The infrared spectrum has absorption maxima that are attributable to hindered carbonyl, 1,2,4-trisubstituted phenyl and *t*-butyl groups and to the fluorine atom.

Action of *t*-Butylmagnesium Chloride on Duryl *p*-Fluorophenyl Ketone.[12]—The procedure was similar to that just described. The crude product weighed 2.45 g. (84% yield) and, after crystallization from ethanol, melted at 126–127°. A mixed melting point with an authentic sample of *p*-*t*-butylphenyl duryl ketone (V) was not depressed.

URBANA, ILLINOIS

[CONTRIBUTION FROM THE STERLING–WINTHROP RESEARCH INSTITUTE]

Derivatives of 4-Amino-2-hydroxybenzoic Acid. III. Thiol Esters

BY R. O. CLINTON, U. J. SALVADOR AND S. C. LASKOWSKI

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Dialkylaminoalkyl ester derivatives of 4-amino- and 4-alkylamino-2-alkoxythiolbenzoic acids have been prepared for testing as local anesthetics. Among these thiol esters have been found compounds far surpassing in activity any local anesthetics hitherto known.

In previous communications[1,2] from these laboratories there have been described several series of dialkylaminoalkyl-4-amino- and 4-alkylamino-2-hydroxybenzoates and the corresponding 2-alkoxy- and 2-benzyloxybenzoates, prepared in a search for more active local anesthetics.

Prior investigations[3] have shown that basic esters derived from thiolbenzoic acids are frequently more active as local anesthetics than the corresponding oxygen analogs (*e.g.*, 2-diethylaminoethyl 4-aminothiolbenzoate (Thiocaine) *vs.* 2-diethylaminoethyl 4-aminobenzoate (procaine)[4]), although this increase in activity is usually accompanied by a proportionate increase in toxicity and a *more* than proportionate increase in irritancy.[5] However, when we extended previous series[1,2] to include the corresponding thiol esters, a surprisingly large increase in local anesthetic activity was noted and, further, this increase in activity was not accompanied by a proportionate increase in irritancy.[6]

A single position isomer of the compounds presently described has appeared in the literature. Harris and Braker[7] have prepared 2-diethylaminoethyl 3-amino-4-butoxythiolbenzoate, but no pharmacological data on this compound have been published.

The dialkylaminoalkyl 2-alkoxy-4-nitrothiolbenzoates were readily synthesized from the parent 2-alkoxy-4-nitrobenzoic acids[2] and dialkylaminoalkylthiols[8] by a modification of the procedure used for the preparation of the corresponding oxygen analogs.[2] Reduction to the 4-amino derivatives and subsequent reductive alkylation to the 4-alkylamino derivatives offered no difficulties. The alternative path used for the preparation of the latter compounds, *i.e.*, through condensation of a dialkylaminoalkanethiol with a 4-alkylamino-2-alkoxybenzoyl chloride hydrochloride, gave poor yields, and purification of the products was very difficult.

The thiol ester derivatives of 4-amino-2-hydroxybenzoic acid are listed in Tables I–III.

In order to determine whether the unexpectedly high local anesthetic activity found with the dialkylaminoalkyl 2-alkoxy-4-aminothiolbenzoates was anomalous, or could be ascribed to other alkoxy aminothiolbenzoates, we also prepared several position isomers of this type. In these isomers the alkoxy group occupied the 2-, 3- or 4-position and the amino group occupied the 3-, 4- or 5-position; included was the example given by Harris and Braker.[7] All of these compounds proved to be much less active and much more irritating than the corresponding 2-alkoxy-4-amino analogs. The properties of these compounds are listed in Table IV.

Experimental[9]

Dialkylaminoalkyl 2-Alkoxy-4-nitrothiolbenzoates.—All of the nitrothiolbenzoates were prepared by a standard procedure, involving a definite ratio of components. An example follows.

To a stirred mixture of 26.7 g. (0.10 mole) of 2-hexyloxy-4-nitrobenzoic acid, 9.5 g. (0.12 mole) of pure, dry pyridine and 300 ml. of dry benzene was added, dropwise, a solution of 9.5 g. (0.08 mole) of pure thionyl chloride in 50 ml. of dry benzene during a period of ten minutes. The heterogeneous mixture was stirred and refluxed for ten minutes, cooled to 40°, and there was added slowly with stirring a solution of 8.0 g. (0.06 mole) of 2-diethylaminoethanethiol in 50 ml. of dry benzene. After stirring for ten minutes at 50°, the mixture was cooled, water was added and the aqueous layer was made strongly alkaline by the addition of solid potassium carbonate. The benzene layer was separated and washed with water, dilute sodium bicarbonate solution, and again with water. After drying over Drierite the benzene solution was decolorized by means of Darco G-60 and concentrated to dryness *in vacuo*. The residual oil was diluted with toluene and again concentrated *in vacuo* to remove traces of pyridine. There was thus obtained crude 2-diethylaminoethyl 2-hexyloxy-4-nitrothiolbenzoate as a pale yellow oil. The yields varied from 80–90%. The derivatives were prepared by the usual methods.

Dialkylaminoalkyl 2-Alkoxy-4-aminothiolbenzoates.—These compounds were prepared by the iron–hydrochloric acid reduction of the nitrothiol esters by the general method

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[1] R. O. Clinton, S. C. Laskowski, U. J. Salvador and Mary Wilson, THIS JOURNAL, **73**, 3674 (1951).

[2] R. O. Clinton, S. C. Laskowski, U. J. Salvador and Mary Wilson, *ibid.*, **74**, 592 (1952).

[3] R. O. Clinton, U. J. Salvador and S. C. Laskowski, *ibid.*, **71**, 3366 (1949); N. F. Albertson and R. O. Clinton, *ibid.*, **67**, 1222 (1945).

[4] L. S. Fosdick and H. L. Hansen, *J. Pharmacol.*, **50**, 323 (1934); Y. K. Nolle, *Farm. i. Farmacol.* (*U.S.S.R.*), No. 2, 1 (1937) [*C. A.* **34**, 3820 (1940)].

[5] *Inter al.*, Thiocaine is more than seven times as irritating as procaine by the Standard Trypan Blue test (unpublished observations of Dr. F. P. Luduena).

[6] Preliminary pharmacological results: F. P. Luduena, R. O. Clinton and S. C. Laskowski, *Science*, **118**, 138 (1953).

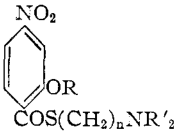
[7] S. E. Harris and W. Braker, U. S. Patent 2,342,142 (1944).

[8] *Cf.* ref. 3 and references cited therein.

[9] All melting points are corrected. They were determined in a modified Hershberg apparatus, using total-immersion N.B.S. calibrated thermometers. The sample was immersed 15° below the melting point, 3° rise per minute. The analyses were done by Mr. M. E. Auerbach, Mr. K. D. Fleischer, and their staffs.

TABLE I

DIALKYLAMINOALKYL 2-ALKOXY-4-NITROTHIOLBENZOATES

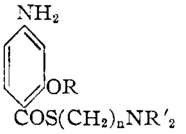


			Hydrochloride						Derivative					
					Analyses, %						Analyses, %			
					Sulfur		Nitrogen				Nitrogen[b]		Nitrogen[c]	
n	R	R'$_2$	M.p., °C.	Formula	Calcd.	Found	Calcd.	Found	Type[a]	M.p., °C.	Calcd.	Found	Calcd.	Found
2	CH_3	$(C_2H_5)_2$	146.4–147.9	$C_{14}H_{21}ClN_2O_4S$	[d]	[d]	[d]	[d]						
3	CH_3	$(C_2H_5)_2$	157.0–158.0	$C_{15}H_{23}ClN_2O_4S$	8.84	8.80								
4	CH_3	$(C_2H_5)_2$	109.0–110.6	$C_{16}H_{25}ClN_2O_4S$			3.71[c]	3.56[c]						
3	CH_3	C_5H_{10}[e]	200.0–201.1	$C_{16}H_{23}ClN_2O_4S$	8.56	8.89	[f]	[f]	B	60.1– 61.2	8.27[p]	8.16[p]	4.13	4.10
3	C_2H_5	$(C_2H_5)_2$	117.2–118.8	$C_{16}H_{25}ClN_2O_4S$			3.71[c]	3.74[c]	P	118.2–119.2	2.46	2.43	9.83	9.80
2	C_3H_7[g]	$(C_2H_5)_2$	185.0–185.8	$C_{16}H_{25}ClN_2O_4S$			7.43	7.24	P	131.4–132.2	2.46	2.49	9.83	10.02
2	C_3H_7[g]	C_5H_{10}[e]	185.9–187.7	$C_{17}H_{25}ClN_2O_4S$	8.24	8.38			B	65.6– 67.7			3.97	3.95
									P	134.2–135.0	2.40	2.40	9.63	9.97
3	C_3H_7[g]	C_5H_{10}[e]	196.6–198.0	$C_{18}H_{27}ClN_2O_4S$	7.95	8.10			F	138.4–141.4	[h]	[h]		
2	C_3H_7[g]	C_6H_{12}[i]	210.0–211.5	$C_{18}H_{27}ClN_2O_4S$	7.95	8.15			P	141.0–142.9	2.35	2.33	9.40	9.57
2	C_4H_9[j]	$(C_2H_5)_2$	171.7–173.3	$C_{17}H_{27}ClN_2O_4S$			7.16	6.96	P	110.6–112.2	2.39	2.58	9.60	9.86
2	C_4H_9[j]	C_5H_{10}[e]	179.3–180.5	$C_{18}H_{27}ClN_2O_4S$	7.95	8.10			B	55.3– 57.2	7.64[p]	7.42[p]	3.82	3.79
									P	128.9–129.6	2.35	2.34	9.40	9.47
3	C_4H_9[j]	C_5H_{10}[e]	170.0–171.0	$C_{19}H_{29}ClN_2O_4S$	7.69	7.68			F	164.5–165.2	[k]	[k]		
2	C_4H_9[j]	C_6H_{12}[i]	165.0–165.9	$C_{19}H_{29}ClN_2O_4S$	3.35[c]	3.24[c]	6.72	6.66	P	135.9–137.2	2.29	2.28	9.19	9.24
2	C_4H_9[l]	$(C_2H_5)_2$	193.4–194.6	$C_{17}H_{27}ClN_2O_4S$	8.20	8.36	3.58[c]	3.86[c]	P	103.4–104.4	2.40	2.38	9.60	9.64
2	C_5H_{11}[m]	$(C_2H_5)_2$	129.6–131.2	$C_{13}H_{29}ClN_2O_4S$	7.91	7.94	[n]	[n]	P	115.8–116.6			9.37	9.13
2	C_6H_{13}[o]	$(C_2H_5)_2$	115.4–116.6	$C_{19}H_{31}ClN_2O_4S$	7.65	7.36	3.34[c]	3.40[c]	P	96.4– 97.4	2.29	2.26	9.16	9.14
q	C_4H_9[j]	$(CH_3)_2$	124.4–128.2	$C_{16}H_{25}ClN_2O_4S$	8.50	8.52	7.43	7.49	P	150.0–155.9			9.84	9.71
r	C_4H_9[j]	$(C_2H_5)_2$	151.9–153.0	$C_{19}H_{31}ClN_2O_4S$	8.46[s]	8.48[s]	3.34[c]	3.29[c]	P	117.4–120.4			9.16	9.36

[a] B = base; F = flavianate; P = picrate. [b] Amino nitrogen, by titration with perchloric acid in glacial acetic acid solution. [c] Nitro nitrogen, by titration with titanous chloride. [d] Calcd.: C, 48.24; H, 6.07. Found: C, 48.22; H, 6.11. [e] 1-Piperidyl. [f] Calcd.: Cl, 9.45. Found: Cl, 9.10. [g] *n*-Propyl. [h] Calcd.: S, 9.41. Found: S, 9.37. [i] 2-Methyl-1-piperidyl. [j] *n*-Butyl. [k] Calcd.: S, 9.22. Found: S, 9.26. [l] *i*-Butyl. [m] *n*-Amyl. [n] Calcd.: Cl, 8.75. Found: Cl, 8.83. [o] *n*-Hexyl. [p] Total nitrogen. [q] 1-Dimethylaminopropyl-2. For the thiol used, *cf.* R. R. Renshaw, *et al.*, THIS JOURNAL, 60, 1765 (1938). [r] 1,1-Dimethyl-2-diethylaminoethyl. For the thiol used *cf.* H. R. Snyder, *et al.*, *ibid.*, 69, 2672 (1947). [s] Chlorine analyses.

TABLE II

DIALKYLAMINOALKYL 4-AMINO-2-ALKOXYTHIOLBENZOATES

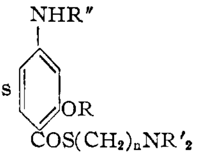


				Salt						Derivative			
						Analyses, %						Analyses, %	
							Nitrogen						
n	R	R'$_2$	Type[a]	M.p., °C.	Formula	Calcd.[c]	Found[c]	Calcd.	Found	Type[b]	M.p., °C.	Calcd.	Found
2	CH_3	$(C_2H_5)_2$	Ph[d]	188.2–190.0	$C_{14}H_{25}N_2O_6PS$	[e]	[e]	7.36	7.35	P	184.5–185.5	5.47[f]	5.31[f]
3	CH_3	$(C_2H_5)_2$	Ph	208.0–208.9	$C_{15}H_{27}N_2O_6PS$	24.85	25.02	8.12[g]	8.24[g]	F	166.2–168.1[h]	10.50[g]	10.60[g]
4	CH_3	$(C_2H_5)_2$	Ph	198.1–199.1	$C_{16}H_{29}N_2O_6PS$	24.00	24.29	7.85[g]	7.85[g]	F	171.3–172.8[i]	10.26[g]	10.02[g]
3	CH_3	C_5H_{10}[j]	Ph	202.5–203.4	$C_{16}H_{27}N_2O_6PS$	24.12	24.20	7.88[g]	7.94[g]	F	183.4–184.2[k,l]	10.30[g]	10.42[g]
3	C_2H_5	$(C_2H_5)_2$	H	175.0–176.3	$C_{16}H_{27}ClN_2O_2S$	10.22	10.40	8.07	7.83	F	175.0–177.0	10.01[g]	10.04[g]
2	C_3H_7[m]	$(C_2H_5)_2$	Ph	150.4–151.8	$C_{16}H_{29}N_2O_6PS$	24.00	23.98	6.85	6.72	..			
2	C_3H_7[m]	C_5H_{10}[j]	H	189.2–190.9	$C_{17}H_{27}ClN_2O_2S$	9.87	9.72	8.93[g]	9.05[g]	F	158.0–163.0[k]	[n]	[n]
3	C_3H_7[m]	C_5H_{10}[j]	DH	170.4–172.0[k]	$C_{18}H_{30}Cl_2N_2O_2S$	7.83[g]	7.77[g]	6.84	6.61				
2	C_3H_7[m]	C_6H_{12}[o]	DH	[p]	$C_{18}H_{30}Cl_2N_2O_2S$	17.32	17.61	6.84	7.11	DF	130.0–140.0[k]	5.80[q]	5.52[q]
2	C_4H_9[r]	$(C_2H_5)_2$	H	155.0–155.5	$C_{17}H_{29}ClN_2O_2S$	9.82	9.55	7.76	7.63	DF	179.0–181.0[k]	5.87[q]	5.84[q]
2	C_4H_9[r]	C_5H_{10}[j]	H	172.3–173.9	$C_{18}H_{29}ClN_2O_2S$	9.50	9.38	7.51	7.38	F	158.0[k]	[s]	[s]
3	C_4H_9[r]	C_5H_{10}[j]	H	155.0–157.0	$C_{19}H_{31}ClN_2O_2S$	9.16	9.35	[t]	[t]	DF	195.0–196.0[k]	5.72[q]	5.46[q]
2	C_4H_9[r]	C_6H_{12}[o]	H	191.2–192.3	$C_{19}H_{31}ClN_2O_2S$	9.16	9.02	7.24	7.28				
2	C_4H_9[u]	$(C_2H_6)_2$	H	163.0–164.2	$C_{17}H_{29}ClN_2O_2S$	9.82	9.61	7.76	7.66	DF	169.6–173.0[k]	5.87[q]	6.14[q]
2	C_5H_{11}[v]	$(C_2H_5)_2$	H	150.2–151.2	$C_{13}H_{31}ClN_2O_2S$	9.45	9.66	7.47	7.45	DF	167–174[k]	[w]	[w]
2	C_6H_{13}[x]	$(C_2H_5)_2$	H	110.2–111.6	$C_{19}H_{33}ClN_2O_2S$	9.11	9.28	7.20	7.02	DF	163–167[k]	5.71[q]	5.88[q]
y	C_4H_9[r]	$(CH_3)_2$	H	122.9–126.8	$C_{16}H_{27}ClN_2O_2S$	10.22	9.94	8.07	7.95	DF	182.9–189.2	[aa]	[aa]
z	C_4H_9[r]	$(C_2H_5)_2$	H	168.1–170.3	$C_{19}H_{33}ClN_2O_2S$	8.24[g]	8.46[g]	7.20	7.17	DF	183[k]	5.71[q,bb]	5.81

[a] Ph = phosphate, H = monohydrochloride, DH = dihydrochloride. [b] P = picrate, F = flavianate, DF = diflavianate. [c] Phosphoric acid analyses on phosphates; chlorine analyses on hydrochlorides. [d] The crystalline base had m.p. 77.0–78.0°. Calcd.: N, 9.92. Found: N, 9.75. [e] Calcd.: C, 44.19; H, 6.58; S, 8.42. Found: C, 44.19; H, 6.44; S, 8.60. [f] Table I, ref. b. [g] Sulfur analysis. [h] Shown to be a monoflavianate by the determination of primary aromatic amine through diazotization: Calcd.: N_{NH_2}, 2.29. Found: N_{NH_2}, 2.32. [i] *Cf.* ref. *h*. Calcd.: N_{NH_2}, 2.24. Found: N_{NH_2}, 2.34. [j] 1-Piperidyl. [k] With decomposition. [l] *Cf.* ref. *h*. Calcd.: N_{NH_2}, 2.25. Found: N_{NH_2}, 2.44. [m] *n*-Propyl. [n] Calcd.: C, 50.93; H, 5.06. Found: C, 51.18; H, 5.09. [o] 2-Methyl-1-piperidyl. [p] Sintered at 175–180°, not melted at 300°. [q] Table I, ref. *c*. [r] *n*-Butyl. [s] Calcd.: C, 51.68; H, 5.26. Found: C, 51.41; H, 5.45. [t] Calcd.: C, 58.97; H, 8.07. Found: C, 58.84; H, 8.10. [u] *i*-Butyl. [v] *n*-Amyl. [w] Calcd.: C, 47.19; H, 4.37. Found: C, 47.23; H, 4.58. [x] *n*-Hexyl. [y] See Ref. *q*, Table I. [z] See Ref. *r*, Table I. [aa] Calcd.: C, 46.04; H, 4.07; N, 8.95. Found: C, 46.20; H, 4.16; N, 8.74. [bb] Calcd.: C, 47.74; H, 4.52. Found: C, 48.02; H, 4.74.

TABLE III

DIALKYLAMINOALKYL 4-ALKYLAMINO-2-ALKOXYTHIOLBENZOATES

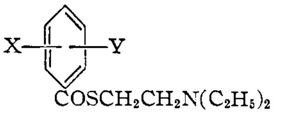


				Salt			Analyses, % Sulfur		Analyses, %		Derivative		Analyses, %	
n	R	R′₂	R″	Type[a]	M.p., °C.	Formula	Calcd.	Found	Calcd.	Found	Type[b]	M.p., °C.	Calcd.	Found
2	CH_3	$(C_2H_5)_2$	C_5H_{11}	DH	149.2–150.5	$C_{19}H_{34}Cl_2N_2O_2S$	7.57	7.44	[c]	[c]				
2	CH_3	$(C_2H_5)_2$	$C_5H_{11}O$[d]	DH	140.0–141.0	$C_{19}H_{34}Cl_2N_2O_3S$	7.25	7.29	16.08[e]	16.19[e]	P	94.2– 96.0	7.03[f]	7.27[f]
3	CH_3	$(C_2H_5)_2$	C_4H_9	B	Oil	$C_{19}H_{32}N_2O_2S$	9.09	8.91	7.94[g]	7.70[g]	F	153.9–155.0[h]	9.61[e]	9.64[e]
3	CH_3	C_5H_{10}[i]	C_4H_9	B	Oil	$C_{20}H_{32}N_2O_2S$	8.79	8.73	7.68[g]	7.94[g]	F	136.0–140.0[h]	9.44[e]	9.48[e]
4	CH_3	$(C_2H_5)_2$	C_4H_9	B	Oil	$C_{20}H_{34}N_2O_2S$	8.74	8.56	7.64[g]	7.49[g]	DF	160.5–161.6[h]	5.63[f]	5.65[f]
2	C_3H_7	$(C_2H_5)_2$	C_4H_9	DH	142.2–143.9	$C_{20}H_{36}Cl_2N_2O_2S$	7.29	7.16	16.13[e]	16.08[e]				

[a] B = base, DH = dihydrochloride. [b] P = picrate, F = flavianate, DF = diflavianate. [c] Calcd.: C, 53.89; H, 8.09. Found: C, 53.82; H, 7.89. [d] 5-Hydroxyamyl. [e] Chlorine analysis. [f] See Table I, ref. *c*. [g] Nitrogen analysis. [h] With decomposition. [i] 1-Piperidyl.

TABLE IV

2-DIETHYLAMINOETHYL ALKOXY-NITRO- AND ALKOXY-AMINOTHIOLBENZOATES



		Hydrochloride		Analyses, % Nitrogen		Analyses, % Chlorine		Derivative		Analyses, % Nitrogen	
X[a]	Y	M.p., °C.	Formula	Calcd.	Found	Calcd.	Found	Type[c]	M.p., °C.	Calcd.	Found
3-OPr	4-NO_2	130.0–131.2	$C_{16}H_{25}ClN_2O_4S$	3.71[b]	3.71[b]	9.41	9.10	P	135.0–136.4	9.84	10.02
2-OBu	3-NO_2	112.6–113.4	$C_{17}H_{27}ClN_2O_4S$	3.58[b]	3.60[b]	9.07	9.05	P	68.4–70.6	9.60	9.56
2-OBu	5-NO_2	169.0–170.5	$C_{17}H_{27}ClN_2O_4S$	3.58[b]	3.55[b]	9.07	9.23	P	110.5–111.9	9.60	9.44
4-OBu	3-NO_2	139.6–141.2[d]	$C_{17}H_{27}ClN_2O_4S$	3.58[b]	3.52[b]	9.07	8.97	P	128.6–129.6	9.60	9.64
3-OPr	4-NH_2	188.8–191.0	$C_{16}H_{28}Cl_2N_2O_2S$	7.30	7.13	18.50	18.30	DF	188.0–189.5[e]	5.96	5.83
2-OBu	3-NH_2	191.0–192.3	$C_{17}H_{30}Cl_2N_2O_2S$	7.05	6.92	17.84	17.53	F	147.7–150.1	[f]	[f]
2-OBu	5-NH_2	123.0–127.0	$C_{17}H_{30}Cl_2N_2O_2S$	7.05	7.35	8.06[g]	7.87[g]	[h]	184.7–185.7	[h]	[h]
4-OBu	3-NH_2	115.2–115.8[i]	$C_{17}H_{30}Cl_2N_2O_2S$	7.05	7.01	17.84	18.00				

[a] OPr = *n*-propoxy; OBu = *n*-butoxy. [b] *Cf.* Table I, footnote *c*. [c] P = picrate, F = flavianate, DF = diflavianate. [d] Harris and Braker, ref. 7, reported m.p. 100–105°. [e] With decomposition. [f] Calcd.: C, 50.77; H, 5.37. Found: C, 51.05; H, 5.48. [g] Sulfur analyses. [h] Hemiflavianate. Calcd.: C, 48.28; H, 4.70; S, 10.07. Found: C, 47.90; H, 4.98; S, 9.97. [i] Reported (ref. 7) m.p. 123–125°.

previously outlined.[2] The aminothiol esters were obtained in yields of 85–95%.

Dialkylaminoalkyl 2-Alkoxy-4-alkylaminothiolbenzoates.—The reductive alkylation of the aminothiol esters by means of an aldehyde, zinc dust and acetic acid[1] gave the bases in yields of 80–85%. This procedure was found preferable to the direct preparation from a 2-alkoxy-4-alkylaminobenzoyl chloride hydrochloride and a dialkylaminoalkanethiol. In certain cases (*cf.* Table III) where a crystalline water-soluble derivative could not be prepared, the bases were purified by a method previously outlined.[1]

Alkoxynitrobenzoic Acids.—The two new alkoxynitrobenzoic acids used in the present work were prepared by alkylation of the hydroxynitrobenzoic acids with butyl benzenesulfonate as previously described,[2] followed by saponification of the oily intermediate esters with sodium hydroxide in dilute alcoholic solution.

2-Butoxy-3-nitrobenzoic acid: yellow needles from Skellysolve B, m.p. 52.5–54.0°.

Anal. Calcd. for $C_{11}H_{13}NO_5$: C, 55.20; H, 5.49; N, 5.86. Found: C, 55.65; H, 5.18; N, 5.81.

2-Butoxy-5-nitrobenzoic acid: yellow prisms from ethanol, m.p. 117.4–118.9°

Anal. Calcd. for $C_{11}H_{13}NO_5$: C, 55.20; H, 5.49; N, 5.86. Found: C, 55.43; H, 5.32; N, 5.92.

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