

Supplementary Materials:

Phenylacetylene and Carbon Dioxide Activation by an Organometallic Samarium Complex

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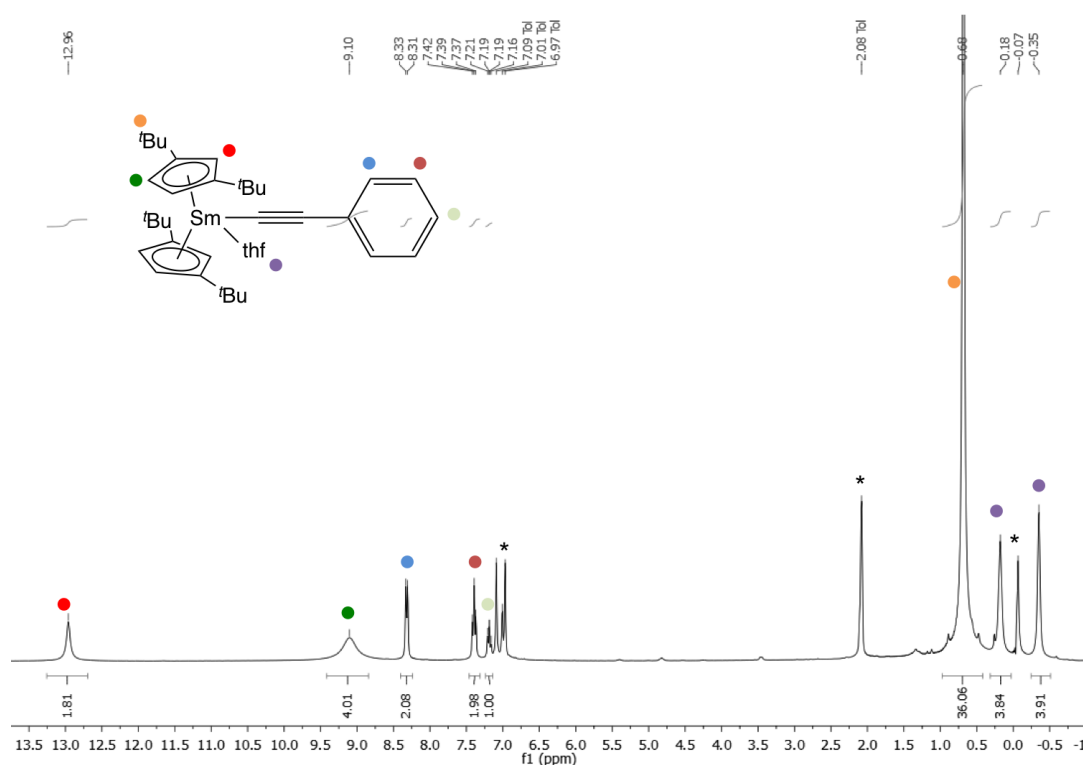
I. ¹H NMR

Figure S1. ¹H NMR of **1** in toluene-*d*₈. * for solvents and impurities.

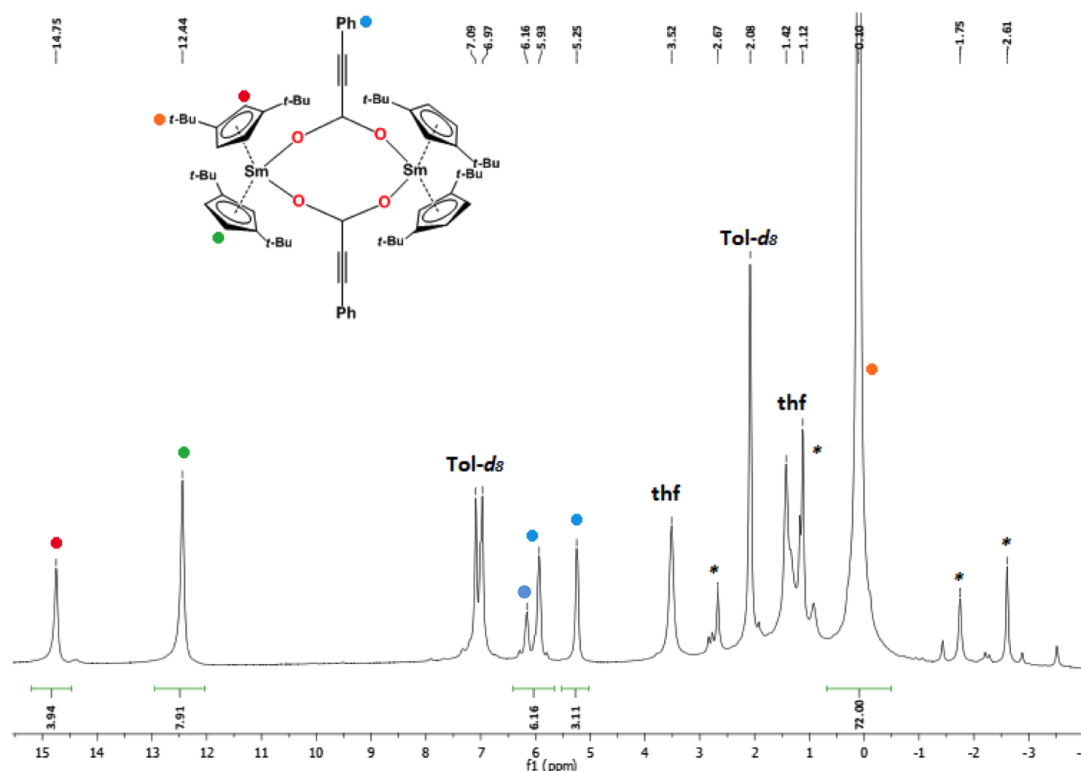


Figure S2. ^1H NMR of **2** in toluene- d_8 . * for solvents and impurities.

II. X-ray Crystal Structure

Single crystals of the compounds **1** and **2** were mounted on a Kapton loop using a Paratone N oil. An APEX II CCD BRUKER detector and a graphite Mo- $K\alpha$ monochromator were used for the data acquisition. All measurements were done at 150 K and a refinement method was used for solving the structure. The structure resolution was accomplished using the SHELXT-2014 [26] program and the refinement was done with the SHELXL-2014/7 [27] program. The structure solution and the refinement were achieved with the PLATON software [28]. Finally, ORTEP of the compounds were obtained using the MERCURY software [29]. During the refinement steps, all atoms- except hydrogens- were refined anisotropically. The position of the hydrogens was determined using residual electronic densities which are calculated by a Fourier difference. Finally, in order to obtain a complete refinement, a weighting step followed by multiples loops of refinement was done. Details on crystal data and structure refinements are summarized in Table S1 in the supplementary materials. CIF files are deposited at the Cambridge Data Base Centre under the reference CCDC numbers 1852482–1852483.

Table S1. Selected Crystal Data Collection Parameters for **1** and **2**.

	1	2
Formula	C ₃₈ H ₅₅ O Sm	C ₇₀ H ₉₄ O ₄ Sm ₂ , 2(C ₇ H ₈)
Crystal size (mm)	0.240x0.140x0.080	0.360x0.060x0.020
Crystal system	Triclinic	Monoclinic
Space group	P -1	C 2/c
Volume (Å ³)	1751.15(8)	7622.3(10)
a (Å)	10.0433(3)	26.008(2)
b (Å)	11.0189(2)	11.2284(9)
c (Å)	16.2612(4)	27.4919(19)
α (deg)	95.701(1)	90
β (deg)	101.441(1)	108.302(4)
γ (deg)	92.611(1)	90
Z	2	4
Formula weight –g/mol)	678.17	1484.41
Density (calcd) (g/cm ⁻³)	1.286	1.294
Absorption coefficient (mm ⁻¹)	0.1702	0.1572
F(000)	706	3080
Temp (K)	150	150
diffractometer ^a	Kappa APEX II CCD	Kappa APEX II CCD
θ range for data collection (deg)	2.3769 – 25.7179	2.6843 – 25.0311
Transmission range	/	/
Absorption correction	Multi-scan; T _{min} : 0.6424 / T _{max} : 0.7456	Multi-scan; T _{min} : 0.6680 / T _{max} : 0.7452
Total no. reflections	12600	12600
Unique reflections [R _{int}]	6033 [0.0413]	6033 [0.0413]
Final R ^b indices [I>2σ(I)]	R = 0.0281, R _w = 0.0700	R = 0.0534, R _w = 0.1259
R indices (all data)	R = 0.0335, R _w = 0.0732	R = 0.0659, R _w = 0.1307
Largest diff. peak and hole (e.Å ⁻³)	0.568(0.068) / - 0.411(0.068)	1.077(0.111) / - 2.131(0.111)
GooF	1.041	1.189

^aRadiation: graphite monochromated Mo Kα (λ = 0.71073 Å). ^bR = Σ ||F_o| - |F_c|| / Σ |F_o|.

Table S2. Bond length (in Å) and angles (in °) for **1**.

Sm(1)–C(1)	2.450(3)	Sm(1)–O(1)	2.504(2)
Sm(1)–C(23)	2.685(3)	Sm(1)–C(13)	2.703(3)
Sm(1)–C(24)	2.705(3)	Sm(1)–C(9)	2.711(3)
Sm(1)–C(22)	2.748(3)	Sm(1)–C(26)	2.773(3)
Sm(1)–C(12)	2.777(3)	Sm(1)–C(25)	2.790(3)
Sm(1)–C(11)	2.794(3)	Sm(1)–C(10)	2.807(3)
O(1)–C(38)	1.437(4)	O(1)–C(35)	1.445(4)
C(1)–C(2)	1.202(4)	C(2)–C(3)	1.438(4)
C(3)–C(8)	1.378(4)	C(3)–C(4)	1.387(5)
C(4)–C(5)	1.382(5)	C(4)–H(4)	0.9500
C(5)–C(6)	1.352(6)	C(5)–H(5)	0.9500
C(6)–C(7)	1.350(5)	C(6)–H(6)	0.9500
C(7)–C(8)	1.373(5)	C(7)–H(7)	0.9500
C(8)–H(8)	0.9500	C(9)–C(10)	1.406(4)
C(9)–C(13)	1.407(4)	C(9)–H(9)	0.9500
C(10)–C(11)	1.412(4)	C(10)–C(14)	1.524(4)
C(11)–C(12)	1.419(4)	C(11)–H(11)	0.9500
C(12)–C(13)	1.409(5)	C(12)–C(18)	1.522(4)
C(13)–H(13)	0.9500	C(14)–C(16)	1.512(5)
C(14)–C(15)	1.526(5)	C(14)–C(17)	1.540(5)
C(15)–H(15A)	0.9800	C(15)–H(15B)	0.9800
C(15)–H(15C)	0.9800	C(16)–H(16A)	0.9800
C(16)–H(16B)	0.9800	C(16)–H(16C)	0.9800
C(17)–H(17A)	0.9800	C(17)–H(17B)	0.9800
C(17)–H(17C)	0.9800	C(18)–C(21)	1.505(5)
C(18)–C(20)	1.521(5)	C(18)–C(19)	1.539(5)
C(19)–H(19A)	0.9800	C(19)–H(19B)	0.9800
C(19)–H(19C)	0.9800	C(20)–H(20A)	0.9800
C(20)–H(20B)	0.9800	C(20)–H(20C)	0.9800
C(21)–H(21A)	0.9800	C(21)–H(21B)	0.9800
C(21)–H(21C)	0.9800	C(22)–C(23)	1.400(4)
C(22)–C(26)	1.409(4)	C(22)–C(27)	1.528(5)
C(23)–C(24)	1.398(4)	C(23)–H(23)	0.9500
C(24)–C(25)	1.405(4)	C(24)–H(24)	0.9500
C(25)–C(26)	1.410(5)	C(25)–C(31)	1.518(5)
C(26)–H(26)	0.9500	C(27)–C(30)	1.511(6)
C(27)–C(29)	1.513(6)	C(27)–C(28)	1.530(5)
C(28)–H(28A)	0.9800	C(28)–H(28B)	0.9800
C(28)–H(28C)	0.9800	C(29)–H(29A)	0.9800
C(29)–H(29B)	0.9800	C(29)–H(29C)	0.9800
C(30)–H(30A)	0.9800	C(30)–H(30B)	0.9800
C(30)–H(30C)	0.9800	C(31)–C(32)	1.515(5)
C(31)–C(33)	1.519(6)	C(31)–C(34)	1.524(5)
C(32)–H(32A)	0.9800	C(32)–H(32B)	0.9800
C(32)–H(32C)	0.9800	C(33)–H(33A)	0.9800
C(33)–H(33B)	0.9800	C(33)–H(33C)	0.9800
C(34)–H(34A)	0.9800	C(34)–H(34B)	0.9800
C(34)–H(34C)	0.9800	C(35)–C(36)	1.487(6)
C(35)–H(35A)	0.9900	C(35)–H(35B)	0.9900
C(36)–C(37)	1.425(7)	C(36)–H(36A)	0.9900
C(36)–H(36B)	0.9900	C(37)–C(38)	1.412(6)
C(37)–H(37A)	0.9900	C(37)–H(37B)	0.9900
C(38)–H(38A)	0.9900	C(38)–H(38B)	0.9900
C(1)–Sm(1)–O(1)	85.0(1)	C(1)–Sm(1)–C(23)	115.4(1)
O(1)–Sm(1)–C(23)	136.1(1)	C(1)–Sm(1)–C(13)	116.8(1)
O(1)–Sm(1)–C(13)	129.1(1)	C(23)–Sm(1)–C(13)	78.1(1)

C (1) –Sm (1) –C (24)	129.6 (1)	O (1) –Sm (1) –C (24)	106.7 (1)
C (23) –Sm (1) –C (24)	30.1 (1)	C (13) –Sm (1) –C (24)	93.7 (1)
C (1) –Sm (1) –C (9)	88.1 (1)	O (1) –Sm (1) –C (9)	121.91 (8)
C (23) –Sm (1) –C (9)	98.4 (1)	C (13) –Sm (1) –C (9)	30.1 (1)
C (24) –Sm (1) –C (9)	121.2 (1)	C (1) –Sm (1) –C (22)	86.2 (1)
O (1) –Sm (1) –C (22)	132.1 (1)	C (23) –Sm (1) –C (22)	29.8 (1)
C (13) –Sm (1) –C (22)	96.6 (1)	C (24) –Sm (1) –C (22)	49.2 (1)
C (9) –Sm (1) –C (22)	104.7 (1)	C (1) –Sm (1) –C (26)	81.3 (1)
O (1) –Sm (1) –C (26)	102.55 (8)	C (23) –Sm (1) –C (26)	48.7 (1)
C (13) –Sm (1) –C (26)	124.9 (1)	C (24) –Sm (1) –C (26)	48.4 (1)
C (9) –Sm (1) –C (26)	133.1 (1)	C (22) –Sm (1) –C (26)	29.6 (1)
C (1) –Sm (1) –C (12)	133.7 (1)	O (1) –Sm (1) –C (12)	101.62 (8)
C (23) –Sm (1) –C (12)	91.4 (1)	C (13) –Sm (1) –C (12)	29.8 (1)
C (24) –Sm (1) –C (12)	92.7 (1)	C (9) –Sm (1) –C (12)	49.4 (1)
C (22) –Sm (1) –C (12)	117.9 (1)	C (26) –Sm (1) –C (12)	138.9 (1)
C (1) –Sm (1) –C (25)	105.9 (1)	O (1) –Sm (1) –C (25)	88.8 (1)
C (23) –Sm (1) –C (25)	49.3 (1)	C (13) –Sm (1) –C (25)	123.0 (1)
C (24) –Sm (1) –C (25)	29.6 (1)	C (9) –Sm (1) –C (25)	147.7 (1)
C (22) –Sm (1) –C (25)	49.2 (1)	C (26) –Sm (1) –C (25)	29.4 (1)
C (12) –Sm (1) –C (25)	119.9 (1)	C (1) –Sm (1) –C (11)	110.9 (1)
O (1) –Sm (1) –C (11)	81.40 (8)	C (23) –Sm (1) –C (11)	120.8 (1)
C (13) –Sm (1) –C (11)	48.4 (1)	C (24) –Sm (1) –C (11)	119.3 (1)
C (9) –Sm (1) –C (11)	48.3 (1)	C (22) –Sm (1) –C (11)	144.8 (1)
C (26) –Sm (1) –C (11)	167.6 (1)	C (12) –Sm (1) –C (11)	29.50 (8)
C (25) –Sm (1) –C (11)	140.8 (1)	C (1) –Sm (1) –C (10)	85.2 (1)
O (1) –Sm (1) –C (10)	92.46 (8)	C (23) –Sm (1) –C (10)	126.0 (1)
C (13) –Sm (1) –C (10)	49.0 (1)	C (24) –Sm (1) –C (10)	140.6 (1)
C (9) –Sm (1) –C (10)	29.5 (1)	C (22) –Sm (1) –C (10)	133.4 (1)
C (26) –Sm (1) –C (10)	158.7 (1)	C (12) –Sm (1) –C (10)	49.1 (1)
C (25) –Sm (1) –C (10)	169.0 (1)	C (11) –Sm (1) –C (10)	29.2 (1)
C (38) –O (1) –C (35)	107.5 (3)	C (38) –O (1) –Sm (1)	128.0 (2)
C (35) –O (1) –Sm (1)	123.9 (2)	C (2) –C (1) –Sm (1)	175.5 (3)
C (1) –C (2) –C (3)	177.9 (4)	C (8) –C (3) –C (4)	117.1 (3)
C (8) –C (3) –C (2)	121.2 (3)	C (4) –C (3) –C (2)	121.8 (3)
C (5) –C (4) –C (3)	120.1 (4)	C (5) –C (4) –H (4)	120.0
C (3) –C (4) –H (4)	120.0	C (6) –C (5) –C (4)	121.2 (4)
C (6) –C (5) –H (5)	119.4	C (4) –C (5) –H (5)	119.4
C (7) –C (6) –C (5)	119.6 (3)	C (7) –C (6) –H (6)	120.2
C (5) –C (6) –H (6)	120.2	C (6) –C (7) –C (8)	120.1 (4)
C (6) –C (7) –H (7)	119.9	C (8) –C (7) –H (7)	119.9
C (7) –C (8) –C (3)	121.9 (3)	C (7) –C (8) –H (8)	119.1
C (3) –C (8) –H (8)	119.1	C (10) –C (9) –C (13)	108.9 (3)
C (10) –C (9) –Sm (1)	79.1 (2)	C (13) –C (9) –Sm (1)	74.6 (2)
C (10) –C (9) –H (9)	125.6	C (13) –C (9) –H (9)	125.6
Sm (1) –C (9) –H (9)	113.0	C (9) –C (10) –C (11)	106.1 (3)
C (9) –C (10) –C (14)	127.3 (3)	C (11) –C (10) –C (14)	125.9 (3)
C (9) –C (10) –Sm (1)	71.5 (2)	C (11) –C (10) –Sm (1)	74.9 (2)
C (14) –C (10) –Sm (1)	126.2 (2)	C (10) –C (11) –C (12)	110.2 (3)
C (10) –C (11) –Sm (1)	75.9 (2)	C (12) –C (11) –Sm (1)	74.6 (2)
C (10) –C (11) –H (11)	124.9	C (12) –C (11) –H (11)	124.9
Sm (1) –C (11) –H (11)	116.4	C (13) –C (12) –C (11)	105.7 (3)
C (13) –C (12) –C (18)	128.3 (3)	C (11) –C (12) –C (18)	125.3 (3)
C (13) –C (12) –Sm (1)	72.2 (2)	C (11) –C (12) –Sm (1)	75.9 (2)
C (18) –C (12) –Sm (1)	124.2 (2)	C (9) –C (13) –C (12)	109.1 (3)
C (9) –C (13) –Sm (1)	75.3 (2)	C (12) –C (13) –Sm (1)	78.1 (2)
C (9) –C (13) –H (13)	125.5	C (12) –C (13) –H (13)	125.5
Sm (1) –C (13) –H (13)	113.4	C (16) –C (14) –C (10)	111.1 (3)
C (16) –C (14) –C (15)	109.9 (3)	C (10) –C (14) –C (15)	111.3 (3)
C (16) –C (14) –C (17)	108.9 (3)	C (10) –C (14) –C (17)	107.5 (3)
C (15) –C (14) –C (17)	108.1 (3)	C (14) –C (15) –H (15A)	109.5

C (14) -C (15) -H (15B)	109.5	H (15A) -C (15) -H (15B)	109.5
C (14) -C (15) -H (15C)	109.5	H (15A) -C (15) -H (15C)	109.5
H (15B) -C (15) -H (15C)	109.5	C (14) -C (16) -H (16A)	109.5
C (14) -C (16) -H (16B)	109.5	H (16A) -C (16) -H (16B)	109.5
C (14) -C (16) -H (16C)	109.5	H (16A) -C (16) -H (16C)	109.5
H (16B) -C (16) -H (16C)	109.5	C (14) -C (17) -H (17A)	109.5
C (14) -C (17) -H (17B)	109.5	H (17A) -C (17) -H (17B)	109.5
C (14) -C (17) -H (17C)	109.5	H (17A) -C (17) -H (17C)	109.5
H (17B) -C (17) -H (17C)	109.5	C (21) -C (18) -C (20)	108.7 (3)
C (21) -C (18) -C (12)	111.8 (3)	C (20) -C (18) -C (12)	110.6 (3)
C (21) -C (18) -C (19)	108.9 (3)	C (20) -C (18) -C (19)	109.1 (3)
C (12) -C (18) -C (19)	107.7 (3)	C (18) -C (19) -H (19A)	109.5
C (18) -C (19) -H (19B)	109.5	H (19A) -C (19) -H (19B)	109.5
C (18) -C (19) -H (19C)	109.5	H (19A) -C (19) -H (19C)	109.5
H (19B) -C (19) -H (19C)	109.5	C (18) -C (20) -H (20A)	109.5
C (18) -C (20) -H (20B)	109.5	H (20A) -C (20) -H (20B)	109.5
C (18) -C (20) -H (20C)	109.5	H (20A) -C (20) -H (20C)	109.5
H (20B) -C (20) -H (20C)	109.5	C (18) -C (21) -H (21A)	109.5
C (18) -C (21) -H (21B)	109.5	H (21A) -C (21) -H (21B)	109.5
C (18) -C (21) -H (21C)	109.5	H (21A) -C (21) -H (21C)	109.5
H (21B) -C (21) -H (21C)	109.5	C (23) -C (22) -C (26)	106.6 (3)
C (23) -C (22) -C (27)	125.9 (3)	C (26) -C (22) -C (27)	127.2 (3)
C (23) -C (22) -Sm (1)	72.6 (2)	C (26) -C (22) -Sm (1)	76.2 (2)
C (27) -C (22) -Sm (1)	122.1 (2)	C (24) -C (23) -C (22)	108.5 (3)
C (24) -C (23) -Sm (1)	75.8 (2)	C (22) -C (23) -Sm (1)	77.6 (2)
C (24) -C (23) -H (23)	125.7	C (22) -C (23) -H (23)	125.7
Sm (1) -C (23) -H (23)	113.2	C (23) -C (24) -C (25)	109.2 (3)
C (23) -C (24) -Sm (1)	74.2 (2)	C (25) -C (24) -Sm (1)	78.6 (2)
C (23) -C (24) -H (24)	125.4	C (25) -C (24) -H (24)	125.4
Sm (1) -C (24) -H (24)	114.0	C (24) -C (25) -C (26)	105.9 (3)
C (24) -C (25) -C (31)	126.1 (3)	C (26) -C (25) -C (31)	126.6 (3)
C (24) -C (25) -Sm (1)	71.8 (2)	C (26) -C (25) -Sm (1)	74.7 (2)
C (31) -C (25) -Sm (1)	128.7 (2)	C (22) -C (26) -C (25)	109.7 (3)
C (22) -C (26) -Sm (1)	74.2 (2)	C (25) -C (26) -Sm (1)	76.0 (2)
C (22) -C (26) -H (26)	125.1	C (25) -C (26) -H (26)	125.1
Sm (1) -C (26) -H (26)	116.5	C (30) -C (27) -C (29)	109.2 (4)
C (30) -C (27) -C (22)	111.2 (3)	C (29) -C (27) -C (22)	108.3 (3)
C (30) -C (27) -C (28)	108.3 (3)	C (29) -C (27) -C (28)	108.5 (3)
C (22) -C (27) -C (28)	111.3 (3)	C (27) -C (28) -H (28A)	109.5
C (27) -C (28) -H (28B)	109.5	H (28A) -C (28) -H (28B)	109.5
C (27) -C (28) -H (28C)	109.5	H (28A) -C (28) -H (28C)	109.5
H (28B) -C (28) -H (28C)	109.5	C (27) -C (29) -H (29A)	109.5
C (27) -C (29) -H (29B)	109.5	H (29A) -C (29) -H (29B)	109.5
C (27) -C (29) -H (29C)	109.5	H (29A) -C (29) -H (29C)	109.5
H (29B) -C (29) -H (29C)	109.5	C (27) -C (30) -H (30A)	109.5
C (27) -C (30) -H (30B)	109.5	H (30A) -C (30) -H (30B)	109.5
C (27) -C (30) -H (30C)	109.5	H (30A) -C (30) -H (30C)	109.5
H (30B) -C (30) -H (30C)	109.5	C (32) -C (31) -C (25)	111.7 (3)
C (32) -C (31) -C (33)	110.1 (4)	C (25) -C (31) -C (33)	112.2 (3)
C (32) -C (31) -C (34)	107.5 (3)	C (25) -C (31) -C (34)	106.6 (3)
C (33) -C (31) -C (34)	108.4 (3)	C (31) -C (32) -H (32A)	109.5
C (31) -C (32) -H (32B)	109.5	H (32A) -C (32) -H (32B)	109.5
C (31) -C (32) -H (32C)	109.5	H (32A) -C (32) -H (32C)	109.5
H (32B) -C (32) -H (32C)	109.5	C (31) -C (33) -H (33A)	109.5
C (31) -C (33) -H (33B)	109.5	H (33A) -C (33) -H (33B)	109.5
C (31) -C (33) -H (33C)	109.5	H (33A) -C (33) -H (33C)	109.5
H (33B) -C (33) -H (33C)	109.5	C (31) -C (34) -H (34A)	109.5
C (31) -C (34) -H (34B)	109.5	H (34A) -C (34) -H (34B)	109.5
C (31) -C (34) -H (34C)	109.5	H (34A) -C (34) -H (34C)	109.5
H (34B) -C (34) -H (34C)	109.5	O (1) -C (35) -C (36)	104.0 (3)

O (1) -C (35) -H (35A)	111.0	C (36) -C (35) -H (35A)	111.0
O (1) -C (35) -H (35B)	111.0	C (36) -C (35) -H (35B)	111.0
H (35A) -C (35) -H (35B)	109.0	C (37) -C (36) -C (35)	107.8 (4)
C (37) -C (36) -H (36A)	110.1	C (35) -C (36) -H (36A)	110.1
C (37) -C (36) -H (36B)	110.1	C (35) -C (36) -H (36B)	110.1
H (36A) -C (36) -H (36B)	108.5	C (38) -C (37) -C (36)	105.4 (4)
C (38) -C (37) -H (37A)	110.7	C (36) -C (37) -H (37A)	110.7
C (38) -C (37) -H (37B)	110.7	C (36) -C (37) -H (37B)	110.7
H (37A) -C (37) -H (37B)	108.8	C (37) -C (38) -O (1)	110.0 (4)
C (37) -C (38) -H (38A)	109.7	O (1) -C (38) -H (38A)	109.7
C (37) -C (38) -H (38B)	109.7	O (1) -C (38) -H (38B)	109.7
H (38A) -C (38) -H (38B)	108.2		

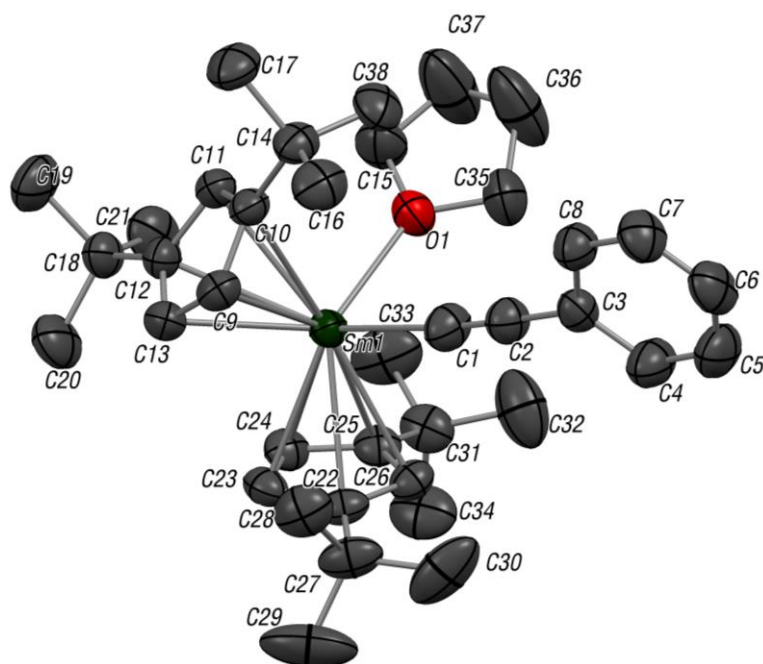


Figure S3. ORTEP of **1** with 50% probability ellipsoids (hydrogen atoms have been removed for clarity).

Table S1. Bond length (in Å) and angles (in °) for **2**.

Sm(1)–O(1)	2.312(4)	Sm(1)–O(2)#2	2.315(5)
Sm(1)–C(13)	2.670(7)	Sm(1)–C(12)	2.696(7)
Sm(1)–C(25)	2.698(7)	Sm(1)–C(26)	2.700(7)
Sm(1)–C(14)	2.720(7)	Sm(1)–C(27)	2.747(7)
Sm(1)–C(24)	2.754(7)	Sm(1)–C(10)	2.759(7)
Sm(1)–C(11)	2.766(7)	Sm(1)–C(23)	2.777(7)
O(1)–C(1)	1.244(8)	O(2)–C(1)	1.266(8)
O(2)–Sm(1)#2	2.315(5)	C(1)–C(2)	1.45(1)
C(2)–C(3)	1.21(1)	C(3)–C(4)	1.43(1)
C(4)–C(9)	1.38(1)	C(4)–C(5)	1.39(1)
C(5)–C(6)	1.38(1)	C(5)–H(5)	0.9500
C(6)–C(7)	1.40(1)	C(6)–H(6)	0.9500
C(7)–C(8)	1.38(1)	C(7)–H(7)	0.9500
C(8)–C(9)	1.38(1)	C(8)–H(8)	0.9500
C(9)–H(9)	0.9500	C(10)–C(11)	1.41(1)
C(10)–C(14)	1.42(1)	C(10)–H(10)	0.9500
C(11)–C(12)	1.42(1)	C(11)–C(15)	1.52(1)
C(12)–C(13)	1.39(1)	C(12)–H(12)	0.9500
C(13)–C(14)	1.40(1)	C(13)–H(13)	0.9500
C(14)–C(19)	1.52(1)	C(15)–C(16)	1.52(1)
C(15)–C(17)	1.53(1)	C(15)–C(18)	1.55(1)
C(16)–H(16A)	0.9800	C(16)–H(16B)	0.9800
C(16)–H(16C)	0.9800	C(17)–H(17A)	0.9800
C(17)–H(17B)	0.9800	C(17)–H(17C)	0.9800
C(18)–H(18A)	0.9800	C(18)–H(18B)	0.9800
C(18)–H(18C)	0.9800	C(19)–C(21)	1.54(1)
C(19)–C(20)	1.54(1)	C(19)–C(22)	1.54(1)
C(20)–H(20A)	0.9800	C(20)–H(20B)	0.9800
C(20)–H(20C)	0.9800	C(21)–H(21A)	0.9800
C(21)–H(21B)	0.9800	C(21)–H(21C)	0.9800
C(22)–H(22A)	0.9800	C(22)–H(22B)	0.9800
C(22)–H(22C)	0.9800	C(23)–C(24)	1.41(1)
C(23)–C(27)	1.43(1)	C(23)–H(23)	0.9500
C(24)–C(25)	1.41(1)	C(24)–C(28)	1.54(1)
C(25)–C(26)	1.41(1)	C(25)–H(25)	0.9500
C(26)–C(27)	1.41(1)	C(26)–H(26)	0.9500
C(27)–C(32)	1.53(1)	C(28)–C(29)	1.53(1)
C(28)–C(30)	1.53(1)	C(28)–C(31)	1.54(1)
C(29)–H(29A)	0.9800	C(29)–H(29B)	0.9800
C(29)–H(29C)	0.9800	C(30)–H(30A)	0.9800
C(30)–H(30B)	0.9800	C(30)–H(30C)	0.9800
C(31)–H(31A)	0.9800	C(31)–H(31B)	0.9800
C(31)–H(31C)	0.9800	C(32)–C(35)	1.52(1)
C(32)–C(33)	1.53(1)	C(32)–C(34)	1.54(1)
C(33)–H(33A)	0.9800	C(33)–H(33B)	0.9800
C(33)–H(33C)	0.9800	C(34)–H(34A)	0.9800
C(34)–H(34B)	0.9800	C(34)–H(34C)	0.9800
C(35)–H(35A)	0.9800	C(35)–H(35B)	0.9800
C(35)–H(35C)	0.9800	C(36A)–C(37A)	1.36(2)
C(36A)–C(41A)	1.41(2)	C(36A)–H(36A)	0.9500
C(37A)–C(38A)	1.35(2)	C(37A)–H(37A)	0.9500
C(38A)–C(39A)	1.42(2)	C(38A)–H(38A)	0.9500
C(39A)–C(40A)	1.36(2)	C(39A)–H(39A)	0.9500
C(40A)–C(41A)	1.37(2)	C(40A)–H(40A)	0.9500
C(41A)–C(42A)	1.52(2)	C(42A)–H(42A)	0.9800
C(42A)–H(42B)	0.9800	C(42A)–H(42C)	0.9800
C(36B)–C(37B)	1.3900	C(36B)–C(41B)	1.3900
C(36B)–H(36B)	0.9500	C(37B)–C(38B)	1.3900

C (37B) -H (37B)	0.9500	C (38B) -C (39B)	1.3900
C (38B) -H (38B)	0.9500	C (39B) -C (40B)	1.3900
C (39B) -H (39B)	0.9500	C (40B) -C (41B)	1.3900
C (40B) -C (42B)	1.50 (3)	C (41B) -H (41B)	0.9500
C (42B) -H (42D)	0.9800	C (42B) -H (42E)	0.9800
C (42B) -H (42F)	0.9800		
O (1) -Sm (1) -O (2) #2	91.3 (2)	O (1) -Sm (1) -C (13)	
116.2 (2)		O (1) -Sm (1) -C (12)	
O (2) #2-Sm (1) -C (13)	130.4 (2)	C (13) -Sm (1) -C (12)	
131.9 (2)		O (2) #2-Sm (1) -C (25)	
O (2) #2-Sm (1) -C (12)	100.9 (2)	C (12) -Sm (1) -C (25)	
30.1 (2)		O (2) #2-Sm (1) -C (26)	
O (1) -Sm (1) -C (25)	101.2 (2)	C (12) -Sm (1) -C (26)	
131.4 (2)		O (1) -Sm (1) -C (14)	
C (13) -Sm (1) -C (25)	85.2 (2)	C (13) -Sm (1) -C (14)	
104.6 (2)		C (25) -Sm (1) -C (14)	
O (1) -Sm (1) -C (26)	131.0 (2)	O (1) -Sm (1) -C (27)	
115.8 (2)		C (13) -Sm (1) -C (27)	
C (13) -Sm (1) -C (26)	77.6 (2)	C (25) -Sm (1) -C (27)	
84.5 (2)		C (14) -Sm (1) -C (27)	
C (25) -Sm (1) -C (26)	30.3 (2)	O (2) #2-Sm (1) -C (24)	
87.0 (2)		C (12) -Sm (1) -C (24)	
O (2) #2-Sm (1) -C (14)	129.5 (2)	C (26) -Sm (1) -C (24)	
30.1 (2)		C (27) -Sm (1) -C (24)	
C (12) -Sm (1) -C (14)	49.8 (2)	O (1) -Sm (1) -C (10)	
98.2 (2)		C (13) -Sm (1) -C (10)	
C (26) -Sm (1) -C (14)	102.5 (2)	C (25) -Sm (1) -C (10)	
130.2 (2)		C (14) -Sm (1) -C (10)	
O (2) #2-Sm (1) -C (27)	86.6 (2)	O (1) -Sm (1) -C (11)	
102.1 (2)		C (13) -Sm (1) -C (11)	
C (12) -Sm (1) -C (27)	97.1 (2)	C (25) -Sm (1) -C (11)	
49.8 (2)		C (14) -Sm (1) -C (11)	
C (26) -Sm (1) -C (27)	30.0 (2)	C (24) -Sm (1) -C (11)	
130.5 (2)			
O (1) -Sm (1) -C (24)	84.5 (2)		
107.7 (2)			
C (13) -Sm (1) -C (24)	114.9 (2)		
132.9 (2)			
C (25) -Sm (1) -C (24)	29.9 (2)		
49.5 (2)			
C (14) -Sm (1) -C (24)	122.3 (2)		
49.6 (2)			
O (1) -Sm (1) -C (10)	83.2 (2)		
99.6 (2)			
C (13) -Sm (1) -C (10)	49.2 (2)		
49.0 (2)			
C (25) -Sm (1) -C (10)	128.3 (2)		
126.7 (2)			
C (14) -Sm (1) -C (10)	30.1 (2)		
146.1 (2)			
C (24) -Sm (1) -C (10)	150.2 (2)		
108.4 (2)			
O (2) #2-Sm (1) -C (11)	83.7 (2)		
49.7 (2)			
C (12) -Sm (1) -C (11)	30.1 (2)		
133.4 (2)			
C (26) -Sm (1) -C (11)	114.3 (2)		
49.8 (2)			
C (27) -Sm (1) -C (11)	120.7 (2)		

162.9 (2)			
C (10) -Sm (1) -C (11)	29.6 (2)	O (1) -Sm (1) -C (23)	
100.4 (2)			
O (2) #2-Sm (1) -C (23)	82.5 (2)	C (13) -Sm (1) -C (23)	
126.7 (2)			
C (12) -Sm (1) -C (23)	127.1 (2)	C (25) -Sm (1) -C (23)	
49.1 (2)			
C (26) -Sm (1) -C (23)	49.2 (2)	C (14) -Sm (1) -C (23)	
147.3 (2)			
C (27) -Sm (1) -C (23)	29.9 (2)	C (24) -Sm (1) -C (23)	
29.6 (2)			
C (10) -Sm (1) -C (23)	175.8 (2)	C (11) -Sm (1) -C (23)	
148.2 (2)			
C (1) -O (1) -Sm (1)	166.4 (5)	C (1) -O (2) -Sm (1) #2	
156.4 (5)			
O (1) -C (1) -O (2)	124.9 (7)	O (1) -C (1) -C (2)	
118.3 (6)			
O (2) -C (1) -C (2)	116.9 (6)	C (3) -C (2) -C (1)	
175.4 (8)			
C (2) -C (3) -C (4)	179.0 (8)	C (9) -C (4) -C (5)	
120.0 (8)			
C (9) -C (4) -C (3)	119.6 (7)	C (5) -C (4) -C (3)	
120.3 (7)			
C (6) -C (5) -C (4)	120.2 (7)	C (6) -C (5) -H (5)	119.9
C (4) -C (5) -H (5)	119.9	C (5) -C (6) -C (7)	
120.3 (8)			
C (5) -C (6) -H (6)	119.8	C (7) -C (6) -H (6)	119.8
C (8) -C (7) -C (6)	119 (1)	C (8) -C (7) -H (7)	120.7
C (6) -C (7) -H (7)	120.7	C (7) -C (8) -C (9)	
121.5 (8)			
C (7) -C (8) -H (8)	119.3	C (9) -C (8) -H (8)	119.3
C (8) -C (9) -C (4)	119.3 (8)	C (8) -C (9) -H (9)	120.3
C (4) -C (9) -H (9)	120.3	C (11) -C (10) -C (14)	
109.3 (6)			
C (11) -C (10) -Sm (1)	75.5 (4)	C (14) -C (10) -Sm (1)	
73.4 (4)			
C (11) -C (10) -H (10)	125.3	C (14) -C (10) -H (10)	125.3
Sm (1) -C (10) -H (10)	117.6	C (10) -C (11) -C (12)	
106.3 (6)			
C (10) -C (11) -C (15)	125.6 (6)	C (12) -C (11) -C (15)	
126.6 (6)			
C (10) -C (11) -Sm (1)	74.9 (4)	C (12) -C (11) -Sm (1)	
72.2 (4)			
C (15) -C (11) -Sm (1)	129.0 (5)	C (13) -C (12) -C (11)	
108.7 (6)			
C (13) -C (12) -Sm (1)	73.9 (4)	C (11) -C (12) -Sm (1)	
77.7 (4)			
C (13) -C (12) -H (12)	125.7	C (11) -C (12) -H (12)	125.7
Sm (1) -C (12) -H (12)	114.9	C (12) -C (13) -C (14)	
109.3 (6)			
C (12) -C (13) -Sm (1)	76.0 (4)	C (14) -C (13) -Sm (1)	
76.9 (4)			
C (12) -C (13) -H (13)	125.4	C (14) -C (13) -H (13)	125.4
Sm (1) -C (13) -H (13)	113.9	C (13) -C (14) -C (10)	
106.4 (7)			
C (13) -C (14) -C (19)	126.0 (6)	C (10) -C (14) -C (19)	
126.4 (6)			
C (13) -C (14) -Sm (1)	73.0 (4)	C (10) -C (14) -Sm (1)	
76.5 (4)			
C (19) -C (14) -Sm (1)	125.7 (4)	C (16) -C (15) -C (11)	
111.3 (6)			

C (16) -C (15) -C (17)	110.2 (7)	C (11) -C (15) -C (17)	
111.6 (6)			
C (16) -C (15) -C (18)	108.2 (6)	C (11) -C (15) -C (18)	
107.7 (7)			
C (17) -C (15) -C (18)	107.8 (6)	C (15) -C (16) -H (16A)	109.5
C (15) -C (16) -H (16B)	109.5	H (16A) -C (16) -H (16B)	109.5
C (15) -C (16) -H (16C)	109.5	H (16A) -C (16) -H (16C)	109.5
H (16B) -C (16) -H (16C)	109.5	C (15) -C (17) -H (17A)	109.5
C (15) -C (17) -H (17B)	109.5	H (17A) -C (17) -H (17B)	109.5
C (15) -C (17) -H (17C)	109.5	H (17A) -C (17) -H (17C)	109.5
H (17B) -C (17) -H (17C)	109.5	C (15) -C (18) -H (18A)	109.5
C (15) -C (18) -H (18B)	109.5	H (18A) -C (18) -H (18B)	109.5
C (15) -C (18) -H (18C)	109.5	H (18A) -C (18) -H (18C)	109.5
H (18B) -C (18) -H (18C)	109.5	C (14) -C (19) -C (21)	
111.0 (6)			
C (14) -C (19) -C (20)	112.1 (6)	C (21) -C (19) -C (20)	
108.7 (6)			
C (14) -C (19) -C (22)	106.7 (6)	C (21) -C (19) -C (22)	
110.0 (6)			
C (20) -C (19) -C (22)	108.2 (6)	C (19) -C (20) -H (20A)	109.5
C (19) -C (20) -H (20B)	109.5	H (20A) -C (20) -H (20B)	109.5
C (19) -C (20) -H (20C)	109.5	H (20A) -C (20) -H (20C)	109.5
H (20B) -C (20) -H (20C)	109.5	C (19) -C (21) -H (21A)	109.5
C (19) -C (21) -H (21B)	109.5	H (21A) -C (21) -H (21B)	109.5
C (19) -C (21) -H (21C)	109.5	H (21A) -C (21) -H (21C)	109.5
H (21B) -C (21) -H (21C)	109.5	C (19) -C (22) -H (22A)	109.5
C (19) -C (22) -H (22B)	109.5	H (22A) -C (22) -H (22B)	109.5
C (19) -C (22) -H (22C)	109.5	H (22A) -C (22) -H (22C)	109.5
H (22B) -C (22) -H (22C)	109.5	C (24) -C (23) -C (27)	
108.5 (7)			
C (24) -C (23) -Sm (1)	74.3 (4)	C (27) -C (23) -Sm (1)	
73.9 (4)			
C (24) -C (23) -H (23)	125.7	C (27) -C (23) -H (23)	125.7
Sm (1) -C (23) -H (23)	118.0	C (25) -C (24) -C (23)	
107.6 (6)			
C (25) -C (24) -C (28)	126.0 (7)	C (23) -C (24) -C (28)	
125.3 (6)			
C (25) -C (24) -Sm (1)	72.8 (4)	C (23) -C (24) -Sm (1)	
76.1 (4)			
C (28) -C (24) -Sm (1)	126.1 (4)	C (24) -C (25) -C (26)	
108.3 (7)			
C (24) -C (25) -Sm (1)	77.2 (4)	C (26) -C (25) -Sm (1)	
74.9 (4)			
C (24) -C (25) -H (25)	125.8	C (26) -C (25) -H (25)	125.8
Sm (1) -C (25) -H (25)	114.2	C (25) -C (26) -C (27)	
108.7 (6)			
C (25) -C (26) -Sm (1)	74.8 (4)	C (27) -C (26) -Sm (1)	
76.8 (4)			
C (25) -C (26) -H (26)	125.7	C (27) -C (26) -H (26)	125.7
Sm (1) -C (26) -H (26)	114.9	C (26) -C (27) -C (23)	
106.9 (7)			
C (26) -C (27) -C (32)	126.3 (7)	C (23) -C (27) -C (32)	
125.9 (7)			
C (26) -C (27) -Sm (1)	73.2 (4)	C (23) -C (27) -Sm (1)	
76.2 (4)			
C (32) -C (27) -Sm (1)	124.8 (5)	C (29) -C (28) -C (30)	
109.9 (6)			
C (29) -C (28) -C (24)	111.4 (6)	C (30) -C (28) -C (24)	
111.2 (6)			
C (29) -C (28) -C (31)	108.2 (6)	C (30) -C (28) -C (31)	
108.1 (6)			

C (24) -C (28) -C (31)	108.0 (6)	C (28) -C (29) -H (29A)	109.5
C (28) -C (29) -H (29B)	109.5	H (29A) -C (29) -H (29B)	109.5
C (28) -C (29) -H (29C)	109.5	H (29A) -C (29) -H (29C)	109.5
H (29B) -C (29) -H (29C)	109.5	C (28) -C (30) -H (30A)	109.5
C (28) -C (30) -H (30B)	109.5	H (30A) -C (30) -H (30B)	109.5
C (28) -C (30) -H (30C)	109.5	H (30A) -C (30) -H (30C)	109.5
H (30B) -C (30) -H (30C)	109.5	C (28) -C (31) -H (31A)	109.5
C (28) -C (31) -H (31B)	109.5	H (31A) -C (31) -H (31B)	109.5
C (28) -C (31) -H (31C)	109.5	H (31A) -C (31) -H (31C)	109.5
H (31B) -C (31) -H (31C)	109.5	C (35) -C (32) -C (33)	
109.9 (8)			
C (35) -C (32) -C (27)	111.4 (6)	C (33) -C (32) -C (27)	
109.8 (7)			
C (35) -C (32) -C (34)	109.1 (7)	C (33) -C (32) -C (34)	
109.4 (7)			
C (27) -C (32) -C (34)	107.1 (7)	C (32) -C (33) -H (33A)	109.5
C (32) -C (33) -H (33B)	109.5	H (33A) -C (33) -H (33B)	109.5
C (32) -C (33) -H (33C)	109.5	H (33A) -C (33) -H (33C)	109.5
H (33B) -C (33) -H (33C)	109.5	C (32) -C (34) -H (34A)	109.5
C (32) -C (34) -H (34B)	109.5	H (34A) -C (34) -H (34B)	109.5
C (32) -C (34) -H (34C)	109.5	H (34A) -C (34) -H (34C)	109.5
H (34B) -C (34) -H (34C)	109.5	C (32) -C (35) -H (35A)	109.5
C (32) -C (35) -H (35B)	109.5	H (35A) -C (35) -H (35B)	109.5
C (32) -C (35) -H (35C)	109.5	H (35A) -C (35) -H (35C)	109.5
H (35B) -C (35) -H (35C)	109.5	C (37A) -C (36A) -C (41A)	
120 (1)			
C (37A) -C (36A) -H (36A)	120.0	C (41A) -C (36A) -H (36A)	120.0
C (38A) -C (37A) -C (36A)	120 (2)	C (38A) -C (37A) -H (37A)	120.2
C (36A) -C (37A) -H (37A)	120.2	C (37A) -C (38A) -C (39A)	
122 (2)			
C (37A) -C (38A) -H (38A)	119.2	C (39A) -C (38A) -H (38A)	119.2
C (40A) -C (39A) -C (38A)	119 (2)	C (40A) -C (39A) -H (39A)	120.7
C (38A) -C (39A) -H (39A)	120.7	C (39A) -C (40A) -C (41A)	
120 (2)			
C (39A) -C (40A) -H (40A)	119.8	C (41A) -C (40A) -H (40A)	119.8
C (40A) -C (41A) -C (36A)	120 (1)	C (40A) -C (41A) -C (42A)	
117 (2)			
C (36A) -C (41A) -C (42A)	123 (2)	C (41A) -C (42A) -H (42A)	109.5
C (41A) -C (42A) -H (42B)	109.5	H (42A) -C (42A) -H (42B)	109.5
C (41A) -C (42A) -H (42C)	109.5	H (42A) -C (42A) -H (42C)	109.5
H (42B) -C (42A) -H (42C)	109.5	C (37B) -C (36B) -C (41B)	120.0
C (37B) -C (36B) -H (36B)	120.0	C (41B) -C (36B) -H (36B)	120.0
C (38B) -C (37B) -C (36B)	120.0	C (38B) -C (37B) -H (37B)	120.0
C (36B) -C (37B) -H (37B)	120.0	C (37B) -C (38B) -C (39B)	120.0
C (37B) -C (38B) -H (38B)	120.0	C (39B) -C (38B) -H (38B)	120.0
C (40B) -C (39B) -C (38B)	120.0	C (40B) -C (39B) -H (39B)	120.0
C (38B) -C (39B) -H (39B)	120.0	C (39B) -C (40B) -C (41B)	120.0
C (39B) -C (40B) -C (42B)	109 (3)	C (41B) -C (40B) -C (42B)	
131 (3)			
C (40B) -C (41B) -C (36B)	120.0	C (40B) -C (41B) -H (41B)	120.0
C (36B) -C (41B) -H (41B)	120.0	C (40B) -C (42B) -H (42D)	109.5
C (40B) -C (42B) -H (42E)	109.5	H (42D) -C (42B) -H (42E)	109.5
C (40B) -C (42B) -H (42F)	109.5	H (42D) -C (42B) -H (42F)	109.5
H (42E) -C (42B) -H (42F)	109.5		

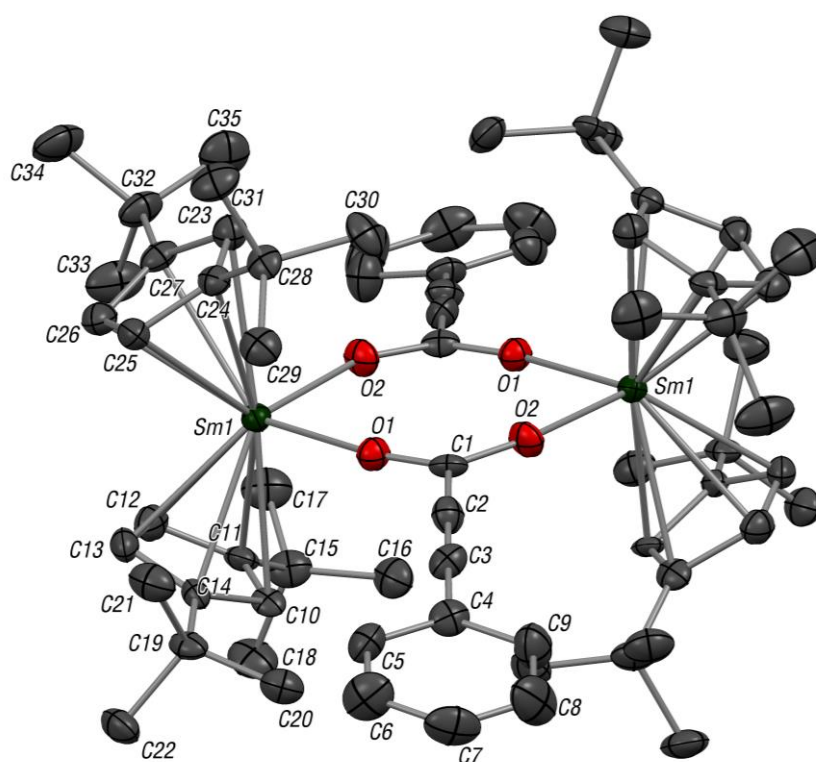


Figure S4. ORTEP of **2** with 50% probability ellipsoids (hydrogen atoms and one molecule of toluene have been removed for clarity).

III. References

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