

## A Convenient Preparation of 10-Hydroxydecanoic Acid

### ABSTRACT

The alkaline cleavage of ricinoleates may be carried out in ethanol at temperatures of 190-200 C using two to three equivalents of NaOH to give yields up to 69% of pure 10-hydroxydecanoic acid. This method avoids the use of large excesses of alkali as well as the use of high boiling alcohols as reaction media.

Formation of 10-hydroxydecanoic acid by caustic fusion of ricinoleates suffers from a number of preparative disadvantages. Alkaline fusion at temperatures lower than those used for production of sebacic acid, i.e., 180-200 C, gives a low yield of hydroxy acid (1). Base treatment in the presence of a high boiling alcohol such as 2-octanol gives much better results, presumably because of the reducing capability of the added alcohol (2,3). However the large excess of this material must be removed during workup. Additionally the amount of NaOH used (up to 8.5:1 over ricinoleate) presents considerable neutralization problem. The method described here eliminates both of these objections and provides a convenient laboratory procedure for preparation of 10-hydroxydecanoic acid. The easily removable solvent, ethanol, is used as reaction medium and reducing agent, and a much lower base ratio is employed.

In this investigation a 300 ml stainless steel autoclave equipped with glass insert was routinely used as a suitable reaction vessel. Some etching of the glass occurred during each run, but the liner remained usable after 10 runs. An ordinary laboratory oven was used to heat the container, and no agitation was found to be necessary.

In a typical experiment we placed 46.8 g (150 mmoles) of methyl ricinoleate and a solution of 12.0 g (300 mmoles) of NaOH in 100 ml of 95% ethanol. The autoclave was placed in an oven at 190-200 C for 16-18 hr and then cooled to room temperature, vented to remove a certain amount of ethylene (identified by its mass spectrum) produced during heating and opened. After dissolving the semisolid contents in about 500 ml of water, the resulting solution was extracted with three 200 ml portions of ether to remove neutral material. This step may be omitted if it is not desired to retain the 2-octanol formed as coproduct. The aqueous phase was then acidified with 30 ml of concentrated hydrochloric acid, and the liberated acids extracted with two 200 ml portions of ether. After drying over magnesium sulfate and evaporation under reduced pressure to give about 35 g of solid, the crude acids were

then dissolved in 100 ml of warm ethyl acetate and 100 ml of heptane was added. After standing overnight at 0 C the mixture deposited 20.2 g of crystals, mp 71.5-74.5 C which on recrystallization from benzene, 140 ml, yielded 19.4 g (104 mmoles, 69%) of 10-hydroxydecanoic acid, mp 73-75 C (lit. 75-76 C [4]).

The above conditions are optimal when methyl ricinoleate is utilized, but the cleavage will proceed at lower temperatures. At 160 C about 41% yield of product was obtained after approximately the same reaction time. Prolonged heating at 190-200 C did not give a higher yield, nor did higher temperatures appear to be beneficial. Neither did it appear necessary to use a base ratio higher than 2:1. The conditions described may be applied to ricinoleic acid; however, the amount of water present in the medium appears to be critical, and 100% ethanol must be used in this case to obtain maximum yields. Sodium ricinoleate may be cleaved in 95% ethanol using one equivalent of added hydroxide. Castor oil may also be used, but best results are obtained with three equivalents of sodium hydroxide for each equivalent of ester in the glyceride, and absolute ethanol is necessary for best results. In none of the examples mentioned does sebacic acid appear as contaminant in the final product.

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### REFERENCES

1. Ansell, M.F., and B.C.L. Weedon, *Ind. Chemist* 40(2):70 (1964).
2. Diamond, M.J., R.G. Binder and T.H. Applewhite, *JAOCS* 42:882 (1965).
3. Diamond, M.J., and T.H. Applewhite, *Ibid.* 44:656 (1967).
4. Lycan, W.H., and R. Adams, *J. Am. Chem. Soc.* 51:625 (1929).

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## High Resolution NMR for Purity Determination of Cyclopropenoid Concentrates

### ABSTRACT

High resolution nuclear magnetic resonance is proposed as a method for assaying the cyclopropenoid concentration of methyl sterculate and methyl malva-

late concentrates. Spectra obtained on a 220 MHz instrument are analyzed and compared with that obtained on a 60 MHz instrument. Calculation of cyclopropene concentration based on methoxy protons and cyclopropene protons is discussed.