Wax Esters From Sunflower Oil Tank Settlings

During storage of crude sunflower oil a viscous sludge is formed in the bottom of the tank. One such tank settling has been reported to contain 4% wax esters (1). Since the acyl and alcohol contents of different preparations of wax esters vary (1,2) and because the waxes of sunflower oil probably contribute to cloud formation in chilled oils, another analysis seemed warranted.

Wax esters from one sample of commercial tank settlings (Minnesota Linseed Oil Co.) were isolated by the method of Popov and Stefanov (1). Also, wax esters were separated from sunflower hull lipids (3) by preparative thin layer chromatography in which silica gel layers were 1 mm thick and hexane-ether (90:10) was used as developing solvent. The wax ester fraction was then extracted from the silica gel with hot benzene. Wax esters were analyzed in an F&M Model 810 gas chromatograph with a 3 ft column packed with 3% OV-1 and temperature programmed from 250 to 400 C at 2 C/min. The wax esters (0.539 g) from the tank settlings were converted to methyl esters and free alcohols by refluxing for 24 hr with 5% hydrochloric acid in 30 ml methanol; 10 ml benzene was added to increase solubility of the wax esters. The mixture was separated on a ½ in. column packed with 17 g of 100/140 mesh Adsorbosil-CAB (Applied Science Laboratories, Inc.); fractions were eluted with hexane-ether (95:5). Products were methyl esters (0.253 g) and alcohols (0.258 g). Methyl esters were analyzed on two gas liquid chromatographs equipped with flame ionization detectors. One, a Packard Model 7401, was run at 210 C and had a 12 ft column packed with 5% LAC-2-R 446 on 60/80 Chromosorb W DMCS. The other, an F&M Model 5750, temperature programmed from 100 to 400 C at 4 C/min, was equipped with a 2 ft column packed with 3% OV-1 on Gas-Chrom Q. Alcohols were analyzed as trifluoroacetates (4) on the same OV-1 column used for methyl esters. All area determinations were made with an Infotronics CRS-40TS integrator system.

TABLE I

GLC Composition of Acids and Alcohols of the Wax Esters From Sunflower Oil Tank Settlings

Component	Acids, as methyl esters, area %	Alcohols, area %
14:0	Trace	......
15:0	Trace	......
16:0	0.6	......
18:0	0.9	1.5
18:1	0.8	0.3
18:2	3.5	
19:0	0.2	......
20:0	43.9	1.2
21:0	1.0	0.1
22:0	22.1	7.9
23:0	0.8	0.7
24:0	7.4	32.1
25:0	0.5	3.3
26:0	6.0	28.5
27:0	0.4	Trace
28:0	8.4	12.3
29:0	0.5	0.4
30:0	3.0	5.6
31:0	......	0.2
32:0	......	5.2
33:0	......	0.1
34:0	......	0.6

TABLE II

Wax Ester Composition, Area Per Cent by GLC

Carbon chain length[a]	From tank settlings	From hull (pericarp) oil	Random calculation
34			Trace
36			0.1
37			Trace
38			0.8
39			Trace
40			1.5
41			0.1
42	4.0	5.1	5.6
43	0.8	......	0.6
44	21.2	16.0	17.5
45	2.3	1.3	2.0
46	23.9	19.4	21.1
47	1.9	1.4	1.4
48	15.4	15.5	15.0
49	1.7	1.8	1.0
50	8.4	12.6	10.2
51	0.2	0.9	0.9
52	7.7	10.0	9.1
53	0.7	Trace	0.8
54	4.8	7.4	5.9
55	0.6	......	0.4
56	3.7	4.4	2.7
57	......	......	0.1
58	1.4	2.1	1.1
59	0.5	......	Trace
60	0.7	1.2	0.6
61		0.7	Trace
62		0.2	0.2
63			Trace
64			Trace

[a] Total number of carbon atoms in alcohol and acid.

Acid and alcohol compositions are given in Table I, and compositions of wax esters from the tank settlings and hull lipids are listed in Table II. The calculated composition of wax esters based on random combination between the acids and alcohols (from tank settlings) is also included in Table II. The data show that the compositions of the two wax preparations are similar to each other and to that calculated from random distribution. This similarity suggests that wax results from a random combination of alcohols and acids, and that wax esters in tank settlings have their origin in sunflower seed hulls.

R. Kleiman
F. R. Earle
I. A. Wolff
Northern Regional Research Laboratory
No. Utiliz. Res. Dev. Div., ARS, USDA
Peoria, Illinois 61604

REFERENCES

1. Popov, A., and K. Stefanov, Fette, Seifen, Anstrichmittel *70*, 234–238 (1968).
2. Kaufmann, H. P., and B. Das, Ibid. *65*, 398–402 (1963).
3. Earle, F. R., C. H. VanEtten, T. F. Clark and I. A. Wolff, JAOCS *45*, 876–879 (1968).
4. Freedman, B., Ibid. *44*, 113–116 (1967).

[Received March 25, 1969]

Power Shaker-Mixer for Use With Small Glass Vials

In a recent publication (1) from this laboratory, reference was made to a high speed mixing device which greatly facilitated two steps in the preparation of fatty acid methyl esters. Although the basic machine (Wig-L-Bug, Crescent Dental Manufacturing Co., Lyons, Ill.) is well known as a dentist's amalgamator and is widely used in spectroscopic laboratories for sample preparation, numerous requests have been received about the machine and the modification employed.

Basically, the standard Wig-L-Bug shaker was adapted to hold a 5 ml glass screw neck vial (A. H. Thomas, Philadelphia, Pa., No. 9802-E) fitted with a molded plastic cap (A. H. Thomas, No. 2849A-13). The dimensions of the vial with cap were ⅝ × 1⅞ in. and the weight of the fully loaded vial was 7.5 g. In the procedure, the glass reaction vial is tightly sealed by the plastic cap with cork backed tinfoil liner and losses of volatiles or contamination of the product are thus avoided. Much of the con-