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Rare earth crown ether complexes: (15-crown-5)tris(nitrato)praseodymium(III)

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
R factor = 0.027
wR factor = 0.075
Data-to-parameter ratio = 14.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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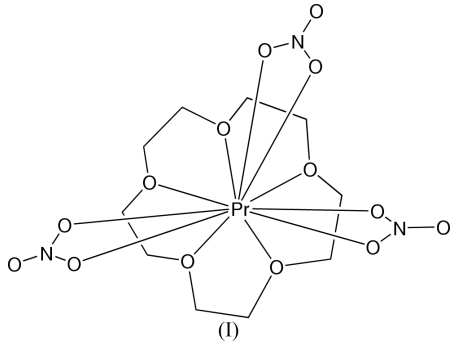
The title compound, tris(nitrato-κ^2O,O')(1,4,7,10,13-pentaoxacyclopentadecane-κ^5O)praseodymium(III), [$Pr(NO_3)_3(C_{10}H_{20}O_5)$], was obtained from a reaction designed to explore CO_2-fixation by lanthanide salts. The structure is the same as found previously for the La, Ce, Eu and Nd analogs.

Comment

We have earlier reported on the conversion of carbon dioxide into oxalate in the presence of Pr^{III} salts (Barrett Adams *et al.*, 1998). As part of a continuing study of this process, the action of carbon dioxide on an ethanolic solution of Pr^{III} nitrate and 15-crown-5 was studied. The crystals obtained at the end of the reaction proved to be [Pr(15-crown-5)$(NO_3)_3$], (I). The structure of the complex is the same as found for the La (Lu *et al.*, 1983; Rogers & Rollins, 1990), Ce (Lin & Xing, 1983), Eu (Bunzli *et al.*, 1982) and Nd (Lu *et al.*, 1983) analogs with the metal ion 11-coordinate and all three nitrate ligands on the same side of the metal.



(I)

Experimental

Crystals of the title compound were obtained from an ethanol solution, originally charged with 1 mmol each of Pr^{III} nitrate and 15-crown-5, that had been treated with carbon dioxide under reflux as part of a study of the chemical activation and reduction of carbon dioxide promoted by trivalent lanthanide ions. Analysis calculated for $C_{10}H_{20}N_3O_{14}Pr$: C 21.94, H 3.88, N 7.68%; found: C 21.4, H 3.6, N 7.7%.

Crystal data

[$Pr(NO_3)_3(C_{10}H_{20}O_5)$]
M_r = 547.20
Monoclinic, $P2_1/c$
a = 9.3236 (5) Å
b = 14.5789 (14) Å
c = 13.6184 (6) Å
β = 95.361 (4)°
V = 1843.0 (2) Å^3
Z = 4
D_x = 1.972 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 24.3–25.8°
μ = 2.722 mm^{-1}
T = 293 (2) K
Plate, light green
0.50 × 0.50 × 0.26 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: empirical *via* ψ scan (North *et al.*, 1968)
T_{min} = 0.227, T_{max} = 0.426
3838 measured reflections
3612 independent reflections
2940 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 26.0°
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 17$
$l = -16 \rightarrow 16$
2 standard reflections
frequency: 120 min
intensity decay: 5.1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.075
S = 1.04
3611 reflections
254 parameters
H atoms constrained
$w = 1/[\sigma^2(F_o^2) + (0.0445P)^2 + 0.7265P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.74 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0083 (4)

Data collection and cell refinement: *CAD*-4 *Software* (Enraf–Nonius, 1989); data reduction: *XCAD*4 (Harms & Wocadlo, 1987); structure solution: *SHELXS*97 (Sheldrick, 1990); structure refinement: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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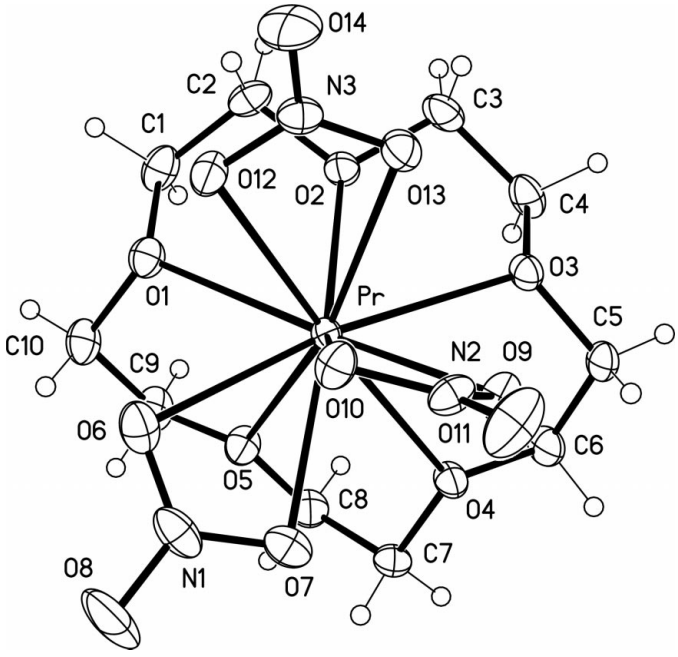

Figure 1
Perspective view of the title molecule.

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