

Modified Color Stability Test for Fatty Acids

The standard color stability test for fatty acids, AOCS Method Td 3a-64, (ASTM D 1981-61) requires approximately 45 g of material. In our experimental and development work, this amount of acid was often more than could be spared for the color stability test.

To alleviate this problem, a smaller size tube, 0.63 in. (1.6 cm) x 5.9 in. (15 cm), was used in place of the standard size tube, 1 in. x 9.5 in. Only 5 g of fatty acid was required per test with this size tube.

TABLE I
Heat Stability of Tall Oil Products

Material ^a	Rosin acid %	Gardner (1933) Color		
		Initial	AOCS stab. method	Modified stab. method
1. Distilled tall oil	29	4-	5-	5-
2. Fatty acid	4.0	7+	9	9
3. Fatty acid	3.8	5-	7	7
4. Fatty acid	1.3	3+	5+	5+
5. Fatty acid	0.6	4-	6+	7-
6. Fatty acid	2.0	3	5-	4+
7. Fatty acid	3.7	3+	4	4
8. Fatty acid	0.7	1-	2-	2-
9. Fatty acid	0.3	1-	1-	1-
10. Fatty acid	0.9	3+	6	6
11. Fatty acid	0.5	1-	4-	4-
12. Stearic acid	None	5	10	10

^a Samples 1 to 11 heated for 1 hr. Sample 12 heated for 2 hr.

To correlate this method (small tube) with the AOCS Method, the procedure was checked over several months by making comparisons using both sizes of tubes. These acids gave a range of heat stability colors from 1- to 10, the normal range used in our work. A few examples are shown in Table I.

Heating was conducted according to the AOCS Method L 15a-58 (1 hr at 205C if iodine value (I.V.) greater than 15, 2 hr at 205C if I.V. less than 15. All samples were blanketed with nitrogen).

There was excellent agreement between both methods in all the fatty acids that were checked. The difference in samples 5 (6+, 7-) and 6 (5-, 4+) is within the normal reproducibility limits.

Aside from the obvious advantage of small sample requirement, this method permits more tubes to be placed in a given size oil bath. Furthermore, the temperature of the bath is easier to maintain within the prescribed limits.

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Detection of Adulteration of Plant Oils with Special Reference to Olive Oil

Standard methods of testing plant oils for adulteration by cheaper fatty materials are still based either on (a) color reactions (Bellier, Halphen, Baudouin, etc.) specific for a type of seed oil, but negative in oils heated above 200C in the presence of oxidants, or (b) the determination of iodine value, refractive index, fatty acid pattern, etc. Since physical and chemical constants have a wide range of variation for each species of plant oil, adulterations up to 25-30% may not be detected even when the adulterated oil is very different from the adulterant as in the case of seed oils added to olive oil, a fruit coat oil (see Table I). The difficulty arises from the fact that the constants depend on the corresponding additive properties of the fatty acid mixtures which consist almost exclusively of the same C₁₆ and C₁₈ fatty acids in various proportions. This is also the reason why glyceride analysis by GLC does not give useful information in this case, in contrast to the case of animal and milk fats where various proportions of triglycerides exist in the range C₂₂ to C₅₄ (or even to C₆₀). Many papers have been published dealing with the adulteration of

oils, but the problem has not been satisfactorily resolved (see the Reports of the Literature Review Committee, *JAACS*, 1958 through 1964).

Triglycerides of plant oils are almost exclusively of the types C₅₄, C₅₂, C₅₀. However, each plant oil contains characteristic triglycerides that can serve as "fingerprints" (Fig. 1) when classified as SSO, SOL, LLLn, etc., where S and U are saturated and unsaturated fatty acids; O, L, Ln are oleic, linoleic, and linolenic acids and designations of SSO, etc., are without regard to position of the fatty acids. Detection of adulteration does not require complete separation and quantitation of the types of glycerides. It is sufficient to isolate a fraction enriched with a specific type of triglyceride and to determine its fatty acid composition. A paper chromatographic method based on an incomplete separation of triglycerides was proposed for this purpose by Kaufmann and Aparicio (1). These authors did not examine extreme cases of each species, i.e., samples containing very high or very low

TABLE I

	Refractive index (40C)	Iodine value	Linoleic acid (%)
Range for olive oils	1.4608-1.4630	78-90	4-20
Typical olive oil "A"	1.4612	82	6
Typical cottonseed oil "B"	1.4660	104	45
Olive oil "A" adulterated with cottonseed oil "B" up to 30%	1.4626	88.6	17.5

TABLE II

Oil tested	Fraction I		Fraction II	
	Oleic acid (%)	Linoleic acid (%)	Linoleic acid	
			(%)	(% of U)
Pure olive oils of linoleic acid contents 6-16%	71-74	3-4.5	0.5-2.0	0.9-3.0
Olive oil plus 6% cottonseed oil				
Olive oil plus 10% soybean oil				
	67	10	6	10
	62	14	12	19