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ORIGINAL ARTICLE

UV Method Development and Validation of Eperisone Hydrochloride in Bulk and Tablet Formulation

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ABSTRACT

Objective: To develop and validate a UV simple, precise and cost saving method for fast analysis of eperisone hydrochloride in eperisone tablets.

Method: The test is developed by using methanol as diluent, performed on UV visible double beam spectrophotometer at range of 200nm-400nm, and found absorbance on maximum wavelength of 260 nm. The test is then validated by using complete parameters of method validation i.e., linearity, precision, selectivity, accuracy, repeatability, robustness, limit of quantitation and detection. The complete procedure is carried out as per United State Pharmacopeia, ICH and WHO guidelines.

Results: Calculate individually all parameters of method validation as per standard that should meet the validation criteria.

Conclusion: As per above discussion the method is precise and accurate for analysis of eperisone hydrochloride in eperisone hydrochloride tablets.

INTRODUCTION

Eperisone hydrochloride which is chemically known as 4'-ethyl-2-methyl-3-piperidino propiophenone hydrochloride, has chemical formula $C_{17}H_{25}NO$ and belongs to the class of muscular relaxants, having molar mass 259.387g/mol [1] (Figure **1**). The drug acts by acting on central nervous stem cells providing relaxation of both skeletal and vascular smooth muscles [2]. Oral eperisone is effectively used three times daily (t.i.d) at dosage regimen of 100 mg. It is well known for use in the treatment of muscular spasm, lower back pain, cervical spondolysis, and in spastic paralysis in terms of cerebrovascular disease. The drug is well tolerated at doses of with mild GI symptoms involving nausea, abdominal cramps, headache and dizziness are the commonly observed adverse effects [2-4]. The drug is rapid absorption after oral administration. It has biological half-life of about 1-4.3 hour, its rapid elimination rules out risk of accumulation [5].

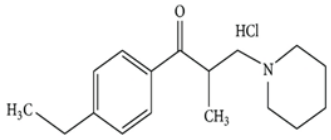

Figure 1. Structure of eperisone hydrochloride.

Research data provides different analytical methods for the estimation of eperisone hydrochloride in single and combined dosage forms such as with Diclofenac sodium and Paracetamol [6] through spectrophotometer [7] and HPLC [8, 9]. The drug is also estimated in human plasma through mass spectrometer [10]. Moreover, these analytical techniques of high-performance liquid chromatography and mass spectrometry (HPLC/MS). The analysis has been also made through gas chromatography/ mass spectrometry (GC/MS) and nuclear magnetic resonance (NMR) have also been acquired for identification of degradation products for the purpose of stability studies in eperisone hydrochloride tablets [11]. However, our study aims for the development and validation of a simple method which is accurate, precise and cost saving for fast analysis of eperisone hydrochloride in eperisone tablets.

EXPERIMENTAL

Instrumentation and Chromatographic Conditions

The test is carried out by using UV/visible spectrophotometer 1700 by setting wavelength range from 200nm-400nm, found wavelength maximum is 260nm. The test fulfills system suitability requirements having RSD not more than 2.0%. Balance Mettler Toledo AG285, ultrasonic bath and magnetic stirrer.

Reagents and Chemicals

A simple method performed by using methanol as diluent using UV/Visible spectrophotometer.

Preparation of Standard Stock Solution

Weigh about 50mg of working standard of eperisone hydrochloride and transfer to 100ml volumetric flask, add 80ml of diluent and dissolve by keeping in ultrasonic bath for 5 minutes. Cool at room temperature and makeup the volume to the mark with methanol. Transfer 5ml of the above solution to 50ml volumetric flask and makeup the volume with, Solution having concentration of 50ppm.

RESULTS

Method Validation

After development it is necessary to perform validation of analytical method by following all parameters of method validation that is linearity, precision, selectivity, accuracy, and robustness, limit of detection and limit of quantification. As per method validation procedure provided in ICH guidelines [12], following steps were carried out.

Linearity

Linearity test is carried out by making solutions of five different concentrations range from 0.008mg/ml to 0.012mg/ml. The linear relationship between concentrations of solutions and instrument response against specific concentrations is confirmed by plotting graph between varying concentrations of analyte in solutions of linearity test and their absorbance (Table **1**). The R^2 of calibration for data point is calculated to be 0.9998 (Figure **2**).

Table 1. Concentration and absorbance of analyte.

S. No.	Concentration of analyte solution	Absorbance of analyte solution
1	0.008 mg / ml	0.4318
2	0.009 mg / ml	0.4858
3	0.010 mg / ml	0.5398
4	0.011 mg / ml	0.5937
5	0.012 mg / ml	0.6477

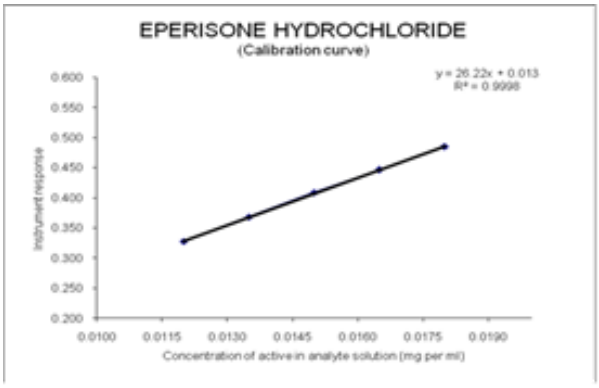

Figure 2. Linearity of eperisone HCl.

A linear relationship has been observed in calibration graph between the absorbance and concentration of analyte in the solution.

Precision

The precision is categorized into two categories.

Repeatability

The test is carried out by making six samples containing analyte of 100% label claim, and analyze against 5 replicates of reference standard, calculate individual assay and RSD of six replicate assays (Table **2**).

Intermediate Precision

Intermediate precision performs by 2 analysts, analyst-1 and analyst-2. It is carried out by preparing 5 samples assayed individually containing 100% of label claim (Table **3a**, **3b**).

Table 2: Assay.

S. No.	Weight of sample (mg)	Assay (mg / tablet)	Assay (% of LC)	Mean	STD %	RSD %
1	160.0	50.13	100.25	50.24 mg/tablet (100.48% of label claim)	0.1356	0.2698%
2	160.4	50.02	100.04			
3	160.9	50.31	100.63			
4	160.8	50.33	100.66			
5	160.2	50.35	100.70			

Table 3a. Analyst-1.

S. No.	Weight of sample (mg)	Assay (mg / tablet)	Assay (% of LC)	Mean	STD %	RSD %
1	160.8	50.72	101.44	50.58 mg/tablet (101.15% of label claim)	0.0892	0.01763%
2	160.7	50.55	101.10			
3	160.6	50.48	100.96			
4	160.8	50.54	101.08			
5	160.9	50.59	101.19			

Table 3b. Analyst-2.

S. No.	Weight of sample (mg)	Assay (mg / tablet)	Assay (% of LC)	Mean	STD %	RSD %
1	161.0	50.08	100.15	50.22 mg/tablet (100.45% of label claim)	0.0870	0.1732%
2	160.7	50.28	100.56			
3	160.8	50.29	100.57			
4	160.6	50.26	100.52			
5	160.8	50.21	100.42			

Table 4. % Recovery.

Theoretical contents of active added in placebo		Assay (mg / Tablet)	Assay (% of label claim)	Difference (%)	Mean difference (%)	Standard Deviation	% recovery
Placebo with 80% of active							
	1.	40.08	80.15	0.152		0.152	100.19
	2.	40.05	80.11	0.106	0.12	0.106	100.13
	3.	40.05	80.10	0.098		0.098	100.12
Placebo with 100% of active							
	1.	50.16	100.33	0.326		0.326	100.33
	2.	50.25	100.50	0.496	0.50	0.496	100.50
	3.	50.34	100.67	0.672		0.672	100.67
Placebo with 120% of active							
	1.	60.33	120.66	0.658		0.658	100.55
	2.	60.14	120.28	0.283	0.59	0.283	100.24
	3.	60.41	120.82	0.825		0.825	100.69

ACCURACY

In accuracy the test method is determined by making spike samples of different concentration i.e., 80%, 100% and 120%. Three samples of each concentration will be prepared according to the procedure (Table **4**).

ROBUSTNESS

The test is performed by making deliberately change in the procedure, to check either the method is stable with slight variations or not. In this procedure test is performed by preparing samples of 100% label claim. Samples kept on 4°C, 25°C and on 35°C for 4hrs and assayed according to the test procedure (Table **5**).

Table 5: Assay in different storage condition.

Storage Condition	Assay (mg/ tablet)	Assay (% of label claim)
4°C	50.15	100.29
25°C (ambient temperature)	50.14	100.27
35°C	50.05	100.10

Selectivity

Selectivity is referred to the ability of test method to detect and quantify the analyte accurately in the presence of impurities (as excipients) used in the formulation. This was established by record the absorbance the diluent (used for preparing test and reference solutions), diluent and the dilution of placebo. The absorbance was recorded. It is also called force degradation test because, acid, base, oxidative and reductive hydrolysis carried out.

Limit of quantitation (loq)

Limit of quantitation for eperisone hydrochloride found is 2.5 ppm.

Limit of detection (lod)

Limit of detection observed for eperisone hydrochloride is 1.0 ppm.

DISCUSSION

All parameters performed in method validation which include linearity, precision, accuracy, robustness, selectivity, limit of quantitation, and detection have been found within the limits. In linearity value of correlation coefficient between response and concentration values found R^2 value of 0.9998, that complies the limit that is $R^2 > 0.9995$, while in repeatability rsd of all six replicates found 0.2698% as well as in intermediate precision all 5 replicates of analyst-

1 found RSD 0.01763% and RSD of analyst-2 is 0.1732%. In accuracy spiked samples shows the contents are equivalent to the theoretical contents in placebo, the deviations found are within the usual limits. In robustness the samples kept on different temperatures as low as 4°C and as high has 35°C, shows no significant change, and all the values are within the limits. The minimum amounts of analyte that can be detected and quantified are determined. In selectivity there is no change in absorbance found during force degradation test.

CONCLUSION

Based on the satisfactory results of all performance parameters the method is validated and found accurate, fast and precise for the estimation of eperisone hydrochloride in bulk and tablet formulation through UV Spectrophotometer.

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