Waters
Lab Highlights
An Internal Communication of Applications and Techniques
LAH 0088 2/83
Doc # M1025
AN/FA,OS/QC,MD/CA/FT

RAPID ANALYSIS OF FATTY ACIDS IN NATURAL OILS AND ALKYD RESINS USING WATERS "FATTY ACID ANALYSIS" COLUMN

Since the introduction of the Fatty Acid Analysis Column for the straightforward separation of free fatty acids (1,2), many applications for improved fatty acid analyses have appeared. These have included its use in margarine analysis (3), in the isolation of minor fatty acids in tall oil (4), and in its use in the coatings industry (5).

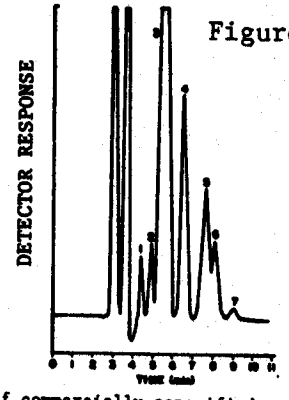
Recently, King et al. (6) have described an improved procedure for the identification and quantitation of free fatty acid mixtures derived from industrial oil and alkyd resin samples. The article includes a saponification procedure tailored for use with the Fatty Acid Analysis Column. The LC sample preparation procedure is a modification of two GC procedures.

The application of this technique to the identification of commercial oil sources of fatty acids is explained in the paper. A typical chromatogram is shown in Figure 1. This method has been applied to natural oils, such as soybean oil. coconut oil (as in Figure 2), linseed oil, tung oil, and castor oil.

When applied to alkyd resin analysis, the LC elution profile of fatty acids has an advantage over the GC procedure. Alkyd resins are derived from polyesterification reactions between selected acids, polyols, and fatty acids from natural oils. In GC the multifunctional alcohols and acids (Tabulated in Table 1) which are byproducts of the reaction, will contaminate the GC column if they are not totally removed. In LC these highly polar compounds are eluted with the solvent front. This simplifies sample prep. Also, unique chromatographic "fingerprints" can be obtained for each oil and used to make semi-quantitative conclusions regarding changes in the fatty acid composition of the oil during alkyd resin synthesis.

FREE FATTY ACIDS

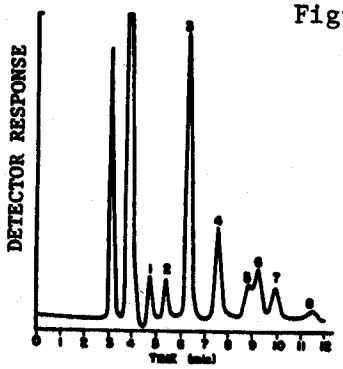
Figure 1



Chromatogram of commercially saponified coconut oil fatty acids. Identification of acids: (1) caprylic, (2) capric, (3) Lauric, (4) myristic, (5) palmitic and linoleic, (6) oleic, (7) stearic. Mobile phase composition: 40/40/25, water acetonitrile/tetrahydrofuran by volume.

FREE FATTY ACIDS

Figure 2



Chromatogram of laboratory saponified non-drying alkyd resin fatty acids. Identification of acids: (1) caprylic, (2) capric, (3) Lauric, (4) myristic, (5) linoleic, (6) palmitic, (7) oleic, (8) stearic

Waters
Division of MILLIPORE 34 Maple St. / Milford, MA 01757 / 617-478-2000

Quantitative Aspects. The LC analysis of coconut, soya, and castor oil derived fatty acid mixtures agree with individual component fatty acids (within 1-3%) as determined by gas-liquid chromatographic analysis of the methyl ester. The results reported were obtained on LC peaks which were uncorrected for detector response. (The paper states that it should be noted that comparisons to reported compositions of commercial oils are not entirely significant, since composition of certain major components in fatty acid mixtures derived from natural oil sources may vary as much as 5% due to the varied history of treatment of the oils.)

The agreement between LC and GC is shown in Table II.

TABLE I

ALKYD CONSTITUENTS NOT RETAINED ON THE FREE FATTY ACID COLUMN

Acids	Alcohols
Fumaric Acid	Pentaerythritol
Maleic Anhydride	Ethylene Glycol
Isophthalic Acid	Diethylene Glycol
Pthalic Anhydride	Mannitol
Benzoic Acid	Glycerol
Succinic Acid	Propylene Glycol
Terephthalic Acid	Trimethylolpropane

TABLE II

COMPARISON OF FATTY ACID COMPOSITIONS DETERMINED BY HPLC AND GC

	HPLC	GC
Fatty Acid	% Total	% Total
Caprylic	1.2	0.3
Capric	2.0	1.4
Lauric	58.3	56.0
Myristic	21.3	23.4
Palmitic	7.9	9.5
Linoleic	0.8	0.3
Stearic	1.6	1.9
Oleic	7.1	7.3

The GC chromatographic results were corrected for detector response while the liquid chromatographic data was obtained by normalizing peak heights uncorrected for refractometer response. In general, the agreement is moderately good and would undoubtedly be better if the liquid chromatographic peaks were corrected for detector response. It should be noted that the HPLC analysis took 4-1/2 minutes per sample.

Brian Bidlingmeyer

References:

1. B.A. Bidlingmeyer, R. Vivilecchia, and D. Clark, Jr., Abstracts, 29th Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, Cleveland, Ohio, March, 1976, No. 120
2. Fatty Acid Analysis Column Technical Bulletin, Waters Associates, Inc., Milford, MA.
3. A.G. Bailie, Jr., J.D. Stuart, and R.G. Jensen, Abstracts, 29th Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, Cleveland, Ohio, March 1978, No. 94.
4. A. Hase, T. Hase, and B. Holmbom, J. Am. Oil Chem. Soc., 57, 115 (1980).
5. T.K. Rehfeld, D.R. Scheuing, Anal. Chem., 50, 980A (1978).
6. J.W. King, E.C. Adams, B.A. Bidlingmeyer, J. Liquid Chromatogr. 5 (2) 275-304 (1982).