

# High Performance Thin Layer Chromatographic Method for Simultaneous Estimation of Ibuprofen and Pseudoephedrine Hydrochloride

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Chitlange, *et al.*: Simultaneous HPTLC Estimation of Ibuprofen and Pseudoephedrine Hydrochloride

High performance thin layer chromatographic method is developed for simultaneous estimation of ibuprofen and pseudoephedrine hydrochloride in tablets. Silica gel 60F<sub>254</sub> plates were used as stationary phase and t.butanol: ethyl acetate: glacial acetic acid: water (7:4:2:2 v/v) as mobile phase. Wavelength selected for analysis was 254 nm. Percent estimation of ibuprofen and pseudoephedrine hydrochloride was found to be 99.56% and 98.77%, respectively. Percent recovery for both the drugs was found in the range of 98.27% to 100.91%, respectively.

Key words: Ibuprofen, pseudoephedrine hydrochloride, HPTLC, t-butanol, ethyl acetate, glacial acetic acid, water

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Ibuprofen (IBU) is a non steroidal antiinflammatory agent with propionic acid group. Chemically it is (RS)-2-(4-isobutyl phenyl) propionic acid. Pseudoephedrine hydrochloride (PEH) is a sympathomimetic agent. Chemically it is (1S,2S)-2-methyl amino-1-phenyl-1-propanol hydrochloride. Fixed dose combination tablet containing IBU (200 mg) and PEH (30 mg) is available for clinical use. IBU is official in IP, BP and USP. PEH is official in IP, BP, EP and USP. Literature survey revealed spectrophotometric<sup>1</sup>, spectrofluorometric<sup>2</sup>, HPLC<sup>3-4</sup>, GC<sup>5</sup> and HPTLC<sup>6</sup> methods for estimation of IBU alone or in combination with other drugs, in pharmaceutical formulation and biological fluids. PEH is reported to be estimated by non-aqueous titrimetry<sup>7</sup>, derivative spectrophotometry<sup>8</sup>, HPLC<sup>9,10</sup>, capillary electrophoresis<sup>11,12</sup> individually or in combination with other drugs, in pharmaceutical formulation and biological fluids. Spectrophotometric<sup>13,14</sup> methods are reported for simultaneous estimation of these drugs in tablet formulation. In the present work a successful attempt has been made to estimate both these drugs simultaneously by economical and less time consuming HPTLC method.

A Camag-HPTLC system comprising of Linomat-IV automatic sample applicator and Camag TLC scanner 3 with CAT'S version 4.0 software were used for sample application and quantitative evaluation respectively. Samples were applied as bands (band size: 6 mm at 6 mm interval) under a stream of nitrogen on aluminium plates coated with silica gel 60F254 (10×10 cm, Merck) and chromatographed using tertiary butanol:ethyl acetate:glacial acetic acid: water (7:4:2:2 v/v) as mobile phase. Ascending development was performed in a saturated twin-trough TLC chamber. Chromatogram was evaluated by scanning in absorbance/reflectance mode at 254 nm using slit dimensions 4×0.45 mm and quantitation was done by comparing peak height of standard and sample peaks.

Standard solution containing 8 mg/ml of IBU and 1.2 mg/ml of PEH was prepared in methanol. To study the linearity of detector response, the standard stock solutions were appropriately diluted and accurately measured volume ranging from 1 to 10  $\mu$ l was applied on the TLC plate. The plate was then chromatographed in the selected chromatographic conditions. A calibration graph was constructed by plotting peak height versus concentration.

For estimation of IBU and PEH in tablets, an accurately weighed quantity of tablet powder equivalent to 200 mg of IBU was transferred to 25 ml volumetric flask, shaken with 10 ml of methanol for 15 min. and the volume was then adjusted to the mark with methanol. The solution was then filtered through whatman Grade I filter paper and 6  $\mu$ l of the filtrate (six bands) and standard solution (one band) was applied on the TLC plate and chromatographed. Amount of both the drugs were estimated by comparing the peak height of standard and sample bands. The results are shown in Table 1 and respective densitogram is shown in fig. 1.

To study the accuracy of the proposed method recovery studies were carried out using standard addition method. The percent recovery was calculated by using the formula, % recovery =  $(T-A)/S \times 100$ , where T is total amount of drug estimated, A is the amount of drug contributed by tablet powder and S is the amount of pure drug added. Result of recovery studies are shown in Table 1.

The system repeatability was studied by applying five replicate applications of standard solution on TLC plate. The plate was chromatographed and the standard deviation for peak height of IBU and PEH was calculated. The robustness of the method was evaluated by studying analyst-to-analyst, intra and inter day variations.

The selected chromatographic conditions were found to effectively separate IBU ( $R_f = 0.91$ ) and PEH ( $R_f = 0.68$ ). The linearity for detector response

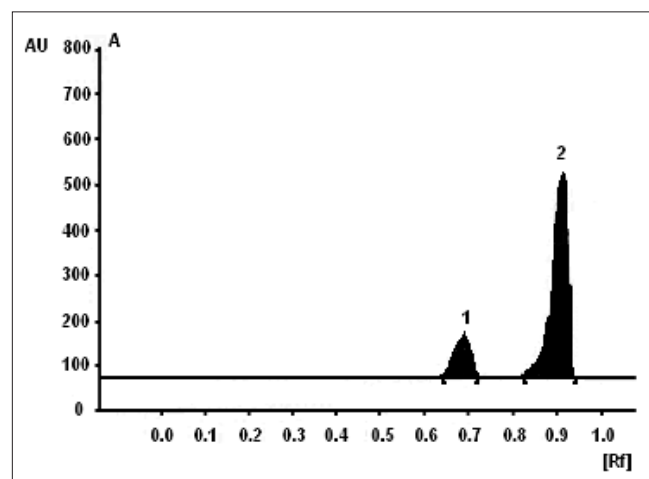


Fig. 1: Chromatogram of marketed formulation. 1- pseudoephedrine hydrochloride and 2- ibuprofen

**TABLE 1: RESULTS OF ESTIMATION IN TABLET AND RECOVERY STUDIES**

| Sample                      | Label claim<br>(mg/tablet) | % Label claim*,<br>SD, CV |                      | % Recovery*,<br>SD, CV |                       |
|-----------------------------|----------------------------|---------------------------|----------------------|------------------------|-----------------------|
|                             |                            | IBU                       | PEH                  | IBU                    | PEH                   |
| Standard laboratory mixture | -                          | 99.01±0.889<br>0.030      | 99.46±0.731<br>0.024 | -                      | -                     |
| Arinac tablet               | IBU 200<br>PEH - 30        | 99.56±1.668<br>0.016      | 98.77±1.452<br>0.024 | 100.69±1.662<br>0.016  | 101.03±1.854<br>0.018 |

\*Indicates mean of five observations, ± indicates standard deviation. IBU stands for ibuprofen and PEH denotes pseudoephedrine hydrochloride.

was observed in the range of 45.6-75.6 µg for IBU (correlation coefficient = 0.9934) and 6.8-11.3 µg for PEH (correlation coefficient = 0.9963). Percent amount of IBU and PEH estimated in the average weight of tablet were found to be 99.56% (standard deviation = ±0.1.668) and 98.77% (standard deviation = ±1.452), respectively. The low values of standard deviation indicate the precision of the method. Percent recovery for IBU and PEH was found to be 100.69% (standard deviation = ±1.662) and 101.03% (standard deviation = ±1.854) indicating that the excipients does not have interference in their estimation. The system repeatability studied by five replicate applications of standard solution. The standard deviations for peak height were found to be ±0.11 for IBU and ±0.06 for PEH. The standard deviation for robustness studies was below 2%.

Based on the above results it can be concluded that the proposed HPTLC method is accurate, precise, specific and reproducible. The method is also economical and less time consuming and can be used for routine analysis of ibuprofen and Pseudoephedrine hydrochloride.

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