Application Note

Quantifying boric acid in a nickel plating solution

Industry	Metals
Instrument	Automatic potentiometric titrator
Measurement method	Potentiometric titration / Neutralization titration
Standards	

1. Scope

The appropriate pH range for a nickel plating solution is 3.0 to 6.2. A good plating film will be formed within this pH range. In general, boric acid is used to adjust the pH. It also serves to whiten the appearance, and to improve the physical properties including the film stress and ductility. Accordingly, it is very important to control the concentration of boric acid in a nickel plating solution.
This Application Note describes an example of measuring the concentration of boric acid in a nickel plating solution by potentiometric titration.
Boric acid is a very weak acid, so direct titration is impossible. However, it forms a chelate complex with mannitol and other polyols, becoming a comparatively strong acid.

$$H_3BO_3 + C_6H_{14}O_6 \rightarrow (C_6H_{12}O_6BO)H + 2H_2O$$

The concentration of boric acid can be determined if the chelate complex formed by adding mannitol is titrated with sodium hydroxide. If metal ions forming hydroxides from the low pH are included, the measurement results will include false positive errors. In this case, add potassium ferrocyanide to mask the coexisting metal ions, preventing the effect mentioned above.

2. Post-measurement procedure

To suppress efflux and enrichment of the electrolyte when storing the electrodes, seal the electrolyte filling port in the combined glass electrode with a rubber stopper.
The performance of the combined glass electrode quickly deteriorates if it is stored while dry. The following storage methods are recommended.

- Short-term storage (less than one month): Store in pure water.
- Long-term storage (one month or longer): Store in a mixture of equal amounts (by volume ratio) of a pH 4 standard solution and a 3.3 mol/L aqueous potassium chloride solution.

3. Apparatus

Main unit	Automatic potentiometric titrator (Preamplifier: STD)
Electrode	Combined glass electrode (Inner solution: 3.3 mol/L potassium chloride solution)
Temperature compensated electrode	

4. Reagents

Titrant	0.1 mol/L sodium hydroxide solution
Additive reagents	10 %-potassium ferrocyanide solution, mannitol

5. Procedure

1) Introduce exactly 1 mL of the sample into a beaker.
2) Add 50 mL of pure water, 10 mL of a 10 % aqueous potassium ferrocyanide solution, and 4 g of mannitol, and stir.
3) When completely dissolved, titrate with a 0.1 mol/L aqueous sodium hydroxide solution.

6. Calculation

Boric acid (g/L) = (EP1 – BL1) × TF × C1 × K1 / S

EP1	Titration volume required to the first endpoint (mL)
BL1	Titration volume required for a blank test (0 mL)
TF	Titration solution factor (0.9916)
C1	Concentration conversion coefficient (6.1831 g/L)
K1	Unit conversion coefficient (1)
S	Amount of sample introduced (mL)

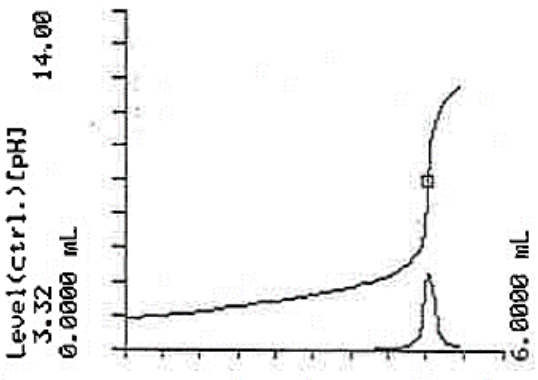
7. Example

— Parameter —

<Titr. Mode>	Auto Int.	**<Ctrl. Para.>**	
<Titr. Form>	Level Stop	Number of EP	1
		End Sense	Auto
<Titr. Para.>		Gain	1
Max Volume	20 (mL)	Data Sampling	Auto
Channel/Unit(Ctrl.)	Ch1, mV	Ctrl. Speed	Standard
Wait Time	0 (s)	Other Control	Standard
Dose Mode	None	Stirrer Speed	4

（The above condition is an example. The setting condition depends on the model.）

— Example of Titration curve —



— Measurement results —

	Sample (mL)	Titration (mL)	Boric acid (g/L)
1	1.00	4.7892	29.4
2	1.00	4.7769	29.3
3	1.00	4.7914	29.4
Average			29.3
SD			0.1
RSD (%)			0.2