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.

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.

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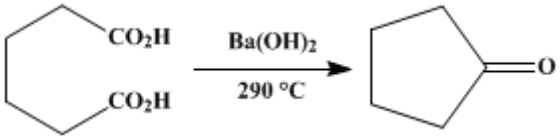
CYCLOPENTANONE



Submitted by J. F. Thorpe and G. A. R. Kon.
Checked by Roger Adams and C. R. Noller.

1. Procedure

In a 1-l. distilling flask (Note 1), fitted with a thermometer reaching within 5 mm. of the bottom, is placed an intimate mixture of 200 g. (1.34 moles) of powdered adipic acid (Note 2) and 10 g. of finely ground crystallized barium hydroxide. The mixture is gradually heated in a fusible alloy bath (Note 3) to 285–295° (Note 4) during about one and one-half hours, and maintained at that temperature until only a small amount of dry residue remains in the flask. This requires about two hours longer. The cyclopentanone distils slowly, accompanied by small quantities of adipic acid.

The ketone is separated from the water in the distillate, either by salting out with calcium chloride or by extracting with a little ether (Note 5). It is washed with a little aqueous alkali and then with water, dried over calcium chloride, and distilled through a good fractionating column (Note 6). The fraction boiling at 128–131° is cyclopentanone. The yield is 86–92 g. (75–80 per cent of the theoretical amount).

2. Notes

1. If larger runs are made, a three-necked flask provided with a mechanical stirrer should be used for the reaction.
2. The unrecrystallized adipic acid, prepared as described on p. 18, may be used.
3. An air bath may be used, but the metal bath insures better temperature control.
4. If the temperature goes above 300°, the adipic acid begins to distil quite rapidly. It is best to hold the temperature as near 290° as is possible.
5. It is reported that potassium carbonate is more satisfactory than calcium chloride for salting out the product from the distillate; traces of adipic acid are removed, and the loss which attends washing with alkali is avoided.[1]
6. Cyclopentanone is quite volatile with ether vapor, and careful fractionation is necessary when ether is used for the separation of the ketone from the water.

3. Discussion

Cyclopentanone can be prepared from adipic acid by distilling the calcium salt,[2] heating alone[3] or with acetic anhydride,[4] or in the presence of various catalysts such as barium hydroxide,[5] barium oxide,[6] thorium oxide,[7] manganous oxide,[7] uranium nitrate,[5] ferrous sulfate[5] and others.[8] A yield of 94 per cent has been reported using barium carbonate as a catalyst.[9]

References and Notes

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3. Aschan, Ber. **45**, 1605 (1912).
4. Blanc, Compt. rend. **144**, 1357 (1907); Godchot and Taboury, Ann. chim. phys. (8) **26**, 43 (1912).

5. Friedr. Bayer and Co., Ger. pat. 256,622 [Frdl. **11**, 49 (1912–14)]; Harries and Wagner, Ann. **410**, 36 (1915).
6. Vavon and Apchie, Bull. soc. chim. (4) **43**, 667 (1928).
7. Sabatier and Maihle, Compt. rend. **158**, 987 (1914).
8. Chavanne and Simon, ibid. **168**, 1326 (1919).
9. Boedtker, J. pharm. Chim. **15**, 225 (1932).

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

calcium salt

calcium chloride (10043-52-4)

potassium carbonate (584-08-7)

ether (60-29-7)

acetic anhydride (108-24-7)

Adipic acid (124-04-9)

barium oxide

ferrous sulfate (13463-43-9)

thorium oxide

Cyclopentanone (120-92-3)

barium hydroxide (17194-00-2)

uranium nitrate

barium carbonate (513-77-9)