lize, it was purified by distillation in vacuum. In this manner the phenylurethans in Table I were obtained.

TABLE I

Phenylurethan of	M. p., °C. (uncor.)	Formula	Analyses,[a] % Calcd. C	H	Found C	H
Dimethylbutylcarbinol	62–63	$C_{14}H_{21}NO_2$	71.5	8.9	71.8	8.9
Diphenylmethylcarbinol	124–125	$C_{21}H_{19}NO_2$	79.5	6.0	79.7	5.7
Triethylcarbinol	61–61.5	$C_{14}H_{21}NO_2$	71.5	8.9	70.9	8.8
Methylethylbenzylcarbinol	83.5–84	$C_{18}H_{21}NO_2$	76.3	7.5	75.9	7.3
Diethylbenzylcarbinol	96–96.5	$C_{19}H_{23}NO_2$	76.7	7.8	76.7	7.4

[a] Analyses by R. W. King.

Discussion of the mechanism of the catalysis will be deferred, but it seems to be a case of general acid–base catalysis. The tests for determining general acid–base catalysis which are employed in aqueous systems cannot be used readily here, since the isocyanates must be kept in inert solvents.

It is hoped to study this catalysis effect in greater detail and to include the reactions of alcohols and isocyanates.

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Optical Properties of 2-Sulfanilamidopyrimidine (Sulfadiazine)

BY ALBERT S. WILKERSON

Thanks to the courtesy of Mr. Leonard Dhein of the American Cyanamid Company, Bound Brook, New Jersey, we have been able to determine the optical and related properties of a sample of sulfadiazine (m. p. 254°)[1] with the following results: monoclinic, colorless, transparent to translucent; luster vitreous. Cleavage pinacoidal at right angles, perfect. $H = 1$–2. Solubility 0.0123 g./100 cc.,[1] H_2O at 37°. Optically biaxial positive; $\alpha = 1.680$, $\beta = 1.695$, $\gamma = 1.788$; all ± 0.002, $2V = 45$–$46°$, $Y//b$, $Z_{\wedge}c = 20°$(?), elongation$//b$.

(1) R. O. Roblin, *et al.*, THIS JOURNAL, **62**, 2002 (1940), found 255–256° with decomposition; they found the solubility to be 0.0123 g./100 cc.

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NEW COMPOUNDS

Certain Naphthylidene Sulfanilamide Derivatives

The procedures employed for the preparation of the following compounds were modifications of methods used in the preparation of the aniline derivatives of the sulfonated α- and β-naphthoquinones.[1,2] To conform with *Chemical Abstracts* the nomenclature of these compounds was suggested by Dr. Austin M. Patterson.

N^4-(3-Hydroxy-4-oxo-1(4)-naphthylidene)-sulfanilamide.—A solution of 8.6 g. of sulfanilamide (Eastman Kodak Co.) in 200 cc. of water at 60–65° was added with stirring to a solution of 13.0 g. of 1,2-naphthoquinone-4-sodium sulfonate (Eastman purified by the method of Folin[3]) in 300 cc. of water at 45–50°. The reaction mixture after standing for fifteen minutes at room temperature was kept in the ice box overnight. The reddish orange precipitate was filtered off, washed with water and dried in a vacuum desiccator over sulfuric acid; yield, 9.9 g. (60%). The compound melted at 271–273° (uncor.). It is insoluble in cold water, very difficultly soluble in hot water, very soluble in alkali, formic acid, slightly soluble in hydrochloric acid, alcohol, dioxane and acetone.

Anal. Calcd. for $C_{16}H_{12}O_4SN_2$: N, 8.53. Found: N, 8.41.

N^4-(3-Hydroxy-4-oxo-7-sulfo-1(4)-naphthylidene)-sulfanilamide, Sodium Salt.—To a solution containing 2.7 g. of 1,2-naphthoquinone-4,6-sodium disulfonate[1] in 75 cc. of water, 1.7 g. of sulfanilamide was added. The mixture was stirred mechanically until all the solid went into solution. This required fifteen minutes. Then 1.5 cc. of superoxol was added and stirring continued for an additional ten minutes. The deep red-brown solution was clarified with a small amount of Merck activated charcoal to remove the brown impurity. Upon addition of 10 g. of powdered sodium chloride the solution became a thick paste in a few minutes. After cooling in the ice box for two hours, the dark red needles were filtered off and dried in a vacuum desiccator over sulfuric acid. The weight of the crude material (contaminated with sodium chloride) was 4.6 g. It was recrystallized by solution in 50 cc. of water at 60° and subsequent chilling. The crystalline compound was filtered off, washed with ice-cold water, ice-cold 95% alcohol, ether and dried in a vacuum desiccator over sulfuric acid; yield, 2.2 g. (41%).

Anal. Calcd. for $C_{16}H_{11}O_7S_2N_2Na$: N, 6.50. Found: N, 6.51.

N^4-[4-Oxo-3-(*p*-sulfamylanilino)-2-sulfo-1(4)-naphthylidene]-sulfanilamide.—A solution of 1.7 g. of sulfanilamide in 100 cc. of water at 80° was added with stirring to a solution of 1.6 g. of 1,4-naphthoquinone-2-potassium sulfonate[4] in 50 cc. of water at 55°. As soon as the temperature of the mixture reached 50°, a small amount of yellow orange crystals (not identified) separated. These were filtered off by suction and discarded. To the filtrate at 30° was added 1 cc. of concentrated hydrochloric acid and the reaction mixture cooled in the ice box for three hours. The bright red needles that separated were filtered off, washed with cold water and dried in a vacuum desiccator over sulfuric acid; yield, 1.4 g. (50%). The material was recrystallized from 40 cc. of boiling water. It is fairly soluble in hot water. Dried in a vacuum desic-

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(1) M. Böniger, *Ber.*, **27**, 23, 3050 (1894).

(2) P. Seidel, *ibid.*, **25**, 423 (1892).

(3) O. Folin, *J. Biol. Chem.*, **51**, 377 (1922).

(4) L. F. Fieser and M. Fieser, THIS JOURNAL, **57**, 491 (1935).

cator over sulfuric acid the compound melted at 276–278° (uncor.).

Anal. Calcd. for $C_{22}H_{18}S_3O_8N_4$: N, 9.96; S, 17.09. Found: N, 9.54; S, 16.85.

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Some Diamino Peptides

Dimethylamine reacted with *p*-nitro-β-bromopropionanilide to form *p*-nitro-β-dimethylaminopropionanilide, m. p. of the hydrochloride, 200–201°. A portion of this was transformed into the methochloride and both substances were reduced catalytically in alcohol containing hydrogen chloride to give, respectively, *p*-amino-β-dimethylaminopropionanilide dihydrochloride (I), colorless felted needles, m. p. 218–219°, and *p*-amino-β-dimethylaminopropionanilide methochloride hydrochloride (II), hygroscopic crystals, m. p. 211–212°.

N,N-Diethylethylenediamine,[1] prepared by the reduction (sodium and alcohol) of diethylglycine nitrile, reacted with *p*-nitrobenzoyl chloride, forming β-[*p*-nitrobenzoylamidoethyl]-diethylamine hydrochloride, colorless needles melting at 164–5°. This substance and its ethochloride were reduced catalytically in alcoholic hydrogen chloride to β-[*p*-aminobenzoylamidoethyl]-diethylamine dihydrochloride (III), m. p. 176.5–178°, and to β-[*p*-aminobenzoylamidoethyl]-triethylammonium chloride, hydrochloride (IV), which forms stubby prisms melting at 228°.

Reduction of β-dimethylaminopropionitrile gave γ-dimethylaminopropylamine, m. p. of the dihydrochloride, 182–184° The base reacted with *p*-nitrobenzoyl chloride to form γ-[*p*-nitrobenzoylamidopropyl]-dimethylamine hydrochloride, m. p. 190–192°. This was reduced catalytically in alcoholic hydrogen chloride solution to γ-[*p*-aminobenzoylamidopropyl]-dimethylamine dihydrochloride (V), m. p. 184–185°.

Reduction of the *p*-nitrophenylurethan of β-hydroxyethyltriethylammonium chloride yielded β-[*p*-aminophenylcarbamatoethyl]-triethylammonium chloride, hydrochloride (VI), which forms irregular prisms melting at 138–139°.

By the reduction of β-[*p*-nitrobenzoyloxyethyl]-triethylammonium chloride, there was obtained β-[*p*-aminobenzoyloxyethyl]-triethylammonium chloride, hydrochloride (VII), m. p. 214–215°. This has been reported[2] previously, but the synthesis used was not free from ambiguity.

The salts described here were crystallized from absolute alcohol or from alcohol–ethyl acetate mixtures. The analytical data are in the table.

No.	Formula	Analyses, % Calcd. C	H	Found C	H
I	$C_{11}H_{19}ON_3Cl_2$	47.12	6.84	47.38	6.61
II	$C_{12}H_{21}ON_3Cl_2$	48.96	7.20	48.67	7.27
III	$C_{13}H_{23}ON_3Cl_2$	50.63	7.52	50.67	7.80
IV	$C_{15}H_{27}ON_3Cl_2$	53.56	8.13	53.45	8.04
V	$C_{12}H_{21}ON_3Cl_2$	48.96	7.20	49.05	7.51
VI	$C_{15}H_{27}O_2N_3Cl_2$	51.12	7.73	51.27	7.75
VII	$C_{15}H_{26}O_2N_2Cl_2$	53.39	7.77	53.02	7.62

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[1] Ristenpart, *Ber.*, **29**, 2526 (1896).

[2] Einhorn and Uhlfelder, *Ann.*, **371**, 138 (1909).

Some New Quaternary Salts

Methylaniline and octadecyl iodide reacted to yield methyloctadecylaniline, a yellow oil, b. p. (3 mm.), 234°. This was treated in benzene solution with methyl iodide forming octadecylphenyldimethylammonium iodide which crystallized in leaflets from ethyl acetate.

Cyclohexylethylamine (prepared from cyclohexylacetic acid by the sequence: acid ⟶ acid chloride ⟶ amide ⟶ nitrile ⟶ amine) was methylated by the Clarke–Eschweiler method. The resulting tertiary amine reacted with benzyl chloride in ether to give cyclohexylethylbenzyldimethylammonium chloride.

Benzyl-β-bromoethyldimethylammonium bromide was prepared from benzyldimethylamine and ethylene bromide.

Triethylamine and α-menaphthyl chloride (α-naphthylmethyl chloride) yielded α-menaphthyltriethylammonium chloride. Data on these substances are presented in the subjoined table.

Formula	M. p., °C.	Composition	Analyses, % Calcd. C	H	Found C	H
$(n\text{-}C_{18}H_{37})C_6H_5NMe_2I$	93–94	$C_{26}H_{48}NI$	62.25	9.65	62.12	9.69
$(C_6H_{11}CH_2CH_2)(C_6H_5CH_2)NMe_2Cl$	206 dec.	$C_{17}H_{28}NCl$	72.44	10.02	72.15	10.16
$(C_6H_5CH_2)(BrCH_2CH_2)NMe_2Br$	174	$C_{11}H_{17}NBr_2$	40.87	5.31	40.91	5.61
$(\alpha\text{-}C_{10}H_7CH_2)NEt_3Cl$	197 dec.	$C_{17}H_{24}NCl$	73.49	8.71	73.41	8.62

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Some Mono- and Disubstituted Guanidines

The guanidines here described were all prepared by conventional methods. The S-methyl-isothiourea sulfate method (generally applicable with amines of moderate strength and water solubility) was used to prepare the five following: β-[N-morpholinoethyl]-guanidine sulfate (from aminoethylmorpholine), β,β-diethoxyethylguanidine sulfate (from amino acetal), N,N-dicyclohexylguanidine sulfate (from dicyclohexylamine), N-benzyl-N-methylguanidine sulfate (from benzylmethylamine) and δ-phenoxybutylguanidine sulfate (from δ-phenoxybutylamine prepared by reduction of γ-phenoxybutyronitrile). In all these cases the salt was isolated by evaporation of the reaction mixture