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J4584-1

Collection of X-ray Diffraction Data. A colorless crystal of approximate dimensions 0.20 × 0.20 × 0.21 mm was mounted on the Siemens P4 diffractometer. Determination of Laue symmetry, crystal class, unit cell parameters and the crystal's orientation matrix were carried out according to standard procedures[1]. Low temperature (158 K) intensity data were collected via a 2θ-omega scan technique with MoKα radiation.

All 1807 data were corrected for absorption[2] and for Lorentz and polarization effects and placed on an approximately absolute scale. The crystal class is triclinic and the space group is the centrosymmetric $P\bar{1}$.

Solution and Refinement of the Crystal Structure. All crystallographic calculations were carried out using the UCI modified version of the UCLA Crystallographic Computing Package[3] and the SHELXTL[4] program. The analytical scattering factors[5] for neutral atoms were used throughout the analysis. The structure was solved by direct methods and refined by full-matrix least-squares techniques (SHELXTL). The structure is polymeric. Each Ce_2Cl_2 unit is located about an inversion center. Refinement of the model led to convergence with wR2 = 0.0507 and GOF = 1.09 for 100 parameters refined against all 1650 unique data (R1 = 0.021 for those 1584 data with $F_o > 4.0\sigma(F_o)$).

J4584-2

References.

1. XSCAnS Version 2.10, Siemens Analytical X-Ray Instruments, Inc.; Madison, WI 1990-1995.

2. XPREP program (reference 4).

3. UCLA Crystallographic Computing Package, University of California Los Angeles 1981, C. Strouse; personal communication.

4. Sheldrick, G. M., Siemens Analytical X-Ray Instruments, Inc.; Madison, WI 1990-1995.

5. International Tables for X-Ray Crystallography 1992, Vol. C., Dordrecht: Kluwer Academic Publishers.

The thermal elliposid plot is shown at the 50% probability level.

J4584-3

Table 1. Crystal data and structure refinement for 1.

Formula	$C_4H_{10}CeCl_3O_2$
fw	336.59
a	6.691(2) Å
b	7.433(2) Å
c	10.092(2) Å
α	84.46(2)°
β	76.72(2)°
γ	74.76(3)°
V	471.0(2) Å^3
Z	2
Space group	$P\bar{1}$
T	158 K
λ	0.71073 Å
ρ (calcd)	2.374 g/cm^3
μ	5.627mm^{-1}
R1,wR2[I>2σ(I)]	0.0206,0.0499
R1,wR2(all data)	0.0224,0.0507

$R1 = \sum||F_o|-|F_c||/\sum|F_o|$

$wR2 = [\sum[w(F_o^2-F_c^2)^2]/\sum[w(F_o^2)^2]]^{1/2}$

J4584-4

Crystal data.

Empirical formula	$C_4H_{10}CeCl_3O_2$
Formula weight	336.59
Crystal Description	Prism
Crystal Color	Colorless
Crystal size	0.21 x 0.20 x 0.20 mm
Crystal system	Triclinic
Space group	$P\bar{1}$
Unit cell dimensions:	a = 6.691(2) Å
	b = 7.433(2) Å
	c = 10.092(2) Å
	α = 84.46(2)°
	β = 76.72(2)°
	γ = 74.76(3)°
Volume	471.0(2) $Å^3$
Z	2
Density (calculated)	2.374 Mg/m^3
Absorption coefficient	5.627 mm^{-1}
F(000)	318

JU584-5

Data Collection and Reduction.

Diffractometer type	Siemens P4
Radiation source	normal-focus sealed tube
Monochromator type	graphite
Radiation	MoKα
Wavelength	0.71073 Å
Temperature	158 K
Cell measurement reflections	35 ($4.69^{\circ} < \theta < 15.68^{\circ}$)
Intensity measurement method	$2\theta/\omega$ scans
θ range for data collection	2.07 to 25.00°
Limiting indices	$-2 \le h \le 7$, $-8 \le k \le 8$, $-12 \le l \le 11$
Scan width (in ω)	1.2° plus α_1,α_2 separation
Scan speed (in ω)	3.0°/min
Reflections collected	1807
Independent reflections	1650 (R_{int} = 0.0248)
Observed reflections	1584 (I >2sigma(I))
Number of standards	2
Interval between standards	98
Decay of standards	no decay
Absorption correction	Semi-empirical from psi-scans
Max. and min. transmission	0.4904 and 0.3882

Unit cell refinement and data collection were carried out by use of the Siemens XSCAnS system, Version 2.10b. Data were processed with a local version of CARESS (R.W. Broach et al.), which employs a modified version of the Lehman-Larsen algorithm to obtain integrated intensities and standard deviations from the measured 96-step peak profiles.

Structure Solution and Refinement.

JL584-6

Structure solution method	direct methods
Refinement method	Full-matrix least-squares on F^2
Scattering Factor Source	International Tables Vol C Tables 4.2.6.8 and 6.1.1.4
Final refinement: Data	1650
Restraints	0
Parameters	100
Final R indices [$I>2\sigma(I)$]	R1 = 0.0206, wR2 = 0.0499
R indices (all data)	R1 = 0.0224, wR2 = 0.0507
Goodness-of-fit on F^2	1.085
Extinction coefficient	0.0082(8)
Largest diff. peak and hole	0.859 and -1.306 eÅ^{-3}
Maximum Δ/σ in final cycle	< 0.001
Mean Δ/σ in final cycle	< 0.001

Final weighting scheme:

calc $w=1/[\sigma^2(Fo^2)+(0.0260P)^2+1.4804P]$

where $P=(Fo^2+2Fc^2)/3$

R-factor definitions:

$R1 = \sum||F_o|-|F_c||/\sum|F_o|$

$wR2 = [\sum[w(F_o^2-F_c^2)^2]/\sum[w(F_o^2)^2]]^{1/2}$

Structure solution, refinement, and generation of figures and tables were carried out by use of Version 5.03 of the Siemens SHELXTL program package.

JU584-7

Table 2. Crystal data and structure refinement for 1.

Empirical formula	$C_4H_{10}CeCl_3O_2$
Formula weight	336.59
Temperature	158 K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group	$P\bar{1}$
Unit cell dimensions	a = 6.691(2) Å α = 84.46(2)° b = 7.433(2) Å β = 76.72(2)° c = 10.092(2) Å γ = 74.76(3)°
Volume, Z	471.0(2) $Å^3$, 2
Density (calculated)	2.374 Mg/m^3
Absorption coefficient	5.627 mm^{-1}
F(000)	318
Crystal size	0.21 x 0.20 x 0.20 mm
θ range for data collection	2.07 to 25.00°
Limiting indices	$-2 \le h \le 7$, $-8 \le k \le 8$, $-12 \le l \le 11$
Reflections collected	1807
Independent reflections	1650 (R_{int} = 0.0248)
Absorption correction	Semi-empirical from psi-scans
Max. and min. transmission	0.4904 and 0.3882
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1650 / 0 / 100
Goodness-of-fit on F^2	1.085
Final R indices [$I>2\sigma(I)$]	R1 = 0.0206, wR2 = 0.0499
R indices (all data)	R1 = 0.0224, wR2 = 0.0507
Largest diff. peak and hole	0.859 and -1.306 $eÅ^{-3}$

$R1 = \sum||F_o|-|F_c||/\sum|F_o|$

$wR2 = [\sum[w(F_o^2-F_c^2)^2]/\sum[w(F_o^2)^2]]^{1/2}$

J4584-8

Table 3. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [$Å^2$ x 10^3] for 1. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
Ce(1)	-2839(1)	7051(1)	9398(1)	9(1)
Cl(1)	-3989(2)	9886(1)	11452(1)	13(1)
Cl(2)	-3272(2)	3470(1)	8879(1)	15(1)
Cl(3)	-1420(2)	4934(1)	11631(1)	14(1)
O(1)	-2431(5)	7019(4)	6847(3)	16(1)
O(2)	-622(5)	9322(5)	8591(3)	17(1)
C(1)	-1227(8)	8103(7)	5847(4)	23(1)
C(2)	-1586(8)	7747(8)	4479(5)	28(1)
C(3)	-3721(11)	7283(12)	4854(6)	54(2)
C(4)	-3898(9)	6460(9)	6203(5)	37(1)

J4584-9

Table 4. Bond lengths [Å] and angles [°] for 1.

Ce(1)-O(2)	2.492(3)	Ce(1)-O(1)	2.528(3)
Ce(1)-Cl(3)	2.8232(12)	Ce(1)-Cl(3)#1	2.8465(14)
Ce(1)-Cl(2)	2.8606(12)	Ce(1)-Cl(2)#2	2.8747(14)
Ce(1)-Cl(1)#3	2.8883(14)	Ce(1)-Cl(1)	2.9304(13)
Cl(1)-Ce(1)#3	2.8883(14)	Cl(2)-Ce(1)#2	2.8747(14)
Cl(3)-Ce(1)#1	2.8465(14)		

O(1)-C(4)	1.455(6)	O(1)-C(1)	1.458(5)
C(1)-C(2)	1.514(6)	C(2)-C(3)	1.513(8)
C(3)-C(4)	1.429(8)		

O(2)-Ce(1)-O(1)	78.76(10)	O(2)-Ce(1)-Cl(3)	108.17(9)
O(1)-Ce(1)-Cl(3)	142.47(7)	O(2)-Ce(1)-Cl(3)#1	70.79(9)
O(1)-Ce(1)-Cl(3)#1	74.03(7)	Cl(3)-Ce(1)-Cl(3)#1	73.90(4)
O(2)-Ce(1)-Cl(2)	142.59(8)	O(1)-Ce(1)-Cl(2)	74.42(7)
Cl(3)-Ce(1)-Cl(2)	79.83(4)	Cl(3)#1-Ce(1)-Cl(2)	77.03(4)
O(2)-Ce(1)-Cl(2)#2	144.64(8)	O(1)-Ce(1)-Cl(2)#2	118.35(7)
Cl(3)-Ce(1)-Cl(2)#2	77.75(4)	Cl(3)#1-Ce(1)-Cl(2)#2	141.23(3)
Cl(2)-Ce(1)-Cl(2)#2	72.35(4)	O(2)-Ce(1)-Cl(1)#3	81.00(9)
O(1)-Ce(1)-Cl(1)#3	70.91(7)	Cl(3)-Ce(1)-Cl(1)#3	145.81(3)
Cl(3)#1-Ce(1)-Cl(1)#3	138.42(3)	Cl(2)-Ce(1)-Cl(1)#3	113.30(4)
Cl(2)#2-Ce(1)-Cl(1)#3	76.87(4)	O(2)-Ce(1)-Cl(1)	72.22(8)
O(1)-Ce(1)-Cl(1)	136.63(7)	Cl(3)-Ce(1)-Cl(1)	78.38(3)
Cl(3)#1-Ce(1)-Cl(1)	122.98(4)	Cl(2)-Ce(1)-Cl(1)	143.92(3)
Cl(2)#2-Ce(1)-Cl(1)	75.15(4)	Cl(1)#3-Ce(1)-Cl(1)	73.24(4)
Ce(1)#3-Cl(1)-Ce(1)	106.76(4)	Ce(1)-Cl(2)-Ce(1)#2	107.65(4)
Ce(1)-Cl(3)-Ce(1)#1	106.10(4)		

C(4)-O(1)-C(1)	108.4(3)	C(4)-O(1)-Ce(1)	123.6(3)
C(1)-O(1)-Ce(1)	125.0(2)	O(1)-C(1)-C(2)	105.7(4)
C(3)-C(2)-C(1)	102.6(4)	C(4)-C(3)-C(2)	106.4(5)
C(3)-C(4)-O(1)	108.2(4)		

Symmetry transformations used to generate equivalent atoms:

#1 -x,-y+1,-z+2 #2 -x-1,-y+1,-z+2 #3 -x-1,-y+2,-z+2

Table 5. Anisotropic displacement parameters [$Å^2$ x 10^3] for 1. J4584-10

The anisotropic displacement factor exponent takes the form:
$-2\pi^2$ [$(ha^*)^2U_{11}$ + ... + $2hka^*b^*U_{12}$]

	U11	U22	U33	U23	U13	U12
Ce(1)	11(1)	9(1)	5(1)	2(1)	-1(1)	-3(1)
Cl(1)	17(1)	13(1)	10(1)	2(1)	-4(1)	-2(1)
Cl(2)	17(1)	12(1)	15(1)	-1(1)	3(1)	-6(1)
Cl(3)	14(1)	17(1)	7(1)	3(1)	0(1)	-1(1)
O(1)	18(2)	24(2)	8(1)	2(1)	-1(1)	-9(1)
O(2)	14(2)	11(2)	25(2)	-1(1)	1(1)	-5(1)
C(1)	31(3)	34(3)	10(2)	9(2)	-3(2)	-20(2)
C(2)	37(3)	40(3)	9(2)	6(2)	-4(2)	-16(2)
C(3)	64(4)	101(6)	18(3)	14(3)	-15(3)	-59(4)
C(4)	35(3)	73(4)	17(3)	2(3)	-8(2)	-36(3)

Table 6. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for 1.

J4584-11

	x	y	z	U(eq)
H(1A)	299(8)	7692(7)	5866(4)	28
H(1B)	-1738(8)	9447(7)	6038(4)	28
H(2A)	-1626(8)	8870(8)	3862(5)	34
H(2B)	-467(8)	6691(8)	4038(5)	34
H(3A)	-4875(11)	8428(12)	4828(6)	64
H(3B)	-3809(11)	6397(12)	4210(6)	64
H(4A)	-3548(9)	5084(9)	6167(5)	45
H(4B)	-5367(9)	6890(9)	6733(5)	45
H(1)	-1163(79)	10362(78)	8692(50)	12(13)
H(2)	571(109)	9265(88)	8559(66)	41(19)