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# The 6th Metropolises Olympiad



## Chemistry

### Practical examination

December 7, 2021



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### 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 several parts:** 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 or waste container.
- **Answers:** only in the answer boxes in the booklet, nothing else will be graded. Relevant calculations have to be shown when asked for.
- **Photos:** you will be asked to deliver photos at definite stages of the experiment; **ask your lab assistant to take the photos** (quality of a typical smartphone is sufficient), the names of the files with the photos should include **your team and your name**.
- **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 Inorganic and Analytical chemistry and answer boxes: 12 pages** (incl. the cover sheet and Periodic table of elements).



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<b>Glassware and equipment</b>	
Lab coat	1 pc
Goggles or glasses	1 pc
Gloves	1 pair
Magnetic stirrer with heating	1 pc
Stirring bar	1 pc
Fingertips to handle hot things	1 pair
Termometer (0-100 °C)	1 pc
Balances, 0,001 g readability at least	1 pc per 2 students
Weighing paper or weighing containers or watch glass for weighing	10 pcs
Laboratory stand with a ring for funnel and clamps	1 pc
Laboratory stand with burette clamp	1 pc
Pipette filler or 3-way bulb	1 pc
Beaker, 100 mL	4 pcs
Fglass funnel (dimeter of 8-10 cm)	3 pcs
Funnel for free-flowing substances (dimeter of 5-7 cm)	1 pc
Funnel (dimeter of 15 cm)	1 pc
Glass rod	2 pcs
Burette, 25 mL	1 pc
Graduated or Bulb pipette, 10 mL	2 pcs
Small beaker to be used under the burette	1 pc
Funnel for filling the burette	1 pc
Container or vial for 500 mL	1 pc
Measuring cylinder, 10 mL	5 pcs
Measuring cylinder, 25 mL	1 pc
Measuring cylinder, 100 mL	2 pcs
Measuring cylinder, 500 mL	1 pc
Conical flask, 250 mL	2 pcs
Conical flask with stopper, 250 mL	2 pcs
Conical flask, 150-200 mL, with a ground joint stopper and gas-vent tube (optionally)	1 pc
Volumetric flask, 100 mL	1 pc
Volumetric flask, 1000 mL	1 pc
Watch glass (to cover conical flask) or funnel matching the flask neck	4 pcs
Paper filters (matching glass funnel)	10 pcs
Filter paper	10 pcs
Plastic spatula	1 pc
Spoon or spatula for weighing substances	10 pcs
Pipetter for sampling (e.g. Pasteur pipette)	5 pcs



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Mortar with pestle	1 pc
Wash-bottle with distilled water	1 pc
<b>Reagents</b>	
Bleaching powder	30 g
Potassium bicarbonate	100 g
Distilled water	5 L
Iodine standard solution (0.05 M)	500 mL
Sodium thiosulfate	40 g
Starch	5 g
Potassium bromide	30 g
Hydrochloric acid concentrated	150 mL
Sodium hydroxide	2 g
Potassium iodide	30 g



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Question	A1	A2	A3	A4	A5	A6	A7	A8	C1	C2	C3	C4	D1	D2	D3	Total
Points	2	2	2	2	6	4	20	2	4	4	20	2	6	20	4	100
Result																

### **Inorganic synthesis and chemical analysis of chlorine compounds.**

Bleaching powder was first synthesized long ago, in the late 18<sup>th</sup> century. Initially, it was prepared by passing chlorine through aqueous solution of slaked lime, though later it was found that the same reaction occurs when chlorine interacts with dry lime. Bleaching powder is used in industry for paper manufacturing, textile bleaching, and as a disinfectant. Besides, it is a potent oxidizer, a source of active chlorine and oxygen. You will start your work with studying the preparation of bleaching powder.

#### Part A.

**A1.** Write down the equation of the reaction behind the synthesis of bleaching powder mentioned in the text.

1. Weigh on the balance a sample of bleaching powder with the mass of about 7 g and record the exact mass:

Mass = \_\_\_\_\_ g.

**A2.** Write down the chemical composition of bleaching powder. Which of its constituents reveal oxidative properties?

Composition:

Oxidative properties are due to:



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**A3.** Give an example of a redox reaction demonstrating the oxidative properties of bleaching powder. Write down the equation of the chemical reaction.

**A4.** Bleaching powder is known to slowly decompose when stored on air. Write down the equation of the reaction behind the process.

2. Weigh on analytical balance a sample of potassium bicarbonate with the mass of 11 g, write down the exact mass.

Mass = \_\_\_\_\_ g.

**A5.** What processes do occur when bicarbonate is dissolved in water? Write down the equations of chemical processes developing in the aqueous solution. What is the reaction of an aqueous solution of potassium bicarbonate?

Equations of chemical reactions:

  
  
  
  
  
  
  
  
  
  

The reaction of an aqueous solution of potassium bicarbonate is:

pH=7                       pH <7                       pH >7

3. A slight smell of bleach (lower than that when using commercial cleaners) can be felt at this stage. You can follow any of the hereunder variants.

Variant 1. Transfer 40 mL of distilled water into a 150-200 mL beaker using the measuring cylinder. Add weighed samples of bleaching powder and potassium bicarbonate to the beaker.

*Ask you lab assistant to take a photo of your reaction mixture. Send the photo together with your work. Entitle the file «Your city and name\_Picture 1».*



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Put the stirring bar in the suspension in the 150-200 mL beaker and place it onto the magnetic stirrer with heating. Cover the beaker with the watch glass (**obligatory to suppress the odor!**). With constant stirring, heat the reaction mixture up to 65°C. Keep the mixture at this temperature with stirring for 15 min.

Variant 2. Prepare 100 mL of the 3% solution of sodium hydroxide. Using the measuring cylinder, transfer 40 mL of distilled water into the 150-200 mL conical flask. Add the weighed samples of bleaching powder and potassium bicarbonate to the flask.

*Ask you lab assistant to take a photo of your reaction mixture. Send the photo together with your work. Entitle the file «Your city and name\_Picture 1».*

Put the stirring bar in the suspension in the 150-200 mL flask and place it onto the magnetic stirrer with heating. Apply the ground joint stopper with a gas-vent tube and immerge the tube end into the 3% solution of sodium hydroxide in a beaker. With constant stirring, heat the reaction mixture up to 65°C. Keep the mixture at this temperature with stirring for 15 min.

*For both variants: Ask you lab assistant to take a photo of your reaction mixture. Send the photo together with your work. Entitle the file «Your city and name\_Picture 2».*

**A6.** What processes do proceed in the reaction mixture. Write down the equations of the chemical reactions. What process do require heating of the reaction mixture?

Equations of chemical reactions (number the equations):

Heating is needed for process No \_\_\_\_.

4. Filtrate the hot solution through paper filter wet with distilled water into a 100 mL beaker. Place the beaker with the filtrate onto the heater and boil off until crystallization starts.

*Ask you lab assistant to take a photo of your reaction mixture. Send the photo together with your work. Entitle the file «Your city and name\_Picture 3».*



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5. Remove the beaker from the heater and let it cool down to room temperature. Filtrate the precipitated crystals through a dry paper filter. Press the obtained crystals with filter paper several times to remove the moisture.

*Ask you lab assistant to take a photo of your product. Send the photo together with your work. Entitle the file «Your city and name\_Picture 4».*

6. Weigh the obtained product on the analytical balance.

*Ask you lab assistant to take a photo of the balance reading together with the product, so that both are clearly seen on the same picture. Send the photo together with your work. Entitle the file «Your city and name\_Picture 5».*

**A7.** Write down the mass of the obtained product and calculate the yield.

Mass of the product \_\_\_\_\_ g.

Yield = \_\_\_\_\_ %

**A8.** What is the chemical composition of the obtained crystalline compound? What admixtures could be present in it?

Chemical composition:

Admixtures:





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Part B. Quantitative analysis. Preparation of solutions.

Working solutions.

Solution of starch. Prepare 20 mL of 1% solution of starch (0.2 g) in distilled water (readily dissolves when heated on the heater).

Solution of potassium iodide. Prepare 1 L of 1% solution of potassium iodide (10 g) in distilled water.

Solution of sodium thiosulfate. Prepare 500 mL of 0.05 M solution of sodium thiosulfate.

*Ask your lab assistant to prepare 5 portions (10 mL each) of the concentrated hydrochloric acid in 10 mL cylinders. If you will need additional 10 mL portions of hydrochloric acid, approach your lab assistant again.*

Part C. Standardization of the sodium thiosulfate solution using iodine standard solution.

Concentration of iodine standard solution: \_\_\_\_\_ M.

Fill the burette with the sodium thiosulfate solution. Using pipette, transfer 10.00 mL of iodine standard solution into 250 mL conical titration flask. Add 100 mL of water into the flask and promptly titrate with the sodium thiosulfate solution until the pale-yellow coloration of the solution. Add 1–2 mL of the starch solution and continue titration with intense swirling until complete disappearance of the solution coloration.

**C1.** Write down the equation(s) of the conducted chemical reaction(s).

**C2.** Write down the equations of possible side chemical reactions.



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**C3.** Write down the titration results.

Titration No	1	2	3			
Initial volume, cm <sup>3</sup>						
Final volume, cm <sup>3</sup>						
Volume spent for titration, cm <sup>3</sup>						

Your accepted volume,  $V_{\text{reply 1}}$ : \_\_\_\_\_ cm<sup>3</sup>

**C4.** Calculate the concentration of the sodium thiosulfate solution.

Calculation:

Concentration of sodium thiosulfate = \_\_\_\_\_ M.

Part D. Determination of the ions in the crystals obtained in Part A.

Press the crystals obtained in Part A with filter paper several times to remove the moisture. Transfer the substance into the mortar and mill into it a homogeneous bulk. Weigh approximately 0.26 g of the prepared substance and write down the exact mass:

Mass = \_\_\_\_\_ g.

Transfer qualitatively the weighed sample into 100 mL volumetric flask, add distilled water up to the middle of the flask, dissolve the solid by gentle shaking, then bring up to the mark and mix well. Using the pipette, transfer a 10.00 mL of aliquot into a conical titration flask. Add 1 g of potassium bromide, 20 mL of concentrated hydrochloric acid (10 mL from each of the two cylinders), stopper the flask, shake and put aside for 5 min. Then add 100 mL of the 1% solution of potassium iodide and titrate with the standardized solution of sodium thiosulfate until pale-yellow coloration of the solution. Add 1–2 mL of the starch solution and continue titration with intense swirling until disappearance of the solution coloration.

**D1.** Write down the equations of the conducted reactions in the ionic form, your observations of the color change, and propose the ion being titrated.



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Equations of chemical reactions:

The color of the solution after addition of starch:

- |                                 |                                    |
|---------------------------------|------------------------------------|
| <input type="checkbox"/> Vinous | <input type="checkbox"/> Blue      |
| <input type="checkbox"/> Red    | <input type="checkbox"/> Deep blue |
| <input type="checkbox"/> Orange | <input type="checkbox"/> Purple    |
| <input type="checkbox"/> Yellow | <input type="checkbox"/> Brown     |
| <input type="checkbox"/> Green  | <input type="checkbox"/> Black     |

The ion being titrated: \_\_\_\_\_

**D2.** Write down the titration results.

Titration No	1	2	3			
Initial volume, cm <sup>3</sup>						
Final volume, cm <sup>3</sup>						
Volume spent for titration, cm <sup>3</sup>						

Your accepted volume,  $V_{\text{reply2}}$ : \_\_\_\_\_ cm<sup>3</sup>

**D3.** Calculate the percentage of the titrated ion in the sample.

Calculation:

Content of the titrated ion: \_\_\_\_\_ %.



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## The periodic table with the relative atomic masses of elements

1 H 1.008																	18 He 4.003		
2												13 B 10.81	14 C 12.01	15 N 14.01	16 O 16.00	17 F 19.00	10 Ne 20.18		
3 Li 6.94	4 Be 9.01											11 Na 22.99	12 Mg 24.30	3 Al 26.98	4 Si 28.09	5 P 30.97	6 S 32.06	7 Cl 35.45	8 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 -