SwissChO Practical Exam 2024

CHEMISTRY.
OLYMPIAD.CH
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OLYMPIADES DE CHIMIE
OLIMPIADI DELLA CHIMICA

G0-1

English (Official)

General Information

Instructions

- You have 3:00 h to solve this practical exam. Before that, you have 10 min to look through the exam and ask any questions of understanding. Wait for the **START** signal before you begin with any laboratory work.
- Finish your work immediately, once the **STOP** signal has been given.
- Write your name on each question sheet.
- Only answers written on question sheets can be considered. You may use the reverse side if you require more space.
- Note down all necessary calculations, observations and data points in a legible manner.
- Do not communicate with other participants during the exam. You may quietly talk with the laboratory assistants.
- Stay at your designated desk. Only leave the working area if you have been given permission to do so.
- You may ask the laboratory assistants for more deionised water, ice or gloves. Requesting any more materials will lead to deduction of points.
- Hazardous or reckless behaviour may lead to expulsion from this exam.
- This exam has **3** problems.

Viel Erfolg!

Bonne chance!

Buona fortuna!

Good luck!

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Periodic Table of Elements

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1 H 1.008																		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 24.31											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 [98]	44 Ru 101.07	45 Rh 102.91	46 Pd 106.42	47 Ag 107.87	48 Cd 112.41	49 In 114.82	50 Sn 118.71	51 Sb 121.76	52 Te 127.60	53 I 126.90	54 Xe 131.29	
55 Cs 132.91	56 Ba 137.33	57–71	72 Hf 178.49	73 Ta 180.95	74 W 183.84	75 Re 186.21	76 Os 190.23	77 Ir 192.22	78 Pt 195.08	79 Au 196.97	80 Hg 200.59	81 Tl 204.38	82 Pb 207.2	83 Bi 208.98	84 Po [209]	85 At [210]	86 Rn [212]	
87 Fr [223]	88 Ra [226]	89–103	104 Rf [267]	105 Db [268]	106 Sg [269]	107 Bh [270]	108 Hs [270]	109 Mt [278]	110 Ds [281]	111 Rg [282]	112 Cn [285]	113 Nh [286]	114 Fl [289]	115 Mc [290]	116 Lv [293]	117 Ts [294]	118 Og [294]	

57 La 138.91	58 Ce 140.12	59 Pr 140.91	60 Nd 140.24	61 Pm [145]	62 Sm 150.36	63 Eu 151.96	64 Gd 157.25	65 Tb 158.93	66 Dy 162.50	67 Ho 164.93	68 Er 167.26	69 Tm 168.93	70 Yb 173.05	71 Lu 174.97
89 Ac [227]	90 Th 232.04	91 Pa 231.04	92 U 238.03	93 Np [237]	94 Pu [244]	95 Am [243]	96 Cm [247]	97 Bk [247]	98 Cf [251]	99 Es [252]	100 Fm [257]	101 Md [258]	102 No [259]	103 Lr [266]

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Score Sheet

NOT TO BE FILLED OUT BY PARTICIPANT

Name of participant: _____

Problem	Title	Maximum Points	Achieved Points	Pages
P1	Synthesis of Methyl Orange	33.0 pt		7
P2	Titration of an Unknown Acid	18.0 pt		4
P3	Oh my Beautiful Copper	25.0 pt		4
Total		76.0 pt		15

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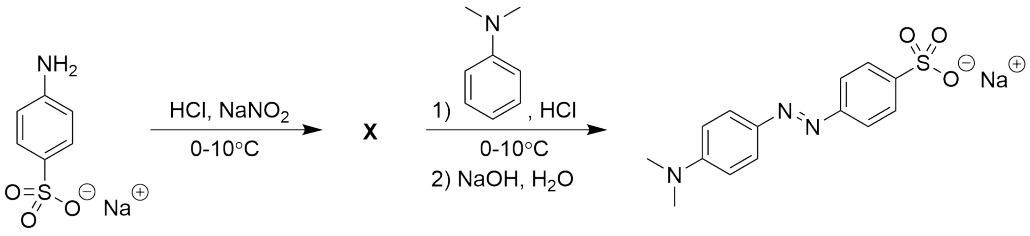
P1-1

English (Official)

Synthesis of Methyl Orange (33.0 pt)



In this task, you will synthesise an azo dye, methyl orange, and analyse the product using thin-layer chromatography. Azo dyes are a class of dyes usually prepared using electrophilic aromatic substitution.

Materials

- 1 × 10 mL measuring pipette
- 1 × 600 mL beaker for aqueous waste
- 1 × 50 mL round-bottomed flask
- 1 × glass vial
- 1 × metal spatula
- 1 × pencil
- 1 × stirring hotplate
- 1 × thermometer (−10 °C – 250 °C)
- 1 × UV lamp (shared by several people)
- 2 × stir bar
- 4 × capillaries
- 4 × Pasteur pipettes
- 1× reflux condenser (already connected to the cooling water)
- pH paper
- 1 × 100 mL round-bottomed flask
- 1 × 50 mL beaker
- 1 × Buchner funnel
- 1 × ice-water bath
- 1 × oil bath
- 1 × ruler
- 1 × suction bottle
- 1 × TLC chamber
- 1× watch glass
- 3 × TLC plates
- 4 × filter paper
- 4 × TLC vials
- 1 × wash bottle with deionised water (to be refilled without penalty)

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P1-2

English (Official)

Chemicals

- 10% aqueous sodium hydroxide solution ($NaOH_{(aq)}$)
- Methanol (CH_3OH)
- Sodium carbonate (Na_2CO_3)
- Hydrochloric acid (HCl): 3 mol L^{-1} aqueous solution and 1:6 conc. HCl:water solution
- N,N-Dimethyl aniline ($C_6H_5N(CH_3)_2$) (provided in a 100 mL round-bottomed flask clamped in an ice-water bath)
- Deionised water (H_2O)
- Sulfanilic acid ($NH_2C_6H_4SO_3H$)
- Sodium nitrite ($NaNO_2$)
- Eluent: 6:1 propan-1-ol (C_3H_7OH):28% ammonia solution ($NH_{3(aq)}$)

Procedure

1. In a little TLC vial, **set aside** a tip of a spatula of sulfanilic acid ($NH_2C_6H_4SO_3H$) and **dissolve** it in approximately 1 mL of methanol.
2. In a 50 mL beaker, **add** the remaining sulfanilic acid (2.1 mmol, $NH_2C_6H_4SO_3H$) and **add** 7 mL of deionised water.
3. Slowly **add** the given amount (0.13 g) of sodium carbonate to neutralise the acid.
4. **Add** a stir bar to the beaker and **stir** until a clear solution is obtained.
5. Meanwhile, **add** the given amount (0.2 g) of sodium nitrite to a small glass vial and **dissolve** it using approximately 1 mL of deionised water.
6. **Dropwise**, **add** this solution to the beaker containing the neutralised sulfanilic acid solution using a Pasteur pipette.
7. In a 50 mL round-bottomed flask, **cool** 4 mL of HCl (1:6 concentrated HCl:water) using an ice-water bath.
8. **Add** a stir bar to the HCl-solution and start stirring.
9. **Slowly add** the solution from step 6 to the cooled HCl-solution via a Pasteur pipette.
10. **Make sure that the temperature of the reaction mixture never exceeds** 10 °C **throughout the addition!**
11. Once you have added the entire solution, **keep stirring** for at least two minutes.
12. **Keep** the solution containing intermediate **X** cold.
13. **Dissolve** the provided amount (2.1 mmol) of N,N-dimethyl aniline, already contained in a 100 mL round-bottomed flask, clamped in an ice-water bath, in 10 mL of 3 mol L^{-1} HCl and **cool** to 5 °C.
14. **Add** the previously prepared cold solution of intermediate **X** to the 100 mL round-bottomed flask dropwise, using a Pasteur pipette.
15. **Keep** the reaction mixture cool during the addition.
16. After the entire addition, **keep** the solution **stirring** for at least 15 minutes.
17. **Slowly add** 10% NaOH solution to your reaction mixture, until a pH of roughly 8 is reached. **Check** the pH with pH paper.
18. Use a Buchner funnel, filter paper and a suction bottle to **filter** the precipitate under vacuum.
19. **Take** a spatula tip's worth of your product, **transfer** it into a separate TLC vial and **dissolve** it in approximately 1 mL of methanol.

Purification

1. **Rinse** the previously used 100 mL round bottom flask several times with deionised water. You may **add** a few drops of aqueous hydrochloric acid to help dissolve any residues. **Don't forget** to remove your stir bar and clean it as well!
2. **Transfer** your product from the filter paper into the 100 mL round bottom flask.
3. **Add** a stir bar to the flask.
4. **Add** approximately 20 mL of deionised water and start stirring.
5. **Add** 10% NaOH solution to your mixture until the solution is slightly basic (check with pH paper).
6. **Connect** the flask to a reflux condenser.
7. **Raise your hand** to show the setup to the assistant before continuing.

8. While stirring, **heat** the mixture to boiling in a hot oil bath (**NEVER HEAT THE OIL BATH ABOVE 110°C, check continuously with a thermometer**) and little by little, add more deionised water, until the solid is dissolved completely.
9. When all of the solid is dissolved, **stop** the heating and **take** the flask out of the hot oil bath.
10. Wait until the mixture **is cooled down** to room temperature, and then further **cool** it to 0 °C in an ice water bath.
11. Once no more solid is crystallising out, use a Buchner funnel, filter paper and a suction bottle to **filter** the precipitate.
12. **Wash** the solid thrice with a little ice-cold deionised water.
13. **Let** air suck through the filtered product for 2-3 min.
14. **Disconnect** the vacuum source and let the product **air-dry** for at least 15 minutes.
15. **Transfer** the product from the filter paper to a watch glass signed with your name and place it on your bench.
16. **Take** a small amount of your product, **transfer** it into a separate TLC vial and **dissolve** it in approximately 1 mL of methanol.

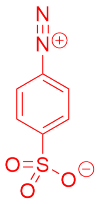
Analysis

1. **Perform** a TLC analysis of the sample of your product before purification and sulfanilic acid using 6:1 propan-1-ol:28% ammonia solution as eluent. **Be careful** when opening the eluent bottle as there can be an evolution of ammonia.
2. **Perform** a TLC analysis of the sample of your product after purification and sulfanilic acid using the same eluent as above. **Be careful** with the release of ammonia.
3. Once you are ready to view all your TLCs under the UV lamp, **raise** your hand. A laboratory assistant will escort you to the lamp.
4. **Make sure** to label your TLC plate with your name such that it can be handed in at the end of the exam.
5. **Make sure** to label the two TLCs you handed in, in a way that it is clear, which one was done before and which one after purification.

Theoretical Questions

1.1 **Draw** the structure of intermediate **X**. 1.0 pt

SOLUTION:



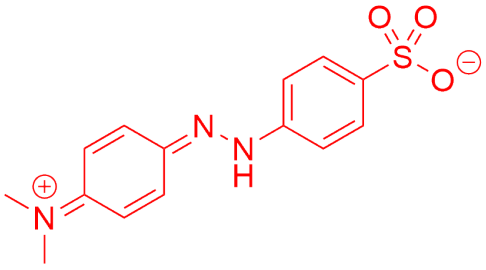
-0.5 pt if wrong charges

1.2 In this reaction, the *para*-product is formed selectively. **Explain** why this is the case, and support your reasoning with resonance structures. 5.0 pt

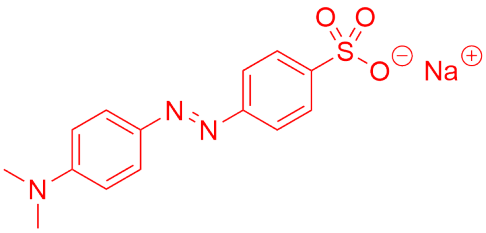
SOLUTION:
See section **SOLUTION TO 1.2** below.

1.3 The formed dye is a pH indicator, with the colour change between pH = 3-4. 2.0 pt
Suggest the structure of the dye in the solution at pH 2 and at pH 5.

SOLUTION:
At pH 2 the dye exhibits a red colour, and it is protonated:



At pH 5 the dye exhibits an orange colour, and it is deprotonated:



1.4 According to your TLC before purification, **provide** the R_f value of the product. 1.0 pt

$R_f =$ _____

SOLUTION:

Full points awarded if the correct spot was chosen and the R_f value correct according to the handed-in TLC.

1.5 According to your TLC before purification, **state**, whether your product still contains starting material. 1.0 pt

☐ Yes ☐ No

SOLUTION:

Full points awarded if the statement is consistent with the handed-in TLC. (Check the spots under UV lamp again)

1.6 **State**, whether you expect the R_f value of the product to change after the purification. **Confirm** your statement by comparing the R_f values from your TLC analyses before and after the purification. 2.0 pt

☐ R_f changes ☐ R_f does not change

$R_f =$ _____

SOLUTION:
1 point for correct calculation of the R_f value (chose the correct spot)
1 point for stating that it does not differ from the value obtained before purification as it is an intrinsic property of the compound itself and does not change upon purification (just the overall looks of the TLC plate will change ;))

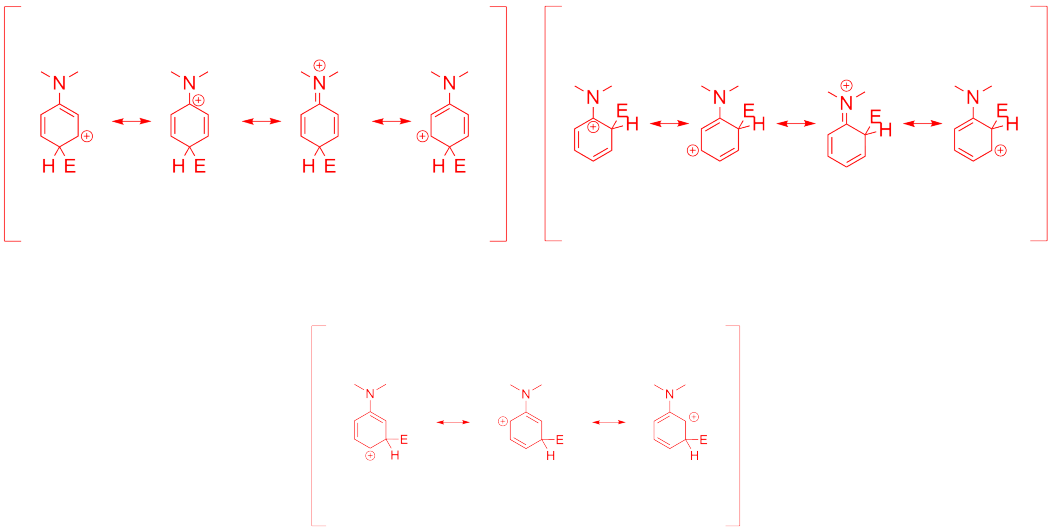
1.7 According to your TLC after purification, **state**, whether your product still contain starting material. 1.0 pt

☐ Still contains starting material ☐ Does not contain starting material

SOLUTION:
Full points awarded if the statement is consistent with the handed in TLC. (Check the spots under UV lamp again)

SOLUTION TO 1.2:

The intrinsic electronic reactivity of the N,N-dimethyl aniline favours otho- and para- electrophilic substitution, as we can rationalise by the more stable cationic intermediate compared to the meta- substitution. We can draw more resonance structures for the ortho- and para-case than for the meta-intermediate:



The para-position is favoured over the ortho-position even though the ortho-position is statistically favoured (2 possible ortho-positions vs. 1 possible para-position) due to steric reasons, as the electrophile in this reaction is rather bulky.
-1 point if no mentioning of ortho- vs. para-
-1 point if no resonance structures for each different case (1 Point for ortho, meta and para separately)
-1 point if no mentioning of ortho- / para- substitution over meta- substitution

POINTS FOR THE PRODUCT

+5 pt for each good TLC that was handed in (no overspotting, letting it run to a good height); 10 pt total!
+10 pt for yield over 50%, afterwards decrease linearly to 0 pt for 0%.

NOTES FOR THE PREPARATION

- Control the cooling water for the reflux condensers the evening before and tighten everything well
- Make sure to also check during the exam that no plastic tubing is ever touching the hot-plate and tell the students as well throughout the week
- Due to safety concerns, the students will not receive the N,N-dimethylaniline in a vial but we will prepare it for them already in a 100 mL round bottom flask and clamp it over the ice bath to avoid exposure to it and any spillage

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P2-1

English (Official)

Titration of an Unknown Acid (18.0 pt)

Materials

- 1 × 10 mL volumetric pipette
- 1 × 100 mL volumetric flask
- 1 × 600 mL beaker for aqueous waste
- 1 × water bottle
- 1 × 100 mL beaker
- 1 × 50 mL burette
- 1 × small glass funnel
- 3 × 200 mL wide-necked Erlenmeyer flask

Chemicals

- 1% phenolphthalein in ethanol
- 0.050 mol L^{-1} sodium hydroxide (NaOH) solution
- Deionised water
- A pre-dissolved sample of an unknown acid in a 100 mL volumetric flask (the possible acids are listed below in the section "list of possible acids in the sample solution")

Procedure

1. You have received a pre-dissolved sample of an unknown acid in a 100 mL volumetric flask. The mass of the dissolved sample is indicated on the label on the volumetric flask. **Write down** the mass of the sample below:

   $m_{\text{sample}} =$ _____ g
2. **Fill up** the 100 mL volumetric flask with deionised water to the marking.
3. **Transfer** 10.0 mL of your solution from the 100 mL volumetric flask into a 200 mL Erlenmeyer flask.
4. You **may** add some deionised water to the solution for better visualisation during the titration.
5. **Add** 4 to 5 drops of phenolphthalein indicator solution.
6. **Fill** a 50 mL burette with the provided 0.050 mol L^{-1} aqueous sodium hydroxide solution.
7. **Titrate** your solution while gently swirling the flask.
8. **Repeat** steps 3 to 8 at least two more times, and note down the volume of consumed sodium hydroxide solution.
9. **Report** the final value used for later calculations in the last row.

Titration run	**Volume consumed** [mL]
1	

2	
3	
4	
5	
Your accepted result	

Theoretical Questions

2.1 **Calculate** the number of moles of acidic protons contained in your provided sample. **Clearly show** your calculations. 3.0 pt

$n_{\text{acidic protons}} =$ _____ mol

SOLUTION:

$$n_{\text{acidic protons}} = 10 \cdot c(\text{NaOH}) \cdot V(\text{NaOH}) \quad (1)$$

+ 1 pt if three or more titrations were preformed
+ 1 pt if correct formula is given, 0.5 if the factor 10 is forgotten (10 mL taken from 100 mL)
+1 pt if correct result for the obtained titration result with the given formula (approx. 15.6 mmol)

2.2 **Derive** a *formula* for the molar mass M_{acid} of the acid, using the following quantities: 3.0 pt

- the mass of your sample m_{sample} in g
- the concentration of your NaOH solution c_{NaOH} in mol L^{-1}
- the volume of your NaOH solution used V_{NaOH} in mL
- the number of acidic protons per acid x

$M_{\text{acid}} =$ _____

SOLUTION:

$$M_{\text{acid}} = \frac{100 \cdot m_{\text{sample}} \cdot x}{c_{\text{NaOH}} \cdot V_{\text{NaOH}}}$$

The factor 100 comes from the fact that an aliquot of a tenth (factor 0.1) was taken, and we are comparing mole/L and mL (factor 1000).
+2 points for the correct derivation of the formula (without the factor)
+1 point for the correct conversion factor (100)

2.3 **Determine** the acid contained in your sample using the table provided below. 3.0 pt
Clearly show your calculations.

Acid: _____

SOLUTION:

The acid contained in the sample can be identified via its molar mass. The students should notice that there are mono-, di- and tri-protic acids in the provided list. To be completely sure, they can repeat the calculations for all these options, and they should end up with the correct result if they have titrated accurately. They can use the formula derived above to determine different molar masses.
+1 point for calculation of molar mass
+2 points if acid correct

POINTS FOR ACCURACY

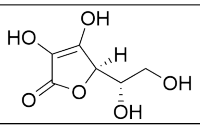
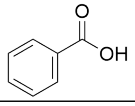
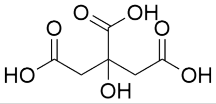
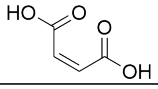
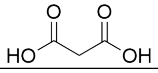
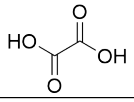
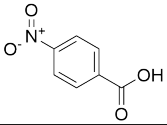
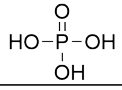
+ 9 points for the titration part: full points awarded if they are within one standard deviation of the master value and then linear decrease to 0 points within an additional standard deviation of the master value

NOTES FOR THE PREPARATION

- The acid to be used is citric acid (use the anhydrous one!)
- The amount they get should be around 1 g, such that the final concentration in the 100 mL volumetric flask is around 0.05 mol L^{-1} and they will thus need around 30 ml of their sodium hydroxide solution per titration

List of possible acids in the sample solution

Name	**Structure**	**pK_a value(s)**	**Molar mass** [g mol^{-1}]
Acetic acid		4.76	60.052

Ascorbic acid		4.10, 11.6	176.12
Benzoic acid		4.20	122.123
Citric acid		3.13, 4.76, 6.40	192.123
Hydrochloric acid	H—Cl	-5.9	36.46
Maleic acid		1.90, 6.07	116.072
Malonic acid		2.83, 5.69	104.061
Oxalic acid		1.25, 4.14	90.034
para-Nitrobenzoic acid		3.41	167.1189
Phosphoric acid		2.14, 7.20, 12.37	97.994

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P3-1

English (Official)

Oh my Beautiful Copper (25.0 pt)

In the following task, you will analyse different complexes and perform ligand-exchange reactions and determine, which metal-ligand combinations afford the most stable complexes.

Materials

- 1 × 10 mL measuring pipette
- 1 × glass filter crucible
- 1 × spatula
- 1 × stirring hotplate
- 1 × 100 mL graduated cylinder
- 1 × 600 mL beaker (labelled with aqueous waste)
- 1 × 100 mL Erlenmeyer flask
- 1 × ice-water bath (to be refilled without penalty)
- 1 × stir bar
- 1 × suction bottle
- 6 × Pasteur pipettes
- 1 × wash bottle with deionised water (to be refilled without penalty)

Chemicals

- Salicylaldoxime ($C_7H_7NO_2$)
- Acetylacetone ($C_5H_8O_2$)
- Copper(II)sulfate ($CuSO_4$)
- Potassium oxalate monohydrate ($K_2C_2O_4 \cdot H_2O$)
- 2 mol L^{-1} acetic acid, AcOH (CH_3COOH)
- Half-conc. Ammonia solution ($NH_{3(aq)}$)
- Deionised water (H_2O)

Procedure

1. **Dissolve** the sample of copper(II)sulfate in 2 mL of deionised water in a 100 mL Erlenmeyer flask. This yields **Complex 1**.
2. **Dropwise, add** concentrated ammonia solution until any intermediately formed precipitate is just dissolved again, and you obtain a clear solution. **Be careful** when adding the ammonia solution due to the evolution of ammonia. This yields **Complex 2**.
3. **Heat** the solution to boiling while stirring.
4. **Dissolve** the provided potassium oxalate in 5 mL of water and **add** it to the boiling solution.
5. **Cool** the solution in an ice-water bath. This yields **Complex 3**.
6. **Let** the solution warm up to room temperature.
7. **Dropwise, add** the provided sample of acetylacetone to the solution.
8. **Stir** the solution for 10 min at room temperature. This yields **Complex 4**.
9. **Add** 20 mL of 2 mol L^{-1} AcOH with a graduated cylinder to the beaker while stirring. The pH value should be between 3 and 5.
10. **Check** the pH of your solution with a pH indicator strip and **add** more AcOH with a plastic pipette

if necessary.

11. **Add** the provided salicylaldoxime to the beaker while stirring.
12. **Stir** the solution for 10 minutes at room temperature. This yields **Complex 5**.
13. While the solution stirs, **legibly label** your glass filter crucible with your name.
14. Using the glass filter crucible, **filter** the formed precipitate under a vacuum and **wash** it a few times with ice-cold deionised water.
15. **Let** air suck through the filtered product for 2 – 3 min.
16. **Hand in** the glass filter crucible with **Complex 5** to the assistant.

Theoretical Questions

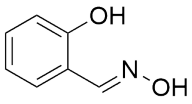
3.1 From the procedure above, **determine** the different complexes formed. **Indicate** their chemical formulae, **describe** their colours, and **draw** out their structures. **Use** the table provided below. **Hint:** use the hints provided below. 18.0 pt

Complex	Chemical formula	Colour	Structure
1			
2			
3			
4			
5			

Hints for 3.1:

- Oxalic acid = ethanedioic acid
- Abbreviations for common ligands: oxalato (ox), acetylacetonato (acac) and salicylaldoximato (sal)

- From **Complex 1** to **Complex 2** only four ligands are exchanged
- From **Complex 2** to **Complex 3** only four coordiation sites are changed
- **Complex 4** has a coordination number of six
- **Complex 5** only has a coordination number of four



Salicylaldoxime

3.2 From the experiments performed above, **order** the ligands in terms of the stability of the formed copper complexes. 2.0 pt

SOLUTION:
The order for the strength of the ligands corresponds exactly to the order, in which the ligands are added after each other since the stronger ligands will be able to displace the weaker ones from the metal centre. The other way around would not be possible.
sal > acac > ox > NH_3 > H_2O
-0.5 Points for each wrongly ordered ligand, minimum 0 Points

3.3 **Name** two aspects, which help explain the stability of **Complex 5**. 2.0 pt

SOLUTION:
1 Point for chelation through the bidentate sal ligands
1 Point for the intramolecular hydrogen bonds

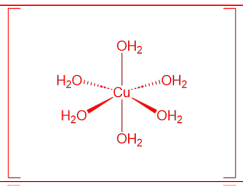
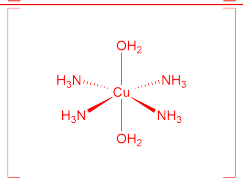
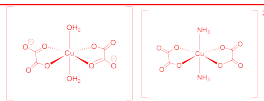
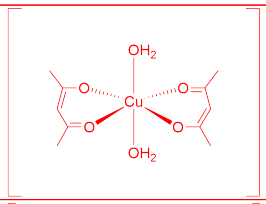
NOTES FOR THE PREPARATION

- Give them anhydrous (white) $CuSO_4$ (0.84 mmol, 0.134 g)
- Potassium oxalate monohydrate (1.7 mmol, 0.31 g)
- Salicylaldoxime (5.1 mmol, 0.700 g)

POINTS FOR COMPLEXES

+ 3 Points for handing in the final complex (they performed the experiment ;))
-2 Points if they hand in the wrong complex e.g. it has a different colour
-1 Points if it's only a little bit of solid

SOLUTION TO 3.1

Complex	Chemical formula	Colour	Structure
1	$[Cu(OH_2)_6]^{2+}$ or $[Cu(OH_2)_6](SO_4)$	Light blue	
2	$[Cu(NH_3)_4(OH_2)_2](SO_4)$ or $[Cu(NH_3)_4(OH_2)_2]^{2+}$, **and 0.5 pt for** $[Cu(NH_3)_6](SO_4)$ or $[Cu(NH_3)_6]^{2+}$	Deep purle/Dark blue	
3	$K_2[Cu(OH_2)_2(ox)_2]$ or $[Cu(OH_2)_2(ox)_2]^{2-}$ **or 0.5 pt for** $K_2[Cu(NH_3)_2(ox)_2]$ or $[Cu(NH_3)_2(ox)_2]^{2-}$	Turquoise blue / light blue	
4	$[Cu(acac)_2(OH_2)]$ **or 0.5 pt for** $[Cu(acac)_2(NH_3)]$	Purple	
5	$[Cu(sal)_2]$ **or 0.5 pt for** $[Cu(sal)_2(OH_2)_2]$ **or** $[Cu(sal_2)(NH_3)_2]$	Green