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Trace determination of isoniazid at micro level using kinetic spectrophotometric method

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Abstract: An effective and fairly inexpensive spectrophotometric method for tracing the determination of isoniazid (INH) in pure form, as well as in pharmaceutical formulations, has been developed through the ligand substitution reaction between INH and aquapentacyanoruthenate (II) ion ($[Ru(CN)_5OH_2]^{3-}$) in aqueous medium at λ_{max} = 502 nm. The fixed time procedure has been employed under optimum reaction conditions. The calibration equations, relating absorbance measured at 502 nm at fixed times (t_n = 2, 5 and 7 min) and c_{INH} in linear range 1.37-27.43 μg mL^{-1}, were used for the trace determination of INH, which has been reported in the present investigation and are in agreement with official and reported methods. The percentage recovery has been calculated and found to be within the range of 99–101 % in the analysis of different pharmaceutical samples. The results reveal that the use of common recipients as the used additives do not produce any type of interference in the suggested method. The validity of the proposed method was also checked by statistical analysis which agreed with the results obtained using the official method. The present method is very simple, reproducible, sensitive and it can be adopted for trace determination of INH in different samples without using extracting agent.

Keywords: ligand substitution reaction; aquapentacyanoruthenate (II) ion; calibration equations; pharmaceutical samples analysis

INTRODUCTION

Tuberculosis, caused by *Mycobacterium tuberculosis*, is a major health hazard for the society and currently a prominent cause of mortality among all existing diseases globally as it affected a mass population worldwide[1]. Patients with the decreased immunity are suspected to be frequent sufferer. At present, the front-line treatment against the infection caused by *Mycobacterium tuberculosis* involves the use of first line drug isoniazid[2] (Fig. 1), prepared first in 1912[3] and was found to be effective against tuberculosis in 1952.[4] Although INH possess a very simple chemical structure and exhibits various pharmaceutical properties

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over last few decades, yet it is possible that the synergistic mechanism of action still remains uncertain.[5]

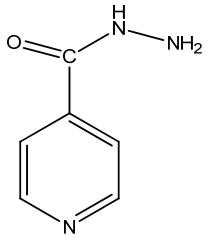


Fig. 1. Structure of isoniazid, INH.

Isoniazid, INH (pyridine-4-carboxylic acid hydrazide), commercially known as isonicotinylhydrazide, is a prodrug and it is activated by the enzyme catalase-peroxidase, produces isonicotinoyl acyl radical[6] through subsequent steps after being added to NAD^+ and forms adduct, INH-NADH. The adduct so formed that it is responsible for producing antitubercular activity in INH[7]. Owing to various biological and medicinal applications of INH in several industrial and pharmaceutical processes, its determination in pure form and in pharmaceutical preparations (dosage form) is of great public interest. The various analytical methods for detection, determination and analysis of INH in pharmaceutical formulations as well as in analytical and biological samples have been described in literature based on colorimetry,[8] spectrophotometry,[9] NMR spectroscopy,[10] spectrofluorimetry,[11] atomic absorption spectrometry,[12] chemiluminescence-based detection,[13] capillary electrophoresis,[14] flow injection analysis,[15] kinetic determination,[16,17] voltammetry/electrochemical[18,19] and different chromategraphic techniques such as capillary gas chromatography,[20] thin layer chromatography,[21,22] electrokinetic chromatography,[23,24] liquid chromatography[25] and high-performance liquid chromatography.[26] Recently, analytical methods for the isoniazid determination has been reviewed. In addition to the above mentioned techniques, some indirect approaches have also been reported for the determination of INH such as the designation of an optical sensor by Safavi and Bagheri.[27] Recently, analytical methods for the isoniazid determination have been reviewed.[28] Many of the methods reported above require expensive and hazardous chemicals and involves sophisticated instrumental techniques. These techniques are very expensive**,** time consuming and not easily accessible in most of the laboratories in India, which limits their direct applications in real samples. In the past, few analytical methods were developed by Naik *et al.* for different analytes[29–35] and Prasad *et al.*[36] Based on the knowledge gained by us in our earlier investigations, here we report a reliable kinetic spectrophotometric method, which has good reproducibility for the trace determination of INH at micro level in pure form as well as in pharmaceutical samples. The present investigation is based on the ligand exchange reaction between aquapentacyanoruthenate(II) ion ($[Ru(CN)_5OH_2]^{3-}$)

and INH in aqueous medium and it is the first indicator reaction of its kind, using aqupentacyanoruthenate(II) for the determination of INH in various pharmaceutical preparations. Further, the novelty of the proposed method lies in the fact that the statistical parameters, as well as recovery experiment data, demonstrates high reproducibility and accuracy of the method and compares adequately with the official reported method[37] for the assay of the INH tablets. Apart from this, the analysis of the pharmaceutical preparations which contain isoniazid as a single drug, as well as the multiple ingredients which show the no interference from the common excipients and does not involve the use of any oxidant, prior heating of a sample containing an extracting agent and uses a simple spectrophotometer for determination.

Therefore, this method could be recommended for the determination of isoniazid in pharmaceutical quality control laboratories.

The applicability of the method was tested on 6 INH containing pharmaceutical samples with the use of pre-established calibration equations, between the absorbance and the time at the fixed wavelength based on the fixed time procedure.

EXPERIMENTAL

Reagents

All analytical reagent grade chemicals and double distilled de-ionised water were used throughout the present kinetic study. All the solutions were stored in dark amber coloured bottles to prevent photodecomposition and oxidation. Potassium hexacyanoruthenate(II) ($K_4[Ru(CN)_6]\cdot 2H_2O$) was obtained from Merck (India) and used as received. The INH was purchased from BDH (UK) and its required stock solution was made in the double distilled de-ionized water by weighing the calculated amount accurately. The sodium perchlorate was purchased from Merck and used without any further purification. Now, a 2×10^{-2} mol dm^{-3} of $NaClO_4$ was prepared and used to maintain the ionic strength (μ) of the reaction mixture at a desired value. A 10^{-2} M stock solution of bromine was standardized regularly using the iodometric method.[38] The pH of the reaction mixture was maintained using KCl/HCl or potassium hydrogen phthalate and HCl/NaOH buffer.[39] Standard BDH buffers were used to standardize the pH meter regularly from time to time.

Apparatus

The ligand substitution kinetics between $[Ru(CN)_5OH_2]^{3-}$ and INH was studied spectrophotometrically using a single beam visible-spectrophotometer model DIGI-110 (SISCO, India), equipped with a circulatory arrangement of water for thermostating the cell compartment. Recording of the absorption spectra was made by the UV–Vis double-beam spectrophotometer (Systronic-2203). All the pH measurements were made by Systronics μ pH system, 361 model. All the volumetric apparatus used were of certified 'A' grade and steamed regularly before each kinetic run. Acetone and 10 % HNO_3 solutions were used for cleaning of the quartz cuvettes from time to time.

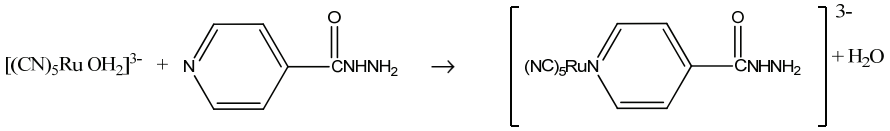
Experimental procedure

All the reaction conditions have been optimized to carry out the experiment optimized in an order to attain the maximum reaction rate and sensitivity. The stock solutions were accur-

ately diluted to the required concentrations. In order to attain the thermal equilibrium, all the working solutions were placed in a self-designed thermostat for at least 30 min at 25.00±0.01 °C, prior to the start of the reaction, by placing them in volumetric flasks. All the thermally pre-equilibrated reagent solutions were added in a reaction mixture in the sequence: $[Ru(CN)_5OH_2]^{3-}$, $NaClO_4$ and INH to initiate the reaction. The reaction mixture was properly shaken and quickly transformed into a 10 mm spectrophotometric quartz cuvette, placed in a pre-thermostatic cell compartment of spectrophotometer. The progress of the reaction was monitored by monitoring the increase in the absorbance due to the formation of pale yellow coloured complex, $[Ru(CN)_5INH]^{3-}$ (λ_{max} = 502 nm) where only product absorbs strongly, while other reactants do not have appreciable absorbances at this wavelength.

The examined reaction is believed to proceed through Eqs. (1) and (2), respectively:

$$\left[Ru(CN)_6\right]^{4-} + Br_2 \xrightarrow{H_2O} \left[Ru(CN)_5OH_2\right]^{3-} + Br^- + BrCN \quad (1)$$



(2)

Eq. (1) represents the generation of $[Ru(CN)_5OH_2]^{3-}$ (pale yellow colour, having λ_{max} = 310 nm) through rapid aquation of $[Ru(CN)_6]^{4-}$, mixing the equimolar concentrations of hexacyanoruthenate(II) and bromine with KBr in tenfold excess other reactants do not have appreciable absorbances at this wavelength.

RESULTS AND DISCUSSION

The incess[37] generates the desired pale yellow product, *i.e.*, $[Ru(CN)_5INH]^{3-}$, in Eq. (2) upon subsequent reaction with INH. The pale yellow colour solution gets intensified and a peak grows at 502 nm due to the formation of the product, *i.e.*, $[Ru(CN)_5INH]^{3-}$. The absorbance spectra for the reactants and the product are given in Fig. 2.

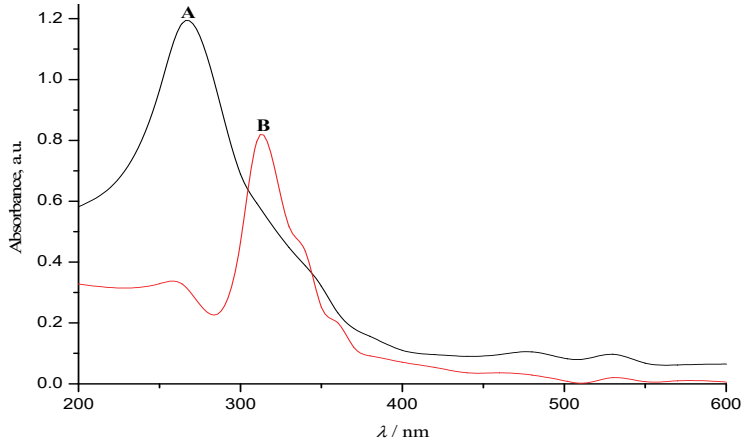


Fig. 2. UV–Vis absorption spectra of the reactants: A – $c_{INH} = 5\times10^{-3}$ mol dm^{-3}; B – $c_{[Ru(CN)5OH2]3-} = 5\times10^{-4}$ mol dm^{-3}.

The repetitive spectral scan for the formation of the product, $[Ru(CN)_5INH]^{3-}$, is given in Fig. 3. and it clearly demonstrates the formation of product which has the wavelength of maximum absorbance (λ_{max} at 502 nm).

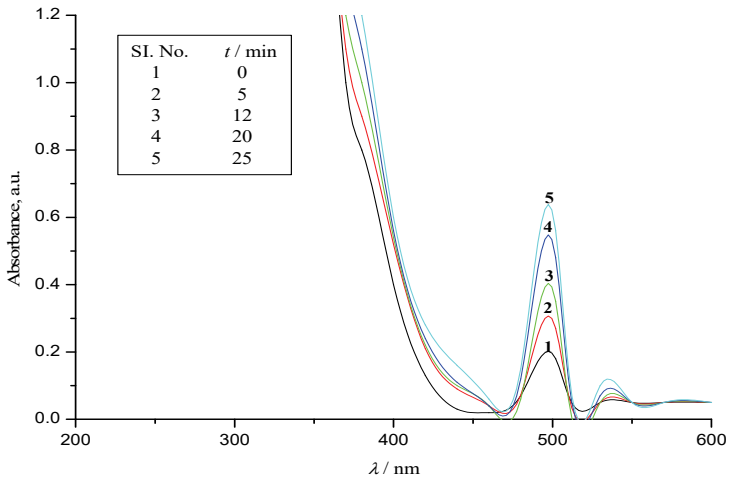


Fig. 3. Repetitive spectral scan of formation of product, $[Ru(CN)_5INH]^{3-}$ under the reaction conditions: $c_{[Ru(CN)_5OH_2]^{3-}} = 5\times10^{-4}$ mol dm^{-3}, $c_{INH} = 5\times10^{-3}$mol dm^{-3}, pH 4.00±0.02, $\mu = 2\times10^{-2}$ mol dm^{-3} ($NaClO_4$) and $t = 25.00\pm0.01$ °C.

In general, the reaction rate is found to be influenced by various parameters, *viz.* pH, concentration of reactant, ionic strength and temperature. Thus, the kinetics of the examined reaction is studied by optimizing the different parameters sequentially, under pseudo-first-order condition by keeping all the parameters fixed at a constant value, except the one whose effect has to be studied. In the present work, the effects of pH, $c_{[Ru(CN)_5OH_2]^{3-}}$, c_{INH}, ionic strength and temperature variation on the reaction rate have been investigated thoroughly. Optimized experimental conditions were made by a precise investigation of the kinetic study of the indicator reaction. The $c_{[Ru(CN)_5OH_2]^{3-}}$ and c_{INH} were varied from 2.0×10^{-4}–7.0×10^{-4} mol dm^{-3} and 10^{-5}–2×10^{-4} mol dm^{-3}, respectively. The reaction was studied at a pH 4.00±0.02. All the results were obtained at $t = 25.00\pm0.01$ °C and $\mu = 2\times10^{-2}$ mol dm^{-3}, respectively.

Effect of pH

The reaction rate depends upon the pH of the reaction mixture and therefore the maintenance of pH is essential to be at an optimum level. In order to select a suitable value of pH, its influence on the reaction rate was investigated in a pH range 2.0–8.0 using the fixed time procedure method, as a measure of the initial rate. The pH of the reaction mixture was varied using potassium hydrogen phthalate/NaOH or potassium hydrogen phthalate/HCl buffer.[40] The variations obs-

erved in initial rate (A_t) after 2 and 8 min of the mixing of the reagents as a function of the pH is provided in Fig. 4. It was observed that the reaction rate was slow at low pH values initially, became the maximum at pH of 4.00±0.02 and then it decreased. At low pH, lower reaction rate is attributed to the formation of various protonated species[41] of $[Ru(CN)_6]^{4-}$ such as monoprotonated; $H[Ru(CN)_6]^{3-}$ and diprotonated, $H_2[Ru(CN)_6]^{2-}$. Again, at higher pH, the low reaction rate is due to lack of protons. Thus, the pH 4.00±0.02 is associated with the maximum absorption and selected as an optimum pH for further study.

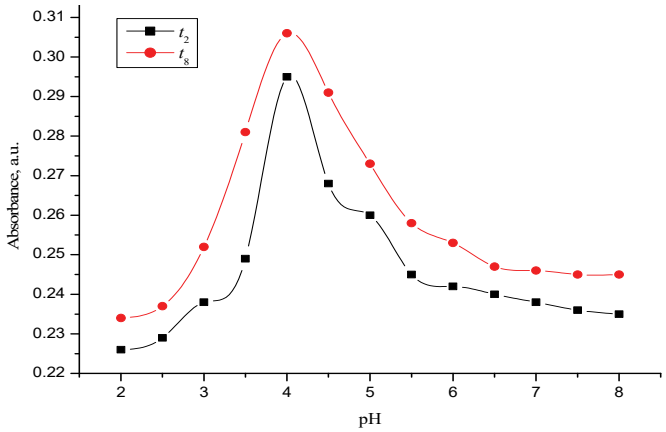


Fig. 4. Effect of pH on the reaction rate under the reaction conditions: $c_{[Ru(CN)_5OH_2]^{3-}} = 6\times10^{-4}$ mol dm^{-3}, $c_{INH} = 5\times10^{-3}$mol dm^{-3}, pH 4.00±0.02, $\mu = 2\times10^{-2}$ mol dm^{-3}($NaClO_4$) and t = 25.00±0.01 °C.

Effect of $[Ru(CN)_5OH_2]^{3-}$

The above selected value of pH 4.00±0.02 facilitated us to do further investigation by varying other parameters sequentially. The influence of $c_{[Ru(CN)_5OH_2]^{3-}}$ upon absorption intensity during the formation of $[Ru(CN)_5INH]^{3-}$ was investigated by the variation of its concentration in the range of 2.0×10^{-4}–7.0×10^{-4} mol dm^{-3}. The plot of absorbance *versus* $c_{[Ru(CN)_5OH_2]^{3-}}$ clearly demonstrates that the reaction attains a steady rate after 5×10^{-4} mol dm^{-3} of $c_{[Ru(CN)_5OH_2]^{3-}}$ (Fig. 5). Thus, 5×10^{-4} mol dm^{-3} $c_{[Ru(CN)_5OH_2]^{3-}}$ is recommended for further study which provides appreciable change in the absorbance value.

Effect of temperature

The dependence of the reaction rate on the temperature was studied by the variation of the temperature in the range of 20 to 45 °C, keeping other reaction variables fixed at optimum values. Higher temperature was strictly avoided due to the possibility of degradation of the product $[Ru(CN)_5INH]^{3-}$. A distinct change in reaction rate was noticed at temperature 25 °C. Hence, the room temperature 25.00±0.01 °C was chosen for further study as an optimum temperature.

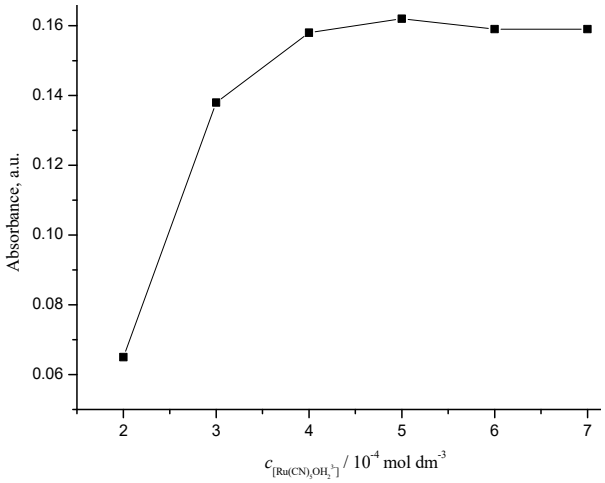


Fig. 5. Effect of $c_{[Ru(CN)_5OH_2]^{3-}}$ on the reaction rate under the reaction conditions: $c_{INH} = 5\times10^{-3}$mol dm^{-3}, pH 4.00±0.02, $\mu = 2\times10^{-2}$mol dm^{-3} ($NaClO_4$) and t = 25.00±0.01 °C.

*Effect of ionic strength (*μ*)*

The dependence of reaction rate on the ionic strength of the medium was investigated by varying $NaClO_4$ concentration (2.5×10^{-2}–2×10^{-1} mol dm^{-3}). The variation of ionic strength on the rate of reaction showed that the ionic strength of 2×10^{-2} mol dm^{-3} of $NaClO_4$ is optimum for the determination of the drug.

Kinetic determination of INH in pharmaceutical formulations

The fixed time procedure method was employed to obtain the calibration equations, under the optimum reaction conditions. A pre-selected time was chosen to record the absorbance for different concentrations of INH. The graph plotted between absorbance *versus* c_{INH} (Fig. 6) displays linear dependence in c_{INH} in the range of (1–20)$\times10^{-5}$ mol dm^{-3} (1.37–27.43 μg mL^{-1}), at different fixed times. Hence, the calibration graphs were obtained in the above-mentioned range. The calibration equations relating absorbance at fixed time (t_n = 2, 5 and 7 min) with c_{INH} have been presented in Table I, along with the values of correlation coefficient (r^2) and detection limit (3σ).

The precision and accuracy of the method.

Although the linearity is obtained in the same ranges of concentrations for all three fixed time intervals, a time of 5 min is recommended for further study, as it is when the best linearity was achieved (maximum correlation coefficients, r^2), which also satisfies the application of short analysis time.

The recovery experiment was performed to check the precision and the accuracy of the given method confirmed, adding the calculated INH amount in the range (3.060–27.382 μg mL^{-1}) in double distilled de-ionised water for the

recovery of drug. The results acquired are provided in Table II along with the errors and standard deviations (±*sd*). The recovered values were measured in the calibration range of 98–102 % using five independent determinations. The results tabulated in Table II show excellent reproducibility of the present method. In order to correspond to the calibration equations, the errors obtained during the measurement of the absorbance at 5 min, *i.e.*, A_5 is comparatively much less than those obtained during the study at 2 and 7 min (*i.e.*, A_2 and A_7), as the study of initial rate in case of A_5 is more adjacent than A_2 or A_7. Consequently, for further study a fixed time interval of 5 min is favoured, which is in accordance with the short analysis time, reproducibility and maximal value of correlation coefficients, r^2 as stated above (*vide infra*). Therefore, in the further investigation, at trace level calibration equation, A_5 was used for INH determination.

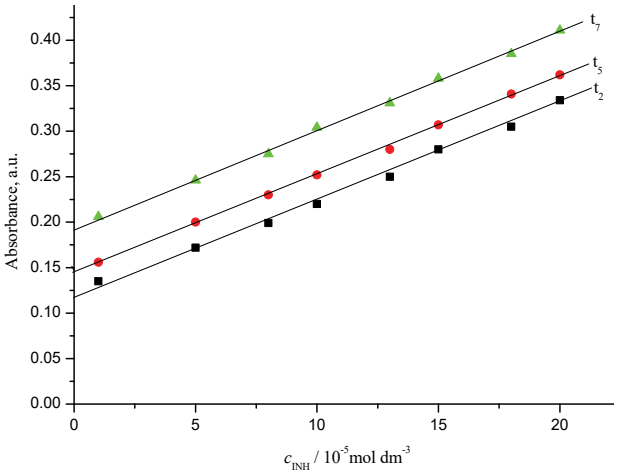


Fig. 6. Linear dependence of c_{INH} on the reaction rate at different times under the reaction conditions: $c_{[Ru(CN)_5OH_2]^{3-}} = 5\times10^{-4}$ mol dm^{-3}, pH 4.00±0.02, $\mu = 2\times10^{-2}$ mol dm^{-3} ($NaClO_4$) and t = 25.00±0.01 °C.

TABLE I. Determination of c_{INH} in the range of (1–20)×10^{-5} mol dm^{-3} under the optimum reaction conditions at $c_{[Ru(CN)_5OH_2]^{3-}} = 5\times10^{-4}$ mol dm^{-3}, pH 4.00±0.02, $\mu = 2\times10^{-2}$ mol dm^{-3} ($NaClO_4$) and t = 25.00±0.01 °C

Calibration equation	Detection limit, μg mL^{-1}	r^2
$A_2 = 1.05\times10^3 c_{INH} + 0.1193$	0.204	0.9957
$A_5 = 1.08\times10^3 c_{INH} + 0.1442$	0.210	0.9990
$A_7 = 1.08\times10^3 c_{INH} + 0.1929$	0.197	0.9983

Study of selectivity

In order to examine the applicability of the proposed method, the magnitude of the interference produced by some excipients in pharmaceutical agents was studied by the above mentioned procedure under the optimal reaction conditions.

The recovery experiments were performed in samples containing fixed concentration of INH (10 μg mL^{-1}) and a huge amount of diverse species. From the results, it is suggested that the chosen method is not affected by interferences of potential excipients even when their concentrations are quite high up to 1000 times (Table III). The tolerance limit (Excipient/INH) was evaluated for different excipients and the maximum error up to ±4 % was considered to be quite tolerable.

TABLE II. Accuracy and precision of c_{INH} determination by the proposed method under the reaction conditions: $c_{[Ru(CN)_5OH_2]^{3-}} = 5\times10^{-4}$ mol dm^{-3}, pH 4.00±0.02, $\mu = 2\times10^{-2}$ mol dm^{-3} ($NaClO_4$) and t = 25.00±0.01°C

c_{INH} added μg mL^{-1}	A_2		A_5		A_7	
	c_{INH} found[a]±sd[b] μg mL^{-1}	Error %	c_{INH} found[a]±sd[b] μg mL^{-1}	Error %	c_{INH} found[a]±sd[b] μg mL^{-1}	Error %
3.060	3.041±0.07	–0.62	3.052±0.03	–0.26	3.078±0.05	+0.59
4.441	4.476±0.04	+0.79	4.468±0.03	+0.61	4.432±0.04	–0.20
6.079	6.058±0.08	–0.34	6.068±0.16	–0.18	6.071±0.08	–0.13
7.402	7.312±0.05	–1.22	7.484±0.06	+1.11	7.436±0.04	+0.46
9.316	9.221±0.08	–1.02	9.186±0.05	–1.40	9.306±0.02	–0.11
13.212	13.362±0.51	+1.13	13.218±0.12	+0.04	13.196±0.26	–0.12
16.451	16.683±0.25	+1.41	16.422±0.02	–0.17	16.384±0.09	–0.41
19.268	19.182±0.18	–0.44	19.274±0.13	+0.03	19.564±0.10	+1.53
21.943	21.952±0.03	+0.04	21.881±0.44	–0.28	21.763±0.39	–0.82
25.324	25.262±0.62	–0.24	25.383±0.09	+0.23	25.427±0.23	+0.40
27.382	27.638±0.02	+0.93	27.394±0.33	+0.04	27.582±0.56	+0.73
Average		+0.42		–0.23		+0.17

[a]Mean of five determinations; [b]standard deviation of the mean of five determinations

TABLE III. Determination of INH concentration at 10 μg mL^{-1} level in presence of excipients under the optimum reaction conditions: $c_{[Ru(CN)_5OH_2]^{3-}} = 5\times10^{-4}$ mol dm^{-3}, pH 4.00±0.02, $\mu = 2\times10^{-2}$ mol dm^{-3} ($NaClO_4$) and t = 25.00±0.01 °C

Additive species	Tolerance limit	Recovery of drug±sd[a], %
Iodide	1000	100.2±0.8
Citrate	500	99.06±0.6
Talc	500	100.46±0.2
Sodium chloride	500	101.3±0.4
Glucose	1000	97.03±0.8
Lactose	500	99.6±0.6
Starch	500	100.2±0.2
Sodium phosphate	1000	101.4±0.6

[a]Standard deviation of the mean of five determinations

Application of the method for the assessment of INH in pharmaceutical preparations

To check the applicability of the developed kinetic spectrophotometric method it was successfully tested on six pharmaceutical samples for INH deter-

mination. The samples were prepared by the method discussed previously by Naik and co-workers.[35] Twenty tablets were weighed, grounded and the average mass per tablet was determined. The finely grounded tablet material was weighed accurately to 200 mg of INH, transferred and solubilized into different 100 mL flasks containing 70 mL de-ionised distilled water. The flasks containing dissolved INH were sonicated for 15 min and further diluted with 100 mL of de-ionised distilled water. In subsequent steps, the solutions were filtered using a 0.45 μm Millipore Whatmann filter paper. The concentration of the drugs within the calibration range was maintained by its accurate dilution using de-ionised distilled water. Further, the diluted samples were analysed directly using the calibration equation for INH determination. Analysis of six samples has been executed and presented in Table IV and the results indicate the quantitative and higher recovery of INH in the range of 99–101 % using size independent determinations.

TABLE IV. Determination of INH concentration in pharmaceutical preparations under the reaction conditions: $c_{[Ru(CN)_5OH_2]^{3-}} = 5\times10^{-4}$ mol dm^{-3}, pH 4.00±0.02, $\mu = 2\times10^{-2}$ mol dm^{-3} ($NaClO_4$) and t = 25.00±0.01 °C

Type of drug used	Amount taken, mg	Amount found±*sd*[a], mg		*t*-value	*F*-value
		Proposed method	Standard method[39]		
Solonex tablet (Macleods)	300	298.4±0.4	298.3±0.5	0.349	1.56
Solonex tablet (Macleods)	100	99.6±0.3	99.4±0.5	0.767	2.77
Isonex tablet (Pfizer)	100	99.6±0.3	99.5±0.4	0.447	1.77
Ipcazide tablet (IPCA)	100	99.2±0.3	99.0±0.4	0.894	1.77
Isokin tablet (Warner)	100	99.5±0.4	99.4±0.5	0.349	1.56
Isokin liquid (Warner)	20	19.6±0.3	19.4±0.5	0.767	2.77

[a]Standard deviation of the mean for five determinations

Validation of the proposed method by statistical analysis of the results in comparison with the official method

The Student's *t*-test and the variance ratio *F*-test was used to judge the performance of the proposed method statistically. At 95 % confidence level, the computed *t*-values from the Student's *t*-test and *F*-values from the variance ratio *F*-test do not exceed the theoretical values, *i.e.*, 2.776 (theoretical *t*-value for n = = 5) and 6.39 (theoretical *F*-value for n = 5), provided in Table IV. Thus, it signifies that there are no remarkable difference between the proposed method and the official method, assuring the suggested method is quite accurate and precise as the official method.[37] The result of INH analysis, using above recommended procedure, was found to be significantlly agreement with that obtained from the standard method.

CONCLUSION

The sugessted spectrophotometric method for INH determination is quite feasible when compared to other developed methods, which are reported in literature and it is of major interest in analytical pharmacy, because it offers the distinct possibility in the assay of isoniazid in pharmaceutical formulations. The main importance of the proposed method lies in the fact that it involves the use of the readily available reagents without any prior heating and without the use of any oxidant, catalyst and process of extraction. The short analysis time and the least interference due to excipients, good reproducibility and the accuracy of the method is further certified by the statistical parameters and by the recovery experiments performed on different samples. Hence, the present method could be used for the routine quality control in pharmaceutical industry.

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И З В О Д

ОДРЕЂИВАЊЕ ТРАГОВА ИЗОНИЈАЗИДА ПРИМЕНОМ КИНЕТИЧКЕ СПЕКТРОФОТОМЕТРИЈСКЕ МЕТОДЕ

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Развијена је ефикасна и економична спектрофотометријска метода за одређивање трагова изонијазида (INH) у чистој форми, као и у фармацеутским формулацијама, базирана на лиганд-супституционој реакцији између INH и аквапентацијанофератног(II) јона ($[Ru(CN)_5OH_2]^{3-}$) у воденој средини на λ_{max} = 502 nm. Примењено је време процедуре за оптималне реакционе услове. Калибрациона једначина, за апсорбанцију на 502 nm, на фиксираном времену (t_n = 2, 5 и 7 min) и INH у линераном опсегу 1,37 до 27,43 μg mL^{-1}, примењена за одређивање трагова INH, у сагласности је са официналним и објављеним методама. Одређен је проценат приноса у анализи различитих фармацеутских узорака и износи 99–101 %. Резултати предложене методе указује на чињеницу да уобичајени адитиви не узрокују било какве интерференције. Резултати валидације предложене методе показују добру сагласност са истим добијеним официналном методом. Презентована метода је веома једноставна, репродуктивна, осетљива и може се применити за одређивање трагова INH у различитим узорцима без коришћења екстракционих агенаса.

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