IR, NMR Spectral Analysis of Newly Synthesized Lactose Containing Heterocyclic Compounds

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ABSTRACT

The IR spectroscopy theory utilizes the concept that molecules tend to absorb specific frequencies of light that are characteristic of the corresponding structure of the molecules.It is an essential tool for the structural analysis of newly synthesized compound. Infrared spectroscopy is the measurement of the interaction of infrared radiation with matter by absorption, emission, or reflection. It is used to study and identify chemical substances or functional groups. It is the spectroscopy that deals with the infrared region of the electromagnetic spectrum that is light with a longer wavelength and lower frequency than visible light. It covers a range of techniques, mostly based on absorption spectroscopy. Many nuclei have spin, and all nuclei are electrically charged, according to the NMR principle. An energy transfer from the base energy to a higher energy level is achievable when an external magnetic field is supplied. Nuclear Magnetic Resonance (NMR) spectroscopy is an analytical chemistry technique used in quality control and research for determining the content and purity of a sample as well as its molecular structure.

Novel 4-aryl-5-p-tolylimino-3-hepta-O-benzoyl-β-D-lactosylimino-1, 2, 4-dithiazolidines (hydrochlorides) have been synthesized by the interaction of several 1-hepta-O-benzoyl-β-D-lactosyl-3-aryl thiocarbamides with N-p-tolyl-S-chloro isothiocarbamoyl chloride. The newly synthesized compounds have been characterized by analytical and IR, 1H NMR. The polarimetric studies of the title compounds have been carried out.

Keywords: - IR, 1H NMR spectroscopy, 1, 2, 4-dithiazolidines etc.

I. INTRODUCTION

Disaccharides & Polysaccharides are important bio macromolecules with numerous beneficial functions and a wide range of industrial applications. Functions and properties of Disaccharides & Polysaccharides are closely related to their structural features. Infrared (IR) spectroscopy is a well-established technique which has been widely applied in polysaccharide structural analysis. In this paper, the principle of IR and interpretation of polysaccharide IR spectrum are briefly introduced. Classical applications of IR spectroscopy in polysaccharide structural elucidation are reviewed from qualitative and quantitative aspects. Some advanced IR techniques including integrating with mass spectrometry (MS), microscopy and computational chemistry are introduced and their applications are emphasized. These emerging techniques can considerably expand application scope

of IR, thus exert a more important effect on carbohydrate characterization. Overall, this review seeks to provide a comprehensive insight to applications of IR spectroscopy in polysaccharide structural analysis and highlights the importance of advanced IR-integrating techniques.

Fundamentals of near-infrared spectroscopy in the study of carbohydrates, as well as the application of partial least squares regression (PLSR) and principal component analysis (PCA), as the most useful chemo metric techniques involved in carbohydrate analysis. The theoretical aspects and practical applications starting from simple to complex carbohydrates mixtures are covered.

For example, NMR can quantitatively analyze mixtures containing known compounds. For unknown compounds, NMR can either be used to match against spectral libraries or to infer the basic structure directly. Once the basic structure is known, NMR can be used to determine molecular conformation in solution as well as studying physical properties at the molecular level such as conformational exchange, phase changes, solubility, and diffusion. In order to achieve the desired results, a variety of NMR techniques are available.

II. RESULTS AND DISCUSSION

Herein, we report the synthesis of various 4-aryl-5-p-tolylimino-3-hepta-O-benzoyl-β-D-lactosylimino-1, 2, 4-dithiazolidines (hydrochlorides) by interaction of several 1-hepta-O-benzoyl-β-D-lactosyl-3-aryl thiocarbamides with N-p-tolyl-S-chloro isothiocarbamoyl chloride. All products were crystallized from ethanol before recording the physical data. The purity of compounds was checked by TLC. The spectral analyses IR, 1H NMR of the product were observed. Optical rotation of the product was also recorded.

III. EXPERIMENTAL

MATERIAL AND METHODS

The identities of these new N- lactocides have been established by the use of 1HNMr spectral study. ^{1}HNMR measurements were performed on a Bruker DRX (300MHz FT NMR) NMR spectrometer in CDCl$_3$ solution with TMS as internal reference 1HNMR spectra of new N-lactosides do contain some well resolved signals, including those of aromatic protons at δ7.45-6.9ppm, lactosyl protons at δ5.35-3.77 ppm, acetyl proton at δ2.45-1.97 ppm, methyl protons at (Ar-CH$_3$) δ1,263 ppm & other protons that are influenced by specific functionality. Similarly influence of structural features on chemical shift & coupling constant was illustrated by NMR spectra. In this way applications of NMR spectroscopy used for studies of carbohydrate are briefly considered.

Experiment No. 1: Synthesis of 4-aryl-5-*p*-tolylimino-3-hepta-*O*-benzoyl-β-D-lactosylimino-1, 2, 4-dithiazolidine (hydrochlorides) (IIIa)

To chloroform solution of *N*-*p*-tolyl *S*-chloro isothiocarbamoyl chloride (0.002M, 0.298g in 15 mL) was added chloroform solution of 1-hepta-*O*-benzoyl-β-D-lactosyl-3-aryl thiocarbamide (0.002M, 2.408g in 20mL) and the reaction mixture was refluxed over boiling water bath for 4 hr. during which evolution of HCl was noticed. The progress of reaction was monitored by TLC. After refluxing, the solvent was distilled off under reduced pressure and sticky mass obtained as residue was triturated several times with petroleum ether (60-80°C). A pale yellow solid was obtained. It was purified by chloroform-petroleum ether

I

N-p-tolyl-S-chloro isothiocarbamoyl chloride

II

1-Hepta-O-benzoyl-β-D-lactosyl-3-aryl thiocarbamides

III

4-Aryl-5-p-tolylimino-3-hepta-O-benzoyl-β-D-lactosylimino-1, 2, 4-dithiazolidines (hydrochlorides)
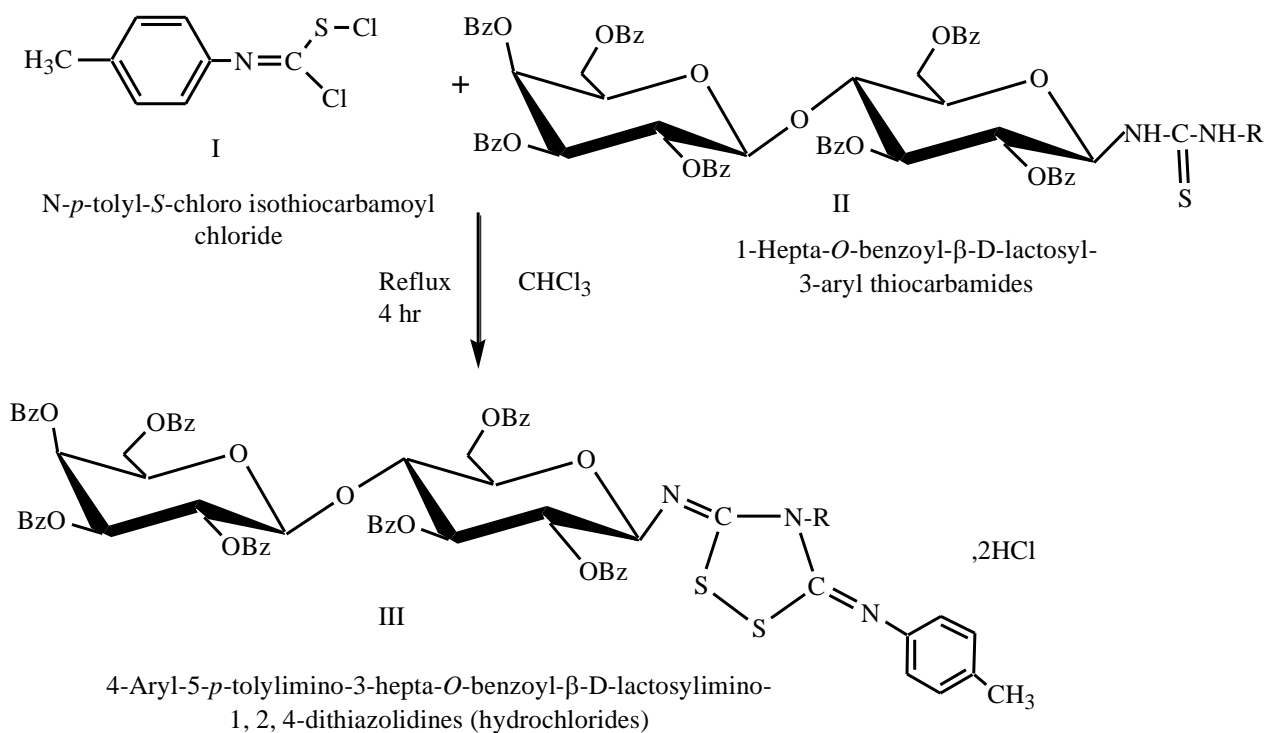
The Infrared spectral[1-16] analysis of the product distinctly showed bands due to Ar. C-H str., Ali. C-H str, C=O str., C=Nstr., C-N str., C-S str., S-S str., and bands due to characteristic of lactose unit.

Table 1.1: IR Spectral analysis of 4-phenyl-5-p-tolylimino-3-hepta-O-benzoyl-β-D-lactosylimino-1, 2, 4-dithiazolidine (hydrochlorides) (IIIa)

Absorption observed (cm^{-1})	Assignment	Absorption expected (cm^{-1})
3066.9	Ar-H stretching	3080-3020[9b, 10b]
2963.2	Ali. C-H stretching	2980-2920[10a]
1730.7	C=O stretching	1730-1715[14, 9c, 10c]
1631.7	C=N stretching	1689-1471[10,13-14]
1270.3	C-N stretching	1400-1280[10f]
1755.6	C-O stretching	1300-1041[10d, 12]
1026, 936.2	characteristic of β- D- lactosyl ring	1100-1000 and 910-900[12,15-16]
709.3	C-S stretching	800-680[8b, 10h]
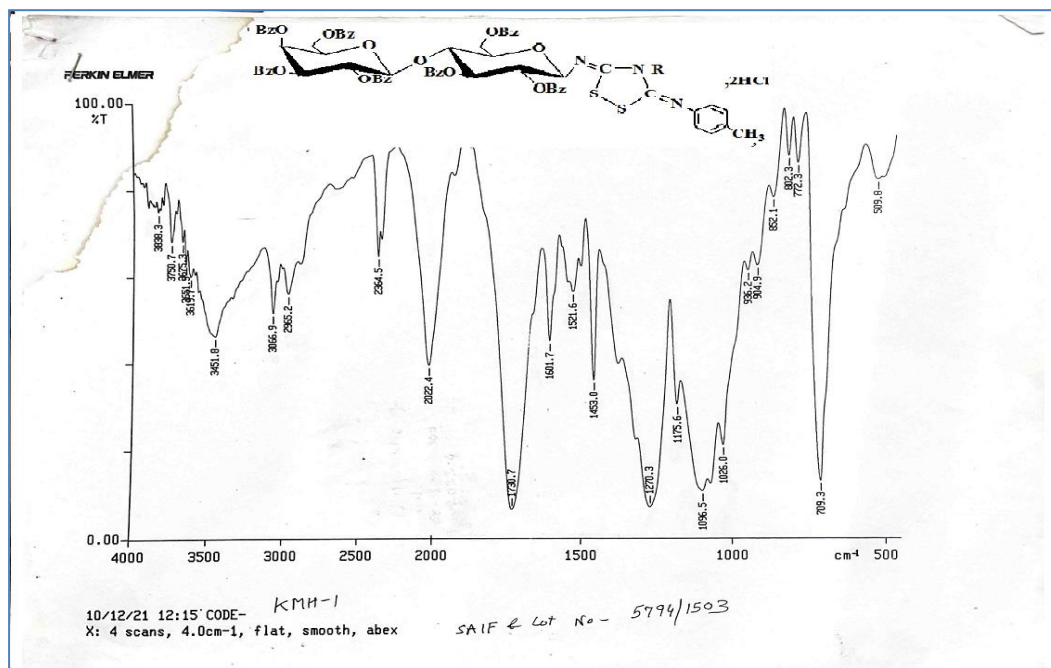
¹H NMR spectral analysis:

The ¹H NMR spectrum[10, 14, 17-24] display the signals due to aromatic protons, lactosyl protons[18-24].

The NMR spectrum of IIIa displayed the signals due to aromatic protons at δ 8.13-7.12 ppm[5, 14d,2-23] (44H, m, 7COC$_6$H$_5$, C$_6$H$_5$, C$_6$H$_4$) lactosyl protons at δ 6.19-3.71 ppm (14H, m)., and methyl protons (Ar-CH$_3$) δ 2.321ppm[15-18] (3H, s, CH$_3$).
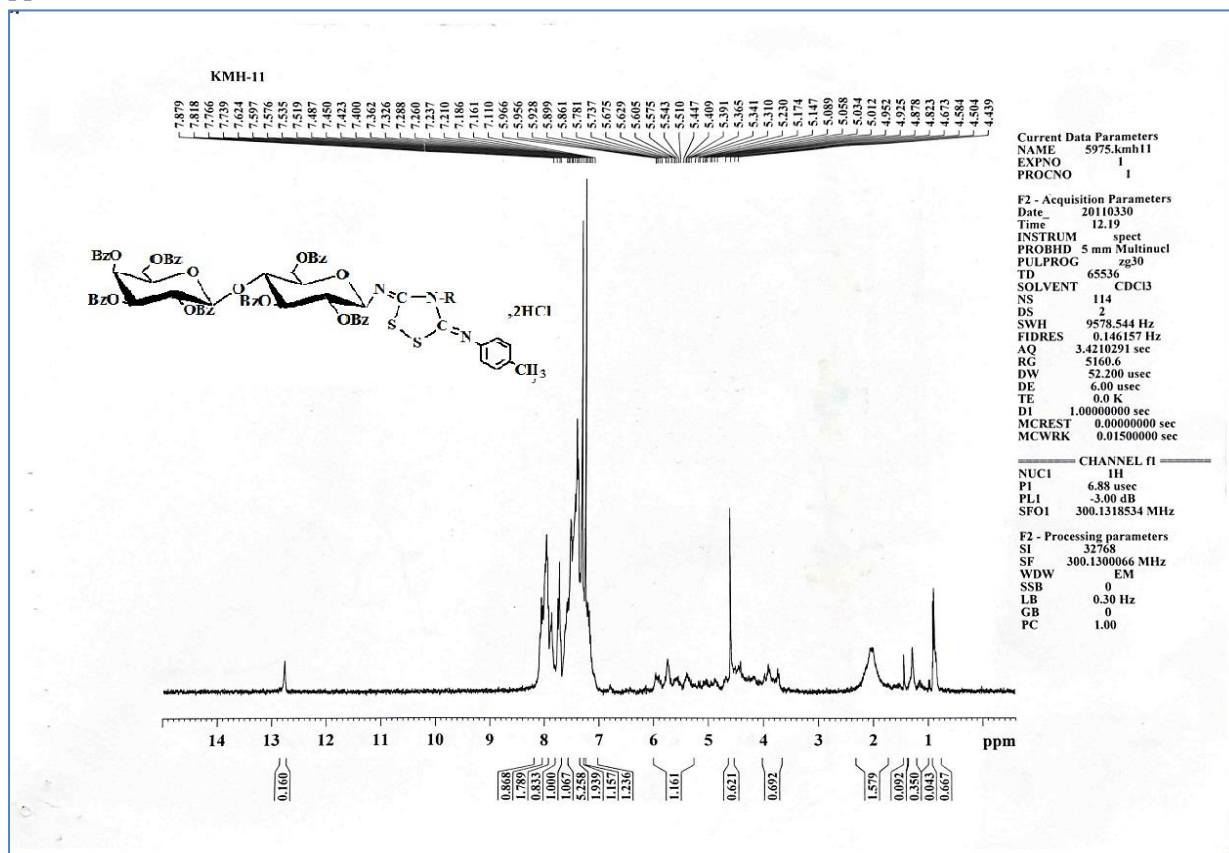
IV. CONCLUSION

In this research work, the characterizations of newly synthesized products were established on the basis of IR, ^{1}H NMR, spectral studies. Various lactose containing Heterocyclic Compounds were synthesized and yield of product ranged from 60-75%.

V. ACKNOWLEDGEMENT

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