

Thermo. Titr. Application Note No. H-105

Title: Determination of Nickel in Hydrometallurgical Leach Liquors

Scope: Determination of the nickel content of hydrometallurgical leach liquors

Principle: A measured amount of acidic hydrometallurgical leach liquor is first treated with hydrogen peroxide to oxidize Fe(II) to Fe(III), then with potassium pyrophosphate solution to mask interference from Fe(III) and other metal ions. Ammonium acetate solution is then added as a pH modifier, before being titrated with standardized disodium dimethylglyoximate to an exothermic endpoint.

Reagents:

Titration: disodium dimethylglyoximate $c(\text{Na}_2\text{DMG}) = 0.5$ mol/L

pH modifier: Ammonium acetate solution $c(\text{NH}_4\text{OAc}) = 5$ mol/L

Masking agent: Potassium pyrophosphate solution $c(\text{K}_4\text{P}_2\text{O}_7) = 1$ mol/L

Oxidizing agent: Hydrogen peroxide solution $c(\text{H}_2\text{O}_2) = 30\%$ w/w

Standard nickel solution: $c(\text{Ni}) = 0.2$ mol/L. Prepared from commercial NiO, with a claimed content of 78.40% Ni. Alternatively, a solution prepared from pure Ni metal by digestion in aqua regia can be used

Method:

Basic Experimental Parameters:

Titration delivery rate (mL/min.)	6
No. of exothermic endpoints	1
Data smoothing factor (DSF)	30
Stirring speed (802 stirrer)	14
Delay before start of titration (secs.)	10

Standardization of Na_2DMG
Aliquots of standard Ni solution of volumes 1, 2, 3, 4 and 5 mL were pipetted by volumetric pipettes into individual

PP titration tubes. DI water was added to bring the titration volume to ~35mL.

The samples were placed in the rack of the 814 Sample Processor, and an automation program was devised which automatically added 5 mL NH₄OAc solution prior to the commencement of the titration.

The automated standardization program computes the titrant molarity from a regression analysis, and determines the coefficient of correlation for the linear regression.

Titrant dose rate: 6 mL/min
 Digital filter factor: 40
 Stirring speed: 10

Basic titration procedure: A 4 mL aliquot of acidic process liquor is pipetted by volumetric glass pipette into a PP titration tube, and 10 mL 30% w/w H₂O₂ solution plus 10 mL DI water added. The sample solution is swirled to mix prior to being placed in the sample rack.

The automation program adds successively with stirring between each addition:

- 10 mL K₄P₂O₇ solution
- 5 mL NH₄OAc solution

Notes:

- *It is critical that the K₄P₂O₇ solution be added and mixed prior to addition of the NH₄OAc solution.*
- *The nickel dimethylglyoximate precipitate formed produces a viscous slurry. A high stirring rate is required to avoid titration noise. A start volume of approximately 40 mL assists in reducing viscosity to tolerable levels.*
- *The red nickel dimethylglyoximate quickly stains the titration assembly. This may be cleaned by a dip rinse in 10% w/v H₂SO₄ solution*

Example: Acidic hydrometallurgical leach liquor containing Fe(II), Fe(III), Mg, Al, Mn, Cr, Cu, Co and Ca.

	6.03 ± 0.06 g/L (n=5), expressed as Ni
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Calculations:

Ni, g/L =	$\frac{((EP \text{ vol.}, mL - \text{Blank}, mL) \times c(\text{Na}_2 \text{DMG}) \text{ mol/L} \times AW \text{ Ni})}{(\text{Sample vol.}, mL \times 2)}$
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Titration Plots:

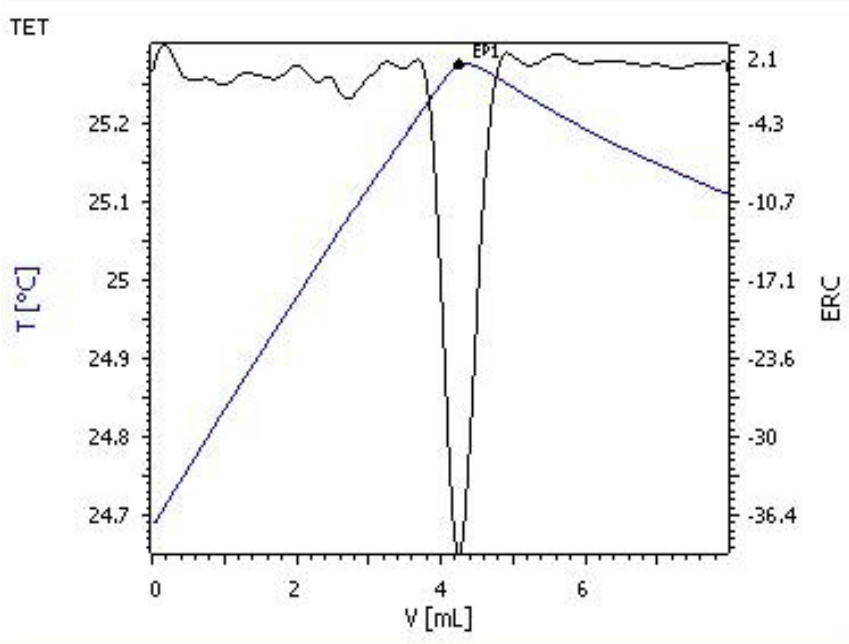


Fig. 1. Titration of standard Ni solution.

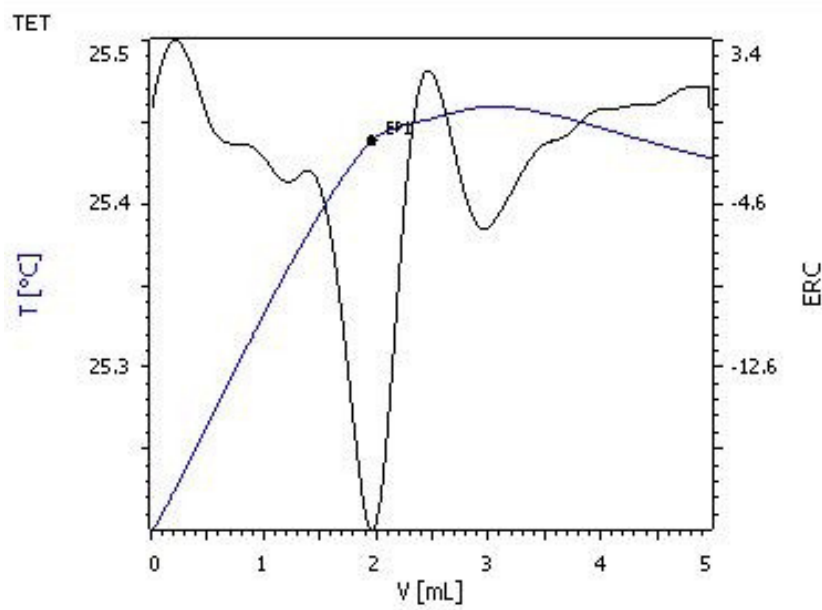


Fig. 2. Titration of sample solution

*Legend:
 Blue curve = solution temperatur
 Black curve = second derivative (ERC)*