B.Sc. Third Year V Semester

ANALYTICAL CHEMISTRY

Paper XIII [Applied Analytical Chemistry-I]

Analysis of Food & Food Products

1. Analysis of Milk

Dr. Subhash M. Lonkar

Professor & Head

Department of Chemistry & Analytical chemistry,
M.S.P. Mandal's
Shri Shivaji College, Parbhani, [MS]431401

Cell : +919421864138, +918999890115

Email: drsubhashlonkar@gmail.com

4. Analysis of Honey

2. Analysis of Wheat Flour

3. Analysis of Fish

Analysis of Food & Food Products

Introduction

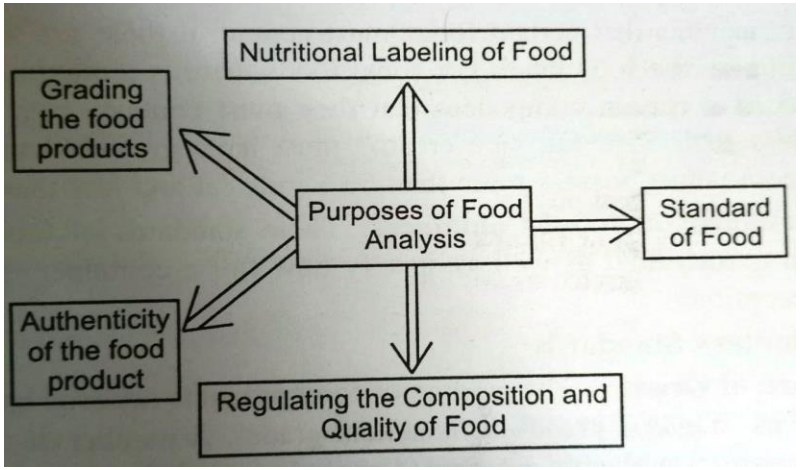
❑ **Food analysis** is the discipline dealing with the development, application and **study** of analytical procedures for characterizing the properties of **foods** and their constituents. ... What types of properties are measured?

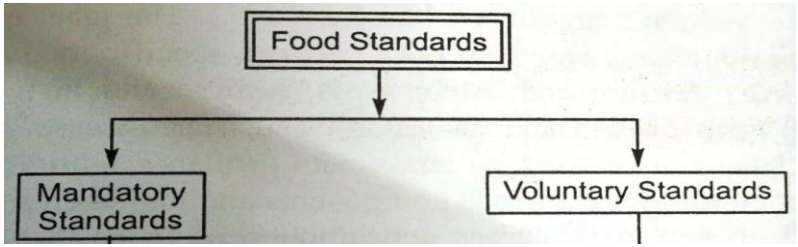
❑ Food analysis is that branch of chemistry which deals with the study of analytical procedures , developments, applications, characterization of the properties of foods and their constituents. These analytical procedures are used to characterize the different properties of foods like their composition, physiochemical properties, structure and sensory attributes. This information provides and idea to consumers about a safe, nutritious, desirable food and also an ability to economically produce foods, suitable for their diet.

Reasons for Analyzing Food

❑ Foods are analyzed by scientists in food industry, research laboratories due to various purposes are as:

❑ Government regulation and recommendations
❑ Standards
❑ Nutritional labeling
❑ Authenticity
❑ Food inspection and grading
❑ Standards: Composition
: Quality
: Inspection
: Labeling of Specific Food products





1. Analysis of Milk

Introduction

❑ **Milk is a colloidal aqueous suspension consisting of many compounds, several of which include**

l. Carbohydrates (sugars)
2. Lipids(fats)
3. Proteins
4. Phosphate
5. And other components

❑ Normal mammary gland secretion of female mammals
❑ It is the first food for the baby mammaline
❑ Freezing point -0^0C (Water) / -0.55^0C (Solids)

Defination of Milk

❑ **Cow milk**, has been defined as the secretion, excluding colostrums, which can be gained by normal milking method from the lactating memory gland of the healthy, normally fed cow.

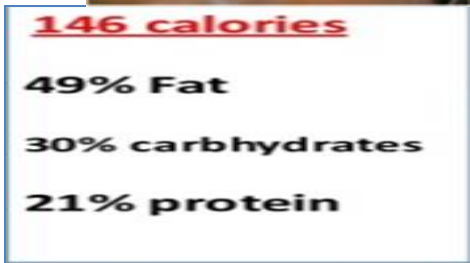
❑ Milk can be considered as containing three basic components, water, fat and non fatty solids (NFS) or solids not fat (SNF).

Cup of Milk

What is Milk?

❑ Milk is normal mammary gland secretion of female mammals or it can be specifically said as the whole, fresh lacteal secretion obtained by the complete milking of one or more healthy cows, excluding that obtained within 15 days before and 5 days after calving, or such longer period as may be necessary to render the milk practically colostrums free.

❑ It consists largely of water, milk fat, lactose or milk sugar, protein and mineral matter. Milk is one of the most important foods in the human diet because It has many components present in very small quantities that are essential to growth and well being.

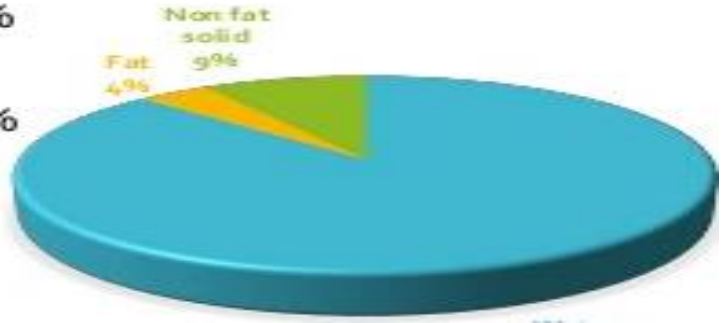


General Composition of Milk

- Water is the principal constituent of milk.
- Milk provides essential nutrients and is an important sources of dietary energy, high quality proteins and fats.
- Milk consist of nutrients like calcium,magnesium,selenium,riboflavin,vitamin B12 and pantothenic acid.
- Milk and milk products are nutrient dense foods and their consumption can add diversity to plant based diets.
- Animal milk can play an important role in the diets of children in populations with very low fat intakes and limited access to other animal source foods.
- The colour,flavour and composition of milk depends on the species of dairy animal, its breed, age and diet, along with the stage of lactation, parity (number of parturition),farming system, physical environment.

What Does Milk Contain?

- Milk primarily consists of water, non fat solid and fat.
- The composition may vary with each breed of the cow.
- Composition may normally contain:
  - Water – 87.3%
  - Non-fat solid – 8.8%
  - Fats – 3.9%*
- *Buffalo milk contain 7.44%
- Cow milk contain 3.66%



% Composition of Milk

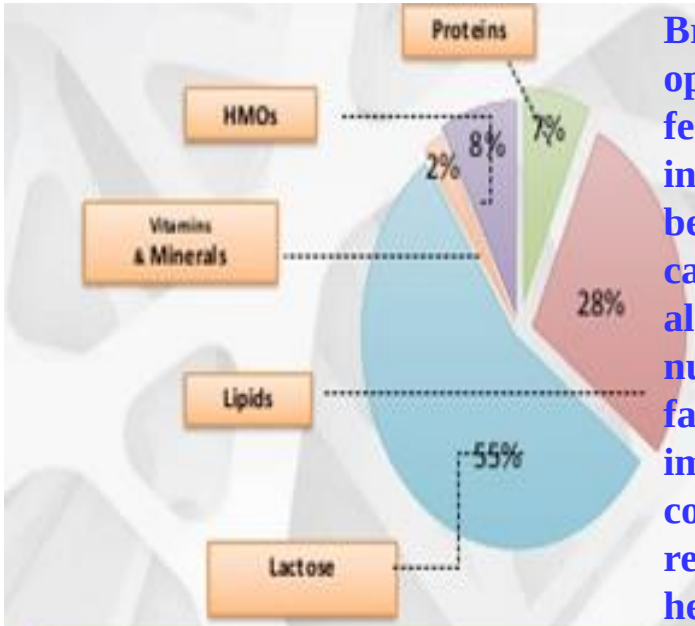
Constituent	Concentration (g/L)	Proportion solids (%)
Fat	37.0	28.9
Protein: casein	27.6	21.6
Protein: whey	6.4	5.0
Non-protein nitrogen	1.9	1.5
Lactose	48.0	37.5
Ash	7.0	5.5
Total solids	127.9	100.0

Milk & Its Components

Milk composition analysis, per 100 grams

Constituents	Unit	Cow	Goat	Sheep	Water buffalo
Water	g	87.8	88.9	83.0	81.1
Protein	g	3.2	3.1	5.4	4.5
Fat	g	3.9	3.5	6.0	8.0
Carbohydrate	g	4.8	4.4	5.1	4.9
Energy	kcal	66	60	95	110
Energy	kJ	275	253	396	463
Sugars (lactose)	g	4.8	4.4	5.1	4.9
Cholesterol	mg	14	10	11	8
Calcium	mg	120	100	170	195
Saturated fatty acids	g	2.4	2.3	3.8	4.2
Monounsaturated fatty acids	g	1.1	0.8	1.5	1.7
Polyunsaturated fatty acids	g	0.1	0.1	0.3	0.2

Composition of Human Milk



Breast feeding is the optimal method of feeding young infants. This is because breast milk can cater to almost all the necessary nutrients. growth factors and immunological components that are required for a healthy term infant

Lactose is the principal sugar of human milk followed by oligosaccharides[2]

If breast milk is not available, the right feed substitute should be used.

Percentage Composition of Milk of the Cow & Other Mammals

Cow milk

Water -87%
Fat – 3-4%
Protein – 3.5%
Lactose - 5%
Minerals -0.8%

Yak milk

Water – 83%
Fat – 5.5 to 9%
Protein – 4 to 5.9%
Solid content – 18%

Buffalo milk

water – 82%
minerals – 0.8%
protein – 4.5%
lactose – 4.8%
fat – 7 to9%
very high fat content

Equine milk(Horse and Donkey milk)

Water – 91%
Fat – 1.9%
Protein – 2.5%(particularly caseins)
Lactose – 6.3% (rich)
Minerals – 0.3%

Goat milk

Water – 87%
Fat – 4%
Protein – 3.6%
Lactose- 4.5%
Minerals – 0.9%

Camel milk

Water – 86.5%
Fat – 4%
Protein – 3.6%
Lactose – 5%
minerals – 0.4%

Sheep milk

Water – 80%
Fat – 8%
Protein – 5.6%
Lactose – 4.8%
Minerals - 0.9%

Human milk

Water – 87.6%
Fat – 4%
Protein – 1.2%
Lactose – 7%
Minerals – 0.4%

Nutrition Milk Provides

Water	→	Hydration
Lactose	→	Carbohydrate
Fat	→	Energy
Protein	→	Builds and repairs muscle
Minerals	→	Calcium for strong bones and teeth

Average Percentage Composition of Milk of the Cow & Other Mammals

	Water	Protein	Fat	Lactose	Ash
Cow	87·2	3·4	3·6	4·9	0·71
Human	87·6	1·2	3·8	7·0	0·21
Ass	89·8	1·9	1·4	6·2	0·45
Buffalo	82·4	4·7	7·4	4·6	0·78
Camel	87·6	3·4	3·0	5·1	0·71
Cat	83·0	7·0	4·5	4·8	0·60
Dog	74·5	3·1	10·2	11·3	0·80
Elephant	85·6	3·2	3·1	7·4	0·63
Ewe	80·6	5·4	8·2	4·7	0·90
Goat	87·8	3·5	3·8	4·1	0·79
Llama	86·5	3·9	3·1	5·6	0·80
Mare	89·8	2·0	1·5	6·1	0·41
Porpoise	41·2	11·2	45·8	1·1	0·57
Rabbit	68·5	12·9	13·6	2·4	2·55
Reindeer	66·1	10·1	19·8	2·5	1·45
Seal	34·0	12·0	54·0	none	0·53
Sow	80·6	6·1	7·6	4·7	0·92
Vixen	81·8	6·3	6·2	4·2	1·31
Whale	69·8	9·4	19·4		0·99
Zebu	86·2	3·0	4·8	5·3	0·70

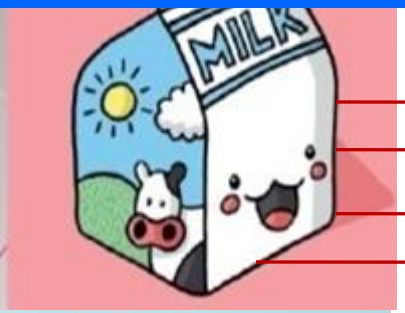
GROSS COMPOSITION OF MILK

Average gross composition of Cow's Milk

Importance of Milk

CHANGES IN MILK COMPOSITION

Analyzing these Contents in Milk



Analysis of Milk

1. **Specific Gravity**
2. **Total Solid**
3. **Acidity**
4. **Fat**
5. **Lactose**
6. **Protein**

Milk is formed of

1) Water: form 87%.

2) Solids: form 13%.
   - Organic constituents of milk.
     1. Protein
     2. Lipid.
     3. Carbohydrate.
   - Inorganic constituents of milk:
     1. Minerals.
     2. Vitamins.

Preparation of Sample

- ❑ Warm the sample to 37·40°C by transferring it to the beaker and keeping it in a water bath.
- ❑ Stirs lowly for proper homogenization.
- ❑ Mix sample thoroughly by pouring back into the bottle, mixing to dislodge any residual fat sticking to the sides and pour it back in the beaker. During mixing do not shake the bottle vigorously.
- ❑ Allow the sample to come to room temperature (26·28°C) and withdraw immediately for analysis.
- ❑ If small clots or lumps are observed in the sample a few drops of liquor ammonia may be used.
- ❑ After homogenization, if the sample shows lumps or clots are visible suggestive of curdling /splitting of milk, the sample should be deemed unfit for analysis and rejected.

1. Determination of Specific Gravity

- **Specific gravity is** also called relative density, ratio of the density of a substance to that of a standard substance. .

$$\text{Specific gravity} = \frac{\text{Density of the object}}{\text{Density of water}} = \frac{\rho_{object}}{\rho_{H_2O}}$$

- Specific gravity, is the ratio between weight of a given volume of milk compared with the same value of water at a specific temperature.
- Normal specific gravity of *cow's milk*: **1020-1030.**
- The **specific gravity of milk** measured at 15°C or 20°C is normally 1.028 - 1,033 kg/ liter.
- The **specific gravity** depends on the protein and fat content. The **specific gravity** of fat is 0.93, solids-non-fat, 1.6 and water 1.0 kg/ liter.

***Material**: Gravity bottle, pycnometer, lactometer, milk, water, chemical balance.*

Method:

- Firstly weigh the empty specific gravity bottle or pyknometer or lactometer & weighed, then filled with milk and weighed again.
- Rinse the gravity bottle with distilled water and again filled with water and weighed.
- Knowing the weight in cases, from this two weights subtract empty weight of specific gravity bottle.
- By knowing weight of water and milk specific gravity calculated as usual method.

Calculation:

$$\text{Specific gravity of milk} = \frac{\text{Wt. of milk}}{\text{Wt. of water}} \times \text{Density of water}$$

2. Determination of Total Solids

❑ **Milk solids** are the non-water components of **milk** – protein , lactose, and minerals. Sometimes the combination of protein, lactose and minerals is called the **solids** not fat content, and when the fat is included it is called **total solids** content.

Material: *Crucible, water bath, oven, milk, chemical balance.*

Method:

❑ Take a weight of empty crucible.
❑ Weigh 5 g of milk in a crucible
❑ Put a crucible in a water bath until dryness.
❑ After complete dryness, put the crucible in an oven, and weigh after cooling.
❑ Determination the percent of total solid.

Calculation:

$$\%\ of\ Total\ Solids = \frac{(Wt.\ of\ crucible + sample)\ after\ drying - Wt.\ of\ crucible}{Wt.\ of\ Sample} X\ 100$$

❑ **Milk** is approximately 87 percent water and 13 percent **solids**. As it comes from the cow, the **solids** portion of **milk** contains approximately 3.7 percent fat and 9 percent **solids**-not-fat. Milk fat carries the fat soluble vitamins A, D, E, and K.

3. Determination of Acidity

❑ **Acidity of Milk** pasteurized, canned, or dry , is an **acid**-forming food. Its pH level is below neutral at about 6.7 to 6.9. This is because it contains lactic **acid**. Remember, though, that the exact pH level is less important than whether it's **acid**-forming or alkaline-forming

❑ The titrable **acidity** test measures the amount of alkali which is required to change the pH of **milk** from its initial value of about 6-6 to 6.8, to the pH of the colour change of phenolphthalein added to **milk** to indicate the end point (pH 8.3)

***Material**: 0.1N NaOH, Phenolphthalein indicator, milk sample.*

Method: Titrable Acidity

10 ml milk + 1 ml phnolpthalein indicator

Titrate with 0.1N NaOH

Color changes, colorless to pink

Calculation:

Acidity = 1 ml 0.1 N NaOH ≈ 0.009 g of lactic acid

4. Determination of Fat

❑ General **Fat Definition** and Chemistry. **Fats** are made from individual fatty acid molecules attached to glycerol, a 3-carbon backbone.

❑ The **fat** content of **milk** is the proportion of **milk**, by weight, made up by butterfat.

❑ The **fat** content, particularly of cow's **milk**, is modified to make a variety of products.

❑ The **fat** content of **milk** is usually stated on the container, and the color of the label or **milk** bottle top varied to enable quick recognition.

❑ Butterfat or milk fat is the **fatty** portion of **milk**. **Milk** and cream are often sold according to the amount of butterfat they contain.

***Material**: Alcohol, ether., centrifuge machine, oven, milk. Conc. HCl.*

Werner Schmidt Method(by Acid Digestion Method):

PRINCIPLE:

- Milk proteins are digested with conc. HCl
- Liberated fat is extracted with alcohol, ethyl ether & petroleum ether
- Ethers are evaporated
- Residue left behind is weighed to calculate the fat content.

Method

10 g milk+10 ml conc.HCl

↓ heat on a Bunsen burner

stir with a glass rod until the contents turn dark brown

↓ cool to room temp

Mojonnier fat extraction flask

↓

10 ml of C_2H_5OH+ 25 ml of ethyl ether

↓ Shake vigorously for 1 min

25 ml of petroleum ether

↓ Shake vigorously for 1 min

Centrifuge Mojonnier flask at about 600 rpm

↓

Decant the ether soln

↓

Repeat extraction

↓

Evaporate the solvent

↓

Dry the fat in oven

↓

Weigh

Calculation:

$$\text{Fat, percent w/w} = \frac{100\,(W1 - W2)}{W3}$$

Where

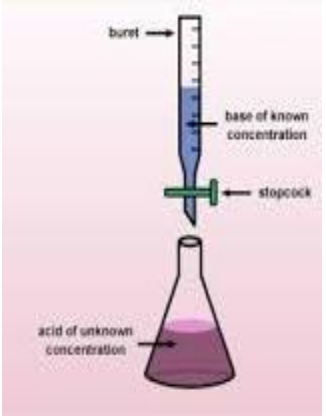
- W1 = Wt in g of contents in the flask before removal of fat.
- W2 = Wt in g of contents in the flask after removal of fat
- W3 = Wt in g of material taken for the test.

5. Determination of Lactose

❑ **Lactose** is a disaccharide sugar that is found in **milk** and is formed from galactose and glucose. **Lactose** makes up around 4.5~5% of **milk** (by weight). The enzyme lactase is essential for digestive hydrolysis of **lactose in milk**. Deficiency of the enzyme causes **lactose** intolerance.

***Material**: N/10 Iodine solution, N/10 NaOH, N/10 sulphuric Acid , N/20 Sodium Thiosulphate*

Method:



Iodimetric method. 50 cc. milk in a litre beaker are diluted with 250 cc. distilled water and treated from a burette with 35 cc. "colloidal iron," at the rate of a few drops a second, mixing the contents constantly by a whirling movement; 165 cc. distilled water are added and the mixture filtered through coarse filter-paper. The filtrate is a 10 % milk-serum.

To 25 cc. of the filtrate in a conical flask are added 25 cc. *N*/10 iodine and 37·5 cc. *N*/10 NaOH. The solution is shaken and after 5 minutes treated with 37·5 cc. *N*/10 H_2SO_4 and titrated with *N*/20 sodium thiosulphate.

25 cc. *N*/10 iodine are titrated with *N*/20 sodium thiosulphate. If the difference between the two titrations = *X* then % lactose in milk-serum = 0·036 *X* (% in milk = 0·36 *X*).

6. Determination of Protein

- **Milk Protein** is a type of **protein** that is derived from filtered **milk** and Is formed from whey and casein **proteins**. The **Milk Protein** yielded is an excellent source of calcium, high in branch chain amino acids and low in fat.
- The standard method for **determining** the percent **protein content in milk** is the Kjeldahl method which, in effect, analyzes total nitrogen. **Protein** nitrogen derived from amino acids represents approximately 95% of nitrogen in **milk**. Non-**protein** nitrogen, such as urea, exists in minor quantities at approximately 5%.

About Protein in Milk

- Proteins are macromolecules that play a crucial role in nutritional growth and development.
- These large biological molecules contain at least one long chain of amino acid residues and are responsible for a multitude of biological functions, including DNA replication and repair, catalyzing metabolic reactions, and providing structure and communication in and between cells. The digestive breakdown of proteins into amino acids provides an important source of fuel and dietary nitrogen.
- Milk naturally contains a number of key nutrients, including protein, which is beneficial to humans regardless of their age.
- Globally, milk is a commonly consumed food product that, due to its high nutrient composition, is regarded as highly beneficial for growth.
- In cow and sheep milk the percent protein can range from 3.3% to 5.8%.

Analyzing Protein Content in Milk with Total Kjeldahl Nitrogen Analysis

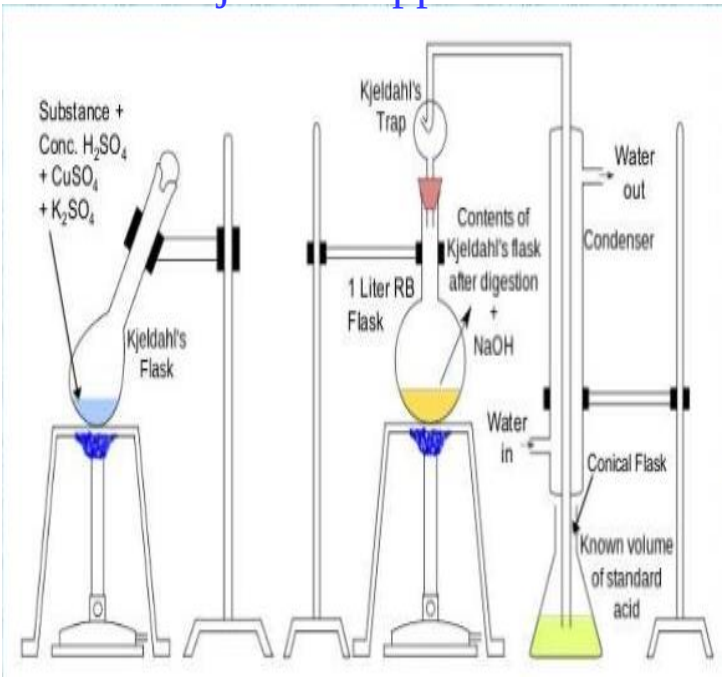
- The standard method for determining the percent protein content in milk is the Kjeldahl method which, in effect, analyzes total nitrogen. Protein nitrogen derived from amino acids represents approximately 95% of nitrogen in milk. Non-protein nitrogen, such as urea, exists in minor quantities at approximately 5%. Therefore, the protein content in milk can be extrapolated by analyzing total nitrogen.
- Because the Total Kjeldahl Nitrogen (TKN) analysis does not directly measure protein, the result of total nitrogen is converted into percent protein by multiplying by a factor of 6.38. The conversion factor of 6.38 is specific to milk in that it accounts for the nitrogen content of the average known amino acid composition that is present.

TKN analysis in milk involves three stages:

1. Digestion

In the initial digestion stage, a mixture of potassium sulfate, copper sulfate, and sulfuric acid are added to a digestion flask containing a pre-weighed, pre-heated (38°C) sample of milk. The digestion solution is heated and kept at a rolling boil for approximately 1.5 to 2 hours. After cooling, purified water is added to the digest. The digest is then transferred to a distillation flask, where sodium hydroxide is added to neutralize the solution and ultimately convert ammonium sulfate to ammonia gas.

Kjeldahl Apparatus

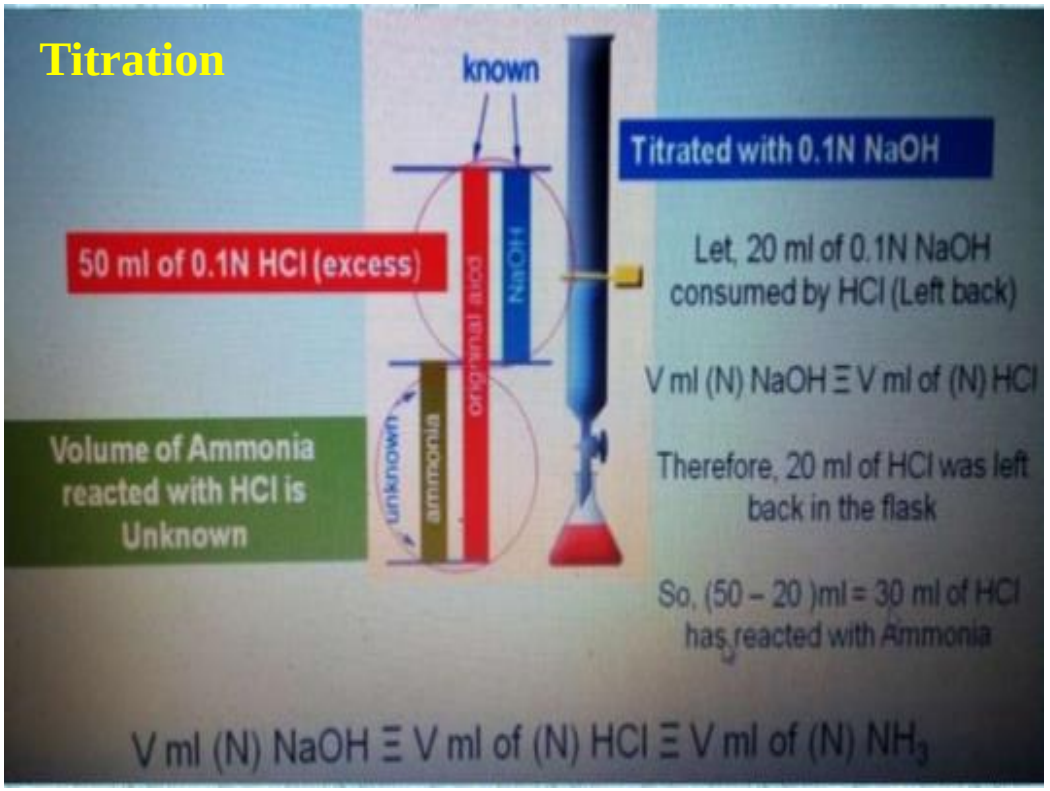


2. Distillation

In the distillation stage, the solution is again heated until all of the ammonia gas is liberated and captured in a boric acid solution.

3. Titration

The analysis stage first involves the titration of a blank sample, containing only digested reagents, followed by the titration of the sample-containing distillate with a standard sulfuric or hydrochloric acid solution



Calculation

- Let the weight of the organic substance be x gm and V ml of (N) HCl is required for complete neutralization of ammonia evolved.
- V ml (N) HCl = V ml of (N) NH3
- 1000 ml of a N NH3 contain 17 gm of NH3 or 14 gm of Nitrogen
- Amount of Nitrogen present in V ml of (N) NH3 = $\frac{14}{1000} \times V \times N = y$ gm
- Percentage of nitrogen = $\frac{\text{Weight of Nitrogen (y gm)}}{\text{Weight of Substance (x gm)}} \times 100$
- Where ,
- N = Normality of Acid Used ; V = Volume of Acid used up
- %N× 6.25(Correction Factor) = %protein

……SUGGESTIONS

Created by, Dr. Subhash Lonkar

Thank you…..