because it is applicable to the analysis of diagnostic meals containing barium sulfate.

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* Present address: Lafayette Pharmacal Inc., Lafayette, IN 47902

Rapid Assay of Hydrogen Peroxide Solution (USP XVIII) *via* UV Spectrophotometry

J. TIETJEN and A. MANCOTT

Abstract □ Assay of hydrogen peroxide solution (USP XVIII) was accomplished by direct UV spectrophotometric analysis. This determination can be done in less than 30 sec. with a calibrated spectrophotometer and is comparable in accuracy to the USP XVIII titrometric procedure.

Keyphrases □ Hydrogen peroxide solution—analysis □ UV spectrophotometry—analysis

The spectrum and absorbance of hydrogen peroxide solutions in the UV range have been investigated (1–6). Bessun and Spitz (6) reported that dilute hydrogen peroxide solutions (10 mcg./ml.) followed Beer's law when measured at wavelengths between 195 and 220 nm. The molar absorptivity of hydrogen peroxide solutions decreases rapidly at wavelengths above 275 nm., and no appreciable absorbance is recorded at wavelengths greater than 375 nm.

To perform a direct UV spectrophotometric assay on 3% hydrogen peroxide solutions (USP XVIII) without employing time-consuming and less accurate dilution techniques, a wavelength of 304 nm. was chosen, which is substantially higher than that of maximum absorption. The very low molar absorptivity at 304 nm. permits direct measurement of hydrogen peroxide solutions in the 2.0–4.5% range.

The UV spectrophotometric assay reported here can be done in a matter of seconds with a calibrated spectrophotometer. It requires no chemicals or reagents and is comparable in accuracy to the USP XVIII titrometric procedure (7).

EXPERIMENTAL

Apparatus—Spectra and absorbance measurements were made with a spectrophotometer[1]. Matched cells with a 1-cm. optical path were used.

Reagents and Chemicals—Hydrogen peroxide solution (30%)[2,3], 0.1 *N* potassium permanganate solution[4], and hydrogen peroxide solutions USP XVIII[5] were assayed. All other reagents used were of the highest commercial grade available.

Procedure—The spectrophotometer (slit width 5 Å, deuterium lamp) was adjusted to zero absorbance with distilled water. The wavelength was calibrated against a mercury emission spectrum and set at 304.0 nm. with the controls locked. Solutions of hydrogen peroxide were prepared by dilution of 30% hydrogen peroxide and standardized with 1.0 *N* potassium permanganate solution. Absorbances of the standard hydrogen peroxide solutions were measured. Samples of hydrogen peroxide solutions USP XVIII were assayed by measuring their absorbance at 304.0 nm. and comparing with the standards. The same samples were also assayed according to the USP XVIII titrometric procedure.

RESULTS AND DISCUSSION

Absorbance readings for standardized hydrogen peroxide solutions in the concentration range of 2.1–4.3 g./100 ml. were obtained (Table I). A graph of concentration *versus* absorbance was linear, with a slope of 4.386. The concentration of hydrogen peroxide in a given sample is found from Eq. 1:

$$C = 4.386A \qquad \text{(Eq. 1)}$$

where A = absorbance, and C = grams of H_2O_2 per 100 ml. of solution.

[1] Bausch & Lomb, model 505.
[2] Fisher certified reagent.
[3] B&A reagent, Allied Chemical.
[4] Fisher certified reagent.
[5] Parke-Davis & Co., Detroit, Mich.

Table I—Absorbance of Known Hydrogen Peroxide Solutions

Concentration of H_2O_2, g./100 ml.	Absorbance at 304.0 nm.
4.335	0.988
3.902	0.890
3.468	0.790
3.034	0.691
2.601	0.593
2.167	0.495

The USP XVIII standard for hydrogen peroxide solution (7)[6] is that 100 ml. of solution contains not less than 2.5 g. and not more than 3.5 g. of H_2O_2.

Determinations of seven random samples of hydrogen peroxide solutions were performed by both spectrophotometric and titrometric procedures. Agreement between both methods was within ±2 parts per 1000. The error introduced by working on a steep area of the absorption band is small when compared to the errors encountered using dilution techniques. Also, an appreciable saving in time, labor, and materials results by direct measurement of the hydrogen peroxide solution. An accurate calibration of the wavelength (either with a mercury emission spectrum or a solution of known hydrogen peroxide concentration) is essential for the reproducibility of this method[7]. By locking the controls of the spectrophotometer once it has been calibrated, many samples of hydrogen peroxide can then be assayed in a very brief period.

[6] Preservatives in hydrogen peroxide solution USP XVIII total not more than 0.05%. The stabilizer did not interfere with the spectrophotometric determination reported here.

[7] Calibration of the spectrophotometer using a sample of hydrogen peroxide whose concentration was found titrometrically requires more time but gives more accurate results.

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Fluorometric Determination of Amphetamines with 3-Carboxy-7-hydroxycoumarin

J. T. STEWART and D. M. LOTTI

Abstract □ A fluorometric method of analysis for amphetamines based on the interaction between aliphatic and/or cyclic amines and 3-carboxy-7-hydroxycoumarin to yield highly fluorescent coumarin–amine salts was investigated. The procedure was applied to the analysis of several amphetamines and amphetamine mixtures. Comparison of the coumarin method to other existing fluorometric methods for amines in the analysis of amphetamines was performed.

Keyphrases □ Amphetamines—analysis □ 3-Carboxy-7-hydroxycoumarin–cyclic amine reaction—fluorescence □ Fluorometry—analysis

The objective of this investigation was to evaluate a fluorometric method of analysis for amphetamines based on the interaction between aliphatic and/or cyclic amines and 3-carboxy-7-hydroxycoumarin to yield highly fluorescent coumarin–amine salts. The assay procedure applied to aliphatic and cyclic amines was reported previously from this laboratory (1). Use of the method permits determination of trace quantities of amines based upon fluorometric measurement of coumarin–amine salt, even in the presence of excess fluorescent coumarin reagent.

In this study, the method was applied to the analysis of several amphetamines and amphetamine mixtures. A study demonstrating the usefulness of the procedure to commercial dosage forms containing amphetamines was made. Comparison of this procedure to other existing fluorometric methods for amines was performed.

EXPERIMENTAL

Apparatus—Fluorescence spectra and measurements were made with a spectrophotofluorometer[1]. Clear, fused quartz cells (12.5 × 47 mm.) were used as sample cells.

Reagents and Chemicals—Powdered samples of dextroamphetamine sulfate[2], methamphetamine hydrochloride[3], benzphetamine hydrochloride[4], chlorphentermine hydrochloride[5], methylphenidate hydrochloride[6], phenmetrazine hydrochloride[7], and phendimetrazine tartrate[8] were used in the analytical procedure for preparation of standard solutions. 3-Carboxy-7-hydroxycoumarin was synthesized according to the procedure of Woods and Sapp (2). All other chemicals used were the highest grade of the commercially available materials.

Solutions of amphetamine salts (8×10^{-5} *M*) and 3-carboxy-7-hydroxycoumarin (6.8×10^{-4} *M*) were prepared by dissolving

[1] Aminco-Bowman equipped with slit arrangement No. 3.
[2] Smith Kline & French Laboratories, Philadelphia, Pa.
[3] Mann Research Laboratories, New York, N. Y.
[4] The Upjohn Laboratories, Kalamazoo, Mich.
[5] Warner-Lambert Research Institute, Morris Plains, N. J.; research affiliate of Warner-Chilcott Laboratories.
[6] Ciba Pharmaceutical Co., Summit, N. J.
[7] Geigy Pharmaceuticals, Ardsley, N. Y.
[8] Ayerst Laboratories, Inc., New York, N. Y.