changed acrylonitrile. I was obtained as amber colored residual oil in quantitative yield [n_D^{30} 1.4740, d_{20}^{20} 1.0665, infrared maxima at 3.03, 3.55 μ (NH_2), 4.3 μ ($C\equiv N$), 6.29, 6.75 μ (NH), 7.1, 7.4 μ (N—N), 8.05, 8.96 μ (C—N)].

Anal. Calcd. for $C_6H_{10}N_4$: C, 52.15; H, 7.25; N, 40.55. Found: C, 52.40; H, 7.30; N, 40.72.

Treatment of I with phenyl isocyanate in benzene gave *1,1-bis(2-cyanoethyl)-4-phenylsemicarbazide)* (m.p. 107° from methanol).

Anal. Calcd. for $C_{13}H_{15}N_5O$: C, 60.68; H, 5.88; N, 27.22. Found: C, 60.81; H, 5.91; N, 28.14.

3-Hydrazino-3-methylpropionitrile. A solution of 100 g. (2.0 moles) of hydrazine hydrate (64%) was held at 30–40° during the addition of 268 g. (4 moles) of crotononitrile and allowed to stand at ambient temperature overnight. The excess crotononitrile was removed by distillation and the residue stripped to 60° at 13 mm. to give a quantitative yield (200 g.) of impure mono-adduct [n_D^{30} 1.4651, d_{20}^{20} 1.0125, infrared maxima at 3.03, 3.55 μ (NH_2), 4.3 μ ($C\equiv N$), 6.22 μ (NH), 7.04, 7.41 μ (N—N) and 7.25 μ (CH_3)].

Anal. Calcd. for $C_4H_9N_3$: N, 42.39. Found: N, 40.1.

Treatment of the above compound with phenyl isocyanate in benzene gave *1-(2-cyanopropyl)-4-phenylsemicarbazide* (m.p. 128.5–129.5° from methanol).

Anal. Calcd. for $C_{11}H_{14}N_4O$: C, 60.53; H, 6.47; N, 25.67. Found: C, 60.52; H, 6.19; N, 26.19.

3-Hydrazino-2-methylpropionitrile. The same procedure as described above was employed using methacrylonitrile to give a quantitative yield of amber oil [n_D^{30} 1.4635, infrared maxima at 3.00, 3.06 μ (NH_2), 4.42 μ ($C\equiv N$), 6.20 μ (NH)].

Anal. Calcd. for $C_4H_9N_3$: C, 48.46; H, 9.08; N, 42.39. Found: C, 48.45; H, 9.08; N, 42.4.

Treatment of the above compound with phenyl isocyanate in benzene gave *1-(1-cyanoisopropyl)-4-phenylsemicarbazide* (m.p. 124–125.5°).

Anal. Calcd. for $C_{11}H_{14}N_4O$: C, 60.53; H, 6.47; N, 25.67. Found: C, 60.8; H, 6.17; N, 26.2.

Treatment of 1,1-bis(2-cyanoethyl)hydrazine (I) *with ethyl chloroformate.* A solution of 140 g. (1.0 mole) of I, 200 ml. of water, and 200 ml. of benzene was stirred at 5–10° during the separate addition in four equal portions of 106 g. (1.0 mole) of ethyl chloroformate and 40 g. (1.0 mole) of sodium hydroxide in 60 ml. of water. The mixture was held at 5° for 1 hr. and then filtered to separate 5 g. of a solid product. Crystallization of this solid from acetone gave a 3.3% yield crystalline product whose analysis indicates it to be III.

1,1,5,5-Tetra(2-cyanoethyl)carbohydrazide (III) [m.p. 191°, infrared maxima at 3.02, 3.12, 6.02, 6.61 μ (NH), 4.41 μ ($C\equiv N$), 5.81–5.83 μ (C=O), 7.06, 7.37 μ (N—N)].

Anal. Calcd. for $C_{13}H_{18}N_8O$: C, 51.85; H, 5.98; N, 37.20. Found: C, 51.93; H, 2.61; N, 38.8.

The aqueous, salt-containing layer of the filtrate was separated and extracted twice with 200 ml. portions of ethyl ether. The combined organic fractions were stripped of solvent on a rotary evaporator to 2 mm. The residual oil on standing partially crystallized. The solid (29 g.) was removed and crystallized from benzene-petroleum ether (b.p. 60–75°) to give a crystalline product whose analysis indicates it to be the expected product II.

Ethyl 2,2-bis(2-cyanoethyl)carbazate (II) [m.p. 91–93°, infrared maxima at 3.02, 6.65 μ (NH), 4.40 μ ($C\equiv N$), 5.80 μ (NH—C=O), 8.05 μ (C—O—C)].

Anal. Calcd. for $C_9H_{14}N_4O_2$: C, 51.42; H, 6.71; 26.68. Found: C, 51.7; H, 6.80; N, 27.3.

Analysis of the mobile amber colored filtrate (125 g.) indicated it was probably V, though this assignment of structure is quite tenuous since it is based solely on analogous reactions, analytical, and spectral data.

1-(2-Cyanoethyl)-3-carboethoxyiminopyrazolidine (Va). [n_D^{30} 1.4673, d_{20}^{20} 1.080, infrared maxima at 3.00 μ (ring NH), 4.40 μ ($C\equiv N$), 5.95 μ (N=C), 5.85 μ (C=O ester), 6.65 μ (ring NH), 7.05 μ (N—N), 8.03, 8.22 μ (C—O—C)]. Molecular weight. Calcd.: 210. Found: 200 ± 5.

Anal. Calcd. for $C_9H_{14}N_4O_2$: C, 51.42; H, 6.71; N, 26.68. Found: C, 51.39; H, 7.03; N, 27.4.

Acknowledgment. The author is grateful to Dr. H. F. White for aid in interpretation of infrared spectra, and to Dr. H. H. Wasserman, Yale University, for his helpful comments.

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The Rearrangement of *N*-(Methylaminoalkyl)anilides[1]

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Received October 3, 1960

Although the intramolecular migration of acyl groups from N→O is well known,[2] fewer examples of intramolecular N→N′ acyl migration have been recorded. Migrations involving loss of amine with the formation of lactams have been reported by Holley and Holley[3] and by Stirling[4] and rearrangements without loss of amine have also been described.[4,5,6] We would like to describe some additional intramolecular N→N′ acyl migrations observed in a series of *N*-[2-(and 3)*sec*-aminoalkyl]-anilides.

N-(2-Methylaminoethyl)acetanilide (I) was prepared by the reductive debenzylation of *N*-(2-benzylmethylaminoethyl)acetanilide. Distillation of the crude product resulted in a mixture of the expected compound (I) and *N*-(2-anilinoethyl)-*N*-methylacetamide (III). The latter compound resulted from the intramolecular rearrangement of I. The structure of III was proved by the lithium aluminum hydride reduction of III to *N*-ethyl-*N*-methyl-*N*′-phenylethylenediamine (IV) identical with the compound obtained by the reaction of 2-chloro-*N*-methyldiethylamine (V) with aniline.

This rearrangement appears to take place through a cyclic transition state (II). A similar mechanism has been described by Stirling[4] for the rearrangement of *N*-(2-aminoethyl)benzanilide, and by others[7] for a variety of intramolecular N→O and O→N acyl migrations.

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[1] Presented in part at the New York Meeting in Miniature, March 1960, and at the 138th meeting of the American Chemical Society, New York, N. Y., September 1960.

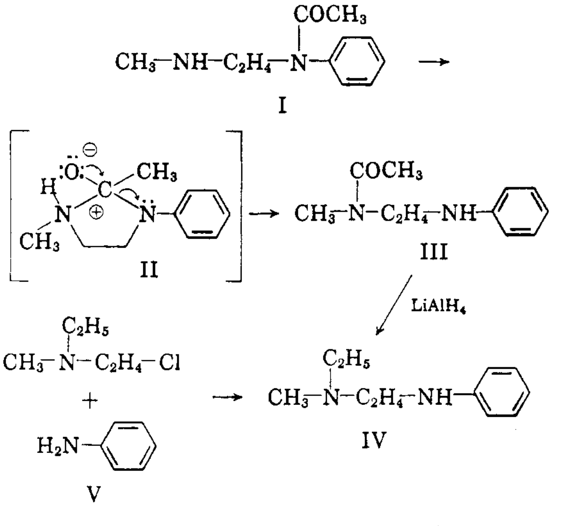
[2] Houben-Weyl, *Methoden der Organischen Chemie*, Thieme, Stuttgart, 1957, Vol. XI, Part 1, p. 936.

[3] R. W. Holley and A. D. Holley, *J. Am. Chem. Soc.*, **74**, 3069 (1952).

[4] C. J. M. Stirling, *J. Chem. Soc.*, 4531 (1958).

[5] O. Widman, *J. prakt. Chem.*, [2] **47**, 343 (1893).

[6] (a) H. Rupe, R. Paltzer, and K. Engel, *Helv. Chim. Acta*, **20**, 209 (1937); (b) A. Gassmann and H. Rupe, *Helv. Chim. Acta*, **22**, 1241 (1939); (c) H. Rupe and W. Frey, *Helv. Chim. Acta*, **22**, 673 (1939).



Other homologs of I (Table I) were also prepared and rearranged. The compounds containing two methylene groups between the nitrogens (I, VI) or a methyl on the carbon atom adjacent to the anilide nitrogen atom (VII) rearranged easily at temperatures of 90–130° and much rearrangement occurred during distillation. *N*-(2-Methylaminopropyl)propionanilide (VIII), in which a methyl group is attached to the carbon atom adjacent to the amine nitrogen atom, was more stable and could be distilled with little rearrangement; however, rearrangement occurred rapidly at 175–185°. *N*-(3-Methylaminopropyl)propionanilide (IX), which would rearrange through a six-membered transition state, was easily distilled and required temperatures above 200° for satisfactory rearrangement. Substitution of a benzoyl for a propionyl group decreased stability as exemplified by *N*-(2-methylaminopropyl)benzamide (X) which rearranged in ten minutes at 120–125°. This is consistent with the facile rearrangement of benzoyl groups observed by Stirling[4] and by Rupe *et al.*[6] The salts of the anilides are stable at room temperature, whereas the bases slowly rearrange on standing.

An experiment designed to investigate the possibility of an *inter*molecular acyl migration emphasized the *intra*molecular requirements of this rearrangement. When *N*-methylacetanilide was heated with *N*-methyl-*N*′-phenylethylenediamine under conditions identical with those used for the rearrangement of *N*-(2-methylaminoethyl)acetanilide (I), no high boiling product formed by migration of the acyl group was observed. Intermolecular N→N′ migration of acyl groups has been reported[8] but acid catalysis was generally required.

The progress of the rearrangement was readily followed by observing increases in viscosity, index of refraction and boiling point, and a decrease in basicity for the rearranged products. Characteristic changes also occurred in the infrared absorption curves: the N—H stretching band became much more intense, confirming a change from an aliphatic to an aromatic amine; the band at 3.58 μ, a characteristic of secondary aliphatic amines,[9] was absent in the rearranged products; the carbonyl band shifted from about 6.02 μ to about 6.10 μ in changing from an aromatic amide to an aliphatic amide. Such a shift in the carbonyl region is consistent with the observed bands at 6.02 μ for *N*-methylpropionanilide and at 6.10 μ for *N*,*N*-diethylacetamide.

EXPERIMENTAL[10]

The following two *N*-(benzylmethylaminoalkyl)anilides are new. The others have been described.[11]

N-(2-Benzylmethylaminopropyl)-2-phenylacetanilide. A mixture of 25.4 g. of N^2-benzyl-N^2-methyl-N^1-phenyl-1,2-propanediamine, 20.2 g. of phenylacetyl chloride, and 50 ml. of benzene was heated at reflux for 3 hr. The reaction mixture was cooled and shaken with 27 ml. of 5*N* sodium hydroxide and the layers were separated. The aqueous layer was extracted with ether. The organic layers were combined, dried over magnesium sulfate, and distilled. *N*-(2-Benzylmethylaminopropyl)-2-phenylacetanilide, b.p. 190–200° (0.2 mm.), n_D^{25} 1.578, was obtained in 44% yield.

Anal. Calcd. for $C_{25}H_{28}N_2O$: C, 80.6; H, 7.6; N, 7.5. Found: C, 80.2; H, 7.9; N, 7.6.

N-(2-Benzylmethylaminopropyl)benzanilide. A solution of 25.4 g. of N^2-benzyl-N^2-methyl-N^1-phenyl-1,2-propanediamine in 50 ml. of benzene was cooled and stirred while 15.4 g. of benzoyl chloride was added. The reaction mixture was heated at reflux for 1 hr. and then stirred with 50 ml. of 2.5*N* sodium hydroxide solution. The benzene layer was separated and the aqueous layer was extracted with ether. The combined organic layers were dried over magnesium sulfate and distilled. *N*-(2-Benzylmethylaminopropyl)-benzanilide, b.p. 190–193° (0.1 mm.), was obtained in 49% yield.

Anal. Calcd. for $C_{24}H_{26}N_2O$: C, 80.4; H, 7.3; N, 7.8. Found: C, 80.3; H, 7.5; N, 7.9.

The preparation and rearrangement of the compounds described in Table I are illustrated by the following examples. Reaction conditions and properties of the compounds are recorded in the tables.

N-(2-Methylaminoethyl)acetanilide (I). A mixture of 15.9 g. of *N*-(2-benzylmethylaminoethyl)acetanilide hydrochloride, 100 ml. of 90% ethanol, and 1.0 g. of 10% palladium-on-carbon catalyst was reduced in a Parr hydrogenator under an initial hydrogen pressure of about 3 atm. until reduction was complete. The mixture was filtered and the filtrate was concentrated to a crystalline residue. Recrystallization from ethanol resulted in a 75% yield of *N*-(2-methylaminoethyl)-acetanilide hydrochloride, m.p. 165–167°.

The hydrochloride was dissolved in water and made basic with sodium hydroxide. The mixture was extracted with ether, and the ether layer was dried over magnesium sulfate and distilled. A 69% yield of *N*-(2-methylaminoethyl)-

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(8)(a) A. Galat and G Elion, *J. Am. Chem. Soc.*, **65**, 1566 (1943); (b) R. Jaunin, M. B. Piccoli, and T. Charalambous, *Helv. Chim. Acta*, **37**, 216 (1954).

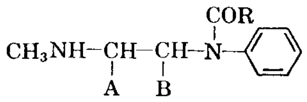
(9) W. B. Wright, Jr., *J. Org. Chem.*, **24**, 1362 (1959).

(10) All temperatures are uncorrected.

(11) W. B. Wright, Jr., H. J. Brabander, and R. A. Hardy, Jr., *J. Org. Chem.*, **26**, 485 (1961).

TABLE I

N-(METHYLAMINOALKYL)ANILIDES

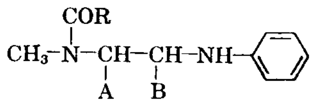


Compound	A	B	R	Yield,[a] %	B.P., mm.	n_D^{25}	Carbonyl[b] μ	Salt,[c] M.P.	Formula[d]	Carbon, % Calcd.	Carbon, % Found	Hydrogen, % Calcd.	Hydrogen, % Found	Chlorine, % Calcd.	Chlorine, % Found	Nitrogen, % Calcd.	Nitrogen, % Found
I	H	H	CH_3	75	115–120 (0.2)	1.531	6.02	165–167	$C_{11}H_{17}ClN_2O$	57.8	58.0	7.5	7.7	15.5	15.4	12.2	12.2
VI	H	H	C_2H_5	84	90– 98 (0.08)	1.526	6.03	156–158	$C_{12}H_{19}ClN_2O$	59.5	59.2	7.5	8.0	14.6	14.9	11.5	11.7
VII	H	CH_3	C_2H_5	76	100–102 (0.2)	1.522	6.02	177–179	$C_{13}H_{21}ClN_2O$	60.8	60.5	8.3	8.5	13.8	13.9	10.9	10.7
VIII	CH_3	H	C_2H_5	71[e]	112–116 (0.2)	1.521	6.02	104–106[d]	$C_{13}H_{22}N_2O_5S$	49.1	49.1	7.0	7.2			8.8	8.7
X	CH_3	H	C_6H_5	89		1.564	6.08	181–183	$C_{17}H_{21}ClN_2O$	67.0	67.1	6.9	6.9	11.6	11.3	9.2	9.2
IX	[f]	[g]	C_2H_5	95	114–118 (0.1)	1.522	6.02	161–163	$C_{13}H_{21}ClN_2O$	60.8	60.5	8.3	8.6	13.8	13.9	10.9	11.0

[a] Isolated as the hydrochloride unless otherwise noted. [b] Location of carbonyl band in the infrared absorption curve. [c] Hydrochloride unless otherwise noted. [d] Formulas and analyses are for the salt. [e] Isolated as the distilled base. [f] Sulfate. [g] *N*-(3-Methylaminopropyl)propionanilide.

TABLE II

N-ANILINOALKYL-*N*-METHYLAMIDES (REARRANGED PRODUCTS)



Compound	Compound Rearranged	Time,[a,b] Hr.	Temp.[a]	B.P., Mm.	n_D^{25}	Carbonyl[c] μ	Picrate, M.P.	Formula[d]	Carbon, % Calcd.	Carbon, % Found	Hydrogen, % Calcd.	Hydrogen, % Found	Nitrogen, % Calcd.	Nitrogen, % Found
III	I	3.5	135–140[e]	134–138 (0.1)	1.560	6.10	132–133	$C_{11}H_{16}N_2O$	68.7	68.7	8.4	8.5	14.6	14.7
XII	VI		100–130[f]	128–134 (0.08)	1.551	6.07	161–162	$C_{12}H_{18}N_2O$	69.9	69.8	8.8	8.8	13.6	13.3
XIII	VII	0.7	160–170	135–140 (0.1)	1.547	6.10	172–173	$C_{13}H_{20}N_2O$	70.9	70.9	9.2	9.7	12.7	12.5
XIV	VIII	1.0	175–185	138–140 (0.15)	1.550	6.12	132–133	$C_{13}H_{20}N_2O$	70.9	70.9	9.2	9.3	12.7	12.5
XV	X	1.0	120–125[g]	[h]		6.16		$C_{17}H_{20}N_2O$	76.1	75.9	7.5	7.7	10.4	10.6
XVI	IX	2.5[i]	215–225	155–160 (0.2)	1.545	6.07	126–128	$C_{13}H_{20}N_2O$	70.9	70.8	9.2	9.4	12.7	12.4

[a] Temperature and time of heating period before distillation. [b] Unless otherwise indicated maximum refractive index was reached by this time, often earlier. [c] Location of carbonyl band in infrared absorption curve. [d] Formulas and analyses are for bases. [e] Also rearranged on heating overnight on the steam bath or in less than 2 weeks at room temperature. [f] This sample was collected as by product of the distillation of the anilide. [g] Sample solidified after 10 min. at 120–125°. [h] M.p. 144–146°. [i] Refractive indices and infrared absorption spectra indicated that rearrangement was incomplete.

acetanilide (I), b.p. 115–120° (0.2 mm.), n_D^{25} 1.531, was obtained. *N*-(2-Anilinoethyl)-*N*-methylacetamide, n_D^{25} 1.560, was isolated from the residue in the flask. Two weeks later the distilled base (I) was reexamined and had rearranged to III, n_D^{25} 1.558.

N-(2-Anilinoethyl)-N-methylacetamide (III). *A. The rearrangement of N-(2-methylaminoethyl)acetanilide* (I). Nine grams of *N*-(2-methylaminoethyl)acetanilide was placed in a distilling flask and warmed in a Woods metal bath held at 135–140°. The index of refraction and infrared absorption curve were measured at intervals. Rearrangement was complete in 3.5 hr. The product was distilled and 7.8 g., n_D^{25} 1.560, was collected at 134–138° (0.1 mm.).

B. Attempted preparation by the reaction of N-methylacetanilide with N-methyl-N′-phenylethylenediamine. N-Methyl-*N′*-phenylethylenediamine hydrochloride, m.p. 167–169°, was prepared by the catalytic debenzylation of *N*-benzyl-*N*-methyl-*N′*-phenylethylenediamine hydrochloride.[10]

Anal. Calcd. for $C_9H_{15}ClN_2$: C, 57.9; H, 8.1; Cl, 19.0; N, 15.0. Found: C, 58.0; H, 8.4; Cl, 19.2; N, 15.3.

The base, b.p. 84–90° (0.2 mm.), n_D^{25} 1.559, was obtained when the hydrochloride was treated with aqueous alkali and the oil which separated was extracted into ether and distilled.

A mixture of 3.0 g. of *N*-methyl-*N′*-phenylethylenediamine and 3.0 g. of *N*-methylacetanilide was heated over a Woods metal bath for 6 hr. at 170–200°. On distillation, all material boiled below 100° (0.1 mm.).

N-Ethyl-N-methyl-N′-phenylethylenediamine (IV). *A. By the reduction of N-(2-anilinoethyl)-N-methylacetamide* (III). A solution of 7.7 g. of *N*-(2-anilinoethyl)-*N*-methylacetamide, obtained by the previously described rearrangement, in 50 ml. of tetrahydrofuran was added to a solution of 3.0 g. of lithium aluminum hydride in 100 ml. of tetrahydrofuran. The mixture was heated at reflux for 4 hr. and then treated with 3 ml. of water, 3 ml. of 15% sodium hydroxide, and 9 ml. of water. The solid was filtered off and washed with tetrahydrofuran. The filtrate was dried over magnesium sulfate and then distilled. The yield of *N*-ethyl-*N*-methyl-*N′*-phenylethylenediamine, b.p. 86–92° (0.3 mm.), n_D^{25} 1.532, was 85%.

Anal. Calcd. for $C_{11}H_{18}N_2$: C, 74.1; H, 10.2; N, 15.7. Found: C, 74.4; H, 10.3; N, 15.9.

Hydrochloride, m.p. 117–119°.

Anal. Calcd. for $C_{11}H_{19}ClN_2$: C, 61.5; H, 8.9; Cl, 16.5; N, 13.0. Found: C, 61.9; H, 9.1; Cl, 16.2; N, 12.9.

Picrate, m.p. 125–127°.

Anal. Calcd. for $C_{17}H_{21}N_5O_7$: C, 50.1; H, 5.2; N, 17.2. Found: C, 50.1; H, 5.0; N, 17.2.

B. By the reaction of aniline with 2-chloro-N-methyldiethylamine (V). A solution of 196.5 g. of thionyl chloride in 1200 ml. of chloroform was cooled to −5° and a solution of 103 g. of 2-(ethylmethylamino)ethanol in 200 ml. of chloroform was added over a 1-hr. period at this temperature. The reaction mixture was allowed to come to room temperature and was then concentrated until most of the chloroform was removed. Ethanol (75 ml.) was added and concentration was continued to dryness. This procedure was repeated twice. The crystalline residue was triturated with acetone, filtered, washed with acetone, and dried at 50°. The yield of 2-chloro-*N*-methyldiethylamine hydrochloride, m.p. 171–173°, was quantitative. Recrystallization from ethanol resulted in a 78% yield of m.p. 174–176°.

Anal. Calcd. for $C_5H_{12}ClN$: C, 38.0; H, 8.3; Cl, 44.9; N, 8.9. Found: C, 38.3; H, 8.5; Cl, 44.9; N, 8.8.

A mixture of 63.2 g. (0.4 mole) of 2-chloro-*N*-methyldiethylamine hydrochloride, 54.8 g. (0.6 mole) of aniline, 85 g. of sodium carbonate, and 200 ml. of toluene was heated at reflux for 16 hr. and then cooled. The reaction mixture was shaken with 80 ml. of 5*N* potassium hydroxide, and the organic layer was separated, dried over magnesium sulfate and distilled. The yield of *N*-ethyl-*N*-methyl-*N′*-phenylethylenediamine, b.p. 88–92° (0.3 mm.), n_D^{25} 1.530, was 62%.

The infrared absorption curves of the two samples of *N*-ethyl-*N*-methyl-*N′*-phenylethylenediamine were identical. Mixture melting points of the salts were not depressed.

Acknowledgment. We wish to thank Mr. W. Fulmor and associates for the infrared absorption curves and Mr. L. Brancone and co-workers for the microanalyses.

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Relative Reactivities of Sites in Salicylanilide. The Bromination of 2′,5-Dibromosalicylanilide

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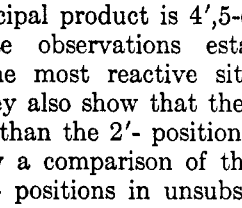
Received August 29, 1960

When salicylanilide is treated with one mole of bromine in glacial acetic acid, substitution takes place primarily in the 5-position, *para* to the phenolic group.[1] When two moles of bromine are used, the principal product is 4′,5-dibromosalicylanilide.[1] These observations establish the 5-position as the most reactive site under these conditions; they also show that the 4′- position is more reactive than the 2′- position. They do not, however, allow a comparison of the reactivity of the 3- and 4′- positions in unsubstituted salicylanilide because, once the first bromine has entered the 5- position, the 3- position is deactivated.

The relative reactivities of the 3- and 4′- positions of salicylanilide were determined by brominating 2′,5-dibromosalicylanilide (I). In this compound both positions are equally deactivated by the two bromine atoms already present in the molecule.



I

Infrared examination of the brominated product was made in dimethylformamide solution between 10.5 μ and 16 μ. All the absorption peaks observed could be ascribed to either 2′,5-dibromosalicylanilide (D) or 2′,3,5-tribromosalicylanilide (T) as follows: 10.8 μ (T); 11.05 μ (D); 11.5 μ (T); 12.1 μ (D); 12.9 μ (shoulder) (D); 13.1 μ (D,T); 13.55 μ (T); 14.05 μ (D); 14.3 μ (T); 14.9 μ − 15.4 μ (D); 16.0 μ (D). Peaks peculiar to 2′,3,4′,5-tetrabromosalicylanilide (13.3 μ) and 2′,4′,5-tribromosalicylanilide (13.8 μ) were not observed. Analysis of the

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(1) Schuler, L., German Patent 920,790; U.S. Patent 2,802,029.