PHENYLACETIC ACID

[α-Toluic acid]

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1. Procedure

In a 5-l. round-bottom flask, fitted with a mechanical stirrer and reflux condenser, are mixed 1150 cc. of water, 840 cc. of commercial sulfuric acid (Note 1) and 700 g. (6 moles) of benzyl cyanide (p. 107). The mixture is heated under a reflux condenser and stirred for three hours (Note 2), cooled slightly, and then poured into 2 l. of cold water. The mixture should be stirred so that a solid cake is not formed; the phenylacetic acid is then filtered off. This crude material is melted under water and washed by decantation several times with hot water. These washings, on cooling, deposit a small amount of phenylacetic acid which is filtered off and added to the main portion of material. The last of the hot water is poured off from the material while it is still molten, and it is then transferred to a 2-l. Claisen distilling flask and distilled under reduced pressure. A small amount of water comes over first and is rejected; about 20 cc., containing an appreciable amount of benzyl cyanide, then distils. This fraction is used in the next run. The distillate boiling at 176–189°/50 mm. is collected separately and solidifies on standing. It is practically pure phenylacetic acid, m.p. 76–76.5°, and weighs 630 g. (77.5 per cent of the theoretical amount) (Note 3). As the fraction which is returned to the second run of material contains a considerable portion of phenylacetic acid, the yield actually amounts to at least 80 per cent.

For the preparation of small quantities of phenylacetic acid, it is convenient to use the modified method given in (Note 3).

2. Notes

1. The standard directions for the preparation of phenylacetic acid specify that the benzyl cyanide is treated with dilute sulfuric acid prepared by adding three volumes of sulfuric acid to two volumes of water. The reaction, however, goes so vigorously that it is always necessary to have a trap for collecting the benzyl cyanide which is blown out of the apparatus. The use of the more dilute acid, as described in the above directions, is more satisfactory.

2. The odor of phenylacetic acid is disagreeable and persistent.

3. The phenylacetic acid may also be made by boiling under a reflux condenser for eight to fifteen hours, without a stirrer, but this method is not nearly so satisfactory as that described in the procedure.

3. Discussion

The standard method for the preparation of phenylacetic acid is the hydrolysis of benzyl cyanide with either alkali or acid. The acid hydrolysis runs by far the more smoothly and so was the only one studied. Phenylacetic acid can also be prepared by the carbonation of benzylmagnesium chloride and by the catalytic reduction of mandelic acid.
This preparation is referenced from:


References and Notes

1. Cannizzaro, Ann. 96, 247 (1855); Mann, Ber. 14, 1465 (1881); Bodroux, Compt. rend. 151, 236 (1910).
4. Zelinsky, Packendorff, and Leder-Packendorff, Ber. 67, 301 (1934).

Appendix

Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

- sulfuric acid (7664-93-9)
- acetic acid (64-19-7)
- Mandelic acid (90-64-2)
- α-Toluic acid (65-85-0)
- Benzyl cyanide (140-29-4)
- Phenylacetic acid (103-82-2)
- benzylmagnesium chloride (6921-34-2)