

COLLEGE PRACTICAL CHEMISTRY

T.Y.B.Sc.



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COLLEGE PRACTICAL CHEMISTRY

T.Y. B.Sc.

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PREFACE TO THE TWENTYFIFTH EDITION

We are very pleased to bring out the Twentyfifth Edition of T.Y. B.Sc. Chemistry Practicals Book for the students of Mumbai University (2019-20).

After a long time, Qualitative Analysis of the mixture containing two cations and two anions, is introduced. Mixture may contain one cation as a major component which is to be estimated as quantitative estimation preferably volumetric estimations. The other cation and the anions are to be estimated as qualitative analysis, without the help of H_2S gas. Even the percentage purity of the major cation is to be determined.

In Organic Chemistry Section, the analysis of binary mixture is modified. Mixture containing solid-solid components is included in Semester V. Likewise, mixture containing liquid-liquid and liquid-solid components, is included in Semester VI.

In Physical Chemistry and Analytical Chemistry Sections, there is no much additions or subtractions.

We have tried our utmost best to simplify the procedures of each and every experiment as far as possible.

We are confident that like all our previous editions, this edition will also be accepted wholeheartedly by the students as well as colleagues.

Date: June 2019

Mumbai

— **AUTHORS**

PREFACE TO THE TWENTYFOURTH REVISED EDITION

We are very pleased and feel honoured to bring out this Twentyfourth Edition of T.Y. B.Sc. Chemistry Practicals Book for the students of Mumbai University (2018-19).

University has made changes in the Syllabus. In Inorganic Chemistry Section, Gravimetric/Volumetric Estimations are removed.

After a long time, Qualitative Analysis of the mixture containing two cations and two anions, is introduced. Mixture may contain one cation as a major component which is to be estimated as quantitative estimation preferably volumetric estimations. The other cation and the anions are to be estimated as qualitative analysis, without the help of H_2S gas. Even the percentage purity of the major cation is to be determined.

In Organic Chemistry Section, the analysis of binary mixture is modified. Mixture containing solid-solid components is included in Semester V. Likewise, mixture containing liquid-liquid and liquid-solid components, is included in Semester VI.

In Physical Chemistry and Analytical Chemistry Sections, there is no much additions or subtractions.

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Date: June 2018
Mumbai

— AUTHORS

SYLLABUS

T.Y. B.Sc. Chemistry Practical

(2018-19)

SEMESTER V

Paper I (Code: USCHP 501)

Physical Chemistry Experiments

NON INSTRUMENTAL:

(I) Colligative Properties:

- (1) To determine the molecular weight of a compound by Rast Method. **(6 Units Only)**.

(II) Chemical Kinetics:

- (1) To determine the order of reaction between $K_2S_2O_8$ and KI by fractional change method. **(3 and Units)**.

(III) Surface Phenomena:

- (1) To investigate the adsorption of acetic acid on activated charcoal and test the validity of *Freundlich Adsorption Isotherm*. **(6 Units Only)**.

INSTRUMENTAL:

(IV) Potentiometry:

- (1) To determine the solubility product and solubility of AgCl potentiometrically using a **chemical cell**. **(6 Units Only)**.

(V) Conductometry:

- (1) To determine the velocity constant of alkaline hydrolysis of ethyl acetate by conductometric method. **(6 Units Only)**.

(VI) pH-metry:

- (1) To determine acidic and basic dissociation constants of amino acid and hence to calculate *isoelectric point*. **(3 and 6 Units)**.

SEMESTER V

Paper II (Code: USCHP 502)

Inorganic Chemistry Experiments

(I) Inorganic Preparations:

- (1) Preparation of potassium diaquo *bis* (oxalato) cuprate (II). **(3 and 6 Units)**.
- (2) Preparation of ferrous ethylene diammonium sulphate. **(6 Units Only)**.
- (3) Preparation of *bis* acetylacetonato copper (II). **(6 Units Only)**.

(II) Determination of percentage purity of the given water soluble salt and qualitative detection with respect to added cation and/or anion (qualitative analysis only by wet tests):

- (1) Any three salts of transition metal ions. **(3 and 6 Units)**.

SEMESTER V

Paper III (Code: UDCHP 503)

Organic Chemistry Experiments

(I) Separation of Binary solid-solid mixture:

(2.0 g mixture to be given)

- (1) Minimum six mixtures to be completed by the students.
- (2) Components of the mixture should include water soluble and water insoluble acids (carboxylic acid), water insoluble phenols (α naphthol, β naphthol), water insoluble bases (nitroanilines), water soluble neutrals (thiourea) and water insoluble neutral compounds (anilides, amides, *m* dinitro benzene, hydrocarbons).
- (3) After correct determination of chemical type, the fixing reagent should be decided by the student for separation
- (4) Follow separation scheme with the bulk sample of binary mixture.
- (5) After separation into component **A** and component **B**,
 - (a) One component (decided by the examiner) is to be analysed and identified, with melting point.
 - (b) The other component is dried and weighed. **(3 and 6 Units)**.

SEMESTER V

Paper IV (Code: USCHP 504)

Analytical Chemistry Experiments

(I) Spectrophotometry:

- (1) Spectrophotometric estimation of fluoride. **(6 Units Only)**.

(II) Analysis of Commercial Samples:

- (1) Estimation of magnesium content in talcum powder by complexometry, using *standardised* solution of *EDTA*. **(3 and 6 Units)**.

(III) Water Analysis:

- (1) Determination of chemical oxygen demand (*COD*) of water sample. **(3 and 6 Units)**.

(IV) Flame Photometry:

- (1) To determine potassium content of a fertilizer by flame photometry. (Calibration curve method). **(3 and 6 Units)**.

(V) Titrimetry Analysis:

- (1) To determine the amount of persulphate in the given sample solution by back titration with standard ferrous ammonium sulphate solution. **(6 Units Only)**.

(VI) Turbidimetry:

- (1) To determine the amount of sulphate in given water sample turbidimetrically. **(6 Units Only)**.

SEMESTER VI

Paper I (Code: USCHP 601)

Physical Chemistry Experiments

NON INSTRUMENTAL:

(I) Chemical Kinetics:

- (1) To interpret the order of reaction graphically from the given experimental data and to calculate the specific rate constant (No fractional order). **(6 Units Only)**.

(II) Viscosity:

- (1) To determine the molecular weight of high polymer polyvinyl alcohols (*PVA*) by viscosity measurements. **(3 and 6 Units)**.

INSTRUMENTAL:

(III) Potentiometry:

- (1) To determine the amount of iodide, bromide and chloride in the mixture by potentiometric titration with silver nitrate. **(6 Units Only)**.
- (2) To determine the number of electrons in the redox reaction between ferrous ammonium sulphate and ceric sulphate potentiometrically. **(3 and 6 Units)**.

(IV) Conductometry:

- (1) To titrate a mixture of weak acid and strong acid against a strong base and to estimate the amount of each acid in the mixture conductometrically. **(6 Units Only)**.

(V) Colorimetry:

- (1) To determine the empirical formula of the complex between Fe (III) and salicylic acid by static method. **(3 and 6 Units)**.

SEMESTER VI

Paper II (Code: USCHP 602)

Inorganic Chemistry Experiments

(I) Inorganic Preparations:

- (1) Preparation of *Tris* (acetyl acetonato) iron (III). **(3 and 6 Units)**.
- (2) Green synthesis of *bis* (dimethylglyoxamato) nickel (II) complex using nickel carbonate and sodium salt of *dmg*. **(6 Units Only)**.
- (3) Preparation of potassium trioxalato aluminate (III). **(6 Units Only)**.

(II) Determination of percentage purity of the given water soluble salt and qualitative detection with respect to added cation and/or anion (qualitative analysis only by wet tests):

- (1) Any three salts of main group metal ions. **(3 and 6 Units)**.

SEMESTER VI

Paper III (Code: USCHP 603)

Organic Chemistry Experiments

(I) Separation of binary liquid-liquid and liquid-solid mixture:

- (1) Minimum six mixtures are to be completed by the students.
- (2) Components of the liquid liquid mixture should include volatile liquids like acetone, methyl acetate, ethyl acetate, isopropyl alcohol, ethyl alcohol, ethyl methyl ketone and non volatile liquids like chlorobenzene, bromobenzene, aniline, N, N dimethylaniline, acetophenone, nitrobenzene, ethyl benzoate.
- (3) Components of the liquid solid mixture should include volatile liquids like acetone, methyl acetate, ethyl acetate, ethyl alcohol, ethyl methyl ketone, isopropyl alcohol and solids such as water insoluble acids, phenols, bases, neutrals.
- (4) A sample of the mixture (01 cm^3) to be given to the student for detection of the physical type of the mixture.
- (5) After correct determination of physical type, separation of the binary mixture to be carried out by distillation method.

- (6) After separation into component **A** and component **B**, the component to be identified will be decided by the examiner.
- (a) In case of liquid solid mixture, the volatile component or the solid component as indicated by the examiner, is to be analysed and detected. The volume/weight of the other component is to be reported. **(3 and 6 Units)**

SEMESTER VI

Paper IV (Code: USCHP 604)

Analytical Chemistry Experiments

(I) Spectrophotometry:

- (1) Estimation of chromium in water sample spectrophotometrically by using diphenyl carbazide. **(3 and 6 Units).**

(II) Analysis of Commercial Samples:

- (1) Estimation of reducing sugar by honey by *Wilstatler method*. **(6 Units Only).**

(III) Ion Exchange Separations:

- (1) Estimate the amount of zinc and magnesium present in the given solution of magnesium zinc mixture, using an anion exchange resin column. **(3 and 6 Units).**

(IV) Potentiometry:

- (1) Estimation of acetic acid in vinegar sample by using quinhydrone electrode potentiometrically. **(3 and 6 Units).**

(V) pH-metry

- (1) Determination of phosphoric acid in cola sample pH-metrically. **(6 Units Only).**

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SEMESTER V

ANALYTICAL CHEMISTRY

XIII

Flame Photometry

Flame photometry is concerned with the emission of characteristic radiation in flames by individual elements and correlation of the emission intensity with the concentration of these elements. It is also called as *flame emission spectroscopy* because of the use of a flame to provide the energy of excitation to atoms introduced into the flame.

The sample is usually presented for analysis in the form of solution. It is transformed into a mist of fine droplets and sprayed into the flame in a reproducible and constant manner. The series of complex processes that occur in the flame begins with the evaporation of the solvent, vapourisation of the solute and dissociation of solute compounds. Formation of atoms of the solute is usually accompanied by association into molecules, by ionisation and by excitation. The excited atoms, which are unstable, quickly emit photons and return to lower energy state, eventually reaching the unexcited state. The measurement of emitted photons, *i.e.*, radiation forms the basis of flame photometry.

The fraction of free atoms that are thermally excited, is governed by a *Boltzmann Distribution* which is as follows:

$$\frac{N^*}{N_0} = A \cdot e^{\Delta E/kT}$$

Where, N^* = the number of excited atoms,

N_0 = the number of atoms in the ground state,

A = A constant for a particular element,

ΔE = the difference in energies of the two levels,

k = the Boltzmann's of the flame,

T = the temperature of the flame.

From the equation, it follows that the fraction of atoms excited depends upon the temperature of the flame. Therefore, one expects higher number of excited atoms if the temperature of flame is increased. This emphasises the importance of controlling temperatures in flame photometry. The temperature of fuel is controlled by the type of fuel and oxident used.

In the case of elements like lithium, sodium or potassium which ionise easily, moderate temperatures of flames are required which only produce excited atoms.

When flame photometry is employed as an analytical tool, the radiations emerging from the flame are passed through an optical filter which permits only the radiation characteristic of the element under investigation to pass through the photocell. The output from the photocell is measured with a suitable galvanometer.

A block diagram of the equipment used in flame photometry is shown in Fig. 13.1. It consists of the basic components as source, monochromator, slit system and detector system. The flame photometer also includes a burner which is utilised for burning the sample solution producing excited atoms.

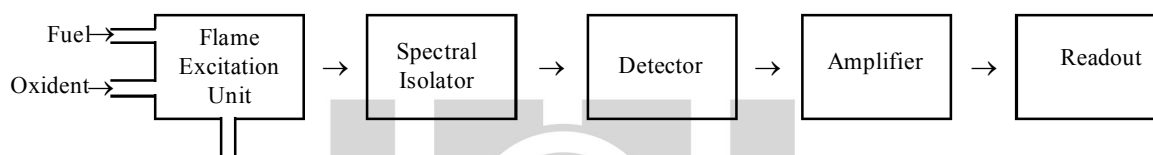


Fig. 13.1

The experimental measurement of a sample demands knowledge of following working conditions:

- (1) *Gases*: The choice of the gas to be used is made from the nature of the element to be analysed. For Li, Na and K, normally a mixture of air + acetylene gas is used.
- (2) *Pressure of atomising gas*: The pressure depends upon the construction of atomiser. Some commercial models use a fixed air/oxygen pressure.
- (3) *Pressure of fuel gas*: The pressure of the fuel gas must be so adjusted that the flame is neither too cold (too little gas) nor too sooty (too much gas).
- (4) *Wavelength selector*: The choice of filter depends upon the element to be analysed.
- (5) *Slit and sensitivity*: To adjust slit, atomise one standard solution of the highest concentration and put potentiometer scale at the upper limit, *i.e.*, galvanometer deflection near the end of scale. Gradually adjust the slit until the maximum deflection (100%) is obtained. Fine adjustments can be made with the sensitivity control. It is better to work with minimum slit and maximum sensitivity.
- (6) *Banks*: *Water* or background solution is measured once, for each series.
- (7) *Washing*: Following order is followed when reading the series of standard solutions:
 - (i) 100% adjustment (with most concentrated solution)
 - (ii) Washing
 - (iii) Zero adjustment (with distilled water)
 - (iv) Washing

- (v) 100% readjustment (As in *i*)
- (vi) Washing
- (vii) Zero adjustment
- (viii) Washing
- (ix) Measurement of standards beginning with the most dilute solution.

Experiment No. 1 (3 and 6 Units)

Aim: To determine potassium content of a fertilizer sample by *flame photometry*.

Theory: In a calibration curve method, the flame is aspirated with known concentrations of the element to be determined and respective readings and measure. A calibration curve is then constructed by plotting signal intensity for each of these standard solutions against the respective concentration. Then the sample having unknown concentration is aspirated and signal intensity is recorded. On the basis of signal intensity, concentration of element in sample can be obtained. If necessary the test solutions may suitably be diluted to get the readings in the range of 0.1 to 0.4. The calibration curve should be checked periodically by making measurements with the standard solutions.

In the standard addition technique, a calibration curve is prepared as described before. Emission readings are then obtained for an aliquot of sample and for an identical aliquot to to which a known quantity of the same elements is added. The concentration of the element in the sample can be calculated from the simple relationship.

$$\frac{X}{X+S} = \frac{I_x}{I_{(x+s)}}$$

- where, X = Concentration of test element in sample,
 S = Concentration increase caused by the addition of standard,
 I_x = Emission intensity of sample solution,
 $I_{(x+s)}$ = Emission intensity after addition of standard.

Normally a series of solution, such as $X + S$, $X + 2S$, $X + 3S$, etc. are prepared and emission intensities are obtained, The emission intensities are then plotted against the concentration of the added standard. Intercept of the resulting line, yields the concentration of the sample.

Requirements: (1) A stock solution of K containing $1 \times 10^{-3} \text{ g/cm}^3$ of the metal can be prepared by dissolving 0.477 g of KCl in 250 cm^3 of distilled water in a standard measuring flask. (*i.e.*, the strength of stock solution = 1000 ppm). (2) Sample solution of K , 100 cm^3 standard measuring flasks, distilled waer, 10 cm^3 pipette, etc.

Procedure:

For calibration curve method:

- (1) Dilute 10 cm^3 of the supplied stock solution of $1 \times 10^{-3} \text{ g/cm}^3$ (1000 ppm) concentration to 100 cm^3 in a standard measuring flask with distilled water. This gives standard solution of 100 ppm.
- (2) Take six serially numbered 100 cm^3 standard measuring flasks.

- (3) Add 5, 10, 15, 20 and 25 cm^3 of 100 ppm K solution to the standard measuring flasks numbered from 1 to 5. Dilute each flask to 100 cm^3 with distilled water.
- (4) Dilute the supplied sample solution to 100 cm^3 in a standard measuring flask with distilled water.
- (5) Pipette out 10 cm^3 of the diluted sample solution of K in 100 cm^3 standard measuring flask and dilute upto the mark (flask No. 6).
- (6) Measure the emission intensity for each solution as described. Note the emission intensities of the different solution of known concentration and sample solution as shown in Table.
- (7) Plot the graph of concentration of solutions (in ppm) against emission intensities. This forms the calibration curve.
- (8) With the help of emission intensity of the sample solution obtain the concentration of K from the calibration curve.

Observations:

Flask Nos.	Volume of standard solution in cm^3	Final volume of solution in cm^3	Concentration of solution in ppm	Emission intensity I
1	05	100	05	
2	10	100	10	
3	15	100	15	
4	20	100	20	
5	25	100	25	
6	10.0 (sample)	100	Unknown	

Graphs:

(1) For the construction of calibration curve plot the graph of *emission intensity* for the solution against concentration of solution in ppm. (Fig. 13.2).

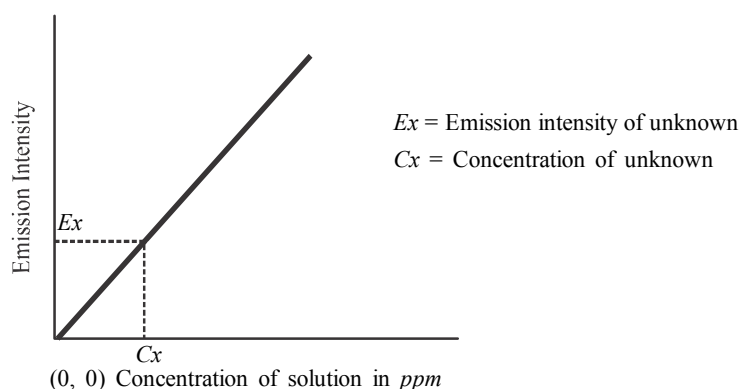


Fig. 13.2

Calculations:

Concentration of unknown from the graph = _____ ppm

10 cm³ of solution contains = $\frac{c}{10}$ ppm

100 cm³ solution contains = $\frac{10c}{100}$ ppm

10 c ppm = 10 c mg/1000 cm³

Hence 10 c ppm = c mg/100 cm³

Amount of K⁺ in the given sample is c mg.

$$\begin{aligned} \text{\% of K}^+ \text{ in the sample is} &= \frac{c \times 100}{\text{weight of the sample}} \\ &= \text{_____ \%} \end{aligned}$$

For calculation of percentage error refer to page No. 88.

Results:

- (1) Amount of K⁺ in the given sample = (c) = _____ mg.
by calibration method
- (2) Percentage of K⁺ in the given sample = _____ %.
- (3) Percentage error = _____ %.

