## Determination of Propyphenazone and Ketoprofen in drug formulations by High Performance Liquid Chromatography

P. PARIMOO\*, A. BHARATHI, AND B.V. ATUL Deptt. of Pharmacy, Birla Institute of Technology and Science, Pilani - 333 031.

Received 27 October 1995

A new simple method is described for the rapid and quantitative estimation of propyphenazone and ketoprofen in drug preparations by high performance liquid chromatgraphy (HPLC). The mobile phase consisted of methanol, acetonitrile and water in the ratio of 10:20:30 using a  $\mu$  Bondapack C-18 column and an UV detector. Phenobarbitone was used as an internal standard.

HE combination of propyphenazone and ketoprofen is one of the marketed drug preparations for analgesic, antipyretic and antiinflammatory action. Many methods have been described in the literature for the determination of propyphenazone and ketoprofen individually and in some cases in the form of a drug combination with other drug constituents. 3-9

In a previous study, we have reported the analysis of the present formulations by quantitative TLC and HPTLC<sup>10</sup>. In the present communication, we describe the results of their simultaneous determination by HPLC in two drug combinations, which has not been reported earlier in the literature.

Pure reference standard propyphenazone (75 mg) was accurately weighed in a 250 ml volumetric flask, dissolved in methanol and the final volume made up to the mark. From this stock solution volumes of 0.5, 1.0, 1.5 and 2.0 ml each were transferred separately into 10 ml volumetric flasks and 1.0 ml of an internal standard (reference standard phenobarbitone, 50 mg in 250 ml methanol) was added to each flask. The final volumes were made up with the mobile phase (10:20:30 methanol: acetonnitril:water). In a similar manner, 25.0 mg of pure reference standard, ketoprofen was accurately

weighed and samples prepared as described for propyphenazone.

Twenty capsules were weighed, their content transferred and finely powdered. The powder equivalent to the average weight of a capsule was calculated. One hundred and twenty mg of the powder was accurately weighed in a 250 ml volumetric flask, dissolved in methanol and final volume made up to the mark. The solution was filtered to obtain a clear solution. Solutions containing the sample and 1.0 ml of internal standard were transferred into 10.0 ml volumetric flasks and final samples prepared by dilutions with mobile phase (10:20:30 methanol:acetonitrile:water) as in the case of standard preparation. The solutions of standard preparation and sample preparation were injected separately through a Rheodyne injector holding a 20 µl loop. Waters associate HPLC system equipped with dual piston reciprocating pumps (model 501), Rheodyne injector and LC spectrophotometer (model 481) was used. The eluent was monitered at 254 nm and chromatograms were recorded on an omniscribe recorder B-5000. The column consisted of a  $\mu$  Bondapack C-18 column and a Hamilton's microsyringe of 20 ul was used for injection. The chromatographic conditions maintained were isocratic with a flow rate of 2 ml/min, chart speed of 1 cm/min at a voltage of 10 mv and the UV. detector was kept at sensitivity

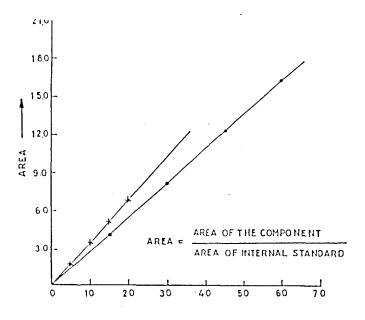
<sup>\*</sup> For correspondence

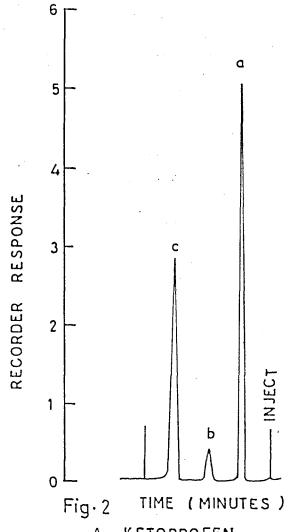
Table 1: Results for the recovery of Propyphenazone and Ketoprofen in Formulation by HPLC

Formulation	Labelled Amount (mg/cap.)	Amount Added (mg)	Amount Found (mg/cap.)*	% Recovery	SD**
Brand A					
Propyphenazone	150	150	149.91	99.94	1.04208
Ketoprofen	50	50	49.95	99.90	0.70052

<sup>\*</sup> Each value is an average of three determinations

of 0.2 AuFs. The linearity of propyphenazone and ketoprofen was studied separately. The drug concentrations were plotted against area under the curve. In case of propyphenazone the curve was found to be linear in the range of 0-60  $\mu$ g/ml and that of the ketoprofen in the range of 0-20  $\mu$ g/ml. The retention times for ketoprofen, phenobarbitone and propyphenazone were 0.93, 2.06, 3.3 minutes respectively (Fig. 2). The percentage of drug per capsule was calculated, using area under the curve.





A = KETOPROFEN

B = PHENOBARBITONE

C = PROPYPHE NAZONE

<sup>\*\*</sup> Standard deviation

The results obtained show that the amounts of propyphenazone and ketoprofen contained in the formulations conform to the specifications of the B.P<sup>3</sup> (Table I). Since an official method of analysis is not available for the simultaneous determination of the present drug combination, the proposed HPLC method is precise, accurate and reproducible. This is evidenced by their separation, quantitation and standard deviations obtained in our experiments. The proposed method thus should become very valuable in the analysis of the present combination preparations.

## **ACKNOWLEDGEMENTS**

The authors wish to thank M/s Plethico Pharmaceuticals Ltd., Indore and Juggat Pharmaceuticals Ltd. Bangalore for the supply of pure drug samples.

## REFERENCES

Martindale, The Extra Pharmacopoeia, Royal Pharmaceutical Society of Great Britain, 30th edition, 1993, 21, 31, 3.

- Liversidge, Gary G. In Florey K., Eds. Analytical Profiles of Drug Substances., Vol. 10, Academic press, N.Y., 1981, 44.
- British Pharmacopoeia, Her Majesty's Stationary Office London 1980 Addendum 1982, 69.
- 4. Benoit, E., Jaussaund, P., Besse, S. and Videmann, B., J. Chromatogr Biomed. Appli., 1992, 583, 167.
- Mamolo, M.G. and Vio, L., J. Pharm. Biomed. Anal., 1985, 3, 157.
- 6. Markovic S. and Kusec, Z., Pharmazie., 1990, 45, 935.
- Menzel Soglowek, S., Geisslinger, G and Brune, K., J. Chromatogr. Biomed. Appli., 1990, 532, 295.
- 8. Sallustio, B.C., Abas, A and Hayball, P.J., J. Chromatogr Biomed. Appli., 1986, 374, 329.
- 9. Hayball, P. J., Nation, R.L., Bochner, F. and Leu, R. K.J. Chromatogr. Biomed. Appli., 1991, 570, 446.
- Parimoo, P., Bharathi, A. and Leela, P., Indian J.
   Pharma. Sci., 1995, 57, 31.

## Simultaneous Spectrophotometric Determination of Rifampicin, Isoniazid and Pyrazinamide in Combined Pharmaceutical Dosage Forms

ROTE A.R. AND SHARMA A.K.

Dept. of Pharmacy, S.G.S.I.T.S., 23, Park Road, Indore. 452 003, M.P.

Received 30 January 1996

A simple spectrophotometric method requiring no prior separation has been developed for the simultaneous determination of Rifampicin, Isoniazid and Pyrazinamide in combination in pharmaceutical formulations. The method described in precise, accurate, reproducible and rapid.

IFAMPICIN (RIF), isoniazid (INH) and pyrazinamide (PYZ) are effective drugs for the treatment of tuberculosis. The drugs are used either

in single or combination formulations which forms the first line treatment for tuberculosis. Fixed combination of RIF (150 mg), INH (100 mg) and PYZ (350 mg) are being marketed as tablet.

<sup>\*</sup> For correspondence