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.

The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries or damages claimed to have resulted from or related in any way to the procedures herein.

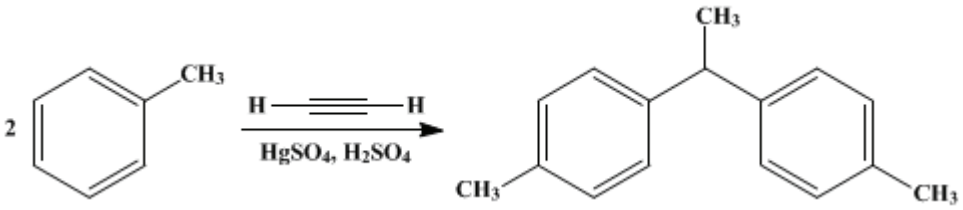
These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

DOI:10.15227/orgsyn.004.0023

Organic Syntheses, Coll. Vol. 1, p.229 (1941); Vol. 4, p.23 (1925).

1,1-DI-*p*-TOLYLETHANE

[Ethane, 1,1-di-*p*-tolyl-]

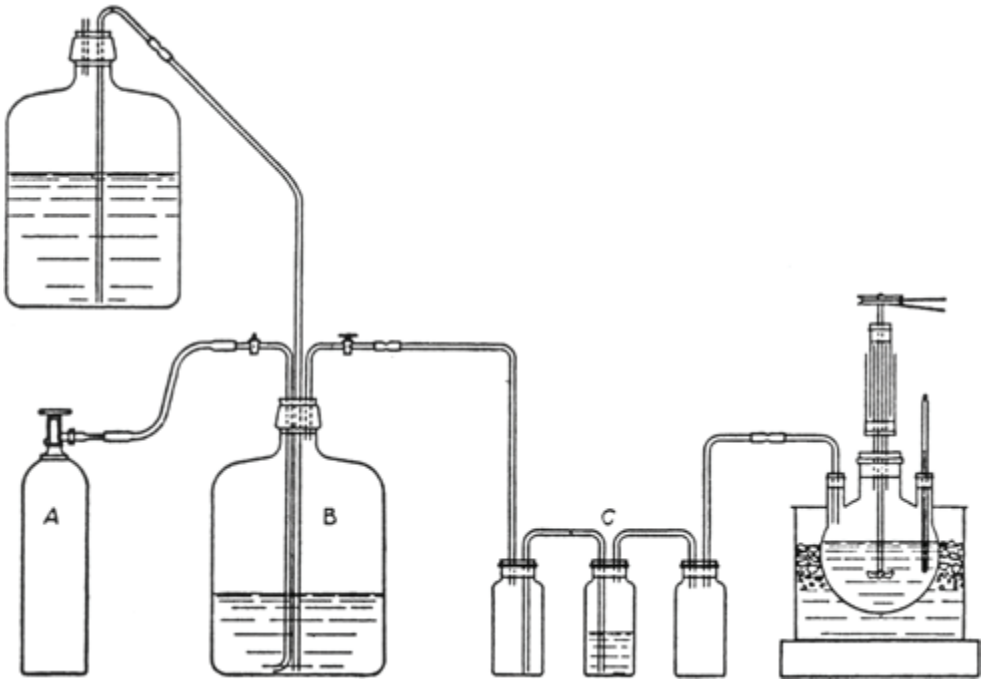


Submitted by J. S. Reichert and J. A. Nieuwland.
Checked by Oliver Kamm

1. Procedure

A 2-l. three-necked flask, containing 606 g. (700 cc., 6.6 moles) of toluene, 70 cc. of concentrated sulfuric acid, and 7 g. of mercuric sulfate, is fitted with a stirrer, a thermometer reaching into the liquid, and an inlet tube connected with a gasometer containing acetylene, as shown in Fig. 11 (Note 1). The flask and its contents are tared and cooled to 10° before the absorption of acetylene is begun. The gas from the tank, A, is washed free from acetone by being passed first through water in the gasometer, B, and then through the concentrated sulfuric acid wash bottle, C.

Fig. 11.



The acetylene is absorbed rapidly, with the evolution of considerable heat. The temperature of the reaction mixture is maintained at 10–15° by immersing the flask in a freezing mixture. When at intervals the reaction slows down, it becomes necessary to sweep the system free from air which accumulates in the flask. The absorption is continued until about 60 g. (2.3 moles) of acetylene has been absorbed, which requires a period of about two hours (Note 2). During the absorption, the mixture turns first a reddish brown, then a dark brown, and finally almost black.

The reaction mixture is freed from the acid by washing once with pure water and then with sodium

carbonate solution, to which some sodium chloride is added to aid the separation of the hydrocarbon layer. If emulsification takes place, the addition of ether will remedy the difficulty.

The toluene layer is transferred to a 1-l. flask, without drying, and the unchanged toluene distilled off; the ditolylethane is then collected over a range of 295–310°. There is practically no intermediate fraction, but a tarry residue of about 75 g. remains in the flask. Upon redistillation, the ditolylethane is collected at 295–300°. The yield is 290–310 g. (60–64 per cent of the theoretical amount) (Note 3) and (Note 4).

2. Notes

1. The acetylene is absorbed with unexpected rapidity so that it is unnecessary to deliver the gas beneath the surface of the liquid. In an ordinary reaction involving a gas it would be advisable to use a special stirrer, and to deliver the gas beneath the distributing tube of the stirrer.
2. It is scarcely necessary to remove the flask for weighing until near the end of the experiment, since the volume of acetylene is known and practically complete absorption takes place. If a tank of compressed gas is not available, the acetylene may be prepared from calcium carbide by the usual laboratory methods.[1]
3. Subsequent redistillation yields a product boiling practically over a 2° range. The best product was obtained by a final fractionation under diminished pressure, when the boiling point was found to be 144–145° /8 mm.
4. Xylene, mesitylene, ethylbenzene, and benzene condense with acetylene in a manner similar to that described above for toluene, although the yields are usually lower.[2]

3. Discussion

1,1-Di-*p*-tolylethane can be prepared from paraldehyde and toluene in the presence of sulfuric acid[3] and by the reduction of 1,1-di-*p*-tolylethylene with sodium in absolute alcohol.[4] The procedure described is essentially that of Reichert and Nieuwland.[2]

This preparation is referenced from:

- Org. Syn. Coll. Vol. 7, 290
- Org. Syn. Coll. Vol. 8, 391

References and Notes

1. Steinkopf, Chem. Ztg. **33**, 969 (1909) [C. A. **3**, 2887 (1909)].
2. Reichert and Nieuwland, J. Am. Chem. Soc. **45**, 3090 (1923).
3. Fischer, Ber. **7**, 1193 (1874).
4. Anschütz and Hilbert, Ber. **57**, 1699 (1924).

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

calcium carbide

alcohol (64-17-5)

sulfuric acid (7664-93-9)

acetylene (74-86-2)

Benzene (71-43-2)

ether (60-29-7)

sodium chloride (7647-14-5)

sodium carbonate (497-19-8)

acetone (67-64-1)

toluene (108-88-3)

sodium (13966-32-0)

xylene (106-42-3)

mercuric sulfate (7783-35-9)

ditolylethane,
1,1-DI-p-TOLYLETHANE,
Ethane, 1,1-di-p-tolyl- (530-45-0)

Mesitylene (108-67-8)

ethylbenzene (100-41-4)

1,1-di-p-tolylethylene (2919-20-2)

paraldehyde (123-53-7)