Crystal structure of isocalamendiol, $C_{15}H_{26}O_2$, *from Baccharis marginalis*

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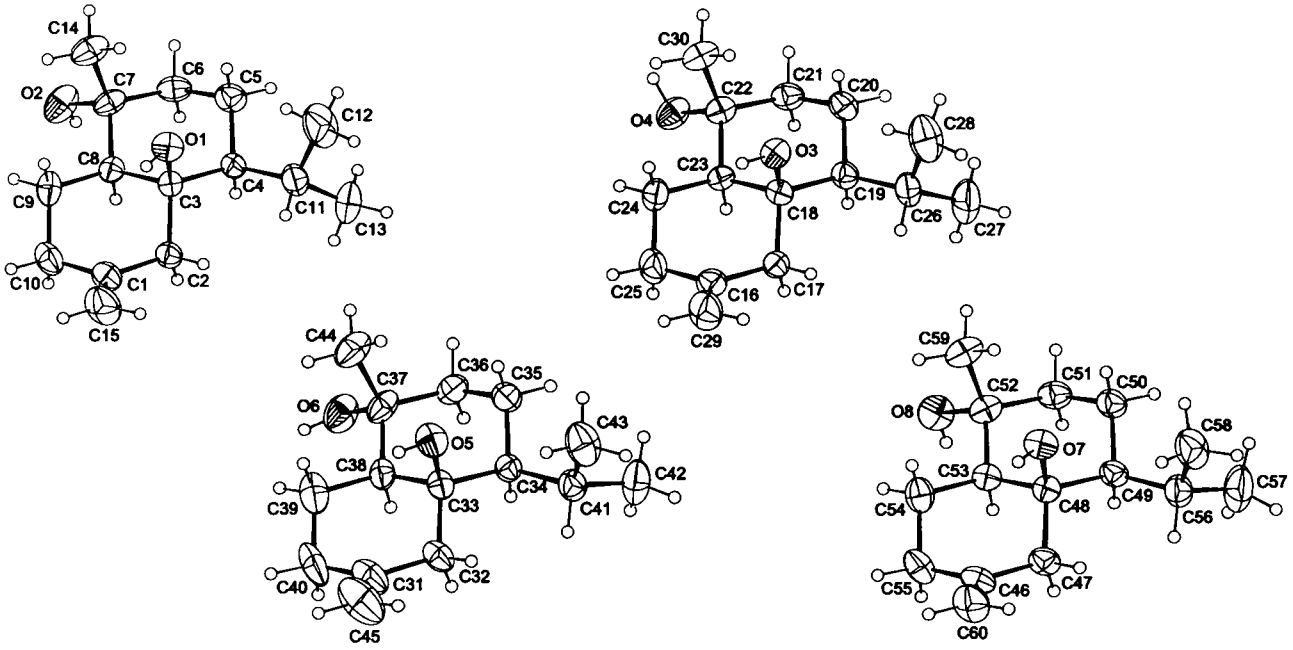

Abstract

$C_{15}H_{26}O_2$, monoclinic, $P12_11$ (no. 4), $a = 10.369(2)$ Å, $b = 27.528(6)$ Å, $c = 10.815(2)$ Å, $\beta = 107.98(3)°$, $V = 2936.3$ Å^3, $Z = 8$, $R_{gt}(F) = 0.041$, $wR_{ref}(F^2) = 0.105$, $T = 293$ K.

Source of material

Plant material (aerial part) of *Baccharis marginalis* were collected in preandine areas of de Fray Jorge Valley (IV Región, Coquimbo). Fresh plant material (12 kg) was extracted with CH_2Cl_2 for 2 h at room temperature. The crude extract was obtained after evaporate of the solvent and fractionated by flash chromatography (silica, petrol/EtOAc mixts of increasing polarity). The combined fractions of 20 % and 30 % left a white powder, the spectroscopic properties were identical with isocalamendiol (20 g).

Discussion

In the course of our continuous research into the chemistry of the American genus *Baccharis* species, we have examined the constituents of *Baccharis marginalis*. In this paper we report the isolation and X-ray structure of isocalamendiol, a sesquiterpene bicyclic diol. This compound was isolated for the first time from the rhizomes of a Japanese plant *Acorus calamus* (Shyobu) [1]. The spectral data of isocalamendiol (UV, IR, MS and ^{1}H NMR) were identical to that of our compound isolated from *Baccharis marginalis*. The most usual type of secondary metabolites isolated from *Baccharis* is that constituted by clerodane-type diterpenes, however, we reported in the previous paper the isolation and chemical characterization of sesquiterpenes that possessing carbon skeleton type: cadinane, amorfane, germacrane and spartidienedione [2-4].

The molecular structure of the title compound consists of two condensed six-membered rings in chair configuration with hydroxy substituents in axial and equatorial positions. The asymmetric unit consists of four similar independent molecules. Differences between them were found for the H–O–C–C torsion angles at O2, O4, O6, and O8 atoms. Moreover, the molecules do not show unusual geometrical features. All bond lengths and angles are within the expected ranges.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.10 × 0.20 × 0.30 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.69 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD4, $\omega/2\theta$
$2\theta_{max}$:	49.28°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5359, 5076
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2640
$N(param)_{refined}$:	634
Programs:	SHELXS-97 [5], SHELXL-97 [6], PLATON [7]

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Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	2a	0.8840	0.2529	0.8040	0.094
H(2)	2a	1.1335	0.3360	0.4981	0.130
H(3)	2a	0.1047	0.7176	0.9996	0.088
H(4)	2a	–0.1769	0.8480	0.7217	0.110
H(5)	2a	0.5770	0.4983	0.9343	0.106
H(6)	2a	0.2715	0.4129	0.5904	0.139
H(7)	2a	0.4717	0.5274	0.0909	0.092
H(8)	2a	0.2396	0.4347	0.3800	0.115
H(2A)	2a	0.7847	0.2196	0.4931	0.065
H(2B)	2a	0.7688	0.1970	0.6205	0.065
H(4A)	2a	1.0225	0.1985	0.5467	0.062
H(5A)	2a	1.2451	0.1937	0.6833	0.082
H(5B)	2a	1.2052	0.2235	0.7897	0.082
H(6A)	2a	1.1909	0.2600	0.5410	0.080
H(6B)	2a	1.3019	0.2752	0.6701	0.080
H(8A)	2a	0.9474	0.2850	0.5103	0.059
H(9A)	2a	0.8769	0.3331	0.7087	0.080
H(9B)	2a	0.8891	0.3592	0.5837	0.080
H(10A)	2a	0.6619	0.3378	0.5605	0.089
H(10B)	2a	0.7121	0.3138	0.4519	0.089
H(11)	2a	0.9190	0.1480	0.6801	0.092
H(12A)	2a	1.0612	0.1081	0.8515	0.183
H(12B)	2a	1.0832	0.1636	0.8836	0.183
H(12C)	2a	1.1888	0.1347	0.8354	0.183
H(13A)	2a	1.1361	0.1143	0.5970	0.187
H(13B)	2a	0.9845	0.1174	0.5092	0.187
H(13C)	2a	1.0270	0.0801	0.6244	0.187
H(14A)	2a	1.2556	0.3368	0.8107	0.126
H(14B)	2a	1.1535	0.3019	0.8473	0.126
H(14C)	2a	1.1071	0.3541	0.7937	0.126
H(15A)	2a	0.5731	0.2260	0.6568	0.127
H(15B)	2a	0.5268	0.2818	0.6278	0.127
H(17A)	2a	0.1592	0.6893	0.7125	0.066
H(17B)	2a	0.2421	0.6726	0.8533	0.066
H(19)	2a	0.2539	0.7721	0.7456	0.064
H(20A)	2a	0.3138	0.8442	0.8628	0.075
H(20B)	2a	0.2427	0.8257	0.9627	0.075
H(21A)	2a	0.0957	0.8771	0.8100	0.076
H(21B)	2a	0.1058	0.8434	0.6964	0.076
H(23)	2a	0.0055	0.7580	0.6825	0.059
H(24A)	2a	–0.1930	0.7421	0.7340	0.080
H(24B)	2a	–0.1061	0.7271	0.8752	0.080
H(25A)	2a	–0.1642	0.6561	0.7542	0.097
H(25B)	2a	–0.1095	0.6769	0.6448	0.097
H(26)	2a	0.4082	0.7187	0.8971	0.084
H(27A)	2a	0.5073	0.8086	0.8431	0.161
H(27B)	2a	0.4766	0.7638	0.7492	0.161
H(27C)	2a	0.5955	0.7615	0.8804	0.161
H(28A)	2a	0.5458	0.7479	1.0889	0.170
H(28B)	2a	0.3964	0.7538	1.0921	0.170
H(28C)	2a	0.4756	0.7990	1.0647	0.170
H(29A)	2a	0.1469	0.6023	0.9112	0.122

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(29B)	2a	–0.0151	0.5951	0.8688	0.122
H(30A)	2a	–0.0495	0.8582	0.9355	0.108
H(30B)	2a	0.0343	0.8119	0.9957	0.108
H(30C)	2a	–0.1221	0.8078	0.9258	0.108
H(32A)	2a	0.6922	0.5116	0.6762	0.078
H(32B)	2a	0.7400	0.5355	0.8144	0.078
H(34)	2a	0.4756	0.5569	0.5956	0.061
H(35A)	2a	0.3236	0.5676	0.7681	0.076
H(35B)	2a	0.2920	0.5967	0.6372	0.076
H(36A)	2a	0.1552	0.5279	0.6065	0.082
H(36B)	2a	0.2503	0.5250	0.5189	0.082
H(38)	2a	0.4662	0.4699	0.6060	0.069
H(39A)	2a	0.5104	0.4236	0.8459	0.106
H(39B)	2a	0.4625	0.3970	0.7111	0.106
H(40A)	2a	0.6986	0.3895	0.8100	0.123
H(40B)	2a	0.6644	0.4143	0.6730	0.123
H(41)	2a	0.6529	0.6022	0.7275	0.079
H(42A)	2a	0.4170	0.6586	0.6259	0.151
H(42B)	2a	0.5056	0.6385	0.5431	0.151
H(42C)	2a	0.5654	0.6776	0.6511	0.151
H(43A)	2a	0.6171	0.6581	0.8777	0.150
H(43B)	2a	0.6187	0.6044	0.9260	0.150
H(43C)	2a	0.4806	0.6318	0.8692	0.150
H(44A)	2a	0.1571	0.4648	0.7597	0.133
H(44B)	2a	0.2891	0.4895	0.8494	0.133
H(44C)	2a	0.2882	0.4337	0.8198	0.133
H(45A)	2a	0.9014	0.4870	0.9564	0.167
H(45B)	2a	0.8890	0.4286	0.9536	0.167
H(47A)	2a	0.5427	0.5650	0.3966	0.072
H(47B)	2a	0.6115	0.5730	0.2879	0.072
H(49)	2a	0.6160	0.4794	0.4377	0.064
H(50A)	2a	0.5815	0.4177	0.2231	0.073
H(50B)	2a	0.6619	0.4026	0.3660	0.073
H(51A)	2a	0.4668	0.4108	0.4277	0.076
H(51B)	2a	0.4405	0.3736	0.3130	0.076
H(53)	2a	0.3759	0.4978	0.3714	0.060
H(54A)	2a	0.2421	0.5218	0.1101	0.081
H(54B)	2a	0.1683	0.5147	0.2155	0.081
H(55A)	2a	0.2080	0.5979	0.1867	0.091
H(55B)	2a	0.2783	0.5817	0.3309	0.091
H(56)	2a	0.7750	0.5256	0.3824	0.082
H(57A)	2a	0.8735	0.4308	0.4428	0.174
H(57B)	2a	0.8399	0.4650	0.5447	0.174
H(57C)	2a	0.9556	0.4790	0.4861	0.174
H(58A)	2a	0.8987	0.4890	0.2524	0.147
H(58B)	2a	0.7585	0.5117	0.1748	0.147
H(58C)	2a	0.7724	0.4553	0.1948	0.147
H(59A)	2a	0.2782	0.3880	0.1076	0.108
H(59B)	2a	0.3646	0.4307	0.0782	0.108
H(59C)	2a	0.2120	0.4392	0.0677	0.108
H(60A)	2a	0.5129	0.6399	0.1567	0.107
H(60B)	2a	0.3529	0.6507	0.1204	0.107

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	2a	0.9552(3)	0.2435(1)	0.7940(3)	0.065(2)	0.076(2)	0.052(2)	–0.004(2)	0.026(2)	0.005(2)
O(2)	2a	1.1301(4)	0.3468(1)	0.5677(3)	0.125(3)	0.070(2)	0.080(3)	–0.031(2)	0.054(3)	0.001(2)
O(3)	2a	0.1617(3)	0.7363(1)	0.9888(3)	0.066(2)	0.064(2)	0.050(2)	0.002(2)	0.023(2)	0.010(2)
O(4)	2a	–0.1322(3)	0.8289(1)	0.6934(3)	0.070(2)	0.081(3)	0.067(2)	0.026(2)	0.020(2)	0.010(2)
O(5)	2a	0.5336(4)	0.5205(1)	0.8899(3)	0.095(3)	0.069(2)	0.047(2)	0.007(2)	0.020(2)	–0.004(2)
O(6)	2a	0.2358(4)	0.4394(1)	0.5684(4)	0.117(3)	0.078(3)	0.087(3)	–0.038(2)	0.039(2)	–0.026(2)
O(7)	2a	0.5013(3)	0.5026(1)	0.1314(3)	0.079(2)	0.060(2)	0.052(2)	–0.000(2)	0.030(2)	0.006(2)
O(8)	2a	0.2212(3)	0.4274(1)	0.3030(4)	0.081(2)	0.073(2)	0.090(3)	–0.017(2)	0.048(2)	0.012(2)
C(1)	2a	0.6918(5)	0.2649(2)	0.5859(5)	0.055(3)	0.074(4)	0.067(3)	0.008(3)	0.023(3)	0.002(3)
C(2)	2a	0.7917(4)	0.2265(2)	0.5829(4)	0.042(3)	0.056(3)	0.066(3)	0.002(2)	0.019(2)	0.001(2)
C(3)	2a	0.9394(4)	0.2404(2)	0.6568(4)	0.048(3)	0.046(3)	0.044(3)	0.001(2)	0.020(2)	0.003(2)
C(4)	2a	1.0386(4)	0.2015(2)	0.6406(4)	0.042(3)	0.049(3)	0.063(3)	0.003(2)	0.013(2)	0.001(2)
C(5)	2a	1.1845(4)	0.2186(2)	0.6969(5)	0.053(3)	0.075(4)	0.078(4)	–0.003(3)	0.022(3)	0.005(3)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(6)	2*a*	1.2077(4)	0.2657(2)	0.6332(5)	0.045(3)	0.086(4)	0.069(3)	–0.014(3)	0.019(3)	–0.005(3)
C(7)	2*a*	1.1175(5)	0.3070(2)	0.6507(4)	0.070(4)	0.064(3)	0.046(3)	–0.026(3)	0.021(2)	–0.005(3)
C(8)	2*a*	0.9681(4)	0.2899(2)	0.6041(4)	0.061(3)	0.048(3)	0.037(3)	–0.001(2)	0.015(2)	–0.001(2)
C(9)	2*a*	0.8681(5)	0.3286(2)	0.6175(5)	0.093(4)	0.047(3)	0.067(3)	0.010(3)	0.036(3)	0.001(3)
C(10)	2*a*	0.7241(5)	0.3141(2)	0.5445(5)	0.073(4)	0.077(4)	0.070(4)	0.029(3)	0.021(3)	0.002(3)
C(11)	2*a*	1.0154(5)	0.1504(2)	0.6876(6)	0.057(3)	0.056(4)	0.116(5)	0.005(3)	0.025(3)	0.017(3)
C(12)	2*a*	1.0944(7)	0.1381(2)	0.8274(7)	0.137(6)	0.093(5)	0.136(6)	0.009(4)	0.041(5)	0.065(5)
C(13)	2*a*	1.0434(7)	0.1119(2)	0.5960(7)	0.148(6)	0.049(4)	0.163(7)	0.012(4)	0.025(5)	–0.005(4)
C(14)	2*a*	1.1625(5)	0.3268(2)	0.7882(5)	0.100(4)	0.093(4)	0.058(4)	–0.031(3)	0.022(3)	–0.016(3)
C(15)	2*a*	0.5870(6)	0.2568(2)	0.6276(6)	0.074(4)	0.113(5)	0.141(6)	0.019(4)	0.048(4)	0.013(4)
C(16)	2*a*	0.0430(5)	0.6560(2)	0.8066(5)	0.070(4)	0.046(3)	0.082(4)	–0.003(3)	0.033(3)	0.000(3)
C(17)	2*a*	0.1576(4)	0.6874(2)	0.8016(4)	0.053(3)	0.047(3)	0.069(3)	–0.003(2)	0.024(3)	–0.002(2)
C(18)	2*a*	0.1482(4)	0.7390(2)	0.8522(4)	0.056(3)	0.045(3)	0.043(3)	–0.003(2)	0.016(2)	0.003(2)
C(19)	2*a*	0.2634(4)	0.7719(2)	0.8386(4)	0.055(3)	0.046(3)	0.059(3)	–0.003(2)	0.018(2)	0.004(2)
C(20)	2*a*	0.2408(4)	0.8240(2)	0.8726(5)	0.067(3)	0.048(3)	0.072(3)	–0.012(3)	0.021(3)	0.000(3)
C(21)	2*a*	0.1059(5)	0.8437(2)	0.7861(5)	0.080(4)	0.043(3)	0.070(3)	0.002(3)	0.028(3)	0.003(2)
C(22)	2*a*	–0.0135(5)	0.8141(2)	0.7976(4)	0.061(3)	0.053(3)	0.044(3)	0.013(3)	0.014(2)	0.009(2)
C(23)	2*a*	0.0087(4)	0.7601(2)	0.7738(4)	0.053(3)	0.049(3)	0.048(3)	0.003(2)	0.020(2)	–0.002(2)
C(24)	2*a*	–0.1072(4)	0.7282(2)	0.7852(5)	0.051(3)	0.065(4)	0.088(4)	0.001(3)	0.028(3)	0.007(3)
C(25)	2*a*	–0.0951(5)	0.6765(2)	0.7377(6)	0.063(4)	0.065(4)	0.117(5)	–0.015(3)	0.033(3)	0.003(3)
C(26)	2*a*	0.4091(4)	0.7541(2)	0.9068(5)	0.046(3)	0.058(3)	0.097(4)	–0.003(3)	0.012(3)	0.011(3)
C(27)	2*a*	0.5060(5)	0.7738(2)	0.8386(6)	0.064(4)	0.114(5)	0.149(6)	–0.009(4)	0.040(4)	0.021(4)
C(28)	2*a*	0.4615(6)	0.7647(2)	1.0514(6)	0.074(4)	0.139(6)	0.100(5)	–0.003(4)	–0.013(4)	0.029(4)
C(29)	2*a*	0.0600(6)	0.6135(2)	0.8684(6)	0.090(4)	0.066(4)	0.154(6)	–0.012(3)	0.045(4)	0.015(4)
C(30)	2*a*	–0.0401(5)	0.8239(2)	0.9253(4)	0.088(4)	0.070(3)	0.064(3)	0.023(3)	0.033(3)	0.005(3)
C(31)	2*a*	0.7447(6)	0.4617(2)	0.8220(6)	0.090(4)	0.085(5)	0.083(4)	0.038(4)	0.020(4)	0.010(4)
C(32)	2*a*	0.6861(5)	0.5094(2)	0.7638(4)	0.071(3)	0.064(3)	0.058(3)	0.022(3)	0.016(2)	0.004(3)
C(33)	2*a*	0.5390(5)	0.5162(2)	0.7589(4)	0.070(3)	0.047(3)	0.036(3)	0.004(2)	0.017(2)	–0.002(2)
C(34)	2*a*	0.4777(4)	0.5625(2)	0.6857(4)	0.050(3)	0.049(3)	0.052(3)	0.003(2)	0.014(2)	0.004(2)
C(35)	2*a*	0.3296(4)	0.5667(2)	0.6804(5)	0.057(3)	0.057(3)	0.077(4)	0.001(3)	0.020(3)	–0.006(3)
C(36)	2*a*	0.2488(5)	0.5242(2)	0.6081(5)	0.065(3)	0.071(4)	0.072(3)	–0.007(3)	0.028(3)	0.006(3)
C(37)	2*a*	0.2998(5)	0.4756(2)	0.6660(5)	0.082(4)	0.067(4)	0.055(3)	–0.035(3)	0.029(3)	–0.017(3)
C(38)	2*a*	0.4545(5)	0.4713(2)	0.6924(4)	0.090(4)	0.043(3)	0.046(3)	0.001(3)	0.032(3)	0.001(2)
C(39)	2*a*	0.5152(7)	0.4241(2)	0.7578(5)	0.128(6)	0.056(4)	0.081(4)	0.004(4)	0.029(4)	0.003(3)
C(40)	2*a*	0.6608(8)	0.4184(2)	0.7610(6)	0.147(6)	0.052(4)	0.105(5)	0.043(4)	0.034(5)	0.013(4)
C(41)	2*a*	0.5602(5)	0.6094(2)	0.7272(5)	0.055(3)	0.051(3)	0.086(4)	–0.004(3)	0.015(3)	–0.004(3)
C(42)	2*a*	0.5071(6)	0.6498(2)	0.6276(6)	0.109(5)	0.058(4)	0.122(5)	–0.016(3)	0.015(4)	0.015(4)
C(43)	2*a*	0.5701(6)	0.6276(2)	0.8625(5)	0.111(5)	0.071(4)	0.100(5)	–0.009(3)	0.005(4)	–0.027(3)
C(44)	2*a*	0.2543(5)	0.4649(2)	0.7847(5)	0.106(4)	0.096(4)	0.075(4)	–0.031(4)	0.044(3)	–0.003(3)
C(45)	2*a*	0.8556(7)	0.4588(3)	0.9200(6)	0.130(6)	0.139(6)	0.123(6)	0.071(5)	0.000(5)	0.018(5)
C(46)	2*a*	0.4143(5)	0.5943(2)	0.2321(5)	0.088(4)	0.040(3)	0.077(4)	0.008(3)	0.032(3)	0.003(3)
C(47)	2*a*	0.5292(5)	0.5621(2)	0.3041(5)	0.069(3)	0.041(3)	0.072(3)	–0.002(3)	0.026(3)	0.004(3)
C(48)	2*a*	0.5055(4)	0.5084(2)	0.2649(4)	0.054(3)	0.051(3)	0.039(3)	0.001(2)	0.015(2)	0.005(2)
C(49)	2*a*	0.6212(4)	0.4762(2)	0.3491(4)	0.059(3)	0.040(3)	0.061(3)	0.002(2)	0.020(2)	0.002(2)
C(50)	2*a*	0.5886(5)	0.4227(2)	0.3137(5)	0.063(3)	0.045(3)	0.074(3)	0.002(2)	0.022(3)	0.004(3)
C(51)	2*a*	0.4574(5)	0.4076(2)	0.3360(4)	0.079(4)	0.045(3)	0.065(3)	–0.004(3)	0.021(3)	0.002(2)
C(52)	2*a*	0.3358(5)	0.4375(2)	0.2573(4)	0.067(3)	0.061(3)	0.047(3)	–0.016(3)	0.023(3)	–0.003(2)
C(53)	2*a*	0.3681(4)	0.4920(2)	0.2800(4)	0.059(3)	0.050(3)	0.044(3)	0.000(2)	0.020(2)	0.003(2)
C(54)	2*a*	0.2524(4)	0.5250(2)	0.2020(5)	0.057(3)	0.076(4)	0.074(3)	0.005(3)	0.026(3)	0.014(3)
C(55)	2*a*	0.2794(5)	0.5778(2)	0.2421(5)	0.076(4)	0.062(4)	0.094(4)	0.024(3)	0.034(3)	0.014(3)
C(56)	2*a*	0.7663(5)	0.4911(2)	0.3589(5)	0.052(3)	0.063(3)	0.089(4)	0.001(3)	0.020(3)	0.005(3)
C(57)	2*a*	0.8682(5)	0.4640(2)	0.4681(6)	0.065(4)	0.120(5)	0.139(6)	0.013(4)	–0.004(4)	0.031(5)
C(58)	2*a*	0.8023(5)	0.4863(2)	0.2337(6)	0.073(4)	0.103(5)	0.134(6)	0.001(3)	0.055(4)	0.004(4)
C(59)	2*a*	0.2937(5)	0.4225(2)	0.1144(4)	0.085(4)	0.077(4)	0.055(3)	–0.029(3)	0.024(3)	–0.009(3)
C(60)	2*a*	0.4280(6)	0.6320(2)	0.1631(5)	0.097(4)	0.058(3)	0.112(5)	0.010(3)	0.032(4)	0.030(3)

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