

spherical particles as well as by scantness of hexane-extractable lipid from the powders that had been put up in a desiccator with silica gel immediately after spray-drying. The microcapsules thus obtained are so resistant to oxidative deterioration during a long-term storage at various A_w that they can be applied to food processing as additives fortified with polyunsaturated lipid. The practical application remains to be further investigated.

ABBREVIATIONS

A_w , water activity; SEM, scanning electron microscopy; TCA, trichloroacetic acid.

Registry No. Linoleic acid, 60-33-3; palmitic acid, 57-10-3; starch, 9005-25-8.

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Pyrazinyl-Substituted 1,3-Cycloalkanediones and Related Compounds: Synthesis and Pesticidal Properties

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2-(2-Pyrazinyl)-1,3-cyclohexanediones with 3-methyl substituents on pyrazine show significant post- and preemergence herbicidal activity, particularly against grass species, and are also active against the adult two-spotted spider mite. While 5- or 6-methyl substituents alone did not impart mite activity, 5-methylpyrazine substitution significantly enhanced activity against grasses. 3,5,6-Trimethyl substitution effected the maximum toxicity against both grass species and adult mites. In the 2-(3,5,6-trimethylpyrazinyl) dione series mite activity appeared to increase with increasing size of the dione C-5 alkyl substituent. No acaricidal or herbicidal activity was observed for 2-(4-pyrimidinyl)-, 2-(3-pyridazinyl)-, or 2-(*N*-methyl-2-imidazolyl)dimedones. Introduction of a 4-methyl group into the pyrimidine ring, however, caused a significant increase in herbicidal activity, particularly against broadleaf species.

2-(2-Pyridinyl)-1,3-cyclohexanediones show marked broad-spectrum herbicidal activity (Manning et al., 1988) without activity on mites or insects, in contrast to appropriately substituted 2-phenyl-1,3-cyclohexanediones and their enol esters, which show selective toxicity to grass species and are also active acaricides, against both adults and eggs (Wheeler, 1980). The two dione classes differ strikingly in their relationships between aryl ring substituents and activity. Whereas the herbicidal 2-pyridinyl diones appear to require the ability to form an internally

hydrogen-bonded N--HO— chelate ring, permitted by essential coplanarity of the pyridine ring and the enolic dione C=C (Manning et al., 1988), a phenyl ortho substituent precluding such coplanarity is a general requirement for both herbicidal and acaricidal activity in the 2-phenyl-1,3-cyclodione series (Wheeler, 1980). Thus, it was of interest to synthesize and test 1,3-cyclohexanediones with pyrazine and other diazines attached at the dione 2-position.

METHODOLOGY

Synthesis. Attachment of diazine rings to the 2-position of 1,3-cyclohexanediones was accomplished by initial synthesis of appropriate 6-diazinyl-5-oxohexanoic acid esters via condensation of lithiated methyl diazines with 3-substituted glutaric esters. Cyclization of the 5-oxohexanoate esters gave the diones, all steps analogous to

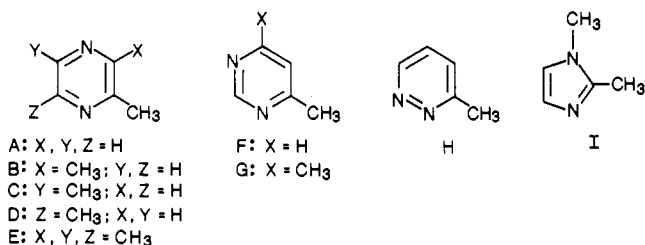
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Table I. pK_a Determinations for 2-Diazinyldimedones

no.	R	pK_a	
		det in 70:30 EtOH-H ₂ O	extrapolated to 100% H ₂ O
1		7.7	6.3
4		7.6	6.2
12		8.6	7.3
13		8.9	7.6
14		8.9	7.6
7		6.3	4.8

those for preparation of the 2-(2-pyridinyl) diones (Manning et al., 1988).



Pyrazines with mono- (A), di- (B-D), and tetramethyl (E) substitution, 4-methyl- (F) and 4,6-dimethylpyrimidine (G), and 3-methylpyridazine (H) provided the dione diazine moieties, and the regioequivalence of methyl groups in all cases precluded isomer formation. A single dimethylimidazole I was derivatized, lithiation occurring at the C-2 methyl. Scheme I depicts the preparation of dione 7 and enol ester 8 from 2,3,5,6-tetramethylpyridazine.

Postemergent Herbicidal Evaluation. The test plants were morningglory (*Ipomoea purpurea*), black nightshade (*Solanum nigrum*), teaweed (*Sida spinosa*), velvetleaf (*Abutilon theophrasti*), jimsonweed (*Datura stramonium*), wild oats (*Avena fatua*), crabgrass (*Digitaria sanguinalis*), giant foxtail (*Setaria faberi*), annual ryegrass (*Lolium multiflorum*), and seedling johnsongrass (*Sorghum halepense*). Test chemicals were dissolved in 50:50 (v/v) acetone-water and applied at 100 gal/acre. The soil and developing plants were sprayed about 2 weeks after the seeds were sown at rates of 8, 1, and 0.5 lb/acre as indicated in the tables. Postemergence activity, as estimated percent control, is reported in Tables II and III.

Preemergent Herbicidal Evaluation. Test solutions of approximately 1% by weight of experimental compound were prepared in acetone. Seeds of the above-described plants and soil were sprayed with the test solutions, immediately after planting, at the rate of 8 lb/acre of soil surface, except where otherwise indicated (Table III). Approximately 3 weeks after spray application, the herbicidal activity of the compound was determined by visual

observation of the treated areas in comparison with untreated controls. These observations are reported in Tables II and III as percent control of plant growth.

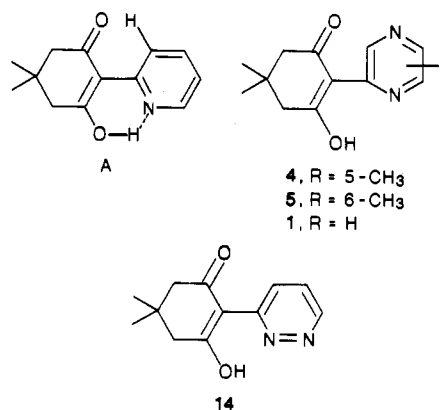
Mite Foliage Spray Tests. Suspensions of the test compounds were prepared by dissolving 1 g of compound in 50 mL of acetone in which 0.1 g of an (alkylphenoxy)-ethanol surfactant had been dissolved. The resulting solution was mixed into 160 mL of water to give approximately 200 mL of a stock suspension. Adults and nymphal stages of the two-spotted spider mite (*Tetranychus urticae* (Koch)) were the test organisms. Infested leaves from a stock culture were placed on the primary leaves of two bean plants 6-8 in. in height, growing in a 2.5-in. clay pot. A total of 150-200 mites transferred from the excised leaves to the fresh plants in a period of 24 h. Following the 24-h transfer period, the excised leaves were removed from the infested plants. The test compounds were formulated by diluting the stock suspensions with water to provide the desired concentrations (ppm). The potted plants (one pot per compound) were placed on a revolving turntable and sprayed with 100-110 mL of test compound formulation with a DeVilbis spray gun at 40 psig air pressure. The sprayed plants were held at 80 ± 5 °F and 50 ± 5% relative humidity for 4 days after which a mortality count of motile forms was made on the test plants.

Mite Ovicide Test. Heavily infested leaves from a stock culture were placed on the primary leaves of two bean plants 6-8 in. in height, growing in a 2.5-in. clay pot. Females were allowed to oviposit for a period of 48 h and the leaves of the infested plants then dipped into an aqueous solution of 800 ppm tetramethyl pyrophosphate in order to destroy the reproductive forms and thus prevent further egg laying. The test compounds were applied as described above and the sprayed plants held at 80 ± 5 °F and 50 ± 5% relative humidity for 4 days, after which a microscopic examination was made of unhatched (dead) and hatched (living) eggs.

RESULTS AND DISCUSSION

Physical Properties and Spectroscopic Evidence.

In a related study the herbicidally active 2-(2-pyridinyl)-1,3-cyclohexanediones A gave evidence of internal hydrogen bonding manifested by (a) a downfield



shift of the 3-pyridinyl proton and (b) the inability to titrate these compounds to an inflection end point (Manning et al., 1988). Among the diazinediones examined, only the two methylpyrazines 4 and 5 and the pyridazine 14 showed isolated protons shifted to the 9-10 ppm region. The unmethylated pyrazine 1 and both pyrimidinyl diones were without this effect. A decreased degree of intramolecular hydrogen bonding in the diazinediones is further indicated by the titratability of all compounds examined, as shown in Table I. That some degree of internal H bonding is present is suggested by the increased acidity

Table II

no.	structure	herbicide act, % control (8 lbs. AI/acre)				acaricidal activity ^c	
		broadleaves ^a		grasses ^b		adults	eggs
		post	pre	post	pre		
1		41	0	36	12	i	i
2		34	10	66	0	i	i
3		60	0	70	0	i	i
4		37	14	82	85	i	i
5		16	0	22	0	i	i
6		64	36	91	86	(500)	(320)
7		21	62	89	98	(40)	(500)
8		19	14	100	83	(7)	(500)
9		50	40	100	100	(10)	(500)
10		46	4	100	80	(70)	(92)
11		6	8	96	100	(3)	(270)
12		6	34	0	0	i	i
13		85	63	37	8	i	i
14		22	0	8	0	i	i
15 ^d		0	0	0	0	i	i

^a Average for morningglory, velvetleaf, black nightshade, teaweed, and jimsonweed. ^b Average for giant foxtail, large crabgrass, wild oats, annual rye, and seedling johnsongrass. ^c i = inactive. Numbers in parentheses are LD₅₀ values. ^d ¹H NMR (D₂O) δ 3.66 (s, 3 H, NCH₃).

Scheme I

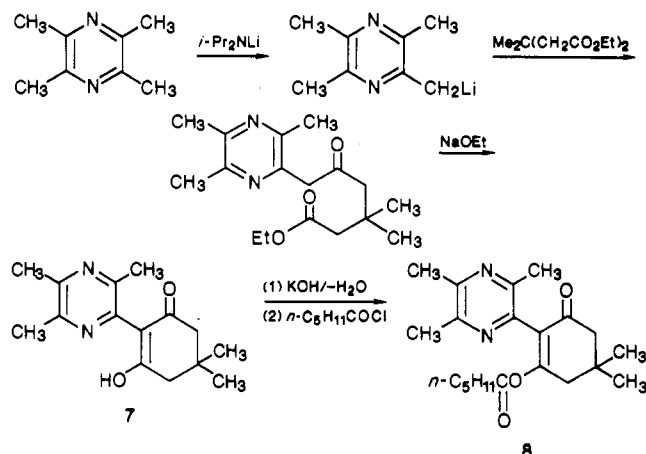
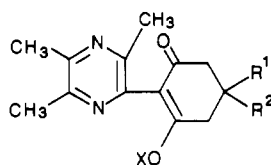


Table III



no.	X	R ¹ , R ²	% control of grasses ^a			
			postemer- gence		preemer- gence	
			1.0 ^b	0.5	1.0	0.5
8	COC ₆ H ₁₁	Me, Me	76	49	7	0
9	H	<i>i</i> -Pr, H	58	31	22	4
10	COC ₆ H ₁₁	<i>i</i> -Pr, H	0		0	
11	H	-(CH ₂) ₄ -	50	12	26	9

^a Average for giant foxtail, large crabgrass, wild oats, annual rye, and seedling johnsongrass. ^b Pounds/acre.

of the pyrazinyl dione 7, in which steric interaction of the 3-methyl group and dione carbonyl tends to rotate the rings out of coplanarity and disrupts the "neutralizing" chelate ring. The diminished hydrogen bonding in the diazinyl diones is evidently a reflection of the lower basicities of diazines (first protonation) relative to pyridine as indicated by the reported conjugate acid pK_a values for pyridine (5.58), pyrazine (0.6), pyrimidine (1.31), and pyridazine (2.33) (Buckingham, 1982).

Structure-Activity Relationships. Table II describes the herbicidal and acaricidal activity of 14 2-diazinyl-1,3-

cyclodiones and enol esters and an imidazolyl dione. In Table III the grass herbicidal activity of four of the more active trimethylpyrazine compounds at lower rates is presented.

Pyrazinyl Derivatives. The herbicidal activity of the pyrazinyl diones is intermediate in type between that of the 2-(2-pyridinyl) diones (Manning et al., 1988) and the 2-phenylcyclohexanediones (Wheeler, 1980). Thus, certain pyrazinyl compounds (2-4, 6, 7) resembled 2-phenyl diones in grass activity while also showing broadleaf activity. 2-(2-Pyrazinyl)-1,3-cyclohexanediones with 3-methyl substituents on pyrazine show significant post- and pre-emergence herbicidal activity, particularly against grass species. Pyrazine substituted in the 5-position with a methyl group gave significantly enhanced activity against grasses (compare 4 and 1) whereas a methyl group at the 6-position alone had little effect (compound 5). 3,5,6-Trimethyl substitution effected the maximum toxicity against both grass species and adult mites. Enol esterification of the normethyl pyrazine 1 increased post-emergence grass activity, but this effect was not seen on esterification of the 3,5,6-trimethylpyrazinyl diones. A possible explanation for this relates to the observation that 2-pyrazinyl-1,3-cyclohexanediones resemble the 2-phenyl-1,3-cyclohexanediones, and not the 2-(2-pyridinyl) diones, in structure-activity relationships. In both the 2-pyrazinyl and 2-phenyl series, development of grass and mite toxicity requires the presence of a substituent adjacent to the ring linkage (pyrazine 3-position), the effect of which is to twist the pyrazine (or phenyl) and dione rings out of coplanarity. In the 3,5,6-trimethylpyrazines (7, 9, 11) this steric effect is achieved by the 3-methyl substituent whereas in the normethylpyrazine 1 enol esterification provides a degree of interference with free rotation, resembling the 3-methylation effect.

As with phytotoxicity, a methyl group at the 3-position of the pyrazine moiety initiated mite activity, and this was enhanced by addition of methyls at positions 5 and 6. In the 3,5,6-trimethylpyrazinyl dione series adult mite activity appeared to increase with increasing size of the C-5 alkyl substituent of the dione.

No acaricidal activity was seen with the pyrimidines, pyridazine, and imidazole, and both the unsubstituted pyrimidine (12) and pyridazine (14) had little activity on plants. A dramatic increase in herbicidal activity, particularly against broadleaf species, occurred, however, on introduction of a 4-methyl group into the pyrimidine ring (see 13). The *N*-methylimidazolyl derivative 15 was completely inactive.

Table IV

no.	mp, °C	analysis					
		calculated			found		
		C	H	N	C	H	N
1	125.5-127.5	66.04	6.47	12.84	65.93	6.59	12.78
2	49.5-51.5	66.65	6.99	9.71	66.61	7.02	9.63
3	oil	69.74	8.19	8.13	69.71	8.33	7.64
4	125-127.5	67.22	6.94	12.06	67.25	6.94	11.96
5	111-112.5	67.22	6.94	12.06	67.36	6.95	11.89
6	75.5-77.5	67.53	7.33	9.26	67.84	7.35	9.35
7	154-155.5	69.20	7.74	10.76	68.95	7.79	10.81
8	oil	70.36	8.43	7.81	71.30	7.96	8.20
9	112-130 dec ^a	67.53	7.33	9.26	67.84	7.35	9.35
10	oil	70.94	8.66	7.52	70.48	8.87	7.27
11	128.5-131.5	71.30	7.74	9.78	70.85	7.57	9.75
12	176-178	66.04	6.47	12.84	65.91	6.47	12.91
13	143.5-145.5	67.22	6.94	12.06	67.18	6.90	12.13
14	148.5-150.5	66.04	6.47	12.84	65.62	6.39	12.81
15	257-260	56.14	6.67	10.91	56.30	6.53	11.02

^a Hydrate.

EXPERIMENTAL CHEMISTRY SECTION

Melting points are uncorrected. ^1H NMR spectra were obtained with a Varian EM-360A spectrometer using Me_4Si as an internal standard. Infrared spectra were recorded on a Perkin-Elmer 197 or Beckman Acculab 2 spectrometer. Elemental analyses were performed by the Union Carbide Analytical Group at the South Charleston, WV, Technical Center. Potentiometric pH measurements were performed on a Beckman Zeromatic pH meter equipped with manual temperature control, fiber junction calomel reference, and glass silver-silver chloride electrodes. The reference electrode contained saturated aqueous potassium chloride solution. The instrument was standardized with commercial aqueous buffer systems. pK_a values were determined by titration in 70% ethanol-30% water (v/v) and extrapolated to 100% aqueous values by graphical relations to values determined in aqueous solutions containing decreasing concentrations of ethanol.

Special Starting Materials. 2,3-Dimethylpyrazine was prepared by the method of Rizzi (1968) from 2,3-dimethyl-5,6-dihydropyrazine, in turn prepared by the procedure of Ishiguro and Matsumura (1958).

Ethyl 3,3-Dimethyl-5-oxo-6-(2-pyrazinyl)hexanoate. To a stirred solution of dry diisopropylamine (60.7 g, 0.6 mol) in 600 mL of dry THF was added a 1.6 M solution of *n*-butyllithium in hexane (375 mL, 0.6 mol) over a 23-min period with cooling to -15°C , after which 56.5 g (0.6 mol) of 2-methylpyrazine were added over 7 min, at -12 to -15°C , giving a dark red solution containing a precipitate. After the mixture was stirred for 27 min, the lithium salt slurry was transferred, portionwise, to a stirred solution of 389.3 g (1.8 mol) of diethyl 3,3-dimethylglutarate in 760 mL of dry tetrahydrofuran, over a 2.3-h period, while being cooled at -78°C . The mixture was allowed to warm to 0 – 2°C at which temperature stirring was continued for about 18 h. Water (200 mL) was then added followed by a stream of gaseous CO_2 until the pH dropped from 12.5 to 8. Tetrahydrofuran was flashed off under reduced pressure and the residue slurred with ether, washing the ether layer with water (2 \times) and then 6 N HCl (4 \times). The acid extract was ether extracted (2 \times) and adjusted to pH 11, causing separation of the crude product. The latter was extracted with ether and the ether solution filtered, washed with saturated NaCl solution, and dried over MgSO_4 . Evaporation of ether and vacuum distillation gave 62.4 g of product (39.3% yield): 132–180 $^\circ\text{C}$ (0.10–0.05 mm) (Kugelrohr air temperature); IR (smear) 1722 cm^{-1} (ester C=O); ^1H NMR (CDCl_3) δ 0.85–1.45 (m, 9 H, 3 CH_3), 2.31–2.86 (m, 4 H, CH_2CCH_2), 3.92–4.41 (m, 4 H, CH_2O and CH_2CO), 8.30–8.69 (m, 3 H, aromatic H).

5,5-Dimethyl-2-(2-pyrazinyl)-1,3-cyclohexanedione (1). A sodium ethoxide solution was prepared from 4.95 g (0.215 mol) of sodium and 300 mL of anhydrous ethanol and heated under reflux while adding 56.92 g (0.215 mol) of ethyl 3,3-dimethyl-5-oxo-6-(2-pyrazinyl)hexanoate over a 13-min period. Refluxing was continued for 2.5 h and ethanol then vacuum flashed, giving a yellow glassy residue. This was dissolved in 100 mL of H_2O , the solution extracted with ether (3 \times), and the pH of the aqueous phase

adjusted to 5, causing a yellow solid to separate. The latter dissolved upon CH_2Cl_2 extraction, and the aqueous-phase pH was readjusted to 5, by adding HCl, causing more precipitate to form. The solid was again extracted into CH_2Cl_2 , and the pH adjustment and CH_2Cl_2 extraction sequence was then repeated several times until no further product precipitated at pH 5. The combined organic extracts were dried (MgSO_4) and vacuum stripped, giving a yellow solid that was crystallized from ethyl acetate to give 32.41 g (69.1% yield) of crystals: mp 125.5–127.5 $^\circ\text{C}$; IR (KBr) 2860–2955, 1630, 1618, 1535, 1469 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.14 (s, 6 H, CH_3CCH_3), 2.52 (s, 4 H, CH_2CCH_2), 8.15–8.56 (m, 3 H, aromatic H), 10.21 (s, 1 H, exch). Anal. Calcd. for $\text{C}_{12}\text{H}_{14}\text{N}_2\text{O}_2$: C, 66.04; H, 6.46; N, 12.83. Found: C, 65.93; H, 6.59; N, 12.78. Conversion of the dione to its potassium salt followed the procedure for the analogous pyridinyl dione (Manning et al., 1987).

5,5-Dimethyl-3-(octanoyloxy)-2-(2-pyrazinyl)cyclohex-2-enone (3). Octanoyl chloride (6.41 g, 0.039 mol) was fed at room temperature, over 5 min, to a stirred slurry of 5,5-dimethyl-2-(2-pyrazinyl)-1,3-cyclohexanedione potassium salt (10.0 g, 0.039 mol) and 1 drop of dicyclohexyl-18-crown-6 in 150 mL of dry tetrahydrofuran. The mixture was stirred for approximately 23 h and then evaporated to dryness under reduced pressure. The residue was stirred with ether and the ether extract washed quickly, in succession, with cold 0.25 N NaOH (2 \times), 7% aqueous Et_3N , and saturated aqueous NaCl. The ether solution was dried (MgSO_4), filtered, and evaporated free of solvent, giving 11.2 g (83.2% yield) of orange oily product: IR (smear) 1755 (ester C=O), 1640–1670 (conj C=O and C=C), 1135, 1090 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.70–1.60 (m, 19 H, 3 CH_3 plus 5 CH_2), 2.08–2.77 (m, 6 H, CH_2CCH_2 plus CH_2CO_2), 8.43–8.73 (m, 3 H, aromatic H). Anal. Calcd for $\text{C}_{20}\text{H}_{28}\text{N}_2\text{O}_3$: C, 69.74; H, 8.19; N, 8.13. Found: C, 69.71; H, 8.33; N, 7.64.

ACKNOWLEDGMENT

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Registry No. 1, 111087-54-8; 1-K⁺, 111087-70-8; 2, 111087-55-9; 3, 111087-56-0; 4, 111087-57-1; 5, 111087-58-2; 6, 111087-59-3; 7, 111087-60-6; 8, 111087-61-7; 9, 111087-62-8; 10, 111087-63-9; 11, 111087-64-0; 12, 111087-65-1; 13, 111087-66-2; 14, 111087-67-3; 15, 111087-68-4; $\text{NH}(\text{Pr}-i)_2$, 108-18-9; $(\text{EtO}_2\text{CH}_2)\text{CMe}_2$, 17804-59-0; $\text{Me}(\text{CH}_2)_6\text{COCl}$, 111-64-8; ethyl 3,3-dimethyl-5-oxo-6-(2-pyrazinyl)hexanoate, 111087-69-5; 2-methylpyrazine, 109-08-0.

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