TABLE II

ANALYSIS OF BIS-(TRIMETHYLENEDIAMINO)-CUPRIC SULFATE HYDRATE[6]

	% C	% H	% Cu	% N	% S
Calcd. for $C_6H_{20}CuN_4SO_4 \cdot H_2O$	22.11	6.81	19.51	17.19	9.84
Determined	21.88	6.40	19.51	16.80	9.66
Determined	21.98	6.44	19.69	16.54	9.99

When heated it changes to the blue anhydrous form and finally decomposes at 276–277° cor. with evolution of gas, leaving a brown residue. Samples of the hydrate were heated to 35, 57, 78 and 100° and kept at these temperatures, all samples being reweighed after 1, 3, 8, 10, and 14 days. At 35° there was no dehydration. At 57° dehydration progressed so slowly that it was not quite complete even after fourteen days. At both 78 and 100° the dehydration was complete after one day. At 100° the change appeared to take place instantly, but at 78° even the visible color change required about an hour. The anhydrous compound apparently is stable at 100° for no additional change in weight occurred during the fourteen-day period of heating. The product does not contain unreacted cupric sulfate because when a sample of it was treated with additional trimethylenediamine and water and dried, it went back to its original weight.

---

[6] All determinations were made by the Laboratory of Michrochemistry, 366 Fifth Avenue, New York, N. Y.

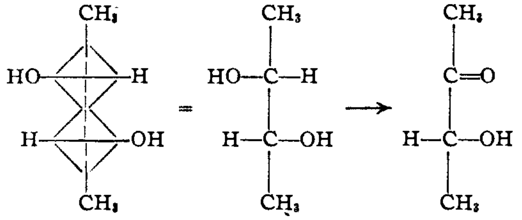
VENEREAL DISEASE RESEARCH LABORATORY
U. S. MARINE HOSPITAL
STATEN ISLAND, NEW YORK

RECEIVED SEPTEMBER 8, 1944

Configuration of Acetylmethylcarbinol

BY R. H. BLOM

The configurations of the optically active 2,3-butylene glycols have recently been correlated with the configurational system which Emil Fischer established for glucose, as D-(−) and L-(+)- for the levo- and dextro-rotatory forms, respectively.[1] The proof depends upon the established relationship of configuration between the methylethylcarbinols and the lactic acids.[2] In turn, the place of the lactic acids in the Fischer sugar system follows, for example, from the oxidation of the methyl 6-desoxy-hexopyranosides to the corresponding lactic acids.[3] In studying the vapor-phase oxidation of D-(−)-2,3-butylene glycol (I), it has been found that the acetylmethylcarbinol so formed (II) is levorotatory. Although extensive racemization occurred during the reaction, the rotation of the product was sufficient to establish the configurational relationship. Since the glycol and the carbinol can exist in only two active forms, D- or L-, racemization would form only the racemic structures in both cases. The acetylmethylcarbinols and the 2,3-butylene glycols which exhibit the same sign of rotation therefore possess the same configuration:



I II

D-(−)-2,3-Butylene glycol[4] D-(−)-Acetylmethylcarbinol

A sample of D-(−)-2,3-butylene glycol,[5] $[\alpha]^{21}D$ −12.20° (C = 100%, 1-dcm. tube) was heated to 140° and vaporized by means of a stream of air. The vapors were passed through a Pyrex tube packed with copper turnings and maintained at 315°. On condensation and fractional distillation, the main products obtained were diacetyl (33% yield), b. r. 88–88.5° (uncor.) and acetylmethylcarbinol (25% yield), b. r. 142–144° (uncor.), $n^{21}D$ 1.4186, which values are in good agreement with the literature.[6] The latter was levorotatory, $[\alpha]^{21}D$ −1.39° (C = 100%, 1-dcm. tube). On standing for twenty-four hours at 4° crystals of the optically inactive dimer of acetylmethylcarbinol were deposited.[7] Since an optically pure isomer of acetylmethylcarbinol has not yet been conclusively obtained,[8] it is not possible to calculate the concentration of the active form present in the product. The acetylmethylcarbinol was identified by acetylation with acetic anhydride, acetoin acetate, b. r. 167–168°[9] being obtained.

The assistance of Dr. S. A. Morell in the preparation of this paper is gratefully acknowledged.

[1] S. A. Morell and A. H. Auernheimer, THIS JOURNAL, **66**, 792 (1944).

[2] P. A. Levene, A. Walti and H. L. Haller, *J. Biol. Chem.*, **71**, 465 (1927).

[3] W. D. Maclay, R. M. Hann and C. S. Hudson, THIS JOURNAL, **61**, 1660 (1939).

[4] The structural formulas used conform with the fundamental convention of Emil Fischer in that the lower edges of the tetrahedra lie in a straight line in the plane of the paper, the corners which carry (H) and (OH) groups thus being above the paper.

[5] G. E. Ward, O. G. Pettijohn, L. B. Lockwood and R. D. Coghill, THIS JOURNAL, **66**, 541 (1944).

[6] J. R. Pound and A. M. Wilson, *J. Phys. Chem.*, **39**, 1135 (1935).

[7] T. M. Lowry and W. C. G. Baldwin, *J. Chem. Soc.*, 704 (1935).

[8] W. Dirscherl and A. Schollig, *Ber.*, **71**, 418 (1938).

[9] M. Bergmann and S. Ludewig, *Ann.*, **436**, 173 (1924).

NORTHERN REGIONAL RESEARCH LABORATORY
BUREAU OF AGRICULTURAL AND INDUSTRIAL CHEMISTRY
AGRICULTURAL RESEARCH ADMINISTRATION
U. S. DEPARTMENT OF AGRICULTURE
PEORIA, ILLINOIS

RECEIVED DECEMBER 14, 1944

Carbobenzoxy Derivatives of Aromatic Amines

BY NORMAN C. BERGSTROM AND A. E. MARTELL[1]

A number of aromatic amines were treated with benzyl chlorocarbonate in order to determine the ease of acylation and the possible use of the reagent for obtaining crystalline derivatives of amines. The acylation of amines and amino acids with benzyl chlorocarbonate has been thoroughly described by Bergmann[2] and his method is essentially the one used here.

[1] Now at Clark University, Worcester, Mass.

[2] Bergmann and Zervas, *Ber.*, **65**, 1192 (1932).