INORGANIC AND QUALITATIVE ANALYTICAL CHEMISTRY (TKBL0511_EN) (Laboratory Practice) for chemical engineering students

This course is consists of laboratory practices (11 weeks, 4 hours per week) and seminars (4 weeks, 1 hour per week).

The objective of this laboratory practice to introduce students to inorganic and analytical laboratory work. In the first part of the semester some test tube reactions relating mostly to inorganic chemistry will be performed and qualitative analysis of some unknown sample will be carried out. These lab practices are accompanied by seminars. From the 6th week analytical topics will be covered relating to the acid-base, redox, precipitation and complex titration. The aim of this work is to gain experience in basic analytical techniques, their implementation and the evaluation of experimental results.

Syllabus lists the tasks in weekly schedule. Descripition of the classical analytical part can be downloaded from the following website: http://www.inorg.unideb.hu/ **First, click on the heading to the left: "OKTATÁS". Second, log in under the brown heading: "Bejelentkezés". You should give the "Felhasználónév" (username): *analitika09*. There is no password, just click on the button: "Belépés". In the list of the subjects under "OKTATÁS" look for: "Inorganic and Qualitative Analytical Chemistry".**

Students are not allowed to miss any laboratory practices or seminars. If a student misses one lab or seminar, medical certification is needed and the practice should be replaced as far as possible. In case of two or more missing practice the course won't be accepted, student will automatically fail the course with a signature denied final mark. In this case she or he has to retake the course next year.

Each week the laboratory practice starts with a short test (not more than 20 minutes). It is based on the theoretical material of the actual week, moreover on the whole material of the previous week including the theoretical background, observations and explanations of the experiments. Unknown samples relating both to quantitative and qualitative determinations will also be marked.

Students must register their observations regarding the experiments carried out in their lab manual. Instructors will check the lab manuals at the beginning of the lab and if it is not with them or is not prepared, after oral questions instructors can ask the student to leave the laboratory and it is taken as an absence.

Grading is based on a five-level scale. The final course grade is based on the results of the short tests and unknown samples. Quality of the lab manual and laboratory work will be also taken into consideration when grading.

Requirements to complete the laboratory practice:

1. Attendance at all of the seminars and lab sessions.
2. Preparation of the lab manuals for the practice and recording the observations.
3. Determination of all the unknown samples.
4. Average of the unknown samples should be at least 2.0.
5. Average of the short tests should be at least 2.0.

If one of the 1-4 requirements is not completed the course cannot be accepted, he will receive a 'signature denied' final course grade and it should be re-take next year. If the 5. requirements is

not completed, the final course grade will be “fail (1)”, but if the average of the sort tests is > 1.5, the student has one time opportunity to improve (get pass(2)) in the examination period. The date of this written test from the whole material of the semester is on 5th January, 2020 at 1 pm.

Recommended reading

1. Inorganic and Qualitative Analytical Chemistry (Lectures)
2. G. Svehla (reviser)

   Vogel’s Qualitative Inorganic Analysis (in the text indicated as: V + page number + paragraph)

   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. Inorganic Chemistry Laboratory Manual
4. 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
5. J. McMurry, R. C. Fay

   Chemistry

   Fourth Edition

   Pearson Education International, Prentice Hall

   New Jersey, 2004. ISBN 0-13-121631-7

WEEKLY SCHEDULE

Practice 1 (14th September)

1. Inorganic and analytical chemistry laboratory rules
2. Laboratory safety
3. Distribution of laboratory pieces of equipment

Seminar 1 (17th September)

Reactions of anion groups I-IV
Anion groups I. and II: The reactions of carbonate, sulfide, sulfate and fluoride ions.

Practice 2 (21st September)

1. The analysis of anion groups I. and II. (V. 297 – 308, 332 – 334, 337 – 340, 346 – 349, 354 – 358)
2. The reactions of carbonate (V. 298. 1,2), hydrogen-carbonate (V. 300, 1, 2, 5), sulfide (V. 308, 1, 2, 3, 6) and sulfite ions (V. 301, 1, 2, 4, 7).
3. The reactions of sulfate (V. 346, 1, 2) and fluoride (V. 332, 2, 4) ions.
4. **Unknown sample:**
   Detection of an anion of group I-II. (CO_3^{2-}(HCO_3^-); S^{2-}; SO_3^{2-}; SO_4^{2-}; F^-)

Seminar 2 (24th September)

Anion groups III-IV: The reactions of chloride, bromide, iodide, nitrite and nitrate ions.

Practice 3 (28st September)

1. The analysis of anion groups III. and IV (V. 310 – 313, 325 – 332, 334 – 337).
2. The reactions of nitrite (V. 310, 1,2, 5, 6, 11) and nitrate ions (3, 4, 8).
3. The reaction of chloride (V. 325, 3, 4), bromide (V. 327, 1, 3, 5) and iodide ions (V. 329, 1, 2, 4, 6, 7, 8).
4. **Unknown sample:**
   Detection of two anions of group III-IV in a solution (NO_2^-; NO_3^-; Cl^-; Br^-; I^-; $Br^- – NO_3^-$ and $I^- – NO_3^-$ are not given).

Seminar 3 (1st October)

Classification of cations according to the Fresenius' system.
The reactions of cation group I: copper(II), silver(I), cadmium(II), mercury(I), mercury(II), lead(II) and bismuth(III) ions

Week 4 (5th October)

1. The reaction of cation group III.
2. The reaction of cation group I and II. (V. 193 – 223)
3. The analysis of cation group IV. (V. 277 - 285) (*reading*)
4. The reactions of cation group I. with iodide ions
5. The reactions of copper(II) (V. 215, 1, 2, 3, 4), silver(I) (V. 204, 1, 2, 3, 4, 5, 6), cadmium(II) (V. 221, 1, 2, 3, 6), mercury(I) (V. 200, 1, 2, 3, 4, 5, 6, 10), mercury(II) (V. 209, 1, 2, 3, 4, 6), lead(II) (V. 194, 1, 2, 3, 4, 5, 6, 7) and bismuth(III) (V. 212, 1, 2, 3, 4).
6. **Unknown sample:**
   Detection of two cations of group I and II in solution (Ag^+; Cd^{2+}; Hg^{2+}; Hg_2^{2+}; Pb^{2+}; Bi^{3+}; $Hg_2^{2+} – Hg^{2+}$ ions are not given).

Seminar 4 (8st October)

The reactions of cation group III: nickel(II), cobalt(II), iron(III), manganese(II), chromium(III) and zinc(II) ions

Week 5 (12th October)

1. The reaction of cation group III (V. 241 - 277).
2. The analysis of cation group V. (V. 285 - 297) (*reading*)
3. The reactions of nickel(II) (V. 264, 1, 2, 3, 8), cobalt(II) (V. 259, 1, 2, 3, 6), iron(III) (V. 245, 1, 2, 4, 5, 6, 7, 8, 11), manganese(II) (V. 268, 1, 2, 3, 5, 6, 7), chromium(III) (V. 254, 1, 2, 4, 7 (a, b, d), 8 (a, b)) and zinc(II) (V. 272, 1, 2, 3, 12).
4. **Unknown sample:**
   Detection of two cations of group I, II and III (Cu^{2+}; Cd^{2+}; Hg^{2+}; Co^{2+}; Ni^{2+}; Zn^{2+}; Mn^{2+}; Fe^{3+}; Cr^{3+}).

Week 6 (19th October)

1. Preparation of ~0.1 mol/dm^3 HCl titrant (250 cm^3). (in Manual: site 1, part *a*)
2. Determination of the exact concentration of the HCl titrant solution using $KHCO_3$ stock solution. (in Manual: site 1, part *b*)
3. Determination of borax (sodium tetraborate) content of a solid sample /**unknown sample**/. (in Manual: site 4)
4. Preparation of ~0.1 mol/dm^3 NaOH titrant by the Sörensen method (250 cm^3) and determination of its exact concentration. (in Manual: site 1, part *a*)

Week 7 (26th October)

1. NaOH titrant by the Sörensen method (250 cm^3) and Determination of the exact concentration of the NaOH titrant (in Manual: site 1, part *b*)
2. Determination of **oxalic acid** /**unknown sample**/ (in Manual: site 3)
3. Simultaneous determination of **sulfuric acid and boric acid** in a mixture /**unknown sample**/ (in Manual: site 3-4)

Week 8 (9th November)

1. Argentometric determination of the composition of a **NaCl-KBr** mixture /**unknown sample**/. (in Manual: site 15)
2. Determination of **ascorbic acid** content in a mixture /**unknown sample**/ (in Manual: site 10)
3. Preparation of 0.02 mol/dm^3 $KMnO_4$ titrant (250.0 cm^3). (in Manual: site 7, part *a*)
4. Preparation of 0.05 mol/dm^3 $Na_2(COO)_2$ solution (100.0 cm^3).

Week 9 (16th November)

1. Determination of the exact concentration of the $KMnO_4$ titrant solution using $Na_2(COO)_2$ stock solution. (in Manual: site 7, part *b*)
2. Determination of **ferrous oxalate** /**unknown sample**/. (in Manual: site 9)
3. Determination of **hydrogen peroxide** /**unknown sample**/. (in Manual: site 9)

Week 10 (23rd November)

1. Preparation of 0.02 mol/dm^3 $Na_2S_2O_3$ titrant (250.0 cm^3) and determination of its exact concentration using 0.003 mol/dm^3 KIO_3 stock solution. (in Manual: site 10-11)
2. Determination of **copper(II)** /**unknown sample**/. (in Manual: site 11)

Week 11 (30th November)

1. Preparation of 0.01 mol/dm^3 Na_2EDTA titrant solution (250.0 cm^3). (in Manual: site 16)
2. Determination of **Bi(III)** /**unknown sample**/.
3. Lab equipment return