



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.

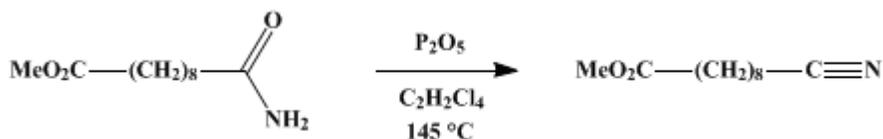
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses, Coll. Vol. 3, p.584 (1955); Vol. 25, p.69 (1945).

METHYL ω -CYANOPELARGONATE

[Pelargonic acid, θ -cyano-, methyl ester]



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1. Procedure

Caution! Tetrachloroethane is toxic; the operations should be conducted in a hood.

In a 1.5-l. Erlenmeyer flask to which is attached a reflux condenser, 190 g. (0.88 mole) of [methyl sebacamate](#) (p. 613) is dissolved in 200 ml. of boiling [tetrachloroethane](#). The solution is allowed to cool to 40–50° ([Note 1](#)), 95 g. (0.67 mole) of [phosphorus pentoxide](#) is added ([Note 2](#)), and the mixture is stirred well by means of a glass rod. The mixture is heated in an oil bath to 120° (thermometer in oil), and a second 95-g. portion of [phosphorus pentoxide](#) is added. After the mixture has been heated at 145° for 30 minutes with occasional hand stirring, the liquid is decanted. The residue is heated at 145° with 200 ml. of [tetrachloroethane](#) for 30 minutes with occasional stirring, and the liquid is decanted. This process is repeated once. The combined extracts are placed in a 1-l. flask, and most of the solvent is distilled under the reduced pressure of a water pump. The residue is transferred to a 300-ml. flask, and the remainder of the solvent is removed ([Note 3](#)). When no more distillate comes over, the receiver is changed, the water pump is replaced by an oil pump, and the residue is fractionated ([Note 4](#)). The yield of [methyl \$\omega\$ -cyanopelargonate](#) boiling at 121–124°/1 mm. ([Note 5](#)) is 119–124 g. (69–71%).

2. Notes

1. The slush that results on cooling is easily mixed with the [phosphorus pentoxide](#).
2. The [phosphorus pentoxide](#) is weighed rapidly on a piece of paper, from which it can be slid quickly into the flask.
3. About 550–560 ml. of [tetrachloroethane](#) is recovered. Ground-glass equipment is preferable for the distillations.
4. There is no fore-run. At 1 mm., the thermometer reads about 118° when the first drop appears at the end of the side tube of the distilling flask. There is a 5- to 6-g. fraction, b.p. 124–135°/1 mm., and some residue.
5. Other boiling points are 154°/5 mm. and 170°/14 mm.

3. Discussion

[Methyl \$\omega\$ -cyanopelargonate](#) has also been prepared by esterification of [\$\omega\$ -cyanopelargonic acid](#) with [methyl sulfate](#)¹ or [methanol](#),² and by dehydration of [methyl sebacamate](#) with [phosphorus pentoxide](#)³ or [thionyl chloride](#).⁴ The procedure described appears in the literature.^{1,3}

References and Notes

1. Biggs and Bishop, *J. Am. Chem. Soc.*, **63**, 944 (1941).
2. Biggs, U. S. pat. 2,339,672 [*C. A.*, **38**, 3990 (1944)].
3. Bishop, U. S. pat. 2,277,033 [*C. A.*, **36**, 4636 (1942)].

4. Soffer, Strauss, Trail, and Sherk, *J. Am. Chem. Soc.*, **69**, 1884 (1947).
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Appendix
Chemical Abstracts Nomenclature (Collective Index Number);
(Registry Number)

Pelargonic acid, θ -cyano-, methyl ester

[methanol \(67-56-1\)](#)

[thionyl chloride \(7719-09-7\)](#)

[methyl sulfate \(75-93-4\)](#)

[tetrachloroethane \(630-20-6\)](#)

[Methyl \$\omega\$ -cyanopelargonate \(53663-26-6\)](#)

[Methyl sebacamate \(53663-35-7\)](#)

[\$\omega\$ -CYANOPELARGONIC ACID \(5810-19-5\)](#)

[phosphorus pentoxide \(1314-56-3\)](#)