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.

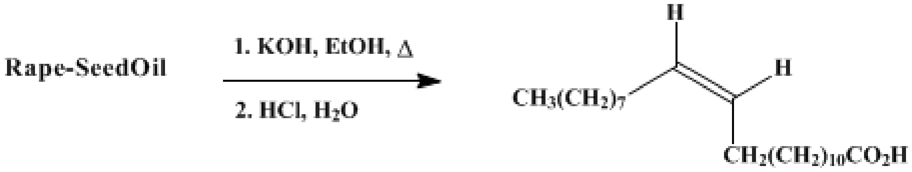
These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

DOI:10.15227/orgsyn.010.0044

Organic Syntheses, Coll. Vol. 2, p.258 (1943); Vol. 10, p.44 (1930).

ERUCIC ACID

[Hydrolysis of Rape-Seed Oil]



Submitted by C. R. Noller and R. H. Talbot.
Checked by H. T. Clarke and E. R. Taylor.

1. Procedure

In a 5-l. flask fitted with a reflux condenser are placed 2.5 l. of 95 per cent ethyl alcohol and 340 g. (4.5 moles) of commercial (73–75 per cent) potassium hydroxide. The mixture is gently shaken until the hydroxide is dissolved, 1330 g. (1.5 l., approximately 4 equivalents) of rape-seed oil is added with shaking (Note 1), and the mixture is refluxed on a steam bath for twenty-five to thirty hours.

The hot mixture is poured, with stirring, into 15 l. of warm water (50–60°), and is followed by 700 cc. (8.2 moles) of concentrated (36 per cent) hydrochloric acid (Note 2). After standing until the layers are distinct (ten to fifteen minutes), the lower layer is siphoned off as completely as possible and the oil is washed with two 1-l. portions of warm water.

The oil thus obtained, which should amount to 1460–1600 cc., is dissolved in three times its volume of 95 per cent ethyl alcohol and the mixture cooled to −10 to 0°, when the erucic acid crystallizes (Note 3). After six to eight hours at this temperature, the crude erucic acid is collected on a basket centrifuge (Note 4). The mother liquor, on chilling, yields a second crop of erucic acid. The combined product (800–1100 g.) melts either partially or wholly at room temperature, owing to the presence of oleic acid. It is dissolved in an equal volume of alcohol, chilled for six hours at 0°, and centrifuged as before, when it is obtained in well-defined crystalline form. The second crop of this recrystallization resembles that of the first crystallization and must again be recrystallized. The product is finally recrystallized once again from an equal volume of 95 per cent alcohol. The recrystallized acid contains alcohol, which is removed by heating to constant weight on the steam bath under diminished pressure. The yield of acid is 260–360 g. (Note 5); the acid melts at 31–32° (Note 6).

2. Notes

1. If the solution is mixed in this manner, the rape-seed oil emulsifies on being poured into the alkali and refluxing begins more smoothly.
2. If the acid is added before the water, esterification occurs to an appreciable extent, and the yield may fall to less than 200 g.
3. The crystallization is best accomplished by cooling for several hours at 0°. Cooling in an ice-salt mixture is much quicker but the erucic acid so obtained contains more oleic acid.
4. The centrifuge affords an easy way of filtering the erucic acid, since if the acid is not quickly separated from the mother liquor it melts and makes separation impossible. If a centrifuge is not available, the acid must be filtered at 0°, which is inconvenient except during cold weather.
5. The mother liquors from the recrystallizations may be combined, evaporated, and distilled under reduced pressure, taking two equal fractions. The lower (b.p. 200–220°/5 mm.) consists mainly of oleic acid; the higher (b.p. 220–230°/5 mm.) is solid at 20° and yields a further quantity of erucic acid on recrystallization.
6. The acid obtained contains a small percentage of arachidic acid and other higher saturated fatty acids, and has an iodine number of 66.9 (instead of about 75). If not entirely colorless, the product may be distilled under reduced pressure, when it boils at 241–243°/5 mm. or 252–254°/12 mm.; there is

practically no loss, only a minute amount of higher-boiling material remaining in the flask.

3. Discussion

Erucic acid is a constituent of various natural oils but is most conveniently obtained from rape-seed oil. The process described above is essentially that of Reimer and Will.[1] Methods have been developed for obtaining pure erucic acid free from saturated acids,[2] but these involve time-consuming procedures of fractional precipitation and crystallization, and necessarily give poor yields. The product obtained above is satisfactory for most purposes.

References and Notes

1. Reimer and Will, Ber. **19**, 3320 (1886); Müller, Rölz, and Wiener, ibid. **67**, 296 (1934).
2. Holde and Wilke, Z. angew. Chem. **35**, 105, 186, 289 (1922); Täufel and Bauschinger, ibid. **41**, 157 (1928).

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

ethyl alcohol,
alcohol (64-17-5)

hydrochloric acid (7647-01-0)

potassium hydroxide (1310-58-3)

oleic acid (112-80-1)

Erucic acid (112-86-7)

arachidic acid (506-30-9)