hydrochloric acid was added to the filtrate causing the formation at *p*H 3 of a gelatinous yellow precipitate which was washed twice with a liter of slightly acidified water. The precipitate was removed and dried in a vacuum desiccator; the slightly brown powder was ground and washed with ether.[7] The **1,2-benzanthryl-10-carbamidoacetoglycine** (I) thus obtained in 82% yield darkened at 210° and had not completely melted at 260°.

Anal. Calcd. for $C_{23}H_{19}O_4N_3$: C, 68.9; H, 4.78; N, 10.45. Found: C, 67.8; H, 4.88; N, 10.43.

Chloroacetylglycylglycine, prepared from 2,5-diketopiperazine and chloroacetyl chloride,[8] was treated with ammonium hydroxide to give triglycine[9] which turned yellow at 215° and melted with decomposition at 240°. This compound was conjugated with the isocyanate under conditions similar to those used above to form **1,2-benzanthryl-10-carbamidoacetoglycylglycine** (II) obtained in 68% yield as a slightly brown powder which darkened at 200° and decomposed at 250°.

Anal. Calcd. for $C_{25}H_{22}O_6N_4$: C, 65.6; H, 4.84. Found: C, 66.36, 66.15; H, 4.78, 4.81.

For the preparation of α-(1,2-benzanthryl-10-carbamido)-glutaric acid (III), l(+)-glutamic acid was employed. The procedures of coupling and isolation were the same as those used before. The brown product, obtained in 44% yield, darkened slightly at 235°, softened at 247° and melted at 252-254° with decomposition.

Anal. Calcd. for $C_{24}H_{20}O_6N_2$: C, 69.2; H, 4.83; N, 6.74. Found: C, 68.92; H, 4.90; N, 6.46.

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[7] Attempts to crystallize this compound from aqueous dioxane or from other solvents were not successful; usually such attempts led to serious decomposition.

[8] Fischer, *Ber.*, **39**, 2931 (1906).

[9] Fischer, *ibid.*, **37**, 2500 (1904).

DEPARTMENT OF CHEMISTRY
UNIVERSITY OF MARYLAND
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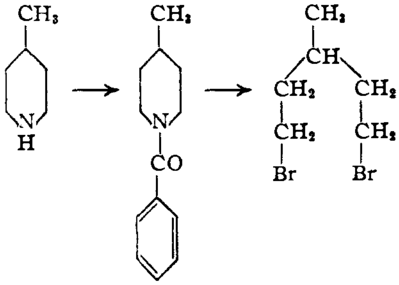
RECEIVED JULY 23, 1946

1,5-Dibromo-3-methylpentane

BY NELSON J. LEONARD AND ZENO W. WICKS[1]

1,5-Dibromo-3-methylpentane (III) has not been prepared previously. This compound is of interest as an intermediate in the synthesis of certain heterocycles,[2] and as a building unit for certain saturated isoprenoid molecules in which synthesis may be effected by combination of alternating symmetrical six- and four-carbon units rather than the customary unsymmetrical five- and five-carbon units.

4-Methylpiperidine (I) was converted to 1-benzoyl-4-methylpiperidine (II) by a Schotten-Baumann reaction with benzoyl chloride,[3] and this, in turn, by a von Braun reaction with phosphorus pentabromide, gave III.[4]



1-Benzoyl-4-methylpiperidine (II).—To a mixture of 340 g. of I (3.4 moles), 180 g. of sodium hydroxide (4.5 moles), and 1400 ml. of water, 476 g. of benzoyl chloride (3.4 moles) was added with stirring at 35-40° during one hour. The non-aqueous layer and the ether extracts of the aqueous layer were combined and evaporated to dryness. The solid residue was recrystallized from ethanol as colorless prisms; m. p. 83.5-84°; yield, 635 g. (92%).

Anal. Calcd. for $C_{13}H_{17}NO$: C, 76.81; H, 8.43; N, 6.89. Found: C, 76.92; H, 8.26; N, 6.72.

1,5-Dibromo-3-methylpentane (III).—During cooling and stirring, 575 g. of phosphorus tribromide (2.15 moles) was added to 426.5 g. of II (2.10 moles), followed by 325 g. of bromine (2.13 moles). The reaction mixture was distilled under reduced pressure from 65 (30 mm.) to 112° (20 mm.), until a yellow solid collected in the condenser and extensive decomposition occurred in the distilling flask. The total distillate was poured onto ice, the mixture stirred for several hours and allowed to stand overnight. The oily layer was separated and boiled with 625 ml. of 40% hydrobromic acid solution under reflux for four hours. After steam distillation, the oily layer in the distillate was separated and washed twice with 10% sodium carbonate, once with water, dried over Drierite, then distilled *in vacuo*. The product boiled at 97-98.5° (10 mm.); yield, 333 g. (65%); n^{20}_D 1.5073; d^{20}_4 1.607.

Anal. Calcd. for $C_6H_{12}Br_2$: C, 29.53; H, 4.96; Br, 65.51; MR_D, 45.44. Found: C, 29.68; H, 5.19; Br, 65.38; MR_D, 45.21.

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[1] Present address: Interchemical Corporation, New York, N. Y.

[2] *Cf.* Prelog and Seiwerth, *Ber.*, **72**, 1638 (1939).

[3] "Organic Syntheses," Coll. Vol. I, 1941, p. 101; Adams and Leonard, THIS JOURNAL, **66**, 257 (1944).

[4] "Organic Syntheses," Coll. Vol. I, 1941, p. 428.

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RECEIVED JULY 26, 1946

Osmotic and Activity Coefficients of Lithium Nitrate Solutions

BY R. A. ROBINSON

Vapor pressure measurements of lithium nitrate solutions have been made by the isopiestic method[1] at 25° up to a concentration of 3.8 *M*. The range has now been extended by further measurements in more concentrated solution. Lithium nitrate was prepared by double recrystallization from the salt obtained from a sample of lithium carbonate which had been purified by the method outlined by Caley and Elving.[2]

TABLE I

m_{LiNO_3}	m_{NaCl}	m_{LiNO_3}	m_{NaCl}	m_{LiNO_3}	m_{NaCl}
3.316	3.676	3.500	3.877	3.607	3.996
3.864	4.273	4.661	5.136	4.750	5.221
5.292	5.796	5.554	6.061	5.614	6.132
m_{LiNO_3}	$m_{H_2SO_4}$	m_{LiNO_3}	$m_{H_2SO_4}$	m_{LiNO_3}	$m_{H_2SO_4}$
5.456	4.246	6.925	5.161	8.682	6.202
9.522	6.691	10.33	7.124	11.22	7.635
11.78	7.909	11.94	8.012	12.29[a]	8.161
13.36	8.757	13.72	8.966		

[a] In equilibrium with saturated solution.

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[1] R. A. Robinson, THIS JOURNAL, **57**, 1165 (1935).

[2] H. S. Booth, "Inorganic Syntheses," Vol. I, McGraw-Hill Book Co., Inc., New York, N. Y., 1939.

Solutions were equilibrated against sodium chloride or sulfuric acid solutions and the following pairs of solutions (Table I) were found to have equal vapor pressure at 25°.

Osmotic and activity coefficients were evaluated relative to the corresponding values for the reference electrolytes[3] with the following results (Table II).

TABLE II

m	φ	γ	m	φ	γ
0.1	0.938	0.788	4.0	1.270	1.125
.2	.935	.752	5.0	1.352	1.310
.3	.940	.736	6.0	1.426	1.515
.5	.954	.726	7.0	1.490	1.734
.7	.970	.729	8.0	1.544	1.960
1.0	.997	.743	9.0	1.593	2.202
1.5	1.043	.783	10.0	1.637	2.455
2.0	1.088	.835	11.0	1.674	2.711
2.5	1.134	.896	12.0	1.704	2.967
3.0	1.181	.966	13.0	1.735	3.242
3.5	1.227	1.044	13.5	1.754	3.398

The saturated solution contained 45.87% of lithium nitrate. Campbell[4] gives 46.0% as the solubility. The water activities corresponding to the above osmotic coefficients are higher by approximately 0.0020 over the range 2 to 9 *M* than those measured by Pearce and Nelson,[5] the difference rising to 0.0080 at 12.87 *M*.

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[3] S. Shankman and A. R. Gordon, THIS JOURNAL, **61**, 2370 (1939); P. Olynyk and A. R. Gordon, *ibid.*, **65**, 224 (1943); R. A. Robinson, *Trans. Roy. Soc.*, *N. Z.*, **75**, 203 (1945); R. H. Stokes and B. J. Levien, THIS JOURNAL, **68**, 333 (1946).

[4] A. N. Campbell, THIS JOURNAL, **64**, 2680 (1942).

[5] J. N. Pearce and A. F. Nelson, *ibid.*, **54**, 3544 (1932).

CHEMISTRY DEPARTMENT
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RECEIVED AUGUST 2, 1946

Ultraviolet Absorption Spectra of Mescaline Sulfate and β-Phenylethylamine Sulfate

BY KURT SALOMON AND ALBERT F. BINA

The following is a report on the results of a spectrographic study of the absorption spectra of mescaline sulfate (3,4,5-trimethoxy-β-phenylethylamine sulfate) and β-phenylethylamine sulfate.

Mescaline has specific pharmacological properties, such as production of color hallucinations in man, while β-phenylethylamine does not exhibit the aforementioned effects. It has been shown by various workers[1,2,3] that the introduction of methoxy groups in the phenylethylamine molecule greatly influences the metabolic fate of this compound by rendering it more resistant to oxidation. Further information about the interrelationship of methoxy groups and the physical and chemical properties of the molecule is therefore desirable.

Only a few experimental studies concerning the influence of methoxy groups on the ultraviolet absorption spectra of benzene and its derivatives have been reported. Hillmer and Schornig[4] have established the fact that introduction of methoxy groups into the benzene ring in presence or absence of various side chains produces simplification of the fine structure of the absorption curve as well as a shifting of the maxima toward the visible region. This shift was in the order of magnitude of ten to twenty millimicrons, depending on the number of methoxy groups introduced and the character of the side chain present. Furthermore, they state that the intensity of the absorption increases with the introduction of methoxy groups. Our results are in agreement with their findings.

Experimental

The ultraviolet absorption spectra were measured with a Beckman Quartz Spectrophotometer. Measurements were made in 80% ethyl alcohol. Crystalline mescaline sulfate was used in these experiments; the melting point of this preparation was 181–184° (uncor.).[5] The β-phenylethylamine used was obtained from Eastman Kodak Company with the information that it has a boiling point from 89–90.5° at 15 mm. pressure. The measurements on this compound were made immediately after redistillation *in vacuo*. In order to produce β-phenylethylamine sulfate for measurements, 0.1 cc. of concentrated sulfuric acid was added to the β-phenylethylamine solution to be measured. The concentrations of mescaline sulfate used were in the range of 25 micrograms per ml. (8×10^{-5} molar) to 100 micrograms per ml. (32×10^{-5} molar). The concentrations of β-phenylethylamine sulfate were in the range of 400 micrograms per ml. (36×10^{-4} molar) to 800 micrograms per ml. (72×10^{-4} molar).

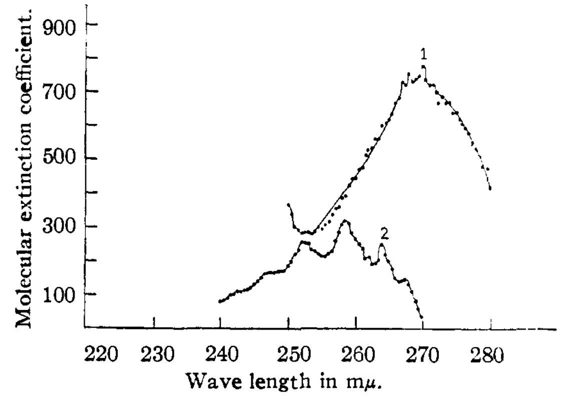

Fig. 1.—1, Mescaline sulfate in 80% ethyl alcohol; 2, β-phenylethylamine sulfate in 80% ethyl alcohol.

Preliminary experiments show that mescaline can be extracted from an alkaline water solution into isobutyl alcohol. The amount of mescaline can then be estimated by measuring the intensity of absorption at the absorption peak. The absorption spectrum of mescaline is identical in ethyl alcohol and in isobutanol.

CONTRIBUTION FROM THE
BIOCHEMICAL LABORATORY OF THE
DEPARTMENTS OF NEUROPSYCHIATRY AND RADIOLOGY
WASHINGTON UNIVERSITY MEDICAL SCHOOL
ST. LOUIS, MO.
RECEIVED APRIL 8, 1946

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[1] K. H. Slotta and J. Müller, *Z. physiol. Chem.* (Hoppe-Seyler's), **238**, 14 (1936).

[2] D. Richter, *Biochem. J.*, **31**, 2022 (1937).

[3] F. Bernheim and M. L. C. Bernheim, *J. Biol. Chem.*, **123**, 317 (1938).

[4] A. Hillmer and P. Schornig, *Z. physik. Chem.*, **A167**, 407 (1933).

[5] The mescaline sulfate was kindly furnished by Hoffmann-LaRoche, Inc., Nutley, New Jersey.