EXPERIMENT 23  DETERMINATION OF IODINE VALUE IN OILS AND FATS

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23.0 OBJECTIVES

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

- learn to perform determination of iodine value in oils and fats.

23.1 INTRODUCTION

The glycerides of the unsaturated fatty acids unite with a definite amount of iodine and the iodine value is therefore a measure of the degree of unsaturation.

23.2 PRINCIPLE

The material is treated, in carbon tetrachloride medium, with a known excess of iodine monochloride solution in glacial acetic acid (Wijs solution). The excess of iodine monochloride is treated with potassium iodide and the liberated iodine estimated by titration with sodium thiosulphate solution.

23.3 REQUIREMENTS

Reagents

Potassium Dichromate.
Concentrated Hydrochloric Acid.
Potassium Iodide Solution - Prepare a fresh solution by dissolving 10 g of KI free from potassium iodate, in 90 ml of water.

Starch Solution - Triturate 5 g of starch and 0.01 g of mercuric iodide with 30 ml of cold water and slowly pour it with stirring into one litre of boiling water. Boil for three minutes. Allow to cool and decant off the supernatant clear liquid.

Standard Sodium Thiosulphate Solution (0.1N).
Glacial acetic Acid.

Iodine Monochloride (ICl) - 98 %.

Wijs Iodine Monochloride Solution

Dissolve 10 ml of iodine monochloride in about 1800 ml of glacial acetic acid (chemically pure) and shake vigorously. Pipette 5 ml of this, add 10 ml of KI solution and titrate with 0.1 N standard Na$_2$S$_2$O$_3$ solution, using starch solution as indicator. Adjust the volume of the solution till it is approximately 0.2 N.

Carbon Tetrachloride or Chloroform - inert to Wijs solution.

23.4 PROCEDURE

Melt the sample if it is not already completely liquid, and filter through a filter paper to remove any impurities and the last traces of moisture. Make sure that the sample as well as the glass apparatus used is absolutely clean and dry. Weigh accurately, by difference, an appropriate quantity of the oil or fat, into a clean dry 500 ml iodine flask or well ground glass-stoppered bottle to which 25 ml of carbon tetrachloride have been added and agitate to dissolve the contents. Add 25 ml of Wijs solution and replace the glass stopper after wetting with KI solution; swirl for intimate mixing, and allow to stand in the dark for 30 min. in the case of non-drying and semi-drying oils and 1 hr. in the case of drying oils. Carry out a blank test simultaneously under similar experimental conditions. After standing, add 15 ml of KI solution and 100 ml of water, rinsing in the stopper also, and titrate the liberated iodine with standard Na$_2$S$_2$O$_3$ solution, swirling the contents of the bottle continuously to avoid any local excess until the colour of the solution is straw yellow. Add 1 ml of the starch solution and continue the titration until the blue colour formed disappears after thorough shaking with the stopper on.

23.5 CALCULATION

\[
\text{Iodine value} = \frac{12.69 \times (B-S) \times N}{W}
\]

Where,

\(B\) = Volume, in ml, of Na$_2$S$_2$O$_3$ solution required for the blank,

\(S\) = volume, in ml, of Na$_2$S$_2$O$_3$ solution required for the sample,

\(N\) = normality of Na$_2$S$_2$O$_3$ solution, and

\(W\) = weight, in g, of the material taken for the test.

23.6 RESULTS AND INFERENCE

The mean of the results of two determinations 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. The range of iodine value for animal fats (30-70), non-drying oils (80-110), semi-drying oils (80-140) and drying oils (125-200) and very small value for waxes. The iodine value of commonly used edible oils is given as below.
<table>
<thead>
<tr>
<th>Type of Oil</th>
<th>Iodine Value</th>
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</tr>
</thead>
<tbody>
<tr>
<td>Coconut oil</td>
<td>7.5-10.5</td>
<td>Safflower oil</td>
<td>138-146</td>
</tr>
<tr>
<td>Cottonseed oil</td>
<td>98-115</td>
<td>Sunflower oil</td>
<td>100-140</td>
</tr>
<tr>
<td>Groundnut oil</td>
<td>87-98</td>
<td>Soybean oil</td>
<td>125-140</td>
</tr>
<tr>
<td>Mustard oil</td>
<td>98-110</td>
<td>Rice bran oil</td>
<td>90-105</td>
</tr>
<tr>
<td>Sesame oil</td>
<td>103-115</td>
<td>Palm oil</td>
<td>44-58</td>
</tr>
</tbody>
</table>

### 23.7 PRECAUTIONS

- As soon as Wij’s solution is added stopper the flask immediately.
- Exactly for 30 minutes the flask should be kept in dark.
- Exact and accurate weighing should be done according to the iodine value expected in the sample.
- If B-S is greater than B/2, the test must be repeated using a lesser quantity of the sample.