

# Application Bulletin

Of interest to: Water analysis; Food; Fertilizers  
Detergents; Mineral resources

A 1, 2, 7, 8, 11, 12, 15

## Potentiometric determination of orthophosphates, metaphosphates and polyphosphates

### Summary

After acid digestion, the sample solution is neutralized with sodium hydroxide to form sodium dihydrogen phosphate. An excess of lanthanum nitrate is added, then the released nitric acid is titrated with sodium hydroxide solution.



This determination method is suitable for higher phosphate concentrations.

### Instruments and accessories

- 702 SET/MET Titrino, 716 DMS Titrino, 719 SET Titrino, 736 GP Titrino, 751 GPD Titrino or 785 DMP Titrino or 726 Titroprocessor with 700 Dosino or 685 Dosimat
- 2.728.0040 Magnetic Stirrer
- 6.3014.223 Exchange Unit 20 mL (with flat PCTFE/PTFE stopcock)
- 6.0239.100 combined pH glass electrode with 6.2104.020 electrode cable

### Reagents

- Titrant  $c(\text{NaOH}) = 0.1 \text{ mol/L}$  or  $0.01 \text{ mol/L}$
- Lanthanum nitrate solution,  $c[\text{La}(\text{NO}_3)_3] = 0.1 \text{ mol/L}$ , pH value adjusted to 4.2
- Sulfuric acid  $c(\text{H}_2\text{SO}_4) = 1 \text{ mol/L}$
- Sodium hydroxide  $c(\text{NaOH}) = 2 \text{ mol/L}$
- Concentrated acids for sample digestion:  
 $w(\text{H}_2\text{SO}_4) = 96\%$  and  $w(\text{HClO}_4) = 60\%$

### Sample preparation

#### Inorganic salts

Dissolve the sample in dist. water. Orthophosphates can be titrated straightaway.

If, however, the sample contains metaphosphates and/or polyphosphates, 1 mL conc.  $\text{HClO}_4$  and 1 mL conc.  $\text{H}_2\text{SO}_4$  are added. The sample solution is then heated and boiled down until acid steam evaporates. After cooling down, add 10 mL dist. water and boil again until acid steam evaporates. Allow the solution to cool down, then rinse it with dist. water into a 100 mL volumetric flask, fill to the mark and mix.

***Samples containing organic material: food, animal feed, sewage sludge, etc.***

The sample is dried, then reduced to ashes at 800 °C in a muffle furnace. The cooled residue is rinsed with dist. water into a Kjeldahl flask and 1 mL conc. HClO<sub>4</sub> as well as 1 mL conc. H<sub>2</sub>SO<sub>4</sub> are added. Afterwards proceed as described above under metaphosphates and polyphosphates.

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***Analysis***

Pipet an aliquot of the prepared sample solution that contains no more than 30 mg P into a glass beaker and adjust the pH value to 4.2 with c(NaOH) = 2 mol/L or c(H<sub>2</sub>SO<sub>4</sub>) = 1 mol/L. Add 10 mL La(NO<sub>3</sub>)<sub>3</sub> solution, then titrate back to pH = 4.2 with sodium hydroxide solution using the SET mode of the titrator.

***Calculation***

1 mL c(NaOH) = 0.1 mol/L corresponds to 1.5489 mg P or 3.5486 mg P<sub>2</sub>O<sub>5</sub> or 4.7486 mg PO<sub>4</sub>

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***Remarks***

- Instead of La(NO<sub>3</sub>)<sub>3</sub>, AgNO<sub>3</sub> could also be used. This is, however, more expensive and in addition it can cause problems with the combined pH glass electrode (precipitation of AgCl).
- The limit of quantitation is 1 mg/L P [sample volume used: 100 mL, titrant: c(NaOH) = 0.01 mol/L].
- The titration endpoint at pH = 4.2 must be observed as precisely as possible. If the endpoint lies outside the range pH = 4.0 ... 5.0 erroneous results will be obtained.