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 $C_{25}H_{22}O_5N_4$: 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), 1(+)-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 $C_{24}H_{20}O_5N_2$: 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
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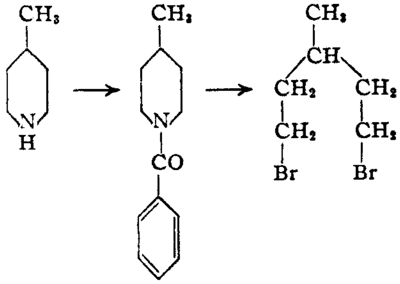
RECEIVED JULY 23, 1946

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 Bráun reaction with phosphorus pentabromide, gave III.[4]



I II III

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.