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Novel spectrophotometric method for selective determination of compounds in ternary mixtures (dual wavelength in ratio spectra)

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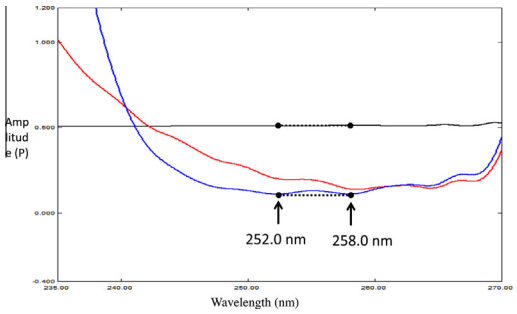
HIGHLIGHTS

- Simple spectrophotometric method for selective determination of ternary mixtures.
- No reported spectrophotometric method for the determination of the studied mixture.
- Merging ratio difference & dual wavelength for determination of ternary mixtures.
- Application of dual wavelength in the ratio spectrum.
- New hybrid method by merging two analytical methods used for binary mixtures.

GRAPHICAL ABSTRACT



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ABSTRACT

A simple selective spectrophotometric method for determination of compounds in ternary mixture was developed by combining the resolution power of two well-known methods that are commonly used for binary mixtures; namely ratio difference method and dual wavelength. The new method (dual wavelength in ratio spectra) was successfully applied for the determination of a ternary mixture of betamethasone dipropionate (BM), clotrimazole (CT) and benzyl alcohol (BA) in pure powder form and in their pharmaceutical preparation. The difference in amplitudes (ΔP) in the ratio spectra at 252.0 and 258.0 nm ($\Delta P_{252.0-258.0nm}$) corresponds to BM, while $\Delta P_{266.8-255.4nm}$ and $\Delta P_{254.2-243.5nm}$ corresponds to CT and BA, respectively. The method was validated as per the USP 2005 guidelines. The developed method can be used in quality control laboratories for routine analysis of compounds in ternary mixtures.

Introduction

Spectrophotometry can be regarded as a cheap alternative for the costly HPLC technique, however spectrophotometric determination of complex mixtures represents a challenging obstacle for analysts against the application of spectrophotometry. On the other hand chemometrics and multivariate analysis though efficient but complicated procedures are required starting from data preprocessing followed by careful preparation of several calibration and validation sets are mandatory for building a successful model [1].

Since the introduction of the ratio spectra and early application of the derivative ratio to determine compounds in binary mixtures by Salinas [2], which was further modified to the double divisor method for the determination of ternary mixture [3]. El-Bardicy et al. developed the ratio subtraction method to get rid of the interference of the component with extended spectrum in binary mixtures [4].

Ratio difference method has been recently introduced for the determination of binary mixture by Elzanfaly et al. [5–7], and

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further modified to the ratio difference at isoabsorptive points for simple single step determination of binary mixtures [8]. The ratio difference method was further modified to determine ternary mixtures after mathematical manipulation of the spectral data [9].

The article represents a simple merge between ratio difference and the dual wavelength where a special prerequisites was introduced for the wavelength selection in the ratio difference method. The new method was able to determine the three components of the ternary mixture.

The method was successfully applied for the accurate and selective determination of betamethasone dipropionate (BM), clotrimazole (CT) and benzyl alcohol (BA) in pure powder form as well as in their pharmaceutical formulation.

Theory of dual wavelength in ratio spectra (DWRS)

i. Ratio difference

The difference between two wavelengths in the ratio spectrum will get rid of the interference of the component used as the divisor [5,6,10].

ii. Dual wavelength

Application of dual wavelength can get rid of a component by subtracting two wavelengths showing similar absorptivities, for that components [11].

Hybridization of the two principles successfully developed a method that can completely remove interference of two components in a ternary mixture for the selective determination of a third component.

Experimental

Apparatus

SHIMADZU dual beam (Kyoto/Japan) UV–visible spectrophotometer model UV-1650 PC connected to IBM compatible and an hp1020 LaserJet printer. The bundle software, UVProbe version 2.21 (SHIMADZU) was used to process absorption and ratio spectra, the spectral band width was 2 nm and scanning speed was 2800 nm/min.

Reference samples

BM, CT and BA working standards were kindly supplied by Memphis Pharmaceutical Co. (Cairo, Egypt).

Pharmaceutical formulation

Lotriderm® cream manufactured by Memphis Co. for Pharmaceuticals and Chemical industries/Cairo/Egypt Under authority of: Schering-Plough Corporation/USA. Batch No. 111325, labeled to contain 0.064 g% BM (equivalent to 0.05 g% betamethasone), 1 g% CT and 1–3 g% BA as a preservative [12], was purchased from local market.

Materials and reagents

Methanol (Adwic) spectroscopic grade.

Standard solutions

(1) Stock standard solution of BM equivalent to 80 μg/mL betamethasone in methanol.
(2) Stock standard solution 1 mg/mL CT in methanol.
(3) Stock standard solution 2 mg/mL BA in methanol.

Procedures

Construction of calibration curves

Calibration curve of BM. Aliquots equivalent to 20–200 μg of BM from stock standard solution (80 μg/mL) were transferred into a series of 10 mL measuring flasks, and the volume was completed with methanol. The zero order spectra of the prepared solutions were divided by the spectrum of 1 mg/mL BA. The amplitudes of the ratio spectra were measured at 252.0 and 258.0 nm.

Calibration graphs relating the differences in the amplitudes at the chosen wavelength couple ($\Delta P_{252.0-258.0nm}$) to the corresponding concentration of BM were constructed, and the corresponding regression equation was computed.

Calibration curve of CT and BA. Aliquots equivalent to 200–2000 μg of CT from stock standard solution (1 mg/mL) and aliquots equivalent to 1–10 mg of BA from stock standard solution (2 mg/mL) were separately transferred into a series of 10 mL measuring flasks, and the volume was completed with methanol. The zero order spectra of the prepared solutions were divided by the spectrum of 20 μg/mL BM. The amplitudes of the ratio spectra were measured at 266.8, 243.5, 254.2 and 255.4 nm.

Calibration graphs relating the differences in the amplitudes at the chosen wavelength couple ($\Delta P_{266.8-255.4nm}$) to the corresponding concentration of CT were constructed, and the corresponding regression equations were computed.

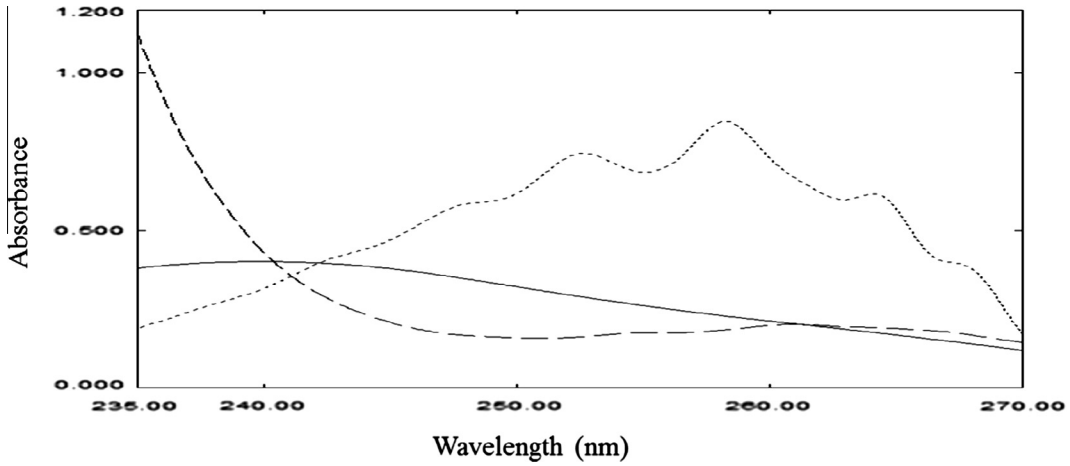

Fig. 1. Absorbance spectra of 10 μg/mL BM (—), 100 μg/mL CT (---) and 500 μg/mL BA (. . ..) in methanol.