Asian Journal of Chemistry Vol. 9, No. 4 (1997), 879–880

NOTE

Synthesis of Some Novel [2-(substituted phenoxyacetylamino)]-5(β–naphthoxymethylene) -1-3,4-thiadiazoles as potential Fungicides

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A series of [2-(substituted phenoxyacetylamino)]-5-(β-naphthoxymethylene)-1,3,4-thiadiazoles have been synthesized. All such compounds have shown appreciable antifungal activity against two fungi *viz. A. niger* and *H. oryzae.*

Thiadiazoles display a wide spectrum of antimicrobial activities[1–3]. In this connection a number of 4-chloroaryloxy-1,3,4-thiadiazolyl amides having excellent fungicidal properties have already been prepared [4]. The present investigation was undertaken for synthesis of title compounds and their antifungal activity against two fungi *viz.*, *A. niger* and *H. oryzae.*

In general the synthesis of compounds involved in the preparation of 2-amino-5-(β-naphthoxymethylene)-1,3,4-thiadiazole (1) and its condensation with substituted aryloxyacetyl chlorides at –20°C.

All melting points recorded were uncorrected. Infrared spectra of compounds have been scanned on Perkin-Elmer-710 spectrophotometer by KBr disc method. All the compounds were purified by crystallization and thin-layer chromatography.

Refluxing of mixture of β-naphthoxyacetic acid (0.2 mole), thiosemicarbazide (0.075 mole) and conc. H_2SO_4 (5.0 mL) for 6 h afforded 2-amino-5-(β-naphthoxymethylene)-1,3,4-thiadiazoles (1). The product was crystallized from ethanol.

m.p. 131°C, Yield 68%, $C_{13}H_{11}N_3SO$ requires (found): C, 60.70 (58.92); H, 4.28 (4.18); N, 17.39% (17.28) and S, (12.,30) %. IR (KBr): 3400 cm^{-1} $\nu(NH_2)$, 1720 cm^{-1} $\nu((C{=}O)$, 1680 cm^{-1} $\nu(C{=}N)$ and 1475–1460 cm^{-1} $\nu(C{-}CH_2)$).

[2-(Substituted phenoxyacetylamino)]-5-β-naphthoxymethylene)-1,3,4-thiadiazoles 1(a–d) have been synthesized by stirring the equimolar mixture of substituted phenoxyacetyl chloride and (1) in required amount of pyridine for 2–4 h at –20°C. The product was precipitated by cold water and crystallized from ethanol (Fig. 1).

1(a) : R = *p*-Cl-
1(b) : R = *o*-CH_3-
1(c) : R = *p*-CH_3-
1(d) : R = *m*-CH_3-

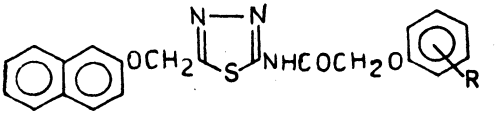

Fig. 1

1(a): m.p. 167°C; yield 50%. $C_{21}H_{16}N_3SClO_3$ requires (found): N, 9.87 (9.83)%; IR (KBr): 1720 cm^{-1} ν(C=O), 1660 cm^{-1} ν(C=N) and 1475–1450 cm^{-1} ν(C—CH_2). 1(b); m.p. 184°C; yield 63%. $C_{22}H_{19}N_3SO_3$ requires (found): N, 10.37 (10.29)%; IR (KBr): 1700 cm^{-1} ν(C=O), 1660 cm^{-1} ν(C=N) and 1475–1460 cm^{-1} ν(C—CH_2). 1(c): m.p. 159°C; yield 56%, $C_{22}H_{19}N_3SO_3$ N, 10.41%. IR (KBr): 1720 cm^{-1} ν(C=O), 1660 cm^{-1} ν(C=N) and 1475–1450 cm^{-1} ν(C—CH_2). 1(d): m.p. 172°C; yield 65%. $C_{22}H_{19}N_3SO_3$ (found): N, 10.34%. IR (KBr): 1730 cm^{-1} ν(C=O), 1650 cm^{-1} ν(C=N) and 1475–1460 cm^{-1} ν(C—CH_2).

All the compounds synthesized 1(a–d) have been screened for their antifungal activity against two fungi, *viz.*, *A . niger* and *H. oryzae*, by agar bath technique[5, 6]. The fungus was planted in agar growth media mixed with test compounds. The activity of test compound was compared with a commercial fungicide (carbendiazim). The diameter of fungus colony was recorded at 10^3, $10^2$and 10 ppm. The percentage inhibition was evaluated as:

$$\text{Percentage inhibition} = \frac{C-T}{T} \times 10^2$$

where C is the diameter of fungus colony in mm in control plate after 96 h and T is for treated plate at the same hour.

All the compounds have shown moderate to fairly good level of fungicidal activity, but their activity was appreciably reduced on dilution. They were slightly more fungicidal against *H. oryzae* than *A. niger*. 4-Chloro substituted derivatives have maximum fungicidal activity while a change in the position of methyl group did not show any marked effect towards fungicidal activity.

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(*Received:* 29 March 1997; *Accepted:* 12 June 1997) AJC-1317