

44th Austrian Chemistry Olympiad

Federal Competition

Practical Part

June 1st, 2018

Solutions

Problem 6 56 bp ≙ 16 rp

Synthesis of a Sweetener

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| 6.1 Present your raw product to the supervisor to obtain confirmation. |
| Raw product was obtained: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ (paraph)  **3 bp\*** |
| 6.2 Caclulate your yield in g and % of theory. |
| mass tara: 48.32 g mass product *m*p: 1.76 g **2 bp**rating of the yield **0-29 bp\***theor. yield.: *m* = $1.70∙\frac{180.20}{137.18}=2.23 g$ (KOCN - 17 mmol excess )own yield in $\%=\frac{m\_{p}}{2.23}∙100=…\%$ **2 bp**appearance of the product: **0-3 bp** |

\*if $1.76 g \leq m\_{p} \leq 2.23 g$ → 29 bp; if *m*p > 2.23 g → 0 bp

 otherwise: $bp= 29 ⋅\left(\frac{m\_{p}}{1.76}\right)$ ; if no product but raw product obtained: 3 bp

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| 6.3 Give the melting point of your product: |
| 174-176°C **0-3 bp** |
| 6.4 Give the Rf values: |
| Rf-value educt: 0.35 Rf-value crude product: 0.64 Rf-value product: 0.64 **3 bp**TLC-Grading: 2 lines, labeling **2 bp**size of the spots & labeling, TLC-quality **5 bp** |
| 6.5 Briefly explain the reason for the different Rf values of educt and product. |
| educt is more polar than the product therefore, retention factor is smaller for the educt **2 bp** |
| 6.6 Tick all correct ways of interpreting the thin layer chromatogramme. You will lose points for ticking wrong boxes. However, you cannot reach a negative number of points within 6.6. |
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|  | Two substance spots for RP indicate complete reaction. |
|  | Two substance spots for RP indicate a high yield. |
| X | Two substance spots for RP indicate contamination by a side product. |
| X | Two substance spots for RP indicate contamination by the educt. |
|  | Two substance spots for the RP and one substance spot for P indicate further reaction during work-up. |
| X | Two substance spots for the RP and one substance spot for P indicate that the contamination has been removed during work-up. |

each correctly ticked box 1 bp, the wrong one - 1bp min 0 bp **max. 3 bp** |

Problem 7 51 bp ≙ 8 rp

Qualitative Analysis

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| Complete the table based on the results of your analyses. |
|  | formula | justification |
| 1 | Na2S2O3**1 bp**  **3 bp** | Na+: exclusion **1 bp**S2O32-: reactions with Ag+  **1 bp** |
| 2 | Fe(NO3)3**2 bp** **2 bp** | Fe3+: typical own color, precipitate with OH-, red color with NaSCN **1 bp**NO3-: Kein precipitate with Ag+ und Ba2+ **1 bp** |
| 3 | NaSCN**1 bp** **2 bp** | Na+: exclusion **1 bp**SCN-: whit precipitate with Ag+,red color with Fe3+ **1 bp** |
| 4 | Pb(NO3)2**3 bp** **2 bp** | Pb2+: yellow precipitate I-, white precipitate with OH- (soluble in excess of OH-) **1 bp**NO3-: no precipitate with Ag+ and Ba2+ **1 bp** |
| 5 | ZnI2**3 bp** **3 bp** | Zn2+: white precipitate with S2-, white precipitate with OH- (soluble in excess of OH-) **1 bp**I-: yellow precipitate with Pb2+, yellowish precipitate with Ag+ **1 bp** |
| 6 | Na3PO4**1 bp** **4 bp** | Na+: exclusion **1 bp**PO43-: alkaline pH-valuegelber precipitate with Ag+ (soluble in HNO3), **1 bp** |
| 7 | HNO3**2 bp** **2 bp** | H3O+: acidic pH value **1 bp**NO3-: no precipitate with Ag+ and Ba2+ **1 bp** |
| 8 | NaHSO4**1 bp** **3 bp** | Na+: exclusion **1 bp**HSO4-: white precipitate with Ba2+, acidic pH-value **1 bp** |

Aufgabe 8 54 bp ≙ 16 rp

Quantitative Analyse:
Bestimmung von Eisen und Aluminium in einer Probe

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| 8.1 Report your titration volumes. |
| „VZn“ = 11 mL **0-12 bp**\* | „VFe“ = 15 mL **0-16 bp** | „VAl“ = 15 mL **0-22 bp** |

$$ΔV = \left|V\_{actual}-V\_{theoretical}\right|$$

Zn: $ΔV\leq 0.05 mL⇒12 bp$; $ΔV>0.50 mL⇒0 bp$ $bp=12\*(1-\frac{\left|∆V\right|-0.05}{0.45})$

Fe: $ΔV\leq 0.09 mL⇒16 bp$; $ΔV>0.70 mL⇒0 bp$ $bp=16\*(1-\frac{\left|∆V\right|-0.09}{0.61})$

Al: $ΔV\leq 0.15 mL⇒22 bp$; $ΔV>0.90 mL⇒0 bp$ $bp=22\*(1-\frac{\left|∆V\right|-0.15}{0.75})$

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| 8.2 Calculate the concentrations. |
| c(Zn2+) = 0.055M  | c(Fe3+) = 0.0745M  | c(Al3+) = 0.0724M  |
| *c*(Zn2+):*V*(EDTA) = 11 mL *c*(EDTA) = 0.05 mol/L => *n*(EDTA) = *c* ⋅ *V* = 0.05 ⋅ 11 = 0.55 mmol*n*(Zn2+) = *n*(EDTA) = 0.55 mmol => *c*(Zn2+) = $\frac{n}{V}=\frac{0.55}{10}=$ $0.055 mol/L$ **1 bp***c*(Fe3+):*V*(EDTA) = 14.9 mL *c*(EDTA) = 0.05 mol/L=> *n*(EDTA) = *c* ⋅ *V* = 0.05 ⋅ 14.9 = 0.745 mmol*n*(Fe3+) = *n*(EDTA) = 0.745 mmol => *c*(Fe3+) = $\frac{n}{V}=\frac{0.745}{10}=$ $0.0745 mol/L$ **1 bp***c*(Al3+):*V*(EDTA) = 14.1 mL *c*(Zn2+) = 0.055 mol/L=> *n*(Zn2+) = c ⋅ V = 0.055 ⋅ 14.1 = 0.7755 mmol*n*Zn(EDTA) = *n*(Zn2+) = 0.7755 mmol*n*ges(EDTA) = *c* ⋅ *V* = 0.05 ⋅ 30 = 1.50 mmol*n*Al(EDTA) = *n*ges(EDTA) – *n*Zn(EDTA) = 1.50 – 0.7755 = 0.725 mmol*n*(Al3+) = *n*Al(EDTA) = 0.725 mmol => *c*(Al3+) = $\frac{n}{V}=\frac{0.725}{10}=$ $0.0725 mol/L$ **2 bp** |