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SYNTHESIS OF SOME NOVEL HYDRAZONES AND THEIR THIAZOLIDINE-4-ONES

ANJANI SOLANKEE*, PANKIT SOLANKEE and HIRAL PATEL

Department of Chemistry, B. K. M. Science College, VALSAD – 396001 (Guj.) INDIA

ABSTRACT

6-Methylpyridine-3-carbohydrazide on condensation with different aromatic aldehydes resulted in hydrazones, which on cyclocondensation with thioglycolic acid resulted in thiazolidine-4-ones. The structures of all the synthesized compounds were confirmed by analytical and spectral data.

Key words: Hydrazones, Thiazolidine-4-ones, Thioglycolic acid.

INTRODUCTION

In continuation of our work[1–5], we have synthesized 2-(phenyl/substituted phenyl)-3-(6'-methylpyridine-3'-carboxamido)-thiazolidine-4-ones by cycloaddition of hydrazones and thioglycolic acid. Hydrazones have been synthesized by using 6-methylpyridine-3-carbohydrazide with different aromatic aldehydes. The structures of all the synthesized compounds were confirmed by analytical and spectral data.

EXPERIMENTAL

All the melting points were determined in an open capillary and are uncorrected. The reactions were monitored on TLC. The IR spectra were recorded in KBr pellets on a Perkin-Elmer 237 spectrophotometer. ^{1}H NMR spectra were recorded on a Bruker Avance DPX 400 MHz spectrometer with $CDCl_3$ as a solvent and using TMS as internal reference. Elemental analyses were carried out on a Carlo Erba 1108 model analyzer.

Preparation of N - (4 - methoxy benzylidene) – 6 - methylpyridine – 3 - carbonyl hydrazone (3e)

A mixture of 6-methylpyridine-3-carbohydrazide (0.01 mol) and 4-methoxy benzaldehyde (0.01 mol) in dry toluene (30 mL) was refluxed on a water bath using Dean-

* Author for correspondence; E-mail: dranjani_solankee@yahoo.com

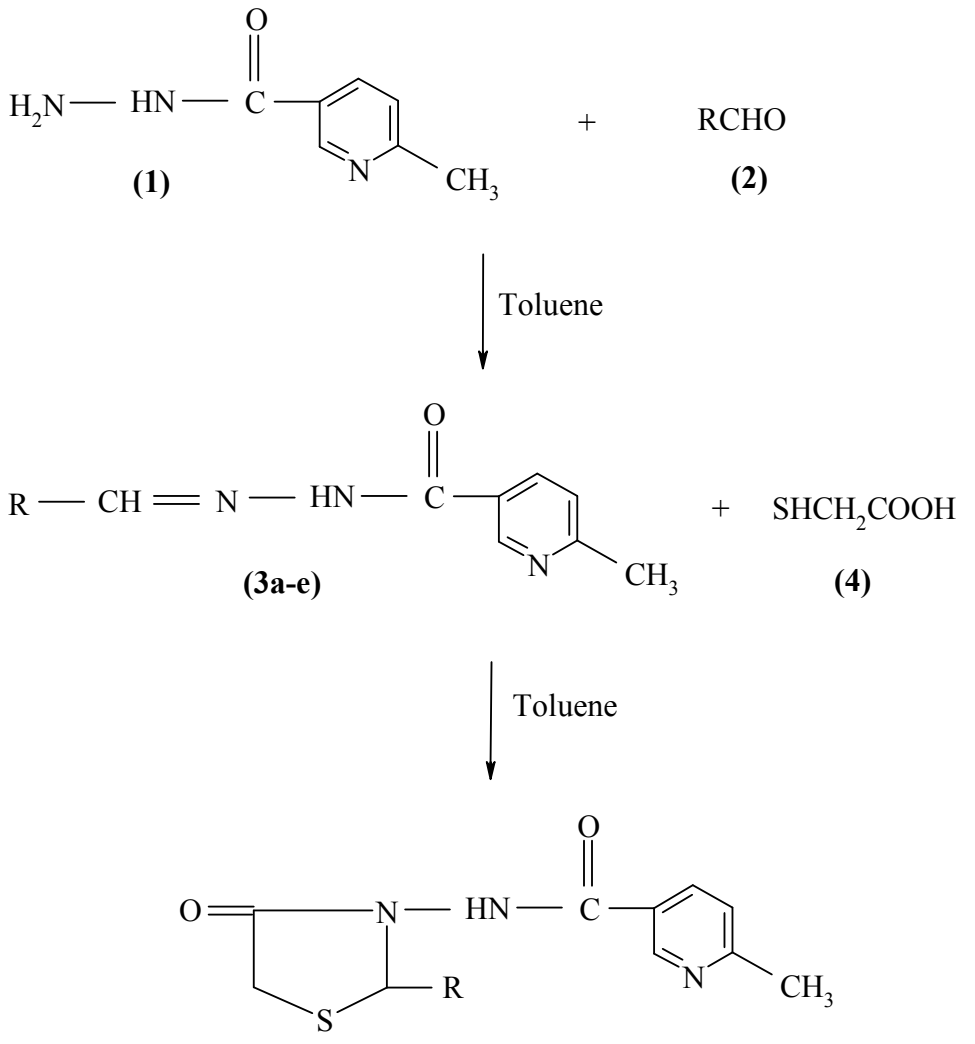
Stark water separator for 4-5 hours. The excess of toluene was evaporated and the residue of hydrazone was recrystallized from alcohol. Yield 71%; m.p. 178 °C; IR (KBr): (cm^{-1}) 1677 (>C=O, -CONH), 1648 (-N=CH-). ^{1}H NMR ($CDCl_3$): (δ ppm), 2.5 (s, 3H, $-CH_3$), 3.8 (s, 3H, $-OCH_3$), 7.00 to 9.00 (m, 7H, Ar-H), 8.4 (s, 1H, -N=CH-), 11.80 (s, 1H, -CONH-). Similarly remaining compounds were prepared by this method.

Table 1. Physical and analytical data of compounds (3a-e) and (5a-e)

R	Molecular formula	Melting point (°C)	Elemental analyses			
			C (%)		N (%)	
			Found	Calcd.	Found	Calcd.
Phenyl	$C_{14}H_{13}N_3O$	180	70.32	70.29	17.59	17.57
2-Chlorophenyl	$C_{14}H_{12}N_3OCl$	123	61.44	61.43	15.34	15.36
3-Chlorophenyl	$C_{14}H_{12}N_3OCl$	121	61.41	61.43	15.37	15.36
4-Chlorophenyl	$C_{14}H_{12}N_3OCl$	215	61.45	61.43	15.35	15.36
4-Methoxyphenyl	$C_{15}H_{15}N_3O_2$	178	66.93	66.91	15.60	15.61
Phenyl	$C_{16}H_{15}N_3O_2S$	114	61.32	61.34	13.39	13.42
2-Chlorophenyl	$C_{16}H_{14}N_3O_2SCl$	181	55.26	55.25	12.12	12.09
3-Chlorophenyl	$C_{16}H_{14}N_3O_2SCl$	107	55.28	55.25	12.11	12.09
4-Chlorophenyl	$C_{16}H_{14}N_3O_2SCl$	95	55.24	55.25	12.08	12.09
4-Methoxyphenyl	$C_{17}H_{17}N_3O_3S$	105	59.51	59.48	12.25	12.24

Preparation of 2-(4-chlorophenyl)-3-(6'-methylpyridine-3'-carboxamido)-thiazolidine-4-one (5d)

A mixture of hydrazone (0.01 mol) and thioglycolic acid (0.01 mol) in dry toluene (60 mL) was refluxed on a water bath using Dean-Stark water separator for 10-12 hours. Excess of toluene was then distilled off and the resulting viscous liquid was treated with saturated $NaHCO_3$ solution to remove unreacted thioglycolic acid. The resulting product separated was washed with water, dried and recrystallized from alcohol. Yield 71%; m.p. 95°C; IR (KBr): (cm^{-1}) 1673 (>C=O, -CONH), 1716 (>C=O, Thiazolidine ring). ^{1}H NMR ($CDCl_3$): (δ ppm), 2.5 (s, 3H, $-CH_3$), 3.75 (dd, 2H, $-CH_2$), 6.00 (s, 1H, -CH-Ar), 7.10 to 8.70 (m, 7H, Ar-H), 9.00 (s, 1H, -CONH-). Similarly remaining compounds were prepared by this method.



Scheme 1

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