EXPERIMENT 27  DETERMINATION OF REICHERT MEISSL (RM) VALUE AND POLENSKE VALUE (PV) IN OILS AND FATS

Structure

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27.0 OBJECTIVES

After attending to this experiment, we shall be able to:

- learn to perform determination of Reichert Meissl (RM) value and Polenske value (PV) in oils and fats.

27.1 INTRODUCTION

Reichert Meissl value refers to the number of ml of 0.1N NaOH solution required to neutralize the steam volatile, water soluble fatty acids distilled from oil or fat under precise specified conditions. It is the measure of water soluble steam volatile fatty acids chiefly butyric and caproic acids present in oil or fat. Polenske value refers to the number of ml of 0.1N aqueous NaOH solution required to neutralize the steam volatile, water insoluble fatty acid distilled from oil or fat under precise specified conditions. PV is measure of steam volatile and water insoluble fatty acids chiefly caprylic, capric and lauric acids present in oil or fat.

27.2 PRINCIPLE

The material is saponified by heating with glycerol and sodium hydroxide solution and then split by treatment with dilute sulphuric acid. The volatile acids are immediately steam distilled. The soluble volatile acids in the distillate are filtered out and estimated by titration with standard sodium hydroxide solution. The condenser, the 25 ml cylinder and the receiver used in the Reichert-Meissl value determination are washed into the filter paper through which the distillate was filtered for that determination. After rinsing, the residue on the filter paper is taken up with ethyl alcohol and titrated with standard sodium hydroxide solution.
27.3 REQUIREMENTS

Apparatus

Flat-Bottom Boiling Flask- The flask (A) shall be made of resistance glass.

Still-Head - The still-head (B) shall be made of glass tubing of wall thickness $1.25 \pm 0.25$ 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 center of the side-hole of the still-head.

a) Condenser- The condenser (C) shall be made of glass.

b) Receiver - The receiver (D) shall be a flask, with two graduation marks on the neck.

c) Asbestos Board - An asbestos board (E), 120 mm diameter, 6 mm in thickness, with a circular hole about 65 mm in diameter, shall be used to support the flask over the burner.

d) Bunsen Burner

Reagents

Glycerine

Conc. NaOH Solution : 50 % (w/w). Dissolve NaOH in an equal weight of water and store the solution in a bottle protected from carbon dioxide. Use the clear portion free from deposit.

Pumice Stone Grains : 1.4 to 2.0 mm in diameter.

Dilute $H_2SO_4$ Solution : 1 N.

Standard NaOH solution : 0.1 N.

Phenolphthalein Indicator : Dissolve 0.1 g of phenolphthalein in 100 ml of 60 % rectified spirit.

Ethyl Alcohol : 90%, v/v neutral to phenolphthalein.
27.4 PROCEDURE

**RM value**

Weigh accurately 5.00 ± 0.01 g of the filtered oil or fat into the boiling flask. Add 20 g of glycerol and 2 ml of conc. NaOH solution from a burette to which access of carbon dioxide is prevented and whose orifice is wetted before running in the liquid, the first few drops from the burette being rejected. Heat the flask and its contents with continuous shaking on a gauze over the naked flame until the fat, including any drops adhering to the upper parts of the flask, has been saponified and the liquid becomes perfectly clear. Avoid overheating during this saponification. Cover the flask with a watch glass, and allow the flask to cool a little. Add 90 ml of boiling distilled water, which has been vigorously boiled for about 15 min. After thorough mixing, the solution should remain clear. If the solution is not clear (indicating incomplete saponification) or is darker than light yellow (indicating overheating), repeat the saponification with a fresh sample of the oil or fat. If the sample is old, the solution may sometimes be dark and not clear.

Add 0.6 to 0.7 g of pumice stone grains and 50 ml of dilute sulphuric acid, and immediately connect the flask with the distilling apparatus. Place the flask on the asbestos board. After the fatty acids have melted and separated into a clear liquid layer on gentle warming, heat the flask without altering the flame so that 110 ml of liquid distils over in the course of 19-21 min. The distillation is considered to begin when the first drop forms in the still head. Keep the water flowing in the condenser at a sufficient speed to maintain the temperature of the outgoing water from the condenser between 15°C and 20°C. Collect the distillate in a graduated flask.

As soon as 110 ml have distilled over, stop heating the boiling flask and replace the graduated flask by a measuring cylinder of about 25 ml capacity to catch washings. Close the graduated flask with the stopper, and, without making the contents, place it in a water-bath at 15°C for 10 min, making sure that the 110 ml graduation mark is below the level of the water. Swirl round the contents of the flask from time to time. Dry the outside of the flask and then mix the distillate by closing the flask and inverting it four or five times, but do not shake. Filter through a dry Whatman No. 4 filter paper. Reject the first 2-3 ml of the filtrate and collect the rest in a dry flask.

Pipette 100 ml of the filtrate in a titration flask, add 0.1 ml of phenolphthalein indicator solution and titrate with standard 0.1N NaOH solution until the liquid becomes slightly pink. Run a blank test without the fat but using the same quantities of reagents and following the same procedure.

**Polenske value**

After determining the Reichert-Meissl value, detach the still-head and wash the condenser with three successive 15 ml portions of cold distilled water, passing each washing separately through the measuring cylinder, the 110 ml flask with stopper, and the filter paper nearly filling the paper each time to the brim and draining each washing before filtering the next. The last 10 ml of wash-water should require not more than one drop of 0.1N sodium hydroxide solution for neutralization. Discard all the washings. Dissolve the insoluble acids by three similar washings of the condenser, the measuring cylinder, the 110 ml flask with stopper, and the filter paper with 15 ml portions of ethyl alcohol. Combine the alcoholic washings in a clean flask (total volume thus amounting to 45 ml), add 0.25 ml of phenolphthalein indicator solution, and titrate with standard 0.1N NaOH solution until the solution turns slightly pink.
27.5 CALCULATION

Reichert-Meissl value = \((A - B) \times N \times 11\)

Polenske value = \(10 \times V \times N\)

where,

- \(A\) = volume, in ml, of NaOH solution required for the test,
- \(B\) = volume, in ml, of NaOH solution required for blank, and
- \(N\) = normality of NaOH solution.

27.6 RESULTS AND INFEERENCE

The mean of duplicate results should be reported. The difference between the results of two determinations carried out simultaneously or in rapid succession by the same analyst (repeatability) shall not exceed 0.5 in case of RM value and 0.1 in case of PV. The RM value and Polenske value of oils and fat is given as follows:

<table>
<thead>
<tr>
<th>Oils/fats</th>
<th>RM Value</th>
<th>Polenske Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ghee</td>
<td>22-34</td>
<td>2-4</td>
</tr>
<tr>
<td>Coconut oil</td>
<td>6-8</td>
<td>14-18</td>
</tr>
<tr>
<td>Palm kernel oil</td>
<td>5-7</td>
<td>10-12</td>
</tr>
<tr>
<td>Other oils/fats</td>
<td>&lt;1</td>
<td>&lt;1</td>
</tr>
</tbody>
</table>

27.7 PRECAUTIONS

- If the strength of acid and alkali is not appropriate, errors creep in. If the acid is weak with respect to alkali then the values shall be lower.
- The time of distillation (19-21 min) is also important. If more time is given to distil 110 ml of the distillate, the resulting RM value shall be lower and PV higher and reverse is vice versa.
- Accurate weighing of 5 g sample is necessary.
- During the heating with NaOH, avoid overheating which will be indicated by the darkening of the solution.
- Bumping is often observed at the time of heating to acidify soaps to release fatty acids completely. Often it happens during distillation, also breaking the flask violently and splashing the corrosive liquid. This is caused by the crust of thick soap working as a pressure lid for a while. Care must be taken, to liquify the crust before the contents are exposed to heating by warming the flask from the sides and slowly extending the heat to the bottom part of the flask.
- The filtrate should be free from insoluble fatty acids. When operating on coconut oil, it has been noticed that this is not easily achieved. In cases where liquid insoluble fatty acids pass through the filter, transfer the filtrate to a separating funnel and after the separation of the lower aqueous layer, add the insoluble acids to the main bulk of insoluble acids.