Application Bulletin

Of interest to: Metal industry A 10

Determination of nickel in gold and silver plating baths by potentiometric titration

Summary

A potentiometric method for the determination of nickel in gold and silver electroplating baths is described. The titration is done with KCN. Gold and silver are removed before titration by a reduction process. It is also possible to determine nickel in alloys, etc. (see the literature reference).

$$Ni(II) + 4\,KCN + NH_3 + H_2O \longrightarrow (NH_4)_2[Ni(CN)_4]$$

Apparatus and accessories

- Titrino or Titrando with Dosino or Dosimat
- Magnetic Swing-out Stirrer
- Exchange unit
- Ag Titrode with Ag_2S coating 6.0430.100 with electrode cable 6.2104.020

Reagents

- Titrant, *c*(KCN) = 0.2 mol/L:
  Dissolve 4 g KOH and 13.03 KCN in dist. H_2O and fill up to 1 L. Standardize by titrating against Ni standard.
- Ni standard, *c*(Ni) = 0.01 mol/L:
  Dissolve 2.809 g $NiSO_4$ * 7 H_2O in dist. H_2O and fill up to 1 L.
  1 mL = 0.5869 mg Ni
- Hydrazine solution: *w*(hydrazine sulfate) = 2% in dist. H_2O
- Ammonia: *w*(NH_3) = 25%
- Digestion acids: conc. HCl and conc.HNO_3.

Sample preparation

Heat 10.0 mL of the bath sample together with 10 mL HCl. Evaporate down to ¼ of the original volume. Work in a fume cupboard, since cyanide is given off!!! Add conc. HNO_3 drop by drop until the precipitate is redissolved and then evaporate down to ¼ of the volume again. After cooling, rinse with dist. H_2O into a 50 mL graduated flask, fill up to the mark and mix.

Analysis

Pipet out 10.0 mL of the sample solution as prepared above, (corresponding to 2 mL original bath) into a beaker, add 10 mL dist. H_2O, 10 mL ammonia and 5 mL hydrazine solution. Boil for a short time and (use a fume cupboard) and after cooling, titrate with *c*(KCN) = 0.2 mol/L.

Calculations

1 mL *c*(KCN) = 0.2 mol/L = 2.935 mg Ni

g/L Ni = EP1 * C01 * C02 / C00

C00 = Sample size in mL (2)
C01 = 2.935
C02 = Titer of the KCN solution

Literature

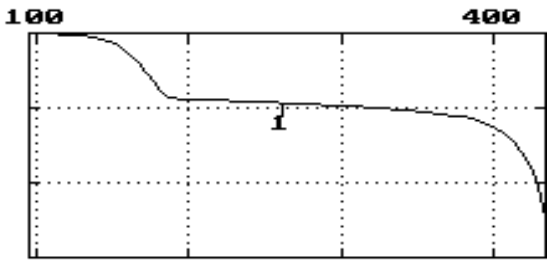
- Trepka-Bloch,E.
  Potentiometrische Bestimmung von Nickel neben Ag, As, Bi, Co, Cu und Fe
  Chemist Analyst 43, (1954) 63-65
  Ref: Fresenius,Z.Anal.Chem. 147, (1955) 143
- Luke,C.L.
  New rapid method for the determination of nickel in ferrous and ferromagnetic metals
  Anal.Chem. 33, (1961) 96-98

Figures

```
'pa
785 DMP Titrino          02287   785.0010
□R□date  1999-07-15    time  08:54        2
MET  U             ********
parameters
>titration parameters
  V step                 0.10 ml
  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                   5 ml
  stop U                 OFF mV
  stop EP                  9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                      30 mV
  EP recognition:         all
  fix EP1 at U            OFF mV
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  limit smpl size:        OFF
  activate pulse:         OFF
          ------------
```

Fig. 1 Parameters Titrino

```
 'fr
785 DMP Titrino         02287
785.0010
□R□date  1999-07-15    time  08:22
1
card label:Appl.751
U(init)           94 mV MET U
********
smpl size         10 ml
EP1            1.900 ml            262
mV
Ni              0.56 g/l
time             188 s
stop V reached
          ============
```




```
 'cu
785 DMP Titrino         02287
785.0010
□R□date  1999-07-15    time  08:22
1
start V      0.000 ml  MET U
********
2.0 ml/div      dU=100.0 mV/div
```

```
 'BMP-File:
C:\METROHM\DATASCAN\ASWNH4.bmp
□2          ============
```

Fig. 2 Results and titration curve