

Application Note

Determination of sodium phosphite pentahydrate

Industry	Chemicals
Instrument	Automatic potentiometric titrator
Measurement method	Potentiometric titration / Oxidation-reduction titration
Standards	

1. Scope

The following reaction occurs in the nickel plating solution used for electroless plating (Formula 1). As the plating reaction progresses, by-products (such as phosphite) accumulate and interfere with the deposition of the plating.



This Application Note introduces an example of measuring sodium phosphite in plating solution (Note 1).

2. Post-measurement procedure

Seal the refill port for electrolyte of reference electrode by rubber septum so that electrolyte is prevented from leaking out or concentrating, and store the electrode.

3. Apparatus

Main unit	Automatic potentiometric titrator (Preamplifier STD)
Electrode	Combined platinum electrode (Inner solution : 3.3 mol/L potassium chloride solution)

4. Reagents

Titrant	0.1 mol/L Ammonium iron (II) sulfate (0.1 mol/L Mohr's salt)
Additive	0.1 mol/L Ammonium vanadate (0.1 mol/L Ammonium metavanadate) 5 w/v %-Silver sulfate solution (Preparation Dissolve 5 g of silver sulfate in concentrated sulfuric acid to make the total volume 100 mL)

5. Procedure

- 1) Accurately collect 5 mL of sample into a beaker.
- 2) Accurately add 20 mL of 0.1 mol/L ammonium metavanadate.
- 3) Add 4 mL of 5 % silver sulfate solution (Note 1, Formula 2).
- 4) Add pure water to make the volume about 60 mL, and boil for 5 minutes.
- 5) After cooling to room temperature, titrate with 0.1 mol / L Mohr's salt solution (Note 1, Formula 3).

* The Blank test is performed under the same conditions as the sample measurement.

6. Calculation

$$\text{Sodium phosphite (g/L)} = (\text{BL1} - \text{EP1}) \times \text{TF} \times 10.802 / \text{S}$$

BL1	Titration amount (mL) of Blank test = 20.0825
EP1	Titration amount (mL)
TF	Factor of Titrant = 0.9992
S	Sample size (g)

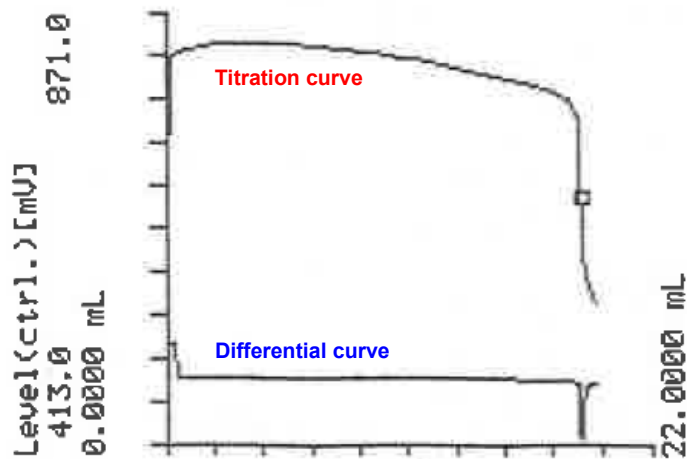
7. Example

— Parameter —

<u><Titr. Mode></u>	Auto Int.	<u><Ctrl. Para.></u>	
<u><Titr. Form></u>	EP	Number of EP	1
		End Sense	Auto
<u><Titr. Para.></u>		Gain	1
Max Volume	40mL	Data Sampling	Auto
Channel/Unit(Ctrl.)	Ch1, mV	Ctrl. Speed	Standard
Wait Time	0s	Other Control	Standard
Tit. Type Check	No Check	Stirrer Speed	4
		Auto Int. Mode	Standard

(Listed above are example settings. Availability of settings may vary by instrument model.)

— Example of Titration curve —



— Measurement results —

n	Sample (mL)	Titration (mL)	Result (g/L)
1	5.0	18.6226	3.151
2	5.0	18.6218	3.153
3	5.0	18.6406	3.113
Average	-	-	3.139
SD	-	-	0.023
RSD (%)	-	-	0.72

8. Notes

Note 1) The measurement method used was oxidation-reduction titration. A constant excess amount of vanadate oxidizes phosphorous acid. The addition of silver salt as a catalyst is needed in this reaction to allow the oxidation of phosphorous acid to proceed quantitatively (Formula 2). The unreacted vanadate is then titrated with Mohr's salt (Formula 3).

