



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.

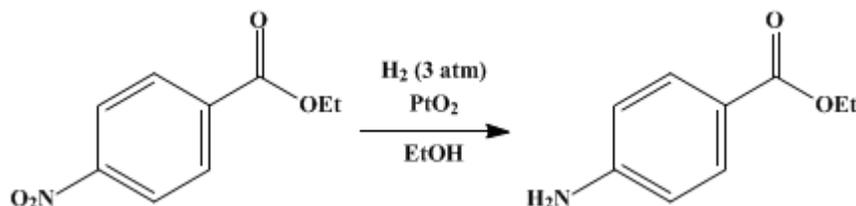
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses, Coll. Vol. 1, p.240 (1941); Vol. 8, p.66 (1928).

ETHYL *p*-AMINO BENZOATE

[Benzocaine]



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Checked by Henry Gilman and S. A. Harris.

1. Procedure

A solution of 19.5 g. (0.1 mole) of ethyl *p*-nitrobenzoate (Note 1) in 150 cc. of 95 per cent alcohol is placed in the reaction bottle of the catalytic reduction apparatus (p. 61), and 0.2 g. of platinum oxide catalyst (p. 463) is added. The mixture is shaken with hydrogen until three molecular equivalents have been absorbed. The time required is about seven minutes. The platinum is filtered off and the alcohol removed from the filtrate by distillation. The ethyl *p*-aminobenzoate, recrystallized from about 40 cc. of ether, melts at 89–90°. The yield is 15–16.5 g. (91–100 per cent of the theoretical amount).

2. Notes

1. The ethyl *p*-nitrobenzoate should be freed from acid and recrystallized from alcohol until it melts at 57°.

3. Discussion

Ethyl *p*-aminobenzoate can be prepared by the esterification of *p*-aminobenzoic acid,¹ and by the reduction of ethyl *p*-nitrobenzoate with ammonium sulfide,² electrolytically,³ and with tin and alcoholic hydrochloric acid.⁴ Although the reducing agent used commercially is generally iron and water in the presence of a little acid, the catalytic reduction procedure described is by far the most convenient for the laboratory. The catalytic reduction of ethyl *p*-nitrobenzoate has been described in the literature.⁵

This preparation is referenced from:

- Org. Syn. Coll. Vol. 1, 61
- Org. Syn. Coll. Vol. 1, 463
- Org. Syn. Coll. Vol. 2, 299

References and Notes

1. Salkowski, Ber. **28**, 1921 (1895); Vorländer and Meyer, Ann. **320**, 135 (1902); Chemnitius, Pharm. Zentralh. **68**, 765 (1927) [C. A. **22**, 583 (1928)]; Org. Syn **13**, 54.
 2. Limpricht, Ann. **303**, 278 (1898).
 3. Alvarez, Mon. farm. therap. **34**, 281 (1928) [C. A. **23**, 1575 (1929)].
 4. Hill and Cox, J. Am. Chem. Soc. **48**, 3218 (1926).
 5. Ferber and Bendix, Ber. **72**, 841 (1939).
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Appendix
Chemical Abstracts Nomenclature (Collective Index Number);
(Registry Number)

Benzocaine

alcohol (64-17-5)

hydrochloric acid (7647-01-0)

ether (60-29-7)

hydrogen (1333-74-0)

iron (7439-89-6)

tin (7440-31-5)

platinum oxide

platinum (7440-06-4)

aminobenzoic acid (118-92-3)

ammonium sulfide

Ethyl p-aminobenzoate (94-09-7)

ethyl p-nitrobenzoate (99-77-4)