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Crystal structure of tetrakis[(4-methyl-pyridin-2-yl)-trimethylsilanyl-amido]zirconium(IV), $Zr(C_9H_{15}N_2Si)_4$

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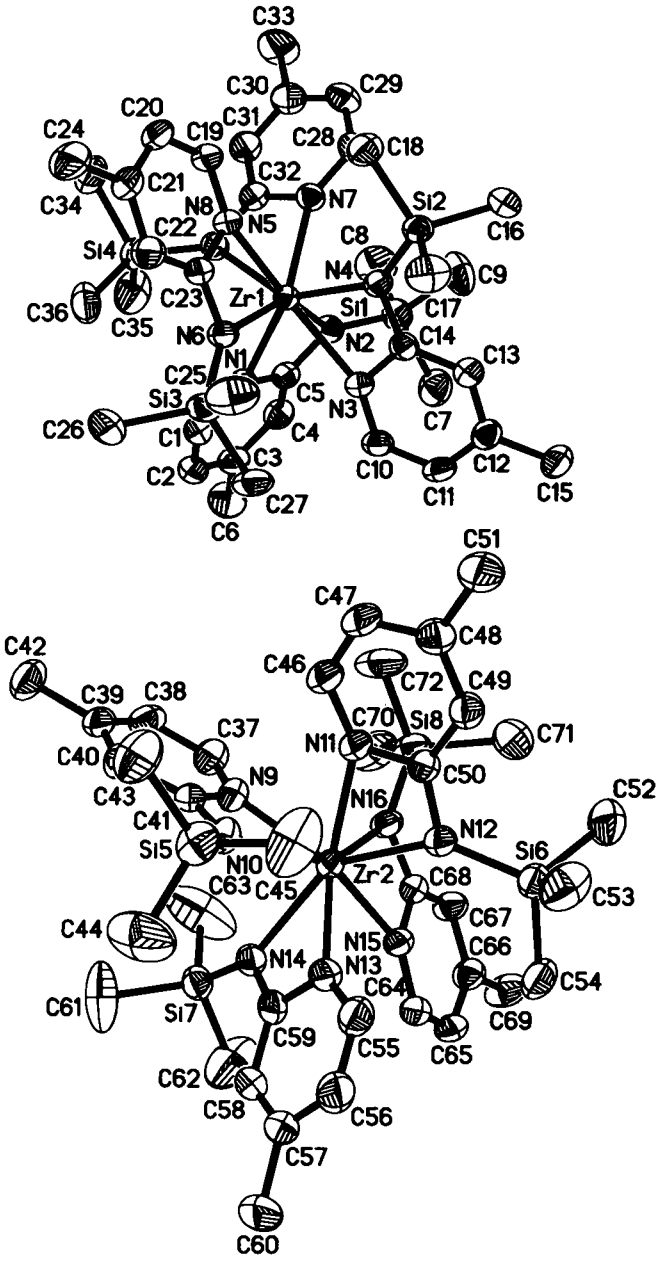

Abstract

$C_{36}H_{60}N_8Si_4Zr$, triclinic, $P\bar{1}$ (no. 2), a = 12.0360(6) Å, b = 18.8040(9) Å, c = 21.581(1) Å, α = 69.448(4)°, β = 75.278(4)°, γ = 88.159(4)°, V = 4414.5 Å^3, Z = 4, $R_{gt}(F)$ = 0.035, $wR_{ref}(F^2)$ = 0.101, T = 191 K.

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Source of material

(4-Methyl-pyridin-2-yl)-trimethylsilanyl-amine (1.44 g, 8 mmol) in ether (5 ml) was added dropwise to $[((C_2H_5)_2N)_4Zr]$ (0.67 g, 2 mmol) in ether (5 ml) at room temperature as color started to change from brown yellow to bright yellow. The reaction mixture was further stirred over night. Volume of the solvent was reduced and allowed to stand at room temperature to afford yellow crystals suitable for X-ray analysis (yield 1.34 g, 83 %).

Discussion

Group 4 metal chemistry of the aminopyridinato ligands [1,2] has been studied extensively. The title compound, an eight-coordinated zirconium complex, is stabilized by four aminopyridinato ligands. The bulky trimethylsilyl substituents at the amido N atoms force the aminopyridinato ligands into a *transoid* arrangement. The chelating Zr-N_{amido}-C-$N_{pyridine}$ planes are planar. The averaged deviation of all eight planes is 0.018 Å. The bond lengths for Zr1—N_{amido} (average 2.271 Å) and Zr2—N_{amido} (average 2.286 Å) are longer and for Zr1—$N_{pyridine}$ (average 2.309 Å) and Zr2—$N_{pyridine}$ (average 2.300 Å) are shorter than those found in [3] for such high coordination number complexes with arylamino-pyridinato ligands and indicate a delocalized bonding mode. The small chelating angles of N_{amido}–Zr1–$N_{pyridine}$ (average 58.69°) and N_{amido}–Zr2–$N_{pyridine}$ (average 58.43°) underline the strained nature of the aminopyridinato coordination.

Table 1. Data collection and handling.

Crystal:	yellow prism, size 0.35 × 0.4 × 1.1 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	3.91 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS II, ω
$2\theta_{max}$:	51.6°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	15600, 15600
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 13181
$N(param)_{refined}$:	884
Programs:	SIR97 [4], SHELXL-97 [5]

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

Atom	Site	x	y	z	U_{iso}
H(1)	2*i*	0.4035	0.1316	0.2642	0.045
H(2)	2*i*	0.4561	0.0568	0.1983	0.052
H(4)	2*i*	0.7748	0.1618	0.1262	0.048
H(6A)	2*i*	0.5894	0.0225	0.1153	0.093
H(6B)	2*i*	0.7189	0.0245	0.1216	0.093
H(6C)	2*i*	0.6805	0.0895	0.0611	0.093
H(7A)	2*i*	0.9269	0.1352	0.2384	0.090
H(7B)	2*i*	1.0350	0.1824	0.1787	0.090

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(7C)	2*i*	0.9321	0.1491	0.1602	0.090
H(8A)	2*i*	0.8969	0.3113	0.0640	0.104
H(8B)	2*i*	1.0062	0.3397	0.0806	0.104
H(8C)	2*i*	0.8874	0.3798	0.0925	0.104
H(9A)	2*i*	0.9011	0.2640	0.2890	0.098
H(9B)	2*i*	0.8900	0.3488	0.2396	0.098
H(9C)	2*i*	1.0088	0.3087	0.2275	0.098
H(10)	2*i*	0.6636	0.0798	0.3232	0.044
H(11)	2*i*	0.7846	0.0181	0.3899	0.046
H(13)	2*i*	0.7801	0.1961	0.4541	0.044
H(15A)	2*i*	0.8930	0.0140	0.4682	0.076
H(15B)	2*i*	0.8374	0.0557	0.5210	0.076
H(15C)	2*i*	0.9460	0.0956	0.4580	0.076
H(16A)	2*i*	0.8512	0.3097	0.4266	0.076
H(16B)	2*i*	0.8118	0.3832	0.4456	0.076
H(16C)	2*i*	0.8465	0.3890	0.3673	0.076
H(17A)	2*i*	0.6216	0.2246	0.5267	0.085
H(17B)	2*i*	0.5027	0.2616	0.5169	0.085
H(17C)	2*i*	0.5893	0.3008	0.5425	0.085
H(18A)	2*i*	0.5012	0.4084	0.4021	0.072
H(18B)	2*i*	0.6193	0.4530	0.3515	0.072
H(18C)	2*i*	0.5850	0.4468	0.4300	0.072
H(19)	2*i*	0.4753	0.4728	0.2628	0.046
H(20)	2*i*	0.2956	0.5078	0.3080	0.051
H(22)	2*i*	0.1971	0.2848	0.4105	0.053
H(24A)	2*i*	0.1057	0.4743	0.3807	0.102
H(24B)	2*i*	0.0949	0.3990	0.4469	0.102
H(24C)	2*i*	0.0516	0.3962	0.3835	0.102
H(25A)	2*i*	0.2466	0.2037	0.5025	0.100
H(25B)	2*i*	0.3616	0.1628	0.5154	0.100
H(25C)	2*i*	0.2441	0.1129	0.5353	0.100
H(26A)	2*i*	0.1468	0.1863	0.3881	0.100
H(26B)	2*i*	0.1450	0.0962	0.4261	0.100
H(26C)	2*i*	0.2107	0.1343	0.3467	0.100
H(27A)	2*i*	0.4297	0.0616	0.3754	0.076
H(27B)	2*i*	0.3606	0.0222	0.4540	0.076
H(27C)	2*i*	0.4781	0.0721	0.4344	0.076
H(28)	2*i*	0.7746	0.4432	0.2545	0.048
H(29)	2*i*	0.8230	0.5558	0.1623	0.056
H(31)	2*i*	0.6252	0.4810	0.0689	0.051
H(33A)	2*i*	0.8184	0.6329	0.0493	0.106
H(33B)	2*i*	0.6968	0.6280	0.0333	0.106
H(33C)	2*i*	0.8029	0.5862	0.0024	0.106
H(34A)	2*i*	0.3568	0.4690	0.1379	0.090
H(34B)	2*i*	0.3292	0.4476	0.0776	0.090
H(34C)	2*i*	0.4531	0.4852	0.0672	0.090
H(35A)	2*i*	0.5706	0.3496	0.0335	0.113
H(35B)	2*i*	0.4445	0.3188	0.0396	0.113
H(35C)	2*i*	0.5235	0.2650	0.0838	0.113
H(36A)	2*i*	0.2800	0.3134	0.2322	0.103
H(36B)	2*i*	0.3310	0.2411	0.2145	0.103
H(36C)	2*i*	0.2527	0.2956	0.1701	0.103
H(37)	2*i*	0.9889	0.3501	0.5916	0.050
H(38)	2*i*	0.8674	0.4260	0.5334	0.053
H(40)	2*i*	0.6005	0.3325	0.6998	0.048
H(42A)	2*i*	0.6833	0.4674	0.5274	0.088

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(42B)	2*i*	0.6045	0.4698	0.5985	0.088
H(42C)	2*i*	0.5797	0.4044	0.5713	0.088
H(43A)	2*i*	0.5736	0.3632	0.8269	0.102
H(43B)	2*i*	0.4626	0.3083	0.8749	0.102
H(43C)	2*i*	0.4961	0.3270	0.7939	0.102
H(44A)	2*i*	0.5776	0.1057	0.8622	0.159
H(44B)	2*i*	0.5001	0.1587	0.8163	0.159
H(44C)	2*i*	0.4652	0.1392	0.8974	0.159
H(45A)	2*i*	0.7096	0.2679	0.9173	0.133
H(45B)	2*i*	0.7099	0.1779	0.9345	0.133
H(45C)	2*i*	0.5955	0.2148	0.9629	0.133
H(46)	2*i*	0.8491	0.4149	0.7372	0.054
H(47)	2*i*	0.8461	0.4897	0.8024	0.058
H(49)	2*i*	0.9874	0.3211	0.9295	0.048
H(51A)	2*i*	0.8853	0.5098	0.8960	0.091
H(51B)	2*i*	0.9956	0.4699	0.9180	0.091
H(51C)	2*i*	0.8690	0.4346	0.9628	0.091
H(52A)	2*i*	1.1851	0.2798	0.9005	0.101
H(52B)	2*i*	1.2464	0.2330	0.8534	0.101
H(52C)	2*i*	1.2414	0.2019	0.9333	0.101
H(53A)	2*i*	0.9578	0.2099	1.0119	0.098
H(53B)	2*i*	1.0247	0.1351	1.0394	0.098
H(53C)	2*i*	0.9071	0.1288	1.0195	0.098
H(54A)	2*i*	1.1396	0.0852	0.8682	0.081
H(54B)	2*i*	1.0244	0.0485	0.9271	0.081
H(54C)	2*i*	1.1419	0.0555	0.9470	0.081
H(55)	2*i*	0.8173	0.0765	0.9456	0.055
H(56)	2*i*	0.7341	−0.0436	0.9815	0.072
H(58)	2*i*	0.7623	−0.0182	0.7844	0.061
H(60A)	2*i*	0.6754	−0.1482	0.9590	0.145
H(60B)	2*i*	0.7492	−0.1552	0.8886	0.145
H(60C)	2*i*	0.6222	−0.1254	0.8942	0.145
H(61A)	2*i*	0.6825	0.1501	0.6491	0.143
H(61B)	2*i*	0.6788	0.0594	0.6836	0.143
H(61C)	2*i*	0.7287	0.0998	0.6026	0.143
H(62A)	2*i*	1.0206	0.0286	0.6623	0.161
H(62B)	2*i*	0.9494	0.0191	0.6121	0.161
H(62C)	2*i*	0.8999	−0.0202	0.6933	0.161
H(63A)	2*i*	1.0315	0.1936	0.5902	0.167
H(63B)	2*i*	0.9172	0.2371	0.5805	0.167
H(63C)	2*i*	0.9601	0.1780	0.5425	0.167
H(64)	2*i*	1.0436	0.0224	0.8001	0.042
H(65)	2*i*	1.2227	−0.0111	0.7538	0.045
H(67)	2*i*	1.3194	0.2120	0.6533	0.048
H(69A)	2*i*	1.4132	0.0230	0.6840	0.084
H(69B)	2*i*	1.4669	0.1012	0.6811	0.084
H(69C)	2*i*	1.4237	0.0982	0.6177	0.084
H(70A)	2*i*	1.2974	0.3089	0.5624	0.101
H(70B)	2*i*	1.3103	0.3991	0.5426	0.101
H(70C)	2*i*	1.1932	0.3599	0.5440	0.101
H(71A)	2*i*	1.3642	0.2962	0.6973	0.107
H(71B)	2*i*	1.2903	0.3393	0.7446	0.107
H(71C)	2*i*	1.3711	0.3872	0.6707	0.107
H(72A)	2*i*	1.0575	0.4321	0.6413	0.103
H(72B)	2*i*	1.1761	0.4733	0.6352	0.103
H(72C)	2*i*	1.0952	0.4252	0.7090	0.103

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}
C(1)	2*i*	0.4801(2)	0.1374(2)	0.2362(1)	0.035(1)	0.035(2)	0.045(1)	0.001(1)	−0.013(1)	−0.014(1)
C(2)	2*i*	0.5103(3)	0.0934(2)	0.1968(2)	0.049(2)	0.036(2)	0.052(2)	0.003(1)	−0.023(1)	−0.018(1)
C(3)	2*i*	0.6218(3)	0.1027(2)	0.1543(1)	0.055(2)	0.036(2)	0.042(1)	0.009(1)	−0.019(1)	−0.017(1)
C(4)	2*i*	0.6988(2)	0.1545(2)	0.1550(1)	0.042(1)	0.042(2)	0.039(1)	0.011(1)	−0.010(1)	−0.019(1)
C(5)	2*i*	0.6655(2)	0.1973(2)	0.1987(1)	0.036(1)	0.031(2)	0.033(1)	0.005(1)	−0.011(1)	−0.011(1)
C(6)	2*i*	0.6556(3)	0.0556(2)	0.1091(2)	0.073(2)	0.066(3)	0.064(2)	0.011(2)	−0.020(2)	−0.042(2)
C(7)	2*i*	0.9516(3)	0.1713(2)	0.1912(2)	0.045(2)	0.067(3)	0.070(2)	0.013(2)	−0.014(2)	−0.029(2)
C(8)	2*i*	0.9222(3)	0.3314(2)	0.0947(2)	0.061(2)	0.071(3)	0.054(2)	−0.006(2)	0.008(2)	−0.010(2)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(9)	2*i*	0.9248(3)	0.3004(3)	0.2419(2)	0.036(2)	0.093(3)	0.081(2)	0.001(2)	–0.014(2)	–0.049(2)
C(10)	2*i*	0.6875(2)	0.1035(2)	0.3503(1)	0.041(1)	0.028(2)	0.041(1)	0.002(1)	–0.010(1)	–0.013(1)
C(11)	2*i*	0.7595(2)	0.0665(2)	0.3896(1)	0.041(1)	0.025(2)	0.046(2)	0.007(1)	–0.008(1)	–0.011(1)
C(12)	2*i*	0.7951(2)	0.1015(2)	0.4293(1)	0.032(1)	0.037(2)	0.039(1)	0.003(1)	–0.008(1)	–0.006(1)
C(13)	2*i*	0.7576(2)	0.1719(2)	0.4269(1)	0.039(1)	0.035(2)	0.038(1)	0.003(1)	–0.013(1)	–0.011(1)
C(14)	2*i*	0.6858(2)	0.2084(2)	0.3844(1)	0.030(1)	0.032(2)	0.032(1)	–0.001(1)	–0.005(1)	–0.010(1)
C(15)	2*i*	0.8748(3)	0.0634(2)	0.4729(2)	0.043(2)	0.045(2)	0.060(2)	0.010(1)	–0.020(1)	–0.009(2)
C(16)	2*i*	0.8098(3)	0.3562(2)	0.4144(2)	0.056(2)	0.037(2)	0.069(2)	–0.000(1)	–0.030(2)	–0.020(2)
C(17)	2*i*	0.5839(3)	0.2725(2)	0.5128(2)	0.076(2)	0.052(2)	0.039(2)	0.005(2)	–0.009(2)	–0.017(2)
C(18)	2*i*	0.5817(3)	0.4203(2)	0.3986(2)	0.064(2)	0.044(2)	0.048(2)	0.014(1)	–0.026(1)	–0.023(1)
C(19)	2*i*	0.4181(2)	0.4343(2)	0.2937(1)	0.043(2)	0.029(2)	0.042(1)	0.004(1)	–0.016(1)	–0.010(1)
C(20)	2*i*	0.3117(2)	0.4556(2)	0.3202(2)	0.046(2)	0.035(2)	0.053(2)	0.014(1)	–0.023(1)	–0.017(1)
C(21)	2*i*	0.2278(2)	0.3990(2)	0.3654(2)	0.037(1)	0.042(2)	0.062(2)	0.013(1)	–0.016(1)	–0.023(2)
C(22)	2*i*	0.2537(2)	0.3241(2)	0.3803(2)	0.034(1)	0.039(2)	0.054(2)	0.003(1)	–0.005(1)	–0.016(1)
C(23)	2*i*	0.3638(2)	0.3053(2)	0.3509(1)	0.032(1)	0.029(2)	0.037(1)	0.003(1)	–0.010(1)	–0.012(1)
C(24)	2*i*	0.1097(3)	0.4188(2)	0.3969(2)	0.046(2)	0.055(3)	0.103(3)	0.019(2)	–0.010(2)	–0.036(2)
C(25)	2*i*	0.2904(4)	0.1589(2)	0.5024(2)	0.081(3)	0.051(3)	0.048(2)	0.001(2)	0.007(2)	–0.009(2)
C(26)	2*i*	0.1911(3)	0.1411(2)	0.3911(2)	0.043(2)	0.050(2)	0.096(3)	–0.010(2)	–0.015(2)	–0.015(2)
C(27)	2*i*	0.4083(3)	0.0672(2)	0.4203(2)	0.047(2)	0.027(2)	0.059(2)	0.000(1)	0.002(1)	–0.004(1)
C(28)	2*i*	0.7423(2)	0.4515(2)	0.2169(1)	0.038(1)	0.044(2)	0.038(1)	–0.005(1)	–0.009(1)	–0.014(1)
C(29)	2*i*	0.7730(3)	0.5179(2)	0.1620(2)	0.049(2)	0.040(2)	0.047(2)	–0.012(1)	–0.007(1)	–0.012(1)
C(30)	2*i*	0.7291(3)	0.5292(2)	0.1051(2)	0.051(2)	0.042(2)	0.040(2)	–0.004(1)	–0.006(1)	–0.005(1)
C(31)	2*i*	0.6541(2)	0.4748(2)	0.1075(1)	0.048(2)	0.042(2)	0.033(1)	0.000(1)	–0.011(1)	–0.007(1)
C(32)	2*i*	0.6194(2)	0.4089(2)	0.1672(1)	0.037(1)	0.035(2)	0.030(1)	0.004(1)	–0.008(1)	–0.011(1)
C(33)	2*i*	0.7649(4)	0.6003(2)	0.0420(2)	0.086(3)	0.051(3)	0.053(2)	–0.023(2)	–0.017(2)	0.009(2)
C(34)	2*i*	0.3889(3)	0.4504(2)	0.1003(2)	0.055(2)	0.062(3)	0.064(2)	0.016(2)	–0.027(2)	–0.015(2)
C(35)	2*i*	0.5023(4)	0.3175(3)	0.0647(2)	0.088(3)	0.098(3)	0.073(2)	0.034(2)	–0.046(2)	–0.055(2)
C(36)	2*i*	0.3109(3)	0.2938(2)	0.1951(2)	0.049(2)	0.071(3)	0.082(2)	–0.005(2)	–0.035(2)	–0.010(2)
C(37)	2*i*	0.9094(2)	0.3472(2)	0.6140(1)	0.044(2)	0.045(2)	0.035(1)	0.006(1)	–0.010(1)	–0.013(1)
C(38)	2*i*	0.8379(3)	0.3918(2)	0.5790(1)	0.063(2)	0.033(2)	0.037(1)	0.008(1)	–0.018(1)	–0.009(1)
C(39)	2*i*	0.7203(3)	0.3865(2)	0.6114(2)	0.056(2)	0.032(2)	0.048(2)	0.013(1)	–0.028(1)	–0.019(1)
C(40)	2*i*	0.6804(2)	0.3374(2)	0.6777(2)	0.036(1)	0.044(2)	0.048(2)	0.013(1)	–0.020(1)	–0.021(1)
C(41)	2*i*	0.7587(2)	0.2938(2)	0.7137(1)	0.036(1)	0.027(2)	0.041(1)	0.006(1)	–0.015(1)	–0.015(1)
C(42)	2*i*	0.6399(3)	0.4364(2)	0.5738(2)	0.073(2)	0.056(2)	0.058(2)	0.025(2)	–0.039(2)	–0.019(2)
C(43)	2*i*	0.5266(3)	0.3182(2)	0.8338(2)	0.053(2)	0.082(3)	0.067(2)	0.027(2)	–0.009(2)	–0.030(2)
C(44)	2*i*	0.5298(4)	0.1496(3)	0.8565(3)	0.057(2)	0.081(4)	0.159(5)	–0.017(2)	0.018(3)	–0.047(3)
C(45)	2*i*	0.6635(4)	0.2223(3)	0.9244(2)	0.075(3)	0.132(4)	0.046(2)	0.039(3)	–0.004(2)	–0.027(2)
C(46)	2*i*	0.8779(3)	0.3960(2)	0.7767(2)	0.052(2)	0.037(2)	0.053(2)	0.016(1)	–0.024(1)	–0.019(1)
C(47)	2*i*	0.8757(3)	0.4406(2)	0.8150(2)	0.054(2)	0.037(2)	0.062(2)	0.021(1)	–0.024(1)	–0.023(2)
C(48)	2*i*	0.9180(2)	0.4126(2)	0.8733(2)	0.045(2)	0.043(2)	0.053(2)	0.009(1)	–0.014(1)	–0.024(1)
C(49)	2*i*	0.9581(2)	0.3409(2)	0.8902(1)	0.042(2)	0.042(2)	0.042(1)	0.008(1)	–0.016(1)	–0.019(1)
C(50)	2*i*	0.9560(2)	0.2966(2)	0.8498(1)	0.029(1)	0.033(2)	0.037(1)	0.004(1)	–0.010(1)	–0.013(1)
C(51)	2*i*	0.9169(3)	0.4610(2)	0.9163(2)	0.077(2)	0.053(2)	0.069(2)	0.021(2)	–0.030(2)	–0.037(2)
C(52)	2*i*	1.1999(3)	0.2288(2)	0.8991(2)	0.052(2)	0.067(3)	0.086(3)	0.004(2)	–0.036(2)	–0.018(2)
C(53)	2*i*	0.9777(4)	0.1605(2)	1.0079(2)	0.088(3)	0.065(3)	0.041(2)	0.023(2)	–0.016(2)	–0.019(2)
C(54)	2*i*	1.0957(3)	0.0799(2)	0.9147(2)	0.072(2)	0.050(2)	0.052(2)	0.023(2)	–0.037(2)	–0.020(2)
C(55)	2*i*	0.8059(2)	0.0572(2)	0.9124(2)	0.042(2)	0.047(2)	0.044(2)	0.001(1)	–0.016(1)	–0.006(1)
C(56)	2*i*	0.7576(3)	–0.0142(2)	0.9339(2)	0.057(2)	0.052(2)	0.056(2)	–0.013(2)	–0.026(2)	0.007(2)
C(57)	2*i*	0.7423(3)	–0.0444(2)	0.8857(2)	0.063(2)	0.035(2)	0.087(3)	–0.010(2)	–0.045(2)	–0.000(2)
C(58)	2*i*	0.7736(3)	0.0009(2)	0.8178(2)	0.052(2)	0.035(2)	0.071(2)	–0.002(1)	–0.035(2)	–0.013(2)
C(59)	2*i*	0.8226(2)	0.0758(2)	0.7968(2)	0.028(1)	0.039(2)	0.051(2)	0.005(1)	–0.017(1)	–0.018(1)
C(60)	2*i*	0.6929(4)	–0.1255(2)	0.9089(3)	0.124(4)	0.045(3)	0.115(4)	–0.031(2)	–0.078(3)	0.013(2)
C(61)	2*i*	0.7224(3)	0.1044(4)	0.6472(2)	0.054(2)	0.183(6)	0.067(2)	0.015(3)	–0.031(2)	–0.055(3)
C(62)	2*i*	0.9433(5)	0.0245(3)	0.6563(3)	0.182(5)	0.094(4)	0.102(3)	0.079(4)	–0.084(4)	–0.074(3)
C(63)	2*i*	0.9543(5)	0.1891(3)	0.5843(2)	0.137(4)	0.124(5)	0.068(3)	–0.061(4)	0.039(3)	–0.067(3)
C(64)	2*i*	1.1011(2)	0.0614(2)	0.7707(1)	0.035(1)	0.030(2)	0.041(1)	0.001(1)	–0.012(1)	–0.012(1)
C(65)	2*i*	1.2069(2)	0.0411(2)	0.7431(1)	0.039(1)	0.027(2)	0.047(2)	0.009(1)	–0.014(1)	–0.014(1)
C(66)	2*i*	1.2914(2)	0.0980(2)	0.6991(1)	0.035(1)	0.037(2)	0.046(2)	0.008(1)	–0.011(1)	–0.015(1)
C(67)	2*i*	1.2637(2)	0.1724(2)	0.6839(1)	0.033(1)	0.030(2)	0.048(2)	0.001(1)	–0.006(1)	–0.007(1)
C(68)	2*i*	1.1534(2)	0.1905(2)	0.7133(1)	0.031(1)	0.029(2)	0.037(1)	0.004(1)	–0.012(1)	–0.011(1)
C(69)	2*i*	1.4090(3)	0.0784(2)	0.6677(2)	0.039(2)	0.042(2)	0.073(2)	0.012(1)	–0.003(1)	–0.012(2)
C(70)	2*i*	1.2568(3)	0.3539(2)	0.5658(2)	0.061(2)	0.056(3)	0.059(2)	0.004(2)	0.000(2)	0.001(2)
C(71)	2*i*	1.3205(3)	0.3411(2)	0.6974(2)	0.056(2)	0.057(3)	0.097(3)	–0.012(2)	–0.030(2)	–0.014(2)
C(72)	2*i*	1.1230(3)	0.4283(2)	0.6613(2)	0.058(2)	0.028(2)	0.104(3)	–0.001(1)	–0.006(2)	–0.014(2)
N(1)	2*i*	0.5542(2)	0.1887(1)	0.2371(1)	0.032(1)	0.035(1)	0.037(1)	0.0031(9)	–0.0092(9)	–0.015(1)
N(2)	2*i*	0.7283(2)	0.2473(1)	0.2093(1)	0.032(1)	0.035(1)	0.035(1)	0.0007(9)	–0.0058(8)	–0.013(1)
N(3)	2*i*	0.6498(2)	0.1714(1)	0.3485(1)	0.035(1)	0.026(1)	0.036(1)	0.0036(8)	–0.0103(9)	–0.0116(9)
N(4)	2*i*	0.6490(2)	0.2804(1)	0.3693(1)	0.035(1)	0.029(1)	0.032(1)	0.0043(9)	–0.0115(8)	–0.0102(9)
N(5)	2*i*	0.4442(2)	0.3620(1)	0.3097(1)	0.035(1)	0.023(1)	0.036(1)	0.0034(8)	–0.0114(9)	–0.0092(9)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(6)	2*i*	0.4047(2)	0.2356(1)	0.3557(1)	0.030(1)	0.028(1)	0.039(1)	0.0016(8)	–0.0043(9)	–0.0106(9)
N(7)	2*i*	0.6693(2)	0.3979(1)	0.2199(1)	0.035(1)	0.032(1)	0.033(1)	–0.0015(9)	–0.0083(9)	–0.0091(9)
N(8)	2*i*	0.5416(2)	0.3524(1)	0.1807(1)	0.036(1)	0.034(1)	0.032(1)	–0.0006(9)	–0.0114(9)	–0.0085(9)
N(9)	2*i*	0.8717(2)	0.2990(1)	0.6792(1)	0.034(1)	0.038(2)	0.034(1)	0.0084(9)	–0.0116(9)	–0.013(1)
N(10)	2*i*	0.7396(2)	0.2478(1)	0.7790(1)	0.030(1)	0.041(2)	0.037(1)	0.0075(9)	–0.0104(9)	–0.013(1)
N(11)	2*i*	0.9187(2)	0.3270(1)	0.7924(1)	0.040(1)	0.031(1)	0.044(1)	0.0111(9)	–0.018(1)	–0.016(1)
N(12)	2*i*	0.9836(2)	0.2234(1)	0.8595(1)	0.039(1)	0.030(1)	0.037(1)	0.0068(9)	–0.0139(9)	–0.0141(9)
N(13)	2*i*	0.8386(2)	0.1019(1)	0.8460(1)	0.032(1)	0.034(1)	0.041(1)	0.0018(9)	–0.0107(9)	–0.013(1)
N(14)	2*i*	0.8602(2)	0.1273(1)	0.7339(1)	0.033(1)	0.035(1)	0.043(1)	0.0053(9)	–0.0142(9)	–0.018(1)
N(15)	2*i*	1.0753(2)	0.1339(1)	0.7580(1)	0.030(1)	0.027(1)	0.036(1)	0.0039(8)	–0.0100(8)	–0.0097(9)
N(16)	2*i*	1.1081(2)	0.2599(1)	0.7024(1)	0.031(1)	0.026(1)	0.042(1)	0.0042(8)	–0.0093(9)	–0.0077(9)
Si(1)	2*i*	0.87641(6)	0.26184(5)	0.18432(4)	0.0297(4)	0.0483(5)	0.0427(4)	0.0000(3)	–0.0018(3)	–0.0179(4)
Si(2)	2*i*	0.65671(6)	0.33085(4)	0.42168(4)	0.0443(4)	0.0296(4)	0.0364(4)	0.0035(3)	–0.0151(3)	–0.0135(3)
Si(3)	2*i*	0.32615(6)	0.15340(5)	0.41499(4)	0.0345(4)	0.0302(5)	0.0468(4)	–0.0026(3)	0.0005(3)	–0.0083(3)
Si(4)	2*i*	0.44190(7)	0.35350(5)	0.13536(4)	0.0432(4)	0.0444(5)	0.0417(4)	0.0074(3)	–0.0213(3)	–0.0159(4)
Si(5)	2*i*	0.61683(7)	0.23436(6)	0.84473(5)	0.0346(4)	0.0552(6)	0.0516(5)	0.0085(4)	–0.0002(3)	–0.0155(4)
Si(6)	2*i*	1.06038(6)	0.17492(5)	0.91827(4)	0.0406(4)	0.0367(5)	0.0350(4)	0.0085(3)	–0.0153(3)	–0.0137(3)
Si(7)	2*i*	0.86768(6)	0.11143(5)	0.65893(4)	0.0380(4)	0.0437(5)	0.0462(4)	0.0069(3)	–0.0169(3)	–0.0233(4)
Si(8)	2*i*	1.19848(7)	0.34208(5)	0.65832(4)	0.0361(4)	0.0274(5)	0.0550(5)	0.0003(3)	–0.0077(3)	–0.0055(3)
Zr(1)	2*i*	0.57776(2)	0.27901(1)	0.28256(1)	0.0259(1)	0.0248(2)	0.0276(1)	0.00113(8)	–0.00671(9)	–0.00862(9)
Zr(2)	2*i*	0.92880(2)	0.21562(1)	0.76931(1)	0.0265(1)	0.0269(2)	0.0304(1)	0.00502(9)	–0.00982(9)	–0.0112(1)

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