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Aziridine Analogues of Some Synthetic Pyrethroids

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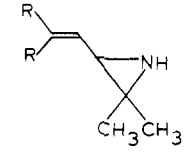
The aziridines 2,2-dimethyl-3-(2-methylpropenyl)aziridine and 2,2-dimethyl-3-(2,2-dichlorovinyl)aziridine were synthesized. These aziridines were coupled with chloroformates prepared from 3-phenoxybenzyl alcohol, 5-benzyl-3-furylmethyl alcohol, and allethrolone. These couplings yielded aziridine analogues (C-1 carbon in the cyclopropane ring has been replaced with a nitrogen atom) of resemethrin, allethrin, phenothrin, NRDC 134, and permethrin. Other aziridinecarbamates were prepared from a variety of chloroformates. Preliminary insecticidal tests on these compounds have been performed. A general mortality rate of less than 10% at the 500-ppm level was found for the majority of the compounds synthesized. A notable exception is the compound containing the 5-benzyl-3-furylmethyl alcohol moiety, which had a mortality rate of 100% at the 500-ppm level in a number of insects.

Recent development in new synthetic pyrethroids have been primarily concerned with the synthesis of either photostabilized alcohol moieties or the replacement of the isobutenyl methyl groups of the acid moiety by functions that are resistant to oxidative degradation (Soderlund and Casida, 1977). There has been very little work reported on structural or compositional variations of the cyclopropane ring with the exception of the acyclic compounds such as in fenvalerate (Ohno et al., 1974). A compositional variation that we have been concerned with recently is the replacement of the C-1 carbon in the cyclopropane ring with a nitrogen atom to form an aziridinecarbamate linkage between the "acid" and alcohol components of the pyrethroid. It has been established that the configuration of the C-1 carbon in the cyclopropane ring must be *R* for insecticidal activity; the *S* configuration confers little or no toxicity (Elliott and Janes, 1973). Replacement of the C-1 carbon with a nitrogen would remove this aspect of configuration; however, the ease of inversion about the nitrogen atom in the aziridine ring system (Acheson, 1976) would allow the molecule to possess, part of the time, the same molecular conformation as that of a pyrethroid having the *R* configuration at the C-1 carbon. Casida and Berteau (1969) reported the synthesis of 5-benzyl-3-furylmethyl 2,2,3,3-tetramethylaziridine-1-carboxylate. Toxicity test data revealed a housefly topical LD_{50} of 13 mg/kg with synergist and 228 mg/kg without synergist. Sammes and Rahman (1972) synthesized methyl and ethyl 2,2-dimethyl-3-(2-methylpropenyl)aziridine-1-carboxylates by means of photochemical decomposition of azidoformates in the presence of 2,5-dimethyl-2,4-hexadiene but did not extend their studies by employing alcohol moieties that would be expected to produce analogues of known active pyrethroids. No additional pyrethroid-like compounds containing the aziridine ring in lieu of the cyclopropane ring have been found in the literature. We wish to report the synthesis and certain toxicity data for compounds obtained from the coupling of 2,2-dimethyl-3-(2-methylpropenyl)aziridine (IV) and 2,2-dimethyl-3-(2,2-



IV R = CH_3
V R = Cl

dichlorovinyl)aziridine (V) with various chloroformates. Initial insecticidal test data have been obtained for the aziridine analogues of resmethrin, allethrin, phenothrin, NRDC 134, and permethrin (Table I). The aziridine moieties were also coupled with chloroformates prepared from *o*-, *m*-, and *p*-cresols, sesamol, phenol, benzyl alcohol, and piperonyl alcohol. Insect toxicity for most of these analogues was low with a general mortality rate of less than 10% at 500 ppm.

EXPERIMENTAL SECTION

Infrared spectra were determined with a Beckman IR-33. ^1H NMR spectra were recorded by using a Hitachi Perkin-Elmer R-24B NMR spectrometer. A Hitachi Perkin-Elmer RMU-6E mass spectrometer was used in obtaining the mass spectra. The photolysis was conducted with a Hanovia, No. 7825-36, mercury-vapor lamp of medium

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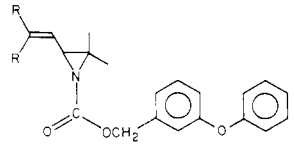
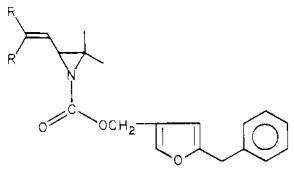
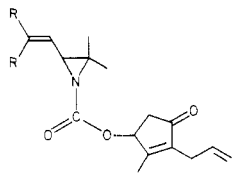
Table I. Toxicity of Aziridine Analogues of Synthetic Pyrethroids

compd structure	R	compd no.	ppm	% mortality (24 h) HF[a]	CRW[a]	SAW[a]	MBB[a]
	CH_3	VI	500	10		0	0
			100	0		0	0
			10		40		
	CL	XI	500	0		0	0
			100	0		0	0
	CH_3	VIII	500	0		0	0
			100	0		0	0
			10		70		
	CL	IX	500	100		100	100
			100	80		20	0
	CH_3	VII	500	10		0	0
			100	0		0	0
			10		20		
	CL	X	500	0		0	0
			100	0		0	0

[a] HF = housefly; SAW = southern armyworm; MBB = mexican bean beetle; CRW = corn footworm.

pressure. Allethrolone was obtained from Roussel UCLAF and 2-Benzyl-4-(hydroxymethyl)furan from S. B. Penick and Co. Where analyses are indicated only by symbols of elements, analytical results were within ±0.4% of the theoretical values.

General Procedure for the Preparation of Chloroformates. Phosgene was condensed into diethyl ether cooled to 0 °C in an ice bath. To this stirring solution the alcohol and dimethyl aniline in diethyl ether were added. The mixture was stirred for 3 h in the ice bath and then washed with 2 × 100 mL of water, 3 × 75 mL of 3% hydrochloric acid, and 1 × 50 mL of saturated sodium chloride solution. The diethyl ether was dried with potassium carbonate.

3-Phenoxybenzyl Chloroformate (I). Phosgene, 28.0 g, and 34.0 g of 3-phenoxybenzyl alcohol were combined with 30 g of dimethylaniline according to the general procedure given above. The diethyl ether was removed to give 40.5 g of a clear oil for a 95% yield: NMR (CCl_4) δ 7.0 (9 H, m) and 5.1 (2 H, s); IR 1790 cm^{-1} (carbonyl).

Allethrolone Chloroformate (II). Five grams each of phosgene, allethrolone, and dimethyl aniline was combined according to the general procedure. After removal of the ether, 4.6 g of a yellow oil was obtained for a 65.2% yield. The NMR was complex but similar to that of allethrolone except for the absence of an exchangeable proton at 4.4 ppm: IR 1800 (carbonyl) and 1735 cm^{-1} (carbonyl).

5-Benzyl-3-furylmethyl Chloroformate (III). Five grams each of phosgene, 2-benzyl-4-(hydroxymethyl)furan and dimethylaniline was combined according to the procedure given. Removal of the ether afforded 3.9 g of product for a 58.5% yield: NMR (CCl_4) δ 6.95 (5 H, s), 5.75 (1 H, s), 4.8 (2 H, s), 4.7 (1 H, s), and 3.7 (2 H, s); IR 1775 cm^{-1} (carbonyl).

3-(2-Methylpropenyl)-2,2-dimethylaziridine (IV). A solution of methylazidoformate, 33 g, and 250 mL of 2,5-dimethyl-2,4-hexaadiene was illuminated for 4–6 h with a mercury-vapor lamp. The photolysis reaction was monitored by the disappearence of the azide band at 2150 cm^{-1}. The methyl aziridinecarboxylate was distilled from the reaction mixture (73 °C; 0.2 mmHg). Forty grams was obtained. The carboxylate, 16 g, was heated to reflux for 24 h in a solution of potassium hydroxide, 7.5 mL of water, and 300 mL of methanol. The mixture was diluted with 700 mL of water and extracted with petroleum ether. Removal of petroleum either gave 8.8 g of crude product. Vacuum distillation (90 °C; 10 mmHg) gave 5 g of a clear oil for a 46% yield: NMR (CCl_4) δ 4.8 (1 H, d, J = 8 Hz), 3.15 (1 H, s, exchangeable), 2.2 (1 H, d, J = 8 Hz), 1.7 (6 H, s), 1.2 (3 H, s), and 1.1 (3 H, s); IR 3280 cm^{-1} (N–H br band); mass spectrum m/e (rel intensity) 125 (M^+, 37), 110 (100), 95 (30), and 85 (95).

3-(2,2-Dichlorovinyl)-2,2-dimethylaziridine (V). 1,1-Dichloro-4-methyl-1,3-pentadiene was treated with *m*-chloroperbenzoic acid, sodium azide, and triphenylphosphine in a manner similar to the method of Ittah et al. (1978). The product was obtained in a 35.5% overall yield: NMR (CCl_4) δ 5.5 (1 H, d, J = 8 Hz), 2.4 (1 H, d, J = 8 Hz), 1.25 (3 H, s), 1.1 (3 H, s), and 0.8 (1 H, s, exchangeable); IR 3240 cm^{-1} (N–H br band).

General Procedure for Aziridinecarboxylates. Triethylamine and the appropriate aziridine were combined in 25 mL of diethyl ether. The aziridine solution was added to a chloroformate dissolved in 75 mL of diethyl ether at 0 °C. The mixture was stirred in an ice bath for 90 min and then 3 h at room temperature. *Workup Method A.* The diethyl ether solution was washed with 5 × 50 mL fractions of deionized water with the pH adjusted to 8 with sodium carbonate. The ether was dried with potassium carbonate. For analytical determinations, sample were dissolved in hexane and passed through a column composed of 60% Celite and 40% Norit A. *Workup Method B.* The diethyl ether was washed with 3 × 100 mL of 3% hydrochloric acid and 2 × 100 mL of 5% sodium bicarbonate. For analytical determinations, samples were dissolved in a 9:1 hexane–ether mixture and passed through a column of silica gel.

3-Phenoxybenzyl 3-(2-Methylpropenyl)-2,2-dimethylaziridine-1-carboxylate (VI). Triethylamine, 1.5 g, and 1.5 g of aziridine IV were combined with 2.5 g of chloroformate I according to the general procedure and workup A. The ether was removed to give 3.4 g of product for a 96.7% yield: NMR (CCl_4) δ 7.2–6.6 (9 H, m), 4.9 (1 H, d, J = 8 Hz), 2.75 (1 H, d, J = 8 Hz), 1.7 (6 H, s) m 1.2 (3 H, s), and 1.15 (3 H, s); IR 1720 cm^{-1} (carbonyl). Anal. $C_{22}H_{25}NO_3$: C, H, and N.

Allethrolone 3-(2-Methylpropenyl)-2,2-dimethyl-

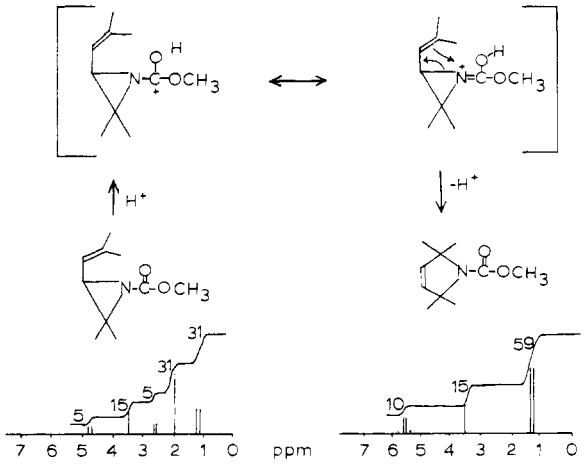


Figure 1. Scheme for the rearrangement of methyl 2,2-dimethyl-3-(2-methylpropenyl)aziridine-1-carboxylate to methyl Δ^3-2,2,5,5-tetramethylpyrrolinecarboxylate with ^1H NMR spectra.

aziridine-1-carboxylate (VII). Chloroformate II, 2.1 g, and 1.5 g each of aziridine IV and triethylamine were combined according to the general procedure and workup method A. The evaporation of the ether gave 2.9 g of product for a 97.5% yield: NMR (CCl_4) δ 5.45 (1 H, m), 4.8 (4 H, m), 2.2–2.8 (5 H, m), 1.9 (3 H, s), 1.7 (6 H, s), 1.2 (3 H, s), and 1.1 (3 H, s); IR 1760 (carbonyl) and 1740 cm^{-1} (carbonyl). Anal. $C_{18}H_{25}NO_3$: C, H, and N.

5-Benzyl-3-furylmethyl 3-(2-Methylpropenyl)-2,2-dimethylaziridine-1-carboxylate (VIII). Chloroformate III, 2.5 g, and 1.5 g each of triethylamine and aziridine IV were combined by using the general procedure and workup A. The evaporation of the ether gave 3.1 g of product for a 92.2% yield: NMR (CCl_4) δ 7.0 ppm (5 H, s), 5.8 (1 H, s), 4.85 (1 H, d, J = 8 Hz), 4.7 (1 H, s), 4.3 (2 H, s), 3.8 (2 H, s), 2.8 (1 H, d, J = 8 Hz), 1.7 (16 H, s), 1.2 (3 H, s), and 1.15 (3 H, s); IR 1765 cm^{-1} (carbonyl).

5-Benzyl-3-furylmethyl 3-(2,2-Dichlorovinyl)-2,2-dimethylaziridine-1-carboxylate (IX). Chloroformate III, 1.25 g, and 1.0 g each of triethylamine and aziridine V were combined by using the general procedure and workup method B. With the removal of the ether, 1.75 g of product was obtained for a 96% yield: NMR (CCl_4) δ 7.05 (5 H, s), 5.8 (1 H, s), 5.65 (1 H, d, J = 8 Hz), 4.7 (1 H, s), 4.3 (2 H, s), 3.8 (2 H, s), 2.9 (1 H, d, J = 8 Hz), and 1.3 (6 H, s); IR 1735 cm^{-1} (carbonyl). Anal. $C_{19}H_{19}Cl_2NO_3$: C, H, and N.

Allethrolone 3-(2,2-Dichlorovinyl)-2,2-dimethylaziridine-1-carboxylate (X). Chloroformate II, 1.07 g, and 1.0 g each of triethylamine and aziridine V were combined according to the general procedure and workup B. The evaporation of the diethyl ether gave 1.5 g of product for a 91.2% yield: NMR (CCl_4) δ 5.5 (2 H, m), 4.9 (3 H, m), 2.65 (5 H, m), 1.35 (3 H, s), and 1.3 (3 H, s); IR 1720 (carbonyl) and 1710 cm^{-1} (carbonyl).

3-Phenoxybenzyl 3-(2,2-Dichlorovinyl)-2,2-dimethylaziridine-1-carboxylate (XI). One gram each of triethylamine and aziridine V was combined with 1.24 g of chloroformate I according to the general procedure and workup B. Rotary evaporation of the ether gave 1.8 g of product for a 99% yield: NMR (CCl_4) δ 7.1 (9 H, m), 5.65 (1 H, d, J = 8 Hz), 5.0 (2 H, s), 2.95 (1 H, d, J = Hz), and 1.3 (6 H, s). IR 1740 cm^{-1} (carbonyl). Anal. $C_{20}H_{19}Cl_2NO_3$: C, H, and N.

BIOLOGICAL TESTING

The compounds were evaluated by a standard greenhouse insecticide test (Strong, 1976) using housefly (*Musca domestica*), Mexican bean bettle (*Epilachna varivestis*), southern armyworm (*Spodopter ericania*), and corn rootworm (*Diabrotica 12-punctata*). The rates of application were 500 and 100 ppm of active ingredient except in the case of corn rootworm where the test rate was 10 ppm of active ingredient. The biological testing was performed by an independent laboratory.

RESULTS AND DISCUSSION

The photolysis of methylazidoformate with 2,5-dimethyl-2,4-hexadiene, to give methyl 3-(2-methylpropenyl)-2,2-dimethylaziridine-1-carboxylate, was found to proceed with the highest yield when the reaction was conducted without solvent. When solvents such as hexane or cyclohexane were used, little or no product was obtained. The resulting dimethylvinylaziridinecarboxylate, an analogue of methyl chrysanthemate, was found to be very acid sensitive. NMR studies revealed that under even mild conditions this compound rearranged to a Δ^3-pyrroline. NMR data and a possible process for this ring expansion are given in Figure 1. Rearrangement of vinylaziridines to Δ^3-pyrrolines has been reported by Lwowski et al. (1968). The acid sensitivity resulting from the dimethylvinyl substitutent of the methyl carbamate and the carbamates of "pyrethroid" alcohols made purification difficult, since the acidity of even silica gel or Florisil was sufficient to promote the rearrangement. The lack of activity in the dimethylvinyl series may be attributed to this ease of rearrangement. The dichlorovinyl compounds were synthesized in hopes of eliminating this sensitivity to acid, since a deactivated vinyl side chain would be a poorer nucleophile for the ring expansion process. Further, the resulting (dichlorovinyl)aziridine is an obvious analogue of 2,2-dimethyl-3-(2,2-dichlorovinyl)cyclopropanecarboxylic acid (DV-acid), the acid moiety of permethrin, and NRDC-149 (Elliott, 1977). The dihalo intermediates and final products proved to be quite stable to acidic conditions and the purification on silica gel or Florisil was easily accomplished.

Preliminary insecticidal test data (Table I) indicate that among the "pyrethroid" carbamates prepared, only the 5-benzyl-3-furylmethyl aziridinecarboxylates, VIII and IX, have appreciable activity. This follows the same trend seen in synthetic pyrethroids where the esters of 5-benzyl-3-furylmethyl alcohol are usually 2–3 times as potent as those of 3-phenoxybenzyl alcohol (Elliott, 1977). These toxicity test data were obtained without the benefit of a synergist. As seen in the studies of Casida and Berteau (1969), the toxicity of 5-benzyl-3-furylmethyl 2,2,3,3-tetramethylaziridine-1-carboxylate is increased more than 17-fold with the addition of a synergist. If a comparable increase in toxicity in the presence of a synergist is found with the compounds reported herein (e.g., IX), further investigations would be warranted.

The finding of little or no insect toxicity in the aziridine analogues having alcohol moieties other than known active alcohols (e.g., the cresols, phenol, benzyl alcohol, piperonyl alcohol, etc.) would indicate that the insecticidal activity is due to mimicry of the pyrethroid structure and not due to a general toxicity of aziridinecarbamates.

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Measurement of Unfrozen and Free Water in Soy Proteins by Differential Scanning Calorimetry

Dorothy J. Muffett[1] and Harry E. Snyder*[2]

Soy isolates, soy concentrate, soy flour, and ovalbumin were analyzed for amounts of unfrozen water at different total moisture contents. Analysis was by differential scanning calorimetry to determine heats of fusion and integral heats of vaporization. Unfrozen water did increase with increased total water content. When plotted against total moisture (dry basis), the unfrozen water was a linear function of total water above a concentration of 1 g of water/g of solids, and ~10% of added water was unfrozen. Plots of integral heats of vaporization vs. total water gave peaks below the critical moisture content.

Bound water can be defined as the water that remains unfrozen in samples held well below 0 °C (Fennema, 1977). The amounts of bound (unfrozen) water can be analyzed by differential scanning calorimetry (DSC) (Ross, 1978; Hansen, 1976), by differential thermal analysis (DTA) (Bushuk and Mehrotra, 1977), or by nuclear magnetic resonance spectroscopy (NMR) (Kuntz et al., 1969; Hansen, 1976). The amounts of bound water measured by different methods at low total moisture (below 0.3 g of water/g of solids) generally agree well.

Some analyses of bound (unfrozen) water by DSC and DTA show that the amount of bound water increases as the total moisture content increases above the critical moisture content (Ross, 1978; Bushuk and Mehrotra, 1977; Biswas et al., 1975). There are no NMR studies to our knowledge that show comparable increases above 0.5 g of water bound/g of solids. Hansen (1978) using NMR showed that soy isolates increased in unfrozen water up to 0.5 g/g of solids as total water increased, but the same increase was not found for soy concentrate or for ovalbumin. Conceptually, it is difficult to imagine how primary and multilayer water would continue to increase after the bonding sites are saturated or how capillary water would increase after the capillaries are filled.

We chose to investigate by DSC the water-binding relationships of soy protein as total moisture was increased. We found large increases in unfrozen water as total water increased, but we prefer the term unfrozen to bound water until a suitable explanation for this phenomenon is available.

EXPERIMENTAL SECTION

Reagents. Soy protein isolates were from the Ralston Purina Co. Supro 610 is a hydrolyzed isolate that contains 95% protein and has a pH of 6.7 ± 0.1. Edipro A is 92.5% protein and has a pH of 4.6 ± 0.2. Edipro N is 92.5% protein and has a pH of 6.7 ± 0.1.

Promosoy 100, a concentrate with 70% protein and a pH of 7.0 ± 0.1, was from Central Soya Co., Inc. Defatted soy flour, toasted Nutrisoy flour 40, was from Archer Daniels Midland Co. The ovalbumin, salt free and 99% pure, was from Sigma Chemical Co.

Procedures. A Perkin-Elmer differential scanning calorimeter, Model 1B, was used to measure unfreezable water in the protein systems. Each sample, in a volatile sample pan, was cooled to −30 °C and then heated at 20 °C/min to at least 20 °C. Sample size ranged from 2 to 12 mg, depending on moisture content. Samples that contained freezable (free) water produced an endothermic peak, the area of which was measured with a planimeter to determine the total heat of fusion. This was divided by the heat of fusion of pure water, 79.6 cal/g, to determine the weight of freezable water in the sample. This weight was subtracted from the total water to determine the amount of unfreezable water. The total water was determined by drying each sample in its punctured volatile sample pan at 105 °C after completion of the DSC scan.

All weighings were done with a Cahn electrobalance to the nearest 10 μg. The average heat of vaporization of each sample was measured by placing it in a volatile sample pan, sealing, weighing, and then puncturing the lid and placing the sample in a DSC sample holder that had been cooled to 17 °C. The sample was then cooled to 0 °C and scanned to 200 °C at 20 °C/min. All samples produced endothermic peaks, the areas of which gave the total energy needed to vaporize the water. The total energy divided by the weight of water evaporated gave the integral heat of vaporization in calories per gram. Scanning to 200 °C caused all the water to vaporize, and further heating at 105 °C produced no weight change.

Samples that contained less than 40% water were prepared by humidification over distilled water in a vacuum desiccator at room temperature. Water was added directly to produce samples with more than 40% moisture.

RESULTS AND DISCUSSION

Upon measuring the unfrozen water content of Edipro N at different total moisture levels, we found that unfrozen water increased as total water increased. Figure 1 shows

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