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NEW ANALYTICAL METHOD DEVELOPMENT AND VALIDATION OF UV -VISIBLE SPECTROSCOPY ANDUSING FTIR STUDY OF CHEMICAL STRUCTURE OF SULPIRIDE

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ABSTRACT

Development and implementation of a simple, precise, and Accurate Zero order spectroscopic method for the estimation of Sulpiride in bulk and Tablet dosage form. The drug indicates the maximum absorption (λ_{max}) at 212 nm in 0.2N HCl solutions and AUC in the absorption spectrum is measured in the wavelength range from 207 to 218 nm at a concentration range of 1-5µg/ml according to Beer's law. Linearity analysis showed an R^2 value of 0.998. The LOD & LOQ were found to be 0.0110 and 0.33 µg/ml. The %RSD was found less than 2%. The method has been validated according to Linearity, Precision, Accuracy, Robustness, Ruggedness, LOD and LOQ according to ICH guidelines. This method obeys the Beer's law and met all the required specifications within the limit. FTIR analysis of Sulpiride active pharmaceutical ingredient gives different peaks of wavenumbers. FTIR data interpretation also gives information about purity and quality of sample and it also meets the required specifications.

KEYWORDS: Sulpiride, UV Spectroscopy, FTIR, ICH Guidelines.

INTRODUCTION

Sulpiride belongs to the Benzamide class of Antipsychotic drugs and is primarily used in the treatment of schizophrenia and depression. It acts as a selective dopamine receptor antagonist, mainly affecting the limbic system while sparing the motor system, thus reducing the risk of extrapyramidal side effects. Sulpiride exhibits both antipsychotic and antidepressant properties, and it is used in the management of acute and chronic schizophrenia, vertigo, and certain cases of psychosomatic disorders. Its therapeutic effects are particularly useful in treating negative symptoms of schizophrenia and dysthymia. Moreover, Sulpiride is often employed in low doses for the treatment of functional gastrointestinal disorders due to its Prokinetic properties. Its efficacy and tolerability make it a valuable choice in both psychiatric and gastroenterological settings.

This fundamental law governs UV-Vis spectrophotometry, relating the absorbance (A) of a solution to its molar absorptivity (a), the path length of the light through the sample (b), and the concentration of the analyte (c). The formula is **A = a * b * c**.

The instrument measures the light intensity after it passes through a sample and compares it to the initial intensity of the light source. This process is repeated across a range of wavelengths, generating a UV-Vis spectrum that shows the wavelengths of light absorbed and their respective intensities.

1. MATERIALS AND METHODS

Instrument: UV-Visible double beam spectrophotometer, SHIMADZU (model UV-1800) with UV probe software. All weights were taken in analytical balance.

Chemicals: Sulpiride was given as a gift sample by Pharmaceutical manufacturing company Medreich Pvt LTD, Bengaluru.

Solvent: 0.2 N HCl is used as a solvent and distilled water and Concentrated Hydrochloric acid. (Analytical grade). All the reagents and chemicals are provided by our Institution.

Selection of analytical wavelength: Appropriate dilutions were prepared for drug from the standard stock solution and the solution was scanned in the wavelength range of 200-400 nm. The trial and error method[1] gives absorption spectra in 0.2 N HCl acid and it shows maximum absorbance at 212 nm & Good solubility and Stability.

2. PREPARATIONS OF ANALYTICAL SAMPLES

a) **Preparation of 0.2 N HCl solution:** Measure accurately about 8.26ml of Concentrated Hydrochloric acid (HCl) of having 37.2% w/w concentration and transferred into a 500 ml volumetric flask then make up the volume up to 500 ml by using Distilled water to get a 0.2 N HCl solution.

b) **Preparation of standard stock solution:** Accurately weigh 100mg of Sulpiride[3] was transferred into 100ml volumetric flask and diluted with 0.2N HCl up to the mark. From this pipette out 10ml into 100ml volumetric flask and diluted with 0.2N HCl up to the mark, from this solution pipette out **(1, 2, 3, 4 and 5ml)** into 10ml individual volumetric flask and add 0.2N HCl up to the mark, this gives **1 , 2 , 3, 4, & 5 µg/ml** concentrations.

c) **Preparation of sample solution:** Sulpiride API was obtained from Medreich Pvt LTD, Bengaluru. From this weighed 100mg of drug and transferred into 100 ml volumetric flask then it was diluted with the 0.2N HCl solution and made up to the mark and the solution was filtered through what man filter paper NO. 41. From the above solution 10 ml was pipette out into 100 ml volumetric flask and the volume was made up to the mark with 0.2N HCl. The final concentration of Sulpiride was brought to 100µg/ml. From that above solution make required concentrations of **1, 2, 3, 4, & 5 µg/ml** concentrations.

1. METHOD AND VALIDATION

The method was validated according to the ICH guidelines[16] of Q2R2 & ICH Q14.

2. RESULTS AND DISCUSSION

Method: Zero order derivative spectroscopy

Linearity: The linearity of an analytical method is its dimension to show the test results that are directly proportional to the concentration of the analytic in the sample within the determined range. The linearity was established in the range of 1-5µg/ml was measured at 212nm and absorbance values are shown in (Table-02). The calibration curve was prepared by plotting graph against the concentration and absorbance and therefore the graph shown in Fig-05. Statistical variables like slope, intercept, regression equation, correlation coefficient and sandell's sensitivity were determined[16] (Table-03).

Precision: The precision of an analytical method expresses the closeness of a series of individual analytic measurements obtained from multiple sampling of the equivalent sample. Precision was established by intra-day and inter-day studies. Intra-day precision was determined by analyzing the same concentration for five times within same day. Inter-day precision was determined by analyzing the same concentration daily for five days[16] (Table-04).

Ruggedness: The ruggedness is defined as the reliability of results when the method is performed under the variation in conditions. This includes distinct analyst, laboratories, instruments, temperature etc. Ruggedness was determined between distinct analysts, the value of %RSD was found to be less than 2.[16] (Table-05).

LOD and LOQ: The limit of detection is an individual analytical method is the smallest amount of analytic in a sample which can be reliably detected by the analytical method. The limit of quantitation is a discrete analytical procedure is the smallest amount of analytic in a sample which can be quantitatively determined. LOD and LOQ were calculated by using following formula.

LOD = 3.3(SD)/S and LOQ = 3(LOD)

LOD and LOQ value of were found Sulpiride 0.029 and 0.089µg/ml.

FTIR: Fourier Transform Infrared (FTIR) spectroscopy has emerged as a powerful analytical tool in medical research, offering non-invasive and precise examination of the molecular composition of biological samples. The primary objective of this review is to underscore the benefits of FTIR spectroscopy in medicinal research, emphasizing its ability to delineate molecular fingerprints and assist in the identification of biochemical structures and key peaks in biological samples.

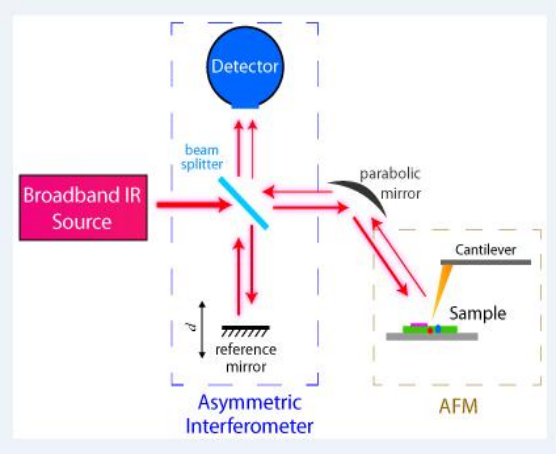

Fig. No, 01: FTIR Interferometer diagram.

FTIR STUDY & INTERPRETATION OF PURE SULPIRIDE

FTIR stands for **Fourier Transform Infrared Spectroscopy**. It is an analytical technique used to identify organic, polymeric, and, in some cases, inorganic materials by measuring how infrared light is absorbed by a sample. When a sample is exposed to infrared radiation, different chemical bonds vibrate at characteristic frequencies, absorbing specific wavelengths of the IR light. The resulting absorption spectrum acts as a molecular "fingerprint" that can be used to identify and analyze the material.

The infrared spectra of Sulpiride pure drug were recorded at a range of 400-4000 cm−1 on QATR-S IR Spirit **FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR)**[17] spectra of the pure Sulpiride show spectrum peak points as below Table No. 01.

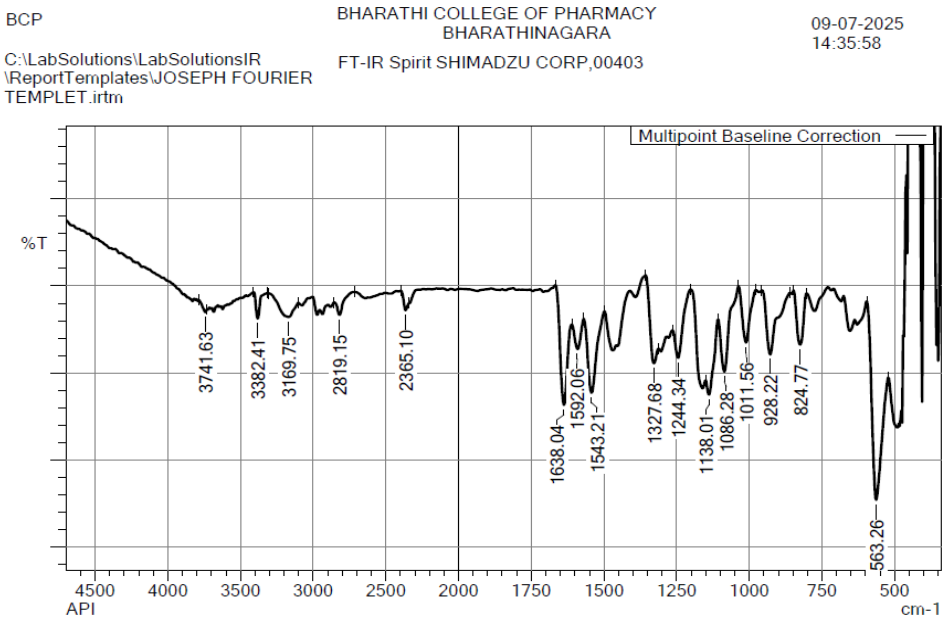

Fig.02: IR Spectra of Sulpiride drug.

Table 01: Interpretation of FTIR data of Sulpiride.[17-19]

Group frequency range(cm^{-1})	Peak obtained range (cm^{-1})	Confirmation of functional groups
1626-1662 cm^{-1}	1638.04 cm^{-1}	Presence of C=C Alkene (M)
790-830 cm^{-1}	824.77 cm^{-1}	Presence of C-H in bending (Disubstituted Benzene)
1089cm^{-1} -1332 cm^{-1}	1086 cm^{-1} &1327 cm^{-1}	Symmetric and Assymetric SO_2 –Sulphonyl group
2815-2850 cm^{-1}	2819 cm^{-1}	Presence of methoxy ($-OCH_3$)
1590-1650	1543 cm^{-1}	Primary amine, NH bend
3500-3300 cm^{-1}	3382.41 cm^{-1}	N-H Streching 'M' (Aliphatic primary amines)

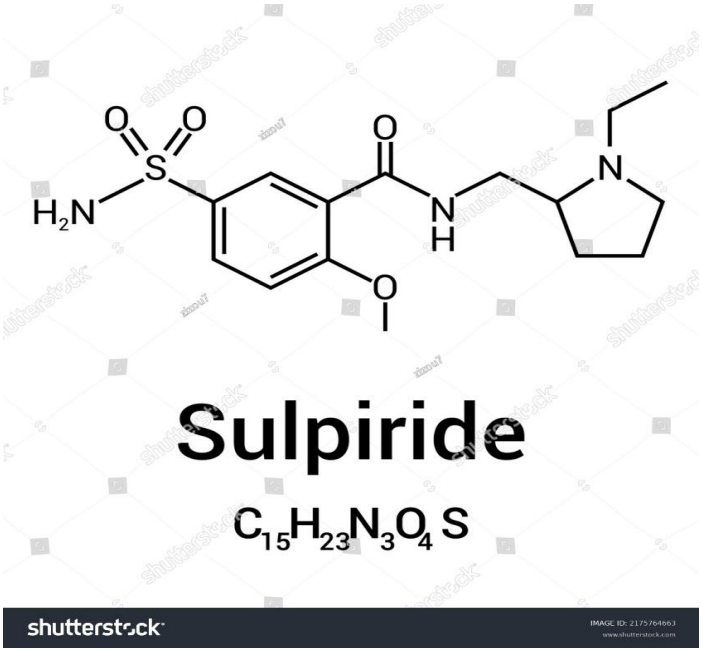

Fig. No. 03: Structure of Sulpiride.

Table 02: Results of calibration curve at 212nm by zero order spectroscopy.

SL NO	Concentration in µg/ml	Absorbance ± Standard deviation*
01	1	0.1445±0.00263
02	2	0.3153±0.004028
03	3	0.4625±0.001893
04	4	0.5723±0.006549
05	5	0.71683±0.00508

**Average of six determinations.*

Table 03: Regression parameter Sulpiride for by zero order spectroscopy.

Regression parameter	Results
Range(µg/ml)	1 – 5
λ_{max}(nm)	212 nm
RegressionEquation	y = 0.1383x+ 0.0067
Slope(b)	0.1383
Intercept(a)	0.0067
Correlationcoefficient(r^2)	0.9987
Sandells sensitivity(µg/cm^2)	0.0064
Limit of detection(µg/ml)	0.029
Limit of quantitation(µg/ml)	0.089

** All the values are there with in the Specified standards.*

Table 04: Determination of precision results for Sulpiride at 212 nm by zero order spectroscopy.

Concentration (µg/ml)	Intra-day Absorbance ±SD**	%RSD	Inter-day Absorbance ±SD**	%RSD
1	0.146±0.00216	1.479	0.133±0.002409	1.811
2	0.314±0.00414	1.318	0.306±0.001886	0.616
3	0.465±0.002427	0.521	0.433±0.00256	0.591
4	0.580±0.00415	0.715	0.556±0.002544	0.457
5	0.717±0.004933	0.688	0.691±0.003145	0.455

***Average of five determinations, **percentage relative standard deviation.*

Table 05: Determination of Ruggedness results for Sulpiride at 212 nm by Zero order spectroscopy.

Analysts	Analyst 1	Analyst 2
Mean absorbance	0.465	0.463
±Standard deviation*	0.002427	0.002986
%RSD	0.521	0.644

***Average of six determinations, **percentage relative standard deviation.*

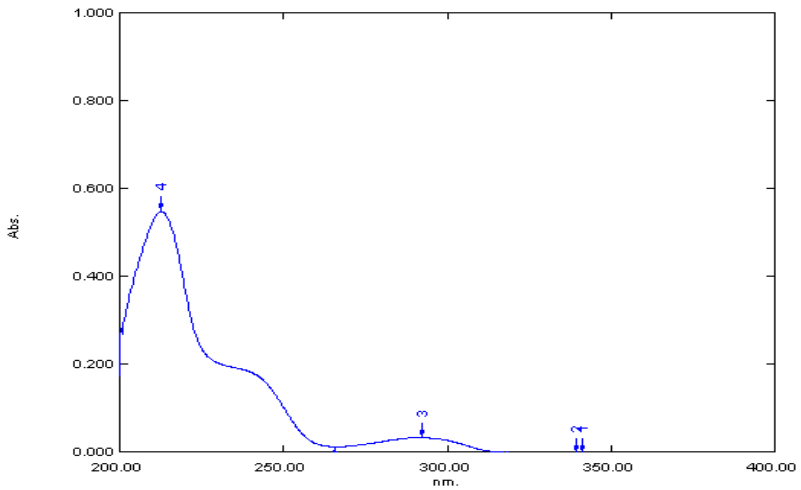

Fig.04: Zero order spectrum of Sulpiride (3µg/ml) at 212nm.

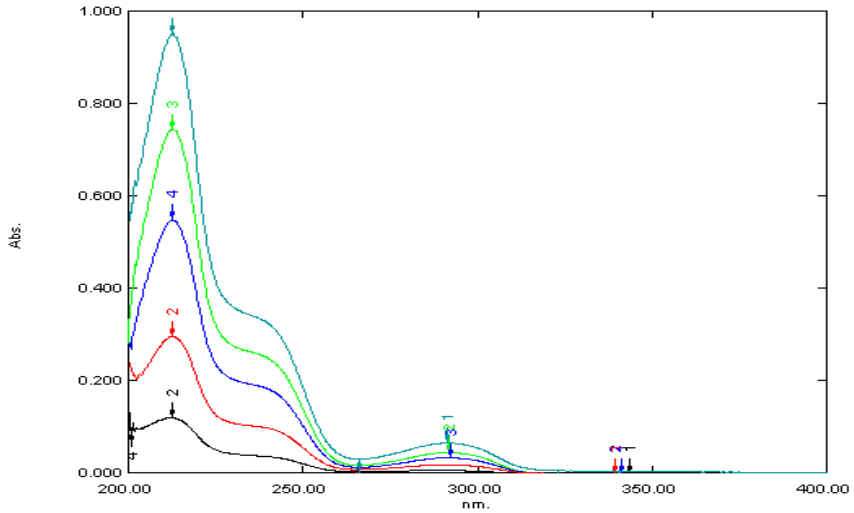

Fig.05: Zero order overlain spectra of Sulpiride showing absorbance at 212nm.

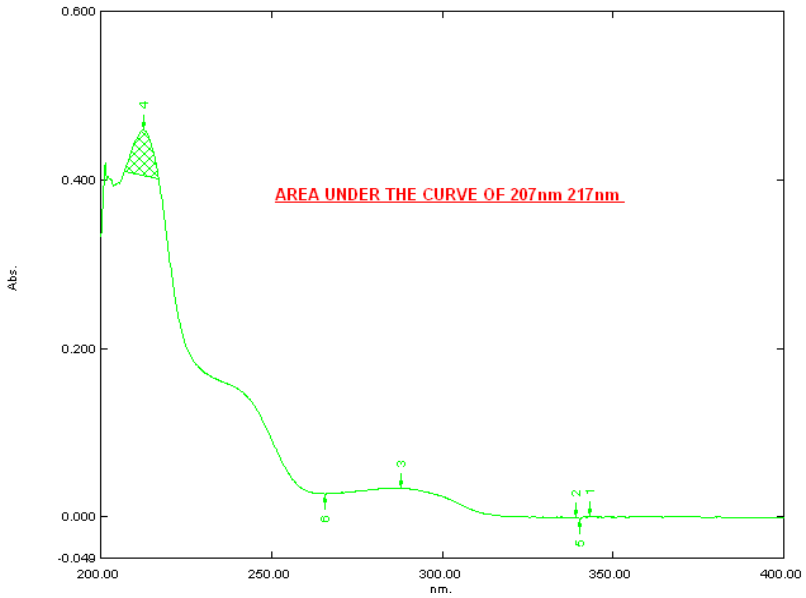

Fig.06: Area under the curve spectra of Sulpiride showing peak area at 207-217nm.

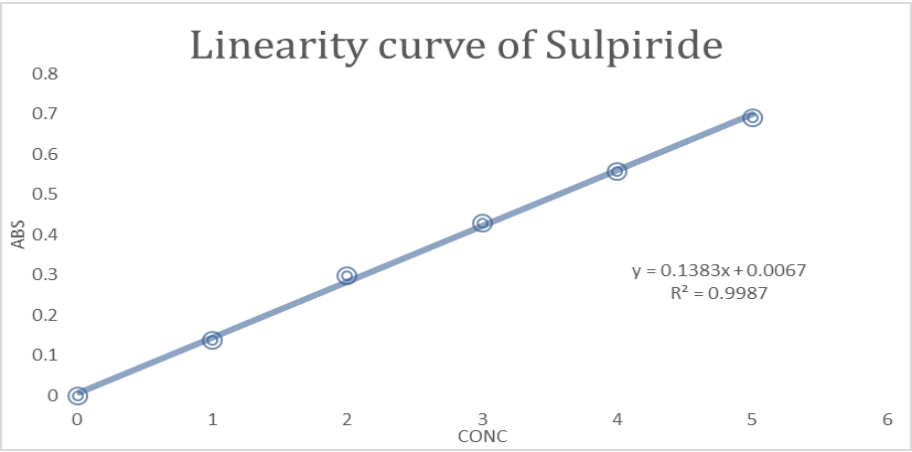

Fig.07: Calibration curve of Sulpiride by zero order spectroscopy.

CONCLUSION

According to the ICH guidelines[16], this method was validated. It is a simple, accurate, precise, and reproducible method for estimating and validating the Sulpiride drug using a UV-Vis Spectrophotometer. It also meets the required specifications of a standard

reference Infrared Spectrum (IR) and provides pivotal insights into the structure, functional groups, & Interpretation. This method is novel and less expensive compared to other developments, which we refer to as literature reviews.

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