Organic Syntheses

A Publication
of Reliable Methods
for the Preparation
of Organic Compounds

Working with Hazardous Chemicals

The procedures in *Organic Syntheses* are intended for use only by persons with proper training in experimental organic chemistry. All hazardous materials should be handled using the standard procedures for work with chemicals described in references such as "Prudent Practices in the Laboratory" (The National Academies Press, Washington, D.C., 2011; the full text can be accessed free of charge at http://www.nap.edu/catalog.php?record_id=12654). All chemical waste should be disposed of in accordance with local regulations. For general guidelines for the management of chemical waste, see Chapter 8 of Prudent Practices.

In some articles in *Organic Syntheses*, chemical-specific hazards are highlighted in red "Caution Notes" within a procedure. It is important to recognize that the absence of a caution note does not imply that no significant hazards are associated with the chemicals involved in that procedure. Prior to performing a reaction, a thorough risk assessment should be carried out that includes a review of the potential hazards associated with each chemical and experimental operation on the scale that is planned for the procedure. Guidelines for carrying out a risk assessment and for analyzing the hazards associated with chemicals can be found in Chapter 4 of Prudent Practices.

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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

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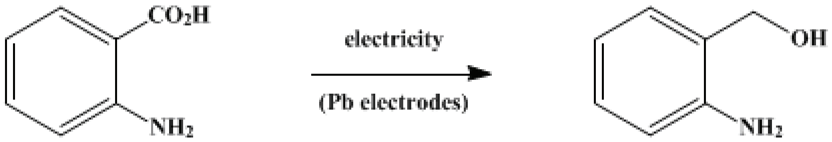
o-AMINOBENZYL ALCOHOL

[Benzyl alcohol, *o*-amino-]



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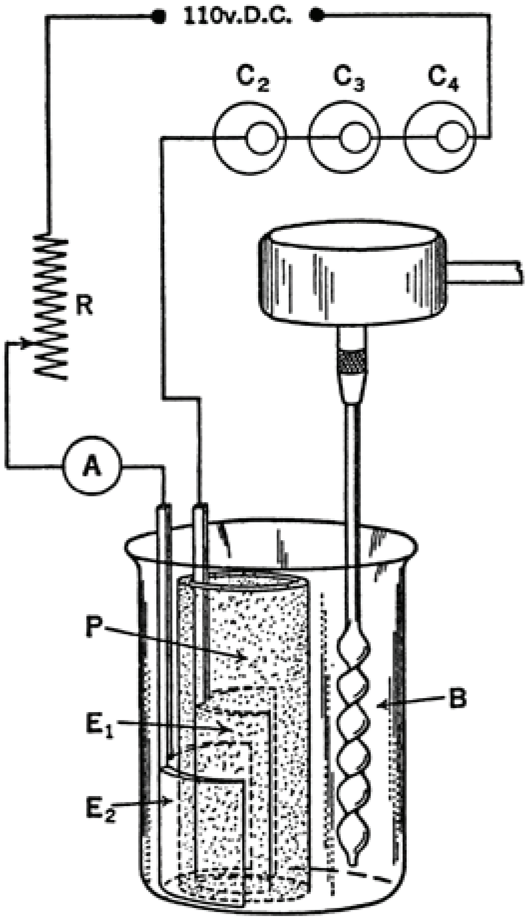
1. Procedure

The reduction is carried out in four cells of the type shown in Fig. 2. Each cell consists of a 1-l. beaker (*B*), a porous cup (*P*), a mechanical stirrer, and sheet lead electrodes (E_1 and E_2) each having a total surface area of 100 sq. cm. (Note 1). In the cathode space of each cell are placed 25 g. (0.18 mole) of anthranilic acid (Note 2) and 400 ml. of 15% sulfuric acid. In each porous cup is placed 200 ml. of 15% sulfuric acid. The cells are connected in series as shown in Fig. 5 with an ammeter (*A*) and suitable resistance (*R*) (Note 3) also in the circuit.

Fig. 2.



The stirrers are started, the current (110 volt d.c.) is turned on, and the resistance is so adjusted that the ammeter records 10–12 amperes. The temperature of the solution in the cells is maintained at 20–

30° by surrounding them with a bath of cool water (Note 4). The reduction is complete after 60–70 ampere-hours. This fact is indicated by the increased evolution of hydrogen and the complete solution of the anthranilic acid.

The cathode liquid is removed from the cells and neutralized with solid ammonium carbonate or concentrated aqueous ammonia. The solution is filtered to remove any resinous material, then saturated with ammonium sulfate and extracted with five 80-ml. portions of chloroform (Note 5). The chloroform solution is dried with 20 g. of anhydrous sodium or magnesium sulfate and filtered, and the chloroform is removed by evaporation on a steam bath (Note 6). The yield of *o*-aminobenzyl alcohol obtained from the four cells is 62–70 g. (69–78%).

This product is light brown and melts at 75–80°. After one recrystallization from petroleum ether the melting point is 80–81° (Note 7).

2. Notes

1. Ordinary sheet lead of 99.9% purity is satisfactory.
2. The anthranilic acid melted at 143–144°.
3. A resistance of 10–12 ohms is required for the apparatus described. It may be necessary to adjust the rheostat several times during the course of the reaction.
4. The checkers found it necessary to cool the cells by frequent addition of ice to the water bath surrounding them.
5. Ethyl ether may be used in place of chloroform but is not so satisfactory since several more extractions are necessary to remove the same amount of product from the aqueous solution.
6. The evaporation may be carried out in an apparatus that permits recovery of the solvent.
7. Petroleum ether boiling at 65–75° is used for recrystallization. The product has a limited solubility even in the hot solvent, and a relatively large volume is therefore required.

3. Discussion

o-Aminobenzyl alcohol has been prepared by the reduction of *o*-nitrobenzaldehyde[1,2] or *o*-nitrobenzyl alcohol[3,4] with zinc and hydrochloric acid in alcoholic solution, and by the reduction of anthranilic acid with lithium aluminum hydride.[5,6] The present method of preparing *o*-aminobenzyl alcohol is a modification of that described by Mettler.[7] Other substituted benzyl alcohols have been obtained by the same method.

This preparation is referenced from:

- Org. Syn. Coll. Vol. 3, 276

References and Notes

1. Friedländer and Henriques, *Ber.*, **15**, 2109 (1882).
2. Paal and Senniger, *Ber.*, **27**, 1084 (1894).
3. Gabriel and Posner, *Ber.*, **27**, 3512 (1894).
4. Auwers, *Ber.*, **37**, 2260 (1904).
5. Nystrom and Brown, *J. Am. Chem. Soc.*, **69**, 2548 (1947).
6. Conover and Tarbell, *J. Am. Chem. Soc.*, **72**, 3586 (1950).
7. Mettler, *Ber.*, **38**, 1751 (1905).

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

petroleum ether

sodium or magnesium sulfate

sulfuric acid (7664-93-9)

hydrochloric acid (7647-01-0)

ammonium carbonate (506-87-6)

ammonia (7664-41-7)

ethyl ether (60-29-7)

hydrogen (1333-74-0)

chloroform (67-66-3)

zinc (7440-66-6)

Anthranilic Acid (118-92-3)

ammonium sulfate (7783-20-2)

o-Nitrobenzaldehyde (552-89-6)

lithium aluminum hydride (16853-85-3)

o-Aminobenzyl alcohol,
Benzyl alcohol, o-amino- (5344-90-1)

o-nitrobenzyl alcohol (612-25-9)