Application Note

Determination of free potassium cyanide in silver plating solution

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

1. Scope

CAUTION

Silver plating solution is generally harmful to the human body. When you follow this application note, wear masks, gloves, protective equipment, etc., and conduct the operations inside fume hoods.

The free cyanide concentration of planting solution in high-speed plating bath is one of the most important factors for plating quality and plating speed. To prepare the solution suitable for the planting, the free cyanide concentration is measured.
This application shows an example of measurement of free potassium cyanide in a silver cyanide plating bath. Silver nitrate solution was used as the titrant for this measurement. In this titration, silver ion reacts with cyanide ion to form dicyanoargentate ion.

$$Ag^+ + 2CN^- \rightarrow [Ag(CN)_2]^-$$

In this method, the potential changes with the concentration ratio of silver ions. The inflection point on the titration curve is detected as the endpoint.

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	0.1 mol/L silver nitrate solution	
Additive reagents	0.5 g potassium iodide	(Note 1)

5. Procedure

1) The sample is collected with a volumetric pipette and added to a beaker, then 100 mL of pure water is added.
2) Add 0.5 g of potassium iodide and stir until completely dissolved.
3) Titrate with 0.1mol/L silver nitrate solution.

6. Calculation

Free potassium 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 (1.0016)
C1	Concentration conversion coefficient (13.024)
K1	Unit conversion coefficient (1)
S	Amount of sample introduced (mL)

7. Example

— Parameter —

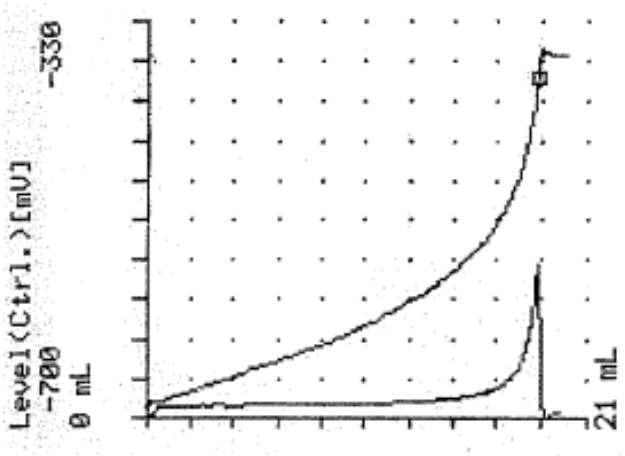
A titration mode "Automatic intermit control" used in this application note is recommended to use for slow chemical reactions such as precipitation titration.

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>	Level Stop	Number of EP	1
		End Sense	Auto
<Titr. Para.>		Gain	1
Max Volume	30 (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 free potassium cyanide in silver plating solution

	Sample (mL)	Titration (mL)	Free potassium cyanide (g/L)
1	2	18.5643	121.08
2	2	18.5138	120.75
3	2	18.5386	120.92
Average	-	-	120.92
SD	-	-	0.16
RSD (%)	-	-	0.14

8. Note

Note 1: The addition of potassium iodine is recommended to detect the endpoint easily from our experience when the potentiometric titrator is used because it enables the endpoint to be clear on the titration curve.