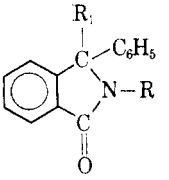
TABLE I
3-PHENYLPHTHALIMIDINES



R	R_1	M.p., °C.	Yield, %	Formula	N, % Calcd.	N, % Found
3-(2-Propylamino)propyl·HCl	OH	189–190	91	$C_{20}H_{25}ClN_2O_2$	7.76	7.70
3-(2-Propylamino)propyl	OH	99–102	80	$C_{20}H_{24}N_2O_2$	8.64	8.79
3-Diethylaminopropyl	OH	76–77	75	$C_{21}H_{26}N_2O_2$	8.28	8.41
2-Aminoethyl·HCl	OH	261–263	37	$C_{16}H_{17}ClN_2O_2$	9.19	9.20
3-Dimethylaminopropyl·HCl	OH	201–202	69	$C_{19}H_{23}ClN_2O_2$	8.08	8.30
Diethylaminoethyl·HCl	OH	190–192	73	$C_{20}H_{25}ClN_2O_2$	7.77	7.18
1-(3-Methoxypropyl)	OH	127–128.5	91	$C_{18}H_{19}NO_3$	4.72	4.87
Allyl	OH	145–147	85	$C_{17}H_{15}NO_2$	4.98	5.10
t-Butyl	OH	115–116	83	$C_{18}H_{19}NO_2$	4.98	5.10
Cyclohexyl	OH	224–226	92	$C_{20}H_{21}NO_2$	4.56	4.61
o-Tolyl	OH	174–175.5	45	$C_{21}H_{17}NO_2$	4.44	4.51
m-Tolyl	OH	178–180	75	$C_{21}H_{17}NO_2$	4.44	4.71
p-Tolyl	OH	222–224	89	$C_{21}H_{17}NO_2$	4.44	4.68
o-Methoxyphenyl	OH	151–153	37	$C_{21}H_{17}NO_3$	4.23	4.46
p-Methoxyphenyl	OH	191–191.5	28	$C_{21}H_{17}NO_3$	4.23	4.44
o-Nitrophenyl	OH	147.5–149	43	$C_{20}H_{14}N_2O_4$	8.09	8.24
n-Nitrophenyl	OH	197–198	41	$C_{20}H_{14}N_2O_4$	8.09	7.97
p-Nitrophenyl	OH	187–190	48	$C_{20}H_{14}N_2O_4$	8.09	8.00
m-Aminophenyl	OH	192–193	21	$C_{20}H_{16}N_2O_4$	8.86	8.92
2-Chloro-4-nitrophenyl	OH	170.5–172.5	33	$C_{20}H_{13}ClN_2O_4$	7.36	7.52
4-Propionylphenyl	OH	185–186.5	20	$C_{23}H_{19}NO_3$	3.93	4.09
4-Pyridyl	OH	211–215	34	$C_{19}H_{14}N_2O_3$	9.23	9.22
2-Pyridyl	OH	173.5–175	20	$C_{19}H_{14}N_2O_3$	9.23	9.04
2-(4-Methylpyridyl)	OH	207–208.5	40	$C_{20}H_{16}N_2O_2$	8.86	8.88
2-(6-Methylpyridyl)	OH	186–187	45	$C_{20}H_{16}N_2O_2$	8.86	8.96
2-Pyrimidyl	OH	222–223.5	41	$C_{18}H_{13}N_3O_2$	13.85	13.98
Cyclohexyl	$N(C_2H_4Cl)_2$	131–132	31	$C_{24}H_{28}Cl_2N_2O$	6.50	6.77
Phenyl	$N(C_2H_4Cl)_2$	137–139	55	$C_{24}H_{22}Cl_2N_2O$	6.59	6.65
Phenyl	Piperidino	210–212	57	$C_{25}H_{24}N_2O$	7.62	7.98
Phenyl	N-Methylpiperazino	205–207	35	$C_{25}H_{25}N_3O$	10.96	11.24
Phenyl	Ethoxyl	139–140	84	$C_{22}H_{19}NO_2$	4.25	4.22
Phenyl	$N(C_2H_5)_2$	192–194	63	$C_{24}H_{24}N_2O$	7.86	7.99
Phenyl	Morpholino	210–212	42	$C_{24}H_{24}N_2O$	7.56	7.43
Phenyl	$C_2H_5SO_2$	166–168	80	$C_{22}H_{19}NO_3S$	3.72	3.86
Phenyl	$C_6H_5CH_2SO_2$	180–192	37	$C_{26}H_{19}NO_3S$	3.18	3.14
p-Tolyl	$(NCH_2CH_2Cl)_2$	153–154	38	$C_{25}H_{24}Cl_2N_2O$	6.38	6.40
p-Tolyl	Piperidino	114–115	57	$C_{26}H_{26}N_2O$	7.32	7.58
p-Tolyl	N-Methylpiperazino	181–183	24	$C_{26}H_{27}N_3O$	10.58	10.59

of these compounds warranted further study. All of the other phthalimidines were devoid of antitumor effects.

Experimental[6]

3-Hydroxy-3-phenyl-2-substituted phthalimidines were prepared *via* previously reported procedures[3,4] and were recrystallized from methanol or methanol and water. The compounds are tabulated in Table I.

2,3-Diphenyl-3-ethylsulfonylphthalimidine.—A mixture of 9.4 g. of 3-chloro-2,3-diphenylphthalimidine[4] and 50 ml. of chloroform was cooled and added slowly to a cold, stirred solution of 3 g. of ethanethiol in 30 ml. of $CHCl_3$. The solution was stirred and allowed to reach room temperature, then evaporated *in vacuo*. The gummy solid was taken up in 50 ml. of glacial acetic acid and cooled, and 10 ml. of 30% H_2O_2 was added dropwise to the cold solution. The mixture was diluted with 100 ml. of water, and the solid was removed. Recrystallization from ethanol gave 10 g. (98%) of the expected sulfone, m.p. 166–168°.

3-Alkoxy, 3-piperidino, and 3-morpholino derivatives were prepared in the fashion described by von Graf and co-workers[4] and the data are included in Table I.

(6) Melting points, corrected, were obtained with a Thomas–Hoover apparatus.

Some Compounds Derived from 1-Cyano- and 1-Bromobenzocyclobutene

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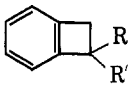
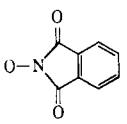
As part of an investigation into the biological significance of benzocyclobutene derivatives,[1] we have synthesized a number of 1,1-disubstituted compounds (Table I) from the readily accessible 1-cyanobenzocyclobutene. In addition, the oxygen isostere XV of the previously described 1-aminomethylbenzocyclobutene[1] and the unique amino acid, 1-benzocyclobutenylglycine (XVI), were prepared from 1-bromobenzocyclobutene. The pharmacological evaluation of these compounds as potential antihypertensive and analgetic agents is in progress.

(1) J. A. Skorcz and J. E. Robertson, *J. Med. Chem.*, **8**, 255 (1965

TABLE I
1-SUBSTITUTED AND 1,1-DISUBSTITUTED BENZOCYCLOBUTENES



No.	R	R′	B.p. (mm.) or m.p., °C.	Yield, %	Formula	Calcd., % C	Calcd., % H	Calcd., % N	Found, % C	Found, % H	Found, % N
I	CN	$CH_2C_6H_5$	118–122 (0.01)	77	$C_{16}H_{13}N$	87.64	5.97	6.39	87.28	6.20	6.41
II	CO_2H	$CH_2C_6H_5$	90–92	90	$C_{16}H_{14}O_2$	80.65	5.92		80.63	5.86	
III	$CONH_2$	$CH_2C_6H_5$	81–83	63	$C_{16}H_{15}NO$	80.98	6.37	5.90	81.01	6.37	5.96
IV	$CH_2NH_2 \cdot HCl$	$CH_2C_6H_5$	221–223	85	$C_{16}H_{18}ClN$[a]			5.39			5.36
V	$CH_2NHCO_2C_2H_5$	$CH_2C_6H_5$	159–161 (0.07)	68	$C_{19}H_{21}NO_2$	77.26	7.16	4.74	77.37	7.16	4.77
VI	CN	$CH_2CH_2N(CH_3)_2$	93–95 (0.05)	67	$C_{13}H_{16}N_2$	77.96	8.04	13.99	77.84	8.13	13.62
VII	$CONH_2$	$CH_2CH_2N(CH_3)_2$	97–99	72	$C_{13}H_{18}N_2O$	71.53	8.31	12.83	71.42	8.33	12.90
VIII	$CO_2C_2H_5$	$CH_2CH_2N(CH_3)_2 \cdot HCl$	159–161	52	$C_{15}H_{22}ClNO_2$[b]			4.94			5.06
IX	CN	CH_2CH_2CN	127–130 (0.15)	71	$C_{12}H_{10}N_2$	79.09	5.53	15.37	78.94	5.65	15.12
X	$CONH_2$	$CH_2CH_2CONH_2$	189–191	70	$C_{12}H_{14}N_2O_2$	66.04	6.47	12.84	66.09	6.36	12.83
XI	CO_2H	$CH_2CH_2CO_2H$	151–154	80	$C_{12}H_{12}O_4$[c]	65.45	5.49		65.51	5.28	
XII		$CO_2COCH_2CH_2$[d]	87–89	55	$C_{12}H_{10}O_3$	71.27	4.98		71.05	5.06	
XIII		$CONHCOCH_2CH_2$[d]	189–191	52	$C_{12}H_{11}NO_2$	71.62	5.51	6.96	71.59	5.35	7.00
XIV	H		146–149	57	$C_{16}H_{11}NO_3$	72.44	4.18	5.28	72.45	3.93	5.27
XV	H	$ONH_2 \cdot HCl$	191–192	88	$C_8H_{10}ClNO$	55.98	5.87	8.16	55.87	5.90	8.17
XVI	H	$CH(NH_2)CO_2H$	260–262	17	$C_{10}H_{11}NO_2$	67.78	6.26	7.91	67.57	6.17	8.25

[a] *Anal.* Calcd.: Cl, 13.64. Found: Cl, 13.64. [b] *Anal.* Calcd.: Cl, 12.49. Found: Cl, 12.72. [c] *Anal.* Calcd.: Neut. equiv., 110.1. Found: Neut. equiv., 109.9. [d] Spiro compound.

In agreement with reports on various benzocyclobutene derivatives,[2] the compounds described here show typical indane-like absorption in the ultraviolet with peaks near 272, 265, and 260 mμ. With the exception of the imide XIV, each of the compounds has a band of moderate to weak intensity in the 10.0–10.1-μ region of the infrared.[2a,3]

Experimental[4]

1-Benzyl-1-cyanobenzocyclobutene (I).—A solution of 1-cyanobenzocyclobutene[5] (25.8 g., 0.20 mole) and benzyl chloride (26.6 g., 0.21 mole) in 200 ml. of anhydrous benzene was treated portionwise with 7.8 g. (0.20 mole) of sodamide. The temperature was maintained below 40° during the addition. The well-stirred mixture then was heated to 80° and was kept at that temperature for 4 hr. Water (100 ml.) was added, and the benzene layer was separated, dried (Na_2SO_4), and evaporated. Distillation provided 33.6 g. of a viscous liquid; λ_{max}^{EtOH} 293 mμ (ε 211), 271 (1765), 265 (1930), 259 (1370), and a shoulder at 254 (831). Infrared bands appeared at 4.49 and 10.07 μ (neat).

Hydrolysis of 11 g. (0.05 mole) of the nitrile I with ethanolic KOH by the procedure of Cava and Mitchell[6] gave 10.7 g. of the acid II as a pale yellow solid from Skelly B; λ_{max}^{Nujol} 5.90 and 10.00 μ.

Treatment of the nitrile I (6.6 g., 0.03 mole) with 20% NaOH (10 ml.) and 30% H_2O_2 (10 ml.) in aqueous methanol[7] gave 4.1 g. of the amide III as a white, crystalline powder after recrystallization from chloroform–Skelly B; λ_{max}^{Nujol} 5.98 and 10.04 μ.

1-Aminomethyl-1-benzylbenzocyclobutene hydrochloride (IV) was prepared by reduction of I (11 g., 0.05 mole) with $LiAlH_4$ (0.15 mole) in anhydrous ether. Treatment of the undistilled amine with ethereal HCl provided 11 g. of the hydrochloride as white needles after recrystallization from ethanol; λ_{max}^{Nujol} 10.03 μ.

The reaction of 5.7 g. (0.022 mole) of IV in a solution of triethylamine (4.5 g.) and chloroform (125 ml.) with an equivalent amount of ethyl chloroformate gave 4.4 g. of **N-carbethoxy-1-aminomethyl-1-benzylbenzocyclobutene (V)** as a viscous liquid; λ_{max}^{Neat} 5.89 and 10.04 μ.

1-Cyano-1-(2-dimethylaminoethyl)benzocyclobutene (VI) was synthesized from 11.6 g. (0.09 mole) of 1-cyanobenzocyclobutene, 0.1 mole of N,N-dimethylaminoethyl chloride, and 0.09 mole of sodamide as described for the preparation of I. The distilled product amounted to 12 g.; λ_{max}^{EtOH} 270 mμ (ε 1530), 264 (1580), 258 (1040), and a shoulder at 253 (587). Infrared bands appeared at 4.49 and 10.07 μ (neat).

A 6-g. (0.03 mole) sample of VI in 15 ml. of concentrated H_2SO_4 was allowed to stand at 25° for a total of 4 days and then was poured into ice water. The aqueous solution was neutralized (NH_4OH) and saturated with NaCl. Chloroform extraction afforded 5.4 g. of an oil which slowly crystallized during refrigeration under Skelly B. The yield of the amide VII was 4.7 g. after recrystallization from chloroform–Skelly B; λ_{max}^{Nujol} 6.02 and 10.05 μ.

The ester VIII was prepared from 6 g. (0.03 mole) of VI and 0.03 mole of water in 75 ml. of ethanolic HCl by the method of Goering and co-workers.[8] The product amounted to 4.4 g. after recrystallization from ethanol–ether; λ_{max}^{Nujol} 5.81 and a shoulder at 10.03 μ.

1-Cyano-1-(2-cyanoethyl)benzocyclobutene (IX).—A stirred solution of 25.8 g. (0.20 mole) of 1-cyanobenzocyclobutene and 2 ml. of 30% methanolic KOH in 75 ml. of *t*-butyl alcohol at 10° was treated dropwise with 11.7 g. (0.22 mole) of acrylonitrile in 30 ml. of *t*-butyl alcohol. The temperature was maintained

(2) (a) F. R. Jensen and W. E. Coleman, *J. Am. Chem. Soc.*, **80**, 6149 (1958); (b) M. P. Cava, R. J. Pohl, and M. J. Mitchell, *ibid.*, **85**, 2080 (1963); (c) A. T. Blomquist and C. G. Bottomley, *Ann.*, **653**, 67 (1962).

(3) M. P. Cava and D. R. Napier, *J. Am. Chem. Soc.*, **80**, 2255 (1958).

(4) Melting points were taken with a Thomas–Hoover capillary apparatus and are corrected. Analyses were performed in our laboratories under the direction of Mr. E. Kluchesky and by Drs. G. Weiler and F. B. Strauss, Oxford, England. The ultraviolet spectra were obtained with a Beckman spectrophotometer, Model DK2A, and the infrared spectra with a Beckman spectrophotometer, Model IR8.

(5) J. F. Bunnett and J. A. Skorcz, *J. Org. Chem.*, **27**, 3836 (1962).

(6) M. P. Cava and M. J. Mitchell, *ibid.*, **27**, 631 (1962).

(7) M. P. Cava, R. L. Litle, and D. R. Napier, *J. Am. Chem. Soc.*, **80**, 2257 (1958).

(8) H. L. Goering, S. J. Cristol, and K. Dittmer, *ibid.*, **70**, 3314 (1948).

below 20° during the 1-hr. addition period. The solution was stirred overnight at 25°, neutralized with 20% HCl, diluted with water, and extracted with two 200-ml. portions of ether, which were combined, dried (Na_2SO_4), and evaporated. Distillation provided 25.8 g. of product; λ_{max}^{EtOH} 270 mμ (ε 1490), 264 (1530), 258 (1020), and a shoulder at 253 (580). Infrared bands appeared at 4.46, 4.48, and 10.03 μ (CCl_4).

A mixture of the dinitrile IX (1.8 g., 0.01 mole) and 25 ml. of concentrated HCl was stirred vigorously at 25°; complete solution occurred within 30 min. After 20 min. of additional stirring, the yellow solution was poured onto ice, and the deposited solid was filtered, washed with water, and dried. The diamide X weighed 1.4 g. and was recrystallized from ethanol; λ_{max}^{Nujol} 6.05, 6.14, and 10.03 μ.

A mixture of the dinitrile IX (3.6 g., 0.02 mole) and 60 ml. of concentrated HCl was refluxed for 4 hr. The solution was cooled, and the precipitated solid was filtered, washed with water, and dried. Recrystallization from water afforded 3.55 g. of the diacid XI as a white, crystalline powder; λ_{max}^{Nujol} 5.80 and a shoulder at 10.07 μ.

The diacid XI (4.4 g., 0.02 mole) and 4.1 g. (0.04 mole) of acetic anhydride were heated at an oil bath temperature of 130° for 2 hr. The resulting solution was refrigerated for 24 hr., and the precipitated material was filtered with the aid of a minimum amount of benzene. Recrystallization from benzene yielded 2.2 g. of **1-(2-carboxyethyl)benzocyclobutene-1-carboxylic acid anhydride (XII)** as a white, crystalline powder; λ_{max}^{Nujol} 5.53, 5.70, and 10.07 μ.

Spiro[benzocyclobutene-1,3′-(2′,6′-dioxopiperidine)] (XIII).—A solution of 5.6 g. (0.03 mole) of the dinitrile IX in 10 ml. of glacial acetic acid and 4 ml. of concentrated H_2SO_4 was heated at an oil bath temperature of 120° for 10 min.[9] The warm solution was poured onto ice and immediately neutralized ($NaHCO_3$). The precipitated material was taken up in two 100-ml. portions of chloroform, which were combined, washed with saturated brine solution, dried (Na_2SO_4), and evaporated. The remaining solid was recrystallized from ethyl acetate–Skelly B to afford 3.1 g. of a white powder; λ_{max}^{EtOH} 272 mμ (ε 1590), 265 (1810), 259 (1400), 253 (1130), 245 (1110), and 238 (1120). Infrared bands appeared at 6.02, 6.12, and 10.01 μ (Nujol).

N-Phthalyl-O-(1-benzocyclobutenyl)hydroxylamine (XIV).—A solution of 1-bromobenzocyclobutene[8,10] (1.83 g., 0.01 mole), prepared in 31% yield from benzocyclobutene-1-carboxylic acid by a modified[11] Hunsdiecker reaction, N-hydroxyphthalimide (1.63 g., 0.01 mole), and triethylamine (1 g., 0.01 mole) in 35 ml. of acetonitrile was refluxed for 24 hr. The solvent was evaporated; the residual solid was washed repeatedly with water and dried. Recrystallization from aqueous ethanol provided 1.5 g. of pale tan needles; λ_{max}^{Nujol} 5.59 and 5.80 μ.

O-(1-Benzocyclobutenyl)hydroxylamine Hydrochloride (XV).—The imide XIV (4 g., 0.015 mole) and 0.9 g. (0.015 mole) of 85% aqueous hydrazine hydrate in 60 ml. of methanol was refluxed with stirring for 3 hr. The methanol was removed under vacuum, and the residue was treated with 40 ml. of 10% HCl and 10 ml. of water. The mixture was stirred overnight at 25°, filtered, and evaporated to dryness. Recrystallization of the residue from ethanol–ether provided 2.3 g. of white flakes; λ_{max}^{EtOH} 273 mμ (ε 1680), 267 (1720), 261 (1120), and a shoulder at 254 (614). An infrared band appeared at 10.05 μ as a shoulder (Nujol).

1-Benzocyclobutenylglycine (XVI).—To a solution of 1.73 g. (0.075 g.-atom) of sodium in 100 ml. of absolute ethanol under nitrogen was added 12.8 g. (0.075 mole) of ethyl acetamidocyanoacetate. The pale yellow solution was stirred for 30 min. and then was treated with 13.8 g. (0.075 mole) of 1-bromobenzocyclobutene. The darkened reaction mixture was refluxed for 40 hr., cooled, and filtered. The filtrate was evaporated to near dryness, and the residue was diluted with water. The organic layer was taken up in 150 ml. of ethyl acetate, which was washed with saturated brine and dried (Na_2SO_4). Solvent evaporation afforded 16.6 g. of a brown oil which was put on a column of 450 g. of alumina. Elution with chloroform afforded 9.55 g. (47%) of ethyl 1-benzocyclobutenylacetamidocyanoacetate as a viscous liquid. A 9-g. sample of this material was refluxed in a solution of methanol (75 ml.) and 10% NaOH (75 ml.) for 2 days. The amber solution was concentrated under vacuum, diluted with water, and extracted with ethyl acetate. Neutralization with 4 *N* HCl caused the immediate precipitation of a pale tan flocculent solid. Recrystallization was effected by addition of 500 ml. of ethanol to a cold, aqueous (450 ml.) solution of the material. The white flakes weighed 2.2 g. Thin layer chromatography on silica gel G with *n*-butyl alcohol–acetic acid–water (7:1:2) gave a single spot (ninhydrin), R_f 0.43. An infrared band appeared at 10.07 μ (Nujol).

(9) E. Tagmann, E. Surry, and K. Hoffmann, *Helv. Chim. Acta*, **35**, 1235 (1952).

(10) L. Horner, A. Kirmse, and K. Muth, *Chem. Ber.*, **91**, 430 (1958).

(11) F. W. Baker, H. D. Holtz, and L. M. Stock, *J. Org. Chem.*, **28**, 514 (1963).

Some N-Substituted Dimethoxyphenylacetamides and Dimethoxyphenylethylamines

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In connection with a study of compounds related to sympathomimetic amines, a number of N-substituted dimethoxyphenylethylamines were prepared by the reduction of the corresponding amides with $LiAlH_4$.

Experimental[2]

Ethyl 3,4-dimethoxyphenylacetate[3] [b.p. 143° (2.5 mm.)] and ethyl 2,5-dimethoxyphenylacetate[4] [b.p. 139–140° (2.5 mm.)] were prepared in 65 and 82% yields, respectively, by Fisher esterification of the corresponding acids. 2-Amino-6-chlorobenzothiazole[5] and 2-amino-6-methoxybenzothiazole[6] were prepared by published procedures.

Preparation of the Amides (Table I).—Fifty milliliters of a 0.213 *M* solution of ethylmagnesium bromide in tetrahydrofuran was added dropwise to 10.7 mmoles of amine in 20 ml. of tetrahydrofuran cooled in an ice bath. The cooling bath was removed and the mixture was stirred until the initially formed precipitate dissolved. To the stirred solution was added 10.7 mmoles of ester in 20 ml. of tetrahydrofuran. After 2 hr. the solvent was removed at the water pump. The residue was stirred with 40 ml. of 5% NH_4Cl solution; the mixture was cooled in an ice bath, and the amide was then collected on a filter and crystallized from hot aqueous ethanol subsequent to decolorization with Norit. The results are summarized in Table I.

Preparation of the Amines (Table II).—A solution of 1.1 g. of $LiAlH_4$ in 100 ml. of diethyl ether was added to 4 mmoles of amide dissolved in 80 ml. of hot benzene. The mixture was refluxed for 72 hr. The solution was then cooled and cautiously heated with 25 ml. of water followed by 25 ml. of 10% H_2SO_4. The aqueous phase was separated, brought to pH 5 by addition of solid Li_2CO_3, and filtered. The filtrate, heated to 70°, was treated with 1.2 g. of picric acid. The picrate which separated was filtered from the cooled solution, and crystallized from aqueous ethanol. The purified picrate was suspended in 10% aqueous NaOH and the liberated amine was extracted from the mixture with ether. The ether extract was evaporated and the residual amine crystallized from the solvent indicated in Table II.

(1) Chemistry Department, State University of New York at Buffalo, Buffalo, N. Y. 14214.

(2) All melting points were determined on a hot stage apparatus and are uncorrected.

(3) J. G. Watkinson, W. Watson, and B. L. Yates, *J. Chem. Soc.*, 5437 (1963); R. Quelet and J. Gavavett, *Compt. rend.*, **230**, 394 (1950); C. Schoepf, G. Goettmann, E. Meisel, and L. Neuroth, *Ann.*, **563**, 86 (1949).

(4) G. Leaf and A. Neuberger, *Biochem. J.*, **43**, 606 (1948).

(5) G. M. Dyson, R. F. Hunter, and R. W. Morris, *J. Chem. Soc.*, 1186 (1927).

(6) C. G. Stuckwisch, *J. Am. Chem. Soc.*, **71**, 3417 (1949).