Supporting Material

Contents

1. Crystallographic data of the compounds and selected bond distances

Crystallographic data of the compounds **1** and **2** are given in Table S1. Selected bond distances d(M-O) and d(M-C_π) [pm] are presented in Table S2 and Table S3.

Table S1. Summarized crystallographic data of the compounds {[((18C6-κ^6*O*)Rb$^+$)$_2$-*μ*-(*η*:*η*-L_{DOPT}^{2-})][((18C6-κ^6*O*)Rb$^+$)$_2$-*μ*-(*η'*:*η'*-L_{DOPT}^{2-})](THF$_{solv}$)$_2$} (**1**) and [*μ*-(*η*:*η*-L_{DOPT}^{2-})$_{0.5}$((tetraglyme-κ^4*O*)(tetraglyme-κ^2*O*)Cs$^+$)$_2$-*μ*-(*η'*:*η'*-L_{DOPT}^{2-})$_{0.5}$]$_n$ (**2**).

Molecule	**1**	**2**
crystal size	0.267x0.110x0.083 mm^3	0.354x0.352x0.344 mm^3
crystal system	triclinic	triclinic
space group, Z	$P\bar{1}$ (2), Z = 2	$P\bar{1}$ (2), Z = 2
unit cell	a = 10.9805(18)Å b = 12.636(2) Å c = 22.734(4) Å α = 98.144(3)° β = 97.696(3)° γ = 109.057(3)°	a = 11.1914(14)Å b = 11.5224(14) Å c = 20.297(2) Å α = 74.5693(19)° β = 77.5731(19)° γ = 73.379(2)°
V	2896.1(8) Å^3	2390.0(5) Å^3
empirical formula	$C_{62}H_{80}Rb_2O_{14}$	$C_{50}H_{56}Cs_2O_{10}$
formula weight	1220.20 g/mol	1082.76 g/mol
density (calcd)	1.399 g/cm^3	1.505 g/cm^3
absorption coefficient	1.755 mm^{-1}	1.581 mm^{-1}
F(000)	1276	1092
*Θ*range	2.997 to 30.507°	3.309 to 36.313°
index ranges	-15/15, -18/18, -32/32	-18/18, -17/19, -33/33
reflections collected	81216	92073
independent reflections	17652 [*R*(int) = 0.1152]	22883 [*R*(int) = 0.0308]
completeness	99.8 %	99.8 %
obsd	[*I*>2σ (*I*)]	[*I*>2σ (*I*)]
reflections used for refinement	10491	17653
absorption correction	semi-empirical from equivalents	semi-empirical from equivalents
largest difference peak and hole	0.728 and -0.707 e Å^3	3.202 and -2.589 e Å^3
treatment of hydrogen atoms	H atoms were positioned geometrically	H atoms were positioned geometrically
refined parameters	703	582
GOF on F^2	0.854	1.072
wR2 (all data)	0.1123	0.1101
R1 [*I*>2σ (*I*)]	0.0494	0.0431

Table S2. Distances d(M-O) and d(M-C_π) [pm] in the solvent-shared ion pair of the dianionic DOPT structure **1** in (a) *η*:*η*- and (b) η′:η′ coordination of Rb^+. The mirror symmetry along the central C-C bond is highlighted in transparent depiction. Hydrogen atoms are omitted for clarity.

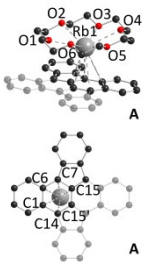
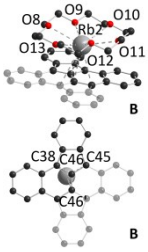
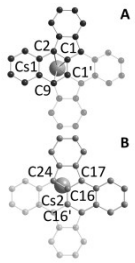
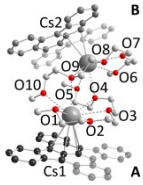
(a)		Rb1	O1	287.81(26)
		Rb1	O5	289.82(24)
		Rb1	O3	290.90(23)
		Rb1	O6	299.32(24)
		Rb1	O4	301.98(22)
		Rb1	O2	310.60(23)
		Rb1	C15'	316.47(27)
		Rb1	C15	322.06(27)
		Rb1	C14	326.34(32)
		Rb1	C7	338.37(32)
		Rb1	C1	344.45(37)
		Rb1	C6	351.57(37)
		C15	C15'	138.8(6)
		C14	C15'	139.7(4)
		C7	C15	140.8(4)
(b)		Rb2	O9	287.44(18)
		Rb2	O13	289.33(24)
		Rb2	O11	293.53(26)
		Rb2	O12	298.82(22)
		Rb2	O8	299.14(24)
		Rb2	O10	305.09(25)
		Rb2	C46	302.05(44)
		Rb2	C38	319.39(30)
		Rb2	C46'	323.83(47)
		Rb2	C45	349.94(38)
		C46	C46'	133.5(8)
		C45	C46	142.6(5)
		C38	C46	144.5(5)

Table S3. Distances d(M-O) and d(M-C_π) [pm] in the solvent-shared ion pair of the dianionic DOPT structure **2** in (a) η:η- and (b) η′:η′ coordination of Cs^+. The mirror symmetry along the central C-C bond is highlighted in transparent depiction. Hydrogen atoms are omitted for clarity.

(a)		Cs1	C1	322.46(3)
		Cs1	C1'	331.01(4)
		Cs1	C2	340.58(3)
		Cs1	C9	359.86(3)
		C1A	C1A'	137.11(1)
		C1A'	C9	141.78(2)
		C1A	C2	141.84(1)
		Cs2	C24	321.98(3)
		Cs2	C16	324.92(3)
		C16A	C16A'	137.60(1)
		C16A	C17	142.65(2)
		C16A	C24	142.83(2)
(b)		Cs1	O2	301.47(3)
		Cs1	O10	307.26(3)
		Cs1	O1	313.32(4)
		Cs1	O9	314.29(4)
		Cs1	O3	314.37(4)
		Cs1	O4	328.24(3)
		Cs2	O7	309.66(4)
		Cs2	O4	310.23(4)
		Cs2	O5	314.52(2)
		Cs2	O6	315.35(2)
		Cs2	O8	316.49(3)
		Cs2	O9	325.83(4)

1.1 Crystal structure data of DOPT Dirubidium bis(18-crown-6-ether) **1**

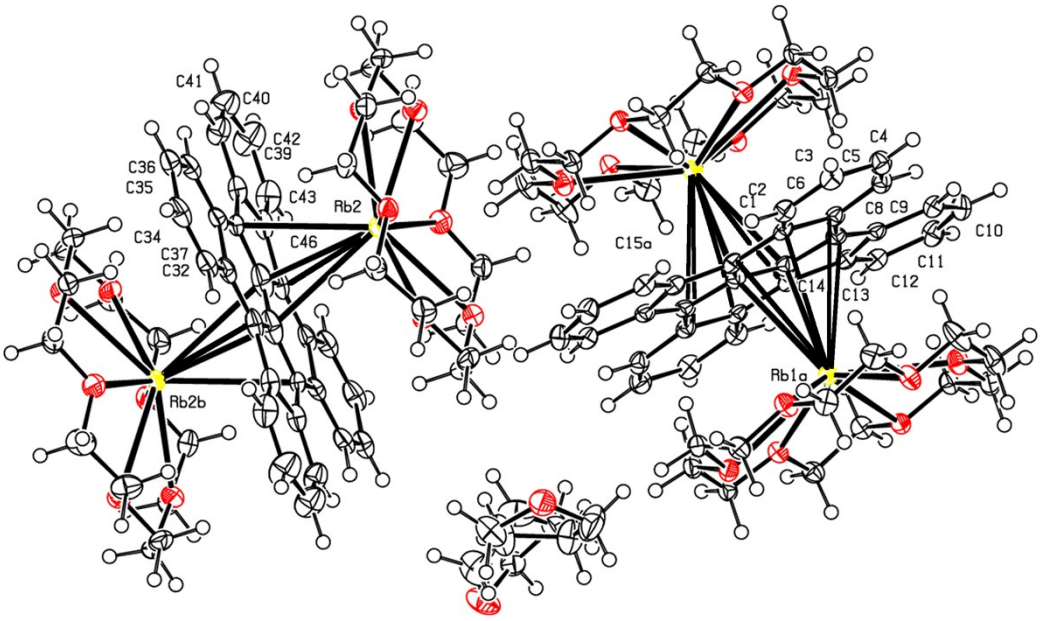


Figure S1. ORTEP drawing of the structure of **1** at the 50% probability level, with two differently coordinated triple-decker units in the crystal lattice (left: *η*:*η* coordination; right: η′:η′ coordination; bottom: interstitial THF molecules).

Table S4. Atomic coordinates and equivalent isotropic displacement parameters (Å^2). U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
C(1)	1.1943(3)	0.4337(3)	0.5244(1)	0.019(1)
C(2)	1.3073(3)	0.4029(3)	0.5273(1)	0.024(1)
C(3)	1.3416(3)	0.3460(3)	0.5706(1)	0.025(1)
C(4)	1.2641(3)	0.3179(3)	0.6137(1)	0.026(1)
C(5)	1.1545(3)	0.3481(3)	0.6133(1)	0.023(1)
C(6)	1.1159(3)	0.4068(3)	0.5703(1)	0.020(1)
C(7)	0.9995(3)	0.4377(3)	0.5697(1)	0.020(1)
C(8)	0.8970(3)	0.4271(3)	0.6043(1)	0.020(1)
C(9)	0.8744(3)	0.3835(3)	0.6569(1)	0.023(1)
C(10)	0.7639(3)	0.3814(3)	0.6802(1)	0.026(1)
C(11)	0.6728(3)	0.4235(3)	0.6522(1)	0.027(1)
C(12)	0.6926(3)	0.4689(3)	0.6009(1)	0.023(1)
C(13)	0.8031(3)	0.4722(3)	0.5753(1)	0.020(1)
C(14)	0.8467(3)	0.5111(3)	0.5227(1)	0.019(1)
C(15)	0.9628(3)	0.4882(3)	0.5222(1)	0.020(1)
C(16)	0.8900(3)	0.0919(3)	0.4997(1)	0.023(1)
C(17)	0.8021(3)	0.1341(3)	0.5337(1)	0.024(1)
C(18)	0.5976(3)	0.1622(3)	0.5236(1)	0.025(1)
C(19)	0.4799(3)	0.1496(3)	0.4784(1)	0.025(1)
C(20)	0.4100(3)	0.2254(3)	0.3966(2)	0.029(1)
C(21)	0.4587(3)	0.3091(3)	0.3573(2)	0.030(1)
C(22)	0.5884(3)	0.3476(3)	0.2827(2)	0.037(1)
C(23)	0.6719(3)	0.3036(3)	0.2467(2)	0.035(1)
C(24)	0.8735(3)	0.2718(3)	0.2550(1)	0.026(1)
C(25)	0.9916(3)	0.2813(3)	0.2997(1)	0.024(1)
C(26)	1.0594(3)	0.2078(3)	0.3821(1)	0.023(1)
C(27)	1.0124(3)	0.1257(3)	0.4225(1)	0.023(1)
C(28)	0.1933(4)	-0.0102(4)	0.2290(2)	0.054(1)
C(29)	0.1548(4)	-0.1114(4)	0.2602(2)	0.051(1)
C(30)	0.0530(4)	-0.0911(3)	0.2949(2)	0.036(1)
C(31)	-0.0062(3)	-0.0260(3)	0.2567(2)	0.032(1)

C(32)	0.6899(3)	0.4442(3)	-0.0367(1)	0.028(1)
C(33)	0.8068(3)	0.4367(3)	-0.0527(2)	0.032(1)
C(34)	0.8129(3)	0.3394(3)	-0.0861(2)	0.034(1)
C(35)	0.7001(3)	0.2424(3)	-0.1055(2)	0.032(1)
C(36)	0.5826(3)	0.2447(3)	-0.0904(1)	0.029(1)
C(37)	0.5713(3)	0.3424(3)	-0.0566(1)	0.026(1)
C(38)	0.4506(3)	0.3495(3)	-0.0423(1)	0.023(1)
C(39)	0.3159(3)	0.2771(3)	-0.0508(1)	0.027(1)
C(40)	0.2513(3)	0.1628(3)	-0.0796(2)	0.034(1)
C(41)	0.1186(4)	0.1105(4)	-0.0823(2)	0.045(1)
C(42)	0.0455(4)	0.1696(4)	-0.0562(2)	0.048(1)
C(43)	0.1044(3)	0.2818(4)	-0.0275(2)	0.038(1)
C(44)	0.2388(3)	0.3399(3)	-0.0234(1)	0.029(1)
C(45)	0.3222(3)	0.4553(3)	0.0033(1)	0.028(1)
C(46)	0.4453(3)	0.4542(4)	-0.0097(2)	0.040(1)
C(47)	0.6247(4)	0.6419(3)	0.2041(2)	0.033(1)
C(48)	0.7556(4)	0.6285(3)	0.2148(2)	0.035(1)
C(49)	0.8896(3)	0.5411(3)	0.1700(2)	0.034(1)
C(50)	0.8899(3)	0.4580(3)	0.1162(2)	0.031(1)
C(51)	0.7991(3)	0.2611(3)	0.0709(1)	0.029(1)
C(52)	0.7064(3)	0.1497(3)	0.0794(2)	0.030(1)
C(53)	0.4836(3)	0.0515(3)	0.0801(2)	0.030(1)
C(54)	0.3501(3)	0.0598(3)	0.0678(2)	0.031(1)
C(55)	0.2149(3)	0.1551(3)	0.1034(2)	0.038(1)
C(56)	0.2081(4)	0.2414(3)	0.1540(2)	0.039(1)
C(57)	0.2969(3)	0.4364(3)	0.1985(1)	0.029(1)
C(58)	0.3978(3)	0.5460(3)	0.1944(2)	0.030(1)
C(59)	-0.3571(5)	-0.0638(4)	0.3533(2)	0.059(1)
C(60)	-0.4813(4)	-0.0454(4)	0.3292(2)	0.045(1)
C(61)	-0.4804(4)	-0.0536(5)	0.2616(2)	0.054(1)
C(62)	-0.3474(4)	-0.0547(4)	0.2557(2)	0.046(1)
O(1)	0.9277(2)	0.1610(2)	0.4564(1)	0.021(1)
O(2)	0.6830(2)	0.1189(2)	0.4933(1)	0.022(1)
O(3)	0.5183(2)	0.2281(2)	0.4392(1)	0.025(1)
O(4)	0.5402(2)	0.2717(2)	0.3222(1)	0.026(1)
O(5)	0.7872(2)	0.3099(2)	0.2862(1)	0.026(1)
O(6)	0.9504(2)	0.2070(2)	0.3403(1)	0.020(1)
O(7)	0.1026(3)	0.0474(2)	0.2365(1)	0.045(1)
O(8)	0.5244(2)	0.5367(2)	0.2051(1)	0.028(1)
O(9)	0.7626(2)	0.5482(2)	0.1660(1)	0.030(1)
O(10)	0.8005(2)	0.3481(2)	0.1181(1)	0.026(1)
O(11)	0.5774(2)	0.1522(2)	0.0707(1)	0.027(1)
O(12)	0.3406(2)	0.1435(2)	0.1140(1)	0.029(1)
O(13)	0.2977(2)	0.3497(2)	0.1520(1)	0.031(1)
O(14)	-0.3069(2)	-0.1026(2)	0.3041(1)	0.037(1)
Rb(1)	0.7810(1)	0.2943(1)	0.4118(1)	0.019(1)
Rb(2)	0.5284(1)	0.3666(1)	0.1001(1)	0.022(1)

Table S5. Bond lengths [Å] and angles [°].

C(1)-C(2)	1.412(4)	C(1)-C(6)	1.449(4)
C(1)-C(14)#1	1.457(4)	C(1)-Rb(1)#1	3.444(3)
C(2)-C(3)	1.380(4)	C(3)-C(4)	1.391(4)
C(4)-C(5)	1.376(4)	C(5)-C(6)	1.408(4)
C(6)-C(7)	1.452(4)	C(6)-Rb(1)#1	3.516(3)
C(7)-C(15)	1.408(4)	C(7)-C(8)	1.439(4)
C(7)-Rb(1)#1	3.384(3)	C(8)-C(9)	1.404(4)
C(8)-C(13)	1.459(4)	C(9)-C(10)	1.381(4)
C(10)-C(11)	1.400(4)	C(11)-C(12)	1.383(4)
C(12)-C(13)	1.406(4)	C(13)-C(14)	1.438(4)
C(14)-C(15)	1.397(4)	C(14)-C(1)#1	1.457(4)
C(14)-Rb(1)	3.263(3)	C(15)-C(15)#1	1.388(6)
C(15)-Rb(1)	3.165(3)	C(15)-Rb(1)#1	3.220(3)
C(16)-O(1)	1.422(3)	C(16)-C(17)	1.494(4)
C(17)-O(2)	1.434(3)	C(17)-Rb(1)	3.683(3)
C(18)-O(2)	1.434(4)	C(18)-C(19)	1.489(4)
C(18)-Rb(1)	3.699(3)	C(19)-O(3)	1.425(3)
C(20)-O(3)	1.418(4)	C(20)-C(21)	1.494(5)
C(21)-O(4)	1.421(4)	C(21)-Rb(1)	3.662(3)
C(22)-O(4)	1.430(4)	C(22)-C(23)	1.492(5)
C(22)-Rb(1)	3.655(3)	C(23)-O(5)	1.422(4)
C(24)-O(5)	1.419(4)	C(24)-C(25)	1.496(4)
C(25)-O(6)	1.422(3)	C(25)-Rb(1)	3.684(3)
C(26)-O(6)	1.421(3)	C(26)-C(27)	1.494(4)
C(26)-Rb(1)	3.678(3)	C(27)-O(1)	1.424(4)
C(28)-O(7)	1.427(5)	C(28)-C(29)	1.518(6)
C(29)-C(30)	1.516(5)	C(30)-C(31)	1.501(5)
C(31)-O(7)	1.429(4)	C(32)-C(33)	1.408(5)
C(32)-C(45)#2	1.439(5)	C(32)-C(37)	1.462(4)
C(33)-C(34)	1.377(5)	C(34)-C(35)	1.393(5)
C(35)-C(36)	1.387(5)	C(36)-C(37)	1.410(5)
C(37)-C(38)	1.434(5)	C(37)-Rb(2)	3.640(3)
C(38)-C(39)	1.432(5)	C(38)-C(46)	1.445(5)
C(38)-Rb(2)	3.194(3)	C(39)-C(40)	1.399(5)
C(39)-C(44)	1.475(5)	C(39)-Rb(2)	3.678(3)
C(40)-C(41)	1.378(5)	C(41)-C(42)	1.396(6)
C(42)-C(43)	1.371(6)	C(43)-C(44)	1.400(5)
C(44)-C(45)	1.435(5)	C(45)-C(46)	1.426(5)
C(45)-C(32)#2	1.440(5)	C(45)-Rb(2)	3.499(3)
C(45)-Rb(2)#2	3.662(3)	C(46)-C(46)#2	1.335(8)
C(46)-Rb(2)	3.021(3)	C(46)-Rb(2)#2	3.238(3)
C(47)-O(8)	1.428(4)	C(47)-C(48)	1.493(5)
C(47)-Rb(2)	3.661(4)	C(48)-O(9)	1.422(4)
C(49)-O(9)	1.418(4)	C(49)-C(50)	1.496(5)
C(50)-O(10)	1.425(4)	C(50)-Rb(2)	3.705(3)
C(51)-O(10)	1.417(4)	C(51)-C(52)	1.499(5)
C(51)-Rb(2)	3.731(3)	C(52)-O(11)	1.415(4)
C(53)-O(11)	1.415(4)	C(53)-C(54)	1.496(5)
C(54)-O(12)	1.421(4)	C(54)-Rb(2)	3.641(3)
C(55)-O(12)	1.428(4)	C(55)-C(56)	1.496(5)
C(55)-Rb(2)	3.621(4)	C(56)-O(13)	1.411(4)
C(57)-O(13)	1.413(4)	C(57)-C(58)	1.488(5)
C(58)-O(8)	1.424(4)	C(58)-Rb(2)	3.655(3)
C(59)-O(14)	1.406(5)	C(59)-C(60)	1.503(5)
C(60)-C(61)	1.528(5)	C(61)-C(62)	1.488(5)
C(62)-O(14)	1.417(4)	O(1)-Rb(1)	2.8781(19)
O(2)-Rb(1)	3.106(2)	O(3)-Rb(1)	2.909(2)
O(4)-Rb(1)	3.020(2)	O(5)-Rb(1)	2.898(2)
O(6)-Rb(1)	2.993(2)	O(8)-Rb(2)	2.992(2)
O(9)-Rb(2)	2.875(2)	O(10)-Rb(2)	3.050(2)
O(11)-Rb(2)	2.935(2)	O(12)-Rb(2)	2.988(2)
O(13)-Rb(2)	2.894(2)	Rb(1)-C(15)#1	3.220(3)
Rb(1)-C(7)#1	3.384(3)	Rb(1)-C(1)#1	3.444(3)
Rb(1)-C(6)#1	3.516(3)	Rb(2)-C(46)#2	3.238(3)

C(2)-C(1)-C(6)	117.5(3)	C(2)-C(1)-C(14)#1	123.2(3)
C(6)-C(1)-C(14)#1	119.4(2)	C(2)-C(1)-Rb(1)#1	120.3(2)
C(6)-C(1)-Rb(1)#1	80.76(17)	C(14)#1-C(1)-Rb(1)#1	70.58(16)
C(3)-C(2)-C(1)	122.5(3)	C(2)-C(3)-C(4)	119.8(3)
C(5)-C(4)-C(3)	119.7(3)	C(4)-C(5)-C(6)	122.6(3)
C(5)-C(6)-C(1)	117.9(3)	C(5)-C(6)-C(7)	122.4(3)
C(1)-C(6)-C(7)	119.6(2)	C(5)-C(6)-Rb(1)#1	124.7(2)
C(1)-C(6)-Rb(1)#1	75.23(17)	C(7)-C(6)-Rb(1)#1	72.81(16)
C(15)-C(7)-C(8)	103.6(2)	C(15)-C(7)-C(6)	118.4(3)
C(8)-C(7)-C(6)	137.9(3)	C(15)-C(7)-Rb(1)#1	71.25(17)
C(8)-C(7)-Rb(1)#1	114.84(19)	C(6)-C(7)-Rb(1)#1	82.99(17)
C(9)-C(8)-C(7)	132.7(3)	C(9)-C(8)-C(13)	118.9(3)
C(7)-C(8)-C(13)	108.4(2)	C(10)-C(9)-C(8)	120.5(3)
C(9)-C(10)-C(11)	120.8(3)	C(12)-C(11)-C(10)	120.5(3)
C(11)-C(12)-C(13)	120.8(3)	C(12)-C(13)-C(14)	132.8(3)
C(12)-C(13)-C(8)	118.5(3)	C(14)-C(13)-C(8)	108.6(2)
C(15)-C(14)-C(13)	103.8(2)	C(15)-C(14)-C(1)#1	118.1(3)
C(13)-C(14)-C(1)#1	138.1(3)	C(15)-C(14)-Rb(1)	73.54(17)
C(13)-C(14)-Rb(1)	110.27(19)	C(1)#1-C(14)-Rb(1)	84.52(17)
C(15)#1-C(15)-C(14)	122.9(3)	C(15)#1-C(15)-C(7)	121.5(3)
C(14)-C(15)-C(7)	115.6(3)	C(15)#1-C(15)-Rb(1)	79.7(2)
C(14)-C(15)-Rb(1)	81.42(18)	C(7)-C(15)-Rb(1)	109.3(2)
C(15)#1-C(15)-Rb(1)#1	75.2(2)	C(14)-C(15)-Rb(1)#1	112.4(2)
C(7)-C(15)-Rb(1)#1	84.29(17)	Rb(1)-C(15)-Rb(1)#1	154.92(10)
O(1)-C(16)-C(17)	108.8(2)	O(2)-C(17)-C(16)	109.7(2)
O(2)-C(17)-Rb(1)	55.55(13)	C(16)-C(17)-Rb(1)	84.71(16)
O(2)-C(18)-C(19)	108.7(3)	O(2)-C(18)-Rb(1)	54.97(13)
C(19)-C(18)-Rb(1)	83.99(16)	O(3)-C(19)-C(18)	108.7(2)
O(3)-C(20)-C(21)	108.7(3)	O(4)-C(21)-C(20)	109.0(3)
O(4)-C(21)-Rb(1)	52.68(14)	C(20)-C(21)-Rb(1)	84.99(17)
O(4)-C(22)-C(23)	109.3(3)	O(4)-C(22)-Rb(1)	53.04(14)
C(23)-C(22)-Rb(1)	84.50(17)	O(5)-C(23)-C(22)	109.2(3)
O(5)-C(24)-C(25)	109.1(3)	O(6)-C(25)-C(24)	108.5(2)
O(6)-C(25)-Rb(1)	50.69(13)	C(24)-C(25)-Rb(1)	84.10(17)
O(6)-C(26)-C(27)	109.5(2)	O(6)-C(26)-Rb(1)	50.94(13)
C(27)-C(26)-Rb(1)	83.84(16)	O(1)-C(27)-C(26)	109.4(2)
O(7)-C(28)-C(29)	107.2(3)	C(30)-C(29)-C(28)	103.6(3)
C(31)-C(30)-C(29)	102.4(3)	O(7)-C(31)-C(30)	104.2(3)
C(33)-C(32)-C(45)#2	125.1(3)	C(33)-C(32)-C(37)	117.3(3)
C(45)#2-C(32)-C(37)	117.6(3)	C(34)-C(33)-C(32)	123.0(3)
C(33)-C(34)-C(35)	120.0(3)	C(36)-C(35)-C(34)	119.4(4)
C(35)-C(36)-C(37)	122.7(3)	C(36)-C(37)-C(38)	124.2(3)
C(36)-C(37)-C(32)	117.6(3)	C(38)-C(37)-C(32)	118.1(3)
C(36)-C(37)-Rb(2)	123.47(18)	C(38)-C(37)-Rb(2)	60.74(15)
C(32)-C(37)-Rb(2)	87.88(18)	C(39)-C(38)-C(37)	138.5(3)
C(39)-C(38)-C(46)	100.3(3)	C(37)-C(38)-C(46)	121.2(3)
C(39)-C(38)-Rb(2)	98.02(18)	C(37)-C(38)-Rb(2)	96.20(17)
C(46)-C(38)-Rb(2)	69.96(17)	C(40)-C(39)-C(38)	131.0(3)
C(40)-C(39)-C(44)	118.5(3)	C(38)-C(39)-C(44)	110.5(3)
C(40)-C(39)-Rb(2)	123.5(2)	C(38)-C(39)-Rb(2)	59.30(15)
C(44)-C(39)-Rb(2)	84.96(18)	C(41)-C(40)-C(39)	120.6(4)
C(40)-C(41)-C(42)	121.1(4)	C(43)-C(42)-C(41)	120.5(4)
C(42)-C(43)-C(44)	121.2(4)	C(43)-C(44)-C(45)	131.9(3)
C(43)-C(44)-C(39)	118.2(3)	C(45)-C(44)-C(39)	109.9(3)
C(46)-C(45)-C(44)	100.9(3)	C(46)-C(45)-C(32)#2	121.4(3)
C(44)-C(45)-C(32)#2	137.6(3)	C(46)-C(45)-Rb(2)	58.90(16)
C(44)-C(45)-Rb(2)	92.54(18)	C(32)#2-C(45)-Rb(2)	107.42(18)
C(46)-C(45)-Rb(2)#2	61.65(17)	C(44)-C(45)-Rb(2)#2	116.53(19)
C(32)#2-C(45)-Rb(2)#2	87.32(18)	Rb(2)-C(45)-Rb(2)#2	117.36(8)
C(46)#2-C(46)-C(45)	121.4(5)	C(46)#2-C(46)-C(38)	120.2(5)
C(45)-C(46)-C(38)	118.4(3)	C(46)#2-C(46)-Rb(2)	87.0(3)
C(45)-C(46)-Rb(2)	97.26(19)	C(38)-C(46)-Rb(2)	83.34(18)
C(46)#2-C(46)-Rb(2)#2	68.7(3)	C(45)-C(46)-Rb(2)#2	95.6(2)
C(38)-C(46)-Rb(2)#2	108.4(2)	Rb(2)-C(46)-Rb(2)#2	155.69(13)
O(8)-C(47)-C(48)	109.5(3)	O(8)-C(47)-Rb(2)	51.62(15)
C(48)-C(47)-Rb(2)	83.48(19)	O(9)-C(48)-C(47)	109.7(3)

O(9)-C(49)-C(50)	109.0(3)	O(10)-C(50)-C(49)	108.4(3)
O(10)-C(50)-Rb(2)	52.34(15)	C(49)-C(50)-Rb(2)	83.61(19)
O(10)-C(51)-C(52)	108.0(3)	O(10)-C(51)-Rb(2)	51.23(14)
C(52)-C(51)-Rb(2)	83.70(18)	O(11)-C(52)-C(51)	109.0(3)
O(11)-C(53)-C(54)	109.1(3)	O(12)-C(54)-C(53)	109.5(3)
O(12)-C(54)-Rb(2)	52.18(14)	C(53)-C(54)-Rb(2)	84.54(18)
O(12)-C(55)-C(56)	109.2(3)	O(12)-C(55)-Rb(2)	53.00(15)
C(56)-C(55)-Rb(2)	85.2(2)	O(13)-C(56)-C(55)	108.9(3)
O(13)-C(57)-C(58)	108.5(3)	O(8)-C(58)-C(57)	109.3(3)
O(8)-C(58)-Rb(2)	51.81(14)	C(57)-C(58)-Rb(2)	85.22(19)
O(14)-C(59)-C(60)	108.7(3)	C(59)-C(60)-C(61)	103.1(3)
C(62)-C(61)-C(60)	104.4(3)	O(14)-C(62)-C(61)	106.1(3)
C(16)-O(1)-C(27)	111.7(2)	C(16)-O(1)-Rb(1)	123.18(16)
C(27)-O(1)-Rb(1)	121.39(15)	C(18)-O(2)-C(17)	111.6(2)
C(18)-O(2)-Rb(1)	102.83(16)	C(17)-O(2)-Rb(1)	102.08(16)
C(20)-O(3)-C(19)	112.1(2)	C(20)-O(3)-Rb(1)	120.75(17)
C(19)-O(3)-Rb(1)	120.86(16)	C(21)-O(4)-C(22)	111.6(2)
C(21)-O(4)-Rb(1)	105.34(17)	C(22)-O(4)-Rb(1)	104.74(18)
C(24)-O(5)-C(23)	112.6(2)	C(24)-O(5)-Rb(1)	121.26(16)
C(23)-O(5)-Rb(1)	119.97(18)	C(26)-O(6)-C(25)	111.2(2)
C(26)-O(6)-Rb(1)	107.43(17)	C(25)-O(6)-Rb(1)	107.75(16)
C(28)-O(7)-C(31)	108.2(3)	C(58)-O(8)-C(47)	111.1(2)
C(58)-O(8)-Rb(2)	106.22(18)	C(47)-O(8)-Rb(2)	106.41(18)
C(49)-O(9)-C(48)	112.4(3)	C(49)-O(9)-Rb(2)	123.1(2)
C(48)-O(9)-Rb(2)	120.14(19)	C(51)-O(10)-C(50)	113.1(2)
C(51)-O(10)-Rb(2)	107.54(17)	C(50)-O(10)-Rb(2)	105.97(18)
C(53)-O(11)-C(52)	112.8(2)	C(53)-O(11)-Rb(2)	117.52(18)
C(52)-O(11)-Rb(2)	121.32(19)	C(54)-O(12)-C(55)	111.1(3)
C(54)-O(12)-Rb(2)	105.76(17)	C(55)-O(12)-Rb(2)	104.56(18)
C(56)-O(13)-C(57)	112.4(3)	C(56)-O(13)-Rb(2)	119.9(2)
C(57)-O(13)-Rb(2)	121.66(19)	C(59)-O(14)-C(62)	105.8(3)
O(1)-Rb(1)-O(5)	113.23(6)	O(1)-Rb(1)-O(3)	112.64(6)
O(5)-Rb(1)-O(3)	113.82(6)	O(1)-Rb(1)-O(6)	57.09(5)
O(5)-Rb(1)-O(6)	56.60(5)	O(3)-Rb(1)-O(6)	142.05(6)
O(1)-Rb(1)-O(4)	141.44(6)	O(5)-Rb(1)-O(4)	57.48(6)
O(3)-Rb(1)-O(4)	56.74(6)	O(6)-Rb(1)-O(4)	106.23(6)
O(1)-Rb(1)-O(2)	56.26(5)	O(5)-Rb(1)-O(2)	141.66(6)
O(3)-Rb(1)-O(2)	56.75(5)	O(6)-Rb(1)-O(2)	105.73(5)
O(4)-Rb(1)-O(2)	105.55(6)	O(1)-Rb(1)-C(15)	84.45(7)
O(5)-Rb(1)-C(15)	123.57(7)	O(3)-Rb(1)-C(15)	105.78(7)
O(6)-Rb(1)-C(15)	108.92(7)	O(4)-Rb(1)-C(15)	133.26(7)
O(2)-Rb(1)-C(15)	93.55(7)	O(1)-Rb(1)-C(15)#1	85.12(7)
O(5)-Rb(1)-C(15)#1	100.25(7)	O(3)-Rb(1)-C(15)#1	128.58(7)
O(6)-Rb(1)-C(15)#1	88.71(7)	O(4)-Rb(1)-C(15)#1	131.89(7)
O(2)-Rb(1)-C(15)#1	114.00(6)	C(15)-Rb(1)-C(15)#1	25.08(10)
O(1)-Rb(1)-C(14)	104.60(7)	O(5)-Rb(1)-C(14)	125.03(7)
O(3)-Rb(1)-C(14)	84.27(7)	O(6)-Rb(1)-C(14)	132.71(6)
O(4)-Rb(1)-C(14)	110.28(7)	O(2)-Rb(1)-C(14)	92.45(7)
C(15)-Rb(1)-C(14)	25.04(7)	C(15)#1-Rb(1)-C(14)	44.32(7)
O(1)-Rb(1)-C(7)#1	105.80(7)	O(5)-Rb(1)-C(7)#1	80.16(7)
O(3)-Rb(1)-C(7)#1	127.30(7)	O(6)-Rb(1)-C(7)#1	89.20(7)
O(4)-Rb(1)-C(7)#1	108.55(6)	O(2)-Rb(1)-C(7)#1	136.95(6)
C(15)-Rb(1)-C(7)#1	43.57(7)	C(15)#1-Rb(1)-C(7)#1	24.46(7)
C(14)-Rb(1)-C(7)#1	51.35(7)	O(1)-Rb(1)-C(1)#1	127.50(6)
O(5)-Rb(1)-C(1)#1	102.16(7)	O(3)-Rb(1)-C(1)#1	84.47(7)
O(6)-Rb(1)-C(1)#1	132.16(6)	O(4)-Rb(1)-C(1)#1	90.15(6)
O(2)-Rb(1)-C(1)#1	112.66(6)	C(15)-Rb(1)-C(1)#1	43.21(7)
C(15)#1-Rb(1)-C(1)#1	50.29(7)	C(14)-Rb(1)-C(1)#1	24.90(7)
C(7)#1-Rb(1)-C(1)#1	43.09(7)	O(1)-Rb(1)-C(6)#1	127.60(6)
O(5)-Rb(1)-C(6)#1	81.82(7)	O(3)-Rb(1)-C(6)#1	104.33(6)
O(6)-Rb(1)-C(6)#1	109.59(6)	O(4)-Rb(1)-C(6)#1	89.89(6)
O(2)-Rb(1)-C(6)#1	135.42(6)	C(15)-Rb(1)-C(6)#1	49.70(7)
C(15)#1-Rb(1)-C(6)#1	42.50(7)	C(14)-Rb(1)-C(6)#1	43.25(7)
C(7)#1-Rb(1)-C(6)#1	24.20(7)	C(1)#1-Rb(1)-C(6)#1	24.01(7)
O(9)-Rb(2)-O(13)	114.14(7)	O(9)-Rb(2)-O(11)	111.17(7)
O(13)-Rb(2)-O(11)	115.48(6)	O(9)-Rb(2)-O(12)	140.07(6)
O(13)-Rb(2)-O(12)	57.57(6)	O(11)-Rb(2)-O(12)	58.07(6)

O(9)-Rb(2)-O(8)	57.88(7)	O(13)-Rb(2)-O(8)	56.57(7)
O(11)-Rb(2)-O(8)	141.97(6)	O(12)-Rb(2)-O(8)	104.86(6)
O(9)-Rb(2)-C(46)	104.48(9)	O(13)-Rb(2)-C(46)	96.46(8)
O(11)-Rb(2)-C(46)	113.81(9)	O(12)-Rb(2)-C(46)	115.04(8)
O(8)-Rb(2)-C(46)	104.20(9)	O(9)-Rb(2)-O(10)	56.06(7)
O(13)-Rb(2)-O(10)	145.85(6)	O(11)-Rb(2)-O(10)	55.86(6)
O(12)-Rb(2)-O(10)	106.70(6)	O(8)-Rb(2)-O(10)	107.60(6)
C(46)-Rb(2)-O(10)	117.46(8)	O(9)-Rb(2)-C(38)	118.17(7)
O(13)-Rb(2)-C(38)	108.12(7)	O(11)-Rb(2)-C(38)	87.15(7)
O(12)-Rb(2)-C(38)	100.41(7)	O(8)-Rb(2)-C(38)	130.79(7)
C(46)-Rb(2)-C(38)	26.70(10)	O(10)-Rb(2)-C(38)	104.43(7)
O(9)-Rb(2)-C(46)#2	80.23(8)	O(13)-Rb(2)-C(46)#2	108.26(8)
O(11)-Rb(2)-C(46)#2	122.78(9)	O(12)-Rb(2)-C(46)#2	139.33(8)
O(8)-Rb(2)-C(46)#2	92.76(9)	C(46)-Rb(2)-C(46)#2	24.31(13)
O(10)-Rb(2)-C(46)#2	102.16(7)	C(38)-Rb(2)-C(46)#2	44.01(9)
O(9)-Rb(2)-C(45)	114.08(8)	O(13)-Rb(2)-C(45)	72.63(7)
O(11)-Rb(2)-C(45)	124.14(8)	O(12)-Rb(2)-C(45)	100.78(7)
O(8)-Rb(2)-C(45)	90.75(7)	C(46)-Rb(2)-C(45)	23.84(8)
O(10)-Rb(2)-C(45)	141.23(6)	C(38)-Rb(2)-C(45)	42.92(8)
C(46)#2-Rb(2)-C(45)	41.65(8)	O(9)-Rb(2)-C(55)	148.19(8)
O(13)-Rb(2)-C(55)	40.69(8)	O(11)-Rb(2)-C(55)	77.89(7)
O(12)-Rb(2)-C(55)	22.43(7)	O(8)-Rb(2)-C(55)	95.62(8)
C(46)-Rb(2)-C(55)	98.69(9)	O(10)-Rb(2)-C(55)	129.12(7)
C(38)-Rb(2)-C(55)	92.06(9)	C(46)#2-Rb(2)-C(55)	121.65(9)
C(45)-Rb(2)-C(55)	80.54(8)	O(9)-Rb(2)-C(37)	104.18(7)
O(13)-Rb(2)-C(37)	130.88(7)	O(11)-Rb(2)-C(37)	74.60(7)
O(12)-Rb(2)-C(37)	108.33(7)	O(8)-Rb(2)-C(37)	141.29(7)
C(46)-Rb(2)-C(37)	43.01(9)	O(10)-Rb(2)-C(37)	81.39(6)
C(38)-Rb(2)-C(37)	23.06(7)	C(46)#2-Rb(2)-C(37)	48.74(9)
C(45)-Rb(2)-C(37)	64.10(7)	C(55)-Rb(2)-C(37)	107.63(8)

Symmetry transformations used to generate equivalent atoms: #1 -x+2,-y+1,-z+1; #2 -x+1,-y+1,-z

Table S6. Anisotropic displacement parameters (Å^2). The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11} + ... + 2\ h\ k\ a^*\ b^*\ U_{12}]$.

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(1)	0.016(1)	0.017(2)	0.018(1)	-0.003(1)	0.001(1)	0.002(1)
C(2)	0.019(1)	0.025(2)	0.024(2)	0.002(1)	0.005(1)	0.005(1)
C(3)	0.020(2)	0.030(2)	0.027(2)	0.002(1)	0.003(1)	0.011(1)
C(4)	0.026(2)	0.029(2)	0.024(2)	0.006(1)	0.001(1)	0.011(1)
C(5)	0.019(1)	0.025(2)	0.019(1)	0.002(1)	0.002(1)	0.003(1)
C(6)	0.018(1)	0.018(2)	0.019(1)	0.000(1)	0.002(1)	0.003(1)
C(7)	0.018(1)	0.014(2)	0.021(1)	0.002(1)	-0.002(1)	0.000(1)
C(8)	0.018(1)	0.016(2)	0.020(1)	0.000(1)	0.002(1)	0.002(1)
C(9)	0.023(2)	0.024(2)	0.020(1)	0.001(1)	0.002(1)	0.007(1)
C(10)	0.027(2)	0.033(2)	0.021(2)	0.006(1)	0.008(1)	0.011(2)
C(11)	0.023(2)	0.034(2)	0.025(2)	0.003(2)	0.010(1)	0.010(2)
C(12)	0.022(2)	0.023(2)	0.023(2)	0.000(1)	0.003(1)	0.008(1)
C(13)	0.017(1)	0.019(2)	0.019(1)	-0.001(1)	0.000(1)	0.004(1)
C(14)	0.012(1)	0.020(2)	0.019(1)	-0.002(1)	-0.001(1)	0.001(1)
C(15)	0.017(1)	0.020(2)	0.020(1)	0.002(1)	0.002(1)	0.003(1)
C(16)	0.024(2)	0.022(2)	0.019(1)	0.006(1)	0.001(1)	0.005(1)
C(17)	0.026(2)	0.027(2)	0.018(1)	0.007(1)	0.002(1)	0.006(1)
C(18)	0.029(2)	0.023(2)	0.023(2)	0.005(1)	0.010(1)	0.008(1)
C(19)	0.022(2)	0.026(2)	0.027(2)	0.008(1)	0.012(1)	0.003(1)
C(20)	0.021(2)	0.036(2)	0.031(2)	0.006(2)	0.005(1)	0.011(2)
C(21)	0.025(2)	0.035(2)	0.036(2)	0.009(2)	0.004(1)	0.017(2)
C(22)	0.027(2)	0.047(2)	0.046(2)	0.032(2)	0.008(2)	0.015(2)
C(23)	0.025(2)	0.049(2)	0.031(2)	0.023(2)	-0.001(1)	0.008(2)
C(24)	0.029(2)	0.029(2)	0.021(2)	0.008(1)	0.004(1)	0.009(2)
C(25)	0.026(2)	0.026(2)	0.022(2)	0.009(1)	0.008(1)	0.008(1)
C(26)	0.017(1)	0.026(2)	0.025(2)	0.006(1)	0.002(1)	0.005(1)
C(27)	0.024(2)	0.023(2)	0.026(2)	0.007(1)	0.005(1)	0.012(1)
C(28)	0.052(3)	0.063(3)	0.065(3)	0.025(3)	0.031(2)	0.030(2)
C(29)	0.046(2)	0.036(2)	0.075(3)	0.011(2)	0.016(2)	0.017(2)
C(30)	0.034(2)	0.031(2)	0.034(2)	0.009(2)	-0.001(2)	0.003(2)
C(31)	0.031(2)	0.030(2)	0.030(2)	0.005(2)	0.005(2)	0.006(2)
C(32)	0.022(2)	0.034(2)	0.019(1)	0.014(2)	-0.004(1)	-0.002(1)
C(33)	0.025(2)	0.037(2)	0.026(2)	0.016(2)	0.001(1)	0.000(2)
C(34)	0.025(2)	0.047(2)	0.033(2)	0.023(2)	0.008(1)	0.009(2)
C(35)	0.031(2)	0.039(2)	0.025(2)	0.015(2)	0.004(1)	0.010(2)
C(36)	0.023(2)	0.032(2)	0.026(2)	0.016(2)	0.001(1)	0.001(1)
C(37)	0.023(2)	0.026(2)	0.020(2)	0.013(1)	-0.003(1)	-0.002(1)
C(38)	0.033(2)	0.023(2)	0.013(1)	0.005(1)	-0.001(1)	0.009(1)
C(39)	0.027(2)	0.039(2)	0.020(2)	0.012(2)	0.005(1)	0.014(2)
C(40)	0.029(2)	0.041(2)	0.028(2)	0.002(2)	0.007(2)	0.009(2)
C(41)	0.030(2)	0.046(3)	0.045(2)	-0.002(2)	0.005(2)	0.002(2)
C(42)	0.026(2)	0.057(3)	0.048(2)	-0.005(2)	0.005(2)	0.003(2)
C(43)	0.030(2)	0.056(3)	0.032(2)	0.009(2)	0.008(2)	0.020(2)
C(44)	0.028(2)	0.045(2)	0.020(2)	0.012(2)	0.006(1)	0.017(2)
C(45)	0.017(1)	0.048(2)	0.020(1)	0.017(2)	0.006(1)	0.008(2)
C(46)	0.028(2)	0.061(3)	0.029(2)	0.023(2)	0.003(2)	0.010(2)
C(47)	0.049(2)	0.018(2)	0.023(2)	0.002(1)	0.004(2)	0.003(2)
C(48)	0.042(2)	0.023(2)	0.023(2)	-0.002(2)	0.002(2)	-0.005(2)
C(49)	0.025(2)	0.033(2)	0.032(2)	0.009(2)	0.002(1)	-0.007(2)
C(50)	0.024(2)	0.035(2)	0.029(2)	0.012(2)	0.008(1)	-0.001(2)
C(51)	0.031(2)	0.037(2)	0.021(2)	0.004(2)	0.008(1)	0.013(2)
C(52)	0.036(2)	0.035(2)	0.022(2)	0.007(2)	0.007(1)	0.015(2)
C(53)	0.042(2)	0.018(2)	0.025(2)	0.003(1)	0.009(2)	0.004(2)
C(54)	0.035(2)	0.020(2)	0.029(2)	-0.001(1)	0.008(2)	0.000(2)
C(55)	0.023(2)	0.030(2)	0.050(2)	0.004(2)	0.007(2)	-0.001(2)
C(56)	0.032(2)	0.035(2)	0.049(2)	0.010(2)	0.019(2)	0.006(2)
C(57)	0.035(2)	0.034(2)	0.023(2)	0.007(2)	0.009(1)	0.015(2)
C(58)	0.045(2)	0.028(2)	0.022(2)	0.008(1)	0.009(2)	0.018(2)
C(59)	0.091(4)	0.069(3)	0.031(2)	-0.002(2)	0.004(2)	0.055(3)
C(60)	0.040(2)	0.061(3)	0.034(2)	0.007(2)	0.008(2)	0.019(2)
C(61)	0.044(2)	0.088(4)	0.041(2)	0.029(2)	0.013(2)	0.028(2)
C(62)	0.046(2)	0.047(3)	0.052(2)	0.026(2)	0.016(2)	0.018(2)
O(1)	0.022(1)	0.022(1)	0.021(1)	0.007(1)	0.005(1)	0.009(1)
O(2)	0.021(1)	0.024(1)	0.019(1)	0.003(1)	0.004(1)	0.005(1)
O(3)	0.017(1)	0.027(1)	0.029(1)	0.010(1)	0.004(1)	0.003(1)
O(4)	0.026(1)	0.030(1)	0.027(1)	0.013(1)	0.006(1)	0.012(1)
O(5)	0.022(1)	0.032(1)	0.023(1)	0.009(1)	0.002(1)	0.008(1)
O(6)	0.018(1)	0.021(1)	0.020(1)	0.005(1)	0.002(1)	0.005(1)
O(7)	0.046(2)	0.038(2)	0.066(2)	0.026(2)	0.031(1)	0.019(1)
O(8)	0.038(1)	0.024(1)	0.022(1)	0.006(1)	0.007(1)	0.010(1)
O(9)	0.029(1)	0.024(1)	0.023(1)	0.001(1)	0.001(1)	-0.003(1)
O(10)	0.025(1)	0.027(1)	0.020(1)	0.006(1)	0.005(1)	0.000(1)
O(11)	0.028(1)	0.023(1)	0.028(1)	0.009(1)	0.009(1)	0.005(1)
O(12)	0.028(1)	0.024(1)	0.030(1)	0.004(1)	0.009(1)	0.002(1)
O(13)	0.035(1)	0.027(1)	0.028(1)	0.005(1)	0.011(1)	0.007(1)
O(14)	0.044(2)	0.035(2)	0.036(1)	0.007(1)	0.006(1)	0.019(1)
Rb(1)	0.017(1)	0.019(1)	0.019(1)	0.003(1)	0.002(1)	0.004(1)
Rb(2)	0.026(1)	0.020(1)	0.016(1)	0.005(1)	0.002(1)	0.003(1)

Table S7. Hydrogen coordinates and isotropic displacement parameters (Å²).

	x	y	z	U_{eq}
H(2)	1.3617	0.4221	0.4984	0.028
H(3)	1.4179	0.3261	0.5709	0.031
H(4)	1.2868	0.2779	0.6433	0.031
H(5)	1.1028	0.3287	0.6432	0.027
H(9)	0.9357	0.3553	0.6766	0.028
H(10)	0.7496	0.3510	0.7156	0.032
H(11)	0.5970	0.4208	0.6686	0.032
H(12)	0.6307	0.4983	0.5827	0.028
H(16A)	0.9688	0.0959	0.5281	0.027
H(16B)	0.8433	0.0113	0.4790	0.027
H(17A)	0.7815	0.0914	0.5666	0.029
H(17B)	0.8471	0.2161	0.5523	0.029
H(18A)	0.6440	0.2437	0.5427	0.030
H(18B)	0.5707	0.1192	0.5557	0.030
H(19A)	0.4419	0.0705	0.4545	0.030
H(19B)	0.4124	0.1655	0.4993	0.030
H(20A)	0.3453	0.2455	0.4179	0.035
H(20B)	0.3664	0.1476	0.3715	0.035
H(21A)	0.3835	0.3148	0.3302	0.036
H(21B)	0.5093	0.3855	0.3826	0.036
H(22A)	0.6406	0.4248	0.3068	0.045
H(22B)	0.5138	0.3532	0.2552	0.045
H(23A)	0.6225	0.2235	0.2260	0.042
H(23B)	0.6961	0.3498	0.2156	0.042
H(24A)	0.9006	0.3189	0.2246	0.032
H(24B)	0.8282	0.1914	0.2337	0.032
H(25A)	1.0554	0.2598	0.2783	0.029
H(25B)	1.0347	0.3610	0.3223	0.029
H(26A)	1.1057	0.2856	0.4067	0.028
H(26B)	1.1218	0.1857	0.3599	0.028
H(27A)	0.9647	0.0481	0.3978	0.028
H(27B)	1.0884	0.1230	0.4503	0.028
H(28A)	0.2839	0.0419	0.2473	0.065
H(28B)	0.1893	-0.0366	0.1854	0.065
H(29A)	0.1170	-0.1843	0.2303	0.061
H(29B)	0.2314	-0.1129	0.2881	0.061
H(30A)	-0.0136	-0.1641	0.2978	0.043
H(30B)	0.0944	-0.0455	0.3361	0.043
H(31A)	-0.0729	-0.0784	0.2219	0.038
H(31B)	-0.0477	0.0189	0.2808	0.038
H(33)	0.8850	0.5018	-0.0400	0.038
H(34)	0.8941	0.3384	-0.0958	0.041
H(35)	0.7037	0.1753	-0.1289	0.038
H(36)	0.5066	0.1776	-0.1034	0.034
H(40)	0.2993	0.1209	-0.0976	0.041
H(41)	0.0763	0.0329	-0.1022	0.054
H(42)	-0.0458	0.1318	-0.0584	0.058
H(43)	0.0532	0.3208	-0.0101	0.045
H(47A)	0.6226	0.7031	0.2359	0.040
H(47B)	0.6093	0.6638	0.1644	0.040
H(48A)	0.8259	0.7032	0.2181	0.042
H(48B)	0.7685	0.6018	0.2532	0.042
H(49A)	0.9134	0.5155	0.2077	0.041
H(49B)	0.9552	0.6172	0.1711	0.041
H(50A)	0.8629	0.4818	0.0785	0.038
H(50B)	0.9793	0.4554	0.1171	0.038
H(51A)	0.8883	0.2580	0.0725	0.035
H(51B)	0.7697	0.2770	0.0311	0.035
H(52A)	0.7106	0.0861	0.0498	0.036
H(52B)	0.7315	0.1374	0.1206	0.036
H(53A)	0.5046	0.0421	0.1223	0.036
H(53B)	0.4857	-0.0158	0.0526	0.036
H(54A)	0.3348	0.0811	0.0280	0.037
H(54B)	0.2823	-0.0151	0.0668	0.037
H(55A)	0.1451	0.0806	0.1010	0.045
H(55B)	0.2011	0.1800	0.0644	0.045
H(56A)	0.1180	0.2433	0.1497	0.047
H(56B)	0.2305	0.2205	0.1933	0.047
H(57A)	0.3169	0.4172	0.2385	0.035
H(57B)	0.2092	0.4436	0.1938	0.035
H(58A)	0.3797	0.5634	0.1538	0.035
H(58B)	0.3947	0.6089	0.2249	0.035
H(59A)	-0.2919	0.0086	0.3777	0.071
H(59B)	-0.3755	-0.1211	0.3795	0.071
H(60A)	-0.4801	0.0306	0.3481	0.054
H(60B)	-0.5594	-0.1050	0.3364	0.054
H(61A)	-0.5495	-0.1245	0.2377	0.065
H(61B)	-0.4947	0.0129	0.2475	0.065
H(62A)	-0.3508	-0.1016	0.2163	0.055
H(62B)	-0.2857	0.0238	0.2585	0.055

1.2 Crystal structure data of **2**

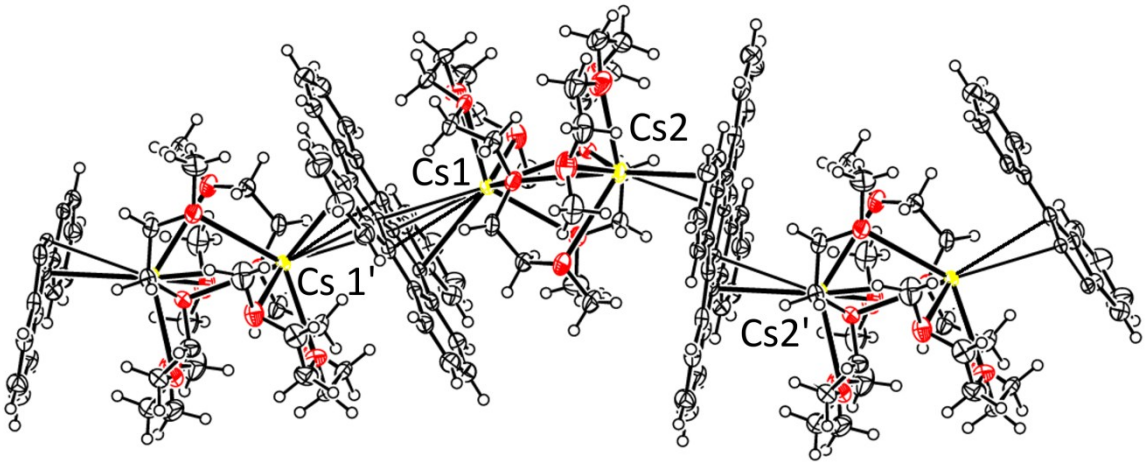


Figure S2. ORTEP drawing of the structure of 1D polymeric DOPT Dicesium bis(tetraglyme) **2** at the 50% probability level, with two differently coordinated alternating dianionic DOPT-planes in the crystal lattice (left: half of η′:η′ coordinated unit B; middle-left: η:η coordination in **2**A; middle-right: η′:η′ coordination in **2**B; right: half of η:η coordinated unit A). Two tetraglyme ligands between the alternating units A and B are connected to both Cs(I) cations Cs1 and Cs2 (yellow).

Table S8. Atomic coordinates and equivalent isotropic displacement parameters (Å^2). U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
C(1A)	0.9530(2)	0.5510(2)	0.4891(1)	0.019(1)
C(2)	0.9818(3)	0.6637(2)	0.4495(1)	0.027(1)
C(3)	0.8588(3)	0.7396(3)	0.4381(1)	0.029(1)
C(4)	0.8192(3)	0.8650(3)	0.4024(2)	0.039(1)
C(5)	0.6908(4)	0.9189(4)	0.4021(2)	0.056(1)
C(6)	0.6012(4)	0.8513(4)	0.4330(2)	0.054(1)
C(7)	0.6345(3)	0.7286(3)	0.4686(2)	0.039(1)
C(8)	0.7639(3)	0.6693(3)	0.4715(1)	0.029(1)
C(9)	0.8250(2)	0.5458(3)	0.5040(1)	0.026(1)
C(10)	0.7904(3)	0.4317(3)	0.5423(1)	0.028(1)
C(11)	0.6652(3)	0.4170(3)	0.5562(1)	0.034(1)
C(12)	0.6355(3)	0.3067(3)	0.5935(2)	0.041(1)
C(13)	0.7297(3)	0.2056(3)	0.6174(2)	0.042(1)
C(14)	0.8537(3)	0.2145(3)	0.6049(1)	0.035(1)
C(15)	0.8894(3)	0.3250(2)	0.5670(1)	0.027(1)
C(16A)	0.5321(3)	0.5367(2)	1.0078(1)	0.016(1)
C(17)	0.6604(2)	0.4879(3)	1.0179(1)	0.027(1)
C(18)	0.6857(2)	0.5961(2)	1.0287(1)	0.027(1)
C(19)	0.7967(3)	0.6140(3)	1.0436(1)	0.033(1)
C(20)	0.7965(3)	0.7279(3)	1.0555(2)	0.039(1)
C(21)	0.6890(3)	0.8262(3)	1.0522(2)	0.042(1)
C(22)	0.5779(3)	0.8134(3)	1.0369(1)	0.033(1)
C(23)	0.5731(2)	0.6991(2)	1.0252(1)	0.027(1)
C(24)	0.4704(3)	0.6619(2)	1.0116(1)	0.025(1)
C(25)	0.3426(2)	0.7176(2)	0.9982(1)	0.027(1)
C(26)	0.2798(3)	0.8423(3)	0.9987(1)	0.032(1)
C(27)	0.1574(3)	0.8930(3)	0.9842(2)	0.035(1)
C(28)	0.0932(3)	0.8194(3)	0.9679(1)	0.035(1)
C(29)	0.1506(3)	0.6957(3)	0.9673(1)	0.031(1)
C(30)	0.2746(2)	0.6401(3)	0.9821(1)	0.027(1)
C(31)	1.2457(4)	0.3506(3)	0.7150(2)	0.049(1)
C(32)	1.2906(3)	0.5301(3)	0.6377(2)	0.040(1)

C(33)	1.2552(3)	0.6680(3)	0.6226(2)	0.040(1)
C(34)	1.0884(3)	0.8411(3)	0.5914(2)	0.033(1)
C(35)	0.9632(3)	0.8772(2)	0.5666(1)	0.031(1)
C(36)	0.7505(3)	0.8706(3)	0.6002(1)	0.032(1)
C(37)	0.6555(3)	0.8458(3)	0.6632(2)	0.036(1)
C(38)	0.6103(3)	0.6545(3)	0.6597(1)	0.036(1)
C(39)	0.5993(3)	0.5330(3)	0.7063(1)	0.040(1)
C(40)	0.4807(5)	0.4423(5)	0.8069(2)	0.066(1)
C(41)	0.2845(4)	0.8333(5)	0.7810(2)	0.058(1)
C(42)	0.3437(4)	1.0028(4)	0.7982(2)	0.052(1)
C(43)	0.4490(4)	1.0603(4)	0.7889(2)	0.055(1)
C(44)	0.6450(4)	1.0338(3)	0.8234(2)	0.048(1)
C(45)	0.7444(4)	0.9419(3)	0.8587(2)	0.046(1)
C(46)	0.9100(3)	0.7654(3)	0.8434(2)	0.039(1)
C(47)	0.9652(3)	0.6772(3)	0.7959(2)	0.038(1)
C(48)	0.9284(3)	0.4780(3)	0.8572(1)	0.032(1)
C(49)	0.8721(3)	0.3786(3)	0.8511(1)	0.033(1)
C(50)	0.8997(6)	0.2329(4)	0.7839(2)	0.076(2)
Cs(1)	0.9501(1)	0.5436(1)	0.6536(1)	0.022(1)
Cs(2)	0.5936(1)	0.6933(1)	0.8512(1)	0.029(1)
O(1)	1.2159(2)	0.4822(2)	0.6988(1)	0.038(1)
O(2)	1.1305(2)	0.7102(2)	0.6077(1)	0.033(1)
O(3)	0.8725(2)	0.8353(2)	0.6213(1)	0.027(1)
O(4)	0.6673(2)	0.7160(2)	0.6926(1)	0.029(1)
O(5)	0.5069(3)	0.5529(3)	0.7635(1)	0.051(1)
O(6)	0.3828(2)	0.8918(3)	0.7725(1)	0.049(1)
O(7)	0.5435(3)	0.9790(2)	0.8263(1)	0.047(1)
O(8)	0.8009(2)	0.8465(2)	0.8198(1)	0.036(1)
O(9)	0.8943(2)	0.5862(2)	0.8050(1)	0.029(1)
O(10)	0.9266(3)	0.3431(2)	0.7873(1)	0.046(1)
C(16B)	0.4482(7)	0.5485(7)	0.9985(4)	0.016(2)
C(1B)	0.9563(13)	0.4674(12)	0.5128(6)	0.015(3)

Table S9. Bond lengths [Å] and angles [°].

C(1A)-C(1B)	0.949(13)	C(1A)-C(1B)#1	0.970(14)
C(1A)-C(1A)#1	1.378(5)	C(1A)-C(9)	1.415(3)
C(1A)-C(2)	1.417(3)	C(1A)-Cs(1)#1	3.225(2)
C(1A)-Cs(1)	3.311(2)	C(2)-C(3)	1.433(4)
C(2)-C(15)#1	1.444(4)	C(2)-C(1B)#1	1.537(13)
C(2)-Cs(1)#1	3.406(2)	C(3)-C(4)	1.422(4)
C(3)-C(8)	1.461(4)	C(4)-C(5)	1.394(5)
C(5)-C(6)	1.386(6)	C(6)-C(7)	1.389(5)
C(7)-C(8)	1.421(4)	C(8)-C(9)	1.437(4)
C(9)-C(10)	1.449(4)	C(9)-C(1B)	1.509(14)
C(9)-Cs(1)	3.599(2)	C(9)-Cs(1)#1	3.799(2)
C(10)-C(11)	1.418(4)	C(10)-C(15)	1.456(4)
C(10)-C(1B)	1.951(13)	C(10)-Cs(1)	3.817(2)
C(11)-C(12)	1.385(4)	C(12)-C(13)	1.386(5)
C(13)-C(14)	1.385(5)	C(14)-C(15)	1.418(4)
C(15)-C(2)#1	1.444(4)	C(15)-C(1B)	1.963(12)
C(15)-Cs(1)	3.707(2)	C(16A)-C(16B)	0.962(8)
C(16A)-C(16B)#2	0.979(8)	C(16A)-C(16A)#2	1.385(5)
C(16A)-C(17)	1.423(3)	C(16A)-C(24)	1.425(4)
C(16A)-Cs(2)	3.248(2)	C(16A)-Cs(2)#2	3.683(3)
C(17)-C(18)	1.433(4)	C(17)-C(30)#2	1.446(4)
C(17)-C(16B)#2	1.517(8)	C(17)-Cs(2)	3.663(2)
C(18)-C(19)	1.422(4)	C(18)-C(23)	1.463(4)
C(18)-Cs(2)	3.753(2)	C(19)-C(20)	1.395(5)
C(20)-C(21)	1.396(5)	C(21)-C(22)	1.398(5)
C(22)-C(23)	1.416(4)	C(23)-C(24)	1.437(4)
C(23)-Cs(2)	3.509(2)	C(24)-C(25)	1.442(4)
C(24)-C(16B)	1.498(8)	C(24)-Cs(2)	3.220(2)
C(25)-C(26)	1.410(4)	C(25)-C(30)	1.460(3)
C(25)-C(16B)	1.968(8)	C(25)-Cs(2)	3.642(2)
C(26)-C(27)	1.389(4)	C(27)-C(28)	1.394(4)
C(28)-C(29)	1.388(4)	C(29)-C(30)	1.412(4)
C(30)-C(17)#2	1.446(4)	C(30)-C(16B)	1.979(8)
C(31)-O(1)	1.418(4)	C(31)-Cs(1)	3.678(3)
C(32)-O(1)	1.413(4)	C(32)-C(33)	1.487(5)
C(32)-Cs(1)	3.716(3)	C(33)-O(2)	1.411(4)
C(34)-O(2)	1.414(3)	C(34)-C(35)	1.499(4)
C(35)-O(3)	1.416(3)	C(35)-Cs(1)	3.810(3)
C(36)-O(3)	1.433(4)	C(36)-C(37)	1.498(4)
C(36)-Cs(1)	3.797(3)	C(37)-O(4)	1.434(3)
C(37)-Cs(2)	3.757(3)	C(38)-O(4)	1.432(3)
C(38)-C(39)	1.488(4)	C(38)-Cs(1)	3.635(3)
C(39)-O(5)	1.401(4)	C(39)-Cs(2)	3.848(3)
C(39)-Cs(1)	3.869(3)	C(40)-O(5)	1.411(4)
C(40)-Cs(2)	3.837(4)	C(41)-O(6)	1.406(5)
C(41)-Cs(2)	3.782(4)	C(42)-O(6)	1.427(5)
C(42)-C(43)	1.466(6)	C(42)-Cs(2)	3.905(4)
C(43)-O(7)	1.405(5)	C(44)-O(7)	1.434(5)
C(44)-C(45)	1.462(6)	C(45)-O(8)	1.443(4)
C(45)-Cs(2)	3.767(3)	C(46)-O(8)	1.397(4)
C(46)-C(47)	1.500(4)	C(46)-Cs(2)	3.825(3)
C(47)-O(9)	1.439(3)	C(47)-Cs(1)	3.670(3)
C(48)-O(9)	1.421(3)	C(48)-C(49)	1.500(4)
C(48)-Cs(2)	3.863(3)	C(49)-O(10)	1.420(3)
C(50)-O(10)	1.408(4)	Cs(1)-O(2)	3.0149(19)
Cs(1)-O(10)	3.077(2)	Cs(1)-O(1)	3.133(2)
Cs(1)-O(9)	3.1433(18)	Cs(1)-O(3)	3.1439(18)
Cs(1)-C(1B)	3.187(12)	Cs(1)-C(1A)#1	3.225(2)
Cs(1)-O(4)	3.280(2)	Cs(1)-C(1B)#1	3.339(12)
Cs(1)-C(2)#1	3.406(2)	Cs(2)-O(7)	3.097(2)
Cs(2)-O(4)	3.1039(18)	Cs(2)-O(5)	3.145(3)
Cs(2)-O(6)	3.153(3)	Cs(2)-O(8)	3.165(2)
Cs(2)-O(9)	3.257(2)	Cs(2)-C(16B)	3.343(7)
Cs(2)-C(16B)#2	3.591(7)	C(16B)-C(16A)#2	0.979(8)

C(16B)-C(16B)#2	1.360(16)	C(16B)-C(17)#2	1.517(8)
C(16B)-Cs(2)#2	3.591(7)	C(1B)-C(1A)#1	0.970(14)
C(1B)-C(1B)#1	1.34(2)	C(1B)-C(2)#1	1.537(13)
C(1B)-Cs(1)#1	3.339(12)		

C(1B)-C(1A)-C(1B)#1	88.2(11)	C(1B)-C(1A)-C(1A)#1	44.7(8)
C(1B)#1-C(1A)-C(1A)#1	43.5(7)	C(1B)-C(1A)-C(9)	76.5(8)
C(1B)#1-C(1A)-C(9)	164.5(8)	C(1A)#1-C(1A)-C(9)	121.1(3)
C(1B)-C(1A)-C(2)	165.3(9)	C(1B)#1-C(1A)-C(2)	77.7(7)
C(1A)#1-C(1A)-C(2)	121.1(3)	C(9)-C(1A)-C(2)	117.7(2)
C(1B)-C(1A)-Cs(1)#1	88.6(8)	C(1B)#1-C(1A)-Cs(1)#1	79.1(7)
C(1A)#1-C(1A)-Cs(1)#1	81.35(19)	C(9)-C(1A)-Cs(1)#1	102.86(14)
C(2)-C(1A)-Cs(1)#1	84.92(13)	C(1B)-C(1A)-Cs(1)	74.2(7)
C(1B)#1-C(1A)-Cs(1)	83.2(7)	C(1A)#1-C(1A)-Cs(1)	74.35(18)
C(9)-C(1A)-Cs(1)	89.91(13)	C(2)-C(1A)-Cs(1)	107.53(14)
Cs(1)#1-C(1A)-Cs(1)	155.70(9)	C(1A)-C(2)-C(3)	101.5(2)
C(1A)-C(2)-C(15)#1	120.3(2)	C(3)-C(2)-C(15)#1	138.2(2)
C(1A)-C(2)-C(1B)#1	38.0(5)	C(3)-C(2)-C(1B)#1	139.5(5)
C(15)#1-C(2)-C(1B)#1	82.3(5)	C(1A)-C(2)-Cs(1)#1	70.59(13)
C(3)-C(2)-Cs(1)#1	105.19(14)	C(15)#1-C(2)-Cs(1)#1	90.33(14)
C(1B)#1-C(2)-Cs(1)#1	68.7(5)	C(4)-C(3)-C(2)	131.1(3)
C(4)-C(3)-C(8)	119.1(3)	C(2)-C(3)-C(8)	109.8(2)
C(5)-C(4)-C(3)	119.3(3)	C(6)-C(5)-C(4)	121.4(3)
C(5)-C(6)-C(7)	121.7(4)	C(6)-C(7)-C(8)	119.3(3)
C(7)-C(8)-C(9)	131.4(3)	C(7)-C(8)-C(3)	119.1(3)
C(9)-C(8)-C(3)	109.5(2)	C(1A)-C(9)-C(8)	101.5(2)
C(1A)-C(9)-C(10)	120.2(2)	C(8)-C(9)-C(10)	138.3(2)
C(1A)-C(9)-C(1B)	37.7(5)	C(8)-C(9)-C(1B)	139.2(5)
C(10)-C(9)-C(1B)	82.5(5)	C(1A)-C(9)-Cs(1)	66.94(12)
C(8)-C(9)-Cs(1)	111.70(15)	C(10)-C(9)-Cs(1)	87.35(13)
C(1B)-C(9)-Cs(1)	62.2(5)	C(1A)-C(9)-Cs(1)#1	55.85(12)
C(8)-C(9)-Cs(1)#1	98.07(14)	C(10)-C(9)-Cs(1)#1	104.14(15)
C(1B)-C(9)-Cs(1)#1	61.0(5)	Cs(1)-C(9)-Cs(1)#1	119.44(7)
C(11)-C(10)-C(9)	123.6(3)	C(11)-C(10)-C(15)	117.8(2)
C(9)-C(10)-C(15)	118.6(2)	C(11)-C(10)-C(1B)	172.8(4)
C(9)-C(10)-C(1B)	50.1(4)	C(15)-C(10)-C(1B)	68.6(4)
C(11)-C(10)-Cs(1)	127.10(16)	C(9)-C(10)-Cs(1)	70.36(13)
C(15)-C(10)-Cs(1)	74.62(13)	C(1B)-C(10)-Cs(1)	56.5(4)
C(12)-C(11)-C(10)	122.0(3)	C(11)-C(12)-C(13)	120.1(3)
C(14)-C(13)-C(12)	120.3(3)	C(13)-C(14)-C(15)	121.9(3)
C(14)-C(15)-C(2)#1	123.5(3)	C(14)-C(15)-C(10)	117.9(3)
C(2)#1-C(15)-C(10)	118.6(2)	C(14)-C(15)-C(1B)	174.1(5)
C(2)#1-C(15)-C(1B)	50.9(4)	C(10)-C(15)-C(1B)	67.7(4)
C(14)-C(15)-Cs(1)	121.90(15)	C(2)#1-C(15)-Cs(1)	66.74(12)
C(10)-C(15)-Cs(1)	83.12(14)	C(1B)-C(15)-Cs(1)	59.3(4)
C(16B)-C(16A)-C(16B)#2	88.9(7)	C(16B)-C(16A)-C(16A)#2	45.0(5)
C(16B)#2-C(16A)-C(16A)#2	44.0(5)	C(16B)-C(16A)-C(17)	164.8(5)
C(16B)#2-C(16A)-C(17)	75.9(5)	C(16A)#2-C(16A)-C(17)	119.8(3)
C(16B)-C(16A)-C(24)	74.9(5)	C(16B)#2-C(16A)-C(24)	163.8(5)
C(16A)#2-C(16A)-C(24)	119.9(3)	C(17)-C(16A)-C(24)	120.3(2)
C(16B)-C(16A)-Cs(2)	87.3(5)	C(16B)#2-C(16A)-Cs(2)	102.6(5)
C(16A)#2-C(16A)-Cs(2)	97.0(2)	C(17)-C(16A)-Cs(2)	95.26(15)
C(24)-C(16A)-Cs(2)	76.17(13)	C(16B)-C(16A)-Cs(2)#2	77.0(5)
C(16B)#2-C(16A)-Cs(2)#2	62.4(4)	C(16A)#2-C(16A)-Cs(2)#2	61.09(19)
C(17)-C(16A)-Cs(2)#2	96.10(15)	C(24)-C(16A)-Cs(2)#2	113.46(15)
Cs(2)-C(16A)-Cs(2)#2	158.07(8)	C(16A)-C(17)-C(18)	99.4(2)
C(16A)-C(17)-C(30)#2	122.4(2)	C(18)-C(17)-C(30)#2	138.2(2)
C(16A)-C(17)-C(16B)#2	38.7(3)	C(18)-C(17)-C(16B)#2	138.1(4)
C(30)#2-C(17)-C(16B)#2	83.7(3)	C(16A)-C(17)-Cs(2)	61.98(13)
C(18)-C(17)-Cs(2)	82.41(14)	C(30)#2-C(17)-Cs(2)	117.29(14)
C(16B)#2-C(17)-Cs(2)	75.3(3)	C(19)-C(18)-C(17)	130.5(2)
C(19)-C(18)-C(23)	118.8(3)	C(17)-C(18)-C(23)	110.6(2)
C(19)-C(18)-Cs(2)	123.48(16)	C(17)-C(18)-Cs(2)	75.35(13)
C(23)-C(18)-Cs(2)	69.11(12)	C(20)-C(19)-C(18)	120.0(3)
C(19)-C(20)-C(21)	121.1(3)	C(20)-C(21)-C(22)	121.0(3)
C(21)-C(22)-C(23)	120.0(3)	C(22)-C(23)-C(24)	130.4(2)
C(22)-C(23)-C(18)	119.1(3)	C(24)-C(23)-C(18)	110.5(2)

C(22)-C(23)-Cs(2)	115.02(15)	C(24)-C(23)-Cs(2)	66.57(12)
C(18)-C(23)-Cs(2)	87.97(13)	C(16A)-C(24)-C(23)	99.2(2)
C(16A)-C(24)-C(25)	122.4(2)	C(23)-C(24)-C(25)	138.2(2)
C(16A)-C(24)-C(16B)	38.3(3)	C(23)-C(24)-C(16B)	137.6(4)
C(25)-C(24)-C(16B)	84.0(3)	C(16A)-C(24)-Cs(2)	78.37(13)
C(23)-C(24)-Cs(2)	89.26(13)	C(25)-C(24)-Cs(2)	95.05(13)
C(16B)-C(24)-Cs(2)	81.5(3)	C(26)-C(25)-C(24)	124.2(2)
C(26)-C(25)-C(30)	117.9(2)	C(24)-C(25)-C(30)	117.9(2)
C(26)-C(25)-C(16B)	173.3(3)	C(24)-C(25)-C(16B)	49.2(3)
C(30)-C(25)-C(16B)	68.7(3)	C(26)-C(25)-Cs(2)	111.48(15)
C(24)-C(25)-Cs(2)	61.72(12)	C(30)-C(25)-Cs(2)	95.87(14)
C(16B)-C(25)-Cs(2)	65.4(2)	C(27)-C(26)-C(25)	122.4(3)
C(26)-C(27)-C(28)	119.6(3)	C(29)-C(28)-C(27)	120.1(3)
C(28)-C(29)-C(30)	122.2(2)	C(29)-C(30)-C(17)#2	124.7(2)
C(29)-C(30)-C(25)	117.7(3)	C(17)#2-C(30)-C(25)	117.5(2)
C(29)-C(30)-C(16B)	174.1(3)	C(17)#2-C(30)-C(16B)	49.7(3)
C(25)-C(30)-C(16B)	67.9(3)	O(1)-C(31)-Cs(1)	56.73(15)
O(1)-C(32)-C(33)	110.1(3)	O(1)-C(32)-Cs(1)	55.23(14)
C(33)-C(32)-Cs(1)	87.35(17)	O(2)-C(33)-C(32)	108.9(2)
O(2)-C(34)-C(35)	109.4(2)	O(3)-C(35)-C(34)	109.2(2)
O(3)-C(35)-Cs(1)	52.04(12)	C(34)-C(35)-Cs(1)	84.32(14)
O(3)-C(36)-C(37)	108.1(2)	O(3)-C(36)-Cs(1)	52.74(12)
C(37)-C(36)-Cs(1)	89.44(16)	O(4)-C(37)-C(36)	113.1(2)
O(4)-C(37)-Cs(2)	52.66(12)	C(36)-C(37)-Cs(2)	147.8(2)
O(4)-C(38)-C(39)	108.6(2)	O(4)-C(38)-Cs(1)	64.35(14)
C(39)-C(38)-Cs(1)	87.58(19)	O(5)-C(39)-C(38)	109.5(3)
O(5)-C(39)-Cs(2)	50.35(14)	C(38)-C(39)-Cs(2)	84.40(16)
O(5)-C(39)-Cs(1)	135.41(19)	C(38)-C(39)-Cs(1)	69.82(16)
Cs(2)-C(39)-Cs(1)	86.25(6)	O(5)-C(40)-Cs(2)	50.94(17)
O(6)-C(41)-Cs(2)	53.40(18)	O(6)-C(42)-C(43)	111.2(3)
O(6)-C(42)-Cs(2)	48.77(16)	C(43)-C(42)-Cs(2)	84.1(2)
O(7)-C(43)-C(42)	109.8(3)	O(7)-C(44)-C(45)	109.8(3)
O(8)-C(45)-C(44)	108.8(3)	O(8)-C(45)-Cs(2)	54.86(15)
C(44)-C(45)-Cs(2)	88.4(2)	O(8)-C(46)-C(47)	107.8(2)
O(8)-C(46)-Cs(2)	52.10(14)	C(47)-C(46)-Cs(2)	86.86(17)
O(9)-C(47)-C(46)	113.2(2)	O(9)-C(47)-Cs(1)	57.60(12)
C(46)-C(47)-Cs(1)	151.7(2)	O(9)-C(48)-C(49)	109.8(2)
O(9)-C(48)-Cs(2)	54.76(13)	C(49)-C(48)-Cs(2)	86.14(17)
O(10)-C(49)-C(48)	108.7(2)	O(2)-Cs(1)-O(10)	128.56(6)
O(2)-Cs(1)-O(1)	54.65(6)	O(10)-Cs(1)-O(1)	77.80(7)
O(2)-Cs(1)-O(9)	92.00(5)	O(10)-Cs(1)-O(9)	54.11(5)
O(1)-Cs(1)-O(9)	75.01(5)	O(2)-Cs(1)-O(3)	54.54(5)
O(10)-Cs(1)-O(3)	133.28(5)	O(1)-Cs(1)-O(3)	102.74(6)
O(9)-Cs(1)-O(3)	80.49(5)	O(2)-Cs(1)-C(1B)	101.1(2)
O(10)-Cs(1)-C(1B)	116.7(2)	O(1)-Cs(1)-C(1B)	113.9(3)
O(9)-Cs(1)-C(1B)	166.8(2)	O(3)-Cs(1)-C(1B)	105.8(2)
O(2)-Cs(1)-C(1A)#1	89.54(6)	O(10)-Cs(1)-C(1A)#1	116.78(6)
O(1)-Cs(1)-C(1A)#1	96.65(6)	O(9)-Cs(1)-C(1A)#1	168.39(6)
O(3)-Cs(1)-C(1A)#1	109.60(5)	C(1B)-Cs(1)-C(1A)#1	17.4(2)
O(2)-Cs(1)-O(4)	107.74(5)	O(10)-Cs(1)-O(4)	93.47(6)
O(1)-Cs(1)-O(4)	137.24(5)	O(9)-Cs(1)-O(4)	66.47(5)
O(3)-Cs(1)-O(4)	54.19(5)	C(1B)-Cs(1)-O(4)	107.4(3)
C(1A)#1-Cs(1)-O(4)	123.84(6)	O(2)-Cs(1)-C(1A)	88.70(6)
O(10)-Cs(1)-C(1A)	133.19(6)	O(1)-Cs(1)-C(1A)	115.36(6)
O(9)-Cs(1)-C(1A)	167.21(6)	O(3)-Cs(1)-C(1A)	89.57(5)
C(1B)-Cs(1)-C(1A)	16.7(2)	C(1A)#1-Cs(1)-C(1A)	24.30(9)
O(4)-Cs(1)-C(1A)	101.18(6)	O(2)-Cs(1)-C(1B)#1	77.7(2)
O(10)-Cs(1)-C(1B)#1	133.3(2)	O(1)-Cs(1)-C(1B)#1	98.6(2)
O(9)-Cs(1)-C(1B)#1	169.7(2)	O(3)-Cs(1)-C(1B)#1	93.3(2)
C(1B)-Cs(1)-C(1B)#1	23.5(4)	C(1A)#1-Cs(1)-C(1B)#1	16.5(2)
O(4)-Cs(1)-C(1B)#1	116.4(2)	C(1A)-Cs(1)-C(1B)#1	16.8(2)
O(2)-Cs(1)-C(2)#1	110.50(6)	O(10)-Cs(1)-C(2)#1	93.08(6)
O(1)-Cs(1)-C(2)#1	98.25(6)	O(9)-Cs(1)-C(2)#1	147.14(5)
O(3)-Cs(1)-C(2)#1	131.98(5)	C(1B)-Cs(1)-C(2)#1	26.7(2)
C(1A)#1-Cs(1)-C(2)#1	24.49(6)	O(4)-Cs(1)-C(2)#1	124.19(6)
C(1A)-Cs(1)-C(2)#1	42.46(6)	C(1B)#1-Cs(1)-C(2)#1	40.7(2)
O(2)-Cs(1)-C(9)	107.70(6)	O(10)-Cs(1)-C(9)	121.39(6)

O(1)-Cs(1)-C(9)	137.47(5)
O(3)-Cs(1)-C(9)	90.06(6)
C(1A)#1-Cs(1)-C(9)	41.32(6)
C(1A)-Cs(1)-C(9)	23.16(6)
C(2)#1-Cs(1)-C(9)	47.78(6)
O(7)-Cs(2)-O(5)	124.80(7)
O(7)-Cs(2)-O(6)	53.43(7)
O(5)-Cs(2)-O(6)	74.06(8)
O(4)-Cs(2)-O(8)	79.81(5)
O(6)-Cs(2)-O(8)	99.83(7)
O(4)-Cs(2)-C(24)	169.65(6)
O(6)-Cs(2)-C(24)	104.11(6)
O(7)-Cs(2)-C(16A)	118.50(7)
O(5)-Cs(2)-C(16A)	102.23(6)
O(8)-Cs(2)-C(16A)	118.27(6)
O(7)-Cs(2)-O(9)	107.38(6)
O(5)-Cs(2)-O(9)	95.19(6)
O(8)-Cs(2)-O(9)	54.74(5)
C(16A)-Cs(2)-O(9)	104.87(6)
O(4)-Cs(2)-C(16B)	146.18(14)
O(6)-Cs(2)-C(16B)	107.22(14)
C(24)-Cs(2)-C(16B)	26.29(14)
O(9)-Cs(2)-C(16B)	119.27(14)
O(4)-Cs(2)-C(23)	165.99(6)
O(6)-Cs(2)-C(23)	115.33(6)
C(24)-Cs(2)-C(23)	24.17(6)
O(9)-Cs(2)-C(23)	103.08(5)
O(7)-Cs(2)-C(16B)#2	133.91(13)
O(5)-Cs(2)-C(16B)#2	89.66(13)
O(8)-Cs(2)-C(16B)#2	125.09(13)
C(16A)-Cs(2)-C(16B)#2	15.43(13)
C(16B)-Cs(2)-C(16B)#2	22.3(3)
O(7)-Cs(2)-C(25)	85.82(6)
O(5)-Cs(2)-C(25)	104.05(5)
O(8)-Cs(2)-C(25)	120.93(5)
C(16A)-Cs(2)-C(25)	42.29(6)
C(16B)-Cs(2)-C(25)	32.37(14)
C(16B)#2-Cs(2)-C(25)	53.87(13)
C(32)-O(1)-Cs(1)	103.03(16)
C(33)-O(2)-C(34)	113.3(2)
C(34)-O(2)-Cs(1)	122.22(16)
C(35)-O(3)-Cs(1)	107.16(14)
C(38)-O(4)-C(37)	115.2(2)
C(37)-O(4)-Cs(2)	105.80(15)
C(37)-O(4)-Cs(1)	113.42(15)
C(39)-O(5)-C(40)	113.4(3)
C(40)-O(5)-Cs(2)	108.7(2)
C(41)-O(6)-Cs(2)	105.6(2)
C(43)-O(7)-C(44)	112.4(3)
C(44)-O(7)-Cs(2)	119.5(2)
C(46)-O(8)-Cs(2)	107.51(16)
C(48)-O(9)-C(47)	114.4(2)
C(47)-O(9)-Cs(1)	99.65(15)
C(47)-O(9)-Cs(2)	112.39(17)
C(50)-O(10)-C(49)	112.3(3)
C(49)-O(10)-Cs(1)	119.85(16)
C(16A)-C(16B)-C(16B)#2	46.0(5)
C(16A)-C(16B)-C(24)	66.8(5)
C(16B)#2-C(16B)-C(24)	112.8(8)
C(16A)#2-C(16B)-C(17)#2	65.4(5)
C(24)-C(16B)-C(17)#2	136.8(6)
C(16A)#2-C(16B)-C(25)	155.3(7)
C(24)-C(16B)-C(25)	46.8(3)
C(16A)-C(16B)-C(30)	156.9(7)
C(16B)#2-C(16B)-C(30)	156.9(9)
C(17)#2-C(16B)-C(30)	46.6(2)
C(16A)-C(16B)-Cs(2)	76.0(5)
O(9)-Cs(1)-C(9)	147.52(5)
C(1B)-Cs(1)-C(9)	24.8(3)
O(4)-Cs(1)-C(9)	82.75(5)
C(1B)#1-Cs(1)-C(9)	39.6(2)
O(7)-Cs(2)-O(4)	88.77(6)
O(4)-Cs(2)-O(5)	54.56(5)
O(4)-Cs(2)-O(6)	69.45(6)
O(7)-Cs(2)-O(8)	53.97(7)
O(5)-Cs(2)-O(8)	133.53(5)
O(7)-Cs(2)-C(24)	93.69(6)
O(5)-Cs(2)-C(24)	116.49(6)
O(8)-Cs(2)-C(24)	109.72(6)
O(4)-Cs(2)-C(16A)	152.46(6)
O(6)-Cs(2)-C(16A)	122.40(7)
C(24)-Cs(2)-C(16A)	25.46(6)
O(4)-Cs(2)-O(9)	67.19(5)
O(6)-Cs(2)-O(9)	132.67(6)
C(24)-Cs(2)-O(9)	121.30(6)
O(7)-Cs(2)-C(16B)	116.98(14)
O(5)-Cs(2)-C(16B)	91.76(14)
O(8)-Cs(2)-C(16B)	132.51(14)
C(16A)-Cs(2)-C(16B)	16.71(14)
O(7)-Cs(2)-C(23)	84.59(6)
O(5)-Cs(2)-C(23)	138.67(6)
O(8)-Cs(2)-C(23)	86.31(6)
C(16A)-Cs(2)-C(23)	37.40(6)
C(16B)-Cs(2)-C(23)	46.98(14)
O(4)-Cs(2)-C(16B)#2	137.09(13)
O(6)-Cs(2)-C(16B)#2	127.66(13)
C(24)-Cs(2)-C(16B)#2	40.47(13)
O(9)-Cs(2)-C(16B)#2	97.44(13)
C(23)-Cs(2)-C(16B)#2	51.72(13)
O(4)-Cs(2)-C(25)	147.63(5)
O(6)-Cs(2)-C(25)	81.87(6)
C(24)-Cs(2)-C(25)	23.23(6)
O(9)-Cs(2)-C(25)	144.48(5)
C(23)-Cs(2)-C(25)	44.14(6)
C(32)-O(1)-C(31)	112.1(3)
C(31)-O(1)-Cs(1)	101.0(2)
C(33)-O(2)-Cs(1)	121.35(18)
C(35)-O(3)-C(36)	111.01(19)
C(36)-O(3)-Cs(1)	105.99(14)
C(38)-O(4)-Cs(2)	118.32(13)
C(38)-O(4)-Cs(1)	92.47(16)
Cs(2)-O(4)-Cs(1)	111.43(6)
C(39)-O(5)-Cs(2)	109.59(17)
C(41)-O(6)-C(42)	113.0(3)
C(42)-O(6)-Cs(2)	111.3(2)
C(43)-O(7)-Cs(2)	122.4(2)
C(46)-O(8)-C(45)	112.8(2)
C(45)-O(8)-Cs(2)	103.26(19)
C(48)-O(9)-Cs(1)	115.28(15)
C(48)-O(9)-Cs(2)	104.36(15)
Cs(1)-O(9)-Cs(2)	111.00(5)
C(50)-O(10)-Cs(1)	118.6(2)
C(16A)-C(16B)-C(16A)#2	91.0(7)
C(16A)#2-C(16B)-C(16B)#2	45.0(5)
C(16A)#2-C(16B)-C(24)	157.8(8)
C(16A)-C(16B)-C(17)#2	156.4(8)
C(16B)#2-C(16B)-C(17)#2	110.4(8)
C(16A)-C(16B)-C(25)	113.5(6)
C(16B)#2-C(16B)-C(25)	159.5(9)
C(17)#2-C(16B)-C(25)	90.0(4)
C(16A)#2-C(16B)-C(30)	111.9(6)
C(24)-C(16B)-C(30)	90.2(4)
C(25)-C(16B)-C(30)	43.43(19)
C(16A)#2-C(16B)-Cs(2)	102.6(5)

C(16B)#2-C(16B)-Cs(2)	89.2(6)	C(24)-C(16B)-Cs(2)	72.2(3)
C(17)#2-C(16B)-Cs(2)	108.5(3)	C(25)-C(16B)-Cs(2)	82.2(2)
C(30)-C(16B)-Cs(2)	96.0(3)	C(16A)-C(16B)-Cs(2)#2	87.8(5)
C(16A)#2-C(16B)-Cs(2)#2	61.9(4)	C(16B)#2-C(16B)-Cs(2)#2	68.6(6)
C(24)-C(16B)-Cs(2)#2	115.5(4)	C(17)#2-C(16B)-Cs(2)#2	80.6(3)
C(25)-C(16B)-Cs(2)#2	118.8(3)	C(30)-C(16B)-Cs(2)#2	104.5(3)
Cs(2)-C(16B)-Cs(2)#2	157.7(3)	C(1A)-C(1B)-C(1A)#1	91.8(11)
C(1A)-C(1B)-C(1B)#1	46.5(8)	C(1A)#1-C(1B)-C(1B)#1	45.3(8)
C(1A)-C(1B)-C(9)	65.8(8)	C(1A)#1-C(1B)-C(9)	157.4(12)
C(1B)#1-C(1B)-C(9)	112.3(14)	C(1A)-C(1B)-C(2)#1	155.8(13)
C(1A)#1-C(1B)-C(2)#1	64.3(7)	C(1B)#1-C(1B)-C(2)#1	109.4(14)
C(9)-C(1B)-C(2)#1	137.8(9)	C(1A)-C(1B)-C(10)	113.2(11)
C(1A)#1-C(1B)-C(10)	154.7(11)	C(1B)#1-C(1B)-C(10)	159.5(16)
C(9)-C(1B)-C(10)	47.4(4)	C(2)#1-C(1B)-C(10)	90.5(6)
C(1A)-C(1B)-C(15)	156.6(12)	C(1A)#1-C(1B)-C(15)	111.1(10)
C(1B)#1-C(1B)-C(15)	156.0(15)	C(9)-C(1B)-C(15)	91.1(6)
C(2)#1-C(1B)-C(15)	46.8(4)	C(10)-C(1B)-C(15)	43.7(3)
C(1A)-C(1B)-Cs(1)	89.1(8)	C(1A)#1-C(1B)-Cs(1)	83.5(7)
C(1B)#1-C(1B)-Cs(1)	84.7(10)	C(9)-C(1B)-Cs(1)	93.1(6)
C(2)#1-C(1B)-Cs(1)	84.6(5)	C(10)-C(1B)-Cs(1)	92.8(4)
C(15)-C(1B)-Cs(1)	88.8(4)	C(1A)-C(1B)-Cs(1)#1	74.9(7)
C(1A)#1-C(1B)-Cs(1)#1	80.0(7)	C(1B)#1-C(1B)-Cs(1)#1	71.9(10)
C(9)-C(1B)-Cs(1)#1	95.8(5)	C(2)#1-C(1B)-Cs(1)#1	102.9(6)
C(10)-C(1B)-Cs(1)#1	109.1(5)	C(15)-C(1B)-Cs(1)#1	112.7(5)
Cs(1)-C(1B)-Cs(1)#1	156.5(4)		

Symmetry transformations used to generate equivalent atoms: #1 -x+2,-y+1,-z+1; #2 -x+1,-y+1,-z+2

Table S10. Anisotropic displacement parameters (Å^2). The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11} + ... + 2\ h\ k\ a^*\ b^*\ U_{12}]$.

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(1A)	0.023(1)	0.022(1)	0.014(1)	-0.006(1)	-0.001(1)	-0.008(1)
C(2)	0.046(1)	0.021(1)	0.016(1)	-0.004(1)	-0.004(1)	-0.013(1)
C(3)	0.040(1)	0.033(1)	0.021(1)	-0.007(1)	-0.006(1)	-0.016(1)
C(4)	0.049(2)	0.038(2)	0.034(1)	-0.004(1)	-0.010(1)	-0.017(1)
C(5)	0.058(2)	0.042(2)	0.061(2)	0.009(2)	-0.024(2)	-0.011(2)
C(6)	0.043(2)	0.048(2)	0.068(2)	-0.003(2)	-0.019(2)	-0.008(2)
C(7)	0.040(2)	0.047(2)	0.034(1)	-0.005(1)	-0.011(1)	-0.015(1)
C(8)	0.035(1)	0.038(1)	0.020(1)	-0.007(1)	-0.006(1)	-0.016(1)
C(9)	0.024(1)	0.040(1)	0.017(1)	-0.010(1)	-0.002(1)	-0.009(1)
C(10)	0.034(1)	0.033(1)	0.019(1)	-0.010(1)	-0.003(1)	-0.007(1)
C(11)	0.037(1)	0.042(2)	0.026(1)	-0.012(1)	-0.002(1)	-0.011(1)
C(12)	0.042(2)	0.052(2)	0.036(1)	-0.016(1)	0.002(1)	-0.023(1)
C(13)	0.056(2)	0.043(2)	0.033(1)	-0.009(1)	0.001(1)	-0.024(2)
C(14)	0.049(2)	0.034(1)	0.026(1)	-0.008(1)	-0.005(1)	-0.013(1)
C(15)	0.037(1)	0.029(1)	0.017(1)	-0.009(1)	-0.004(1)	-0.007(1)
C(16A)	0.018(1)	0.016(1)	0.014(1)	-0.002(1)	-0.003(1)	-0.005(1)
C(17)	0.017(1)	0.047(1)	0.015(1)	-0.004(1)	-0.002(1)	-0.011(1)
C(18)	0.026(1)	0.032(1)	0.015(1)	-0.002(1)	-0.001(1)	0.000(1)
C(19)	0.030(1)	0.042(2)	0.023(1)	-0.004(1)	-0.003(1)	-0.005(1)
C(20)	0.038(2)	0.044(2)	0.037(1)	-0.008(1)	-0.010(1)	-0.011(1)
C(21)	0.049(2)	0.038(2)	0.039(2)	-0.003(1)	-0.009(1)	-0.015(1)
C(22)	0.037(1)	0.029(1)	0.027(1)	0.000(1)	-0.003(1)	-0.006(1)
C(23)	0.028(1)	0.028(1)	0.016(1)	0.001(1)	-0.001(1)	-0.001(1)
C(24)	0.041(1)	0.015(1)	0.017(1)	-0.003(1)	0.002(1)	-0.009(1)
C(25)	0.034(1)	0.034(1)	0.016(1)	-0.004(1)	0.000(1)	-0.020(1)
C(26)	0.036(1)	0.038(1)	0.026(1)	-0.006(1)	-0.001(1)	-0.018(1)
C(27)	0.035(1)	0.038(1)	0.033(1)	-0.009(1)	-0.001(1)	-0.012(1)
C(28)	0.031(1)	0.047(2)	0.028(1)	-0.008(1)	-0.003(1)	-0.013(1)
C(29)	0.032(1)	0.044(2)	0.022(1)	-0.008(1)	0.001(1)	-0.020(1)
C(30)	0.032(1)	0.039(1)	0.016(1)	-0.007(1)	0.002(1)	-0.021(1)
C(31)	0.051(2)	0.040(2)	0.045(2)	-0.005(1)	-0.012(2)	0.009(1)
C(32)	0.027(1)	0.046(2)	0.044(2)	-0.014(1)	-0.005(1)	-0.002(1)
C(33)	0.029(1)	0.048(2)	0.049(2)	-0.014(1)	-0.008(1)	-0.014(1)
C(34)	0.038(1)	0.027(1)	0.034(1)	-0.003(1)	0.000(1)	-0.017(1)
C(35)	0.036(1)	0.025(1)	0.026(1)	0.000(1)	0.004(1)	-0.011(1)
C(36)	0.035(1)	0.029(1)	0.023(1)	0.001(1)	0.002(1)	-0.006(1)
C(37)	0.037(1)	0.031(1)	0.029(1)	-0.002(1)	0.005(1)	-0.003(1)
C(38)	0.037(1)	0.058(2)	0.018(1)	-0.002(1)	-0.002(1)	-0.024(1)
C(39)	0.049(2)	0.056(2)	0.024(1)	-0.009(1)	0.004(1)	-0.034(2)
C(40)	0.096(3)	0.093(3)	0.029(1)	0.000(2)	0.004(2)	-0.073(3)
C(41)	0.049(2)	0.078(3)	0.041(2)	-0.005(2)	-0.005(2)	-0.014(2)
C(42)	0.054(2)	0.048(2)	0.038(2)	-0.004(1)	-0.005(1)	0.006(2)
C(43)	0.063(2)	0.038(2)	0.045(2)	0.001(1)	-0.002(2)	0.005(2)
C(44)	0.067(2)	0.031(2)	0.043(2)	-0.005(1)	0.003(2)	-0.017(2)
C(45)	0.068(2)	0.036(2)	0.036(1)	-0.014(1)	0.002(1)	-0.020(2)
C(46)	0.052(2)	0.041(2)	0.032(1)	-0.010(1)	-0.007(1)	-0.020(1)
C(47)	0.040(2)	0.044(2)	0.036(1)	-0.014(1)	0.002(1)	-0.022(1)
C(48)	0.039(1)	0.038(1)	0.021(1)	-0.005(1)	-0.008(1)	-0.010(1)
C(49)	0.041(1)	0.033(1)	0.022(1)	-0.002(1)	-0.003(1)	-0.013(1)
C(50)	0.145(5)	0.041(2)	0.056(2)	-0.010(2)	-0.015(3)	-0.043(3)
Cs(1)	0.031(1)	0.022(1)	0.016(1)	-0.005(1)	-0.003(1)	-0.008(1)
Cs(2)	0.038(1)	0.031(1)	0.017(1)	-0.002(1)	-0.002(1)	-0.010(1)
O(1)	0.036(1)	0.040(1)	0.031(1)	-0.007(1)	-0.009(1)	0.003(1)
O(2)	0.030(1)	0.029(1)	0.040(1)	-0.006(1)	-0.005(1)	-0.011(1)
O(3)	0.032(1)	0.023(1)	0.020(1)	0.000(1)	0.002(1)	-0.009(1)
O(4)	0.030(1)	0.033(1)	0.022(1)	0.000(1)	-0.002(1)	-0.012(1)
O(5)	0.063(2)	0.069(2)	0.027(1)	-0.012(1)	0.016(1)	-0.042(1)
O(6)	0.040(1)	0.059(2)	0.037(1)	-0.008(1)	-0.001(1)	-0.003(1)
O(7)	0.058(2)	0.030(1)	0.042(1)	0.002(1)	-0.006(1)	-0.006(1)
O(8)	0.052(1)	0.033(1)	0.025(1)	-0.008(1)	-0.004(1)	-0.015(1)
O(9)	0.035(1)	0.031(1)	0.025(1)	-0.008(1)	-0.004(1)	-0.014(1)
O(10)	0.086(2)	0.032(1)	0.025(1)	-0.005(1)	-0.001(1)	-0.027(1)
C(16B)	0.016(4)	0.018(4)	0.014(3)	-0.003(2)	-0.001(2)	-0.003(3)
C(1B)	0.021(7)	0.013(6)	0.011(5)	0.000(4)	-0.002(4)	-0.008(4)

Table S11. Hydrogen coordinates and isotropic displacement parameters (Å²).

	x	y	z	U_{eq}
H(4)	0.8796	0.9115	0.3791	0.047
H(5)	0.6641	1.0039	0.3804	0.067
H(6)	0.5148	0.8898	0.4296	0.065
H(7)	0.5713	0.6849	0.4907	0.047
H(11)	0.5997	0.4848	0.5394	0.041
H(12)	0.5504	0.3004	0.6027	0.050
H(13)	0.7091	0.1298	0.6425	0.051
H(14)	0.9168	0.1445	0.6223	0.042
H(19)	0.8710	0.5485	1.0455	0.040
H(20)	0.8707	0.7387	1.0661	0.047
H(21)	0.6913	0.9029	1.0603	0.050
H(22)	0.5058	0.8813	1.0344	0.040
H(26)	0.3228	0.8937	1.0093	0.038
H(27)	0.1177	0.9773	0.9853	0.043
H(28)	0.0099	0.8540	0.9573	0.042
H(29)	0.1051	0.6468	0.9565	0.037
H(31A)	1.2419	0.3074	0.7643	0.059
H(31B)	1.3104	0.3064	0.6826	0.059
H(32)	1.3603	0.4786	0.6100	0.048
H(33A)	1.2613	0.6976	0.6630	0.048
H(33B)	1.3135	0.7011	0.5826	0.048
H(34A)	1.1506	0.8767	0.5550	0.039
H(34B)	1.0803	0.8741	0.6329	0.039
H(35A)	0.9370	0.9685	0.5511	0.037
H(35B)	0.9700	0.8396	0.5270	0.037
H(36A)	0.7494	0.8221	0.5668	0.038
H(36B)	0.7303	0.9598	0.5775	0.038
H(37A)	0.6655	0.8862	0.6984	0.043
H(37B)	0.5698	0.8836	0.6509	0.043
H(38)	0.5663	0.6949	0.6184	0.044
H(39A)	0.5761	0.4820	0.6810	0.047
H(39B)	0.6814	0.4876	0.7219	0.047
H(40A)	0.4583	0.3953	0.7797	0.099
H(40B)	0.4101	0.4626	0.8434	0.099
H(40C)	0.5554	0.3922	0.8280	0.099
H(41A)	0.2193	0.8878	0.7537	0.087
H(41B)	0.2479	0.8160	0.8299	0.087
H(41C)	0.3178	0.7552	0.7652	0.087
H(42A)	0.3094	0.9832	0.8480	0.062
H(42B)	0.2756	1.0621	0.7736	0.062
H(43A)	0.4839	1.0797	0.7392	0.066
H(43B)	0.4188	1.1388	0.8052	0.066
H(44A)	0.6134	1.1055	0.8459	0.057
H(44B)	0.6795	1.0640	0.7746	0.057
H(45A)	0.8092	0.9822	0.8624	0.055
H(45B)	0.7084	0.9048	0.9060	0.055
H(46A)	0.8892	0.7190	0.8912	0.047
H(46B)	0.9714	0.8126	0.8433	0.047
H(47)	1.0467	0.6278	0.8124	0.046
H(48A)	0.8976	0.4980	0.9034	0.039
H(48B)	1.0214	0.4482	0.8522	0.039
H(49A)	0.8890	0.3060	0.8900	0.039
H(49B)	0.7796	0.4100	0.8530	0.039
H(50A)	0.8082	0.2443	0.7896	0.114
H(50B)	0.9342	0.1651	0.8207	0.114
H(50C)	0.9381	0.2127	0.7388	0.114

1.3 Additional crystallographic details for compound **2**

Compound **2** additionally exhibits a positional disorder at the central carbon bond in both discussed types **2**A (Fig. 4a) and **2**B (Fig. 4b) what has to be denoted for completeness (Fig. S3).

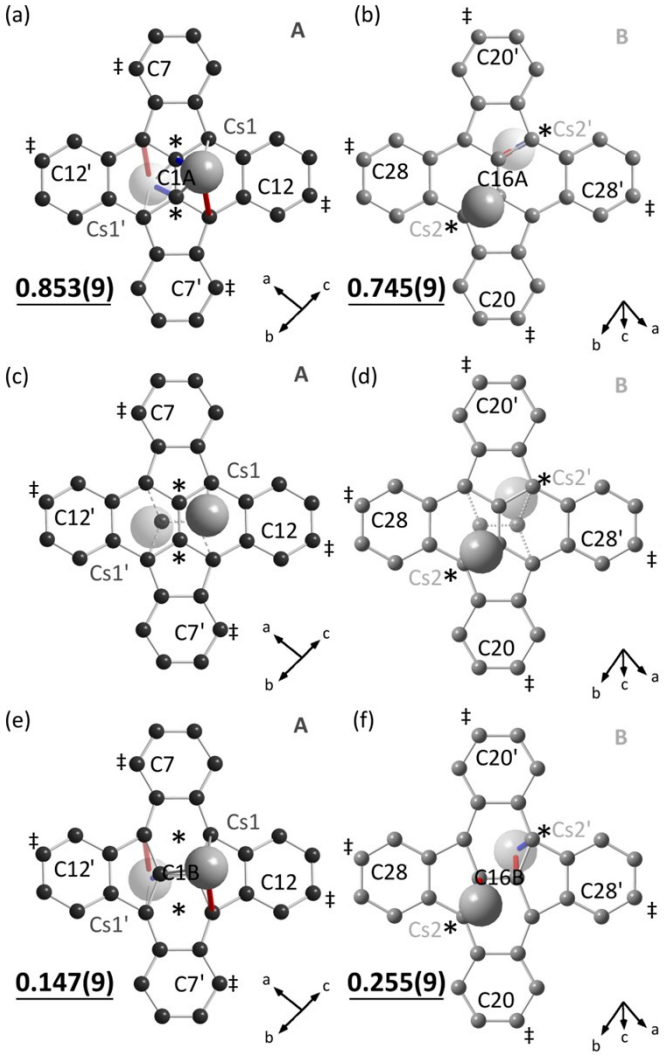


Figure S3. Depiction of the detected positional disorder for both types A and B in the cesium complex **2**. (a+b) Represent the major constitution found in the crystalline state of **2** with a frequency in the crystalline state of 85.3(9) % for **2**A resp. 74.5(9) % for **2**B. (c+d) The positional disorder is given in dashed lines, accounting for a formal rotation of the charged $DOPT^{2-}$ plane by 90°. (e+f) The de facto coordination modes onto the formally rotated dianionic DOPT plane is given with similar coordination modes of η:η for type **2**A resp. η':η' for type **2**B with a minor occupancy of the C1B positions of 14.7(9) % (**2**A) resp. of the C16B positions of 25.5(9) % (**2**B). (*) Carbon species with shortest d(Cs-C_π) bond are highlighted. The overall shape of the dianionic DOPT plane is fully maintained as represented in Fig. 4c and Fig. 4d.

Due to the exhibited C_2 symmetry of the PAH dianion, a rotation of the L_{DOPT}^{2-} plane by 180° with fixed Cs^+ positions fully maintains the discussed η:η resp. η':η' coordination of the contacting Cs(I)-ions and cannot be distinguished by X-ray analysis. Structural solution of the crystal data set for compound **2** reveals an occupancy ratio for the rotational isomers of type A with 0.853(9) (Fig. 4a) : 0.147(9) at positions C1A / C1B and for type B with 0.745(9) (Fig. 4b) : 0.255(9) at positions C16A / C16B. The detected rotational isomerization results in an additional set of coordination geometries for **2**A (Fig. S3e) and **2**B (Fig. S3f) resulting in slightly different cesium to carbon distances of d(Cs-C_π)$_{\eta4,0.147(9)}$ = 319 - 360 pm (vs. d(Cs-C_π)$_{\eta4,0.853(9)}$ = 323 - 360 pm) resp. d(Cs-C_π)$_{\eta2,0.255(9)}$ = 322 - 334 pm (vs. of d(Cs-C_π)$_{\eta2,0.745(9)}$ = 322 - 325 pm).

2. Experimental details

General: Synthesis was carried out in Schlenk type glassware using 4.8 argon (99.998 %). The gas was further purified and dried by passing it over (a) activated carbon, (b) BTS-copper catalyst (Sigma-Aldrich), (c) pre-dried molecular sieve (3 Å) and (d) Sicapent® with indicator (Merck Millipore). THF (technical grade), 18C6 (≥99.0 %), tetraethylene glycol dimethyl ether (tetraglyme, ≥99 %) and rubidium ingots (99.6 %) were received from Sigma Aldrich. Cesium (purum, ≥99.5 %) was purchased from Fluka. 5,6;11,12-di-*o*-phenylenetetracene (DOPT, L_{DOPT}) was synthesized according to our earlier published reaction procedure[1] and sublimed (ca. 350°C) under a gentle flow of argon prior to use. THF was dried with Na/benzophenone and stored over activated molecular sieve (4 Å). Hygroscopic 1,4,7,10,13,16-hexaoxacyclooctadecane (18-crown-6 ether, 18C6) was pre-dried in a ball tube vacuum distillation apparatus (ca. 200°C and 10^{-3} mbar), dissolved in dry THF (0.1 M), degassed and stored static over activated molecular sieve for at least 1 week (4 Å). As received tetraglyme was dried over activated molecular sieve and degassed prior to use. Suitable crystals for structural analysis were obtained upon cooling from concentrated solution at 0°C to -30°C (Fig. S4). The X-ray diffraction data were collected on a Bruker AXS Mach3 Apex II diffractometer at 100 K in perfluorinated polyether. Structures were solved by direct methods (SHELXT) and refined by full-matrix least-squares (SHELXL).[2] Graphics of the molecules and unit cells were generated with the latest version of software PLATON[3] and Diamond[4]. All rotational ellipsoids of the ORTEP[5]-plots were drawn with 50 % probability level.

Due to the proven potent carcinogenic activity of some polyarenes an uncontrolled release of PAHs contributes to a serious environmental contamination which is potentially hazardous for health.[6] Thus handling and synthesis of PAHs has to be taken with great care and any exposure should be precluded. In general the use of any silicon based grease should be avoided due to the proven chance of irreversible incorporation of $(H_3C)_2Si$-groups into the π-perimeter via reductive reaction with alkaline metal.[7] Nonetheless, we never observed for DOPT any competing reaction during synthesis or significant disturbance of the reaction solution impeding crystal growth. *Note: The very limited solubility of the compounds (e.g., in THF) together with their extremely high air and temperature sensitivity prevented their full analytical characterization testing several different crystals of different batches with reproducible uniform crystal shape.*

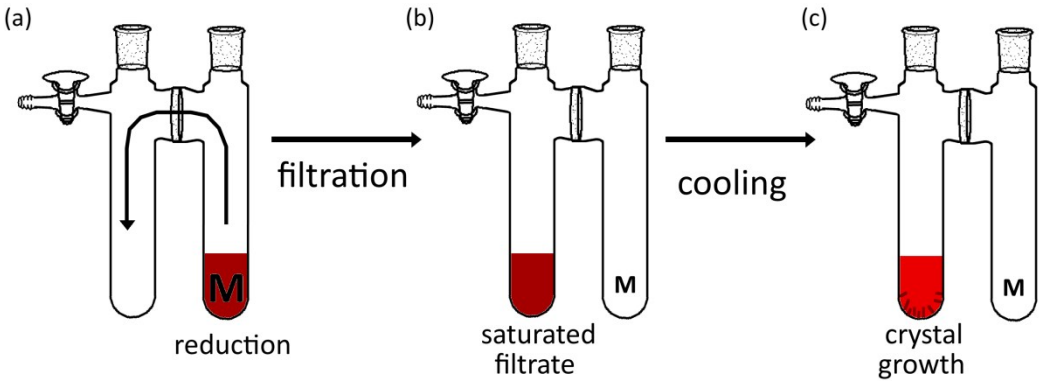


Figure S4. H-shaped flasks† were used for successful synthesis and crystallization of the highly sensitive complexes **1** and **2**. (a) An excess of the particular alkaline metal (M = Rb, Cs) is placed in one leg of the flame-dried flask, together with predired solvent and purified PAH compound. (b) After sealing of the flask and full conversion to the desired anion separation from excessive metal (M) can be carried out through the embedded fritted glass (P4(P16)) without air contact. (c) A saturated clear solution is obtained, suitable for crystal growth at lower temperatures.

Preparation of DOPT Dirubidium bis(18-crown-6-ether), {[((18C6-κ^6*O*)Rb$^+$)$_2$-*μ*-(*η*:*η*-L$_{DOPT}^{2-}$)][((18C6-κ^6*O*)Rb$^+$)$_2$-*μ*-(*η*′:*η*′-L$_{DOPT}^{2-}$)](THF$_{solv}$)$_2$} 1. DOPT (10 mg, 0.027 mmol, 1 eq.) was reacted with freshly cut rubidium (100 mg, excess) in dry THF and 18C6 (3 mL, 0.1 M, ca. 10 eq.). The mixture was placed in one side of an H-shaped flask† (Fig. S4) with an embedded fritted glass (medium sized, P4(P16); 10 - 16 µm) between the connection of both legs. Sonication at 0°C for 1 h instantly converted the suspension to an instable deep-blue solution due to solvation of excessive rubidium by the crown-ether. After extended sonification the deep-red reaction mixture was filtered into the empty leg of the H-shaped flask† and stored at -30°C for 7 d. Highly air and moisture sensitive red rhombic crystals of **1** were obtained directly from the pale-red mother liquor. Crystals of **1** have to be kept under a solvent atmosphere to preclude instant decomposition.

Preparation of DOPT Dicesium bis(tetraglyme), [*μ*-(*η*:*η*-L$_{DOPT}^{2-}$)$_{0.5}$((tetraglyme-κ^4*O*)(tetraglyme-κ^2*O*)Cs$^+$)$_2$-*μ*-(*η*′:*η*′-L$_{DOPT}^{2-}$)$_{0.5}$]$_n$ 2. *Note: Tetraglyme might decompose when exposed to the applied metal-organic reactants owing to rapid ether-cleavage at elevated temperatures. Thus all manipulations have to be carried out well below 0°C.* DOPT (10 mg, 0.027 mmol, 1 eq.) was reacted with flakes of cesium metal (100 mg, excess) in a solution of dry tetraglyme (3 mL) at 0°C then following the same procedure as described for **1** given above. Finally, the highly viscous reaction mixture was filtered into the empty leg of the H-shaped flask† eventually by applying of vaccum and finally at -30°C for 7 d. Extremely air, moisture and temperature sensitive deep-red rhombs of **2** were obtained directly from the almost colorless mother liquor. Attempts of aprotic washing (e.g. cold *n*-pentane, *n*-hexane) or separation of the crystals from the mother liquor at low temperatures failed due to a quick decomposition of the material which is accompanied by a color change from deep-red to green and finally dark-blue. Crystals of **2** have best to be kept under a minimum amount of tetraglyme solvent to preclude instant decomposition.

3. References

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