



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](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.

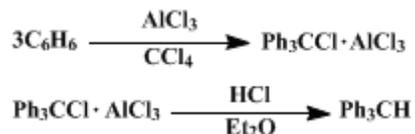
The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries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

*Organic Syntheses, Coll. Vol. 1, p.548 (1941); Vol. 4, p.81 (1925).*

## TRIPHENYLMETHANE

### [Methane, triphenyl-]



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### 1. Procedure

To a mixture of 292 g. (332 cc., 3.7 moles) of dry benzene and 116 g. (73.2 cc., 0.75 mole) of dry carbon tetrachloride (Note 1), in a 1-l. flask provided with a reflux condenser having a calcium chloride tube at its upper end, is added 100 g. (0.75 mole) of anhydrous aluminum chloride in lumps (Note 2). The flask is at once immersed in ice water to within 5 cm. of the top, and allowed to stand for twenty-four hours, the temperature of the water being allowed to rise to that of the room (Note 3). One hundred and ten grams (153 cc., 1.48 moles) of anhydrous ether is added in small portions, through the condenser, the flask being shaken occasionally during the twenty minutes required for the addition.

The mixture is allowed to stand for twenty-four hours, and is then poured into a 5-l. flask containing 650 g. of ice and 25 cc. of concentrated hydrochloric acid. One liter of benzene is then added, and the mixture heated on a steam bath under a return condenser. After gently boiling for five or ten minutes, the mixture is allowed to cool to 40–50°, when the benzene layer is separated, washed with 700 cc. of warm water containing 25 cc. of concentrated hydrochloric acid, and distilled. After the benzene has been removed under atmospheric pressure, the residue is transferred to a 300-cc. flask and distilled under reduced pressure, and a fraction boiling over the range 190–215°/10 mm. collected (Note 4). This weighs 135–160 g.; it solidifies on cooling. It is recrystallized from 550–650 cc. of ethyl alcohol (Note 5), and a colorless product, crystallizing in needles which melt sharply at 92°, is obtained. This is filtered off and washed twice with 30-cc. portions of fresh alcohol. The alcoholic mother liquor is concentrated and the residue distilled under reduced pressure, material which distils over at 190–200°/10 mm. being collected. This is recrystallized from ethyl alcohol, and 6–7 g. more of pure material is thus obtained. The total yield of recrystallized product is 125–154 g. (68–84 per cent of the theoretical amount).

### 2. Notes

1. A convenient method for removing moisture from the carbon tetrachloride and the benzene consists in distilling off about one-tenth of the liquid; this first fraction contains all the moisture which may have been dissolved in the commercial products. It is, as a rule, unnecessary to distil the remaining liquid before use.

2. The aluminum chloride may be a good grade of technical anhydrous material. If a powdered product is employed, the reaction is apt to proceed too rapidly. The lump material appears to be somewhat more efficient.

The calcium chloride tube can be connected with a trap (Fig. 7 on p. 97) for removal of the evolved hydrogen chloride.

3. With shorter periods of standing and lower temperatures, the yield falls off materially. Thus, in one experiment in which the mixture was allowed to stand for twenty-four hours at 5–8°, a large proportion of a lower-boiling product (apparently diphenylmethane) was formed, and only 109 g. of crude triphenylmethane was obtained.

4. A dark, tarry residue amounting to 25–40 g. remains in the flask.

5. Methyl alcohol may also be employed, but it is necessary to use 1600–1700 cc. of it. Triphenylmethane dissolves in one-third of its weight of warm benzene; it separates from this solution

with [benzene](#) of crystallization, which is lost on exposure to air or recrystallization from alcohol.

### 3. Discussion

Triphenylmethane can be prepared by the interaction of [benzene](#) and [chloroform](#) in the presence of [aluminum chloride](#)<sup>1</sup> or [ferric chloride](#);<sup>2</sup> by the reduction of [triphenylchloromethane](#) by ether under the influence of [aluminum chloride](#),<sup>3</sup> [ferric chloride](#),<sup>2</sup> or [zinc chloride](#);<sup>4</sup> by the reduction of [triphenylchloromethane](#) or [triphenylcarbinol](#) by means of alcohol in the presence of [sulfuric acid](#);<sup>5</sup> by heating [triphenylcarbinol](#) with [formic acid](#);<sup>6</sup> and from [benzene](#) and [benzal chloride](#) in the presence of [beryllium chloride](#).<sup>7</sup>

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### References and Notes

1. Schwartz, Ber. **14**, 1516 (1881); Friedel and Crafts, Bull. soc. chim. (2) **37**, 6 (1882); Allen and Kölliker, Ann. **227**, 108 (1885); Biltz, Ber. **26**, 1961 (1893).
  2. Meissel, Ber. **32**, 2422 (1899).
  3. Gomberg, Ber. **33**, 3147 (1900); Norris and Young, J. Am. Chem. Soc. **46**, 2580 (1924).
  4. Gomberg, J. Am. Chem. Soc. **35**, 204 (1913).
  5. Schmidlin and Garcia-Banús, Ber. **45**, 3188 (1912).
  6. Satisfactory yields can be obtained by refluxing practical triphenylcarbinol with an excess of formic acid. (W. W. Hartman, private communication). This general method, without statement of yields, has been mentioned by the following: Guyot and Kovache, Compt. rend. **154**, 121 (1912); Kauffmann and Pannwitz, Ber. **45**, 766 (1912); Kovache, Ann. chim. (9) **10**, 196 (1918).
  7. Bredereck, Lehmann, Schönfeld, and Fritzsohe, Ber. **72**, 1415 (1939).
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### Appendix Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

[ethyl alcohol](#),  
[alcohol](#) (64-17-5)

[sulfuric acid](#) (7664-93-9)

[hydrogen chloride](#),  
[hydrochloric acid](#) (7647-01-0)

[Benzene](#) (71-43-2)

[methyl alcohol](#) (67-56-1)

[ether](#) (60-29-7)

[chloroform](#) (67-66-3)

[formic acid](#) (64-18-6)

[carbon tetrachloride](#) (56-23-5)

[aluminum chloride](#) (3495-54-3)

Diphenylmethane (101-81-5)

zinc chloride (7646-85-7)

ferric chloride (7705-08-0)

Triphenylmethane,  
Methane, triphenyl- (519-73-3)

Triphenylchloromethane (76-83-5)

Triphenylcarbinol (76-84-6)

benzal chloride (98-87-3)

beryllium chloride (7787-47-5)