

<b>HIRANUMA APPLICATION DATA</b>	Automatic Titrator	Data No.	E10	Apr. 19, 2018
<b>PLATING &amp; ETCHING SOLUTION</b>	<b>Quantitative determination of total nickel (Ni<sup>2+</sup>) in nickel plating solution</b>			

## 1. Abstract

The control and analysis of nickel plating solution is very important process for the product quality. Analytical components of the general nickel plating solution are 1) nickel sulfamate, 2) nickel chloride, and 3) boric acid. The example of quantitative determination for total nickel which is total amount of nickel chloride and nickel sulfamate in nickel plating solution is introduced in this report. The total nickel is determined by photometric titration with EDTA standard solution using indicator MX.



## 2. Configuration of instruments and reagents

### (1) Configuration

Main unit : Hiranuma Automatic Titrator COM series (Photometric titrator unit type M) with 530 nm optical filter

### (2) Reagents

Titrant : 0.1 mol/L EDTA standard solution  
 Buffer solution : Diluted ammonia solution (1:1, v/v)  
 Mix 28 ~30 % ammonia solution and DI water with 1:1 volume ratio.  
 Indicator : MX indicator  
 0.1 g of murexide and 10 g of potassium sulfate are well-mixed by mortar.

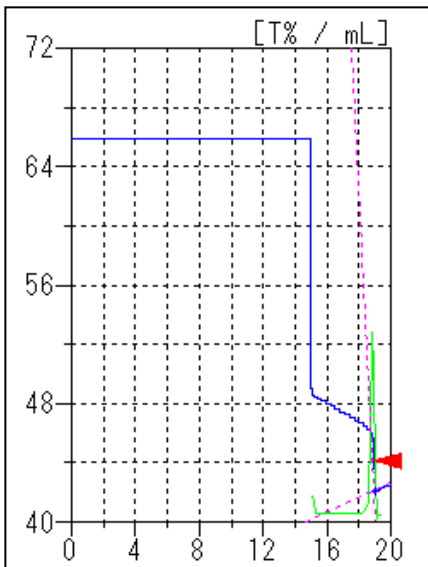
## 3. Measurement procedure

- (1) Dispense 1 mL of sample with volumetric pipette into a 100 mL beaker.
- (2) Add 60 mL of DI water.
- (3) Add 20 mL of diluted ammonia solution.
- (4) Add 0.2 g of MX indicator.
- (5) Immerse photometric probe into sample solution and titrate with 0.1 mol/L EDTA standard solution.

## 4. Measurement conditions and results

### Examples of titration conditions

Cnd. No.	1	Constant No.	1	Mode No.	20
Method	B cross	Size	0 g	Pre Int	0 sec
Buret No.	1	Blank	0 mL	Del K	5
Amp No.	2	Morality	0.1 mol/L	Del Sens	0 mV
D.Unit	T%	Factor	1.007	Int Time	5 sec
S- Timer	5 sec	K	58.69	Int Sens	0 mV
C.P. mL	15 mL	L	0	BrT Speed	2
T.Timer	0 sec	Unit	g/L	Pulse	80
D.P. mL	0 mL	Formula	(D-B)*K*F*M/S		
End Sens	200	Digits	4		
Over mL	1 mL	Auto input parameter	None		
Max Vol.	40 mL				



Example of titration curve

### Measurement results

Number of Measurement	Size (mL)	Titration Volume (mL)	Total Nickel Concentration (g/L)
1	1	19.093	112.841
2	1	19.003	112.309
3	1	18.970	112.114
Statistic calculation			Average
			Standard deviation
			Coefficient of variation
			112.4 g/L
			0.376 g/L
			0.33 %

## 5. Note

### (1) Measurement condition

“Method” on condition parameter is set to “B Cross” because the endpoint is defined as the point where the change in color of the indicator is completed. The titrant is continuously added by using the function “CP mL”. This function is useful to add titrant until just before it reaches endpoint when the titrated volume is relatively large as described in this report. The measurement time can be reduced by “CP mL” function.

### (2) Calculation of nickel sulfamate

Nickel sulfamate concentration can be calculated by the subtraction of nickel chloride from total nickel.

Calculation of nickel sulfamate concentration is possible by the following formula.

$$\text{Nickel sulfamate tetrahydrate concentration (g/L)} = (CA - K \times 0.247) \times 5.502$$

CA : Measurement result of total nickel concentration (g/L)

K : Measurement result of nickel chloride hexahydrate (g/L)

0.247 : Coefficient for converting from nickel chloride hexahydrate to nickel  
(Ni(58.69) / NiCl<sub>2</sub>·6H<sub>2</sub>O(237.69))

5.502 : Coefficients for converting from nickel to nickel sulfamate tetrahydrate  
(Ni(NH<sub>2</sub>SO<sub>3</sub>)<sub>2</sub>·4H<sub>2</sub>O (322.93) / Ni (58.69))

When it is calculated as nickel sulfate hexahydrate concentration, change the coefficient for converting from 5.502 to 4.479.(NiSO<sub>4</sub>·6H<sub>2</sub>O (262.85) / Ni (58.69))

Keywords : Nickel plating solution, Total nickel, Photometric titration, Chelatometric titration

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