Inorganic chemistry III (TTKBL0201en) laboratory practice

Required literature:

József Emri, Béla Győri, István Lázár:
Inorganic Chemistry Laboratory Manual (ed. István Lázár, University of Debrecen, Department of Inorganic and Analytical Chemistry, 2019)

Meaning of the latter type in the schedule:

CAPITAL : obligatory task
Lower case : recommended task
reading : There is no experiment to do, but it could be asked in the short test.

TIMING OF THE PRACTICE:

5 hours of laboratory practice plus 1 hour seminar in each week.

Requirements of passing the course:

Visiting of the laboratory practices and the seminars are obligatory.

Only one, justified missing is allowed during the semester. If a student misses a laboratory practice, he or she has to participate a make-up practice (organized according to the instructors).

During the practices the students have to prepare their own lab manual. A simple A4 size exercise book could be used as lab manual, using individual sheets are not allowed. The way of the preparation of the lab manual is free, but the title of the tasks, the equations of the chemical processes, the observations of the experiments should be noted regarding the tasks (eg. test tube experiments, analysis of unknown samples, demonstrations etc.). Preparations, and the results of the unknown analysis could be due in only after showing the detailed, well-organized lab manual.

Each week, the laboratory practice begins with a short test (not more than 25 minutes) based exclusively on the material of the actual, and that of the previous weeks. If a student is late from the short test, his /her grade will be automatically 1 (failed) for that week.

For the "Passed (2)" grade, requirements are listed below:

- The average grade of the short tests has to be at least 2.00
- Preparation and due in the solid compounds until the deadline given.
- Analysis of all the unknown samples and due in the results.
- Compliance of the above detailed requirements for the lab manual.

The unacceptable test results (the average grade of the shot tests 2.00 > average > 1.70) will result in "Failed (1)" final grade, but there will be a chance to get "Passed (2)" by taking a written make-up exam.

If

-the laboratory work of the student is not acceptable (more than 2 missing, or bare lab manual)

- or final average of the short tests is lower than 1.70 "signature denied" as a final course grade will be given, and the course has to be retaken in the next year.

University of Debrecen, Department of Inorganic and Analytical Chemistry

2019/20 1st semester

SCHEDULE OF INORGANIC CHEMISTRY III LABORATORY PRACTICE

WEEK 1

(*lab*.:10.09.)

INORGANIC CHEMISTRY LABORATORY RULES

LABORATORY SAFETY

DISTRIBUTION OF LABORATORY EQUIPMENT

PRACTICAL TASKS:

1. Hydrogen generation from aqueous solution of acids and bases **1.1**
2. GENERATION OF HYDROGEN IN KIPP'S APPARATUS AND BURNING OF THE PURIFIED HYDROGEN GAS, ACTIVATION OF HYDROGEN, PREPARATION OF ATOMIC HYDROGEN (demonstration) **1.2** and **1.5**

WEEK 2

(*sem*.: 16.09.; *lab*.: 17.09.)

1. Formation of chlorine in the reactions of oxidizing agents and hydrochloric acid **2.1**
2. LABORATORY SCALE PREPARATION OF CHLORINE GAS **2.2**
3. Preparation of iodine via oxidation and reduction **2.7**
4. THE REACTION OF CHLORINE WITH METALS (in two membered groups, using Cl_2 cylinder)) **2.6**
5. THE COLOR AND THE REACTIVITY OF THE HALOGENS (demonstration) **2.5**
6. BURNING OF HYDROGEN IN CHLORINE (demonstration) **2.10**
7. The reactions of alkali bromides and iodides with concentrated sulfuric acid **2.13**
8. DETECTION OF FLUORIDE IONS **2.14**
9. FORMATION AND DISSOLUTION SILVER HALIDES **2.15**
10. DETECTION OF BROMIDE AND IODIDE IONS NEXT TO EACH OTHER BY USING CHLORINE WATER **2.16**
11. DETECTION OF CHLORIDE ION NEXT TO BROMIDE AND IODIDE IONS (BERG REACTION)) **2.17**
12. The effect of oxidizing agents on iodide ion **2.18**

WEEK 3

(*sem*.: 23.09.; *lab*.: 24.09.)

1. UNKNOWN SAMPLE I., detection of 1-3 ions from those of as follows: F^-, Cl^-, Br^-, I^-
2. Experiments with hypohalite ions **2.22**
3. EXPERIMENTS WITH HALOGENATE IONS, THE DISCRIMINATION OF HALOGENATE IONS **2.23**
4. REACTION OF POTASSIUM CHLORATE WITH RED PHOSPHOROUS (demonstration) **2.24 a**
5. Preparation of oxygen gas from hydrogen-peroxide with potassium-dichromate (olvasmány) **3.2**
6. BURNING ELEMENTS IN OXYGEN (demonstration) (O_2 cylinder) **3.3**
7. ELECTROLYTIC PREPARATION, DETECTION AND OXIDIZING ABILITY OF OZONE (demonstration) **3.6**
8. OXIDIZING AND REDUCING CHARACTER OF HYDROGEN PEROXIDE **3.7**
9. DETECTION OF HYDROGEN PEROXIDE **3.8**

WEEK 4

(*sem.*: 30.09.; *lab.*: 01.10.)

1. ALTERATION OF THE VISCOSITY OF LIQUID SULFUR; PREPARATION OF AMORPHOUS SULFUR **3.10**
2. Preparation of monoclinic sulfur in toluene **3.11**
3. Dissolution of sulfur and selenium in oleum, formation of E_8^{2+} cations (reading) **3.13**
4. Preparation of hydrogen sulfide and its aqueous solution, detection of H_2S. (reading) **3.14**
5. Preparing ammonium sulfide solution (reading) **3.16**
6. Preparation of sulfur dioxide in a laboratory and its analysis **3.19**
7. REACTION OF HYDROGEN SULPHIDE WITH SULFUR DIOXIDE (demonstration) **3.20**
8. CHEMICAL PROPERTIES OF SULFURIC ACID **3.23**
9. DIFFERENTIATION OF SULFITE AND SULFATE IONS **3.27**
10. Water-insoluble sulfates (reading) **3.24**
11. Reactions of peroxodisulfate ions **3.28**
12. Reactions of thiosulfate ions **3.29**

WEEK 5

(*sem.*: 07.10.; *lab.*: 08.10.)

1. UNKNOWN SAMPLE II., detection of two ions from those of as follows: Cl^-, Br^-, I^-, ClO_3^-, IO_3^-, S^{2-}, SO_3^{2-}, SO_4^{2-}. *(SO_3^{2-} and SO_4^{2-} or Cl^- and ClO_3^-, furthermore those ions which are able to react with each other under acidic or neutral conditions are not given together, detailed discussion on the seminar).*
2. FORMATION OF NITROGEN BY SYNPROPORTIONATION **4.1**
3. Transformation of white phosphorus to red species **4.3**
4. Oxidation of white phosphorus with Cu(II) ions (in two membered groups) **4.9**
5. CONDENSATION OF AMMONIA, DISSOLUTION OF SUBSTANCES IN LIQUID AMMONIA (demonstration) **4.10** and **7.7 a. and b**
6. Oxidation of ammonia with halogens (reading) **4.11**
7. DETECTION OF AMMONIA AND AMMONIUM IONS **4.13**

WEEK 6

(*sem.*: 14.10.; *lab.*: 15.10.)

1. PREPARATUM I.
    - a./ KNO_3 **7.14**
    - b./ $CuCl_2 \cdot 2H_2O$ **9.47**
    - c./ $KClO_3$ **2.25** (by using Cl_2 cylinder)
    - d./ $NaIO_3$ **2.27**
    - e./ $Na_3H_2IO_6$ **2.28**
    - f./ $Mg(ClO_4)_2 \cdot 6H_2O$ **8.10**
2. Reductions with hydrazine and hydrazinium(2+) salts **4.15 a. and b**
3. Preparation of phosphine (reading) **4.16**
4. DETECTION OF ARSENIC BY THE MARSH TEST (DEMONSTRATION) **4.17**
5. REDUCTION OF ARSENIC(III) CHLORIDE WITH LEAD(II) CHLORIDE (BETTENDORF TEST) **4.21**
6. Formation of bismuth(III) iodides **4.23**
7. Preparation and properties of nitrogen monoxide (reading) **4.24**

WEEK 7

(*sem.*: 21.10.; *lab.*: 22.10.)

1. Finishing and due in the preparatums
2. PREPARATION AND PROPERTIES OF NITROGEN-DIOXIDE (demonstration) **4.25**
3. EXPERIMENTS WITH NITRIC ACID **4.27**
4. Thermal decomposition of nitrates (reading) **4.28**
5. IDENTIFICATION OF NITRITE IONS **4.29**
6. IDENTIFICATION OF NITRATE IONS **4.30**
7. IDENTIFICATION OF NITRITE AND NITRATE IONS WITH GRIESS-ILOSVAY REAGENT **4.31**
8. Formation and study of phosphorous acid (in two membered groups) **4.33**
9. Reaction of phosphorous(V) oxide with water and concentrated sulfuric acid (in two membered groups) **4.34**
10. PROPERTIES OF PHOSPHORIC ACID **4.35**
11. IDENTIFICATION OF ORTHOPHOSPHATE IONS **4.36**

WEEK 8

(*sem.*: 04.11.; *lab.*: 05.11.)

1. UNKNOWN SAMPLE III., detection of two ions from those of as follows: Cl^-, Br^-, I^-, S^{2-}, SO_3^{2-}, SO_4^{2-}, NO_2^-, NO_3^-, NH_4^+, PO_4^{3-} (NO_2^- *and* NO_3^-, SO_3^{2-} *and* SO_4^{2-} *furthermore those ions which are able to react with each other are not given together*).
2. Chemical properties of arsenic(III) and arsenic(V) oxides (reading) **4.40**
3. Distinguishing of arsenite and arsenate ions (reading) **4.41**
4. ARSENIC(III), ANTIMONY(III) AND BISMUTH(III) SULPHIDES **4.42**
5. DISSOLUTION OF SILICON IN SODIUM HYDROXIDE **5.2**
6. Interaction of tin with acids and bases **5.3**
7. Interaction of lead with acids **5.4**
8. Formation of tin and lead by cementation (reading) **5.5**
9. PREPARATION OF SILANE FROM MAGNESIUM SILICIDE (demonstration) **5.6**

WEEK 9

(*sem.*: 11.11.; *lab.*: 12.11.)

1. Comparison of the hydrolytic stability of CCl_4 and $SiCl_4$ as well as $SiCl_4$ and SiH_4 **5.8**
2. Formation and hydrolysis of silicon tetrafluoride **5.9**
3. PROPERTIES OF TIN(II) CHLORIDE **5.10 a. b. and c**
4. LEAD(II) HALIDES **5.12**
5. PREPARATION AND PROPERTIES OF CARBON MONOXIDE (demonstration) **5.13**
6. Preparation and properties of carbon dioxide and carbonic acid (reading) **5.14**
7. HYDROGEN CARBONATES (BYCARBONATES) AND CARBONATES **5.15**
8. DETECTION OF SILICATE IONS ($[SiO_2(OH)_2]^{2-}$) **5.18**
9. OXYGEN COMPOUNDS OF TIN **5.22 a. b. and c**
10. OXYGEN COMPOUNDS OF LEAD **5.23**
11. reaction of cyanide ion **5.26 a. and b.**
12. Formation and detection of thiocyanate ion (reading) **5.26 c.**
13. Preparation of aqueous solution of thiocyanic acid by ion exchange (reading) **5.27**
14. SULPHIDES OF TIN AND LEAD **5.25**

WEEK 10

(*sem.*: 18.11.; *lab.*: 19.11.)

1. CHEMICAL REACTIONS OF ALUMINUM WITH AIR AND WATER **6.2**
2. CHEMICAL REACTIONS OF ALUMINUM WITH ACIDS AND BASES **6.3**
3. Study of diborane (B_2H_6) (reading) **6.6**
4. CHEMICAL PROPERTIES OF SODIUM TETRAHYDROBORATE **6.7** (d. e. and f. are readings)
5. EXPERIMENTS WITH LITHIUM TETRAHYDROALUMINATES (demonstration) **6.8**
6. PREPARATUM II.
   a./ $CuSO_4 \cdot 5H_2O$ 1st method **3.26**
   b./ $CuSO_4 \cdot 5H_2O$ 2nd method **3.26**
   c./ $Na_4P_2O_7 \cdot 10H_2O$ **4.39**
   d./ CuCl **9.48**
   e./ $[Co(NH_3)_6]Cl_3$ **9.27**
   f./ $[Co(NH_3)_4(NO_2)_2]Cl$ **9.42**
   g./ $CaO_2 \cdot 8\ H_2O$ **8.11**
   h./ $[Ni(CN)_2 \cdot NH_3] \cdot nC_6H_6$ **9.38**

WEEK 11

(*sem.*: 25.11.; *lab.*: 26.11.)

1. Finishing and due in the preparatums
2. Formation and properties of boron trifluoride **6.9**
3. FORMATION OF SODIUM HEXAFLUOROALUMINATE **6.10**
4. Experiments whit tallium halides (reading) **6.12**
5. Liberation of hydrogen chloride by boric acid (reading) **6.13**
6. REACTION OF BORIC ACID WITH MANNITOL **6.14**
7. Formation and properties of methyl ester of boric acid (trimethoxyborane) **6.15 a. b. and c**
8. FORMATION, AMPHOTERIC CHARACTER AND APPLICATIONS OF ALUMINUM HYDROXIDE **6.17**