Material Analysis: Nickel in Nickel-plated Component by Colorimetry

1. Importance of the experiment:

Nickel is a transition element and commonly exists in +2 oxidation state, though +1, +3 and +4 states are alsoobserved in Nickel complexes.Nickel plays an important role in biological systems as a constituent of several enzymes. Ni is also present in soils and plants, and its concentration varies widely from trace quantities to being a major constituent. Therefore, determination of nickel at different concentration levels in variety of samples becomes very important.

2. Nickel Toxicity: Compared with other transition metals, Nickel is a moderately toxic element. However, it is known that inhalation of nickel and its compounds can lead to serious problems, including cancer in the respiratory system. Moreover, Nickel can cause a skin disorder known as nickel-eczema(10.1016/j.kijoms.2016.08.003).

3. Nickel in Industries:A thin layer of nickel onto a metal object can be decorative, provide corrosion resistance, wear resistance, or used to build up worn or undersized parts for salvage purposes.Nickel alloys are used extensively because of their corrosion resistance, high temperature strength and special magnetic and thermal expansion properties.

The major alloy types that are used are:

- Iron-Nickel-Chromium alloys
- Stainless Steels
- Copper-Nickel alloys and Nickel-Copper alloys
- Nickel-Chromium Alloys
- Nickel-Chromium-Iron alloys
- Low Expansion Alloys
- Magnetic Alloys (http://www.nickel-alloys.net/nickelalloys.html)

Expt. No.: 2 **Date:** 08/09/2018

Experiment	Material Analysis: Nickel in Nickel-plated Component by Colorimetry
Problem definition	Corrosion protection in steel depends on the amount of Ni (acts as passivating metal) in its composition. Hence, it is important to analyze the amount of Ni in steel for its use in industry.
Methodology	Ni-DMG forms a stable colored complex, and its intensity depends on the concentration of Ni present in solution. Colorimetric method is used to measure the absorbance of the colored complex solution.
Solution	Estimation of Ni concentration in the unknown sample from the calibration graph plotted based on different known Ni concentrations.
Student learning outcomes	Students will learn to a) perform colorimetric method b) analyze the composition of Ni in different grades of steel

Principle:

Dimethylglyoxime (DMG) reacts with nickel ions and forms a pink-colored $Ni(dmg)_2$ complex in alkaline medium. It gets oxidized by potassium ferricyanide ($K_3[Fe(CN)_6]$) in alkaline medium to form a brown-red, water soluble oxidized $Ni(dmg)_2$ complex as shown in the Scheme 1. Absorption spectrum of the oxidized complex shows absorption maxima at a wavelength of 440 nm (Fig. 1). Concentration of Ni in the given unknown sample is determined from the calibration graph as shown in Fig. 2.

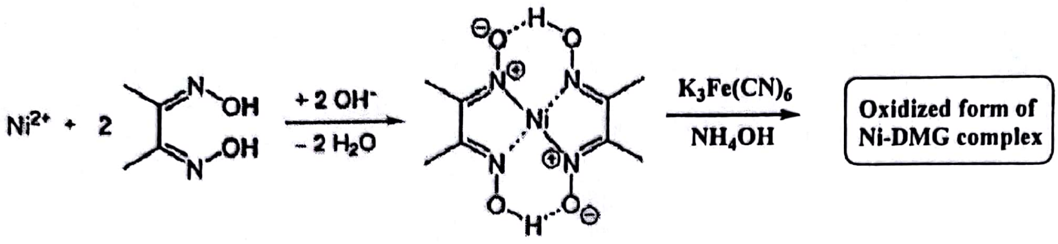
Requirements:

Reagents and solutions: $NiSO_4$ (100 ppm), NaOH (1 N) solution, DMG, $K_3[Fe(CN)_6]$

Apparatus: Colorimetry, Volumetric flasks, Beakers.

Procedure: Take 6 standard 100 mL volumetric flasks (including the unknown). To every flask add 1 mL of DMG solution followed by 1 mL of $K_3[Fe(CN)_6]$ solution using a burette. Further, add 1, 2, 3, 4 and 5 mL of the working Ni stock solution (100 ppm)from a burette toget 1, 2, 3, 4 and 5 ppm of steel containing nickel(II) solutions. All the flasks are shaken well and made up to 100 mL mark with 1N NaOH solution. Allow the flasks at least 15 minutes after adding all the reagents for the complete complex formation. Then the absorbance of the formed brown-red solution is measured at 440 nm against distilled water (blank). Record these absorbance readings in Table 1. Draw a calibration graph taking concentration of Ni^{2+} (in ppm) as X-axis and absorbance readings as Y-axis. A straight line that passes through the origin (see Fig. 2) is an indication that the measured data obeys Beer's

Law. From the calibration plot, measure the concentration of nickel in the given unknown sample.



Scheme - 1

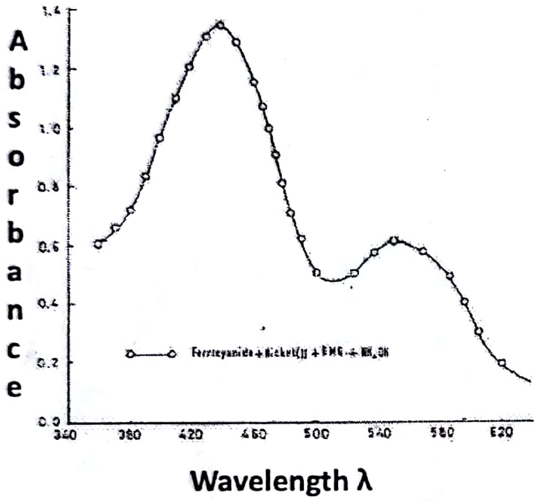


Fig. 1: Absorption spectrum of oxidized Ni(II)-DMG complex showing λ_{max} at 440 nm

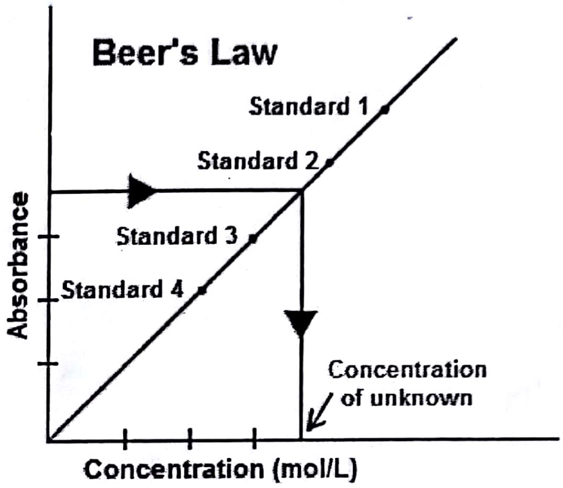


Fig. 2: Model calibration curve for Ni(II) determination

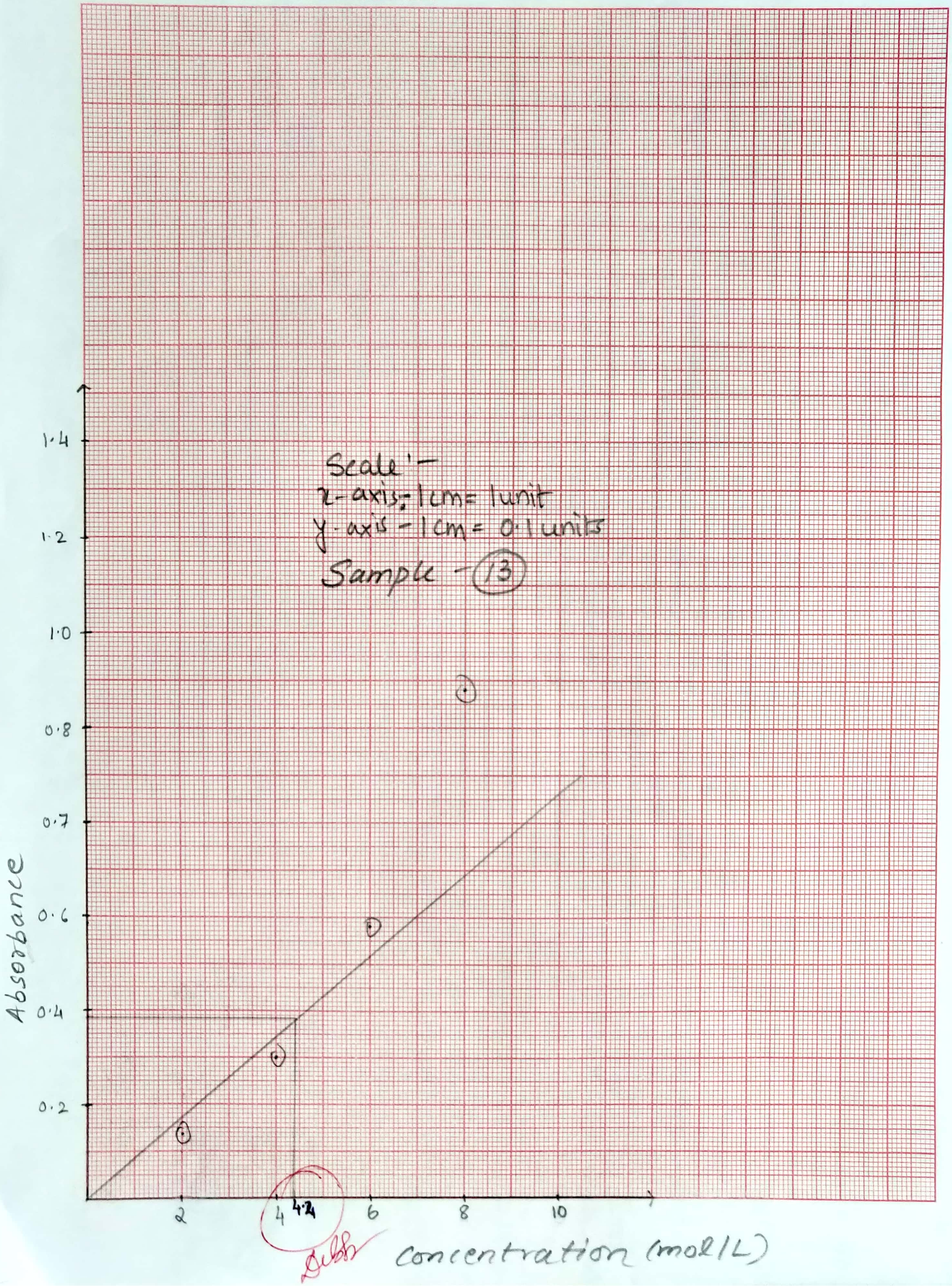
Table 1: Experimental Data

S. No.	Concentration (ppm) (X-axis)	Absorbance (Y-axis)
1.	2	0·137
2.	4	0·300
3.	6	0·582
4.	8	0·874
5.		
6.	Unknown	0·385

Result: Concentration of nickel in the steel sample = 4.4 ppm (mg/L)

Evaluation of Result:

Sample number	Experimental value	Actual Value	Percentage of error	Marks awarded
13				


Scale :-
x-axis:- 1cm = 1unit
y-axis - 1cm = 0.1 units
Sample - 13
Absorbance
1.4
1.2
1.0
0.8
0.7
0.6
0.4
0.2
2
4
4.2
6
8
10
concentration (mol/L)