

43rd Austrian Chemistry Olympiad

National Competition

Practical Tasks

2017-05-26

Solutions

Task 8 42.5 bp ≙ 16 rp

Synthesis of a white powder out of the natural identic compound „vanillin“

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| 8.1 Show the lab assistant the crude product and wait for confirmation. |
| Crude product was present: **0-3 bp** |
| 8.2 Write down the equation for the synthesis. Use constitutional formulae for this purpose. |
| D:\Schule_privatDatensicherung_20.8.2015\Schule_Uni_20.8.2015\Chemie\Bundeskoordinatorenteam_Olympiade\Bundeswettbewerb2017\Wettbewerb\Praxis\Reaktion.png **2 bp** |

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| 8.3 Calculate your yield in g and % of the theoretical mass. |
| mass tara: 10.3g mass product: 1.5 g **0-19 bp\****m* = $3.00∙\frac{154.17}{152.15}=3.04 g$ $\%=\frac{1.50}{3.04}∙100=49.34$ **2 bp**appearance of product: **0-3 bp** |

\*yield >= 30% – 19 bp yield < 30%: $bp=\left(\frac{19}{0.3}\right)⋅\left(\frac{m}{3.04}\right)$

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| 8.4 Determine and write down the melting point. |
| 118-124°C **0-3 bp** |
| 8.5 Determine and write down your Rf values.... |
|  Rf-value starting material: 0.76 **1 bp** Rf-value product: 0.65 **1 bp**Criteria fort he TLC plate: 2 lines (1bp), labeling (1bp), spot size and spot labeling (3bp) **5 bp** |
| 8.6 Explain verbally and with an equation why the use of NaOH in the first step is necessary. |
|  *D:\Schule_privatDatensicherung_20.8.2015\Schule_Uni_20.8.2015\Chemie\Bundeskoordinatorenteam_Olympiade\Bundeswettbewerb2017\Wettbewerb\Praxis\Reaktion.png* The addition of NaOH causes an acid base reaction between the phenol (acid) and base NaOH. The resulting sodium phenolat is dissociated in water and exhibits a higher solubility caused by the ionic charge. **2 bp** |
| 8.7 Explain the reason for the different Rf-values between starting material and product. |
| The different Rf-values reflect the different polarities between starting material (less polar) and the product (more polar). The product exhibits a hydroxyl group which has a stronger interaction with the polar stationary phase. Thus the Rf-value of the product is decreased. **1.5 bp** |

Task 9 52 bp ≙ 8 rp

Qualitative Analysis

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| 9.1 Complete the table according to your analysis results. |
| Sample no. | formula | reasons |
| 1A | CuCO3**2bp 2bp** | Cu2+: natural color, flame color, color of the precipitate with hydroxide ions **1 bp**CO32-: reaction wih HNO3 **1 bp** |
| 1B | FeSO4**3bp 4bp** | Fe2+: natural color, color of the precipitate with hydroxide ions, reaction with KMnO4 **1 bp**SO42-: reaction with neutralized Ba(OH)2 **1 bp** |
| 2 | Ba(OH)2**2bp 2bp** | Ba2+: flame color + natural color (colorless) **1 bp**OH-: reaction with AgNO3 **1 bp** |
| 3 | CaC2O4**3bp 4bp** | Ca2+: flame color, sample is not soluble in water **1 bp**C2O42-: reaction with acidic KMnO4 -solution **1 bp** |
| 4 | CrCl3**3bp 2bp** | Cr3+: natural color, color of the precipitate with hydroxide ions, color of the precipitate with sulfide ions **1 bp**Cl-: color of the precipitate with AgNO3 **1 bp** |
| 5 | ZnI2**3bp 2bp** | Zn2+: color of the precipitate with sulfide ions **1 bp**I-: color of the precipitate with AgNO3 **1 bp** |
| 6 | Al(NO3)3**4bp 2bp** | Al3+: natural color (colorless), precipitate with hydroxide ions is soluble in excess of hydroxide ions + no precipitation with sulfide ions **1 bp**NO3-: no precipitation with Ag+ or Ba2+ **1 bp** |

Task 10 53 bp ≙ 16 rp

Quantitative Analysis: Determination of a complex formation constant

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| 10.1 Write down your titration volume (average value). |
| V = 9.62 mL **0-16 bp\*** |
| 10.2 Calculate the concentration of your potassium permanganate solution. |
| c(C2O42-)=0.005M → 0.125 mmol C2O42- in 25mL → 0.05 mmol MnO4- necessaryc(MnO4-) = 0.05 mmol/9.62 mL = 0.0052 mol/L **2 bp** |
| 10.3. Write down your titration volume (average value). |
| V = 9.42 mL **0-16 bp\*** |
| 10.4. Calculate the concentration of your iron(III) chloride solution. |
| *n*(MnO4-) = 0.0052 mol/L · 9.42 mL = 0.049 mmol → *n*(Fe3+) = 0.245 mmol **1 bp***c*(FeCl3) = 0.245 mmol/25.0 mL = 0.0098 mol/L **1 bp** |

\*: $∆V=V\_{ideal}-V\_{actual}$

 $\left|∆V\right|\leq 0.15mL⇒16 bp$

$ \left|∆V\right|>0.95 mL⇒0 bp$

 $0.15<\left|∆V\right| \leq 0.95 mL ⇒$ $bp=16\*(1-\frac{\left|∆V\right|-0.15}{0.8})$

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| 10.5. Write down the measured absorption values and the calculated concentrations of FeCl3 on the evaluation sheet and hand it over to the lab assistant. |

points for the **photometry**: 0.5 bp per measurement **max** **2.5 bp**

absorptions (example with correct values):

1: 0.203 – 2: 0.342 - 3: 0.490 – 4: 0.585 - 5: 0.663 ; rating for each measurement:

 $\frac{|ε\_{ideal}- ε\_{actual}|}{ε\_{ideal}}\leq 0.1⇒2.5 bp$ **max. 12.5 bp**

$0.1<\frac{|ε\_{ideal}- ε\_{actual}|}{ε\_{ideal}}<0.2⇒1 bp$

measurement less accurate: 0 bp

Example for computer evaluation on the last page

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| 10.6. Calculate $ε$and $K\_{β}$ using these numeric values. |
| d = 1cm$\frac{1}{εd}=2.105⋅10^{-4}\frac{mol}{L} ⇒ ε=4963 L mol^{-1}cm^{-1}$ 1 bp$\frac{1}{K\_{β}εd}=1.328⋅10^{-6}\frac{mol}{L} ⇒ K\_{β}=\frac{1}{1.328⋅10^{-6}⋅ ε⋅d }≈152 $ 1 bp |

