EXPERIMENT-5

To perform the estimation of Acid value of supplied oil.

References:

1. Hand Book of Analysis and Quality Control for Fruit and Vegetable Products, second edition by S. Ranganna, Published by Publishing Company limited, New Delhi, Page 224.
2. Indian Pharmacopoea, Part I, Page 84.

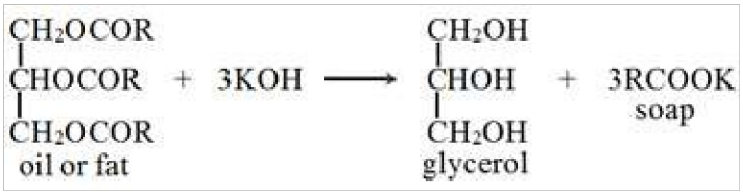
Requirements:

Chemical: Ethanol, Ether, 0.1 M potassium hydroxide, Phenolphthalein, etc.

Apparatus: Volumetric flask, Pipette, Burette, etc.

Theory:

The acid value is the number which expresses in milligrams the amount of potassium hydroxide required to neutralize the free acids present in 1 g of the substance. Here direct titration is done by taking 'n' volumes neutralized of ethanol (95%) and ether as solvent and phenolphthalein as indicator.



Rancidity: On long exposure to air and moisture, fats and oils develops a disagreeable smell and taste. This is termed as rancidity.

Acid value is useful for determining the freshness, adultration, rancidity, purity of oil/fat

Procedure:

1. About 10 g (w) of the sample is dissolved, in 50 ml of a mixture of equal volume of 95% Ethanol and Ether, previously neutralized with 0.1M Potassium hydroxide to Phenolphthalein solution.
2. If the sample does not dissolve in the cold solvent, the flask is connected with a reflux condenser and slowly warmed shaking, until the sample dissolves.
3. 1 ml of phenolphthalein solution is added and titrated with 0.1 M potassium hydroxide until the solution remains faintly pink after shaking for 30 seconds.
4. The titration was repeated thrice to get three concordant readings and the volumes of Alkali consumed were recorded in the table.

Observation Table:

Sl. No.	Initial Burette Reading (ml)	Final Burette Reading (ml)	Difference (ml)	Titer Value (Average in ml)
1				
2				
3				

Calculation

$$\text{Acid value (mg KOH/g)} = \frac{\text{titer value} \times \text{N of KOH} \times 56.1}{\text{wt. of sample (g)}}$$

Where, N = the Normality of KOH; 56.1 = Equivalent weight of KOH

Result:

The acid value of the supplied plant oil was found to be _____

Uses:

The significance of the acid value

1. The acidity of oil is due to hydrolysis or oxidation of oil by atmospheric moisture leading to the formation of fatty acids.
2. Lubricants oil with acid values greater than 0.1 corrode metals, form gum and sludge during operation.

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EXPERIMENT-6

To perform the estimation of Saponication value of supplied oil..

References:

1. Advanced Practical Organic Chemistry by O.P.Agrawal. Published by Krishan Prakashan Media (P) Ltd. Page-275.
2. Indian Pharmacopoea, Part I, Page 93.

Requirements:

Chemical: Ethanol, Ether, 0.1 M potassium hydroxide, Phenolphthalein, etc.

Apparatus: Volumetric flask, Pipette, Burette, etc.

Theory:

Saponification value is the number of milligram of potassium hydroxide required to neutralize the fatty acid resulting from the complete hydrolysis of 1g of oil or fat. It is determined by boiling a weighed amount of substance with a measured volume of standard alcoholic KOH and back titrating with 0.5M HCl. Saponification value is a measure of the size of the fat molecule or the size or molecular weight of fatty acids in the fat. It also indicates the quantity of alkali which must be used to convert a blend of fats to soap. Saponification value is also useful for detecting adulteration of a given fat by one of the higher or lower Saponification value. Some average accepted saponification values for common oils:

Olive oil =185-196 Linseed oil =192-195

Coconut oil= 246-260 Palm oil= 242-250

Procedure:

Standardization of 0.5N HCl:

1. Weigh accurately about 0.75g anhydrous sodium carbonate, previously heated at about 2700c for 1 hour.
2. Dissolve it in 100ml of water and add 0.1ml of methyl red solution.
3. Add the acid slowly from the burette with constant stirring, until the solution becomes faintly pink.
4. Heat the solution to boiling, cool and continue the titration.
5. Heat again to boiling and titrate further as necessary until the faint pink colour is no longer affected by continued boiling.

Determination of Saponification value:

1. Weigh accurately about 2g of sample into a clean 250ml round bottom flask.

2. To this add 25ml alcoholic potassium hydroxide and reflux for 30minutes. Cool and add 1ml phenolphthalein indicator.

3. Immediately titrate with 0.5N HCl. End point is the appearance of white colour. This titre value can be considered as 'A'.

4. Repeat the titration for a blank determination, without taking the sample being examined. The titre value obtained can be considered as 'B'.

5. Calculate the Saponification value of supplied oil by using the formula.

Observation Table for Sample:

Sl. No.	Initial Burette Reading (ml)	Final Burette Reading (ml)	Difference (ml)	Titer Value A (Average in ml)
1				
2				
3				

Observation Table for Blank:

Sl. No.	Initial Burette Reading (ml)	Final Burette Reading (ml)	Difference (ml)	Titer Value (Average in ml)
1				
2				
3				

Calculation

Saponification Value = [(B - A) x N x 56.1]/ W

Where

A = Titer Value of Acid for sample in mL B = Titer Value of Acid for Blank in mL

W = Weight of sample (dry basis) in g N = Normality Acid solution

56.1 = Equivalent weight of potassium hydroxide

Result:

The Saponification value of the supplied plant oil was found to be _____.

Significance:

It helps us determining the stability of the oil during processing and storage and also suggests the molecular weight and chain length of fatty acid in fat or oil. A smaller Saponifocation value indicates a longer chain fatty acid and vice versa.

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Experiment-7

To determine the Iodine value of the given sample of oil.

Reference:

Practical pharmaceutical chemistry IVth edition; Part I by A. H Beckett, J. B Stenlake; Page no 158.

Requirements:

Chemical/ Reagents: Oil sample, carbon tetra chloride, iodine monochloride solution, potassium iodide solution, 0.1 M sodium thiosulphate, starch solution, etc.

General/Glassware: Weight box, weighing bottle, measuring cylinder, burette, conical flask, pipette, round bottom flask, condenser, etc.

Theory:

The iodine value is the number which expresses in grams the quantity of halogen, calculated as iodine, which is absorbed by 100g of substance under the described conditions. It may be determined by three methods.

1. Iodine monochloride method or Wijs method.

2. Iodine monobromide method or Hanus method

3. Pyridine monobromide method

Iodine value gives indication of proportion of unsaturated acid present in fat or oils. If the fatty acid is more unsaturated it will give a high iodine value. Solid fat have low iodine value. Non drying oils have iodine value of 80-100; whereas drying oil have high iodine value.

Iodine value of some oils;

Castor oil -82-90

Olive oil- 79-88

Cod liver oil- 145-180

Linseed oil 175-200

Iodine Monochloride Solution, Wijs Reagent is prepared by dissolving 26.0 g of reagent grade iodine (I2) in 2 L of reagent grade glacial acetic acid, heating gently if necessary to hasten solution. Cool to room temperature.

The Hanus iodobromide solution is made by dissolving 13 2 g of iodine in 1 litre of glacial acetic acid and then, to the cold solution, a quantity of bromine is added equivalent to that of the iodine present the iodine present is determined by titration.

Standardization of Iodine solution:

Pipet 25 mL into a 250 mL Erlenmeyer flask. Add 100 mL purified water and titrate immediately with standard 0.1 N thiosulfate solution until the yellow iodine color almost disappears. Add 1 mL of starch indicator solution and complete the titration dropwise with vigorous swirling to the disappearance of the blue starch iodine complex. This is the original titer. Remove 100 mL of the iodine solution (solution A), and store in a glass-stoppered bottle, for adjustment of the halogen ratio if necessary. Dry chlorine gas from a cylinder by passing through a gas-washing bottle charged with concentrated sulfuric acid, and bubble the dried gas through a gas dispersion tube into the bulk of the iodine solution. Pass chlorine into the solution until the original titer is not quite doubled. Check titer of reagent after chlorine addition by pipetting a 25.0 mL aliquot into 250 mL Erlenmeyer flask. Add 100 mL of distilled water, 10 mL of 30% potassium iodide solution, and titrate with standard 0.1 N thiosulfate solution as

Procedure:

Standardisation of 0.1 M sodium thiosulphate: Dissolve accurately weighed potassium dichromate in 25 ml of water in a 25l of erlenmayer flask.Add 10 ml of hydrochloric acid and 2 g of potassium iodide, stopper, shake and keep dark for 15 minutes. Add 100ml of water to the above mixture and titrate with sodium thiosulphate u sing starch as indicator. Near end point the color will be changed from dark blue to bottle green. Each ml of 0.1 M sodium thiosulphate is equivalent to 0.04904g of potassium dichromate.

Iodine monochloride method or Wijs method: Place an accurately weighed quantity of the substance in a 500ml iodine flask, add 10 ml carbon tetra chloride and dissolve.

Add 20 ml iodine monochloride solution, insert the stopper and allow to stand in the dark at a temperature between 150- 250 for 30 minutes. Place 15 ml potassium iodide solution in the cup top, carefully remove the stopper, rinse the stopper and sides of the flask with 100 ml of water, shake and titrate with 0.1 M sodium thiosulphate using starch solution, added towards the end of the titration, as indicator. Note the no. of ml required(a). Repeat the operation without the substance under examination and note the no of ml required (b). Calculate the iodine value from the expression Iodine value = 1.269(b-a)/w

Where w= weight in gm of the substance.

REPORT

The iodine value of the given sample was found to be _____

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