Synthesis and X-ray diffraction crystallographic characterization of compound 2-(α-naphtyl)-3-(α-pyridinyl)-1,3-thiazolidin-4-one

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Thiazolidinones present a wide range of useful applications especially in the biological aspect. Based on these facts, the compound of interest 2-(α-naphthyl)-3-(α-pyridinyl)-1,3-thiazolidine-4-one ($C_{18}H_{14}N_2OS$), was synthesized via multi-component reaction with the aim of obtaining a compound that would show activity against fungi and bacteria. The synthesis of 2-(α-naphthyl)-3-(α-pyridinyl)-1,3-thiazolidine-4-one, was carried out from the respective α-aminopyridine with α-naphthylaldehyde and α-mercaptoacetic acid, under reflux in dry toluene for 8 h, obtaining a solid compound. Molecular characterization of the compound was carried out by infrared spectrometry, mass spectrometry, and nuclear magnetic resonance. The study of the crystallization and the calculation of the unit-cell constants were determined by the technique of X-ray diffraction of polycrystalline samples. It was determined that the compound crystallizes in a monoclinic system with space group $P2_1/c$ [No. 14] and the constants of the unit cell $a = 11.958$ (3), $b = 9.675$ (4), $c = 12.661$ (4) Å, $\beta = 96.960°$ (2), $V = 1454.01$ (Å^3).
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Key words: crystallographic characterization, X-ray powder diffraction, thiazolidinones

I. INTRODUCTION

Five member heterocyclic compounds are known for their multiple applications which have motive their wide study. Among this group of heterocycles, thiazolidinones have performed a wide range of biological activity (antifungal, antibacterial, analgesic, pesticide, herbicide, antitubercular, local anesthetic, and antimicotic) (Brown, 1961; Singh, 2014). The biological meaning of this class of compounds stimulates the study upon the synthesis and properties of our compound of interest 2-(α-naphtyl)-3-(α-pyridinyl)-1,3-thiazolidin-4-one, because several protocols have been developed for the synthesis of these kind of materials (Kouznetsov *et al.*, 2006; Pânzariu *et al.*, 2016). Thiazolidinone of interest was synthesized via multicomponent reaction in order to obtain a new compound with prominent antifungal and antibacterial activity.

II. EXPERIMENTAL

A. Synthesis

Compound 2-(α-naphtyl)-3-(α-pyridinyl)-1,3-thiazolidin-4-one (4) was synthesized by means of a multicomponent reaction promoted by glacial acetic acid in stoichiometric quantities between α-aminopyridine (1), α-naphtylaldehyde (2), and α-mercaptoacetic acid (3), employing anhydride toluene as solvent to reflux for 8 h. The preparation route of the interest compound is shown in Figure 1.

Once the synthesis process was accomplished, the melting point (measured on a Fisher Johns melting point apparatus) and density (by floating method) were determined, and the molecular characterization of the compound was developed through instrumental methods of infrared spectrometry (IR) employing a Lumex Infralum FT-02 (KBr) spectrophotometer, gas chromatography coupled to mass spectrometry (GC–MS) using a gas chromatograph Agilent Technologies 6890 with an interface to a mass selective detector Agilent Technologies MSD 5963, and nuclear magnetic resonance (NMR) with a Bruker AM-400 or AC-300.

B. Powder data collection

A small amount of the compound, $C_{18}H_{14}N_2OS$ was gently ground in an agate mortar and sieved to a grain size of <38 μm. The specimen was mounted on a zero-background

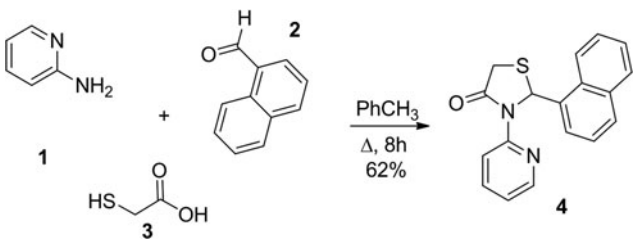

Figure 1. Synthesis of 2-(α-naphtyl)-3-(α-pyridinyl)-1,3-thiazolidin-4-one via multicomponent reaction.

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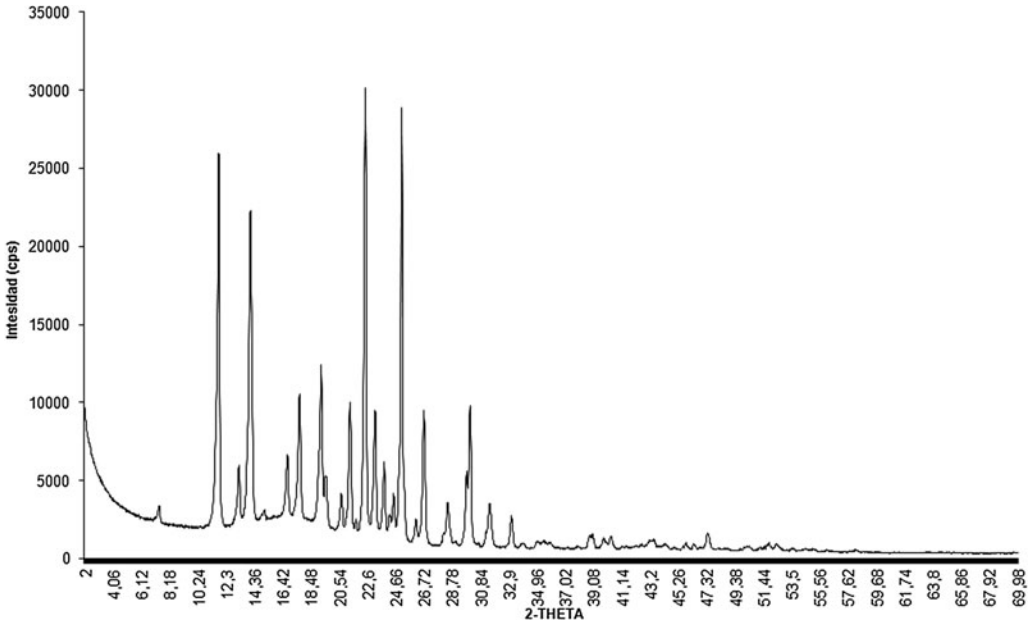

Figure 2. X-ray powder diffraction pattern of 2-(α-naphtyl)-3-(α-pyridinyl)-1,3-thiazolidin-4-one.

TABLE I. X-Ray diffraction data for the compound 2-(α-naphtyl)-3-(α-pyridinyl)-1,3-thiazolidin-4-one.

$2\theta_{obs}$ (°)	d_{obs} (Å)	$(I/I_0)_{obs}$	h	k	L	$2\theta_{calc}$ (°)	d_{calc} (Å)	$\Delta 2\theta$ (°)
7.445	11.8646	3	1	0	0	7.442	11.8699	−0.003
11.784	7.5039	71	−1	1	0	11.791	7.4995	0.007
13.276	6.6637	11	−1	1	1	13.266	6.6686	−0.010
14.086	6.2823	59	0	0	2	14.083	6.2838	−0.003
14.197	6.2334	38	1	1	1	14.197	6.2334	0.000
15.125	5.8530	2	−1	0	2	15.121	5.8547	−0.004
16.784	5.2780	14	0	1	2	16.810	5.2698	0.026
17.692	5.0136	26	−1	1	2	17.692	5.0090	0.016
19.084	4.6468	14	1	1	2	19.093	4.6446	0.009
19.253	4.6064	32	−2	0	2	19.271	4.6020	0.018
19.562	4.5343	10	2	1	1	19.565	4.5336	0.003
19.645	4.5153	9	0	2	1	19.648	4.5147	0.003
20.712	4.2851	7	−1	2	1	20.726	4.2822	0.014
21.351	4.1582	26	1	2	1	21.342	4.1600	−0.009
			−2	1	2	21.363	4.1558	
21.784	4.0766	3	2	0	2	21.791	4.0752	0.007
22.458	3.9557	88	3	0	0	22.453	3.9566	−0.005
23.172	3.8354	24	0	2	2	23.185	3.8332	0.013
23.841	3.7293	15	−1	2	2	23.841	3.7293	0.000
24.240	3.6688	5	−2	2	1	24.225	3.6710	−0.015
			3	1	0	24.284	3.6622	
24.524	3.6270	9	−3	1	1	24.524	3.6270	0.000
24.923	3.5698	14	1	2	2	24.912	3.5714	−0.011
			−3	0	2	25.081	3.5476	
25.111	3.5435	100	1	1	3	25.110	3.5436	−0.001
25.315	3.5154	10	2	2	1	25.280	3.5201	−0.035
26.162	3.4035	5	−2	1	3	26.159	3.4039	−0.003
26.725	3.3330	27	−2	2	2	26.715	3.3343	−0.010
			−3	1	2	26.743	3.3308	
28.154	3.1670	2	0	2	3	28.156	3.1668	0.002
			0	0	4	28.384	3.1419	
28.460	3.1337	9	−1	0	4	28.469	3.1326	0.009
			−1	2	3	28.475	3.1321	
28.690	3.1091	2	1	3	0	28.660	3.1122	−0.030
29.095	3.0667	1	3	2	0	29.134	3.0627	0.039
			1	3	1	29.768	2.9988	
29.851	2.9907	15	1	2	3	29.835	2.9923	−0.016
			0	1	4	29.876	2.9883	

Continued

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TABLE I. Continued

$2\theta_{obs}$ (°)	d_{obs} (Å)	$(I/I_0)_{obs}$	h	k	L	$2\theta_{calc}$ (°)	d_{calc} (Å)	$\Delta 2\theta$ (°)
30.097	2.9668	30	4	0	0	30.090	2.9675	−0.007
30.722	2.9079	<1	−2	2	3	30.734	2.9067	0.012
31.269	2.8583	3	−3	2	2	31.240	2.8608	−0.029
31.521	2.8360	9	4	1	0	31.509	2.8370	−0.012
			−2	3	0	31.547	2.8337	
			−4	1	2	33.078	2.7060	
33.112	2.7033	7	4	1	1	33.141	2.7010	0.029
33.814	2.6487	1	2	0	4	33.833	2.6473	0.019
34.015	2.6335	1	0	2	4	33.996	2.6349	−0.019
35.421	2.5322	1	−4	2	1	35.440	2.5308	0.019
			4	2	0	35.460	2.5295	
35.645	2.5168	1	1	2	4	35.616	2.5187	−0.029
35.890	2.5001	1	−3	3	0	35.894	2.4998	0.004
36.024	2.4911	1	−3	3	1	36.063	2.4885	0.039
			−4	1	3	36.092	2.4866	
38.816	2.3181	3	1	4	1	38.804	2.3188	−0.012
			−5	1	1	38.842	2.3166	
39.038	2.3055	3	−5	1	0	39.036	2.3156	−0.002
			−3	2	4	39.068	2.3038	
39.804	2.2628	2	3	1	4	39.811	2.2625	0.007
			0	4	2	39.905	2.2573	
			−1	4	2	40.311	2.2355	
40.381	2.2318	3	4	1	3	40.377	2.2320	−0.004
			0	4	3	43.152	2.0947	
43.166	2.0941	2	0	0	6	43.155	2.0946	−0.11
43.471	2.0801	2	2	4	2	43.471	2.0800	0.002
			−4	2	4	43.518	2.0779	
			1	1	6	45.826	1.9785	
45.843	1.9778	1	6	0	0	45.831	1.9783	−0.012
			−6	0	2	46.381	1.9561	
46.402	1.9553	1	5	2	2	46.419	1.9546	0.017
			−6	1	2	47.376	1.9173	
47.395	1.9166	4	0	4	4	47.395	1.9166	0.000
50.275	1.8134	1	−6	2	2	50.271	1.8135	−0.001
51.883	1.7601	2	4	4	2	51.909	1.7601	0.026
			0	5	3	52.016	1.7567	
			0	3	6	52.018	1.7566	
			−1	4	5	52.372	1.7456	
52.411	1.7444	2	−6	1	4	52.399	1.7447	−0.012
53.569	1.7094	1	3	5	1	53.561	1.7096	−0.008
			−5	3	4	53.582	1.7090	
			−7	1	1	54.508	1.6821	
54.573	1.6803	1	5	2	4	54.554	1.6808	
55.107	1.6652	1	−6	2	4	55.101	1.6654	−0.006
			4	4	3	55.136	1.6644	
			2	3	6	56.042	1.6396	
56.089	1.6384	<1	2	1	7	56.050	1.6394	−0.039

specimen holder (Buhrke *et al.*, 1998) for the respective measurement. The data were collected at 298 K using a Rigaku model D/MAX IIIB diffractometer with a graphite monochromator operating in Bragg–Brentano geometry equipped with an X-ray tube (CuKα radiation: $\lambda = 1.5406$ Å, 40 kV and 35 mA), a NaI (Tl) scintillation detector, and fixed scatter and divergence slits of 1° and 0.03 mm receiving slit. The scan range was from 2 to 70° 2θ with a step size of 0.02° 2θ and a counting time of 15 s step^{-1}.

POWDERX program (Dong, 1999) was used to remove the background (Sonneveld and Visser, 1975), smooth the data (Savitzky and Golay, 1964), eliminate the Kα_2 component (Rachinger, 1948), and to determine the positions and intensities of the diffraction peaks, using the second derivative method.

III. RESULTS AND DISCUSSION

A. Synthesis

Compound 2-(α-naphtyl)-3-(α-pyridinyl)-1,3-thiazolidin-4-one was obtained with a 62% yield, with a melting point of 141–143 °C, and a measured density of 1.32 g cm^{-3}. Molecular characterization of the compound was developed by IR spectrometry: tension C(sp^3)–H (3054.74); tension C (sp^2)–H (3000.75); vibration tension C(=O)–N (1689.37); vibration tension C(sp^2)–C (1581.37); enlargement C–N (1432.22); asymmetric tension C(sp^2)–O (1288.24); deformation vibration C(sp^2)–H (779.11), deformation vibration C–S (685.33); GC–MS: $m/z = 306.38$ (M^+) and NMR: ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.39 (1H, *d*, $J = 8.4$ Hz, 14-H),

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TABLE II. Crystal data for 2-(α-naphtyl)-3-(α-pyridinyl)-1,3-thiazolidin-4-one via multicomponent reaction.

a (Å)	11.958 (3)
b (Å)	9.675 (4)
c (Å)	12.661 (4)
β (°)	96.960 (2)
V (Å^3)	1454.01 (1)
Z	4
Space group	$P2_1/c$ [No. 14]
M_{20}	29.1
F_{30}	48.6 (0.018, 57)
D_m	1.32 g cm^{-3}

8.12 (1H, *d*, *J* = 4.1 Hz, 4-H), 8.05 (1H, *d*, *J* = 8.4 Hz, 12-H), 7.89 (1H, *d*, *J* = 8.0 Hz, 10-H), 7.74 (2H, *t*, *J* = 8.4 Hz, 11-H, 13-H), 7.68 (1H, *s*, 3-H), 7.63 (1H, *t*, *J* = 7.6 Hz, 7-H), 7.55 (1H, *t*, *J* = 7.4 Hz, 7-H), 7.30 (1H, *t*, *J* = 7.7 Hz, 9-H), 7.19 (1H, *d*, *J* = 7.1 Hz, 6-H), 6.97 (1H, *t*, *J* = 6.0 Hz, 5-H), 3.97 (1H, *d*, *J* = 16.1 Hz, 1-H), 3.79 (1H, *d*, *J* = 16.1 Hz, 2-H). ^{13}C NMR (101 MHz, DMSO) δ 172.19, 151.04, 147.96, 137.98, 136.19, 134.30, 129.92, 129.24, 128.68, 126.73, 126.19, 125.21, 122.83, 121.00, 120.57, 116.08, 59.84, 34.51.

B. X-ray diffraction of polycrystalline samples

The X-ray powder diffraction (XRPD) pattern of 2-(α-naphthyl)-3-(α-pyridinyl)-1,3-thiazolidine-4-one is shown in Figure 2; a small amount of amorphous component in the background was observed because of the type of mount since paraffin was used as a support, which results in some discrepancies in peak intensities of the reflection list, compared with the simulated pattern, using a Le Bail refinement. The peak list for this compound are given in Table I. The XRPD pattern was successfully indexed using the DICVOL06 program (Boultif and Louër, 2006) on a monoclinic cell with an absolute error of ±0.03°2θ in the calculations. The space group, $P2_1/c$ [No. 14] was estimated by the CHEKCELL program (Laugier and Bochu, 2002), which was compatible with the systematic absences and the crystal density, 1.320 g cm^{-3}. The unit-cell parameters were refined with the NBS*AIDS83 program using the total observed reflex ions (Miguell *et al.*, 1981). The crystal data, X-ray density, as well as figures of merit M_{20} (de Wolff, 1968) and F_{30} (Smith and Snyder, 1979) are compiled in Table II.

SUPPLEMENTARY MATERIAL

The supplementary material for this article can be found at https://doi.org/10.1017/S0885715618000453

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