



IR Quantification of Isoniazid In Bulk and Oral Dosage Form

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ABSTRACT

Simple and sensitive infrared spectrophotometric method have been developed for the estimation of isoniazid in tablet dosage form and the Beer's concentration range was found to be 0.5mg-2.0 mg. The correlation coefficient for the method was found to be 0.9 and the developed method was analyzed for specificity, 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), Potassium thiocyanate(KSCN), Potassium bromide disc.

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INTRODUCTION

Isoniazid is pyridine-4-carboxylic acid hydrazide, used as an antitubercular drug. It is the drug of choice in the treatment of pulmonary & extrapulmonary tuberculosis¹⁻⁵. It is used with other antitubercular drugs including rifampicin, ethambutol and pyrazinamide. Isoniazid is also used in risk subjects for the prophylaxis of tuberculosis. Literature survey revealed that few sophisticated analytical methods⁽⁶⁻¹¹⁾ inclusive of sensitive HPLC method, difference spectrophotometry and some UV spectrophotometric methods have been reported for raw material, dosage forms and biological fluids. The present work reports on a simple, precise, IR spectrophotometric method for the determination of INH in pure and pharmaceutical formulations which has not been reported till date. The structure of isoniazid was given in Figure.1.

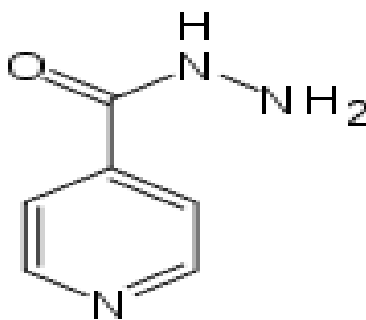


Figure .1: The structure of isoniazid

MATERIALS AND METHOD

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

- Potassium bromide (KBr)
- Internal standard: Potassium thiocyanate (KSCN)
- Bulk material: sample of isoniazid was gifted from Medopharm pharmaceuticals.
- Dosage form: Isoniazid 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)¹²⁻¹⁷

Method

Calibration of the standard :

Potassium thiocyanate was used as an internal standard which was pre-ground, dried, and then reground with dry KBr to make a concentration of about 0.2% by weight of potassium thiocyanate. The final mixture was stored over phosphorus pentoxide. Five different

concentration of standard and KBr-KSCN were prepared by mixing known weights of the standard substance with a known weight of the KBr-KSCN mixture and then grinded by using agate mortar & pestle under IR lamp. A standard calibration curve was constructed using peak area and concentration as presented in Figure.2 and the values obtained by proposed methods are presented in Table 1.

Table 1 : Concentration of KBr/KSCN mixture and standard

| | | | | | |
|-------------------|-----|-----|-----|-----|-----|
| KBr/KSCN (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 2067.54 cm⁻¹ (prominent band) against the concentration of the substance .

Assay

10 tablets of isoniazid were weighed and ground to fine powder. Accurately weighed tablet powder equivalent to 10mg of isoniazid is dissolved in 100ml of ethanol to make a concentration of 100 µg/ml. From that 10 ml of solution which was equivalent to 1mg was taken in a porcelain dish and evaporated. Then it was mixed with the KBr /KSCN mixture and then homogenized by using agate mortar & pestle under IR lamp. The final powder was transferred to KBr press to form a disc and the infrared spectrum in absorbance mode was recorded. The ratio in sample peak area was interpolated on the respective linearity chart of the isoniazid 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.

RESULTS AND DISCUSSION

Isoniazid was found to obey Beer's law in the concentration range of 0.5mg-2.0mg. Isoniazid showed good linearity as indicated by correlation coefficient value (0.9). 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%.

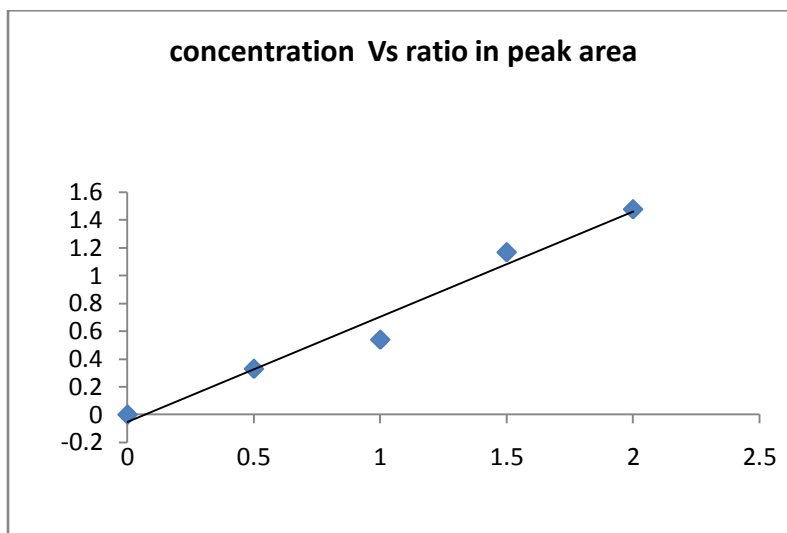


Figure 2 : Calibration graph

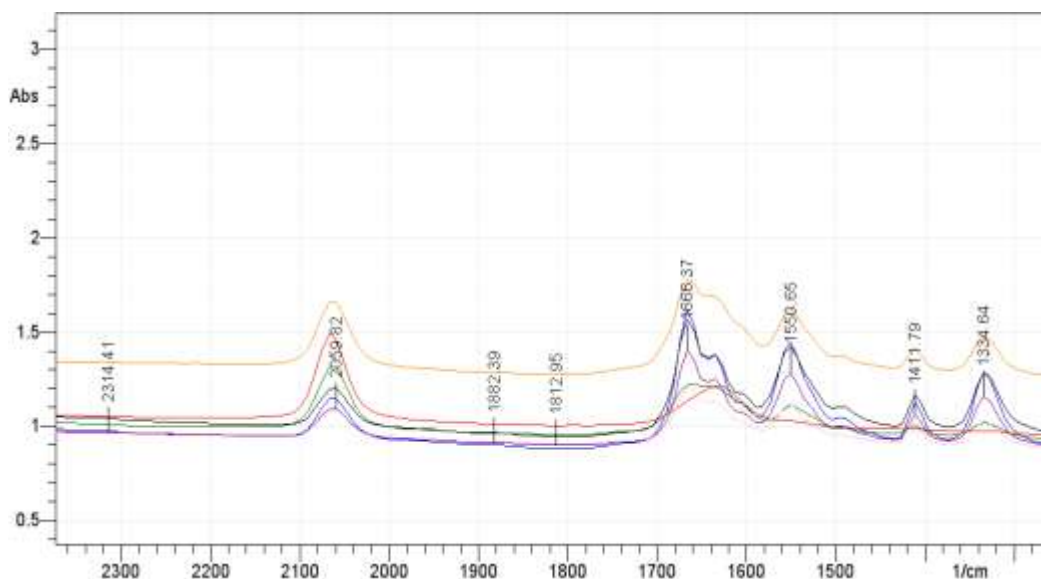


Figure. 3 : IR spectra of standard isoniazid with KBr disc method using internal method

The percentage recovered were found to be in the range of 95-100% as represented in **Table 3**. The optical characteristics such as Beer's law limits and Sandell's sensitivity, the regression characteristics like slope (m), intercept (c), correlation co-efficient (r) were calculated and the results were summarized in the Table- 4. The IR quantification process does not involve prior extraction and is independent of drug material.

Table 2 : Concentration of a Peak area at 2059.82 cm^{-1} wave number

| Concentration | Absorbance |
|---------------|------------|
| 0.5 mg | 0.33 |
| 1.0 mg | 0.54 |
| 1.5 mg | 1.17 |
| 2.0 mg | 1.48 |

*Each value is the mean of three readings.

Table 3 : Recovery study for INH by Infrared spectrophotometric method

| S.N O | Method | Label claim | Amount of drug added (mg)* | Amount of drug recovered (mg)* | % Recovery |
|----------|---|-------------|-----------------------------------|-----------------------------------|---------------|
| 1 | KBr disc method using internal standard | 300mg | 1.5 | 1.47 | 98.29 |
| | | | 2.0 | 1.97 | 98.65 |

*Each value is the mean of three readings

Table 4 : Optical parameters for the proposed methods

| S.NO | Parameters | Infrared method |
|------|--------------------------------------|---------------------|
| 1 | Beer's law limit (mg) | 0.5- 2.0 |
| 2 | Regression equation ($y= m x + c$) | $0.76 x + (-0.056)$ |
| 3 | Slope (m) | 0.76 |
| 4 | Intercept (c) | - 0.056 |
| 5 | Correlation coefficient | 0.987189 |
| 6 | Sandell's | 0.0012 |

CONCLUSION

The percentage recovery of the method lies between 95- 100 %. The correlation coefficient for the method was found to be 0.9 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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