H atoms constrained to give C—H = 0·96 Å, H—C—H = 109·5°, aromatic H on ring-angle external bisectors, $U(\text{H}) = 1{\cdot}2U_{eq}(\text{C})$, isotropic extinction parameter $x = 3{\cdot}6\,(2) \times 10^{-7}$ [$1{\cdot}42\,(4) \times 10^{-6}$] to give $F_c' = F_c/(1 + xF_c^2/\sin2\theta)^{1/4}$ [absolute structure determination by refinement of $\eta = -1{\cdot}04$ (1), followed by inversion to give the results here (Rogers, 1981)]. 317 [387] parameters, $R = 0{\cdot}0279$ [0·0253], $wR = 0{\cdot}0283$ [0·0318], mean $\Delta/\sigma = 0{\cdot}010$ [0·006], max. = 0·047 [0·018], max. $\Delta\rho = 0{\cdot}57$ [0·97], min. = −0·45 [−1·59] e Å^{-3}, slope of normal probability plot = 1·72 [1·20]. Scattering factors from *International Tables for X-ray Crystallography* (1974), *SHELXTL* programs (Sheldrick, 1985).

Atomic parameters are given in Tables 1 and 2,* selected bond lengths and angles in Tables 3 and 4. Atomic numbering for the anions is shown in Figs. 1 and 2.

Related literature. The two Re complexes are isostructural with their Mo analogues (Ellis *et al.*, 1986; Boyde *et al.*, 1986), and can thus be used as diamagnetic hosts for single-crystal ESR studies of the paramagnetic Mo complexes.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and complete lists of bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44334 (45 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Hexaaquatris(oxalato)dipraseodymium(III) Pentahydrate (a Redetermination)

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Abstract. $[Pr_2(C_2O_4)_3(H_2O)_6].5H_2O$, $M_r = 744{\cdot}0$, monoclinic, $P2_1/c$, $a = 11{\cdot}236\,(2)$, $b = 9{\cdot}634\,(3)$, $c = 10{\cdot}320\,(2)$ Å, $\beta = 114{\cdot}55\,(1)°$, $V = 1016{\cdot}1$ Å^3, $Z = 2$, $D_x = 2{\cdot}44$ Mg m^{-3}, $F(000) = 720$, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 4{\cdot}60$ mm^{-1}, $T = 293$ K, $R = 0{\cdot}046$ for 2162 unique reflexions [$F > 3\sigma(F)$]. This work is a redetermination of an earlier photographic structural investigation [Ollendorff & Weigel (1969). *Inorg. Nucl. Chem. Lett.* **5**, 263–269] for which $R = 0{\cdot}130$. The nine O atoms which make up the praseodymium coordination sphere are derived from three water and three oxalate ligands. Each oxalate group lies on a crystallographic inversion centre, thereby linking the Pr atoms into infinite hexagonal nets parallel to the *ac* plane. An interplanar separation of *b*/2 is sufficient to accommodate 'zeolitic' water, which shows minor differences in hydrogen bonding compared to an otherwise isostructural neodymium analogue {structure determined at 223 K by Hansson [*Acta Chem Scand.* (1973), **27**, 2852–2860], reported with a different choice of *c* relative to the title compound}.

Experimental. The title compound was prepared by diffusion of an aqueous solution of a Pr^{3+} salt in silica gel containing oxamic acid at pH 5·5 to 7·5. Single crystals suitable for X-ray work formed as the oxamate anions were slowly hydrolysed to oxalate.

Colourless trapezoid 0·26 × 0·1 × 0·1 mm, Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized Mo $K\alpha$ radiation, unit-cell dimensions from setting angles of 25 accurately centred reflexions ($6{\cdot}1 < \theta < 17{\cdot}1°$), $\omega/2\theta$ scan mode used to measure

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Table 1. *Fractional atomic coordinates, equivalent isotropic thermal parameters* (Å^2) *and occupancy factors*

	x	y	z	U_{eq}*	
Pr(1)	0·18935 (3)	0·04570 (4)	0·33153 (3)	0·0192	1·00
O(1)	0·3923 (4)	−0·0702 (5)	0·3393 (5)	0·0293	1·00
O(2)	0·3942 (4)	0·1057 (6)	0·5388 (5)	0·0332	1·00
O(3)	0·1262 (5)	−0·1019 (6)	0·1172 (5)	0·0383	1·00
O(4)	0·0086 (4)	0·1408 (5)	0·1096 (5)	0·0294	1·00
O(5)	−0·0291 (5)	−0·0265 (6)	0·3228 (5)	0·0320	1·00
O(6)	0·1588 (5)	0·0634 (6)	0·5657 (5)	0·0397	1·00
O(7)	0·2081 (6)	−0·1960 (6)	0·4249 (7)	0·0538	1·00
O(8)	0·2977 (5)	0·1927 (7)	0·2053 (6)	0·0550	1·00
O(9)	0·1687 (6)	0·2962 (6)	0·3850 (6)	0·0535	1·00
C(1)	0·4994 (6)	0·0502 (7)	0·5579 (7)	0·0244	1·00
C(2)	0·0344 (6)	−0·0701 (7)	0·0020 (7)	0·0252	1·00
C(3)	0·0545 (6)	0·0256 (7)	0·5704 (7)	0·0262	1·00
O(1w)	0·4253 (14)	−0·1908 (27)	0·7040 (26)	0·1109	0·60 (4)
O(2w)	0·4382 (16)	−0·3022 (44)	0·6213 (29)	0·1204	0·49 (4)
O(3w)	0·3385 (18)	−0·0975 (25)	−0·1749 (29)	0·1546	0·58 (3)
O(4w)	0·3882 (26)	0·0424 (37)	0·0495 (38)	0·2008	0·48 (4)
O(5w)	0·4324 (35)	0·0214 (37)	−0·0733 (43)	0·1946	0·42 (4)

$*U_{eq} = \frac{1}{3}\sum_i \sum_j U_{ij}\mathbf{a}_i.\mathbf{a}_j a_i^* a_j^*$.

2490 reflexions with $I > 0$, scan width of 0·80 + 0·35 × tanθ and scan speed ranging from 1·2 to 5° min^{-1} according to the intensity gathered in a pre-scan, $0 < h < 14$, $0 < k < 12$, $-13 < l < 13$, $0 < \theta < 28°$, 2162 unique structure amplitudes with $F > 3\sigma(F)$, $R_{int} = 0{\cdot}007$, no drift in intensity standards ($\bar{4}02$, $22\bar{2}$, $\bar{5}04$) measured every 2 h, Lp and absorption corrections (transmission factors, max. 0·6640, min. 0·4363), Patterson synthesis used to locate Pr, remaining non-hydrogen atoms from ΔF maps, hydrogen ignored, full-matrix least squares based on F using *SHELX* (Sheldrick, 1976), final $R = 0{\cdot}046$, $wR = 0{\cdot}055$, $w = 1{\cdot}0/[\sigma^2(F) + 0{\cdot}000314F^2]$, anisotropic thermal parameters throughout, empirical extinction correction applied $[F_c^* = F_c(1{\cdot}0\text{–}0{\cdot}0000000054F_c^2/\sin\theta)]$. Population parameters for the 'zeolitic' water, O(1w) to O(5w), were also refined, converging to 10·4 (8) per unit cell compared with a possible maximum of 10. The compound was therefore formulated as $(H_2O)_6(C_2O_4)_3Pr_2.5H_2O$ as microanalytical results proved inconclusive. Residual electron density in final ΔF map in range −2·8 to 3·2 e Å^{-3} near Pr and ±0·9 e Å^{-3} elsewhere, maximum Δ/σ 0·044 [U_{22}, O(4w)]. Scattering factors from *International Tables for X-ray Crystallography* (1974), computations carried out on the joint CDC 7600/Amdahl 470 system of the University of Manchester Regional Computing Centre.

Table 1† contains final atomic parameters, selected molecular geometry is in Table 2. The crystal structure is illustrated in Fig. 1.

Table 2. *Selected bond lengths* (Å) *and angles* (°)

Pr(1)–O(1)	2·510 (5)	Pr(1)–O(2)	2·473 (4)
Pr(1)–O(3)	2·473 (5)	Pr(1)–O(4)	2·519 (4)
Pr(1)–O(5)	2·516 (5)	Pr(1)–O(6)	2·584 (5)
Pr(1)–O(7)	2·495 (6)	Pr(1)–O(8)	2·550 (5)
Pr(1)–O(9)	2·507 (6)	O(2)–C(1)	1·236 (8)
O(3)–C(2)	1·246 (8)	O(6)–C(3)	1·248 (8)
C(1)–C(1^{i})	1·54 (1)	C(2)–C(2^{ii})	1·55 (1)
C(3)–C(3^{iii})	1·54 (1)		
O(4)–Pr(1)–O(1)	124·7 (2)	O(5)–Pr(1)–O(3)	84·0 (2)
O(6)–Pr(1)–O(1)	118·1 (2)	O(6)–Pr(1)–O(4)	117·2 (2)
O(7)–Pr(1)–O(2)	89·2 (2)	O(7)–Pr(1)–O(3)	75·9 (2)
O(7)–Pr(1)–O(5)	71·3 (2)	O(8)–Pr(1)–O(2)	80·9 (2)
O(8)–Pr(1)–O(3)	83·3 (2)	O(9)–Pr(1)–O(2)	73·9 (2)
O(9)–Pr(1)–O(5)	95·5 (2)	O(9)–Pr(1)–O(8)	71·7 (2)

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

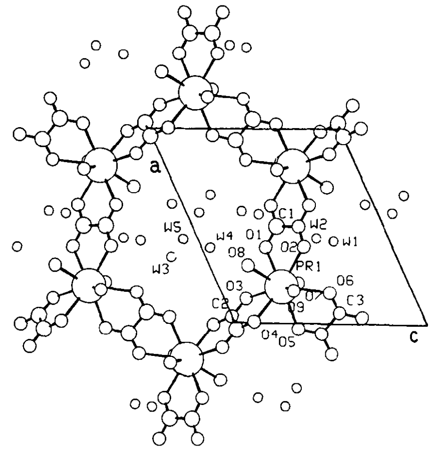

Fig. 1. A part of the Pr–oxalate layer at $y = 0$ produced using *PLUTO* (Motherwell & Clegg, 1978). A second layer at $y = \frac{1}{2}$ has been omitted for clarity.

Related literature. A room-temperature structure determination of neodymium(III) oxalate 10·5 hydrate has been reported by Hansson (1970).

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† Lists of structure factors, anisotropic vibrational parameters and distances between disordered water molecules have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44401 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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