

(Deemed University Established Under Section 3 of UGC Act 1956)

COIMBATORE-21

DEPARTMENT OF CHEMISTRY

SYLLABUS

B.Sc Chemistry	y Semester-VI	2018-2019)
18CHU611A	BASIC ANALYTICAL CHEMISTRY	-PRACTICAL	3H 1C
Instruction Ho	urs/week: L:0 T:0 P:3	Marks: Internal:40	External: 60
Total:100			

Course Objectives

The course enables the students to

- 1. Understand the methods to analyse gasoline, arson accelerants and use of phenolphthalein in trap cases.
- Analytical tests for testing macronutrients, Iron in food products and caffeine and 2. benzoic acid in soft drinks.

Course Outcome

The course enables the students to

- 1. Analyse gasoline, arson accelerants and use of phenolphthalein in trap cases.
- 2. Do analytical tests for testing macronutrients, Iron in food products and caffeine and benzoic acid in soft drinks.

Experiments

Applications (Any one):

- a. To study the use of phenolphthalein in traps cases.
- To analyze arson accelerants. b.
- To carry out analysis of gasoline. c.

Instrumental demonstrations:

- a. Estimation of macro nutrients: Potassium, Calcium, Magnesium in soil samples by flame photometry.
- b. Spectrophotometric determination of Iron in Vitamin / Dietary Tablets.
- Spectrophotometric Identification and Determination of Caffeine and Benzoic Acid in c. Soft Drink.

Suggested Reading

Text Books:

1. Willard, H.H., Merritt, L.L., Dean, J. & Settoe, F.A. (1988). Instrumental Methods of Analysis (VII Edition). Wadsworth Publishing Co. Ltd., USA.

- 2. Skoog, D.A., Holler, J. & Crouch, S.R. (2009). *Instrumental Analysis* (India Edition). Cengage Learning India Private Limited, New Delhi.
- 3. Skoog, D.A., West, D.M. & Holler, F.J. (1992). *Fundamentals of Analytical Chemistry* (VI Edition). Fort Worth: Saunders College Publishing.
- 4. Harris, D. C. (2006). *Quantitative Chemical Analysis*. W. H. Freeman and Company Ltd.
- 5. Dean, J. A. (1992). Analytical Chemistry Notebook. McGraw Hill.

Reference Books:

- 1. Day, R. A. & Underwood, A. L. (1991). *Quantitative Analysis*. Prentice Hall of India.
- 2. Freifelder, D. (1982). *Physical Biochemistry* (II Edition). W.H. Freeman and Co., USA.
- 3. Cooper, T.G. (1977). *The Tools of Biochemistry*. John Wiley and Sons, USA.
- 4. Robinson, J.W. (1995). Undergraduate Instrumental Analysis (V Edition). Marcel Dekker Inc., New Delhi.



CLASS : III B.Sc CHEMISTRY COURSE NAME : BASIC ANALYTICAL CHEMISTRY PRACTICAL DE : 16CHU611A SEMESTER : VI BATCH-2016-2019

COURSE CODE : 16CHU611A

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Suggested Readings:

Text Books:

 Khosla, B. D., Garg, V. C. & Gulati, A.(2011). Senior Practical Physical Chemistry. New Delhi : R. Chand &Co.

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Experiment No: 1

DETERMINATION OF PH VALUER OF SOIL SAMPLES

Aim

To Determine the pH value of soil samples.

Materials required

pH meter, Beaker, glass rod, funnel, filter paper and chemical balance.

Principle :

Determination of pH is actually a measurement of hydrogen ions activity in soil – water system. It is defined as negative logarithm of the hydrogen ion activity. Mathematically, it is expressed as:

 $pH = -\log a H^+$

The pH value of a soil is an indication of soil reaction i.e. acidic, neutral or alkaline. The nutrient availability is governed by soil reaction. It is maximum at neutral pH and decreases with increase in acidity or alkalinity. Thus, pH value gives an idea about the availability of nutrients to plants.

The pH is usually measured by pH meter, in which the potential of hydrogen ion indicating electrode (glass electrode) is measured potentiometrically against calomel saturated reference electrode which also serves as salt bridge. Now a days, most of the pH meters have single combined electrode. Before measuring the pH of the soil, the instrument has to be calibrated with standard buffer solution of known pH. Since, the pH is also affected by the temperature, hence, the pH meter should be adjusted to the temperature of the solution by temperature correction knob.

Reagents :

Standard buffer solutions: These may be of pH 4.0, 7.0 or 9.2 and are prepared by dissolving one standard buffer tablet in 100 ml distilled water, It is necessary to prepare fresh buffer solution after few days. In absence of buffer tablet, a 0.05 M potassium hydrogen phthalate solution can be used which gives a pH of

4.0 (Dissolve 10.21 g. of A.R. grade potassium hydrogen phthalate in distilled water and dilute to 1 litre. Add 1 ml of chloroform or a crystal of thymol per litre as a preservative).

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Procedure :

Soil to water ratio of 1:2 (pH₂)

Take 20 g soil in 100 ml beaker and add 40 ml. of distilled water to it. The suspension is stirred at a regular interval for 30 minutes. Determine the pH by immersing electrodes in suspension. For soils containing high salts, the pH should be determined by using 0.01M calcium chloride solution. (Dissolve 0.110 g of CaCl₂ in water and dilute to 1 litre).

Saturates soil paste (pHs)

Add small amount of distilled water to 250g of air dried soil. Stir the mixture with a spatula. At saturation, the soil paste glistens and flows slightly when the container is tapped it slides freely and ensures cleanly off the spatula. After mixing, allow the sample to stand for an hour. If the paste has stiffened markedly or lost its glistening, add more water or if free water has collected on the surface of the paste, add an additional weighed quantity of dry soil and mix it again. Then insert the electrode carefully in the paste and measure the pH.

Saturation extract (pHe)

The soil is extracted using vacuum extractor and the pH is measured in the saturation extract.

Result:-

The pH value of the soil sample is = ------

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Experiment No: 2

DETERMINATION OF SOIL MOISTURE

Aim

To determine the moisture content of soil samples.

Equipments :

- Sampling tube/auger Moisture cans (numbered)
- Balance with weights/automatic Drying oven
- > Desicator

Procedure :

Collect soil samples by tube or auger from a number of points within the experimental site and mix thoroughly. Place composite sub samples of about 50 gm to 100 gm in soil moisture cans with tight fitting lids. Take atleast three sub- samples. The moist samples are weighed immediately, dried to constant weight in an oven at 105°C (for about 24 hrs) and reweighed after cooling in a desicator. Determine the tare weight of moisture cans. Calculate the soil moisture content by determining the loss in weight on drying and the weight of the oven dry soil as follows :

Soil moisture content by weight $\% = \frac{(\text{weight of wet soil} + tare) - (\text{Weight of dry soil} + tare)}{(\text{weight of dry soil} + tare) - tare}$

$$Mw (\%) = \frac{Loss \, of \, wweight \, on \, dry \, soil}{t \, of \, oven \, dry \, soil} \times 100$$

% of moisture on volume basis = % of moisture on weight basis × bulk density

Result:

The percentage of moisture content of soil sample is = ----- %

Prepared by Dr.M.Makeswari, Department of chemistry, KAHE

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Experiment No:3

DETERMINATION OF SOIL ELECTRICAL CONDUCTIVITY

Aim:-

To Determine the electrical conductivity of soil samples.

Apparatus:-

Beaker, glass rod, funnel, filter paper, Conductivity meter and chemical balance.

Reagents :

Potassium chloride: Dissolve 0.7456g dry potassium chloride (AR) in distilled water and make up the volume to one litre.

Conductivity :

The knowledge of total soluble salts is essential in crop production, specially during the process of salinization. Since, there is a direct relationship between the quantity of soluble salts and the electrical conductance. Hence soluble salts in soils are measured indirectly by measuring the electrical conductance of the soil.

Principle :

The electrical conductivity is measured with the help of solubridge. The instrument is calibrated and cell constant is determined with the help of 0.1 N KCl solution. This solution gives an electrical conductance of 1.41 mmhos/cm or dSm^{-1} at $25^{0}C$.

Procedure :

Take 20 g of soil in 100 ml beaker, add 40 ml of distilled water and shake intermitantly for 30 minutes. Determine the conductivity of the supernatant liquid with the help of conductivity meter. The electrical conductivity of saturation extract (E.C.e) is also determined for salinity ratings.

Result:-

The electrical conductivity of the soil sample is =

Prepared by Dr.M.Makeswari, Department of chemistry, KAHE

Experiment No: 4

DETERMINATION OF CAFFEINE IN TEA

Aim

To determine the amount of caffeine present in tea by gravimetric method.

Reagents and Materials

- Conical flask, beaker,
- o **funnel**,
- Sintered crucibles,
- \circ Na₂HPO₄,
- NaOH, Na₂SO₄,
- Distilled water,
- \circ $\,$ Tea powder.

Theory:

Gravimetric method of determination of caffeine in tea

Caffeine is a weak base, which does not form stable salts with acids, and even if salts are formed it is easily dissociated in aqueous media, consequently, volumetric aqueous titration assay is unsuitable for determination of caffeine contents. Three different methods can be applied such as (i) Gravimetric; (ii) Colorimetric; and (iii) Nonaqueous titration methods can be used for assay of caffeine.

Isolation of Caffeine from Tea

The powdered tea leaves are extracted with boiling water and filtered; The filtrate is purified with Pb acetate solution (to precipitate tannins and other impurities); Excess lead is removed by addition Na_2HPO_4 followed by filtration; Caffeine is extracted from the filtrate with chloroform and is purified by recrystallization from water.

Procedure :

Gravimetric method of analysis

This method essentially consists of the following steps:

- (i) Weigh accurately 2 g of powdered tea and boil in a conical flask under reflux condenser with 100 ml of water for 30 min;
- (ii) Filter the solution while hot on a piece of cotton and reflux again with 50 ml water for 15 min;
- (iii) Filter the aqueous extract through the same filter and wash with 10 ml of H₂O;
- (iv) Concentrate the combined aqueous extracts to about 50 ml in a porcelain dish, (with continuous stirring with a glass rod to prevent sublimation of caffeine), then cool;
- (v) Transfer the concentrated solution to a separating funnel, wash the dish with 10 ml of water then add the washings to the separator;

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- (vi) Extract with three successive quantities of 40, 40, and 20 ml of chloroform, wash the mixed chloroformic extracts with 10 ml of N/1 NaOH (to remove any resinous matter), then wash with 10 ml water. Test for complete extraction with Wagner's reagent;
- (vii) Filter the chloroformic extract on anhydrous Na₂SO₄; and
- (viii) Distil off the chloroformic extract in a pre-weighed flask on a water bath. Transfer the flask to a desiccator for 2 h and weigh till constant weight.

Calculation

% of caffeine (w/w) = wt. of the residue $\times 100/wt$. of tea

Result :

% of caffeine present in tea powder is =



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Experiment No 5

ESTIMATION OF IRON IN VITAMINE TABLETS

Aim:

To determine amount of iron present in vitamine tablets by volumetrically.

Requirements:

Burette, pipette, Conical flask, beaker, Measuring jar, Potassium permanganate, iron tablets.

Theory :

To estimate the iron(II) content of an iron tablet, a small number of tablets are first dissolved in dilute sulfuric acid. This solution is then titrated against previously standardised potassium manganate(VII) solution. The reaction is represented by the equation:

$MnO_{4} + 8H^{+} + 5Fe^{+2} \rightarrow Mn^{+2} + 5Fe^{+3} + 4H_2O$

Method Preparation of tablets

Find the mass of five iron tablets. (Note that if Feospan tablets are used, each capsule should be opened, and the contents weighed.) Crush the weighed tablets in a mortar and pestle. Transfer all the ground material to a beaker where it is dissolved in about 100 cm^3 of dilute sulfuric acid.

All of this solution (including washings) is transferred to a 250 cm³ volumetric flask and the solution made up to the mark with deionised water. The volumetric flask should be stoppered and inverted several times. This is the solution containing iron(II) ions.

Titration:

Wash the pipette, burette and conical flask with deionised water. Rinse the burette with the potassium manganate(VII) solution and the pipette with the iron(II) solution.

Using a pipette filler, fill the pipette with the iron(II) solution and transfer the contents of the pipette to the conical flask. Acidify this solution by adding about 10 cm^3 of dilute sulfuric acid.

Using a funnel, fill the burette with potassium manganate(VII) solution, making sure that the part

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below the tap is filled before adjusting to zero. Because of the intense colour of KMnO₄ solution, readings are taken from the top of the meniscus.

With the conical flask standing on a white tile, add the solution from the burette to the flask. Swirl the flask continuously and occasionally wash down the walls of the flask with deionised water using a wash bottle.

The end-point of the titration is detected by 'the first persisting pink colour'. Note the burette reading.

Repeat the procedure two or three times, adding the potassium manganate(VII) dropwise approaching the endpoint. These accurate titres should agree to within 0.1 cm³.

Calculate the concentration of the iron(II) solution, and from this calculate the mass of iron in an iron tablet.

Specimen Results:

Mass of iron tablets = 1.81 g Rough titre = 17.0 cm³ Second titre =

16.7 cm^3 Third titre = 16.7 cm^3

Average of accurate titres = 16.7 cm^3

Volume of iron(II) solution used in each titration = 25.0 cm^3 Concentration of potassium manganate(VII) solution = 0.005 M

Calculation:

 $\begin{aligned} V_A x M_A x n_B &= V_B x M_B x n_A 25.0 x M_A x 1 \\ M_A &= 16.7 x 0.005 x 5 / (25.0 x 1) \\ &= 0.0167 M \end{aligned} = 16.7 x 0.005 x 5 / (25.0 x 1) \end{aligned}$

Volume of Fe²⁺ solution in total = 250.0 cm³ Moles of iron in this volume = 0.0167 = 0.004175 Mass of iron in this volume = 0.004175 x 56 g = 0.2338 g Percentage of iron in the tablets $= \frac{mass of iron \times 100}{mass of tablets}$

$$=\frac{0.2238 \times 100}{1.81}$$
= 12.92%



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Mass of iron in each tablet = 0.2338 / 5 = 46.76 mg.

Result :

Mass of iron present in tablet is =