The 5th Metropolises Olympiad

Chemistry

Practical exam

December 17, 2020

General directions

- **Lab safety:** follow the general rules accepted in chemistry labs; you must wear your lab coat and goggles or own glasses whenever present in the lab; no eating or drinking in the lab.
- **Violation of lab safety rules:** you get one warning only; offend again: you are disqualified.
- **The exam includes two parts:** on Analytical and Inorganic chemistry. Fulfill the task only in the suggested order.
- **Time:** 4 h to complete all the procedures. 30 min warning before the end.
- **Use only the calculator and pen.**
- **Questions** concerning safety or toilet break: **ask your lab assistant**.
- **Chemical waste:** carefully pour in the sink.
- **Answers:** only in the answer boxes in the booklet, nothing else will be graded. Relevant calculations have to be shown when asked for.
- **After the stop signal:** put your booklet aside and leave it at your working place.
- **You must stop your work immediately after the stop signal has been given. A 2 min delay will result in zero points for the current part of the task.**
- **During the Practical exam, some items of the glassware are expected to be used several times. Clean these carefully.**
- **This booklet with the task on Inorganic and Analytical chemistry and answer boxes: 12** pages (incl. the cover sheet and Periodic table of elements).

Labware and equipment	
Gloves	1 pair
Fingertips to handle hot things	1 pair
Beaker, 50 or 100 mL	4 pcs
Beaker, 250 mL	2 pcs
Crystallizing dish or water bath	2 pcs
Measuring cylinder, 10 mL	2 pcs
Glass rod	2 pcs
Spatula	1 pc
Gas-fired burner with asbestus plate fixed on a stand or electric heater	1 pc
Matches (if using the gas-fired burner)	1 box
Balances, 0,001 g readability at least	1 pc per 2 students
Paper filters (matching glass funnel)	10 pcs
Glass funnel	2 pcs
Laboratory stand with a ring and clamps	1 pc
Ice	~ 1 kg
Weighing paper or weighing containers	10 pcs
Wash-bottle with distilled water	1 pc
Watch glass for weighing	2 pcs
Drop funnel, 50 mL	1 pc
Mortar with pestle	1 pc
Optical microscope	1 pc per 2 students
Thermometer	1 pc
Laboratory stand with burette clamp	1 pc
Burette, 25 mL	1 pc
Graduated or Bulb pipette, 10 mL	2 pcs
Small beaker to be used under the burette	1 pc
Measuring cylinder, 100 mL	2 pcs
Conical flask, 250 mL	4 pcs
Volumetric flask, 100 mL	3 pcs
Funnel for filling the burette	1 pc
Beaker for filling the burette, 50 or 100 mL	1 pc
Pipette filler or 3-way bulb	1 pc
Watch glass (to cover conical flask) or funnel matching the flask neck	2 pcs
Pipettes for sampling (e.g. Pasteur pipette)	5 pcs
Petri dish	2 pcs

Reagents	
Sodium chloride, technical glade	200 g
Potassium dichromate	8 g
Sulfuric acid conc. (□94%)	10 mL
Ethanol (96%)	20 mL
Hydrochloric acid (24%)	8 mL
Indicator paper, universal	5 strips
Sodium tetraborate	10 g
Sodium hydrocarbonate	10 g
Sodium chloride	10 g
Agar-agar, 0.1% solution (or Gelatin, 5g)	10 mL
Fluorescein (or fluorescein sodium salt 1 g, or dichlorofluorescein 1 g, or dichlorofluorescein sodium salt 1 g, or starch 10 g plus diphenylcarbazone 1 g)	1 g

Question	A1	A2	B1	B2	B3	B4	B5	B6	B7	D1	D2	D3	D4	E1	E2	E3	Total
Points	15	3	2	2	2	2	2	3	15	3	2	15	2	8	20	4	100
Result																	

Problem 1. Inorganic synthesis and chemical analysis of chromium compounds

Chemical plants are located in many metropolises. They provide inhabitants with workplaces and allow manufacturing valuable products, still, despite all measures, often leading to environmental pollution. Environmental monitoring requires knowledge of both pollutants and possible ways of their transformation into other compounds. You are suggested to carry out two syntheses with chemical analysis on one of the products, using one and the same chromium compound as the starting material.

Part A. Using the balances, weigh a sample of potassium dichromate of about 4 g and write down the exact mass:

Mass = _____ g

Transfer the weighed amount into the mortar and pound with the pestle under the fume hood. Transfer the entire potassium dichromate into the 50 mL beaker and add 8 mL of 24% hydrochloric acid solution under the fume hood. Heat the mixture with constant mixing under the fume hood until the entire potassium dichromate dissolves. Avoid boiling. Once all the crystals dissolved, terminate heating and cool down the beaker to room temperature. In 15 min filter off the precipitated crystals, using the glass funnel with paper filter. To do so transfer all the crystals and the solution onto the filter and let the major part of the solution pass through. Transfer the crystals into the Petri dish, cover the crystals with another filter and press the crystals through the filter removing as much of the residual solution as possible.

A1. Weigh the product and calculate the yield. Ask your lab assistant to take the picture of both the balances reading and the product on the balances. Attach the picture to your exam answers; use the file name «Product 1».

Mass of the product = _____ g
Calculation:

Yield = _____ %
Maximum 13 points for the yield (the grading scheme will be given separately), 2 points for the calculation

A2. Investigate crystals of the product and initial potassium dichromate under the microscope. Draw their shape and compare their color.

The shape of the crystals: prismatic ($K_2Cr_2O_7$) needle-like (product)

The crystals ☒ are close in color ☐ have completely different colors

1 point for each shape and 1 point for the information about the color

Part B. Using the balances, weigh a sample of potassium dichromate of about 2.5 g and write down the exact mass:

Mass = _____ g

Place 25 mL of water into the 50 mL beaker and dissolve there the weighed amount of potassium dichromate. With intense mixing **carefully** add 10 mL of concentrated (94-98%) sulfuric acid to the prepared solution under the fume hood. Since the mixture warms up as a result of adding the acid, first cool the mixture to the room temperature and then place the beaker with the solution into the crystallizing dish with the coolant mixture (water with ice).

B1. Do you observe any change of the solution color? If so, explain the reason (write down the reaction behind the change).

The color change:

☒ Yes ☐ No

1 point

If Yes, write down the reaction behind:

$3Cr_2O_7^{2-} + 2H^+ = 2Cr_3O_{10}^{2-} + H_2O$ or similar

1 point

Fill the dropping funnel with 7 mL of ethanol and clamp the funnel at the stand over the beaker with the solution. Slowly add ethanol drop-wise to the solution in the beaker with intense mixing. Using the thermometer, maintain the temperature of the solution below 40°C.

Once the reaction is complete, cover the beaker with the watch glass or filter paper and keep it aside for cooling.

B2. Write down the reaction occurring.

$K_2Cr_2O_7 + 4H_2SO_4 + 3CH_3CH_2OH = K_2SO_4 + Cr_2(SO_4)_3 + 3CH_3CHO + 7H_2O$

2 points

B3. What is the color of the obtained solution?

☐ Wine-red	☐ Light blue
☐ Red	☐ Blue
☐ Orange	☒ Purple
☐ Yellow	☐ Brown
☐ Green	☐ Black
☐ Colorless	2 points

B4. What particle(s) is(are) responsible for the coloration of the obtained solution?

☐ $Cr_2O_7^{2-}$	☐ Cr^{3+}
☒ $Cr(H_2O)_6^{3+}$	☐ $[Cr(SO_4)(H_2O)_5]^{+}$
☐ Cr^{2+}	☐ $[Cr(OH)(H_2O)_5]^{2+}$
☐ $Cr(H_2O)_6^{2+}$	☐ $Cr(OH)_3$
☐ $Cr(OH)_2$	☐ K^{+} 2 points

Continue with the next part of the task. You will return to the solution obtained in Part B afterwards.

Part C. Quantitative analysis. Preparation of the solutions.

Prepare **one** of the hereunder indicator solutions:
Fluorescein (or dichlorofluorescein). Prepare 5 mL of 0.2% solution of the indicator (0.01 g) in 60-70% ethanol.
Fluorescein (or dichlorofluorescein) sodium salt. Prepare 5 mL of 0.2% solution of the indicator (0.01 g) in distilled water.
Diphenylcarbazone. Prepare 5 mL of 0.2% solution of the indicator (0.01 g) in 60-70% ethanol.

Work solutions.
Starch solution. Prepare 20 mL of 0.4% solution of starch (0.08 g) in distilled water.
Gelatin solution. Prepare 25 mL of 0.1% solution of gelatin (0.025 g) in distilled water.
Silver nitrate solution. Prepare 100 mL of 0.1 M solution of solver nitrate.

Part D. Standardization of silver nitrate solution with sodium chloride.

If using fluorescein or dichlorofluorescein or their sodium salts as the indicator, weigh about 0.6 g of sodium chloride and write down the exact mass:

Mass = _____ g

Quantitatively transfer the weighed amount of sodium chloride into the 100 mL volumetric flask and bring up to the mark with distilled water. Using the pipette, transfer a 10.00 mL aliquot of sodium chloride solution into 250 mL titration flask, add 10 mL of distilled

water and 3-5 drops of the indicator. Titrate with the silver nitrate solution until abrupt color change from white to pink.

If using diphenylcarbazone as the indicator, weigh about 0.6 g of sodium chloride and write down the exact mass:

Mass = _____ g

Quantitatively transfer the weighed amount of sodium chloride into the 100 mL volumetric flask and bring up to the mark with distilled water. Using the pipette, transfer a 10.00 mL aliquot of sodium chloride solution into 250 mL titration flask, add 10 mL of distilled water. Check the solution pH using the indicator strips (must be around 7), neutralize the solution with sodium hydrocarbonate or sodium tetraborate, if needed. Add 2 mL of the starch solution and 5-8 drops of the indicator. Titrate with the silver nitrate solution until abrupt color change from light-red to purple.

D1. Calculate the exact concentration of the sodium chloride solution.

Calculation:

$c = m/(V*M)$

2 points

Concentration of the NaCl solution = ___about 0.100____ M 1 point for the correct range

D2. Write down the chemical reaction conducted.

$NaCl + AgNO_3 = AgCl + NaNO_3$

2 points

D3. Write down the titration results Max. 15 points (the grading scheme will be given separately)

Titration No	1	2	3			
The initial burette reading, cm^3						
The final burette reading, cm^3						
The volume consumed in the titration, cm^3						

The accepted volume, $V_{reply\ 1}$: _____ cm^3

D4. Calculate the concentration of the silver nitrate solution.

Calculation:

$c_1V_1=c_2V_2$

2 points

Concentration of silver nitrate solution = _____ M.

Once you are finished with standardization of the silver nitrate solution, place the beaker with the solution obtained in Part B into the crystallizing dish with the coolant mixture (ice mixed with sodium chloride of technical grade).

Part E. Determination of ions in the crystals prepared in Part A.

Weigh about 0.9 g of the crystals obtained in Part A and write down the exact mass:

Mass = _____ g.

Quantitatively transfer the weighed sample into the 100 mL volumetric flask, bring with water up to the middle of the flask and dissolve the solid by gently swirling the flask. Then bring up to the mark and mix well. Using the pipette, transfer a 10.00 mL aliquot of the solution into the titration flask. Check the solution pH using the indicator strips, add tiny amount of sodium hydrocarbonate (or sodium tetraborate) on the spatula tip to attain the pH value of 7-8. Note the change of the solution color. Add 5 mL of the gelatin solution (or 2 mL of the agar-agar solution) and 15 mL of distilled water to the analyzed solution. Titrate with the standardized silver nitrate solution until the solution changes to orange-brown, the color being stable for at least 30 s.

E1. Write down ionic equations of the conducted reactions and observations of the color changes. Suppose what ion is being titrated.

The reaction occurring upon dissolving of the crystals:

$2KCrO_3Cl + H_2O = 2K^+ + Cr_2O_7^{2-} + 2H^+ + 2Cl^-$

What is the solution color?

- ☐ Wine-red
- ☐ Red
- ☒ Orange
- ☐ Yellow
- ☐ Green
- ☐ Colorless
- ☐ Light blue
- ☐ Blue
- ☐ Purple
- ☐ Brown
- ☐ Black

Reaction(s) occurring upon addition of sodium hydrocarbonate:

$H^+ + HCO_3^- = CO_2 + H_2O$

$HCO_3^- + H_2O = H_2CO_3 + OH^-$

$(CO_3^{2-} + H_2O = HCO_3^- + OH^-)$

$Cr_2O_7^{2-} + 2OH^- = 2CrO_4^{2-} + H_2O$

What is the solution color?

- ☐ Wine-red
- ☐ Red
- ☐ Orange
- ☐ Yellow
- ☐ Green
- ☐ Colorless
- ☐ Light blue
- ☐ Blue
- ☐ Purple
- ☐ Brown
- ☐ Black

Reaction behind the change of the precipitate color at the titration end-point:

$2Ag^+ + CrO_4^{2-} = Ag_2CrO_4$

The ion titrated: ___ Cl^- _____ 1 point for each correct reaction or choice

E2. Write down the titration results. Max. 20 points (the grading scheme will be given separately)

Titration No	1	2	3			
The initial burette reading, cm^3						
The final burette reading, cm^3						
The volume consumed in the titration, cm^3						

The accepted volume, $V_{reply\ 2}$: _____ cm^3

E3. Calculate the number of water molecules per one compound molecule.

Calculation:

4 points for any reasonable calculation and correct answer.

The crystal hydrate formula. The ratio of anhydrous salt to water: _____ .

Part B (continuation). When finished with the quantitative analysis, continue with the solution left in the coolant mixture.

Filter off the precipitated crystals using the glass funnel with paper filter. What the crystals with 5 mL of ethanol. Transfer the crystals onto the dry filter paper or filter, cover the crystals with another piece of the filter paper or another filter and press the crystals heavily through the filter to remove as much of the solvent as possible.

B5. Write down the formula of the synthesized compound.

$KCr(SO_4)_2 \cdot 12H_2O$ or $K_2SO_4 \cdot Cr_2(SO_4)_3 \cdot 24H_2O$ or $[K(H_2O)_6][Cr(H_2O)_6](SO_4)_2$

2 points

Take a few crystals and study them using the microscope.

B6. What is the shape of the crystals? Sketch the crystals.

Prismatic

3 points

B7. Transfer the crystals into the Petri dish or onto the watch glass, weigh them and calculate the product yield. Ask your lab assistant to take the picture of both the balances reading and the product on the balances. Attach the picture to your exam answers; use the file name «Product 2».

Mass of the product = _____ g

Calculation:

Yield = _____ %

Maximum 13 points for the yield (the grading scheme will be given separately), 2 points for the calculation

Periodic table with relative atomic masses

1																	18
1 H 1.008	2											13	14	15	16	17	2 He 4.003
3 Li 6.94	4 Be 9.01											5 B 10.81	6 C 12.01	7 N 14.01	8 O 16.00	9 F 19.00	10 Ne 20.18
11 Na 22.99	12 Mg 24.30	3	4	5	6	7	8	9	10	11	12	13 Al 26.98	14 Si 28.09	15 P 30.97	16 S 32.06	17 Cl 35.45	18 Ar 39.95
19 K 39.10	20 Ca 40.08	21 Sc 44.96	22 Ti 47.87	23 V 50.94	24 Cr 52.00	25 Mn 54.94	26 Fe 55.85	27 Co 58.93	28 Ni 58.69	29 Cu 63.55	30 Zn 65.38	31 Ga 69.72	32 Ge 72.63	33 As 74.92	34 Se 78.97	35 Br 79.90	36 Kr 83.80
37 Rb 85.47	38 Sr 87.62	39 Y 88.91	40 Zr 91.22	41 Nb 92.91	42 Mo 95.95	43 Tc -	44 Ru 101.1	45 Rh 102.9	46 Pd 106.4	47 Ag 107.9	48 Cd 112.4	49 In 114.8	50 Sn 118.7	51 Sb 121.8	52 Te 127.6	53 I 126.9	54 Xe 131.3
55 Cs 132.9	56 Ba 137.3	57-71	72 Hf 178.5	73 Ta 180.9	74 W 183.8	75 Re 186.2	76 Os 190.2	77 Ir 192.2	78 Pt 195.1	79 Au 197.0	80 Hg 200.6	81 Tl 204.4	82 Pb 207.2	83 Bi 209.0	84 Po -	85 At -	86 Rn -
87 Fr -	88 Ra -	89-103	104 Rf -	105 Db -	106 Sg -	107 Bh -	108 Hs -	109 Mt -	110 Ds -	111 Rg -	112 Cn -	113 Nh -	114 Fl -	115 Mc -	116 Lv -	117 Ts -	118 Og -

57 La 138.9	58 Ce 140.1	59 Pr 140.9	60 Nd 144.2	61 Pm -	62 Sm 150.4	63 Eu 152.0	64 Gd 157.3	65 Tb 158.9	66 Dy 162.5	67 Ho 164.9	68 Er 167.3	69 Tm 168.9	70 Yb 173.0	71 Lu 175.0
89 Ac -	90 Th 232.0	91 Pa 231.0	92 U 238.0	93 Np -	94 Pu -	95 Am -	96 Cm -	97 Bk -	98 Cf -	99 Es -	100 Fm -	101 Md -	102 No -	103 Lr -