

harvested at various intervals after the terminal EBDC application.

Since many households cook with fresh tomatoes and commercial tomato products, some samples were heated for 10 min in the laboratory and then analyzed for ETU (Tables V and VIII). Elevated ETU levels were found in all but one sample after cooking; in three samples ETU was found after cooking at trace levels (0.01–0.02 ppm) in samples that previously showed no apparent ETU residues.

In conclusion, field tomatoes treated at the recommended rate for EBDC fungicides contain EBDC residues of <4 ppm and ETU residues that were detectable, but <0.05 ppm, at the suggested harvest date. Tomato products prepared from treated fruit had EBDC concentrations of <1 ppm and ETU concentrations of <0.1 ppm; commercial tomato products had trace levels of EBDC and ETU concentrations of <0.05 ppm. Lye washing to remove tomato skins appeared to be beneficial in reducing both EBDC and ETU residues. Boiling of fresh fruit and commercial products resulted in increased levels of ETU.

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## Residues in Crops Irrigated with Water Containing Simazine

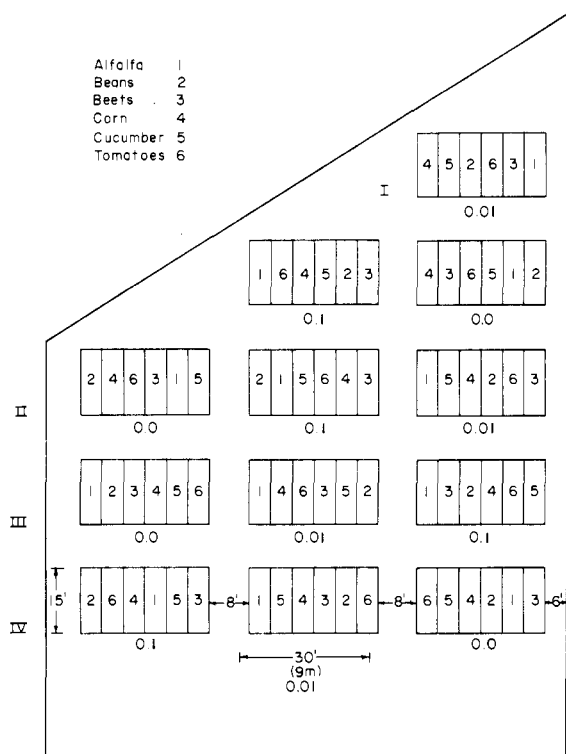
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Annual ditchbank vegetation growing on the inside slopes of irrigation canals can be managed with low-rate applications of simazine. When a herbicide is applied to the bank of either a flowing or a dry canal, some residue might be expected in the irrigation water applied to crops. Six crops representing nine commodity groupings were irrigated with water containing simazine [2-chloro-4,6-bis(ethylamino)-s-triazine] at 0.01 and 0.10 mg/L. The treatment levels were selected to simulate the maximum amount and ten times the maximum that might be expected to enter irrigation water after bank application for weed control. Herbicide was applied to randomized test plots through sprinkler and furrow irrigation, and crops were harvested 7 and 30 days after treatment. Samples were analyzed for residues with a gas chromatograph equipped with a nitrogen and phosphorus detector. No simazine residue was found in corn grain and pinto bean pods while trace amounts were found in pinto bean foliage and cucumbers. Amounts ranging from 0.6 to 2.9  $\mu\text{g}/\text{kg}$  were found in sugar beets, corn foliage, and tomatoes. Sugarbeet foliage collected 7 days after application of simazine at 0.01 and 0.10 mg/L by sprinkler irrigation contained 5  $\mu\text{g}/\text{kg}$ . Alfalfa contained the most residue of the crops tested; samples collected from plots that were sprinkler-irrigated with water containing simazine at 0.10 mg/L contained 6.4  $\mu\text{g}/\text{kg}$ .

Simazine [2-chloro-4,6-bis(ethylamino)-s-triazine] is one of the most widely used preemergence herbicides for the

control of annual grasses and broadleaf weeds in orchards and in such crops as corn, alfalfa, and sugar cane. The persistence and movement of simazine after such applications have been extensively studied, but less effort has been directed to following its accumulation and dissipation after use for weed control in or along irrigation channels (Smith et al., 1975). In 1957, B. H. Grigsby of Michigan

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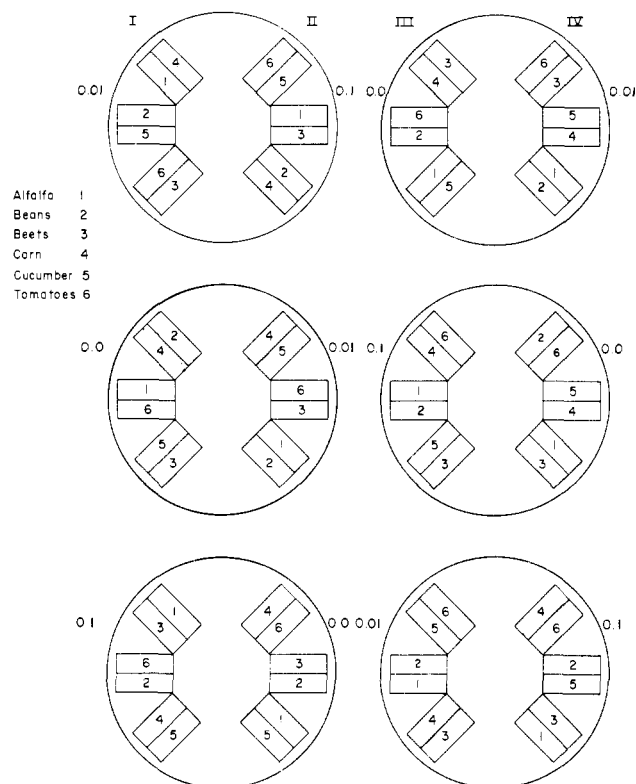


**Figure 1.** Furrow-irrigation plot layout for simazine treatments.

State University described the application of simazine for the control of algae and aquatic vascular plants (Ellis et al., 1976). At present, the only approved use for simazine in or near aquatic sites is in ponded water, where the aquatic formulation, Aquazine, is employed. The study reported here was designed to provide information on the amount of simazine that might be absorbed by crops irrigated with water containing the herbicide. The treatment levels were selected to simulate the maximum level and ten times the maximum that might be expected to enter irrigation water after bank application for weed control. The study was correlated with another activity in which simazine was applied at rates of 2.25 and 4.5 kg/ha to sections of bank along one side of both flowing and dry canals in California, Colorado, and Washington (Anderson et al., 1978). Water samples were taken immediately after herbicide application adjacent to flowing canals and at the time of first spring flow in 1977 from dry application sites.

#### METHODS AND MATERIALS

**Study Site, Plots, and Plants.** The study was conducted within the Denver Federal Center, which is located on the western edge of the metropolitan area at an elevation of 1680 m. The soil was a sandy-clay loam (Denver Clay Loam series) and contained 1.8–2.6% organic matter. The total land area devoted to the study was 0.76 ha, of which 0.2 ha was set aside for the furrow treatments and 0.56 ha for the sprinkler treatments. The area was large enough for the plots to be moved from year to year, to avoid the possibility of soil residue carryover and buildup from previous herbicide uptake studies. Individual plots were arranged as shown in Figures 1 and 2. The 4.5 m × 9 m furrow-irrigated plots were divided into six 1.5 m × 4.5 m subplots, each to contain one crop. Six circular sprinkler-irrigated plots, each with a radius of 9 m and spaced 21 m on center, were divided into 3 m × 4.5 m radially arranged subplots. The subplots were further divided into 1.5 m × 4.5 m planting areas, each to contain one crop. The six crops that were treated and the com-



**Figure 2.** Sprinkler-irrigation plot layout for simazine treatments.

modity groups that they represented were: tomatoes, fruiting vegetable; cucumbers, cucurbit; alfalfa, forage (legume); sugar beets, root crop, and leafy vegetable; corn, grain crop, and grass forage; and pinto beans, seed pod vegetable, and stored grain.

**Irrigation and Herbicide Application.** The six crops were furrow- and sprinkler-irrigated with irrigation water containing simazine at levels of 0.0, 0.01, and 0.10 mg/L with the twofold purpose of obtaining residue data and observing injury to sensitive crops. The experiment was replicated four times.

The arrangement of plots and the apparatus for the application of herbicide were patterned after those developed by Bruns (Bruns and Kelley, 1974; Bruns et al., 1974). The application techniques were tested and modified during the 1974 and 1975 seasons, when dalapon and MSMA were applied. For the furrow-irrigated treatments, simazine was mixed with water in stock-watering tanks with capacities of 1130 L and distributed to the individual furrows by a manifold device that provided equal volumes to each replicate plot. For the sprinkler applications, a pressurized tank and metering device were used to deliver a concentrated solution to the individual sprinkler lines for final dilution. A controlled heating unit installed in the bottom of the tank maintained a solution temperature of 50–60 °C. At this temperature, simazine was soluble without agitation. After dilution, the simazine was applied with pulsating-type sprinklers. The herbicide was applied in 50.8 mm of water for both sprinkler and furrow treatments. The crops were treated in August 1976 at a stage considered to be mid-maturity; the plants had flowered, but fruit had not set. Rainfall, in the form of light thundershowers, totaled 63.5 mm during August and 47.75 mm during September. This was supplemented with irrigation as dictated by soil moisture content.

**Sample Collection and Analysis.** Crop samples totaling 1 kg were collected from the plots at 7 and 30 days after treatment. The samples, taken at random from

plants distributed throughout the plots, were placed in plastic bags and frozen until analyzed. The extraction procedures were based on the methods of Mattson et al. (1965, 1970), with modifications to suit the individual crops and the detection system used. For extraction, 75 g of plant material was chopped and homogenized in a blender with 150 mL of distilled water. The slurry was transferred to a 1-L glass jar with a Teflon-lined cap and mixed with 160 mL of chloroform for 0.5 h on a mechanical shaker. The mixture was decanted through a glass wool-lined Buchner funnel into a 500-mL separatory funnel. The glass jar was rinsed with 20 mL and 10 mL of chloroform, which was also decanted through the Buchner funnel. The glass wool mat was compressed to remove as much liquid as possible and rinsed with 10 mL of chloroform. After separation of the chloroform-water-plant material mixture in the separatory funnel, the chloroform was drained through a fritted glass funnel containing 20 g of anhydrous sodium sulfate. The sodium sulfate was rinsed with 10 mL of chloroform and the entire extract was diluted to a standard volume of 200 mL, and a 100-mL aliquot was removed. A stream of dry air and a water bath heated to 30 °C were used to evaporate the sample to dryness, after which the residue was dissolved in benzene and poured onto a clean-up column containing 24 g of activity grade V aluminum oxide. The various contaminants, including large amounts of pigment from some crops, were removed with a rinse of 75 ml of high-purity hexane. Simazine was eluted from the column with 150 mL of a 1:1 high-purity benzene and hexane mixture. The solvent was evaporated to dryness with a stream of air and the remaining residue was transferred to a graduated 15-mL centrifuge tube with volumes of 5, 4, and 3 mL of high-purity ethyl ether. After evaporation of the ethyl ether, the residue was dissolved in benzene before analysis.

Simazine recovery levels were determined from fortified samples of each crop. Untreated 75-g plant samples were chopped in a blender, and simazine was added to duplicate samples to produce concentrations of 0.10, 0.5, and 1.0 mg/L. The percent recovery was calculated for each concentration and an average simazine recovery value was determined for each crop.

Residues were analyzed on a Hewlett-Packard Model 5730A gas chromatograph equipped with a nitrogen and phosphorus detector. The standard flame collector assembly was replaced with one that contained an alumina cylinder coated with rubidium bromide. The response of the detector to compounds containing nitrogen and phosphorus appears to involve a low-temperature plasma layer surrounding the alkali pellet (Burgett et al., 1976; Maier-Bode and Riedmann, 1975).

The detector was sensitive to simazine in the range of 0.2 µg/L from a crop extract and was selective enough to require only minimal clean-up procedures. However, the lifespan of the collector units was unexpectedly short, possibly because of contaminants in the extracts. A 2 mm i.d. × 1.2 m glass column packed with 100–120 mesh Chromosorb W coated with 2% OV-101 was used throughout the analysis. The rate of nitrogen flow through the column was maintained at 30 mL/min. The optimum rate of hydrogen and air flow through the detector was determined and maintained for each collector unit. The column temperature was 200 °C, with injection port and detector settings of 250 and 300 °C, respectively.

## RESULTS AND DISCUSSION

The simazine recovery levels from fortified crop samples are shown in Table I. Average recoveries ranged from 79%, for alfalfa, to 93%, for pinto bean pods and foliage.

Table I. Recovery of Simazine from Fortified Crops

crop	simazine added, mg/L	recovery, %	Av recovery for each crop, %
tomato	0.1	96	93
	0.5	83	
	1.0	97	
cucumber	0.1	89	88
	0.5	89	
	1.0	87	
alfalfa	0.05	88	79
	0.1	78	
	0.5	75	
	1.0	75	
sugar beet	0.05	80	88
	0.1	90	
	0.5	89	
	1.0	93	
sugar beet foliage	0.05	84	89
	0.10	89	
	0.50	91	
	1.0	91	
corn	0.05	86	89
	0.10	88	
	0.50	88	
	1.0	92	
corn foliage	0.05	86	89
	0.10	86	
	0.50	93	
	1.0	90	
pinto beans and pods	0.05	93	93
	0.10	90	
	0.50	93	
	1.0	94	
pinto bean foliage	0.05	84	93
	0.10	96	
	0.50	95	
	1.0	95	

The residue levels detected in each test crop are shown in Table II. All values are means of four replicate treatments, corrected for recovery and reported in terms of µg/kg of fresh plant material. In one instance (7-day samples from the 0.01 mg/L treatment of alfalfa), the reported amount of simazine residue is below the minimum level of sensitivity of the detector. This resulted from averaging three alfalfa replicates which contained no simazine residue and one which contained 0.3 µg/kg. Most samples taken from the untreated control plots at 7 and 30 days showed no trace of simazine. No residue was found in tomato samples taken 7 days following treatment with simazine at rates of 0.01 or 0.10 mg/L by either sprinkler or furrow irrigation. At 30 days after treatment, however, the 0.01 mg/L sprinkler-application samples contained 0.9 µg/kg and the 0.10 mg/L sprinkler- and furrow-application samples contained 1.7 and 2.9 µg/kg of simazine, respectively.

The extraction of simazine from cucumber samples, which were weighed and homogenized with the peel intact, was complicated by the formation of heavy emulsions before filtration. The test samples were, however, analyzed with no interference. The only simazine residue found was 0.3 µg/kg in the samples taken 30 days after the sprinkler application of 0.01 mg/L. However, only two of four replicates showed any residue and the chromatogram peak heights on which the calculations were based were 2 and 3 mm.

Homogenization of alfalfa samples before extraction of simazine residues was complicated by the fibrous nature of the plant material and the dense pigmentation of the extract. Table II shows that some of the untreated alfalfa check samples contained simazine. Because of the specificity of the detection system to nitrogen and the high

Table II. Simazine Residues ( $\mu\text{g}/\text{kg}$ ) in Field and Vegetable Crops after Irrigation with Water Containing Simazine

treatment and rate, ppm	tomatoes	cucumbers	alfalfa	sugar beets		corn		pinto beans	
				roots	foliage	grain	foliage	bean + pods	foliage
7 Days after Treatment									
sprinkler									
0.0	0	0	$0.2 \pm 0.08^a$	0	0	0	0	0	$0.7 \pm 0.34$
0.01	0	0	$3.8 \pm 0.07$	0	$5.1 \pm 0.73$	0	$0.6 \pm 0.17$	0	0
0.10	0	0	$6.4 \pm 0.17$	$1.2 \pm 0.25$	$5.0 \pm 0.51$	0	$2.5 \pm 0.33$	0	0
furrow									
0.0	0	0	$1.3 \pm 0.24$	0	0	0	0	0	$0.6 \pm 0.29$
0.01	0	0	$0.1 \pm 0.04$	0	$0.2 \pm 0.11$	0	0	0	0
0.10	0	0	$5.1 \pm 0.63$	$0.7 \pm 0.21$	$2.6 \pm 0.50$	0	$0.8 \pm 0.06$	0	0
At Harvest 30 Days after Treatment									
sprinkler									
0.0	0	0	0	0	0	0	0	0	0
0.01	$0.9 \pm 0.37$	$0.3 \pm 0.09$	$1.0 \pm 0.22$	$0.6 \pm 0.19$	$2.8 \pm 0.28$	0	0	0	0
0.10	$1.7 \pm 0.40$	0	$2.1 \pm 0.24$	0	$1.5 \pm 0.15$	0	$0.7 \pm 0.10$	0	0
furrow									
0.0	0	0	$1.6 \pm 0.22$	0	$0.8 \pm 0.39$	0	0	0	$4.5 \pm 2.25$
0.01	0	0	$0.6 \pm 0.11$	$0.7 \pm 0.21$	$0.8 \pm 0.16$	0	0	0	0
0.10	$2.9 \pm 0.22$	0	$0.6 \pm 0.11$	0	$0.9 \pm 0.15$	0	0	0	0

<sup>a</sup> Standard error (four replicates).

nitrogen content of the alfalfa, we felt that re-fractionation and re-analysis were in order. In most instances, the second analysis confirmed the original values. However, at this level of sensitivity, the heights of the simazine peaks from the untreated samples ranged from 1 to 11 mm, and proportionately small variations resulted in large differences in calculated simazine levels. Two of four replicates from the 7-day sampling of untreated sprinkler-irrigated plots contained 1.4 and 0.6  $\mu\text{g}/\text{kg}$  of residue as calculated from chromatogram peaks of 4 and 2 mm, respectively. Re-fractionation and analysis indicated no simazine residue. The apparently nonuniform contamination of these two plots could have resulted from wind drift. In both cases, the plot was located directly downwind (prevailing westerly flow) from a 0.10 mg/L treatment. The two sets of values were averaged to result in the 0.2  $\mu\text{g}/\text{kg}$  of residue shown in Table II. With the 7-day sampling of untreated furrow-irrigated alfalfa plots, it was found that three of four replicates contained simazine. Amounts ranged from 0.34  $\mu\text{g}/\text{kg}$  (1 mm peak height) to 2.0  $\mu\text{g}/\text{kg}$  (6 mm peak height). Re-fractionation and analysis resulted in residue levels ranging from 0.88  $\mu\text{g}/\text{kg}$  to 1.89  $\mu\text{g}/\text{kg}$ . As noted with the sprinkler-irrigated control plots, it would appear that contamination was nonuniform among plants within the plots. Averaging the two sets of values produced the reported 1.3  $\mu\text{g}/\text{kg}$  of residue shown in Table II. Analysis of 30-day plant samples taken from the same control plots indicated that only those subject to furrow irrigation still contained herbicide residue. Simazine is known to be absorbed mostly through plant roots with little foliar absorption (Gysin and Knusli, 1960), indicating that residue found in 7-day sprinkler-irrigated samples was probably surface contamination which was removed with subsequent sprinkling. Amounts of simazine in the four replicates ranged from 0.67 to 3.3  $\mu\text{g}/\text{kg}$  based on chromatogram peak heights of 2 to 11 mm. Re-fractionation and analysis eliminated or reduced the residues detected, again indicating the possibility that only a few plants in each plot contained simazine. The two sets of values were averaged and a residue level of 1.6  $\mu\text{g}/\text{kg}$  resulted. The explanation for this apparent contamination of some plants within the untreated furrow-irrigated plots is less obvious than that for those subject to sprinkler irrigation. The land slopes at a rate of 3.2% along an axis which runs diagonally through the plot area. In nearly every instance where simazine was found in untreated samples, the plot was

within 3 m and downslope of a 0.10 mg/L treatment. These factors, together with a nonuniform hardpan layer at varying depths throughout the area, could have resulted in some subsurface channeling of simazine-treated water.

Simazine was found in alfalfa samples from plots treated at both the 0.01 and 0.10 mg/L rates. In most instances, the levels detected declined from 7 to 30 days. The largest amounts of simazine were found in alfalfa collected 7 days after sprinkler treatment in which 3.8 and 6.4  $\mu\text{g}/\text{kg}$  were found in the 0.01 and 0.10 mg/L treated plots, respectively.

Sugar beets represented two separate commodity groups, root crops and leafy vegetables. No simazine was detected in root samples of any of the untreated plots. Simazine levels of 1.2 and 0.7  $\mu\text{g}/\text{kg}$  were detected in root samples from sprinkler- and furrow-irrigated plots, respectively, collected 7 days after treatment at the 0.10 mg/L rate. No simazine was found in 7-day samples from plots treated at the 0.01 mg/L rate or in 30-day samples from plots treated at the 0.10 mg/L rate. Residue levels of 0.6  $\mu\text{g}/\text{kg}$  and 0.7  $\mu\text{g}/\text{kg}$  were found in root samples from sprinkler- and furrow-irrigated plots, respectively, collected 30 days after treatment at the 0.01 mg/L rate.

Simazine content was higher in sugar beet foliage samples than in root samples, particularly with sprinkler irrigation. Regardless of treatment rate, 7-day sprinkler-irrigated samples contained nearly identical amounts of simazine (5.1 and 5.0  $\mu\text{g}/\text{kg}$  for the 0.01 and 0.10 mg/L rates, respectively). After 30 days, simazine levels in sugar beet foliage from sprinkler-irrigated plots had declined to 2.8  $\mu\text{g}/\text{kg}$  for the 0.01 mg/L treatment rate and 1.5  $\mu\text{g}/\text{kg}$  for the 0.10 mg/L treatment rate. The highest level of simazine found in foliage samples from furrow-irrigated plants was 2.6  $\mu\text{g}/\text{kg}$  in 7-day samples from plots treated at the 0.10 mg/L rate. The untreated plants were free of simazine residue, except for one 30-day sample from a furrow-irrigated plot. This plot was adjacent to (within 3 m) and slightly down-slope of a 0.10 mg/L treatment rate and, as with alfalfa, some subsurface water movement might have occurred.

The recovery of simazine from fortified corn was 89% for both grain and foliage. Corn grain was the most difficult of the crops to extract. Portions of the cob were included with the grain to simulate its possible use as cattle feed. Heavy emulsions formed after shaking, and they separated only after gentle agitation of the separatory funnel for 20–30 min. After filtration through an alu-

minum oxide column, however, the extract appeared clear and was analyzed with no interference on the chromatograms. No simazine was detected in any of the corn grain samples.

Some samples of corn foliage contained simazine. The pattern of its occurrence was similar to that noted with alfalfa and sugar beet foliage: simazine levels were higher in the sprinkler-irrigated samples than in the furrow-irrigated samples, and the levels detected decreased from 7 to 30 days. With the 0.01 mg/L application rate, simazine (0.6  $\mu\text{g}/\text{kg}$ ) was found only in samples from the sprinkler-irrigated plots taken 7 days after treatment; the 30-day samples from the same plots contained no simazine residue. With the 0.10 mg/L treatment rate, the highest level of simazine (2.5  $\mu\text{g}/\text{kg}$ ) was found in the 7-day samples from sprinkler-irrigated plots, and this level dropped to 0.7  $\mu\text{g}/\text{kg}$  at 30 days. Foliage from the corresponding furrow-irrigated plots contained simazine at 0.8  $\mu\text{g}/\text{kg}$  in 7-day samples but none in 30-day samples.

Pinto bean pods and foliage were sampled separately. No trace of simazine was detected in any of the pod samples. The same was true of the foliage samples, except for three from untreated plots. One of four replicate 7-day samples from the sprinkler- and furrow-irrigated control plots and one of four 30-day samples from the furrow-irrigated control plots contained simazine. Samples were re-fractionated and analyzed and the original residue levels were confirmed. As with alfalfa, the contaminated sprinkler-irrigated plot was directly downwind from a plot treated at the 0.10 mg/L rate. Also, the lack of residue at 30 days indicates surface contamination removed with subsequent irrigation. The furrow-irrigated control plot presents a different picture in that the amount of residue increased significantly from 7 to 30 days. Again, as with alfalfa and sugarbeet foliage, the contaminated plot was adjacent to (within 3 m) and downslope of a 0.10 mg/L treated plot. It would appear that some subsurface lateral movement and subsequent accumulation occurred.

In summary, no simazine residue was found in corn grain and pinto bean pods while trace amounts were found in pinto bean foliage and cucumbers. Amounts ranging from 0.6 to 2.9  $\mu\text{g}/\text{kg}$  were found in sugar beets, corn foliage, and tomatoes. Sugar beet foliage collected 7 days after the application of simazine at 0.01 and 0.10 mg/L by sprinkler irrigation contained 5  $\mu\text{g}/\text{kg}$  of residue. Alfalfa contained the most residue of the crops tested. Samples collected 7 days following treatment from plots sprinkler-irrigated with water containing 0.10 mg/L simazine contained 6.4

$\mu\text{g}/\text{kg}$  of residue. This amount declined to 2.1  $\mu\text{g}/\text{kg}$  30 days following treatment.

In the related simazine water residue study (Anderson et al., 1978), the amount of herbicide found in flowing canal water immediately following application to one bank did not exceed 60  $\mu\text{g}/\text{L}$ . Residue levels detected in first flow water samples collected 4 to 6 months after application, peaked at 250  $\mu\text{g}/\text{L}$  within the treated section but decreased rapidly to less than 5  $\mu\text{g}/\text{L}$ .

Climatic as well as edaphic factors would be expected to influence simazine accumulation in crops. However, the data gathered in this study indicate that at concentrations up to 0.10 mg/L, the possibility for significant simazine accumulation in a variety of crops is minimal. Further, it appears that with sufficient downstream dissipation or wasting of first flow irrigation water, the exposure of crops to simazine levels in excess of this amount following ditchbank spraying of irrigation channels for weed control purposes would be remote.

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