

Iron and Steel

Free cyanide in Silver plating solution

Precipitation titration by
Automatic Potentiometric Titrator

Standard

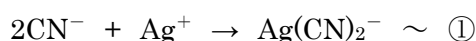
1. Abstract

Here, we demonstrate how to measure concentration of free cyanide in silver plating solution which contains cyanide. Potassium iodide (KI) as indicator is added to a sample solution which is first alkalified with ammonia water, and the sample is titrated with 0.1mol/L silver nitrate solution up to the endpoint. The titration volume is then converted to potassium cyanide to obtain free cyanide concentration.

The endpoint (EP) is the maximum inflexion point on titration curve.

This method is referred to Liebig-Deniges Method.

By adding KI, silver iodide (AgI) is precipitated following the below chemical reaction (Eq.1), which clearly indicates EP. Add ammonia water in order to avoid early EP reached in titration. AgI is insoluble in ammonia water, and therefore, a sample of ammonia alkaline solution can be titrated.



2. Reference

- 1) Experiment and Calculation in Quantitative Analysis – Vol.2 – (Kyoritsu Publication)
- 2) Quantitative Analytical Chemistry (Baifuh Publication)

3. Cautions in measurement

- 1) Note that cyanide generates toxic gas of hydrogen cyanide if contacts acid including carbon dioxide in the atmosphere. Due care must be taken.
- 2) In order to avoid measurement error due to diffused chlorine, use a reference electrode of mercury sulfide or use a silver chloride reference electrode of which junction liquid is replaced with 1mol/L potassium nitrate solution.
- 3) Clean the tip of silver electrode with polishing paper before use.
- 4) A convenient combination silver electrode is used in this test, however, a combination of single silver electrode and reference electrode also can be used.

4. Post-measurement care

After a series of measurements is over, clean the tip of electrode with polishing paper as noted above in 3. Cautions in measurement.

5. Test equipment

Main unit: Automatic potentiometric titrator (Standard preamplifier STD—)
Electrode: Option Combination silver electrode (Junction liquid: 1mol/L-Potassium nitrate solution [№ 988110026])

6. Reagent

Reagent : 0.1mol/L Silver nitrate solution
Additive : 10% Potassium iodide
 : 10% Ammonia water

7. Measurement procedure

—Measurement—

- 1) Precisely take in 5.0mL sample liquid into a 100mL beaker.
- 2) Add 3mL of 10% ammonia water.
- 3) Add 50mL pure water.
- 4) Add 2mL of 10% potassium iodide.
- 5) Titrate with 0.1mol/L silver nitrate to obtain concentration of free cyanide.

8. Formula

Concentration of free cyanide (g / L) = (EPl - BLl) × TF × Cl × K1 / SIZE
(converted to KCN)

EPl : Titration volume (mL)
BLl : Blank level (0.0mL)
TF : Factor of reagent (1.008)
Cl : Concentration conversion coefficient (13.024mg/mL)
 (Potassium cyanide (mg) equivalent to 1mL of 0.1mol/L AgNO₃)
K1 : Unit conversion coefficient (1)
SIZE : Sample size (mL)

9. Example of measurement

— Ambient condition —

Room temperature : 22 °C	Humidity : 20 %	Weather : Fair
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-Titration parameters-

Model : AT-510 Method No. : 10 Titr.mode : Auto Titration Titr.form : EP Stop	
[Titration parameter]	[Result parameter]
Form : EP Stop APB No. : 1 Unit No. : 1 Detector No. : 1 Unit : mV Max. Volume : 20.0mL Wait Time : 0s Direction : Auto	<Calculation> Calc.Type : Sample Conc.1 : Set CO1= (EP1-BL1)*TF*C1*K1/SIZE Unit : g/L EP No. : 1 Temp.Comp. : Off
[Control parameter]	<Constant>
End Point No. : 1 End sense : Auto End Point Area : Off Separation : Off Over Titr.Vol. : 0mL Gain : 1 Data samp.Pot. : 4.0mV Data samp.Vol. : 0.5mL Control Speed : Medium	C1(mg/mL) : 13.024 K1 : 1 [Titr. constant] Factor : 1.008 [Blank list] Blank1 : 0.0

-Titration curve-

*** Result *** Sample No. : 01-01 Date : 2001/1/17 11:51 Sample ID :
Method No. : 10 <Auto Titration> Method Name :
Titr.Time : 00:03:55 Size : <u>5.0mL</u>
Conc-1 : <u>36.203g/L</u>
End point-1 Volume : <u>13.7884mL</u> Potential : - 124.4mV

(The above parameters and titration curve are obtained from titration by AT-510 unit.)

<<Titration parameter>> Form: of titration / APB No. the burette used in titration / Unit No.: APB Unit File number Detector No.: the detector used in titration / Unit: of potential / Max Volume: of titration Wait Time: before titration starts / Direction.: of titration
<<Control parameter>> End Point No. number of EPs detected / End sense: EP detection / End Point Area: detection area Separation: of potential/ Auto Simulation: EP redetection / Over Titr.Vol. over-titration volume Gain: sensitivity of detection signal / Data samp.Pot.: potential changes of sampling signal Data samp.Vol.: titration volume of sampling signal / Control Speed: of dosing
<<Result parameter>> Calc.Type of formula / Conc.1: formula 1 / Unit: of result / EP No.: for calculation Temp.Comp.: temperature compensation of titration liquid / C1(mg/mL): concentration coefficient K1: unit conversion coefficient / Factor: of titration liquid / Blank1: blank level 1

—Measurement results—

n	Sample (mL)	Titrate (mL)	Free cyanide (g/L)	Concentration of free cyanide	
1	<u>5.0</u>	<u>13.7884</u>	<u>36.203</u>	Mean	36.454 g/L
2	5.0	13.9800	36.706	SD	0.2515 g/L
3	5.0	13.8839	36.454	RSD	0.6899 %

* The above results were obtained by 3 tests of the same sample.

* Red underline shows the data from page 3/4.

10. Summary

Plating means surface finishing of materials like metals by coating metallic film or its method. In this note, an example of quantitative method for toxic cyanide which is used in plating is shown. The sample measurement resulted with significant repeatability of 0.7% relative standard deviation.

The automated potentiometry assures precise and reliable measurement.

The analysis of free cyanide (KCN conversion) in silver plating solution can be made by any of the following titration systems manufactured by Kyoto Electronics (KEM).

【AT-610】



Awarded Product of Supreme Technology from Kyoto City

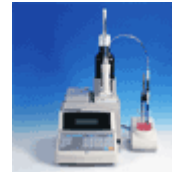
- Easy key entry by touch panel of large color LCD (8-inch wide)
- Simultaneous titration in parallel
- Both potentiometric and Karl Fischer moisture titration (coulometric • volumetric) can be performed at a time.

【AT-510】



- Compact and cost performance model
- PC card expands data memory for convenience and versatility.

【AT-500N-1】



- Low cost and high performance
- Easy view with back light LCD
- GLP/GMP conformed model

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