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Application Note

Determination of copper (I) cyanide in a copper plating solution

Industry	Metals
Instrument	Automatic potentiometric titrator
Measurement method	Photometric titration / Chelatometric titration
Standards	

1. Scope

CAUTION

Copper plating solutions are generally harmful to the human body. When following this application note, wear masks, gloves, protective equipment, etc., and conduct the operations inside a fume hood.

The concentration of copper cyanide in the copper plating solution depends on the plating application (strike plating bath, bright plating bath, etc.). The concentration is also extremely important for controlling the quality of the copper plating. The determination of copper cyanide requires pretreatment to decompose the copper cyanide complex ions (Note 1). The divalent copper ions produced in the pretreatment are titrated with ethylene diamine tetra-acetic acid disodium salt (EDTA-2Na) solution to determine the copper (I) cyanide concentration. In this measurement, a photometric sensor is used to detect the discoloration of the indicator.

$$Cu^{2+} + EDTA\text{-}2Na \rightarrow Cu\text{-}EDTA + 2Na^{+}$$

This application note shows an example of the determination of copper (I) cyanide in a copper plating solution.

2. Post-measurement procedure

Clean the photometric sensor carefully in pure water. Wipe off any moisture with a paper waste cloth, etc., and store it in a dry place.

3. Apparatus

Main unit	Automatic potentiometric titrator (photometric preamplifier PTA)
photometric sensor	Band pass filter wavelength 530nm

4. Reagents

Titrant	0.1mol / L EDTA solution
Additional reagent	Ammonium persulfate, Ammonia water (28%)
Indicator	Pyridylazo-naphthol (PAN) solution in ethanol (0.1%)

5. Procedure

1) Dilute the sample exactly ten times with pure water.
2) Accurately collect 5 mL of diluted solution in a beaker, then add 3g of ammonium persulfate.
3) Add 150mL of pure water, and stir to dissolve the solid.
4) Add 5mL of Ammonia water and add 0.5mL of PAN ethanol solution.
5) Titration with 0.1 mol / L EDTA solution.

6. Calculation

Copper (I) cyanide (g / L) = (EP1 – BL1) × TF × C1 × K1 / (S × R)

EP1	Titration volume (mL)
EP2	Titration volume (mL) of blank test = 0
TF	Factor of titrant = 1.0001
C1	Concentration conversion coefficient = 8.9563
K1	Unit conversion coefficient = 1
S	Collected volume of sample after dilution (mL)
R	Dilution factor = 0.1

7. Example

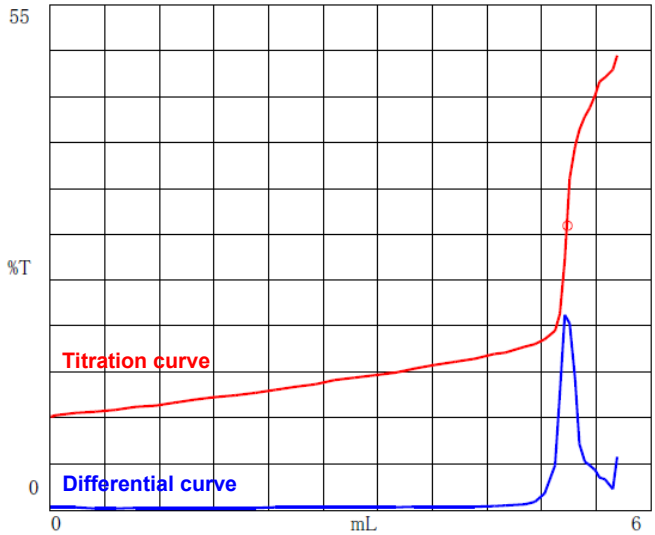
— Parameter —

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	20 (mL)	Data Sampling	On
Channel/Unit(Ctrl.)	Ch3, 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 volume (mL)	Copper(I) cyanide (g/L)
1	5	5.1710	92.64
2	5	5.1849	92.88
3	5	5.1797	92.79
Average	-	-	92.77
SD	-	-	0.13
RSD (%)	-	-	0.14

*Sample amount after dilution

8. Notes

Note 1) The copper cyanide complex ion is decomposed by the chemical reaction with ammonium persulfate added, and the copper ions is oxidized from monovalent to divalent in this pretreatment process.

$$2Na_2Cu(CN)_3 + 7(NH_4)_2S_2O_8 + 12H_2O \rightarrow$$
$$\underline{2Cu^{2+}} + 2SO_4^{2-} + 2Na_2SO_4 + 10(NH_4)_2SO_4 + 6CO_2$$