

Thermo. Titr. Application Note No. H-049

Title: Determination of nickel by titration with disodium dimethylglyoximate

Scope: Determination of nickel in solution by titration with standard disodium dimethylglyoximate .

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 possibly containing Al(III) and Fe(III) should be complexed with tartrate prior to basification. Ideally, amounts of Ni to be titrated should be in the range 0.3-1 mmole.

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.

NH₃/NH₄Cl buffer: Dissolve 17.5 g A.R. NH₄Cl in 172 mL A.R. conc. NH₃ soln. Make to 250 mL with deionized water.

KNa tartrate solution, 450g/L.

Method:

Basic Experimental Parameters:

Titrant delivery rate (mL/min.)	2
No. of exothermic endpoints	1
Data smoothing factor	60
Stirrer speed	12*

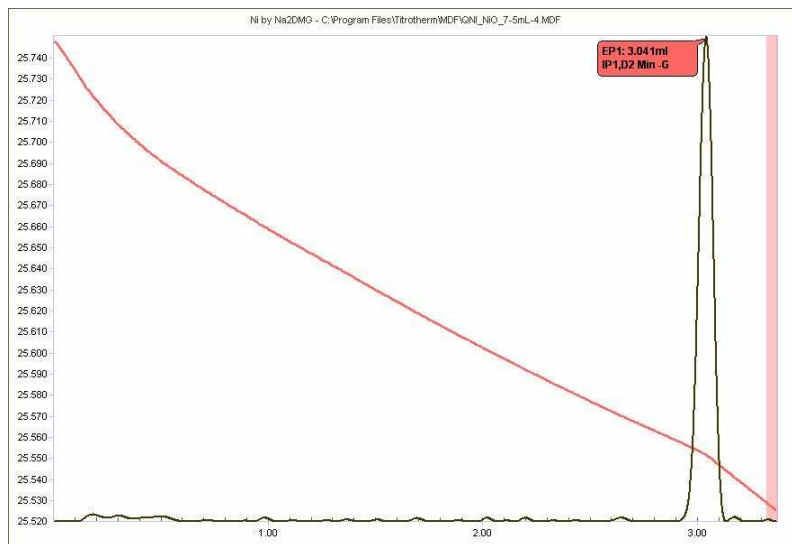
Procedure: The sodium dimethylglyoximate was dispensed from a 10mL Dosino. The KNa tartrate solution was also dispensed from a Dosino, as was the $\text{NH}_3/\text{NH}_4\text{Cl}$ buffer solution using the pre-dose function of the Titrotherm software.

The tartrate solution is used to prevent interference from Al(III) and Fe(III) impurities in samples containing Ni, and is added in the case of high purity Ni solution to “matrix match”. The aim is to have a solution volume of 40-45mL prior to the titration. Adjust the aliquot size to have an estimated Ni content of the solution in the range 0.3-1mmole Ni, and add DI water to make up the balance of the volume (including buffer and tartrate) of approximately 45mL. This higher than normal volume together with the high stirrer speed* reduces the detrimental effect that the highly viscous nickel dimethylglyoximate precipitate has on the endpoint.

Equipment stained by the Ni dimethylglyoximate precipitate may be cleaned by soaking in a solution of a strong mineral acid; eg, 1 mol/L HCl. During the analytical exercise reported here, the titration assembly was cleaned between each titration, and rinsed thoroughly with DI water.

Results:	Titration of high purity Ni solution dispensed by Dosino. Each aliquot was equivalent to 0.06372g of original sample.
	% Ni = 78.44±0.09 (n=12)

Thermometric Titration Plot:



Legend:

Red = solution temperature curve

Black = second derivative curve

Note that the solution temperature falls during the determination. Prior to the titration, an exothermic reaction (neutralization) occurs between the NH_3 in the $\text{NH}_3/\text{NH}_4\text{Cl}$ buffer and the mineral acids in the standard Ni solution. The exothermic formation of nickel dimethylglyoximate occurs against a background of falling solution temperature as a result of cooling.