Application Bulletin

Of interest to: General analytical laboratories, Metallurgical laboratories A 1, 10

Determination of zinc by potentiometric titration with potassium hexacyanoferrate(II)

Summary

Zinc, such as that occurring as a constituent of light alloys, can be determined by precipitation titration with potentiometric end-point indication. The determination of zinc next to cadmium is also possible.

$$2\ K_4[Fe(CN)_6]\ +\ 3\ ZnCl_2\ \rightarrow\ K_2Zn_3[Fe(CN)_6]_2\ +\ 6\ KCl$$

Apparatus and accessories

- Titrino or Titrando with Dosino or Dosimat
- Magnetic Swing-out Stirrer
- Exchange unit
- Pt-Titrode 6.0431.100 with electrode cable 6.2104.020
- Thermostated titration vessel, including titration vessel lid 6.1414.010 and titration vessel with thermostatic jacket 6.1418.250

Reagents

- Titrant, $K_4[Fe(CN)_6]$ c =0.05 mol/L:
  Dissolve 21.12 g $K_4[Fe(CN)_6] * 3\ H_2O$ in dist. H_2O and fill up to 1 L.
- Sulfuric acid: H_2SO_4 conc. p.A.
- Zn standard:
  Dissolve 22.0 g $ZnSO_4 * 7\ H_2O$ in dist. H_2O, add 1 mL conc. H_2SO_4 and fill up to 1 L with dist. H_2O.
  1 mL = 5.0 mg Zn

Analysis

Pipet 5 mL sample, resp. standard solution, and approx. 50 mL dist. H_2O into the titration vessel. Add carefully 5 mL H_2SO_4 and heat the mixture up to 65 ... 70 °C, then titrate with $K_4[Fe(CN)_6]$ in the MET mode of the titrator (parameters, see under appendix).

Calculation

1 mL $K_4[Fe(CN)_6]$ *c* = 0.05 mol/L = 4.904 mg Zn

g/L Zn = EP1 * C01 / C00

C00 = Sample weight in mL
C01 = 4.904

Remarks

- Do not allow temperature to fall below 60 °C during titration.
- The titrant must be standardized with Zn-standard because the stoichiometric factor does not quite agree.
- Cadmium also reacts with $K_4[Fe(CN)_6]$. Precipitation, however, is slow and takes place after that of Zn. It must be indicated potentiometrically. Thus, it is possible to determine Zn next to Cd.

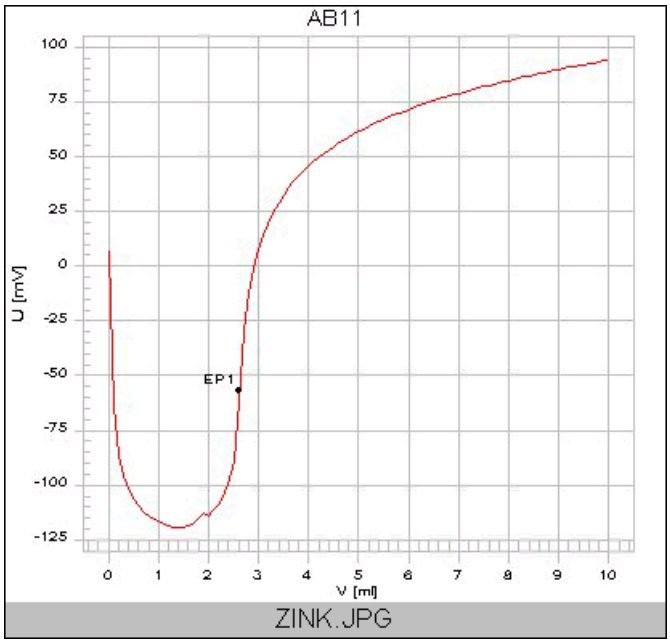
Literature

- Vojlosnikova,M.T. / Kozlovkij,M.T. / Songina,A.O.
  Eine amperometrische Methode zur Bestimmung sehr kleiner Zinkmengen
  Zavodsk. Laborat. 23, (1957) 273-276 (Russ)
  Ref:Fresenius,Z.Anal.Chem. 159, (1957) 59
- Agasjan,P.K.
  Zur Bestimmung von Zink und Cadmium nebeneinander
  Zavodsk. Laborat. 24, (1958) 532-534 (Russ)
  Ref:Fresenius,Z.Anal.Chem. 167, (1959) 281
- Kao,S.S. / Chuang,W.T.
  Simultantitration von Zink und Cadmium mit Hexacyanoferrat(II) - Lösung nach der Dead-Stop-Methode
  Acta Chim. Sinica 24, (1958) 25-29 (Chin)
  Ref:Fresenius,Z.Anal.Chem. 167, (1959) 282
- Malur,J. / Treptow,H.
  Untersuchung über die Grenzen der potentiometrischen Simultanbestimmung von Zink und Cadmium
  Fresenius,Z.Anal.Chem. 183, (1961) 426 - 432

Figures

```
'pa
736 GP Titrino          04268   736.0011
date  99-09-06      time  10:00        2
MET  U             AB11CdZn
parameters
>titration parameters
  V step               0.05 ml
  titr.rate           max. ml/min
  signal drift          50 mV/min
  equilibr.time          26 s
  start V:             OFF
  pause                 10 s
  dos.element:     internal D0
  meas.input:             1
  temperature           63.1 °C
>stop conditions
  stop V:              abs.
  stop V                20 ml
  stop U               OFF mV
  stop EP                9
  filling rate        max. ml/min
>statistics
  status:              OFF
>evaluation
  EPC                   50 mV
  EP recognition:        all
  fix EP1 at U          OFF mV
  pK/HNP:               OFF
>preselections
  req.ident:           OFF
  req.smpl size:         all
  activate pulse:        OFF
         ------------
```

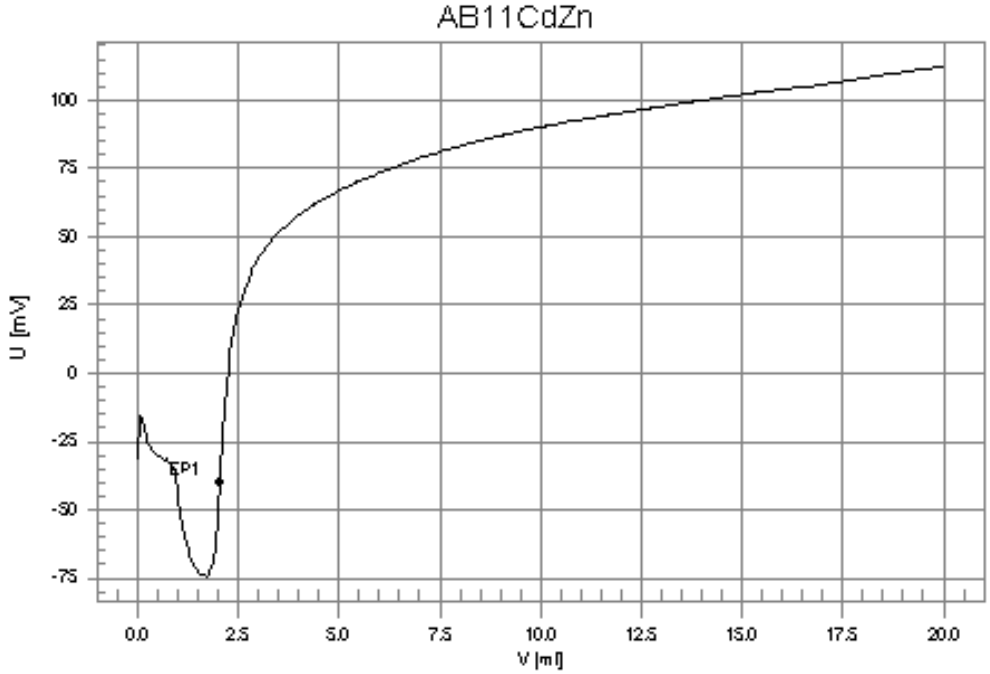
Fig. 1 Parameter report Titrino




```
'fm
736 GP Titrino          04268   736.0011
date  99-09-03      time  14:09        6
MET  U                 AB11
>calculations
Zn=EP1*C01/C00;2;g/l
C00=                    2.5
C01=                  4.904
         ============
```

```
 'fr
736 GP Titrino          04268   736.0011
date  99-09-03      time  14:09        6
U(init)           8 mV  MET U        AB11
smpl size        2.5 ml
EP1            2.614 ml            -57 mV
Zn             5.13 g/l
stop V reached
         ============
```

Fig. 2 Titration curve and results, Zn determination



'fm
736 GP Titrino 04268 736.0011
date 99-09-06 time 10:01 2
MET U AB11CdZn
>calculations
Zn=EP1*C01/C00;2;g/l
Cd=(EP2-EP1)*C02/C00;2;g/l
C00= 2
C01= 5.62
C02= 4.904

'fr
736 GP Titrino 04268 736.0011
date 99-09-06 time 10:00 2
U(init) -29 mV MET U AB11CdZn
smpl size 2 ml
EP1 2.041 ml -40 mV
Zn 5.74 g/l
Cd missing EP

Fig. 3 Titration curve and results, zinc next to cadmium