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ISO RECOMMENDATION

R 660

CRUDE VEGETABLE OILS AND FATS

DETERMINATION OF ACIDITY

1st EDITION

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BRIEF HISTORY

The ISO Recommendation R 660, *Crude vegetable oils and fats – Determination of acidity,* was drawn up by Technical Committee ISO/TC 34, *Agricultural food products,* the Secretariat of which is held by the Magyar Szabványügyi Hivatal (MSZH).

Work on this question by the Technical Committee began in 1961 and led, in 1963, to the adoption of a Draft ISO Recommendation.

In March 1966, this Draft ISO Recommendation (No. 901) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Argentina	Hungary	Romania
Australia	India	South Africa, Rep. of
Belgium	Iran	Turkey
Bulgaria	Ireland	U.A.R.
Chile	Israel	United Kingdom
Colombia	Italy	U.S.S.R
Czechoslovakia	Netherlands	Yugoslavia
Finland	New Zealand	
France	Norway	
Germany	Poland	

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council which decided, in February 1968, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 660 February 1968

CRUDE VEGETABLE OILS AND FATS

DETERMINATION OF ACIDITY

1. SCOPE

This ISO Recommendation describes a method for the determination of the free fatty acid content of crude vegetable oils and fats. This can be expressed as an acid value or as conventional acidity.

2. DEFINITIONS

2.1 *Acid value.* Number of milligrammes of potassium hydroxide required to neutralize the free fatty acids in 1 g of the oil or fat.

2.2 *Acidity.* Conventional expression of the percentage of free fatty acids (see clauses 7.2 and 7.3).

3. PRINCIPLE

Solution of a known quantity of the oil or fat to be analysed in a mixture of ethanol and diethyl-ether, followed by titration of the free fatty acids present with an ethanolic solution of potassium hydroxide.

4. REAGENTS

4.1 *Mixture 1 + 1 v/v ethanol 95 % v/v and diethyl ether.* Neutralize exactly, just before use, by means of the ethanolic potassium hydroxide solution (4.2), in the presence of 0.3 ml of indicator (4.3) per 100 ml of this mixture (phenolphthalein or alkali blue 6 B, as the case may be (see section 6).

4.2 *Potassium hydroxide,* approximately 0.1 N or, if necessary, approximately 0.5 N solution in ethanol 95 % v/v. The exact concentration should be known, and checked immediately before use. Use a solution prepared at least five days previously and decanted into a bottle of brown glass, provided with a rubber stopper. The solution should be colourless or straw yellow.

NOTE. – It is recommended that the ethanol should be purified by adding 5 to 10 g of potassium hydroxide to 1 litre of the ethanol, boiling for one hour under reflux and finally distilling over.

4.3 *Indicator.* Phenolphthalein, 10 g per litre of ethanol 95 % v/v or, if necessary, alkali blue 6 B, 20 g per litre of ethanol 95 % v/v.

5. APPARATUS

5.1 *Conical flask,* 250 ml.

5.2 *Burette* graduated in 0.1 ml, complying with the specification for class A of ISO Recommendation R 385, *Burettes.*

5.3 *Analytical balance.*

6. PROCEDURE

6.1 Preparation of sample

Prepare the contract sample as described in ISO Recommendation R 661, *Crude vegetable oils and fats – Preparation of contract sample for analysis.*

6.2 Test portion

Weigh into the 250 ml conical flask, to the nearest 0.01 g, 5 to 10 g of the oil or fat, according to the acidity expected.

6.3 Determination

Dissolve the test portion in about 150 ml of the 1 + 1 mixture of ethanol and diethyl ether (4.1), previously neutralized.

If the solution obtained is not perfectly clear, add a further quantity of the mixture (4.1).

Titrate, with shaking, with the 0.1 N ethanolic solution of potassium hydroxide (4.2) to the end point of the indicator (pink colour of phenolphthalein persisting for at least 10 seconds). If the quantity of 0.1 N potassium hydroxide solution required exceeds 20 ml, a 0.5 N solution should be used (see clauses 8.1, 8.2, 8.3).

Carry out two determinations on the same prepared sample.

7. EXPRESSION OF RESULTS

7.1 It is recommended that the result of the analysis should be expressed as an acid value (see clause 2.1). Since 1 ml of N potassium hydroxide solution corresponds to 56.1 mg of potassium hydroxide,

$$\text{acid value} = \frac{V \times 56.1}{M}$$

where

V is the volume, expressed as millilitres of N solution, of ethanolic potassium hydroxide solution used,

M is the mass, in grammes, of the test portion.

Take as a result the arithmetic means of two determinations.

7.2 According to the nature of the oil or fat, the acidity can also be expressed in terms of the percentage of

- lauric acid (for coconut, palm kernel and similar oils),
- palmitic acid (for palm oil),
- erucic acid (for oils from certain cruciferae),
- oleic acid.

When the result shows simply "acidity", without further definition, it is conventionally always expressed as a percentage of oleic acid.

7.3 Whatever the mode of expression, the acidity can be calculated from the formula

$$\text{acidity, per cent} = V \times \frac{A}{1000} \times \frac{100}{M} = \frac{V \times A}{10 \times M}$$

where

A is the molar mass of the acid used for expressing the result, i.e.

– lauric acid	200
– palmitic acid	256
– oleic acid	282
– erucic acid	338

V and M have the same meanings given in clause 7.1.

8. NOTES ON PROCEDURE

8.1 If the solution becomes turbid during titration, add a sufficient quantity of the mixture specified in clause 4.1.

8.2 Until the potentiometric method is standardized, alkali blue 6 B may be used as indicator instead of phenolphthalein for dark oils or fats, the colour of which would not allow the end-point of the latter indicator to be seen clearly. In this case also the colour obtained after the change of the indicator from blue to red should persist for at least 10 seconds.

8.3 For oils or fats containing lauric acid, the temperature of the ethanol/ether solution should be kept between 15 and 20 °C during the titration.

8.4 In the presence of mineral acids it may be necessary to apply special techniques.