

Application Bulletin

Of interest to: Mineral resources, Fertilisers, Food,
Washing agents, Water

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

Potentiometric determination of ortho-, meta- and polyphosphates

Summary

After acid digestion, the sample is neutralised with sodium hydroxide to form sodium dihydrogen phosphate. A lanthanum nitrate excess is added, then the released nitric acid is titrated:



The method is suitable for high phosphate concentrations.

Apparatus and accessories

- Titrimo or Titrimo with Dosino or Dosimate
- Magnetic Swing-out Stirrer
- Exchange Unit
- 6.0259.100 comb. pH glass electrode with 6.2104.020 electrode cable

Reagents

- Titrant: $c(\text{NaOH}) = 0.1 \text{ mol/L}$ or 0.01 mol/L
- Lanthanum nitrate: $\text{La}(\text{NO}_3)_3$ $c = 0.1 \text{ mol/L}$. pH-value set to 4.2
- Sulphuric acid: $c(\text{H}_2\text{SO}_4) = 1 \text{ mol/L}$
- Sodium hydroxide: $c(\text{NaOH}) = 2 \text{ mol/L}$
- Acids for digestion: $w(\text{H}_2\text{SO}_4) = 96 \%$ and $w(\text{HClO}_4) = 60 \%$

Sample preparation

Inorganic salts

Dissolve the sample in distilled water. Orthophosphates can be used straightaway. If the samples contain meta-and/or polyphosphates, add 1 mL each conc. HClO_4 and conc. H_2SO_4 . Afterwards, heat up the sample and boil until acid steam evaporates. Let cool, then add 10 mL dist. H_2O and bring to a boil again until acid steam appears. Allow the sample to cool, then rinse the sample with dist. H_2O in a 100 mL measuring flask, fill up to the mark and mix.

Samples containing organic material

(Food, animal feed, sewage sludge etc.)

The samples are dried, then reduced to ashes at 800 °C in a muffle furnace. The cooled residue is rinsed with dist. H₂O into a Kjeldahl flask, whereupon 1 mL each conc. HClO₄ and con. H₂SO₄ is added. Afterwards proceed as described under meta- and polyphosphates.

Analysis

Pipette an aliquot of the prepared sample solution (may not contain more than 30 mg P) into a glass beaker and adjust the pH value to 4.2 with $c(\text{NaOH}) = 2 \text{ mol/L}$ or $c(\text{H}_2\text{SO}_4) = 1 \text{ mol/L}$. Add 10 mL La(NO₃)₃-solution and in SET mode of the titrator, titrate back to pH = 4.2.

Calculation

1 mL $c(\text{NaOH}) = 0.1 \text{ mol/L} = 1.5489 \text{ mg P}$ or $3.5486 \text{ mg P}_2\text{O}_5$
or 4.7486 mg PO_4

Remarks

- In place of La(NO₃)₃, AgNO₃ can be used. This is, however, more expensive and can cause problems with the combined glass electrode (AgCl precipitation).
- The determination limit lies at 1 mg/L P. (100 mL sample size, 0.01 mol/L titrant).
- The titration end-point of pH = 4.2 must be held as precisely as possible. You will receive erroneous results if the end-point is not within the limits of pH = 4.0 ... 5.0.