



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.

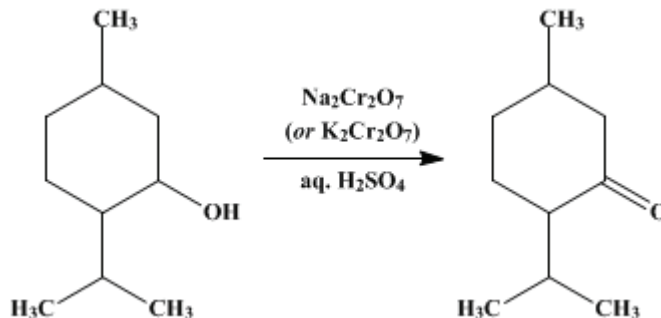
In some articles in *Organic Syntheses*, chemical-specific hazards are highlighted in red "Caution Notes" within a procedure. It is important to recognize that the absence of a caution note does not imply that no significant hazards are associated with the chemicals involved in that procedure. Prior 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.

Organic Syntheses, Coll. Vol. 1, p.340 (1941); Vol. 9, p.52 (1929).

***l*-MENTHONE**



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1. Procedure

In a 1-l. round-bottomed flask provided with a mechanical stirrer is placed 120 g. (0.4 mole) of crystallized [sodium dichromate](#) (or an equivalent amount of [potassium dichromate](#)), and to this is added a solution of 100 g. (54.3 cc., 0.97 mole) of concentrated [sulfuric acid](#) (sp. gr. 1.84) in 600 cc. of water. To this mixture 90 g. (0.58 mole) of [menthol](#) (crystals, m.p. 41–42°) is added in three or four portions and the mixture stirred ([Note 1](#)). Heat is evolved, and the temperature of the mixture rises to about 55° ([Note 2](#)). As soon as the reaction is complete the temperature falls. The oil is mixed with an equal volume of [ether](#), separated in a separatory funnel, and washed with three 200-cc. portions of 5 per cent [sodium hydroxide](#) solution ([Note 3](#)). The [ether](#) is then removed by distillation and the residue distilled under reduced pressure, the [menthone](#) being collected at 98–100°/18 mm. If distilled under atmospheric pressure it boils at 204–207°. The yield is 74–76 g. (83–85 per cent of the theoretical amount).

2. Notes

1. On addition of the [menthol](#) a black spongy mass forms which softens as the temperature rises and finally forms a dark brown oil.
2. The temperature may not reach 55°, in which case the mixture may be warmed gently with a small flame. If the reaction is slow in starting, gentle heating with a small flame is advantageous.
3. The oil, which is dark brown before washing with [sodium hydroxide](#), becomes light yellow. If three washings are not sufficient to remove the dark color another portion of [sodium hydroxide](#) solution is used.

3. Discussion

l-Menthone can be prepared by the oxidation of rhodinol with a [chromic-sulfuric acid](#) mixture;¹ by heating [menthol](#) with [copper](#) at 300°;² and by heating [3-menthene oxide](#) alone or in the presence of a catalyst.³ The procedure described is that of Beckmann.⁴

This preparation is referenced from:

- [Org. Syn. Coll. Vol. 8, 522](#)

References and Notes

1. Bouveault, Bull. soc. chim. (3) **23**, 464 (1900).
2. Neave, J. Chem. Soc. **101**, 513 (1912).

3. Schering-Kahlbaum A.-G., Fr. pat. 723,395 [C. A. **26**, 4063 (1932)].
 4. Beckmann, Ann. **250**, 325 (1889).
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Appendix
Chemical Abstracts Nomenclature (Collective Index Number);
(Registry Number)

rhodinol

sulfuric acid (7664-93-9)

ether (60-29-7)

sodium hydroxide (1310-73-2)

copper (7440-50-8)

sodium dichromate (7789-12-0)

potassium dichromate (7778-50-9)

menthol (15356-60-2)

menthone,
1-MENTHONE (1196-31-2)

chromic-sulfuric acid (15244-38-9)

3-menthene oxide