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Heptaaqua[tetrachlorophthalato(2−)]praseodymium(III) tetrachlorophthalate(−) tetrachlorophthalic acid monohydrate

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The title complex, $[Pr(C_8Cl_4O_4)(H_2O)_7](C_8HCl_4O_4)\cdot C_8H_2Cl_4O_4\cdot H_2O$, has been synthesized and structurally characterized. The praseodymium(III) ion is coordinated by an O atom of a tetrachlorophthalate ligand and seven O atoms of water molecules. The molecular packing is reinforced by an extensive network of O—H···O hydrogen bonds.

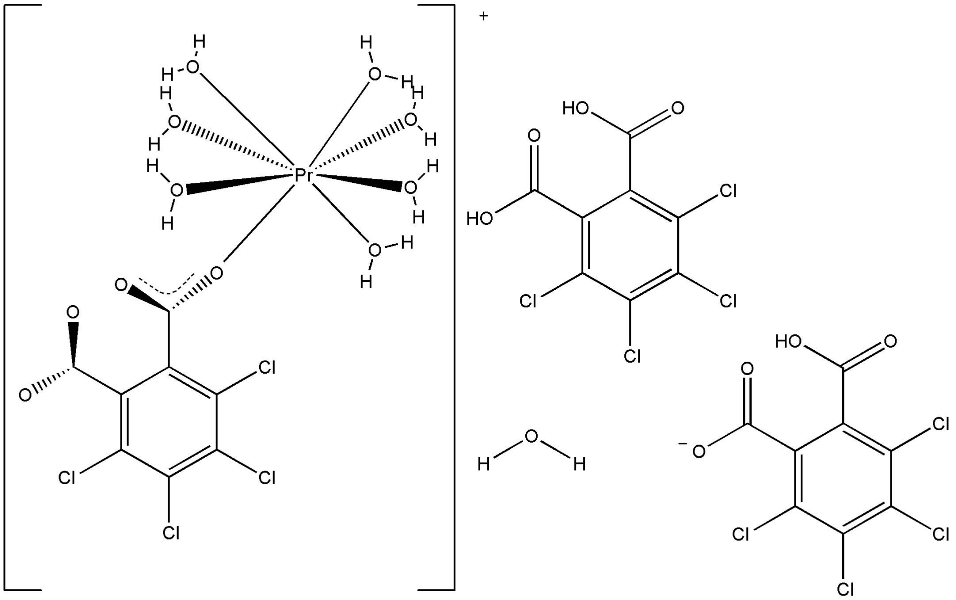
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.023
wR factor = 0.057
Data-to-parameter ratio = 13.3

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

Comment

Owing to their variety of structures and unusual properties, transition metal complexes of benzenedicarboxylate dianions are of great interest (Jian *et al.*, 1993; Bakalbassis *et al.*, 1998; Lewinski *et al.*, 1998; Yang *et al.*, 2002). To date, most of the published work concerns transition metal phthalate complexes. An interesting example of a lanthanide–tetrachlorophthalate coordination polymer has been reported (Liang *et al.*, 2004). We combined praseodymium(III) and tetrachlorophthalic acid (H_2tcph), and the title complex, (I) (Fig. 1), was obtained.



(I)

Compound (I) consists of a $[Pr(tcph)(H_2O)_7]^+$ cation, a neutral (H_2tcph) molecule, an (Htcph)$^-$ anion and an uncoordinated water molecule. The Pr^{III} atom is surrounded by eight O atoms, one from a tcph ligand and the others from coordinated water molecules. The Pr—O bond distances (Table 1) range from 2.424 (2) to 2.507 (2) Å.

Two adjacent $[Pr(tcph)(H_2O)_7]^+$ cations are linked by two short hydrogen bonds [O···O = 2.732 (4) Å] between uncoordinated carboxylate O atoms and coordinated water

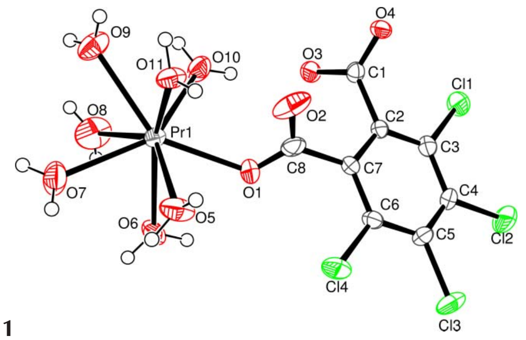


Figure 1
A view of the $[Pr(tcph)(H_2O)_7]^+$ cation in (I), showing 30% probability displacement ellipsoids.

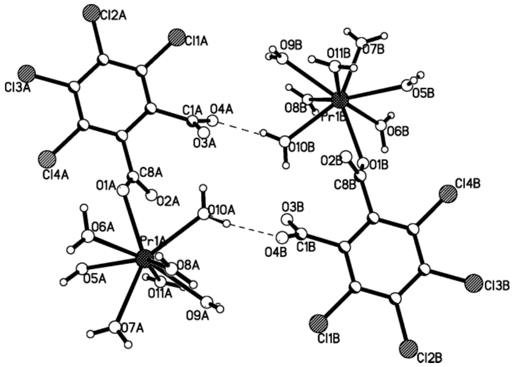


Figure 2
A fragment of (I), showing the hydrogen-bonded (dashed lines) dimer of $[Pr(tcph)(H_2O)_7]^+$ units. The atom labels of the asymmetric unit have suffix A and those atoms generated by the symmetry operation $(-x, 2 - y, 2 - z)$ have suffix B.

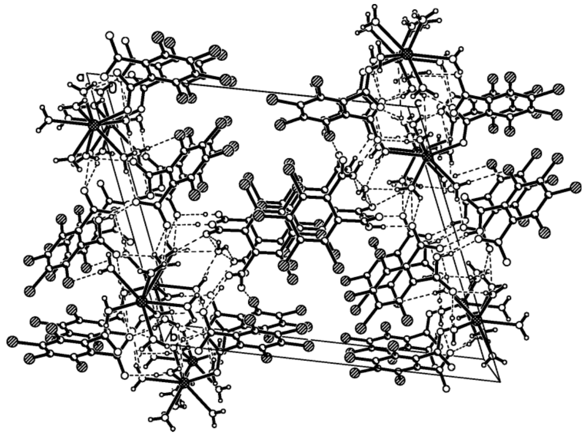


Figure 3
A packing diagram for (I). Dashed lines indicate hydrogen bonds.

molecules, as shown in Fig. 2. In addition, the packing of the molecules in (I) involves intermolecular hydrogen bonding (Table 2), also involving the other uncoordinated carboxylate O atoms and water molecules [O$\cdots$O distances range from 2.558 (3) to 3.171 (4) Å].

Experimental

A solution of $Pr(ClO_4)_3$ (0.1 mmol) in H_2O (10 ml) was added to a suspension of H_2tcph (0.1 mmol) in H_2O (10 ml). The mixture was stirred at room temperature for 30 min. After filtration, the solution was left undisturbed and green crystals of (I) were obtained after several days. Analysis calculated for $C_{24}H_{19}Cl_{12}O_{20}Pr$: C 24.12, H 1.59, Cl 35.68 O 26.80%; found: C 24.19, H 1.62%.

Crystal data

$[Pr(C_8Cl_4O_4)(H_2O)_7](C_8HCl_4O_4)\cdot$-
$C_8H_2Cl_4O_4\cdot H_2O$
$M_r = 1193.70$
Triclinic, $P\bar{1}$
$a = 6.9395$ (5) Å
$b = 16.2519$ (13) Å
$c = 19.1287$ (15) Å
$\alpha = 67.382$ (1)°
$\beta = 86.524$ (1)°
$\gamma = 81.535$ (1)°
$V = 1969.7$ (3) $Å^3$
$Z = 2$
$D_x = 2.013$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5233 reflections
$\theta = 2.2$–27.7°
$\mu = 2.13$ mm^{-1}
$T = 293$ (2) K
Block, green
0.42 × 0.28 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
$T_{min} = 0.414$, $T_{max} = 0.711$
10795 measured reflections
6883 independent reflections
6066 reflections with $I > 2\sigma(I)$
$R_{int} = 0.017$
$\theta_{max} = 25.0°$
$h = -8 \rightarrow 7$
$k = -17 \rightarrow 19$
$l = -21 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.023$
$wR(F^2) = 0.057$
$S = 1.07$
6883 reflections
518 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0273P)^2 + 0.4655P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.54$ e $Å^{-3}$
$\Delta\rho_{min} = -0.50$ e $Å^{-3}$
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.0061 (3)

Table 1
Selected bond lengths (Å).

Pr1—O1	2.424 (2)	Pr1—O6	2.486 (2)
Pr1—O11	2.432 (2)	Pr1—O5	2.491 (2)
Pr1—O10	2.451 (2)	Pr1—O9	2.505 (2)
Pr1—O8	2.466 (3)	Pr1—O7	2.507 (2)

Table 2
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O5—H5$A\cdots$O3^{i}	0.85	1.92	2.727 (4)	158
O5—H5$B\cdots$O19^{ii}	0.85	1.95	2.791 (4)	172
O6—H6$A\cdots$O17^{ii}	0.85	1.89	2.732 (4)	170
O7—H7$A\cdots$O19^{ii}	0.85	2.25	3.034 (4)	153
O7—H7$B\cdots$O14^{iii}	0.85	2.05	2.897 (3)	172
O8—H8$A\cdots$O20^{iv}	0.85	1.88	2.715 (4)	167
O8—H8$B\cdots$O16^{iv}	0.85	2.15	2.760 (4)	129
O9—H9$A\cdots$O20^{iv}	0.85	1.95	2.787 (4)	166
O9—H9$B\cdots$O2^{v}	0.85	2.10	2.916 (4)	161
O10—H10$A\cdots$O4^{vi}	0.85	1.90	2.736 (3)	168
O10—H10$B\cdots$O3	0.85	2.03	2.858 (3)	164
O11—H11$A\cdots$O2^{v}	0.85	1.87	2.716 (4)	174
O11—H11$B\cdots$O3^{i}	0.85	1.89	2.692 (3)	158
O13—H13$\cdots$O4^{vii}	0.82	1.75	2.558 (3)	167
O15—H15$\cdots$O16^{viii}	0.82	1.78	2.590 (3)	168
O18—H18$\cdots$O17^{ix}	0.82	1.78	2.580 (3)	166
O20—H20$A\cdots$O14^{ii}	0.85	2.05	2.817 (4)	149
O20—H20$B\cdots$O13^{viii}	0.85	2.18	2.850 (4)	135
O20—H20$B\cdots$O15^{viii}	0.85	2.59	3.171 (4)	127

Symmetry codes: (i) $x+1, y, z$; (ii) $-x+1, -y+1, -z+1$; (iii) $-x+1, -y+1, -z+2$; (iv) $x, y, z+1$; (v) $-x+1, -y+2, -z+2$; (vi) $-x, -y+2, -z+2$; (vii) $x, y-1, z$; (viii) $-x, -y+1, -z+1$; (ix) $-x, -y+1, -z$.

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All H atoms were located in difference Fourier maps, relocated in idealized positions (O—H = 0.82–0.85 Å) and refined as riding, with $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(O).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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