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Validation of omeprazole from pharmaceutical dosages by non-aqueous potentiometric titration method

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Abstract

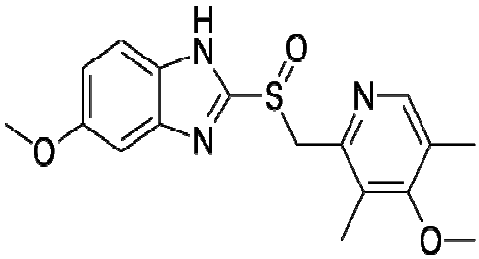
A validation method of omeprazole was developed by non-aqueous potentiometric titration method from API and dosages. The titration technique has developed with 0.1 N perchloric acid which was standardized by potassium hydrogen phthalate. The precise study was found with % Relative Standard Deviation which is less than one where no of observations were six. The method was observed with linearity and Correlation Coefficient as $r^2 > 0.9999$ for 100 mg of drug substance weight in 20% to 100%. The recovery in percentage of omeprazole was observed with 99.90 to 100.27%. The method has ruggedness in variety of conditions like different batches of reagents and titrators of different manufactures.

Keywords: Omeprazole, glacial acetic acid, potassium hydrogen phthalate, perchloric acid.

Introduction

Omeprazole is (RS)-6-methoxy-2-((4-methoxy-3,5-dimethyl pyridin-2-yl)methylsulfinyl)-1H-benzo(d)imidazole. It shows 'PPI' i.e. is proton pump inhibition property. It inhibits the hydrogen-potassium adenosine triphosphatase gastric acid secretion by of the gastric parietal cell.

HPLC[1-6], UVspectrophotometric methods[7] and miscellaneous[8-12] were reported for determination of omeprazole in pharmaceutical dosage. For validation of omeprazole, a new non aqueous potentiometric method was suggested for quantification of API and its formulations.



Scheme-1: Structure of Omeprazole.

Methodology

Instrumentation: A Lab- India auto titrator was used for development of validation method.

All weighing of standard and sample was done on Simadzu make, analytical balance.

Chemicals: Omeprazole of known purity was used as standard. All A.R. grade reagents were utilized in assay development.

Different steps in procedure: 0.1N perchloric acid Standardization: A previously powdered, dried at 120°C for 2 hours of potassium hydrogen phthalate of 350mg was weighed accurately. It was transferred into titration vessel. It was titrated with o.1N Perchloric acid in presence of 50ml of glacial acetic acid in auto titrator to determine exact normality of perchloric acid.

For standardization of perchloric acid the equivalent weight of potassium hydrogen phthalate ($C_8H_5KO_4$) is taken as 0.2042.

$$\text{Normality of perchloric acid} = \frac{\text{Amount used for titration}}{\text{Constant burette reading}} x \text{ Equivalent weight of } (C_8H_5KO_4)$$

Quantitative determination of omeprazole: In various replicate (n= 6) 0.100g. of omeprazole were titrated with 0.1N perchloric acid in auto titrator in presence of 35ml of glacial acetic acid as solvent.

For determination of purity of omeprazole equivalent weight of omeprazole was taken as 0.1151g. The purity in % (Percentage) of Omeprazole was given by equation as:

$$\% \text{ assay} = \frac{\text{Constant burette reading x exact normality of titrant x 0.1151 x 100}}{\text{Weight (omeprazole taken)}}$$

Result and discussion

Determination of omeprazole from formulation: A aim of such newly developed has to determined the amount of omeprazole present in sample. The validation of omeprazole for batches of omeprazole in formulation has carried by using the new method. The range observed with 99.90 to 100.27 %.

Precision with replicate analysis: The precision study was performed for test sample of omeprazole.

The % COV value is 0.1342 showed good precision of the method. The Table-1 shows method precision study.

Table-1: Method of precision with replicate analysis.

Omeprazole (g)	Instrumental values (ml)	Exact Normality of perchloric acid used in titration	Assay (%)
0.1	8.69	0.09991	99.931
0.1	8.71	0.09991	100.161
0.1	8.7	0.09991	100.046
0.1	8.71	0.09991	100.161
0.1	8.72	0.09991	100.276
0.1	8.72	0.09991	100.276
		Average	100.142
		STD	0.1344
		% COV	0.1342

Study of Linearity omeprazole in different amount of drug: A 0.02, 0.04, 0.06, 0.08 and 0.1g as replicates of test samples were used in linearity study. The result of replicate values are given in Table-2.

The auto titration performed at each level of linearity sample hence the graph was plotted for linearity study. The regression equation was given y = 87.05x-0.001 and graph is given in Figure-1.

Table-2: Linearity study of omeprazole in different amount of drug.

Omeprazole (g)	Instrumental Values (ml)	Exact Normality of perchloric acid used in titration	Assay (%)
0.02	1.74	0.09991	100.046
0.04	3.48	0.09991	100.046
0.06	5.23	0.09991	100.238
0.08	6.95	0.09991	99.903
0.1	8.71	0.09991	100.161
		Average	100.079
		STD	0.1277
		% COV	0.1276

Accuracy and recovery study of drug in 3 replicate analysis: Accuracy study was performed at 0.02, 0.04, 0.06, 0.08 % and 0.100 g of the standard in triplicate titration. Instrument reading (values) were noted. The instrument reading obtained in linearity study was used for percentage (%) recovery study. For percentage recovery calculation following equation was used.

$$\text{Percentage recovery} = \frac{\text{Observed Instrumental reading}}{\text{Mean reading}} x100$$

The values of percentage recovery in Table-3 of omeprazole were in 99.747 - 100.325%.

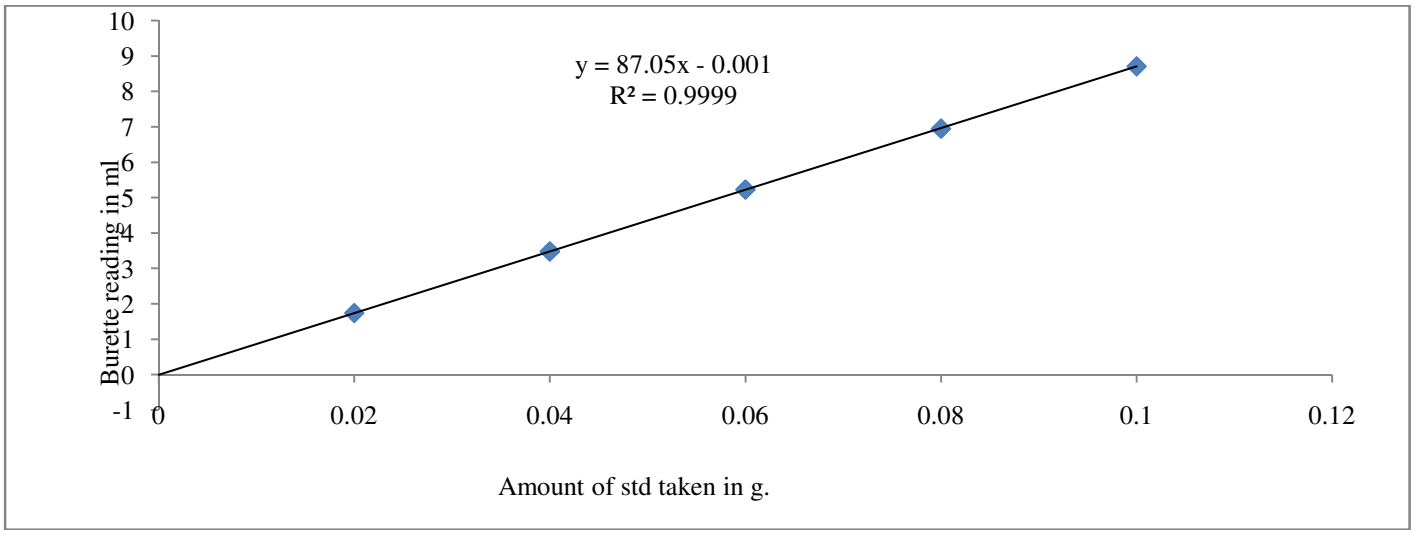


Figure-1: Linearity graph omeprazole in different amount of drug.

Table-3: The data of recovery study of drug in 3 replicate analysis.

Steps	Instrument reading, ml	Omeprazole added in titration jar, g	Omeprazole found by calculation, g	Values of Assay, %	Values of Assay, % (Mean)
1	1.74	0.02	0.0200	100.046	100.046
	1.75	0.02	0.0201	100.621	
	1.73	0.02	0.01977	99.4718	
2	3.48	0.04	0.04023	100.0468	100.142
	3.49	0.04	0.04011	100.3343	
	3.48	0.04	0.03988	100.0468	
3	5.24	0.06	0.06022	100.4301	100.238
	5.23	0.06	0.05988	100.2385	
	5.22	0.06	0.05988	100.0468	
4	6.96	0.08	0.08000	100.0468	99.903
	6.94	0.08	0.07977	99.75938	
	6.95	0.08	0.08011	99.90313	
5	8.7	0.1	0.10014	100.0468	100.0468
	8.71	0.1	0.10011	100.1618	
	8.69	0.1	0.09977	99.93188	

For reproducibility study, ruggedness of the method of omeprazole sample was studied under different conditions such as laboratories, analysts and reagents. Quantification of omeprazole was conducted potentiometrically on different conditions lay down by ICH.

Conclusion

The newly developed method of auto titration i.e. non aqueous, has found to be rugged. The Table- 1, 2 and 3 shows good percentage recovery and low values standard deviation of new method. The values in different table has data for various study parameters essential for of validation of any drug. Therefore such non aqueous auto titration method is useful in validation of omeprazole of in API and formulations.

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