Original Research Article

A comparative study of Cilnidipine and Telmisartan in tablets by high performance thin-layer chromatography with ultraviolet absorption densitometric detection

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Abstract

Background: Analysis of pharmaceutical compounds and newer drugs is commonly used in all the stages of drug discovery and development process.The present study was conducted to compare Cilnidipine and Telmisartan in tablets by high performance thin-layer chromatography with ultraviolet absorptiondensitometric detection.**Materials & Methods:**The present study was conducted on Cilnidipine and Telmisartan tablets. Separate HPTLC pre-coated plates of silica gel G 60 F254 (10x10) were employed for the spotting of standard solutions. 2µl of standard solutions of concentration 50,100, 200, 400, and 600µg/ml of Cilnidipine and Telmisartan standard solutions were applied in the five tracks respectively in two different plates.**Results:** Rf value for Cilnidipine was 0.46, height was 436.52 and area was 11834.2. Rf value for Telmisartan was 0.18, height was 372.74 and area was 2234.56. The accuracy of Cilnidipine was 98.12% and Telmisartan was 97.2%. Repeatability was 0.96 and 0.74 in cilnidine and Telmisartan respectively, intra-day value was 0.84-1.22 and 0.62-0.89 in cilnidine and Telmisartan respectively. Inter-day value was 0.92-1.36 and 1.24-1.48 in cilnidine and Telmisartan respectively. LOD was 12.8 ng/spot and 62.08 ng/spot in cilnidine and Telmisartan respectively. LOQ was 38.28 ng/spot and 185.0 ng/spot in cilnidine and Telmisartan respectively.**Conclusion:**Authors found that Rf value for Cilnidipine was 0.46 and for Telmisartan was 0.18. This method was found to be accurate and precise and canbe used for routine analysis of Cilnidipineand Telmisartan in pure and combined dosage form.

Key words: Cilnidipine, Telmisartan, Thin-layer chromatography

Introduction

Analysis of pharmaceutical compounds and newer drugs is commonly used in all the stages of drug discovery and development process. These analytical techniques provide more accurate and precised data, not only supporting drug discovery and development but also postmarket surveillance[1].Pharmaceutical analysts work regularly to improve the reliability of existing techniques to cope up the demands for better chemical measurements. Modern pharmaceutical analysis is mainly dominated by costlier instrumental analysis. Hence, many analysts' focus is on developing newer applications, discoveries, and new methods of analysis to increase the specificity and sensitivity of a method[2]. Cilnidipine (CIL) O3-(2-methoxyethyl) O5-[(E)-3-phenylprop-2-enyl] 2,6-dimethyl-4- (3-nitro phenyl)-1,4-dihydropyridine- 3,5- dicarboxylate is a novel and unique dihydropyridine calcium antagonist that possesses a slow-onset, long-lasting vasodilating effect[3]. Telmisartan (TEL), 4-((2-n-propyl-4- methyl-6-(1- methylbenzimidazol – 2 – yl) – benzimidazol – 1 - yl) methyl) biphenyl2- carboxylic acid, is an angiotensin II receptor blocker that shows high affinity for the angiotensin II receptor type 1 (AT1), with a binding affinity 3000 times greater for AT1 than AT2 2 [4]. Literature review revealed spectrophotometric and

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RP-HPLC method for the estimation of these drugs individually. Thin layer chromatography studies are among the key identity tests in most pharmacopoeial monographs. Pharmacopoeial standards are typically used by industry as a basis for meeting QC requirements and current good manufacturing practices (cGMPs). An extension of TLC is high-performance thin layer chromatography (HPTLC) is robust, simplest, rapid, and efficient tool in quantitative analysis of compounds[5]. The present study was conducted to compare Cilnidipine and Telmisartan in tablets by high performance thin-layer chromatography with ultraviolet absorption densitometric detection.

Materials & Methods

The present study was conducted on Cilnidipine and Telmisartan tablets. The study approval was obtained before hand from institutional ethical committee.
20 tablets containing 10 mg of Cilnidipine and 40 mg of Telmisartan were weighed, average weight determined and finely powdered. Appropriate quantity of powder equivalent to 10 mg of Cilnidipine and 40 mg Telmisartan was accurately weighed, transferred to a 10 ml volumetric flask and volume was made up to 10 ml with methanol and shaken vigorously for 15 minutes. The solution was then sonicated for 5 minutes and filtered through the Whatman filter paper no.41. Necessary dilutions of filtrate were made with methanol to get final concentration 100 µg/ml of Cilnidipine and 400 µg/ml of Telmisartan. Selection of mobile phase A trial and error process was done to select the appropriate mobile phase. The solvent system of Toluene: Ethyl acetate: DMF in the ratio 6.5: 3.0: 0.5 was the most appropriate solvent system for the HPTLC analysis of Cilnidipine and Telmisartan in methanol. Separate HPTLC pre-coated plates of silica gel G 60 F254 (10x10) were employed for the spotting of standard solutions. 2µl of standard solutions of concentration 50,100, 200, 400, and 600µg/ml of Cilnidipine and Telmisartan standard solutions were applied in the five tracks respectively in two different plates.Results thus obtained were subjected to statistical analysis. P value less than 0.05 was considered significant.

Results

Table 1: Rf factor, height and area of Cilnidipine peak

Vial	Rf	Amount- ng/spot	Height	Area
S1	0.47	100	102.4	2136.4
S2	0.48	200	146.32	3356.6
S3	0.46	400	230.82	5845.4
S4	0.46	800	350.16	9264.8
S5	0.46	1200	436.52	11834.2

Table I shows that Rf value for Cilnidipine was 0.46, height was 436.52 and area was 11834.2.

Table 2: Rf factor, height and area of Telmisartan peak

Vial	Rf	Amount- ng/spot	Height	Area
S1	0.16	100	104.2	660.4
S2	0.17	200	160.74	776.24
S3	0.18	400	242.32	1536.20
S4	0.18	600	334.56	1872.74
S5	0.18	1200	372.74	2234.56

Table II shows that Rf value for Telmisartan was 0.18, height was 372.74 and area was 2234.56.

Table 3:Validation parameters for the proposed methods

Parameter	Cilnidipine	Telmisartan
Accuracy %	98.12	97.24
Precision (RSD, %)		
Repeatability(n=6)	0.96	0.74
Intraday(n=3)	0.84-1.22	0.62-0.89
Interday(n=3)	0.92-1.36	1.24-1.48
LOD	12.8ng/spot	62.08ng/spot
LOQ	38.28 ng/spot	185.0ng/spot

Table III shows that accuracy of Cilnidipine was 98.12% and Telmisartan was 97.2%. Repeatability was 0.96 and 0.74 in cilnidine and Telmisartan respectively, Intraday(n=3)value was 0.84-1.22 and

0.62-0.89 in cilnidine and Telmisartan respectively. Interday(n=3) value was 0.92-1.36 and 1.24-1.48 in cilnidine and Telmisartan respectively. LOD was 12.8 ng/spot and 62.08 ng/spot in cilnidine and Telmisartan respectively. LOQ was 38.28 ng/spot and 185.0 ng/spot in cilnidine and Telmisartan respectively.

Discussion

HPTLC is one of the most widely applied methods for the analysis in pharmaceutical industries, clinical chemistry, forensic chemistry, biochemistry, cosmetology, food and drug analysis, environmental analysis, and other areas. It is due to its numerous advantages, for example, it is the only chromatographic method offering the option of presenting the results as an image. Other advantages include simplicity, low costs, parallel analysis of samples, high sample capacity, rapidly obtained results, and possibility of multiple detection[6]. High-throughput analysis using HPLTC is being aimed at the rapid analysis of large numbers of compounds. This field has been expedited by the requirement to provide analytical support for multiple drug targets emerging from the field of molecular biology, human genetics, and functional genomics[7]. Further, drivers for development have been in the support for the analysis of large compound libraries arising from parallel and combinatorial chemistry, and economic pressure to reduce time-to-market for new drug candidates[8].

The present study was conducted to compare Cilnidipine and Telmisartan in tablets by high performance thin-layer chromatography with ultraviolet absorption densitometric detection. In present study we found that Rf value for Cilnidipine was 0.46, height was 436.52 and area was 11834.2. Rf value for Telmisartan was 0.18, height was 372.74 and area was 2234.56. Jayasekhar in their study found that the Rf value for Cilnidipine and Telmisartan was found to be 0.47and 0.17 respectively[9]. The correlation coefficients was 0.9917 and 0.9852 for Telmisartan respectively. The calibration curve was found to be linear between 100 to 1200 ng/spot for both Cilnidipine and Telmisartan. The limits of detection and quantitation were found to be 12.6 and 38.28 ng/spot, respectively for Cilnidipine and 61.05 and 185.0 ng/spot, respectively for Telmisartan.

We observed that accuracy of Cilnidipine was 98.12% and Telmisartan was 97.2%. Repeatability was 0.96 and 0.74 in Cilnidioine and Telmisartan respectively, Intraday(n=3) value was 0.84-1.22 and 0.62-0.89 in cilnidine and Telmisartan respectively. Interday(n=3) value was 0.92-1.36 and 1.24-1.48 in cilnidine and Telmisartan respectively. LOD was 12.8 ng/spot

Puranik et al developed and validated a simple, rapid, and accurate chromatographic methods (HPLC and HPTLC) for simultaneous determination of ofloxacin and ornidazole in solid dosage form. The amount of ofloxacin and ornidazole estimated as percentage of label claimed was found to be 100.23 and 99.61% with mean percent recoveries 100.47 and 99.32%, respectively. Both these methods were found to be simple, precise, accurate, selective, and rapid and could be successfully applied for the determination of pure laboratory prepared mixtures and tablets[10].

Małgorzata Starek et al reported that the procedure can be readily used for selective analysis of drugs, and repeatable results are obtained without interference from auxiliary substances. Similarly, HPTLC method was successfully used to analyze fixed-dose tablets samples of lamivudine, stavudine, and nevirapine. Patel et al[12] developed a simple and rapid HPTLC method and validated for quantitative determination of olanzapine on silica gel 60F254 layers using methanol-ethyl acetate (8.0 + 2.0, v/v) as the mobile phase. The developed method was found to be simplest among existing analytical methods[11].

Conclusion

Authors found that Rf value for Cilnidipine was 0.46 and for Telmisartan was 0.18. This method was found to be accurate and precise and can be used for routine analysis of Cilnidipine and Telmisartan in pure and combined dosage form.

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