Synthesis and X-ray diffraction data of 4-benzyloxy-1-oxaspiro-[4.6]-undec-3-en-2-one

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(Received 13 June 2013; accepted 21 June 2013)

The 4-benzyloxy-1-oxaspiro-[4.6]-undec-3-en-2-one ($C_{17}H_{20}O_3$) was prepared through a domino reaction from benzyl α-hydroxycycloheptanecarboxylate and the cumulated ylide $Ph_3P{=}C{=}C{=}O$ by: (i) addition and (ii) intramolecular Wittig Olefination reaction. The reaction was carried out using anhydrous toluene as solvent under an argon atmosphere in a Schlenk flask. Molecular characterization was performed by Fourier transform infrared spectroscopy, gas chromatography-mass spectrometry, (^{1}H,^{13}C – mono and bidimensional) nuclear magnetic resonance spectroscopy; crystallographic characterization was completed by X-ray diffraction of polycrystalline samples (XRPD). The title compound crystallized in a monoclinical system and unit-cell parameters are reported [a = 13.207(3) Å, b = 5.972(1) Å, c = 19.719(4) Å, β = 105.67(2)°, unit-cell volume V = 1497.5 (4) Å^3, Z = 4]. All of the measured lines were indexed with the $P2_1/n$ (No. 14) space group. © *2013 International Centre for Diffraction Data.* [doi:10.1017/S088571561300064X]

Key words: *O*-Bencyl tetronate, X-ray powder diffraction data, spiro tetronic acid

I. INTRODUCTION

There are many studies related to the synthesis of compounds structurally related to conjugated systems derived from the 4-hydroxyfuran-2[5*H*]-one core; many of these derivatives have shown several activities in different biological reactions and they are usually employed as precursors of substances of high relevance in the battle against many diseases. Most of the methodologies reported for its preparation are generally limited by the instability of the substances employed and the difficulty of obtaining the desired structure (Tejedor, 2004).

Until now, one of the best methodologies used to obtain butenolides (furan-2[5*H*]-ones) is Wittig Olefination; using the cumulated ylide $Ph_3P{=}C{=}C{=}O$ as a Wittig reagent and different α-hydroxyesters, it is possible to achieve the synthesis of several analogs of substances that have shown biological activity (antitumor and anticancer among the most important); the use of $Ph_3P{=}C{=}C{=}O$ also allows us to obtain molecules with spiroatoms that usually are not easy to access by the conventional reaction methods (Schobert, 2007).

In this work, the preparation of 4-benzyloxy-1-oxaspiro-[4.6]-undec-3-en-2-one (**2**) using $Ph_3P{=}C{=}C{=}O$ and the corresponding benzyl α-hydroxycycloheptanecarboxylate (**1**) is presented (Figure 1), reporting molecular characterization [Fourier transform infrared spectroscopy, gas chromatography-mass spectrometry (GC-MS), ^{1}H, nuclear magnetic resonance spectroscopy on carbons (^{13}C NMR), and X-ray powder diffraction (XRPD)] data. Crystallographic information by X-ray diffraction about this type of derivatives has been little explored. This compound has been obtained in an easy and fast way and owing to its similarity with the analogs already reported is expected to be of biological interest.

Figure 1. Synthesis of 4-benzyloxy-1-oxaspiro-[4.6]-undec-3-en-2-one (**2**).

II. EXPERIMENTAL

A. Synthesis

In a 250-ml round bottom Schlenk flask, 2.18 g (7.21 mmol) of keteneylidenetriphenylphosphorane ($Ph_3P{=}C{=}C{=}O$) were loaded under argon atmosphere; 150 ml of anhydrous toluene were added and the mixture was magnetically stirred. Approximately 0.87 g (3.5 mmol) of benzyl 1-hydroxycycloheptanecarboxylate (**1**) (previously dried under vacuum for 1 h) were added. The reaction mixture was refluxed for 72 h, after completion of the reaction indicated by thin layer chromatography (TLC). Toluene was removed under vacuum using a rotary evaporator. To remove the phosphine oxide formed during the reaction, the residue was initially dissolved in dichlorometane (DCM) and filtered over silica gel (60–120 mesh). Then, the residual crude product was purified by column chromatography using silica gel (60–120 mesh) and hexane–ethyl acetate (5:1) as eluents

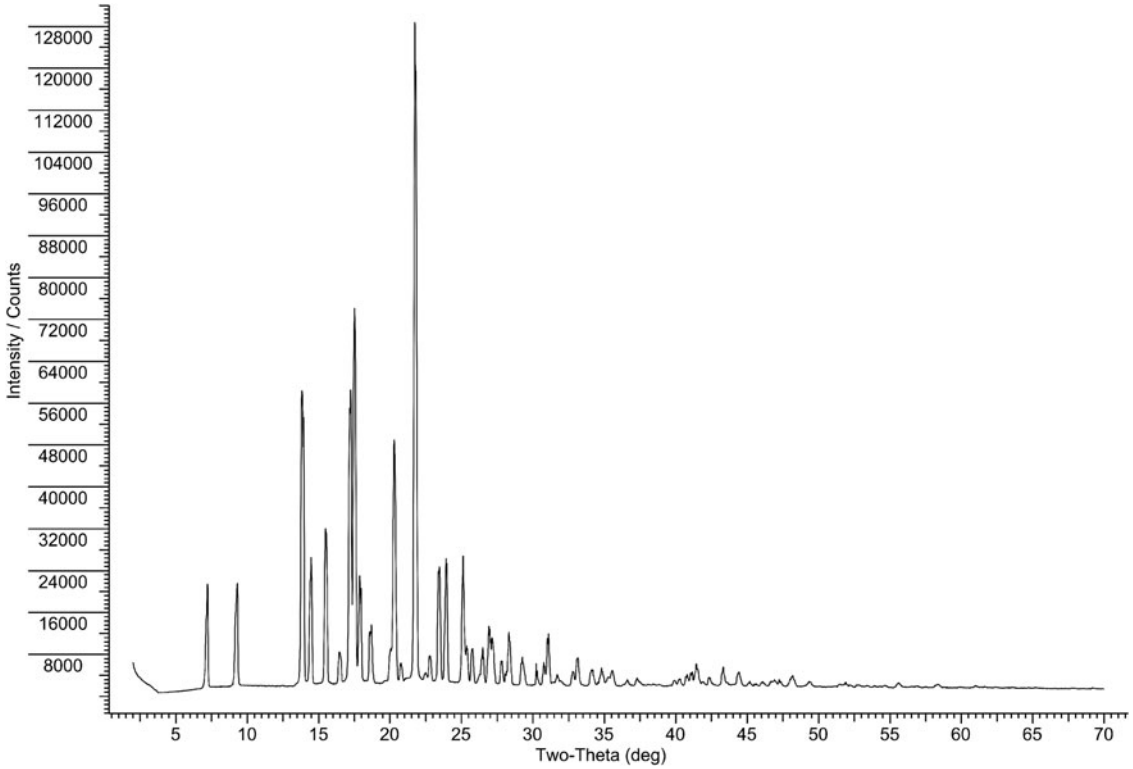

Figure 2. X-ray powder diffraction pattern of 4-benzyloxy-1-oxaspiro-[4.6]-undec-3-en-2-one.

TABLE I. X-ray powder diffraction data of 4-benzyloxy-1-oxaspiro-[4.6]-undec-3-en-2-one. Cu$K\alpha_1$ radiation ($\lambda = 1.5406$ Å).

$2\theta_{obs}$ (°)	d_{obs} (Å)	$(I/I_0)_{obs}$	h	k	l	$2\theta_{calc}$ (°)	d_{calc} (Å)	$\Delta 2\theta$ (°)
7.243	12.1950	13	−1	0	1	7.241	12.1978	−0.002
9.315	9.4865	13	0	0	2	9.308	9.4932	0.007
			0	0	1	9.350	9.4514	
13.846	6.3907	40	−1	0	3	13.833	6.3966	−0.013
14.505	6.1018	14	−2	0	2	14.512	6.0989	0.007
15.521	5.7046	21	0	1	1	15.543	5.6966	0.022
16.482	5.3740	4	−1	1	1	16.515	5.3635	0.033
17.269	5.1309	38	1	0	3	17.240	5.1393	−0.029
17.534	5.0539	50	0	1	2	17.531	5.0548	−0.003
			1	1	1	17.553	5.0485	
17.903	4.9506	14	−1	1	2	17.913	4.9479	0.010
18.709	4.7391	7	0	0	4	18.679	4.7466	0.030
			−2	1	1	20.051	4.4247	
20.075	4.4196	4	−2	0	4	20.081	4.4196	0.006
20.325	4.3658	33	−1	1	3	20.328	4.3652	0.003
20.762	4.2749	2	−2	1	2	20.801	4.2669	0.039
21.772	4.0788	100	2	1	1	21.756	4.0817	−0.016
21.851	4.0642	76	−3	0	3	21.842	4.0659	−0.009
			−2	1	3	22.532	3.9429	
22.537	3.9420	1	−1	0	5	22.537	3.9421	0.000
22.791	3.8987	3	1	1	3	22.810	3.8954	0.019
23.459	3.7891	15	−1	1	4	23.461	3.7888	0.002
23.961	3.7109	16	0	1	4	23.929	3.7158	−0.032
			2	1	2	23.995	3.7058	
25.130	3.5408	17	−3	1	1	25.130	3.5409	0.000
25.396	3.5044	5	−3	1	2	25.387	3.5056	−0.009
25.750	3.4570	4	3	1	0	25.753	3.4566	0.003
			1	0	5	26.222	3.3958	
26.271	3.3896	1	2	0	4	26.267	3.3901	−0.004
26.514	3.3591	4	−3	1	3	26.499	3.3609	−0.015
			2	1	3	26.898	3.3120	
26.963	3.3041	8	−3	0	5	26.941	3.3067	−0.022
			−2	1	1	20.051	4.4247	

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)	$\Delta 2\theta$ (°)
27.180	3.2783	6	3	1	1	27.199	3.2760	0.019
27.808	3.2056	3	0	1	5	27.819	3.2043	0.011
28.128	3.1699	2	−2	1	5	28.162	3.1661	0.034
			0	0	6	28.178	3.1644	
			3	0	3	28.305	3.1505	
28.351	3.1455	7	−3	1	4	28.375	3.1429	0.024
29.306	3.0451	3	−4	0	4	29.263	3.0495	−0.043
			1	1	5	30.253	2.9519	
30.268	2.9505	3	0	2	1	30.277	2.9496	0.009
			2	1	4	30.293	2.9481	
30.802	2.9005	3	−1	2	1	30.805	2.9002	0.003
			−1	1	6	31.041	2.8787	
31.063	2.8767	7	−4	1	1	31.059	2.8771	−0.004
			0	2	2	31.381	2.8483	
31.402	2.8465	<1	1	2	1	31.394	2.8472	−0.008
31.734	2.8174	1	−2	1	6	31.711	2.8194	−0.023
31.973	2.7969	1	0	1	6	31.982	2.7961	0.009
			4	0	2	31.986	2.7958	
32.090	2.7870	<1	3	1	3	32.096	2.7865	0.006
32.771	2.7306	2	1	2	2	32.746	2.7326	−0.025
			−1	2	3	33.082	2.7056	
33.115	2.7030	4	2	2	0	33.119	2.7027	0.004
			0	2	3	33.148	2.7004	
			2	1	5	34.053	2.6307	
34.072	2.6293	2	−3	0	7	34.066	2.6297	−0.006
34.200	2.6197	2	−5	0	3	34.236	2.6170	0.036
			−5	0	1	34.257	2.6155	
34.575	2.5921	<1	−2	2	3	34.529	2.5955	−0.046
34.734	2.5806	1	1	2	3	34.718	2.5818	−0.016
34.867	2.5711	2	2	0	6	34.887	2.5697	0.020
			−4	1	5	34.906	2.5684	
35.192	2.5481	1	−1	2	4	35.165	2.5500	−0.027
35.299	2.5406	1	3	1	4	35.299	2.5407	0.000
			2	2	2	35.536	2.5242	
35.567	2.5221	2	−2	1	7	35.588	2.5207	0.021
			−3	2	1	36.340	2.4702	
36.357	2.4691	<1	0	1	7	36.350	2.4696	−0.007
36.638	2.4508	1	−2	0	8	36.637	2.4508	−0.001
36.869	2.4360	<1	5	0	1	36.872	2.4358	0.003
37.312	2.4080	1	−3	1	7	37.334	2.4067	0.022
			−3	2	3	37.335	2.4066	
			−5	1	3	37.491	2.3970	
37.516	2.3954	<1	−5	1	1	37.510	2.3958	−0.006
37.859	2.3745	<1	3	2	1	37.853	2.3749	−0.006
			0	0	8	37.879	2.3733	
			4	0	4	38.053	2.3629	
38.093	2.3605	<1	2	1	6	38.094	2.3604	0.001
			−5	1	4	38.401	2.3422	
38.437	2.3401	<1	5	1	0	38.440	2.3400	0.003
38.571	2.3323	<1	−2	2	5	38.576	2.3320	0.005
38.761	2.3213	<1	−3	2	4	38.737	2.3227	−0.024
			1	1	7	38.786	2.3199	
38.877	2.3146	<1	3	1	5	38.871	2.3150	−0.006
39.634	2.2722	<1	−1	1	8	39.652	2.2722	0.018
39.889	2.2582	1	−5	1	5	39.886	2.2584	−0.003
40.268	2.2378	1	2	2	4	40.215	2.2407	−0.053
40.332	2.2344	1	−4	1	7	40.330	2.2345	−0.002
40.765	2.2117	1	−4	2	2	40.752	2.2124	−0.013
			−1	2	6	40.802	2.2097	
			4	1	4	41.048	2.1971	
41.087	2.1951	2	−3	1	8	41.089	2.1950	0.002
41.389	2.1798	2	−1	0	9	41.418	2.1783	0.029
41.477	2.1754	3	−4	2	0	41.455	2.1765	−0.022
			0	2	6	41.550	2.1717	
			−5	0	7	41.571	2.1707	

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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)	$\Delta 2\theta$ (°)
41.609	2.1688	1	3	2	3	41.641	2.1672	0.032
			5	0	3	41.661	2.1662	
			−6	0	4	41.682	2.1651	
41.881	2.1553	<1	−5	1	6	41.894	2.1547	0.013
			−4	2	4	42.330	2.1335	
42.350	2.1325	1	−3	0	9	42.356	2.1322	0.006
			2	1	7	42.362	2.1319	
			6	0	0	42.624	2.1194	
42.652	2.1181	<1	4	2	1	42.645	2.1184	−0.007
43.336	2.0862	3	1	1	8	43.326	2.0867	−0.010
43.649	2.0720	<1	−4	1	8	43.651	2.0719	0.002
			−6	1	2	43.844	2.0632	
43.879	2.0617	<1	−6	1	3	43.888	2.0613	0.009
			−4	2	5	43.924	2.0597	
44.151	2.0496	<1	2	0	8	44.153	2.0495	0.002
			3	0	7	44.201	2.0474	
			−1	2	7	44.207	2.0471	
			−6	1	1	44.343	2.0412	
			4	2	2	44.351	2.0408	
44.390	2.0391	2	−5	1	7	44.368	2.0401	−0.022
			4	1	5	44.445	2.0367	
			5	1	3	44.454	2.0364	
			−6	1	4	44.474	2.0355	
44.501	2.0343	2	−2	2	7	44.488	2.0348	−0.013
			−6	0	6	44.532	2.0330	
			−3	1	9	45.114	2.0081	
45.151	2.0065	1	0	2	7	45.123	2.0077	−0.028
			4	0	6	45.544	1.9901	
45.575	1.9888	<1	0	1	9	45.567	1.9891	−0.008
			−6	1	5	45.583	1.9885	
46.080	1.9682	1	−5	2	3	46.083	1.9681	0.003
			−5	2	1	46.099	1.9674	
			1	3	0	46.119	1.9666	
46.694	1.9437	1	−1	3	2	46.740	1.9419	0.046
46.950	1.9337	1	6	1	1	46.896	1.9358	−0.054
			−5	1	8	47.254	1.9220	
			3	2	5	47.256	1.9219	
47.287	1.9207	1	−4	1	9	47.296	1.9204	0.009
47.956	1.8955	1	−5	0	9	47.984	1.8944	0.028
			−2	2	8	47.985	1.8944	
			1	1	9	48.022	1.8930	
48.041	1.8923	1	−2	3	2	48.041	1.8923	0.000
			4	1	6	48.157	1.8880	
			5	2	1	48.174	1.8874	
48.213	1.8860	1	−7	0	3	48.200	1.8865	−0.013
			0	2	8	48.989	1.8579	
			−7	0	1	49.027	1.8566	
49.046	1.8559	<1	1	3	3	49.037	1.8562	−0.009
			−3	2	8	49.166	1.8516	
49.242	1.8490	<1	−6	1	7	49.253	1.8586	0.011
			−6	0	8	49.266	1.8481	
49.364	1.8447	1	−1	3	4	49.374	1.8443	0.010
			−3	1	10	49.375	1.8443	
			−7	0	5	49.381	1.8441	
50.697	1.7992	<1	−7	1	3	50.709	1.7988	0.012
51.147	1.7845	<1	1	2	8	51.125	1.7852	−0.022
			−1	0	11	51.426	1.7755	
			−3	0	11	51.448	1.7747	
			3	3	1	51.449	1.7747	
51.470	1.7740	<1	2	1	9	51.469	1.7741	−0.001
			−7	1	1	51.506	1.7729	
51.566	1.7710	<1	−6	2	2	51.582	1.7704	0.016
			−6	2	3	51.621	1.7692	
			6	0	4	51.725	1.7659	
51.747	1.7652	<1	−6	1	8	51.738	1.7655	−0.009
			−2	2	9	51.781	1.7641	

Continued

https://doi.org/10.1017/S088571561300064X Published online by Cambridge University Press

TABLE I. Continued

$2\theta_{obs}$ (°)	d_{obs} (Å)	$(I/I_0)_{obs}$	h	k	l	$2\theta_{calc}$ (°)	d_{calc} (Å)	$\Delta 2\theta$ (°)
51.939	1.7591	1	−1	2	9	51.918	1.7598	−0.021
			−3	2	9	52.709	1.7352	
52.734	1.7344	<1	3	3	2	52.745	1.7341	0.011
			0	2	9	53.113	1.7230	
			−7	1	6	53.122	1.7227	
53.127	1.7217	<1	−6	2	5	53.127	1.7225	−0.027
53.440	1.7132	<1	3	0	9	53.442	1.7131	0.002
53.936	1.6986	<1	2	0	10	53.959	1.6979	0.023
			3	2	7	54.282	1.6886	
54.309	1.6878	<1	6	2	1	54.304	1.6880	−0.005
			4	3	0	54.332	1.6872	
			−5	2	8	54.625	1.6788	
54.654	1.6780	<1	−4	2	9	54.663	1.6777	0.009
			5	2	4	54.713	1.6763	
55.516	1.6539	1	−6	0	10	55.536	1.6539	0.020
55.627	1.6509	1	−5	0	11	55.653	1.6509	0.026
			−3	3	6	55.686	1.6493	
56.117	1.6376	<1	6	2	2	56.104	1.6380	−0.013
			−1	2	10	56.159	1.6365	
56.506	1.6273	<1	−3	2	10	56.545	1.6262	0.039
56.852	1.6182	<1	−2	3	7	56.838	1.6186	−0.014
56.983	1.6148	<1	−7	1	8	56.968	1.6152	−0.015
			−8	0	6	57.368	1.6049	
57.417	1.6036	<1	0	3	7	57.371	1.6048	−0.046
			−5	2	9	57.573	1.5996	
57.600	1.5989	<1	−4	0	12	57.592	1.5992	−0.008
58.187	1.5842	<1	−5	3	3	58.181	1.5844	−0.006
			−2	1	12	58.195	1.5840	
			−5	3	1	58.195	1.5840	
			0	0	12	58.268	1.5822	
58.313	1.5811	1	6	2	3	58.312	1.5811	−0.001
58.444	1.5779	<1	2	2	9	58.459	1.5775	0.015
			−7	2	1	58.493	1.5766	
58.821	1.5686	<1	−7	2	5	58.808	1.5690	−0.013
			−5	3	4	58.838	1.5682	
			−1	1	12	58.856	1.5678	

to afford the pure *O*-benzyltetronate (**2**) (yield 70%). $R_f = 0.4$ (SiO_2, hexane:ethyl acetate, 5:2).

4-O-(bencyloxy)-1-oxaspiro[4.6]undec-3-en-2-one (***2***): White solid, m.p. 95 °C. IR [KBr, ν (cm^{-1})] = 3472 (w), 3119 (w), 2924 (m), 2862 (w), 1747 (vs), 1624 (vs), 1460 (w), 1033 (m) [ν(C–O)], 812 (m), 736 (w), 700 (m). ^{1}H NMR (400 MHz, $CDCl_3$), δ (ppm): 7.42 (2H, d, *J**, H_{ortho}), 7.40 (2H, td, *J**, H_{meta}), 7.36 (1H, t, *J**, H_{para}), 5.05 (2H, s, ϕ–CH_2–), 4.95 (1H, s, H–C=), 1.50–2.03 (12H, m, cycloheptane ring) [* *J* not resolved, appear as broad signal]. ^{13}C-NMR (100 Hz, $CDCl_3$), δ (ppm): 185.99 (C = O), 172.27 (4-C), 134.34 (C_{ipso}), 128.96 (2C, C_{meta}), 128.89 (C_{para}), 127.60 (2C,C_{ortho}), 87.39 (5-C_{spiro}), 87.22 (H–C =), 74.18 (ϕ–CH_2–), 36.94 (2C, *a-C*), 29.24 (2C, *b-C*), 22.67 (2C, *c-C*). GC-MS (EI) *m/z* (%): 272 (1, M^+), 254 (2, M^+–H_2O), 228 (1, M^+–CO_2), 181 (2, M^+–C_7H_7), 127 (1, M^+–CO–C_8H_5O), 91 (100, M^+–$C_{10}H_{13}O_3$), 95 (10, M^+–$C_{10}H_9O_3$,), and 56 (4, M^+–$C_{13}H_{12}O_3$). Anal. Calcd for $C_{17}H_{20}O_3$ (272 g mol^{-1}): C, 74.97; H, 7.40.

B. Powder data collection

The title compound was ground and sieved to a grain size <38 μm. The compound was mounted on a zero-background specimen holder. The diffraction pattern was collected at room temperature (298 K) in the range from 2 to 70° 2θ with a step size of 0.015 26° 2θ and a count time of 0.4 s per step, using an D8 ADVANCE BRUKER with geometry DaVinci diffractometer operating in Bragg–Brentano geometry quipped with a Cu-target X-ray tube (40 kV and 30 mA), a nickel filter and a one-dimensional LynxEye detector. A fixed antiscatter slit of 8 mm, receiving slit of 1 mm, soller slits of 2.5°, and a detector slit of 0.6 mm were used.

PowderX program (Dong, 1999) was used to remove the background (Sonnerveld and Visser, 1975), smoothing (Savitzky and Golay, 1964), to eliminate the $K\alpha_2$ component

TABLE II. Crystal-structure data for 4-benzyloxy-1-oxaspiro-[4.6]-undec-3-en-2-one.

Crystal system	Monoclinic
a (Å)	13.207 (3)
b (Å)	5.972 (1)
c (Å)	19.719 (4)
β (°)	105.67 (2)
V (Å^3)	1497.5 (4)
Z	4
M_{20}	17.6
F_{30}	33.1 (0.0168, 54)
D_m	1.211 g cm^{-3}

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(Rachinger, 1948) and the second derivative method was used to determine the peak-observed positions and intensities.

III. RESULTS AND DISCUSSION

The experimental XRPD pattern is depicted in Figure 2. XRPD data for the title compound is given in Table I. Indexing of the experimental XRPD pattern was performed using the DICVOL06 program (Boultif and Louër, 2006) with an absolute error of 0.03° 2θ. The title compound crystallized in a monoclinical system with space group $P2_1/n$ (No. 14) estimated by the CHEKCELL program (Laugier and Bochu, 2002), which was compatible with the systematic absences and with the crystal density (1.211 g cm^{-3}). The unit-cell parameters were refined with the NBS*AIDS83 program (Mighell *et al.,* 1981). Unit-cell data, values of M_{20} (de Wolff, 1968) and F_{30} (Smith and Snyder, 1979) are presented in Table II.

ACKNOWLEDGEMENTS

This work was supported by internal grant 5165 (VIE-UIS). The authors express their acknowledgement to Universidad Industrial de Santander-UIS and Parque Tecnológico Guatiguará-PTG (Bucaramanga-Colombia) for data collection. José H. Quintana Mendoza thanks COLCIENCIAS and Universidad Industrial de Santander for his scholarship (Programa Jóvenes Investigadores e Innovadores, año 2012).

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