

Chapter 2

Experimental

2.0 Experimental

2.1 Reagents and Instruments

Reagents:

The following chemicals and solvents are all obtained from commercial sources without any further purification in the titrimetric and spectrophotometric methods.

- Benzethonium chloride (BDH Chemicals)
- Sodium lauryl chloride (Fluka Chemika)
- Azure A (Fluka Chemika)
- Mercuric acetate (Merck)
- Potassium hydrogen phthalate
- Glacial acetic acid (J.T. Baker)
- Perchloric acid, 70% (Merck)
- Methyl yellow indicator (Riedel-de Haen)
- Bromophenol blue

Instrument:

The concentration of sodium lauryl sulfate in the selected detergents is determined by using the Shimadzu 1601 UV-visible spectrophotometer for the spectrophotometric analysis.

Apparatus:

- Measuring cylinders 100 ml, 50 ml
- Burette 100 ml
- One-mark volumetric flask 50 ml, 100 ml, 250 ml, 500 ml and 1 L
- One-mark pipettes 5 ml, 10 ml, 20 ml
- Conical flasks 250 ml
- Magnetic stirrer
- Hot plate with stirrer
- Pipette pump
- Beakers
- Droppers
- Filter paper

All the apparatus are washed thoroughly and dried in the oven before use. However, the measuring apparatus such as volumetric flask and pipettes should be soaked in the acid bath and should be dried in open air.

2.2 Preparation of 0.1 M Standard Acetous Perchloric Acid solution

Approximately 8.5 ml of 70-72% perchloric acid was added to 500 ml of glacial acetic acid in the 1 L volumetric flask. After that, 30 ml of anhydrous acetic anhydride was added to the mixture and diluted the mixture to 1 L with glacial acetic acid.

2.3 Standardisation of 0.1 Standard Acetous Perchloric Acid Solution

Potassium hydrogen phthalate was first dried in the oven at 105°C for one hour. 0.4500 g of potassium hydrogen phthalate was then weighed accurately and transferred to the 250 ml conical flask. 80 ml of glacial acetic acid and a few drops of bromophenol blue indicator were added to the conical flask. After the potassium hydrogen phthalate was dissolved in the mixture, it was titrated with acetous perchloric acid solution which was prepared previously until the colour become crystal clear at the end point.

Blank titration was performed using the same procedure but without the addition of the potassium hydrogen phthalate. This was corrected from the volume of titrant.

2.4 Preparation of 6% Mercuric Acetate Solution

6.0005 g of mercuric acetate crystals was weighed accurately and then dissolved in 100 ml of glacial acetic acid. In order to ensure that the mercuric acetate crystals were fully dissolved, the mixture was heated gently on a hot plate.

2.5 Preparation of Azure A Solution

Azure A solution which acts as an indicator was freshly prepared by dissolving 0.0402 g of the dye Azure A in distilled water and to it was added 10 ml of 0.1 M H_2SO_4 and made up to 100 ml using distilled water.

2.6 Preparation of Methyl Yellow Solution

0.0400 g of p-dimethylaminoazobenzene was weighed accurately and dissolved in 100 ml of MeOH. The methyl yellow solution was used as an indicator for the titrimetric method.

2.7 Determination of the Purity of Benzethonium Chloride

About 1 g of benzethonium chloride was weighed accurately in a 250 ml conical flask. (The exact amount of benzethonium chloride used can be obtained from Table 7). It was dissolved in 80 ml of glacial acetic acid. To it was added 10 ml of 6% mercuric acetate solution prepared previously. A few drops of bromophenol blue indicator was added and the mixture was titrated with 0.1 M acetous perchloric acid solution to yellow-crystal clear end point.

A blank determination was performed on 80 ml of glacial acetic acid and 10 ml of mercuric acetate solution. This was then use to correct the volume of titrant.

2.8 Preparation of 0.005 M Benzethonium Chloride Standard Solution

2.3449 g of dried benzethonium chloride was weighed accurately and diluted to 1 L using distilled water in a volumetric flask.

2.9 Preparation of 0.050 M Benzetbonium Chloride Standard Solution

2.3445 g of dried benzethonium chloride was weighed accurately and diluted to 100 ml using distilled water.

2.10 Determination of the Purity of Sodium Lauryl Sulfate

0.5002 g of sodium lauryl sulfate was weighed and it was transferred to a clean and dry 250 ml volumetric flask. The solid was dissolved in distilled water and diluted to the mark. Care should be taken during the process of dissolving the sodium lauryl sulfate in water as to avoid the formation of bubbles, which will cause error in the subsequent analysis. An aliquot of 25 ml of this solution was pipetted into a 250 ml conical flask. To it was added 50 ml CHCl_3 , 10 ml of 1 M sulfuric acid and 1 ml of methyl yellow indicator.

The mixture was titrated with 0.005 M of benzethonium chloride. During the titration, the mixture must be shaken vigorously until the end-point is reached in which the pink colour chloroform layer will change to orange. The titration was continued until the end point of yellow colour chloroform layer was obtained.

The titration was carried out for 2 more samples.

2.11 Preparation of 50 $\mu\text{g/ml}$ Standard Sodium Lauryl Sulfate (SLS) Solution

0.0501 g of pure SLS was transferred to 1 L volumetric flask and diluted to the mark with distilled water.

2.12 Preparation of a Series of Standard Sodium Lauryl Sulfate Solution for Spectrophotometric Analysis

A series of standard solution was prepared by pipetting 1, 3, 5, 8 and 10 ml of the standard SLS solution prepared previously into 6 separating funnels. To each of these funnels was added 25, 24, 22, 20, 17 and 15 ml of distilled water respectively, 10 ml of 0.1 M sulfuric acid and 1 ml of Azure A solution.

The mixture was extracted with two 20 ml portions of water-saturated chloroform. The chloroform layer was filtered through the filter paper into 50 ml volumetric flask and diluted to the mark with chloroform. The absorbance of these solutions was recorded at 637 nm against water-saturated chloroform as the blank.

A standard graph was prepared by plotting absorbance against concentration of SLS is shown in figure 11a and 11b in chapter 3.

2.13 Preparation of SLS Samples from the Commercial Detergent Products

About 10 g of the sample was weighed accurately into a 250 ml beaker (The exact amount of sample used in each determination can be obtained from Table 12, 13, 16 and 17). To it was added 20 ml of dilute HCl (2 M) and 50 ml of distilled water.

The mixture was heated until the oil liquefied and separated. The resulting

mixture was cooled until the oil solidified. The aqueous layer was decanted through fluted filter paper into a 250 ml volumetric flask. The filter paper was macerated thoroughly and the extraction process was repeated twice, decanted as before and washed with distilled water. The combined filtrate was cooled to room temperature and diluted to the mark with distilled water.

2.14 Spectrophotometric Analysis of the Samples

2 aliquots of 2 ml sample solution was pipetted into 2 separating funnels respectively. To each of the sample was added 23 ml of distilled water, 10 ml of 0.1 M H_2SO_4 and 1 ml of Azure A. One of them was added 1 ml of 0.05 M benzethonium chloride to serve as blank.

The mixture was extracted with two portion of 20 ml water-saturated chloroform. The chloroform layer was filtered through filter paper into 50 ml volumetric flask and diluted to volume. The absorbance of these sample and blank solutions were recorded at 637 nm using a UV-visible spectrophotometer. The blank absorbance was then subtracted from the sample absorbance. The concentration of the sample can be determined from the standard calibration graph, which was prepared earlier.