GUIDE FOR M.Sc. INORGANIC CHEMISTRY PRACTICALS

For use by students of M.Sc. Chemistry – Semesters 1 & 2, Mahatma Gandhi University.
(2001 admission onwards)

Prepared by:

Dr. Ravi Divakaran, Lecturer in Chemistry, St. Albert's College, Ernakulam 682 018, Kerala.

Phone: 09847219522 Off: 0484-2394225

M.Sc. Chemistry Practical Syllabus for semesters 1 & 2

CH-205 INORGANIC CHEMISTRY – PRACTICALS-I (60 + 60 hours)

A – Qualitative analysis

I. Separation and identification of four metal ions including two less familiar elements such as Tl, W, Se, Mo, Ce, Th, Ti, Zr, V, U and Li. (Na, K and eliminating anions not to be given. A minimum of 5 mixtures containing 5 different rare ions have to be analysed by a student)

B – Quantitative analysis

II. Complexometric titration for the estimation of hardness of water, Zn, Mg, Ca, Ni ions.

III. Colourimetric estimation of Fe, Cu, Ni, Mn, Cr, NH_4^+, phosphate and nitrate ions.

References

1. Vogel – A Text Book of Qualitative Inorganic Analysis – Longman
2. Kolthoff & Stenger – Volumetric Analysis – Intersience
3. Vogel – A Text Book of Quantitative Inorganic Analysis – Longman
4. Kolthoff & Sandell - Text Book of Qualitative Inorganic Analysis.
5. G. Schwarzen Back "Complexometric Titration" Interscience.

Note to Examiners:

1. Candidates may be asked to report four metal ions present in the given mixture.
2. While reporting the scheme of analysis the student is expected to indicate the chemistry involved in the relevant reactions.
3. The candidates may be asked to give the procedure for the quantitative analysis giving the chemistry behind the experiments.
4. Each student has to carry out I, II and III experiments for the practical examination.

VOLUMETRIC ANALYSIS

TOTAL HARDNESS IN WATER

Aim of the experiment: To determine the total hardness of water samples from (1) the corporation water supply and (2) the college well.

Theory: [Ref: Eaton AD, Clesceri LS, Greenberg AE, editors. Standard Methods for the Examination of Water and Waste Water. 19th ed. APHA, 1995. Method 2-36]

Hardness in water is caused by the presence of Ca^{2+} and Mg^{2+} ions. Total hardness is defined as the sum of Ca^{2+} and Mg^{2+} ion concentrations, expressed in *milligrams of $CaCO_3$ per litre*. If Eriochrome Black T (= EBT) is added to an aqueous solution containing Ca^{2+} and Mg^{2+} ions at a pH of 10.0 ± 0.1, the solution becomes wine red. Both will be complexed by EDTA. When all the Ca^{2+} and Mg^{2+} ions present are complexed by EDTA, the solution changes to blue. Mg^{2+} ions *must* be present to yield a satisfactory end point. [If Mg^{2+} is not present in the sample water, small amounts of complexometrically neutral Mg salt of EDTA is added to the buffer]. The titration should be completed in less than 5 minutes to minimize the tendency for $CaCO_3$ precipitation.

Note: (1) If murexide is used as indicator, the titration gives hardness due to Ca^{2+} alone. A dilute solution of NaOH is used instead of ammonia buffer in this case. The colour change is from pink to purple.

(2) Publications in the area of water analysis still use 'mL' instead of 'cm^3' and 'L' instead of 'dm^3'. The same terminology is used here.

Apparatus required:

(1) One 50 mL burette.
(2) One 250 mL glass beaker.
(3) Two 250 mL and one 1000 mL volumetric flasks.
(4) One 500 mL Beaker.
(5) One 100 mL measuring cylinder.
(6) Dropper and glass rod.

Chemicals required: (per student)

(1) EDTA disodium salt hydrate, Analar, 1 gram.
(2) Calcium carbonate powder, anhydrous, Analar, 1 gram.
(3) Eriochrome Black T indicator, a few crystals.
(4) Methyl orange indicator solution, 1 mL.
(5) Ammonium chloride, 17 g.
(6) Concentrated NH_3 solution, 150 mL
(7) Hydrochloric acid, 1:1, about 50 mL.

Preparation of reagents:

Note: Prepare all reagents using distilled water only! Reagents 1 and 2 may be used in common by all students. All preparations are to be recorded by students.

1. ***Standard $CaCO_3$ solution***: Accurately weigh out exactly 1.000g of analar anhydrous $CaCO_3$ powder into a clean 500 mL beaker. Add carefully just sufficient 1:1 HCl to dissolve the powder completely. Add 200 mL distilled water, cover with a watch glass and boil for a few minutes to expel CO_2. Cool and add a few drops of methyl orange indicator and adjust to the intermediate orange colour by adding drops of dilute ammonia or HCl as required. Transfer quantitatively into a 1000 mL volumetric flask and make up using distilled water. 1 mL of this solution ≡ 1.00 mg $CaCO_3$.
2. ***Buffer solution***: Dissolve 17g of NH_4Cl in 150 mL concentrated NH_3 solution in a 250 mL volumetric flask and make up with distilled water. Keep in a clean stoppered bottle.
3. ***EDTA solution***: Weigh out about 0.93g of EDTA disodium salt hydrate into a 250 mL volumetric flask, add a little ammonia solution and about 200 mL of distilled water and swirl gently to dissolve completely (presence of ammonia makes dissolution of EDTA faster). Make up to the mark to get approximately 0.01 M solution. Standardise against standard $CaCO_3$ solution. Obtain result in the form "1 mL EDTA solution ≡ ____ mg $CaCO_3$. (*Note: Standardisation to be recorded in the usual form*).

Procedure:

(1) *Standardisation of EDTA*: Pipette out 20 mL of standard $CaCO_3$ solution into a 250 mL beaker and add 1 to 2 mL of buffer solution. Add 2 or 3 small crystals (count! Do not use more indicator than necessary to get pale colour) of EBT and stir using a glass rod to get wine red colour. Titrate with EDTA solution, stirring after each addition, till the colour just changes to blue. Repeat.
(2) *Estimation of hardness in sample*: Measure out 100 mL of sample water (using cylinder) into a clean 250 mL beaker and titrate using EDTA exactly as above. Repeat.

Calculation:

Standardisation of EDTA: V mL of EDTA ≡ 20 mL $CaCO_3$ solution ≡ 20 mg $CaCO_3$. Therefore 1 mL EDTA = _____ mg $CaCO_3$.
Estimation of hardness in sample: 100 mL water ≡ V mL EDTA ≡ _____ mg $CaCO_3$. Therefore 1000 mL water = _____ mg $CaCO_3$.

Result:

(1) Total hardness in corporation tap water = _____ mg $CaCO_3$/L
(2) Total hardness in college well water = _____ mg $CaCO_3$/L

ESIMATION OF CALCIUM

Aim of the experiment: To determine the mass of calcium in the whole of the given solution.

Theory: Eriochrome Black T (= EBT) forms a wine-red coloured complex with Ca^{2+} ions in solution at a pH of about 10 (obtained by adding ammonia solution). EDTA forms a stronger complex with the Ca^{2+} ions and liberates free EBT, which has a blue colour. One mole of EDTA complexes with one mole of Ca^{2+} ions.

$$Na_2H_2EDTA + Ca^{2+} \rightarrow CaH_2EDTA(complex) + 2\ Na^{+}$$

Apparatus required:

(1) One 50 cm^3 burette.
(2) One 250 cm^3 conical flask.
(3) Two 100 cm^3 and one 250 cm^3 volumetric flasks.
(4) One 250 cm^3 Beaker.
(5) One 100 mL measuring cylinder.
(6) Dropper, glass rod and watch glass.

Chemicals required: (per student)

(1) EDTA disodium salt hydrate, Analar, 5 grams.
(2) Calcium carbonate powder, anhydrous, Analar, 1 gram.
(3) Eriochrome Black T indicator, a few crystals.
(4) Methyl orange indicator solution, 1 mL.
(5) Ammonium chloride, 2 g.
(6) Concentrated NH_3 solution, 15 mL
(7) Hydrochloric acid, 1:1, about 10 mL.

Preparation of reagents:

Note: Prepare all reagents using distilled water only! All preparations are to be recorded by students.

Buffer solution: Dissolve 17g of NH_4Cl in 150 cm^3 concentrated NH_3 solution in a 400 cm^3 beaker and dilute to 250 cm^3 with distilled water. Keep in a clean stoppered bottle. (Enough for all students)

EDTA solution: Weigh out about 4.65g of EDTA disodium salt hydrate into a 250 mL volumetric flask, add a little ammonia solution and about 200 mL of distilled water and swirl gently to dissolve completely (presence of ammonia makes dissolution of EDTA faster). Make up to the mark to get approximately 0.05 M EDTA solution.

Procedure:

Preparaion of standard 0.05M $CaCO_3$ solution: Accurately weigh out about 500 mg of analar anhydrous $CaCO_3$ powder into a clean 250 cm^3 beaker. Add about 20 cm^3 distilled water. Carefully add just sufficient 1:1 HCl in drops and sir to dissolve the powder completely. Cover with a watch glass

and boil for a few minutes to expel CO_2. Cool and add a few drops of methyl orange indicator and adjust to the intermediate orange colour by adding drops of dilute ammonia or HCl as required. Transfer quantitatively into a 100 cm^3 volumetric flask and make up using distilled water. Calculate molarity of the solution.

Standardisation of EDTA: Pipette out 20 cm^3 of standard $CaCO_3$ solution into a 250 cm^3 conical flask and add 1 to 2 cm^3 of buffer solution. Add 2 or 3 small crystals (count! Do not add more than necessary to get a pale colour) of EBT and stir using a glass rod to get wine red colour. Titrate with EDTA solution, stirring after each addition, till the colour just changes to blue. Repeat to get concordant values.

Estimation of calcium: Make up the given calcium solution to 100 cm^3. Pipette out 20 cm^3 into a clean 250 cm^3 conical flask and titrate using EDTA exactly as above. Repeat to get concordant values. Calculate molarity, and hence mass of Ca^{2+} in the whole of the given solution.

Calculation:

Standardisation of EDTA: Mass of $CaCO_3$ weighed out = *w*. Molar mass of $CaCO_3$ = 100

Therefore molarity $M_1 = \frac{w}{100} \times \frac{1000}{100}$ = _____.

V_1 cm^3 of EDTA ≡ 20 cm^3 $CaCO_3$ solution. Therefore molarity M_2 of EDTA = $\frac{20 \times M_1}{V_1}$ = _____.

Estimation of Ca^{2+} *in sample*: V_2 cm^3 of EDTA ≡ 20 cm^3 $CaCO_3$ solution.

Therefore molarity M_3 of the Ca^{2+} soluion = $\frac{V_2 \times M_2}{20}$ = _____.

Molar mass of Ca^{2+} = 40.078

Therefore mass of Ca^{2+} in the whole of the given solution = $\frac{M_3 \times 40.078}{10}$ = _____.

Result:

Mass of Ca^{2+} in the whole of the given solution = _____ g.

ESIMATION OF MAGNESIUM

Aim of the experiment: To determine the mass of magnesium in the whole of the given solution.

Theory: Eriochrome Black T (= EBT) forms a wine-red coloured complex with Mg^{2+} ions in solution at a pH of about 10 (obtained by adding ammonia solution). EDTA forms a stronger complex with the Mg^{2+} ions and liberates free EBT, which has a blue colour. One mole of EDTA complexes with one mole of Mg^{2+} ions.

$$Na_2H_2EDTA + Mg^{2+} \rightarrow MgH_2EDTA(complex) + 2\ Na^+$$

Apparatus required:

(1) One 50 cm^3 burette.
(2) One 250 cm^3 conical flask.
(3) Two 100 cm^3 and one 250 cm^3 volumetric flasks.
(4) One 250 cm^3 Beaker.
(5) One 100 mL measuring cylinder.
(6) Dropper, glass rod and watch glass.

Chemicals required: (per student)

(1) EDTA disodium salt hydrate, Analar, 5 grams.
(2) $MgSO_4.7H_2O$, Analar, 2 grams.
(3) Eriochrome Black T indicator, a few crystals.
(4) Methyl orange indicator solution, 1 mL.
(5) Ammonium chloride, 2 g.
(6) Concentrated NH_3 solution, 15 mL
(7) Hydrochloric acid, 1:1, about 10 mL.

Preparation of reagents:

Note: Prepare all reagents using distilled water only! All preparations are to be recorded by students.

Buffer solution: Dissolve 17g of NH_4Cl in 150 cm^3 concentrated NH_3 solution in a 400 cm^3 beaker and dilute to 250 cm^3 with distilled water. Keep in a clean stoppered bottle. (Enough for all students)

EDTA solution: Weigh out about 4.65g of EDTA disodium salt hydrate into a 250 mL volumetric flask, add a little ammonia solution and about 200 mL of distilled water and swirl gently to dissolve completely (presence of ammonia makes dissolution of EDTA faster). Make up to the mark to get approximately 0.05 M EDTA solution.

Procedure:

Preparaion of standard 0.05M $MgSO_4$ solution: Accurately weigh out about 1.2324 g of analar $MgSO_4.7H_2O$ into a 100 cm^3 volumetric flask, dissolve and make up using distilled water. Calculate molarity of the solution.

Standardisation of EDTA: Pipette out 20 cm^3 of standard magnesium sulphate solution into a 250 cm^3 conical flask and add 1 to 2 cm^3 of buffer solution. Add 2 or 3 small crystals (count! Do not add more than necessary to get a pale colour) of EBT and stir using a glass rod to get wine red colour. Titrate with EDTA solution, stirring after each addition, till the colour just changes to blue. Repeat to get concordant values.

Estimation of magnesium: Make up the given magnesium solution to 100 cm^3. Pipette out 20 cm^3 into a clean 250 cm^3 conical flask and titrate using EDTA exactly as above. Repeat to get concordant values. Calculate molarity, and hence mass of Mg^{2+} in the whole of the given solution.

Calculation:

Standardisation of EDTA: Mass of $MgSO_4.7H_2O$ weighed out = *w*. Molar mass of $MgSO_4.7H_2O$ = 246.48

Therefore molarity $M_1 = \frac{w}{100} \times \frac{1000}{246.48}$ = _____.

V_1 cm^3 of EDTA ≡ 20 cm^3 Mg solution. Therefore molarity M_2 of EDTA = $\frac{20 \times M_1}{V_1}$ = _____.

Estimation of Mg^{2+} *in sample*: V_2 cm^3 of EDTA ≡ 20 cm^3 Mg^{2+} solution.

Therefore molarity M_3 of the Mg^{2+} soluion = $\frac{V_2 \times M_2}{20}$ = _____.

Molar mass of Mg^{2+} = 24.3050

Therefore mass of Mg^{2+} in the whole of the given solution = $\frac{M_3 \times 24.3050}{10}$ = _____.

Result:

Mass of Mg^{2+} in the whole of the given solution = _____ g.

ESIMATION OF ZINC

Aim of the experiment: To determine the mass of zinc in the whole of the given solution.

Theory: Eriochrome Black T (= EBT) forms a wine-red coloured complex with Zn^{2+} ions in solution at a pH of about 10 (obtained by adding ammonia solution). EDTA forms a stronger complex with the Zn^{2+} ions and liberates free EBT, which has a blue colour. One mole of EDTA complexes with one mole of Zn^{2+} ions.

$$Na_2H_2EDTA + Zn^{2+} \rightarrow ZnH_2EDTA(complex) + 2\ Na^+$$

Apparatus required:

(1) One 50 cm^3 burette.
(2) One 250 cm^3 conical flask.
(3) Two 100 cm^3 and one 250 cm^3 volumetric flasks.
(4) One 250 cm^3 Beaker.
(5) One 100 mL measuring cylinder.
(6) Dropper, glass rod and watch glass.

Chemicals required: (per student)

(1) EDTA disodium salt hydrate, Analar, 5 grams.
(2) $ZnSO_4.7H_2O$, Analar, 2 grams.
(3) Eriochrome Black T indicator, a few crystals.
(4) Methyl orange indicator solution, 1 mL.
(5) Ammonium chloride, 2 g.
(6) Concentrated NH_3 solution, 15 mL
(7) Hydrochloric acid, 1:1, about 10 mL.

Preparation of reagents:

Note: Prepare all reagents using distilled water only! All preparations are to be recorded by students.

Buffer solution: Dissolve 17g of NH_4Cl in 150 cm^3 concentrated NH_3 solution in a 400 cm^3 beaker and dilute to 250 cm^3 with distilled water. Keep in a clean stoppered bottle. (Enough for all students)

EDTA solution: Weigh out about 4.65g of EDTA disodium salt hydrate into a 250 mL volumetric flask, add a little ammonia solution and about 200 mL of distilled water and swirl gently to dissolve completely (presence of ammonia makes dissolution of EDTA faster). Make up to the mark to get approximately 0.05 M EDTA solution.

Procedure:

Preparaion of standard 0.05M $ZnSO_4$ ***solution***: Accurately weigh out about 1.4377 g of analar $ZnSO_4.7H_2O$ into a 100 cm^3 volumetric flask, dissolve and make up using distilled water. Calculate molarity of the solution.

Standardisation of EDTA: Pipette out 20 cm^3 of standard zinc sulphate solution into a 250 cm^3 conical flask and add 1 to 2 cm^3 of buffer solution. Add 2 or 3 small crystals (count! Do not add more than necessary to get a pale colour) of EBT and stir using a glass rod to get wine red colour. Titrate with EDTA solution, stirring after each addition, till the colour just changes to blue. Repeat to get concordant values.

Estimation of zinc: Make up the given zinc solution to 100 cm^3. Pipette out 20 cm^3 into a clean 250 cm^3 conical flask and titrate using EDTA exactly as above. Repeat to get concordant values. Calculate molarity, and hence mass of Zn^{2+} in the whole of the given solution.

Calculation:

Standardisation of EDTA: Mass of $ZnSO_4.7H_2O$ weighed out = *w*. Molar mass of $ZnSO_4.7H_2O$ = 287.54

Therefore molarity $M_1 = \frac{w}{100} \times \frac{1000}{287.54}$ = _____.

V_1 cm^3 of EDTA ≡ 20 cm^3 Zn solution. Therefore molarity M_2 of EDTA = $\frac{20 \times M_1}{V_1}$ = _____.

Estimation of Zn^{2+} *in sample*: V_2 cm^3 of EDTA ≡ 20 cm^3 Zn^{2+} solution.

Therefore molarity M_3 of the Zn^{2+} soluion = $\frac{V_2 \times M_2}{20}$ = _____.

Molar mass of Zn^{2+} = 65.39

Therefore mass of Zn^{2+} in the whole of the given solution = $\frac{M_3 \times 65.39}{10}$ = _____.

Result:

Mass of Zn^{2+} in the whole of the given solution = _____ g.

ESIMATION OF NICKEL

Aim of the experiment: To determine the mass of nickel in the whole of the given solution.

Theory: Eriochrome Black T (= EBT) forms a wine-red coloured complex with Ni^{2+} ions in solution at a pH of about 10 (obtained by adding ammonia solution). EDTA forms a stronger complex with the Ni^{2+} ions and liberates free EBT, which has a blue colour. One mole of EDTA complexes with one mole of Ni^{2+} ions.

$$Na_2H_2EDTA + Ni^{2+} \rightarrow NiH_2EDTA(complex) + 2\ Na^{+}$$

Apparatus required:

(1) One 50 cm^3 burette.
(2) One 250 cm^3 conical flask.
(3) Two 100 cm^3 and one 250 cm^3 volumetric flasks.
(4) One 250 cm^3 Beaker.
(5) One 100 mL measuring cylinder.
(6) Dropper, glass rod and watch glass.

Chemicals required: (per student)

(1) EDTA disodium salt hydrate, Analar, 5 grams.
(2) $NiSO_4.7H_2O$, Analar, 2 grams.
(3) Eriochrome Black T indicator, a few crystals.
(4) Methyl orange indicator solution, 1 mL.
(5) Ammonium chloride, 2 g.
(6) Concentrated NH_3 solution, 15 mL
(7) Hydrochloric acid, 1:1, about 10 mL.

Preparation of reagents:

Note: Prepare all reagents using distilled water only! All preparations are to be recorded by students. Reaction of Ni^{2+} with EDTA is slow. So warm the solution and titrate slowly.

Buffer solution: Dissolve 17g of NH_4Cl in 150 cm^3 concentrated NH_3 solution in a 400 cm^3 beaker and dilute to 250 cm^3 with distilled water. Keep in a clean stoppered bottle. (Enough for all students)

EDTA solution: Weigh out about 4.65g of EDTA disodium salt hydrate into a 250 mL volumetric flask, add a little ammonia solution and about 200 cm^3 of distilled water and swirl gently to dissolve completely (presence of ammonia makes dissolution of EDTA faster). Make up to the mark to get approximately 0.05 M EDTA solution.

Procedure:

Preparaion of standard 0.05M $NiSO_4$ ***solution***: Accurately weigh out about 1.4035 g of analar $NiSO_4.7H_2O$ into a 100 cm^3 volumetric flask, dissolve and make up using distilled water. Calculate molarity of the solution.

Standardisation of EDTA: Pipette out 20 cm^3 of standard nickel sulphate solution into a 250 cm^3 conical flask and warm to about 50°C. Add 1 to 2 cm^3 of buffer solution. Add 2 or 3 small crystals (count! Do not add more than necessary to get a pale colour) of EBT and swirl to get a uniform wine red colour. Titrate slowly with EDTA solution, swirling after each addition, till the colour just changes to blue. Repeat to get concordant values.

Estimation of nickel: Make up the given nickel solution to 100 cm^3. Pipette out 20 cm^3 into a clean 250 cm^3 conical flask and titrate using EDTA exactly as above. Repeat to get concordant values. Calculate molarity, and hence mass of Ni^{2+} in the whole of the given solution.

Calculation:

Standardisation of EDTA: Mass of $NiSO_4.7H_2O$ weighed out = *w*. Molar mass of $NiSO_4.7H_2O$ = 280.69

Therefore molarity $M_1 = \frac{w}{100} \times \frac{1000}{280.69}$ = _____.

V_1 cm^3 of EDTA ≡ 20 cm^3 Ni solution. Therefore molarity M_2 of EDTA = $\frac{20 \times M_1}{V_1}$ = _____.

Estimation of Ni^{2+} *in sample*: V_2 cm^3 of EDTA ≡ 20 cm^3 Ni^{2+} solution.

Therefore molarity M_3 of the Ni^{2+} soluion = $\frac{V_2 \times M_2}{20}$ = _____.

Molar mass of Ni^{2+} = 58.69

Therefore mass of Ni^{2+} in the whole of the given solution = $\frac{M_3 \times 58.69}{10}$ = _____.

Result:

Mass of Ni^{2+} in the whole of the given solution = _____ g.