LAB EXPERIMENT 9

Determination of Iodine number.

Introduction:

Current medical thinking suggests that a higher proportion of unsaturated fats in the diet are beneficial. Unsaturated fats tend to spoil (become rancid) more readily than saturated ones which presents a storage problem. The amount of unsaturation in fat samples is therefore often determined industrially and is termed the iodine number of the fat.

The iodine number is defined as the percentage of iodine absorbed by a fat or wax, or the number of grams of iodine absorbed per 100 g of fat or wax. It is a measure of the unsaturated bonds present in the fat under investigation, i.e.: a low iodine number indicates few unsaturated bonds.

Principle:

In the following procedure a known amount of bromine solution is added to a fixed weight of oil.

$$\boxed{-HC{=}CH- \; + \; 2\,Br_2 \xrightarrow{\text{Halogenation}} -\underset{\substack{|\\ Br}}{CH}-\underset{\substack{|\\ Br}}{CH}- \; + \; Br_2}$$

The excess bromine not taken up by the unsaturated bonds displaces an equimolar quantity of iodine from potassium iodide,to liberate iodine.

$$\boxed{Br_2\ (\text{excess}) \; + \; 2\,KI \longrightarrow 2\,KBr \; + \; I_2\ (\text{Liberated})}$$

The iodine so liberated is then titrated with standardized sodium thiosulphate.

$$I_2 + 2\,Na_2S_2O_3 \longrightarrow 2\,KBr + 2\,NaI$$

The reaction mixture is kept in the dark and the titration is carried out quickly as possible since halogens are oxidized in the light.

N.B.

- The sample titration **(T)** gives a measure of the amount of (iodine) remaining in solution after halogenation.
- The titration of a blank **(B)** gives a measure of the total amount of halogenating reagent present initially.
- A subtraction of the values **(B)** from **(T)** indicates the quantity of reagent utilized in the halogenation of a 5 ml sample of all from an original 27 ml volume.

Reagents:

1- The halogenation reagent:- Pyridine sulphate dibromide in glacial acetic acid is made as follows:
8 ml of pyridine and 5.5 ml concentrated sulphuric acid are separately added to 20 ml of glacial acetic acid. A 2.5 ml aliquot of bromine is then added to another 20 ml portion of the volume made up to 1 litre with glacial acetic acid giving an approximately 0.1 M solution of Bromine.

2- 10% aqueous solution of potassium iodide.
3- 1% aqueous solution of starch.
4- 0.1 mol/l standard thiosulphate solution.
6- various fats to be assigned.

Procedure:

1- To 2.0 ml of the fat, add 25 ml of chloroform to dissolve the material.

2- Pipette 5 ml sample of this solution into two separate Erlenmeyer flasks. To a third flask add 5 ml of chloroform to form a blank.

3- Add 10 ml of halogenation reagent to each flask.Cover the flasks with aluminum foil,shake and allow to stand for 15 minutes.

4- Add 7.5 ml of potassium iodide KI solution to each flask and titrate the liberated iodine using the thiosulphate solution.

5-Titrate the test solution until a light brown colour is obtained then add 5 drops of starch indicator solution immediately and titrate to a colourless end point.

7- Record the **total volume** of the thiosulphate required to reach the end point.

Calculations:

The difference **C** between the blank thiosulphate titer **B** and the sample titer **T** is the thiosulphate equivalent of iodine which combined with the fat. This titer volume is used in calculations.

(I)

1ml (0.1N) = 0.0127 g I_2
C ml = Y g I_2 reacted with the unsaturated bonds in the oil used

$$Y = \frac{C \times 0.0127}{1} = \quad g\ I_2$$

(II)

27ml of oil solution contain = 2ml oil
5ml = X ml oil

$$X = \frac{5 \times 2}{27} = 0.37 \text{ ml oil in 27 ml}$$

Meaning that the 5ml of the test sample contain 0.37 ml oil. and to convert this amount from milliliters to grams this formula is used:
W = V x d
Where,
W weight in gm
V volume in milliliters
D density of oil = 0.39 g/ml

By substitution, W = 0.3441 g oil

(III)

Since, 0.3441 g oil = Y g I_2
100 g oil = Z

$$Z = 100 \times \frac{Y}{0.3441} = \qquad \% \text{ the iodine number}$$

Typical iodine numbers for some fats and oils:

Fat or Oil	Iodine No
Coconut oil	**8-10**
Butter	25-40
Beef tallow	30-45
Palm oil	37-54
Lard	45-70
Oliver oil	75-95
Peanut oil	85-100
Cottonseed oil	100-117
Corn oil	115-130
Fish oils	**120-180**
Canola oil	125-135
Soybean oil	125-140
Safflower oil	130-140
Sunflower oil	130-145

Name: ***No.***

Experiment 9:

Results Sheet

1-Calculate the iodine number of your oil sample.
2-Compare your results to those in the previous table.