

NOTE.

The Resolution of ν -Mandelic Acid with (-)Ephedrine. By ROBERT ROGER.

ν -MANDELIC acid was resolved by Manske and Johnson (*J. Amer. Chem. Soc.*, 1929, **51**, 1909) and Skita, Keil, and Meiner (*Ber.*, 1933, **66**, 974) by means of both optically active forms of ephedrine. By the following process the resolution has been achieved with (-)ephedrine alone.

ν -Mandelic acid (50 g. in 60 c.c. of absolute alcohol) was added slowly to (-)ephedrine (50 g. in 90 c.c. of absolute alcohol). The solution was gently warmed for 2 hours and then cooled in the ice-chest. The crystalline complex was collected, washed with fresh alcohol (30 c.c.), boiled with alcohol (75 c.c.), collected when cold, and decomposed with hydrochloric acid; the mandelic acid was then extracted (crop A, 22 g., $[\alpha]_{5461} - 177^\circ$ in acetone). (-)Ephedrine (40 g.) was dissolved in the original resolution liquor and to it was added ν -mandelic acid (40 g.) dissolved in the alcoholic washings from the previous stage. After gentle warming for an hour, the complex was separated and treated as before (crop B, 20 g., $[\alpha]_{5461} - 147.4^\circ$). This process was repeated with a further 40 g. of (-)ephedrine and 40 g. of ν -mandelic acid, giving crop C (24 g., $[\alpha]_{5461} - 78.3^\circ$). The final liquors from this series of operations were concentrated under diminished pressure to half the volume; on cooling, a solid separated, which was collected, washed with alcohol, and decomposed (crop D, 12 g., $[\alpha]_{5461} + 90^\circ$). The combined filtrate and washings were finally completely denuded of alcohol under diminished pressure at as low a temperature as possible; decomposition of the residue gave crop E (44 g., $[\alpha]_{5461} + 160^\circ$).

Crop A, recrystallised from acetone-benzene, gave 15 g. of (-)mandelic acid, $[\alpha]_{5461} - 189.8^\circ$. Similarly, crop E after recrystallisation gave 30 g. of (+)mandelic acid, $[\alpha]_{5461} + 189.1^\circ$. Crops B and C were united and quickly combined with fresh (-)ephedrine; a crop of lævo-acid (30 g.) was then obtained, $[\alpha]_{5461} - 182^\circ$ (the rotatory power of pure lævorotatory mandelic acid in acetone is $[\alpha]_{5461}^{20} - 189.9^\circ$; Roger, J., 1932, 2168).

130 G. of ν -mandelic acid thus gave 45 g. of the lævorotatory acid and 30 g. of the dextro-rotatory acid; 124 g. of ephedrine were recovered.—UNIVERSITY COLLEGE, DUNDEE, UNIVERSITY OF ST. ANDREWS. [Received, June 21st, 1935.]
