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.

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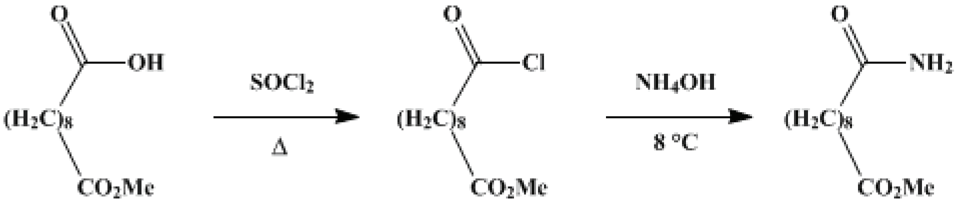
Organic Syntheses, Coll. Vol. 3, p.613 (1955); Vol. 25, p.71 (1945).

METHYL SEBACAMATE

[Sebacamic acid, methyl ester]



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1. Procedure

A. *ω-Carbomethoxypelargonyl chloride*. A mixture of 216 g. (1 mole) of methyl hydrogen sebacate (Note 1) and 147 g. (1.24 moles) of thionyl chloride in a 2-l. round-bottomed flask attached by a standard ground-glass joint to an upright condenser is refluxed on a steam bath for 5 hours. The condenser is then replaced by a still head having a modified side arm, and the unused thionyl chloride is removed. The residual ω-carbomethoxypelargonyl chloride is suitable for the next step. If a purer product is desired it is distilled under reduced pressure. The yield of ester chloride boiling at 158–160°/10 mm. is 194–201 g. (83–86%).

B. *Methyl sebacamate.* Two and one-half liters of concentrated aqueous ammonia (about 28%) in a 4-l. beaker or enameled pot (Note 2) is stirred vigorously with an off-center stirrer and chilled to 8° in a cooling bath. The crude chloride from part A is added slowly from a dropping funnel to the solution, which is kept below 8° throughout the addition. A vigorous reaction takes place, and methyl sebacamate precipitates immediately. After the addition has been completed, the product is filtered by suction and washed with 200 ml. of cold water. After 3 days' drying in a vacuum desiccator, the methyl sebacamate weighs 200–204 g. (93–95%) and melts at 72–74° (Note 3).

2. Notes

1. Methyl hydrogen sebacate (b.p. 168–170°/3 mm.) was prepared by the checkers in yields of 52–61% from sebacic acid and methanol by the procedure described for the ethyl analog.[1] The ethyl ester can be used to obtain ethyl sebacamate. The preparation of the acid chloride from ethyl hydrogen sebacate is mentioned in Note 12, p. 171.
2. A variety of enameled-steel pots known as bain-marie is available from the American Specialty Company, Rochester, New York.
3. The yield is no higher with the distilled chloride.

3. Discussion

These compounds have been described in the literature.[2,3]

This preparation is referenced from:

- Org. Syn. Coll. Vol. 3, 584
- Org. Syn. Coll. Vol. 3, 768

References and Notes

1. *Org. Syntheses* Coll. Vol. **2**, 276 (1943).
2. Biggs and Bishop, *J. Am. Chem. Soc.*, **63**, 944 (1941).
3. Bishop, U. S. pat. 2,277,033 [C. A., **36**, 4636 (1942)].

Appendix

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

ammonia (7664-41-7)

methanol (67-56-1)

thionyl chloride (7719-09-7)

Ethyl hydrogen sebacate (693-55-0)

methyl hydrogen sebacate (818-88-2)

sebacic acid (111-20-6)

Methyl sebacamate,
Sebacamic acid, methyl ester (53663-35-7)

ω-Carbomethoxypelargonyl chloride

ethyl sebacamate