



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.

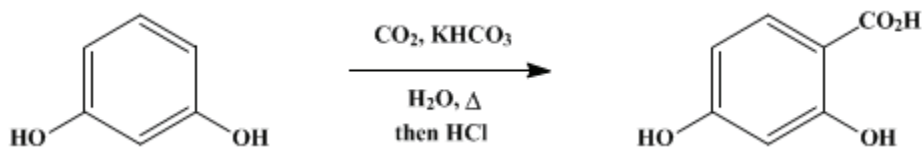
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses, Coll. Vol. 2, p.557 (1943); Vol. 10, p.94 (1930).

β-RESORCYLIC ACID



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1. Procedure

In a 5-l. flask fitted with a reflux condenser, a solution containing 200 g. (1.8 moles) of [resorcinol](#), 1 kg. (9.9 moles) of potassium acid carbonate ([Note 1](#)), and 2 l. of water ([Note 2](#)) is heated slowly on a steam bath for four hours. The flask is then placed over a flame and refluxed vigorously for thirty minutes; a rapid stream of [carbon dioxide](#) is passed through the solution during this heating process.

While still hot the solution is acidified by adding 900 cc. of concentrated [hydrochloric acid](#) (sp. gr. 1.19) from a separatory funnel with a tube delivering the acid to the bottom of the flask; this prevents the formation of a layer of acid over the unneutralized solution. The flask is allowed to cool to room temperature and is then chilled in an ice bath. The resorcylic acid crystallizes in prisms which are almost colorless but which, on exposure to air, turn pink owing to contamination with a small amount of [resorcinol](#). The yield of crude acid is 225 g. By extracting the mother liquor with [ether](#) several times, 35 g. of resorcylic acid and some unchanged [resorcinol](#) can be recovered. The resorcylic acid is extracted from the [ether](#) by shaking with an aqueous solution of [sodium bicarbonate](#). The aqueous solution is acidified with [hydrochloric acid](#) and again extracted with [ether](#). The [ether](#) is then evaporated, leaving the resorcylic acid, which is usually highly colored and must be recrystallized several times from boiling water and charcoal to remove the color.

The combined yield of crude acid (260–270 g.) is dissolved in 1 l. of water, boiled with about 25 g. of [Norite](#), filtered through a heated filter, and crystallized by placing in an ice-salt freezing mixture and stirring vigorously. A finely crystalline, colorless product is obtained in this way. If the acid is allowed to crystallize slowly the crystals are somewhat colored. The yield of pure resorcylic acid melting at 216–217° is 160–170 g. (57–60 per cent of the theoretical amount) ([Note 3](#)).

2. Notes

1. Instead of potassium acid carbonate, the sodium salt in corresponding quantity may be used.
2. If less than ten parts of water to one of [resorcinol](#) is used, the yield is diminished.
3. The air-dried crystals lose at 110° a quantity of water corresponding to a half mole of water of crystallization.

3. Discussion

The method of preparing [β-resorcylic acid](#) described above is a modification¹ of the procedure given by Bistrzycki and Kostanecki.² A more rapid but less efficient variant of this procedure has been described.³

This preparation is referenced from:

- [Org. Syn. Coll. Vol. 2, 100](#)

References and Notes

1. Clibbens and Nierenstein, J. Chem. Soc. **107**, 1494 (1915).
 2. Bistrzycki and Kostanecki, Ber. **18**, 1984 (1885).
 3. Couturier, Ann. chim. (11) **10**, 570 (1938).
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Appendix
Chemical Abstracts Nomenclature (Collective Index Number);
(Registry Number)

potassium acid carbonate

resorcyclic acid

resorcylic acid

hydrochloric acid (7647-01-0)

ether (60-29-7)

sodium bicarbonate (144-55-8)

carbon dioxide (124-38-9)

Norite (7782-42-5)

resorcinol (108-46-3)

β-Resorecylic acid (89-86-1)