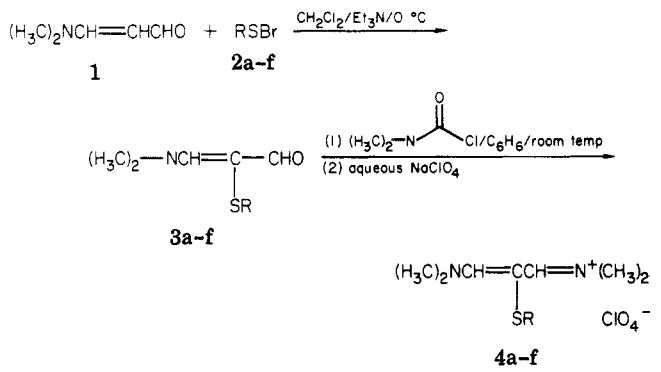
Table I

	3			4		
R	mp, °C	% yield	^{1}H NMR ($CDCl_3$/Me_4Si), δ H_a/H_b	mp, °C	% yield	^{1}H NMR ($CDCl_3$/Me_4Si), δ H_c
CH_3	oil	30.3	7.07/8.96	84.5–85.5	30	7.85
$CH_2CH_2CH_3$	oil	63	7.25/9.08	73–74	57	7.93
C_6H_{11}	oil	10	7.17/8.95	130–132	21	7.90[b]
C_6H_5	90–92	39	7.48/9.11	134–136	33	8.24
4-$CH_3C_6H_4$	112–113	55	7.10/9.17	112–113	12	8.09[b]
$CH_2C_6H_5$	oil	22.6	7.15/9.05	175–177	32	7.78[b]

[a] Satisfactory analyses (±0.4 for C,H,N) were obtained for 3d,3e and 4a-f. NMR and mass spectra (MN^+ peaks) consistent with the desired structure were obtained for all compounds. [b] DMSO-d_6.

Scheme I



a, R = CH_3; b, R = $CH_2CH_2CH_3$; c, R = C_6H_{11}; d, R = C_6H_5; e, R = 4-$CH_3C_6H_4$; f, R = $CH_2C_6H_5$

bromides **2** were conveniently prepared in situ from the appropriate disulfide and bromine. At 0 °C, these sulfenyl bromides were reacted with 3-(dimethylamino)acrolein to give novel 2-(substituted thio)-3-(dimethylamino)acroleins **3** that can be readily converted to 2-(substituted thio) methanaminium salts **4** by stirring with dimethylcarbamyl chloride in benzene for 3 days.[4]

Experimental Section

All melting points were determined on a Thomas-Hoover "Uni-Melt" capillary melting apparatus and are uncorrected. NMR spectra were recorded on either a Varian EM 360A spectrometer at 60 MHz or a Perkin-Elmer R-32 spectrometer at 90 MHz with Me_4Si as an internal standard. Low-resolution mass spectra were recorded on a Finnigan 4023 GC/MS/DS instrument (chemical ionization, methane). Microanalyses were performed by the Analytical Laboratories of The Dow Chemical Co., Midland, MI. No attempt was made to optimize yields.

3-(Dimethylamino)-2-(phenylthio)-2-propenal (3d). Under nitrogen, a CH_2Cl_2 solution of 32.75 g (0.15 mol) of phenyl disulfide was cooled to 0 °C. Bromine, 23.9 g (0.15 mol), was added neat. After 10 min, a CH_2Cl_2 solution of 25 g (0.25 mol) of 3-(dimethylamino)acrolein (Fluka Chemical Co.) and 50 mL of Et_3N was added dropwise while the temperature was held at 0 °C. After being stirred overnight at room temperature, the reaction was shaken with water (200 mL × 2). The CH_2Cl_2 layer was dried (Na_2SO_4) and concentrated to give 49.5 g of crude **3d** as a dark oil. The aldehyde was purified by preparative HPLC (EtOAc) to give 20.2 g (39%) of pure **3d** as light tan crystals: mp 90–92 °C; NMR ($CDCl_3$) δ 3.22 (s, 6 H), 7.13 (s, 5 H), 7.48 (s, 1 H), 9.11 (s, 1 H); mass spectrum, m/z 208 (MH^+).

Anal. Calcd for $C_{11}H_{13}NOS$: C, 63.79; H, 6.33; N, 6.76. Found: C, 63.54; H, 6.41; N, 6.70.

In a similar manner, aldehydes **3a**, **3b**, **3c**, **3e**, and **3f** were prepared.

***N*-(3-(Dimethylamino)-2-(phenylthio)-2-propenylidene)-*N*-methylmethanaminium Perchlorate (4d).** A 17.5-g (0.085-mol) sample of **3d** was mixed with 10.7 g (0.1 mol) of dimethylcarbamyl chloride and 60 mL of benzene. The reaction was stirred at room temperature for 3 days and then extracted with 100 mL of water. The aqueous layer was treated with 15 g of $NaClO_4 \cdot H_2O$. The solid was collected and after recrystallization (1/1 CH_3OH/C_2H_5OH) yielded 9.5 g (33%) of **4d** as a light orange solid: mp 134–136 °C; NMR ($CDCl_3$) δ 3.39 (s, 6 H), 3.48 (s, 6 H), 7.05–7.48 (m, 5 H), 8.24 (s, 2 H); mass spectrum, m/z 235 (M^+).

Anal. Calcd for $C_{13}H_{19}ClN_2O_4S$: C, 46.64; H, 5.72; N, 8.36. Found: C, 47.0; H, 5.82, N, 8.46.

Using a similar procedure, compounds **4a**, **4c**, **4e**, and **4f** were prepared.

Conversion of Perchlorate 4b to the Iodide Salt. A 25.0-g (0.083-mol) sample of the perchlorate salt **4b** was dissolved in CH_2Cl_2 and shaken vigorously with 75 g of KI/200 mL of water (2×). The CH_2Cl_2 layer was dried (Na_2SO_4) and then concentrated to give a white solid. Recrystallization (2-propanol) gave 16.5 g (61%) of the iodide salt as white crystals: mp 168–171 °C; NMR ($CDCl_3$) δ 0.95 (t, 3 H, J = 8 Hz), 1.58 (m, 2 H), 2.48 (t, 2 H, J = 8 Hz), 3.52 (s, 6 H), 3.72 (s, 6 H), 8.88 (s, 2 H); mass spectrum, m/z 201 (M^+).

Anal. Calcd for $C_{10}H_{21}IN_2S$: C, 36.60; H, 6.45; N, 8.53. Found: C, 36.80; H, 6.35; N, 8.35.

Acknowledgment. The author expresses his appreciation to Mr. Robert J. Barbuch and Robert G. Dull for much of the spectral data.

Registry No. **3a**, 90584-65-9; **3b**, 90584-66-0; **3c**, 90584-67-1; **3d**, 74093-76-8; **3e**, 74093-77-9; **3f**, 90584-68-2; **4a**, 90584-70-6; **4b**, 90584-72-8; **4b** iodide salt, 90584-81-9; **4c**, 90584-74-0; **4d**, 90584-76-2; **4e**, 90584-78-4; **4f**, 90584-80-8; $(MeS)_2$, 624-92-0; $(PrS)_2$, 629-19-6; $(c\text{-}HxS)_2$, 2550-40-5; $(PhS)_2$, 882-33-7; $(p\text{-}MeC_6H_4S)_2$, 103-19-5; $(PhCH_2S)_2$, 150-60-7; dimethylcarbamyl chloride, 79-44-7; 3-(dimethylamino)acrolein, 927-63-9.

(3) Cook, G. A. "Enamines: Synthesis, Structure and Reactions"; Marcel Dekker: New York, 1969; pp 148–149.
(4) Arnold, Z. *Collect. Czech. Chem. Commun.* **1974**, *24*, 760.

Self-Condensation of 3*H*-Pyrrol-3-one 1-Oxides

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Received February 29, 1984

Whereas the parent 3-aza- and 3,4,-diazacyclopentadienones are unknown, phenyl-substituted derivatives **1**[1,2] and **2**[3,4] have been detected despite their relative lability

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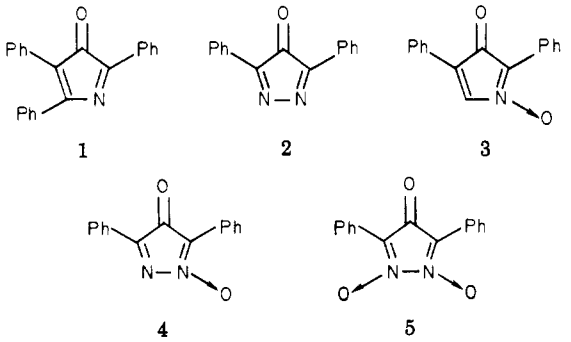


1 2 3 4 5

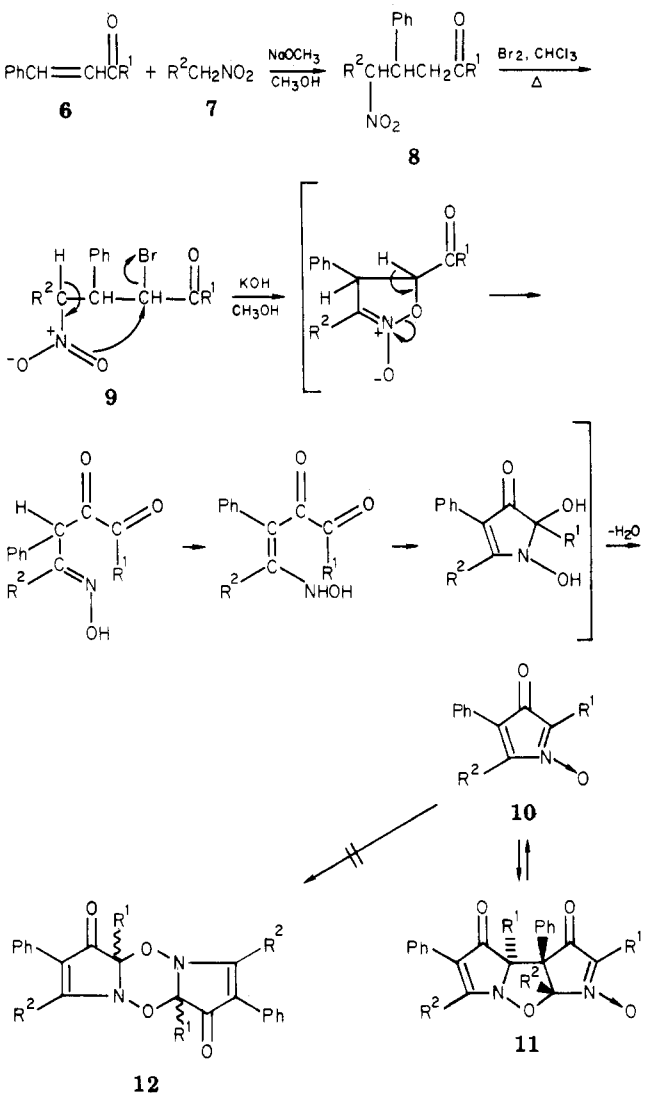
at room temperature. On the other hand, introduction of an *N*-oxide function in these systems increases their stability markedly (e.g., **3**,[5] **4**,[6] and **5**[6]). It had been noted many years before that oxygen substitution in the 3- and 4-positions also stabilizes cyclopentadienones themselves.[7]

The only derivatives of the 3*H*-pyrrol-3-one 1-oxide system that have been reported[5] bear three aryl substituents; phenyl, *p*-bromophenyl, or *p*-chlorophenyl. It was of interest to determine whether the stability of **3** was dependent upon the presence of aryl groups or whether unsubstituted or alkyl-substituted derivatives could be prepared. The only reported route to this ring system is shown in Scheme I.

In this work the alkyl-substituted bromo nitro ketones **9** were prepared first by Michael addition of the nitroalkanes **7** in base to α,β-unsaturated ketones **6**, followed by bromination at the reflux temperature of chloroform. Treatment of **9a**, **9b**, or **9c** with alcoholic base followed by acidification gave mixtures of deep purple and bright yellow products in poor yields. Attempts to separate these mixtures by crystallization resulted in conversion of the purple component to the yellow dimers **11**. In the case of **9d** only traces of the mixture could be obtained. In this case the mixture could not be separated except on thin-layer plates. Dissolution of either component immediately reformed the mixture.

The structure of the dimers **11** was determined from their spectroscopic properties. (See Experimental Section.) For example, the parent peak in the mass spectrum of **11b** is the molecular ion; the $M^+/2$ peak is also strong (48%, M^+). The proton NMR spectra show the presence of two alkyl groups, one on a saturated carbon and one on an unsaturated carbon. Some aliphatic nitrones such as 3,4,5,6-tetrahydropyridine *N*-oxide exist in a head-to-tail dimeric structure analogous to **12**, but such a structure is ruled out in the present case by the NMR data. The proton NMR spectrum of **11b** (R^1 = Ph, R^2 = C_2H_5) is of special significance. A molecular model of **11b** shows that a syn arrangement of R^2 and the phenyl group at C4 places the methyl group of R^2 close to the center of the phenyl group, which should significantly shield it. Indeed the triplet signal for this methyl group appears at δ 0.0 instead of near δ 1.0. The group R^1 is likely to be anti to the phenyl group at C4 to minimize steric repulsion. The regiochemistry of the dimerization of **10** is consistent with the infrared spectrum of **11** which shows two carbonyl absorption bands at 1725 (sh) and 1710 cm^{-1}. Upon heating, the yellow dimers revert to the deep purple compounds.

Scheme I[a]



6 7 8 9 10 11 12

[a] **a**, R^1 = Ph, R^2 = CH_3; **b**, R^1 = Ph; R^2 = C_2H_5; **c**, R^1 = Ph, R^2 = *n*-C_4H_9; **d**, R^1 = $C(CH_3)_3$, R^2 = CH_3.

It is assumed that the purple materials observed are the desired pyrrolone oxides **10**, since both the triphenyl derivative **3** and tetraphenylcyclopentadienone also have deep purple colors. It is evident that replacement of any of the aryl substituents of **3** results in a significant destabilization of this heterocyclic system.

Experimental Section

Melting points were taken on a Fisher-Johns melting point apparatus and are uncorrected. Infrared spectra were recorded on a Perkin-Elmer 398 spectrophotometer using potassium bromide disks or Nujol mulls. ^1H magnetic resonance spectra were taken on a Varian EM 360L NMR spectrometer in $CDCl_3$ with Me_4Si as an internal reference. TLC was carried out on freshly prepared Merck GF_{254} type (60) silica gel plates. Elemental analyses were performed by E. Pascher, Bonn, West Germany. The α,β-unsaturated ketones **6a–c** were prepared according to literature methods.[8,9]

General Procedure for the Preparation of Nitro Ketones[9] 8a–d. A solution of equal molar quantities of the specific unsaturated ketone, the aliphatic nitro compound (nitroethane,

(1) Eicher, T.; Abdesaken, F.; Franke, G.; Weber, J. L. *Tetrahedron Lett.* **1975**, 3915.
(2) Freeman, J. P.; Haddadin, M. J. *J. Org. Chem.* **1980**, *45*, 4898.
(3) Fagan, P. J.; Neidert, E. E.; Nye, M. J.; O'Hare, M. J.; Tang, W. P. *Can. J. Chem.* **1979**, *57*, 904.
(4) Trost, B. M.; Whitman, P. J. *J. Am. Chem. Soc.* **1974**, *96*, 7421.
(5) Kohler, E. P.; Addinall, G. R. *J. Am. Chem. Soc.* **1930**, *52*, 1590.
(6) Freeman, J. P.; Gannon, J. J.; Surbey, D. L. *J. Org. Chem.* **1969**, *34*, 187.
(7) Claisen, L.; Evan, T. *Liebigs Ann. Chem.* **1895**, *284*, 245–299.
(8) (a) Vogel, A. I. "Practical Organic Chemistry", 3rd ed.; Longman, Green and Co.: London, 1956; p 718. (b) Conia, J. M. *Bull. Soc. Chim. Fr.* **1956**, *1392*.
(9) Kohler, E. P. *J. Am. Chem. Soc.* **1924**, *46*, 1733.

1-nitropropane, or 1-nitropentane), and sodium methylate were dissolved in the appropriate volume of methanol. The solution was refluxed for 2–3 h, cooled, and diluted with water. Extraction with ether and evaporation of the dried ether solution gave an oily residue which was triturated with methanol to give a white solid which was recrystallized from methanol. The present method is more convenient than the literature[10,11] methods for preparing nitro ketones **8a,d**.

4-Nitro-1,3-diphenyl-1-pentanone (8a). A solution of benzalacetophenone (4 g, 19 mmol), nitroethane (1.4 g, 18 mmol), and sodium methylate (1.02 g, 19 mmol) in methanol (50 mL) was used: yield 3.25 g (60%); mp 102–104 °C (lit.[11] mp 90–92 °C).

4-Nitro-1,3-diphenyl-1-hexanone (8b). A solution of benzalacetophenone (4 g, 19 mmol), 1-nitropropane (1.69 g, 19 mmol), and sodium methylate (1.02 g, 19 mmol) in methanol (50 mL) was used: yield 2.62 g (46%); mp 157–158 °C (lit.[12] mp 156–158 °C).

4-Nitro-1,3-diphenyl-1-octanone (8c). A solution of benzalacetophenone (10 g, 45 mmol), 1-nitropentane (5.62 g, 48 mmol), and sodium methylate (2.6 g, 48 mmol) in methanol (100 mL) was used: yield 8.59 g (55%); mp 70–71 °C; IR (KBr) 1683, 1545, 1450, 1370, 1240, 764, 750, 702, 688 cm^{-1}; NMR ($CDCl_3$) δ 0.98 (m, 9 H), 3.39 (m, 2 H), 3.94 (m, 1 H), 4.85 (m, 1 H), 7.65 (m, 10 H). Anal. Calcd for $C_{20}H_{23}NO_3$: C, 73.82; H, 7.12; N, 4.32. Found: C, 73.68; H, 7.06; N, 427.

6-Nitro-5-phenyl-2,2-dimethyl-3-heptanone (8d). A solution of 5-phenyl-2,2-dimethyl-4-penten-3-one (**6b**, 3 g, 20 mmol), nitroethane (1.5 g, 20 mmol), and sodium methylate (1.2 g, 20 mmol) in methanol (50 mL) was used: yield 1.35 g (30%); mp 57–59 °C (lit.[10] mp 61–62 °C).

General Procedure for the Preparation of Bromo Nitro Ketones[9] 9a–d. To a solution of the specific nitro ketone in chloroform was added bromine dropwise during refluxing. A drop of acetone was added to initiate the reaction. In cases where the red color of bromine disappeared, more bromine was added until a reddish color persisted. The extent of the reaction was periodically monitored by TLC and refluxing was stopped upon the disappearance of all or most of the starting material. The solvent was evaporated under reduced pressure and the yellow oily residue was triturated with methanol to yield a white solid which was recrystallized from methanol.

4-Nitro-1,3-diphenyl-2-bromo-1-pentanone (9a): 4-nitro-1,3-diphenyl-1-pentanone (**8a**, 2 g, 7 mmol) in chloroform (10 mL) and bromine; reflux time 15 min; yield 1.2 g (67%); mp 154–155 °C; IR (KBr) 1685, 1545, 1448, 1388, 1330, 1280, 805, 740, 680 cm^{-1}; NMR ($CDCl_3$) δ 1.38 (d, 3 H), 3.94 (d of d, 1 H), 5.07 (m, 1 H), 5.98 (d, 1 H), 7.47 (m, 8 H), 8.21 (m, 2 H). Anal. Calcd for $C_{17}H_{16}BrNO_3$: C, 56.37; H, 4.45; N, 3.86; Br, 22.06. Found: C, 56.38; H, 4.49; H, 3.83; Br, 22.27.

4-Nitro-1,3-diphenyl-2-bromo-1-hexanone (9b): 4-nitro-1,3-diphenyl-1-hexanone (**8b**, 1 g, 3.3 mmol) in chloroform (10 mL) and bromine; reflux time 15 min; yield 0.79 g (62%); mp 151–153 °C; IR (KBr) 1680, 1590, 1545, 1448, 1368, 1273, 1225, 740, 695, 682, 662 cm^{-1}; NMR ($CDCl_3$) δ 0.93 (t, 3 H), 1.72 (m, 2 H), 4.06 (d of d, 1 H), 4.84 (m, 1 H), 5.86 (d, 1 H), 7.31 (m, 8 H), 8.33 (m, 2 H). Anal. Calcd for $C_{18}H_{18}BrNO_3$: C, 57.46; H, 4.82; N, 3.72; Br, 21.24. Found: C, 57.46; H, 4.82; N, 3.74; Br, 21.4.

4-Nitro-1,3-diphenyl-2-bromo-1-octanone (9c): 4-nitro-1,3-diphenyl-1-octanone (**8c**, 2 g, 6 mmol) in chloroform (10 mL) and bromine reflux time 2 h; yield 0.74 g (30%); mp 137–138 °C; IR (KBr) 1682, 1550, 1450, 1365, 1277, 810, 700, 685 cm^{-1}; NMR ($CDCl_3$) δ 1.19 (m, 9 H), 3.91 (d of d, 1 H), 5.43 (m, 1 H), 6.06 (d, 1 H), 7.35 (m, 8 H), 7.93 (m, 2 H). Anal. Calcd for $C_{20}H_{22}BrNO_3$: C, 59.39; H, 5.53; N. 3.46; Br, 19.75. Found: C, 59.49; H, 5.46; N, 3.41; N, 3.41; Br, 19.3.

6-Nitro-5-phenyl-4-bromo-2,2-dimethyl-3-heptanone (9d): 6-nitro-5-phenyl-2,2-dimethyl-3-heptanone (**8d**, 0.5 g, 2.16 mmol) in chloroform (10 mL) and bromine; reflux time 2.5 h; yield 0.23 g (34%); mp 145–146 °C; IR (KBr) 2980, 1708, 1500, 1400, 1370, 1350, 1105, 1062, 761, 745, 708 cm^{-1}; NMR ($CDCl_3$) δ 0.99 (s, 9 H), 1.48 (d, 3 H), 3.70 (d of d, 1 H), 5.56 (m, 2 H), 7.42 (s, 5 H). Anal. Calcd for $C_{15}H_{20}BrNO_3$: C, 52.64; H, 5.89; N, 4.09; Br, 23.35. Found: C, 52.55; H, 5.90; N, 4.07; Br, 23.3.

General Procedure for the Preparation of Dihydrodipyrrol[1,2-*b*:3′,2′-*d*]isoxazole-3,4-dione 1-Oxides 11a–c. The specific bromo nitro ketone was treated with methanolic potassium hydroxide (0.5 N). The mixture was magnetically stirred for 30–60 min until all the solid was dissolved. The resulting solution was poured into iced hydrochloric acid and extracted with chloroform, the solvent was evaporated under reduced pressure, and the dark purple oily residue was triturated with ethanol to give a yellow solid which was recrystallized from toluene–ethanol (2:1).

(3a*R,3b*S**,8a*S**)-3a,8a-Dihydro-6,8a-dimethyl-2,3a,3b,5-tetraphenyl-3*H*-dipyrrol[1,2-*b*:3′,2′-*d*]isoxazole-3,4(3b*H*)-dione 1-Oxide (11a).** 4-Nitro-1,3-diphenyl-2-bromo-1-pentanone (**9a**, 1 g, 2.7 mmol) was treated with 5% methanolic potassium hydroxide solution (40 mL): yield 76 mg (10%); mp 195–196 °C; IR (KBr) 1720 (sh), 1710, 1610, 1540, 1490, 1450, 1405, 1387, 1373, 1320, 1200, 870, 690 cm^{-1}; NMR ($CDCl_3$) δ 1.08 (s, 3 H), 2.38 (s, 3 H), 7.37 (m, 16 H), 8.37 (m, 4 H). Anal. Calcd for $C_{34}H_{28}N_2O_4$: C, 77.55; H, 4.98; N, 5.32. Found: C, 77.55; H, 5.08; N, 5.29.

(3a*R,3b*S**,8a*S**)-6,8a-Diethyl-3a,8a-dihydro-2,3a,3b,5-tetraphenyl-3*H*-dipyrrol[1,2-*b*:3′,2′-*d*]isoxazole-3,4(3b*H*)-dione 1-Oxide (11b).** 4-Nitro-1,3-diphenyl-2-bromo-1-hexanone (**9b**, 0.5 g, 1.3 mmol) was treated with 5% methanolic potassium hydroxide (20 mL): yield 40 mg (10%); mp 193–194 sC; IR (KBr) 1720 (sh), 1710, 1610, 1532, 1490, 1450, 1400, 1320, 877, 749, 690 cm^{-1}; NMR ($CDCl_3$) δ 0.00 (t, 3 H), 1.28 (t, 3 H), 2.68 (m, 4 H), 7.36 (m, 16 H), 8.42 (m, 4 H); mass spectrum, *m/e* 554 (M^+, 100); 227 ($M^+/2$, 48). Anal. Calcd for $C_{36}H_{30}N_2O_4$: C, 77.96; H, 5.45; N, 5.05. Found: C, 77.59; H, 5.59; N, 5.01.

(3a*R,3b*S**,8a*S**)-6,8a-Dibutyl-3a,8a-dihydro-2,3a,3b,5-tetraphenyl-3*H*-dipyrrol[1,2-*b*:3′,2′-*d*]isoxazole-3,4(3b*H*)-dione 1-Oxide (11c).** 4-Nitro-1,3-diphenyl-2-bromo-1-octanone (**9c**, 0.5 g, 1 mmol) was treated with 5% methanolic potassium hydroxide (20 mL): yield 40 mg (10%); mp 159–160 °C; IR (KBr) 1725 (sh), 1710, 1630, 1532, 1450, 1395, 1370, 1320, 750, 693 cm^{-1}; NMR ($CDCl_3$) δ 1.5 (m, 18 H), 7.34 (m, 16 H), 8.44 (m, 4 H). Anal. Calcd for $C_{40}H_{38}N_2O_4$: C, 78.66; H, 6.27; N, 4.59. Found: C, 78.50; H, 6.30; N, 4.52.

Registry No. **6** (R^1 = *t*-Bu), 538-44-3; **6** (R^1 = Ph), 94-41-7; **8a**, 6277-76-5; **8b**, 80460-05-5; **8c**, 90552-85-5; **8d**, 90552-86-6; **9a**, 6289-91-4; **9b**, 90552-87-7; **9c**, 90552-88-8; **9d**, 90552-89-9; **10a**, 90552-90-2; **10b**, 90552-91-3; **10c**, 90552-92-4; **11a**, 90552-93-5; **11b**, 90552-94-6; **11c**, 90552-95-7; nitroethane, 79-24-3; 1-nitropropane, 108-03-2; 1-nitropentane, 628-05-7.

(10) Kozlov, L. M.; Fink, E. F. *Tr. Kazan. Khim.-Tehknol. Inst.* **1954–55**, *No. 19–20*, 49–54; *Chem. Abstr.* **1955**, *51*, 11267*h*.
(11) Colonna, S.; Re, A. *J. Chem. Soc., Perkin Trans. 1* **1981**, 547.
(12) Rogers, M. A. T. *J. Chem. Soc.* **1943**, 590.

A Convenient Synthesis of 12,12,12-Trifluorododecanoic Acid

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Received November 14, 1983

Reactions between anodically generated trifluoromethyl radicals and various olefins have been described several times,[1-5] but they have been little used for the practical synthesis of trifluoromethyl derivatives because of their somewhat low yields and because they often give mixtures containing about half a dozen products in which dimeric material or bis(trifluoromethyl) derivatives predominate. That the method deserves further exploration is suggested by the fact that electrolysis of trifluoroacetic acid (1) can

(1) Renaud, R. N.; Champagne, P. J. *Can. J. Chem.* **1975**, *53*, 529.
(2) Renaud, R. N.; Champagne, P. J.; Savard, M. *Can. J. Chem.* **1979**, *57*, 2617.
(3) Brookes, C. J.; Coe, P. L.; Owen, D. M.; Pedler, A. E.; Tatlow, J. C. *J. Chem. Soc., Chem. Commun.* **1974**, 323.
(4) Brookes, C. J.; Coe, P. L.; Pedler, A. E.; Tatlow, J. C. *J. Chem. Soc., Perkin Trans. 1* **1978**, 202.
(5) Muller, N. *J. Org. Chem.* **1983**, *48*, 1370.

0022-3263/84/1949-2826$01.50/0