Crystal structure of η^5-3,6-di-*tert*-butyl-4-(tris(pentafluorophenyl)boranyl-oxycarbonyl)-5-(η^5-tetramethylcyclopentadienyl-methyl-9,10,11-trimethyl-bicyclo(6.3.0)undeca-4-en-8,10-dienyl)titanium(III) toluene hemisolvate, $Ti(C_{51}H_{47}BF_{15}O_2) \cdot 0.5\,C_7H_8$

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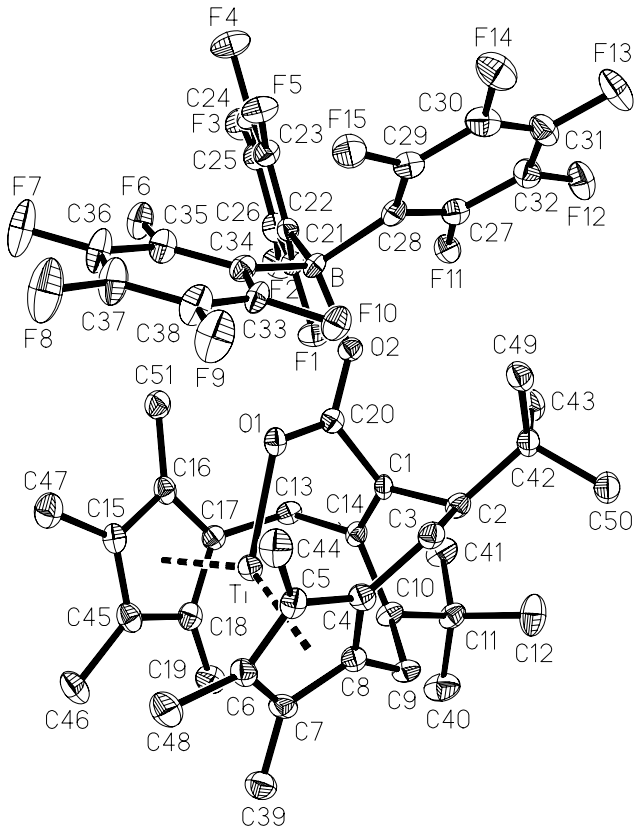

Abstract

$C_{54.50}H_{51}BF_{15}O_2Ti$, triclinic, $P\bar{1}$ (no. 2), a = 11.603(2) Å, b = 12.872(3) Å, c = 18.142(4) Å, α = 76.47(3)°, β = 77.99(3)°, γ = 69.13(3)°, V = 2438.2 Å^3, Z = 2, $R_{gt}(F)$ = 0.048, $wR_{obs}(F^2)$ = 0.114, T = 200 K.

Source of material

$B(C_6F_5)_3$ (268 mg, 0.52 mmol) was dissolved in 5 mL of toluene under Ar. The resulting solution was filtered and added to a solution of pentamethylcyclopentadienyl-trimethylcyclopentadienyl-cyclooctyne-Ti(II) [1] (252 mg, 0.52 mmol) in 10-12 ml of toluene. The brownish solution was filtered and exposed to carbon dioxide at room temperature after Ar was removed in vacuo. After 10 days at room temperature the red-brown solution was cooled down to –78 °C. After 2 days crystals had formed which were separated from the mother liquor by decanting, washed with cooled toluene and dried in vacuum to give 10 mg of the title compound as dark blue material.

Discussion

Decamethylmetallocenes of titanium and zirconium couple under C—H bond activations of methyl groups of its pentamethylcyclopentadienyl ligands with butadiynes *R*C≡C–C≡C*R*, which has been verified for *M* = Ti, *R* = Me, Ph, *t*-Bu and *M* = Zr, *R* = *t*-Bu [1-3]. Additionally, certain of these complexes insert carbon dioxide to give a further facile method for functionalizing metallocenes [1]. The best investigated example is a formal Ti(II) complex with one pentamethylcyclopentadienyl and one anellated trimethylcyclopentadienyl-cyclooctyne ligand [1-5].

In the molecular structure of the zwitterionic title complex, the titanium(III) is coordinated by two differently substituted cyclopentadienyl ligands. From the former pentamethylcyclopentadienyl ligand one methyl group is C—H activated and the formed methylene group couples with one of the former alkyne carbon atoms giving an alkenyl group. The other former alkyne carbon atom is connected with the carbon atom of the inserted carbon dioxide. This carboxylate bridges the titanium and the boranate centers.

Table 1. Data collection and handling.

Crystal:	dark blue prism, size 0.2 × 0.3 × 0.4 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.76 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS I, φ
$2\theta_{max}$:	45.4°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10948, 6047
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3622
$N(param)_{refined}$:	647
Programs:	SHELXS-86 [6], SHELXL-93 [7]

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

Atom	Site	Occ.	x	y	z	U_{iso}
H(2)	2*i*		0.2260	0.0046	0.9119	0.037
H(3A)	2*i*		0.0316	0.0912	0.9725	0.038
H(3B)	2*i*		0.0209	0.2082	0.9149	0.038
H(9A)	2*i*		0.1559	–0.1772	0.8398	0.041
H(9B)	2*i*		0.1777	–0.1190	0.9020	0.041
H(10)	2*i*		0.2768	–0.1083	0.7459	0.037

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Table 2. Continued.

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(12A)	2*i*		0.3471	–0.2181	0.9320	0.091
H(12B)	2*i*		0.4079	–0.1204	0.9194	0.091
H(12C)	2*i*		0.4952	–0.2477	0.9150	0.091
H(13A)	2*i*		0.3470	0.1332	0.6695	0.035
H(13B)	2*i*		0.3744	0.0052	0.6609	0.035
H(19A)	2*i*		0.2687	–0.1151	0.6404	0.064
H(19B)	2*i*		0.1891	–0.0943	0.5730	0.064
H(19C)	2*i*		0.1319	–0.1258	0.6602	0.064
H(39A)	2*i*		0.0224	–0.1738	0.7737	0.069
H(39B)	2*i*		–0.0695	–0.0899	0.7148	0.069
H(39C)	2*i*		–0.1250	–0.1358	0.7996	0.069
H(40A)	2*i*		0.4270	–0.2719	0.7405	0.085
H(40B)	2*i*		0.3651	–0.3146	0.8243	0.085
H(40C)	2*i*		0.5120	–0.3360	0.8071	0.085
H(41A)	2*i*		0.5141	–0.1123	0.7201	0.074
H(41B)	2*i*		0.5964	–0.1837	0.7869	0.074
H(41C)	2*i*		0.5092	–0.0564	0.7913	0.074
H(43A)	2*i*		0.3960	0.1662	0.8704	0.064
H(43B)	2*i*		0.4397	0.0367	0.9114	0.064
H(43C)	2*i*		0.4360	0.1335	0.9543	0.064
H(44A)	2*i*		–0.2754	0.2951	0.8321	0.070
H(44B)	2*i*		–0.1558	0.3232	0.8410	0.070
H(44C)	2*i*		–0.2278	0.2631	0.9137	0.070
H(46A)	2*i*		–0.0625	–0.0051	0.5994	0.074
H(46B)	2*i*		–0.0889	0.1056	0.5346	0.074
H(46C)	2*i*		–0.1671	0.1096	0.6183	0.074
H(47A)	2*i*		–0.0810	0.3959	0.5865	0.074
H(47B)	2*i*		–0.1756	0.3270	0.6245	0.074
H(47C)	2*i*		–0.1194	0.3301	0.5359	0.074
H(48A)	2*i*		–0.2668	0.0692	0.7369	0.073
H(48B)	2*i*		–0.2829	0.1995	0.7311	0.073
H(48C)	2*i*		–0.3380	0.1321	0.8078	0.073
H(49A)	2*i*		0.1887	0.2994	0.9229	0.059
H(49B)	2*i*		0.2318	0.2624	1.0060	0.059
H(49C)	2*i*		0.1043	0.2507	0.9943	0.059
H(50A)	2*i*		0.3036	–0.0322	1.0225	0.065
H(50B)	2*i*		0.1729	0.0526	1.0536	0.065
H(50C)	2*i*		0.3004	0.0644	1.0654	0.065
H(51A)	2*i*		0.0938	0.3922	0.5884	0.060
H(51B)	2*i*		0.2307	0.3191	0.5559	0.060
H(51C)	2*i*		0.1970	0.3295	0.6446	0.060
C(58)	2*i*	0.50	0.321(2)	0.040(1)	0.434(1)	0.107(5)
H(58A)	2*i*	0.50	0.2785	0.1219	0.4233	0.160
H(58B)	2*i*	0.50	0.2625	0.0032	0.4663	0.160
H(58C)	2*i*	0.50	0.3508	0.0109	0.3861	0.160
C(57)	2*i*	0.50	0.4250(5)	0.0177(6)	0.4740(4)	0.058(3)
C(52)	2*i*	0.50	0.4962(7)	–0.0945(5)	0.4942(4)	0.041(2)
H(52)	2*i*	0.50	0.4751	–0.1529	0.4822	0.049
C(53)	2*i*	0.50	0.5983(6)	–0.1214(5)	0.5322(4)	0.056(3)
H(53)	2*i*	0.50	0.6469	–0.1981	0.5460	0.068
C(54)	2*i*	0.50	0.6292(6)	–0.0360(8)	0.5498(5)	0.086(5)
H(54)	2*i*	0.50	0.6990	–0.0544	0.5758	0.103
C(55)	2*i*	0.50	0.5580(7)	0.0762(7)	0.5296(5)	0.070(3)
H(55)	2*i*	0.50	0.5792	0.1346	0.5416	0.083
C(56)	2*i*	0.50	0.4560(6)	0.1031(5)	0.4916(4)	0.042(2)
H(56)	2*i*	0.50	0.4073	0.1798	0.4778	0.050

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*	0.2288(3)	0.1191(3)	0.8140(2)	0.022(2)	0.033(2)	0.020(3)	–0.010(2)	–0.006(2)	0.001(2)
C(2)	2*i*	0.1972(4)	0.0890(3)	0.9003(2)	0.030(2)	0.031(2)	0.030(3)	–0.012(2)	–0.001(2)	–0.001(2)
C(3)	2*i*	0.0544(3)	0.1251(3)	0.9189(3)	0.029(2)	0.036(2)	0.030(3)	–0.014(2)	–0.001(2)	–0.001(2)
C(4)	2*i*	–0.0051(4)	0.0904(3)	0.8669(2)	0.027(2)	0.039(3)	0.030(3)	–0.013(2)	0.000(2)	–0.007(2)
C(5)	2*i*	–0.1199(4)	0.1563(3)	0.8404(2)	0.023(2)	0.041(3)	0.028(3)	–0.010(2)	0.001(2)	0.000(2)
C(6)	2*i*	–0.1477(4)	0.0924(4)	0.7965(3)	0.024(2)	0.048(3)	0.031(3)	–0.018(2)	–0.002(2)	–0.004(2)
C(7)	2*i*	–0.0502(4)	–0.0126(3)	0.7958(3)	0.031(2)	0.041(3)	0.033(3)	–0.021(2)	0.001(2)	–0.006(2)
C(8)	2*i*	0.0412(4)	–0.0135(3)	0.8379(2)	0.027(2)	0.038(3)	0.027(3)	–0.015(2)	0.001(2)	–0.003(2)
C(9)	2*i*	0.1631(4)	–0.1067(3)	0.8483(3)	0.035(3)	0.031(2)	0.038(3)	–0.018(2)	–0.001(2)	–0.002(2)
C(10)	2*i*	0.2800(3)	–0.0846(3)	0.7940(3)	0.026(2)	0.024(2)	0.036(3)	–0.003(2)	0.002(2)	–0.005(2)
C(11)	2*i*	0.4095(4)	–0.1691(3)	0.8185(3)	0.030(3)	0.029(2)	0.042(4)	–0.005(2)	–0.004(2)	0.000(2)
C(12)	2*i*	0.4154(5)	–0.1907(4)	0.9038(3)	0.052(3)	0.063(3)	0.046(4)	0.009(3)	–0.016(3)	–0.004(3)
C(13)	2*i*	0.3102(4)	0.0718(3)	0.6795(2)	0.027(2)	0.029(2)	0.028(3)	–0.010(2)	0.005(2)	–0.006(2)
C(14)	2*i*	0.2717(3)	0.0415(3)	0.7663(2)	0.019(2)	0.030(2)	0.031(3)	–0.005(2)	–0.007(2)	0.001(2)
C(15)	2*i*	0.0057(4)	0.2248(4)	0.6077(3)	0.035(3)	0.043(3)	0.030(3)	–0.010(2)	–0.008(2)	–0.006(2)
C(16)	2*i*	0.1251(4)	0.2226(4)	0.6171(2)	0.034(3)	0.045(3)	0.022(3)	–0.014(2)	–0.004(2)	–0.006(2)
C(17)	2*i*	0.1995(4)	0.1093(3)	0.6364(2)	0.030(2)	0.036(3)	0.023(3)	–0.014(2)	0.001(2)	–0.004(2)
C(18)	2*i*	0.1307(4)	0.0380(3)	0.6310(2)	0.038(3)	0.040(3)	0.021(3)	–0.011(2)	0.001(2)	–0.009(2)
C(19)	2*i*	0.1848(4)	–0.0849(4)	0.6257(3)	0.042(3)	0.047(3)	0.043(3)	–0.015(2)	0.001(2)	–0.017(2)
C(20)	2*i*	0.1819(4)	0.2436(3)	0.7812(2)	0.027(3)	0.029(2)	0.024(3)	–0.007(2)	0.000(2)	–0.006(2)
C(21)	2*i*	0.4354(4)	0.3718(3)	0.6564(3)	0.038(3)	0.031(3)	0.031(3)	–0.013(2)	–0.008(2)	–0.001(2)
C(22)	2*i*	0.3403(4)	0.4528(3)	0.6896(2)	0.032(2)	0.032(3)	0.024(3)	–0.013(2)	–0.005(2)	–0.003(2)
C(23)	2*i*	0.3527(4)	0.5598(4)	0.6676(3)	0.038(3)	0.036(3)	0.029(3)	–0.011(2)	0.000(2)	–0.005(2)
C(24)	2*i*	0.4481(4)	0.5850(4)	0.6170(3)	0.054(3)	0.041(3)	0.040(4)	–0.027(3)	–0.003(3)	0.004(2)
C(25)	2*i*	0.5396(4)	0.5008(4)	0.5842(3)	0.042(3)	0.055(3)	0.028(3)	–0.030(3)	0.001(2)	0.004(2)
C(26)	2*i*	0.5334(4)	0.3933(4)	0.6032(3)	0.026(3)	0.049(3)	0.034(3)	–0.007(2)	0.003(2)	–0.013(2)
C(27)	2*i*	0.3697(4)	0.4088(3)	0.8586(3)	0.033(3)	0.034(3)	0.030(3)	–0.012(2)	0.007(2)	–0.009(2)
C(28)	2*i*	0.2561(4)	0.4688(3)	0.8329(2)	0.033(3)	0.033(2)	0.028(3)	–0.012(2)	–0.001(2)	0.003(2)
C(29)	2*i*	0.1814(4)	0.5500(4)	0.8757(3)	0.035(3)	0.033(3)	0.041(4)	–0.012(2)	0.000(2)	–0.008(2)
C(30)	2*i*	0.2133(4)	0.5684(4)	0.9404(3)	0.048(3)	0.047(3)	0.040(4)	–0.021(3)	0.014(3)	–0.019(3)
C(31)	2*i*	0.3247(4)	0.5007(4)	0.9655(3)	0.046(3)	0.064(3)	0.035(4)	–0.031(3)	–0.001(3)	–0.012(3)
C(32)	2*i*	0.4043(4)	0.4232(4)	0.9234(3)	0.032(3)	0.046(3)	0.042(4)	–0.015(2)	–0.007(2)	–0.005(2)
C(33)	2*i*	–0.0186(4)	0.5049(3)	0.7878(3)	0.040(3)	0.032(2)	0.024(3)	–0.005(2)	–0.004(2)	0.002(2)
C(34)	2*i*	0.0860(4)	0.5074(3)	0.7352(3)	0.035(3)	0.027(2)	0.031(3)	–0.006(2)	–0.002(2)	–0.005(2)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(35)	2*i*	0.0573(4)	0.5723(4)	0.6649(3)	0.035(3)	0.046(3)	0.027(3)	–0.006(2)	0.005(2)	–0.009(2)
C(36)	2*i*	–0.0631(4)	0.6288(4)	0.6486(3)	0.040(3)	0.058(3)	0.036(4)	0.006(3)	–0.008(3)	0.006(3)
C(37)	2*i*	–0.1608(4)	0.6203(4)	0.7028(3)	0.029(3)	0.063(3)	0.053(4)	0.016(3)	–0.011(3)	–0.006(3)
C(38)	2*i*	–0.1394(4)	0.5588(4)	0.7742(3)	0.031(3)	0.054(3)	0.034(4)	–0.003(2)	0.010(2)	–0.002(2)
C(39)	2*i*	–0.0561(4)	–0.1116(4)	0.7686(3)	0.047(3)	0.051(3)	0.046(4)	–0.025(2)	–0.005(2)	–0.009(2)
C(40)	2*i*	0.4303(4)	–0.2833(4)	0.7955(3)	0.041(3)	0.033(3)	0.088(5)	–0.004(2)	–0.010(3)	–0.007(3)
C(41)	2*i*	0.5170(4)	–0.1265(4)	0.7753(3)	0.028(3)	0.043(3)	0.067(4)	–0.008(2)	–0.006(2)	0.003(2)
C(42)	2*i*	0.2600(4)	0.1256(3)	0.9549(2)	0.030(2)	0.042(3)	0.026(3)	–0.013(2)	–0.006(2)	0.000(2)
C(43)	2*i*	0.3951(4)	0.1145(4)	0.9195(3)	0.036(3)	0.057(3)	0.036(3)	–0.017(2)	–0.007(2)	–0.004(2)
C(44)	2*i*	–0.2018(4)	0.2691(4)	0.8584(3)	0.032(3)	0.057(3)	0.045(4)	–0.007(2)	–0.002(2)	–0.011(2)
C(45)	2*i*	0.0109(4)	0.1113(4)	0.6143(3)	0.035(3)	0.051(3)	0.029(3)	–0.014(2)	–0.004(2)	–0.011(2)
C(46)	2*i*	–0.0854(4)	0.0774(4)	0.5895(3)	0.050(3)	0.069(3)	0.042(4)	–0.025(3)	–0.012(2)	–0.018(3)
C(47)	2*i*	–0.1019(4)	0.3285(4)	0.5868(3)	0.042(3)	0.052(3)	0.047(4)	–0.010(2)	–0.011(2)	–0.001(2)
C(48)	2*i*	–0.2696(4)	0.1263(4)	0.7654(3)	0.032(3)	0.060(3)	0.056(4)	–0.015(2)	–0.009(2)	–0.012(3)
C(49)	2*i*	0.1899(4)	0.2452(4)	0.9710(3)	0.042(3)	0.053(3)	0.030(3)	–0.022(2)	–0.003(2)	–0.009(2)
C(50)	2*i*	0.2591(4)	0.0455(4)	1.0308(3)	0.043(3)	0.052(3)	0.036(3)	–0.019(2)	–0.011(2)	0.000(2)
C(51)	2*i*	0.1651(4)	0.3246(4)	0.6000(3)	0.043(3)	0.047(3)	0.031(3)	–0.019(2)	–0.005(2)	–0.002(2)
B	2*i*	0.2260(4)	0.4373(4)	0.7577(3)	0.034(3)	0.026(3)	0.024(3)	–0.010(2)	–0.004(2)	0.004(2)
F(1)	2*i*	0.4419(2)	0.2616(2)	0.6729(2)	0.036(1)	0.036(2)	0.054(2)	–0.013(1)	0.003(1)	–0.010(1)
F(2)	2*i*	0.6215(2)	0.3091(2)	0.5712(2)	0.035(2)	0.062(2)	0.058(2)	–0.016(1)	0.008(1)	–0.023(1)
F(3)	2*i*	0.6349(2)	0.5228(2)	0.5337(2)	0.054(2)	0.077(2)	0.051(2)	–0.038(2)	0.007(1)	–0.002(1)
F(4)	2*i*	0.4524(3)	0.6912(2)	0.5981(2)	0.072(2)	0.047(2)	0.074(2)	–0.037(2)	0.005(2)	0.002(1)
F(5)	2*i*	0.2633(2)	0.6470(2)	0.6975(2)	0.052(2)	0.033(1)	0.061(2)	–0.014(1)	0.002(1)	–0.010(1)
F(6)	2*i*	0.1477(2)	0.5825(2)	0.6052(2)	0.043(2)	0.056(2)	0.033(2)	–0.010(1)	–0.005(1)	0.002(1)
F(7)	2*i*	–0.0826(3)	0.6891(3)	0.5780(2)	0.055(2)	0.097(2)	0.045(2)	0.013(2)	–0.014(2)	0.017(2)
F(8)	2*i*	–0.2784(3)	0.6716(3)	0.6864(2)	0.037(2)	0.114(3)	0.075(3)	0.020(2)	–0.012(2)	0.006(2)
F(9)	2*i*	–0.2353(2)	0.5525(2)	0.8284(2)	0.036(2)	0.081(2)	0.058(2)	0.001(1)	0.005(2)	–0.002(2)
F(10)	2*i*	–0.0031(2)	0.4436(2)	0.8586(2)	0.036(1)	0.045(2)	0.036(2)	–0.011(1)	–0.000(1)	0.000(1)
F(11)	2*i*	0.4557(2)	0.3306(2)	0.8204(1)	0.033(1)	0.046(2)	0.039(2)	–0.012(1)	–0.005(1)	–0.010(1)
F(12)	2*i*	0.5148(2)	0.3559(2)	0.9458(2)	0.045(2)	0.083(2)	0.045(2)	–0.021(2)	–0.013(1)	–0.013(1)
F(13)	2*i*	0.3562(2)	0.5164(3)	1.0281(2)	0.060(2)	0.107(2)	0.045(2)	–0.039(2)	–0.003(2)	–0.035(2)
F(14)	2*i*	0.1356(2)	0.6498(2)	0.9794(2)	0.062(2)	0.071(2)	0.055(2)	–0.022(2)	0.006(2)	–0.035(2)
F(15)	2*i*	0.0714(2)	0.6232(2)	0.8559(2)	0.050(2)	0.040(2)	0.054(2)	–0.003(1)	–0.010(1)	–0.016(1)
Ti	2*i*	0.04200(7)	0.13305(6)	0.73636(5)	0.0270(4)	0.0353(5)	0.0274(5)	–0.0113(4)	–0.0027(3)	–0.0053(4)
O(1)	2*i*	0.0783(2)	0.2769(2)	0.7586(2)	0.027(2)	0.037(2)	0.027(2)	–0.008(1)	–0.007(1)	–0.003(1)
O(2)	2*i*	0.2464(2)	0.3070(2)	0.7820(2)	0.027(2)	0.028(2)	0.030(2)	–0.009(1)	–0.003(1)	–0.004(1)

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