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METHODS FOR ANALYSIS OF ANIMAL AND VEGETABLE FATS AND OILS

PART 2 – DETERMINATION OF CHEMICAL CHARACTERISTICS

Section 5 : Determination of volatile acids (Reichert-Meissl, Polenske and Kirschner values)

(Second Revision)

SRI LANKA STANDARDS INSTITUTION

Sri Lanka Standard

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Gr. 4

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Sri Lanka

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This standard does not purport to include all the necessary provisions of a contract.

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METHODS FOR ANALYSIS OF ANIMAL AND VEGETABLE FATS AND OILS
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FOREWORD

This Sri Lanka Standard was approved by the Sectoral Committee on Agricultural and Food Products and was authorized for adoption and publication as a Sri Lanka Standard by the Council of the Sri Lanka Standards Institution on 2009-07-23.

This standard was first published in 1976 and subsequently revised in 1993. The standard prescribes the general methods for determining whether the material conforms to the requirements of the relevant individual standards and thus form a necessary adjunct to series of Sri Lanka Standard Specification for individual oils and fats. However keeping in view the experience gained during the years and various International standards brought out by the International Organization for Standardization (ISO) on the subject of testing animal and vegetable fats and oils, it was decided to revise it with a view to updating the existing methods of test and by incorporating those not covered earlier.

In order to accommodate large number of test methods within the scope of one standard, this standard is published in four parts covering different characteristics as indicated below.

Part 1 Determination of physical characteristic
Part 2 Determination of chemical characteristics
Part 3 Determination of foreign substances and parameters affecting quality and stability
Part 4 Determination of principle constituents and natural constituents.

1 SCOPE

1.1 This section prescribes the determination of values to characterize the volatile low molecular weight fatty acids.

NOTE : The *methods for the determination of volatile acids are empirical and determine only a part of these acids. Some acids still remain in the distillation flask. By working under specific constant conditions, a definite portion is distilled and hence comparable results are obtained. Hence the method must be strictly followed in its details.*

1.2 This method is applicable for animal and vegetable fats and oils. It is not applicable for waxes.

3 DEFINTIONS

For the purpose of this section the following definitions shall apply:

3.1 Reichert – Meissl value (R) (soluble volatile acid value) : The number of millilitres of 0.1 N aqueous alkali solution required to neutralize the water-soluble volatile fatty acids distilled from 5 g of the oil or fat under the precise conditions specified in the method.

3.2 Polenske value (P) (insoluble volatile acid value) : The number of millilitres of 0.1 N aqueous alkali solution required to neutralize the water-insoluble volatile fatty acids distilled from 5 g of the oil or fat under the precise conditions specified in the method.

3.3 Kirschner value (K) : The number of millilitres of 0.1 N aqueous alkali solution required to neutralize the water-soluble volatile fatty acids, which form water-soluble silver salts, distilled from 5 g of the oil or fat under the precise conditions specified in the method.

4 PRINCIPLE

The fat or oil is saponified with sodium hydroxide, acidified and distilled under standard conditions. The distillate is filtered and the soluble acids, the filtrate, is titrated. This is the Reichert- Meissl value. The insoluble acids are dissolved in neutral alcohol and titrated. This is the Polenske value. The titrated soluble acids are treated with silver sulphate and filtered and the filtrate is acidified and the distillate is titrated. This is the Kirschner value.

The Reichert-Meissl value reflects the amount of butyric acid and caproic acid, the Kirschner value reflects the amount of butyric acid alone and the Polenske chiefly caprylic, capric and lauric acids with some contribution from myristic and even palmic acids.

5 APPARATUS

5.1 *Beaker,* 25-ml capacity

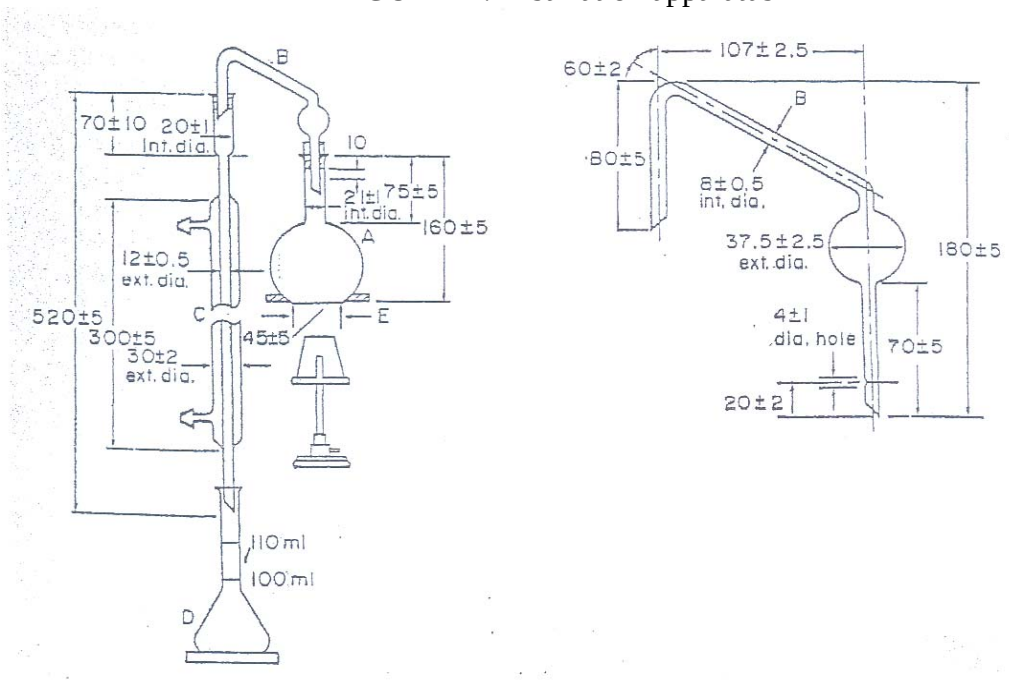
5.2 *Graduated cylinder,* 100-ml capacity

5.3 *Pipette,* 50-ml capacity

5.4 *Graduated burette*

5.5 *Distillation apparatus, special unit with diemensions all clearly mentioned (see figure 1)*

FIGURE 1 : Distillation apparatus



A. *Flat- bottomed boiling flask* of 300-ml capacity and base diameter 45 ± 5 mm.

B. *Still-head*, of glass tubing of wall-thickness 1.25 ± 0.25 mm. A rubber stopper fitted below the bulb of the longer arm of the still- head, and used for connecting it to the flask, shall have its lower surface 10 mm above the centre of the side hole of the still head.

C. *Condenser of glass tubing.* The wall thickness of the inner tube shall be 1.0 ± 0.2 mm and that of the outer jacket and of the wider part at the top 1.25 ± 0.25 mm.

D. *Receiver*, consisting of a flask, carrying two graduation marks on the neck, one at 100 ml and the other at 110 ml.

E. *Asbestos sheet* or similar material to support the flask (A) and to protect the sides of the flask from direct flame, with diameter 120 mm and thickness 6 mm and a central hole 65 mm in diameter.

5.6 *Bunsen burner*

5.7 *Water bath*

5.8 *Filter paper*, 90-mm diameter

5.9 *Pumice powder*

6 REAGENTS

All the reagents shall be of recognized analytical grade and the water used shall be distilled water or water of equivalent purity.

6.1 *Ethanol,* 95 per cent (v/v), neutralized to phenolphthalein immediately before use.

6.2 *Glycerol,* 98 per cent (v/v), relative density = 1.26 and neutral to phenolphthalein.

6.3 *Silver sulphate,* powdered.

6.4 *Sodium hydroxide,* 50 per cent (m/m) solution. Dissolve sodium hydroxide in an equal weight of water and store the solution in a bottle protected from carbon dioxide. Use the clear portion free from deposit.

6.5 *Sulphuric acid, dilute*. Dilute approximately 25 ml concentrated sulphuric acid to 1 litre and adjust until 40 ml neutralize 2 ml of the 50 per cent sodium hydroxide solution.

6.6 *Sodium hydroxide,* approximately 0.1 N (0.1 M) solution accurately standardized.

6.7 *Barium hydroxide,* approximately 0.1 N (0.05M) solution accurately standardized.

6.8 *Phenolphthalein indicator,* 0.5 per cent solution in 95 per cent (v/v) ethanol.

7 PROCEDURE

7.1 *Determination of Reichert-Meissl value (Water-soluble volatile acids)*

7.1.1 Weigh, to the nearest 0.01 g, 5.0 g of the fat sample into the flask (A) of the apparatus (5.5). Add 20 g (about 16 ml) of glycerol (6.2). Add 2 ml of sodium hydroxide (6.4) from the burette (5.4) protected against the entry of carbon dioxide; clean the nozzle before use by discarding the first few drops from the tap.

7.1.2 Heat the flask gently over a naked flame until the fat is melted and then with continuous shaking until the fat, including droplets adhering to the upper part of the flask, is saponified and the liquid has become perfectly clear. Avoid over heating during the process.

7.1.3 Allow to cool to about 90 ^{o}C. Add 93 ml of water, previously boiled for 15 minutes (free from carbondioxide) from the graduated cylinder (5.2), and mix by shaking. The liquid must remain limpid. If the solution is not clear (incomplete saponification) or if it is darker than a clear yellow (overheated), repeat the saponification on a fresh test portion. Add 0.1 g of pumice powder (5.9) and then 50 ml of sulphuric acid (6.5). Connect the flask to the distillation apparatus.

7.1.4 Heat very gently until the liberated fatty acids are completely melted. Then set the flame so that 110 ml of distillate is collected within 19 minutes to 21 minutes. The beginning of the distillation is to be taken as the moment when the first drop appears at the condenser end of the stilhead (B). Collect the distillate in the graduated flask (D). The temperature of the cooling water leaving the condenser should be within 15 ^{o}C to 20 ^{o}C.

7.1.5 When the distillate exactly reaches the 110-ml mark of the flask, remove the flame and quickly replace the flask by the beaker (5.1). Stopper the graduated flask and place it in a water bath (5.7) maintained at 15 ^{o}C, so that the 110-ml graduation is 1 cm below the water level in the bath, keep it there for 10 minutes.

7.1.6 Remove the graduated flask from the water bath, dry the outside and mix the contents by inverting the flask 4 to 5 times without shaking, avoid wetting the stopper with the insoluble acids. Filter the liquid through a dry, plain filter paper (5.8), fitting snugly into a funnel. The filtrate should be clear (see Note 1).

7.1.7 Pipette 100 ml of filtrate and add five drops of phenolphthalein (6.8) Titrate with barium hydroxide (6.7); the flask should be dry before use. If the volume of barium hydroxide required for the titration is less than 5 ml, add 5 ml of water previously boiled for 15 minutes (see Note 2). Close the flask with a cork and retain the solution for the determination of Kirschner value as prescribed in 7.3. If the determination of the Kirschner value is not required, sodium or potassium hydroxide (6.6) may be used for the titration.

7.1.8 Carry out a blank test.

7.2 *Determination of Polenske value (water-insoluble volatile acids)*

7.2.1 After titrating the soluble volatile acids, detach the stillhead (B) and rinse the condenser (C) with 15 ml of water at 15 ^{o}C, previously boiled for 15 minutes. Collect the washing in the beaker (5.1). Rinse graduated flask (D) with this liquid, pour into the filter, and allow all of it to pass through.

7.2.2 Repeat this operation twice more, using 15 ml of water at each time. The size of the filter is such that each 15 ml portion can be poured straight into it and will fill it to the brim. The last wash should be collected separately and should require not more than one drop of alkali solution (6.6) for neutralization. Reject the aqueous washings.

7.2.3 Place the funnel on a clean, dry flask. Dissolve the water-insoluble fatty acids by washing the condenser with 15 ml of ethanol (6.1). Collect the washing in the beaker (5.1). Then use the liquid to rinse the volumetric flask (D) and pour it in one lot onto the filter. Wash twice more in the same way.

7.2.4 Titrate the total ethanolic washing (45 ml in all), with barium hydroxide (6.7) or sodium or potassium hydroxide (6.6) using phenolphthalein (6.8) as the indicator.

7.3 ***Determination of Kirschner value.***

7.3.1 Add 0.5 g of powdered silver sulfate (6.3) to the solution, neutralized with the barium hydroxide (6.7) obtained in 7.1. Allow the flask to stand in the dark for 1 hour with occasional shaking. Filter the contents away from the light through a dry filter. Transfer 100 ml of the filtrate into a dry flask similar to flask A (see Note 2). Add 35 ml of cold water recently boiled for 15 minutes, 10 ml of sulfuric acid (6.5) and 0.1 g of pumice powder (5.9) (see Note 3).

7.3.2 Attach the flask to the distillation apparatus (5.5) and distil as before so that 110 ml of distillate is collected in 19 minutes to 21 minutes. The beginning of distillation is taken as the moment when the first drop is formed at the lower (condenser) tip of the still head (B). Collect the distillate in the graduated flask (D). When the distillate reaches the 110-ml mark, remove the flame and quickly replace the flask by the beaker (5.1).

7.3.3 Stopper the flask (D) and mix the distillate by inverting the flask 4 times or 5 times without shaking. Filter through a dry plain filter (5.8) fitting snugly in a funnel. The filtrate should be clear. To 100 ml of the filtrate add 5 drops of phenolphthalein (6.8) and titrate with barium hydroxide (6.7). Carry out a blank test using the solution obtained from the blank test prescribed in 7.1, and neutralize with the barium hydroxide (6.7).

NOTES:

1. The filtrate should be free from insoluble fatty acids. This cannot easily be achieved in coconut oil as liquid insoluble fatty acids may pass through the filter. This may be avoided by using a wetted filter paper, or by collecting the filtrate in a separating funnel and, after settling, removing the lower aqueous layer, the insoluble acids will float at the surface. Add these to the bulk of the insoluble acids.

2. *If the Reichert-Meissl value is low, the volume of the filtrate may not be enough to provide 100 ml for the distillation flask. In this case, 5 ml of water, previously boiled for 15 minutes, may be added to the filtrate and the factor* $(100 + V_1)$ *should be substituted by* $(105 + V_1)$ *in the equation 8.3.*

3. In the determination of Kirschner value, the pumice powder may be replaced by a 5 - mm coil of aluminium wire, 30 cm in length and 1 mm in diameter.

8 EXPRESSION OF RESULTS

8.1 *Reichert – Meissl value = 11 ($V_1 – V_2$) c.*

where,

V_1 = volume in ml, of the barium hydroxide required for the titration;
V_2 = volume in ml, of barium hydroxide required for the blank test; and
c = concentration, in mol/l of the alkali solution.

8.2 *Polenske value (see Note) = 10 V_3 c*

where,

V_3 = volume in ml, of the standard alkali solution required for the titration; and
c = concentration, in mol/l, of the alkali solution.

8.3 *Kirschner value = 0.121 ($V_4 – V_5$) (100 + V_1) c_1*

where,

V_1 = volume in ml, of the barium hydroxide solution used for the determination of the Reichert- Miessl value;

V_4 = volume in ml, of barium hydroxide required for the titration;

V_5 = volume in ml, of the barium hydroxide solution for the blank test; and

c_1 = concentration, in mol/l, of the barium hydroxide solution.

NOTE : *The Polenske value, and to a less extent the Reichert-Meissl value, may come out too low if the determination is carried out at low barometric pressure such as may occur at high altitudes.*

The following formulae may be used to correct the observed values found at a barometric pressure of h mmHg *to values appropriate to normal pressure.*

$$Corrected\ \mathrm{Re}ichert - Meissl\ \ value = \frac{(Observed\ \ value - 10)x\log 760}{\log h}$$

$$Corrected\ \ Polenske\ \ value = Observed\ \ value\ \ x\ \ \frac{760 - 45}{h - 45}$$

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