Practical problems. SwissChO. Easter 2017.

Task 1. Perfect gases and molecular mass (4 points)

Take two erlenmeyers 250 or 300 mL, if possible with thin necks. Mark them 1 or 2 with ink. Use scissors to cut two squares in an aluminium foil about 10 x 10 cm. Cover the top of each erlenmeyer with such a square, and fold the foil back around the neck, as tightly as possible, as if to make believe that the flask is closed. Weigh each erlenmeyer to ±0.01 g, and note their weights (m_1)

Take a small piece of dry ice (CO_2) about as big as a hazelnut. Remove the aluminium cap carefully and introduce the dry ice into the erlenmeyer. Close immediately with the aluminium cap, and weigh. The increase of weight should be bigger than 2 g. If not, add some more dry ice. Repeat with the other erlenmeyer. Note the weights (m_2).

Deposit the two erlenmeyers on a metallic table. The ice will slowly evaporate inside. Keep having a look on them. After about half an hour, the dry ice has disappeared. Don't wait any longer ! Take the erlenmeyer and weigh it : m_3. This weight is continuously decreasing with time. The new weight should be at least 0.12 g heavier that the original value m_1. Keep the erlenmeyer that has the greatest increase of weight. Forget the other one.

Remove the aluminum cap of the chosen erlenmeyer. Fill it with tap water. And transfer this water into a big 1-Liter cylinder. The obtained water volume V gives you the volume of the gas in the erlenmeyer.

Use your values m_1, m_2, m_3 and V to calculate the molecular mass of the gas coming out of the dry ice. Of course, you know that it is CO_2. The final result is not important. What is important is the reasoning you have to do to get it. Anyway you will obtain a little bit less than the theoretical value 44 g/mol. Please explain with great care your reasoning, supposing that :

the gases are perfect,

air is made of 20% O_2 and 80% N_2, with an apparent molar mass 28.9

the temperature is 22°C (a small change in temperature has a negligible effet).

Don't use too many significant numbers in your calculations ! You will be judged by the quality of your explanations.

Task 2. Titrations (9 points)

1st Titration (Backtitration). To be done three times.

Take an erlenmeyer (250 or 300 mL). Introduce 50 mL water with a cylinder, then about 5 g ± 1 g urotropin as a buffer to be later on, then enough xylenolorange powder so that the color is a visible violet (not too dim). Mix well. Use a pipette to add 10 mL of the stock solution Cu(II)-Zn(II). Add 25 mL Na_2EDTA 0.05 M. The color of the solution should turn to green-brown. All the ions Zn and Cu are complexed by EDTA, and the solution contains an excess of EDTA, that has now to be determined.

Fill the burette with the 0.050 M $ZnSO_4$ solution. Add the $ZnSO_4$ solution drop by drop into the brownish-green solution. Observe the point where the color changes from green to violet. Mind ! The necessary volume is small ! Don't go too quickly. The reaction Zn + EDTA may be slow near the end point. Note the volume *V* used. Repeat three times.

Calculations from the backtitrations

a) Write the equation of the reactions of EDTA with Zn and Cu (1 point)

b) Calculate the number of moles of EDTA added (1 point)

c) Calculate the average number of moles of Zn used for the titration (1 point)

d) Calculate by difference the total number of moles of Zn+Cu from the 10 mL sample. Calculate then the total concentration Zn+Cu in mol/liter of the stock solution. Attention of well separating Zn from the original solution and Zn from the titration. (1 point)

2nd Titration (Redox). To be done three times.

Take an erlenmeyer (250 or 300 mL). Introduce 50 mL of the stock solution containing Cu(II) and Zn(II). Add 2 mL H_2SO_4 1 M with a plastic pipette. Add about 2 g ± 1 g NaI. Stir well. The solution gets brown and turbid, because of the reaction between Cu^{2+} and iodide I^- producing insoluble CuI and some iodine I_2. This is a redox reaction, as the precipitate CuI is copper(I) iodide. Zn does not react.

Fill a burette with a 0.1 M $Na_2S_2O_3$ solution. Add the $Na_2S_2O_3$ drop by drop. The color of the solution is slowly fading, so I_2 is consumed. When it is pale yellow, add a spatula of soluble starch, and stir. The solution turns deep blue or deep violet, due the presence of a dark complex starch-iodine. Continue the titration up to the disappearance of the colour. Note the used volume *V*.

Calculations from the redox titrations.

e) Write the equations of the reactions, one for I2 production, one for consuming I_2 (1 p.)

f) Calculate the average number of moles of thiosulfate ($Na_2S_2O_3$) used in the redox titrations (1 p.)

g) Calculate the number of moles of I_2 (1 p.)

h) Calculate the number of moles of Cu in the sample, and the concentration of Cu in the stock solution (1 pt.)

i) Calculate the molar concentration of Zn in the stock solution (1 p.)

Task 3. Organic synthesis. To be done once. (9 points)

Using plastic pipettes, introduce 1 mL tert-butanol $(CH_3)_3COH$ (10 mmol) and 2 mL pure acetic acid CH_3COOH in a 50 mL erlenmeyer. Add 0.69 g *p*-dimethoxybenzene $CH_3OC_6H_4OCH_3$ (5 mmol). Stir for dissolving, with a magnetic bar. When it is dissolved, cool the solution in ice. After 1 or 2 minutes, some precipitate appears. Add slowly, drop by drop, with a plastic pipette, 2.5 mL concentrated sulfuric acid H_2SO_4 (2 drops per second) .

Put the flask on the stirrer at room temperature for 15 minutes. Answer the theoretical questions when waiting. Then put the flask back into the ice and add 20 mL water at 0°C. A nice white precipitate should appear, lighter than water. Filtrate on paper in a funnel or a Buchner. Wash five times with 20 mL cold water (0°C).

Put the filter paper with the wet product on a watch glass. Add your name, and give it to the staff. Its weight will be measured later on.

The obtained product has the structure of *p*-dimethoxybenzene, where two H atoms attached to the ring have been removed and replaced by two tert-butyl groups $(CH_3)_3C\cdot$. The H atoms are situated as far away as possible. .

Questions.

a) Draw the developed structures of the tert-butanol, of the acetic acid, and of the *p*-dimethoxybenzene. (3 x 1 p)

b) Draw a possible structure of the final product. (2 p)

c) Calculate the molar weight of the final product (1 p)

d) What is the theoretical amount of the final product ? (1 p)

e) The approximate yield of your operation will be determined by the staff (2 p).