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# Study of Characterization of Commercial Drugs Using FTIR, XRD and TGA Technique

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***ABSTRACT--****Minerals are minute amounts of metallic elements that are vital for the healthy growth of teeth and bones. 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 in understanding the mechanism of drug action. Hence an attempt has been made to study the physico-Chemical characteristic of commercial drugs in solid state using FT-IR, Thermal and X-ray diffraction measurement. Some specific modes of vibrations are identified and the absorbance values are noted. The intensity ratio of Phosphate to Silicate* $I_{1028}/I_{756}$ *were found to 0.66 for drug A, 0.33 for drug B and 0.83 for drug C. The presence of certain minerals such as Phosphate, Silicate, and Sulphate were identified.*

***Key Words--****Characterization, Commercial Minerals drugs, FTIR, XRD, TGA.*

## I. INTRODUCTION

Vitamin, are the organic compounds required by the body in small amounts for metabolism, to protect health, and for proper growth in children. Vitamins also assist in the formation of hormones, blood cells, nervous-system chemicals, and genetic material. The various vitamins are not chemically related, and most differ in their physiological actions. Minerals are minute amounts of metallic elements that are vital for the healthy growth of teeth and bones. They also help in such cellular activity as enzyme action, muscle contraction, nerve reaction, and blood clotting [1]. Mineral nutrients are classified as major elements (calcium, chlorine, magnesium, phosphorus, potassium, sodium, and sulfur) and trace elements (chromium, copper, fluoride, iodine, iron, selenium, and zinc). The 13 well-identified vitamins are classified according to their ability to be absorbed in fat or water. The fat-soluble vitamins A, D, E, and K are generally consumed along with fat-containing foods, and because they can be stored in the body's fat, they do not have to be consumed every day. The water-soluble vitamins and vitamin C cannot be stored and must be consumed frequently, preferably every day. Both vitamins and minerals are needed by the body in very small amounts to trigger the thousands of chemical reactions necessary to maintain good health [2]. Many of these chemical reactions are linked, with one triggering another. If there is a missing or deficient vitamin or mineral or link anywhere in this chain, this process may break down, with potentially devastating health effects. In the U.S., since 1940, the Food and Nutrition Board of the National Research Council has published recommended dietary allowances (RDA) for vitamins, minerals, and other nutrients. Expressed in milligrams or international units (IU) for adults and children of normal health,

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these recommendations are useful guidelines not only for professionals in nutrition but also for the growing number of families and individuals who eat irregular meals and rely on prepared foods, many of which are now required to carry nutritional labeling[3]. 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 1. Synthesis of new compounds with actions that are more specific and fewer adverse reactions, 2. synthesis of competitive antagonist and 3. Understanding the mechanism of drug action. Hence keeping in view , the present study aims to characterizing commercial available drug using FT-IR, XRD and TGA techniques.

## II. MATERIALS AND METHODS

The commercially fast moving Multi-Vitamin/minerals drug of same identical composition and of different company has been purchased from Pharmaceutical Shops and they are taken as Drug A, Drug B and Drug C (Brand name not specified). The selected samples have undergone a thorough analysis through the following instruments namely FT-IR spectroscopy, X-ray diffractometer and Thermo gravimetric Analysis. [4, 5, 6]. Absorbance spectra were recorded using Nicolet Avatar-360 FT-IR spectrometer equipped with a KBr beam splitter and a DTGS detector installed at the, I.I.T, Chennai, Tamil Nadu, India.

## III. RESULTS AND DISCUSSION

### *3.1 Infrared Spectral analysis of commercial mineral drugs*

The spectra recorded for commercial minerals drugs (Drug A, Drug B and Drug C) are presented in Fig 1.

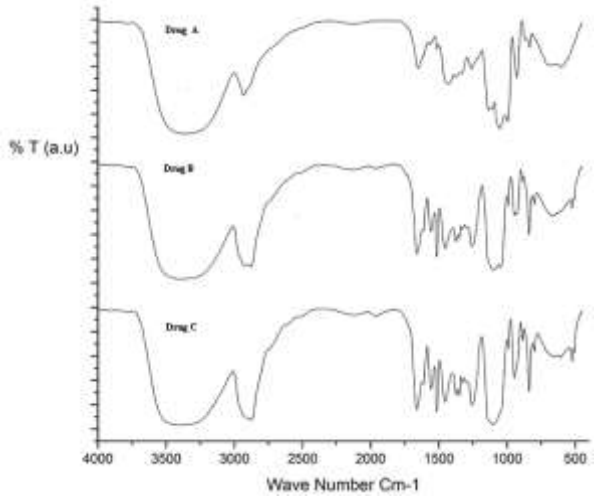


Fig.1. FTIR spectra of Commercial multivitamin/minerals Drugs

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The tentative frequency assignment of this commercial drugs A, B and C are given in Table 1.

By observing the position, shape and relative intensities of the vibrational bands in FTIR spectra, a satisfactory vibrational band assignment has been made [4]. The spectra of the drug c and b are more or less same as that of drug A. The most intense bands of the three drugs are 3377, 1749, 1658, 1407, 1149 and 1037$cm^{-1}$. The assignments of some of the observed frequencies are given below.

The sharp band 2917 $cm^{-1}$ found in the drug B and C is referred to us S-H stretching of methyl free bonded group. The weak band at the region of ~ 1270 $cm^{-1}$ shows the presence of C-O stretching. The band observed ~ 1112 $cm^{-1}$ is due to the presence of sulphate group. The Carbonyl group is determined at the wavelength 1661$cm^{-1}$ 1659$cm^{-1}$ and 1658$cm^{-1}$ of drug A, B and C respectively. The strong peaks observed ~ 1028 $cm^{-1}$ indicates presence of Phosphate group. The band observed

~755 $cm^{-1}$ shows a weak band representing presence of minerals Silicates. The water contribution to the very

| **Wavelength( $Cm^{-1}$)** | | | **FrequencyAssignment** |
|---|---|---|---|
| **Drug A** | **Drug B** | **Drug C** | |
| 3408 (vs.) | 3377(vs.) | 3377(vs.) | OH stretching water molecule |
| 2917(S) | 2917(s) | - | S-H stretching free bonded. |
| 1754(m) | 1749(m) | 1749(m) | CH methyl ester |
| 1661(vs.) | 1659(s) | 1658(Vs) | C=C carbonyl group |
| 1370,1318(s) | 1407(s) | 1407(s) | Nitrate group |
| 1276(m) | 1271(w) | 1271(w) | c-o stretch |
| 1203(s) | 1223(w) | - | Sulphite $(Ro)_2SO$ |
| 1028(vs.) | 1037(s) | 1037(s) | Phosphate |
| 1112(vs.) | 1121(m) | 1133(m) | Sulphate |
| - | 1077(m) | 1077(m) | Silicate S=O (sulphur compound) |
| - | 989 | - | Magnate |
| - | 934 | 934 | P-O-P(phosphate) |
| 870 | 870 | 870 | P-F bond |
| 821 | 821 | 821 | $SiCH_3$ Stretching |
| 756 | 755 | 755 | Silicates |
| 670 | 690 | 690 | Sulphate |

broad band 3377$cm^{-1}$ is partly present in the sample is also due to water vapour uptake during grinding with potassium bromide. The fingerprint region shows more or less weaker bands in the region around 900-500 $cm^{-1}$. The medium intense band observed around 870 $cm^{-1}$ is found in all the three sample spectrum of FT-IR indicating the presence of P-F group. The intense band like 1407 $cm^{-1}$, 1370 $cm^{-1}$ found in sample A, B and C indicates the presence of Nitrate group. In altogether the presence of the minerals like Phosphate, silicate, sulphate etc., are determined from the absorbing bands of the FT-IR spectrum of the drug samples A, B and C. The FT-IR spectrum for the samples A and B indicates some common wavelength bands at 1077$cm^{-1}$(m) 934$cm^{-1}$ (w), 755 $cm^{-1}$ (w), 690 $cm^{-1}$ (w), 1271 $cm^{-1}$ (w), 1749 $cm^{-1}$ (m), 3377 $cm^{-1}$ (Vs.) 870 $cm^{-1}$ (w)and 1470 $cm^{-1}$ (s). It is found that shift in their frequency is absorbed for the sample A when compared to the sample B and C. In all the three spectrums of FT-IR the strong bands are 3377 $cm^{-1}$ (vs.), 1659 $cm^{-1}$ (vs.) indicates the OH stretching water molecule and C=C carbonyl group.

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But the strongest band 2917 $cm^{-1}$ of sample A and B is absent in sample C. This indicates the presence of S-H stretching free bonded in A and B but not in C. Sulphate is concentrated more in sample A because of its strongest peak 1112 $cm^{-1}$ but it is shifted to 1121 $cm^{-1}$ (m) and 1133 $cm^{-1}$ in sample B and C.The peak found in sample at 989 $cm^{-1}$ indicates the presence of Magnate. The Phosphate concentrations in all the three samples are strong which is observed by the wavelength 1028 $cm^{-1}$ (vs.),1037$cm^{-1}$(s), 1037$cm^{-1}$(s). The presence of minerals like phosphate, sulphate, silicates and Magnate are determined from the FT-IR spectrum and similar results were given by Martin et.al. [7]. The very common band 870$cm^{-1}$ found in the sample A, B and C refers to P-F group.

### *3.2 Internal Standard calculations and qualitative Analysis*

The study of the quality assurance undertaken in the pharmaceutical laboratory is the main parameters. The FT-IR spectra have been recorded for the commercial minerals drug. Some specific modes of vibrations are identified and the absorbance values are noted in Table 2.

| S No. | Drug Sample | $I_{1028}/I_{756}$ | $I_{1112}/I_{1028}$ |
|---|---|---|---|
| 1 | Sample A | 0.66 | 0.83 |
| 2 | Sample B | 0.33 | 1.5 |
| 3 | Sample C | 0.83 | 0.3 |

The ratio of absorbance among the various modes is calculated, which represents the internal standards. The sets of internal standards of the drug are compared to check the qualitative/quantitative of the sample.

The intensity ratio of absorbance was calculated using baseline method. The intensity ratio of Phosphate to Silicate $I_{1028}/I_{756}$ were found to 0.66 for drug A, 0.33 for drug B and 0.83 for drug C. It is found form the intensity ratio was highest in the drug C indicating higher content of Phosphate minerals. Next to the drug C higher content of mineral Phosphate present in the drug A and least in the drug B. Further the intensity ratio for the sample sulphate to Phosphate $I_{1112}/I_{1028}$ is higher in the drug C and least in the drug B. As in general the ratio of the intensity of the peaks phosphate/silicate and Sulphate/phosphate minerals shows the presence of qualitative minerals present in the drug sample and this can account to crystallinity of the sample. The intensity ratio of Phosphate ratio to Silicate $I_{1028}/I_{756}$ is increased in the sample B (0.83) than sample A (0.66). This increase in ratio may be attributes to increase in Phosphate mineral for the sample B than A. The intensity ratio of sulphate to Phosphate $I_{1112}/I_{1028}$ increases in the sample A (0.83) than in the sample B(0.3) but for the sample C(1.3) is greater than the sample A(0.83) and sample B(0.3).This are further confirmed using X-ray difractometry.

### *3.3 X-Ray powder diffraction study.*

The experimentally obtained XR diffractogram of the commercial drug sample (A) Fig 2. The relative intensity (%) I/Io are found for those peaks having 2ɵ value listed in the Table 3-5 respectively for the sample A, B and C. Since the commercial drugs do not give large crystals sufficient for a detailed single crystal examination, the structure of these drugs was characterized by their powder XRD patterns. In this, diffraction patterns of commercial drugs a large number of intense peaks arising from the diffraction of X-rays by planes of

minerals have been observed over the range of 3-65$^o$ of diffraction angle. The inter-planer spacing (d) were calculated from the positions of intense peaks using Bragg's relationship, $n\lambda = 2d\sin\theta$, where $\lambda$ is the wavelength of radiation. The calculated spacing together with the relative intensities with respect to the most intense peaks is given in that

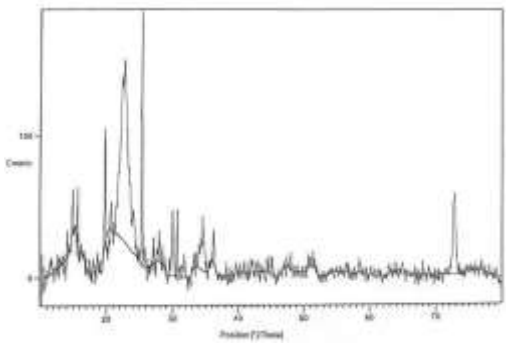

Fig. 2.X Ray diffractogram of Commercial Drug A

Tables 3, similarly average d value for X-ray diffraction analysis of commercial mineral drug for Samples B d =6.6565 and C d=5.0494.

Table 3.X-ray diffraction analysis of commercial mineral drug

| **2θ** | **θ** | **sinθ** | **λ/ 2sinθ** | **D** | **N** | **I/Io** |
|---|---|---|---|---|---|---|
| 13.72 | 6.86 | 0.1194 | 6.2814 | 6.4634 | 1 | 0.07 |
| 15.60 | 7.80 | 0.1375 | 5.5268 | 5.6802 | 1 | 0.38 |
| 16.31 | 8.15 | 0.1417 | 5.2928 | 5.4378 | 1 | 0.68 |
| 18.20 | 9.10 | 0.1582 | 4.7408 | 4.8742 | 1 | 0.07 |
| 19.28 | 9.64 | 0.1675 | 4.4776 | 4.6225 | 1 | 0.06 |
| 19.60 | 9.80 | 0.1702 | 4.4065 | 4.5291 | 1 | 0.86 |
| 23.89 | 11.95 | 0.2071 | 3.6214 | 3.7385 | 1 | 0.14 |

Average d=3.3568

The appearance of diffraction of commercial drugs in the diffraction pattern confirms good crystallinity. The average planar distance i.e., long spacing for commercial drug A, B and C are 3.3568, 6.6565 and 5.0494 respectively. The difference in the observed values of long spacing of commercial drugs A, B and C is ≅ 2.0 A$^o$ which corresponds double the length of additional S=O and P-O-P group of molecules. These results are in good agreement with FT-IR study. The sulphur and Phosphate group of molecule fit into the spaces between Oxygen atoms without a large strain of bonds. Many diffraction peaks in the intermediate range are also observed in the diffraction pattern of these drugs and are attributed to the diffraction of X-rays by planes of atoms of much smaller separation than the basal planes [6]. The calculated spacing from these peaks correspond to the shorter side spacing i.e., the lateral distance between one drug molecule and the next in a layer. It is proposed that the minerals like phosphate, sulphur, calcium etc., are arranged in a parallel planes i.e., a basal plane equally spaced in the drug with fully extended Zig-Zag chain of ions on both sides of each basal planes and the durg molecules have double layer structure as proposed by Upadhyay et.al [6].

From the XRD diffractorgram, it is observed that the degree of crystallinity is more in the drug sample C than that of A and B. The 2θ, d spacing, $I/I_0$ are shown in Table 3. By comparing with the JCPD file the some of the minerals observed in the drug sample are Calcium Phosphate, Magnesium Calcium Phosphate, Calcium Magnesium Phosphate and Calcium Carbonate. The resolution of closely spaced lines can be influenced by both the nature of the sample and by instrumental factors. The width of the X-ray lines (full width at half maximum) is dependent on the sample particle size as well as its crystalline[5]. Thus X ray diffraction permitted us to identify the active ingredients present in the samples A, B and C respectively.

The XRD diffractometer offers numerous advantages. It is not only simple and direct but it also permits unambiguous identified from the drug. The JCPDS file for Magnesium calcium phosphate, calcium carbonate; calcium phosphate is compared with the XRD pattern of the drugs Drug-A, Drug-B and Drug-C. Since the samples taken are multivitamin tablets they have shown common peaks which has been noted as some common minerals found in them. The XRD spectrum is so intense and the required d-spacing values are found for corresponding values of 2ɵ. Then the relative intensity in percentage is found for those peaks having 2ɵ value listed in the Table 3 of the XRD spectrum for the samples A, B and C. These spectra also indicate the presence of Calcium Phosphate mineral deposition more in all the three samples. Only a small trace level of Calcium Carbonate is found in the sample A. The peaks of the sample B are less intense and this proves that the ingredients are less in concentration than in sample A and C. Finally the particular mineral Phosphate is more concentrated in the taken samples A, B and C which has been determined from the FT-IR and XRD spectrum. The other minerals like Magnesium, silicates, sulphates deposition is also traced out from the experiments of FT-IR spectrometer and XRD diffractometer. These results are in good agreement with FT-IR study

### *3.4 Thermal analysis technique*

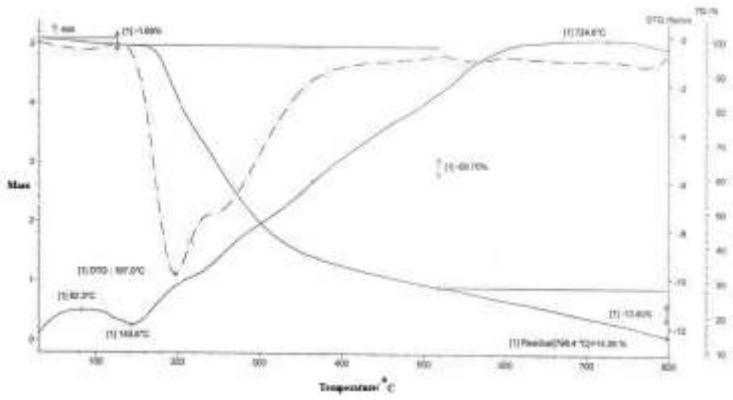

Fig.3. Thermogravimeteric analysis of commercial drug.

The thermal analysis for the drug A is shown in the Figure 3.

The graph shows the decomposition rate in three stage process when the material is subjected to an atmosphere of nitrogen and heated at uniform heating rate. The TGA curve shows the variation of weight loss with Temperature. The curve shows the melting point at 197°C. The decomposition ends at 798 °C and the residues of 14.25% were left at 798.4°C. This was some black carbonaceous residue left in the crucible at the

end of the experiment. This was formed from the liquid state being that the melting point had occurred at 197°C the decomposition rate starts from 82.3 °C to 724.8°C. The curve shows straight and stable to certain extent and it represents no weight loss due to dehydration or formation of volatile products. The weight loss occurs at three stages as shown in the Fig 3. At the points on the curve TGA the mild weight loss (1.89%) occurs at 143.6°C, the second weight loss is more than the first of around (69.70%) at 536°C and the last stage nearly 13.80% of sample were left out at 798.4°C The decomposition stops finally at the temperature 798.4°C. The process of decomposition was going on between the stages 200°C -536°C and this shows no weight loss occurs during that temperature range. This technique is unique and accurate to identify the minerals through their decomposition rate. Thermogravimetric data were used to calculated the energy of activation and to find the order of reaction for the decomposition of commercial drugs using the equation of Freeman and Caroll which is written as $\Delta\log(dw/dt)/\ \Delta(\log W_r) = (-E_a/2.303R)\ (\Delta(1/T)/\ \Delta(\log W_r) + n$

$E_a$= energy of activation,
R= gas constant,
n=order of reaction,

Table 4 Freeman Carroll's Coefficients obtained from the Thermogram of commercial multivitamin/mineral drug rate of weight loss obtained from the loss in weight vs. time curve at appropriate time.

| dw/dt (mg/sec) | Time(Sec) | $W_r$ mg | 1/T $sec^{-1}$ | Log $W_r$ | $\Delta\log(dw/dt)/\ \Delta(\log W_r)$ | $\Delta(1/T)/\ \Delta(\log W_r)$ |
|---|---|---|---|---|---|---|
| 4009.08 | 2.6 | 4.0 | 0.385 | 0.602 | 6.19 | 0.478 |
| 3731.67 | 3.4 | 4.1 | 0.294 | 0.613 | 6.63 | 0.332 |
| 3454.26 | 4.0 | 3.4 | 0.250 | 0.531 | 7.70 | 0.388 |
| 3176.85 | 4.6 | 3.1 | 0.208 | 0.491 | 10.38 | 0.364 |
| 2899.45 | 5.5 | 2.9 | 0.187 | 0.462 | 12.62 | 0.400 |
| 2622.04 | 6.2 | 2.6 | 0.161 | 0.414 | 73.56 | 0.498 |
| 2344.62 | 6.9 | 2.8 | 0.145 | 0.447 | | |
| 2067.22 | 7.5 | 2.1 | 0.133 | 0.322 | | |
| 1789.81 | 8.2 | 1.8 | 0.122 | 0.255 | | |
| 1512.40 | 8.9 | 1.5 | 0.112 | 0.176 | | |
| 1234.99 | 9.6 | 1.1 | 0.104 | 0.041 | | |
| 957.58 | 10.3 | 1 | 0.097 | 0 | | |
| 680.17 | 11.0 | 0.6 | 0.091 | -0.222 | | |
| 402.76 | 11.7 | 0.7 | 0.085 | -0.155 | | |
| 125.35 | 12.4 | 0.2 | 0.081 | -0.699 | | |

The plots of $\Delta\log(dw/dt)/\ \Delta(\log W_r)$ vs $(\Delta(1/T)/\ \Delta(\log W_r)$ have been found to be linear with intercept equal to zero(Fig.4).

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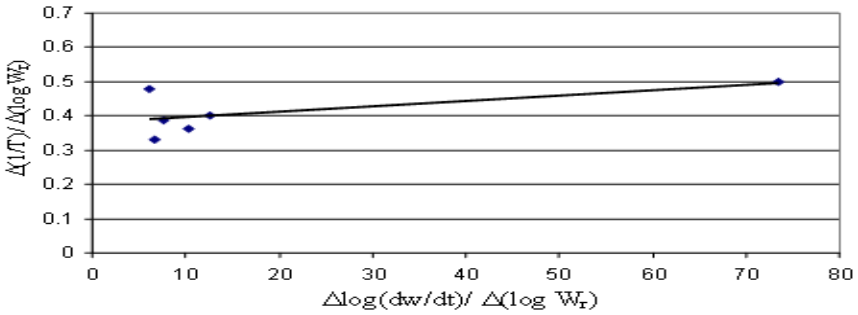


Fig.4. Freeman Carroll's type plots of commercial multivitamin drug

It is therefore concluded that the order of reaction for the decomposition of commercial drug is zero and the value of energy of activation from the slope (-Ea/2.303 R) of the plot is 0.992 Kcal/mol.

Thermo analysis is very useful and time saving for verifying results from other measurement methods and vice versa. It is found from the study that the melting point is 196° c and decomposition occurs in the range 82° c -800° c. Many works were reported for the various drugs using thermal analysis by Ensiolaine [8] and Lori Burnham [9] on Ibopamun and Acetazolamide. It is concluded that in a system of where a material is degrading by a series of reaction mechanisms, the rate of controlling mechanisms varies in different temperature regions. The TGA method provided gives sufficiently precise measurement on thermo analytical approach at the trace level. Thermogravimetric data were used to calculated the energy of activation and to find the order of reaction for the decomposition of commercial drugs using the equation of Freeman and Caroll. The plots of $\Delta\log(dw/dt)/\Delta(\log W_r)$ vs $(\Delta(1/T)/\Delta(\log W_r)$ have been found to be linear with intercept equal to zero. It is therefore concluded that the order of reaction for the decomposition of commercial drug is zero and the value of energy of activation from the slope (-Ea/2.303 R) of the plot is 0.992 Kcal/mol.

## IV. CONCLUSION

In conclusion FT-IR spectroscopy together with X-ray diffraction and thermal analysis enabled us to identify and characterize the different functional group/minerals present in the commercial drugs A, B and C. The results permit us to assert that the three multivitamins drugs show some common minerals in all the three drugs. Therefore in general the spectroscopy study plays a vital role in the field of pharmaceutical and its analysis of drug contents both in the qualitative and quantitative analysis. The following research on the drug samples with the instrumentation of spectroscopic study like FTIR, XRD and TGA provides a successful and powerful tool in both academic and industrial research.

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## REFERENCES

1. Thomas Nogrady, Medicinal Chemistry-A Biochemical Approach, oxford university press-2004. P405
2. Mir Misbahuddin, Rabil Islam M., General Principles of Pharmacology, Books and allied (P) Ltd., Calcutta, 1998.
3. 24- 189.
4. Robert G., White, Handbook of Industrial Infrared Analysis, Plenum Press, New York, 1964. P 2170.
5. Siegfried Waterewig,Reinhard H., Neubert H., (2005) Pharmaceutical applications of Mid-IR and Raman Spectrocopy, Advanced Drug Delivery Reviews, 57, 1144-1170.
6. Ali altharawi, Khondaker Miraz rahman and K. L. andrew chan, (2019) Towards identifying the mode of action of drugs using live-cell FTIR spectroscopy Analyst, 144, 2725-2735.
7. Upadhyay s.K., Shukla R.K., Govind sharma.,( 2007) Infrared, Thermal and X-rays Diffraction Analysis of Dysprosium Soaps", Asian Journal of Chemistry, 19(5), 3487-3492.
8. Martin E. Auer, Ulrich J., Griesser Juergen Sawatzhi, (2003) Journal of Molecular structure , study of polymorphic forms in drug formulatin by near infrared FT-IR spectroscopy, 661-662, 307-317.
9. Ensio Laine, Jukka Pirttimaki, and Ritta Rajala, (1995) Thermal studies on polymorphic structure of ibopamin" thermochemica Acta, 248, 205-216.
10. Lori Burnham, David Dollimore, Kenneth S. Alexander, (2002) Thermogravimetry study of drug Acetazolamide Thermochimica Acta, 392-393, 127-133.

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