



## Spectrophotometric Determination of Nickel (II) using 2-Acetylpyridine semicarbazone (APS)

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**Abstract** For the determination of Nickel (II), a straight forward, sensitive, and accurate spectrophotometric approach has been created. The reagent 2-Acetylpyridine semicarbazone (APS) react with nickel (II) in aqueous solution in the pH range 0.5-12. In the pH range of 8.0 to 10.0, 2-Acetylpyridine semicarbazone (APS) interacts with nickel (II) to produce a light yellow tinted complex. At 350 nm, the compound has the highest absorption. The pH of the buffer (ammonium chloride - ammonium hydroxide) solution was selected because the complex exhibited a maximum and stable absorbance in the range of 8-10. The present approach successfully determines the presence of nickel in alloy steels, aluminum-based alloys, vegetable oil, and water.

**Keywords** Nickel (II), UV-VIS Spectrophotometer, 2-Acetylpyridine semicarbazone (APS)

### Introduction

The metal nickel is crucial for both industrial and biological purposes. Together with cobalt, copper, zinc, and manganese, it is one of the crucial trace elements needed in the human diet [1-4]. Nutritional phenomena are directly influenced by nickel and cobalt. Ni, often known as nickel, is a crucial trace element that the human body needs in extremely minute levels to operate properly [7-8].

Among Ni's crucial functions in human physiology are the following:

- Enzymatic activities: Ni affects the activity of various enzymes in the body, such as urease, which aids in the urea breakdown process and is involved in amino acid metabolism.
- DNA synthesis: Ni contributes to the synthesis of DNA and is necessary for the preservation of DNA's structural integrity.
- Immune system: Ni is necessary for the immune system to operate correctly and is involved in the development of specific types of white blood cells that aid in the fight against infection.
- Bone Development: Ni is involved in calcium metabolism and may be important for supporting bone health [9-10].

It's important to note, however, that excessive amounts of Ni can be toxic and can lead to various health problems. Therefore, it's important to maintain a balanced intake of Ni through a healthy and varied diet [11-13].

While only being needed in extremely minute amounts, nickel, often known as Ni, is a crucial micronutrient for the growth and development of plants. In plants, Ni is essential for a number of physiological activities, including:

- Enzymatic functions: Ni is a part of various enzymes in plants, including hydrogenase, which is involved in nitrogen fixation, and urease, which is involved in the metabolism of urea.



- Photosynthesis: Ni contributes to photosynthesis' effectiveness, the process by which plants turn light energy into chemical energy.
- Plant defense: Ni is involved in how plants fight against infections and pests. It contributes to the creation of certain chemicals that shield plants from biotic stress.
- Seed germination: Ni aids in the growth of seedlings and the germination of seeds.
- Nutrient uptake: Iron and nitrogen are two more crucial elements that Ni can help the body absorb and use. [14-15].

Ni shortage affects plants often despite its significance, particularly in crops like rice and wheat. Reduced growth and productivity, as well as increased vulnerability to disease and pests, can all result from a Ni deficit. Nevertheless, too much Ni may be hazardous to plants and cause a number of symptoms, such as leaf necrosis, slowed development, and decreased yield. As a result, it's critical to maintain a suitable balance of Ni in soils and plant development medium [16-20].

One of the crucial alloying components for steel and cast iron is nickel. A review of the literature revealed that numerous spectrophotometric techniques utilising different chromogenic reagents were published for the detection of nickel (II) [21-30].

The present study describes, a simple, rapid, selective and sensitive direct spectrophotometric methods for the determination of trace amount of nickel (II) by complexing with 2-Acetylpyridine semicarbazone (APS).

## Experimental Work

### Preparation of Inorganic Salt Solutions

#### Nickel (II) solution

Stock solution of Ni (II) ( $1 \times 10^{-2}M$ ) was prepared ( $NiSO_4 \cdot 7H_2O$ ) with double distilled water containing few drops of concentrated  $H_2SO_4$  and made up to 100ml. The stock solution was standardized gravimetrically.

### Preparation of Buffer Solutions

The conventional techniques described in the literature were used to create the buffer solutions. The following solutions were used to make buffers.

pH	Constituents
0.5-3.0	1M Sodium acetate + 0.1M hydrochloric acid
3.5 -6.0	0.2M Sodium acetate + 0.2M acetic acid
6.5 - 7.5	1M Sodium acetate + 0.2M acetic acid
8.0-12.0	2M Ammonia + 2M ammonium chloride

### Details of Instruments Employed in the Present Investigations

#### UV-visible double beam recording spectrophotometer

The present investigation made use of a UV-160A double beam spectrophotometer. Shimadzu Corporation in Japan created it. The following are some of its most notable characteristics:

1. High-speed wavelength scanning is achieved by employing a CPU-controlled scanning device without the use of a sine bar.
2. All-in-one corporate spectrophotometer with CRT and printer.
3. Single-action operation is enabled by providing backup mode settings.
4. Ease of data processing because the received spectrum is available simply talking to CRO.

**Table 1:** Specifications of UV 160a Spectrophotometer

Measuring wavelength range	200-1100 nm
Spectral band width	2 nm
Wavelength readability	0.1 nm increment
Wavelength scanning speed	Monochromator setting speed is nearly - 3600 nm/min Fast nearly 2400 nm/min



Wavelength accuracy	Medium nearly 1500 nm/min Slow nearly 480 nm/min ± 0.5 nm with automatic wavelength correction
Light source switching	Automatic wavelength change is possible between 295 and 364 nm.
Photometric system	Double beam system
Recording system	Printout of measured data and calculated results
Multicomponent	Samples from various sources are included. Mixed samples can be used as standards to determine up to eight different components. Up to sixteen (16) standards' worth of data can be kept in the backup memory.
Light sources	Halogen lamp, 50 W, 2000-hour life span, socket type, deuterium lamp, automated sensitivity control, monochromator, monochromator.
Mono chromator	Aberration - corrected concave holographic grating with $f = 4.2$
Detector	A matched pair of silicon photodiode
Recorder	Computer controlled thermal graphic printer
CRT	9-inch with graphic function 240 x 320.
Sample compartmen	Inner size: 1100 mm wide
Distance between sample and reference beam	100 mm
Power requirements	With line voltage selector for 100
Weight	42 kgs

### ELICO digital pH meter

The pH of buffer solutions is measured with an ELICO digital pH meter (Model LI 610), made by M/s ELICO private limited, Hyderabad, India. The devices are equipped with a system to automatically adjust for changes in temperature. The reproducibility of measurements is within  $\pm 0.01$  pH.

### Instruments used in the characterization of ligands

#### Infrared spectrophotometer

Using a Perkin-Elmer 983G infrared double beam spectrophotometer, we measured the ligand's infrared spectra, which ranged from 400 to 4000  $\text{cm}^{-1}$ . Anhydrous conditions were used to form a thin pellet from 10 mg of finely powdered material, which was then fully mixed with spectral grade KBr. IR spectrum of ligands were recorded using this device

#### Absorption spectra of reagent solutions and metal complexes

It was necessary to make up to the specified concentration of dimethyl formamide (DMF) in a 25-ml volumetric flask using an aliquot of reagent (typically 1 ml of  $1 \times 10^{-2}\text{M}$ ) solution, and this was done by adding distilled water to a 10 ml volumetric flask containing buffer solution. The reagent solution's absorbance was compared to a water blank. The absorbance versus wavelength relationship was shown using a graph.

In order to determine the absorption spectra of a complex (metal + reagent), the following approach was used. Metal complexes were produced in a 25 ml standard flask by adding 10 ml of buffer, acceptable amounts (1 ml or 2.5 ml) of DMF and the appropriate concentration of metal ions to the flask. It was decided to compare the absorbance of the complexes against a reagent blank that had been made identically. It was decided on the analytical wavelength based on an absorbance vs. wavelength plot.



### Effect of pH on the absorbance of the metal complexes

To see if pH has an impact on colour intensity, a metal complex in solution was created using buffer solutions with varying pH values.

One set of standard flasks was filled with 10 ml of buffer solution, 10 ml of metal-ion-reagent solution, and one or two millilitres of dimethylformamide (DMF). The absorbance of each solution (metal complex) was compared to a comparable reagent blank that had been produced according to a predetermined wavelength ( $X_{\max}$ ). After analysing the absorbance versus pH data, the decision was reached on what pH to use.

### Effect of reagent concentration on the absorbance

To determine the optimal reagent concentration for complete colour development, the following technique was followed. Every flask included 10 millilitres of buffer solutions as well as the appropriate volume of DMF and a fixed quantity of metal ion. This method yielded the same results each time since each flask had the same aliquot of reagent solution every time. In each flask, distilled water was used to dilute the contents to volume before measuring absorbance at a specific wavelength ( $X_{\max}$ ) against a reagent blank generated in the same way. This experiment determined the needed reagent molar excess for complete colour development.

### Effect of time on the absorbance of reaction mixture and stability

There were 10 ml of buffer solution, metal ion and reagent and DMF added to a 25-ml calibrated flask, then the solution was diluted up to the mark with distilled water. The colour 56 complex solution's absorbance was compared to a reagent blank generated in the same way but over a longer period of time. This experiment established the stability of the compound and the time elapsed until the entire spectrum

## Results and Discussion

In the pH range of 8.0 to 10.0, **2-Acetylpyridine semicarbazone** (APS) interacts with nickel (II) to produce a light yellow tinted complex. The colour reaction was studied in order to create a spectrophotometric technique for measuring nickel (II) in an aqueous media.

### Absorption spectra of APS and its nickel complex in aqueous solutions

Following the techniques outlined in the research, "the absorbance of the complex in solution was measured at pH 8.5 against a reagent blank in the wavelength range of 250 to 600 nm. a. The spectra are shown in Figure 1. At 350 nm, the compound has the highest absorption. As a result, all further research was conducted at this wavelength."

### Effect at pH on the absorbance of the Ni (II) - APS complex

"Buffer solutions were prepared with different pH values, which allowed researchers to examine the effect of the pH on colour intensity. Figure depicts the link between pH and absorbance. The pH of the buffer (ammonium chloride - ammonium hydroxide) solution was selected because the complex exhibited a maximum and stable absorbance in the range of 8-10."

### Effect of reagent concentration on the absorbance of the complex

With the approach described in the study, we looked at the impact of the reagent concentration on complex absorbance at 350 nm. In order to achieve full colour development, researchers found that just a five-fold molar excess of the reagent was necessary.



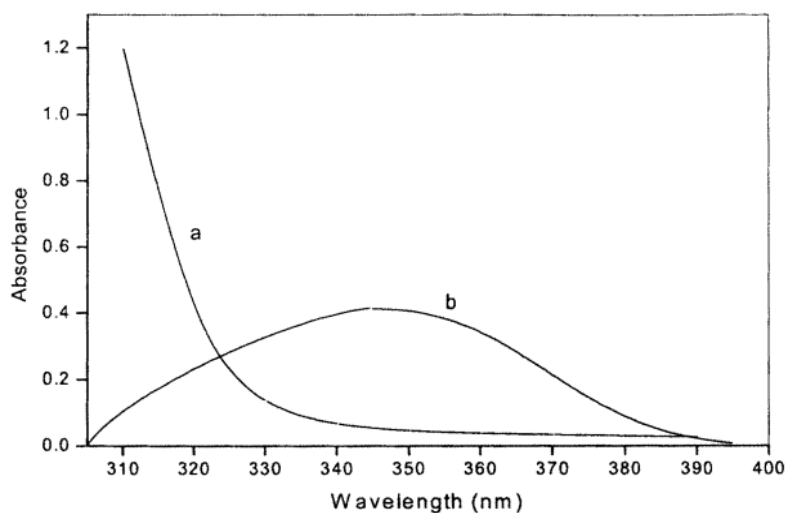


Figure 1: Absorbance spectrum of a) APS Vs Water blank b) Ni (II) - APS complex Vs APS solution ( $[Ni(II)] = 1.6 \times 10^{-5} M$ ,  $[APS] = 4 \times 10^{-4} M$ ,  $pH = 9.0$ )

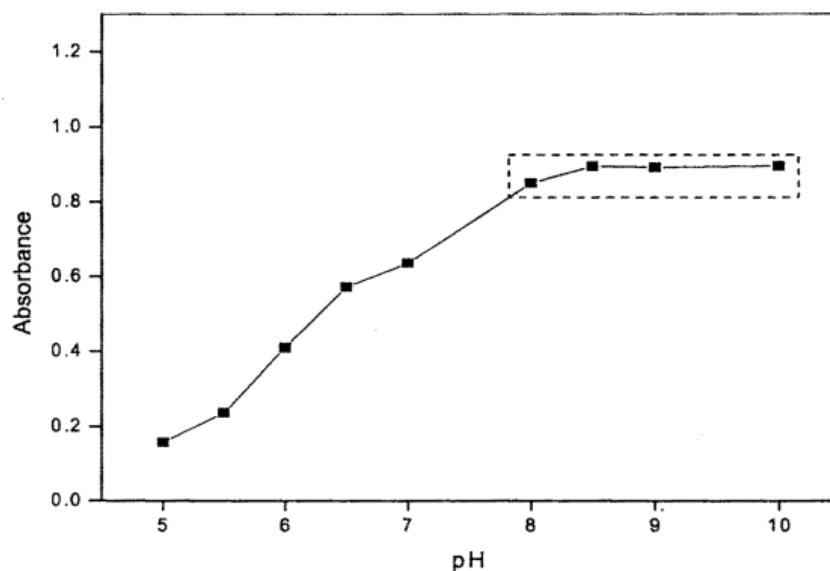


Figure 2: Effect of pH on the absorbance of Ni (II) - APS system ( $[Ni(II)] = 1.6 \times 10^{-5} M$ ,  $[APS] = 4 \times 10^{-4} M$ , Wavelength = 350 nm)

#### Effect of time on the absorbance of the complex

“At 350 nm, the nickel (II) complex's absorbance was measured at different time points over a period of several minutes. There is a near-instantaneous colour interaction between APS and nickel (II), which results in a constant absorbance for 75 minutes, according to the results.”

#### Order of addition of constituents on the absorbance of the complex

The absorbance of the nickel (II) - APS complex in solution is unaffected by the sequence in which the elements (buffer, nickel (II), DMF, and APS) are added to the solution.



### Applicability of Beer's law

“The applicability of Beer's law to the existing system was explored using the research approach. In the range of 0.094 - 0.940 g/ml, the calibration graph shows that the system follows Beer's law (II). The straight line is governed by the equation  $A_{350} = 0.4682C + 0.0308$ .  $2.81 \times 10^4 \text{ Lit Mol}^{-1} \text{ cm}^{-1}$  Ni (II) Absorbance and Sandell's sensitivity for Ni (II) are  $0.021 \mu\text{g}/\text{cm}^2$  for Ni (II). There is a  $0.479\text{-ml g}^{-1} \text{ cm}^{-1}$  specific absorptivity for the system in question. There is a 0.0144 standard deviation in the  $0.47 \mu\text{g}/\text{ml}$  Ni (II) measurement. Relative standard deviation and mean absorbance are 1.02 percent and 0.642-20.0024 percent, respectively, for a sample of 10 measurements.”

### Tolerance limits of foreign ions

To test the reagent's selectivity, the impact of different foreign ions was studied using the technique described in study. The goal of this experiment was to determine the tolerance limits for difference.

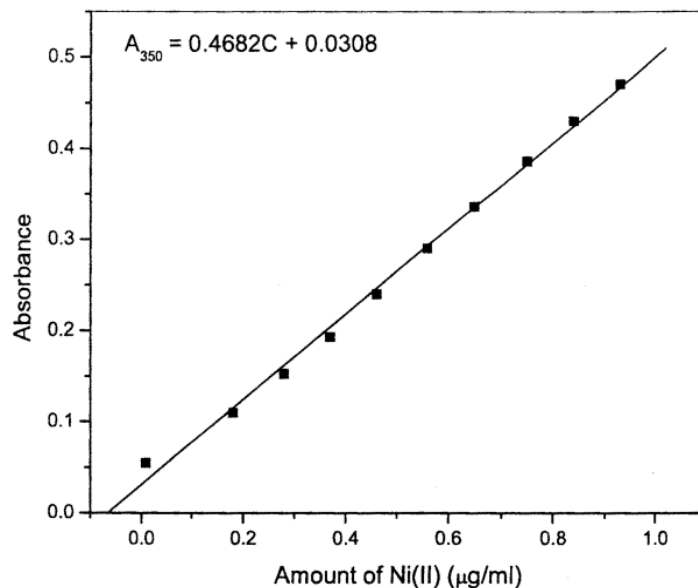


Figure 3: Absorbance Vs Amount of Ni (II) (pH = 9.0, [APS] =  $6 \times 10^4 \text{M}$ , Wavelength = 350 nm)

Ions that form a chain The amount of foreign ion that results in a 2% error in absorbance was selected as a tolerance limit. Reference samples were also prepared in accordance with research guidelines. Table shows the statistics on foreign ion tolerance levels. Table shows that when there are a large number of cations and anions, they do not interact. On the other hand, the tolerance limit values for metal ions that are connected to each other are lower.

### Composition and stability constant of the Ni (II) - APS complex

“Job's continuous variation and molar ratio method was used to determine the nickel (II) - APS complex's composition. An analysis was performed using the data from Job's plot in order to calculate the stability constant of nickel (IV)-APS.”

### Job's continuous variation method

“Job's methods are being examined. According to Job's plot, three moles of reagent react with one mole of metal ion. Consequently, the ratio is one to three. The complex's stability constant was calculated using the following equation, as the complex's composition is 1:3 (M ; L)”.

$$\beta = \frac{1 - \alpha}{27\alpha^4 C^3} \quad (1)$$

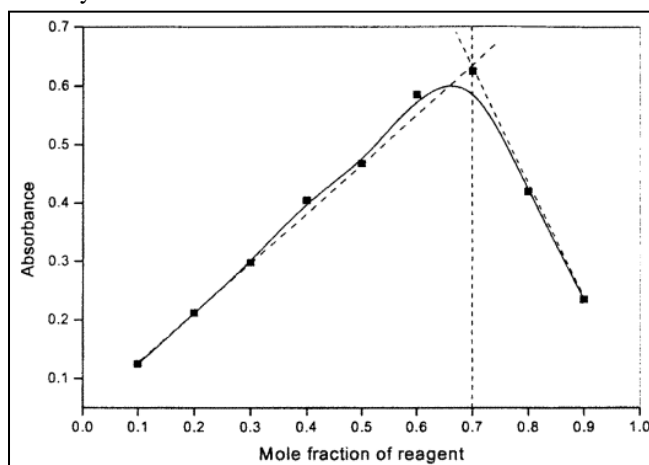
There is a correlation between Job's plot and the values (0.07131)  $\alpha$  and C ( $5.6 \times 10^{-6}$ ).  $1.3 \times 10^{14}$  is determined to be the stability constant.

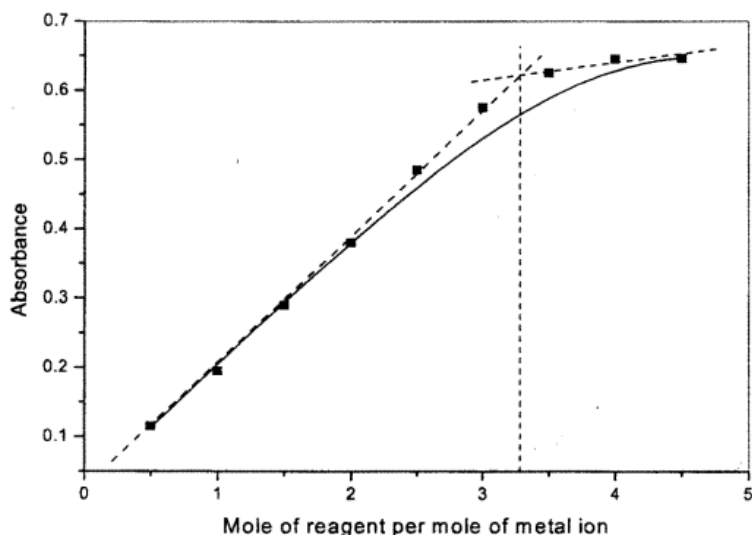


**Table 2:** Tolerance limit of foreign ions in the determination of 0.938 µg/ml of nickel (II)

Ion added	Tolerance limit (µg/ml)	Ion added	Tolerance limit (µg/ml)
Thiocyanate	720	Ba (II)	33
Cynate	702	Cd (II)	27
Iodate	507	Sr (II)	21
Citrate	461	Mg(II)	15
Thiosulphate	455	Au(III)	10.25
Iodide	411	Pt(II)	10.25
Tartarate	355	Tl(II)	10.25
Bicarbonate	288	Ca (II)	10
Nitrate	248	Pb (II)	8.2
Carbonate	240	Hg (II)	4.8
Phosphate	232	Sn (II)	4.7
Sulphate	230	Zn (II)	3.6
Ascorbate	217	Pd(II)	2.6
Oxalate	211	Cu (II)	2.5
Bromide	198	Mo (VI)	2.3
Thiourea	185	W (VI)	2.2
EDTA	148	Zn (II)	1.5
Chloride	141	Co (II)	1.4
Acetate	112	Mn (II)	1.3
Fluoride	75	Cr (VI)	1.2
		Ag (II)	1.2
		Cr (VI)	1.2
		Fe (II)	0.67
		Al (III)	0.6
		V (V)	0.6
		Fe (II)	0.6

- Masked with 70 µg/ml pf fluoride
- Masked with 140 µg/ml of chloride
- Masked with 700 µg/ml of thiocyanate

Figure 4: Job's curve of Ni (II) = APS =  $2 \times 10^{-4}$  M (stock solution) (Wavelength = 350 nm, pH = 9.0)



### Molar ratio method

The complex's composition was also established using the molar ratio approach, as detailed in the study.

1 ml of  $2 \times 10^{-4}$  M nickel (II) solution was added to 10 ml of buffer (pH 8.5) solution in a set of (ten) 25-ml standard flasks. Each flask received known aliquots (1-10 ml) of reagent ( $2 \times 10^{-4}$  M) solution. The absorbance was measured against a comparable reagent blank after the contents were plotted with distilled water. The complex's composition was determined via a molar ratio plot, which revealed a 1: 3 ratio (M: L). As a result, the molar ratio approach confirms the composition found by Job's method.

### Conclusion

**The present method is simple, inexpensive, and accurate. The present approach successfully determines the presence of nickel in alloy steels, aluminum-based alloys, vegetable oil, and water.**

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