Application Bulletin

Of interest to: Metal industry A 10

Routine determination of copper in brass, bronze, German silver and electroplating baths

Summary

A routine method for the determination of copper is described. After dissolving the sample and adding a KI/KCNS solution, the freed iodine is back-titrated with thiosulfate. The endpoint indication is potentiometric.

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

Reagents

- Titrant $c(Na_2S_2O_3)$ = 0.2 mol/L:
  Dissolve 49.64 g $Na_2S_2O_3$ * 5 H_2O in CO_2 - free dist. H_2O and fill up to 1 L.
- Copper standard $c(Cu^{+2})$ = 0.1 mol/L:
  Dissolve 24.968 g $CuSO_4$ * 5 H_2O in dist. H_2O, add 10 mL conc. H_2SO_4 and fill up to 1 L with dist. H_2O.
- Reacting solution:
  Dissolve 7 g KI and 53 g KCNS in dist. H_2O and fill up to 1 L.
- Urea solution: w(urea) = 10% in dist. H_2O
- Sulfuric acid: $w(H_2SO_4)$ = 96%
- Nitric acid: $w(HNO_3)$ = 35%

Sample preparation

Acidic copper baths only containing Cu(II) can be used directly. In all other cases, place an electroplating bath sample or alloy containing approx. 200 mg Cu in a glass beaker and in a fume cupboard carefully add 10 mL HNO_3 . When the greatest reaction is over, add 2 mL H_2SO_4, and heat until the white sulfuric acid fumes are given off. Allow to cool, then mix with 1 mL urea solution and 25 mL distilled H_2O.

Titer determination of the thiosulfate solution

Pipet 20.0 mL Cu standard into a glass beaker, mix with 1 mL H_2SO_4 and 25 mL reacting solution and immediately titrate the freed iodine with thiosulfate.

$$2\ Cu^{2+} + 2\ KI \rightarrow I_2 + 2\ Cu^{+}$$

Titer: C01 / EP1 = C31

EP1 = mL thiosulfate up to the 1. EP

C01 = 10 (20 mL 0.1 mol/L corresponds to 10 mL 0.2 mol/L Cu^{2+})

Analysis

Add 25 mL reacting solution to the digestion solution and immediately titrate the freed iodine with thiosulfate.

Calculation

1 mL $c(Na_2S_2O_3)$ = 0.1 mol/L = 12.709 mg Cu

% Cu = EP1 * C01 * C02 * C31 / C00

C00 = Sample size in g
C01 = 12.709
C02 = 0.1 (for %)
C31 = Titer of the thiosulfate solution

Remarks

- After digestion, no excess HNO_3 should be present, otherwise this would also react with KI, falsifying the result.
- The more economical KI/KCNS solution can be used instead of the expensive KI.

Literature

- Agterdenbos,J. / Elberse,P.A.
  Rapid iodometric determination of copper in some copper-base alloys
  Talanta 13, (1966) 523-524
- Bastius,H.
  Zur jodometrischen Bestimmung des Kupfers
  Fresenius,J.Anal.Chem. 250, (1970) 169-172

Figures

```
'pa
785 DMP Titrino          02287   785.0010
user                  MEIER
date  1999-06-18     time  07:10         0
DET  U               ********
parameters
>titration parameters
  meas.pt.density            4
  min.incr.               10.0 µl
  dos.rate                max. ml/min
  signal drift              50 mV/min
  equilibr.time             26 s
  start V:                 OFF
  pause                      0 s
  meas.input:                1
  temperature             25.0 °C
>stop conditions
  stop V:                 abs.
  stop V                    30 ml
  stop U                   OFF mV
  stop EP                    9
  filling rate            max. ml/min
>statistics
  status:                  OFF
>evaluation
  EPC                        5
  EP recognition:    greatest
  fix EP1 at U             OFF mV
  pK/HNP:                  OFF
>preselections
  req.ident:               OFF
  req.smpl size:           OFF
  limit smpl size:         OFF
  activate pulse:           ON
          ------------
```

Fig. 1 Parameter report Titrino

```
 'fr
785 DMP Titrino          02287   785.0010
user                  MEIER
date  1999-06-17     time  17:04         6
U(init)          -58 mV DET U     ********
smpl size    0.36476 g
EP1           16.854 ml             113 mV
Kupfer       54.9528 %
stop V reached
          ============
```

```
 'cu
785 DMP Titrino          02287   785.0010
user                  MEIER
date  1999-06-17     time  17:04         6
start V       0.000 ml  DET U     ********
2.0 ml/div       dU=100.0 mV/div
```

```
 'BMP-File: C:\DATASCAN\ARQUYX.bmp
          ============
```

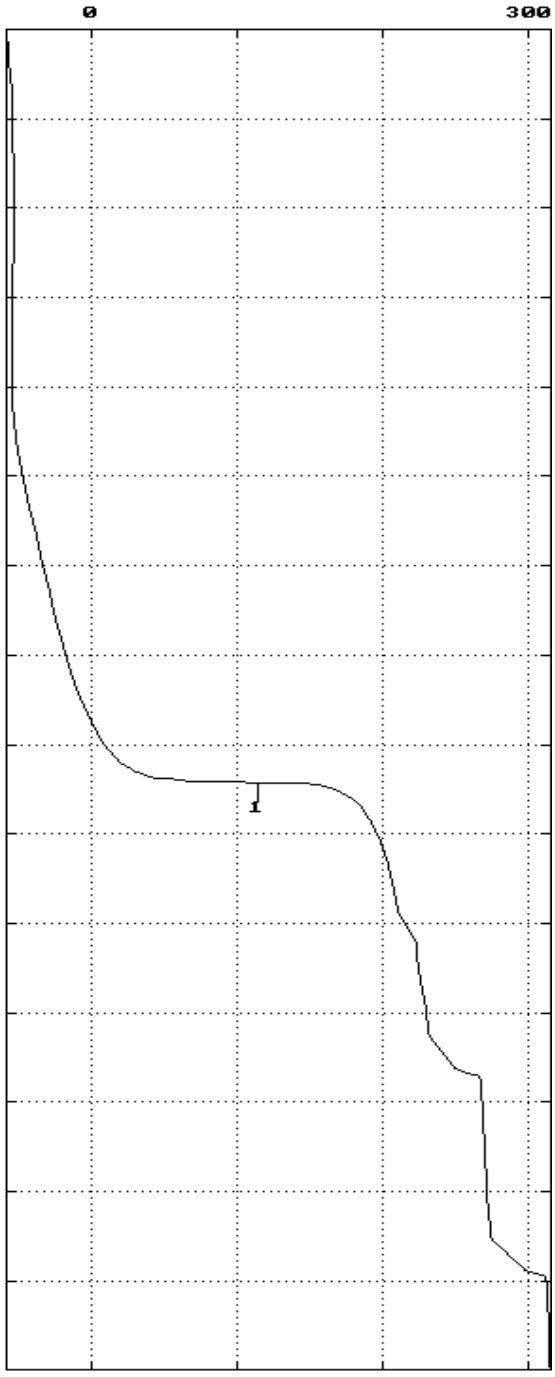

Fig. 2 Results report and titration curve: Cu in brass