



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.

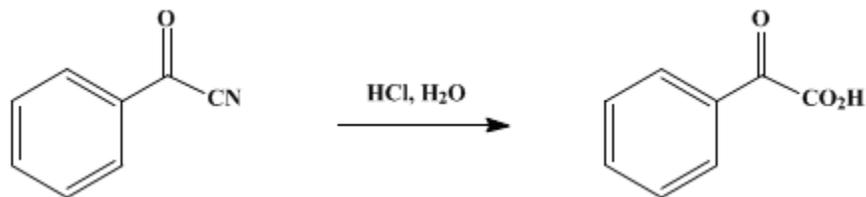
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses, Coll. Vol. 3, p.114 (1955); Vol. 24, p.16 (1944).

BENZOYLFORMIC ACID

[Glyoxylic acid, phenyl-]



Submitted by T. S. Oakwood and C. A. Weisgerber.
Checked by R. L. Shriner and Charles R. Russell.

1. Procedure

In a 1-l. flask are placed 50 g. (0.38 mole) of [benzoyl cyanide](#) (p. 112) and 500 ml. of concentrated [hydrochloric acid](#) (sp. gr. 1.18). The mixture is shaken occasionally until the solid is dissolved completely and is then allowed to stand at room temperature for 5 days ([Note 1](#)). At the end of this time the clear yellow solution is poured into 2 l. of water and extracted with one 400-ml. portion and three 250-ml. portions of [ether](#). The [ether](#) is removed by distillation from a steam bath, and the residual oil is placed in a vacuum desiccator containing [phosphorus pentoxide](#) and solid [sodium hydroxide](#) and allowed to remain there until dry ([Note 2](#)). The yield of crude solid acid melting from 57° to 64° is about 55–56 g. (96–98%). The crude acid is dissolved in 750 ml. of hot [carbon tetrachloride](#), and 2 g. of [Norit](#) is added ([Note 3](#)). The solution is filtered and allowed to cool to room temperature and then cooled in an ice-water mixture until crystallization is complete. The solid acid is filtered with suction, and the solvent remaining on the crystals is removed by placing the product in a vacuum desiccator for about 2 days. The yield of slightly yellow [benzoylformic acid](#) melting at 64–66° is 42–44 g. (73–77%) ([Note 4](#)).

2. Notes

1. With occasional shaking about a day is necessary for complete solution of the solid [benzoyl cyanide](#). A yellow oil separates which dissolves on shaking. At the end of this time some [ammonium chloride](#) occasionally separates.
2. If the oil does not crystallize it is cooled in an ice-water mixture until solidification is complete. The solid is returned to the desiccator for complete drying. The desiccator must be evacuated slowly or spattering will take place.
3. If [decolorizing carbon](#) is not used, the acid tends to separate as an oil which solidifies on cooling.
4. Titration with standard [sodium hydroxide](#) solution showed the acid to be about 99% pure.

3. Discussion

[Benzoylformic acid](#) can be prepared by the oxidation of [acetophenone](#) with [potassium permanganate](#) in alkaline solution,¹ by the oxidation of [mandelic acid](#) with [potassium permanganate](#) in alkaline solution,^{2,3} and by the hydrolysis of [benzoyl cyanide](#) with concentrated [hydrochloric acid](#).⁴

References and Notes

1. Claus and Neukranz, *J. prakt. Chem.* (2) **44**, 80 (1891).
2. Acree, *Am. Chem. J.*, **50**, 391 (1913).
3. *Org. Syntheses Coll. Vol. 1*, 241 (1941).
4. Boeseken and Felix, *Rec. trav. chim.*, **40**, 569 (1921).

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

hydrochloric acid (7647-01-0)

ether (60-29-7)

ammonium chloride (12125-02-9)

sodium hydroxide (1310-73-2)

Mandelic acid (90-64-2)

potassium permanganate (7722-64-7)

carbon tetrachloride (56-23-5)

Acetophenone (98-86-2)

decolorizing carbon,
Norit (7782-42-5)

Benzoylformic acid,
Glyoxylic acid, phenyl- (611-73-4)

Benzoyl cyanide (613-90-1)

phosphorus pentoxide (1314-56-3)