Table III. Percent Recovery of Chlorinated Phenols in Urine—Subdivided Samples

day of sampling[a]	% recovery		
	2,4,6-trichlorophenol	2,3,5,6-tetrachlorophenol	pentachlorophenol
0	85	89	91
6	83	89	90
17	79	86	87
26	81	87	89
36	83	83	85

[a] Duplicate analysis.

Table IV. Actual Levels of Biologically Incorporated Chlorinated Phenols in Urine—Subdivided Samples

day of sampling[a]	level, ng/mL			
	2,5-dichlorophenol	2,4,5-trichlorophenol	2,3,4,6-tetrachlorophenol	pentachlorophenol
0	51	3	18	31
5	54	3	20	27
13	47	4	16	26
34	50	2	18	30

[a] Duplicate analysis.

Table III lists results from first void, fresh human urine fortified at the same levels as sample 1, but which were subdivided into 5-mL sample aliquots and frozen. Individual aliquots were removed from the freezer, thawed, and analyzed on the designated days. The remaining subsamples were left in the freezer. This sample handling technique was applied to urine containing biologically incorporated chlorophenols, and the results are shown in Table IV.

These experiments demonstrate the importance of proper sampling and storage of urine samples when analyzing for chlorophenols over an extended period of time. Our laboratory now subdivides all urine samples into 5-mL aliquots when first receiving the sample so that repetitive analysis can be performed at later times. Little loss is note when the urine samples are subdivided in this manner before freezing and subsequently analyzed over a 40-day period.

CONCLUSION

A storage technique for the assurance of quantifiable analytical results for chlorinated phenols in urine has been presented. Conventional thawing, refreezing, and rethawing of an entire urine sample exhibited as much as a 40% decrease in the levels of chlorinated phenols in fortified and biologically incurred samples. Urine samples that were subdivided into individual sample bottles and kept frozen until analysis exhibited little or no decomposition over a 40-day period.

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2-Chloro-*N*-[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)aminocarbonyl]benzenesulfonamide, a New Herbicide

2-Chloro-*N*-[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)aminocarbonyl]benzenesulfonamide (DPX 4189), a novel chemical compound, controls a wide spectrum of weeds in greenhouse studies. It is especially effective as a selective herbicide at extremely low rates in both preemergence and postemergence treatments of broadleaf and certain grass weeds commonly found in a number of cereal crops. Data demonstrating this activity and selectivity are presented.

Novel *N*-(1,3,5-triazinylaminocarbonyl)benzenesulfonamides were recently reported to be highly herbidical by Levitt (1978) and Finnerty et al. (1979). In greenhouse studies, one of these compounds, 2-chloro-*N*-[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)aminocarbonyl]benzenesulfonamide (DPX 4189), provided control of a large number of weed species. It is particularly useful at low rates of application in both preemergence and postemer-

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Table I. Postemergence Applications of DPX 4189

	% control or injury					
rate, g/ha	velvetleaf[a]	annual morning glory[b]	cocklebur[c]	mustard[d]	jimsonweed[e]	wheat
1	100	90	80	100	50	0
2	95	90	100	100	70	0
4	100	100	80	100	60	0
8	100	100	100	100	90	0
60	100	100	100	100	100	30
120	100	100	100	100	95	60
0	0	0	0	0	0	0

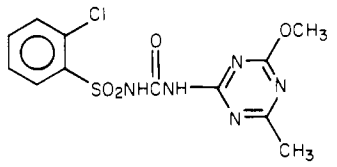
[a] *Abutilon* sp. [b] *Ipomoea* sp. [c] *Xanthium* sp. [d] *Brassica* sp. [e] *Datwia* sp.

Table II. Preemergence Applications of DPX 4189

	% control or injury								
rate, g/ha	mustard[a]	cocklebur[b]	velvetleaf[c]	annual morning glory[d]	giant foxtail[e]	cheatgrass[f]	wild oats[g]	crabgrass[h]	wheat
2	80	30	80	85	40	0	0	0	0
4	90	60	70	80	60	0	0	30	0
8	100	60	80	90	60	30	0	50	0
16	100	80	90	90	60	60	0	50	0
32	100	80	95	95	80	60	0	60	0
64	100	80	100	90	80	60	0	70	0
125	100		100	95	95	65	20	70	20
0	0	0	0	0	0	0	0	0	0

[a] *Brassica* sp. [b] *Xanthium* sp. [c] *Abutilon* sp. [d] *Ipomoea* sp. [e] *Setaria* sp. [f] *Bromus* sp. [g] *Avena sp.* [h] *Digitaria* sp.

gence treatments of weeds associated with cereal crops such as wheat, oats, and barley. This communication reports the synthesis, herbicidal efficacy, crop tolerance, and mammalian toxicity of this novel herbicide.



Chemical Methods. DPX 4189 was synthesized by adding an equivalent of 2-chlorobenzenesulfonyl isocyanate, prepared according to the method of Ulrich and Sayigh (1966), to a suspension of 2-amino-4-methoxy-6-methyl-1,3,5-triazine (Huffman and Schaeffer, 1963) in acetonitrile. After being stirred for 2–16 h at room temperature, the mixture is filtered and the precipitate thus obtained is washed with ethyl ether to yield the desired product, melting at 174–178 °C. Nuclear magnetic resonance, infrared absorption spectra, and elemental analysis were consistent with the proposed structure. The compound is moderately soluble in methylene chloride, less soluble in acetone and acetonitrile, and of very low solubility in hydrocarbon solvents. Its solubility in water is 125 ppm. The sodium salt of DPX 4189 has a solubility of 5–10% in water.

Toxicology. The experimental methods used in the toxicity studies have been described by Sherman and Kaplan (1975). DPX 4189 has a low order of acute oral toxicity to rats. Its LD_{50} for fasted male rats is 5545 mg/kg and for fasted female rats 6293 and mg/kg.

Exposure of the rabbit eye to the chemical as a solid produced very mild temporary conjunctival irritation with no other effect. DPX 4189 was not an irritant or a sensitizer when applied to shaved, intact guinea pig skin at 30 percent and 3% concentrations in an inert carrier. DPX 4189 was not mutagenic in the *Salmonella*/microsome assay in strains TA 1535, TA 1537, TA 1538, TA 98, and TA 100 either in the presence or in the absence of an activation system using the methods of Ames et al. (1975).

The feeding of male and female rats for 3 months at dietary levels of 0, 100, 500, and 5000 ppm produced a decreased rate of body weight gain only in females at 5000 ppm, and slight hematologic and other clinical laboratory effects were observed at 5000 ppm, the only level so evaluated; however, no gross or microscopic pathology finding was attributed to DPX 4189.

Biological Methods. The biological activity of DPX 4189 was delineated in a number of greenhouse tests. These tests are described and the data resulting from them are shown below. The numbers presented are visual observations of percent control (chlorosis, necrosis, or retardation). Complete control is indicated by 100 and no effect is indicated by 0.

Test Procedure I. Wheat (Redcoat) in the three-leaf stage and a number of weed species, 10 cm in height, growing in Fallsington silt loam soil were sprayed overall with DPX 4189 dissolved in a solution containing 95% acetone, 0.2% poly(oxyethylene) (20), sorbitan monolaurate (Tween 20), and water. Percent weed control and the degree of retardation or other injury exhibited by the wheat evaluated 24 days after treatment are shown in Table I.

Test Procedure II. Wheat and a number of weed species planted in Fallsington silt loam soil were treated with DPX 4189 (dissolved as in test procedure I) preemergence as a surface spray. Weed control ratings and the response of wheat taken 28 days after application are presented in Table II.

Test Procedure III. DPX 4189, dissolved as in test procedure I, was applied as an overall spray on preemergence and postemergence plantings in Fallsington silt loam soil. The postemergence plantings included wheat (same variety) in the two- to three-leaf stage, barley (Besbar) in the one- to two-leaf stage, and several weed species (4 cm tall) that are known to infest wheat and barley cultures. Treated plants and controls were maintained for 4 weeks and ratings were then taken. These are shown in Table III.

Table III. Postemergence and Preemergence Applications of DPX 4189

		% control or injury							
application	rate, g/ha	wheat	barley	black grass[a]	kochia[b]	false chamomile[c]	wild mustard[d]	dog fennel[e]	wild buckwheat[f]
postemergence	16	0				100	100	90	100
postemergence	31	0	0	80	100	100	100	100	100
postemergence	62	0	0	90	100	100	100	100	100
postemergence	0	0	0	0	0	0	0	0	0
preemergence	16	0				100	90	90	90
preemergence	31	0	30	80	90	100	90	100	100
preemergence	62	10	40	90	100	100	100	100	100
preemergence	0	0	0	0	0	0	0	0	0

[a] *Alopecurus myosuroides.* [b] *Kochia scoparia.* [c] *Matricaria inodora.* [d] *Brassica* sp. [e] *Eupatorium* sp. [f] *Polygonum convolvulus.*

Table IV. Postemergence Treatment of Water Hyacinth[b]

	% chlorotic or necrotic tissue	
rate, g/ha	10 days	45 days
8	30[a]	100
0	0	0

[a] Plant growth strongly retarded. [b] These data indicate that DPX 4189 may be useful for the control of water hyacinth, an aquatic plant that infests many bodies of water in tropical and subtropical areas. This pattern of activity (rapid growth retardation followed by chlorosis and gradual decline) should permit control with minimal side effects on the ecosystem.

Test Procedure IV. DPX 4189, dissolved as in test procedure I, was applied as an overall spray to small ponds containing water hyacinth (*Eichornia crassipes*) plants typically 18 cm tall with five leaves per plant. Treated plants and controls were maintained in a greenhouse, and visual observations, which were taken 10 and 45 days after application, are presented in Table IV.

The data from the above-described tests were an early indication that DPX 4189 held promise for selective weed control in cereal crops such as wheat and barley, especially when applied postemergence. This compound provides control of a large number of weeds as either a pre- or postemergence treatment. Injury symptons are somewhat slow to develop, and a typical effect of DPX 4189 is almost complete inhibition of plant growth frequently followed by chlorosis and death. These data indicate weed control at extremely low application rates under greenhouse conditions. In field tests, DPX 4189 is effective against weeds at application rates considered extremely low.

Subsequent greenhouse and field tests have contributed much to our knowledge of this novel herbicide. The results of these studies will be reported in future publications.

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Constituents of Mustard, Goldenrod, and Croton—Three Host Plants of the Tarnished Plant Bug

The volatile compounds of three plants, mustard [*Brassica juncea* (L.)], goldenrod (*Solidago nemoralis* Ait.), and croton (*Croton capitatus* Michx.), were examined. Though all three are hosts of the *Lygus* bug, there is little similarity in the compounds found in the volatile extracts.

The tarnished plant bug, *Lygus lineolaris* (Palisot de Beauvois), is a pest of cotton, *Gossypium hirsutum* L., and many other crops. Although cotton is damaged by the *Lygus*, it is not a preferred host. Mustard, *Brassica juncea* (L.) Czern. E Coss., for instance, will attract *Lygus* to cotton fields when the mustard is interplanted with cotton. As a result, such interplanting is used to attract *Lygus* to experimental cotton lines that are to be assessed for resistance to *Lygus* (Laster and Meredith, 1974). During the growing season, *Lygus* are found on many hosts, perhaps 20–25 varied species of plants. In late summer and early fall, croton, *Croton capitatus* Michx., and goldenrod, *Solidago nemoralis* Ait., are hosts, as well as domesticated mustard plants, though other weeds are also hosts at this time of year.

Although it was expected that vast differences in volatile