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.

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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.

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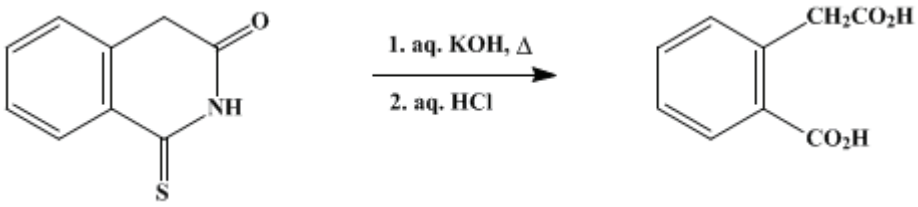
Organic Syntheses, Coll. Vol. 5, p.612 (1973); Vol. 44, p.62 (1964).

HOMOPHTHALIC ACID



Submitted by P. A. S. Smith and R. O. Kan[1].
Checked by Melvin S. Newman and Bernard Darre.

1. Procedure

Ten grams (0.056 mole) of 2*a*-thiohomophthalimide[2] and a solution of 30 g. of potassium hydroxide in 125 ml. of water are placed in a 300-ml., one-necked, round-bottomed flask (Note 1). The mixture is refluxed for 48 hours, filtered, and acidified with 12*N* hydrochloric acid. The solid that forms on cooling is collected by filtration and recrystallized from a mixture of 25 ml. of water and as much acetic acid (about 7 ml.) as necessary to dissolve the solid in the boiling solution, with addition of a little activated carbon. The yield of homophthalic acid, m.p. 181° (Note 2), is 6.1–7.5 g. (60–73%) (Note 3).

2. Notes

1. Because base can attack glass vessels, possibly introducing difficultly removable silicates into the reaction mixture, a copper flask is recommended for routine operations.
2. The melting point depends on the rate of heating. When the solid is heated slowly, the melting range can be as low as 172–174°.
3. An alternative procedure involves 3 days of refluxing in a mixture of 75 ml. of glacial acetic acid, 50 ml. of 12*N* hydrochloric acid, and 30 ml. of water. The product separates on cooling in a slightly lower yield (48%).

3. Discussion

Homophthalic acid may be obtained by the oxidation of indene,[3,4] the reduction of phthalonic acid,[5,6] and the hydrolysis of *o*-carboxyphenylacetonitrile.[7] Other methods are listed in an earlier volume.[3]

4. Merits of the Preparation

This is a general method for converting 2*a*-thiohomophthalimides to homophthalic acids. Since 2*a*-thiohomophthalimides are readily obtained from phenylacetyl chlorides,[2] this is a convenient method for preparing homophthalic acids.

This preparation is referenced from:

- Org. Syn. Coll. Vol. 5, 1051

References and Notes

1. Department of Chemistry, University of Michigan, Ann Arbor, Michigan.
2. P. A. S. Smith and R. O. Kan, this volume, p. 1051.
3. O. Grummitt, R. Egan, and A. Buck, *Org. Syntheses*, Coll. Vol. **3**, 449 (1955).
4. W. F. Whitmore and R. C. Cooney, *J. Am. Chem. Soc.*, **66**, 1239 (1944).
5. K. Miescher and J. R. Billeter, *Helv. Chim. Acta*, **22**, 608 (1939).

6. W. Davies and H. G. Poole, *J. Chem. Soc.*, 1617 (1928).
7. *C. C. Price and R. G. Rogers, Org. Syntheses, 22, 30 (1942).*

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

phthalonic acid

hydrochloric acid (7647-01-0)

acetic acid (64-19-7)

carbon (7782-42-5)

potassium hydroxide (1310-58-3)

indene (95-13-6)

Homophthalic acid (89-51-0)

thiohomophthalimide

o-CARBOXYPHENYLACETONITRILE (6627-91-4)