Organic Syntheses

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.*

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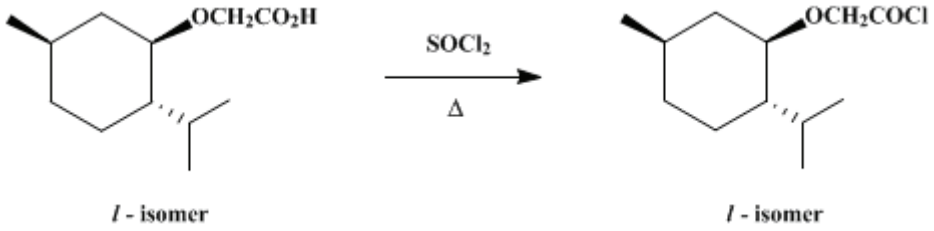
*Organic Syntheses, Coll. Vol. 3, p.547 (1955); Vol. 23, p.55 (1943).*

# *l*-MENTHOXYACETYL CHLORIDE

## [Acetyl chloride, (menthyloxy)-, *l*-]

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## 1. Procedure

A 1-l. three-necked round-bottomed flask is mounted on a steam cone and is fitted with a 250-ml. separatory funnel and a reflux condenser connected to a trap (Note 1) for absorbing gases. In the flask is placed 325 g. (198 ml., 2.73 moles) of thionyl chloride (Note 2), and to it is added, during the course of 1 hour, 125 g. (0.58 mole) of *l*-menthoxyacetic acid (p. 544). The flask is shaken frequently during the addition of the acid and, if necessary, is warmed to start the reaction. When all the acid has been added (Note 3), the reaction mixture is refluxed gently for 5 hours. After the reaction is complete, the excess of thionyl chloride is removed by distillation on the steam bath (Note 4) and the residue is distilled under reduced pressure. The yield of *l*-menthoxyacetyl chloride boiling at 117–120° /3 mm. (120–125° /5 mm.), $[\alpha]_D^{25}$ −89.6°, amounts to 115–118 g. (Note 5) (85–87%). The product turns dark on standing; it should be stored in a glass-stoppered amber bottle.

## 2. Notes

1. The gas-absorption trap shown in *Org. Syntheses* Coll. Vol. **1**, 97 (1941) may be used.
2. The commercial grade (b.p. 74–78°) of thionyl chloride was used.
3. Owing to the high viscosity of the acid, it is desirable to rinse the separatory funnel with a little thionyl chloride which is then added to the reaction mixture.
4. The recovered thionyl chloride may be redistilled for future runs. It is best to remove the last traces of thionyl chloride by heating the crude product to about 140° under the vacuum of the water pump.
5. The submitters report the same yields (per cent) when twice the amounts of materials are used.

## 3. Discussion

The procedure given above is adapted from that described by Read and Grubb.[1] No other methods for the preparation of *l*-menthoxyacetyl chloride have been described.

## References and Notes

**1.** Read and Grubb, *J. Soc. Chem. Ind.*, **51**, 330T (1932).

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

thionyl chloride (7719-09-7)

l-MENTHOXYACETIC ACID (40248-63-3)

L-Menthoxyacetyl chloride,
Acetyl chloride, (menthyloxy)-, l- (15356-62-4)