
Application Bulletin

Of interest to: General analytical laboratories; Pharmaceutical industry;
Metals, electroplating

A 1, 4, 10

Potentiometric titration of aluminum and magnesium in the same solution

Summary

Mixtures of aluminum and magnesium ions (digestion solutions of alloys, antacids, etc.) can be analyzed automatically by potentiometric titration. After addition of 1,2-diaminocyclohexanetetraacetic acid (DCTA) and complex formation the DCTA excess is titrated back with copper(II) sulfate solution. The ion-selective copper electrode is used as indicator electrode. First the aluminum is determined in acidic solution, then the magnesium in alkaline solution.

Instruments and accessories

- 702 SET/MET Titrino, 716 DMS Titrino, 736 GP Titrino, 751 GPD Titrino or 785 DMP Titrino or 796 Titroprocessor with 700 Dosino or 685 Dosimat
 - Possibly two additional 776 auxiliary Dosimats or 700 Dosinos
 - 2.728.0040 Magnetic Stirrer
 - 6.3014.223 Exchange Unit(s)
 - 6.0502.140 ion-selective copper electrode (Cu ISE) with 6.2104.020 electrode cable
 - 6.0726.107 double-junction Ag/AgCl reference electrode [filled with $c(\text{KCl}) = 3 \text{ mol/L}$] with 6.2106.020 electrode cable
-

Reagents

- DCTA solution, $c(\text{DCTA}) = 0.1 \text{ mol/L}$:
Dissolve 36.463 g 1,2-diaminocyclohexanetetraacetic acid monohydrate in 400 mL $c(\text{NaOH}) = 0.5 \text{ mol/L}$ and make up to 1 L with dist. water.
- Titrant: copper(II) sulfate solution, $c(\text{CuSO}_4) = 0.1 \text{ mol/L}$:
Dissolve 24.968 g $\text{CuSO}_4 \cdot 5 \text{ H}_2\text{O}$ in approx. 500 mL dist. water, add 0.5 mL conc. H_2SO_4 and make up to 1 L with dist. water.
- Buffer solution pH = 4.7:
Dissolve 123 g sodium acetate and 86 mL conc. acetic acid in dist. water and make up to 1 L.
- Ammonia solution, $w(\text{NH}_3) = 10\%$

Analysis

Strongly acidic sample solutions (e.g. from acid digestions) are pre-neutralized to pH = 2 ... 3 with NaOH.

Pipet a sample volume containing no more than 12 mg Al³⁺ or 20 mg Mg²⁺ into a glass beaker and make up to approx. 50 mL with dist. water. Add 6.00 mL c(DCTA) = 0.1 mol/L and 5 mL buffer solution pH = 4.7 and allow to react for 1 min under stirring. Afterwards titrate back the DCTA excess with c(CuSO₄) = 0.1 mol/L (**de-termination of aluminum**). The end volume of titrant is automatically stored as constant C41 in the titrator.

Now the **magnesium** can be determined. Add anew 6.00 mL c(DCTA) = 0.1 mol/L and 20 mL w(NH₃) = 10% to the titrated sample solution and again, titrate back the DCTA excess with c(CuSO₄) = 0.1 mol/L.

Calculation

1 mL c(DCTA) = 0.1 mol/L corresponds to 2.6982 mg Al³⁺ or 2.4305 mg Mg²⁺

Determination of aluminum:

RS1 = C41 – EP1_a; mL

RS2 = g/L Al³⁺ = (C30 – EP1_a) * C01 / C00

Determination of magnesium:

RS3 = g/L Mg²⁺ = (C30 – EP1_b – C31) * C02 / C00

EP1_a = titrant consumption for the Al³⁺ titration in mL

EP1_b = titrant consumption for the Mg²⁺ titration in mL

C00 = sample volume in mL

C01 = 2.6982

C02 = 2.4305

C30 = 6.00 [added volume of c(DCTA) = 0.1 mol/L in mL]

C31 = RS1

C41 = end volume of titrant for the Al³⁺ titration in mL

Remark

The surface of the Cu ISE has to be polished from time to time with aluminum oxide powder (6.2802.000 polishing set).

```

'pa
736 GP Titrino          04268  736.0011
date 99-12-14          time 10:01  5
TIP                    AB 181
parameters
>sequence
  1.method:            DCTA
  2.method:            Al
  3.method:            DCTA
  4.method:            NH3
  5.method:            Mg
>statistics
  status:              OFF
>preselections
  req.ident:           OFF
  req.smpl size:       OFF
  meas.mode:           OFF
  temperature          25.0 °C
-----
'pa
736 GP Titrino          04268  736.0011
date 99-12-14          time 10:00  5
DOS                    DCTA
parameters
>dosing parameters
  dispensing type:     volume
  volume              6.000 ml
  disp.crit:          rate
  rate                max. ml/min
  pause               0 s
  time interval       10 s
  dos.element:        external D1
  temperature         25.0 °C
>stop conditions
  stop V:              OFF
  filling rate        max. ml/min
>statistics
  status:              OFF
>monitoring
  meas.mode:           OFF
  temperature:         OFF
  assign output:       none
>preselections
  req.ident:           OFF
  req.smpl size:       OFF
  activate pulse:     OFF
-----
'pa
736 GP Titrino          04268  736.0011
date 99-12-14          time 10:28  7
MET U                  Al
parameters
>titration parameters
  V step              0.05 ml
  titr.rate           max. ml/min
  signal drift        25 mV/min
  equilibr.time       34 s
  start V:            OFF
  pause               60 s
  dos.element:        internal D0
  meas.input:         1
  temperature         25.0 °C
>stop conditions
  stop V:              abs.
  stop V              3 ml
  stop U              OFF mV
  stop EP              1
  filling rate        max. ml/min
>statistics
  status:              OFF
>evaluation
  EPC                 30 mV
  EP recognition:     greatest
  fix EP1 at U       OFF mV
  pK/HNP:             OFF
>preselections
  req.ident:           OFF
  req.smpl size:       OFF
  activate pulse:     OFF
-----
>evaluation
  EPC                 30 mV
  EP recognition:     greatest
  fix EP1 at U       OFF mV
  pK/HNP:             OFF
>preselections
  req.ident:           OFF
  req.smpl size:       OFF
  activate pulse:     OFF
-----
'pa
736 GP Titrino          04268  736.0011
date 99-12-14          time 10:01  5
DOS                    NH3
parameters
>dosing parameters
  dispensing type:     volume
  volume              20 ml
  disp.crit:          rate
  rate                max. ml/min
  pause               0 s
  time interval       10 s
  dos.element:        external D2
  temperature         25.0 °C
>stop conditions
  stop V:              OFF
  filling rate        max. ml/min
>statistics
  status:              OFF
>monitoring
  meas.mode:           OFF
  temperature:         OFF
  assign output:       none
>preselections
  req.ident:           OFF
  req.smpl size:       OFF
  activate pulse:     OFF
-----
'pa
736 GP Titrino          04268  736.0011
date 99-12-14          time 10:35  7
MET U                  Mg
parameters
>titration parameters
  V step              0.05 ml
  titr.rate           max. ml/min
  signal drift        25 mV/min
  equilibr.time       34 s
  start V:            OFF
  pause               60 s
  dos.element:        internal D0
  meas.input:         1
  temperature         25.0 °C
>stop conditions
  stop V:              abs.
  stop V              3 ml
  stop U              OFF mV
  stop EP              9
  filling rate        max. ml/min
>statistics
  status:              OFF
>evaluation
  EPC                 30 mV
  EP recognition:     greatest
  fix EP1 at U       OFF mV
  pK/HNP:             OFF
>preselections
  req.ident:           OFF
  req.smpl size:       OFF
  activate pulse:     OFF
-----

```

Fig. 1: Parameter settings on the 736 GP Titrino for the automatic determination of aluminum and magnesium. The TIP method used comprises three dosing steps and two titrations.

```
'fr
736 GP Titrino          04268  736.0011
date 99-12-14         time 10:28    7
U(init)              92 mV MET U      Al
spl size             2 ml
EP1                  2.294 ml          154 mV
RS1                   0.16 ml
Al                    5.00 g/l
stop #EP reached
=====
```

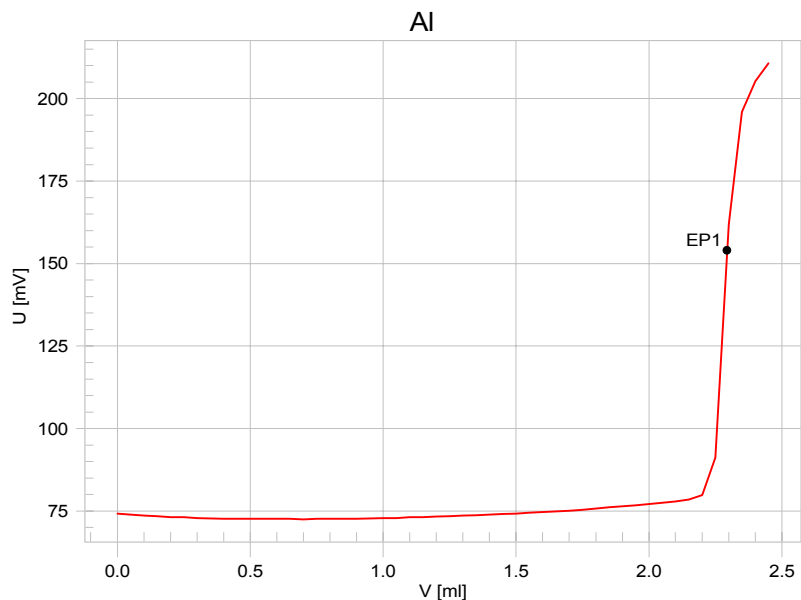


Fig. 2: Result block and titration curve for the Al^{3+} determination.

```
'fr
736 GP Titrino          04268  736.0011
date 99-12-14         time 10:36    7
U(init)             -164 mV MET U      Mg
spl size             2 ml
EP1                  1.703 ml          -112 mV
Mg                    5.03 g/l
stop V reached
=====
```

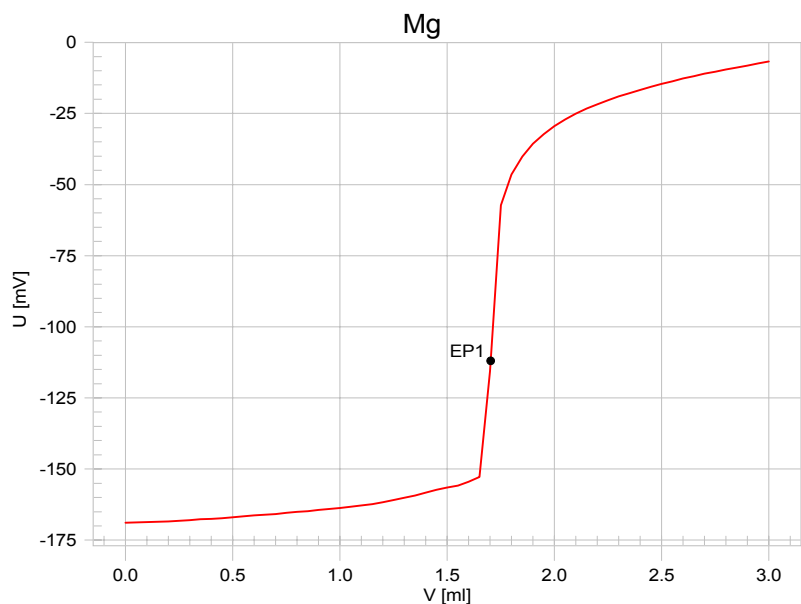


Fig. 3: Result block and titration curve for the Mg^{2+} determination.