Derivatization of Corn Oil for Analysis by GC

Instrument: Perkin Elmer Clarus 500 GC with FID

In the derivatization of corn oil fatty acids with methanolic HCl, a general purpose transesterification reagent, removal of one of the reaction products drives the reaction toward completion. 2,2-Dimethoxypropane increases the methyl ester yield by reacting with glycerol as it is formed, but causes reaction byproducts to appear in the chromatogram. This effect can be avoided by adding dimethylsulfoxide to the reaction mixture.

Table 1. 2,2-DMP to Glycerol Ratio Affects the Reaction

Volume 2,2-DMP (µL)	2,2-DMP: Glycerol Molar Ratio	Observations
50	37:1	incomplete reaction
100	74:1	incomplete reaction
250	185:1	complete reaction, solution yellow, extra peaks in chromatogram
500	370:1	complete reaction, solution orange/red, extra peaks in chromatogram
750	546:1	complete reaction, solution black, extra peaks in chromatogram
1000	739:1	complete reaction, solution black, extra peaks in chromatogram

Calculations:

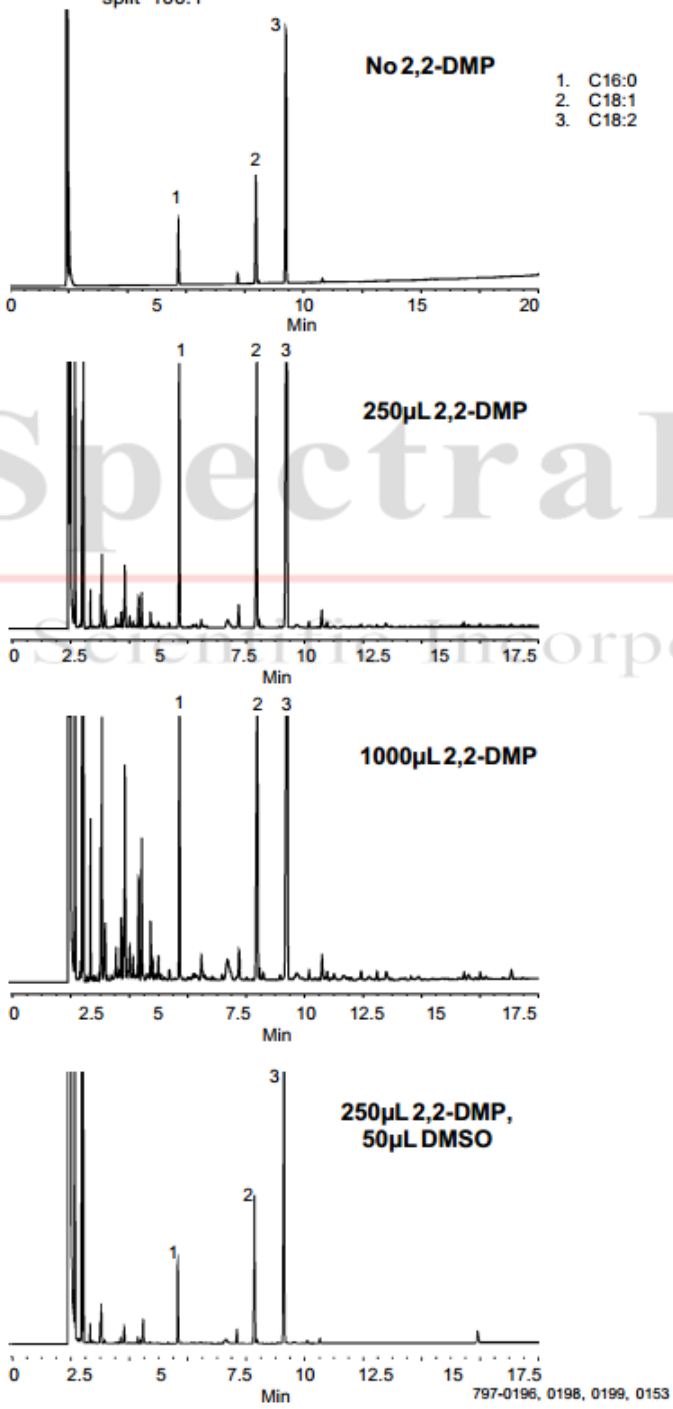
Moles Linoleic Acid
(mol. wt. linoleic acid) (3) + mol. wt. glycerol = moles linoleic acid
(280.50) (3) + 92.09 = 933.59 moles linoleic acid

Moles 2,2-DMP
(volume 2,2-DMP) (density 2,2-DMP) =
grams 2,2-DMP/mol. wt. 2,2-DMP = moles 2,2-DMP
(0.05mL) (0.847g/mL) = 4.07×10^{-4} moles 2,2-DMP

Molar Ratio 2,2-DMP:Glycerol
moles 2,2-DMP/moles linoleic acid
(4.07×10^{-4}) (0.11×10^{-4}) =37, etc.

Figure A. Fatty Acid Methyl Esters from Corn Oil

Column: **SP-2380, 30m x 0.25mm ID, 0.20µm film**
Cat. No.: **24110-U**
Oven: 150°C to 250°C at 4°C/min
Carrier: helium, 20cm/sec, set at 150°C
Det.: FID, 300°C
Inj.: 3µL corn oil derivatized with methanolic HCL +/- 2,2-DMP, split 100:1



References

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