



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.

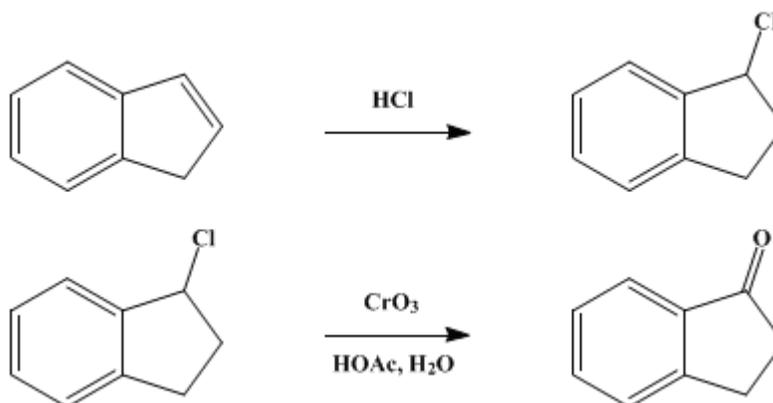
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

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α -HYDRINDONE

[1-Indanone]



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1. Procedure

A 250-cc. flask, fitted with an inlet tube reaching to the bottom, a thermometer, and an exit tube for conducting away unused gas, is immersed in an ice-water bath, and to it is added 80 g. (0.69 mole) of freshly distilled **indene** (Note 1). While the temperature of the liquid is kept at 5–10°, dry **hydrogen chloride** is passed in at a moderate rate until 24–27 g. of the gas has been absorbed. The addition takes from eight to ten hours and requires little attention from the operator. The crude **α -chlorohydrindene** is then transferred to a 250-cc. Claisen flask and distilled at diminished pressure. After a small fore-run (5–10 g.) containing **indene**, **α -chlorohydrindene** boiling at 90–103°/15 mm. is collected; the yield is 80–90 g. (Note 2).

In a 500-cc. three-necked flask, fitted with a stirrer, thermometer, and dropping funnel, is placed 100 g. (1 mole) of **chromic anhydride** dissolved in 100 cc. of water, and 100 cc. of glacial **acetic acid** is added. The **α -chlorohydrindene** is then admitted through the funnel at such a rate as to keep the temperature at 35–40°, the flask being cooled externally. This addition takes about one and one-half hours; after it is complete, stirring is continued for fifteen minutes before the mixture is poured into a large beaker and diluted with 300 cc. of water. After the acid present is neutralized by the addition of solid **sodium carbonate** (Note 3), the **α -hydrindone** is expelled from the mixture by steam distillation, care being taken to avoid entrainment of froth at the beginning of the process. When about 2.5 l. of distillate has been collected, the solution again begins to froth. The operation is stopped at this point, since further distillation does not yield an appreciable quantity of product. The **α -hydrindone** usually solidifies, as soon as the distillate is chilled, to a mixture of colorless crystals and a yellow solid; the product is collected after thorough cooling in an ice bath. This moist solid is dissolved in 200 cc. of **benzene**, and the solution is subjected to distillation until free from water. After the **benzene** is removed by vacuum distillation on the steam bath, the **α -hydrindone** is distilled. It boils at 125–126°/17 mm., and the distillate is a pale yellow solid, m.p. 39–41°. The yield is 46–55 g. (50–60 per cent of the theoretical amount, based on the **indene** used) (Note 4) and (Note 5).

2. Notes

1. If technical **indene** is employed, the fraction boiling at 178–182° is suitable for the preparation. The checkers used **indene**, b.p. 181.6–183.3°, obtained from the Barrett Company, New York City.
2. Most of the material distils at 100–103°/15 mm., but the yield of **α -hydrindone** is increased by using the material collected over the wider range.

3. An excess of [sodium carbonate](#) promotes foaming during the distillation and is to be avoided. Since the neutral point is not easily recognized with test paper, the carbonate is added in decreasing amounts until a fresh portion is no longer decomposed with gas evolution.
4. It is not practicable to dry the steam-distilled product in the air because [α-hydrindone](#) has such a high vapor pressure that the loss is appreciable. Drying can be accomplished in a vacuum desiccator, but this takes several days. The distillation specified gives a completely anhydrous product with the loss of no more than 2–5 g.
5. Crystallization is conveniently accomplished by dissolving the [α-hydrindone](#) in alcohol (1 cc. per gram) at room temperature, adding water until solid just begins to separate, and cooling to 0°. The substance crystallizes either as long plates or as leaflets.

3. Discussion

[α-Hydrindone](#) has been prepared from indene chlorohydrin,¹ the procedure described above; from indene bromohydrin by heating with dilute [sulfuric acid](#);² by cyclization of [hydrocinnamoyl chloride](#) with [ferric chloride](#)³ or [aluminum chloride](#);⁴ by the cyclization of [hydrocinnamic acid](#) with [hydrogen fluoride](#)⁵ or [sulfuric acid](#);⁶ and by the interaction of [acrylyl chloride](#) with [benzene](#) in the presence of [aluminum chloride](#).⁷

References and Notes

1. Hückel, Sachs, Yantschulewitsch, and Nerdel, *Ann.* **518**, 172 (1935).
2. Porter and Suter, *J. Am. Chem. Soc.* **57**, 2022 (1935).
3. Wedekind, *Ann.* **323**, 255 (1902).
4. Kipping, *J. Chem. Soc.* **65**, 484 (1894).
5. Fieser and Hershberg, *J. Am. Chem. Soc.* **61**, 1278 (1939).
6. Price and Lewis, *ibid.* **61**, 2553 (1939).
7. Moureu, *Bull. soc. chim. (3)* **9**, 570 (1893); *Ann. chim. phys. (7)* **2**, 198 (1894). Kohler, *Am. Chem. J.* **42**, 380 (1909).

Appendix

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

indene chlorohydrin

indene bromohydrin

[sulfuric acid](#) (7664-93-9)

[hydrogen chloride](#) (7647-01-0)

[acetic acid](#) (64-19-7)

[Benzene](#) (71-43-2)

[sodium carbonate](#) (497-19-8)

[hydrogen fluoride](#) (7664-39-3)

aluminum chloride (3495-54-3)

ferric chloride (7705-08-0)

Hydrocinnamic acid (501-52-0)

chromic anhydride

α -Hydrindone,
1-Indanone (83-33-0)

indene (95-13-6)

α -Chlorohydrindene (35275-62-8)

hydrocinnamoyl chloride (645-45-4)

acrylyl chloride (814-68-6)