Application Note

Determination of silver cyanide in silver plating solution

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

1. Scope

Warning

- ***Toxic gas and Exothermic reaction***

  The decomposition of cyanide ion by sulfuric acid and nitric acid releases hydrogen cyanide via an exothermic reaction. Decomposition operations should be performed in fume hoods, and protective masks and gloves should be worn.

The concentration of silver cyanide in the plating bath greatly affects the quality of silver plating. Therefore, controlling its concentration is extremely important. The concentration of silver cyanide can be determined by decomposing the cyanide ions and titrating the silver ions produced with a sodium chloride standard solution (Note 1).

$$Ag^+ + Cl^- \rightarrow AgCl$$

This application shows an example of the determination of silver cyanide in a silver plating solution.

2. Post-measurement procedure

- Polish the detection area of the combined silver electrode with polishing paper or other material.
- Reference internal liquid (the combined silver electrode) is replaced about once every two weeks.

3. Apparatus

Main unit	Automatic potentiometric titrator (Preamplifier STD)
Electrode	Combined silver electrode (Reference internal solution 1 mol/L potassium nitrate solution)

4. Reagents

Titrant	0.1mol / L Sodium chloride solution
Additive reagents	Concentrated sulfuric acid, Concentrated nitric acid

5 Procedure

1) Add exactly 10 mL of the sample into a beaker.
2) **Warning (Hydrogen cyanide and Exothermic reaction)**
   Add 20 mL of concentrated sulfuric acid and three drops of concentrated nitric acid and then gently stir to induce the reaction.
3) After the solution cools to room temperature, add 100 mL of pure water and stir until all solids dissolve.

4) Titrate with 0.1 mol/L sodium chloride solution.

6. Calculation

Silver cyanide (g/L) = (EP1 - BL1) × TF × C1 × K1/S

EP1	Titration volume (mL) for sample measurement
BL1	Titration volume for blank test (0 mL)
TF	Titrant factor (1.0137)
C1	Concentration conversion coefficient (13.389)
K1	Unit conversion coefficient (1)
S	Amount of sample introduced (mL)

7. Example

— Parameter —

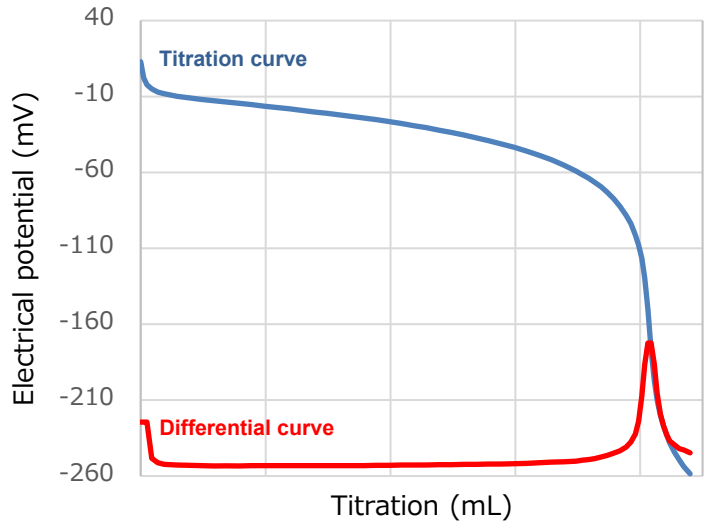
A titration mode "Automatic intermit control" used in this application note is recommended to use for slow chemical reactions such as precipitation titration.

Note: The below titration parameters including "Max. Volume" in "Titr. Para" should be adjusted by the operators depending on the sample concentration.

<Titr. Mode>	Auto Int.	**<Ctrl. Para.>**	
<Titr. Form>	EP Stop	Number of EP	1
		End Sense	Auto
<Titr. Para.>		Gain	1
Max Volume	40 (mL)	Data Sampling	On
Channel/Unit(Ctrl.)	Ch1, mV	Data sampling potential	20 (mV)
Wait Time	0 (s)	Data sampling volume	0.5 (mL)
Dose Mode	None	Ctrl. Speed	Standard
		Other Control	Standard
		Stirrer Speed	4

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

— Example of titration curve —



— Measurement results —

Table 1 Measurement results of silver cyanide in silver plating solution

	Sample (mL)	Titration (mL)	Silver cyanide (g/L)
1	10	31.8950	43.29
2	10	31.9126	43.31
3	10	31.5238	42.79
Average	-	-	43.13
SD	-	-	0.30
RSD (%)	-	-	0.69

8. Note

Note 1) The cyanide ion tends to form complex with the silver ion, so it must be decomposed with sulfuric acid and nitric acid.