

## Determination of the Amount of Phosphorus in a Washing Detergent Component – Determination of Acid Capacity of Washing Alkali.

### Part 1: The determination of the amount of phosphorus in a washing detergent component

#### Principle

The orthophosphate ion reacts with ammonium molybdate and antimony ion under acidic conditions to form a complex. This complex is reduced with ascorbic acid to form a blue complex which absorbs light at 880 nm. The absorbance is proportional to the concentration of orthophosphate in the sample.

Polyphosphates and some organophosphorus compounds undergo hydrolysis upon treatment with sulfuric acid and are converted to molybdate-reactive orthophosphate. Many organophosphorus compounds can be converted to orthophosphate by oxidation with peroxide sulfate. The conversion of more stable phosphorus containing compounds to orthophosphate require more rigorous oxidation with nitric/sulfuric acid.

### Determination of Orthophosphate after dissolution with Peroxodisulfate

#### 1 Reagents

##### 1.1 Already prepared – HTL Dornbirn

A) Sulfuric acid solution,  $c(\text{H}_2\text{SO}_4) = 9 \text{ mol/L}$

B) Orthophosphate stock solution,  $\rho_P = 50 \text{ mg/L}$ .

##### C) Acid Molybdate Solution

Sulfuric acid solution ( $c(\text{H}_2\text{SO}_4) = 9 \text{ mol/L}$ )

Ammonium-Molybdate Hydrate

Potassium Antimony(III) Tartrate hydrate

D) Hydrochloric acid for glassware cleaning,  $c(\text{HCl}) = 2,5 \text{ mol/L}$ .

##### 1.2 to prepare by GPCh-competitor

E) Sulfuric acid solution,  $c(\text{H}_2\text{SO}_4) = 4,5 \text{ mol/L}$ .

Fill beaker with  $50 \text{ mL} \pm 5 \text{ mL}$  water. Add slowly at continuous stirring and cooling  $50 \text{ mL} \pm 5 \text{ mL}$  of sulfuric acid  $c(\text{H}_2\text{SO}_4) = 9 \text{ mol/L}$  (A). Mix well and let the solution cool down to room temperature.

**F) Sulfuric acid solution**,  $c(\text{H}_2\text{SO}_4) = 2 \text{ mol/L}$ .

Fill beaker with  $60 \text{ mL} \pm 3 \text{ mL}$  water. Add slowly at continuous stirring and cooling  $22 \text{ mL} \pm 1 \text{ mL}$  of sulfuric acid (A). Adjust the volume with water using  $100 \text{ mL}$  volumetric flask. Mix the solution thoroughly.

**G) Sodium Hydroxide**,  $c(\text{NaOH}) = 2 \text{ mol/L}$ .

Dissolve  $8 \text{ g} \pm 1 \text{ g}$  of sodium hydroxide pellets in water. Let the solution cool down. Adjust the volume with water to  $100 \text{ mL}$ .

**H) Ascorbic Acid Solution**,  $\rho = 100 \text{ g/L}$ .

Dissolve  $10 \text{ g} \pm 0,5 \text{ g}$  ascorbic acid ( $\text{C}_6\text{H}_8\text{O}_6$ ) in  $100 \text{ mL} \pm 5 \text{ mL}$  water.

**I) Orthophosphate Standard/Stock Solution**,  $\rho_P = 2 \text{ mg/L}$ .

Pipet  $20 \text{ mL} \pm 0,01 \text{ mL}$  orthophosphate stock solution ( $\rho_P = 50 \text{ mg/L}$ , (B)) to a  $500 \text{ mL}$  volumetric flask, adjust the volume with water, mix the solution thoroughly.

Remark:  $1 \text{ mL}$  standard solution contains  $2 \mu\text{g P}$ .

**J) Potassium Peroxodisulfate Solution**

Dissolve  $5 \text{ g} \pm 0,1 \text{ g}$  potassium peroxodisulfate ( $\text{K}_2\text{S}_2\text{O}_8$ ) in  $100 \text{ mL} \pm 5 \text{ mL}$  water.

## 2 Devices / Apparatus

### 2.1 Photometer

Absorbance is measured photometrically at  $880 \text{ nm}$ .

### 2.2 Glassware

All glassware used in the analysis must be free of phosphate. Wash the glassware with hydrochloric acid ( $c(\text{HCl}) = 2,5 \text{ mol/L}$ , D) at  $40 - 50 \text{ }^\circ\text{C}$  and rinse thoroughly with water.

## 3 Procedure: Determination of total phosphorus after dissolution with Peroxodisulfate

### 3.1 Sample preparation

**K) Preparation of sample solution 1**

Weight  $2,5 \text{ g}$  of the sample to be analyzed, transfer to  $250 \text{ mL}$  flask and dissolve in water at  $60 \text{ }^\circ\text{C}$ . Cool the solution to room temperature and bring the volume to  $250 \text{ mL}$ .

$1 \text{ mL}$  of obtained solution contains  $10 \text{ mg}$

**L) Preparation of sample solution 2**

Transfer  $20 \text{ mL}$  of detergent solution 1 (K) to a volumetric flask and add water to total  $1 \text{ L}$ .

## 3.2 Procedure

### 3.2.1 Analysis procedure

Transfer 10 mL of sample solution 2 (L) to 100 mL Erlenmeyer flask. Add 4 mL potassium peroxodisulphate (J) and bring to a simmer over medium heat for 30 min. Keep up the total volume in the range of 25 - 35 mL by adding water. Cool down the solution and adjust the pH with sodium hydroxide - solution (G) or sulfuric acid ( $c(\text{H}_2\text{SO}_4) = 2 \text{ mol/L}$ , F). pH values should be in the range 3 to 10. Transfer the solution into 50 mL volumetric flask. The total volume of the solution at this step must not exceed ~40 mL.

### 3.2.2 Blank value

Prepare a blank solution in exactly the same manner as reagent solution except use water instead of orthophosphate standard solution

### 3.2.3 Calibration

#### 3.2.3.1 Preparation of standard solutions

Prepare a series of standards solution by pipetting 1; 2; 4; 6; 8 and 10 mL of orthophosphate standard solution ( $\rho_P = 2 \text{ mg/L}$ , I) in 100 mL Erlenmeyer flask. Fill the volume of each flask approx. up to 40 mL. The concentration of the orthophosphate in the obtained solutions is in the range from 0,04 to 0,4 mg/L. Add 4 mL potassium peroxodisulfate (J) and bring to a simmer over medium heat for 30 min. Keep up the total volume in the range of 25 - 35 mL by adding water. Cool down the solution and adjust the pH with sodium hydroxide - solution (G) or sulfuric acid ( $c(\text{H}_2\text{SO}_4) = 2 \text{ mol/L}$ , F). pH values should be in the range 3 to 10. Transfer the solution into 50 mL volumetric flask. The total volume of the solution at this step must not exceed ~40 mL (see 3.2.1).

#### 3.2.3.2 Color development

Add 1 mL ascorbic acid solution (H) under continuous stirring to each 50 mL volumetric flask (3.2.3.1), then 2 mL acid molybdate solution (C). Fill up the volume to 50 mL. Mix well the solutions.

#### 3.2.3.3 Photometric measurement

Absorbance is measured photometrically at 880 nm against water as a reference within 10 - 30 min after the preparation.

#### 3.2.3.4 Calibration curve

Plot a graph of absorbance (y-axis) against concentration (x-axis) in mg/L of standard solutions of phosphorus. The relation between absorbance and concentration is linear. Calculate the slope of the straight line.

### 3.2.4 Analyte determination

#### 3.2.4.1 Color development

Prepare the partial sample according to 3.2.1 and proceed further according to 3.2.3.2.

#### 3.2.4.2 Photometric measurement (See 3.2.3.3)

### 3.3 Results evaluation

#### 3.3.1 Calculation

The total amount of phosphate,  $\rho_P$  in mg/L is calculated according to the formula:

$$\rho_P = n \cdot \frac{(A - A_0) \cdot V_{max}}{f \cdot V_S}$$

Where

- A Absorbance of the sample;  
A<sub>0</sub> Absorbance of the blank solution;  
f Slope of the curve (L/mg);  
V<sub>max</sub> Volume of the volumetric flask (50 mL);  
V<sub>S</sub> actual volume of the sample (mL).  
n 1

Express phosphorus concentration in the detergent component as: **mg P per g detergent component** with three significant digits.

#### 3.3.2 Analysis report

The report must include the following details:

- Details for sample identification
- Information about basic standard operation procedure
- Result of the amount of phosphate in mg P per g detergent component
- Discussion of calibration curve

#### Literature:

Photometrisches Verfahren mittels Ammoniummolybdat (ISO 6878:2004);  
Graffmann G., Fres. Z. Anal. Chem. 301, 364-372 (1980)

## Part 2

### Determination of acid capacity of alkaline washing liquors

#### 1. Principle

The buffering capacity is the molar quantity of hydrogen- or hydroxide-ions in relation to the water volume used, which is necessary to reach an appointed pH value (mmol/L).

#### 2. Procedure

The acid capacity of a solution of the washing alkali sample specified (10 g of sample / L) is determined by titration with hydrochloric acid solution  $c(\text{HCl}) = 0,1 \text{ mol/L}$ .

The hydrochloric acid  $c=0,1 \text{ mol/L}$  required must be produced by dilution of the given hydrochloric acid  $\tilde{c} = 0,5 \text{ mol/L}$ .

The titer of this hydrochloric acid ( $\tilde{c} = 0,1 \text{ mol/L}$ ) has to be determined with anhydrous soda ( $M(\text{Na}_2\text{CO}_3)=105,99 \text{ g/mol}$ ) and end point determination with pH-Indicator.

Sample solution (10 g/L):

Produce a solution basing on the sample material obtained.

By titration with hydrochloric acid  $\tilde{c} = 0,1 \text{ mol/L}$  and with the pH-indicators Methylorange and Phenolphthalein the buffer capacities  $K_{\text{S}8,2}$  and  $K_{\text{S}4,3}$  must be measured.

The titration must be performed manually without the help of a magnetic stirrer!

#### 3. Calculation:

$$K_{\text{S}8,2} = \frac{c(\text{HCl}) \cdot V_1 \cdot 1000}{V_3}$$

$c(\text{HCl})$  molar concentration of hydrochloric acid used [mol/L].  
 $V_1$  required volume of HCl in mL, to reach pH-value of 8,2  
 $V_3$  volume of sample in mL

$$K_{\text{S}4,3} = \frac{c(\text{HCl}) \cdot V_2 \cdot 1000}{V_3}$$

$c(\text{HCl})$  molar concentration of hydrochloric acid used [mol/L].  
 $V_2$  volume of HCl in mL, to reach pH-value of 8,2  
 $V_3$  volume of sample in mL

#### 4. Analysis report

The report must include the following details:

- a) Details for sample identification
- b) Results in mmol/L