



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](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.

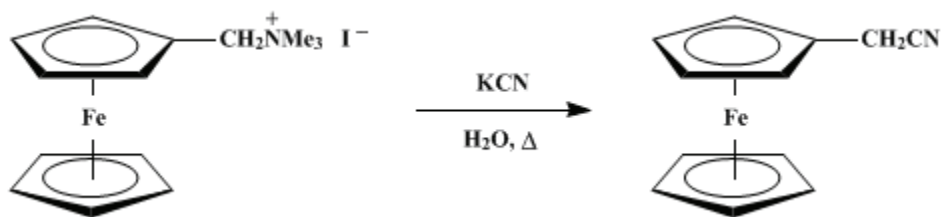
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*These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.*

*Organic Syntheses, Coll. Vol. 5, p.578 (1973); Vol. 40, p.45 (1960).*

## FERROCENYLACETONITRILE

### [Iron, [(cyanomethyl)cyclopentadienyl]cyclopentadienyl]



Submitted by Daniel Lednicer and Charles R. Hauser<sup>1</sup>.

Checked by B. C. McKusick and R. D. Vest.

### 1. Procedure

*Caution! This preparation should be carried out in a hood since trimethylamine is evolved.*

A solution of 57 g. (0.88 mole) of potassium cyanide in 570 ml. of water is placed in a 1-l. three-necked flask equipped with a stirrer and a reflux condenser. Fifty-eight grams (0.15 mole) of N,N-dimethylaminomethylferrocene methiodide<sup>2</sup> is added and the mixture is heated to boiling with good stirring. As the mixture is brought to boiling, the solid goes into solution. Within a few minutes of the onset of boiling, evolution of trimethylamine begins and a steam-volatile oil starts to separate from the solution. The reaction mixture is boiled vigorously with stirring for 2 hours and then is allowed to cool to room temperature. During the cooling the oil that has separated solidifies.

The solid is separated by filtration and the filtrate is extracted with three 150-ml. portions of ether. (*Caution! Gloves should be worn when handling this solution because of the large amount of cyanide it contains.*) The solid is dissolved in ether and this solution is combined with the extracts. The combined ethereal solutions are washed with water and dried over 5 g. of sodium sulfate. Removal of the solvent by distillation leaves crude ferrocenylacetonitrile as a solid or as an oil that crystallizes on being scratched. The nitrile is dissolved in about 200 ml. of boiling technical grade hexane. The hot solution is decanted from a small amount of insoluble black tar and is cooled to room temperature. Ferrocenylacetonitrile is deposited as bright yellow crystals, m.p. 79–82° (Note 1). The yield of the nitrile is 24–26 g. (71–77%) (Note 2).

### 2. Notes

1. The pure nitrile melts at 81–83° after further recrystallization from hexane.<sup>3</sup>
2. The yield is directly dependent on the quality of the methiodide employed. Yields as high as 95% have been obtained.<sup>3</sup>

### 3. Discussion

This method is that described by Lednicer, Lindsay, and Hauser.<sup>3</sup> No other procedure appears to have been employed to prepare this compound.

Essentially the present procedure converted 1-methylgramine to 1-methyl-3-indoleacetonitrile,<sup>4</sup> but it failed to convert benzyldimethylphenylammonium chloride to phenylacetonitrile.<sup>5</sup>

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### References and Notes

1. Department of Chemistry, Duke University, Durham, North Carolina. The work was supported by the Office of Ordnance Research, U. S. Army.
  2. See p. 434, this volume.
  3. D. Lednicer, J. K. Lindsay, and C. R. Hauser, *J. Org. Chem.*, **23**, 653 (1958).
  4. H. R. Snyder and E. L. Eliel, *J. Am. Chem. Soc.*, **70**, 1857 (1948).
  5. H. R. Snyder and J. C. Speck, *J. Am. Chem. Soc.*, **61**, 668 (1939).
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**Appendix**  
**Chemical Abstracts Nomenclature (Collective Index Number);**  
**(Registry Number)**

N,N-Dimethylaminomethylferrocene methiodide

Ferrocenylacetonitrile

ether (60-29-7)

sodium sulfate (7757-82-6)

potassium cyanide (151-50-8)

phenylacetonitrile (140-29-4)

Trimethylamine (75-50-3)

hexane (110-54-3)

1-methylgramine

1-methyl-3-indoleacetonitrile

benzyltrimethylphenylammonium chloride

Iron, [(cyanomethyl)cyclopentadienyl]cyclopentadienyl