



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.

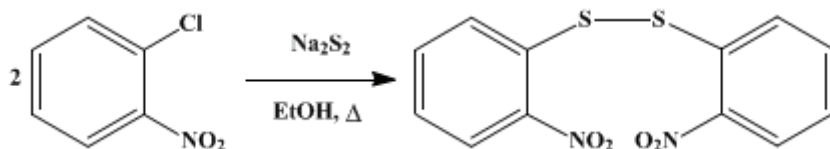
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses, Coll. Vol. 1, p.220 (1941); Vol. 8, p.64 (1928).

DI-*o*-NITROPHENYL DISULFIDE

[Disulfide, bis (*o*-nitrophenyl)]



Submitted by Marston T. Bogert and Arthur Stull.

Checked by Frank C. Whitmore, W. F. Singleton, and F. E. Smith.

1. Procedure

In a 3-l. round-bottomed flask fitted with a reflux condenser are placed 360 g. (1.5 moles) of crystalline [sodium sulfide](#) ([Note 1](#)) and 1.5 l. of 95 per cent [alcohol](#). The flask is heated on a steam bath until the sulfide dissolves. Then 48 g. (1.5 atoms) of finely ground [sulfur](#) is added, and the heating is continued until the [sulfur](#) has dissolved, forming a brownish-red solution of [sodium disulfide](#) ([Note 2](#)). A solution of 315 g. (2 moles) of [o-chloronitrobenzene](#) ([Note 3](#)) in 500 cc. of 95 per cent [alcohol](#) is prepared in a 5-l. round-bottomed flask fitted with a reflux condenser. The [sodium disulfide](#) solution is added slowly to this solution through a funnel placed in the top of the reflux condenser. The addition should be slow until the violence of the reaction abates somewhat.

The mixture is then heated on a steam bath, gently at first ([Note 4](#)), and then at full heat for two hours. After cooling, it is filtered by suction. The mixture of the organic disulfide and the [sodium chloride](#) is transferred to a 1-l. beaker and stirred thoroughly with 500 cc. of water to remove the [sodium chloride](#). It is then filtered by suction and the crystalline residue is washed on the filter with 100 cc. of [alcohol](#) to remove any [o-chloronitrobenzene](#). The product melting at 192–195° weighs 180–210 g. (58–66 per cent of the theoretical amount).

2. Notes

1. Crystals as dry as possible should be selected. If desired, an equivalent amount of analyzed fused [sodium sulfide](#) may be used. In this case, solution takes a longer time. Since an excess of [sodium sulfide](#) is added, slight inaccuracies in the amount used are not important.
2. In case a small amount of [sodium disulfide](#) separates in a layer at the bottom, it should be taken up with a little more alcohol and added to the [chloronitrobenzene](#) solution.
3. Commercial [o-chloronitrobenzene](#) containing 95–98 per cent of the ortho compound was used.
4. If too much heat is applied at first, the reaction becomes very violent.

3. Discussion

[Di-*o*-nitrophenyl disulfide](#) can be prepared by heating [o-chloronitrobenzene](#) in alcohol with [sodium sulfide](#) and [sulfur](#)¹ and from [o-nitrophenylsulfur chloride](#) and [potassium hydrosulfide](#).² The procedure described is that of Blanksma³ as elaborated by Wohlfahrt.⁴

This preparation is referenced from:

- [Org. Syn. Coll. Vol. 2, 455](#)
- [Org. Syn. Coll. Vol. 2, 471](#)
- [Org. Syn. Coll. Vol. 5, 843](#)

1. Elgersma, Rec. trav. chim. **48**, 752 (1929).
 2. Dougherty and Haas, J. Am. Chem. Soc. **59**, 2469 (1937).
 3. Blanksma, Rec. trav. chim. **20**, 127 (1901).
 4. Wohlfahrt, J.prakt. Chem. (2) **66**, 553 (1902).
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Appendix
Chemical Abstracts Nomenclature (Collective Index Number);
(Registry Number)

alcohol (64-17-5)

sodium chloride (7647-14-5)

sulfur (7704-34-9)

chloronitrobenzene,
o-chloronitrobenzene,
ochloronitrobenzene (88-73-3)

sodium sulfide (1313-82-2)

sodium disulfide

potassium hydrosulfide (1310-61-8)

Di-o-nitrophenyl disulfide,
Disulfide, bis (o-nitrophenyl) (1155-00-6)

o-Nitrophenylsulfur chloride (7669-54-7)