



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.

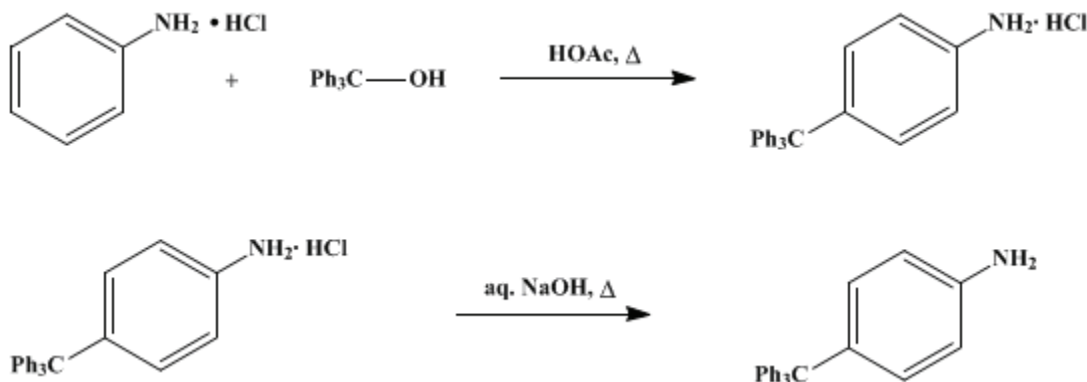
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses, Coll. Vol. 4, p.47 (1963); Vol. 30, p.5 (1950).

p-AMINOTETRAPHENYLMETHANE

[*p*-Toluidine, α,α,α -triphenyl-]



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1. Procedure

Into a 1-l. round-bottomed flask equipped with a reflux condenser are introduced 100 g. (0.385 mole) of technical grade [triphenylcarbinol](#) (Note 1), 105 g. (0.81 mole) of dry [aniline hydrochloride](#) (Note 2), and 250 ml. of glacial [acetic acid](#). The mixture is heated at the reflux temperature for 3 hours. During the period of reflux a clear brown homogeneous solution is formed. The solution while still hot is poured with stirring into a 4-l. beaker containing 2 l. of water. [p-Aminotetraphenylmethane hydrochloride](#), which is not very soluble in water, separates as a light-brown solid. It is collected on a Büchner funnel and washed with 1 l. of water. The solid is then put back into the beaker, and a solution of 40 g. of [sodium hydroxide](#) in 2 l. of water is added. The mixture is heated to boiling for 1 hour to convert the hydrochloride to the free base (Note 3), which likewise is not very soluble in water. The mixture is allowed to cool to room temperature and is filtered with suction through a Büchner funnel. The solid material is washed with 500 ml. of water and is dried in an oven at 110–120°. The crude substance melts at 243–247°. It is purified by crystallization from 1.7 l. of [toluene](#). The purified product (90–95 g., 70–74%) melts at 249–250° (Note 4).

2. Notes

1. Technical grade [triphenylcarbinol](#) is satisfactory, provided it is dry. The checkers obtained a final product having a higher melting point by starting with Eastman Kodak Company purest grade [triphenylcarbinol](#).
2. The [aniline hydrochloride](#) must be dry if a good yield of product is to be obtained. [Aniline hydrochloride](#) can be prepared conveniently by mixing 75 g. of [aniline](#) and 80 ml. of concentrated [hydrochloric acid](#) in an evaporating dish and evaporating to dryness. The [aniline hydrochloride](#) should be dried in an oven at 110–120° before use. [Aniline hydrochloride](#) (Merck) which has been washed with [ether](#) and dried at 110–120° can be employed satisfactorily.
3. The mixture tends to bump during the period of heating. This bumping can be overcome by stirring the solution mechanically.
4. A product melting at 256–257° (uncor.)² was obtained by the checkers (Note 1).

3. Discussion

The procedure given is similar to one described by Ullmann and Münzhuber,² except that one-half as much [aniline hydrochloride](#) and two-thirds as much glacial [acetic acid](#) are used, and the time of reflux is reduced from 6 to 3 hours. [p-Aminotetraphenylmethane](#) can be prepared from

triphenylchloromethane and aniline hydrochloride, following the same procedure outlined for triphenylcarbinol and aniline hydrochloride, except that a reaction time of 1 hour is sufficient. It has been prepared also from triphenylchloromethane and aniline.³

References and Notes

1. Chemical Warfare Center, Edgewood Arsenal, Maryland.
 2. Ullmann and Münzhuber, *Ber.*, **36**, 407 (1903).
 3. MacKenzie and Chuchani, *J. Org. Chem.*, **20**, 336 (1955).
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Appendix Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

hydrochloric acid (7647-01-0)

acetic acid (64-19-7)

ether (60-29-7)

aniline (62-53-3)

sodium hydroxide (1310-73-2)

aniline hydrochloride (142-04-1)

toluene (108-88-3)

Triphenylchloromethane (76-83-5)

Triphenylcarbinol (76-84-6)

p-Aminotetraphenylmethane,
p-Toluidine, α,α,α -triphenyl- (22948-06-7)

p-Aminotetraphenylmethane hydrochloride