Research Article

IR Quantification of Isoxsuprine Hydrochloride in Bulk and Oral Dosage Form

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ABSTRACT

Simple and sensitive Infrared spectrophotometric method have been developed for the estimation of isoxsuprine hydrochloride in tablet dosage form and the Beer's concentration range was found to be 0.5mg-2.0mg. The correlation coefficient for the method was found to be 0.998 and the developed method was analyzed for specificity, limit of detection (LOD), limit of quantification (LOQ), linearity of response, precision and accuracy; thus the proposed method could be adopted for routine analysis of bulk drug and its formulation.

Keywords: Infrared spectroscopy (IR), Beer's law, Limit of detection (LOD), Limit of quantification (LOQ).

INTRODUCTION

Isoxsuprine[1] (ISX), 4-Hydroxy-α-[1-[(1-methyl-2-phenoxy-ethyl)amino]ethylbenzen methanol, is a vasodilator that produces the effects of β-adrenoreceptor stimulation and α-adrenoreceptor antagonism; the former effect is more predominant. It is used in the treatment of cerebral and peripheral vascular diseases. It is also used to arrest premature labour. Several analytical methods (colorimetric methods[2], MS-MS identification[3] in post administration equine urine, spectrophotometric methods[4]) have been reported for the determination of isoxsuprine hydrochloride in raw material, dosage forms and biological fluids. Literature survey revealed that few sophisticated analytical methods have been reported for the estimation of isoxsuprine hydrochloride. The present work aims to devise a novel method by Infrared spectrophotometry (IR) which has not been reported till date.

MATERIALS AND METHODS

All the chemicals used throughout the experiment were of highest purity of (IR grade).

1. Potassium bromide (KBr)

2. Internal standard: potassium thiocyanate (KCNS)

3. Bulk material: sample of isoxsuprine hydrochloride was gifted from Juggat Pharmaceutical Limited.

4. Dosage form: Isoxsuprine hydrochloride tablets was purchased from local market.

Instrumentation

All spectral measurements were made on ABB-IR instrument (model no: MB 3000) with KBr press (model no: M 15).

Method

Calibration of the standard: Potassium thiocyanate was used as an internal standard which was preground, dried, and then reground with dry KBr to make a concentration of about 0.2% by weight of thiocyanate. The final mixture was stored over phosphorus pentoxide. Five different concentration of standard and KBr-KCNS were prepared by mixing known weights of the standard substance with a known weight of the KBr-KCNS mixture and then grinding by using agate mortar & pestle. A standard calibration curve was constructed using absorbance and concentration.

Table 1: Concentration of KBr/KCNS mixture and standard

KBr/KCNS (in mg)	50	50	50	50	50
Standard (in mg)	0.0	0.5	1.0	1.5	2.0

The discs were prepared by using KBr press and the infrared spectrum was recorded in absorbance mode; the calibration curve was obtained by plotting the amplitude of the IR absorption at 2068 cm^{-1} (prominent band) against the concentration of the substance.

Assay[5]

Weighed 20 tablets of isoxsuprine hydrochloride and ground to fine powder. Accurately weighed tablet powder equivalent to 1mg of isoxsuprine hydrochloride was mixed with the KBr/KCNS mixture and then homogenized by using agate mortar & pestle. The final powder was transferred to KBr press to form a disc and the infrared spectrum in absorbance mode was recorded. The sample peak area was interpolated on the respective linearity chart of the isoxsuprine hydrochloride and the concentration was determined.

Recovery Studies

The recovery studies were carried out on spiked samples by adding predetermined amount of standard drugs to the respective sample. About 50 and 100% of standard drugs were added to the sample and the absorbance was measured. The percentage recovery was calculated. The recovery study was performed at two levels to confirm the precision and accuracy of the above said method.

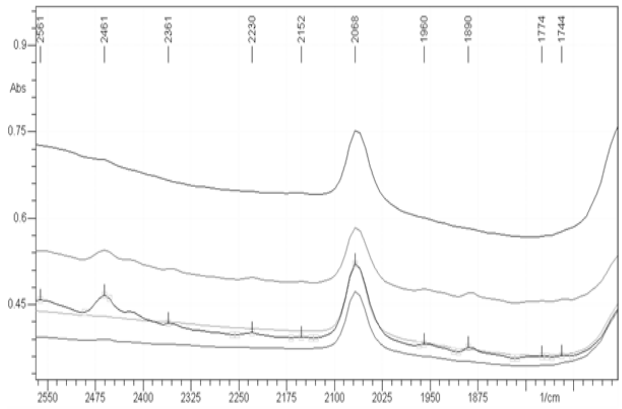

Figure 1: IR Spectra of standard isoxsuprine hydrochloride with internal standard (KBr/KCNS)

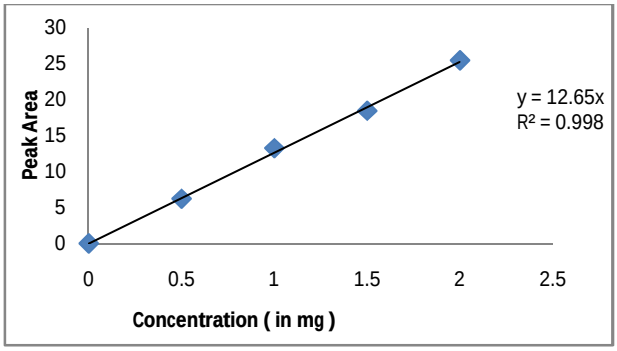

Figure 2: Calibration curve of Isoxsuprine hydrochloride versus Peak area

RESULTS AND DISCUSSION

Isoxsuprine hydrochloride was found to obey Beer's law in the concentration range of 0.5mg-2.0mg. Isoxsuprine hydrochloride showed good linearity as indicated by correlation coefficient value of to 0.998. The optical parameters of isoxsuprine hydrochloride are presented in table no.2. The percentage of the drug in the formulation was calculated and presented in table no.3.

Table 2: Optical parameters of isoxsuprine hydrochloride by IR spectrophotometry

Parameters	Infrared spectroscopy quantification method
Beer's law limit (mg)	0.5-2.0
Regression equation (y= mx+c)	12.65x+0.00
Slope (m)	12.65
Intercept (c)	0.00
Correlation coefficient	0.998

Table 3: Result of tablet Assay and statistical parameters for isoxsuprine hydrochloride by IR spectrophotometry

Method	Label claim	Amount found by proposed method (mg)*	% label claim	SD	SE	(95%)CI	%RSD
KBr Disc method using internal standard.	10mg	9.993	99.93	0.011547	0.006667	0.340042	0.115547

*Each value is a mean of 3 determinations

Table 4: Recovery study for isoxsuprine hydrochloride by infrared spectrophotometric method

Method	Label claim	Amount of drug added (mg)*	Amount of drug recovered (mg)*	% Recovery
Infrared quantification method	10mg	1.5 2	1.498 1.99	99.86 99.50

*Each value is a mean of 3 determinations

The results of the analysis showed that the amount of drug present in the formulation was in good agreement with the label claim of the formulation. The accuracy of the proposed method was determined by recovery study. The recovery studies were carried out on spiked samples at two levels 50%, 100%. The percentage recovered were found to be in the range of 99-100% represented in table.4. Which showed that the excipients in the formulation did not interfere with the analysis. The IR quantification process does not involve prior extraction and is independent of drug materials solubility.

CONCLUSION

The percentage recovery of the method lies between 99-100 %. The correlation coefficient for the method was found to be 0.998 and the recovery studies indicates that there is no interference of other ingredients present in the formulation. Thus the method is simple, precise, accurate, less time consuming and could be used for routine analysis.

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REFERENCES

1. Indian Pharmacopeia: Vol 2; 2010. p.1527-15292.
2. Kalsang K Tharpa, KanaKapura K Basavaiah, HosaKereDoddarevanna HD Revanasiddappa and KanaKapura Basavaiah KB Vinay Spectrophotometric determination of isoxsuprine hydrochloride using 3-methyl-2-benzothiazolinone hydrazone hydrochloride in spiked human urine and Pharmaceuticals. Talanta, 81(4- 5), 2010, 1216-1223.
3. Niraimathi V, Jerad Suresh A, NanjappanK Spectrophotometric estimation of Nislodipine in bulk and tablet dosage formulation. Acta Ciencia Indica, xxxvi, C(4), 2010, 393.
4. Bosken J.M, A F Lehner, A Hunsucker, J D Harkins, W E Woods, W Karpiesiuk, W G Carter, J Boylers , M Fisher,and T Tobin. Direct MS-MS identification of isoxsuprine-glucuronide in post-administration equine urine. Can J Vet Res, 64(2), 2000, 112–116.
5. Andreia de Haro Moreno, Herida Regina NunesSalgado Development and validation of the quantitative analysis of ceftazidime in powder for injection by infrared spectroscopy. Physical Chemistry, 2(1), 2012, 6-11.
6. Vogel's Textbook of Quantitative Chemical Analysis by J mendhan, Denney Thomas, Sixth Edition, New York, Pearson education publisher, 1989, 734-745.

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