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Analysis of Drug Preparation Containing Chlorpropamide and Phenformin Hydrochloride by Thin-Layer Chromatography

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KLORPROPAMİD VE FENFORMİN-HCL İÇEREN TABLETLERİN İNCE TABAKA KROMATOGRAFİSİ YÖNTEMİYLE ANALİZİ

Özet

Eczacılık preparatlarının içerdikleri bileşenlerin belirtiminde genellikle farmakopilere uygun yöntemler kullanılır. Ancak bu yöntemlerin bir bölümü oldukça pahalı gereçleri veya uzun süren işlemleri gerektirir. Bu nedenle, klorpropamid ile fenformin-HCl içeren ve antidiabetik olarak kullanılan tabletlerin analizi için bir yöntem geliştirdik. Bu yöntemde, önce etkinleştirilmiş Silikajel G tabakaları ve metanol : amonyak : su (49:49:2) mobil fazı kullanılarak bileşenler birbirlerinden ayrılmakta , daha sonra *Shimadzu UV 240* spektrofotometresiyle miktarları tesbit edilmektedir. Yapılan literatür taramasında benzer bir yönteme rastlanmamış olup, farmasötik preparatlarda hızlı, ucuz ve basit bir şekilde klorpropamid ve fenformin belirtimi için önerilmektedir.

Summary

The pharmaceutical preparations are usually analyzed by pharmacopeial methods for their individual components. This involves either sophisticated instruments or time consuming factors. Therefore a method was developed to analyze a pharmaceutical antidiabetic preparation in the form of a tablet containing chlorpropamide and phenformin hydrochloride. This involves, first, separation of the individual components by a simple thin-layer chromatography (TLC), and then estimating the same by spectrophotometric method *(Shimadzu UV 240)*. Literature survey does not reveal any work on such above mentioned formulation. Therefore, the devised method was found simple, economical and time saving.

Keywords: *Chlorpropamide - Phenformin HCl - TLC analysis - Spectrophotometric method*

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INTRODUCTION

Mill and *Chamberlain* (1) determined phenformine in human body fluids by using *HPLC*. *Allesandro* et *al* (2) have estimated biguanides by complexometric titration. Their determination was by formation of Cu complexes from cuprammonium or Fehling's solution.*Wickramsingha* and *Shaws* (3) estimated phenformine and other biguanides by using gas chromatography. *Joshi* (4) determined chlorpropamide and its metabolites in plasma by using *HPLC*. *Takla* and *Joshi* (5) identified, assayed and determined the purity of chlorpropamide, glibenclamide and tolbutamide by *TLC*. They have used cyclohexane : chloroform: acetic acid : ethanol (10/1/2/1). *Zecca* and*Colombo* (6) estimated glibenclamide, chlorpropamide and tolbutamide in plasma by *HPLC* with *UV* detection.

MATERIAL AND METHODS

The label claims per tablet as : chlorpropamide 50 mg ; phenformin 25 mg.

Preparation of test solution

Five tablets were triturated in a glass mortar, and then the powder equivalent to two tablets was extracted in 70 mL ethanol. The solution was filtered through Whatman no. 42 paper in a 100 mL volumetric flask. The residue was washed with 5 mL each of ethanol 4 times and then added to the original to make up the volume.

Preparation of standand solution

100 mg of chlorpropamide and 50 mg of phenformin were weighed accurately and dissolved in 100 mL of ethanol. The above samples were checked for its purity by IP specifications (Figures 1,2).

Separation and quantitation

The chromoplates of 20x20 cm size were prepared with silica gel G of thickness 500 μm and then activated at 100 -105°C for one hour. Each three activated chromoplates were taken and streaked using 1, 1.25 and 1.5 mL of test and standard solution. The plates were developed in a saturated developing chamber using methanol : water : ammonia solution (49:49:2) as a mobile phase. The plates were run to a 12 cm which took 25 minutes. Visualization was done by spraying the plates with 2% potassium permanganate reagent in water. For the purpose of scrapping a reference plate under the same conditions was prepared and then knowing the Rf values scrapping was done.

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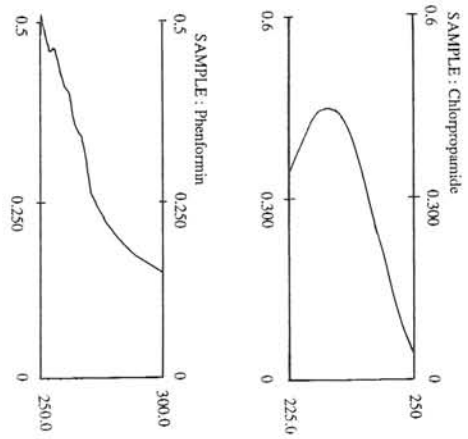

Figure 1. Spectra of chlorpropamide and phenformin.

Table 1. TLC - data of the analysis of chlorpropamide and phenformin HCL containing tablets.

Drug	Content/tab. by proposed method.	Drug added (mg)	Drug recovered (mg)	Recovery (%)	S.D.	C.V.
Chlorpro-pamide	54.08	100	102.6	102	0.3836	0.7080
Phenformin-HCl	27.42	50	51.9	103	0.3919	1.4453

The drugs were separated at following Rf values:

Chlorpropamide 0.85, Phenformin 0.25.

The resulting band each of above test and standard solution was scrapped out and treated in the following manner:

a) Scrapping corresponding to ref. chlorpropamide spot was extracted in 100 mL 0.01N HCl and then analyzed by *Shimadzu* UV 240 / Visible spectrophotometer at 232 nm (E 1 % 1 cm 600).

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b) Scrapping corresponding to ref. phenformin spot was extracted in 5 mL of 0.1N sulfuric acid and then analyzed by *Shimadzu* UV 240/Visible spectrophotometer at 251 nm (E 1 % 1 cm 11).

RESULTS AND DISCUSSION

The content of two tablets 100 mg chlorpropamide and 50 mg phenformin were added.From this mixture the quantity of content equivalent to 100 mg of chlorpropamide was taken and analyzed by proposed method (Table 1). The percentage recovery of the active ingredients was computed from the results obtained. Further statistical evaluation indicates the precision of the proposed method.

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