Application Note
Determination of potassium carbonate in a silver plating solution

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

1. Scope

Warning

- ***Toxic hydrogen cyanide***

  Toxic hydrogen cyanide is released during titration (Note 1). When you follow this application note, wear masks, gloves, protective equipment, etc. The automatic potentiometric titrator should be installed and used in fume hoods where local exhaust ventilation is possible.

The concentration of potassium carbonate in the silver plating solution affects the plating quality and plating rate. Therefore, the concentration of potassium carbonate should be controlled so that it does not exceed the standard value. In the determination by neutralization titration, potassium carbonate is neutralized in two steps (reaction formula (1), (2)) by titrating with hydrochloric acid (Note 2).

$$K_2CO_3 + HCl \rightarrow KHCO_3 + KCl \quad (1)$$
$$KHCO_3 + HCl \rightarrow KCl + CO_2 + H_2O \quad (2)$$

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

2. Post-measurement procedure

Seal the refill port for electrolyte of electrode by rubber septum.
The performance of the electrode quickly degrades if it is stored while dry. The following storage methods are recommended.

- Short-term storage (less than one month) Store it immersed in pure water.
- Long-term storage (one month or longer) Store it immersed in a 1:1 volume ratio mixture of a standard solution of pH4 and a 3.3 mol/L KCl solution.

3. Apparatus

Main unit	Automatic potentiometric titrator (preamplifier STD)
Electrode	Combined silver electrode (Reference internal solution 1mol / L Potassium chloride solution)
Temperature	Compensation electrode

4. Reagents

Titrant	0.5mol / L Hydrochloric acid solution

5. Procedure

1) Dilute the sample exactly ten times with pure water.
2) Accurately collect 5 mL of diluted solution in a beaker.
3) Add 100mL of pure water.
4) Titrate with 0.5 mol / L hydrochloric acid solution.

6. Calculation

Potassium carbonate (g / L) = (EP2 – EP1) × TF × C1 × K1 / (S × R)

EP1	First endpoint titration volume (mL)
EP2	Second endpoint titration volume (mL)
TF	Factor of titrant = 1.0033
C1	Concentration conversion coefficient = 69.1
K1	Unit conversion coefficient = 1
S	Collected volume of sample after dilution (mL)
R	Dilution factor = 0.1

7. Example

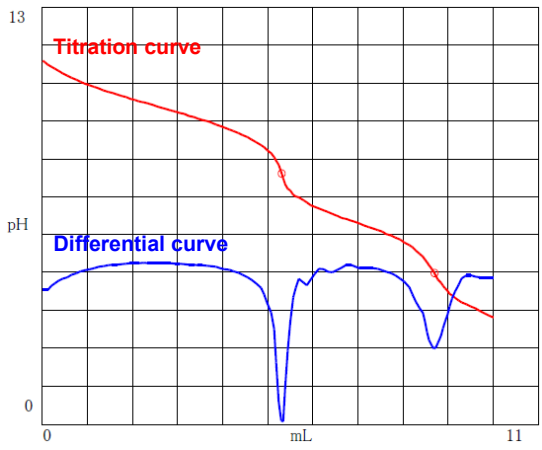
— Parameter —

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	2
		End Sense	Auto
<Titr. Para.>		Gain	1
Max Volume	30 (mL)	Data Sampling	On
Channel/Unit(Ctrl.)	Ch1, pH	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 —

Table 1 Measurement results of potassium carbonate in silver plating solution

	Sample (mL)*	First endpoint titration volume (mL)	Second endpoint titration volume (mL)	Potassium carbonate (g/L)
1	5	5.3128	8.7090	470.90
2	5	5.3136	8.7239	472.86
3	5	5.3141	8.7216	472.47
Average	-	-	-	472.08
SD	-	-	-	1.04
RSD (%)	-	-	-	0.22

*Sample amount after dilution

8. Notes

Note 1) **Warning**
This titration should be conducted in a fume hood, as Hydrogen cyanide, which is highly toxic, is produced on the chemical reaction between potassium cyanide and hydrochloric acid.

$$HCl + KCN \rightarrow HCN + KCl$$

Note 2) In this titration, two endpoints are obtained. The first endpoint as neutralization equivalence point is obtained by the chemical reaction (formula (1) above) between hydrochloric acid and potassium carbonate including the potassium cyanide and potassium hydroxide, which are generally added in the silver plating solution. The second point is obtained by the chemical reaction (formula (2) above) between hydrochloric acid and potassium hydrogen carbonate. The concentration of potassium carbonate is calculated from the difference between the two endpoints (endpoint 2 – endpoint 1).