(0.248 mol) of 4-cyclooctenone[19] and 73 g (0.27 mol) of methylene iodide was added in one portion. The mixture was refluxed for 2 days, after which glpc analysis of the ether solution indicated that the reaction had only gone to a small extent (*ca.* 30%). Another 18 g of the zinc–copper couple was added to the mixture and this was followed by another 73 g (0.27 mol) of methylene iodide. The mixture was then refluxed for another 2 days. The mixture was then filtered and the filtrate was washed with two 100-ml portions of a 5% HCl solution, two 100-ml portions of saturated Na_2CO_3 solution, and two 100-ml portions of water, dried ($MgSO_4$), and concentrated. The product was distilled, yielding 16.2 g (47%) of **7**, bp 97–100° (15 mm). Glpc analysis of the distilled product indicated that two products were present in a ratio of 16:84. The first product was 4-cyclooctenone, while the second was bicyclo[6.1.0]nonan-4-one (**7**): ir (CCl_4) 3075, 3000, 2940, 2870, 1705, 1355, 1345, 1170 cm^{-1}; nmr ($CDCl_3$) τ 7.4–8.7 (10 H), 9.3 (m, 3 H), 10.15 (m, 1 H).[4]

Irradiation of 7 in Ether.—A solution of 0.66 g of **7** in 250 ml of ether was irradiated for 3 hr through a Corex filter. At the end of this period, the ether layer was concentrated. Glpc analysis of the crude product indicated the presence of three products ratio of 8:32:60. The first product had the same retention time and ir spectrum as **7**. The second product was bicyclo[6.1.0]nonan-4-ol (**8**): ir (CCl_4) 3610, 3320, 3070, 3000, 2920, 2860, 1030 cm^{-1}; nmr (CCl_4) τ 5.9 (m, 1 H), 6.6 (s, 1 H), 7.5–9.1 (m, 13 H), 10.1 (m, 1 H).

Anal. Calcd for $C_9H_{16}O$: C, 77.08; H, 11.50. Found: C, 77.11; H, 11.65.

The third product was assigned the structure of 4-(1-ethoxyethyl)bicyclo[6.1.0]nonan-4-ol (**9**): ir (CCl_4) 3570, 3080, 1110 cm^{-1}; nmr (CCl_4) τ 6.1–7.0 (m, 3 H), 7.6–9.0 (broad, 17 H), 9.2 (m, 3 H), 10.15 (m, 1 H); mass spectrum (70 eV) *m/e* 212, 139, 73.

Anal. Calcd for $C_{13}H_{24}O_2$: C, 73.53; H, 11.39. Found: C, 73.70; H, 11.20.

Oxymercuration of Bicyclo[6.1.0]non-4-ene.—To a solution of 3.19 g (0.01 mol) of $Hg(OAc)_2$ in 10 ml of tetrahydrofuran and 10 ml of water was added 1.08 g (0.01 mol) of bicyclo[6.1.0]non-4-ene.[20] The mixture was stirred for 10 min. To the solution was added a 10-ml solution of 3 *M* NaOH and this was followed by a 10-ml solution of 3 *M* $NaBH_4$ in 3 *M* NaOH solution. The mercury was allowed to settle, and the mixture was saturated with NaCl. The upper layer was separated, dried ($MgSO_4$), and concentrated. Glpc analysis indicated that one major fraction is present which has the same retention time and ir spectrum as those of **8**.

Irradiation of 7 in *tert*-Butyl Alcohol.—A sample of 1.24 g of bicyclo[6.1.0]nonan-4-one (**7**) was irradiated through a Corex filter for 3 hr. The solution was then concentrated. Glpc analysis of the crude product on silicone grease (130°) indicated the presence of three products in a ratio of 22:15:62. The first product is believed to be a mixture of the aldehyde **10** and **11**: ir (CS_2) 3080, 3000, 2720, 1730 cm^{-1}; nmr (CCl_4) τ 0.34 (m, 1 H), 3.7–4.7 (m, 1 H) 4.75–5.25 (m, 2 H), 7.2–9.5 (m, 9), 10.05 (m, 1 H). The second product had the same retention time and ir spectrum as **7**.

The third product is believed to be a mixture of the *tert*-butyl esters **12** and **13**: ir (CCl_4) 3070, 2990, 1735, 1400, 1375, 1160 cm^{-1}; nmr (CCl_4) τ 7.6–8.0 (m, 2 H), 8.2–8.9 (15 H), 8.9–9.2 (m, 3 H), 9.35 (m, 3 H), 10.2 (m, 1 H).

Anal. Calcd for $C_{13}H_{24}O_2$: C, 73.53; H, 11.39. Found: C, 73.37; H, 11.05.

Registry No.—**1,** 28399-86-2; **2,** 94-66-6; **3,** 28399-94-2; **4,** 28405-45-0; **6,** 28405-46-1; **7,** 28405-47-2; **8,** 28405-40-5; **9,** 28405-48-3; **14,** 502-49-8; **15,** 28405-49-4; **16,** 5457-66-9; **18,** 28405-50-7; **22,** 28405-52-9.

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[20] H. E. Simmons, E. P. Blanchard, and H. D. Hartzler, *J. Org. Chem.*, **31**, 295 (1966).

Notes

Ynamines from 1,1-Difluoro-2-aryl- and -2-alkylethylenes

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The ynamines (**3** and **4**) are a well-known class of organic compounds and have been the subject of a review article.[1] We observed recently that the action of lithium diethylamide on a 1,1-difluoroalkene (**1**) or a β,β-difluorostyrene (**2**) is a convenient, general laboratory route to ynamines (**3** and **4**).[2] **1** and **2** are obtained readily from aliphatic or aromatic aldehydes and sodium chlorodifluoroacetate by the procedure of Fuqua, *et al.*,[3] and an overall yield of 30–40% of the ynamine was obtained from the commercially available aldehyde. The aromatic ynamines were pale yellow liquids, whereas the aliphatic compounds were colorless. All were stable at room temperature and, as expected, each had a characteristic strong band at 2220 cm^{-1} in the infrared spectrum.[1]

$$\underset{\mathbf{1}}{RCH{=}CF_2} + 2LiN(C_2H_5)_2 \longrightarrow \underset{\mathbf{3}}{RC{\equiv}CN(C_2H_5)_2}$$

$$\underset{\mathbf{2}}{ArCH{=}CF_2} + 2LiN(C_2H_5)_2 \longrightarrow \underset{\mathbf{4}}{ArC{\equiv}CN(C_2H_5)_2}$$

The proton and fluorine nmr spectra of **1** and **2** were recorded (Table I). All the spectra were 12-line ABX patterns and were readily interpreted on this basis assuming that $J_{HF}(trans) > J_{HF}(cis)$. It is interesting to note that the chemical shifts of the fluorine atoms of β,β-difluorostyrene are at lower field than those of the 1,1-difluoroalkenes, and, significantly, F-1 (cis to the phenyl ring) is at lower field than F-2 (trans to the phenyl ring). A similar pattern is seen in the pmr spectra of the protonated series where the proton cis to the phenyl ring of styrene appears at lower field than the trans

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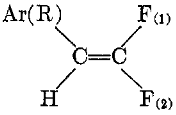
[1] H. G. Viehe, *Angew. Chem., Int. Ed. Engl.*, **6**, 767 (1967).

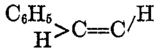
[2] When this work was initiated, the author was unaware of H. G. Viehe, U. S. Patent 3,369,047 (1968), which claims but does not give examples of the preparation of ynamines from lithium dialkylamides and 1,1-difluoroethylenes.

[3] S. A. Fuqua, W. G. Duncan, and R. M. Silverstein, *J. Org. Chem.*, **30**, 1027 (1965).

TABLE I

NMR PARAMETERS FOR 1,1-DIFLUORO-2-ARYL(ALKYL)ETHYLENES



		Chemical shift				J, Hz		
Ar or R group	Registry no.	H, δ	F-2, ppm	F-1, ppm	(F-2) − (F-1), ppm	HF-1	HF-2	FF
$CH_3(CH_2)_4$	4980-67-0	4.07	90.1	92.8	−2.7	25	3.5	49
$CH_3(CH_2)_5$	592-93-8	4.06	90.5	93.0	−2.5	25	3.5	50
C_6H_5	405-42-5	5.13	85.4	83.6	1.8	26	4.5	33
$4\text{-}CH_3C_6H_4$	28321-07-5	5.13	86.4	84.3	2.1	26	4.5	34
$4\text{-}CH_3OC_6H_4$	1608-24-8	5.13	87.7	86.0	1.7	25	4.5	38
$4\text{-}ClC_6H_4$	28321-09-7	5.15	83.7	82.0	1.7	26	4.5	31
$2\text{-}CH_3C_6H_4$	28321-10-0	5.25	85.1	86.0	−0.9	24	5.5	32
$2\text{-}ClC_6H_4$	28321-11-1	5.65	86.8	87.9	−1.1	25	4.5	28
 $C_6H_5(H)C{=}C(H)$	28321-12-2	4.90	88.4	86.7	1.7	24	2.0	28

proton,[4] which is at lower field than the *gem*-vinyl protons of 1-alkenes.[5] The anomalous low-field chemical shift of the vinyl protons of styrene has been explained in terms of the diamagnetic anisotropy of the phenyl ring.[4] A similar argument can be applied to explain qualitatively the low F-1 and F-2 chemical shift values of β,β-difluorostyrene relative to 1,1-difluoroalkenes. If the 4 position of β,β-difluorostyrene is substituted, both F-1 and F-2 are shifted by the same amount and in the same direction of field. The shifts observed are those anticipated based on the inductive and mesomeric effects of the particular substituent. The effect of a 2 substituent on the fluorine chemical shift is more complex and the paucity of data precludes the establishment of a discernible pattern. Drieding molecular models show that the aryl ring and vinyl system are not coplanar in the 2-substituted compounds and, as a result, one would expect F-1 to be shielded by the aromatic ring currents. Presumably the electronic effects of the substituent are also important; however, a better understanding of all the systems presented in Table I must await future studies.

Experimental Section

Infrared spectra were obtained on neat samples. Proton nmr (pmr) spectra were recorded at 60 MHz and fluorine spectra at 56.4 MHz in carbon tetrachloride solution. Internal references, tetramethylsilane and trichlorofluoromethane, were used. Proton chemical shifts are δ values; fluorine chemical shifts are upfield from the reference. The abbreviations s, d, t, q, and m refer to singlet, doublet, triplet, quartet, and multiplet.

β,β-Difluorostyrenes and 1,1-Difluoroalkenes.—The procedure of Fuqua, *et al.*,[3] was used with only minor modification.

2-Chloro-β,β-difluorostyrene was obtained from *o*-chlorobenzaldehyde: bp 60–61° (13 mm); 66% yield; $n^{25}D$ 1.5118; infrared bands at 1730, 1470, 1430, 1340, 1275, 1235, 1175, 1125, 1030, 945, 815, and 748 cm^{-1}.

Anal. Calcd for $C_8H_5ClF_2$: C, 55.03; H, 2.89; F, 21.76. Found: C, 54.8; H, 2.98; F, 22.3.

4-Chloro-β,β-difluorostyrene was obtained from *p*-chlorobenzaldehyde: bp 67–71° (15 mm); 17% yield; $n^{25}D$ 1.5169; infrared bands at 1730, 1485, 1350, 1245, 1165, 1090, 1010, 938, and 840 cm^{-1}.

Anal. Calcd for $C_8H_5ClF_2$: C, 55.03; H, 2.89; Cl, 20.31. Found: C, 54.8; H, 2.91; Cl, 20.5.

2-Methyl-β,β-difluorostyrene was obtained from *o*-tolualdehyde: bp 59–61° (20 mm); 44% yield; $n^{25}D$ 1.4909; infrared bands at 1730, 1450, 1335, 1235, 1205, 1165, 935, 815, and 745 cm^{-1}.

Anal. Calcd for $C_9H_8F_2$: C, 70.10; H, 5.23; F, 24.65. Found: C, 69.7; H, 5.13; F, 24.7.

4-Methyl-β,β-difluorostyrene was obtained from *p*-tolualdehyde: bp 63–64° (22 mm); 35% yield; $n^{25}D$ 1.4909; infrared bands at 1725, 1340, 1240, 935, and 830 cm^{-1}.

Anal. Found: C, 69.8; H, 5.41; F, 24.6.

1,1-Difluoro-4-phenyl-*trans*-1,3-butadiene was obtained from *trans*-cinnamaldehyde: bp 66–67° (2.0 mm); 40% yield; $n^{25}D$ 1.5535; infrared bands at 1730, 1355, 1330, 1295, 1275, 1190, 965, 935, 830, 745, and 688 cm^{-1}.

Anal. Calcd for $C_{10}H_8F_2$: C, 72.26; H, 4.85; F, 22.86. Found: C, 72.0; H, 4.96; F, 23.2.

1,1-Difluoro-1-heptene was obtained from hexanal: bp 94–96°; 49% yield; $n^{25}D$ 1.3669; infrared bands at 2960, 2880, 1740, 1460, 1315, 1200, 1170, 1120, 1040, 930, and 800 cm^{-1}.

Anal. Calcd for $C_7H_{12}F_2$: C, 62.66; H, 9.02; F, 28.32. Found: C, 62.8; H, 9.10; F, 28.1.

Preparation of Ynamines. General Procedure.—The apparatus consisted of a 1-l. two-necked flask equipped with magnetic stirrer and fitted with a serum cap and Claisen adapter on which were placed a dropping funnel and a condenser with nitrogen bubbler. From a syringe, 90 ml (144 mmol) of 1.6 *M* *n*-butyllithium in hexane[6] was placed in the flask through the serum cap in a nitrogen atmosphere. A solution of 14.85 ml of diethylamine in 55 ml of ether was placed in the dropping funnel and the butyllithium solution was cooled in an ice bath. The amine solution was added, with stirring, over 30 min. After the addition, 64.3 mmol of neat difluoro compound was placed in the dropping funnel and the ice bath was replaced with a Dry Ice–acetone bath. The difluoro compound was added during 10–15 min and the funnel was rinsed with several milliliters of ether which was also added to the reaction vessel. The cooling bath was removed and the mixture warmed slowly. In the case of the *aryl difluoro compounds*, at some temperature below 0°, *a rapid reaction occurred with vigorous gas evolution*. This was not noted with the alkyl derivatives where the reaction was much more moderate. The reaction mixture was brought to room temperature and stirred overnight under nitrogen. Solids were removed by filtration and washed with hexane, all under a nitrogen blanket. The filtrates were concentrated on the water pump and the residue was distilled *in vacuo*.

***N,N*-Diethylphenylethynylamine** was obtained from β,β-difluorostyrene: bp 87–91° (45 mm); 73% yield; $n^{25}D$ 1.5618; infrared bands at 2980, 2880, 2220, 1600, 1375, 1355, 1330, 1185, 1070, 750, and 688 cm^{-1} (the spectrum was essentially identical

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(4) C. N. Banwell and N. Sheppard, *Mol. Phys.*, **3**, 351 (1960).

(5) A. A. Bothner-By and C. Naar-Colin, *J. Amer. Chem. Soc.*, **83**, 231 (1961).

(6) Foote Mineral Co., Exton, Pa.

with the published spectrum);[7] pmr resonances at δ 1.22 (t, 6 H), 2.95 (q, 4 H), 7.20 (m, 5 H).

N,N-**Diethyl-4-methoxyphenylethynylamine** was obtained from 4-methoxy-β,β-difluorostyrene: bp 98–100° (0.08 mm); 77% yield; n^{25}D 1.5106; infrared bands at 2950, 2220, 1600, 1500, 1455, 1405, 1390, 1365, 1320, 1280, 1240, 1180, 1030, 830, and 760 cm^{-1}; pmr resonances at δ 1.22 (t, 6 H), 2.93 (q, 4 H), 3.68 (s, 3 H), 6.73 and 7.20 (AB quartet, 4 H).

Anal. Calcd for $C_{13}H_{17}NO$: C, 76.80; H, 8.43; N, 6.89. Found: C, 76.6; H, 8.64; N, 7.17.

N,N-**Diethyl-2-chlorophenylethynylamine** was obtained from 2-chloro-β,β-difluorostyrene: bp 84–86° (0.03 mm); 84% yield; n^{25}D 1.5785; infrared bands at 2980, 2850, 2220, 1600, 1435, 1415, 1380, 1340, 1250, 1190, 1080, 1050, 1030, and 745 cm^{-1}; pmr resonances at δ 1.27 (t, 6 H), 3.00 (q, 4 H), 7.10 (m, 4 H).

Anal. Calcd for $C_{12}H_{14}ClN$: C, 69.39; H, 6.79; Cl, 17.07; N, 6.74. Found: C, 69.0; H, 6.96; Cl, 16.8; N, 7.36.

N,N-**Diethyl-4-chlorophenylethynylamine** was obtained from 4-chloro-β,β-difluorostyrene: bp 87–89° (0.03 mm); 67% yield; n^{25}D 1.5772; infrared bands at 2950, 2220, 1490, 1185, 1170, 1090, 1060, 1010, and 823 cm^{-1}; pmr resonances at δ 1.22 (t, 6 H), 2.95 (q, 4 H), 7.28 (s, 4 H).

Anal. Found: C, 69.1; H, 6.96; Cl, 16.9; N, 7.23.

N,N-**Diethyl-*o*-tolylethynylamine** was obtained from 2-methyl-β,β-difluorostyrene: bp 74–76° (0.03 mm); 80% yield; n^{25}D 1.5563; infrared bands at 2950, 2220, 1600, 1460, 1370, 1280, 1200, 1070, 1040, 750, and 710 cm^{-1}; pmr resonances at δ 1.22 (t, 6 H), 2.93 (q, 4 H), 2.33 (s, 3 H), 7.00 (m, 4 H).

Anal. Calcd for $C_{13}H_{17}N$: C, 83.36; H, 9.14; N, 7.48. Found: C, 82.8; H, 9.04; N, 8.34.

N,N-**Diethylamino-*p*-tolylethynylamine** was obtained from 4-methyl-β,β-difluorostyrene: bp 72–75° (0.03 mm); 73% yield; n^{25}D 1.5578; infrared bands at 2950, 2900, 2850, 2220, 1600, 1500, 1440, 1365, 1185, 1100, 1065, and 813 cm^{-1}; pmr resonances at δ 1.22 (t, 6 H), 2.95 (q, 4 H), 2.26 (s, 3 H), 6.93 and 7.11 (AB quartet, 4 H).

Anal. Found: C, 82.3; H, 9.20; N, 7.87.

N,N-**Diethyl(4-phenyl-but-*trans*-3-ene-1-ynyl)amine** was obtained from 1,1-difluoro-4-phenyl-*trans*-1,3-butadiene: bp 99–104° (0.03 mm); 43% yield; n^{25}D 1.6288; infrared bands at 3000, 2850, 2200, 1620, 1590, 1490, 1440, 1410, 1370, 1350, 1250, 950, 750, and 690 cm^{-1}; pmr resonances at δ 1.20 (t, 6 H), 2.92 (q, 4 H), 6.16 and 6.52, (AB quartet, 2 H, J = 16 Hz (vinyl protons)), 7.18 (m, 5 H).

Anal. Calcd for $C_{14}H_{17}N$: C, 84.36; H, 8.60; N, 7.03. Found: C, 83.3; H, 9.00; N, 7.17.

N,N-**Diethylamino-1-heptyne** was obtained from 1,1-difluoro-1-heptene: bp 59–61° (0.70 mm); 74% yield; n^{25}D 1.4454; infrared bands at 2950, 2850, 2220, 1460, 1375, 1320, 1250, 1180, 1090, and 1065 cm^{-1}; pmr resonances at δ 1.12 (t, 2.78 (q), 0.80–1.7 (m), 2.18 (t, $CH_2C{\equiv}C$).

Anal. Calcd for $C_{11}H_{21}N$: C, 78.97; H, 12.66; N, 8.38. Found: C, 78.8; H, 12.4; N, 8.64.

N,N-**Diethylamino-1-octyne** was obtained from 1,1-difluoro-1-octene: bp 56–58° (0.10 mm); 45% yield; n^{25}D 1.4490; infrared bands at 2950, 2850, 2220, 1450, 1360, 1290, 1240, 1170, 1080, and 790 cm^{-1}; pmr resonances at δ 1.12 (t), 2.78 (q), 0.80–1.7 (m), 2.18 (t, $CH_2C{\equiv}C$).

Anal. Calcd for $C_{12}H_{23}N$: C, 79.49; H, 12.79; N, 7.73. Found: C, 78.6; H, 12.8; N, 6.90.

Registry No.—*N,N*-Diethylphenylethynylamine, 4231-26-9; *N,N*-diethyl-4-methoxyphenylethynylamine, 28321-14-4; *N,N*-diethyl-2-chlorophenylethynylamine, 28321-15-5; *N,N*-diethyl-4-chlorophenylethynylamine, 28321-16-6; *N,N*-diethyl-*o*-tolylethynylamine, 28321-17-7; *N,N*-diethylamino-*p*-tolylethynylamine, 28321-18-8; *N,N*-diethyl(4-phenylbut-*trans*-3-ene-1-ynyl)amine, 28321-19-9; *N,N*-diethylamino-1-heptyne, 28321-20-2; *N,N*-diethylamino-1-octyne, 4231-37-2.

Acknowledgment.—The author is indebted to Dr. F. J. Weigert for helpful discussions about nuclear magnetic resonance and Mrs. Sharon Wheeler for technical assistance.

(7) V. Wolf and F. Kowitz, *Justus Liebigs Ann. Chem.*, **638**, 33 (1960).

2,3-Dimethyl-1-phenylnaphthalene from Thermal Dimerization of Phenylallene[1]

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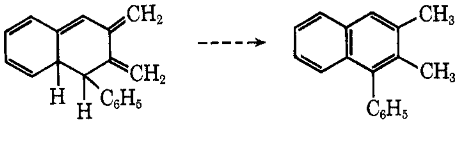
We report that phenylallene may be thermally converted to 2,3-dimethyl-1-phenylnaphthalene and suggest initial Diels–Alder cycloaddition producing a pentaene system, followed by tautomerization, as a mechanistically likely sequence.



A solution of phenylallene in inert solvents at 175° for 1 hr gave a low yield of colorless crystalline product, mp 84–85°, after recrystallization and sublimation. It proved to be a $C_{18}H_{16}$ isomer having ultraviolet absorption bands, λ_{max} 230 nm (log ϵ ~5), 276 (4.1), 285 (4.15), and 294 (4.0) in CCl_4, unlike those expected for a 2-phenylnaphthalene[4] but in close correspondence with those reported for 1-phenylnaphthalene,[4] 2-methyl-1-(*o*-tolyl)naphthalene[4] [227 (4.91), 275 (3.84), 281 (3.87), 284 (3.87), and 292 (3.76) in petroleum ether], and 2,3-dibenzyl-1-phenylnaphthalene[5] [λ_{max} 237.5 nm (4.87) and 286 (3.85) in CH_2Cl_2].

The nmr spectrum of the phenylallene dimer had methyl singlets at δ 2.10 and 2.43, and ten aromatic protons at δ 6.9–7.8 ppm.

The physical data and mechanistic possibilities prompted an assignment of 2,3-dimethyl-1-phenylnaphthalene as the probable structure for the $C_{18}H_{16}$ compound. The picrate derivative had mp 110–111°, in good agreement with the literature value, mp 112°, for authentic 2,3-dimethyl-1-phenylnaphthalene.[6] The melting point observed for the phenylallene dimer, 84–85°, was close to that for 2,3-dimethyl-1-phenylnaphthalene cited by Müller and K. Körmendy,[6] 85–86°.

Allene–allene thermal dimerizations giving dimethylenecyclobutanes and derived structures are common.[7–9] The present result, isolation of a 2,3-dimethyl-1-phenylnaphthalene as a thermal dimer from phenylallene, indicates a new mode of dimerization available to arylallenes. Formation of 2,3-dibenzyl-1-phenylnaphthalene through elimination of hydrogen chloride from 2-chloro-1,3-diphenylpropene[5] may well go by way of 1,3-diphenylallene and may then represent another example of this type of arylallene dimerization.

(1) Supported in part by National Science Foundation Grant GP-5226.

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(3) National Science Foundation Cooperative Graduate Fellow, 1963–1966.

(4) "UV Atlas of Organic Compounds," Vol. II, Plenum Press, New York, N. Y., 1966, plates E 1/2, E 1/3, and E 1/4.

(5) H. A. Staab and H. A. Kurmeier, *Chem. Ber.*, **101**, 2697 (1968).

(6) A. Müller and K. Körmendy, *J. Org. Chem.*, **18**, 1237 (1953).

(7) J. D. Roberts and C. M. Sharts, *Org. React.*, **12**, 1 (1962).

(8) D. R. Taylor, *Chem. Rev.*, **67**, 317 (1967).

(9) J. E. Baldwin and R. H. Fleming, *Fortschr. Chem. Forsch.*, **15** (3), 281 (1970).