



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.

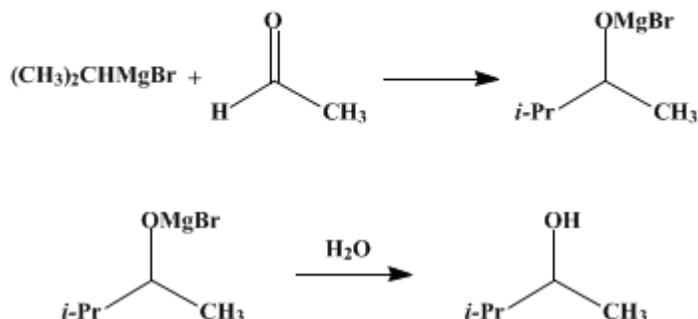
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses, Coll. Vol. 2, p.406 (1943); Vol. 12, p.48 (1932).

METHYL ISOPROPYL CARBINOL

[2-Butanol, 3-methyl-]



Submitted by Nathan L. Drake and Giles B. Cooke.

Checked by C. S. Marvel and B. H. Wojcik.

1. Procedure

In a 3-l. three-necked flask, fitted with a mechanical stirrer, a separatory funnel, and a reflux condenser the upper end of which is protected by a calcium chloride tube, are placed 146 g. (6 gram atoms) of dry magnesium turnings (Note 1) and about 250 cc. of dry ether (Note 2).

A solution of 600 g. (4.9 moles) of isopropyl bromide (Note 3) in 300 cc. of dry ether is then added through the separatory funnel. The reaction begins after about 15 cc. of the solution has been added (Note 4). The solution is added at such a rate that the reaction mixture refluxes gently. It is well to arrange to cool the flask with running water if the refluxing becomes too vigorous. The addition of the isopropyl bromide solution should require from three and one-half to four hours. The reaction mixture is refluxed on the water bath for forty minutes after addition of the isopropyl bromide solution is complete.

The flask is then cooled to -5° (Note 5), and a solution of 200 g. (4.5 moles) of acetaldehyde (Note 6) in 250 cc. of dry ether is added at this temperature over a period of one hour.

After addition of the acetaldehyde solution is complete, the product is decomposed by pouring the reaction mixture onto 2 kg. of cracked ice. The excess magnesium may be removed conveniently by decantation at this point. The basic magnesium halide is dissolved by addition of about 1 l. of 15 per cent sulfuric acid. The ether solution is separated, and the aqueous layer is extracted with four 150-cc. portions of ether. The ether solutions are combined, dried over 25 g. of calcined potassium carbonate, filtered, and fractionally distilled, using a short column. The methyl isopropyl carbinol distils at $110-111.5^\circ$. The fraction boiling at $37-109^\circ$ should be dried and refractionated. The total yield is 210-215 g. (53-54 per cent of the theoretical amount).

2. Notes

1. The excess of magnesium is used to increase the yield of Grignard reagent.
2. More ether may be added during the preparation to replace any which may be lost. The ether used should be dried over bright sodium wire.
3. The isopropyl bromide (b.p. $59-60^\circ$) was obtained from the Eastman Kodak Company and was used without further treatment. Directions for preparing isopropyl bromide are given in *Org. Syn. Coll. Vol. I, 1941, 37*, and on p. 359 above.
4. Heating on the water bath to start the reaction should be unnecessary if all the apparatus and reagents

are completely dry.

5. The temperature must not be allowed to rise above -5° .

6. The [acetaldehyde](#) may be prepared conveniently by depolymerizing pure, dry [paraldehyde](#) with [toluenesulfonic acid](#) as a catalyst. [Acetaldehyde](#) boiling at $20.5-21^{\circ}$ must be used for this preparation. There is no advantage in using gaseous [acetaldehyde](#).

3. Discussion

[Methyl isopropyl carbinol](#) has been prepared by reduction of [methyl isopropyl ketone](#) with [sodium amalgam](#),¹ and with [sodium](#);² by treatment of [chloroacetyl chloride](#) with zinc methyl;³ by treatment of [bromoacetyl bromide](#) with zinc methyl;⁴ by treatment of [isobutyraldehyde](#) with [methylmagnesium bromide](#);⁵ and as a by-product of the reaction between [chloroacetone](#) and [methylmagnesium iodide](#).⁶

This preparation is referenced from:

- [Org. Syn. Coll. Vol. 3, 200](#)

References and Notes

1. Münch, *Ann.* **180**, 339 (1876).
 2. Michael and Zeidler, *Ann.* **385**, 262 (1911).
 3. Bogomolez, *J. Russ. Phys.-Chem. Ges.* **13**, 396 (1881) [*Ber.* **14**, 2066 Ref. (1881)]; *Ann.* **209**, 86 (1881).
 4. Winogradow, *ibid.* **191**, 128 (1878).
 5. Henry, *Compt. rend.* **145**, 22 (1907).
 6. Fourneau and Tiffeneau, *ibid.* **145**, 438 (1907).
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Appendix Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

zinc methyl

[acetaldehyde](#) (75-07-0)

[potassium carbonate](#) (584-08-7)

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

[ether](#) (60-29-7)

[magnesium](#),
[magnesium turnings](#) (7439-95-4)

[Isopropyl bromide](#) (75-26-3)

[sodium](#),
[sodium wire](#) (13966-32-0)

[chloroacetyl chloride](#) (79-04-9)

methylmagnesium iodide (917-64-6)

bromoacetyl bromide (598-21-0)

chloroacetone (78-95-5)

Methyl isopropyl carbinol,
2-Butanol, 3-methyl- (598-75-4)

toluenesulfonic acid (88-20-0)

Methyl isopropyl ketone (563-80-4)

isobutyraldehyde (78-84-2)

methylmagnesium bromide (75-16-1)

paraldehyde (123-53-7)