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Simultaneous Determination of Dexibuprofen and Tramadol HCl by HPTLC Method

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Abstract

Introduction: Aim of the study is to develop simple, specific, accurate HPTLC method for simultaneous determination of the Dexibuprofen and Tramadol HCl in pharmaceutical dosage form.

Materials and Method: Stationary phase was precoated silica gel 60 F_{254}. The Mobile Phase used was mixture of Chloroform: toluene: ethanol: glacial acetic acid (8:4:1:0.5 v/v/v/v). The detection of the spots was carried out at 264nm and 271nm for Dexibuprofen and Tramadol HCl respectively.

Result: The method was validated in terms of linearity, accuracy, precision and specificity. The Limit of detection for Dexibuprofen and Tramadol HCl was found to be 1µg and 0. 2 µg respectively. The Limit of quantification for Dexibuprofen and Tramadol HCl was found to be 1.6µg and 0.4µg respectively.

Conclusion: The method is free from the interferences due to excipients present in the formulation and can be successfully used for determine drug in pharmaceutical dosage form.

Keywords: Dexibuprofen, Tramadol HCl, HPTLC method, Simultaneous determination

Introduction

Dexibuprofen is chemically (S)-Alpha-Methyl-4-(2-Methyl Propyl) Benzene Acetic Acid. Dexibuprofen is a pharmacologically active enantiomer of racemic ibuprofen. Racemic ibuprofen is a non-steroidal substance with anti-inflammatory and analgesic effects. Its mechanism of action is due to inhibition of prostaglandin synthesis.[1] Tramadol Hclischemically*rac*-(1*R*,2*R*)-2-(dimethylaminomethyl)-1-(3-methoxyphenyl)-cyclohexanolit work through modulation of the GABAergic, noradrenergic and serotonergic systems, in addition to its mild agonism of the µ-opioid receptor.[2]

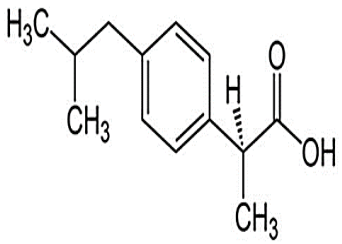

Fig. 1: Dexibuprofen

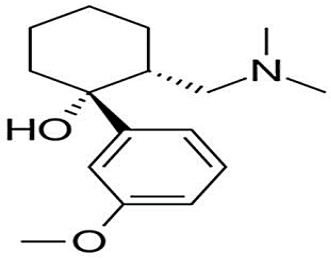

Fig. 2: Tramadol

It is reported that the addition of Tramadol HCl 37.5mg tablets (four times daily) for 5 days to existing NSAID therapy provides effective pain relief in patients with osteoarthritis flare pain. Liquid filled capsules containing 475mg Dexibuprofen and Tramadol HCl combination if taken twice in a day are effective novel dosage forms for osteoarthritis.[2] The objective of the work is to develop the new HPTLC method for simultaneous of Dexibuprofen and Tramadol HCl in pharmaceutical dosage form.

Materials and Method

Dexibuprofen and Tramadol HCl pure powder sample were gifted by Glochem Industries Limited and Oranosys Pharma Respectively. The HPTLC system (Linomate IV Applicator and Camag scanner IV) was used for the development of the method.

Instrumentation

Camag Linomat IV as sample applicator, Camag TLC scanner II, Camag Twin tough chamber were used.

HPTLC pre-coated plates with silica gel 60F (20x10 cm) Merck 5642 were used.

Development of mobile phase

Number of polar and non-polar solvent combinations were attempted to develop the mobile phase. Mobile phase containing chloroform: toluene: ethanol: glacial acetic acid (8:4:1:0.5 v/v/v/v) gave best resolution without tailing so it was finalized as mobile phase for method development for simultaneous determination of Dexibuprofen and Tramadol HCl.

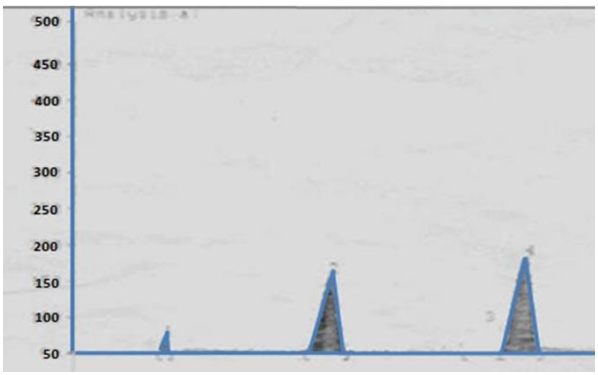

Fig. 3: Chromatographic scan showing resolved peaks of Tramadol HCl and Dexibuprofen by simultaneous HPTLC analysis method

Preparation of Calibration Curve

Sample Preparation for calibration curve: Tramadol HCl and Dexibuprofen have good solubility in methanol so it was used for sample preparation. Dexibuprofen and Tramadol were present in combination formulation as 400mg: 75mg i.e. used in ratio of 16:3 hence for linearity study sample was prepared by dissolving 160mg of Dexibuprofen and 30mg of Tramadol HCl in methanol and volume was made upto 25ml. Sample volumes ranging from 1-5 µl were spotted. TLC chamber was saturated using developed mobile phase chloroform: toluene: ethanol: glacial acetic acid (8:4:1:0.5) for 30 mins and plates were scanned at λ_{max}264nm for Dexibuprofen and 271nm for Tramadol HCl.

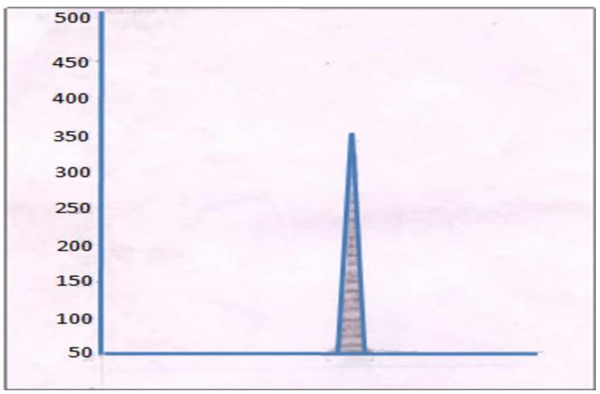

Fig. 4: Chromatogram of Dexibuprofen

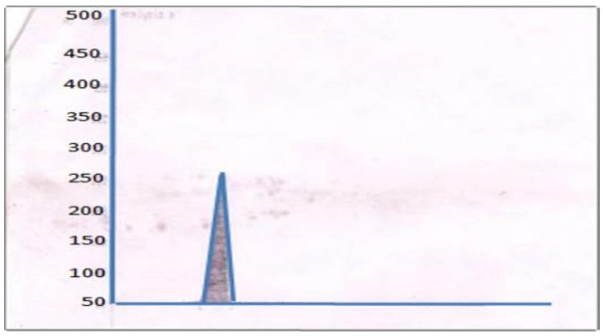

Fig. 5: Chromatograms of Tramadol HCl

Table 1: Calibration curve of Dexibuprofen by HPTLC method

Sr. No	Amount of Dexibuprofen (µg)	Area at 263nm
1.	6.4	1498.4
2.	12.8	3087.1
3.	19.2	4163.4
4.	25.6	4953.2
5.	32.0	6126.5

Table 2: Calibration curve of Tramadol HCl by HPTLC method

Sr. No	Amount of Tramadol HCl (µg)	Area at 271nm
1.	1.2	1371
2.	2.4	2450
3.	3.6	3336
4.	4.8	4075
5.	6.0	5377

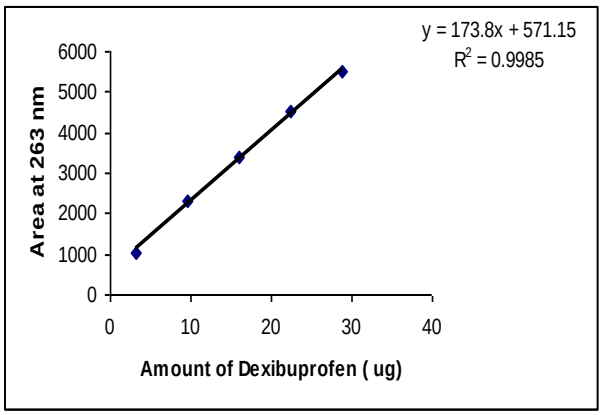

Fig. 6: Calibration curve of Dexibuprofen

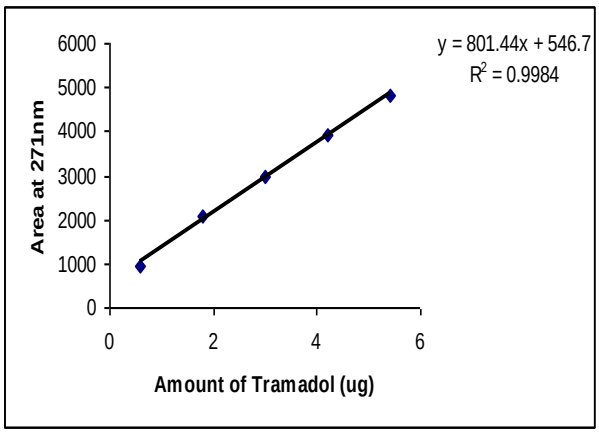

Fig. 7: Calibration curve of Tramadol HCl

Validation of HPTLC Method for Simultaneous Analysis of Dexibuprofen and Tramadol HCl

1. **Linearity:** Linearity of method was studied by preparing linearity curve of Dexibuprofen and Tramadol HCl simultaneously. Results are

recorded in Table 3 and 4.

Standard solution preparation: As Dexibuprofen and Tramadol HCl used in ratio of 16:3 in the formulation. Standard solution was prepared by dissolving 64mg of Dexibuprofen and 12mg of Tramadol HCl in methanol and volume was made upto 100ml by methanol.

2. **Precision:** From the stock solution prepared for linearity study, sample of 2µl was spotted with the help of applicator on precoated HPTLC plates and HPTLC chromatograms were obtained as described earlier. This procedure was performed six times. From the absorbance reading, mean, SD and % RSD were calculated. The results are recorded in Tables 5 and 6.

Table 3: **Linearity studies of developed HPTLC method for analysis of Dexibuprofen**

Sr. No	Amount of Dexibuprofen(µg)	Area at 263nm
1.	3.2	1050.5
2.	9.6	2292.3
3.	16	3406.2
4.	22.4	4508.1
5	28.8	5505.1

Table 4: Linearity studies of developed HPTLC method for analysis of Tramadol HCl

Sr. No	Amount of Tramadol HCl (µg)	Area at 271nm
1.	0.6	950.3
2.	1.8	2063.8
3.	3	2998.3
4.	4.2	3903.1
5.	5.4	4839.3

Table 5: Precision studies of developed HPTLC method for analysis of Dexibuprofen

Sr. No	Reference solution	Area at 263nm
1.	Sample 1	3070
2.	Sample 2	3105
3.	Sample 3	3100
4.	Sample 4	3090
5.	Sample 5	3002
6.	Sample 6	3011
	Mean	3063
SD: ±41.50502% **RSD:** 1.355045 **(RSD limit: < 2.0%)**		

Table 6: Precision studies of developed HPTLC method for analysis of Tramadol HCl

Sr. No	Reference solution	Area at 271nm
1.	Sample 1	2400
2.	Sample 2	2380
3.	Sample 3	2460
4.	Sample 4	2350
5.	Sample 5	2300
6.	Sample 6	2369
	Mean	2376.5
SD: ± 48.55838 **% RSD:** 2.043273 **RSD limit: (< 2.0%)**		

3. **Accuracy:** It was expressed in terms of percentage recovery.

The amount of Dexibuprofen and Tramadol HCl that is added in the solution and the amount exhibited by the chromatogram were determined to check the accuracy of the developed method. The procedure was performed three times and the concentrations of drugs in the solution were calculated. Mean of percent recovery was found to be 99.35 and 99.15 for Dexibuprofen and Tramadol HCl respectively. The results are shown in tables 7and 8.

4. **Limit of Detection (LOD):** It is the minimum concentration of Dexibuprofen and Tramadol HCl solution that is detectable but not quantifiable by HPTLC method. The smallest peak on the chromatogram that could be detected was obtained. The concentration at the smallest peak was determined by spotting the concentration of Dexibuprofen and Tramadol standard solutions. It was found to be 1µg and 0. 2 µg respectively.
5. **Limit of Quantification (LOQ):** It is minimum concentration of Dexibuprofen and Tramadol that was detectable as well as quantifiable. The smallest peak on the chromatogram that could be detected as well as quantified was obtained. The concentration of the smallest peak was determined using standard solution of Dexibuprofen and Tramadol HCl. It was found to be 1.6µg and 0.4µg respectively.

Table 7: Percentage recovery of Dexibuprofen by HPTLC method

Sr. No	Solutions	Area at 263nm	Drug content (%)
1.	Sample 1	4908.4	99.16
2.	Sample 2	4916.8	99.33
3.	Sample 3	4928.2	99.56
Mean 99.35% **SD:** ±0.163911 **% RSD:** 0.164983 **(RSD limit :< 2.0%)**			

Table 8: Percentage recovery of Tramadol HCl by HPTLC method

Sr. No	Solutions	Area at 271nm	Drug content (%)
1.	Sample 1	0.4044	99.26
2.	Sample 2	0.4034	99
3.	Sample 3	0.4042	99.2
Mean 99.15**% SD:** ±0.153810 % **RSD:** 0.15486 **(RSD limit: < 2.0%)**			

Results and Discussion

The proposed methods for simultaneous

determination of Dexibuprofen and Tramadol HCl in combined dosage form were found to be simple, rapid, accurate, precise, specific and economical. The developed method can be used for routine analysis of two drugs in combined dosage forms. This method permits determination of drug without previous separation with good accuracy and precision.

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