TABLE 1

AMIDES AND HETEROCYCLIC COMPOUNDS FROM PHENYL SALICYLATE AND FROM PHENYL-1-HYDROXYNAPHTHALENE-2-CARBOXYLATE

Amine used	M. p., °C.	Yield, %	Formula	% Nitrogen Calcd.	% Nitrogen Found
		With Phenyl Salicylate			
Piperidine	142–143	69	$C_{12}H_{15}NO_2$	6.8	6.9
Cyclohexylamine	85–86	79	$C_{13}H_{17}NO_2$	6.4	6.4
Benzylamine	135–136	77	$C_{14}H_{13}NO_2$	6.2	6.2
n-Butylamine[11]	[a]	81			
Laurylamine	71–72	75	$C_{19}H_{31}NO_2$	4.6	4.6
Diethylamine	[b]	68	$C_{11}H_{15}NO_2$	7.3	7.2
Ethylenediamine	183–184	69	$C_{16}H_{16}N_2O_4$	9.4	9.5
Chloroaniline	155	83	$C_{13}H_{11}ClNO$	15.3[c]	15.0[c]
Aminobiphenyl	110	85	$C_{19}H_{16}NO_2$	[d]	[d]
o-Aminophenol	125	22.4	$C_{13}H_9NO_2$	6.63[e]	6.63[e]
p-Aminophenol[12]	176	57			
m-Aminophenol	184	58	$C_{13}H_{11}NO_2$	6.11	6.10
5-Aminoindazole	230	37	$C_{14}H_{11}N_3O_2$	16.62	16.57
6-Aminoindazole	234–235	31	$C_{14}H_{11}N_3O_2$	16.62	16.56
m-Phenylenediamine[12]	199–200	49			
5-Aminobenzotriazole	245	42	$C_{13}H_{10}N_4O_3$	22.03	21.74
1,2,3,4-Tetrahydroquinoline	138–139	34	$C_{16}H_{15}NO_2$	5.5	5.6
		With Phenyl-1-hydroxynaphthalene-2-carboxylate			
Diethylamine	[f]	63	$C_{15}H_{17}NO_2$	5.77	5.6
o-Phenylenediamine	>265	78	$C_{17}H_{12}N_2O$	[g]	[g]
o-Aminophenol	188	89	$C_{17}H_{11}NO_2$	[h]	[h]

[a] B. p. 153–156° (3 mm.). [b] B. p. 146–148° (4 mm.). [c] Chlorine. [d] Calcd.: C, 78.80; H, 5.18. Found: C, 78.8; H, 5.1. [e] Calcd.: C, 73.8; H, 4.3. Found: C, 74.24; H, 4.53. [f] B. p. 130–133° (1 mm.). [g] Calcd.: C, 78.40; H, 4.6. Found: C, 78.6; H, 4.4. [h] Calcd.: C, 78.0; H, 4.22. Found: C, 77.9; H, 4.3.

acetanilide[9] or aniline[10], from methyl salicylate and an aliphatic amine.[11,12]

(9) German Patent 289,027 [*Frdl.*, **12**, 184 (1914–1916)].

(10) Schöpff, *Ber.*, **25**, 2740 (1892).

(11) Hurd, Fancher and Bonner, THIS JOURNAL, **68**, 2745 (1946).

(12) Fargher, Galloway and Probert, *J. Textile Inst.*, **21**, 245T (1930) [*C. A.*, **24**, 6026 (1930)].

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A New Process for the Preparation of Thioglycolylamides

BY JAMES A. VANALLAN

It is known that thioglycolylamides may be obtained by alkaline hydrolysis of carbamyl thioglycolylanilides[1] but the yields are low (15%) and several steps are required to obtain the product. Also acetothioglycolylamides, which are obtained from the acid chloride and an amine, may be saponified to the required thioglycolylamides but the intermediate acid chloride[2] is difficult to obtain, and again the process consists of several steps.

It has now been found that thioglycolylamides may be made in excellent yield and in a high state of purity without protecting the thiol group. The process consists of mixing an amine and thioglycolic acid in molecular proportions with benzene as a solvent and utilizing a Clarke–Rahrs ester column[3] to remove the water as it is formed. The reactants are at all times in an atmosphere of benzene during the course of the reaction, which minimized the formation of disulfide. The crude product, therefore, usually possesses a higher degree of purity than that obtained by other processes. The process is illustrated by the preparation of thioglycolylanilide.

Thioglycolylanilide.—Thioglycolic acid (46 g.) and aniline (45 g.) are mixed in 250 ml. of benzene. This solution is refluxed, using an ester column, until approximately 9 ml. of water has separated (about nine hours). The benzene solution is then treated with an equal volume of petroleum ether and chilled. The product (70 g., 85%) separates as a mass of white crystals; m. p. 103–105°. A recrystallization from dilute alcohol raises the melting point to 110°.

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(1) Beckurts and Frerichs, *J. prakt. Chem.*, [2] **66**, 174 (1902).

(2) Benary, *Ber.*, **46**, 2105 (1913).

(3) Eastman Kodak Company, "Syn. Org. Chem.," **9**, No. 3, May (1936).

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Resonance and Hydrogen Bond Effects on the Basic Strengths of Certain Arylalkyl Azomethines

BY CHARLES D. WAGNER AND EDWARD D. PETERS

When aliphatic primary amines are treated with most of the common aromatic aldehydes, azo-