Synthesis IR AND NMR STUDIES OF 2-(4-methoxybenzyl)-6-phenyl-5-thiocyanatoimidazo[2,1-b][1,3,4]thiadiazole

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Procedure:

Step: 01: Synthesis of 2-amino-5-(4-methoxybenzyl)-1, 3, 4-thiadiazole:

0.1mol of 4-methoxy phenyl acetic acid and thiosemicarbazide were refluxed gently in 30 ml of phosphorous oxychloride for 30 minutes. The resulting solution was cooled and added to 90 ml of ice cold water slowly. The separated solid was carefully filtered and suspended in water and was neutralized with potassium hydroxide. The solid mass obtained after neutralization was filtered and recrystallized from DMF-Ethanol mixture,with yield of 62 %.The melting point of the compound was found to be ----------and uncorrected.

Step: 02: 2-(4-methoxybenzyl)-6-phenyl-imidazo[2,1-b][1,3,4]thiadiazole:

2-amino-5-(4-methoxybenzyl)-1,3,4-thiadiazole (30 mmol) was treated with the commercially availablephenacyl bromide (30 mmol), in ethanol (150 mL). The mixture was refluxed for 10-12 hrs. Excess of solvent was removed under reduced pressure and the solid hydrobromide was separated by filtration, washed with cold ethanol and dried. Neutralization of hydrobromide salts with cold aqueous solution of sodium carbonate yielded the corresponding free base which was filtered with yield of 62 %. Melting point of the compound was found to be---------- and uncorrected.

Step:03:2-(4-methoxybenzyl)-benzyl-5-thiocyanato-6-(4-chlorophenyl)imidazo[2,1b][1,3,4]thiadiazole:(359)

To a mixture 2-(4-methoxybenzyl)-6-phenyl-imidazo[2,1-b][1,3,4]thiadiazole(10 mmol) and potassium thiocyanate (1.56 g, 16 mmol) in glacial acetic acid (50 mL), bromine (1.6 g, 10 mmol)) in glacial acetic acid (20 mL) was added at 0-5°C drop wise with stirring. Stirring was continued for 30 min at 15-20°C and then at room temperature for 1 h. The reaction mixture was poured into ice water, filtered, washed with water and recrystallized from EtOH-$CHCl_3$ with a yield of 50-55 %. The melting point of the pure compound was found to be 114-16(C.

IR SPECTRAL DETAILS OF THE 359: The synthesized compound (359) shows transmittance peak for the following groups:-

($_{max}$ cm^{-1} 3058.55-3028.66: Ar,-CH

2966.95-2843.52: Aliphatic -CH

2156.99: -SCN

1608.34: >C=C<

1469.49: >C=N-

1HNMR Data: 7.49-7.93 (m,2H,ar), 7.52-7.47 (m,2H,ar), 7.45-7.41> (m,1H,ar), 7.29-7.24 (m,2H,ar), 6.94-6.92 (m,2H,ar), 4.34 (s,2H,ar),> 3.84 (s,3H,ar)>>>> 13C NMR Data: 167.35(s), 159.59(s), 152.39(s), 142.29(s), 132.04(s),> 130.26(s), 130.19(s), 129.22(s), 128.78(s), 128.48(s), 128.02(s),> 126.85(s), 126.72(s), 114.76(s), 114.65(s), 108.50(s), 96.50(s),> 55.37(s), 37.52(s)