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SENSITIVE AND VALIDATED U.V.SPECTROPHOTOMETRIC METHODS FOR THE ESTIMATION OF NIMESULIDE IN PHARMACEUTICAL AND BULK FORMULATIONS

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ABSTRACT

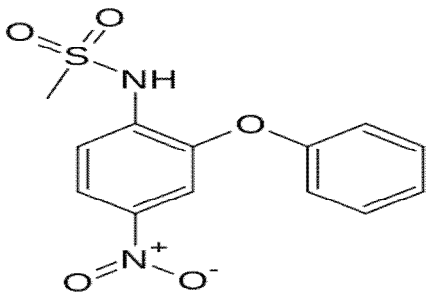
Two simple precise and accurate U.V Spectrophotometric methods were developed and validated according to ICH guidelines. Method A: methanol was used as a solvent and absorption maximum was found at 297nm.method B: methanol: acetonitrile (50:50) was used as a solvent and absorption maximum was found at 295nm.Validation parameters such as accuracy, linearity, robustness were performed. % assay and %RSD was calculated and results were found within the predetermined limits. This indicated that the proposed methods can be used in routine analysis of Nimesulide in bulk and pharmaceutical formulations.

Keywords: U.V., RSD, acetonitrile.

INTRODUCTION

It was launched in Italy for the first time as Aulin and Mesulid in 1985 and is presently available in more than 50 countries worldwide. Nimesulide is a relatively COX-2 selective, non-steroidal anti-inflammatory drug (NSAID) with analgesic and antipyretic properties. Its approved indications are the treatment of acute pain, the symptomatic treatment of osteoarthritis and primary dysmenorrhoea in adolescents and adults above 12 years old. It has a multifactorial mode of action and is characterized by a fast onset of action.

Structure



[2]Chemical formula:C_{13}-H_{12}-N_2-O_{5-S}

molecular weight:308[2]

[3]Solubility in water:10 mg/L (20 C)

Melting Point:143 - 144

Boiling Point:442[3]

MATERIALS AND METHODS

Reagents and materials

Acetonitrile and methanol was purchased from U.V.scientifics.Distilled water was prepared in the laboratory itself using Millipore apparatus.Nemisulide was obtained as a gift sample from Pharma train.Nemisulide tablet was purchased from a retail pharmacy store near Chandhanagar, Hyderabad.

Instrumentation

U V Spectrophotometer was used which was manufactured by PG instruments and model number is T190.

Digital Analytical weighing balance shimadzu model no.S903.

Preparation of stock solution

10mg is taken and diluted with 100ml of the solvent. From the above prepared solution 1ml is taken and then diluted to 10 ml using the same solvent. This gives a concentration of 10µg/ml.From the above stock solution required dilutions were prepared and scanned under U.V.Spectrophotometer from a range of 200-400nm against the solvents of method A(100% methanol) and method B[acetonitrile:methanol(50:50)] as blank. The maximum absorbance for method A and method B was found to be 297nm and 295nm respectively.

Spectrophotometric characteristics

Method A:

Wavelength maximum: 297nm

Solvent taken: methanol 100%

Weight of the drug taken = 10mg

Method B

Wavelength maximum: 295nm

Solvent taken: methanol: acetinitrile (50:50)

Weight of drug taken: 10mg

Calculation of average weight

S.No	Weight in gms.	S.No	Weight in gms.
1		11	
2		12	
3		13	
4		14	
5		15	
6		16	
7		17	
8		18	
9		19	
10		20	

Total weight of 20 tablets:

No. Of Tablets: 20

Average weight of each tablet:

RESULTS AND DISCUSSIONS

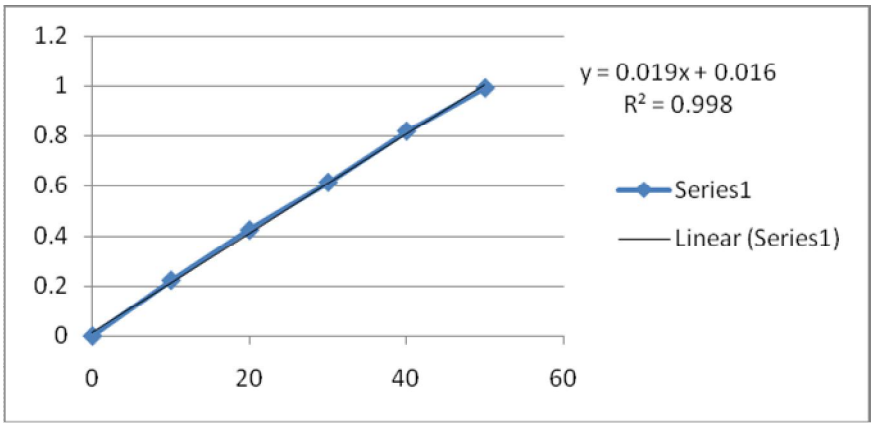
VALIDATION

Method A:

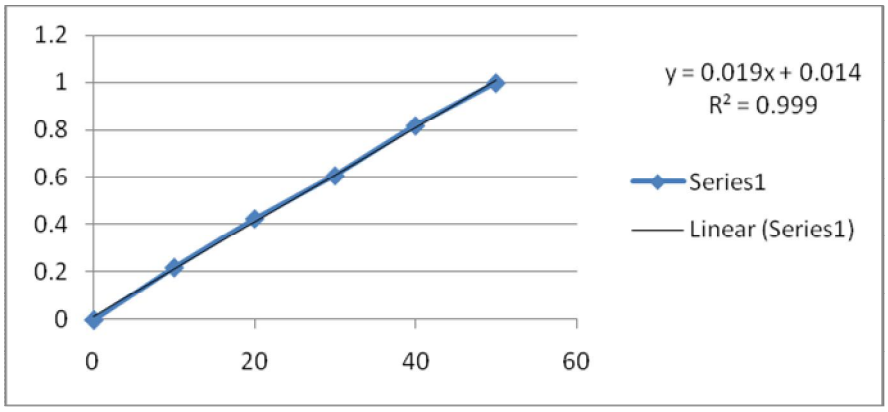
Linearity:

S.No	Concentration	Absorbance		%Assay
		Standard	Sample	
1	10	0.223	0.220	98.65
2	20	0.424	0.426	100.47
3	30	0.612	0.609	99.50
4	40	0.817	0.819	100.24
5	50	0.990	0.998	100.80
Arithematic Mean				99.932
Standard Deviation				0.861
%Relative Standard Deviation				0.862

Standard linearity chart



Sample linearity chart



Accuracy

S.No	Concentration	Absorbance		%Assay
		Standard	Sample	
1	50%	0.210	0.208	99.05
2	50%	0.210	0.206	98.09
3	50%	0.210	0.208	99.05
1	100%	0.223	0.220	98.65
2	100%	0.223	0.221	98.97
3	100%	0.223	0.221	98.97
1	150%	0.269	0.272	101.12
2	150%	0.269	0.271	100.74
3	150%	0.269	0.267	99.25
Arithematic Mean				99.32
Standard deviation				0.975.
% Relative Standard Deviation				0.982

Robustness

Differential Wavelength

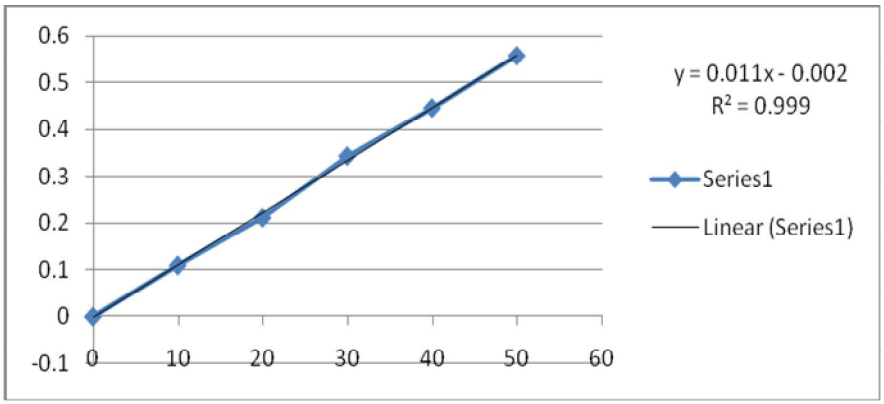
wavelength	Absorbance		%Assay
	Standard	Sample	
282nm	0.099	0.098	100
	0.099	0.100	101.01
	0.099	0.100	101.01
297nm	0.223	0.220	98.65
	0.223	0.221	98.97
	0.223	0.221	98.97
312nm	0.190	0.193	101.58
	0.190	0.189	99.47
	0.190	0.192	101.1
Arithematic Mean			100.0844
Standard deviation			1.112858
% Relative Standard Deviation			1.11

Method B

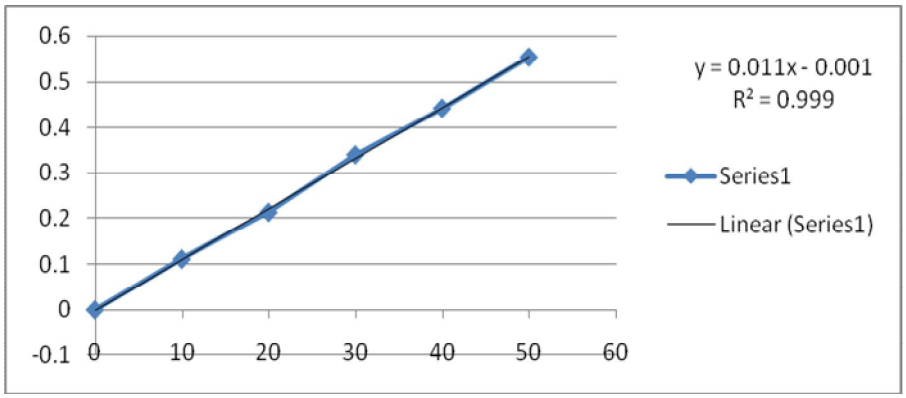
Linearity

S.No	Concentration	Absorbance		%Assay
		Standard	Sample	
1	10	0.109	0.111	101.83
2	20	0.211	0.213	100.94
3	30	0.343	0.340	99.12
4	40	0.445	0.442	99.3
5	50	0.556	0.553	99.4
Arithematic Mean				100.118
Standard Deviation				1.20
%Relative Standard Deviation				1.20

Standard linearity chart



Sample linearity chart



Robustness

(Differential Wavelength)

wavelength	Absorbance		%Assay
	Standard	Sample	
280nm	0.100	0.098	98
	0.100	0.100	100
	0.100	0.100	100
295nm	0.109	0.111	100.83
	0.109	0.109	100
	0.109	0.110	100.91
315nm	0.102	0.104	101.9
	0.102	0.104	101.9
	0.102	0.103	100.9
Arithematic Mean			100.49
Standard deviation			1.194
% Relative Standard Deviation			1.18

Accuracy

S.No	Concentration	Absorbance		%Assay
		Standard	Sample	
1	50%	0.101	0.103	101.98
2	50%	0.101	0.103	101.98
3	50%	0.101	0.103	101.98
1	100%	0.109	0.111	101.83
2	100%	0.109	0.111	101.83
3	100%	0.109	0.111	101.83
1	150%	0.120	0.122	101.66
2	150%	0.120	0.122	101.66
3	150%	0.120	0.122	101.66
Arithematic Mean				101.83
Standard deviation				0.138
% Relative Standard Deviation				0.136

SUMMARY

The proposed methods provides simple,accurate,economical and convenient methods for the analysis of nimesulide using U.V.spectrophotometry. The wavelength corresponding to maximum absorbance in 100%methanol was found to be 297nm and 50:50 (acetonitrile : methanol)was found to be 295nm.Beer's law was obeyed in the concentration range of 10-50µg/ml. Correlation coefficient was found to be 0.999. Accuracy of the proposed methods was determined by %Assay and was found to be 98.09%-101.12% for method A and 101.66%-101.98% for method B. This indicates that the proposed methods were found to be accurate. Thus by the results obtained from the assay were found to be in good agreement with label claim, indicating the absence of interference of excipients.

CONCLUSION

The present analytical methods were validated as per ICH guidelines and meets the acceptance criteria. It is concluded that the analytical method is accurate, linear, robust .The present analytical methods can be used for its intend purpose.

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