KRATKA SAOPĆENJA SHORT COMMUNICATIONS

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Absolute Configuration of 3-Amino-2-methylpropanol*

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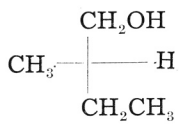
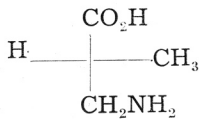
It is known that natural (—)-2-methylbutanol, a convenient reference standard for branched-chain structures, has the configuration I[1].

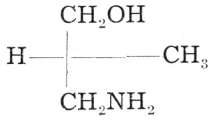
3-Amino-2-methylpropanol can also be useful for correlations of branched-chain compounds. This compound can be obtained by reduction of α-methyl-β--alanine. The correlation of the configuration of (—)-α-methyl-β-alanine II and (—)-2-methylbutanol was effected by the conversion of both compounds into the same, optically active, 2-methyl-1-phthalimidobutane[2,3].**



I



II



III

We prepared (+)-3-amino-2-methylpropanol by the reduction of (—)-α-methyl-β-alanine methyl ester with lithium aluminium hydride in a quantitative yield. (+)-3-Amino-2-methylpropanol has, therefore, the configuration III, and in the earlier proposed terms[4] would be (*R*)-3-amino-2-methylpropanol. We have characterized this compound as the hydrochloride and the picrate.

EXPERIMENTAL

(+)-3-Amino-2-methylpropanol

A suspension of α-methyl-β-alanine (0.9 g., $[\alpha]_D$ — 5.4°, in water) in anhydrous methanol (10 ml.) was saturated with dry gaseous hydrogen chloride. After standing for 24 hours at room temperature the α-methyl-β-alanine methyl ester was isolated in the usual way. A solution of the freshly distilled ester (0.8 g.) in anhydrous ether (20 ml.) was added over a period of one hour to a vigourously stirred solution of lithium aluminium hydride (1 g.) in ether (20 ml.). After stirring for 6 hours

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** In our paper[3] we determined the optical rotations of 2-methyl-1-phthalimidobutane in benzene. At present we have determined the optical rotation of this compound without solvent [2], and the sign of optical rotation was the same as was expected.

the reaction mixture was decomposed with wet ether, water was added (20 ml.), and extracted with ether in a continuous extractor for 24 hours. The ethereal extract was dried (Na_2SO_4) and evaporated to dryness. The oily residue of 3-amino-2-methylpropanol (0.6 g., 98%) was distilled over powdered sodium hydroxide, b. p. 100—105°/14 mm. (bath temperature), $[\alpha]_D^{19} + 2.8° \pm 0.6°$ (c, 3.12 in water).

Anal. 8.008 mg. subst.: 15.941 mg. CO_2, 8.672 mg. H_2O
$C_4H_{11}ON$ (89.136) calc'd.: C 53.90; H 12.44%
found: C 54.32; H 12.12%

The crystalline 3-amino-2-methylpropanol hydrochloride was prepared by dissolving the base in diluted hydrochloric acid and evaporating to dryness. It showed $[\alpha]_D^{19} + 6.6° \pm 0.4°$ (c, 2.11 in water).

3-Amino-2-methylpropanol picrate, thin yellow needles from ethyl acetate — petroleum ether, m. p. 108—109°.

Anal. 6.201 mg. subst.: 8.498 mg. CO_2, 2.474 mg. H_2O
$C_{10}H_{14}O_8N_4$ (318.24) calc'd.: C 37.74; H 4.43%
found: C 37.40; H 4.47%

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IZVOD

Apsolutna konfiguracija 3-amino-2-metilpropanola

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(+)-3-Amino-2-metilpropanol priređen je redukcijom (-)-α-metil-β-alanina, koji ima konfiguraciju II. Prema tome (+)-3-amino-2-metilpropanol ima konfiguraciju III.

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