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Determination of polythiazide in the presence of vanillin in Renese tablets by second-order derivative UV spectroscopy

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A second-derivative spectroscopic method for simultaneous determination of polythiazide and vanillin in Renese tablets has been developed. Methanol solutions of the tablets were analyzed by measurement of the amplitudes of the positive peak at 282 nm with respect to the negative peak at 270 nm for polythiazide, and the amplitude of the negative peak at 218 nm with respect to the base line for vanillin. The method allows specific, rapid and accurate determination of the binary mixture in the tested concentration range of 1–10 μg ml^{-1} for polythiazide and 1.5–15 μg ml^{-1} for vanillin.

Polythiazide is a well-known diuretic and antihypertension agent (1–3). Besides polythiazide, vanillin is one of the ingredients of Renese tablets. The chemical structures of polythiazide and vanillin are shown in Fig. 1.

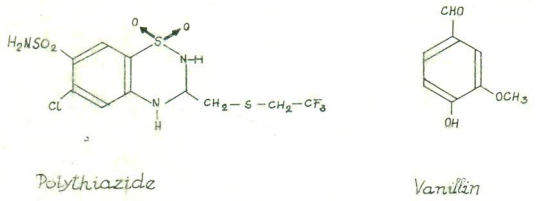

Fig. 1. Chemical structures of polythiazide and vanillin.

Several analytical methods have been described for assaying either polythiazide (4–10) or vanillin (11–16). These include the spectrophotometric method (4, 11, 13), liquid chromatography determination (5, 12), HPLC (6, 8, 9, 14, 16), thin-layer chromatography (10, 15) and determination by iodometric titrations (7). However, no method has been described for their simultaneous quantitation in two-component mixtures.

The zero-order UV spectra of polythiazide and vanillin overlap in the 200–280 nm region and the corresponding absorption maxima differ only by approximately 5 nm, which makes their simultaneous determination by conventional UV spectroscopy difficult. In recent years, the derivative transformation of spectral data has been shown to be

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a powerful tool for both quantitative and qualitative analyses of drug mixtures (17– 20) owing to its ability to eliminate matrix interferences (21, 22) as well as enhance resolution (19, 21, 23).

This paper describes the novel application of a second–order derivative method for simultaneous determination of polythiazide and vanillin in Renese tablets that overcomes the problems due to overlapping spectral bands and eliminates the need for separation procedures.

EXPERIMENTAL

Instrument

A Hewlett Packard Model 8452A Diode Array UV–VIS Spectrophotometer, equipped with a derivative module and 1–cm quartz cells, was used. The zero– and second-derivative spectra were recorded in a wavelength range of 190-350 nm.

Material

Methanol stock solutions of polythiazide (Pfiser Co., 200 μg ml^{-1}) and vanillin (Aldrich Chemical, 150 μg ml^{-1}) were prepared. A series of working standards (1–10 μg ml^{-1} polythiazide and 1.5–10 μg ml^{-1} vanillin) were obtained by appropriate dilution with methanol. Polythiazide–vanillin binary mixtures were also prepared so that the concentration ratio between the analyte and the potentially interfering components could span the range from 50–200% of their ratio in the assayed pharmaceutical preparation.

RESULTS AND DISCUSSION

The second-order derivative spectrum was recorded against methanol and peak amplitudes between the negative peak at 270 nm with respect to the positive peak at 282 nm ($^2D_{270, 282}$) and the negative peak at 218 nm with respect to the base line ($^2D_{218}$) were measured for polythiazide and vanillin, respectively.

The transformation of zero-order data resolved the broad absorption band of polythiazide into the component bands (Fig. 2 a, b) and new profiles clearly showed peaks where previously shoulders and inflection had been seen. Similarly (Fig. 2 c, d), the spectrum of vanillin was resolved into two sharp peaks. On observing the superimposed second-order derivative spectra of polythiazide and vanillin (Fig. 3 c), it became evident that, due to some interferences, not all of the peaks recorded would be useful in the quantitation of mixtures. The spectrum analysis revealed that the derivative signal $^2D_{270, 282}$ was specific for polythiazide and $^2D_{218}$ for vanillin; these amplitudes were selected because the respective signal magnitude of the interfering component was negligible at the chosen wavelength. Calculations were made from the calibration curve (Fig. 4), plotting the peak amplitude (mm) against concentration (mg ml^{-1}). The 95% confidence limits for the calibration graphs were typically ±0.01 μg ml^{-1} for polythiazide and ±0.02 μg ml^{-1} for vanillin at the central calibration concentration of 5.0 μg ml^{-1} for both components.

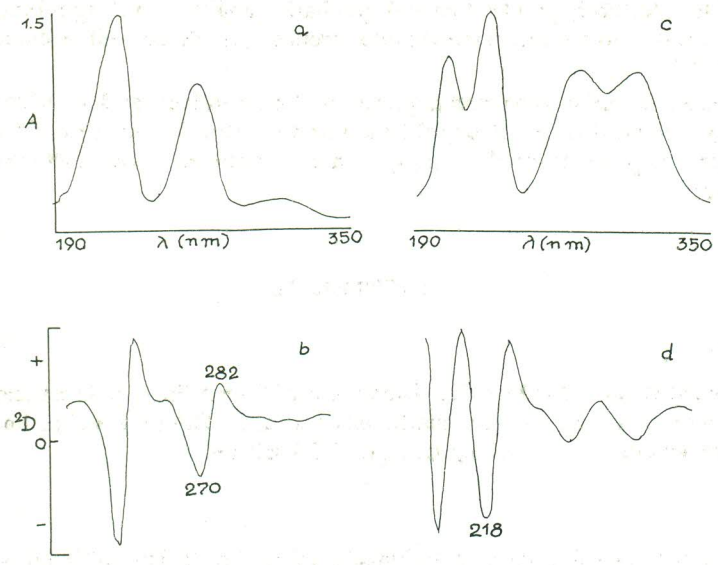

Fig. 2. Zero-order (a, c) and second-order (b, d) derivative UV spectra of polythiazide (a, b; 10 μg ml^{-1}) and vanillin (c, d; 7.5 μg ml^{-1}) in methanol. The amplitudes of the positive peak at 282 nm with respect to the negative peak at 270 nm, and the negative peak at 218 nm with respect to the base line, were used for quantitation.

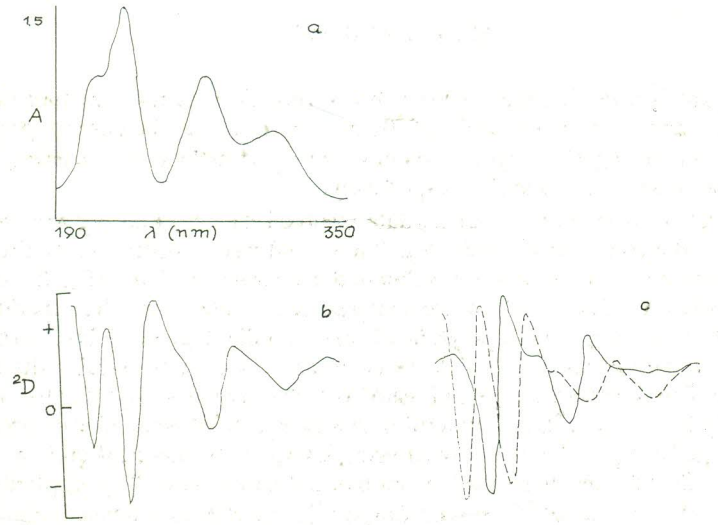

Fig. 3. Zero-order (a) and second-order (b) derivative UV spectra of a binary mixture of polythiazide (10 μg ml^{-1}) and vanillin (7.5 μg ml^{-1}) in methanol. (c) Second-order derivative of polythiazide (solid line) and vanillin (broken line) overlaid to show areas of spectral overlap.

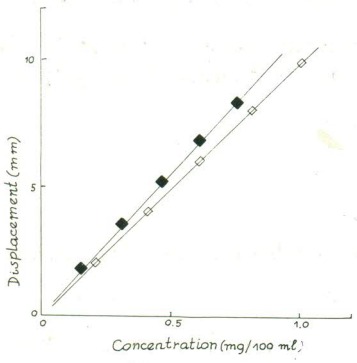

Fig. 4. Second-order derivative calibration curve for polythiazide for the amplitude of the positive peak at 282 nm with respect to the negative peak at 270 nm (❑) and for vanillin for the amplitude of the negative peak at 218 nm with respect to the base line (■).

The zero-order and second derivative spectra of a polythiazide-vanillin mixture (at the same concentration as in Fig. 3 c) are presented in Fig. 3 a, b; in particular, $^2D_{270,\,282}$ for polythiazide and $^2D_{218}$ for vanillin are unchanged. Linear correlations were obtained between the respective derivative amplitude of polythiazide-vanillin mixture and the corresponding component concentration over the range of 1–10 μg ml^{-1} for polythiazide and 1.5–10 μg ml^{-1} for vanillin. The least-square regression equation (for polythiazide) was:

$$y = 0.107x + 0.002, \quad n = 5, \quad r = 0.9996,$$

and for vanillin:

$$y = 0.733\,x + 0.038, \quad n = 5, \quad r = 0.9915,$$

where y is concentration in mg ml^{-1} and x is ^2D in mm.

Interaction studies for constant polythiazide or vanillin levels, but varying polythiazide or vanillin concentrations, showed that the selected derivative amplitude did not depend on the presence of the other component; in fact, the recovery was in every instance close to quantitative.

The results of the analyses of a Renese dosage form are presented in Table I. The relative standard deviations for both drugs were less then 2%.

Table I. Analyses of Renese tablets labelled to contain 0.625% polythiazide and 0.47% vanillin

Batch	Average recovery, %; CV (n = 5) Polythiazide	Vanillin
1	102.1 (1.4)	99.4 (1.1)
2	101.2 (0.9)	97.5 (1.3)
3	98.7 (1.5)	97.1 (1.0)
4	102.4 (0.7)	98.2 (1.9)

We conclude that the described second-order derivative spectroscopic method is suitable for being applied to quality control since it permits rapid, precise, accurate and low-cost analyses of polythiazide-vanillin mixtures in Renese tablets without extraction proce-

dures, and is easily applied to routine usage, thus confirming its potentials as an analytical tool for simultaneous quantitation of drugs in multicomponent pharmaceutical preparations.

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SAŽETAK

Određivanje politiazida u prisutnosti vanilina u Renese tabletama upotrebom derivacijske spektrofotometrije drugog reda

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Za simultano određivanje politiazida i vanilina u Renese tabletama upotrebljena je derivacijska spektroskopija drugog reda. Mjerenjem amplitude pozitivnog pika na 282

nm u odnosu na negativni pik na 270 nm određivan je politiazid, dok je za određivanje vanilina upotrebljen negativni pik na 218 nm u odnosu na baznu liniju. Ovaj postupak omogućava specifičnu, brzu i točnu analizu binarne smjese u koncentracijskom području 1–10 μg ml^{-1} za politiazid i 1,5–10 μg ml^{-1} za vanilin.

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