The Gravimetric Estimation of Nickel:

The nickel is precipitated as nickel dimethyl glyoxime by adding alcoholic solution of dimethyl glyoxime $C_4H_6(NOH)_2$ and then adding a slight excess of aqueous ammonia solution.

NiSO₄ + 2 C₄H₈O₂N₂ \rightarrow Ni(C₄H₇O₂N₂)₂ + H₂SO₄

When the pH is buffered in the range of 5 to 9, the formation of the red chelate occurs quantitatively in a solution. The chelation reaction occurs due to donation of the electron pairs on the four nitrogen atoms, not by electrons on the oxygen atoms. The reaction is performed in a solution buffered by either an ammonia or citrate buffer to prevent the pH of the solution from falling below 5. If the pH does become too low the equilibrium of the above reaction favors the formation of the nickel (II) ion, causing the dissolution of Ni (DMG)₂ back into the mother liquor.

A slight excess of the reagent has no action on the precipitate, but a large excess should be avoided because of the possible precipitation of the reagent itself. The precipitate is soluble in the free mineral acids. It is therefore crucial to avoid the addition of too large and excess of the reagent because it may crystallize out with the chelate. It is also important to know that the complex itself is slightly soluble to some extent in alcoholic solutions. By adding small amount of chelating agents will minimize the errors from these sources. The amount of the reagent added is also governed by the presence of other metals such as cobalt, which form soluble complexes with the reagent. If a high quantity of these ions is present, a greater amount of DMG must be added. The nickel dimethylglyoximate is a very bulky precipitate. Therefore, the sample weight used in the analysis must be carefully controlled to allow more convenient handling of the precipitate during the transfer to the filtering crucible. The compactness of the precipitate is improved by adjusting the pH to 3 or 4, followed by the addition of ammonia solution.

A slow increase in the concentration of ammonia in the solution causes a slight increase in the pH gradually and results in the precipitation of the complex. The result is the formation of a denser precipitate. Once the filtrate has been collected and dried, the nickel content of the solution is calculated stoichiometrically from the weight of the precipitate.

The structure of DMG & the complex with nickel ions is given below;





DMG	но N OH CH ₃ OH 1% dimethylglyoxime and 10% ammonium hydroxide in 75% ethanol
Nickel solution	Ni ²⁴⁴
Ni-DMG Complex	
Funnel and flask	



Ref: http://amrita.vlab.co.in/?sub=2&brch=193&sim=348&cnt=1

MW of dimethylglyoxime: 116.12 g/mol

NiDMG2 complex MW: 288.9155 ± 0.0002 g/mol

Nickel MW: 58.6934 g/mol