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PMR Spectrometric Analysis of Chloramphenicol in Pharmaceutical Preparations

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Key Words PMR Spectrometry, Chloramphenicol pmr analysis

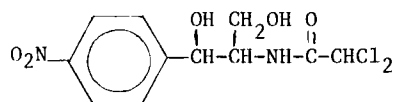
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Abstract

A new method is developed using PMR for the assay of chloramphenicol and its pharmaceutical forms. The method is rapid, accurate, precise and helps in confirming the identity and purity of the drug. The principle of the method involves comparing the integral of the well-defined doublet system of chloramphenicol spectrum (positioned at 7.6δ and 7.5δ) to that of the sharp singlet (positioned at 6.25δ) of maleic acid which is used as internal standard.

Introduction

Chloramphenicol D-(-)-threo-1-(p-nitrophenyl)-2-(2,2-dichloroacetamido)-1,3 propanediol is an official antibiotic in both B.P.



(1973)⁽¹⁾ and USP XIX (1975)⁽²⁾. The official method of assay of chloramphenicol (B.P. 1973) is a U.V. spectrometric method which has also been employed to determine the antibiotic in pharmaceutical preparation in methods involving separation prior to quantitation (3,4).

Different colorimetric methods (5,6,7) and microbiological methods (8) has been reported for the assay of chloramphenicol and its dosage forms, but most of these methods are not only time-consuming but also not very specific. This work describes a new method involving the application of PMR spectroscopy.

Experimental

A varian T60-A NMR spectrometer was used throughout this study.

Materials and methods

Materials

Standard chloramphenicol (B.D.H. Chemicals Ltd., Poole England), chloramphenicol capsules and oral suspension were used as the samples; maleic acid (B.D.H. Chem. Ltd., Poole, England), internal standard, and chloroform and dimethylsulfoxide as solvents.

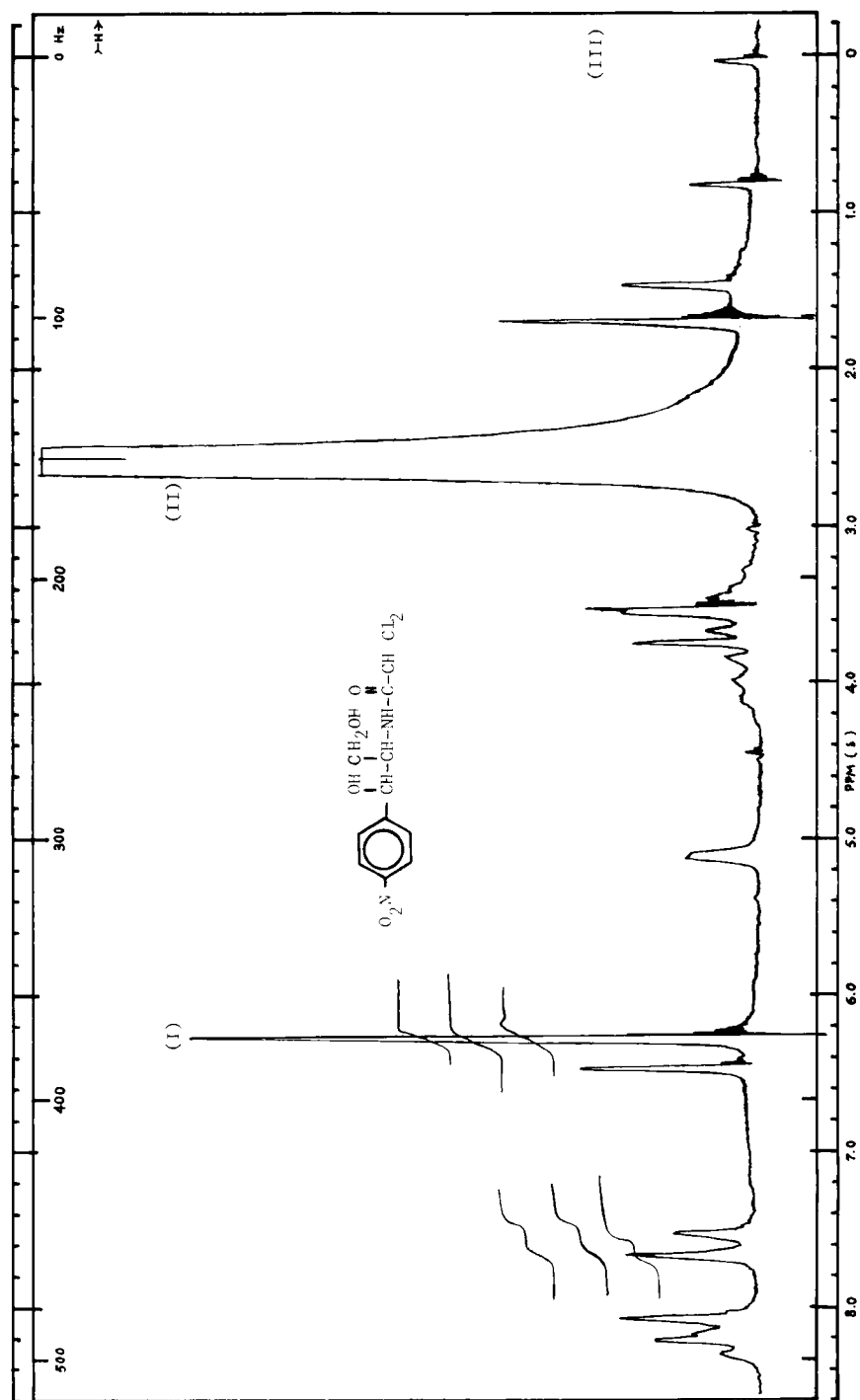


Figure 1 - NMR spectra of chloramphenicol, maleic acid (I) in dimethyl sulfoxide (DMS) and TMS (tetramethylsilane) [1].

ProcedureFor standard chloramphenicol

Dissolve 100 mg of chloramphenicol and 50 mg of maleic acid in 2 ml of dimethylsulfoxide, transfer 0.4 ml of the clear solution to an PMR tube. Place the tube in an NMR spectrometer, adjusting the spin rate to eliminate spinning side bands as much as possible; and record the spectrum. Integrate the peaks of interest (the aromatic protons of chloramphenicol at 7.6 δ and 7.5 δ and the the CH = CH protons of maleic acid at 6.25 δ) and record the mean of the three integrations. The amount of chloramphenicol may be calculated as follows:

$$\text{mg of chloramphenicol} = \frac{A_1}{A_2} \times \frac{EW_1}{EW_2} \times W_2$$

A_1 = integral value of the aromatic doublet of chloramphenicol.

A_2 = integral value of the CH=CH singlet of maleic acid.

EW_1 = mol. wt. of chloramphenicol/2.

EW_2 = mol. wt. of maleic acid/2.

W_2 = wt. (mg) of maleic acid.

Samples of pure chloramphenicol in the range of 100 - 150 mg were analysed (maleic acid in approximately the same amount was added to each tube as an internal standard). The mean recovery in ten separate experiments is shown in Table 1.

TABLE 1 - Analysis of chloramphenicol standard, by PMR.

Sample No.	internal standard added mg	chlorampheni- col added mg	Found	Recoveries %
1	50	100	99.81	99.81
2	50	105	106.12	101.07
3	50	110	110.29	100.26
4	50	115	114.82	99.84
5	50	120	119.90	99.91
6	50	120	118.31	98.59
7	50	125	124.20	99.36
8	50	130	132.20	101.69
9	50	140	144.21	103.00
10	50	150	148.12	98.67
Average =				100.219
SD =				1.367

For chloramphenicol capsules*

Dissolve a quantity of the mixed contents of 20 capsules equivalent to 250 mg of chloramphenicol, and 50 mg of maleic acid in 2 ml of dimethylsulfoxide. In an NMR tube pipette 0.4 ml. of the solution, run the PMR spectrum of the solution and proceed as previously described under standard chloramphenicol. Results are shown in Table 2.

* Park Davis & Co., Penly Pool, Man, U.K.

TABLE 2 - Analysis of chloramphenicol capsule, by PMR.

Sample No.	internal standard added mg	chlorampheni- col declared mg	Found	Recoveries %
1	50	250	245.71	98.28
2	50	250	246.22	98.49
3	50	250	252.93	101.17
4	50	250	244.52	97.81
5	50	250	253.11	101.24
6	50	250	248.21	99.28
7	50	250	244.10	97.64
8	50	250	247.25	98.90
9	50	250	254.10	101.64
10	50	250	253.29	101.32
			Average	= 99.57
			S.D.	= 1.595

For chloramphenicol palmitate oral suspension **

Extract a volume of the suspension equivalent to 125 mg of chloramphenicol with 3 portions of 5 ml of chloroform, evaporate the extract to dryness. Dissolve the residue in 2 ml of dimethylsulfoxide containing 50 mg of maleic acid.

** Park Davis & Co., Ponly Pool, Man, U.K.

In an NMR tube, place 0.4 ml of the solution, run the PMR spectrum and proceed as previously mentioned under standard chloramphenicol. Results are shown in Table 3.

Results and Discussions

Table I shows the percent recoveries obtained when the proposed method is used for the assay of authentic chloramphenicol. The results demonstrated good precision (average recovery = 100.219, s.d. = 1.367.

TABLE 3 - Analysis of chloramphenicol oral suspension, by PMR

Sample No.	internal standard added mg	chlorampheni- col declared mg	Found	Recoveries %
1	50	125	122.51	98.00
2	50	125	127.25	101.80
3	50	125	123.53	98.82
4	50	125	123.51	98.80
5	50	125	128.26	102.60
6	50	125	122.72	98.18
7	50	125	121.89	97.51
8	50	125	126.13	100.90
9	50	125	122.44	97.95
10	50	125	127.29	101.83
Average =				99.64
S.D. =				2.008

When the proposed method is used for the assay of two batches of chloramphenicol capsules and chloramphenicol palmitate, oral suspensions gave reasonable results as indicated in Table 2 and 3 respectively.

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