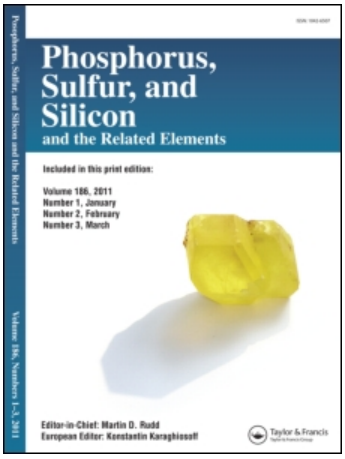

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A CONVENIENT AND GENERAL SYNTHESIS OF A NOVEL HETEROCYCLIC SYSTEM; 5H-[1,3,4]THIADIAZOLO[2,3-d] [1,2,4]TRIAZIN-5-ONES

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5H-[1,3,4]Thiadiazolo[2,3-d][1,2,4]triazin-5-ones were synthesized by cyclocondensation of 4-amino-5-mercapto-1,2,4-triazin-3-ones with a variety of carboxylic acids in the presence of phosphorus oxychloride.

Keywords: Thiadiazolo-triazin-5-ones; cyclocondensation

INTRODUCTION

In continuation of our interest in the synthesis of heterocycles containing sulfur and nitrogen atoms[1], we wish to communicate our results of the synthesis of a novel heterocyclic system 3H-[1,2,4]]thiadiazolo[2,3-d][1,2,4]triazin-3-one.

The synthetic route to 4H-[1,3,4]thiadiazolo[2,3-c][1,2,4]triazin-4-ones **2** has been achieved through ring closure of 4-amino-3-thio-1,2,4-triazin-5-ones **1** with a number of carboxylic acids in the presence of sulfuric acid[2]. Thus treatment of the isomeric 4-amino-5-thio-1,2,4-triazin-3-ones[3]

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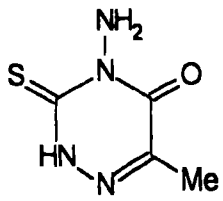
3 with an excess of carboxylic acid in concentrated sulfuric acid should afford the derivative of 3H-[1,3,4]thiadiazolo[2,3-d][1,2,4]triazin-3-ones **4**. Unexpectedly this attempt was unsuccessful. However when **3** was refluxed with benzoic acid in the presence of phosphorus oxychloride cyclocondensation occurred to afford (**4**, R=Ph). The structure of 2-phenyl-8-methyl-5H-[1,3,4]thiadiazolo-[2,3-d][1,2,4]triazin-5-one (**4**, R=Ph) was confirmed on the basis of spectroscopic data. The IR spectrum of this compound showed an absorption at 1700 cm^{-1} indicating the existence of a carbonyl group. The ^{1}HNMR spectrum of the compound exhibited signals at 2.65 belonging to a methyl group and at 7.6–8.1 due to aromatic protons. Accurate mass measurment showed exact m/z at 243 for the cyclized product.



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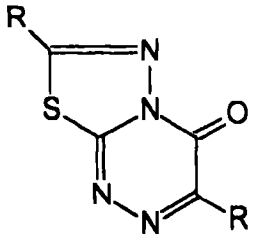

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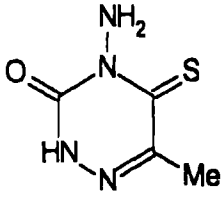

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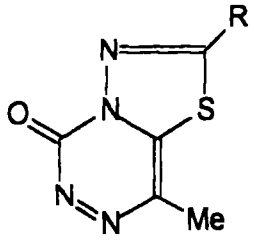

4

R= **a**) -Ph

b) -C_6H_4-*p*-CH_3

c) -$CH_2C_6H_5$

In conclusion, the use of phosphorus oxychloride as a dehydrating agent for cyclocondensation to obtain [1,3,4]thiadiazolo[2,3-d][1,2,4]]triazine seems to be convenient and general.

EXPERIMENTAL SECTION

Mps were determined on a Reichert apparatus and are uncorrected. IR spectra were recorded on a Shimatzu spectrometer as KBr disc. ^{1}HNMR spectra were recorded on a Bruker (100 MHz) instrument. Mass spectra were obtained from Varian CH-7 at 70 eV.

Synthesis of 5H-[1,3,4]Thiadiazolo[2,3-d][1,2,4]triazin-5-ones

General Procedure

Compound **3** (1 mmol) and an appropriate carboxylic acid (2 mmol) in phosphorus oxychloride (6 mL) was refluxed for 1hr. The excess of phosphorus oxychloride was distilled off and the residue was taken up in water (5 mL), neutralized by addition of ammonium hydroxide solution and extracted with chloroform. The crude product which was obtained by removal of chloroform was crystallized from an appropriate solvent.

Selected data for 4a

Yield: 53%, mp: 278–9°C (from acetic anhydride), ^{1}HNMR δ ($CDCl_3$) 2.62 (s, 3H, Me), 7.5 (m, 3H, aromatic protons), 8.0 (m, 2H, aromatic protons), IR, $\tilde{\nu}$ (KBr disc): 2990, 1700, 1600, 1580, 1480, 1100, 800, 750 cm^{-1}, Ms, m/z, M^+ 244(4.1), 242(26.4), 240(17.1), 235(24.2), 185(27), 166(100), 157(55), 125(38).

Selected data for 4b

Yield: 51%, mp: 325–6°C (from acetic anhydride), ^{1}HNMR δ ($CDCl_3$) 2.4 (s, 3H, Me), 2.6(s, 3H, Me), 7.3 (d, 2H, aromatic protons), 7.9 (d, 2H, aromatic protons), IR, $\tilde{\nu}$ (KBr disc): 2990, 1690, 1600, 1510, 1500, 1190, 1100, 810 cm^{-1}, Ms, m/z, M^+ 258(20), 256(27), 223(46), 199(76), 195(46), 165(42), 163(42), 133(53), 117(26)98(42), 57(100), 44(84).

Selected data for 4c

Yield: 55%, mp: 260–62°C (from ethanol), ^{1}HNMR δ ($CDCl_3$) 2.46 (s, 3H, Me), 4.45 (s, 2H, CH_2), 7.3 (m, 5H, aromatic protons), IR, $\tilde{\nu}$ (KBr disc): 3100, 1700, 1600, 1510, 1450, 1150, 1050, 700 cm^{-1}, Ms, m/z, M^+ 258(6), 255(16), 254(45), 229(21), 228(50), 227(82), 196(68), 146(26), 145(100), 131(54), 111(86), 91(78), 77(82).

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