Crystal structure of 3*S*,4′*S*,6′*S*-2-benzyl-3-[(*tert*-butyldimethylsiloxy)-(2′,2′-dimethyl-1,3-dioxolan-4′-yl)-6′-methyl]-4-[2-(trimethylsilyl)ethoxy]-3.6-dihydro-2*H*-1,2-oxazine, $C_{28}H_{49}NO_5Si_2$

A. Al-Harrasi, I. Brüdgam, H. Hartl and H.-U. Reißig*

Freie Universität Berlin, Institut für Chemie und Biochemie, Takustr. 3, 14195 Berlin, Germany

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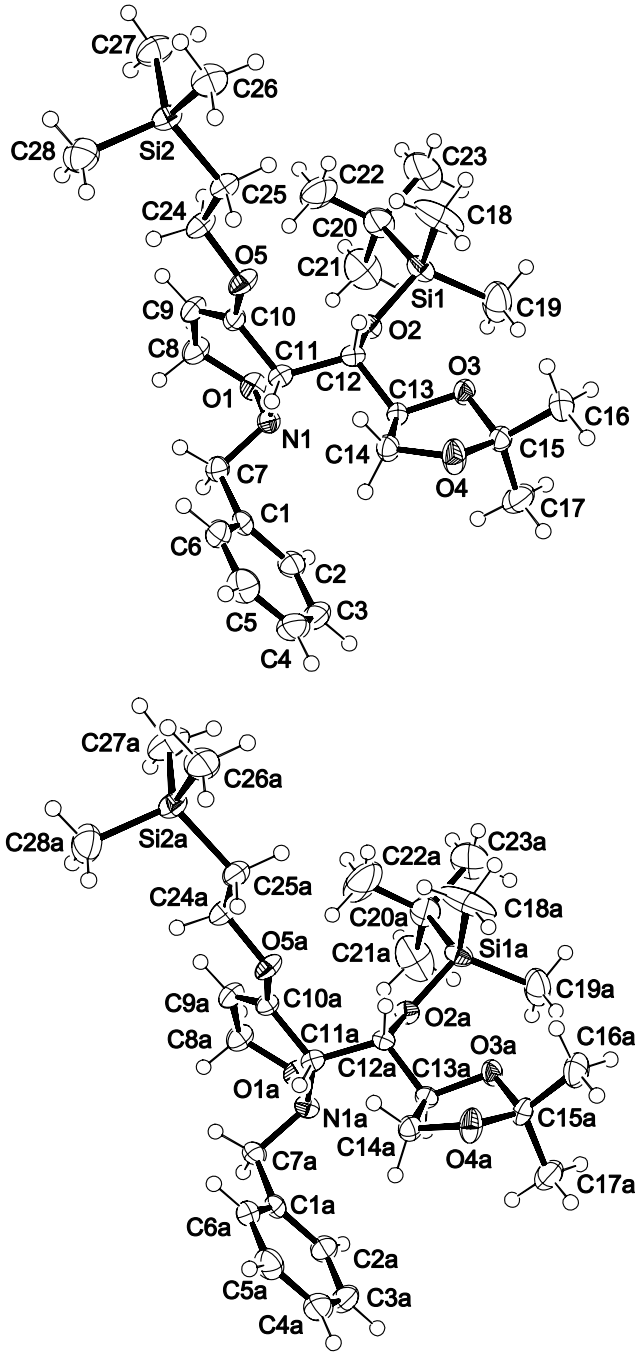

* Correspondence author (e-mail: hans.reissig@chemie.fu-berlin.de)

Abstract

$C_{28}H_{49}NO_5Si_2$, monoclinic, $P12_11$ (no. 4), a = 14.325(3) Å, b = 11.850(2) Å, c = 19.459(4) Å, β = 102.978(5)°, V = 3218.8 Å^3, Z = 4, $R_{gt}(F)$ = 0.046, $wR_{ref}(F^2)$ = 0.103, T = 173 K.

Source of material

The title compound was obtained by [3+3] cyclization of lithiated 2-(trimethylsilylethoxy)allene with L-therose derived nitrone as described in [1,2], purified by chromatography on silica gel and recrystallized from hexane (m.p. 366-368 K).

Discussion

The crystal structure proves the constitution and configuration of the title compound and also shows details of the conformation. It proves the newly formed stereogenic center at C11 (C11a). Bond distances and bond angles show normal values. The crystal structure contains two crystallographically independent molecules with marginal differences of the bond lengths and torsion angles. The absolute structure was confirmed by the value of Flack parameter of 0.01(8).

Table 1. Data collection and handling.

Crystal:	colorless plate, size 0.05 × 0.13 × 0.65 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.43 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	55.16°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	22282, 14174
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 8416
$N(param)_{refined}$:	669
Programs:	SHELXS-97 [3], SHELXL-97 [4], ORTEP-3 [5]

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

Atom	Site	x	y	z	U_{iso}
H(26A)	2*a*	0.7747	0.0380	0.0367	0.062
H(26B)	2*a*	0.6771	0.0269	0.0632	0.062
H(26C)	2*a*	0.7640	–0.0577	0.0924	0.062
H(27A)	2*a*	0.9437	0.0259	0.1929	0.068
H(27B)	2*a*	0.9507	0.1531	0.2211	0.068
H(27C)	2*a*	0.9573	0.1272	0.1416	0.068
H(28A)	2*a*	0.6703	0.0960	0.2126	0.074
H(28B)	2*a*	0.7643	0.1440	0.2650	0.074
H(28C)	2*a*	0.7572	0.0129	0.2448	0.074
H(25A)	2*a*	0.7904	0.2778	0.0760	0.034
H(25B)	2*a*	0.6899	0.2651	0.0974	0.034

Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(24A)	2*a*	0.7677	0.3437	0.2121	0.038
H(24B)	2*a*	0.8553	0.3763	0.1768	0.038
H(9)	2*a*	0.8480	0.5223	0.2546	0.036
H(8A)	2*a*	0.7994	0.7119	0.2938	0.039
H(8B)	2*a*	0.8994	0.7137	0.2702	0.039
H(11)	2*a*	0.6207	0.6218	0.1228	0.026
H(7A)	2*a*	0.6535	0.8277	0.2485	0.036
H(7B)	2*a*	0.6373	0.6938	0.2428	0.036
H(6)	2*a*	0.4759	0.6343	0.2025	0.041
H(5)	2*a*	0.3178	0.6655	0.1421	0.052
H(4)	2*a*	0.2759	0.8336	0.0806	0.049
H(3)	2*a*	0.3917	0.9713	0.0812	0.044
H(2)	2*a*	0.5496	0.9398	0.1402	0.038
H(12)	2*a*	0.6995	0.5865	0.0300	0.025
H(13)	2*a*	0.6220	0.8115	0.0286	0.026
H(14A)	2*a*	0.5248	0.6013	0.0112	0.033
H(14B)	2*a*	0.4816	0.7226	0.0248	0.033
H(16A)	2*a*	0.5890	0.7569	–0.1989	0.057
H(16B)	2*a*	0.4779	0.7265	–0.2092	0.057
H(16C)	2*a*	0.5579	0.6353	–0.1761	0.057
H(17A)	2*a*	0.5158	0.9065	–0.0583	0.072
H(17B)	2*a*	0.4529	0.8940	–0.1370	0.072
H(17C)	2*a*	0.5636	0.9281	–0.1240	0.072
H(19A)	2*a*	0.7894	0.8142	–0.0926	0.091
H(19B)	2*a*	0.9001	0.8004	–0.0937	0.091
H(19C)	2*a*	0.8690	0.8838	–0.0381	0.091
H(18A)	2*a*	0.8432	0.4967	–0.0124	0.102
H(18B)	2*a*	0.8903	0.5447	–0.0737	0.102
H(18C)	2*a*	0.7780	0.5605	–0.0786	0.102
H(22A)	2*a*	0.9905	0.5277	0.0865	0.100
H(22B)	2*a*	0.9516	0.6040	0.1419	0.100
H(22C)	2*a*	1.0638	0.6001	0.1439	0.100
H(21A)	2*a*	0.9499	0.8096	0.1337	0.093
H(21B)	2*a*	0.9846	0.8723	0.0710	0.093
H(21C)	2*a*	1.0615	0.8151	0.1337	0.093
H(23A)	2*a*	1.1292	0.6947	0.0515	0.100
H(23B)	2*a*	1.0585	0.7553	–0.0133	0.100
H(23C)	2*a*	1.0595	0.6206	–0.0072	0.100
H(25C)	2*a*	0.7848	0.0280	0.5710	0.036
H(25D)	2*a*	0.6861	0.0133	0.5950	0.036
H(17D)	2*a*	0.5031	0.6434	0.4221	0.064
H(17E)	2*a*	0.4503	0.6199	0.3418	0.064
H(17F)	2*a*	0.5571	0.6690	0.3601	0.064
H(16D)	2*a*	0.6073	0.4996	0.2937	0.060

Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(16E)	2*a*	0.5016	0.4476	0.2797	0.060
H(16F)	2*a*	0.5912	0.3751	0.3208	0.060
H(14C)	2*a*	0.5253	0.3537	0.5060	0.032
H(14D)	2*a*	0.4762	0.4760	0.5057	0.032
H(13A)	2*a*	0.6181	0.5629	0.5206	0.026
H(12A)	2*a*	0.7014	0.3410	0.5233	0.022
H(24C)	2*a*	0.7623	0.0992	0.7062	0.038
H(24D)	2*a*	0.8515	0.1279	0.6718	0.038
H(11A)	2*a*	0.6173	0.3722	0.6136	0.025
H(8A1)	2*a*	0.7874	0.4601	0.7884	0.038
H(8A2)	2*a*	0.8896	0.4659	0.7681	0.038
H(9A)	2*a*	0.8423	0.2734	0.7486	0.035
H(22D)	2*a*	1.0625	0.3649	0.6450	0.152
H(22E)	2*a*	0.9924	0.2912	0.5863	0.152
H(22F)	2*a*	0.9496	0.3657	0.6405	0.152
H(21D)	2*a*	0.9444	0.5698	0.6333	0.112
H(21E)	2*a*	0.9821	0.6354	0.5729	0.112
H(21F)	2*a*	1.0568	0.5774	0.6366	0.112
H(19D)	2*a*	0.7950	0.5696	0.4024	0.080
H(19E)	2*a*	0.9083	0.5650	0.4089	0.080
H(19F)	2*a*	0.8650	0.6442	0.4607	0.080
H(18D)	2*a*	0.8472	0.2567	0.4837	0.120
H(18E)	2*a*	0.8987	0.3065	0.4253	0.120
H(18F)	2*a*	0.7855	0.3210	0.4162	0.120
H(2A)	2*a*	0.5418	0.6913	0.6380	0.038
H(7A1)	2*a*	0.6416	0.5711	0.7421	0.033
H(7A2)	2*a*	0.6269	0.4374	0.7324	0.033
H(5A)	2*a*	0.3143	0.4034	0.6162	0.045
H(4A)	2*a*	0.2719	0.5784	0.5635	0.045
H(6A)	2*a*	0.4703	0.3740	0.6815	0.037
H(27D)	2*a*	0.9506	–0.2064	0.6984	0.087
H(27E)	2*a*	0.9474	–0.0788	0.7247	0.087
H(27F)	2*a*	0.9610	–0.1044	0.6468	0.087
H(28D)	2*a*	0.6682	–0.1501	0.7074	0.081
H(28E)	2*a*	0.7609	–0.1051	0.7626	0.081
H(28F)	2*a*	0.7530	–0.2354	0.7406	0.081
H(26D)	2*a*	0.7726	–0.3067	0.5958	0.067
H(26E)	2*a*	0.7888	–0.2142	0.5396	0.067
H(26F)	2*a*	0.6871	–0.2237	0.5604	0.067
H(3A)	2*a*	0.3849	0.7217	0.5751	0.046
H(23D)	2*a*	1.1306	0.4607	0.5557	0.106
H(23E)	2*a*	1.0625	0.5236	0.4905	0.106
H(23F)	2*a*	1.0638	0.3886	0.4943	0.106

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}
Si(1)	2*a*	0.86556(6)	0.69446(7)	0.00109(4)	0.0221(5)	0.0404(6)	0.0285(5)	–0.0061(4)	0.0075(4)	–0.0099(4)
Si(2)	2*a*	0.79462(6)	0.11889(6)	0.15080(5)	0.0280(5)	0.0212(5)	0.0375(5)	0.0021(4)	0.0031(4)	0.0018(4)
O(1)	2*a*	0.7918(1)	0.7808(2)	0.1998(1)	0.030(1)	0.025(1)	0.029(1)	–0.0054(9)	0.0009(9)	–0.0025(9)
O(2)	2*a*	0.7915(1)	0.7121(2)	0.05369(9)	0.020(1)	0.029(1)	0.024(1)	–0.0027(8)	0.0040(8)	–0.0030(9)
O(3)	2*a*	0.6353(1)	0.7405(2)	–0.06208(9)	0.021(1)	0.040(1)	0.021(1)	0.0026(9)	0.0033(8)	0.0062(9)
O(4)	2*a*	0.4804(1)	0.6923(2)	–0.0759(1)	0.025(1)	0.066(2)	0.023(1)	–0.007(1)	0.0004(9)	0.007(1)
O(5)	2*a*	0.7332(1)	0.4590(2)	0.1346(1)	0.033(1)	0.018(1)	0.033(1)	0.0026(9)	–0.0050(9)	0.0003(9)
N(1)	2*a*	0.6937(2)	0.7581(2)	0.1644(1)	0.024(1)	0.024(1)	0.022(1)	0.001(1)	0.003(1)	–0.002(1)
C(26)	2*a*	0.7470(2)	0.0198(3)	0.0771(2)	0.043(2)	0.030(2)	0.048(2)	–0.005(2)	0.007(2)	–0.005(2)
C(27)	2*a*	0.9273(2)	0.1046(3)	0.1801(2)	0.035(2)	0.045(2)	0.050(2)	0.008(2)	–0.005(2)	0.001(2)
C(28)	2*a*	0.7401(3)	0.0894(3)	0.2274(2)	0.054(2)	0.037(2)	0.059(2)	–0.000(2)	0.016(2)	0.004(2)
C(25)	2*a*	0.7601(2)	0.2641(2)	0.1162(2)	0.027(2)	0.024(2)	0.034(2)	0.002(1)	0.002(1)	0.003(1)
C(24)	2*a*	0.7855(2)	0.3615(2)	0.1670(2)	0.033(2)	0.022(2)	0.037(2)	0.004(1)	–0.002(1)	0.005(1)
C(10)	2*a*	0.7514(2)	0.5595(2)	0.1682(1)	0.026(2)	0.021(2)	0.023(2)	0.002(1)	0.005(1)	0.002(1)
C(9)	2*a*	0.8135(2)	0.5821(2)	0.2278(2)	0.033(2)	0.027(2)	0.027(2)	0.004(1)	–0.002(1)	0.004(1)
C(8)	2*a*	0.8298(2)	0.7006(2)	0.2534(2)	0.034(2)	0.029(2)	0.028(2)	0.001(1)	–0.004(1)	–0.003(1)
C(11)	2*a*	0.6879(2)	0.6491(2)	0.1274(1)	0.021(1)	0.017(1)	0.025(2)	–0.002(1)	0.001(1)	–0.001(1)
C(7)	2*a*	0.6321(2)	0.7647(3)	0.2153(1)	0.035(2)	0.032(2)	0.022(2)	0.006(1)	0.004(1)	–0.001(1)
C(1)	2*a*	0.5294(2)	0.7832(2)	0.1771(1)	0.033(2)	0.030(2)	0.018(2)	0.003(1)	0.008(1)	–0.003(1)
C(6)	2*a*	0.4594(2)	0.7029(3)	0.1775(2)	0.043(2)	0.029(2)	0.033(2)	0.005(2)	0.013(2)	0.006(2)

Table 3. Continued.

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(5)	2*a*	0.3651(2)	0.7215(3)	0.1416(2)	0.038(2)	0.045(2)	0.047(2)	–0.009(2)	0.011(2)	–0.000(2)
C(4)	2*a*	0.3402(2)	0.8212(3)	0.1054(2)	0.030(2)	0.047(2)	0.044(2)	0.007(2)	0.005(2)	0.001(2)
C(3)	2*a*	0.4088(2)	0.9021(3)	0.1055(2)	0.044(2)	0.028(2)	0.036(2)	0.009(2)	0.004(2)	0.004(2)
C(2)	2*a*	0.5027(2)	0.8834(2)	0.1408(2)	0.038(2)	0.026(2)	0.034(2)	0.001(1)	0.012(2)	–0.001(1)
C(12)	2*a*	0.7012(2)	0.6629(2)	0.0520(1)	0.019(1)	0.019(1)	0.024(2)	–0.001(1)	0.002(1)	–0.004(1)
C(13)	2*a*	0.6215(2)	0.7341(2)	0.0080(1)	0.023(1)	0.023(2)	0.019(1)	0.001(1)	0.005(1)	0.002(1)
C(14)	2*a*	0.5207(2)	0.6817(3)	–0.0031(1)	0.023(1)	0.037(2)	0.022(2)	–0.001(1)	0.004(1)	0.003(1)
C(15)	2*a*	0.5435(2)	0.7600(2)	–0.1064(1)	0.023(2)	0.034(2)	0.022(2)	0.002(1)	0.003(1)	0.003(1)
C(16)	2*a*	0.5419(2)	0.7158(3)	–0.1789(2)	0.044(2)	0.046(2)	0.025(2)	0.002(2)	0.009(2)	0.003(2)
C(17)	2*a*	0.5166(3)	0.8829(3)	–0.1065(2)	0.058(2)	0.039(2)	0.044(2)	0.019(2)	0.004(2)	0.006(2)
C(19)	2*a*	0.8547(3)	0.8122(4)	–0.0635(2)	0.043(2)	0.095(3)	0.045(2)	–0.006(2)	0.013(2)	0.019(2)
C(18)	2*a*	0.8414(3)	0.5580(3)	–0.0465(2)	0.044(2)	0.083(3)	0.085(3)	–0.016(2)	0.031(2)	–0.054(3)
C(20)	2*a*	0.9878(2)	0.6973(3)	0.0623(2)	0.023(2)	0.062(2)	0.039(2)	–0.001(2)	0.004(1)	–0.005(2)
C(22)	2*a*	0.9994(3)	0.5985(4)	0.1132(2)	0.039(2)	0.086(3)	0.073(3)	0.023(2)	0.008(2)	0.024(3)
C(21)	2*a*	0.9968(3)	0.8087(3)	0.1040(2)	0.043(2)	0.087(3)	0.049(2)	–0.021(2)	–0.003(2)	–0.023(2)
C(23)	2*a*	1.0659(2)	0.6915(4)	0.0194(2)	0.022(2)	0.118(4)	0.061(3)	–0.004(2)	0.012(2)	–0.006(3)
Si(1A)	2*a*	0.86744(6)	0.45459(7)	0.49913(4)	0.0232(5)	0.0299(5)	0.0364(5)	–0.0027(4)	0.0123(4)	–0.0050(4)
Si(2A)	2*a*	0.79669(6)	–0.12738(7)	0.65017(5)	0.0351(5)	0.0213(5)	0.0383(5)	0.0034(4)	0.0066(4)	0.0045(4)
O(4A)	2*a*	0.4912(1)	0.4256(2)	0.4107(1)	0.031(1)	0.044(1)	0.020(1)	–0.010(1)	–0.0034(9)	0.005(1)
O(3A)	2*a*	0.6410(1)	0.4947(2)	0.43111(9)	0.021(1)	0.040(1)	0.018(1)	0.0003(9)	0.0026(8)	0.0038(9)
O(2A)	2*a*	0.7903(1)	0.4695(2)	0.54954(9)	0.019(1)	0.024(1)	0.027(1)	–0.0032(8)	0.0042(8)	–0.0012(9)
O(1A)	2*a*	0.7837(1)	0.5326(2)	0.6958(1)	0.025(1)	0.026(1)	0.029(1)	–0.0041(9)	–0.0026(9)	–0.0044(9)
O(5A)	2*a*	0.7314(1)	0.2113(1)	0.6272(1)	0.033(1)	0.016(1)	0.029(1)	0.0029(8)	–0.0033(9)	0.0023(9)
N(1A)	2*a*	0.6872(2)	0.5090(2)	0.6580(1)	0.022(1)	0.025(1)	0.023(1)	–0.001(1)	0.004(1)	–0.005(1)
C(25A)	2*a*	0.7566(2)	0.0151(2)	0.6123(2)	0.032(2)	0.025(2)	0.031(2)	0.005(1)	0.004(1)	0.003(1)
C(17A)	2*a*	0.5120(2)	0.6184(3)	0.3760(2)	0.051(2)	0.037(2)	0.038(2)	0.013(2)	0.005(2)	0.009(2)
C(15A)	2*a*	0.5516(2)	0.4992(2)	0.3821(1)	0.028(2)	0.030(2)	0.020(2)	0.001(1)	–0.001(1)	0.006(1)
C(16A)	2*a*	0.5640(2)	0.4512(3)	0.3131(1)	0.055(2)	0.039(2)	0.023(2)	–0.001(2)	0.002(2)	0.001(2)
C(14A)	2*a*	0.5214(2)	0.4304(2)	0.4854(1)	0.021(1)	0.034(2)	0.023(2)	–0.000(1)	0.002(1)	0.003(1)
C(13A)	2*a*	0.6208(2)	0.4861(2)	0.4999(1)	0.023(1)	0.021(1)	0.022(2)	0.000(1)	0.006(1)	–0.002(1)
C(12A)	2*a*	0.7003(2)	0.4170(2)	0.5455(1)	0.018(1)	0.015(1)	0.023(1)	–0.003(1)	0.006(1)	–0.003(1)
C(24A)	2*a*	0.7815(2)	0.1143(2)	0.6613(2)	0.033(2)	0.021(2)	0.036(2)	0.003(1)	0.000(1)	0.005(1)
C(10A)	2*a*	0.7477(2)	0.3122(2)	0.6611(1)	0.024(2)	0.018(2)	0.024(2)	0.001(1)	0.004(1)	–0.000(1)
C(11A)	2*a*	0.6843(2)	0.4010(2)	0.6198(1)	0.019(1)	0.017(1)	0.026(2)	–0.002(1)	0.003(1)	–0.002(1)
C(8A)	2*a*	0.8205(2)	0.4514(2)	0.7492(2)	0.030(2)	0.032(2)	0.028(2)	0.001(1)	–0.003(1)	–0.000(1)
C(9A)	2*a*	0.8076(2)	0.3333(2)	0.7221(1)	0.032(2)	0.025(2)	0.027(2)	0.003(1)	–0.001(1)	0.002(1)
C(22A)	2*a*	0.9990(3)	0.3619(4)	0.6130(3)	0.041(2)	0.132(5)	0.133(4)	0.034(3)	0.023(3)	0.084(4)
C(20A)	2*a*	0.9876(2)	0.4609(3)	0.5624(2)	0.019(2)	0.070(3)	0.056(2)	0.004(2)	0.011(2)	0.019(2)
C(21A)	2*a*	0.9932(3)	0.5708(4)	0.6052(2)	0.043(2)	0.125(4)	0.052(2)	–0.030(3)	0.002(2)	–0.017(3)
C(19A)	2*a*	0.8578(2)	0.5722(3)	0.4353(2)	0.041(2)	0.079(3)	0.043(2)	–0.006(2)	0.014(2)	0.017(2)
C(18A)	2*a*	0.8473(3)	0.3187(3)	0.4503(2)	0.061(3)	0.064(3)	0.133(4)	–0.020(2)	0.063(3)	–0.060(3)
C(2A)	2*a*	0.4956(2)	0.6327(2)	0.6342(2)	0.034(2)	0.027(2)	0.033(2)	0.000(1)	0.009(1)	0.000(1)
C(7A)	2*a*	0.6225(2)	0.5102(2)	0.7069(1)	0.030(2)	0.031(2)	0.022(2)	0.005(1)	0.008(1)	–0.003(1)
C(1A)	2*a*	0.5214(2)	0.5291(2)	0.6664(1)	0.033(2)	0.027(2)	0.018(2)	0.004(1)	0.010(1)	–0.001(1)
C(5A)	2*a*	0.3603(2)	0.4622(3)	0.6210(2)	0.030(2)	0.047(2)	0.039(2)	–0.006(2)	0.011(2)	0.002(2)
C(4A)	2*a*	0.3354(2)	0.5657(3)	0.5897(2)	0.031(2)	0.047(2)	0.033(2)	0.006(2)	0.005(1)	0.001(2)
C(6A)	2*a*	0.4533(2)	0.4450(3)	0.6595(2)	0.032(2)	0.032(2)	0.031(2)	0.005(1)	0.014(1)	0.007(1)
C(27A)	2*a*	0.9297(3)	–0.1295(3)	0.6840(2)	0.046(2)	0.046(2)	0.073(3)	0.012(2)	–0.006(2)	0.007(2)
C(28A)	2*a*	0.7377(3)	–0.1581(3)	0.7239(2)	0.074(3)	0.042(2)	0.049(2)	–0.003(2)	0.021(2)	0.009(2)
C(26A)	2*a*	0.7566(2)	–0.2301(3)	0.5780(2)	0.055(2)	0.030(2)	0.053(2)	–0.001(2)	0.018(2)	–0.004(2)
C(3A)	2*a*	0.4025(2)	0.6504(3)	0.5965(2)	0.045(2)	0.031(2)	0.038(2)	0.010(2)	0.007(2)	0.008(2)
C(23A)	2*a*	1.0685(2)	0.4582(4)	0.5221(2)	0.022(2)	0.110(4)	0.081(3)	0.002(2)	0.015(2)	0.013(3)

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