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Synthesis, Spectral analysis, Biochemical and Chelating properties of m - amino salicylic acid Derivative

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ABSTRACT

In the present investigation Synthesis, spectral analysis and Biochemical studies on some newly derivative of m – Amino Salicylic acid. A newly ester derivative was synthesized by reflux method. The resulting crude precipitates were recrystallized from the organic solvent. The derivative was Characterized using Elemental Analysis, Infrared spectra, Proton and 13C NMR spectroscopic. This compound has been screened for their biochemical activities.

Keywords: Synthesis, Spectral analysis, biochemical and 5 -ASA derivative.

I. INTRODUCTION

m-amino salicylic acid is also known as 5 –Amino salicylic acid or mesalamine or mesalamine, is an anti-inflammatory drug used to treat inflammation of the digestive treat Ulcerative colitis [1]and mild – to – moderate Crohn's disease [2]. It is also recommended therapy for the induction and maintenance of remission of ulcerative colitis (UC) [3-4]. The drug acts topically at the colonic mucosa to reduce mucosal inflammation [5] yet because the active drug is rapidly absorbed in the stomach and small intestine [6] a number of oral formulations have been developed to deliver m-ASA to the colon [5,7]. The most common side effects of m-ASA are headache and flatulence. Hair loss and itching also may occur. Infrequent side effects include increased hearth rate, Pancreatitis, back pain, fatigue, tremor, and ear pain and blood disorders.

The most important bimolecular, now a day with drastically different properties is required for various applications. Chelates of biologically important molecules are also being investigated for various requirements of human life. Organic molecules with donor atoms like N, O etc. are very good examples that can form coordination compounds. They show important biological and chemical properties. The derivatives of m – Amino salicylic acid is used of medicinal purpose. Practically only few scientists have made attempt to study with m – Amino salicylic acid derivatives or biochemical formation and catalytic behavior of m –Amino salicylic acid derivatives.

Looking to the literature survey carried out as well as the significance of the m – Amino salicylic acid derivatives as well as its coordination compounds, it is quite likely to give modified and improvised

biochemical. Prompted by the above biological properties of m – Amino salicylic acid, it was contemplated to synthesize a novel series of m – Amino salicylic acid derivatives. Antibacterial and antifungal activities of the newly synthesized compounds are discussed in this paper.

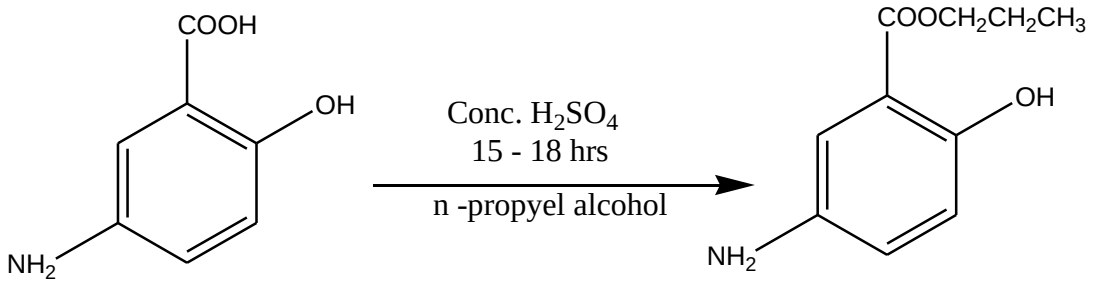
II. EXPERIMENTAL

All chemicals used were of A.R. grade and used as such without further purification except for ethanol. 5 – Amino salicylic acid was obtained from S-d fine chemical company (Properties: White to pink crystals, dec ~ 280°, Slightly soluble in cold water, more soluble in hot water.

Melting points were determined in open capillary tubes and are uncorrected. IR spectra (4000 – 400 cm^{-1}) were recorded on Shimadzu Perkin – Elmer 8201 FT-IR with KBr pellets. The electronic spectra were recorded on Shimadzu- 1800 PTE Ltd. Japan. The ^{1}H-NMR spectra and ^{13}CMR spectra were recorded on BRUKER AVANCE II 400 MHz Spectrometer. Chemical shift values are reported as values in ppm relative to TMS (δ = 0) as internal standard in $CDCl_3$ solvent. Elemental analyses were performed on Vario MICRO C, H, N, S Elemental Analyzer system. Thermogravimetric analysis was carried out under atmospheric condition with heating rate 50 – 1000 @ 10℃ min^{-1} on Mettler Toledo.

DIFFERENTIAL EQUATIONS OF SECOND ORDER

Synthesis of n- propyl 5 -amino-2 hydroxy benzoate



5- amino salicylic acid **n-propyl 5-amino 2-hydroxy benzoate**

A solution of 5-amino salicylic acid (10gm, 65.3 mmol) and concentrated sulfuric acid in n-propyl alcohol was heated under reflux for 15 – 18 hrs. in water bath. After addition of sodium bicarbonate (until the evolution of carbon dioxide). The reaction mixture was filtered. The filtrate was poured into water and extracted with solvent. The combine organic layer was dried over magnesium sulphate and the solvent was removed [8-10].

SPECTRAL ANALYSIS:

5 – Amino Salicylic acid

^{1}H-NMR: δ =8.585 (1H, - COOH), δ =8.077 (1H, -OH), δ = 2.412 (2H, $-NH_2$, Primary amine), δ = 6.431 – 7.531 (6H, Aromatic).

IR Spectra: (KBr) 3100 (N-H), 3160 (O-H), 2850 (C-H),1650 (C=O), 1370 - 1600 (C=C & C-N)

^{13}C-NMR: (Solvent $CDCl_3$) δ = 176.05(-COOH), δ = 112.10 – 138.27 (Aromatic – C), and δ = 170.01 (-C=O)

n-propyl m-amino salicylic acid

^{1}H-NMR: δ = 10.315 (1H, -OH), δ = 3.336-3.31 (2H, -NH_2, Primary amine), δ = 6.820 – 7.279 (6H, Aromatic**),** δ =4.277- 4.309(2H, CH_2), δ = 1.764 -1.851(Multiple 2H, CH_2), δ = 1.024-1.061(3H**,** CH_3).

IR Spectra: (KBr) 3360 (N-H), 3230 (O-H), 2890-2930 (C-H), 1870 (C=O), 1295(C-O), 1690 (C=C & C-N) and 725-720 (C-C-C).

^{13}C-NMR: (Solvent DMSO) δ = 10.49 (-CH_3), δ = 21.97 (– CH_2), δ = 66.81 (-CH_2), δ = 112.45-154.87(Aromatic -C), δ = 170.06(-C=O)

ANALYTICAL DATA AND PHYSICAL PARAMETERS

Name of Compounds: m **-amino salicylic acid** and n**- propyl m- amino salicylic acid**

Molecular formula:		$C_7H_7NO_3$	and	$C_{10}H_{13}NO_3$	
Color:		Light pink	and	reddish black	
Molecular weight:		153 gm	and	195 gm	
Elemental analysis:	found	calculated	and	found	calculated
%C	---	54.90		60.50	61.53
%H	---	4.61		6.17	6.72
%N	---	9.15		6.98	7.17

Melting point: 151 and ----

BIOCHEMICAL PROPERTIES

n-propyl m-amino salicylic acid compound was screened for their antimicrobial and antifungal activity by Agar diffusion method [11]. n-propyl m-amino salicylic acid synthesized compound was evaluated for antimicrobial activity by E. coli, S. aureus, B. subtilis and S. typhi by measuring the zone of inhibition in mm. The activities were performed at a conc. of 50 µg / ml. Streptomycin sulphate (20 µg / ml.) was used as a standard drug for antimicrobial and antifungal activity respectively. Alcohol was used as solvent control for antimicrobial activity.

For the biochemical activity the n-propyl m-amino salicylic acid compound show antibacterial activity and show maximum inhibitory activity against S. aureus. Results of sensitivity against B. subtilis is lower. Again, the inhibitory activity good against E. coli where slightly poor sensitive against Typhi. The assay of bacterial sensitivity was conducted under standard conditions of antibacterial assay technique (Methods in microbiology, A/P, 1978). The results were averaged from the duplicate plates of the concerned set of experiment.

Table – 1. Antimicrobial activity data

Compounds	Diameter of zone of inhibition in (mm)			
	Escherichia Coli	Staphylococcus Aureus	Bacillus Subtilis	Salmonella Typhi
n**-PROPYL m-AMINO SALICYLIC ACID**	20	22	16	18
Streptomycin sulphate (std. drug)	11	11	11	11

III.RESULT AND DISCUSSION

A newly synthesized derivative of m – amino salicylic was reported in this paper. The target compound was synthesized by reflux method in water bath at room temperature. The structure of the newly synthesized derivative has been elucidated on the basis of Elemental, 1H-NMR, ^{13}C-NMR, IR Spectra and biochemical activities. See spectral analysis.

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Fig: 1 (a) ^{1}HNMR Spectrum of m – amino salicylic acid

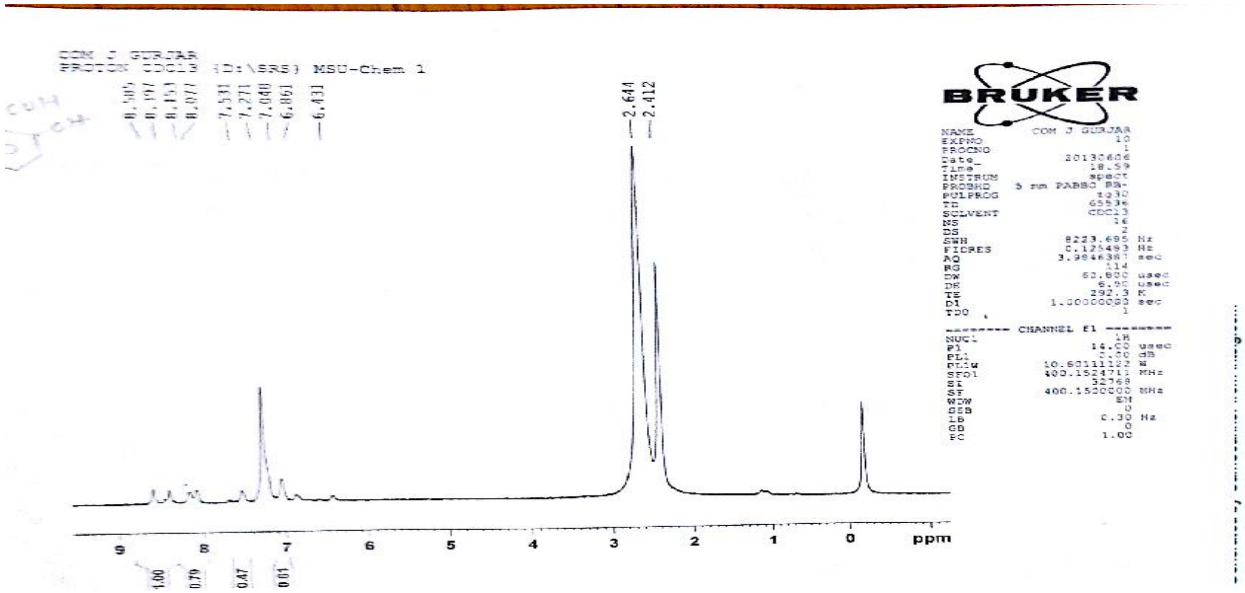


fig: 1(b) ^{1}HNMR spectrum of n-propyl m-amino 2-hydroxy benzoate

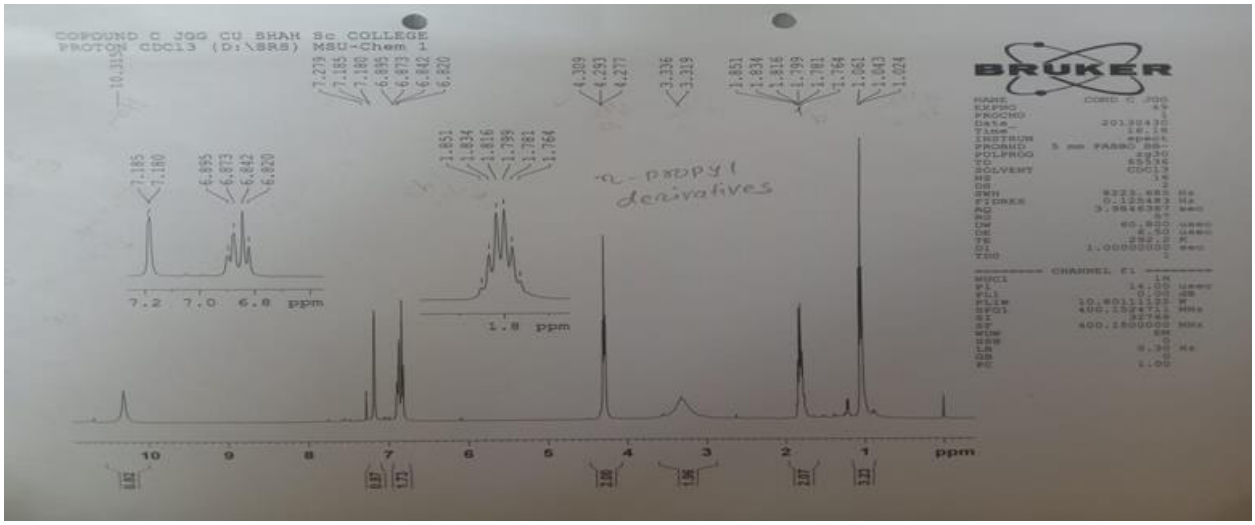


fig: 2 (a) IR Spectra of m – amino salicylic acid

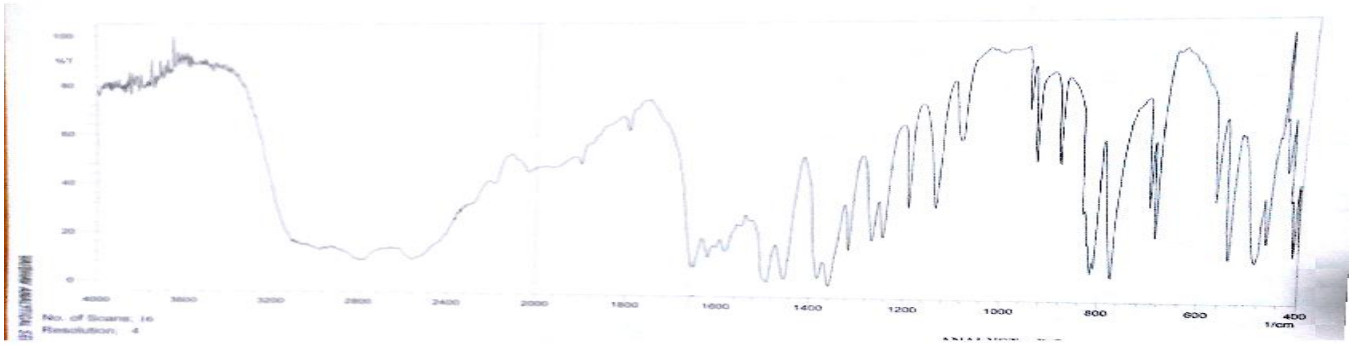
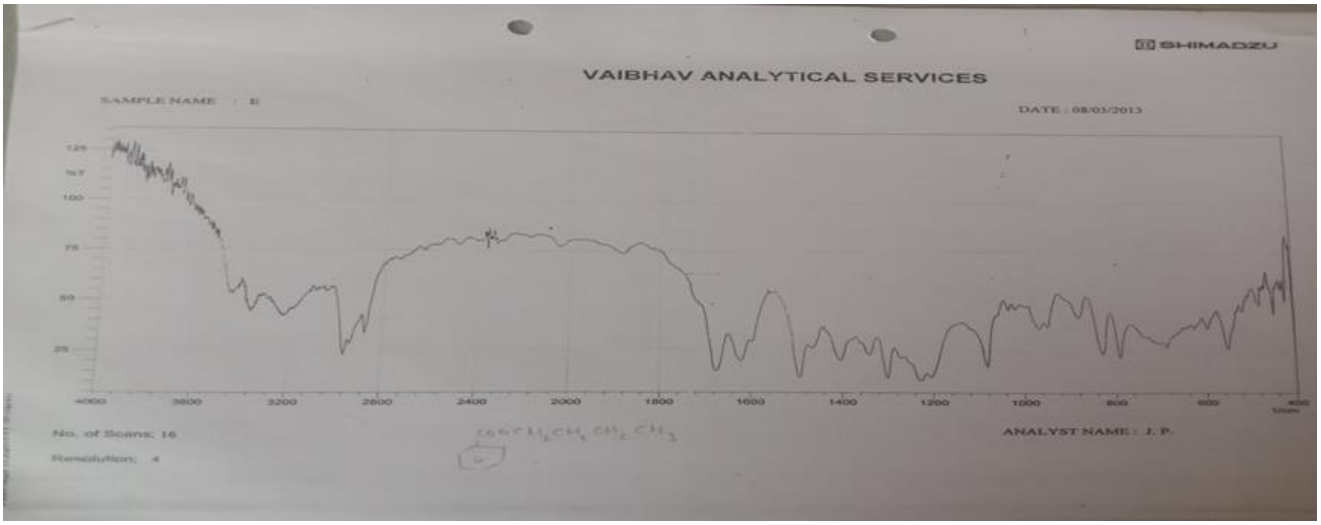


fig:2(b) IR spectra of n-propyl m-amino 2-hydroxy benzoate



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