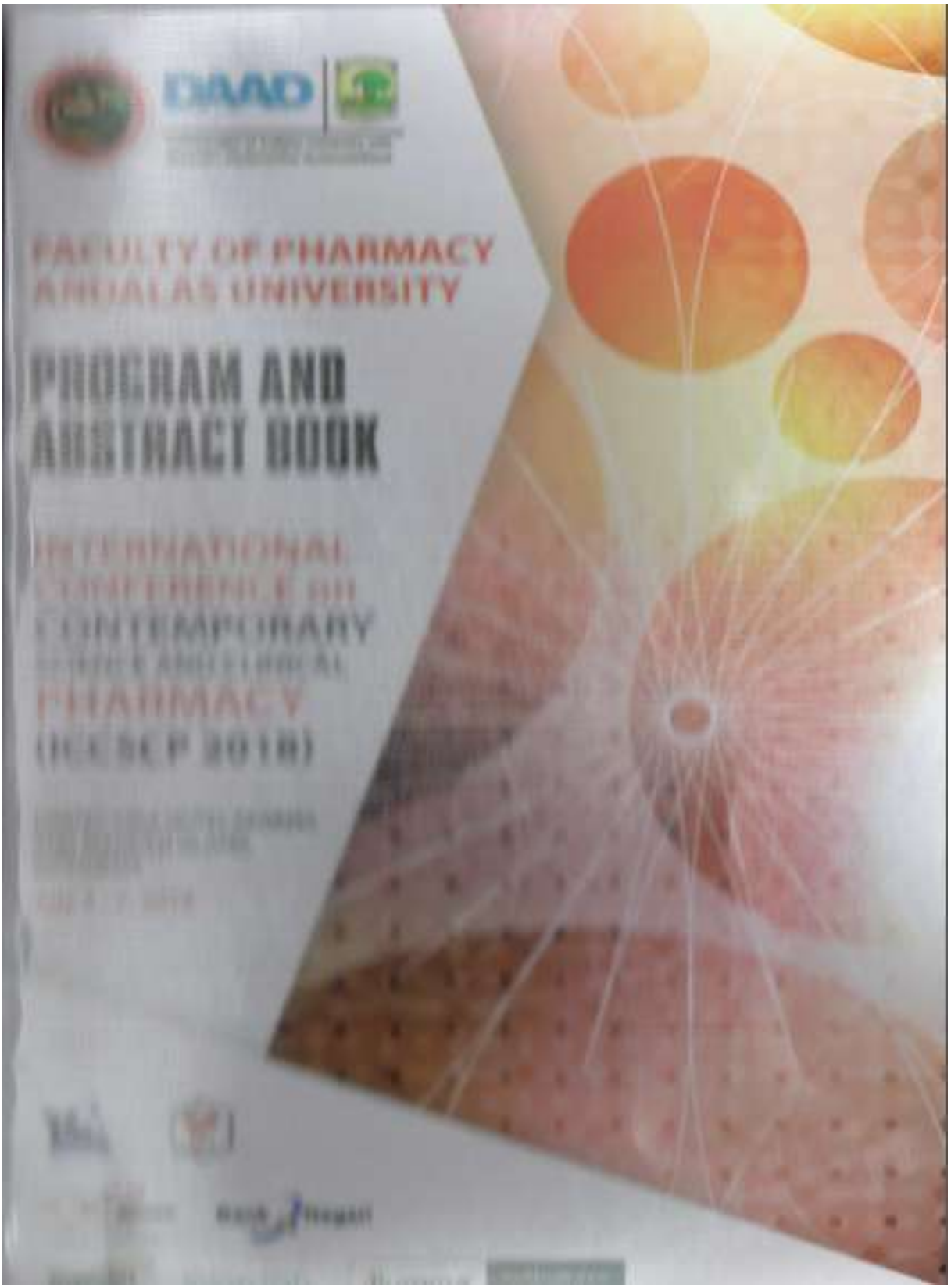
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PROGRAM AND ABSTRACT BOOK

INTERNATIONAL CONFERENCE ON CONTEMPORARY SCIENCE AND CLINICAL PHARMACY (ICCSCP 2018)

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ID-147

Validation of Analysis Method and Determination of Citicoline in Tablet Dosage Form by Thin Layer Chromatography-Densitometry

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A simple, precise, accurate and rapid thin layer chromatography-densitometry (TLC-Densitometry) has been developed and validated for determination citicoline in tablet dosage form. Normal phase thin layer chromatography plate (silica gel 60 F254) was used as stationary phase and methanol: water: Ammonia (8:1:1) as the mobile phase. Citicoline showed Rf value of 0,71 and evaluation was performed by densitometry (TLC-scanner) at 273 nm. The calibration curve was found to be linear with the correlation coefficient R= 0,9993. The limit of detection (LOD) and the limit of quantification (LOQ) of the method was respectively 15,744 µg/mL and 52,48 µg/mL. Precision (% RSD intraday was 0,86-1,41 and interday was 1,43-1,65). Recovery analysis were found to be 101,32%, 97,70% and 98,06 %. According to the results, this method was in accordance with good validation requirements.

Keywords: thin layer chromatography-densitometry; citicoline; validation.

VALIDATION OF ANALYSIS METHOD AND DETERMINATION OF CITICOLINE IN TABLET DOSAGE FORM BY THIN LAYER CHROMATOGRAPHY-DENSITOMETRY

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ABSTRACT

A simple, precise, accurate and rapid thin layer chromatography-densitometry (TLC-Densitometry) has been developed and validated for determination citicoline in tablet dosage form. Normal phase thin layer chromatography plate (silica gel 60 F254) was used as stationary phase and methanol: water: Ammonia (8:1:1) as the mobile phase. Citicoline showed Rf value of 0,71 and evaluation was performed by densitometry (TLC-scanner) at 273 nm. The calibration curve was found to be linear with the correlation coefficient R= 0,9993. The limit of detection (LOD) and the limit of quantification (LOQ) of the method was respectively 15,744 µg/mL and 52,48 µg/mL. Precision (% RSD intraday was 0,95-1,90 and interday was 1,43-1,65). Recovery analysis were found to be 101,32%, 97,70% and 98,06 %. According to the results, this method was in accordance with good validation requirements.

Keywords: TLC-Densitometry, Citicoline, Validation

Background

Citicoline consisting of ribose, pyrophosphate, cytosine (a nitrogenous base) and choline [1]. Citicoline can increase blood flow and oxygen to the brain and has been

given in the treatment of cerebrovascular disorders (including ischemic stroke), Parkinson's, and a head injury

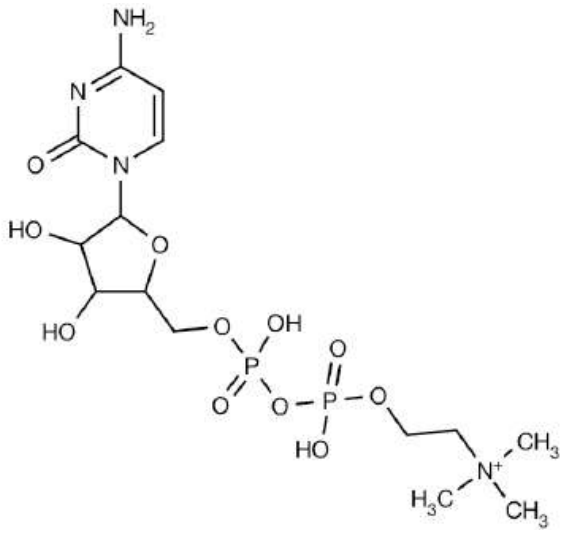

Figure 1. Structure of Citicoline

Analysis compound citicoline has been done by some researchers previously using the spectrophotometric UV-Vis (λ = 272 nm) [3], spectrophotometric methods UV-Vis in the area of visible light [4] and the use of high performance liquid chromatography [5]. Literature survey does not reveal any TLC Densitometry method for the determination of citicoline in tablet dosage forms. TLC densitometry method is simple, precise, and accurate for the determination citicoline in tablet dosage forms suitable good method validation requirements.

METHODS

EXPERIMENTAL

Materials and reagents

Citicoline standard was were provided by biometrik riset indonesia, Jakarta, Indonesia. Methanol, asetic acid, and ammonia (Merck®), aquades (Bratachem) were of pharmaceutical grade reagents. Commercial tablet containing 500 mg

citicoline were purchased from local pharmacy in Padang, West Sumatera, Indonesia (March, 2016).

Standar preparation

Stock standard solution was prepared by dissolving citicoline standard (4 mg) in 50 ml methanol (800µg/ml).

Sample Preparation

Two brands of tablets A and B were selected. Twenty tablets were weighed and the average weight was calculated. The tablets were then powdered and an amount equivalent to 40 mg of citicoline was dissolved in 100 volumetric flask, and approximately 25 ml of methanol was added. The mixture was ultrasonicated for 15 min. This solution was filtered through a filter paper. Then diluted to 100 mL with the same solvent to obtained 800 µg/mL citicoline

Instrumentation and chromatographic conditions

The TLC system consisted of a twin trough chamber (20 x 20 cm). Pre-coated silica gel 60 F254 TLC plates (20 x 20 cm, Merck, Darmstadt, Germany) were used as stationary phase. The standard and formulation samples of citicoline were spotted manually on pre-coated TLC plates 5 µL respectively. The mobile phase consists of Methanol:Water:Ammonia (8:1:1). Densitometric scanning was performed on CAMAG TLC Scanner 4 in Absorbance mode, operated by winCATS software. The spots were analyzed at wavelength 273 nm. Evaluation was performed using linear regression analysis of peak areas. citicoline was detected at Rf of 0.71.

RESULTS AND DISCUSSION

Optimum Condition

Table 1: Optimum condition for analysis of citicoline

Parameters	Data
Solute	Methanol
Eluen	Methanol:water:ammonia (8:1:1 v/v)
Stationary phase	Silica gel GF 254
λmax	273 nm

Table 1 showed optimum conditions for analysis citicoline using TLC. The mobile phase of methanol:water:ammonia (8:1:1 v/v) gave efficiency chromatogram with Rf value 0.71 for citicoline and a sharp and symmetrical peak (figure 1). The analytical wavelength, 273 nm, was chosen on the basis of the absorption spectrum recorded in the range 200-400 nm.

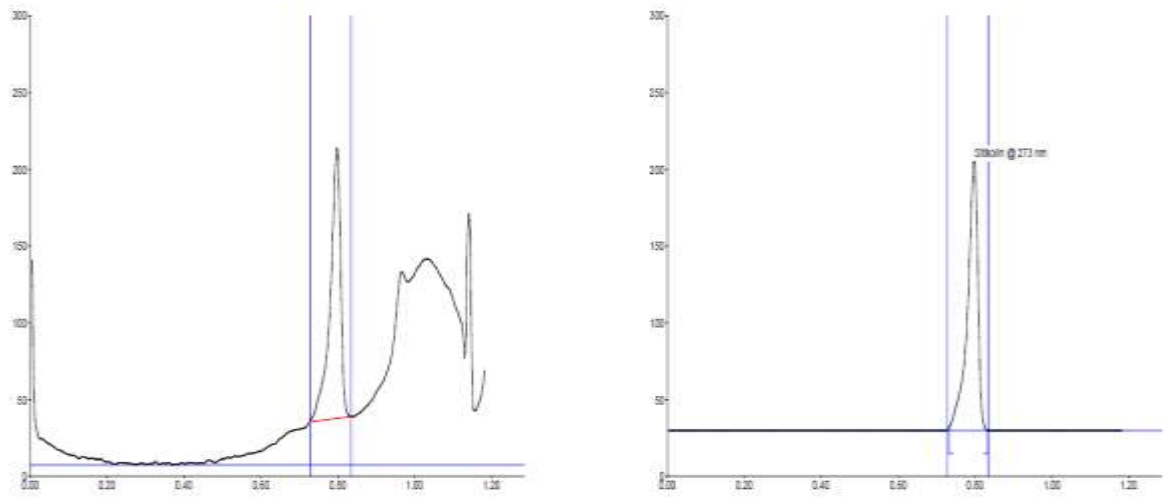

Figure 1. Densitogram of standard citicoline (400 µg/ mL); peak 1 (R_f = 0.71).

Table 2. Summary of Validation Parameters of Proposed TLC Densitometry Method

Parameter	Value
R_f	0,71
Linearity and range	Y= 1925 + 4,8663X with r=0,9993. 240 - 560 µg/ml
Limit of detection	15,74 µg/ml
Limit of quantification	52,48 µg/mL
Precision	
Intraday (% RSD)	0,95 – 1,90
Interday (%RSD)	1,43 – 1,65
% Accuracy (n=6)	98,06 -101,32

Table 3. Accuracy result of commercial tablets

Label claim (%) ± SD	Added (%)	Recovery
100,157 ± 0,738	40	101,32%
	80	97,70%
	120	98,06 %

Analysis of marketed formulations

A single spot at Rf 0,71 was observed in the densitogram of the drug samples extracted from tablets. There was no interference from the excipients commonly present in the tablets. The results, given in Table 4. The percentage recovery values for brand A and brand B were found to be 100,157 ± 0,738 % and 101,590 ± 0,879% respectively.

Table 4. Results of analysis of citicoline in pharmaceutical formulation

Sample	% recovery ± SD
Brand A	100,157 ± 0,738 %
Brand B	101,590 ± 0,879%

CONCLUSIONS

The developed TLC-Densitometry method is simple, precise, and accurate, and can be used for simultaneous determination of Citicoline in tablet dosage forms. The method was validated and in accordance with good validation requirements.

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