INORGANIC AND QUALITATIVE ANALYTICAL CHEMISTRY
(Laboratory course for students of pharmacy)
2011/2012 2nd semester

The **laboratory course** of 84 hours consists of seminars (1 class hours per week total 14 hours) and real laboratory practices (5 hours per week total 70 hours). The course is given during 14 weeks. The first introductory practice takes 5 hours as well, because some demonstration will be given after the general instruction part.

In the seminars the theoretical background of the laboratory investigations and some special or particular problems of analytical operations of the current experiments are discussed. The practices help students to get knowledge of material and to have training in the qualitative analytical laboratory operations and in compilation of laboratory reports. It is not allowed to miss any laboratory practicies/seminars. If a student misses one or two lab practicies (medical sertification is needed form the doctor), she or he has to participate at a make-up laboratory practice. If a student misses three lab practices/seminars even for any medical reason, the student's lecture book won't be signed and she or he has to retake the course next year.

In the first four practices some experiments and test tube reactions relating mostly to inorganic chemistry are required to perform. From Practice 5 the sequence of the analytical topics follows the classical Fresenius' system. In the first part of the practices it is required to obtain some skills and experiences in the identification and separation of the relevant species. This work is followed by the analysis of "unknown samples". Sometimes special experiments are performed collectively by small teams (team study). The demonstration experiments are similar. In these cases the experiments are supervised by the teacher. Some purity tests were taken from the official European Pharmacopoeia or Hungarian Pharmacopoeia. Students who finish the actual practice sooner can analyse an extra "voluntary test", too.

At the beginning of every practice the students are required to write a test relating to the theoretical background and practical questions of the current experiments. For these tests and for the analysis of unknown samples, grades are given. The purity tests are qualified as "acceptable" or "not acceptable". The final qualification is determined by the grades and by the quality of the laboratory reports. Depending on the qualification of purity tests and the volume of voluntary tests, the final grade can be rounded.

Recommended reading

1 Inorganic and Qualitative Analytical Chemistry,

Supplementary material for laboratory course for students of pharmacy and Chemical Engenniers.

Compiled by Róbert Király and Gábor Lente

Department of Inorganic and Analytical ChemistryFaculty of Science, University of Debrecen

Debrecen, 2011 … (in the text indicated as: S.I. + Practice #))

2 G. Svehla (reviser)

Vogel's Qualitative Inorganic Analysis

Sixth edition

Longman Scientific & Technical

Copublished in the United States with John Wiley & Sons, Inc.,

New York, 1994

ISBN 0-582-45090-X
ISBN 0-470-20710-8 (USA only)

3 N. N. Greenwood and A. Earnshaw
Chemistry of the Elements
Butterworth-Heinemann
Reed Educational and Professional Publishing Ltd, 2 ed, 1997
ISBN 0-7506-3365-4

4 J. McMurry, R.C. Fay
Chemistry
Fourth Edition
Pearson Education International, Prentice Hall
New Jersey, 2004
ISBN 0-13-121631-7

5 H. F. Holtzlaw, Jr., W. R. Robinson
College Chemistry with Qualitative Analysis
Eighth edition
D. O. Heath and Company,
Lexington, Massachusetts, Toronto, 1988
ISBN 0-669-12862-7

DETAILED PROGRAMME

Practice 1 *(2012. Feb. 08.)*. The chemistry will be discussed on the 1st seminar *(2012. Feb. 06.)*

1. Inorganic and analytical laboratory rules ***(exposition).***
2. Laboratory safety ***(exposition).***
3. Distribution of laboratory equipment.
4. Reaction of potassium chlorate with sulphur and red phosphorus ***(demonstration) (S.I. Practice 1).***
5. Reaction of hydrogen sulfide with sulfur dioxide ***(demonstration) (S.I. Practice 1).***
6. Preparation of solutions of ammonium sulfide and polysulfide The decomposition of polysulfide ***(demonstration) (S.I. Practice 1).***
7. Laboratory preparation of hydrogen with the use of Kipp-apparatus and combustion of hydrogen ***(reading) (S.I. Practice 1).***

Demonstrations taken from the lectures

8. Reaction of potassium chlorate with sulfur and confectionery sugar (GEL 2.24).
9. Detection and confirmation of SO_2 and H_2S gases (SO_2 + KIO_3, H_2S + $Pb(NO_3)_2$ and PbS + H_2O_2)).

Practice 2 *(2012. Feb. 15.)* The chemistry will be discussed on the 2nd seminar *(2012. Feb. 13.)*

1. Laboratory preparation of chlorine and its reaction with metals ***(team study) (S.I. Practice 2).***
2. Preparation of chlorine by reating NaClO (hypo) with HCl ***(reading) (S.I. Practice 2).***

3. Reaction of alkali-chroides, -bromides and ioidides with concentrated (cc) H_2SO_4 ***(S.I. Practice 2).***
4. Reactions of hypochlorite ion ***(S.I. Practice 2).***
5. Laboratory preparation of oxygen gas ***(team study) (S.I. Practice 2).***
6 Combustion of elements in oxygen ***(team study) (S.I. Practice 2).***
7. Reactions of hydrogen peroxide ***(S.I. Practice 2).***
8. Preparation and reactions of hydrogen sulfide ***(S.I. Practice 2).***
9. Chemical properties of sulfurous and sulfuric acid ***(S.I. Practice 2).***

Demonstrations taken from the lectures

11. Preparation of peroxymonosulfuric (Caro's) acid and its strong oxidizing properties (GEL 3.25).
12. Allotropic modifications of sulfur (GEL 3.10).
13. Preparation and colour of chromium(VI) peroxide ($CrO(O_2)_2$) (GEL 9.34e).

Practice 3 *(2012. Feb. 22.)*. The chemistry will be discussed on the 3rd seminar *(2012. Feb. 20.)*

1. Laboratory preparation of nitrogen ***(S.I. Practice 3).***
2. Chemical properties of ammonia, oxidation of NH_3 by halogens. ***(team study) (S.I. Practice 3).***
3. Preparation and study of nitrogen monoxide ***(team study) (S.I. Practice 3).***
4. Preparation and chemical properties of nitric acid and nitrates ***(S.I. Practice 3).***
5. Experiments with phosphorus and with phosphorus pentoxide ***(S.I. Practice 3).***

Demonstrations taken from the lectures

6. Preparation and properties of O_3 (LB 16.6).
7. Ammonia fountain (LP).

Practice 4 *(2012. Feb. 29.)*. The chemistry will be discussed on the 4th seminar *(2012. Feb. 27.)*

1. Properties of carbon dioxide ***(team study) (S.I. Practice 4).***
2. Preparation and propertietudy of carbon monoxide ***(team study) (S.I. Practice 4).***
3. Experiments with boric acid and reactions of borate ion ***(S.I. Practice 4).***
4. Reactions of alkali and alkaline earth metals with water ***(team study) (S.I. Practice 4).***
5.Solution of alkali and alkaline earth metals in liquid ammonia ***(demonstration) (S.I. Practice 4).***
6. Interaction of aluminium, lead and tin with acids and alkalies ***(S.I. Practice 4).***
7. Interaction of iron, copper and zinc with acids and alkalies ***(S.I. Practice 4).***

Demonstrations taken from the lectures

8. Cooling in the laboratory, cooling mixtures (solid CO_2 – acetone cooling mixture).

Practice 5 *(2012. Mar. 07.)*. The chemistry will be discussed on the 5th seminar *(2012. Mar. 05.)*

1. Practical classification of reactions and ions.
2. The reactions of anions.
3. The analysis of anion group I (carbonate, hydrogen carbonate, silicate, sulfide, polysulfide and sulfite ions).
4. Identification of halogenate ions.

5. Purity tests: Investigation of bromate impurity in potassium bromide.

Purity tests

6. Investigation of bromate impurity in potassium bromide.

Practice 6 ***(2012. Mar. 14.).*** **The chemistry will be discussed on the 6th seminar** ***(2012. Mar. 12.)***

1. The analysis of anion group II (phosphate, sulfate, fluoride, bromate and iodate ions).
2. The analysis of anion group III (chloride, bromide and iodide ions).
3. Removal of orthophosphate ions from aqueous solutions ***(team study).***
4. „Eaching test" ***(demonstration).***

Unknown sample

5. Detection of an anion of group I-II in a solid salt of an alkali metal (CO_3^{2-} ; HCO_3^{-} ; S^{2-} ; SO_3^{2-} ; SO_4^{2-} ; PO_4^{3-} (HPO_4^{2-} ; $H_2PO_4^{-}$); F^{-} ; BrO_3^{-} ; IO_3^{-}).

Voluntary test

– The same as unknown sample, but solution is given.

Demonstrations taken from the lectures

6. Color of halogens and their aqueous solutions.

7. Color of I_2 in aqueous solution and in organic solvents (iodine water + $CHCl_3$ in comparison with $FeCl_3$ + $CHCl_3$).

8. Color of the-starch-iodine complex (the iodine test for starch).

9. Eacing test (detection of F^{-} ions) (GEL 2.14c).

Practice 7 ***(2012. Mar. 21.).*** **The chemistry will be discussed on the 7th seminar** ***(2012. Mar. 19.)***

1. Identification of bromide and iodide ions coexisting in solution with the use of chlorine water.
2. Identification of chloride ion in the presence of bromide or/and iodide (Berg's reaction).

Unknown sample

3. Detection of two anions of group I-III in a solution of two alkali metal salts (CO_3^{2-} S^{2-} ; SO_3^{2-} ; SO_4^{2-} ; PO_4^{3-} (HPO_4^{2-} ; $H_2PO_4^{-}$); F^{-} ; BrO_3^{-} ; IO_3^{-} ; Cl^{-} ; Br^{-} ; I^{-} ; SO_3^{2-} and SO_4^{2-} ions do not coexist).

Voluntary test

– Detection of one or two anions of group I-III in solution of two alkali metal salts (CO_3^{2-} S^{2-} ; SO_3^{2-} ; SO_4^{2-} ; PO_4^{3-} (HPO_4^{2-} ; $H_2PO_4^{-}$); F^{-} ; BrO_3^{-} ; IO_3^{-} ; Cl^{-} ; Br^{-} ; I^{-} ; SO_3^{2-} and SO_4^{2-} ions do not coexist).

Practice 8 ***(2012. Mar. 28.).*** **The chemistry will be discussed on the 8th seminar** ***(2012. Mar. 26.)***

1. The analysis of anion group IV (nitrite, nitrate and chlorate ions).
2. Detection of nitrite and nitrate ions with Griess-Ilosvay reagent.

Unknown sample

3. Detection of two anions of group I–IV in a mixture of two alkali metal salts (CO_3^{2-} S^{2-} ; SO_3^{2-} ; SO_4^{2-} ; PO_4^{3-} (HPO_4^{2-} ; $H_2PO_4^-$); F^- ; BrO_3^- ; IO_3^- ; Cl^- ; Br^- ; I^- ; NO_2^- and NO_3^-). The pairs of : SO_3^{2-} – SO_4^{2-} ; Br^- – NO_3^- and I^- – NO_3^- are not given).

Voluntary test

4. The same as unknown sample, but solution is given.

Demonstrations taken from the lectures

5. Preparation of nitrous acid (cc. NO_2^- solution + ice + HCl) (GEL 30a).
6. The brown ring test for nitrit ions (GEL 4.29c).
7. The Gries-Ilosvay reaction for the nitrit ions (GEL 4.31).
8. Reaction of chlorate ions with cc. H_2SO_4 (GEL 4.31).

Practice 9 *(2011. Apr. 04.)*. The chemistry will be discussed on the 9th seminar *(2012. Apr. 02.)*

1. The reactions of cations
2. The analysis of cation group I and group IIA (Copper(II), silver(I), cadmium(II), mercury(I), mercury(II), lead(II) and bismuth(III) ions).

Purity test

3. Investigation of lead impurity in boric acid.

No practice will be held on *2012. April 11*.

Practice 10 *(2012. Apr. 18.)*. The chemistry will be discussed on the 10th seminar *(2012. Apr. 16.)*

1. Sanger – Black's test for trace analysis of arsenic impurity in solution ***(demonstration)*.**

Purity test

2. Investigation of silver impurity in "bismuth subnitrate, heavy".

Unknown sample

3. Detection of two cations of group I or IIA in a solution (Ag^+, Cd^{2+}, Cu^{2+}, Hg_2^{2+}, Hg^{2+}, Pb^{2+}, Bi(III) (Hg_2^{2+} – Hg^{2+} and Cu^{2+} – Hg_2^{2+} ions are not given together).

Voluntary test

– Detection of one or two cations of group I and IIA in solution (Hg_2^{2+} – Hg^{2+} and Cu^{2+} – Hg_2^{2+} ions are not given together).

Demonstrations taken from the lectures

4. The reactions of cations of cation group I and group IIA (Copper(II), silver(I), cadmium(II), mercury(I), mercury(II), lead(II) and bismuth(III) ions) with KI and $K_2Cr_2O_7$.

Practice 11 *(2012. Apr. 25.)*. The chemistry will be discussed on the 11th seminar *(2012. Apr. 23.)*

1. The analysis of cation group IIB (arsenic(III), arsenic(V), antimony(III), antimony(V), tin(II) and tin(IV)).
2. Reactions of permanganate, chromate and dichromate ions ***(S.I. Practice 11).***
3. Preparation and properties cyanide complexes of some transition metal ions ***(S.I. Practice 11).***
4. Oxydation states of transition metals belonging to 3d row in aqueous solutions ***(S.I. Practice 11).***
5. Use of organic reactions in analysis ***(S.I. Practice 11).***

Purity test

6. Investigation of iron impurity in citric acid.

Practice 12 *(2012. May 02.)*. The chemistry will be discussed on the 12th seminar *(??? 2012. Apr. 23., 2012. Apr. 30. or other date).*

1. The analysis of cation group III (nickel(II), cobalt(II), iron(II), iron(III), manganese(II), chromium(III), zinc(II) and aluminium(III) ions).
2. "Fluoride test" for aluminium ***(demonstration).***
3. Detection of traces of nickel in cobalt salts.

Unknown sample

4. Detection of two cations of group III in solution (the oxidation state of Fe and Cr can be +3, and the oxidation state of Mn can be +2 only).

Voluntary test

– Detection of one or two cations of group III in solution (the oxidation state of Fe and Cr can be +3, and the oxidation state of Mn can be +2 only).

Practice 13 *(2012. May 09.)*. The chemistry will be discussed on the 13th seminar *(2012. May. 07.)*

1. The analysis of cation group IV (calcium(II), strontium(II) and barium(II) ions).
2. The analysis of cation group V (magnesium(II), lithium(I), sodium(I), potassium(I) and ammonium ions).
3. Reaction of Sr^{2+} and Ba^{2+} ions with sodium rhodizonate ***(S.I. Practice 11).***
4. Salts of alkali metal ions with poor solubility in water ***(S.I. Practice 11).***
5. Detection of traces of ammonia ***(demonstration).***

Unknown sample

6. Detection of two cations of group I, IIA, III, IV or V in solution (**One** component is a cation of group I, IIA or III (Cu^{2+} ; Ag^{+} ; Cd^{2+} ; Hg_2^{2+} ; Hg^{2+} ; Pb^{2+} ; Bi(III) ; Ni^{2+} ; Co^{2+} ; Fe^{2+} ; Fe^{3+} ; Mn^{2+} ; Cr^{3+} ; Zn^{2+} ; Al^{3+}) and the **other one** is a cation of group IV or V (Ca^{2+} ; Sr^{2+} ; Ba^{2+} ; Li^{+} ; Na^{+} ; K^{+} ; NH_4^{+}). The oxidation state of Cr is +3, and the oxidation state of Mn is +2. Fe can be in oxidation state +2 or +3).

Voluntary test

– The same as the unknown sample (solution is given).

Demonstrations taken from the lectures

7. Preparation and colour of chromium(VI) peroxide ($CrO(O_2)_2$) (GEL 9.34e).

Practice 14 ***(2012. May 16.).*** **The chemistry will be discussed on the 14th seminar** ***(2012. May. 14.)***

1. Summary on group reactions.
2. Complete qualitative analysis of a solid sample.

Unknown sample

3. Complete qualitative analysis (cations, anions) of a solid mixture of two components. The cations or the anions in the two components are the same. This way **the number of the detectable ions is 3.**

The same **cations** can be in the sample which were investigated formerly (Cu^{2+} ; Ag^{+} ; Cd^{2+} ; Hg^{2+} ; Pb^{2+} ; Bi(III) ; Ni^{2+} ; Co^{2+} ; Fe^{3+} ; Mn^{2+} ; Cr^{3+} ; Zn^{2+} ; Al^{3+} ; Ca^{2+} ; Sr^{2+} ; Ba^{2+} ; Li^{+} ; Na^{+} ; K^{+} ; NH_4^{+}), but Mg^{2+} is not given, and also two cations of group IV and of group V can not be together. The oxidation state of Hg, and Mn can be +2 only, oxidation state of Fe and Cr can be +3.

The possible **anions** are as follows : CO_3^{2-} (HCO_3^{-}) ; SO_4^{2-} ; PO_4^{3-} (HPO_4^{2-} , $H_2PO_4^{-}$); F^{-} ; Cl^{-} ; Br^{-} ; I^{-} ; NO_3^{-} The various protonated forms of the anions cannot be identified.

4. Inventory and return of laboratory equipments.