
Application Bulletin

Of interest to: Metals, electroplating

A 10

Titrimetric determination of free boric acid and tetrafluoroboric acid in nickel plating baths

Summary

This bulletin describes the simultaneous potentiometric titration of free boric acid and free tetrafluoroboric acid in nickel plating baths. After addition of mannitol, the formed mannitol complexes are titrated with sodium hydroxide solution. The determination is carried out directly in the plating bath sample; nickel and other metal ions do not interfere.

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
 - 2.728.0040 Magnetic Stirrer
 - 6.3014.223 Exchange Unit
 - 6.0222.100 combined LL pH glass electrode with 6.2104.020 electrode cable
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Reagents

- Titrant: sodium hydroxide solution, $c(\text{NaOH}) = 0.1 \text{ mol/L}$ (or more diluted)
 - D-Mannitol solution, $w(\text{mannitol}) = 10\%$ in dist. water
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Analysis

Pipet a defined volume of the sample into a plastic beaker, add 30 mL dist. water and 10 mL $w(\text{mannitol}) = 10\%$ and titrate with $c(\text{NaOH}) = 0.1 \text{ mol/L}$.

Calculation

Two equivalence points are obtained, the first of which corresponds to the HBF_4 content and the difference between the second and the first equivalence point to the H_3BO_3 content.

1 mL $c(\text{NaOH}) = 0.1 \text{ mol/L}$ corresponds to 8.781 mg HBF_4 or 6.183 mg H_3BO_3

$$\text{g/L HBF}_4 = \text{EP1} * \text{C01} / \text{C00}$$

$$\text{g/L H}_3\text{BO}_3 = (\text{EP2} - \text{EP1}) * \text{C02} / \text{C00}$$

EP1 = titrant consumption to reach the first EP in mL

EP2 = titrant consumption to reach the second EP in mL

C00 = sample volume in mL

C01 = 8.781

C02 = 6.183

Figures

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'pa
736 GP Titrino           04268   736.0011
date 99-12-17           time 14:56     9
DET pH                   AB 195
parameters
>titration parameters
  meas.pt.density        4
  min.incr.              10.0 µl
  titr.rate              max. ml/min
  signal drift           20 mV/min
  equilibr.time          38 s
  start V:               OFF
  pause                  0 s
  dos.element:           internal D0
  meas.input:            1
  temperature             25.0 °C
>stop conditions
  stop V:                 abs.
  stop V                  4 ml
  stop pH                 OFF
  stop EP                  9
  filling rate            max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                     5
  EP recognition:         all
  fix EP1 at pH           OFF
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:          value
  activate pulse:         OFF
=====

'fm
736 GP Titrino           04268   736.0011
date 99-12-17           time 14:56     9
DET pH                   AB 195
>calculations
HBF4=EP1*C01/C00;2;g/l
H3BO3=(EP2-EP1)*C02/C00;2;g/l
C00=                      0.250
C01=                      8.781
C02=                      6.183
=====
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Fig. 1: Parameter settings and calculation formulae for the determination of free boric acid and tetrafluoroboric acid.

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'fr
736 GP Titrino          04268  736.0011
date 99-12-17         time 14:56    9
pHc(init)            3.10    DET pH    AB 195
smp1 size            0.250 ml
EP1                  0.834 ml          4.19
EP2                  2.786 ml          8.34
HBF4                 29.29 g/l
H3BO3                48.28 g/l
stop V reached
=====
    
```

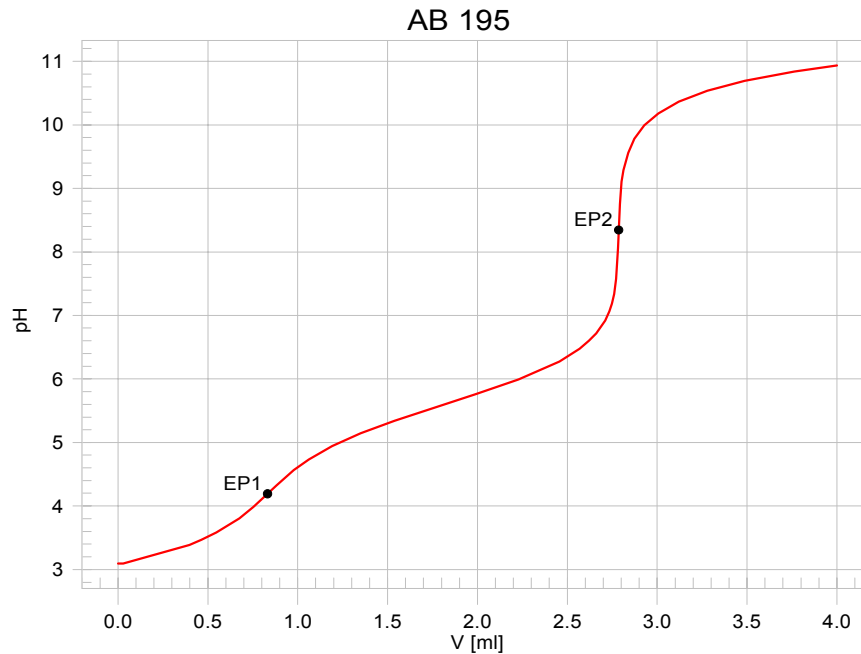


Fig. 2: Result block and titration curve for the determination of free boric acid and tetrafluoroboric acid in a nickel plating bath.

Literature

- E. Scholz
Die Analyse von Fluorboratbädern und anderen Fluorboratlösungen
Galvanotechnik 66 (1975) 811–819.
- D. Strohm
Automation komplexer Titrations am Beispiel eines galvanischen Nickelbades
GIT 32 (1988) 369–373.