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.

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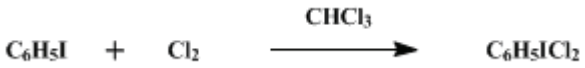
These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

DOI:10.15227/orgsyn.022.0069

Organic Syntheses, Coll. Vol. 3, p.482 (1955); Vol. 22, p.69 (1942).

IODOBENZENE DICHLORIDE

[Benzene, iodo-, dichloride-]



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1. Procedure

In a 1-l. three-necked flask, protected from the light and equipped with a mechanical stirrer, an inlet tube for the introduction of chlorine (Note 1), and an exit tube carrying a calcium chloride drying tube, are placed 150 ml. of dry chloroform (Note 2) and 102 g. (0.5 mole) of iodobenzene. The flask is cooled in an ice-salt mixture, and dry chlorine (Note 3) is introduced, as rapidly as the solution will absorb it, until an excess is present (usually about 3 hours is required). The yellow, crystalline iodobenzene dichloride is filtered with suction, washed sparingly with chloroform, and dried in the air on filter paper. The yield is 120–134 g. (87–94%) (Note 4) and (Note 5). The product is quite pure and may be used directly for the preparation of iodosobenzene and iodoxybenzene. Since iodobenzene dichloride decomposes slowly on standing, it should not be stored indefinitely.

2. Notes

1. The delivery tube should be at least 10 mm. in diameter and should terminate about 5 mm. above the surface of the liquid.
2. Chloroform was dried and rendered free of ethanol by allowing it to stand over anhydrous calcium chloride for 24 hours. It was then decanted through a filter and distilled through dry apparatus. The initial low-boiling fraction was rejected.
3. Chlorine was dried by passing it through at least two wash bottles containing sulfuric acid. Spray was removed by passing the gas through a plug of glass wool. The yields are reduced appreciably if the reactants are not dry.
4. In some runs an additional quantity of the product may be obtained by evaporating the chloroform filtrate to a small volume under reduced pressure. The solvent must not be evaporated completely if a pure product is desired.
5. The submitters report that the three isomeric iodotoluene dichlorides may be prepared in good yields by a similar procedure, but in these cases the chloroform solution must be concentrated by evaporation under reduced pressure (Note 4), since the tolyl homologs are more soluble in chloroform.

3. Discussion

The best preparative method for iodobenzene dichloride is the direct combination of iodobenzene and chlorine in chloroform.[1]

This preparation is referenced from:

- Org. Syn. Coll. Vol. 3, 483
- Org. Syn. Coll. Vol. 3, 485

References and Notes

1. Willgerodt, *J. prakt. Chem.*, (2) **33**, 155 (1886).

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

Benzene, iodo-, dichloride-

ethanol (64-17-5)

calcium chloride (10043-52-4)

chloroform (67-66-3)

chlorine (7782-50-5)

Iodobenzene (591-50-4)

iodobenzene dichloride (2401-21-0)

Iodosobenzene (536-80-1)

Iodoxybenzene (696-33-3)