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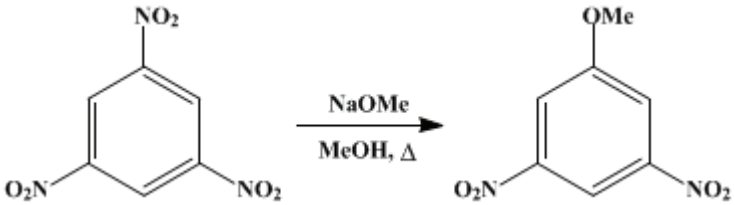
These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

DOI:10.15227/orgsyn.007.0028

Organic Syntheses, Coll. Vol. 1, p.219 (1941); Vol. 7, p.28 (1927).

3,5-DINITROANISOLE

[Anisole, 3,5-dinitro-]



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1. Procedure

A solution of sodium methoxide is prepared by dissolving 6.3 g. (0.27 atom) of sodium in 150 cc. of absolute methyl alcohol (Note 1) in a 1-l. flask provided with a reflux condenser. To this is added a solution of 50 g. (0.23 mole) of 1,3,5-trinitrobenzene (p. 541) in 550 cc. of absolute methyl alcohol. The mixture is boiled for about twenty minutes. The reflux condenser is then replaced by a still head and condenser, and about 300–350 cc. of alcohol is distilled. The residue in the flask is cooled to 20° and filtered.

The crude product is purified by recrystallization from hot ordinary methyl alcohol. On account of the low solubility of the dinitroanisole in hot methyl alcohol, the best results are obtained by boiling the crude product with a few grams of decolorizing carbon (Norite) in about 500 cc. of methyl alcohol, filtering the hot solution through a hot funnel, cooling the solution, filtering, and using the mother liquors for another extraction. After five extractions no more of the product is dissolved. The yield of pure product melting at 105° is 29–35 g. (63–77 per cent of the theoretical amount).

2. Notes

1. One liter of ordinary absolute methyl alcohol was treated with 5 g. of magnesium turnings and 0.1 g. of mercuric chloride, and then distilled as soon as the magnesium had dissolved.

3. Discussion

3,5-Dinitroanisole can be prepared by the action of sodium methoxide on 1,3,5-trinitrobenzene.[1]

References and Notes

1. Lobry de Bruyn, Rec. trav. chim. **9**, 208 (1890).

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

alcohol (64-17-5)

methyl alcohol (67-56-1)

magnesium,
magnesium turnings (7439-95-4)

sodium methoxide (124-41-4)

decolorizing carbon (Norite) (7782-42-5)

sodium (13966-32-0)

mercuric chloride (7487-94-7)

3,5-DINITROANISOLE,
Anisole, 3,5-dinitro- (5327-44-6)

1,3,5-Trinitrobenzene (99-35-4)

dinitroanisole

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