Crystal structure of bis(aminoguanidinium) (hydrogen triethylene-tetramine-hexaacetato)praseodymate(III) hydrate, $C_{18}H_{25}N_4O_{12}Pr \cdot 2CH_7N_4 \cdot 3.7H_2O$

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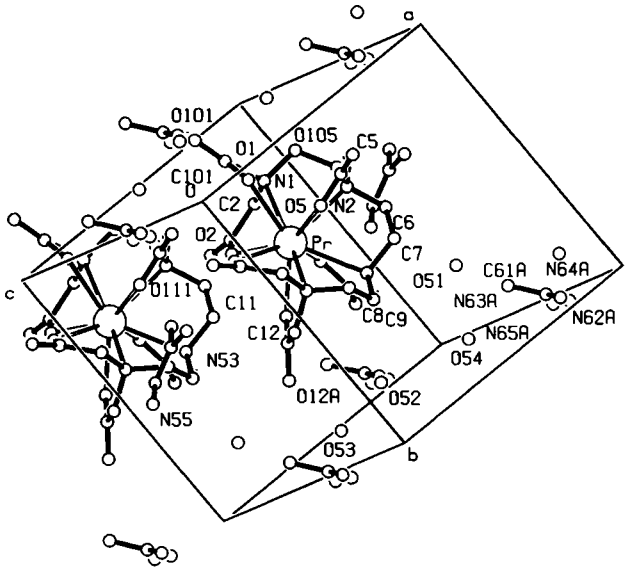

Abstract

$C_{20}H_{46.40}N_{12}O_{15.70}Pr$, triclinic, $P\bar{1}$ (No. 2), a = 10.329(2) Å, b = 10.725(3) Å, c = 15.635(4) Å, α = 107.05(2)°, β = 90.43(2)°, γ = 93.35(2)°, V = 1652.5 Å^3, Z = 2, $R_{gt}(F)$ = 0.050, $wR_{ref}(F^2)$ = 0.149, T = 293 K.

Source of material

Within the context of preparation of inorganic materials, the so-called chelate method, involving the low temperature decomposition of solid metal precursors obtained from a solution of the complexed metals has proved to be an advantageous way to get multimetallic oxides [1]. During the search for potential precursors for Pr-based ceramics, the title compound was isolated. The complex was obtained in the same way as the equivalent Bi complex $(gu)_2Bi(Httha)$ but $Pr_2(CO_3)_3$ was used in place of Bi oxocarbonate and methanol was added to the final solution to get suitable crystals for X-ray diffraction.

Experimental details

All the hydrogen atoms were refined with a common isotropic temperature factor (U_{iso} = 0.059 Å^2). There are two positions for atom O112: O12A with a site occupation factor (sof) of 0.73 and O12B for which the occupation is 0.27. One aminoguanidinium is also disordered: in the first position the amino group (N65) is bonded to N64 (sof 0.70) and also involved in an hydrogen bond to the water molecule O53. In the second orientation (sof 0.30), the terminal NH_2 is bonded to N63 but it is distant from O53 by only 1.6 Å. The presence of O53 is thus linked to the occupation of the first position; this explains the number of 3.7 water molecules by unit of $(agu)_2Pr(Httha)$ complex.The hydrogen atoms of water molecules were not localized.

Discussion

The structure of the complex $(agu)_2Pr(Httha) \cdot 3.7H_2O$ is very similar to that of $(gu)_2Pr(Httha) \cdot 3.5H_2O$ reported in the preceeding paper [2]. The unit cell is 50 Å^3 larger to accomodate the two bigger aminoguanidinium cations. Nevertheless, the structures are not isomorphous, the position of Pr cation in this structure being at 0.9 Å from that observed in $(gu)_2Pr(Httha)$. The metal is tenfold coordinated by 6 carboxylic oxygen and 4 nitrogen atoms (see figure of the preceeding paper [2]). The average values for Pr—O and Pr—N distances are 2.474(3) Å and 2.793(3) Å, respectively. The coordination distance involving the COOH group, d(Pr···O101) = 2.704(3) Å, is significantly longer than the mean Pr—O value. The coordination polyhedron is similar to that observed in the bis(guanidinium) (hydrogen triethylene-tetraminehexaacetato)bismuthate(III) [3].

Table 1. Data collection and handling.

Crystal:	yellow parallelipiped, size 0.16 × 0.24 × 0.30 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	15.62 cm^{-1}
Diffractometer, scan mode:	Huber, ω
$2\theta_{max}$:	70.06°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	14591, 14591
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 10933
$N(param)_{refined}$:	466
Programs:	SHELXS-86 [4], SHELXL-93 [5], PLATON [6]

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Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	x	y	z	U_{iso}
H(1A)	2*i*		0.2563(5)	–0.0129(4)	0.2813(3)	0.059(3)
H(1B)	2*i*		0.3783(5)	–0.0785(4)	0.2331(3)	0.059(3)
H(101)	2*i*		0.165(6)	–0.154(6)	0.068(4)	0.059(3)
H(2A)	2*i*		0.4478(4)	0.2431(4)	0.3876(2)	0.059(3)
H(2B)	2*i*		0.4018(4)	0.1035(4)	0.3930(2)	0.059(3)
H(3A)	2*i*		0.5342(4)	0.0339(4)	0.1836(3)	0.059(3)
H(3B)	2*i*		0.5769(4)	0.0673(4)	0.2846(3)	0.059(3)
H(4A)	2*i*		0.6068(4)	0.2873(4)	0.2945(3)	0.059(3)
H(4B)	2*i*		0.6926(4)	0.2049(4)	0.2190(3)	0.059(3)
H(5A)	2*i*		0.5559(4)	0.1062(4)	0.0791(3)	0.059(3)
H(5B)	2*i*		0.6409(4)	0.2179(4)	0.0568(3)	0.059(3)
H(6A)	2*i*		0.6944(4)	0.3997(4)	0.1542(3)	0.059(3)
H(6B)	2*i*		0.6256(4)	0.4545(4)	0.2453(3)	0.059(3)
H(7A)	2*i*		0.5891(4)	0.5757(4)	0.1437(3)	0.059(3)
H(7B)	2*i*		0.5137(4)	0.4520(4)	0.0797(3)	0.059(3)
H(8A)	2*i*		0.3786(4)	0.6908(4)	0.2949(3)	0.059(3)
H(8B)	2*i*		0.5267(4)	0.6715(4)	0.2807(3)	0.059(3)
H(9A)	2*i*		0.3968(4)	0.6580(4)	0.1121(3)	0.059(3)
H(9B)	2*i*		0.2853(4)	0.6694(4)	0.1809(3)	0.059(3)
H(10A)	2*i*		0.2056(4)	0.5686(4)	0.0347(3)	0.059(3)
H(10B)	2*i*		0.3006(4)	0.4565(4)	0.0250(3)	0.059(3)
H(11A)	2*i*		0.1365(4)	0.2979(4)	–0.0075(2)	0.059(3)
H(11B)	2*i*		0.0156(4)	0.3834(4)	0.0086(2)	0.059(3)

Table 2. Continued.

Atom	Site	Occ.	x	y	z	U_{iso}
H(12A)	2*i*		–0.0265(4)	0.4956(4)	0.1486(3)	0.059(3)
H(12B)	2*i*		0.0661(4)	0.6161(4)	0.1478(3)	0.059(3)
H(52A)	2*i*		–0.0377(5)	0.3457(6)	0.5679(3)	0.059(3)
H(52B)	2*i*		0.0403(5)	0.2900(6)	0.4875(3)	0.059(3)
H(53A)	2*i*		–0.0229(4)	0.3073(5)	0.3498(3)	0.059(3)
H(53B)	2*i*		–0.1409(4)	0.3773(5)	0.3422(3)	0.059(3)
H(54)	2*i*		–0.2157(4)	0.4546(5)	0.5568(3)	0.059(3)
H(55A)	2*i*		–0.2864(5)	0.4575(5)	0.3914(3)	0.059(3)
H(55B)	2*i*		–0.3611(5)	0.5166(5)	0.4729(3)	0.059(3)
H(62A)	2*i*	0.67(1)	0.8618(5)	1.0307(4)	0.0739(3)	0.059(3)
H(62B)	2*i*	0.67	0.7590(5)	0.9221(4)	0.0484(3)	0.059(3)
H(63A)	2*i*	0.67	0.7063(5)	0.8044(5)	0.1431(3)	0.059(3)
H(63B)	2*i*	0.67	0.7762(5)	0.8334(5)	0.2311(3)	0.059(3)
H(64A)	2*i*	0.67	0.9779(5)	1.0695(5)	0.2031(3)	0.059(3)
H(65A)	2*i*	0.67	0.875(1)	0.954(1)	0.3210(7)	0.059(3)
H(65B)	2*i*	0.67	0.987(1)	1.053(1)	0.3434(7)	0.059(3)
H(62C)	2*i*	0.33	0.862(2)	1.031(2)	0.074(1)	0.059(3)
H(62D)	2*i*	0.33	0.759(2)	0.922(2)	0.048(1)	0.059(3)
H(63C)	2*i*	0.33	0.969(2)	1.079(2)	0.206(1)	0.059(3)
H(63D)	2*i*	0.33	0.938(2)	1.002(2)	0.270(1)	0.059(3)
H(64B)	2*i*	0.33	0.696(2)	0.821(2)	0.147(1)	0.059(3)
H(65C)	2*i*	0.33	0.850(2)	0.814(2)	0.282(1)	0.059(3)
H(65D)	2*i*	0.33	0.729(2)	0.729(2)	0.251(1)	0.059(3)

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pr	2*i*		0.28422(2)	0.31236(2)	0.20536(1)	0.02346(8)	0.02185(7)	0.02410(8)	0.00127(5)	–0.00026(5)	0.00653(5)
C(101)	2*i*		0.2543(4)	–0.0302(3)	0.1488(3)	0.040(2)	0.024(1)	0.039(2)	0.001(1)	–0.007(1)	0.008(1)
C(1)	2*i*		0.3207(5)	–0.0088(4)	0.2372(3)	0.057(2)	0.028(2)	0.039(2)	0.000(2)	–0.011(2)	0.014(1)
O(1)	2*i*		0.2514(4)	0.0562(3)	0.1128(2)	0.067(2)	0.034(1)	0.046(2)	–0.009(1)	–0.021(2)	0.018(1)
O(101)	2*i*		0.1976(3)	–0.1466(3)	0.1169(2)	0.053(2)	0.028(1)	0.045(2)	–0.006(1)	–0.014(1)	0.010(1)
C(102)	2*i*		0.2492(4)	0.2162(4)	0.3883(3)	0.038(2)	0.039(2)	0.034(2)	0.003(2)	0.000(1)	0.014(1)
C(2)	2*i*		0.3842(4)	0.1704(4)	0.3648(2)	0.041(2)	0.035(2)	0.028(2)	0.005(1)	–0.004(1)	0.009(1)
O(102)	2*i*		0.2130(4)	0.2272(4)	0.4657(2)	0.059(2)	0.078(2)	0.037(2)	0.018(2)	0.008(1)	0.026(2)
O(2)	2*i*		0.1817(3)	0.2431(3)	0.3285(2)	0.035(1)	0.047(2)	0.040(1)	0.007(1)	0.003(1)	0.024(1)
N(1)	2*i*		0.3959(3)	0.1174(3)	0.2675(2)	0.038(2)	0.028(1)	0.028(1)	0.003(1)	–0.004(1)	0.005(1)
C(3)	2*i*		0.5322(4)	0.0996(4)	0.2414(3)	0.042(2)	0.035(2)	0.037(2)	0.014(2)	–0.002(2)	0.006(1)
C(4)	2*i*		0.6039(4)	0.2234(4)	0.2358(3)	0.029(2)	0.042(2)	0.034(2)	0.010(1)	–0.003(1)	0.001(1)
C(105)	2*i*		0.4513(4)	0.2103(3)	0.0148(2)	0.035(2)	0.028(1)	0.030(2)	–0.001(1)	0.004(1)	0.005(1)
C(5)	2*i*		0.5574(4)	0.1965(4)	0.0787(3)	0.037(2)	0.045(2)	0.032(2)	0.014(2)	0.007(1)	0.002(2)
O(105)	2*i*		0.4718(3)	0.1739(3)	–0.0668(2)	0.044(2)	0.055(2)	0.030(1)	–0.000(1)	0.007(1)	0.007(1)
O(5)	2*i*		0.3462(3)	0.2550(3)	0.0485(2)	0.033(1)	0.035(1)	0.030(1)	0.005(1)	0.0024(9)	0.007(1)
N(2)	2*i*		0.5442(3)	0.2806(3)	0.1707(2)	0.029(1)	0.034(1)	0.030(1)	0.006(1)	0.001(1)	0.002(1)
C(6)	2*i*		0.6105(4)	0.4111(4)	0.1820(3)	0.027(2)	0.041(2)	0.050(2)	–0.005(1)	0.002(2)	0.011(2)
C(7)	2*i*		0.5349(4)	0.4980(4)	0.1419(3)	0.032(2)	0.036(2)	0.045(2)	–0.008(1)	0.007(2)	0.013(2)
C(108)	2*i*		0.4450(4)	0.5592(4)	0.3547(3)	0.030(2)	0.035(2)	0.036(2)	0.003(1)	–0.003(1)	0.005(1)
C(8)	2*i*		0.4428(4)	0.6258(4)	0.2807(3)	0.045(2)	0.027(2)	0.040(2)	–0.006(1)	–0.004(2)	0.007(1)
O(108)	2*i*		0.4756(4)	0.6314(3)	0.4307(2)	0.067(2)	0.041(2)	0.037(2)	0.003(2)	–0.015(2)	–0.001(1)
O(8)	2*i*		0.4125(3)	0.4392(3)	0.3350(2)	0.039(1)	0.030(1)	0.031(1)	–0.001(1)	–0.007(1)	0.0065(9)
C(9)	2*i*		0.3373(4)	0.6081(4)	0.1392(3)	0.041(2)	0.027(2)	0.044(2)	–0.006(1)	–0.005(2)	0.018(1)
N(3)	2*i*		0.4136(3)	0.5376(3)	0.1895(2)	0.033(1)	0.027(1)	0.032(1)	–0.002(1)	–0.001(1)	0.007(1)
C(10)	2*i*		0.2493(4)	0.5181(4)	0.0670(3)	0.038(2)	0.032(2)	0.034(2)	–0.001(1)	–0.001(1)	0.018(1)
N(4)	2*i*		0.1518(3)	0.4459(3)	0.1064(2)	0.032(1)	0.026(1)	0.031(1)	0.002(1)	0.000(1)	0.009(1)
C(111)	2*i*		0.0079(4)	0.2482(4)	0.0787(3)	0.030(2)	0.033(2)	0.038(2)	–0.003(1)	–0.006(1)	0.013(1)
C(11)	2*i*		0.0781(4)	0.3439(4)	0.0375(2)	0.035(2)	0.034(2)	0.032(2)	–0.002(1)	–0.007(1)	0.012(1)
O(11)	2*i*		0.0528(3)	0.2369(3)	0.1506(2)	0.033(1)	0.041(1)	0.045(2)	–0.010(1)	–0.012(1)	0.021(1)
O(111)	2*i*		–0.0913(3)	0.1842(4)	0.0377(2)	0.050(2)	0.059(2)	0.054(2)	–0.022(2)	–0.023(2)	0.024(2)
C(112)	2*i*		0.0851(5)	0.5641(5)	0.2616(3)	0.063(3)	0.060(3)	0.039(2)	0.034(2)	0.015(2)	0.014(2)
C(12)	2*i*		0.0611(4)	0.5345(4)	0.1630(3)	0.037(2)	0.033(2)	0.037(2)	0.012(1)	0.002(1)	0.011(1)
O(12)	2*i*		0.1618(3)	0.4997(3)	0.2910(2)	0.044(2)	0.042(1)	0.032(1)	0.018(1)	0.006(1)	0.010(1)
O(12A)	2*i*	0.73(7)	0.038(3)	0.668(2)	0.3112(5)	0.11(1)	0.077(8)	0.046(3)	0.065(9)	0.008(4)	0.007(4)
O(12B)	2*i*	0.27	–0.025(6)	0.60(1)	0.301(2)	0.09(3)	0.14(5)	0.05(1)	0.08(3)	0.02(1)	0.03(2)
C(51)	2*i*		–0.1061(5)	0.3734(5)	0.4627(3)	0.038(2)	0.059(3)	0.044(2)	–0.002(2)	0.006(2)	0.003(2)
N(52)	2*i*		–0.0252(5)	0.3316(6)	0.5117(3)	0.056(3)	0.091(4)	0.050(2)	0.027(3)	0.013(2)	0.018(2)
N(53)	2*i*		–0.0878(4)	0.3498(5)	0.3742(3)	0.052(2)	0.077(3)	0.037(2)	0.011(2)	0.007(2)	–0.001(2)
N(54)	2*i*		–0.2061(4)	0.4368(5)	0.5000(3)	0.047(2)	0.069(3)	0.039(2)	0.011(2)	0.008(2)	0.002(2)
N(55)	2*i*		–0.2958(5)	0.4751(5)	0.4483(3)	0.052(2)	0.077(3)	0.044(2)	0.015(2)	0.003(2)	0.001(2)

Table 3. Continued.

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(61A)	2*i*	0.67(1)	0.8355(4)	0.9447(4)	0.1624(3)	0.040(2)	0.037(2)	0.050(2)	0.004(2)	0.003(2)	0.010(2)
N(62A)	2*i*	0.67	0.8166(5)	0.9686(4)	0.0863(3)	0.061(3)	0.045(2)	0.054(2)	–0.011(2)	–0.006(2)	0.015(2)
N(63A)	2*i*	0.67	0.7644(5)	0.8498(5)	0.1811(3)	0.059(3)	0.054(2)	0.063(3)	–0.013(2)	–0.011(2)	0.028(2)
N(64A)	2*i*	0.67	0.9241(5)	1.0168(5)	0.2192(3)	0.059(3)	0.053(2)	0.060(3)	–0.012(2)	–0.006(2)	0.017(2)
N(65A)	2*i*	0.67	0.929(1)	1.007(1)	0.3054(7)	0.13(1)	0.13(1)	0.094(8)	–0.078(8)	–0.046(7)	0.055(7)
C(61B)	2*i*	0.33	0.8355(4)	0.9447(4)	0.1624(3)	0.040(2)	0.037(2)	0.050(2)	0.004(2)	0.003(2)	0.010(2)
N(62B)	2*i*	0.33	0.8166(5)	0.9686(4)	0.0863(3)	0.061(3)	0.045(2)	0.054(2)	–0.011(2)	–0.006(2)	0.015(2)
N(63B)	2*i*	0.33	0.9241(5)	1.0168(5)	0.2192(3)	0.059(3)	0.053(2)	0.060(3)	–0.012(2)	–0.006(2)	0.017(2)
N(64B)	2*i*	0.33	0.7644(5)	0.8498(5)	0.1811(3)	0.059(3)	0.054(2)	0.063(3)	–0.013(2)	–0.011(2)	0.028(2)
N(65B)	2*i*	0.33	0.784(2)	0.791(2)	0.246(1)	0.07(1)	0.067(9)	0.08(1)	–0.012(7)	–0.005(8)	0.054(9)
O(51)	2*i*	0.67	0.7662(9)	0.720(1)	0.3259(7)	0.074(5)	0.154(9)	0.122(8)	–0.012(5)	–0.019(5)	0.083(7)
O(52)	2*i*		0.393(2)	0.8845(9)	0.472(1)	0.52(3)	0.082(6)	0.29(2)	0.10(1)	–0.22(2)	–0.013(7)
O(53)	2*i*		0.116(2)	0.908(2)	0.387(1)	0.54(3)	0.34(2)	0.34(2)	–0.27(2)	–0.02(2)	0.18(2)
O(54)	2*i*		0.706(4)	0.946(3)	0.436(1)	1.03(7)	0.61(5)	0.27(2)	0.44(4)	0.31(3)	0.21(3)

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