Letter to the Editor

Sir:

A number of years ago, one of the undersigned (1) published a modification of the classical method of Emil Fischer and coworkers (2) for preparing α-monoglycerides (1-monoacylglycerols), based on the use of isopropylidene-glycerol. According to the modification, isopropylidene-glycerol was obtained by condensing acetone and glycerol, using toluene-p-sulfonic acid as the catalyst and chloroform as the carrier for removing the water formed. The esterification was achieved by subsequent heating with a chosen fatty acid. The progress of both reactions was indicated by the amount of water collected in a glass cylinder placed in a column packed with glass tubes. The isopropylidene group was removed with boric acid.

Anfinsen and Perkins (3), in a *Letter to the Editor* in *JAOCS*, suggested a simplification of the esterification step by using a Soxhlet extraction apparatus, anhydrous magnesium sulfate for the absorption of the water formed during the reaction and benzene instead of chloroform as the water carrier. According to them, this method considerably reduced the time for esterification, the course of which was monitored by thin layer chromatography. Isopropylidene-glycerol would be obtained in the Soxhlet apparatus by the original method or by purchasing the commercially available product.

Having to produce recently a number of pure α-monoglycerides, an attempt was made to use benzene instead of chloroform in preparing isopropylidene-glycerol, while maintaining the collection of water as the indicator of the reaction progress. Since the boiling point of acetone is 19 C lower than that of benzene, the distillate consisted mainly of acetone, and the collection of water was very slow. The problem was solved by condensing glycerol with methyl ethyl ketone (2-butanone) instead of acetone in a Butt-type extractor or in a Dean Stark separator. Since the boiling point of methyl ethyl ketone is 80 C, benzene could be used as the water carrier and the time for preparing the glycerol intermediate and its subsequent esterification with fatty acid was reduced as a result of the higher reaction temperature. A further shortening of the procedure was achieved by using methyl isobutyl ketone (4-methyl-2-pentanone) with a boiling point of 116 C instead of acetone. This made possible the use of toluene (b.p. 110 C) as the water carrier and to reduce the total reaction time to about 2 hr.

Water collection as the indicator of the reaction progress appears preferable to thin layer chromatography, which requires additional work and the periodical withdrawal of samples. The use of a Butt extractor or Dean Stark separator, available in most fats research laboratories, shortens the time of synthesis compared with the use of a packed column as suggested originally, or a Soxhlet apparatus as proposed by Anfinsen and Perkins. The Dean Stark separator requires no adaptation. When using a Butt extractor, insert a small measuring cylinder or a calibrated test tube for water collection, using a piece of filter paper as a base. When the reaction is complete, the increase of the water volume stops.

We wish to conclude with an acknowledgement to Anfinsen and Perkins, whose findings have prompted the present investigation, and to apologize to the spirit of the great Emil Fischer for attempting, once more, to modify his work. This apology could be best expressed quoting a distich of one of his countrymen, which in translation runs as follows:

"'S wondrous how one wealthy man can provide for so many paupers;
Where the kings start to build, toilers have plenty to do."
—Schiller

Sincerely yours,
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REFERENCES

1. Hartman, L., Chem. & Ind. 711 (1960).
2. Fischer, E., M. Bergmann and H. Bärwind, Ber. 53:1589 (1920).
3. Anfinsen, J.R., and E.G. Perkins, JAOCS 41:779 (1964).