**ANALYSIS OF OILS- IODINE NUMBER- SAPONIFICATION VALUE -ACID NUMBER**

**IODINE VALUE OF OIL**

**PRINCIPLE:**

The oils are rich in unsaturated fatty acids. Hence iodine adds at the double bonds of unsaturated fatty acids when iodine monochloride is allowed to react with the oil. The amount of iodine consumed is determined by titrating the iodine released (after adding Kl) with standard thiosulphate. This amount of iodine is expressed as iodine value which is defined as grams of iodine taken up by 100 g oil.

ICI + Kl---> HCI + I2

I2+2Na2S2O3---> 2NaI+ Na2S4O6

Iodine value is thus, a measure of degree of unsaturation in an oil. It is used in determining oxidative rancidity of oils since higher the iodine value (i.e. unsaturation)/greater the

possibility of the oils to go rancid.

**MATERIALS REQUIRED:**

I Given oil sample.

ii Chloroform.

iii Iodine monochloride (0.2 M) -Dissolve 2.25 g iodine monochloride in dH20 and make up

the volume to I 00 mi.

iv Potassium iodide (10%) -Dissolve 10 g KI in 100 ml distilled water (dH20).

v Sodium thiosulphate (0.1 M) -Dissolve 24.819 g of sodium thiosulphate in distilled water and make up the volume to 1 lit.

vi Starch solution (1%}) dissolve 1 g starch in 100 ml dH20.

vii Stoppered flasks (250 ml}, burette, pipettes etc.

**PROCEDURE:**

1. Weight 0.5 g of the given oil sample and dissolve in 10 ml chloroform in a flask. In another flask take only 10 ml chloroform which would act as blank. Proceed with both the flasks as under.
2. Add 25 ml of iodine chloride solution using measuring cylinder, mix well and allow to stand in dark for 30 min with occasional shaking.
3. Rinse the stoppers and sides of the flask with -50 ml water washing down any free iodine.
4. Add 10 ml of potassium iodide solution and titrate the liberated iodine with the standard thiosulphate solution until yellow solution turns almost colourless.
5. Add 1 ml of starch solution and continue titrating until blue colour completely disappears. The flask must be shaken vigorously throughout the titration so that iodine remaining in chloroform is removed and taken up by potassium iodide solution.

**RESULT:**

Iodine value of the given sample is =

**OBSERVATIONS AND CALCULATION:**

Titration II

Determination of iodine number

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| S.No | Volume of Sample (ml) | Burette Readings | | Volume of tiosulphate (ml) | Concordant volume (ml) | Indicator |
|  |  | Initial (ml) | Final (ml) |  |  | Iodine |
|  |  |  |  |  |  |  |

Volume of thiosulphate used for blank = B ml

Volume of thiosulphate used for sample = **S** ml

Hence,

Thiosulphate equivalent of iodine

solution, used by the sample = B-S ml

Milligram of iodine used

by the sample = Volume (ml) x Molarity x Atomic wt. of iodine

= (B-5) X 0.1 X 127 mg

Quantity of iodine (in grams) used by 100 g sample

(i.e. iodine value) = (B-5) X 0.1 X 127 x 100g  
 0.5 X 1000

(B-S) X 127 g

50

= (B-5) X 2.54 g

**SAPONIFICATION VALUE**

**AIM:**

To determine the saponification number of edible oil.

**PRINCIPLE:**

The saponification number of oil is defined as the number of milligrams of potassium hydroxide required to saponify 1 gram of oil. During saponification, triglycerides are converted to glycerol and potassium salt of fatty acid using a known excess of alkali to saponify the known quantity of oil. The excess unused alkali is determined by titration. The saponification number gives an idea of the average molecular weight of the fat. High saponification number indicates low molecular weight and vice versa. The saponification value indicates the quantity of alkali, which must be used to convert fat to soap. The main use of saponification number is in detecting adulteration of a given fat by one of the higher (or) lower saponification value.

**REAGENTS:**

Ethanolic potassium hydroxide

To about 500 ml of ethanol (or) alcohol, 14 grams of potassium hydroxide is added and dissolved to give a solution of 0.5 N potassium hydroxide in ethanol. The solution is prepared fresh.

Potassium hydroxide solution

Approximately, 0.5N solution is prepared by dissolving 2.8 g of potassium hydroxide in 100 ml of ethanol.

Oxalic acid solution 3.15 gms of oxalic acid is dissolved and made upto 100 ml with distilled water (0.5 N).

Phenolphthalein indicator

**PROCEDURE:**

**STANDARDIZATION OF ALCOHOLIC POTASSIUM HYDROXIDE**

The burette is filled with oxalic acid. 15 ml of ethanolic potassium hydroxide is pipetted out into a clean conical flask and a drop of phenolphthalein is added. The solution is titrated against oxalic acid till the pink colour disappears. Titrations are repeated for concordant values form this, the strength of potassium hydroxide is determined.

About 1 gm of oil is weighed and transferred into 100 ml clean conical flask. 15 ml of alcoholic potassium hydroxide solution is added, mixed and air condenser is fixed. Another 100 ml conical flask with 15 ml of alcoholic potassium hydroxide is fixed with an air condenser. This serves as a blank. Both the flasks are refluxed for 30 minuts in a water bath which is in boiling condition. Then the condenser is removed and rinsed with water into the flask. The flaks is cooled and a drop of phenolphthalein is added. The solution is titrated against standard oxalic acid. The end point is then disappearance of the pink colour. From the titre value, saponification value of an oil is calculated.

Titration I

Standardisation of Alcoholic potassium hydroxide

Oxalic acid Vs Alcoholic potassium hydroxide

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| S.No | Volume of Alcoholic potassium hydroxide (ml) | Burette Readings | | Volume of Oxalic acid (ml) | Concordant volume (ml) | Indicator |
|  |  | Initial (ml) | Final (ml) |  |  |  |
|  |  |  |  |  |  |  |

Titration II

Determination of saponification number

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| S.No | Volume of Sample (ml) | Burette Readings | | Volume of Oxalic acid (ml) | Concordant volume (ml) | Indicator |
|  |  | Initial (ml) | Final (ml) |  |  |  |
|  |  |  |  |  |  |  |

**Result:** The saponification number of oil is \_\_\_\_\_

**ACID NUMBER OF EDIBLE OIL**

**AIM:**

To determine the acid number of edible oil.

**PRINCIPLE:**

The acid number of edible oil is defined as the number of milligrams of potassium hydroxide required to neutralize free fatty acids present in 1gm of fat (or) oil. During storage, fats and oils may be hydrolyzed by microorganisms with the formation of free fatty acids. The amount of free fatty acids gives the indication of age and quality of the oil.

**REAGENTS:**

*Oxalic acid solution*

630 mg of oxalic acid is weighed, dissolved and made upto 100 ml with distilled water (0.1 N).

*Potassium hydroxide solution*

5.6 gms of potassium hydroxide is dissolved in 1 lit of distilled water.

Ethanol, Phenolphthalein indicator

**PROCEDURE:**

**STANDARDIZATION OF POTASSIUM HYDROXIDE:**

The burette is filled with potassium hydroxide solution. 10 ml of oxalic acid is pipetted out into a clean conical flask and is titrated against potassium hydroxide taken in the burette with phenolphthalein as an indicator. The end point is the appearance of permanent pale pink colour. Titrations are repeated for concordant values. From the titre value, the normality of potassium hydroxide is calculated.

**DETERMINATION OF ACID NUMBER:**

1 gm of oil is weighed accurately and transferred into a clean conical flask. 15 ml of ethanol is added to dissolve the oil and the contents are shaken well. 15 ml of ethanol is alone taken in another conical flask, which served as a blank. Few drops of phenolphthalein is added in both the flasks. The solutions are titrated against standardized potassium hydroxide taken in the burette. The end point is the appearance of (permanent) pale pink colour, which persists for 10 to 20 seconds, the difference between test and blank values given the volume of potassium hydroxide consumed from this, acid number of oil is determined.

Titration I

Standardisation of Potassium hydroxide

Potassium hydroxide Vs Oxalic acid

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| S.No | Volume of Oxalic acid (ml) | Burette Readings | | Volume of Potassium hydroxide(ml) | Concordant volume (ml) | Indicator |
|  |  | Initial (ml) | Final (ml) |  |  |  |
|  |  |  |  |  |  |  |

Titration II

Determination of Acid number

Potassium hydroxide Vs Unknown

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| S.No | Volume of Unknown (ml) | Burette Readings | | Volume of Potassium hydroxide(ml) | Concordant volume (ml) | Indicator |
|  |  | Initial (ml) | Final (ml) |  |  |  |
|  | 1g oil + 15ml ethanol |  |  |  |  |  |

Titration III

Titration of blank

Potassium hydroxide Vs ethanol

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| S.No | Volume of ethanol (ml) | Burette Readings | | Volume of Potassium hydroxide(ml) | Concordant volume (ml) | Indicator |
|  |  | Initial (ml) | Final (ml) |  |  |  |
|  |  |  |  |  |  |  |

**Result:**

The acid number of given oil is \_\_\_\_\_\_\_.