



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](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.

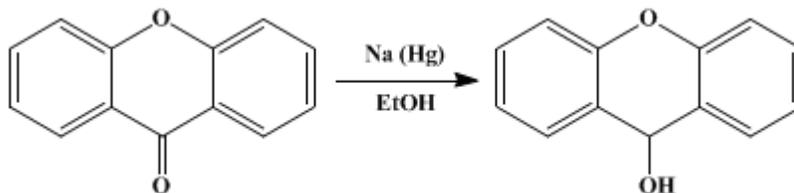
In some articles in *Organic Syntheses*, chemical-specific hazards are highlighted in red "Caution Notes" within a procedure. It is important to recognize that the absence of a caution note does not imply that no significant hazards are associated with the chemicals involved in that procedure. Prior 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.554 (1941); Vol. 7, p.88 (1927).*

## XANTHYDROL



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### 1. Procedure

An amalgam prepared from 9.0 g. (0.39 atom) of sodium and 750 g. (55 cc.) of mercury (Note 1) is warmed to about 50° (Note 2) in a 500-cc. Pyrex round-bottomed flask (Note 3). To it is then added a cold suspension of 25 g. (0.13 mole) of xanthone (p. 552) in 175 cc. of 95 per cent ethyl alcohol. The flask is at once stoppered, held in a cloth, and vigorously shaken, the stopper being raised from time to time to release any pressure. The temperature rises rapidly to 60–70°. The solid xanthone rapidly goes into solution, a very faint and transient blue color being developed. At the end of about five minutes the alcoholic solution is clear and practically colorless.

After a further ten-minute shaking the mercury is separated and washed with 10–15 cc. of alcohol. The alcoholic solution is filtered while warm and slowly poured with stirring into 2 l. of cold distilled water. The precipitated xanthydroxol is filtered with suction, washed with water until free of alkali, and dried at 40–50° to constant weight. The crystalline product, which weighs 23–24 g. (91–95 per cent of the theoretical amount), melts at 121–123° and is practically pure (Note 4) and (Note 5).

### 2. Notes

1. The amalgam may be conveniently prepared by placing the sodium in the 500-cc. flask, covering it with 15–20 cc. of dry toluene, and cautiously melting the metal over a free flame. The flame is then extinguished and the mercury added in drops, with occasional shaking. The reaction is extremely vigorous at first, but the rate of addition may be rapidly increased after the first 2–3 cc. of mercury has been added. The addition is so regulated that the toluene boils continuously, and it is finally allowed to volatilize completely. The toluene vapor remaining above the amalgam tends to protect it from the action of the air.
2. The amalgam is semi-solid at room temperature, but is completely melted at 50°. If made as in (Note 1), it is merely allowed to cool to 50° before the alcoholic xanthone is added.
3. A thick-walled flask is necessary; although no great pressure is developed at any time, the impact of the mercury during shaking might break thin glass.
4. Xanthydroxol is employed as a reagent for the determination of urea, with which it forms an insoluble condensation product. The material obtained is entirely satisfactory for this purpose; if desired, however, it may be recrystallized from alcohol, whereby the melting point is raised by about 1°.
5. Because of the marked instability of xanthydroxol, melting-point determination may afford little assurance of purity. It has been suggested that xanthydroxol be prepared directly prior to use or else kept in an alcohol solution in which it is much more stable than in the solid state (A. M. Ward,<sup>1</sup> private communication).

### 3. Discussion

Xanthydroxol can be prepared by the reduction of xanthone in alcoholic solution with zinc dust and sodium hydroxide,<sup>2</sup> with a low percentage sodium amalgam,<sup>1</sup> or with aluminum isopropoxide.<sup>3</sup> The procedure described is based on the method of Fosse.<sup>1</sup>

This preparation is referenced from:

- [Org. Syn. Coll. Vol. 1, 228](#)
- [Org. Syn. Coll. Vol. 1, 377](#)
- [Org. Syn. Coll. Vol. 1, 552](#)
- [Org. Syn. Coll. Vol. 2, 607](#)

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## References and Notes

1. Fosse, *Ann. chim.* (9) **6**, 58 (1916); Kny-Jones and Ward, *Analyst*, **54**, 574 (1929) [*C. A.* **24**, 39 (1930)]; Graff, Maculla, and Graff, *J. Biol. Chem.* **121**, 73 (1937).
2. Meyer and Saul, *Ber.* **26**, 1276 (1893); Adriani, *Rec. trav. chim.* **35**, 180 (1915); Kirkhgof and Spektor, *Khim. Farm. Prom.* **1934**, No. 2, 12 [*C. A.* **28**, 5451 (1934)].
3. Lund, *Ber.* **70**, 1520 (1937).

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## Appendix

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

amalgam

[ethyl alcohol](#),  
[alcohol \(64-17-5\)](#)

[sodium hydroxide \(1310-73-2\)](#)

[mercury \(7439-97-6\)](#)

[toluene \(108-88-3\)](#)

[zinc \(7440-66-6\)](#)

[sodium \(13966-32-0\)](#)

[aluminum isopropoxide](#)

[urea \(57-13-6\)](#)

[Xanthone \(90-47-1\)](#)

[Xanthidrol \(90-46-0\)](#)