

## Technical Notes: Electroless Nickel Plating

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TITLE: Analysis for Nickel Metal

SCOPE: This method is a direct titration for nickel metal using EDTA. The procedure is quick and will work with automatic titrators using ampermetric or spectrometric probes.

### APPARATUS

1. Erlenmeyer flask 250 ml
2. Automatic burette
3. Chemicals
  - a. Distilled water
  - b. Ammonium hydroxide
  - c. 35% Triethanolamine
  - d. Murexide Indicator in sodium chloride
  - e. 0.1 M EDTA Na<sub>2</sub> Solution

### PROCEDURE:

1. Add exactly 10.0 ml of plating bath into the Erlenmeyer flask.
2. Add about 100 mls of distilled water to the flask.
3. Add 20 mls of Ammonium hydroxide to the flask.
4. Add 10 mls of 35% Triethanolamine
5. Add about 0.2 g of Murexide Indicator Mix to the flask. The solution should be light straw color.
6. Titrate immediately with standard 0.1 M EDTA Na<sub>2</sub> Solution to purple endpoint.

### ANALYSIS

$$\text{Nickel g/l} = (\text{MLS } 0.1 \text{ M EDTA Na}_2) \times 0.576$$



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### TITLE: Analysis for Sodium Hypophosphite

SCOPE: This is a direct titration for hypophosphite using indirect titration for iodate using thiosulfate. The method takes 10 or 30 minutes and can be automated using automatic titrators.

#### APPARATUS

1. Erlenmeyer flask 250 ml with stopper
2. 2 Automatic burettes, 25ml
3. 1x10 ml pipette
4. Chemicals
  - a. Distilled water
  - b. 0.1N Iodine
  - c. 0.1N Thiosulfate
  - d. 6N H<sub>2</sub>SO<sub>4</sub> Analytical Grade
  - e. Starch Indicator

#### PROCEDURE

1. Add exactly 2.0 mls of plating bath into the Erlenmyer flask.
2. Add approximatly 20 ml of 6N H<sub>2</sub>SO<sub>4</sub> to the flask.
3. Add exactly 25 ml 0.1N Iodine to the flask.
4. Stopper flask and place in a dark place for exactly 30 minutes.
5. Remove from dark area and wash stopper and neck of flask into the sample contents with deionized water.
- 6 Titrate immediately with standard 0.1N Thiosulfate Solution to light yellow point. Add a small amount of starch indicator. Solution will go black. Continue titration until solution goes clear.

#### ANALYSIS:

Hypophosphite g/l = (MLS 0.1N Iodine (25) - MLS 0.1N Thiosulfate) x 2.65



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### TITLE: Analysis for Sodium Ortho-phosphite

SCOPE: This is a titration method for ortho-phosphite using indirect titration for iodate using thiosulfate. The method takes 30 minutes and can be automated using automatic titrators.

#### APPARATUS:

1. Erlenmeyer flask 250 ml with stopper
2. 2 Automatic burettes, 25ml
3. 1x10 ml pipette
4. Chemicals
  - a. Distilled water
  - b. 0.1N Iodine
  - c. 0.1N Thiosulfate
  - d. 5% by weight Sodium bicarbonate Solution
  - e. 10% by volume Acetic Acid Solution
  - f. Starch Indicator

#### PROCEDURE:

1. Add exactly 2.0 mls of plating bath into the Erlenmyer flask.
2. Add 20 ml Deionized Water to the Erlenmyer flask.
3. Add 20 ml of Sodium bicarbonate.
4. Add exactly 50 ml 0.1N Iodine to the flask.
5. Stopper flask and place in a dark place for exactly 30 minutes.
6. Remove from dark area and wash stopper and neck of flask into the sample contents with deionized water.
7. Add 20 ml 10% Acetic Acid to Erlenmyer flask.
8. Titrate immediately with standard 0.1N Thiosulfate Solution to light yellow point. Add a small amount of starch indicator. Solution will go black. Continue titration until solution goes clear.

#### ANALYSIS:

Ortho-phosphite = ( 50 - MLS 0.1N Thiosulfate ) X 2.05 g/l

#### EXAMPLE:

Upon titrating with 0.1N Sodium thiosulfate the sample went clear at 27.6 ml.

Ortho-phosphite = ( 50 - 27.6 ) X 2.05

Ortho-phosphite = 45.9 g/l

Note: The amount of iodine may need to be increased if the ortho is greater than 80 g/l in the process. Increase the quantity to a range where the difference is within range.

0-75 g/l	50 ml
75-100 g/l	65 ml
100-150 g/l	100 ml
150-200 g/l	150 ml

