



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.

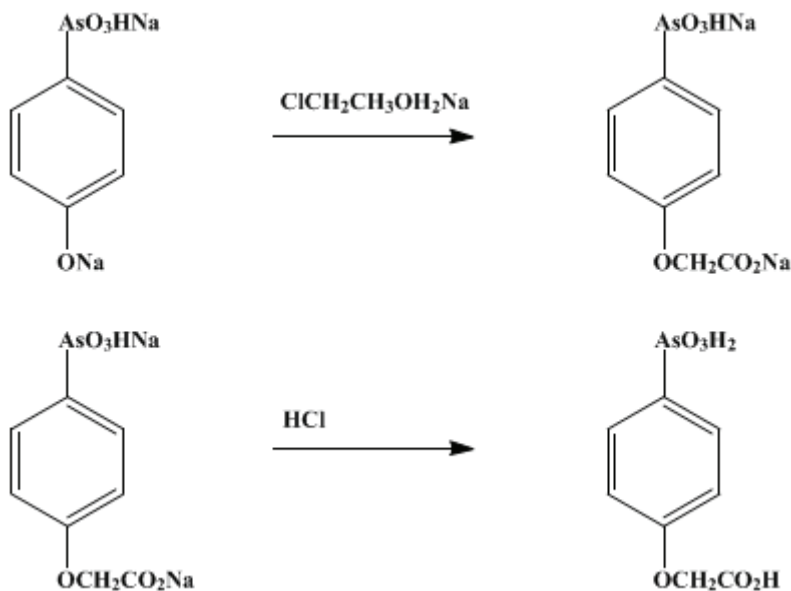
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses, Coll. Vol. 1, p.75 (1941); Vol. 7, p.4 (1927).

***p*-ARSONOPHENOXYACETIC ACID**

[Acetic acid, *p*-arsonophenoxy-]



Submitted by C. S. Palmer and E. B. Kester.

Checked by J. B. Conant

1. Procedure

In a 2-l. round-bottomed flask are placed 218 g. (1 mole) of *p*-hydroxyphenylarsonic acid (Note 1) and 375 cc. of water. A solution of 180 g. (4.5 moles) (Note 2) of sodium hydroxide in 375 cc. of water is added, and the mixture shaken until a homogeneous solution is obtained. After cooling to 40–50° there is added 189 g. (2 moles) of chloroacetic acid in small portions (Note 3) with stirring, and the clear solution is refluxed for four hours. At the end of this time the mixture is cooled to 20°, filtered to remove a slight flocculent precipitate, and the *p*-arsonophenoxyacetic acid precipitated (Note 4) by the addition of 200 cc. (2.1 moles) of hydrochloric acid (sp. gr. 1.19).

The product is filtered on a 10-cm. Büchner funnel and washed with three successive 100-cc. portions of cold water. It is then crystallized from 2 l. of hot water (Note 5), filtered, and washed twice with 100-cc. portions of cold water, once with 25 cc. of acetone, and once with 25 cc. of ether. After drying at 110° for one hour, there remains a pure white, anhydrous product weighing 110–120 g. (40–43 per cent of the theoretical amount) (Note 6). An arsenic analysis shows the product to be about 99 per cent pure.

2. Notes

- Commercial phenolarsonic acid may be used, or 240 g. of sodium *p*-hydroxyphenylarsonate (p. 490) and 140 g. of sodium hydroxide may be substituted for the acid and the larger amount of base.
- The use of less than 4.5 moles of alkali lowers the yield; an increase in the alkali concentration does not increase the yield.
- Unless the chloroacetic acid is added cautiously, there may be a violent reaction and the yield of *p*-arsonophenoxyacetic acid is lowered.
- The product sometimes does not precipitate readily without vigorous agitation and rubbing the inside wall of the vessel with a stirring rod.
- It is sometimes necessary to use decolorizing carbon in order to get a white recrystallized product.
- The odor of phenol is strong at the end of the reaction, but most of the *p*-hydroxyphenylarsonic acid

which does not react with the [chloroacetic acid](#) is not hydrolyzed and can be recovered in the filtrate from the [p-arsonophenoxyacetic acid](#).

3. Discussion

[p-Arsonophenoxyacetic acid](#) can be made by heating aqueous [sodium p-hydroxyphenylarsonate](#), 2 moles of [chloroacetic acid](#), and 4 moles of [sodium hydroxide](#).¹

References and Notes

1. Meister, Lucius and Brüning, Ger. pat. 216,270 [Frdl. 9, 1047 (1908–10)].
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Appendix Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

[hydrochloric acid](#) (7647-01-0)

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

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

[phenol](#) (108-95-2)

[chloroacetic acid](#) (79-11-8)

[acetone](#) (67-64-1)

[phenolarsonic acid](#)

[decolorizing carbon](#) (7782-42-5)

[p-ARSONOPHENOXYACETIC ACID](#),
[Acetic acid, p-arsonophenoxy-](#) (53663-15-3)

[p-hydroxyphenylarsonic acid](#) (98-14-6)

[sodium p-hydroxyphenylarsonate](#) (53663-20-0)