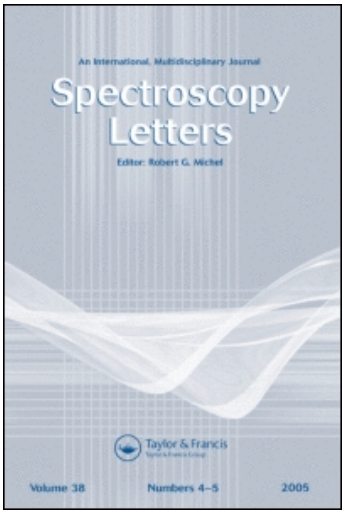

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Dual Wavelength Spectrophotometry. Determination of 1,2,4-Benzenetricarboxylic Acid and Benzenepentacarboxylic Acid

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DUAL WAVELENGTH SPECTROPHOTOMETRY. DETERMINATION OF 1,2,4-BENZENE-TRICARBOXYLIC ACID AND BENZENEPENTACARBOXYLIC ACID

KEY WORDS: Dual wavelength, benzenepolycarboxylic acid

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ABSTRACT

Dual wavelength ultraviolet spectrophotometry was utilized for the determination of 1,2,4-benzenetricarboxylic and benzenepentacarboxylic acids in their simultaneous solution.

INTRODUCTION

Dual wavelength spectrophotometry was proposed by Chance [2,3] two decades ago for spectral measurements of turbid samples. Since then, several methods based on this technique have been reported. Shibata, et al.,[4,5] have utilized dual wavelength spectrophotometry for the analyses of mixtures.

The purpose of this investigation was to apply dual wavelength spectrophotometry to the analysis of simultaneous solutes whose individual absorption spectra are very closely related.

In principle, the method consists of passing two light beams with wavelengths $\lambda_1 \neq \lambda_2$ through a cell containing the solution and measuring $\Delta A = A_{\lambda_2} - A_{\lambda_1}$. Since both beams pass through the same cell, errors encountered in the conventional single wavelength method caused by cell positioning, cell constants, and differences between reference and sample solutions due to concentration and turbidity, are eliminated.

METHOD

Benzenepentacarboxylic acid (BPCA) and 1,2,4-benzenetricarboxylic acid (1,2,4-BTCA) were commerically obtained (Aldrich Chemical Company) and were recrystallized twice from their respective solutions in hot acetone/benzene. The spectra of these two compounds at concentrations of 75 mg/liter in 0.15% HCl solution obtained individually with a Perkin Elmer Model 356 spectrophotometer are shown in Figure 1.

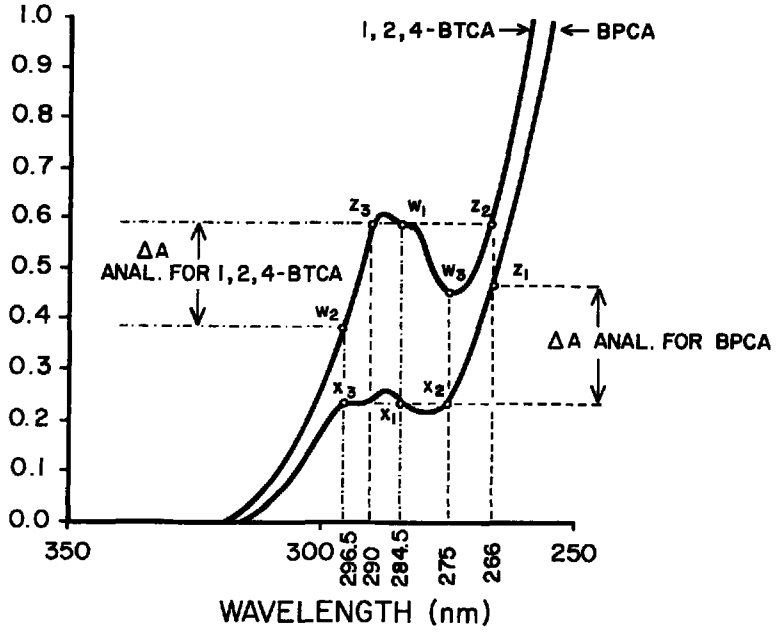

FIGURE 1

Absorption Spectra of Benzenepentacarboxylic Acid (BPCA) and of 1,2,4-Benzenetricarboxylic Acid (1,2,4-BTCA), 75 mg/l in 0.15% HCl.

Determination of λ_1 and λ_2 for the Analysis of 1,2,4-BTCA in the Presence of BPCA

As shown in Figure 1, an analytical wavelength of 284.5 nm was chosen for 1,2,4-BTCA, and a perpendicular line was drawn to the abscissa. This line intercepts the BPCA curve at point x_1 at which a line parallel to the abscissa is drawn. The intercepts x_2 and x_3 are points at which the absorbance due to BPCA equals to that at point x_1. Thus ΔA when λ_2

= 284.5 nm and λ_1 = 296.5 nm or 275 nm will be due to 1,2,4-BTCA. To maximize ΔA analytical for 1,2,4-BTCA ($A_{w_1} - A_{w_2} > A_{w_1} - A_{w_3}$), it is advantageous to choose λ_1 = 296.5 nm.

Alternatively, if λ_2 (analytical) is set on the spectrophotometer at 284.5 nm while λ_1 (reference) is scanned from 350 nm to 250 nm for various concentrations of BPCA, isosbestic points at which A = 0 are obtained. Figure 2 depicts these scans.

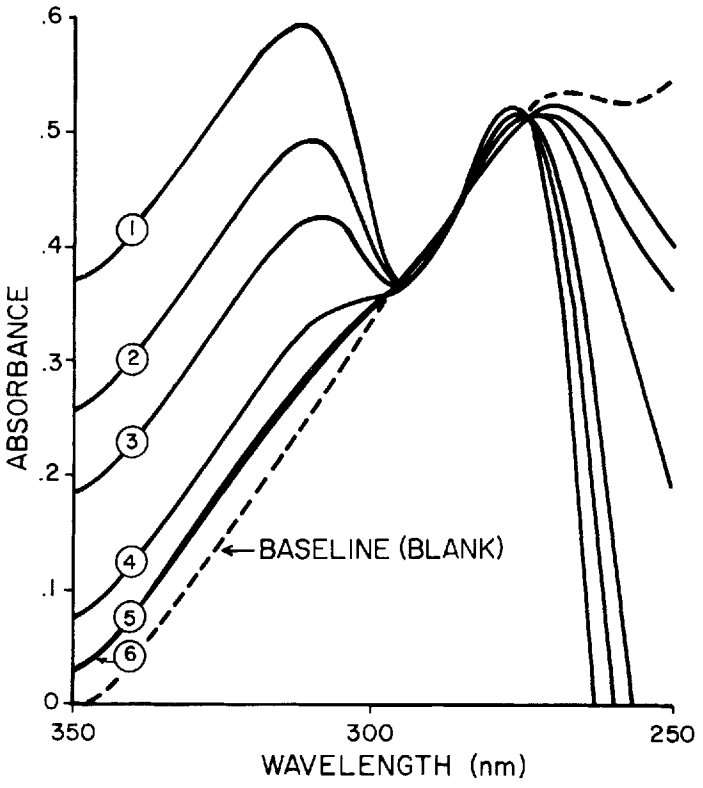

FIGURE 2

Determination of Isosbestic Points for Various Concentrations of BPCA. (1) 100, (2) 70, (3) 50, (4) 20, (5) 10, and (6) 5 mg/l BPCA in 0.15% HCl.

The final and precise choice of λ_1 and λ_2 was made by adjusting λ_1 and/or λ_2 to give minimal change in absorbance as the concentration of BPCA is varied (see Figure 3).

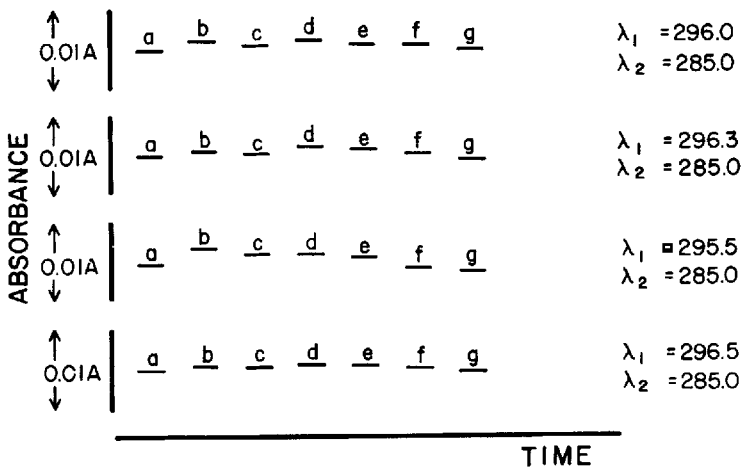

FIGURE 3

Precise Determination of λ_1 and λ_2 for the Analysis of 1,2,4-BTCA in the presence of BPCA. (a)0, (b) 100, (c) 70, (d) 50, (e) 20, (f) 10 and (g) 5 mg/l BPCA in 0.15% HCl.

Determination of λ_1 and λ_2 for the Analysis of BPCA in the Presence of 1,2,4-BTCA

As seen in Figure 1, the choice of λ_2 (analytical) for BPCA must be made on the slope of the absorption spectrum of BPCA (point z_1) so as to yield points z_2 and z_3 on the 1,2,4-BTCA spectrum where $A_{z_2} = A_{z_3}$, and so that ΔA analytical for BPCA will be of appropriate magnitude suitable for analysis. Thus ΔA when λ_2 = 266 nm and λ_1 = 290 nm will be due to BPCA.

Alternatively, if λ_2 (analytical) is set on the spectrophotometer at 266.0 nm while λ_1 (reference) is scanned from 350 nm to 250 nm for various concentrations of 1,2,4-BTCA, isosbestic points at which A = 0 are obtained as shown in Figure 4.

The final and precise choice of λ_1 and λ_2 was made by adjusting λ_1 and/or λ_2 to give minimal change in absorbance as the concentration of 1,2,4-BTCA is varied (see Figure 5).

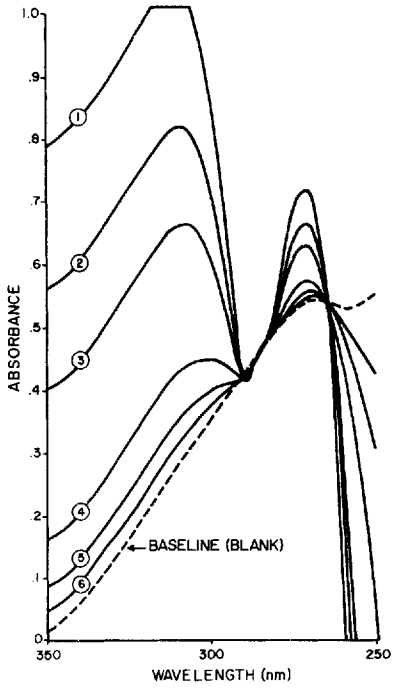

FIGURE 4

Determination of Isosbestic Points for Various Concentrations of 1,2,4-BTCA. (1) 100, (2) 70, (3) 50, (4) 20, (5) 10, and (6) 5 mg/l 1,2,4-BTCA in 0.15% HCl.

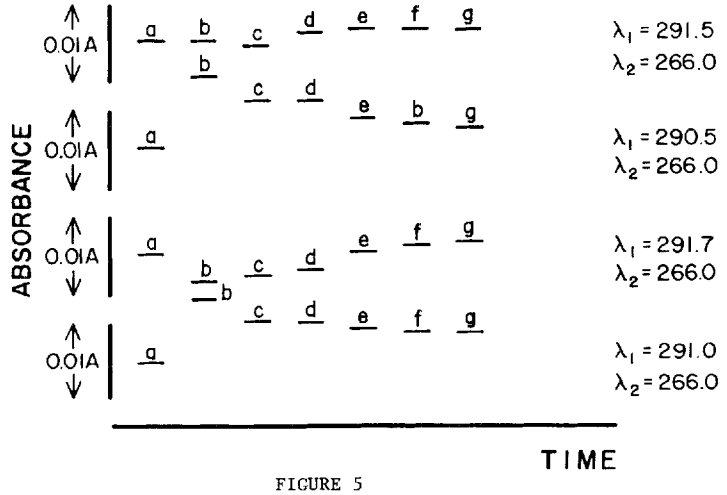

FIGURE 5

Precise Determination of λ_1 and λ_2 for the Analysis of BPCA in the Presence of 1,2,4-BTCA. (a) 0, (b) 100, (c) 70, (d) 50, (e) 20, (f) 10 and (g) 5 mg/l 1,2,4-BTCA in 0.15% HCl.

RESULTS AND DISCUSSION

From Figures 3 and 5, the following wavelengths were chosen: λ_1 (reference) = 296.5 nm and λ_2 (analytical) = 285.0 nm for the analysis of 1,2,4-BTCA in the presence of BPCA, and λ_1 (reference) = 291.5 nm and λ_2 (analytical) = 266.0 nm for the analysis of BPCA in the presence of 1,2,4-BTCA.

An unmistakable linearity between ΔA and concentration is shown in Figure 6. This is remarkable especially since an analytical wavelength was chosen on a steep slope of the absorption spectrum of a substrate.

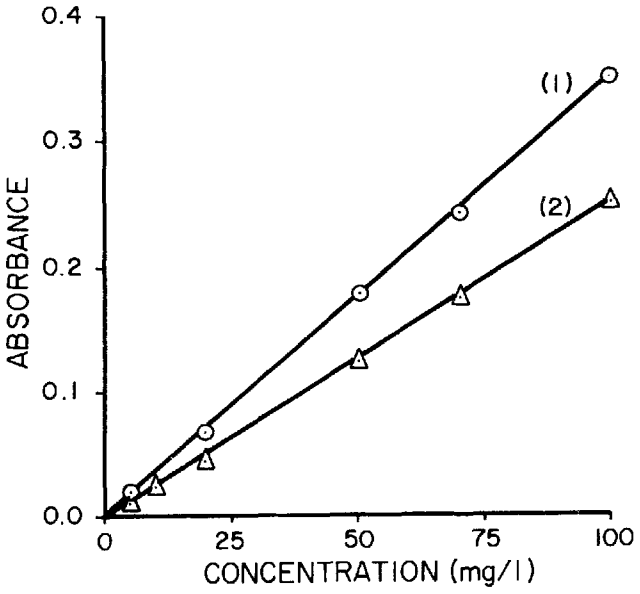

FIGURE 6

Calibration Curves. (1) For BPCA, λ_1 = 291.5 nm, λ_2 = 266.0 nm (2) For 1,2,4-BTCA; λ_1 = 296.5 nm, λ_2 = 285.0 nm.

In order to test the application of this dual wavelength spectrophotometry to the analysis of a simultaneous solution of 1,2,4-BTCA and BPCA each in the concentration range of 0 to 100 mg/l, six solutions were made having different concentrations of substrates. Table 1 describes the results of this test.

TABLE 1

Determination of 1,2,4-BTCA and of BPCA in Their Simultaneous Solution in 0.15% HCl

Sample Number	Added (mg/l) 1,2,4-BTCA	BPCA	Found (mg/l) 1,2,4-BTCA	BPCA	% Recovery 1,2,4-BTCA	BPCA
1	50.0	5.0	49.5	5.0	99.0	100.0
2	20.0	10.0	19.5	10.0	97.5	100.0
3	10.0	50.0	9.3	48.5	93.0	97.0
4	100.0	20.0	105.0	20.0	105.0	100.0
5	70.0	100.0	70.0	95.5	100.0	95.5
6	5.0	70.0	4.5	69.5	90.0	99.3

Dual wavelength spectrophotometry is an extremely useful method for the analysis of two components in simultaneous solution. When the appropriate wavelengths are chosen, the analysis is rapid and does not require the use of matched cells as in the conventional method. Furthermore, as described in this work, substrates having very similar spectra can be analyzed utilizing dual wavelength spectrophotometry.

The dual wavelength spectrophotometric analysis of polycarboxy-polyphenolic benzenes is currently under investigation for utilization in the study of organic-mineral interactions.

ACKNOWLEDGMENTS

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