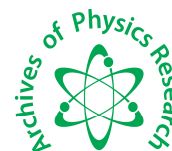




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UV-Visible and infrared analysis of commercial drug and its mixtures

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ABSTRACT

The study of characterization of commercial drug was made through spectroscopic technique. Chloramphenicol and Ranitidine is the commercial drug mostly used for the treatment of bone marrow depression and mycrocytic-anemia. Many researches have been made on structural characterization of this commercial drug. The use of this drug interaction with spectroscopic study has been made to find the possible influence of one drug over the other.

Key Words: FTIR, UV study, commercial drug, characterization.

INTRODUCTION

Pharmacology can be defined as the branch of science that includes history, source, physicochemical properties, dosage forms, methods of administration, absorption, distribution, mechanism of action, physiological and biochemical changes produced within the body, biotransformation and excretion, clinical uses, and adverse effects of the drugs. There is some structure activity relationship with the drug. The activity of drug is intimately related to its chemical structure. Knowledge about the chemical structure a drug is useful for Synthesis of new compounds with more specific actions and fewer adverse reactions and understanding the mechanism of drug action [1]. The main objective of pharmaceutical drug analysis is to offer not only a ready reference, but also an intermediate level for the convenient analysis of pure pharmaceutical substances and their respective dosage forms wherever applicable.

Spectroscopy has recently emerged as a novel bio-medical technique that can potentially reveal a wealth of qualitative and quantitative information about a given pharmaceutical sample. The increasing use of FT-IR spectroscopy demonstrates that this technique is a valuable tool owing to its high sensitivity in detecting changes in functional groups of tissue components, such as membranes, Proteins nucleic acids, as well as for complex drug material.

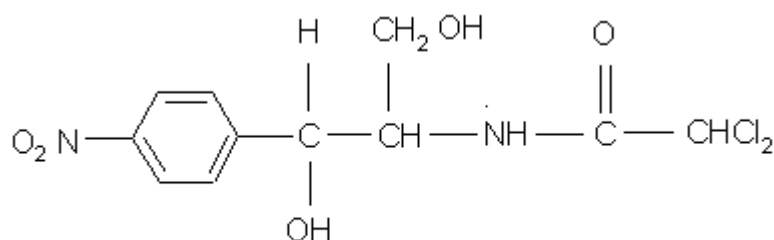
When infrared light is passed through a sample of an organic compound some of the frequencies are absorbed while the others are transmitted. The variation of infrared absorbance against frequency gives the infrared spectrum. The infrared spectrum of a compound is essentially the superposition of absorption bands of specific functional groups. For qualitative analysis, the

absorptions in specific frequency regions can be correlated with specific stretching and bending motions of these groups. Thus by interpreting of the spectrum it is possible to state whether certain functional groups are present or not in a given sample material. Many researches have been carried out to study the structural characterization of drugs using spectroscopic study [2-5]. Keeping in view of this above facts an attempt has been made to study. (1) Structural conformation and to assign the frequencies of different functional group of commercial tablet Chloramphenicol and Ranitidine using FTIR technique. (ii) To find the absorption position in UV-Visible spectrum, and to find any interaction existing among the drugs.

MATERIALS AND METHODS

2.1. Description of Chloramphenicol commercial drug

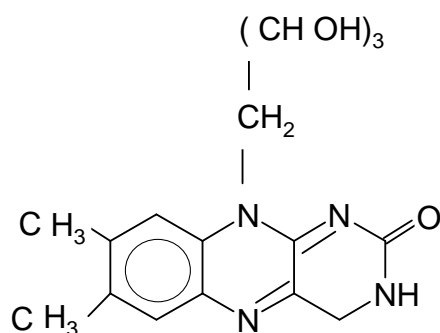
The molecular formula of Chloramphenicol is $C_{11}H_{12}Cl_2N_2O_5$ having the structure as



The use of chloramphenicol has been shown to cause bone marrow depression. This toxicity is usually reversible if the drug is discontinued, but in rare cases patients develop a plastic anemia a bone marrow disease which is often irreversible and fatal.

2.2. Description of Riboflavin commercial drug

The molecular formula of Riboflavin is $C_{17}H_{20}N_4O_6$ having structure as



Riboflavin is used to prevent riboflavin deficiency and to treat ariboflavinosis. Whenever possible, poor dietary habits should be corrected and many clinicians recommended administration of multivitamin preparations containing riboflavin inpatients with vitamin deficiencies since poor dietary habits often result in concurrent deficiencies.[6]

Riboflavin may be useful in treating microcytic-anemia that occurs in patients with a familiar metabolic disease associated with splenomegaly and glutathione reeducates deficiency.

2.3. Sample preparation

The samples were made into fine powder. The drug powder samples and KBr (all solid dry state) were kept in an oven at 25 °C in order to remove most bound water that might interfere with the measurement. Approximately 5mg of the sample is mixed with 100mg of dried KBr and then pressed into a clear pellet of 13mm diameter and 1mm thickness [7]. Absorbance spectra were recorded using Nicolet Avatar –360 FT-IR spectrometer equipped with a KBr beam splitter and a DTGS detector installed at the sophisticated analytical Instrument facility, I.I.I, Chennai. The operating conditions for this FT- IR spectrometer are given below: For each spectrum 100 scans were co-added, at a spectral resolution of 4cm⁻¹.

RESULTS AND DISCUSSION

3.1. UV-Vis Spectral measurements of commercial drugs and its mixtures

The UV-Vis spectral measurements are carried out on Chloramphenicol and Riboflavin. The sample is dissolved in methanol and a solution of 2% concentration is prepared and a smooth spectrum is obtained. The spectral recording shows a peak at 278nm for chloramphenicol. In the case of Riboflavin three absorbance was measured at 446, 270 and 222nm with the absorbance maximum of 0.16 0.43 and 0.39.

To investigate and interaction of the drug existing among the drugs , UV-Visible spectra were recorded at different mixtures of concentration and are listed in Table 1.

In the case of 1:1 mixtures shift in λ_{max} of Riboflavin was observed due to interaction of chloramphenicol. An increase in absorbance recorded in the case of 275nm and 199nm showing the influence of one drug on another. With the addition of chloramphenicol twice that of riboflavin the absorbance peaks decreases further indicating the chloramphenicol influence is much more than that of Riboflavin. This can be confirmed by the ratio of 2:1 mixtures of the drugs i.e increasing the twice the concentration of riboflavin will make not variance on the absorbance value and the λ_{max} value indicating the influence of the drug chlramphenicol is actively involved in interaction with Riboflavin.

TABLE .1 Variation of Absorbance Of λ_{Max} of Chloramphenicol , Riboflavin and its Mixtures

DRUGS	λ_{max}	Absorbance	λ_{max}	Absorbance	λ_{max}	Absorbance
Chloramphenicol	278	0.615	-	-	-	-
Riboflavin	446	0.167	270	0.431	222	0.393
1:1 mixture of Chloramphenicol and Riboflavin	444	0.143	275	1.657	199	2.246
1:2 mixture of Chloramphenicol and Riboflavin	443	0.986	275	1.372	200	1.724
2:1 mixture of Chloramphenicol and Riboflavin	445	0.053	270	0.352	216	0.326

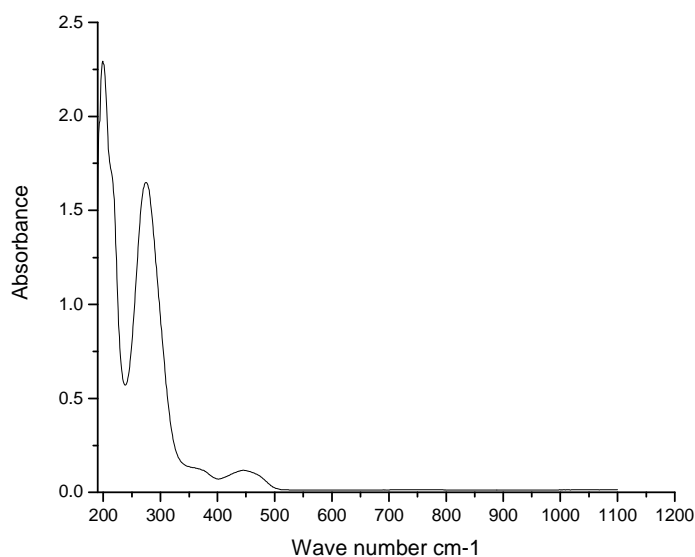


Fig.1 UV-Visible Spectra of Commercial drug Chloramphenicol and Riboflavin-1:1 mixture

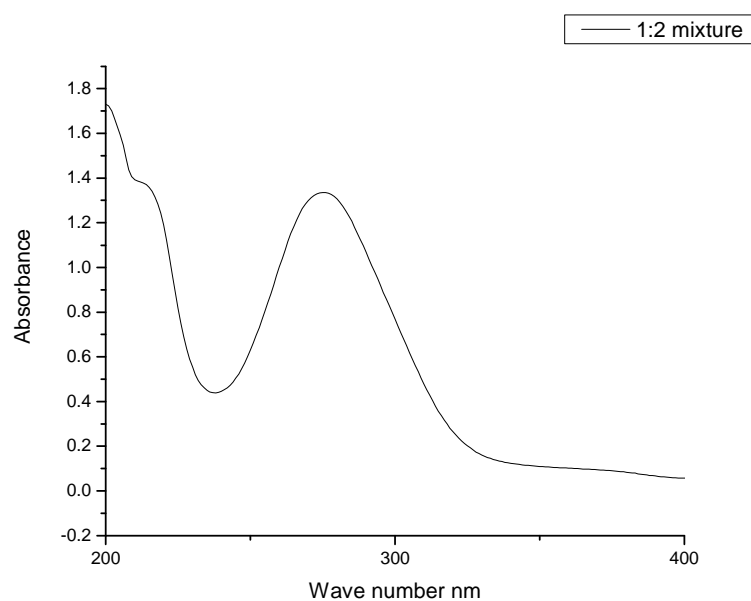


Fig.2 UV-Visible Spectra of Commercial drug Chloramphenicol and Riboflavin-1:2 mixtures

3.2. Infrared Spectral analysis of Commercial Drug –Chloramphenicol and Riboflavin

The FT-IR spectra of Commercial drug Chloramphenicol and Riboflavin are given in Table.2. The spectra recorded for commercial drug and their mixtures at different proportions are presented in Fig.4. By observing the position, shape and relative intensities of the vibrational bands in FT-IR spectra of the drug Chloramphenicol/Riboflavin a satisfactory vibrational band assignment has been made. As solid or liquid, a broad band of high intensity was observed in the region $\sim 3400\text{ cm}^{-1}$ exhibiting the presence of water of crystallization in solid state spectra.

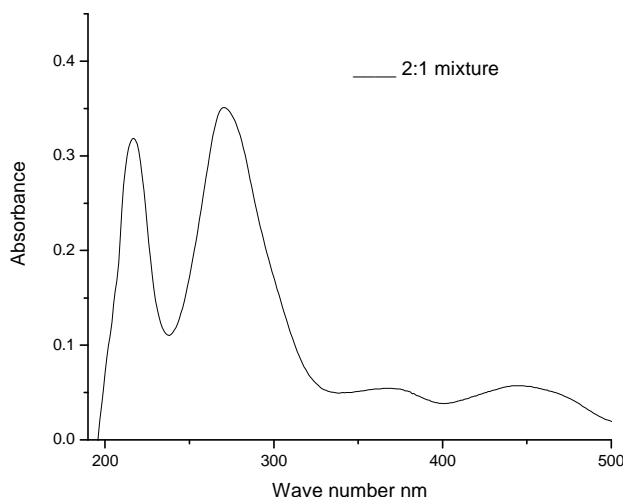


Fig.3 UV-Visible Spectra of Commercial drug Chloramphenicol and Riboflavin- 2:1 mixture

3.2.1 C-N stretching / C-O and C=O vibration

C-O stretching absorption of primary alcohol is strong and occurs in the region $1071-1065\text{cm}^{-1}$. For aromatic and unsaturated amines, two bands are observed at $1360-1250\text{cm}^{-1}$. The spectra of benzene and derivative substituted compound exhibit a band in the region $1220-1210\text{cm}^{-1}$. The band region 1222cm^{-1} in IR and 1226cm^{-1} in Raman has been assigned to C-N symmetry stretching of the compound [8]. In analogy with this bands observed at 1220cm^{-1} in the FT-IR of riboflavin are assigned due to O-H bending vibrations.

Saturated aliphatic ketones absorb strongly in the range $1700-1680\text{cm}^{-1}$ and this band shifted from its expected position by a number of parameters due to the adjacent position. The bands due to C-O stretching vibrations are strong and occur in the region 1260cm^{-1} . In fluorouracil the bands observed at 1621cm^{-1} in IR. A strong absorption band due to C=O stretching occurs in the region $\sim 1371\text{cm}^{-1}$. Because of high intensity and relative interference free region in which it occurs, this band is reasonably easy to recognize.

TABLE.2 Tentative Frequency Assignment of Commercial drug Chloramphenicol Mixed at Different Ratios with Riboflavin

Chloramphenicol drug	1:1 ratio	1:2 ratio	2:1 ratio	Frequency Assignment
Wave number cm^{-1}				
3401	3352(s)	3356(s)	3372(s)	OH stretching
2927	2925(m)	2910(s)	2925(m)	Aromatic C-H stretching
1732	-	-	-	C= stretch amide II
1647	1686(vs)	1686(s)	1685(s)	Amide I
1550	1561(s)	1561(s)	1559(s)	N-O stretch(ArNO_2)
1071	1066(s)	1064(s)	1065(s)	C-O stretch (Primary alcohol)
876	842(m)	817(s)	818(w)	C-N stretch
1346	1348(s)	1348(s)	1348(m)	C=O stretching mode

TABLE. 3 Tentative Frequency Assignments of Commercial Drug Riboflavin Mixed at Different ratios with Chloramphenicol

Riboflavin Drug	1:1 ratio	1:2	2:1	Frequency Assignment
Wave number cm^{-1}				
3400(s)	3352(s)	3356(s)	3372(s)	N-H and O-H stretching, and possibly intra molecular hydrogen bonded -OH groups
1650(s)	1686(vs)	1686(s)	1685(s)	C=O Diene, triens; C=N-
1562(s)	1561(s)	1561(s)	1559(s)	Aryl H- vibration frequencies
1450(s)	1454(m)	1412(s)	1414(s)	-C-H deformations
1388(s)				-CH ₃ symmetrical deformations
1240(m)	1244(s)	1243(s)	1245(m)	-O-H bending
1065(s)	1066(s)	1064(s)	1065(s)	C-O stretching
990(m)	-	-	-	C-O stretching
817(m)	842(m)	817(s)	818(w)	Meta di substituted aromatic ring ortho-di substituted aromatic ring due to -H, moving out of plane of the benzene ring.

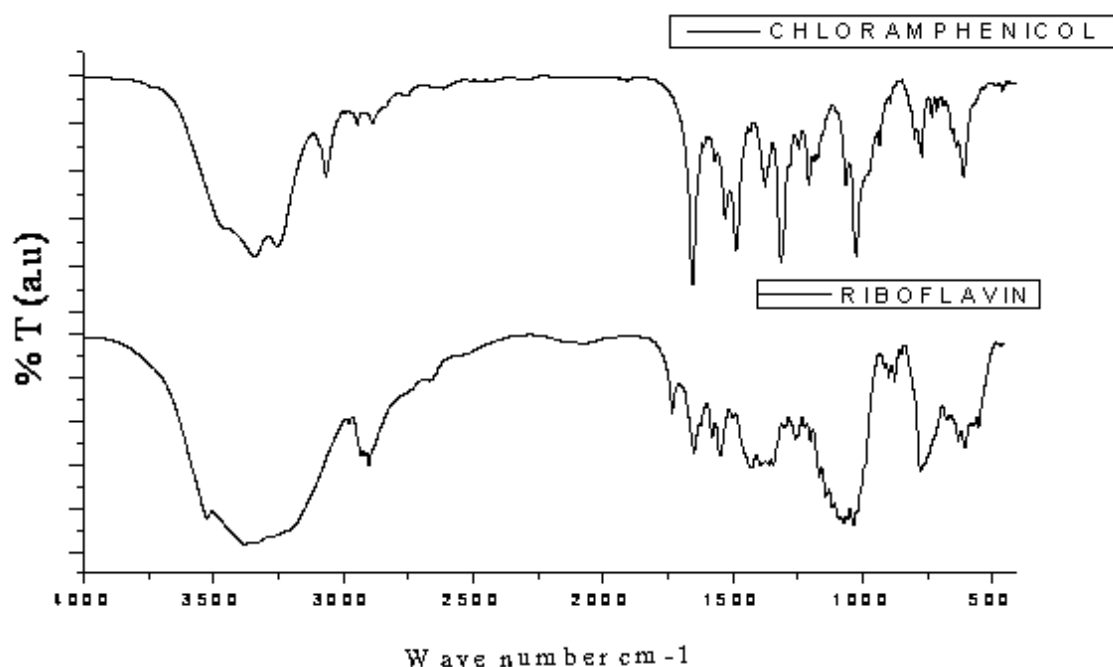


Fig.4. FTIR Spectrum of Commercial drug Chloramphenicol and Riboflavin

A band of medium strong intensity may be found in the region $1325\text{-}115\text{ cm}^{-1}$ for aliphatic ketones. [9] Hence the bands at $\sim 1065\text{ cm}^{-1}$ and 990 cm^{-1} in FT-IR spectrum are allotted to C-O stretching vibration.

3.2.2. Ring Vibration

For aromatic six member rings, there are 2 or 3 bands in this region due to skeletal vibrations, the strongest usually being about 1500 cm^{-1} . In acetaminophen the bands of strong intensity $\sim 1641\text{ cm}^{-1}$ are assigned to asymmetric and symmetric vibrations of the ring C-N stretching vibrations. The ring symmetry and asymmetry bending vibrations results in bands $\sim 808\text{ cm}^{-1}$ in FT-IR [10]. The spectral region $1290\text{-}1000\text{ cm}^{-1}$ is occupied by a number of C-H in plane deformation vibrations which are sharp and of weak to medium intensity. Apart from these, this region also contains vibrations due to C-C and C-O stretching. The symmetrical deformation of the hydrogen atoms of a methyl group results in an absorption band in the range $1385\text{-}1370\text{ cm}^{-1}$

which is stable in position [11]. In the present case the band observed $\sim 1388\text{ cm}^{-1}$ in the FT-IR spectrum is allotted to be due to CH_3/CH_2 deformation.

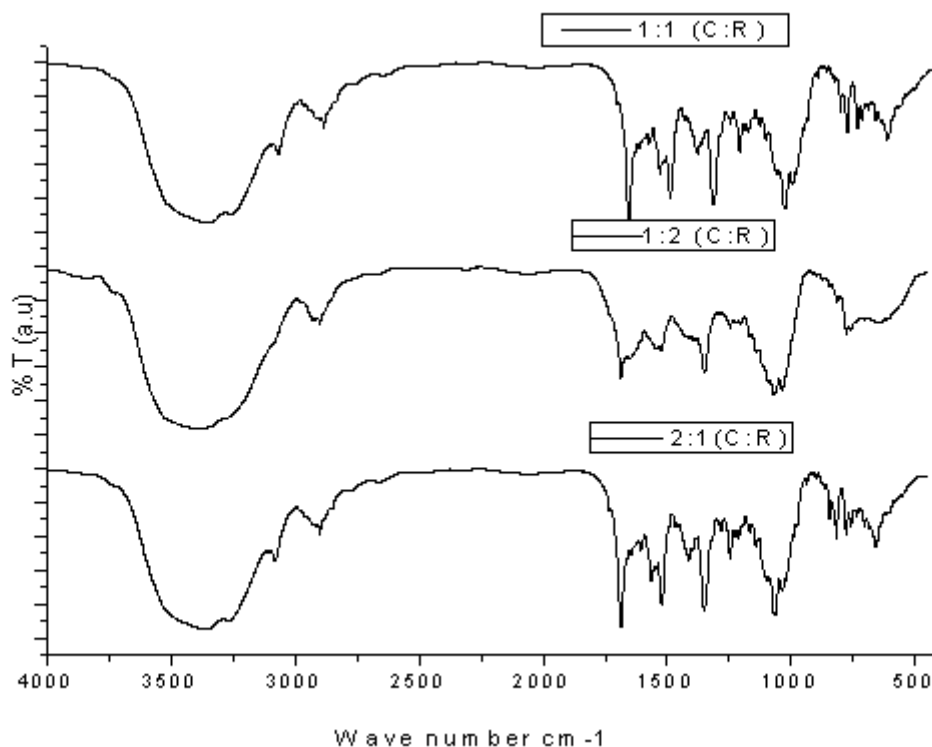


Fig.5. FTIR Spectrum of Commercial drug Chloramphenicol and riboflavin drug treated to different proportion.

The fundamental studies in benzene vibrations show the characteristics skeletal stretching modes. The semi unsaturated carbon-carbon bond leads to the appearance of a group of four bands between $1650\text{--}1450\text{ cm}^{-1}$. The bands observed $\sim 1562\text{ cm}^{-1}$ and $\sim 1550\text{ cm}^{-1}$ are assigned to aromatic ring stretching vibrations.[12] Aromatic ring deformation vibrations occur below 700 cm^{-1} and normally the in plane deformation vibrations is at a higher frequency than the out of plane deformations. Thus a satisfactory vibrational band assignment has been made available for acetaminophen through infrared spectroscopy. The remaining bands observed in the spectra may be due to overtones and combinations of fundamental vibrations. Further shift in frequencies are observed in the interaction of Riboflavin at various combination with Chloramphenicol resulting in influence of riboflavin was more pronounced in case of chloramphenicol and later is less effective. These results are further confirmed by the UV-Visible study. In conclusion the spectroscopic studies FT-IR and UV-Visible can be effectively used for both qualitative and quantitative analysis of commercial drug to the certain limit and further investigation with other spectroscopic technique provides complete characterization of the drug and it plays a vital role in the field of pharmacology.

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