$S = 1.92$
1592 reflections
204 parameters
H atoms not located
$w = 1/[\sigma^2(F_o) + 0.02081F_o + 0.0049F_o^2]$

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å²)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

| | $x$ | $y$ | $z$ | $U_{eq}$ |
|---|---|---|---|---|
| S(1) | 0.0444 (1) | 0.3203 (1) | 0.8346 (2) | 0.040 |
| Na(1) | 0.1888 (2) | 0.4939 (1) | 0.8123 (4) | 0.039 |
| O(1) | 0.3208 (3) | 0.4459 (1) | 1.0278 (6) | 0.037 |
| O(2) | 0.4625 (3) | 0.4015 (1) | 0.8265 (9) | 0.051 |
| O(3) | −0.0239 (3) | 0.4535 (1) | 0.9042 (6) | 0.035 |
| O(4) | −0.1491 (4) | 0.4125 (1) | 1.4301 (7) | 0.044 |
| O(5) | 0.2486 (5) | 0.2999 (1) | 0.4651 (8) | 0.058 |
| O(6) | 0.3569 (3) | 0.5397 (1) | 0.6595 (7) | 0.040 |
| O(7) | 0.1504 (6) | 0.4599 (2) | 0.4524 (9) | 0.086 |
| O(8) | 1/2 | 1/2 | 0.2681 (9) | 0.047 |
| N(1) | 0.0889 (4) | 0.3919 (1) | 1.0352 (7) | 0.030 |
| C(1) | 0.2221 (4) | 0.3828 (1) | 0.9117 (9) | 0.032 |
| C(2) | 0.2119 (5) | 0.3464 (1) | 0.7922 (9) | 0.037 |
| C(3) | −0.0071 (4) | 0.3566 (1) | 1.0747 (8) | 0.030 |
| C(4) | −0.1405 (4) | 0.3854 (1) | 1.0266 (8) | 0.030 |
| C(5) | −0.0240 (5) | 0.4179 (1) | 0.9725 (7) | 0.029 |
| C(6) | 0.3459 (5) | 0.4116 (1) | 0.9209 (9) | 0.032 |
| C(7) | 0.3233 (6) | 0.3255 (1) | 0.639 (1) | 0.045 |
| C(8) | 0.4265 (7) | 0.2979 (2) | 0.777 (2) | 0.072 |
| C(9) | 0.398 (2) | 0.2559 (3) | 0.671 (3) | 0.173 |
| C(10) | 0.317 (1) | 0.2611 (2) | 0.462 (2) | 0.083 |
| C(11) | −0.2373 (4) | 0.3964 (1) | 1.2395 (8) | 0.034 |
| C(12) | −0.3233 (6) | 0.3592 (2) | 1.327 (1) | 0.062 |

Table 2. *Selected geometric parameters* (Å, °)

| | | | |
|---|---|---|---|
| S(1)—C(2) | 1.775 (5) | Na(1)···O(7) | 2.292 (6) |
| S(1)—C(3) | 1.831 (4) | O(6$^{ii}$)···O(2) | 2.691 (6) |
| O(1)—C(6) | 1.281 (6) | O(6$^{ii}$)···O(4$^{iii}$) | 2.766 (5) |
| O(2)—C(6) | 1.235 (7) | O(7)···O(1$^{iv}$) | 2.848 (7) |
| O(3)—C(5) | 1.215 (5) | O(7)···O(3) | 2.963 (7) |
| Na(1)···O(1) | 2.304 (4) | O(7)···O(4$^{iv}$) | 3.160 (7) |
| Na(1)···O(3) | 2.410 (4) | O(8)···O(1$^{iv}$) | 2.748 (6) |
| Na(1)···O(3$^{i}$) | 2.340 (4) | O(8)···O(6) | 2.833 (6) |
| Na(1)···O(6) | 2.305 (4) | | |
| C(2)—S(1)—C(3) | 90.7 (2) | | |

Symmetry codes: (i) $-x, -y+1, z$; (ii) $-x+1, -y+1, z$; (iii) $x+1, y, z-1$; (iv) $x, y, z-1$.

The high values of $wR$ and $(\Delta/\sigma)_{max}$ result from disorder of the tetrahydrofuryl group, the hydroxyethyl group and the water O atom (O7). Data collection: *AFC-5 Data Collection Software* (Rigaku Corporation, 1984*a*). Data reduction: *RASA-5P* (Rigaku Corporation, 1984*b*). Program(s) used to solve structure: *MULTAN*11/84 (Main, Germain & Woolfson, 1984). Program(s) used to refine structure: *RASA-5P*. Molecular graphics: *ORTEP* (Johnson, 1965); *PLUTO* (Motherwell & Clegg, 1978) .

Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: AS1108). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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# Di-$\mu$-chloro-bis[dichlorotris(2-propanol)cerium(III)] at 153 K

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## Abstract

The structure of di-$\mu$-chloro-bis[dichlorotris(2-propanol)cerium(III)], $[CeCl_3(C_3H_8O)_3]_2$, is reported. The Ce atoms are bridged by two Cl atoms and so a dimer is formed.

## Comment

The addition of trimethylsilylpropargyl† magnesium bromide to aldehyde affords, predominantly, allenyl alcohol. The regioselectivity changes dramatically when

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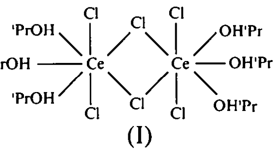
† Propargyl = 2-propynyl.

cerium organyls are used for the addition and homopropargyl alcohols are formed almost exclusively. These organocerium compounds are prepared from $CeCl_3.{}^{i}PrOH$, obtained from the reaction of $Ce^{III}$ chloride with 2-propanol, which forms a dimeric lanthanide chloride–2-propanol complex (Groth & Köhler, 1994). Single crystals were obtained from 2-propanol at room temperature. The molecular structure of the title complex (I) and the atomic numbering scheme are shown in Fig. 1.



(I)

The molecule possesses a centre of inversion in the middle of the $Ce_2Cl_2$ ring. Because of the dimerization the Ce atoms are seven-coordinated. The chloride bridge is asymmetric [Ce1—Cl1 2.980 (3) and Ce1—Cl1$^{i}$ 2.856 (3) Å; symmetry code: (i) $1-x$, $1-y$, $-z$] and the Ce—Cl distances are longer than the terminal Ce—Cl bonds [Ce1—Cl2 2.799 (2) and Ce1—Cl3 2.727 (3) Å]. Because of steric hindrance the Ce—O distances are also different [Ce1—O1 2.478 (7), Ce1—O2 2.525 (7) and Ce1—O3 2.43 (3) Å]. Two of the 2-propanol groups were found to be disordered. In the O1 group, two positions of each C atom were refined to an occupancy of 0.5:0.5 and then fixed. In the O3 group, two positions of each atom were refined to an occupancy of 0.6:0.4 and then fixed. For this part of the structure, distance restraints, the rigid-bond restraint (Rollet, 1970; Hirshfeld, 1976; Trueblood & Dunitz, 1983) and similarity restraints for the anisotropic displacement parameters were used. Because of the disorder, only the H atom bonded to O2 was found in the difference Fourier map and this was refined with a distance restraint. All other H atoms were included in calculated positions and refined using the riding model.

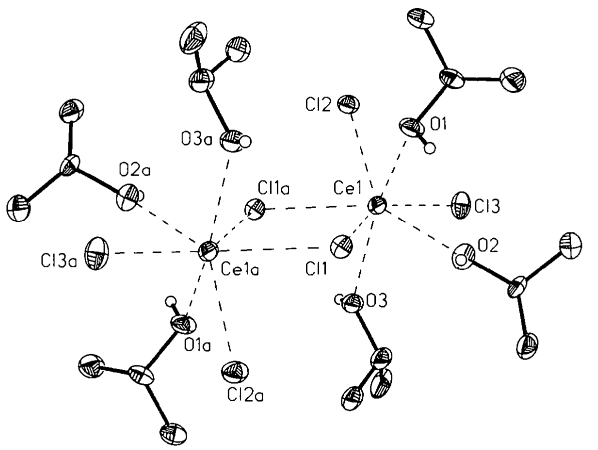


Fig. 1. Structure of the title complex showing 50% probability displacement ellipsoids. The H atoms attached to C atoms are omitted for clarity.

The crystal packing is stabilized by inter- and intramolecular hydrogen-bond interactions involving O—H···Cl interactions between the O1, O2 and O3 atoms, and the Cl3$^{ii}$ and Cl1 atoms [O1···Cl3$^{ii}$ 3.658 (9), O2···Cl3$^{ii}$ 3.105 (8) and O3···Cl1 3.57 (4) Å; symmetry code: (ii) $x-1$, $y$, $z$]. All bond distances and angles are comparable with those of the isostructural neodymium compound (Zhongsheng, Shenglong, Fusong, Cheng, Guangdi & Yuguo, 1985).

## Experimental

*Crystal data*

$[Ce_2Cl_6(C_3H_8O)_6]$
$M_r = 853.5$
Triclinic
$P\bar{1}$
$a = 5.904$ (2) Å
$b = 11.181$ (5) Å
$c = 13.354$ (6) Å
$\alpha = 69.04$ (2)°
$\beta = 77.83$ (2)°
$\gamma = 85.99$ (2)°
$V = 804.7$ (6) Å$^3$
$Z = 1$
$D_x = 1.761$ Mg m$^{-3}$

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 50 reflections
$\theta = 10$–12.5°
$\mu = 3.318$ mm$^{-1}$
$T = 153$ (2) K
Blocks
0.6 × 0.3 × 0.25 mm
Colourless

*Data collection*

Stoe Siemens AED four-circle diffractometer
Profile from $2\theta/\omega$ scans
Absorption correction: refined from $\Delta F$
$T_{min} = 0.539$, $T_{max} = 0.785$
4873 measured reflections
2849 independent reflections

2432 observed reflections [$I > 2\sigma(I)$]
$R_{int} = 0.0373$
$\theta_{max} = 25.20°$
$h = -6 \rightarrow 7$
$k = -13 \rightarrow 13$
$l = -15 \rightarrow 15$
3 standard reflections
frequency: 90 min
intensity variation: none

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.0561$
$R(F^2) = 0.2053$
$S = 1.151$
2847 reflections
222 parameters
H atoms refined using a riding model

Calculated weights $w = 1/[\sigma^2(F_o^2) + (0.1116P)^2 + 11.7681P]$ where $P = (F_o^2 + 2F_c^2)/3$
$\Delta\rho_{max} = 2.471$ e Å$^{-3}$
$\Delta\rho_{min} = -3.263$ e Å$^{-3}$
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å$^2$)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

| | $x$ | $y$ | $z$ | $U_{eq}$ |
|---|---|---|---|---|
| Ce1 | 0.65123 (10) | 0.44660 (5) | 0.16057 (5) | 0.0206 (3) |
| Cl1 | 0.2359 (5) | 0.4314 (2) | 0.0718 (2) | 0.0244 (6) |
| Cl2 | 0.8751 (5) | 0.6697 (2) | 0.1386 (2) | 0.0263 (6) |
| Cl3 | 0.8535 (5) | 0.3374 (3) | 0.3377 (2) | 0.0302 (6) |

| | | | | |
|---|---|---|---|---|
| O1 | 0.3310 (14) | 0.5692 (7) | 0.2308 (6) | 0.027 (2) |
| C1 | 0.346 (4) | 0.618 (2) | 0.3181 (15) | 0.027 (3) |
| C2 | 0.341 (5) | 0.7620 (17) | 0.274 (2) | 0.029 (4) |
| C3 | 0.161 (4) | 0.558 (2) | 0.4171 (14) | 0.031 (3) |
| C1′ | 0.267 (4) | 0.617 (2) | 0.3220 (16) | 0.030 (3) |
| C2′ | 0.280 (5) | 0.7607 (17) | 0.279 (2) | 0.031 (4) |
| C3′ | 0.418 (4) | 0.555 (2) | 0.4037 (15) | 0.030 (3) |
| O2 | 0.3774 (13) | 0.2729 (7) | 0.2972 (6) | 0.025 (2) |
| C4 | 0.381 (2) | 0.1561 (9) | 0.3915 (8) | 0.022 (2) |
| C5 | 0.299 (3) | 0.1826 (12) | 0.4962 (9) | 0.037 (3) |
| C6 | 0.242 (2) | 0.0528 (10) | 0.3835 (10) | 0.032 (3) |
| O3 | 0.780 (8) | 0.273 (2) | 0.093 (4) | 0.030 (3) |
| C7 | 0.726 (4) | 0.138 (2) | 0.131 (2) | 0.035 (3) |
| C8 | 0.946 (4) | 0.062 (2) | 0.131 (2) | 0.040 (4) |
| C9 | 0.574 (4) | 0.114 (2) | 0.061 (2) | 0.036 (3) |
| O3′ | 0.79 (1) | 0.272 (3) | 0.087 (7) | 0.032 (4) |
| C7′ | 0.693 (7) | 0.142 (3) | 0.120 (3) | 0.035 (3) |
| C8′ | 0.868 (6) | 0.047 (3) | 0.168 (3) | 0.038 (4) |
| C9′ | 0.626 (6) | 0.125 (4) | 0.024 (2) | 0.035 (4) |

Table 2. *Selected geometric parameters* (Å, °)

| | | | |
|---|---|---|---|
| Ce1—O1 | 2.478 (7) | Ce1—Cl2 | 2.799 (3) |
| Ce1—O2 | 2.525 (7) | Ce1—Cl1$^{i}$ | 2.856 (3) |
| Ce1—O3 | 2.43 (3) | Ce1—Cl1 | 2.980 (3) |
| Ce1—Cl3 | 2.727 (3) | | |
| O2—Ce1—Cl3 | 73.6 (2) | O1—Ce1—Cl1$^{i}$ | 107.4 (2) |
| O2—Ce1—Cl2 | 141.6 (2) | O3—Ce1—Cl3 | 91.6 (9) |
| Cl3—Ce1—Cl2 | 84.30 (9) | O3—Ce1—Cl2 | 131.6 (11) |
| O2—Ce1—Cl1$^{i}$ | 137.0 (2) | O3—Ce1—Cl1 | 82.0 (8) |
| Cl3—Ce1—Cl1$^{i}$ | 141.46 (8) | O1—Ce1—Cl3 | 101.9 (2) |
| Cl2—Ce1—Cl1$^{i}$ | 78.30 (8) | O1—Ce1—Cl2 | 77.4 (2) |
| O2—Ce1—Cl1 | 69.3 (2) | O1—Ce1—Cl1 | 70.8 (2) |
| Cl3—Ce1—Cl1 | 142.88 (8) | O1—Ce1—O2 | 77.0 (3) |
| Cl2—Ce1—Cl1 | 126.68 (8) | O1—Ce1—O3 | 149.6 (10) |
| Cl1$^{i}$—Ce1—Cl1 | 72.09 (9) | O2—Ce1—O3 | 80.9 (12) |
| O3—Ce1—Cl1$^{i}$ | 75.7 (11) | Ce1$^{i}$—Cl1—Ce1 | 107.91 (9) |

Symmetry code: (i) $1 - x, 1 - y, -z$.

Data collection: *DIF*4 (Stoe & Cie, 1988*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1988*b*). Program(s) used to solve structure: *SHELXS*90 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1081). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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