Thermo. Titr. Application Note No. H-023

Title:	**Determination of Nickel by Dimethylglyoxime Titration**

***Scope*:**	Determination of nickel in the absence of cobalt and other interferences.

***Principle*:**	Titration of Ni(II) with standard sodium dimethylglyoximate (DMG) solution in buffered ammonia solution to an exothermic endpoint. Two moles of DMG react with one mole of Ni(II). Acidic Ni solutions should be complexed with citrate prior to basification.

Reagents:	Titrant: 0.5 mol/L disodium dimethylglyoximate. Dissolve 153.6 g disodium dimethylglyoximate (99% pure, FW = 304.21) in deionized water and make to 1000mL in a volumetric flask. Standardize against a solution prepared from pure metallic nickel. NH_3/NH_4Cl buffer: Dissolve 17.5 g A.R. NH_4Cl in 172 mL A.R. conc. NH_3 soln. Make to 250 mL with deionized water.

Method:	Basic Experimental Parameters:	
	Data rate (per second)	20
	Titrant delivery rate (mL/min.)	2
	No. of exothermic endpoints	1
	Data smoothing factor	50
	Procedure: Pipette an aliquot of solution containing approximately 50-70 mg Ni(II) [for example, 25mL of an approximately 0.05 mol/L Ni(II) solution] into a 140 titration beaker equipped with a spinring. Add 2mL NH_3/NH_4Cl buffer solution, and titrate to a single exothermic endpoint with 0.5 mol/L disodium dimethylglyoximate. This exercise was carried out on an approximately 0.05 mol/L Ni(II) solution prepared from A.R. $NiSO_4.6H_2O$, FW = 262.86, minimum assay = 98%. 6.5853g was dissolved and made to volume in a 500mL volumetric flask with deionized water. A 25 mL aliquot was calculated to contain 0.3293 g of the salt.	

For solutions containing Fe(III), Al(III) or Cr(IV), add 5mL saturated potassium sodium tartrate solution prior to titration to complex these cations and prevent interference.

Equipment stained by the Ni dimethylglyoximate precipitate may be cleaned by soaking in a solution of a strong mineral acid; eg, 10% w/v sulfuric acid.

The thermistor may be cleaned periodically with a soft toothbrush.

Results:	Replicate analysis of nickel sulfate solution
	Mean = 22.29±0.02g/L Ni (n=10)

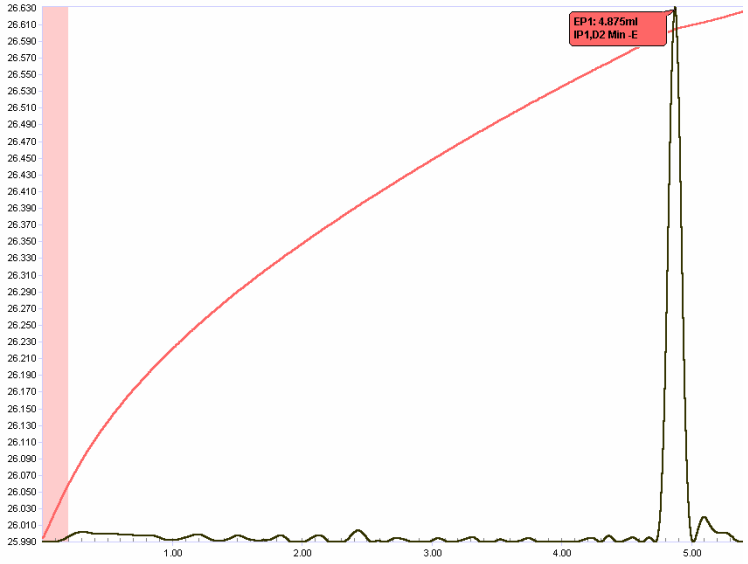
Calculation:

$$\%\, Ni, w/w = \frac{((titre, mL - blank, mL) \times M\ Na_2DMG \times FW\ Ni \times 100)}{(sample\ \ mass, g \times 2 \times 1000)}$$

EXAMPLE :

$$\%\, Ni, w/w = \frac{((4.873 - 0.058) \times 0.5194 \times 58.6934 \times 1000}{(0.3293 \times 2 \times 1000)} = 22.29\%$$

Thermometric Titration Plot:



Legend:

Red = solution temperature curve

Black = second derivative curve