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.

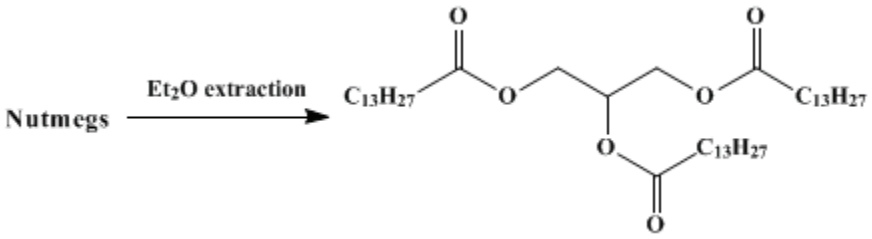
The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries or damages claimed to have resulted from or related in any way to the procedures herein.

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.

DOI:10.15227/orgsyn.006.0100

Organic Syntheses, Coll. Vol. 1, p.538 (1941); Vol. 6, p.100 (1926).

TRIMYRISTIN



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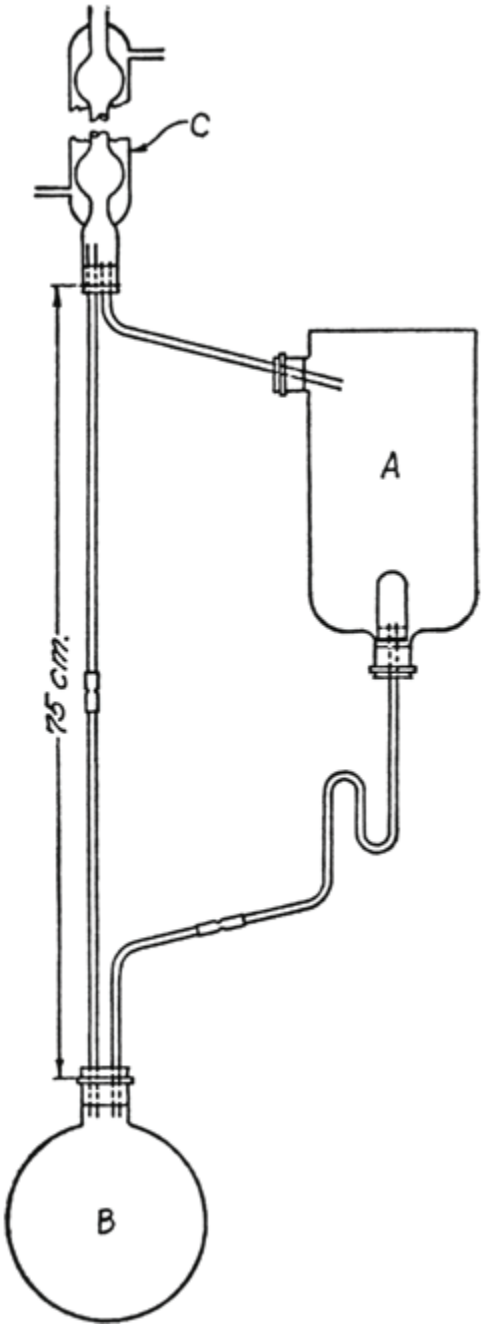
1. Procedure

In the container A (Fig. 30) is placed 1500 g. of crushed nutmegs (Note 1) moistened with ether (Note 2). A is a 3-l. inverted aspirator bottle connected by a 3-mm. glass tube to the efficient condenser C, and by 3-mm. tubing, one end of which is provided with a Soxhlet thimble to the 2-l. round-bottomed flask B. Flask B is connected by 3-mm. tubing of 75-cm. length to C. In B are placed 500 cc. of ether and a few chips of clay plate to prevent superheating. B is then heated on a steam cone so that the ether boils rapidly enough to reach the condenser C and to flow back through A.

Fig. 30.



The extraction with ether is continued until the ether leaving the insoluble solid is entirely colorless. This requires twenty-four to seventy-two hours, according to the state of subdivision of the nutmegs and the rate at which the ether is passed through. The ethereal solution is then freed of a small quantity of entrained insoluble matter by filtering through a folded paper. This filtration may advantageously be completed in the type of extractor described on p. 375. The clear solution is now entirely freed from ether by distillation on the water bath. The residue weighs 640–690 g. On cooling it sets to a mass of crystals of trimyristin which is filtered with suction (Note 3) and washed with 225 cc. of cold 95 per cent ethyl alcohol in small portions. The product is now recrystallized from 3.5 l. of 95 per cent ethyl alcohol; it is stirred mechanically during cooling since the trimyristin tends to separate as an oil at the outset (Note 4). The crystallized trimyristin is then filtered off by suction and washed with 350-400 cc. of 95 per cent alcohol in small portions. The crystals, which are colorless and practically odorless, melt at 54–55°. The yield is 330–364 g.

2. Notes

1. If the nutmegs are crushed to No. 40 powder, as recommended by the author, the extraction is complete in twenty-four to forty-eight hours; in checking it was found more convenient merely to pass the nutmegs through a food chopper (whereby they were broken up into pieces the largest of which were 3–4 mm. across), when the extraction required sixty-six to seventy-two hours for completion.
West India nutmegs are less expensive than the East India variety, and there seems to be no difference in yields of trimyristin between the two kinds.
If nutmeg butter, a commercial fat obtained by the hot pressing of ground nutmegs, is available, the above extraction may be omitted. The only operation necessary is a double crystallization of the crude material from boiling 95 per cent alcohol. Since nutmeg butter is frequently adulterated with foreign fats, the purity of the product should be checked by the saponification number (232 for pure trimyristin).
2. The nutmeg must first be moistened with ether; otherwise the extraction takes much longer. The author has found this apparatus to be generally satisfactory for the extraction of vegetable drugs with volatile solvents.
3. The filtrate from the crude trimyristin contains the odorous oils of the nutmeg. A further quantity of trimyristin may be obtained from it by distilling with steam and recrystallizing the non-volatile residue twice from alcohol; but the amount is not commensurate with the trouble, and this operation is not advised unless the residues from at least 5 kg. of nutmegs are on hand.
4. The alcohol may be distilled from the mother liquor of the recrystallization. The residue from this distillation may be added to the mother liquor of the first crystallization, which is then concentrated to the crystallization point. The crop of crystals thus obtained will usually require double recrystallization. Alcohol recovered from the first mother liquor will contain too much volatile oil of nutmeg to be used for other purposes.

3. Discussion

Trimyristin can be isolated from nutmegs by ether extraction.[1]

This preparation is referenced from:

- Org. Syn. Coll. Vol. 1, 379

References and Notes

1. Playfair, Ann. **37**, 152 (1841); Masino, Ann. **202**, 172 (1880); Power and Salway, J. Chem. Soc. **93**, 1653 (1908); Krafft, Ber. **12**, 1668 (1879); Verkade and Coops, Rec. trav. chim. **46**, 528 (1927).

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

ethyl alcohol,
alcohol (64-17-5)

ether (60-29-7)

trimyristin (555-45-3)