is thought that atrazine is responsible for the majority of the DEA. Furthermore, atrazine was present at a mean concentration that was about twice as much as propazine. Commonly, propazine is detected at much lower concentrations than atrazine because propazine is no longer used (voluntarily removed from the market in early 1990's by the manufacturer); however, supplies remain, and it is apparent from these data that propazine is still being used in the High Plains of West Texas.

An unusual finding was the presence of hydroxyatrazine in many of the playa-lake samples. It was detected in 44 percent of the samples with a mean concentration of 0.56 $\mu\text{g/L}$, a value comparable to that of atrazine. This result is unusual compared to the studies that have been carried out in the Corn Belt of the Midwestern United States (Thurman and others, 1991; 1992). Lerch and others (1998) have shown that hydroxyatrazine is a minor component during the growing season compared to the parent compound, atrazine. The more conservative nature of the hydroxyatrazine has been ascribed to the insolubility and sorption of the hydroxyatrazine metabolite to soil by a mixed-mode mechanism (Lerch and others, 1997). One explanation for the higher concentrations of hydroxyatrazine in the playa-lake samples is the more alkaline soils in this part of West Texas, where soil pH is commonly greater than 7. At these high pH values, the mixed-mode mechanism is inactive because the negative log of the acid dissociation constant (pK_a) of hydroxyatrazine is approximately 5. Thus, at a pH that is 2 units higher than the pK_a , the molecule will not be positively charged, which would lessen the effectiveness of the mixed-mode mechanism. For example, the hydroxyatrazine concentration in the water sample from playa lake 30 was 5.4 $\mu\text{g/L}$, which is one of the highest concentrations for hydroxyatrazine that has been measured (Lerch and others, 1998).

The ratio of metabolites to parent compounds has been suggested as a means of dating the relative age of surface water that contains herbicides. Thurman and Fallon (1996) used the deethylatrazine-to-atrazine ratio to indicate the age of surface water that has received atrazine runoff. Figure 5 shows a comparison of atrazine concentrations to the total concentrations of its metabolites as an indicator of this year's or previous years' application of atrazine. The data show that metabolite concentrations are greater than or equal to atrazine concentrations in many of the playa-lake samples. These data indicate that the water in the lakes contains "old" (degraded) atrazine from previous years' application.

Figure 6 shows a comparison of cotton-herbicide concentrations to the total concentrations of their metabolites in the playa-lake samples. The cotton-herbicide parent compounds exhibit considerably higher concentrations than their metabolites, in spite of the fact that half-lives for the cotton her-

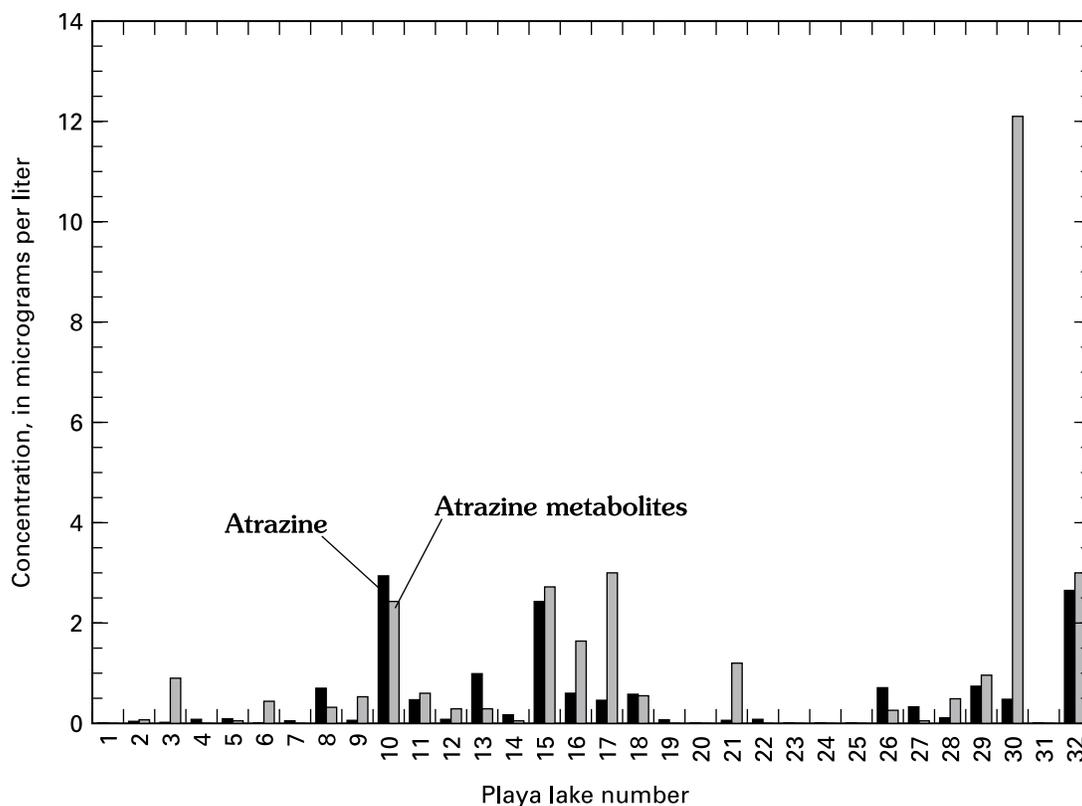


Figure 5. Comparison of atrazine and atrazine metabolite concentrations in playa-lake samples.

bicides are comparable to the half-life for atrazine. Only the sample from playa lake 9 contained about equal parent compound and metabolite concentrations for the herbicides, with a concentration for both of about 6 µg/L. Data from this sampling site suggest that the cotton herbicides may be from previous seasons.

OCCURRENCE OF INSECTICIDES IN PLAYA LAKES

Organophosphate insecticides were detected in only one sample from the 32 playa lakes for a detection frequency of 3 percent. The one detection was dicrotophos. Other organophosphate compounds that were analyzed were azinophos methyl, chlorpyrifos, malathion, methylparathion, and profenfos. Organophosphate insecticide use on cotton in West Texas is extensive, with more than 1.5 million kilograms (Gianessi and Anderson, 1995) applied annually for the State, approximately half of which is used in the High Plains of West Texas. Thus, the amount used is not an explanation for low

detection frequency. However, the time of sampling was early summer so that application may not have occurred prior to sampling. A check of application dates for this area suggests that this could be a possible explanation. Another possible explanation is that the half-life of organophosphate insecticides is generally short, less than 10 days. Thus, degradation of the parent compounds also could be an explanation for the low detection frequency. Another sampling of the playa lakes later in the season to provide more information on concentrations in relation to timing of applications is planned for 1999.

CONCLUSIONS

Cotton and (or) corn herbicides were detected in 97 percent of one-time samples from 32 playa lakes in the High Plains of West Texas. The major cotton herbicides detected were diuron, fluometuron, metolachlor, norflurazon, and prometryn. The corn herbicide atrazine and its metabolites also were commonly found in the playa-lake samples.



Figure 6. Comparison of cotton herbicide and metabolite concentrations in playa-lake samples.

Relative concentrations of parent herbicides and their metabolites may be used as indicators of recent herbicide runoff to the playa lakes as compared to previous years' applications. The presence of hydroxyatrazine was notably high in concentration relative to many surface waters in the Midwestern United States that have been studied. Organophosphate insecticides were detected in only one sample from the playa lakes.

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