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Analysis of Nickel in a Ni-plated Materials for Corrosion Protection by Colorimetry

Expt No. 07

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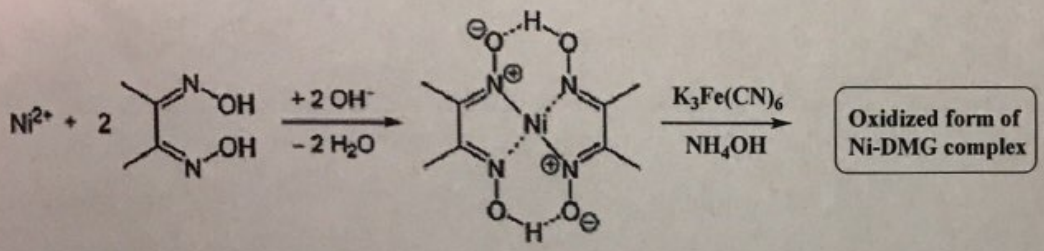
Principle: Dimethylglyoxime reacts with nickel ions and forms a pink coloured $Ni(dmg)_2$ complex in alkaline medium. Nickel forms a brown-red, water soluble oxidized nickel-dimethylglyoxime complex with potassium ferricyanide in alkaline medium as shown in the scheme below. The absorption spectrum of the oxidized complex shows absorption maximum at a wavelength of 440 nm (Figure 1). After drawing calibration graph the determination of nickel concentration in a given unknown sample can be done by colorimetry applying Beer-Lambert law.



Scheme

OBSERVATION AND CALCULATIONS

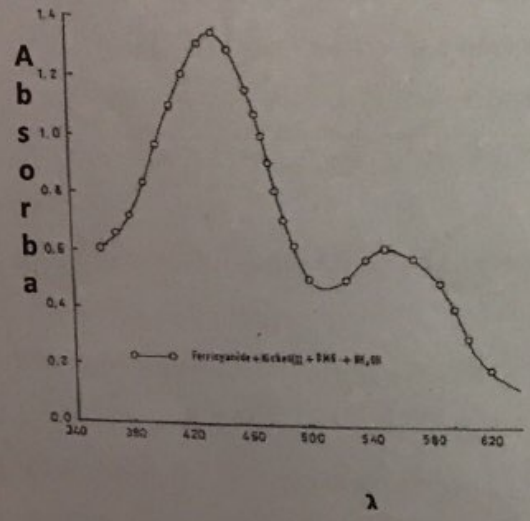


Figure 1: Absorption spectrum of oxidized Ni(II)-DMG complex showing λ_{max} at 440 nm

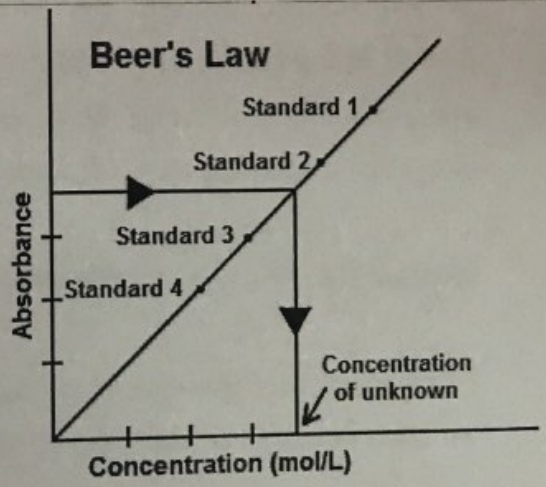


Figure 2: Model calibration carve for Ni(II) determination

27

Table-1: Experimental Data

S. No	Concentration (mg/L) (X axis)	Absorbance (Y axis)
1.	2	0.224
2.	4	0.445
3.	6	0.630
4.	8	0.816
5.		
6.	Unknown	0.547

Requirements:

Solutions required

1. Steel sample containing Nickel(II) solution – Prepare a standard nickel(II) solution by weighing 450 mg of $NiSO_4.6H_2O$. Quantitatively transfer the weighed sample to a 100 mL volumetric flask and dissolve the salt in distilled water. Finally make the volume to 100 mL with distilled water. This solution contains 1000 mg of Ni per liter (1000 ppm). For calibration graph make a working stock solution (100 ppm) by diluting 10 ml of 1000 ppm solution to 100 ml.

2. **Potassium Ferricyanide solution** – Dissolve 1.5 g of $K_3Fe(CN)_6$ in a 100 mL volumetric flask with distilled water.

3. **Dimethylglyoxime in NaOH solution** – Take 2.0 g of dimethylglyoxime and 8 g of NaOH in a 100 mL volumetric flask and dissolve the salts with 25 mL distilled water and make the volume to 100 mL with distilled water.

4. **Sodium Hydroxide solution** – Dissolve 40 g of NaOH in 1000 mL of distilled water.

Procedure:

Take 6 standard 100 mL volumetric flasks (including the unknown). To every flask add 1.00 mL of dimethylglyoxime solution by a pipette followed by 1,00 mL of potassium ferricyanide solution by a burette. Then add 1.00, 2.00, 3.00, 4.00, and 5.00 mL of the working stock solution (100 ppm)to get 1.0, 2.0, 3.0, 4.0 and 5.0 ppm solution respectively of steel

containing nickel(II) solutionfrom a burette to the flask. All flasks are shaken well and made up to 100 mL mark with 1N NaOH solution. Allow the flasks at least for 15 minutes after the addition of all the reagents for the complete complex formation. Then the absorbance of the formed brown-red solution is measured at 440 nm against the distilled water (blank). Record these absorbance readings in Table-1. Draw a calibration graph taking concentration of nickel(II) as X-axis and absorbance readings as Y-axis and a straight line that passes through the origin (see Figure 2). From the molar concentration of nickel solution and cell path length, calculate the molar absorptivity of the oxidized nickel-dimethylglyoxime complex at 440 nm. From the calibration plot, measure the concentration of nickel in the unknown sample.

Result

Concentration of nickel in the steel sample = 5 ppm (mg/L)

Evaluation of result

Sample number	Experimental value	Actual Value	Percentage of error	Marks awarded
				(8/8) 8/2/18