Determination of paracetamol and calcium carbonate by UV-spectrophotometry and complexometric titration

Description of the assignment

The sample flasks contain the material for a tablet, whose composition is given as follows:

ca. 100 mg paracetamol
ca. 350 mg calcium carbonate
ca. 150 mg potassium chloride

This medicine is for the treatment of headaches, the regulation of stomach acid, and in cases of substantial water loss, for the replacement of important minerals.

It is not possible to carry out a direct titration of the calcium ions, because of the presence of paracetamol together with the calcium carbonate. For this reason the two substances must be separated by an analytical extraction with methanol. The determination of the paracetamol can be carried out after the extraction by comparison of the UV-spectrum with that of a similarly-handled standard solution, made by addition of paracetamol to a paracetamol-free mineral-solution.

The determination of calcium carbonate in the extraction residue can be carried out by addition of an EDTA-solution and titration using "Calcon" as indicator.

EDTA-solution

The EDTA-solution will be provided. The titer, t, for this standard solution can be determined using zinc sulfate heptahydrate as primary standard.

Apparatus

A spectrometer operating in the UV region, quartz cuvettes, a centrifuge, centrifuge tubes, 25 mL measuring cylinder, microspatula, 100 mL standard flasks, 1000 mL standard flasks, 10 mL graduated pipettes, 10 mL- and 20 mL-pipettes, 50 mL burette, 300 mL conical flasks (Erlenmeyer flasks), burette funnel, weighing glasses, drying oven, wash-bottles, beakers.

Procedure

Extraction

The contents of the sample flask provided should be placed as completely as possible in a centrifuge tube. The flask should be washed several times with very small quantities of methanol, and the methanol extracts put in the centrifuge tube.

In another centrifuge tube put ca. 350 mg calcium carbonate, ca. 150 mg potassium chloride, and 90 – 110 mg paracetamol weighed to within +/- 0.0005 g. (Reference tube). The solids in both centrifuge tubes should be as fully digested as possible with each 7 mL of methanol, the suspension centrifuged at ca. 4000 rpm for 5 min. The clear supernatant liquid should be carefully removed into two separate 100 mL standard flasks. Take care when removing the supernatant, that no solid is transferred into the standard flasks. The procedure should be repeated four times. Be specially careful that no extract is lost during the first extraction.

The inorganic residue which remains in the tubes should be dried at a temperature not exceeding 45 ^{o}C. The samples should be dried, occasionally gently breaking up the solid, until the mixture is no longer pasty (about 30 minutes drying time).

Determination of paracetamol

Fill both of the flasks containing the methanol extracts to the mark, using deionised water. Pipette out two 10 mL portions of this solution, transfer them to two separate 1000 mL standard flasks, and fill to the mark using deionised water.

For each of the solutions (sample and reference) measure the extinction (absorptivity) in a 1 cm quartz cuvette. Use a double-beam photometer at 247 nm, with a sample of deionised water as blank (in the reference beam).

Using the two absorbance values, and the weight of paracetamol in the reference, calculate the mass of paracetamol in the sample.

Determination of calcium carbonate

Titrimetric determination of the EDTA-solution, c(EDTA) = 0.05 mol L^{-1}

Weigh between 220 and 260 mg of zinc sulfate heptahydrate ($ZnSO_4.7H_2O$) to an accuracy of +/- 0.0005 g into a 300 mL Erlenmeyer (conical) flask, and add 100 mL of deionised water. Add enough ammonium chloride to cover the tip of a spatula, followed by 1.0 mL of ammonia solution (w = 25%) measured using a graduated pipette. The solution should be clear. After the addition of a Riedel indicator-buffer tablet, titrate with the EDTA solution. The end-point is from red to green. Titrate slowly as the end-point is approached. The molar mass of zinc sulfate heptahydrate is 287.55 g mol^{-1}.

Titration of calcium ions

The dried residue remaining in the sample centrifuge tube and in the reference centrifuge tube, should be carefully dissolved by the dropwise addition hydrochloric acid (w = 10%). After the vigorous evolution of carbon dioxide has stopped, the remaining clear solution should be washed quantitatively into a 100 mL standard flask, and the flask made up to the mark.

Pipette 20 mL of this solution into a 300 mL Erlenmeyer flask, and dilute with ca. 100 mL of deionised water. Add 10 mL of a freshly-prepared solution of sodium hydroxide (w = 15%) and a small spatula of the ground Calcon-sodium chloride mixture (Calcon 1% by weight). Titrate this against EDTA solution (0.05 mol L^{-1}, just prepared) to the end-point when the pink colour changes to blue-violet. Titrate slowly as the end-point is approached. The blue-violet colour should last for at least 15 seconds.
The molar mass of calcium carbonate is 100.1 g mol^{-1}.

Results

Report all the measurements, data, calculations and any unexpected observations in the results sheet, together with your name.

Furthermore, provide the following values in the results sheet:

1. Mass of paracetamol determined (mg) in the sample.
2. Mass of calcium carbonate determined (mg) in the sample
3. Mass of calcium carbonate determined (mg) in the reference sample