HIRANUMA APPLICATION DATA		Automatic Titrator	Data No.	G2	Apr. 5, 2019		
Metals	Determination of trace chloride ion in						
Wictais	copper sulfate solution						

# 1. Abstract

This report introduces an example of the determination of trace chloride ion in the solution containing highly concentrated copper sulfate.

The precipitation titration with silver nitrate standard solution is generally used for the determination of chloride ion in copper sulfate solution. The endpoint of the titration is detected with indicator method or the potentiometric method. The potentiometric method is used for this sample because the color change of indicator reagent is obscure in this sample containing highly concentrated copper sulfate. However, it tends to show the difficulty to detect the endpoint because of the less sensitivity of electrode under highly concentrated copper sulfate. Therefore the potentiometric titration is performed with the specially treated silver electrode that the silver chloride is coated.

 $Cl^{-} + AgNO_3 \rightarrow AgCl + NO_3^{-}$ 

### 2. Configuration of instruments and reagents

(1) Configuration of	instruments					
Main unit	: Hiranuma Automatic	Titrator COM Series				
Electrode	: Silver electrode	AG-311A (Silver chloride-coated electrode)				
	Reference electrode	MS-231Z				
	*The following electrode	es are also usable.				
	• AGR-801AZ (Silve	r chloride-coated silver reference combination electrode)				
	• AGR-811AZ (Silver chloride-coated silver reference combination electrode,					
	doub	le junction type)				
	*Remark					
	The general reference	e electrode (RE-201Z) cannot be used for this titration because				
	KCl inner solution m	ight come out to sample solution and it causes measurement error.				
	The inner electrodes	of AGR-801Z and MS-231Z use mercury (I) sulfate. When these				
	electrodes are dispose	ed, please ask the specialized industrial waste disposal operator.				
(2) Reagents						
Titrant	: 0.01 mol/L Silver nitra	ate standard solution				

#### **3.** Measurement procedure

(1) Dispense 20 mL of sample into a 100 mL beaker with volumetric pipette.

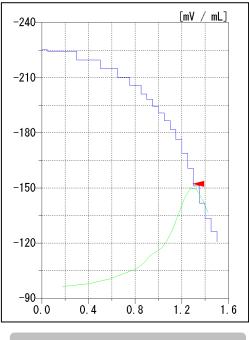
- (2) Add about 20 mL of DI water.
- (3) Immerse electrodes and titrate with 0.01 mol/L Silver nitrate standard solution.



# 4. Measurement conditions and results

			Example of thration	conuni	on			
Cnd. No.	1							
Method	Auto		Constant No.	1		Mode No.	8	
Buret No.	1		Size	20	mL	Pre Int	0	sec
Amp No.	2		Blank	0	mL	Del K	5	
D.Unit	mV		Morality	0.01	mol/L	Del Sens	0	mV
S- Timer	5	sec	Factor	1.005		Int Time	5	sec
C.P. mL	0	mL	Κ	35.45		Int Sens	3	mV
T.Timer	0	sec	L	0		Brt Speed	2	
D.P. mL	0	mL	Unit	PPM		Pulse	40	
End Sens	80		Formula					
Over mL	0.5	mL	(D-B)*K*F*M	*1000/S				
Max Vol.	20	mL	Decimal Places	4				
			Auto input parameter		None			

## Example of titration condition



Measurement results				
Number of	Size	Titrant	Chloride ion	
Measurement	(mL)	Volume (mL)	Concentration (ppm)	
1	20	1.294	23.0508	
2	20	1.294	23.0508	
3	20	1.290	22.9796	
~		Avg.	23.03 ppm	
Statistic calculation	SD		0.04 ppm	
	RSD		0.18 %	

#### Example of titration curve

## 5. Note

· Indicator electrode

The sensitivity of the electrode is important for this measurement. It tends to show the difficulty on the automatic endpoint detection because of the less sensitivity of normal electrode under highly concentrated copper sulfate. The silver chloride-coated silver electrode can provide acceptable results on this measurement. However, it is degraded by prolonged use even if the silver chloride is coated on the electrode. The recoating of silver chloride is required when the sensitivity of electrode gets lowered.

Keywords: Chloride ion, Precipitation titration, Copper sulfate solution, Copper sulfate plating solution, Silver chloride coating

\*Some measurement would not be possible depending on optional configuration of system.

