ŠPŠCH Pardubice
Professional field of action: „Working in a chemical laboratory“
Applied chemistry

LEE3: 1. Determination of 9-ACA in a final product within an output control

You are a laboratory technician in a company which, among other businesses, produces fine chemicals. You are responsible for the output quality control of the final products. Your today's task is to determine the content of 9-ACA in the final product by automatic potentiometric titration. You are provided with the relevant equipment. You are supposed to perform the procedure by following working instructions and specify the content of 9-ACA in the sample in mass%, then to compare your findings with the technical requirement for the final product.

Knowledge of procedure Characterization of working activity		**Expertise** Characterization of working systems	
Working steps	**Skills/abilities**	**Context of natural science**	**Context of technology**
Working tasks: 1. Determine the content of 9-anthracene carboxylic acid in the final product by automatic titration with 0.1M NaOH. 2. Express the content of 9-anthracene carboxylic acid in		Determination of carboxylic functional group is based on an an acid-base titration. The acid is titrated with standard solution of 0,1 M NaOH with the help of automatic titration. The equivalence point is indicated with pH-electrode	

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the sample in mass %. 3. Compare the obtained result with the technical requirements for the quality assurance of the product		and inbuilt reference electrode.	
Analysis of the assignment Analysis of the task. Planning and organizing work. Identifying risks. Identifying workplace health&safety rules.	• analyzing the assignment of the task • planning the partial working steps • organizing work • time-management to finalize the task in given time • researching potential risk of chemicals to be handled with (according to GHS classification) • respecting workplace health&safety rules		
Labware: automatic titrator, pH-electrode with an inbuilt reference electrode, analytical scales, stirrer, common glass ware	• collecting lab-ware, chemicals, devices • proper use of laboratory instruments and equipment • proper use of measuring glass ware (pipettes, burettes, volumetric flasks) • accurate performing of operations	The device consists of automatic burette, titrator equipped with microprocessor, pH measuring cell and stirrer. Microprocessor inbuilt is examining the course of the first derivative and stops the titration when the maximum value of dE/dV is reached.	

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	• operating the device by following a manual • keeping equipment clean and in a good working order • saving energy, water and other consumables	The software enables to input a formula, weights, dilution, titrant´s concentration and figure out the content of the substance in the sample.	
Chemicals: concentrated solution NaOH 50 mass % solution NaOH, c = 0,1 mol/l potassium hydrogenphthalate p.a. mixture EtOH-H_2O in volume ratio 4:1 **Preparation of 2 L NaOH solution c(NaOH) = 0,1 mol/L** Pipette 10,5 mL of NaOH 50% with graduated pipette and transfer the volume into 2 L volumetric flask, fill the solution with distilled water free of CO_2 up to the mark.	• identifying the potential risk (emphasis on H, P sentences, safety of work and environmental protection) • compliance with the occupational health and safety policy • abiding by the respective safety regulations to minimize the risk • preparing solution by dilution of the substance concentrated solution • precise filling up solution in volumetric flask • using protective goggles and gloves when handling with the concentrated solution of NaOH	Potential risks of chemical substances according to GHS.	
Preparation of 2 L of EtOH-H_2O solution (4:1) Mix 1600 mL ethanol + 400 mL water in 2 L volumetric flask.	• calculating volumes of each solvent to prepare 2 L of mixture 4:1. • mixing liquids in a defined ratio		

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Preparation of automatic titrator for analysis Fill the automatic burette with 0,1 M NaOH. Setting-up automatic titrator.	• rinsing and filling up automatic burette • following user manual to set-up device for measurements	Microprocessor inbuilt is examining the course of the first derivative and stops the titration when the maximum value of dE/dV is reached. The software enables to input a formula and figure out the content of the compound in a sample.	
Blank value determination Measure 200 mL of EtOH-H_2O mixture. Titrate it with 0,1 M NaOH. The blank sample value represents one fourth of the consumed 0,1 M NaOH volume. Perform the titration twice.	• setting-up the experiment • inputting data required for calculations • following user manual to perform titration • assembling the titration unit	The volume of NaOH consumed for the blank sample titration is divided by 4 and subtract from the volume consumed for the sample titration	
Standardization of NaOH solution Weigh 0,27g potassium hydrogenphtalate (analytical scales), add 50 mL of water and dissolve the substance, titrate it with 0,1 M NaOH solution. Perform the titration twice.	• weighing (analytical scales) and dissolving the substance • inputting data required for calculations • following user manual to perform titration		
Determination of 9-ACA Weigh 0,2 - 0,25 g of 9-ACA (analytical scales) straight into titration flask, dissolve the sample in	• weighing (analytical scales) and dissolving the substance • inputting data required for calculations		

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50 mL of ethanolic solution and titrate it with 0,1 M NaOH. Perform the titration twice.	• following user manual to perform titration		
Evaluation: Make records on readings. Make calculations. Compare the obtained result with the required value. Interpret the result. Evaluate the measurement.	• making records about measurements • data processing • making calculations • evaluating and presenting results and findings	The minimum content of 9-ACA in the final product is to be 98 mass%.	

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