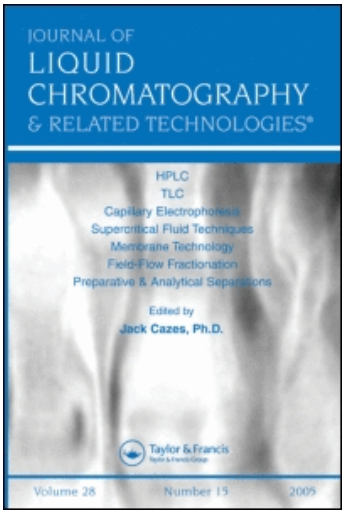

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DETERMINATION OF DIIODOHYDROXYQUIN IN DOSAGE FORMS BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

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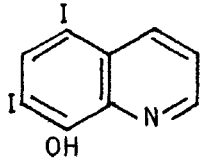
ABSTRACT

A new, simple and sensitive method for the determination of diiodohydroxyquin in dosage forms using high-performance liquid chromatography (HPLC) has been developed. Diiodohydroxyquin is extracted with isopropanol, the solution is evaporated to dryness, the residue is reconstituted in methanol containing 0.01% phenyl salicylate (internal standard) and injected. A plot of peak height ratio (diiodohydroxyquin/internal standard) vs. concentration of diiodohydroxyquin gave a straight line. The column used was a commercially available μ Bondapak C_{18} cartridge 10 cm x 8 mm, and the mobile phase was a methanol: 0.05 M orthophosphoric acid (75:25) mixture at a flow rat of 3 cm^3 per minute. Retention times for diiodohydroxyquin and phenyl salicylate were 3.85 min. and 3.0 min. respectively.

INTRODUCTION

Diiodohydroxyquin (5,7-diiodo-8-quinolionol)I is an amebicide used orally to treat amoebic as well as some bacterial infections of the gastrointestinal tract. It has been reported of value in cases of lambliasis and in balantidial dysentery.

Several methods have been reported for the quantitative determination of Diiodohydroxyquin including colorimetry (1), polarography (2), the oxygen flask method (3), (4), Iodometry (5), non-aqueous titration (6) and spectrophotometry (7). A high-performance liquid chromatographic (HPLC) method has been described (8) for the assay of Iodo chlorhydroxyquin and Hydrocortisone in ointments and creams, but no work seems to have been reported for the determination of I by HPLC. The present work describes a rapid, accurate and sensitive method for the estimation of I in dosage forms using high-performance liquid chromatography (HPLC).



I

EXPERIMENTAL

Apparatus

The HPLC unit used (Model ALG/GPC 244 V, Waters Associates, U.S.A.) had a system controller (Model 720 System Controller, Water Associates, Milford, Milford, Mass. U.S.A., a data Module (Model 730 Data Module, Waters Associates, Milford, Mass., U.S.A., an autosampler (Model 710 B Autosampler, Water Associates, Milford, Mass., U.S.A., two pumps (Model 45 Pumps, Waters Associates, Milford, Mass., U.S.A.) a U.V. detector (Model 480 U.V. detector, Milford, Mass., U.S.A.) and a radial compression separation system (Z. Module Radial Compression Separation System, Milford, Mass., U.S.A.). The column was a commercially microparticular bonded octadecylsilane material (μ Bondapak C 18 Cartridge, Waters Associates, Milford, Mass., U.S.A.)

Reagents

Diiodohydroxyquine (5,7-diiodo-8-hydroxy quinoline) (Riedel-De Haen A G, Seelze, Hannover, West Germany) phenyl salicylate (B.D.H. Chemicals Ltd., Poole England United Kingdom) and orthophosphoric acid (Orthophosphoric Acid (85%) - Riedel-De Haen A G, Seelze, Hannover West Germany) were used as obtained without further purification. Methanol (Fluka A G, Chemische Fabrik, CH 9470, Buchs,

Switzerland) and isopropanol (E. Merck, Darmstadt, West Germany) were of spectroscopic grade. Water used was double distilled in all all-glass still (Corning "Mega Pure" distillation system).

HPLC Parameters

The mobile phase was a methanol: 0.05 M orthophosphoric acid (75:25) mixture at a flow rate of 3 cm^3 per minute. The U.V. detector was set at 254 mm and the optical density setting was 0.05 AUFS. The chart speed was 1 cm per minute. The mobile phase was prepared daily by pre-mixing and passing through a 0.45 m aqeous-type membrane filter (Millipore, Bed ford, Mass., U.S.A.) under vacuum, using a solvent clarification kit (Millipore, Bed ford, Mass., U.S.A.).

Preparation of the Standard Curve

A solution was prepared by dissolving 0.05 g of Diiodohydroxyquin in 100 ml methanol by sonication and subsequent warming of the flask. A solution containing 0.100 g phenyl salicylate (internal standard) in 100 ml was also prepared in methanol. 1 ml samples of 5,7 Diiodo-hydroxyquin and 0.1% phenyl salicylate in methanol to contain 0.005%, 0.01%, 0.015%, 0.02%, 0.025%, 0.03%, 0.035%, 0.04%, and 0.045% 5,7-Diiodohydroxyquin. Triplicate injections of 20 µl each were made onto the column. The peak

heights of diiodohydroxyquin and the internal standard were measured and the peak height ratio (diiodohydroxyquin/ internal standard) was plotted against the concentration of diiodohydroxyquin.

RESULTS AND DISCUSSION

Determination of Diiodohydroxyquin in Dosage Forms (Synthetic mixtures)

About 300 mg of accurately weighed diiodohydroxyquin were mixed with the excipients talc (2g), starch (30g) and lactose (60g). Four aliquots (about 10mg, 20mg, 30mg, and 40mg) were accurately weighed into 100ml. volumetric flasks, 100 ml isopropanol were added and heated to just below the solvent's boiling point for 1 hour. The flasks were then transferred to a ultrasonic bath containing warm water for half an hour, then removed and allowed to cool to room temperature.

The volume was adjusted to 100 ml with isopropanol to replace the amount lost through evaporation. About 12 ml of the solution were transferred to a clean centrifuge tube and spun at 3000 rpm for 5 minutes to bring down any insoluble matter. 10ml were pipetted into a test tube and evaporated to dryness at $80^{o}C$ under a stream of nitrogen. After cooling, the samples were reconstituted in spectromethanol containing 0.01% of the internal/standard and injected onto the column.

Determination of Diiodohydroxyquin in Commercial Tablets

Diiodohydroxyquin was determined in commercial tablets containing 650 mg of the active ingredient.

Five tablets were accurately weighed and finely powdered in a mortar. Accurately weighed amounts of the powder (approximately 20mg) were transferred to 100ml flasks. 100ml isopropanol were added and warmed on a hot plate to just below the solvents boiling point (82°C). The flasks were then transferred to an ultrasonic bath containing warm water for 1 hour sonication, then removed and allowed to cool to room temperature. The volume was adjusted to 100 ml with isopropanol to replace the amount lost through evaporation. About 12ml of the solution were transferred to a clean centrifuge tube and spun at 3000 rpm for 5 minutes to bring down any insoluble matter. 10ml were pipetted into a test tube and evaporated to dryness at 80°C under a stream of nitrogen. After cooling, the samples were reconstituted in spectromethanol containing 0.01% of the internal/standard and injected onto the column.

Fig.1 shows a typical chromatograph obtained in HPLC and indicates the retention times for diiodohydroxyquin and phenyl salicylate. The standard curves were obtained by accurately measuring the peak heights of diiodohydroxyquin and internal standard, calculating the ratio and

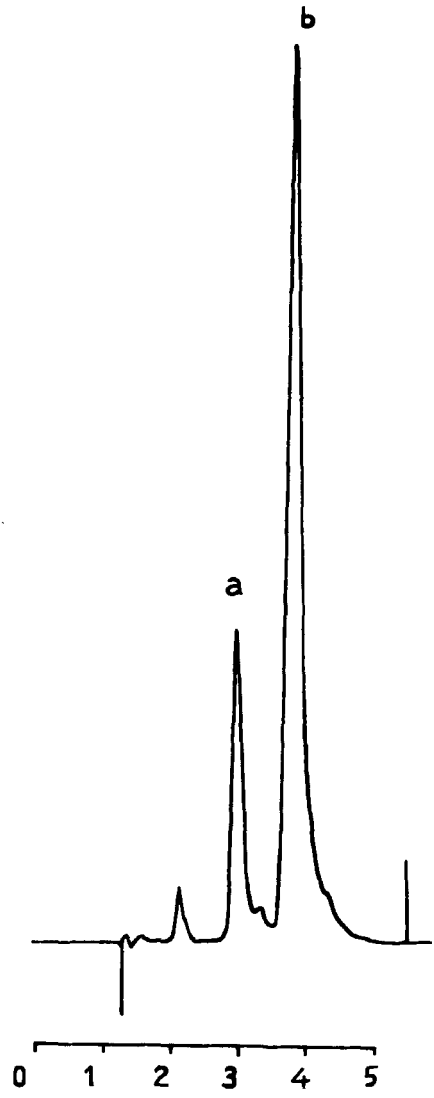


Fig.1

Typical chromatogram

a phenyl salicylate

b diiodohydroxyquin

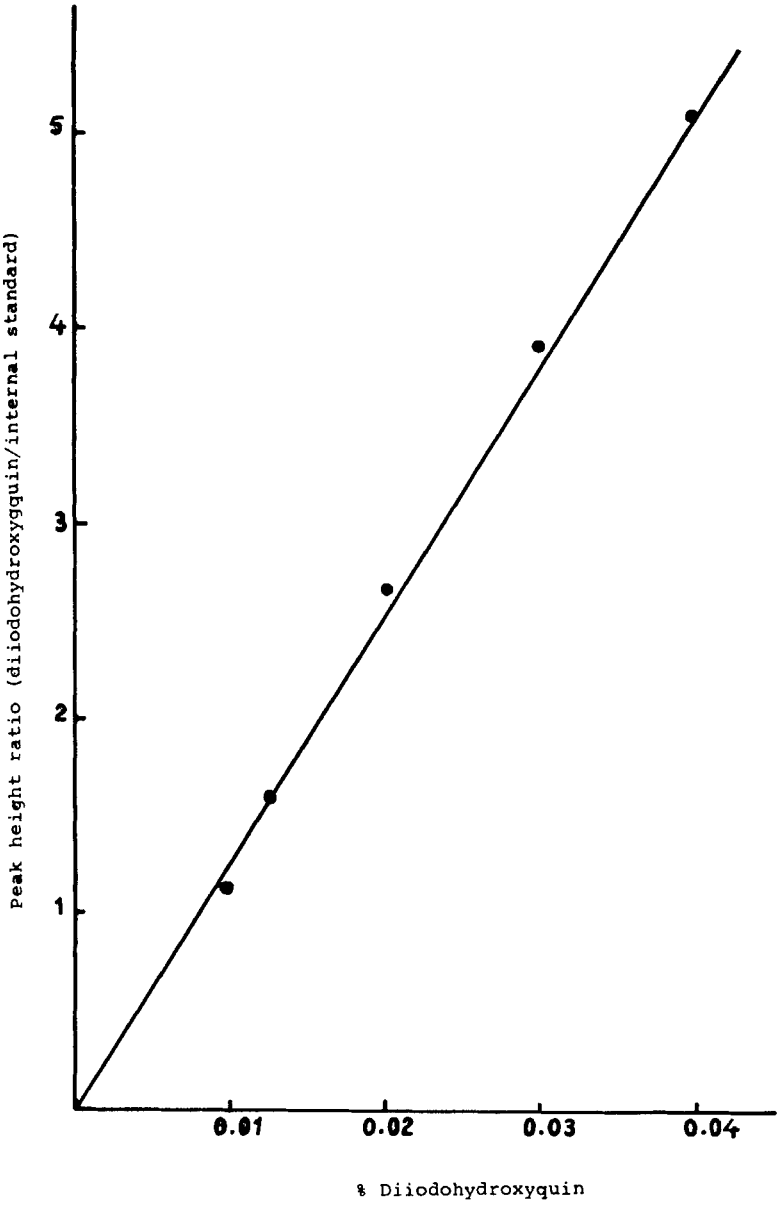


Fig.2

Relation between peak height ratio and concentration of diiodohydroxyquin. (r = 0.98)

TABLE 1

ANALYSIS OF DIIODOHYDROXYQUIN IN DOSAGE FORMS (SYNTHETIC MIXTURES)

	% Diiodohydroxyquin (mg/100 ml)		
Sample	Added	Found[a] ± SD.)	% Recovery[b] (±SD.)
1	9.3	8.3 (0.14)	89.2 (1.5)
2	15.1	14.7 (0.2)	97.37(2.0)
3	22.9	21.4 (0.2)	93.6 (1.14)
4	32.1	30.4 (0.66)	93.9 (1.67)

a mean of three determinations

b mean recovery of three determinations.

plotting against percentage diiodohydroxyquin. Fig.2 shows the straight line relationship between the concentration of diiodohydroxyquin and the peak height ratio (diiodohydroxyquin/internal standard). Table I shows the percent recovery of diiodohydroxyquin from synthetic mixtures.

The results of applying the new HPLC method to the determination of diiodohydroxyquin in two commercial tablets are shown in Table 2. Good recoveries are achieved by applying the new HPLC method.

TABLE 2

ANALYSIS OF DIIODOHYDROXYQUIN IN DOSAGE FORMS (Tablets)

Sample	Labeled amount	% Labeled amount*
A	650mg / tablet	96.97
B	650mg / tablet	98.66

*Each result represents the average of at least three estimations.

In conclusion, a new rapid, accurate and sensitive HPLC method for the determination of diiodohydroxyquin in dosage forms is described.

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