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Synthesis of 3-Keto-6-phenyl-8-methyl-9-oxa-$\Delta^{1,2}$-2-azabicyclo[4.3.0]nonane

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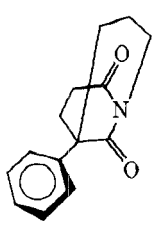
The synthesis and the study of the intramolecular cyclizations of ethyl 3-[3-phenyl-3-(2′-piperidonyl)]propionate (**3**), 2-(3-bromopropyl)-2-phenylglutarimide (**17**), and 2-(2-bromopropyl)-2-phenylglutarimide (**18**) are described. Although both N and O alkylations are possible, only the O-alkylated products were observed.

The synthesis of 5-phenyl-2,9-diketo-1-azabicyclo-[3.3.1]nonane (**1**), a bridged analog of aminoglutethimide (**2**), was undertaken in order to investigate the steric requirements of the antiepileptic action of drugs containing the ureide or imide moiety.



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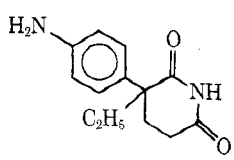

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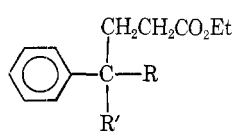
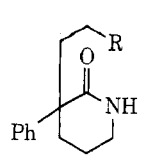
The initial approach to the synthesis of **1** involved the synthesis of ethyl 3-[3-phenyl-3-(2′-piperidonyl)]propionate (**3**). It was proposed that a base-catalyzed intramolecular attack by the amide nitrogen on the ester function would yield **1**.

Diethyl 2-phenylglutarate (**4**) was converted to diethyl 2-cyanoethyl-2-phenylglutarate (**5**) *via* cyanoethylation. Hydrogenation of **5** yielded the primary amine **6** which was converted without purification to the desired lactam ester **3**. Treatment of **3** with a variety of bases (sodium hydride, potassium *tert*-butoxide, thallous ethoxide, sodium hydroxide, and sodium ethoxide) in various solvent systems (dimethylformamide, dimethoxyethane, diethyl ether) failed to yield the desired bicyclic glutarimide **1**. The product isolated was identified as 3-[3-phenyl-3-(2′-piperidonyl)]propionic acid (**7**). This probably results from the initial

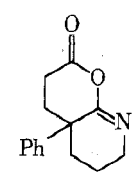


4, R′ = H; R = CO_2Et
5, R′ = CH_2CH_2CN; R = CO_2Et
6, R′ = $CH_2CH_2CH_2NH_2$; R = CO_2Et



3, R = CO_2Et
7, R = CO_2H

formation of **8** *via* intramolecular O-acylation followed by hydrolysis during isolation to give the acid **7**.



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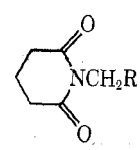
Attempts to cyclize compound **7** to the desired glutarimide **1** under various conditions (acetic anhydride, acetic anhydride and pyridine, thionyl chloride, dicyclohexylcarbodiimide, and polyphosphoric acid) yielded only starting material.

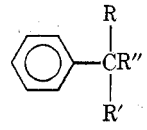
We previously reported[3] the light-catalyzed addition of hydrogen bromide to *N*-allylglutarimide (**9**) to yield *N*-(3-bromopropyl)glutarimide (**10**). If a small amount of acetic acid was added to the reaction *N*-(2-bromopropyl)glutarimide (**11**) was obtained. Attempts to cyclize **10** to the C-alkylated bicyclic system failed.



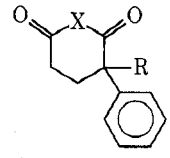
9, R = $CH{=}CH_2$
10, R = CH_2CH_2Br
11, R = $CHBrCH_3$

An alternate approach to the synthesis of **1** involved a base-catalyzed intramolecular alkylation of 2-(3-bromopropyl)-2-phenylglutarimide (**17**). We anticipated that the addition of a suitable base would lead to the abstraction of the relatively acidic imide proton and the resulting anion could afford nucleophilic displacement of the primary bromide of the propyl side chain to yield the desired compound **1**.

Phenylacetonitrile was allowed to react with allyl bromide and sodium hydride in dimethylformamide to yield 2-allylphenylacetonitrile (**12**). Cyanoethylation of **12** yielded 2-allyl-2-cyanoethylphenylacetonitrile (**13**) followed by hydrolysis to yield the diacid **14** which was converted to 2-allyl-2-phenylglutaric anhydride (**15**) with refluxing acetic



12, R = H; R′ = $CH_2CH{=}CH_2$; R″ = CN
13, R = CH_2CH_2CN; R′ = $CH_2CH{=}CH_2$; R″ = CN
14, R = $CH_2CH_2CO_2H$; R′ = $CH_2CH{=}CH_2$; R″ = CO_2H

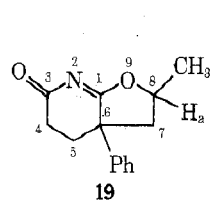


15, X = O; R = $CH_2CH{=}CH_2$
16, X = NH; R = $CH_2CH{=}CH_2$
17, X = NH; R = $CH_2CH_2CH_2Br$
18, X = NH; R = $CH_2CHBrCH_3$
22, X = NH; R = $CH_2CH_2CH_2OH$

anhydride. Treatment of **15** with concentrated ammonium hydroxide afforded 2-allyl-2-phenylglutarimide (**16**) in good yield. Light-catalyzed addition of hydrogen bromide[3] to compound **16** gave 2-(3-bromopropyl)-2-phenylglutarimide (**17**). Addition of a trace of acetic acid or the use of water-saturated toluene as the solvent for the addition yielded 2-(2-bromopropyl)glutarimide (**18**).

Treatment of compound **18** with sodium hydride in dimethylformamide led to the formation of a product with an empirical formula consistent with either 3-keto-6-phenyl-8-methyl-9-oxa-$\Delta^{1,2}$-2-azabicyclo[4.3.0]nonane (**19**) or 2,8-diketo-5-phenyl-7-methyl-1-azabicyclo[3.2.1]octane (**20**).

The ir spectrum in chloroform showed strong absorptions at 1710 (C=O) and 1625 cm^{-1} (OC=N–) and in KBr at 1695 and 1615 cm^{-1}, respectively. The nmr spectrum in deuteriochloroform showed a three-proton doublet at δ 1.46, a complex multiplet of six protons between δ 1.80 and 2.90, and a one-proton multiplet centered at δ 4.50. Structure **20** was eliminated as a possibility on the basis of ir



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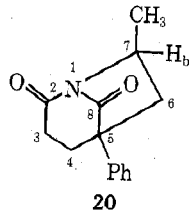

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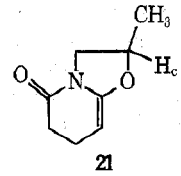

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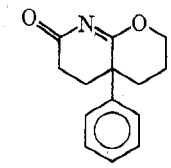
data (1625 cm^{-1}) which indicated the presence of a carbon–nitrogen double bond. In addition, in **20** the proton H_b would be expected to occur further upfield than δ 4.50. However, in **19** this would be a reasonable absorption for H_a. This assignment is supported by the report that the proton H_c in compound **21** also occurs at δ 4.50;[3] therefore, the ir and nmr data support structure **19**. Compound **19** slowly decomposes to a white gum if allowed to stand in the atmosphere.

In a similar manner, treatment of **17** with sodium hydride in benzene yielded a colorless gum which was purified using column chromatography. The ir spectrum showed absorptions at 3400 (–OH), 3210 (–NH), and 1680 cm^{-1} (C=O). The nmr spectrum in chloroform showed one exchangeable proton at δ 2.76 (–OH) and a broad singlet at δ 8.97 (imide H). On the basis of these data and elemental analysis the compound was identified as 2-phenyl-2-(3-hydroxypropyl)glutarimide (**22**). Isolation of this product suggests that base treatment leads to the formation of the O-alkylated bicyclic derivative **23** which is then opened during chromatography on the slightly acidic silica gel to yield the primary alcohol **22**.



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Experimental Section[4]

Diethyl 2-Cyanoethyl-2-phenylglutarate (5). About 10 drops of a solution of acrylonitrile (5.76 g, 0.109 mol) in 25 ml of *tert*-butyl alcohol was added to a solution of diethyl 2-phenylglutarate (16.56 g, 0.062 mol) in 25 ml of *tert*-butyl alcohol. To this mixture was added sodium hydride (0.456 g of a 57% suspension, 0.019 mol) all at once. The remainder of the acrylonitrile solution was added dropwise with stirring and the resulting yellow reaction mixture was stirred at 25° for 8 hr. To this mixture was added 50 ml of H_2O, after which the solution was made acidic with 10% HCl, extracted with 3 × 100 ml of ethyl acetate, and dried ($MgSO_4$), and the solvents were removed to give a cloudy yellow, viscous oil. Distillation afforded the desired product: 12.10 g (0.038 mol, 62%); 165–170° (0.8 mm); nmr ($CDCl_3$, 1% TMS) δ 1.12–1.36 (t, 6, $-CH_2CH_3$), 2.20–2.48 (m, 8, $-CH_2CH_2-$), 3.46–3.96 (m, 4, $-OCH_2CH_3$), 7.28–7.40 (m, 5 H, aromatic); ir (neat) 2980, 3020, 2250, 1735, 1600, 1200 cm^{-1}.

Anal. Calcd for $C_{18}H_{23}NO_4$: C, 68.12; H, 7.30; N, 4.41. Found: C, 68.33; H, 7.51; N, 4.63.

Ethyl 3-[3-Phenyl-3-(2′-piperidonyl)]propionate (3). A solution of diethyl 2-cyanoethyl-2-phenylglutarate (**5**, 20.0 g, 0.063 mol) in 125 ml of glacial acetic acid was hydrogenated at 50 psi (initial pressure) over PtO_2 for 6 hr in a Parr shaker. The contents of the flask were filtered and the acetic acid was removed *in vacuo* to yield a clear viscous oil which was then heated to reflux in anhydrous toluene for 4 hr. The toluene was then removed *in vacuo* leaving a pale green oil which was chromatographed on 300 g of Brinkman silica gel (70–325 mesh) and eluted with ethyl acetate–benzene (2:1). The desired compound **3** was obtained as a clear colorless oil which crystallized immediately upon standing. Recrystallization from diethyl ether yielded 11.0 g (0.040 mol, 64%) of pure white needles: mp 90–91°; nmr ($CDCl_3$, 1% TMS) δ 1.10–1.30 (t, 3, $-CH_2CH_3$), 1.60–2.40 (m, 8, CH_2CH_2), 3.10–3.40 (br m, 2, $-CH_2NH$), 3.85–4.25 (q, 2, OCH_2CH_3), 6.60–6.80 (br s, 1, NH), 7.25 (s, 5, aromatic); ir (KBr) 3250, 2960, 1730, 1665 (shoulder), 1610, 1490, 1200 cm^{-1}.

Anal. Calcd for $C_{16}H_{21}NO_3$: C, 69.79; H, 7.68; N, 5.08. Found: C, 70.03; H, 7.91; N, 5.38.

3-[3-Phenyl-3-(2′-piperidonyl)]propionic Acid (7). Ethyl 3-[3-phenyl-3-(2′-piperidonyl)]propionate (**3**) (2.0 g, 0.008 mol) was heated and stirred with 10 ml of 10% NaOH until the reaction mixture became homogeneous. The solution was extracted with ethyl acetate and the aqueous layer acidified with 10% HCl. The acidic solution was saturated with NaCl, extracted with 3 × 50 ml of ethyl acetate, and dried ($MgSO_4$), and the solvent was removed to yield 1.62 g (0.0064 mol, 80%) of a white solid. Recrystallization (ether–methanol) yielded colorless prisms: mp 158–160°; nmr (CD_3OD) δ 1.50–1.75 (m, 2, $-CH_2CH_2NH$), 1.85–2.20 (m, 6, $-CH_2CH_2COOH$ + $PhCCH_2$), 3.10–3.40 (m, 2, $-CH_2NH$), 7.40 (s, 5, aromatic); ir (KBr) 3300, 3100–2700, 1715, 1600, 1240 cm^{-1}.

Anal. Calcd for $C_{14}H_{17}NO_3$: C, 67.99; H, 6.92; N, 5.66. Found: C, 67.81; H, 7.04; N, 5.93.

2-Allylphenylacetonitrile (12). To NaH (14.4 g, 0.6 mol) in 200 ml of dimethylformamide (DMF) was added phenylacetonitrile (70.3 g, 0.6 mol) in 100 ml of DMF dropwise while stirring the suspension. On addition the reaction mixture became deep red. After the addition the suspension was stirred for an additional 15 min. Allyl bromide (72.6 g, 0.6 mol) in 75 ml of DMF was then added dropwise after which the reaction mixture was refluxed for 5 hr. Crushed ice (800 g) was added, the mixture was extracted with 3 × 250 ml of ethyl acetate and dried ($MgSO_4$), and the solvent was removed to afford 96.4 g of a thick brown oil. Distillation yielded the desired product: 68.2 g (0.434 mol, 72.5%); bp 80–84° (1.0 mm) [lit.[5] bp 122–126° (12 mm)]; nmr ($CDCl_3$, 1% TMS) δ 2.50–2.76 (d, 2, $-CH_2CH{=}CH_2$), 3.50–4.00 (m, 1, benzylic H), 4.90–6.16 (m, 3, $-CH{=}CH_2$), 7.40 (s, 5, aromatic); ir (neat) 3110, 3100, 3060, 2260, 1650, 1610, 1450, 990, 930 cm^{-1}.

2-Allyl-2-cyanoethylphenylacetonitrile (13). Sodium hydride (0.5 g) was dissolved in 200 ml of anhydrous *tert*-butyl alcohol and to this was added 2-allylphenylacetonitrile (**12**, 28.2 g,

0.179 mol) in 50 ml of *tert*-butyl alcohol. Immediately upon addition the reaction mixture darkened. Acrylonitrile (19.0 g, 0.36 mol) in 100 ml of *tert*-butyl alcohol was then added slowly, dropwise, with stirring. The reaction mixture became exothermic and was maintained at 40–50° with a water bath for 2 hr. The reaction mixture was then made acidic with 50 ml of 10% HCl and diluted with 250 ml of H_2O. The aqueous solution was extracted with 3 × 250 ml of ethyl acetate and dried ($MgSO_4$), and the solvent was removed to yield a viscous brown oil. This oil was distilled to yield 13.3 g (0.063 mol, 35%) of the desired product: bp 142–150° (0.4 mm) [lit.[5] bp 200–202° (18 mm)]; nmr ($CDCl_3$, 1% TMS) δ 2.24–2.60 (m, 4, $-CH_2CH_2CN$), 2.68–2.88 (d, 2, $-CH_2CH{=}CH_2$), 5.00–L.10 (m, 3, $CH_2CH{=}CH_2$), 7.50 (s, 5, aromatic); ir (neat) 3120, 3100, 3010, 3060, 2970, 1650, 1610, 990, 935, 760, 690 cm^{-1}.

Anal. Calcd for $C_{14}H_{14}N_2$: C, 79.97; H, 6.71; N, 13.32. Found: C, 80.18; H, 6.49; N, 13.14.

2-Allyl-2-phenylglutaric Anhydride (15). To 50 ml of an aqueous solution of KOH (10.6 g, 0.2 mol) was added 10.0 g (0.047 mol) of 2-allyl-2-cyanoethylphenylacetonitrile (**13**). The reaction mixture was refluxed 48 hr, acidified with 10% HCl, extracted with 3 × 100 ml of ethyl acetate, and dried ($MgSO_4$), and the solvent was removed to give 11.6 g of a pale yellow oil (98.3%). This oil was refluxed in 25 ml of acetic anhydride for 2 hr. The mixture was cooled and the excess volatile reactants were removed *in vacuo* to give a dark, viscous, orange oil. The oil was dissolved in ethyl acetate and decolorized with charcoal. Filtration and distillation yielded 8.62 g (0.037 mol, 80%) of the desired product: bp 142–144° (0.4 mm); nmr ($CDCl_3$, 1% TMS) δ 2.30–2.42 (m, 2, $-CH_2CH{=}CH_2$), 2.52–2.82 (m, 4, $-CH_2CH_2-$), 4.90–6.00 (m, 3, $-CH_2CH{=}CH_2$), 7.25–7.40 (m, 5, aromatic); ir (neat) 3100, 3055, 2960, 1810, 1765, 1640, 1060, 915, 750, 685 cm^{-1}.

Anal. Calcd for $C_{14}H_{14}O_3$: C, 73.02; H, 6.14. Found: C, 73.12; H, 6.28.

2-Allyl-2-phenylglutarimide (16). An aqueous solution of NH_4OH (58% solution, 18.3 g, 0.52 mol) and 30.0 g (0.13 mol) of 2-allyl-2-phenylglutaric anhydride (**15**) were slowly heated to reflux. The water was allowed to distil and heating was continued until the initially clear light yellow solution began to darken (*ca.* 3 hr). The reaction mixture was cooled to 25° to yield a thick tarry material. This material was distilled to give 23.0 g (0.098 mol, 76%) of a clear, viscous oil: bp 158–160° (0.2 mm) [lit.[5] bp 191–193° (5 mm)]; nmr ($CDCl_3$, 1% TMS) δ 2.20–2.80 (m, 6, $CH_2CH{=}CH_2$ + $-CH_2CH_2$), 4.80–5.90 (m, 3, $-CH_2CH_2$), 7.20 (s, 5, aromatic), 9.20 (s, 1, imide H); ir ($CHCl_3$) 3400, 1710, 1645, 1175, 915 cm^{-1}.

Anal. Calcd for $C_{14}H_{15}NO_2$: C, 73.34; H, 6.59; N, 6.10. Found: C, 73.43; H, 6.81; N, 6.03.

2-(3-Bromopropyl)-2-phenylglutarimide (17). Into a solution of 100 ml of anhydrous toluene and 0.25 g of benzoyl peroxide was placed 1.00 g (0.004 mol) of 2-allyl-2-phenylglutarimide. The reaction mixture was irradiated with a Westinghouse sunlamp (275 W, 110–125 V) while HBr was bubbled through the solution. Irradiation and HBr addition was continued for 15 min after which time the solution was purged with N_2. The toluene was removed leaving a thick orange oil which was dissolved in 50 ml of ether and placed in a refrigerator. Crystallization occurred on standing overnight to yield 1.10 g (0.0035 mol, 82.5%) of a solid. Recrystallization (ether) gave white needles: mp 95–97°; nmr ($CDCl_3$, 1% TMS) δ 1.65–2.60 (m, 8, ring H's + $-CH_2CH_2CH_2Br$), 3.18–3.40 (t, 2, $-CH_2Br$), 7.30 (s, 5, aromatic), 9.60 (s, 1, imide H); ir ($CHCl_3$) 3390, 2980, 1700–1720, 1340, 1260, 1170 cm^{-1}.

Anal. Calcd for $C_{14}H_{16}NO_2Br$: C, 54.20; H, 5.19; N, 4.51. Found: C, 54.40; H, 5.30; N, 4.78.

2-(2-Bromopropyl)-2-phenylglutarimide (18). If wet toluene was used in the procedure for **17**, compound **18** was obtained. Removal of the toluene after the addition of HBr to 2.30 g (0.01 mol) of 2-allyl-2-phenylglutarimide afforded a viscous brown oil which was dissolved in hot ethyl acetate. On cooling, a light-brown solid formed which was filtered, washed with cold ehher, and dried to give 2.75 g (0.009 mol, 90%) of **18**. Recrystallization (ether–acetone) produced white crystals: mp 159–160°; nmr ($CDCl_3$, 1% TMS) δ 1.50–1.60 (d, 3, J = 6 Hz, $-HCBrCH_3$), 2.10–2.80 (m, 6, ring H's + $-CH_2CHBr-$), 3.90–4.05 (m, 1, $-CH_2CHBrCH_3$), 7.20 (s, 5, aromatic), 10.40 (s, 1, imide H); ir (KBr) 3210, 1720, 1690, 1360, 1260, 1200, 1180 cm^{-1}.

Anal. Calcd for $C_{14}H_{16}NO_2Br$: C, 54.20; H, 5.19; N, 4.51. Found: C, 54.26; H, 5.07; N, 4.80.

Reaction of 2-Phenyl-2-(3-bromopropyl)glutarimide with Sodium Hydride. In 125 ml of anhydrous benzene was dissolved 2.0 g (0.0065 mol) of 2-phenyl-2-(3-bromopropyl)glutarimide and to this was added 0.24 g (0.01 mol) of NaH [washed free of mineral oil with petroleum ether (bp 60–68°)] all at once. Immediately upon addition, H_2 began to evolve and the reaction mixture was allowed to stir at room temperature for 24 hr. The reaction mixture was then filtered (solid identified as NaBr) and the solvent removed to yield a viscous orange-brown oil. This oil was chromatographed on 50 g of Brinkman silica gel (70–325 mesh) and eluted with ethyl acetate–benzene (1:1). Chromatography yielded 1.10 g (0.0045 mol, 69%) of 2-phenyl-2-(3-hydroxypropyl)glutarimide (**22**), a low-melting, hygroscopic white solid: mp 76–78°; nmr ($CDCl_3$, 1% TMS) δ 1.13–2.50 (m, 8, CH_2CH_2), 2.76 (br s, 1, disappears with addition of D_2O, OH), 3.43–3.63 (t, 2), 7.33 (s, 5, aromatic), 8.80–9.13 (brs, 1, imide H); ir (KBr) 3400, 3210, 1680, 1340, 1175 cm^{-1}.

Anal. Calcd for $C_{14}H_{17}NO_3$: C, 67.99; H, 6.92; N, 5.66. Found: C, 67.79; H, 6.79; N, 5.55.

3-Keto-6-phenyl-8-methyl-9-oxa-$\Delta^{1,2}$-2-azabicyclo[4.3.0]-nonane (19). In 30 ml of anhydrous DMF was placed 1.55 g (0.005 mol) of 2-(2-bromopropyl)-2-phenylglutarimide. Directly to this mixture was added 0.21 g (57% suspension, 0.005 mol) of NaH and the solution was stirred for 24 hr. The DMF was then removed *in vacuo* to leave a tan residue. This was partially dissolved in H_2O and immediately extracted with ether (3 × 50 ml). The ethereal extract was dried (Na_2SO_4) and the solvent removed to yield a white solid (0.52 g, 0.0023 mol, 46%). Recrystallization (ether) yielded thin colorless needles: mp 164–166°; nmr ($CDCl_3$, 1% TMS) δ 1.45–1.55 [d, 3, J = 6 Hz, $-OC(CH)CH_3$], 1.85–2.90 (m, 6, CH_2), 4.30–4.85 [m, 1, $-OC(CH_3)H$], 7.30 (s, 5, aromatic); ir ($CHCl_3$) 3030, 2960, 1710, 1625, 1230, 950 cm^{-1}.

Anal. Calcd for $C_{14}H_{15}NO_2$: C, 73.34; H, 6.59; N, 6.10. Found: C, 73.50; H, 6.76; N, 6.15.

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Registry No.—**3**, 53370-32-4; **4**, 53370-33-5; **5**, 53370-34-6; **7**, 53370-35-7; **12**, 5558-87-2; **13**; 53370-36-8; **15**, 53370-37-9; **16**, 53370-38-0; **17**, 53370-39-1; **18**, 53370-40-4; **19**, 53370-41-5; **22**, 53370-42-6; acrylonitrile, 107-13-1; phenylacetonitrile, 140-29-4; allyl bromide, 106-95-6.

References and Notes

(1) Deceased July 14, 1974.
(2) Taken in part from the dissertation presented by P. J. Wirth, Aug 1974, to the Graduate School of the University of Kansas in partial fulfillment of the requirements for the Doctor of Philosophy Degree.
(3) E. E. Smissman and J. W. Ayres, *J. Org. Chem.*, **37**, 1092 (1972).
(4) Infrared spectra were obtained on a Beckman IR-33 spectrophotometer. Nuclear magnetic resonance spectra were obtained using Varian A-60A and T-60 spectrometers. Elemental analyses were determined on a Hewlett-Packard 185 C, H, N analyzer at the University of Kansas.
(5) M. P. Mertes and C. O. Wilson, *J. Amer. Pharm. Ass.*, **47**, 882 (1958).