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SPECTRAL ANALYSIS OF NOVEL 1-(4-METHYL -2, 5-DIMETHOXYPHENYL) ETHANONE-CLUBBED CHALCONE DERIVETIVES

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ABSTRACT

Taking in view of the applicability of heterocyclic compounds, we have undertaken the preparation of heterocycles bearing chalcone nucleus. The placement of a wide variety of substituent of these nuclei of novel 1-(4-methyl -2, 5-dimethoxyphenyl) ethanone clubbed -chalcone derivetives have been designed in order to evaluate the synthesized products by their spectral profile like IR, ^{1}H-NMR, CMR and LCMS.

KEYWORDS: Clubbed –Chalcone, Spectral profile, ^{1}H-NMR, CMR, LCMS.

INTRODUCTION

The chemistry of chalcones has generated intensive scientific studies throughout the world; especially interest has been focused on the synthesis and biodynamic activities of chalcones. In chalcones, two aromatic rings are linked by an aliphatic three carbon chain. Chalcones are also key precursors in the synthesis of many biologically important heterocycles such as benzothiazepine, pyrazolines, 1, 4- diketones, pyrimidines, isooxazolines, cyclohexanone and flavones. Thus the synthesis of chalcone derivative and structural elucidation by different chemical and physical method like elemental analysis and spectroscopic method like IR, NMR and MASS spectroscopy has generated vast interest to organic as well as for medicinal chemists. Chalcones, belonging to flavonoid family, synthesized or the natural one, displayed many interesting properties including antimalarial,[1,2] anticancer,[3,4] antiviral,[5]

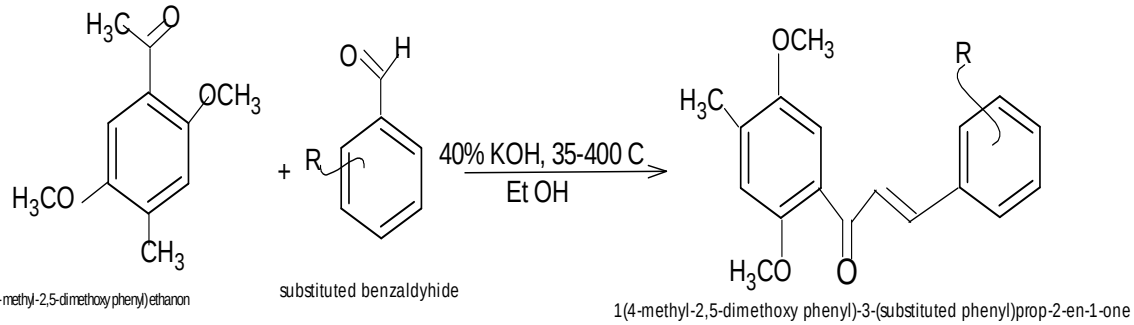
antibacterial,[6,7] antifungal,[8], antihyperglycemic,[9] and tyrosine kinase inhibitors,[10] activities In the chemical structure, three carbon α – β unsaturated carbonyl system, the back bone of the open chain flavonoids, joins two aromatic rings. As part of our ongoing research for newly synthesized molecules, we have synthesized and investigated the molecular structure of the novel substituted chalcone derivative by Spectral techniques like IR, ^{1}H-NMR, CMR, LCMS spectra, which give evident for elucidated final chalcone derivatives structure.

MATERIALS AND METHODS

The ^{1}H NMR Spectra were recorded on a Bruker 300 MHz using TMS as an internal standard. The IR spectra were recorded on a Bruker Spectrum 100 FTIR Spectrophotometer. CMR spectra are run on a Varian XL 100 instrument and the Mass Spectra on a Waters Micromass Q-fit instrument. The chemical used are compound of A.R. grade.

General preparation of novel 1-(4-methyl -2, 5-dimethoxyphenyl) ethanone clubbed -chalcone derivetives

To a mixture of 1-(4-methyl-2, 5-dimethoxyphenyl)ethanone (0.01 mole) and Substituted benzaldehyde (0.01 mole) in ethanol (30 ml) was added a solution of potassium hydroxide (40 ml, 40%) with constant shaking of the reaction flask. The reaction mixture was stirred for a 24 hours on a magnetic stirrer and poured in to crushed ice and acidified with diluted HCl (2N). The solid mass which separated out was filtered, washed with water, dried and crystallized from methanol to give light yellow needles. Completion of reaction were checked on aluminium coated TLC plates 60 F245 (E. Merck) using n-hexane: ethyl acetate (7.5:2.5, v/v).



Where, R = (a) -C_6H_5Cl, (b) -$C_6H_5Cl_2$, (c) -C_6H_5 (-OCH_3) (-OH) (-Br)

RESULT AND DISCUSSION

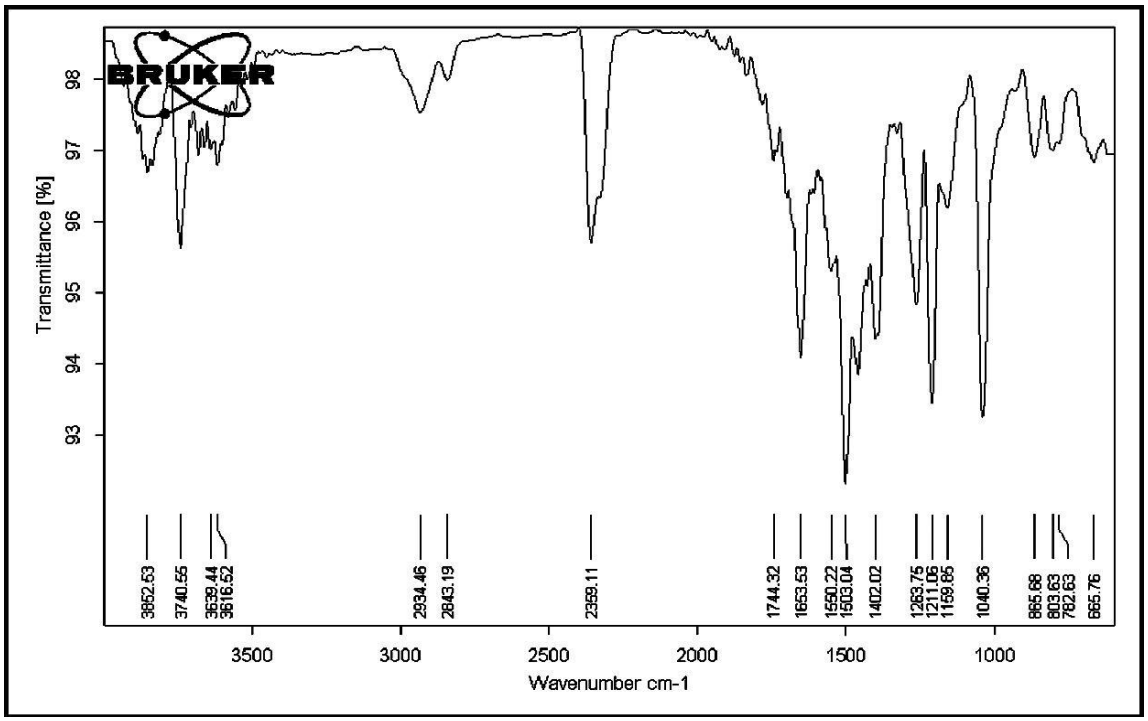
Different clubbed-chalcone derivatives a, b, c were prepared and all the compounds were characterized by their elemental analyses and mostly spectral IR, ^{1}H-NMR, CMR and Mass Spectra.

IR Spectra

Spectroscopic analysis 1-(4-methyl-2, 5-dimethoxyphenyl)-3-(2-chlorophenyl) prop-2-en-1-one. (compound-a)

The IR spectra of of 1-(4, 5-dimethoxy-2-methylphenyl) -3-(2-chlorophenyl) – prop – 2 – en – 1 - one (a) showed a carbonyl abso rption at 1653 cm^{-1} which is characteristic band of the α, β- unsaturated carbonyl group. The absorption band due to C-O stretching appeared at 1263 cm-^{1}. Due to the ether linkage two stretching bands are observed at 1275-1200 cm-^{1} (symmetric) and 1089-1020 cm-^{1} (asymmetric). Ethylinic double bond stretching of chalcone showed at 1661 cm-^{1}. C-Cl stretching displayed at 665 cm-^{1} A medium to strong absorption band seen at 865 cm-^{1} is due to trans CH=CH out of plane deformation (wagging) and Trans CH=CH (vinyl) stretching shown in the range of 3090-3000 cm-^{1}. The aromatic in plane bending was observed at 1159 cm^{-1} and out of plane bending was observed at 833 cm^{-1}**.**

In addition to above mentioned peaks, IR spectrum consists other stretching and bending vibration common to compound under study.



IR (cm^{-1}): 2943 (C-H str. (asym) alkyl), 2834 (C-H str. (sym) alkyl), 1402(C-H def (asym)

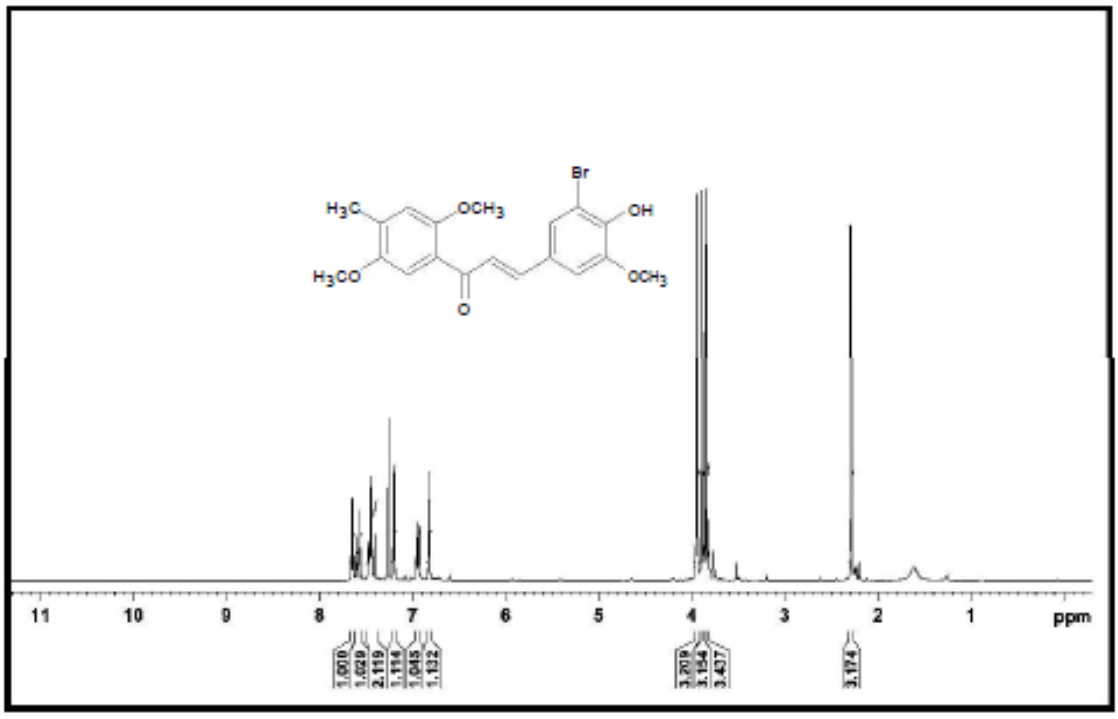
alkyl), 1356 (C-H def (sym) alkyl), 1503 (C=C str. arom.), 1159 (C-H i.p.def arom.), 803 (C-H o.o.p.def.arom.), 1263 (C-O-C (sym) ether), 1040 (C-O-C (asym) ether), 1653 (C=O str., chalcone), 865 (CH=CH def.chalcone), 3081 (CH=CH str. chalcone), 1661 (C=C str. chalcone), 665(C-Clstr.).

^{1}H NMR Spectra

^{1}H NMR spectrum of 1-(2, 5-dimethoxy-4-methylphenyl)- 3-(2-bromo-4-hydroxy-5-imethoxyphenyl)-prop-2-en-1-one (compound-c)

The ^{1}H NMR spectrum of 1-(2, 5-dimethoxy-4-methylphenyl)- 3-(2-bromo-4-hydroxy-5-imethoxyphenyl)-prop-2-en-1-one (1f) showed a pair of doublets at 7.62ppm (J=16.2Hz). and 7.45 ppm (which is merged with aromatic proton) consistent with *trans* olefinic-protons attached to aromatic ring (-CH=CH-Ar) and attached to carbonyl carbon (-COCH=CH) of a chalcone moiety. The signal appeared at 2.28 ppm suggest presence of methyl group. Nine protons of methoxy group displayed in the range of 3.83-3.93 ppm. confirmed the presence of methoxygroup. OH group showed as singlet 6.81 ppm.

In the aromatic region, Singlet of one proton showed at 6.81ppm.Multiplate of the two protons showed in the range of 7.19-7.54ppm, Doublet of proton displayed at 6.94ppm (J=1.8 Hz) and meta coupled proton showed as doublet at 7.58 ppm(J=1.6Hz).respectively.



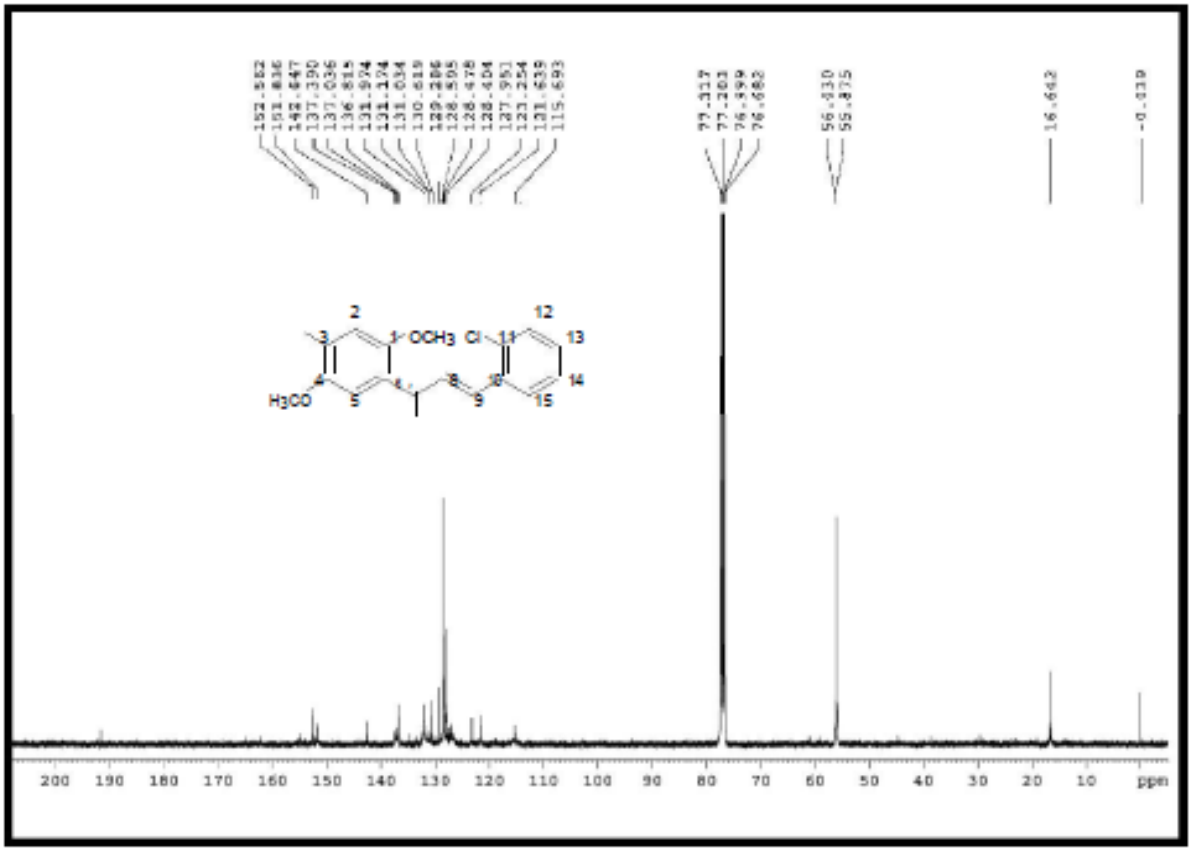
^{1}H NMR ($CDCl_3$) ppm: 2.28 (s, 3H), 3.83 (s, 3H, OCH_3), 3.87 (s, 3H, OCH_3), 3.93 (s, 3H,

OCH_3), 6.81(s, 1H), 6.94(d, 1H, J=1.8Hz), 7.19(S, 1H), 7.45(m, 1H+1H chalcone), 7.58(d, 1H, J=1.6Hz), 7.62(d, 1H, J=15.8Hz, chalcone).

CMR Spectra

^{13}C NMR Spectra of 1-(2, 5-dimethoxy-4-methylphenyl)- 3-(2-chlorophenyl)-prop-2-en-1-one (compound-a)

In the ^{13}C NMR Spectra of 1-(2, 5-dimethoxy-4-methylphenyl)- 3-(2-chlorophenyl)-prop-2-en-1-one showed two carbons of keto-ethylenic group -CO-CH=CH-were resonated at 123.25 ppm (C-8), 142.64 ppm (C-9) and carbonyl carbon showed signal at 191.60 ppm (C-7).

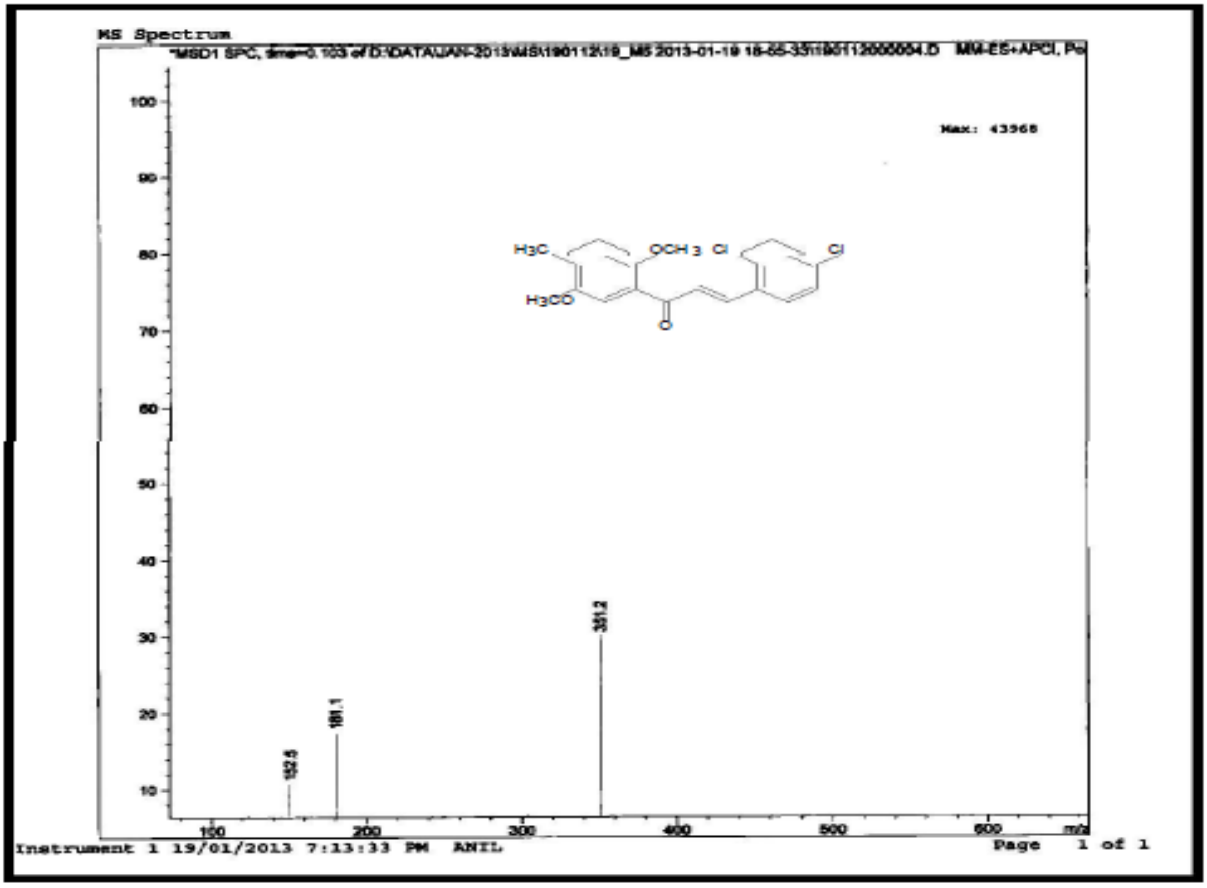


^{13}C NMR ($CDCl_3$) ppm: 151.83(C-1), 130.61(C-2), 131.97(C-3), 152.56(C-4), 115.69(C-5), 121.63(C-6), 191.60(C-7), 123.25(C-8), 142.64(C-9), 129.28(C-10), 136.81(C-11), 128.47(C-12), 128.59(C-13), 127.95(C-14), 137.03(C-15), 56.03(C-16), 55.87(C-17), 16.64(C-18).

LC Mass Spectra

LC MASS Spectra of 1-(2, 5-dimethoxy-4-methylphenyl)- 3-(2, 4di chlorophenyl)-prop-2-en-1-one (compound-b)

The mass spectra of 1-(2, 5-dimethoxy-4-methylphenyl)- 3-(2, 4di chlorophenyl)-prop-2-en-1-one showed strong molecular ion peak at 351*m/e*.



CONCLUSION

Newly synthesized molecules, investigated the molecular structure of the novel substituted chalcone-clubbed derivative by Spectral techniques like IR, ^{1}H-NMR, CMR, LCMS spectra, which give evident for elucidated final chalcone derivatives structure.

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