RJC
http://www.rasayanjournal.com

RASĀYAN *J. Chem.*
Vol.2, No.2 (2009), 293-296
ISSN: 0974-1496
CODEN: RJCABP

ANALYTICAL METHOD DEVELOPMENT AND VALIDATION OF PENDO-PURGATIVE BY RP-HPLC

K. Kathiresan*, M. Kathiravan[1], P. Pandian, S.P. Nayak and R. Manavalan

*Department of Pharmacy, Annamalai University, Annamalai Nagar, India
[1] D.R. Karigowda College of Pharmacy
*Email: kathiresan123@rediffmail.com

ABSTRACT

A simple, rapid, sensitive specific, accurate, RP-HPLC method was developed and validated for determination of Pendo-Purgative containing Sodium picosulphate and Citric acid in a combined formulation. According to the literature survey carried out there is no method developed in determination of these drugs by RP-HPLC. The separation was carried out using HPLC instruments. The coloum used for sodium picosulphate and citric acid was Li, (Hypersil – BDS, 150 x 4.6 mm, 5 μ). L1, (Develosill – MG05, 250 x 4.6 mm, 5μ) respectively. The flow rate selected for sodium picosulphate was 1ml/min and that for citric acid was 0.8 ml/min. The UV visible PDA detector was used at 263 nm and 210 nm for sodiumpico and citric acid respectively. The mobile phase used for sodium picosulphate was prepared by mixing buffer (Na_2HPO_4 +KH_2PO_4+trimethyl amine) with acetonitrile ratio 85:15 and that for citric acid by mixing buffer (triflouro acetic acid) and methanol in ratio 900:100. Developed method was validated in terms of linearity, range, specificity, precession, accuracy, robustness, ruggedness. The linearity range was found to be 50% to 150% with correlation coefficient of 0.998 and 0.9978 for sodium picosulphate and citric acid respective. Content of sodium picosulphate and citric acid was found to be 99.6% and 100.2% respectively.

Keywords: RP-HPLC; Sodium Picosulphate; Citric acid

INTRODUCTION

Pendo-PURGATIVE containing sodium picosulphate chemically known as disodium 4,4-(2-pyirdyl methylene di(phenyl sulphate),which is freely soluble in water and soluble in methanol,slightly soluble in ethanol used as laxative and citric acid chemically known as 2-hydroxy-b-1,2,3-propanee tricarboxylic acid which is soluble in methanol,water and sparingly soluble in ether used as a laxative,binding agent, diluent in a combined formulation.The separation was carried out using HPLC instruments. According to the literature survey carried out there is no analytical method estimation of these drugs in their combined formulation. Hence a RP – HPLC method was developed and validated as per ICH[1] guidelines.

EXPERIMENTAL

HPLC (waters alliance 2695 seperation module), UU (Shimadzu 2450), Analytical balance (melter Toledo AG 204), sonicator (Bandelin) disodium hydrogen phosphate HPLC grade, Potassium dihydrogen phosphate (HPLC grade), Triethylamine (HPLC grade), Phosphoricacid (HPLC grade), water mill - Q. sodiumpico sulphate 99.63% citric acid 99.90% (granules India Ltd).

PARAMETER	SODIUM PICOSULPHATE	CITRIC ACID
Temperature	30 ± 1°C,	25 ± 1°C
Detector	PDA 263 nm	PDA 210nm
Flow rate	1ml/min	0.8ml / min
Injection volume	50 μl	25ml
Column	Hypersil – BDS, 150x4.6 mm, 5μ	Develosil–MG-5, 250x4.6mm, 5μ
Retention time	6.8	6.9
Run time	10 min	15 min

Preparation of standard solution:
For sodium picosulphate 20 mg of sodium picosulphate was weighted and transferred to a 200ml volumetric flask.Additional dilutions were made to get 0.008mg/ml of sodium picosulphate final concentration with milli-Q water.For citric acid 30mg was accuracy weighed and transferred to 25 ml volumetric flask. Additional dilutions were made with mobile phase [mixture of buffer (Trifluoro acetic acid in water) and methanol] to get final concentration of 1.2mg/ml of citric acid.

Preparation of sample solution:
Solution picosulphate solution
Content of 10 sachets were accurately weighted and transferred to motor and pestle and crushed. Sodium picosulphate equivalent to 10gm was accurately weighed and transferred to 100ml volumetric flask. 50ml of milli-Q-water was addedand stand for 5min till effervescence has ceased, cool to room temperature, sonicate for 30 minutes with occasional shaking. Final volume was made up with milli-Q-water.

RESULTS AND DISCUSSION

Given below in the table and figures ate the summary of the results.

Table-1

Sl. No	Parameters	Acceptance criteria	% RSD	Results
1.	Specificity	Number of theoretical plates determined for sodium picosulphate and citric acid is 5010 and 10,100	0.29	Number of theoretical plats determined for sodium picosulphate and citric acid is 5055.7 and 11074.2
		Tailing factor for sodium picosulphate and citric acid should be less than 2.0	-	Tailing factor for sodium picosulphate and citric acid should be less than 0.8 to 0.95
2.	Linearity	Correlation coefficient should be not less than 99.99	-	Correlation coefficient should be not less than 0.9998
		Percentage curve fitting should be less than 99.99		Percentage curve fitting less than 0.9998
3.	Precession	RSD should not be more than 2%	0.2	The results are found to be will within the acceptance limits.
4.	Accuracy	Percentage recovery should be between 98.0 and 102.0	-	Percentage recovery found to be between 98.0 and 100.2
5.	Robustness	The number of theoretical plates determined for sodium picosulphte and citric acid should be between 5010 and 10,100	-	The number of theoretical plates determined for sodium picosulphte and citric acid found to be between 5055.7 and 11074.2
		The tailing factor for sodium picosulphte and citric acid should be less than 2.0	-	The tailing factor for sodium picosulphte and citric acid found to be 0.8 and 0.95
6.	Ruggedness	Percentage RSD of assay result obtained should be not more than 2.0.	-	1.36- 1.55% for sodium picosulphate.For citric acid it was found 0.3 - 1.72%

Citric acid solution preparation
Content of 10 sachets were accurately weighed and transferred into 100ml volumetric flask. 50ml mill-Q-water was added and stand for 5min till effervescence has gone, cool to room temperature, sonicate for 30 minutes and make up the final volume with mill-Q-water.1ml of above solution was taken and transferred to 100ml volumetric flask and final volume was made with with mobile phase

[mixture of buffer (trifluro acetic acid in water) and methanol]. Solution was filtered through 0.45µ filter paper.

ACKNOWLEDGMENTS

The authors are thankful to management of Annamalai University,Department of Pharmacy for their support to carry out the research successfully. The authors would also like to thank GRANULES INDIA LTD, HYEDRABAD for their help during the study.

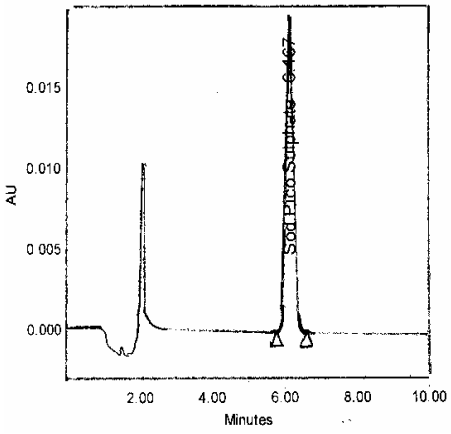


Fig.-1

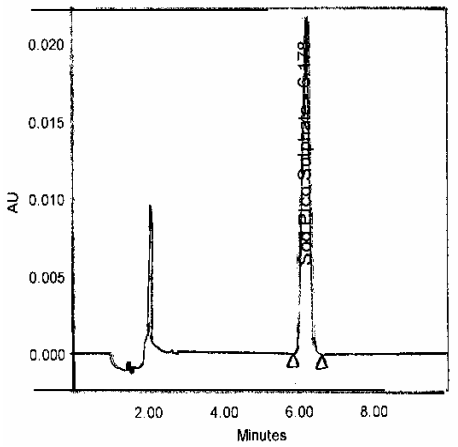


Fig.-2

HPLC Chromatogram of Standard for Sodium Picosulphate

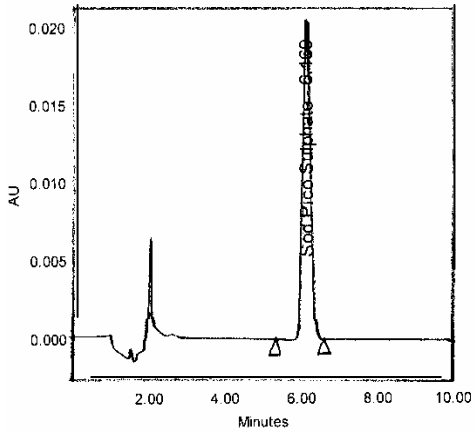


Fig.-3

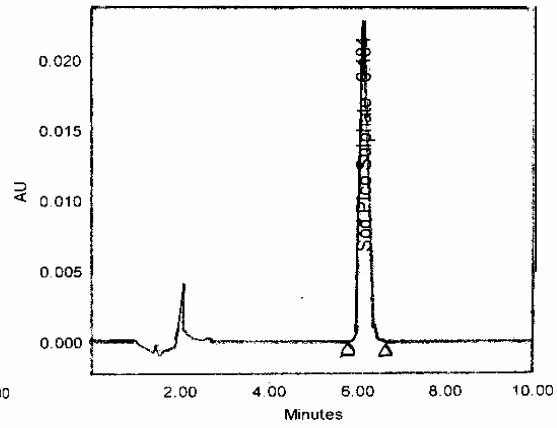


Fig.-4

HPLC Chromatogram of Sample for Sodium Picosulphate

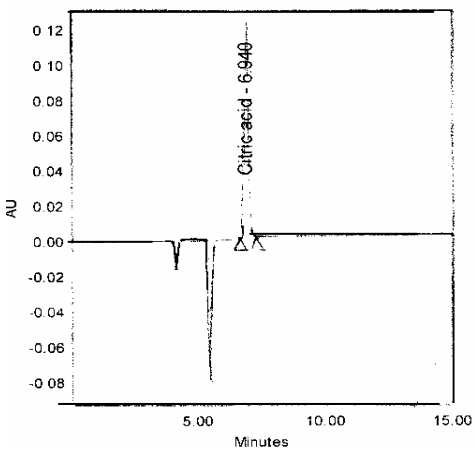


Fig.-5

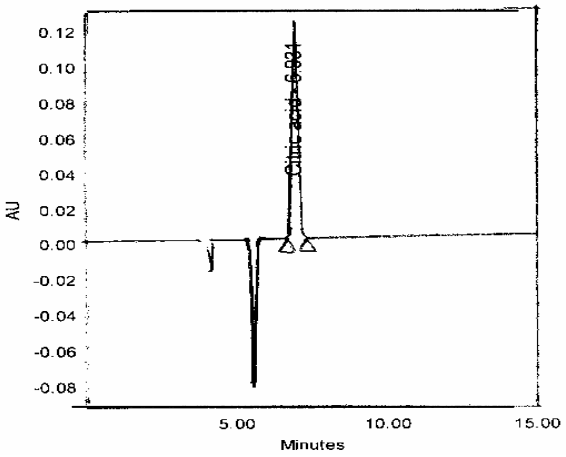
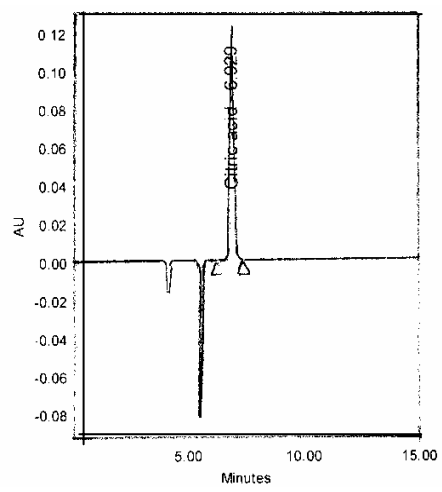


Fig.-6

HPLC Chromatogram of Standard for Citric Acid



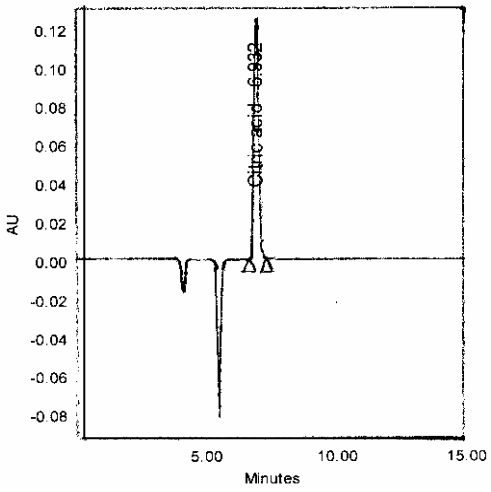
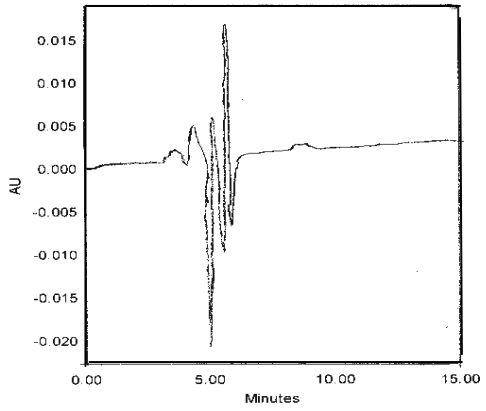
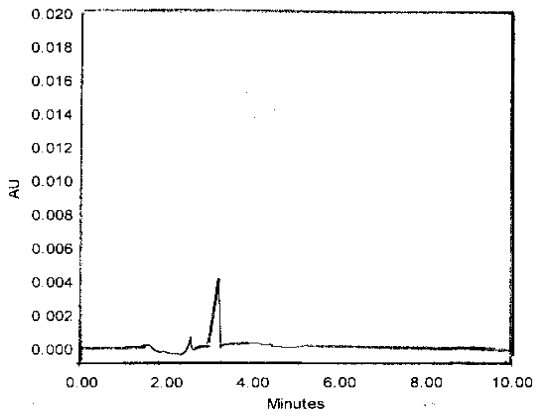


Fig.-7 **Fig.-8**

HPLC Chromatogram of Sample for Citric Acid





Citric acid **Sodium picosulphate**

Fig.-9 **Fig.-10**

HPLC Chromotagram of Blank

REFERENCES

1. International Conference on Harmonization, Validation of Analytical Procedures; Methodology. Federal Register, Nov. 1996, 1-8.
2. International Conference on Harmonization, Draft Guideline on Validation of Analytical Procedures, Definitions and Terminology. Federal registrar, **1260** (1995).
3. British Pharmacopoeia, Stationary Office London, **4**, A 457 (2003).
4. The Merck index. Published by An Encyclopedia of Chemicals Drugs and Biologicals; Merck research lab, Division of Merck & Co. INC, White house station N.J; 13th Edn., **10**, 1070.
5. Martindale, Published by Pharmaceutical Press, London, 32nd Edn., **76**, 1395 (1999).
6. K.D. Tripathi., Essentials of medical pharmacology 5th Edn., Jaypeebrothers Medical Publishers (P) Ltd, New Delhi, **182**,317.
7. United Stated Pharmacopoeia, Asian Edn, United Pharmacopoeia Convention, Inc., Rockville, **1266**, 2610 (2005).
8. Indian Pharmacopoeia, Published by the controller of publications, Delhi, **2**, 554-555 (1996).
9. British Pharmacopoeia, Stationary Office London, **2**, 1417 (2003).

(Received: 13 March 2009 Accepted: 3 April 2009 RJC-351)