



Synthesis of New 5-methylfuran-2-carboxaldehyde thiosemicarbazone (5-MFAT): Selective and Sensitive Spectrophotometric Determination of Co(II) in Vegetable, Soil, water and Alloy Samples

G. Sreenivasula Reddy and P. Raveendra Reddy*

Department of Chemistry, Sri Krishnadevaraya University, Anantapur, India-515003.

Received 26 Jan 2015; Revised 19 Mar 2015; Accepted 19 Mar 2015.

*Corresponding author: Email: raveendrareddy_sku@yahoo.com

Abstract

Cobalt (II) reacts with 5-methylfuran-2-carboxaldehyde thiosemicarbazone (5-MFAT) and forms a light yellow colored complex, which was extracted into isobutanol from sodium acetate and acetic acid buffer at pH 6.0. The absorbance value of the Co (II)–5-MFAT complex was measured at different intervals of time at 360 nm, to ascertain the time stability of the complex. The extraction of the complex into the solvent was instantaneous and stable for more than 24hrs. The system obeyed Beer's law in the concentration range of 0.236 -2.36 $\mu\text{g/ml}$ of cobalt (II), with an excellent linearity and a correlation coefficient of 0.999. The molar absorptivity and Sandell's sensitivity of the extracted species were found to be $1.86 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$ and $3.2 \times 10^{-3} \mu\text{g/cm}^2$ at 360 nm, respectively. Hence, a detailed study of the extraction of cobalt (II) with 5-MFAT has been undertaken with a view to developing a rapid selective, sensitive and extractive spectrophotometric method for the determination of cobalt (II) when present alone or in the presence of diverse ions which are usually associated with cobalt (II) in vegetable, soil, water and various standard alloy samples have been tested for the determination of cobalt (II) for the purpose of validation of the present method. The results of the proposed method are comparable with those from atomic absorption spectrometry and were found to be in good agreement.

Key words: Cobalt (II); Extractive spectrophotometry; 5-methylfuran-2-carboxaldehyde thiosemicarbazone; AAS.

1. Introduction:

Cobalt is an important micronutrient for biological organisms [1]. Its presence is of utmost importance for many organisms, including mammals, because it activates a number of enzymes [2]. Cobalt also plays an important role in the nitrogen fixation of plants by bacteria and is an integral part of vitamins B12. Plants and different diet compositions contain extremely low concentrations of cobalt. Its deficiency is exhibited by a retarded growth rate, loss of appetite and pernicious anemia in human beings [3]. Deficiency of cobalt causes a variety of diseases in animals [4-6]. Over-exposure to cobalt causes irritation of the gastro-intestinal tract, nausea, diarrhoea, and so on. At high concentrations, it inhibits hemi-biosynthesis and enzyme activities. Moreover, the element is toxic when taken directly. Apart from the physiological utility of cobalt, it also finds extensive use in industry. Due to its strength and high resistance to corrosion in many media, it is widely used in high-speed steel tools, magnets and high temperature alloys. The metal finds wide applications in the manufacture of aircraft, electromagnets, paints and ceramics. Cobalt is also used as a catalyst in industrial processes. Recent developments in the determination of cobalt have grown considerably, owing to its involvement in some essential metabolic and industrial processes, which are discussed above. Considering several methods for the determination of transition metal ions, at the micro level, the most frequently adopted methods include the use of analytical techniques, such as atomic absorption spectrometry (AAS), inductively coupled plasma atomic emission spectroscopy (ICP-AES), X-ray fluorescence spectroscopy, spectrophotometry and several other allied techniques. Among these, predominantly spectrophoto-

metric methods are preferred for determination of cobalt (II), as they are less expensive and possess a greater sensitivity in comparison. The main objective of studying its applications is to determine the trace amounts of cobalt (II) in vegetable, water, soil and other natural samples of significant importance.

Thiosemicarbazones are important sulphur and nitrogen bearing organic reagents. The metal chelates of these reagents find a wide range of applications in medicine [7-8] and agriculture [9]. The use of thiosemicarbazones in inorganic analysis has been reviewed [10-12] and the survey of literature reveals that only a few select thiosemicarbazones are employed for the extractive spectrophotometric determination of cobalt (II) [13-32]. The present analytical reagent 5-methylfuran-2-carboxaldehyde thiosemicarbazone (5-MFAT) is tested for the extractive spectrophotometric determination of cobalt (II), and the proposed method is successfully applied for the determination of cobalt (II) in real samples such as vegetable, soil, water, and standard alloy samples.

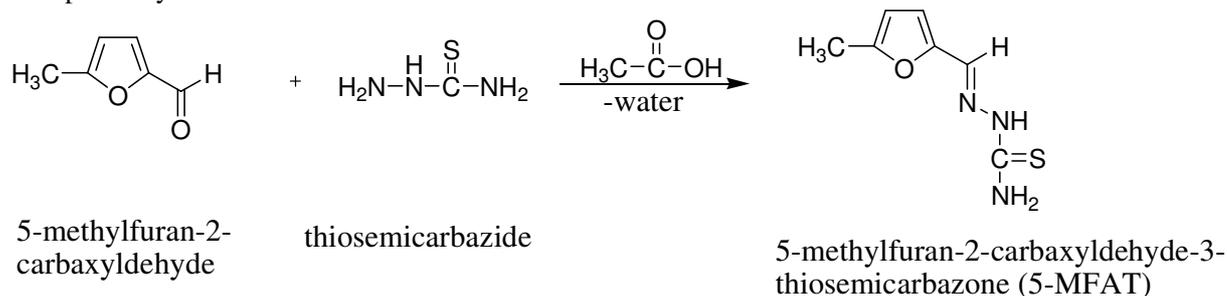
2. Materials and Methods

2.1. Apparatus

A Shimadzu 2450 UV-VIS spectrophotometer with 1.0 cm quartz cell is used for absorbance studies. An Elico LI-120 digital pH meter is used for pH adjustment. A Perkin-Elmer 170-30 atomic absorption spectrometer is used for the comparison of results. A Nicolet FT-IR 560 Magna spectrometer using KBr was used to obtain the infrared spectra of the compound (5-MFAT). The Bruker 300MHz NMR spectrometer was used to obtain the ¹H-NMR spectra of the ligand. The Micro Mass VG 7070 H Mass spectrometer was used to obtain the mass of the 5-MFAT.

2.2. Reagents and chemicals

All reagents used conform to analytical reagent grade, unless otherwise stated. 5-methylfuran-2-carboxaldehyde thiosemicarbazone (5-MFAT) is prepared employing the following the procedure. (2.0 ml of 0.0050 mol) of 5-methylfuran is dissolved in 10 ml of absolute ethanol and mixed in a round bottomed flask with (0.458 gms of 0.0050 mol) of thiosemicarbazide dissolved in 20ml of hot water. The mixture was heated under reflux for 30 minutes and then allowed to cool at room temperature for two hours. The crystals obtained were subjected to filtration, washed with cold ethanol and then recrystallized from ethanol (Scheme I). The melting point was 163-167°C. 5-MFAT dissolve in N,N-dimethylformamide (DMF), acetone and dimethyl sulphoxide. The characterization of 5-MFAT was carried out by IR ¹H NMR and Mass spectroscopy shown Figure 1, 2 and 3 respectively. The IR spectrum of 5-MFAT shows absorption bands around 1485 cm⁻¹ (C = S) 1540 cm⁻¹ (C = N) 2980 cm⁻¹ (Sp3 C-H) 3030 cm⁻¹(Sp2 C-H) and 3300 cm⁻¹ (-NH). The ¹H NMR (DMSO, ppm): 11.4 (-N-H), 7.6 (-NH₂), 9.85 (-sp² C-H), 2.6 (-CH₃), 7.2-7.8 (furan) and Mass spectrum of 5-MFAT shows signal at 184 (M +1) corresponding to its molecular ion peak. The molecular formula and molecular weight of the reagent C₇H₉N₃OS and 183 is respectively.



Scheme-1 preparation of 5-methylfuran-2-carboxaldehyde thiosemicarbazone (5-MFAT)

2.3. Preparation of standard Co (II) solution.

4.0375 g of cobaltous chloride hexahydrate (CoCl₂ · 6H₂O) is dissolved in double distilled water and made up to 1 L. It is standardized gravimetrically [33], with 2-nitroso-1-naphthol. Lower concentrations of cobalt (II) solutions are prepared suitably by diluting the stock solution with double distilled water.



Figure 1. Infrared spectrum of 5-methylfuran-2-carboxaldehyde-3-thiosemicarbazone (5-MFAT) in KBr pellet.

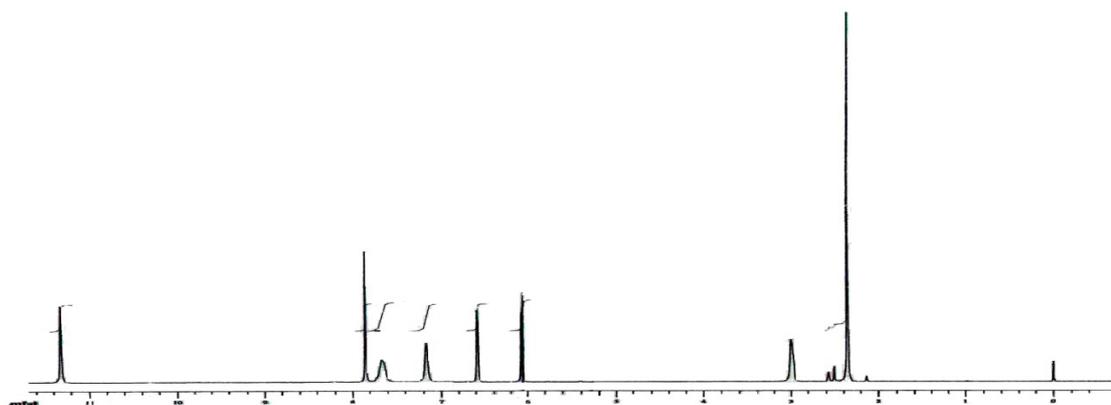


Figure 2. ^1H NMR spectrum of 5-methylfuran-2-carboxaldehyde-3-thiosemicarbazone (5-MFAT) in DMSO-d₆ medium

2.4. Buffer solutions

Solutions of 1.0 M sodium acetate and 1.0 M acetic acid were prepared in double distilled water. Suitable portions of these solutions were mixed to get the desired pH of the solution.

2.5. Analytical Procedure for vegetable samples

Three vegetable samples like Avalu (*Brassica nigra*), Chama (*Colocasia esculenta*) Thotakura (*Amaranthus gangeticus*), Palateega (*Ichnocarpus frutescens*) and Kakara (*Momordica charantia*) were analyzed for cobalt(II). Accurately weighed 5 g of sample was transferred into a Pre cleaned silica dish and 10 mL of 20 percent v/v sulfuric acid (3.3 M) is added. It was gently heated on a water bath for about thirty minutes and retransferred onto a hot plate, continuing until white fumes ceased to evolve, and the residue was completely evaporated to dryness. The dish was then placed in a muffle furnace and reheated up to 500°C. It was either left overnight or not less than a period of at least eight hours to completely decompose. The contents were transferred quantitatively into a 25 mL standard flask, washing the dish with double distilled water to ensure homogeneity.

2.6. Analytical Procedure for Soil samples

Soil samples were collected from in and around Kadapa, A.P and India. The sample was homogenized in the laboratory using a pestle and mortar and air-dried for approximately 24 h before analysis. An aliquot of 500 mg of finely pulverized soil was digested with 5 mL of nitric acid (65%) in a Teflon vessel. The sample was digested for about 3 h at 80°C and again redigested at 160°C for three quarters of an hour. After treating with double distilled water the supernatant liquid was made up to the mark in a 25 mL standard flask.

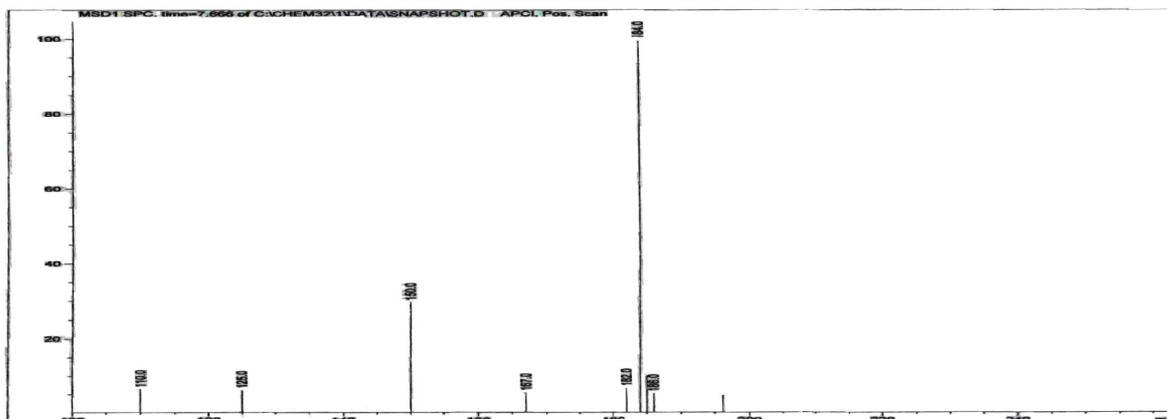


Figure 3. Mass spectrum of 5-methylfuran-2-carboxaldehyde thiosemicarbazone (5-MFAT).

2.7. Analytical Procedure for water samples

A choice of three water samples in and around Kadapa, A.P and India. Township has been made. Each filtered environmental water sample is evaporated nearly to dryness with a mixture of 5 mL of concentrated H_2SO_4 and 10 mL of concentrated HNO_3 in a fume cupboard and then cooled to room temperature. The residue is then heated with 10 mL of double distilled water in order to dissolve the salts. The solution is cooled and neutralized with dilute NH_4OH in the presence of 1-2 mL of 0.01% tartrate solution. The resulting solution was filtered and quantitatively transferred into a 25 mL calibrated flask and made up to the mark with double distilled water.

2.8. Analytical Procedure for water samples

The present method was also applied for the determination of cobalt (II) in alloy samples such as high-speed tool samples (BCS 484 and 485) and alloy steel samples (BCS 233 AND 266). About 0.1 g each of oven dried ($110^\circ C$) alloy sample was dissolved in 15 mL of aquaregia. They were heated to near dryness and the nitrate expelled from the residue using 5 mL of concentrated hydrochloric acid. Each residue was extracted into double distilled water, separately, and made up to 25 mL calibrated flask.

2.9. Recommended procedures

To an aliquot of a working standard solution containing $0.1-10 \mu g ml^{-1}$ cobalt (II) were added pH 6.0 buffer (3 ml), $1 \times 10^{-3} M$ reagent solution (1 ml) and a salting-out agent, 0.1 M magnesium sulfate (1 ml). The mixture was shaken two times with 10 ml portions of isobutanol each time for 1 min and allowed to stand for a few minutes. The two organic phases were collected into a 25.0 ml volumetric flask and made up to the mark with isobutanol. The absorbance's of all the organic phases were measured at 360nm against the reagent blank.

3.0. Results And Discussion

5-methylfuran-2-carboxaldehyde thiosemicarbazone (5-MFAT) forms a 1:2 (M:L) complex with cobalt (II), which is extracted into isobutanol from acetic acid sodium acetate (pH 6.0) buffer. The light yellow Co (II)-5-MFAT complex has a maximum absorbance at 360 nm and is stable for 46 hours. The conditions for effective extraction are established after studying the effects of various factors, such as pH, choice of the solvent, reagent concentration, salting-out agent and influence of diverse ions, in order to develop a rapid and sensitive extractive spectrophotometric method for the determination of cobalt (II) in micro levels.

3.1. Absorption spectra

The absorption spectra of the reagent solution against the corresponding solvent as a blank and that of the solution containing cobalt (II) complex against the reagent as a blank are recorded in the wavelength range 360 nm. The absorption spectra of the reagent and complex are shown in Figure. 4. The spectra reveal that Co (II)-5-MFAT

complex and the reagent have maximum absorbance at 460 nm, respectively. Hence, further absorbance measurements of the Co (II)-5-MFAT complex are recorded at 360 nm against the reagent blank.

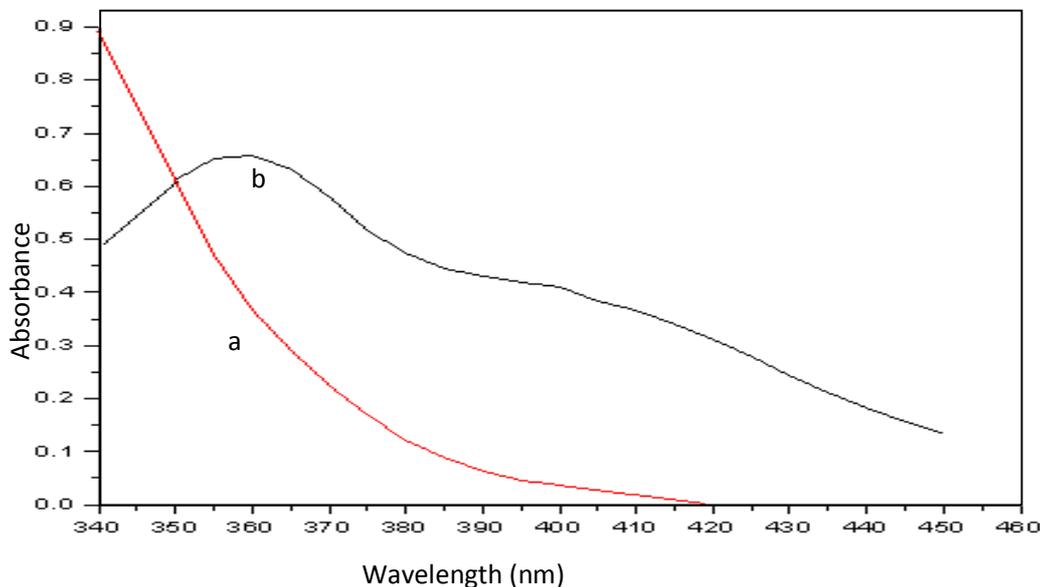


Figure 4: Absorption Spectra of a. 5-MFAT Vs Water blank, b. Co (II) – 5-MFAT complex Vs 5-MFAT solution, Co (II): 4×10^{-5} M, 5-MFAT: 6×10^{-4} M, pH:6.0.

3.2. Effect of pH

To arrive at the optimum pH required for full color development, the effect of pH on the color intensity is studied. In each case, a mixture containing 1.0 mL of 1×10^{-4} M cobalt (II), 3.0 mL of suitable buffer, 1.0 mL of 1×10^{-3} M 5-MFAT solution is taken, and the volume of the aqueous phase is adjusted to 10.0 mL with double distilled water. It is shaken with 10.0 mL portion of isobutanol for one minute. The organic phase is collected into a 25 mL standard flask and made up to the mark with isobutanol. The same procedure is applied for buffers of different pH values, ranging from 1.0 to 10.0. The absorbance's are measured at 360 nm, using their corresponding reagent blanks. A plot is executed between the pH and the absorbance, and the same is represented in Figure. 5. The plot shows that there is maximum absorbance and constancy in the pH range 5.0-7.0. Hence, pH 6.0 is chosen for further studies, considering this as an optimum pH.

3.3. Effect of solvents

The effect of various solvents such as isobutanol, chloroform, toluene, benzene, n-butanol, carbon tetrachloride, ethyl acetate, butyl acetate, xylene, tributyl phosphate, n-propyl alcohol and methyl isobutyl ketone on the extraction of cobalt(II) with 5-MFAT is studied adopting the standard procedure for registering the effect of pH. Among the various solvents studied, isobutanol is selected as the most suitable solvent, because of its maximum absorbance and greater extraction ability. The results are reported in Table 1.

3.4. Effect of reagent concentration

The effect of reagent concentration is studied using different aliquots containing constant volumes of 1.0×10^{-4} M cobalt(II) solution, 3.0 mL of pH 6.0 buffer solution, 1.0 mL of 0.1 m Magnesium sulphate and 1.0 mL of 5-MFAT solution containing different concentrations ranging from 1×10^{-4} to 20×10^{-4} M, in order to obtain the maximum color formation. The total volumes of aqueous phases are brought to 10.0 mL with double distilled

water. The aqueous phases are shaken with 10 mL of isobutanol in each case, and the organic phases are collected into 25 mL standard flasks. The organic phases are made up to the mark with isobutanol. The absorbance's of the organic phases are measured at 360 nm against their corresponding reagent blanks. It is clearly observed from the absorbance values that a maximum fifteen fold molar excess of the reagent is sufficient to get a maximum color formation of the complex.

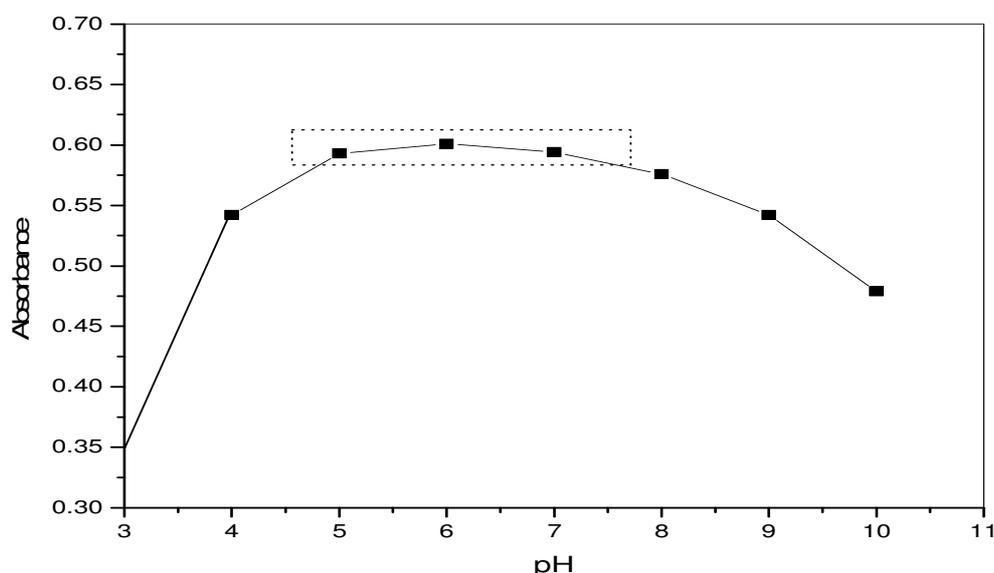


Figure 5: Effect of pH on the absorbance of Co(II) – 5-MFAT complex Co(II): 4×10^{-5} M, 5-MFAT: 4×10^{-4} M, λ_{\max} : 360 nm

Table 2: Effect of solvents on the extraction of Co(II)-5-MFAT complex

Solvent	Absorbance
n-Amyl alcohol	0.731
Isoamyl alcohol	0.672
Benzene	0.613
n-Butanol	0.693
Isobutanol	0.801
n-hexanol	0.615
Xylene	0.754
Chloroform	0.732
Cyclohexanane	0.681
Cyclohexanol	0.785
Carbon tetrachloride	0.793
Methyl isobutyl ketone	0.641

Co(II): 1.0 ml of 4×10^{-5} M; 5-MFAT: 1.0 ml of 4×10^{-4} M; pH: 6.0; λ_{\max} : 360 nm.

3.5. Effect of salting-out agents

Various salting-out agents, such as magnesium sulfate, lithium nitrate, lithium sulfate, lithium chloride, ammonium chloride and ammonium sulfate were attempted, in order to enhance the metal complex extraction into the organic phase in a single step. It is observed that the presence of 1.0 mL of 0.1 M Magnesium sulfate solution ensures complete extraction. The aqueous phase is tested for the complete extraction of cobalt (II) by using 1-nitroso-2-naphthol, disclosing that there is no cobalt (II) content in the aqueous phase. It is established that 1.0 mL of 0.1 M

Magnesium sulfate as salting-out agent and a fifteen-fold molar excess of the reagent quantitatively extracts cobalt (II) into the organic phase.

3.6. Validity of Beer's law, Molar Absorptivity, Sandells' Sensitivity and Correlation Coefficient for Co(II)-5-MFAT complex

The Co(II)-2,6-DAPBPTSC complex followed Beer's law in the range of 0.236-2.36 ppm. The molar absorptivity of the complex was calculated to be $1.8 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$, and Sandell's sensitivity of complex obtained from Beer's law data for absorbance, 0.001, was found to be $3.2 \times 10^{-3} \mu\text{g cm}^{-2}$. The correlation coefficient value of the Co (II)-5-MAFT complex, with an independent variable as concentration in $\mu\text{g mL}^{-1}$ and dependable variable as absorbance, was found to be 0.999. This indicated an excellent linearity between the two variables.

3.7. Precision, accuracy and detection limit of the method

To assess the precision and accuracy of the method, determinations are carried out at different concentrations of cobalt (II) under optimum conditions. The standard deviation of the method is found to be not more than 0.005, and the relative standard deviation was less than 0.434 percent. It is evident from these results that the method is precise, besides being accurate. The detection limit C_{\min} is determined as the amount of cobalt (II) corresponding to five times the standard deviation of the blank values, and a value of $0.0028 \mu\text{g mL}^{-1}$ is obtained.

3.8. Determination of The Composition of Co(II)-5-MFATT complex

The composition of the Co (II) complex with 5-MFAT was studied using Job's method of continuous variation, and the mole ratio method [20]. Extractive spectrophotometric investigation of the metal complex was conducted to obtain the composition of the complex. The composition of the complex was established by Job's method of continuous variation is shown in the Figure.6. Equimolar solutions of Co (II) and 5-MFAT ($2 \times 10^{-4} \text{ M}$) were prepared. The metal and reagent solutions were mixed in different proportions, keeping the total volume constant at 1.0 ml. To each solution, 3.0 ml of buffer (pH 6.0) solution and 1.0 ml of 0.1M magnesium sulphate solution as salting-out agent were added and the volumes of the aqueous phases brought to 10.0 ml with double-distilled water. Each of the aqueous phases was shaken with 10.0 ml of isobutanol for 2 min and allowed to settle. The organic phase was collected into a 25.0 ml standard flask and made up to the mark with isobutanol. The absorbance values of the organic phases were recorded at 360 nm, against their respective reagent blanks. From the above experimental results, it is evident that one mole of cobalt (II) reacts with two mole of 5-MFAT, showing the composition of the complex to be 1:2. This composition was verified using the molar ratio method. From jobs continuous variation method the stability constant of the complex found to be 4.05×10^{11} .

3.9. Effect of foreign ions

The tolerance limit means the limit within which the foreign ion cannot be interfered with for the determination of Co (II). In order to assess the possible analytical applications of this color reaction, the effect of some foreign ions are also examined by carrying out determinations of $75 \mu\text{g}$ of cobalt(II) with a known amount of the ion in question using the recommended general procedure. The criterion for interference is a variation of more than ± 2 percent in absorbance from the expected value for cobalt (II) alone. In the case of some interfering ions, an increased tolerance limit is achieved by the addition of masking agents, such as thiosulfate, fluoride, tartrate or thiocyanate. Increasing the amount of masking agents proportionately could mask a higher amount of interfering ions. In this study, cations like As(III), As(V), Mg(II), Mn(II), Zr(IV), Sb(III), Ca(II), Sr(II), Ba(II), and Tl(III) do not interfere, when present up to $5500 \mu\text{g}$ and cations like Bi(III), Hg(II), Be(II), Th(VI), U(VI), Al(III), and V(V) are tolerated up to $3000 \mu\text{g}$, but Cd(II), Cu(II), Ni(II), Zn(II), Fe(III), Mo(VI) and Pd(II) do interfere with the determination of cobalt(II) when present in more than $2000 \mu\text{g}$. The interference of Cu (II) can be eliminated by using 1.0 mL 0.2% oxalate as a masking agent. Fe (II) & Fe (III) are masked with 1.0 mL of 3% of sodium fluoride. The interference of Zn (II), Cd (II) can be eliminated by using 1.0 mL of 0.5% of thiosulphate solution. Anions like fluoruride, thiocyanate, thiosulfate and thiourea do not interfere when present up to $3000 \mu\text{g}$ with the determination in the method. Citrate and borate are tolerated up to $1500 \mu\text{g}$. Oxalate and phosphate interferes, even when present in trace amounts. EDTA masks cobalt (II) completely in the present determination.

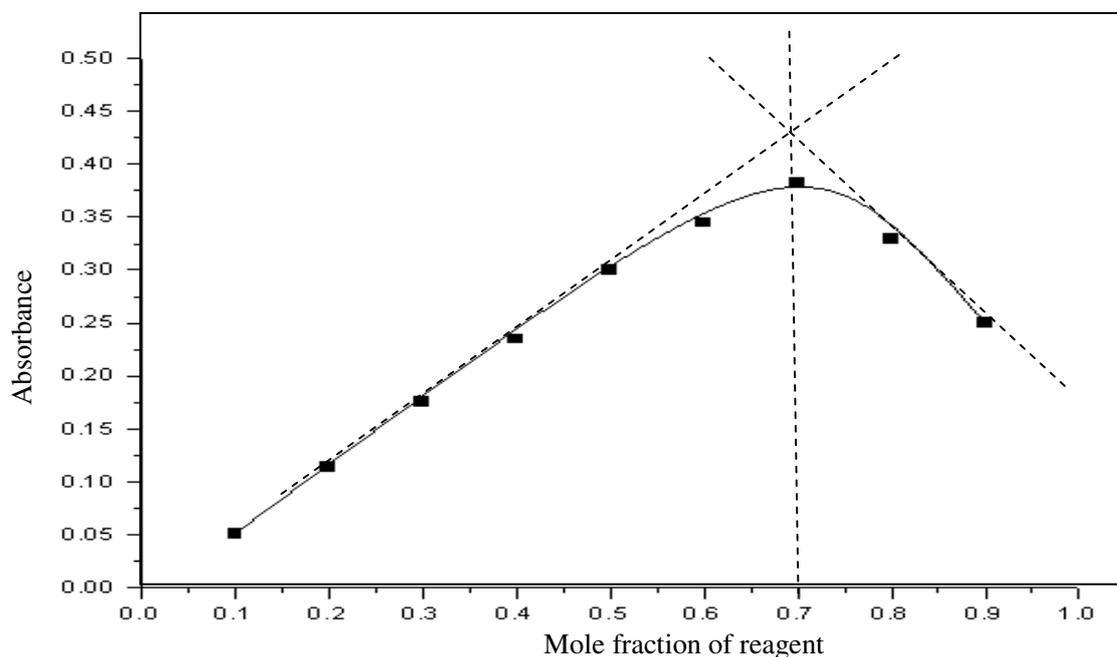


Figure 6: Job's Curve Co (II): 5-MFAT: 2×10^{-4} λ_{max} : 360 nm. pH: 6.0

3.10. Applications

The developed extractive spectrophotometric method for cobalt (II) is applied for its determination in real samples such as vegetable, soil, water and standard alloy samples.

3.11. Determination of cobalt(II) in vegetable samples

Vegetable samples were collected from around Kadapa, AP and India. Each aliquot was analyzed for cobalt(II) by the recommended procedure which was given in the experimental section. Cobalt (II) present in vegetable samples was determined from the calibrated plot (Beer's law plot) using 5-MFAT and the results checked by atomic absorption spectrometry Table 2.

3.12. Determination of cobalt(II) in soil samples

Soil samples were collected from around Kadapa, AP, and India. Each aliquot was analyzed for cobalt (II) by the recommended procedure which was given in the experimental section. Cobalt (II) present in soil samples was determined from the calibrated plot (Beer's law plot) using 5-MFAT and the results checked by atomic absorption spectrometry Table 3.

3.13. Determination of cobalt(II) in water samples

The aliquot was analyzed for Co (II) using 5-MFAT and adopting the recommended procedure. The amounts of cobalt (II) present in the samples were computed from the calibrated plot. These results were then compared by a Atomic Absorption Spectrometer; the derived values are shown in Table 4.

3.14. Determination of cobalt(II) in standard alloy samples

The present method was also applied for the determination of cobalt (II) content in standard alloy samples. The amount of cobalt (II) present in each one of the sample solutions was determined from a calibrated plot and the results checked by direct Atomic Absorption Spectrometer Table 5.

Table 2: Determination of cobalt (II)^a in vegetable samples.

Sample	Amount of Cobalt(II) ^b found $\mu\text{g/g}$			S D	RSD (%)
	Certified value	AAS Method	Present Method		
Avalu (<i>Brassica nigra</i>)	-	0.60	0.54	0.009	1.62
Chama (<i>Colocasia esculenta</i>)	-	1.08	1.09	0.016	1.51
Thotakura (<i>Amaranthus gangeticus</i>)	-	1.09	1.10	0.017	1.58
Pala teega (<i>Ichnocarpus frutescens</i>)	-	1.48	1.46	0.016	1.51
Kakara (<i>Momordica charantia</i>)	-	1.25	1.23	0.025	2.03

^aNo statistically significant differences were found between Co (II) concentrations measured by the AAS method and the present method. ^bAverage of four determinations.

Table 3: Determination of cobalt (II) in soil samples.

Name of the area	Amount of Co(II) ^a found ($\mu\text{g ml}^{-1}$)		S D	RSD (%)
	AAS Method	Present Method		
Rayachoty	24.0	23.7	0.227	0.96
Pamidi	23.0	22.8	0.225	0.99
Madhanapalli	20.0	19.8	0.194	0.98
Narpala	20.0	19.5	0.175	0.90
Vempalli	20.0	19.6	0.180	0.92
Dhadithota	21.0	20.7	0.184	0.89
Penuconda	20.0	19.3	0.231	1.20
S.V. Puram	19.0	18.6	0.167	0.90
Dharmavaram	19.0	18.2	0.145	0.80
Madhavaram	22.0	21.9	0.219	1.00
Reddyvari palli	22.0	21.8	0.239	1.10
Gooty	23.0	22.9	0.286	1.25

^aAverage of five determinations.

Table 4: Determination of trace amounts of Cobalt (II)^a in water samples.

Sample	Amount of Cobalt(II) found $\mu\text{g/ml}$			S D	RSD (%)
	Certified value	AAS Method	Present Method		
River water (Swarnamukhi)	-	1.92	2.01	0.01	0.60
Waste water (Kadapa)	-	2.30	2.34	0.02	1.06
Sea water (Marina Beach)	-	1.90	1.85	0.02	1.16

^aAverage of five determinations.

Table 5: Determination of cobalt (II)^a in standard alloy samples.

Sample	Amount of Cobalt(II) ^b found			S D	RSD (%)
	Certified value	AAS Method	Present Method		
High speed tool (BCS 484)	10.20	10.09	10.02	0.15	1.52
High speed tool (BCS 485)	05.06	05.05	05.02	0.07	1.51
Alloy steel (BCS 233)	23.40	23.25	23.07	0.29	1.26
Alloy steel (BCS 266)	23.40	23.34	23.17	0.38	1.67

^aAverage of five determinations.

^bConcentration in %.

Conclusion

A thorough survey of the literature reveals that many thiosemicarbazones are utilized for the determination of cobalt (II). Studies upon the use for 5-methylfuran-2-carboxaldehyde thiosemicarbazone as an analytical reagent

are however limited. The present investigations are carried out with a view to test the potential of 5-MFAT as a complexing agent for cobalt (II) and its subsequent determination by extraction spectrophotometry. The selectivity of this method is enhanced by using masking agents for Ni (II), Cd (II), Cu (II), Fe (III) and Zn (II). Finally, it is established that this method is suitable for the determination of cobalt (II) in vegetable, soil, water and standard alloy samples.

Acknowledgements-I am highly grateful thanks to G. Ramachandra Reddy, for extending moral support to me use the AAS results from kadapa, India. My sincere thanks to Sri Krishnadevaraya University for conducting this research programme.

References

1. Underwood E. J., "Trace Elements in Human and Animal Nutrition" 4th ed.; Academic Press: New York, (1979).
2. Eichhorn, G. L., "Inorganic Biochemistry" Elsevier Scientific Publications Company: New York, (1975).
3. Oser, B. L., "Hawks Physiological Chemistry" 14th ed.; T. M. H. Publishing Company: New Delhi, (1979).
4. Brown, T., Richardson. and Sinclair, H. M., *Proc. Soc. Exptl. Biol.* 90 (1955) 135.
5. Emerson, G. A., *Ibid.* 70 (1949) 392.
6. Lillie, F. J., Olsen, N. S., and Bird, H. R., *Proc. Soc. Exp. Biol.* 72 (1949) 598.
7. Hall, I. H., Lackey, C. B., Kistler, T. D., Durham, R. W., Joud, E. M., Khan, M., Thanh, D. X., Djebbar-sid, S., Benali-Baitich, O., and Bouet, G. M., *Pharmazie.* 155 (2000) 937.
8. Mille M. C., Basto K. F., Stinema C. N., Vance J. R., West D. X.; Hall I. H. *Anti Cancer Res.* 18 (1998) 4131.
9. Buu-Hoi, Ng. Ph., Loc, T. B., and Xuong, Ng. D. *Bull Soc.Chem. France.* (1955) 694.
10. Pavon, J. M. C. *Mircro chem. J.* 26 (1981) 155.
11. Garg, B. S., and Jain, V. K. *Micro chem. J.* 38 (1988) 144.
12. Casas, J. S., Garcia-Tasende, M., and Sordo, S. *J. Coord. Chem.* 209 (2000) 197.
13. Bendito, D. P., and Pino, F., *Inform. Quim. Anal.* 31 (1968) 721.
14. Reddy, N. S., and Reddy, D. V., *Anal. Chim. Acta.* 120 (1980), 395.
15. Pavon, J. M.C., Bendito, D. P., and Pino, F., *An. Quim.* 65 (1969) 667.
16. Idem., *Analyst.* 99 (1974) 355.
17. Rueda, M. R., Leyva, J. A., and Antonio, M. J. *Quim. Anal.* 29 (1975) 122.
18. Salinas, F., Sanchez, J. C. J., and Gallego, J. M. L., *Ann.Chim. Rome.* 76 (7-8) (1986) 301.
19. Lingappa, Y., Reddy, K. H., and Reddy, D. V. *Talanta.* 34(9) (1987) 789.
20. Reddy, B.K., Kumar, C.J., Sarma, L.S., and Reddy, A.V., *Asian J. Chem.* 9 (1997) 487.
21. Atalay, T., and Akgemci, E.G., *Turk. J. Chem.*, 22 (1998) 123.
22. Reddy, S.A., Reddy, K.J., Narayana, S.L., Sarala, Y., and Reddy, A.V., *J.Chin. Chem. Soc.*, 55 (2008) 326.
23. Akgemci, E.G., and Atalay, T., *Turk. J. Chem.*, 24 (2000) 89.
24. Lokhande, R.S., Jadhav, M., and Dapale, S., *Int. J. Chem. Sci.*, 4 (2004) 296.
25. Kumar, A.P., Reddy, P.R., and Reddy, V.K.. *J. Korean Chem. Soc.*, 51 (2007) 331.
26. Kumar, A.P., Reddy, P.R., and Reddy, V.K. *J. Autom. Methods Manag.Chem.*, 48 (2007) 768.
27. Konstantinovic S.S., Radovanovic B.C., Todorovic Z.B., Ilic S.B., *J. Serb. Chem. Soc.*, 72 (2007) 975.
28. Lokhande R.S., and Pawar R.N., *Asian J. Chem.*, 18 (2006) 3241.
29. Balouch, A., Memon N., Bhangar, M.I., and Khuhawar, M.Y., *J. AOAC Int.*, 92 (2009) 248.
30. Vareda E., Rios, A., and Valcarcel, M., *Analyst*, 122 (1997) 85.
31. Sarma, L.S., Kumar, J.R., Kumar, C.J., and Reddy, A.V., *Anal. Lett.* 36 (2003) 605.
32. Khuhawar, M.Y., and Lanjwani, S.N., *Talanta.* 46 (1998) 485.
33. Vogel, A. I. *A Text Book of Quantitative Inorganic Analysis*; 3rd ed.; Longman: Green London, (1961).

(2015) ; <http://www.jmaterenvironsci.com/>