Application Bulletin

Metrohm

Of interest for:
Cement-work laboratories
General analytical laboratories

No. 100/1 e

Biamperometric determination of potassium and/or ammonium

Summary

The potassium (or ammonium) ion is precipitated with sodium tetraphenyl borate, and the excess of this reagent back-titrated against the thallous ion, using biamperometric endpoint detection. Ammonium can either be titrated together in an acid solution, or driven off by previous boiling in an alkaline solution. Methods are given for determining potassium in the presence of large excesses of sodium, ammonium, calcium and magnesium.

Geräte

- 2.536.0210 Potentiograph with 2.665.0030 Titrating Stand
- 2.585.0010 Polarizer
- 6.0308.100 double platinum wire electrode

Reagents

- **Standard potassium solution**
  Dissolve 1.9068 g KCl in distilled water and make up to 1 litre (1 mL ≙ 1 mg K^+).
- **0.02 mol/L sodium tetraphenyl borate**
  Dissolve 6.8446 g sodium tetraphenyl borate in distilled water and make up to 1 litre.
- **0.02 mol/L thallium nitrate**
  Dissolve 5.3274 g $TlNO_3$ in distilled water and make up to 1 litre.
- **app. 2 mol/L sodium hydroxide**
  Dissolve 80 g NaOH in 1 litre of distilled water.
- **app. 2 mol/L hydrochloric acid**
  Dilute 200 mL conc. HCl with 800 mL distilled water.

Determinations

- **Potassium alone or in the presence of sodium and magnesium**
  Take a sample containing app. 1 to 5 mg potassium and make it up to 20 mL with distilled water. Precipitate out the potassium with 10 mL sodium tetraphenyl borate solution, add 2 mL of 2 mol/L NaOH and back-titrate the excess tetraphenyl borate with 0.02 mol/L $TlNO_3$. Use a polarisation voltage of 300 mV and the 250 mV range of the Potentiograph. There will be a sharp kink in the curve at titration endpoint. At the same time, carry out a blank control titration on a sample made up of 20 mL distilled water, 10 mL tetraphenyl borate and 2 mL 2 mol/L NaOH. The result is obtained by subtracting the reagent consumption of the main titration (B) from that of the blank control titration (A).

  Calculation: $(A - B) \cdot 0.782$ = mg potassium in original sample

  A = mL $TlNO_3$ 0.02 mol/L for blank control
  B = mL $TlNO_3$ 0.02 mol/L for actual sample

Biamperometric determination of potassium and/or ammonium

Determination *(continued)*

- **Potassium in the presence of ammonium**

  Before precipitation, add 1 mL HCl to the sample. After precipitation, add 3 mL 2 mol/L NaOH and proceed further as described in "potassium alone ...". This method gives the sum $K^+ + NH_4^+$.

  Make a second sample alkaline by adding 3 to 5 mL of 2 mol/L NaOH and boil for 5 min, making up the water lost by evaporation during boiling. Allow to cool, precipitate with sodium tetraphenyl borate, and titrate as described in "potassium alone ...". The ammonium content is found from the difference between the two titration results.

  1 mL 0.02 mol/L sodium tetraphenyl borate ≙ 0.361 mg ammonium

- **Potassium in the presence of calcium**

  The important thing here is to use a sufficiently large excess of NaOH so as to ensure that $Ca(OH)_2$ is precipitated quantitatively. In most cases the addition of 5 mL 2 mol/L NaOH before titration will suffice.

Remarks

Powerful oxidising agents, iodide, sulphide, and slightly acid solutions containing Ag^+, Hg^{2+}, Au^{3+} and Cu^{2+} will interfere with the determination. On the other hand, it is unaffected by anions such as Cl^-, NO_3^-, CH_3COO^-, SO_4^{2-} and PO_4^{3-}.

Literature

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