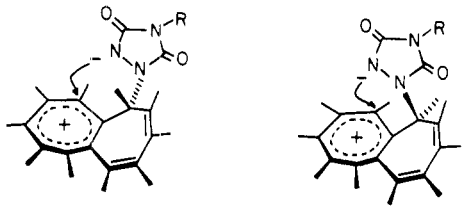
give **9** and **10** followed by rapid closure as indicated. Alternatively, the triazolinedione could be captured stereoselectively from the exo surface to give **9** which cyclizes at a sufficiently slow rate to allow conformational equilibration with **10** to intervene. It is the latter intermediate which serves as precursor to the major product; no interconversion of **6** and **7** has been observed.



9 **10**

Experimental Section

Addition of *N*-Methyltriazolinedione to Heptalene. To a solution of heptalene, prepared from 522 mg (3.34 mmol) of 1,6-dihydroheptalene,[5] in 90 mL of chloroform cooled to −78 °C was added dropwise a solution of *N*-methyltriazolinedione (376 mg, 3.33 mmol) in 25 mL of nitrogen-pruged chloroform. After completion of the addition, the magnetically stirred solution was maintained at room temperature for 1.5 h prior to concentration in vacuo. The residual solid was chromatographed on silica gel (elution with chloroform-ethyl acetate, 9:1) to give 420 mg (47%) of a mixture of **6a** and **7a** as a yellow foam. This material was subjected to preparative TLC on silica gel (elution with ethyl acetate–pentane, 1:1) to separate the isomers.

The high R_f major isomer (**6a**) was obtained as plates, mp 170.5–172.5 °C, after recrystallization from ethanol: IR (KBr) 3002, 1764, 1701, 1466, 1362, 1292, 1251, 1056, 982, 863, 803, 796 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 6.50–6.30 (m, 4 H), 6.15–5.78 (m, 2 H), 5.64–5.34 (m, 2 H), 4.50–4.35 (m, 2 H), 3.02 (s, 3 H); ^{13}C NMR ($CDCl_3$) 155.7, 136.4, 130.8, 129.8, 127.6, 124.9, 58.1, 25.6 ppm; *m/e* calcd 267.1008, obsd 267.1014.

Anal. Calcd for $C_{15}H_{13}N_3O_2$: C, 67.40; H, 4.90; N, 15.72. Found: C, 67.31; H, 5.01; N, 15.58.

The low R_f minor isomer (**7a**) was obtained as needles, mp 198–199 °C, from ethyl acetate–pentane: IR (KBr) 3000, 1765, 1710, 1460, 1387, 1240, 1035, 855, 790, 772, 757, 738, 695, 610 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 6.78–6.59 (m, 4 H), 6.32–5.95 (m, 2 H), 5.60–5.30 (m, 2 H), 4.57–4.38 (m, 2 H), 3.10 (s, 3 H); ^{13}C NMR ($CDCl_3$) 151.6, 131.5, 130.5, 124.8(2C), 123.9, 117.5, 56.6, 25.6 ppm; *m/e* calcd 267.1008, obsd 267.1014.

Anal. Calcd for $C_{15}H_{13}N_3O_2$: C, 67.40; H, 4.90. Found: C, 67.25; H, 4.91.

Addition of *N*-Phenyltriazolinedione to Heptalene. A solution of heptalene, as obtained from 800 mg (5.13 mmol) of the 1,6-dihydro derivative, was treated as above with 630 mg (3.59 mmol) of *N*-phenyltriazolinedione in 50 mL of chloroform. The residue obtained from column chromatography on silica gel (elution with chloroform-ethyl acetate, (1:1) was subjected to preparative TLC on silica gel (same eluent). The R_f 0.5 band was recrystallized from ethanol to give adduct **7b** as off-white microcrystalline needles: mp >310 °C dec; IR (KBr) 3010, 2910, 1770, 1710, 1600, 1505, 1433, 1423, 1272, 1234, 1138, 865, 752, 748, 694, 610 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 7.60–7.23 (m, 5 H), 6.78–6.03 (m, 6 H), 5.73–5.43 (m, 2 H), 4.67–4.50 (m, 2 H); ^{13}C NMR ($CDCl_3$) 150.1 (s), 132.0 (s), 131.5 (d), 130.6 (d), 129.2 (d), 128.2 (d), 125.7 (d), 125.1 (s), 124.9 (d), 123.8 (d), 117.1 (s), 56.9 (d) ppm; *m/e* calcd 329.1164, obsd 329.1172.

Anal. Calcd for $C_{20}H_{15}N_3O_2$: C, 72.93; H, 4.59. Found: C, 72.82; H, 4.63.

The mother liquor from the first crystallization above was recrystallized twice more from ethanol and ethyl acetate–hexane. Each time a small amount of dirty brown sludge was removed. The remaining material was resubjected to TLC as above to finally give **6b** as an amorphous yellow solid: IR (KBr) 3015, 2920, 1770, 1710, 1600, 1503, 1413, 1127, 860, 755, 692, 6.15 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 7.60–7.23 (m, 5 H), 6.62–5.80 (m, 6 H), 5.78–5.48 (m, 2 H), 4.72–4.55 (m, 2 H); ^{13}C NMR ($CDCl_3$) 153.6 (s), 135.8 (s), 131.8 (s), 130.8 (d), 129.8 (d), 129.1 (d), 128.1 (d), 127.8 (s), 127.4 (d), 125.5 (d), 125.0 (d), 58.3 (d) ppm; *m/e* calcd 329.1164, obsd 329.1172.

Addition of (−)-*endo*-Bornyltriazolinedione to Heptalene. A solution of the optically pure triazolinedione in chloroform was added to a solution of heptalene in chloroform in the predescribed manner. The semisolid (80:20 mixture) obtained from preparative TLC was recrystallized twice from ethanol to give major isomer **6c** as pale yellow needles: mp 200–202 °C; IR (KBr) 3025, 2960, 2940, 2475, 1770, 1718, 1420, 1394, 1386, 1308, 1093, 870, 765, 759, 742, 696, 624 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 6.57–5.87 (m, 6 H), 5.73–5.43 (m, 2 H), 4.57–4.40 (m, 2 H), 4.40–4.10 (m, 1 H), 2.67–2.27 (m, 1 H), 1.97–1.50 (m, 6 H), 1.00 (s, 3 H), 0.93 (s, 3 H), 0.88 (s, 3 H); ^{13}C NMR ($CDCl_3$) 156.8, 136.3, 131.5, 130.9, 130.5, 129.8, 127.7, 124.8, 59.4, 58.4, 51.6, 47.8, 45.5, 29.6, 27.1, 26.3, 19.7, 18.8, 14.0 ppm; *m/e* calcd 389.2103, obsd 289.2109.

Anal. Calcd for $C_{24}H_{27}N_3O_2$: C, 74.01; H, 6.99. Found: C, 73.69; H, 7.08.

No attempt was made to purify the minor isomer.

Catalytic Hydrogenation of 6a. A solution of **6a** (117.5 mg, 0.44 mmol) in ethyl acetate containing platinum oxide (102 mg) was hydrogenated in a Parr apparatus at 52 psi for 26 h. At this time, an additional 107 mg of PtO_2 was added and the hydrogenation was continued. Two more portions of PtO_2 (96.6 and 112 mg) were also added over the next 3 days. After a total reaction time of 6 days, the catalyst was separated by filtration through Celite and the filtrate was concentrated. The residual colorless solid was recrystallized from ethyl acetate–hexane and then hexane to give 48.7 mg (40%) of **8** as white needles, mp 122.5–123.5 °C. Very slow recrystallization from hexane at room temperature led to the appearance of a second crystalline form of **8**: thin plates; mp 134.5–135 °C; IR (KBr) 2910, 2900, 2839, 1765, 1710, 1465, 1450, 1395, 1380, 1200, 1175, 1160, 1030, 1020, 930, 765, 760, 735, 685, 640, 588 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 4.25–4.0 (d of t, J = 9, 6 Hz, 2 H), 3.13–2.87 (m with superimposed s, 4 H), 2.34–1.15 (series of m, 17 H); ^{13}C NMR ($CDCl_3$) 154.4, 60.9, 53.8, 40.4, 33.7, 30.3, 28.3, 25.1, 23.9 ppm; *m/e* calcd 277.1790, obsd 277.1799.

Anal. Calcd for $C_{15}H_{23}N_3O_2$: C, 64.95; H, 8.36; N, 15.15. Found: C, 64.89; H, 8.38; N, 14.96.

Acknowledgment. We thank the National Science Foundation (Grant CHE-7900333) for their partial financial support of this research.

Registry No. 6a, 75600-13-4; **6b**, 70528-33-5; **6c**, 75600-14-5; **7a**, 75600-15-6; **7b**, 70528-32-4; **8**, 75600-16-7; heptalene, 257-24-9; *N*-methyltriazolinedione, 13274-43-6; *N*-phenyltriazolinedione, 4233-33-4; (−)-*endo*-bornyltriazolinedione, 73462-83-6.

Bursatellin: A New Diol Dinitrile from the Sea Hare *Bursatella leachii pleii*

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Received August 1, 1980

Sea hares have yielded a fascinating array of natural products, most of which appear to be accumulated from diverse dietary sources.[1] Interest in the chemistry of these opisthobranch molluscs has been stimulated by the reputed toxicity of some species,[1,2] the observation of cytotoxic activity in sea hare extracts,[3,4] and the promise of

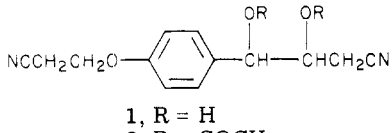
(1) P. J. Scheuer, *Isr. J. Chem.*, **16**, 52 (1977).
(2) B. W. Halstead, "Poisonous and Venomous Marine Animals of the World", Vol. 1–3, U. S. Government Printing Office, Washington, D. C., 1965, 1967, 1970.
(3) F. J. Schmitz, D. P. Michaud, and K. H. Hollenbeak, *J. Org. Chem.*, **45**, 1525 (1980), and references cited therein.

0022-3263/80/1945-5383$01.00/0

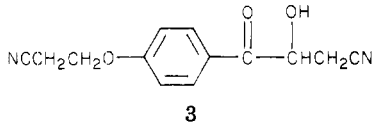
encountering novel products. Prompted by our previous observation[3] that the sea hare *Aplysia dactylomela* contained a wide variety of halogenated and nonhalogenated metabolites, we investigated another species, *Bursatella leachii pleii* (Rang). In contrast to the case of *A. dactylomela*, we have found that extracts of the latter mollusc collected near La Paguera, Puerto Rico, are comprised mostly of aliphatic lipids, but among the minor components we have isolated a novel dinitrile whose structure we report herein.

Specimens of *B. leachii pleii* were dissected and the digestive glands removed. An ethanol extract of the digestive glands, usually the richest source of novel metabolites, was found to contain little other than aliphatic lipid material and sterols. However, from the dichloromethane solubles of a 95% ethanol extract of the remainder of the animal, a fraction exhibiting aromatic proton absorption was obtained upon Sephadex LH-20 chromatography. This fraction was chromatographed over silica gel to yield bursatellin (1) as a colorless glass: $[\alpha]_D$ +12.1°; $C_{13}H_{14}N_2O_3$ (high-resolution mass spectrum). The infrared spectrum of 1 indicated the presence of hydroxyl (3340 cm^{-1}, br) and nitrile groups (2245 cm^{-1}), and ultraviolet absorption at 273 nm (ϵ 946) indicated an aromatic ring. Bursatellin gave a positive periodate test for a 1,2-diol grouping and formed a diacetate (2) whose infrared spectrum possessed nitrile (2260 cm^{-1}) and acetate absorptions (1745 cm^{-1}) but no residual hydroxyl bands. Hence, the third oxygen in 1 was assigned to an ether group.

The ^1H NMR spectrum of 1 contained aromatic proton absorptions at 6.89 and 7.32 ppm (each 2 H, d, 8.7 Hz), indicative of a para-disubstituted benzene moiety, with one substituent likely being an ether oxygen. A pair of two-proton triplets at 2.83 and 4.20 ppm (J = 6.4 Hz) was assigned to adjacent methylene units in a β-oxypropionitrile group. The remaining four one-proton signals were assigned to a 3,4-dihydroxybutyronitrile moiety on the basis of chemical shifts and decoupling: 4.10-ppm multiplet coupled to both a 4.97-ppm doublet (J = 4 Hz) and a pair of one-proton double doublets at 3.63 and 3.71 ppm (J = 11.5, 5.4 Hz for each). The presence of two nitrile groups in 1 is required to account for the four degrees of unsaturation present in addition to those of the aromatic ring. Combination of the above partial structures yields formula 1 for bursatellin.



1, R = H
2, R = $COCH_3$



3

Confirmation of the location of the diol grouping was derived by MnO_2 oxidation of 1 which gave 3 whose IR (1660 cm^{-1}) and UV (λ_{max} 271, ϵ 7700) spectra support the assigned structure, as does its ^1H NMR spectrum: one-proton triplet at 5.64 ppm (J = 4.7 Hz) coupled to a pair of one-proton double doublets at 3.85 and 3.97 ppm (J = 11.5, 4.7 Hz for each). As anticipated, the mass spectra of 1 and 3 show prominent fragment ions at m/e 176 and 174, respectively, corresponding to benzylic cleavage.

The possibility that bursatellin might contain isonitrile groups as have been found[5] in a number of other marine natural products was ruled out on the basis of infrared and ^1H NMR data. Thus, infrared absorption for **1**, **2**, and **3** occurs at higher frequency (2245–2260 cm^{-1}) than is normally observed[5] for isonitriles (2115–2140 cm^{-1}), and the ^1H NMR spectrum of bursatellin does not exhibit the ^{14}N–^1H coupling characteristic of isonitriles.[6]

Experimental Section[7]

Specimens of *Bursatella leachii pleii* (115 animals) collected near La Paguera, Puerto Rico, were dissected and the digestive glands were removed. The animal bodies were stored in ethanol for 3 months. The concentrated ethanol extract was diluted with water and extracted with dichloromethane. A portion (6.5 g) of the dichloromethane solubles (25.5 g) was chromatographed over Sephadex LH-20, using chloroform–methanol (1:1) to give 16 fractions. Fraction 13 (80 mg) was chromatographed over 30 g of TLC-grade silica gel, using chloroform–methanol (19:1) to give nine fractions. Evaporation of fraction 8 gave 15 mg of bursatellin (1) as a colorless glass: $[\alpha]^{25}_D$ +12.1° (*c* 0.45, CH_3OH); IR (Nujol) 3340, 2245 (w) cm^{-1}; UV (CH_3OH) λ_{max} 273 nm (ϵ 946); ^1H NMR (270 MHz, CD_3OD + $CDCl_3$) δ 7.32 (2 H, d, J = 8.7 Hz), 6.89 (2 H, d, J = 8.7 Hz), 4.97 (1 H, d, J = 4 Hz), 4.20 (2 H, t, J = 6.4 Hz), 4.10 (1 H, m), 3.71 (1 H, dd, J = 5.4, 11.5 Hz), 3.63 (1 H, dd, J = 5.4, 11.5 Hz), 2.83 (2 H, t, J = 6.4 Hz); mass spectrum (70 ev), m/e (relative intensity) 246 (M^+, 5), 193 (6), 176 (67), 175 (51), 174 (31), 135 (31), 123 (38), 107 (76), 95 (39), 77 (82), 71 (100); high-resolution mass spectrum, M^+ obsd 246.009, calcd for $C_{13}H_{14}N_2O_3$ 246.100.

Acetylation of Bursatellin (1). Bursatellin (1, 5 mg) was mixed with 0.5 mL of acetic anhydride and 1.0 mL of pyridine at room temperature, and the resulting solution was allowed to stand at 0 °C overnight. A few drops of water was added, and after the reaction mixture had cooled, it was diluted with more water and then extracted with ethyl acetate. The organic layer was washed successively with 1 N HCl, water, 5% $NaHCO_3$, and water. After evaporation of the solvent, 4.8 mg of the diacetate **2** was obtained: oil; IR ($CHCl_3$) 2260, 1745, 1695, 1615, 1515, 1235, 1050 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 7.28 (2 H, d, J = 8.7 Hz), 6.90 (2 H, d, J = 8.7 Hz), 5.92 (1 H, d, J = 7.4 Hz), 4.72 (1 H, m), 4.19 (2 H, t, J = 6.4 Hz), 4.08 (1 H, dd, J = 4.5, 11.4 Hz), 3.88 (1 H, dd, J = 5.4, 11.4 Hz), 2.83 (2 H, t, J = 6.4 Hz), 2.12 (3 H, s), 2.09 (3 H, s); mass spectrum (12 ev), m/e (relative intensity) 303 (M^+ – HCN, 22), 261 (7), 235 (5), 218 (60), 176 (100), 162 (34), 130 (18), 123 (27), 71 (34).

Oxidation of Bursatellin (1). Bursatellin (1, 3 mg) was stirred with 22 mg of MnO_2 in 1.0 mL of acetone at room temperature for 3.5 h. The reaction mixture was filtered, the solvent was evaporated, and the product **3** (1 mg) was isolated by preparative TLC [$CHCl_3$–MeOH (9:1)]: oil; IR (KBr) 3390, 2250, 1660, 1600, 1250 cm^{-1}; UV (CH_3OH) λ_{max} 271 nm (ϵ 7700); ^1H NMR (270 MHz, CD_3OD + $CDCl_3$) δ 8.04 (2 H, d, J = 8.7 Hz), 7.07 (2 H, d, J = 8.7 Hz), 5.64 (1 H, t, J = 4.7 Hz), 4.32 (2 H, t, J = 6.4 Hz), 3.97 (1 H, dd, J = 11.5, 4.7 Hz), 3.85 (1 H, dd, J = 11.5, 4.7 Hz), 2.98 (2 H, t, J = 6.4 Hz); mass spectrum (70 ev), m/e (relative intensity)

(4) G. R. Pettit, C. L. Herald, J. J. Einck, L. D. Vanell, P. Brown, and D. Gust, *J. Org. Chem.*, **43**, 4685 (1978); G. R. Pettit, C. L. Herald, M. S. Allen, R. B. Von Dreele, L. D. Vanell, J. P. Y. Kao, and W. Blake, *J. Am. Chem. Soc.*, **99**, 262 (1977).

(5) See, for example: S. J. Wratten, D. J. Faulkner, K. Hirotsu, and J. Clardy, *Tetrahedron Lett.*, 4345 (1978); B. Di Blasio, E. Fattorusso, S. Magno, L. Mayol, C. Pedone, C. Santacroce, and D. Sica, *Tetrahedron*, **32**, 473 (1976); J. T. Baker, R. J. Wells, W. E. Oberhansli, and G. B. Hawes, *J. Am. Chem. Soc.*, **98**, 4010 (1976); B. J. Burreson, C. Christophersen, and P. J. Scheuer, *Tetrahedron*, **31**, 2015 (1975); L. Minale, R. Riccio, and G. Sodano, *ibid.*, **30**, 1341 (1974); E. Fattorusso, S. Magno, L. Mayol, C. Santacroce, and D. Sica, *ibid.*, **30**, 3911 (1974); F. Cafieri, E. Fattorusso, S. Magno, C. Santacroce, and D. Sica, *ibid.*, **29**, 4259 (1973).

(6) I. D. Kuntz, Jr., P. von R. Schleyer, and A. Allerhand, *J. Chem. Phys.*, **35**, 1533 (1961).

(7) Infrared spectra were taken on Beckman Acculab 3 and Perkin-Elmer 298 spectrometers. ^1H NMR spectra were obtained on a Bruker 270-MHz instrument in the solvents specified; signals are reported in parts per million (ppm) downfield from internal tetramethylsilane. Mass spectra were taken on Hewlett-Packard 5985A and CEC (Du Pont, Monrovia, CA) 110 spectrometers. A Perkin-Elmer Model 141 polarimeter was used for obtaining optical rotations. Chromatographic adsorbents used were Brinkmann silica gel 60H, TLC-mesh silica.

244 (M^+, 8), 199 (5), 175 (10), 174 (86), 167 (3), 121 (48), 93 (21), 92 (10), 53 (100), 52 (68).

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Registry No. 1, 75347-13-6; 2, 75347-14-7; 3, 75347-15-8.

C-Phosphonoketenimines, Characterization and Synthetic Application to Heterocycles

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In recent years, the synthetic applications of multifunctional heteroallenes[1] have been widely investigated. In spite of extensive developments in the chemistry of modified ketenes and isocyanates,[2] little attention has been paid to the uses of ketenimines.[3] From a synthetic point of view it was of interest to synthesize a ketenimine bearing a phosphoryl group[4] as an eliminatable substituent.

Previously we reported[5] a facile preparation of a *C*-phosphonoketenimine[6] which acts as a novel annelation reagent. In this paper we report further investigation of these reactions and their synthetic utility for the preparation of heterocycles.

The ketenimines **2** were successfully synthesized by the dehydration of diethyl (carbamoylalkyl)phosphonates (**1**) prepared from α-halo amides and diethyl phosphite[7] (Scheme I). The dehydration proceeded smoothly with triphenylphosphine, bromine, and triethylamine in dichloromethane at room temperature.[8] For example, *N*-ethylmethyl(diethylphosphono)ketenimine (**2a**), a stable and colorless liquid, was isolated in 82% yield by distillation. The IR spectra of **2a** and **2b** display the characteristic absorptions at 2030 cm^{-1} arising from C=C=N groups. The ^1H NMR spectrum of **2a** showed two splittings at δ 1.69 (d, J_{HP} = 13.6 Hz) and δ 3.51 (dq, J_{HH} = 7.0 Hz, J_{HP} = 4.8 Hz), which were assignable to the allenic methyl and *N*-methylene protons, respectively. The observations of no change in these coupling constants in the 60- and 100-MHz NMR spectra showed that the splittings are not due to the nitrogen atom[9] but to the long-range

Scheme I

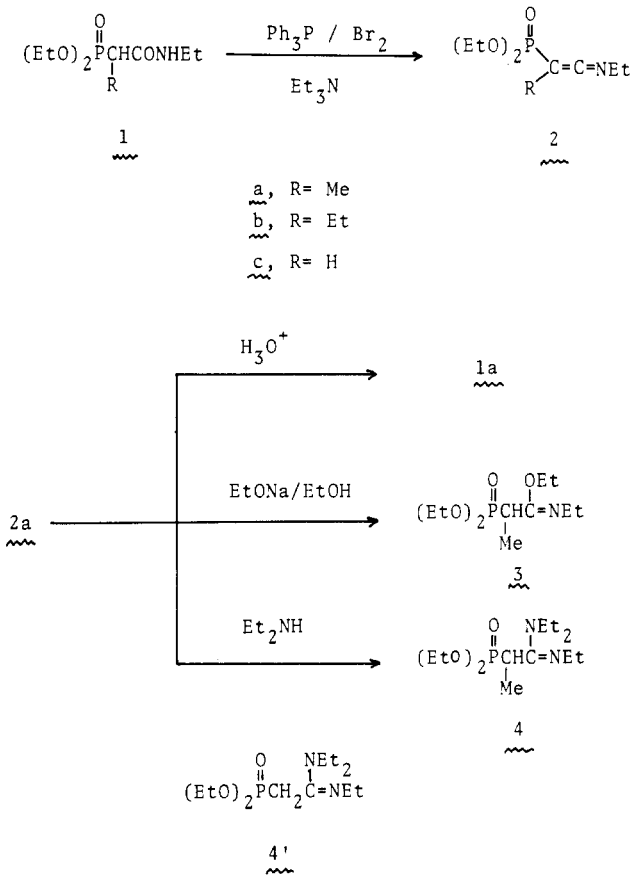
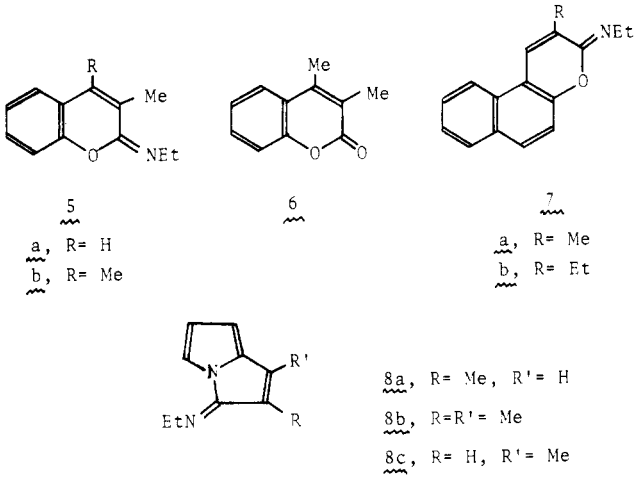


Chart I



(1) For a recent review, see: Reichen, W. *Chem. Rev.* **1978**, *78*, 569.
(2) (a) Ishida, M.; Minami, T.; Agawa, T. *J. Org. Chem.* **1979**, *44*, 2067 and references cited therein. (b) For a recent review, see: Richter, R.; Ulrich, H. "The Chemistry of Cyanates and Their Thio Derivatives"; Patai, S., Ed.; Wiley: New York, 1977: Part 2, p 619.
(3) For a general review, see: Krow, G. R. *Angew. Chem., Int. Ed. Engl.* **1971**, *10*, 435.
(4) A few examples of ketenimines with phosphorus substituents are known: (a) Partos, R. D.; Speziale, A. J. *J. Am. Chem. Soc.* **1965**, *87*, 5068; (b) Foucand, A.; Leblane, R. *Tetrahedron Lett.* **1965**, 509; (c) Bestmann, H. J.; Schmid, G. S. *Angew. Chem.* **1974**, *86*, 274.
(5) Motoyoshiya, J.; Enda, J.; Ohshiro, Y.; Agawa, T. *Chem. Commun.* **1979**, 900.
(6) Recently an alternative preparation of *C*-phosphonoketenimines has been reported: Kolodyazhnyi, O. I.; Yakovlev, V. N. *Zh. Obshch. Khim.* **1980**, *50*, 55; *Chem. Abstr.* **1980**, *92*, 164046.
(7) Diethyl (carbamoylalkyl)phosphonates (**1**) are also prepared in the same yields by the Arbuzov reaction: Speziale, A. J.; Freeman, R. C. *J. Org. Chem.* **1958**, *23*, 1883.
(8) Bestmann, H. J.; Lienert, J.; Mott, L. *Justus Liebigs Ann. Chem.* **1968**, *718*, 24.
(9) Jochims, J. C.; Anet, F. A. L. *J. Am. Chem. Soc.* **1970**, *92*, 5524.

0022-3263/80/1945-5385$01.00/0