Supporting information for

Synthesis and Magnetic Properties of Cerium Macrocyclic Complexes with tmtaaH_2, Tetramethyldibenzotetraaza[14]-annulene

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1. $Ce(tmtaa)_2*(C_5H_{12})_{0.5}$

1.1 Labeling Diagram

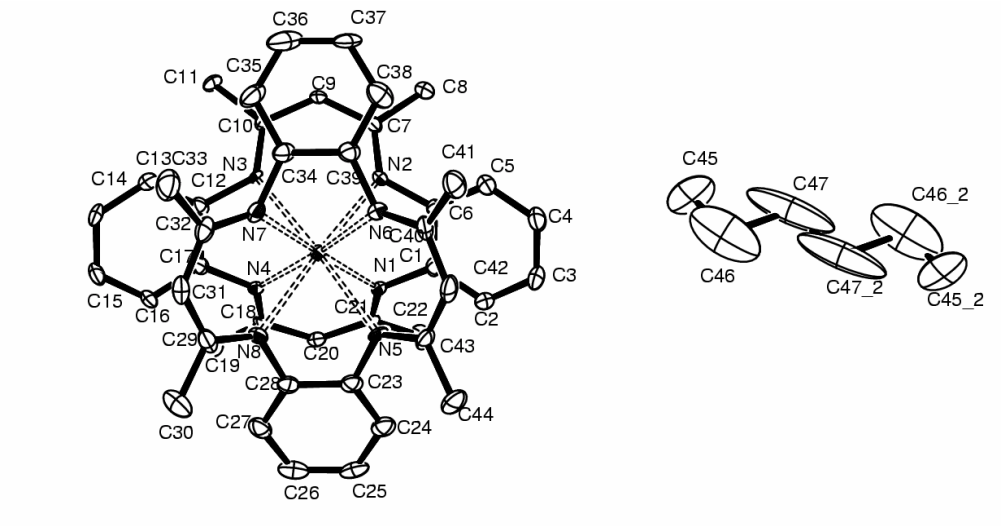

Figure S1. ORTEP diagram of $Ce(tmtaa)_2*(C_5H_{12})_{0.5}$ (50% probability ellipsoids). The atom labels bearing _2 are symmetry related positions. C45 in the disordered pentane molecule has 0.5 occupancy.

1.2 Experimental Details

Data Collection

A fragment of a block green-like crystal of $C_{46.50}H_{44}CeN_8$ having approximate dimensions of 0.32 x 0.24 x 0.16 mm was mounted on a glass fiber using Paratone N hydrocarbon oil. All measurements were made on a Bruker SMART CCD [10] CCD area detector with graphite monochromated MoKα radiation.

Cell constants and an orientation matrix, obtained from a least-squares refinement using the measured positions of 6639 centered reflections with $I > 10\sigma(I)$ in the range $2.2 < \theta < 24.7^o$ corresponded to a primitive Triclinic cell with dimensions:

a = 11.417(1) Å	α = 107.720(2) ^{o}
b = 11.543(1) Å	β = 90.261(2) ^{o}
c = 16.419(2) Å	γ = 107.884(2) ^{o}
V = 1950.1(4)Å^3	

For Z = 2 and F.W. = 855.02, the calculated density is 1.456 g/cm^3.

Analysis of the systematic absences allowed the space group to be uniquely determined to be:

P-1

The data were collected at a temperature of 136(2) K. Frames corresponding to an arbitrary hemisphere of data were collected using ω scans of 0.3^o counted for a total of 10 seconds per frame.

Data Reduction

Data were integrated by the program SAINT[11] to a maximum θ value of 24.72°. The data were corrected for Lorentz and polarization effects. Data were analyzed for agreement and possible absorption using XPREP[12]. An empirical absorption correction based on comparison of redundant and equivalent reflections was applied using SADABS[13]. (Tmax = 0.8297, Tmin = 0.6977). Of the 9863 reflections that were collected, 6189 were unique (R_{int} = 0.0425); equivalent reflections were merged. No decay correction was applied.

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. Non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included in calculated positions using a riding model, but not refined. However, no H atoms corresponding to the disordered pentane were included in the refinement. The final cycle of full-matrix least-squares refinement[3] was based on 6189 reflections (all data) and 514 variable parameters and converged (largest parameter shift was 0.000 times its esd) with conventional unweighted and weighted agreement factors of:

$$R_1 = \Sigma||Fo| - |Fc|| / \Sigma|Fo| = 0.0468 \text{ for 5527 data with } I > 2\sigma(I)$$

$$wR_2 = [(\Sigma w (|Fo|^2 - |Fc|^2)^2 / \Sigma w |Fo|^2)]^{1/2} = 0.1238$$

The standard deviation of an observation of unit weight[4] was 1.046. The weighting scheme was based on counting statistics and included a factor (q = 0.082) to downweight the intense reflections. The maximum and minimum peaks on the final difference Fourier map corresponded to 2.009 and -2.059 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5]. Anomalous dispersion effects were included in Fcalc[6]; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbel[8]. All calculations were performed using the SHELXTL[9] crystallographic software package of Bruker Analytical X-ray Systems Inc.

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{46.50}H_{44}CeN_8$
Formula Weight	855.02
Crystal Color, Habit	block, green
Crystal Dimensions	0.32 x 0.24 x 0.16 mm
Crystal System	Triclinic
Lattice Type	primitive
Lattice Parameters	a = 11.417(1) Å b = 11.543(1) Å c = 16.419(2) Å α= 107.720(2) ^{o} β= 90.261(2) ^{o} γ = 107.884(2)^o V = 1950.1(4) Å^3
Space Group	P-1
Z value	2
D_{calc}	1.456 g/cm^3
F_{000}	874
μ(MoK)	1.21 cm^{-1}

B. Intensity Measurements

Diffractometer	Bruker SMART CCD
Radiation	MoK(λ = 0.71073 Å) graphite monochromated
Detector Position	60.00 mm
Exposure Time	10 seconds per frame.
Scan Type	ω (0.3 degrees per frame)
θ_{max}	24.72^o
No. of Reflections Measured	Total: 9863 Unique: 6189 (R_{int} = 0.0425)
Corrections	Lorentz-polarization Absorption (Tmax = 0.8297, Tmin = 0.6977)

C. Structure Solution and Refinement

Structure Solution	direct (SHELXS-97 (Sheldrick, 1997))
Refinement	Full-matrix least-squares
Function Minimized	$\Sigma w(\|F_o\|^2 - \|F_c\|^2)^2$
Least Squares Weighting scheme	$w = 1/[\sigma^2(F_o^2) + (qP)^2 + 1.347P]$
	where $P = [F_o^2 + 2F_c^2]/3$
q-factor	0.082
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>2.00σ(I))	5527
No. Variables	514
Reflection/Parameter Ratio	10.75
Residuals: R; wR_2; Rall	0.0468; 0.1238; 0.0524
Goodness of Fit Indicator	1.046
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	2.009 $e^-/Å^3$
Minimum peak in Final Diff. Map	-2.059 $e^-/Å^3$

1.3 Tables

Table 1. Atomic coordinates and U_{iso}/U_{eq} and occupancy

atom	x	y	z	U_{eq}	Occupancy
C1	0.4781(4)	0.1794(4)	0.1603(3)	0.018(1)	1
C2	0.4776(4)	0.0746(4)	0.1864(3)	0.023(1)	1
C3	0.5835(5)	0.0724(5)	0.2263(3)	0.029(1)	1
C4	0.6917(5)	0.1783(5)	0.2439(3)	0.029(1)	1
C5	0.6932(4)	0.2828(5)	0.2198(3)	0.023(1)	1
C6	0.5888(4)	0.2852(4)	0.1766(3)	0.016(1)	1
C7	0.6607(4)	0.4574(4)	0.1158(3)	0.018(1)	1
C8	0.7709(4)	0.4171(5)	0.0843(3)	0.025(1)	1
C9	0.6445(4)	0.5611(4)	0.0947(3)	0.018(1)	1
C10	0.5419(4)	0.6025(4)	0.1014(3)	0.017(1)	1
C11	0.5473(4)	0.7061(4)	0.0608(3)	0.020(1)	1
C12	0.3326(4)	0.5822(4)	0.1393(3)	0.016(1)	1
C13	0.3261(4)	0.7063(4)	0.1616(3)	0.020(1)	1
C14	0.2142(4)	0.7286(4)	0.1679(3)	0.024(1)	1
C15	0.1056(4)	0.6275(5)	0.1554(3)	0.026(1)	1
C16	0.1092(4)	0.5040(4)	0.1360(3)	0.020(1)	1
C17	0.2209(4)	0.4770(4)	0.1253(3)	0.015(1)	1
C18	0.1618(4)	0.2478(4)	0.0544(3)	0.018(1)	1
C19	0.0508(5)	0.2431(5)	0.0008(3)	0.028(1)	1
C20	0.1853(4)	0.1307(4)	0.0365(3)	0.017(1)	1
C21	0.2880(4)	0.1067(4)	0.0665(3)	0.017(1)	1
C22	0.3024(4)	-0.0222(4)	0.0197(3)	0.025(1)	1
C23	0.2177(4)	0.2088(4)	0.2875(3)	0.020(1)	1
C24	0.1653(4)	0.0748(4)	0.2593(3)	0.025(1)	1
C25	0.0446(5)	0.0146(5)	0.2210(3)	0.029(1)	1
C26	-0.0244(5)	0.0876(5)	0.2070(3)	0.031(1)	1
C27	0.0252(4)	0.2196(5)	0.2313(3)	0.026(1)	1
C28	0.1455(4)	0.2828(4)	0.2741(3)	0.020(1)	1
C29	0.1612(4)	0.5072(5)	0.3392(3)	0.024(1)	1
C30	0.0315(5)	0.4724(6)	0.3669(4)	0.037(1)	1
C31	0.2304(5)	0.6377(5)	0.3659(3)	0.027(1)	1
C32	0.3567(4)	0.6958(4)	0.3641(3)	0.024(1)	1
C33	0.4081(5)	0.8405(5)	0.4110(4)	0.038(1)	1
C34	0.5591(4)	0.6816(4)	0.3266(3)	0.021(1)	1
C35	0.6197(5)	0.7941(5)	0.3067(3)	0.032(1)	1
C36	0.7447(5)	0.8297(5)	0.3001(4)	0.041(2)	1
C37	0.8116(5)	0.7558(5)	0.3111(3)	0.027(1)	1
C38	0.7565(5)	0.6436(5)	0.3282(3)	0.031(1)	1
C39	0.6295(4)	0.6049(4)	0.3376(3)	0.021(1)	1
C40	0.5934(4)	0.4441(4)	0.4082(3)	0.020(1)	1
C41	0.7090(5)	0.5185(5)	0.4708(3)	0.030(1)	1
C42	0.5181(5)	0.3314(5)	0.4211(3)	0.026(1)	1
C43	0.3983(4)	0.2569(4)	0.3844(3)	0.022(1)	1
C44	0.3311(5)	0.1556(5)	0.4237(3)	0.034(1)	1
C45	0.9284(19)	0.1803(17)	0.4516(10)	0.073(6)	0.50
C46	0.9036(11)	0.1078(15)	0.4883(8)	0.143(6)	1
C47	1.0088(12)	0.0502(14)	0.4804(10)	0.171(12)	1
N1	0.3723(3)	0.1964(3)	0.1285(2)	0.016(1)	1
N2	0.5791(3)	0.3923(3)	0.1569(2)	0.014(1)	1
N3	0.4436(3)	0.5510(3)	0.1371(2)	0.013(1)	1

N4	0.2353(3)	0.3551(3)	0.1103(2)	0.014(1)	1
N5	0.3418(3)	0.2770(3)	0.3213(2)	0.018(1)	1
N6	0.5600(3)	0.4860(3)	0.3479(2)	0.019(1)	1
N7	0.4291(4)	0.6296(4)	0.3278(2)	0.020(1)	1
N8	0.2087(3)	0.4173(4)	0.2978(2)	0.018(1)	1
Ce1	0.3973(1)	0.4121(1)	0.2285(1)	0.015(1)	1

U_{eq} is defined as one third of the orthogonalized U_{ij} tensor

Table 2. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C1	0.018(2)	0.019(2)	0.021(2)	0.009(2)	0.007(2)	0.011(2)
C2	0.020(2)	0.022(2)	0.034(3)	0.016(2)	0.009(2)	0.007(2)
C3	0.030(3)	0.031(3)	0.039(3)	0.023(2)	0.005(2)	0.016(2)
C4	0.023(3)	0.038(3)	0.037(3)	0.022(2)	0.004(2)	0.016(2)
C5	0.018(2)	0.027(3)	0.029(2)	0.014(2)	0.005(2)	0.010(2)
C6	0.016(2)	0.018(2)	0.021(2)	0.010(2)	0.007(2)	0.009(2)
C7	0.010(2)	0.022(2)	0.021(2)	0.009(2)	0.002(2)	0.006(2)
C8	0.015(2)	0.028(3)	0.037(3)	0.019(2)	0.007(2)	0.007(2)
C9	0.011(2)	0.021(2)	0.029(2)	0.017(2)	0.007(2)	0.004(2)
C10	0.018(2)	0.014(2)	0.019(2)	0.009(2)	0.000(2)	0.004(2)
C11	0.022(2)	0.020(2)	0.024(2)	0.017(2)	0.008(2)	0.007(2)
C12	0.012(2)	0.025(2)	0.016(2)	0.012(2)	0.003(2)	0.008(2)
C13	0.019(2)	0.018(2)	0.025(2)	0.012(2)	0.003(2)	0.004(2)
C14	0.028(3)	0.019(2)	0.036(3)	0.017(2)	0.006(2)	0.014(2)
C15	0.017(2)	0.035(3)	0.036(3)	0.019(2)	0.005(2)	0.015(2)
C16	0.010(2)	0.025(2)	0.029(2)	0.013(2)	0.002(2)	0.007(2)
C17	0.013(2)	0.021(2)	0.015(2)	0.012(2)	0.001(2)	0.007(2)
C18	0.010(2)	0.027(3)	0.022(2)	0.015(2)	0.007(2)	0.006(2)
C19	0.025(3)	0.028(3)	0.030(3)	0.008(2)	-0.006(2)	0.008(2)
C20	0.015(2)	0.017(2)	0.021(2)	0.009(2)	0.004(2)	0.002(2)
C21	0.018(2)	0.020(2)	0.020(2)	0.014(2)	0.010(2)	0.007(2)
C22	0.025(3)	0.021(2)	0.031(3)	0.010(2)	0.005(2)	0.008(2)
C23	0.020(2)	0.025(2)	0.018(2)	0.012(2)	0.006(2)	0.005(2)
C24	0.029(3)	0.024(3)	0.025(2)	0.011(2)	0.012(2)	0.008(2)
C25	0.034(3)	0.023(3)	0.025(3)	0.011(2)	0.012(2)	-0.001(2)
C26	0.024(3)	0.035(3)	0.025(3)	0.009(2)	0.008(2)	-0.002(2)
C27	0.021(2)	0.035(3)	0.025(2)	0.013(2)	0.010(2)	0.011(2)
C28	0.020(2)	0.024(2)	0.019(2)	0.011(2)	0.010(2)	0.007(2)
C29	0.020(2)	0.036(3)	0.024(2)	0.015(2)	0.006(2)	0.016(2)
C30	0.022(3)	0.045(3)	0.039(3)	0.004(3)	0.007(2)	0.013(3)
C31	0.030(3)	0.028(3)	0.030(3)	0.010(2)	0.003(2)	0.020(2)
C32	0.030(3)	0.023(3)	0.023(2)	0.010(2)	-0.004(2)	0.011(2)
C33	0.041(3)	0.027(3)	0.046(3)	0.007(2)	0.000(3)	0.017(3)
C34	0.020(2)	0.023(2)	0.019(2)	0.012(2)	-0.003(2)	0.002(2)
C35	0.035(3)	0.024(3)	0.038(3)	0.019(2)	-0.006(2)	0.001(2)
C36	0.034(3)	0.038(3)	0.039(3)	0.021(3)	-0.007(3)	-0.013(3)
C37	0.018(3)	0.025(3)	0.028(3)	0.012(2)	-0.003(2)	-0.010(2)
C38	0.024(3)	0.044(3)	0.023(2)	0.011(2)	-0.001(2)	0.011(2)
C39	0.018(2)	0.023(2)	0.021(2)	0.009(2)	-0.002(2)	0.003(2)
C40	0.021(2)	0.027(3)	0.020(2)	0.010(2)	0.004(2)	0.016(2)
C41	0.028(3)	0.037(3)	0.029(3)	0.013(2)	-0.002(2)	0.013(2)
C42	0.034(3)	0.032(3)	0.025(2)	0.017(2)	0.004(2)	0.020(2)
C43	0.029(3)	0.021(2)	0.025(2)	0.016(2)	0.011(2)	0.013(2)
C44	0.039(3)	0.035(3)	0.036(3)	0.026(2)	0.006(2)	0.009(2)
C45	0.116(15)	0.072(11)	0.040(8)	0.030(8)	0.050(9)	0.032(10)
C46	0.087(8)	0.180(14)	0.088(8)	-0.053(9)	0.026(7)	0.033(9)
C47	0.048(6)	0.20(2)	0.121(13)	-0.090(12)	0.044(6)	-0.027(10)
N1	0.014(2)	0.015(2)	0.023(2)	0.013(2)	0.007(2)	0.006(2)
N2	0.009(2)	0.017(2)	0.020(2)	0.009(2)	0.003(2)	0.005(2)
N3	0.013(2)	0.015(2)	0.020(2)	0.016(2)	0.007(2)	0.006(2)
N4	0.012(2)	0.015(2)	0.020(2)	0.010(2)	0.007(2)	0.005(2)

N5	0.020(2)	0.017(2)	0.021(2)	0.015(2)	0.009(2)	0.004(2)
N6	0.017(2)	0.018(2)	0.023(2)	0.011(2)	0.004(2)	0.005(2)
N7	0.022(2)	0.017(2)	0.025(2)	0.012(2)	0.001(2)	0.007(2)
N8	0.013(2)	0.022(2)	0.021(2)	0.010(2)	0.006(2)	0.006(2)
Ce1	0.014(1)	0.018(1)	0.019(1)	0.012(1)	0.003(1)	0.006(1)

The general temperature factor expression:

$\exp(-2\Pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}\,hl + 2b^*c^*U_{23}kl))$

Table 3. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
C1	C2	1.399(6)	C1	N1	1.405(6)
C1	C6	1.418(6)	C1	Ce1	3.004(4)
C2	C3	1.383(7)	C2	H2	0.9500
C3	C4	1.399(7)	C3	H3	0.9500
C4	C5	1.374(7)	C4	H4	0.9500
C5	C6	1.396(6)	C5	H5	0.9500
C6	N2	1.405(6)	C6	Ce1	2.980(4)
C7	N2	1.332(5)	C7	C9	1.405(6)
C7	C8	1.512(6)	C8	H8A	0.9800
C8	H8B	0.9800	C8	H8C	0.9800
C9	C10	1.389(6)	C9	H9	0.9500
C10	N3	1.328(5)	C10	C11	1.523(6)
C11	H11A	0.9800	C11	H11B	0.9800
C11	H11C	0.9800	C12	C13	1.392(6)
C12	N3	1.418(5)	C12	C17	1.425(6)
C12	Ce1	3.030(4)	C13	C14	1.377(6)
C13	H13	0.9500	C14	C15	1.380(7)
C14	H14	0.9500	C15	C16	1.377(6)
C15	H15	0.9500	C16	C17	1.402(6)
C16	H16	0.9500	C17	N4	1.414(5)
C17	Ce1	3.026(4)	C18	N4	1.320(6)
C18	C20	1.403(6)	C18	C19	1.517(6)
C19	H19A	0.9800	C19	H19B	0.9800
C19	H19C	0.9800	C20	C21	1.404(6)
C20	H20	0.9500	C21	N1	1.331(6)
C21	C22	1.512(6)	C22	H22A	0.9800
C22	H22B	0.9800	C22	H22C	0.9800
C23	C24	1.400(6)	C23	N5	1.406(6)
C23	C28	1.415(6)	C23	Ce1	3.016(4)
C24	C25	1.383(7)	C24	H24	0.9500
C25	C26	1.381(7)	C25	H25	0.9500
C26	C27	1.379(7)	C26	H26	0.9500
C27	C28	1.407(7)	C27	H27	0.9500
C28	N8	1.422(6)	C28	Ce1	3.019(4)
C29	N8	1.324(6)	C29	C31	1.397(7)
C29	C30	1.520(7)	C30	H30A	0.9800
C30	H30B	0.9800	C30	H30C	0.9800
C31	C32	1.396(7)	C31	H31	0.9500
C32	N7	1.319(6)	C32	C33	1.529(7)
C33	H33A	0.9800	C33	H33B	0.9800
C33	H33C	0.9800	C34	C35	1.407(6)
C34	C39	1.413(6)	C34	N7	1.422(6)
C34	Ce1	3.012(4)	C35	C36	1.373(8)
C35	H35	0.9500	C36	C37	1.355(8)
C36	H36	0.9500	C37	C38	1.369(7)
C37	H37	0.9500	C38	C39	1.405(7)
C38	H38	0.9500	C39	N6	1.419(6)
C39	Ce1	3.016(4)	C40	N6	1.327(6)
C40	C42	1.399(7)	C40	C41	1.513(7)
C41	H41A	0.9800	C41	H41B	0.9800
C41	H41C	0.9800	C42	C43	1.388(7)

C42	H42	0.9500	C43	N5	1.333(6)
C43	C44	1.512(6)	C44	H44A	0.9800
C44	H44B	0.9800	C44	H44C	0.9800
C45	C46	1.14(2)	C46	C47	1.53(2)
C47	C47#1	1.45(4)	N1	Ce1	2.460(4)
N2	Ce1	2.428(3)	N3	Ce1	2.460(3)
N4	Ce1	2.462(4)	N5	Ce1	2.450(3)
N6	Ce1	2.449(4)	N7	Ce1	2.456(4)
N8	Ce1	2.448(4)			

Table 4. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
C2	C1	N1	124.7(4)	C2	C1	C6	119.0(4)
N1	C1	C6	115.8(4)	C2	C1	Ce1	136.3(3)
N1	C1	Ce1	54.1(2)	C6	C1	Ce1	75.3(2)
C3	C2	C1	121.0(4)	C3	C2	H2	119.5
C1	C2	H2	119.5	C2	C3	C4	119.8(4)
C2	C3	H3	120.1	C4	C3	H3	120.1
C5	C4	C3	120.0(4)	C5	C4	H4	120.0
C3	C4	H4	120.0	C4	C5	C6	121.3(4)
C4	C5	H5	119.4	C6	C5	H5	119.4
C5	C6	N2	124.7(4)	C5	C6	C1	119.0(4)
N2	C6	C1	115.8(4)	C5	C6	Ce1	134.3(3)
N2	C6	Ce1	53.7(2)	C1	C6	Ce1	77.2(2)
N2	C7	C9	121.8(4)	N2	C7	C8	121.2(4)
C9	C7	C8	116.8(4)	C7	C8	H8A	109.5
C7	C8	H8B	109.5	H8A	C8	H8B	109.5
C7	C8	H8C	109.5	H8A	C8	H8C	109.5
H8B	C8	H8C	109.5	C10	C9	C7	128.6(4)
C10	C9	H9	115.7	C7	C9	H9	115.7
N3	C10	C9	122.2(4)	N3	C10	C11	122.5(4)
C9	C10	C11	115.2(4)	C10	C11	H11A	109.5
C10	C11	H11B	109.5	H11A	C11	H11B	109.5
C10	C11	H11C	109.5	H11A	C11	H11C	109.5
H11B	C11	H11C	109.5	C13	C12	N3	125.1(4)
C13	C12	C17	118.9(4)	N3	C12	C17	115.7(4)
C13	C12	Ce1	137.7(3)	N3	C12	Ce1	53.29(19)
C17	C12	Ce1	76.2(2)	C14	C13	C12	121.3(4)
C14	C13	H13	119.3	C12	C13	H13	119.3
C13	C14	C15	120.2(4)	C13	C14	H14	119.9
C15	C14	H14	119.9	C16	C15	C14	119.8(4)
C16	C15	H15	120.1	C14	C15	H15	120.1
C15	C16	C17	121.5(4)	C15	C16	H16	119.2
C17	C16	H16	119.2	C16	C17	N4	125.8(4)
C16	C17	C12	118.1(4)	N4	C17	C12	115.7(4)
C16	C17	Ce1	137.0(3)	N4	C17	Ce1	53.5(2)
C12	C17	Ce1	76.5(2)	N4	C18	C20	122.1(4)
N4	C18	C19	122.9(4)	C20	C18	C19	114.9(4)
C18	C19	H19A	109.5	C18	C19	H19B	109.5
H19A	C19	H19B	109.5	C18	C19	H19C	109.5
H19A	C19	H19C	109.5	H19B	C19	H19C	109.5
C18	C20	C21	128.8(4)	C18	C20	H20	115.6
C21	C20	H20	115.6	N1	C21	C20	121.4(4)
N1	C21	C22	122.2(4)	C20	C21	C22	116.2(4)
C21	C22	H22A	109.5	C21	C22	H22B	109.5
H22A	C22	H22B	109.5	C21	C22	H22C	109.5
H22A	C22	H22C	109.5	H22B	C22	H22C	109.5
C24	C23	N5	125.0(4)	C24	C23	C28	118.5(4)
N5	C23	C28	116.2(4)	C24	C23	Ce1	137.8(3)
N5	C23	Ce1	53.3(2)	C28	C23	Ce1	76.5(2)
C25	C24	C23	121.4(5)	C25	C24	H24	119.3
C23	C24	H24	119.3	C26	C25	C24	119.5(5)

C26	C25	H25	120.2	C24	C25	H25	120.2
C27	C26	C25	120.8(5)	C27	C26	H26	119.6
C25	C26	H26	119.6	C26	C27	C28	120.4(5)
C26	C27	H27	119.8	C28	C27	H27	119.8
C27	C28	C23	119.2(4)	C27	C28	N8	125.3(4)
C23	C28	N8	115.1(4)	C27	C28	Ce1	136.5(3)
C23	C28	Ce1	76.3(2)	N8	C28	Ce1	53.2(2)
N8	C29	C31	122.1(4)	N8	C29	C30	121.3(4)
C31	C29	C30	116.4(4)	C29	C30	H30A	109.5
C29	C30	H30B	109.5	H30A	C30	H30B	109.5
C29	C30	H30C	109.5	H30A	C30	H30C	109.5
H30B	C30	H30C	109.5	C32	C31	C29	129.0(4)
C32	C31	H31	115.5	C29	C31	H31	115.5
N7	C32	C31	122.4(4)	N7	C32	C33	121.6(4)
C31	C32	C33	116.0(4)	C32	C33	H33A	109.5
C32	C33	H33B	109.5	H33A	C33	H33B	109.5
C32	C33	H33C	109.5	H33A	C33	H33C	109.5
H33B	C33	H33C	109.5	C35	C34	C39	118.3(4)
C35	C34	N7	125.6(4)	C39	C34	N7	115.8(4)
C35	C34	Ce1	136.3(3)	C39	C34	Ce1	76.6(3)
N7	C34	Ce1	53.8(2)	C36	C35	C34	120.8(5)
C36	C35	H35	119.6	C34	C35	H35	119.6
C37	C36	C35	120.5(5)	C37	C36	H36	119.8
C35	C36	H36	119.8	C36	C37	C38	121.1(5)
C36	C37	H37	119.4	C38	C37	H37	119.4
C37	C38	C39	120.4(5)	C37	C38	H38	119.8
C39	C38	H38	119.8	C38	C39	C34	118.9(4)
C38	C39	N6	125.5(4)	C34	C39	N6	115.1(4)
C38	C39	Ce1	136.2(3)	C34	C39	Ce1	76.3(2)
N6	C39	Ce1	53.3(2)	N6	C40	C42	121.2(4)
N6	C40	C41	122.7(4)	C42	C40	C41	115.9(4)
C40	C41	H41A	109.5	C40	C41	H41B	109.5
H41A	C41	H41B	109.5	C40	C41	H41C	109.5
H41A	C41	H41C	109.5	H41B	C41	H41C	109.5
C43	C42	C40	128.6(4)	C43	C42	H42	115.7
C40	C42	H42	115.7	N5	C43	C42	122.5(4)
N5	C43	C44	121.0(4)	C42	C43	C44	116.3(4)
C43	C44	H44A	109.5	C43	C44	H44B	109.5
H44A	C44	H44B	109.5	C43	C44	H44C	109.5
H44A	C44	H44C	109.5	H44B	C44	H44C	109.5
C45	C46	C47	105.7(15)	C47#1	C47	C46	114.3(13)
C21	N1	C1	124.3(4)	C21	N1	Ce1	137.1(3)
C1	N1	Ce1	98.3(3)	C7	N2	C6	123.9(4)
C7	N2	Ce1	137.3(3)	C6	N2	Ce1	98.6(2)
C10	N3	C12	124.5(3)	C10	N3	Ce1	135.8(3)
C12	N3	Ce1	99.2(2)	C18	N4	C17	124.0(4)
C18	N4	Ce1	136.2(3)	C17	N4	Ce1	99.0(2)
C43	N5	C23	123.4(4)	C43	N5	Ce1	137.2(3)
C23	N5	Ce1	99.3(3)	C40	N6	C39	122.5(4)
C40	N6	Ce1	138.4(3)	C39	N6	Ce1	99.0(3)
C32	N7	C34	125.4(4)	C32	N7	Ce1	135.5(3)
C34	N7	Ce1	98.4(3)	C29	N8	C28	124.6(4)
C29	N8	Ce1	136.1(3)	C28	N8	Ce1	99.1(3)
N2	Ce1	N8	176.39(11)	N2	Ce1	N6	76.58(12)
N8	Ce1	N6	104.61(12)	N2	Ce1	N5	112.41(12)
N8	Ce1	N5	65.22(12)	N6	Ce1	N5	69.90(12)
N2	Ce1	N7	112.80(12)	N8	Ce1	N7	70.69(13)

N6	Ce1	N7	64.95(12)	N5	Ce1	N7	103.33(12)
N2	Ce1	N1	65.42(12)	N8	Ce1	N1	111.09(12)
N6	Ce1	N1	113.98(12)	N5	Ce1	N1	77.40(12)
N7	Ce1	N1	178.20(11)	N2	Ce1	N3	70.47(12)
N8	Ce1	N3	111.90(12)	N6	Ce1	N3	111.44(12)
N5	Ce1	N3	177.12(10)	N7	Ce1	N3	75.32(12)
N1	Ce1	N3	104.03(12)	N2	Ce1	N4	104.33(11)
N8	Ce1	N4	74.75(12)	N6	Ce1	N4	175.64(11)
N5	Ce1	N4	113.26(12)	N7	Ce1	N4	110.97(12)
N1	Ce1	N4	70.06(12)	N3	Ce1	N4	65.25(11)
N2	Ce1	C6	27.78(12)	N8	Ce1	C6	149.26(12)
N6	Ce1	C6	70.04(12)	N5	Ce1	C6	85.10(12)
N7	Ce1	C6	127.23(12)	N1	Ce1	C6	51.07(12)
N3	Ce1	C6	97.74(11)	N4	Ce1	C6	112.78(12)
N2	Ce1	C1	50.89(12)	N8	Ce1	C1	125.58(12)
N6	Ce1	C1	86.76(12)	N5	Ce1	C1	69.94(12)
N7	Ce1	C1	151.11(12)	N1	Ce1	C1	27.56(12)
N3	Ce1	C1	112.49(12)	N4	Ce1	C1	97.12(12)
C6	Ce1	C1	27.42(11)	N2	Ce1	C34	85.29(12)
N8	Ce1	C34	98.11(12)	N6	Ce1	C34	50.63(12)
N5	Ce1	C34	112.00(12)	N7	Ce1	C34	27.85(13)
N1	Ce1	C34	150.35(12)	N3	Ce1	C34	68.00(12)
N4	Ce1	C34	125.05(12)	C6	Ce1	C34	100.45(12)
C1	Ce1	C34	126.88(12)	N2	Ce1	C23	125.95(12)
N8	Ce1	C23	50.69(12)	N6	Ce1	C23	96.83(12)
N5	Ce1	C23	27.39(12)	N7	Ce1	C23	112.01(12)
N1	Ce1	C23	69.39(12)	N3	Ce1	C23	150.71(12)
N4	Ce1	C23	86.10(12)	C6	Ce1	C23	99.03(12)
C1	Ce1	C23	75.43(12)	C34	Ce1	C23	131.32(12)

Table 5. Torsion Angles(°)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
N1	C1	C2	C3	-172.3(4)	C6	C1	C2	C3	-0.9(7)
Ce1	C1	C2	C3	-100.2(5)	C1	C2	C3	C4	2.3(8)
C2	C3	C4	C5	-1.4(8)	C3	C4	C5	C6	-0.9(8)
C4	C5	C6	N2	173.8(4)	C4	C5	C6	C1	2.2(7)
C4	C5	C6	Ce1	103.1(5)	C2	C1	C6	C5	-1.3(6)
N1	C1	C6	C5	170.8(4)	Ce1	C1	C6	C5	133.9(4)
C2	C1	C6	N2	-173.6(4)	N1	C1	C6	N2	-1.5(6)
Ce1	C1	C6	N2	-38.4(3)	C2	C1	C6	Ce1	-135.2(4)
N1	C1	C6	Ce1	36.9(3)	N2	C7	C9	C10	-8.9(7)
C8	C7	C9	C10	167.2(4)	C7	C9	C10	N3	5.6(7)
C7	C9	C10	C11	-171.3(4)	N3	C12	C13	C14	-175.4(4)
C17	C12	C13	C14	-1.9(6)	Ce1	C12	C13	C14	-103.7(5)
C12	C13	C14	C15	2.7(7)	C13	C14	C15	C16	-0.5(7)
C14	C15	C16	C17	-2.3(7)	C15	C16	C17	N4	176.1(4)
C15	C16	C17	C12	3.0(6)	C15	C16	C17	Ce1	103.7(5)
C13	C12	C17	C16	-0.9(6)	N3	C12	C17	C16	173.2(4)
Ce1	C12	C17	C16	136.5(4)	C13	C12	C17	N4	-174.7(4)
N3	C12	C17	N4	-0.6(5)	Ce1	C12	C17	N4	-37.4(3)
C13	C12	C17	Ce1	-137.3(4)	N3	C12	C17	Ce1	36.8(3)
N4	C18	C20	C21	-5.2(7)	C19	C18	C20	C21	171.2(4)
C18	C20	C21	N1	9.7(7)	C18	C20	C21	C22	-165.5(4)
N5	C23	C24	C25	-175.2(4)	C28	C23	C24	C25	-1.7(7)
Ce1	C23	C24	C25	-103.5(5)	C23	C24	C25	C26	2.7(7)
C24	C25	C26	C27	-0.5(7)	C25	C26	C27	C28	-2.7(7)
C26	C27	C28	C23	3.6(7)	C26	C27	C28	N8	176.3(4)
C26	C27	C28	Ce1	104.9(5)	C24	C23	C28	C27	-1.4(6)
N5	C23	C28	C27	172.6(4)	Ce1	C23	C28	C27	136.0(4)
C24	C23	C28	N8	-174.8(4)	N5	C23	C28	N8	-0.8(6)
Ce1	C23	C28	N8	-37.4(3)	C24	C23	C28	Ce1	-137.4(4)
N5	C23	C28	Ce1	36.6(3)	N8	C29	C31	C32	-7.7(8)
C30	C29	C31	C32	167.2(5)	C29	C31	C32	N7	6.0(8)
C29	C31	C32	C33	-170.8(5)	C39	C34	C35	C36	-1.3(7)
N7	C34	C35	C36	-174.3(5)	Ce1	C34	C35	C36	-101.9(6)
C34	C35	C36	C37	1.1(8)	C35	C36	C37	C38	0.7(8)
C36	C37	C38	C39	-2.3(8)	C37	C38	C39	C34	2.0(7)
C37	C38	C39	N6	174.2(4)	C37	C38	C39	Ce1	102.6(6)
C35	C34	C39	C38	-0.2(6)	N7	C34	C39	C38	173.4(4)
Ce1	C34	C39	C38	135.5(4)	C35	C34	C39	N6	-173.2(4)
N7	C34	C39	N6	0.5(6)	Ce1	C34	C39	N6	-37.4(3)
C35	C34	C39	Ce1	-135.8(4)	N7	C34	C39	Ce1	37.9(3)
N6	C40	C42	C43	-8.1(8)	C41	C40	C42	C43	167.8(5)
C40	C42	C43	N5	8.1(8)	C40	C42	C43	C44	-168.2(5)
C45	C46	C47	C47#1	-177.0(17)	C20	C21	N1	C1	-178.8(4)
C22	C21	N1	C1	-3.9(6)	C20	C21	N1	Ce1	8.1(7)
C22	C21	N1	Ce1	-177.0(3)	C2	C1	N1	C21	-49.4(6)
C6	C1	N1	C21	138.9(4)	Ce1	C1	N1	C21	-175.2(5)
C2	C1	N1	Ce1	125.8(4)	C6	C1	N1	Ce1	-45.8(4)
C9	C7	N2	C6	177.6(4)	C8	C7	N2	C6	1.7(7)
C9	C7	N2	Ce1	-8.6(7)	C8	C7	N2	Ce1	175.5(3)
C5	C6	N2	C7	52.7(6)	C1	C6	N2	C7	-135.5(4)
Ce1	C6	N2	C7	175.8(5)	C5	C6	N2	Ce1	-123.0(4)

C1	C6	N2	Ce1	48.7(4)	C9	C10	N3	C12	-174.6(4)
C11	C10	N3	C12	2.1(6)	C9	C10	N3	Ce1	14.9(7)
C11	C10	N3	Ce1	-168.3(3)	C13	C12	N3	C10	-46.1(6)
C17	C12	N3	C10	140.2(4)	Ce1	C12	N3	C10	-173.3(5)
C13	C12	N3	Ce1	127.2(4)	C17	C12	N3	Ce1	-46.5(4)
C20	C18	N4	C17	174.8(4)	C19	C18	N4	C17	-1.3(6)
C20	C18	N4	Ce1	-17.6(6)	C19	C18	N4	Ce1	166.3(3)
C16	C17	N4	C18	45.3(6)	C12	C17	N4	C18	-141.4(4)
Ce1	C17	N4	C18	171.3(4)	C16	C17	N4	Ce1	-126.0(4)
C12	C17	N4	Ce1	47.3(4)	C42	C43	N5	C23	-175.2(4)
C44	C43	N5	C23	0.9(7)	C42	C43	N5	Ce1	7.6(7)
C44	C43	N5	Ce1	-176.4(3)	C24	C23	N5	C43	-50.8(7)
C28	C23	N5	C43	135.6(4)	Ce1	C23	N5	C43	-178.1(5)
C24	C23	N5	Ce1	127.3(4)	C28	C23	N5	Ce1	-46.3(4)
C42	C40	N6	C39	174.9(4)	C41	C40	N6	C39	-0.7(7)
C42	C40	N6	Ce1	-7.5(7)	C41	C40	N6	Ce1	176.8(3)
C38	C39	N6	C40	53.4(7)	C34	C39	N6	C40	-134.2(4)
Ce1	C39	N6	C40	178.4(5)	C38	C39	N6	Ce1	-125.0(4)
C34	C39	N6	Ce1	47.4(4)	C31	C32	N7	C34	-177.4(4)
C33	C32	N7	C34	-0.7(7)	C31	C32	N7	Ce1	14.9(7)
C33	C32	N7	Ce1	-168.5(3)	C35	C34	N7	C32	-46.0(7)
C39	C34	N7	C32	140.9(5)	Ce1	C34	N7	C32	-171.4(5)
C35	C34	N7	Ce1	125.4(4)	C39	C34	N7	Ce1	-47.8(4)
C31	C29	N8	C28	175.7(4)	C30	C29	N8	C28	1.0(7)
C31	C29	N8	Ce1	-11.6(7)	C30	C29	N8	Ce1	173.8(3)
C27	C28	N8	C29	49.5(6)	C23	C28	N8	C29	-137.6(4)
Ce1	C28	N8	C29	174.9(5)	C27	C28	N8	Ce1	-125.5(4)
C23	C28	N8	Ce1	47.4(4)	C7	N2	Ce1	N8	148.1(15)
C6	N2	Ce1	N8	-37.1(18)	C7	N2	Ce1	N6	-102.3(4)
C6	N2	Ce1	N6	72.5(3)	C7	N2	Ce1	N5	-163.5(4)
C6	N2	Ce1	N5	11.3(3)	C7	N2	Ce1	N7	-47.2(4)
C6	N2	Ce1	N7	127.6(2)	C7	N2	Ce1	N1	133.1(4)
C6	N2	Ce1	N1	-52.1(2)	C7	N2	Ce1	N3	16.7(4)
C6	N2	Ce1	N3	-168.6(3)	C7	N2	Ce1	N4	73.3(4)
C6	N2	Ce1	N4	-111.9(3)	C7	N2	Ce1	C6	-174.8(6)
C7	N2	Ce1	C1	160.9(5)	C6	N2	Ce1	C1	-24.3(2)
C7	N2	Ce1	C34	-51.7(4)	C6	N2	Ce1	C34	123.1(3)
C7	N2	Ce1	C23	169.0(4)	C6	N2	Ce1	C23	-16.2(3)
C29	N8	Ce1	N2	-175.9(15)	C28	N8	Ce1	N2	-2.0(18)
C29	N8	Ce1	N6	75.3(4)	C28	N8	Ce1	N6	-110.7(3)
C29	N8	Ce1	N5	134.5(5)	C28	N8	Ce1	N5	-51.5(3)
C29	N8	Ce1	N7	19.0(4)	C28	N8	Ce1	N7	-167.0(3)
C29	N8	Ce1	N1	-161.3(4)	C28	N8	Ce1	N1	12.7(3)
C29	N8	Ce1	N3	-45.5(5)	C28	N8	Ce1	N3	128.5(2)
C29	N8	Ce1	N4	-100.2(4)	C28	N8	Ce1	N4	73.7(3)
C29	N8	Ce1	C6	150.7(4)	C28	N8	Ce1	C6	-35.3(4)
C29	N8	Ce1	C1	171.9(4)	C28	N8	Ce1	C1	-14.1(3)
C29	N8	Ce1	C34	24.0(4)	C28	N8	Ce1	C34	-162.0(3)
C29	N8	Ce1	C23	162.2(5)	C28	N8	Ce1	C23	-23.9(2)
C40	N6	Ce1	N2	-106.8(5)	C39	N6	Ce1	N2	71.1(3)
C40	N6	Ce1	N8	69.7(5)	C39	N6	Ce1	N8	-112.4(3)
C40	N6	Ce1	N5	13.5(4)	C39	N6	Ce1	N5	-168.6(3)
C40	N6	Ce1	N7	129.8(5)	C39	N6	Ce1	N7	-52.3(3)
C40	N6	Ce1	N1	-51.8(5)	C39	N6	Ce1	N1	126.1(3)
C40	N6	Ce1	N3	-169.2(4)	C39	N6	Ce1	N3	8.7(3)
C40	N6	Ce1	N4	150.6(12)	C39	N6	Ce1	N4	-31.4(15)
C40	N6	Ce1	C6	-78.5(5)	C39	N6	Ce1	C6	99.4(3)

C40	N6	Ce1	C1	-56.3(5)	C39	N6	Ce1	C1	121.6(3)
C40	N6	Ce1	C34	158.2(5)	C39	N6	Ce1	C34	-23.9(2)
C40	N6	Ce1	C23	18.6(5)	C39	N6	Ce1	C23	-163.5(3)
C43	N5	Ce1	N2	51.9(5)	C23	N5	Ce1	N2	-125.8(3)
C43	N5	Ce1	N8	-131.1(5)	C23	N5	Ce1	N8	51.3(3)
C43	N5	Ce1	N6	-13.4(4)	C23	N5	Ce1	N6	169.0(3)
C43	N5	Ce1	N7	-70.0(5)	C23	N5	Ce1	N7	112.3(3)
C43	N5	Ce1	N1	108.3(5)	C23	N5	Ce1	N1	-69.4(3)
C43	N5	Ce1	N3	-132(2)	C23	N5	Ce1	N3	51(2)
C43	N5	Ce1	N4	169.9(4)	C23	N5	Ce1	N4	-7.8(3)
C43	N5	Ce1	C6	57.1(4)	C23	N5	Ce1	C6	-120.5(3)
C43	N5	Ce1	C1	80.6(4)	C23	N5	Ce1	C1	-97.0(3)
C43	N5	Ce1	C34	-42.2(5)	C23	N5	Ce1	C34	140.2(3)
C43	N5	Ce1	C23	177.7(6)	C32	N7	Ce1	N2	160.4(4)
C34	N7	Ce1	N2	-9.6(3)	C32	N7	Ce1	N8	-20.7(4)
C34	N7	Ce1	N8	169.4(3)	C32	N7	Ce1	N6	-138.0(5)
C34	N7	Ce1	N6	52.1(2)	C32	N7	Ce1	N5	-78.0(4)
C34	N7	Ce1	N5	112.1(2)	C32	N7	Ce1	N1	168(3)
C34	N7	Ce1	N1	-2(3)	C32	N7	Ce1	N3	99.4(4)
C34	N7	Ce1	N3	-70.5(2)	C32	N7	Ce1	N4	43.7(5)
C34	N7	Ce1	N4	-126.2(2)	C32	N7	Ce1	C6	-172.0(4)
C34	N7	Ce1	C6	18.1(3)	C32	N7	Ce1	C1	-150.5(4)
C34	N7	Ce1	C1	39.6(4)	C32	N7	Ce1	C34	169.9(6)
C32	N7	Ce1	C23	-50.7(5)	C34	N7	Ce1	C23	139.4(2)
C21	N1	Ce1	N2	-134.3(4)	C1	N1	Ce1	N2	51.5(2)
C21	N1	Ce1	N8	46.7(4)	C1	N1	Ce1	N8	-127.5(2)
C21	N1	Ce1	N6	164.6(4)	C1	N1	Ce1	N6	-9.6(3)
C21	N1	Ce1	N5	103.6(4)	C1	N1	Ce1	N5	-70.6(2)
C21	N1	Ce1	N7	-142(3)	C1	N1	Ce1	N7	43(3)
C21	N1	Ce1	N3	-73.9(4)	C1	N1	Ce1	N3	111.9(2)
C21	N1	Ce1	N4	-17.2(4)	C1	N1	Ce1	N4	168.6(3)
C21	N1	Ce1	C6	-162.5(5)	C1	N1	Ce1	C6	23.3(2)
C21	N1	Ce1	C1	174.2(6)	C21	N1	Ce1	C34	-143.9(4)
C1	N1	Ce1	C34	41.9(4)	C21	N1	Ce1	C23	76.2(4)
C1	N1	Ce1	C23	-98.0(3)	C10	N3	Ce1	N2	-19.8(4)
C12	N3	Ce1	N2	168.1(3)	C10	N3	Ce1	N8	163.1(4)
C12	N3	Ce1	N8	-9.0(3)	C10	N3	Ce1	N6	46.3(4)
C12	N3	Ce1	N6	-125.7(2)	C10	N3	Ce1	N5	164(2)
C12	N3	Ce1	N5	-8(2)	C10	N3	Ce1	N7	101.4(4)
C12	N3	Ce1	N7	-70.7(3)	C10	N3	Ce1	N1	-76.9(4)
C12	N3	Ce1	N1	111.1(3)	C10	N3	Ce1	N4	-136.7(4)
C12	N3	Ce1	N4	51.2(2)	C10	N3	Ce1	C6	-25.2(4)
C12	N3	Ce1	C6	162.8(2)	C10	N3	Ce1	C1	-49.2(4)
C12	N3	Ce1	C1	138.7(2)	C10	N3	Ce1	C34	73.0(4)
C12	N3	Ce1	C34	-99.0(3)	C10	N3	Ce1	C23	-149.7(4)
C12	N3	Ce1	C23	38.3(4)	C18	N4	Ce1	N2	78.7(4)
C17	N4	Ce1	N2	-111.6(2)	C18	N4	Ce1	N8	-97.6(4)
C17	N4	Ce1	N8	72.0(2)	C18	N4	Ce1	N6	-180(34)
C17	N4	Ce1	N6	-10.1(15)	C18	N4	Ce1	N5	-43.8(4)
C17	N4	Ce1	N5	125.8(2)	C18	N4	Ce1	N7	-159.5(4)
C17	N4	Ce1	N7	10.1(3)	C18	N4	Ce1	N1	22.1(4)
C17	N4	Ce1	N1	-168.3(3)	C18	N4	Ce1	N3	138.9(4)
C17	N4	Ce1	N3	-51.5(2)	C18	N4	Ce1	C6	50.8(4)
C17	N4	Ce1	C6	-139.6(2)	C18	N4	Ce1	C1	27.4(4)
C17	N4	Ce1	C1	-163.0(2)	C18	N4	Ce1	C34	173.1(4)
C17	N4	Ce1	C34	-17.3(3)	C18	N4	Ce1	C23	-47.4(4)
C17	N4	Ce1	C23	122.2(2)	C5	C6	Ce1	N2	105.7(5)

C1	C6	Ce1	N2	-136.1(4)	C5	C6	Ce1	N8	-78.6(5)
N2	C6	Ce1	N8	175.7(2)	C1	C6	Ce1	N8	39.7(4)
C5	C6	Ce1	N6	6.5(4)	N2	C6	Ce1	N6	-99.2(3)
C1	C6	Ce1	N6	124.7(3)	C5	C6	Ce1	N5	-63.9(4)
N2	C6	Ce1	N5	-169.6(3)	C1	C6	Ce1	N5	54.4(2)
C5	C6	Ce1	N7	39.2(5)	N2	C6	Ce1	N7	-66.5(3)
C1	C6	Ce1	N7	157.4(2)	C5	C6	Ce1	N1	-141.6(5)
N2	C6	Ce1	N1	112.7(3)	C1	C6	Ce1	N1	-23.4(2)
C5	C6	Ce1	N3	116.6(4)	N2	C6	Ce1	N3	10.9(3)
C1	C6	Ce1	N3	-125.2(2)	C5	C6	Ce1	N4	-177.1(4)
N2	C6	Ce1	N4	77.2(3)	C1	C6	Ce1	N4	-58.8(3)
C5	C6	Ce1	C1	-118.2(5)	N2	C6	Ce1	C1	136.1(4)
C5	C6	Ce1	C34	47.6(4)	N2	C6	Ce1	C34	-58.1(3)
C1	C6	Ce1	C34	165.9(2)	C5	C6	Ce1	C23	-87.5(4)
N2	C6	Ce1	C23	166.8(2)	C1	C6	Ce1	C23	30.7(3)
C2	C1	Ce1	N2	141.4(5)	N1	C1	Ce1	N2	-113.5(3)
C6	C1	Ce1	N2	24.6(2)	C2	C1	Ce1	N8	-39.5(5)
N1	C1	Ce1	N8	65.5(3)	C6	C1	Ce1	N8	-156.4(2)
C2	C1	Ce1	N6	66.1(5)	N1	C1	Ce1	N6	171.2(3)
C6	C1	Ce1	N6	-50.7(3)	C2	C1	Ce1	N5	-3.6(4)
N1	C1	Ce1	N5	101.4(3)	C6	C1	Ce1	N5	-120.4(3)
C2	C1	Ce1	N7	77.5(5)	N1	C1	Ce1	N7	-177.4(2)
C6	C1	Ce1	N7	-39.3(4)	C2	C1	Ce1	N1	-105.0(5)
C6	C1	Ce1	N1	138.1(4)	C2	C1	Ce1	N3	178.0(4)
N1	C1	Ce1	N3	-76.9(3)	C6	C1	Ce1	N3	61.2(3)
C2	C1	Ce1	N4	-115.8(5)	N1	C1	Ce1	N4	-10.8(3)
C6	C1	Ce1	N4	127.3(2)	C2	C1	Ce1	C6	116.8(6)
N1	C1	Ce1	C6	-138.1(4)	C2	C1	Ce1	C34	99.4(5)
N1	C1	Ce1	C34	-155.6(2)	C6	C1	Ce1	C34	-17.5(3)
C2	C1	Ce1	C23	-31.8(4)	N1	C1	Ce1	C23	73.3(2)
C6	C1	Ce1	C23	-148.6(3)	C35	C34	Ce1	N2	65.1(5)
C39	C34	Ce1	N2	-52.1(3)	N7	C34	Ce1	N2	171.2(3)
C35	C34	Ce1	N8	-116.1(5)	C39	C34	Ce1	N8	126.6(3)
N7	C34	Ce1	N8	-10.1(3)	C35	C34	Ce1	N6	141.6(6)
C39	C34	Ce1	N6	24.4(2)	N7	C34	Ce1	N6	-112.4(3)
C35	C34	Ce1	N5	177.4(5)	C39	C34	Ce1	N5	60.2(3)
N7	C34	Ce1	N5	-76.6(3)	C35	C34	Ce1	N7	-106.1(6)
C39	C34	Ce1	N7	136.7(4)	C35	C34	Ce1	N1	73.8(6)
C39	C34	Ce1	N1	-43.4(4)	N7	C34	Ce1	N1	179.9(2)
C35	C34	Ce1	N3	-5.7(5)	C39	C34	Ce1	N3	-122.9(3)
N7	C34	Ce1	N3	100.3(3)	C35	C34	Ce1	N4	-39.1(6)
C39	C34	Ce1	N4	-156.3(2)	N7	C34	Ce1	N4	66.9(3)
C35	C34	Ce1	C6	88.5(5)	C39	C34	Ce1	C6	-28.7(3)
N7	C34	Ce1	C6	-165.5(2)	C35	C34	Ce1	C1	96.5(5)
C39	C34	Ce1	C1	-20.7(3)	N7	C34	Ce1	C1	-157.4(2)
C35	C34	Ce1	C23	-159.5(5)	C39	C34	Ce1	C23	83.3(3)
N7	C34	Ce1	C23	-53.5(3)	C24	C23	Ce1	N2	-36.1(5)
N5	C23	Ce1	N2	67.9(3)	C28	C23	Ce1	N2	-154.0(2)
C24	C23	Ce1	N8	142.2(5)	N5	C23	Ce1	N8	-113.7(3)
C28	C23	Ce1	N8	24.4(2)	C24	C23	Ce1	N6	-114.5(5)
N5	C23	Ce1	N6	-10.4(3)	C28	C23	Ce1	N6	127.7(3)
C24	C23	Ce1	N5	-104.0(6)	C28	C23	Ce1	N5	138.1(4)
C24	C23	Ce1	N7	179.8(5)	N5	C23	Ce1	N7	-76.1(3)
C28	C23	Ce1	N7	62.0(3)	C24	C23	Ce1	N1	-1.4(5)
N5	C23	Ce1	N1	102.7(3)	C28	C23	Ce1	N1	-119.2(3)
C24	C23	Ce1	N3	80.5(6)	N5	C23	Ce1	N3	-175.4(2)
C28	C23	Ce1	N3	-37.3(4)	C24	C23	Ce1	N4	68.8(5)

N5	C23	Ce1	N4	172.8(3)	C28	C23	Ce1	N4	-49.1(3)
C24	C23	Ce1	C6	-43.7(5)	N5	C23	Ce1	C6	60.3(3)
C28	C23	Ce1	C6	-161.5(3)	C24	C23	Ce1	C1	-29.6(5)
N5	C23	Ce1	C1	74.4(3)	C28	C23	Ce1	C1	-147.5(3)
C24	C23	Ce1	C34	-156.3(5)	N5	C23	Ce1	C34	-52.3(3)
C28	C23	Ce1	C34	85.9(3)					

Symmetry Operators:

x, y, z -x, -y, -z

Table 5. Least-squares planes (x,y,z in crystal coordinates) and deviations from them
(* indicates atom used to define plane)

- 2.1161 (0.0143) x + 0.0471 (0.0148) y + 15.5615 (0.0069) z = 1.2282 (0.0068)

* -0.0072 (0.0017) N1
* 0.0072 (0.0017) N2
* -0.0072 (0.0017) N3
* 0.0072 (0.0017) N4
1.5067 (0.0018) Ce1

Rms deviation of fitted atoms = 0.0072

- 2.2540 (0.0150) x + 0.2707 (0.0160) y + 15.4513 (0.0080) z = 4.2563 (0.0067)

Angle to previous plane (with approximate esd) = 1.17 (0.10)

* 0.0122 (0.0019) N5
* -0.0122 (0.0019) N6
* 0.0121 (0.0019) N7
* -0.0121 (0.0018) N8
-1.5093 (0.0019) Ce1

Rms deviation of fitted atoms = 0.0122

2. Ce(tmtaa)(tmtaaH)

2.1 Diagram

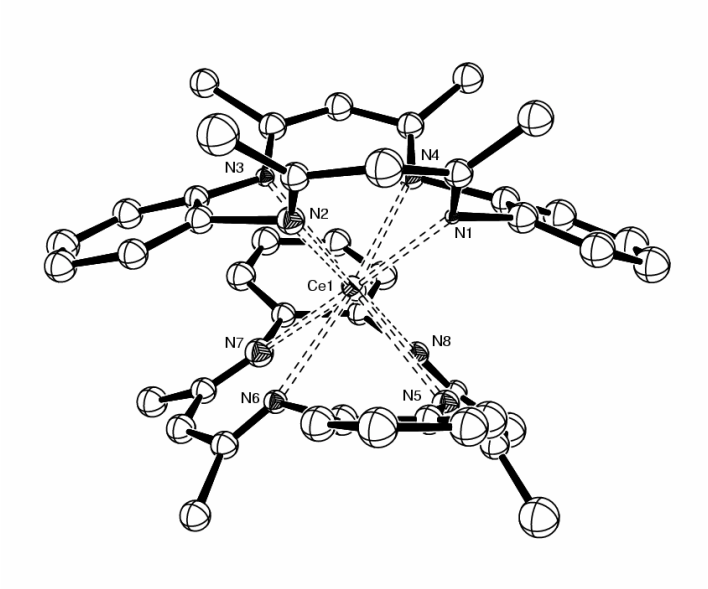

Figure S2. ORTEP diagram of Ce(tmtaa)(tmtaaH). Non-hydrogen atoms are refined isotropically, except cerium (Ce1).

2.2 Experimental Details

Data Collection

A fragment of a red block-like crystal of $C_{44}H_{45}CeN_8$ having approximate dimensions of 0.19 x 0.14 x 0.02 mm was mounted on a glass fiber using Paratone N hydrocarbon oil. All measurements were made on a Bruker SMART CCD[10] CCD area detector with graphite monochromated MoKα radiation.

Cell constants and an orientation matrix, obtained from a least-squares refinement using the measured positions of 2465 centered reflections with $I > 10\sigma(I)$ in the range $2.5 < \theta < 24.2^o$ corresponded to a primitive triclinic cell with dimensions:

a = 8.919(1) Å	α = 117.853(1) ^{o}
b = 11.424(1) Å	β = 102.987(1) ^{o}
c = 11.472(1) Å	γ = 101.300(1) ^{o}
V = 943.63(16)Å^3	

For Z = 1 and F.W. = 826.00, the calculated density is 1.454 g/cm^3.

Analysis of the systematic absences allowed the space group to be uniquely determined to be:

P1

The data were collected at a temperature of 119(2) K. Frames corresponding to an arbitrary

hemisphere of data were collected using ω scans of 0.3^o counted for a total of 15 seconds per frame.

Data Reduction

Data were integrated by the program SAINT[11] to a maximum θ value of 24.73^o. The data were corrected for Lorentz and polarization effects. Data were analyzed for agreement and possible absorption using XPREP[12]. An empirical absorption correction based on comparison of redundant and equivalent reflections was applied using SADABS[13]. (Tmax = 0.9754, Tmin = 0.7972). Of the 4812 reflections that were collected, 3489 were unique (R_{int} = 0.0385); equivalent reflections were merged. No decay correction was applied.

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. Cerium atoms were refined anisotropically, while the rest were refined isotropically. Methyl and methane hydrogen atoms were included in calculated positions using a riding model, but not refined. The amido hydrogen was neither located nor included in the refinement. The crystal was a racemic twin (twin percentage = 41.1 %), and the twin law (-1 0 0, 0 -1 0, 0 0 -1) was incorporated in the refinement. The final cycle of full-matrix least-squares refinement[3] was based on 3489 reflections (all data) and 227 variable parameters and converged (largest parameter shift was 0.000 times its esd) with conventional unweighted and weighted agreement factors of:

$$R_1 = \Sigma||Fo| - |Fc|| / \Sigma|Fo| = 0.0675 \text{ for 3391 data with } I > 2\sigma(I)$$

$$wR_2 = [(\Sigma w (|Fo|^2 - |Fc|^2)^2 / \Sigma w |Fo|^2)]^{1/2} = 0.1826$$

The standard deviation of an observation of unit weight[4] was 1.100. The weighting scheme was based on counting statistics and included a factor (q = 0.1394) to downweight the intense reflections. The maximum and minimum peaks on the final difference Fourier map corresponded to 1.841 and -1.804 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5]. Anomalous dispersion effects were included in Fcalc[6]; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbel[8]. All calculations were performed using the SHELXTL[9] crystallographic software package of Bruker Analytical X-ray Systems Inc.

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	C44 H45 Ce N8
Formula Weight	826.00
Crystal Color, Habit	red, block
Crystal Dimensions	0.19 x 0.14 x 0.02 mm
Crystal System	triclinic
Lattice Type	primitive
Lattice Parameters	a = 8.919(1) Å b = 11.424(1) Å c = 11.472(1) Å α= 117.853(1) ^{o} β= 102.987(1) ^{o} γ = 101.300(1)^{o} V = 943.63(16) Å^{3}
Space Group	P1
Z value	1
D_{calc}	1.454 g/cm^{3}
F_{000}	423
μ(MoK)	1.25 cm^{-1}

B. Intensity Measurements

Diffractometer	Bruker SMART CCD
Radiation	MoK(λ = 0.71073 Å) graphite monochromated
Detector Position	60.00 mm
Exposure Time	15 seconds per frame.
Scan Type	ω (0.3 degrees per frame)
θ_{max}	24.73^{o}
No. of Reflections Measured	Total: 4812 Unique: 3489 (R_{int} = 0.0385)
Corrections	Lorentz-polarization Absorption (Tmax = 0.9754, Tmin = 0.7972)

C. Structure Solution and Refinement

Structure Solution	direct
Refinement	Full-matrix least-squares
Function Minimized	$\Sigma w(\|F_o\|^2 - \|F_c\|^2)^2$
Least Squares Weighting scheme	$w = 1/[\sigma^2(F_o^2) + (qP)^2 + 0.000P]$
	where $P = [F_o^2 + 2F_c^2]/3$
q-factor	0.1394
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>2.00σ(I))	3391
No. Variables	227
Reflection/Parameter Ratio	15
Residuals: R; wR_2; Rall	0.0675; 0.1826; 0.0694
Goodness of Fit Indicator	1.100
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	1.841 $e^-/Å^3$
Minimum peak in Final Diff. Map	-1.804 $e^-/Å^3$

2.3 Tables

Table 1. Atomic coordinates and U_{iso}/U_{eq} and occupancy

atom	x	y	z	U_{eq}	Occupancy
C1	0.2579(19)	-0.0243(15)	0.6698(16)	0.022(3)	1
C2	0.287(2)	-0.1658(18)	0.6035(19)	0.032(4)	1
C3	0.401(2)	0.0958(19)	0.781(2)	0.038(4)	1
C4	0.4367(19)	0.2434(15)	0.8161(16)	0.022(3)	1
C5	0.620(3)	0.347(3)	0.895(3)	0.044(8)	1
C6	0.3555(19)	0.4282(16)	0.8111(16)	0.022(3)	1
C7	0.4361(19)	0.5464(16)	0.9427(17)	0.025(3)	1
C8	0.450(2)	0.6809(17)	0.9704(18)	0.029(4)	1
C9	0.370(2)	0.6905(18)	0.8585(18)	0.032(4)	1
C10	0.285(2)	0.5727(17)	0.7237(18)	0.028(4)	1
C11	0.2763(18)	0.4371(15)	0.6945(16)	0.020(3)	1
C12	0.158(2)	0.2806(16)	0.4392(17)	0.025(3)	1
C13	0.253(2)	0.3922(16)	0.4170(17)	0.028(4)	1
C14	0.065(2)	0.1503(16)	0.3193(17)	0.027(3)	1
C15	-0.0125(19)	0.0246(16)	0.3044(16)	0.024(3)	1
C16	-0.068(2)	-0.106(2)	0.1539(19)	0.032(4)	1
C17	-0.105(2)	-0.1141(16)	0.4012(17)	0.026(3)	1
C18	-0.255(2)	-0.2139(18)	0.307(2)	0.036(4)	1
C19	-0.328(2)	-0.329(2)	0.314(2)	0.041(4)	1
C20	-0.248(2)	-0.341(2)	0.428(2)	0.039(4)	1
C21	-0.094(2)	-0.2412(19)	0.5243(19)	0.035(4)	1
C22	-0.021(2)	-0.1280(17)	0.5146(17)	0.027(4)	1
C23	-0.039(2)	0.1129(16)	0.8842(16)	0.024(3)	1
C24	-0.005(2)	0.0167(19)	0.9107(19)	0.035(4)	1
C25	0.128(2)	0.0542(19)	1.0242(19)	0.036(4)	1
C26	0.239(2)	0.1956(19)	1.109(2)	0.038(4)	1
C27	0.208(2)	0.2917(18)	1.0791(18)	0.031(4)	1
C28	0.0719(19)	0.2571(16)	0.9690(17)	0.025(3)	1
C29	0.0498(19)	0.4754(16)	0.9986(16)	0.022(3)	1
C30	0.089(2)	0.5403(18)	1.1566(17)	0.026(4)	1
C31	0.017(2)	0.5578(17)	0.9428(17)	0.026(3)	1
C32	-0.066(2)	0.5158(15)	0.8036(16)	0.022(3)	1
C33	-0.117(2)	0.6301(18)	0.7856(18)	0.032(4)	1
C34	-0.1815(19)	0.3449(15)	0.5547(16)	0.021(3)	1
C35	-0.152(2)	0.4163(18)	0.4889(18)	0.031(4)	1
C36	-0.229(2)	0.3590(18)	0.3437(18)	0.031(4)	1
C37	-0.342(2)	0.2173(17)	0.2584(18)	0.030(4)	1
C38	-0.365(2)	0.1388(18)	0.3207(18)	0.031(4)	1
C39	-0.2917(18)	0.2004(15)	0.4654(15)	0.020(3)	1
C40	-0.4376(18)	0.0505(15)	0.5231(15)	0.020(3)	1
C41	-0.603(3)	0.059(2)	0.451(2)	0.022(6)	1
C42	-0.449(2)	-0.031(2)	0.592(2)	0.041(4)	1
C43	-0.318(2)	0.0053(16)	0.7200(17)	0.025(3)	1
C44	-0.387(3)	-0.042(2)	0.806(2)	0.049(5)	1
N1	0.124(2)	-0.0031(17)	0.6322(18)	0.006(4)	1
N2	0.320(2)	0.2702(19)	0.794(2)	0.021(5)	1
N3	0.181(2)	0.311(2)	0.572(2)	0.011(5)	1
N4	-0.022(2)	0.0450(18)	0.4088(18)	0.021(4)	1
N5	-0.161(3)	0.077(2)	0.761(3)	0.022(6)	1
N6	0.0486(19)	0.3705(16)	0.9163(16)	0.016(4)	1

N7	-0.093(3)	0.399(2)	0.706(2)	0.027(6)	1
N8	-0.304(2)	0.1079(19)	0.547(2)	0.020(4)	1
Ce1	0.0075(1)	0.1942(1)	0.6641(1)	0.019(1)	1

U_{eq} is defined as one third of the orthogonalized U_{ij} tensor

Table 2. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ce1	0.023(1)	0.017(1)	0.016(1)	0.008(1)	0.007(1)	0.008(1)

The general temperature factor expression:

$\exp(-2\Pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}\,hl + 2b^*c^*U_{23}kl))$

Table 3. Selected Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
N1	Ce1	2.571(17)	N2	Ce1	2.594(19)
N3	Ce1	2.56(2)	N4	Ce1	2.524(17)
N5	Ce1	2.57(2)	N6	Ce1	2.520(16)
N7	Ce1	2.54(2)	N8	Ce1	2.555(19)

3. References

(1) XS: Program for the Solution of X-ray Crystal Structures, Part of the SHELXTL Crystal Structure Determination Package, Bruker Analytical X-ray Systems Inc.: Madison, WI, (1995-99)

(2) XL: Program for the Refinement of X-ray Crystal Structures, Part of the SHELXTL Crystal Structure Determination Package, Bruker Analytical X-ray Systems Inc.: Madison, WI, (1995-99)

(3) Least-Squares:

Function minimized: $\Sigma w\ (|Fo|^2 - |Fc|^2)^2$

(4) Standard deviation of an observation of unit weight:

$[\Sigma w(|Fo|^2 - |Fc|^2)^2/(N_o - N_v)]^{1/2}$
where: N_o = number of observations
N_v = number of variables

(5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(8) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(9) XP: Molecular Graphics program. Part of the SHELXTL Structure Determination Package. Bruker Analytical X-ray Systems Inc.: Madison, WI, (1995-99)

(10)SMART: Area-Detector Software Package, Bruker Analytical X-ray Systems, Inc.: Madison, WI, (1995-99)

(11)SAINT: SAX Area-Dectector Integration Program, V5.04; Siemens Industrial Automation, Inc.: Madison, WI, (1995)

(12)XPREP:(v 5.03) Part of the SHELXTL Crystal Structure Determination Package, Siemens Industrial Automation, Inc.: Madison, WI, (1995)

(13)SADABS: Siemens Area Detector ABSorption correction program, George Sheldrick, (1996). Advance copy, private communication.