INORGANIC AND QUALITATIVE ANALYTICAL CHEMISTRY
(Laboratory practice and seminar)
2016/2017 2nd semester

This **course** of 78 hours consists of seminars (1 hour per week, sum. 13 hours) and laboratory practices (5 hours per week, sum. 65 hours). The course is given during 13 weeks. Even the first introductory practice takes 5 hours, because some demonstrations will be given after the general introduction part.

The theoretical background of laboratory experiments and some special or practical problems are discussed in the seminars. Students are required to obtain knowledge in the field of inorganic chemistry and training in qualitative analytical laboratory operations. Students are also required to individually document their work in the form of laboratory reports. Students are not allowed to miss any laboratory practices or seminars. If a student misses one lab practice because of medical reasons (certified by an MD), he or she has to participate in a make-up laboratory practice (organized at the end of the semester). **If a student misses two lab practices or seminars even for medical reasons**, the student's lecture book will not be signed and has to retake the full course.

In the first four laboratory practices some experiments and test tube reactions relating mostly to inorganic chemistry will be performed. From Practice 5 the sequence of selected topics from analytical chemistry will follow the classical system of Fresenius. In the first part of the practices students are required to experiment individually on the identification and separation of inorganic anions and cations. This work is followed by the analysis of "unknown samples". Sometimes special experiments are performed by teams of 4 students (team study). Demonstration experiments are similar to team studies but in this case the experiments are supervised by an instructor. Some purity tests are taken from the official European Pharmacopoeia or Hungarian Pharmacopoeia. Students who complete all mandatory tasks earlier before the end of the laboratory practice can volunteer to anaylse extra unknown samples.

Each week the laboratory practice starts with a short test (not more than 25 minutes) based exclusively on the material of the actual week and that of the previous week. Results of the experiments carried out the previous week can be also asked. Each short test worth 30 points. Altogether 12 short tests will be written during the semester totalling 360 points. The average score from the short tests have to be above 50 % (more than 180 points) to earn "pass (2)" grade. In addition to collecting minimum 180 points from the short tests, the average grade earned for analyzing unknown samples have to be minimum 2.00 in order to successfully complete the course. The purity tests will be graded "acceptable" or "not acceptable", and the result of the four purity tests will form a single grade which will be averaged together with the results of the unknown samples. The final grade for the course is determined by the average result of the short tests and that of the unknown samples. The quality of the laboratory reports will be assessed to decide uncertain grades. Students with "fail (1)" final course grades (A exam) thanks to unacceptable test results can take a comprehensive test exam during the examination period once (B exam). **Please note:** students with results lower than 35% (126 points) from short tests or having failed to reach the 2.00 average for analysis of unknown samples cannot take a final exam, will receive a 'signature denied' final course grade and will need to retake the course.

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 ***(2017. Feb. 22.).*** **The chemistry will be discussed on the 1st seminar** ***(2017. Feb. 20.)***

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 ***(demonstration) (S.I. Practice 1).***

Demonstrations taken from the lectures

8. Reaction of melted potassium chlorate with gummy bear.
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 ***(2017. Mar. 1.).*** **The chemistry will be discussed on the 2nd seminar** ***(2017. Feb. 27.).***

1. Laboratory preparation of chlorine and its reaction with metals ***(team study, the chlorine gas is taken form cylinder) (S.I. Practice 2).***
2. Preparation of chlorine by reacting 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. Chemical properties of sulfurous and sulfuric acid ***(S.I. Practice 2).***

Demonstrations taken from the lectures

9. Preparation of peroxymonosulfuric (Caro's) acid and its strong oxidizing properties

Practice 3 ***(2017. Mar. 8.).*** **The chemistry will be discussed on the 3rd seminar** ***(2017. Mar. 6.)***

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 ***(2017. Mar. 22.).*** **The chemistry will be discussed on the 4th seminar** ***(2017. Mar. 20.)***

1. Properties of carbon dioxide ***(team study) (S.I. Practice 4).***
2. Preparation and properties of carbon monoxide ***(reading) (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 ***(2017. Mar. 29.).*** **The chemistry will be discussed on the 5th seminar** ***(2017. Mar. 27.)***

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 ***(2017. Apr. 5.).*** **The chemistry will be discussed on the 6th seminar** ***(2017. Apr. 3.)***

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. „Etching test" ***(demonstration).***
5. Reactions of the members of II[nd] anion group with $[Fe(SCN)_4]^-$ ***(demonstration).***

Unknown sample

6. 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

7. Color of halogens and their aqueous solutions.
8. Color of the-starch-iodine complex (the iodine test for starch).

Practice 7 ***(2017. Apr. 12.).*** **The chemistry will be discussed on the 7th seminar** ***(2017. Apr. 10.)***

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 ***(2017. Apr. 19.).*** **The chemistry will be discussed on the 8th seminar** ***(The exact date and place of the 8th seminar will be disclosed later.)***

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)
6. The brown ring test for nitrite ions
7. The Griess-Ilosvay reaction for the nitrite ions
8. Reaction of chlorate ions with cc. H_2SO_4

Practice 9 ***(2017. Apr. 26.).*** **The chemistry will be discussed on the 9th seminar** ***(2017. Apr. 24.)***

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.
4. Investigation of silver impurity in “bismuth subnitrate, heavy”.

Practice 10 ***(2017. May 3.).*** **The chemistry will be discussed on the 10th seminar** ***(The exact date and place of the 10th seminar will be disclosed later.)***

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

Purity test

2. Investigation of iron impurity in citric acid.

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 ***(2017. May 10.).*** **The chemistry will be discussed on the 11th seminar** ***(2017. May 8.)***

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.
4. Preparation and properties of cyanide complexes of some transition metal ions ***(S.I. Practice 11).***
5. Use of organic reactions in analysis: determination of Fe^{2+} and Fe^{3+} ions with 2,2'-dipyridyl reagent, determination of Ni^{2+} ions with dimethylglyoxime reagent and determination of Zn^{2+} ions with dithizone.***(S.I. Practice 11).***

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 12 ***(2017. May 17.).*** **The chemistry will be discussed on the 12th seminar** ***(2017. May. 15.)***

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 12).***
4. Salts of alkali metal ions with poor solubility in water ***(S.I. Practice 12).***
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 ($CrO(O_2)_2$) (**V**.III.24.8.b.).*

Practice 13 ***(2017. May 24.).*** **The chemistry will be discussed on the 13th seminar** ***(2017. May 22.)***

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.