Crystal structure of diaqua(hydrogen 5-isonicotinamidoisophthalato)-(μ_2-5-isonicotinamidoisophthalato-κ^2O^1,O^3)praseodymium(III) monohydrate, $Pr(H_2O)_2(C_{14}H_9N_2O_5)(C_{14}H_8O_5N_2) \cdot H_2O$

Dai-Zhi Kuang*, Yong-Lan Feng, Jin-Sheng Xu and Zhi-Jian Zhang

Hengyang Normal University, Key Laboratory of Functional Organometallic Materials, Department of Chemistry and Materials Science, Hengyang 421088, Hunan, P. R. China

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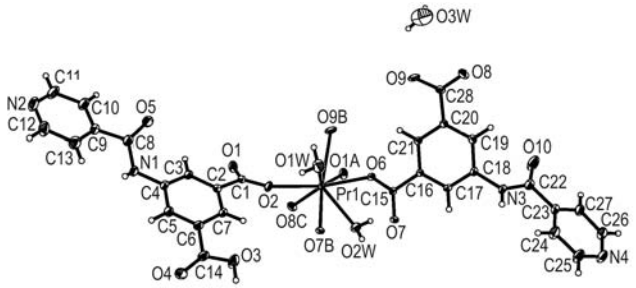

Abstract

$C_{28}H_{23}N_4O_{13}Pr$, triclinic, $P\bar{1}$ (no. 2), a = 9.6645(8) Å, b = 10.6237(8) Å, c = 15.860(1) Å, α = 81.453(1)°, β = 78.763(2)°, γ = 64.177(1)°, V = 1433.9 Å^3, Z = 2, $R_{gt}(F)$ = 0.034, $wR_{ref}(F^2)$ = 0.088, T = 293 K.

Source of material

A mixture of $Pr(NO_3)_3 \cdot 6H_2O$ (0.031 g, 0.07 mmol), 5-isonicotinamidoisophthalic acid (H_2L, 0.028 g, 0.1 mmol), NaOH (0.061 g, 0.15 mmol), CH_3CH_2OH (4 mL) and H_2O (6 mL) was sealed in a 15 ml Teflon-lined stainless steel reactor, which was heated at 433 K for 72 h and then cooled to room temperature. Green needle-like crystals of the title compound were collected.

Experimental details

All the hydrogen atoms except for those of water molecules and one carboxylate group of HL^- were generated geometrically and refined isotropically using the riding model, while those ones of water molecules and carboxylate group were found directly. The water H atoms found from difference Fourier maps were refined with restraints for d(O—H) = 0.85 Å with U_{iso}(H) = 1.2 U_{eq}(O).

Discussion

Recently, a great deal of interest in transition metal complex assemblies has been devoted to the development of rational synthetic routes to novel one-, two- and three-dimensional crystal frameworks, due to their potential applications in many areas [1]. However, the design and control over high-dimensional lanthanide frameworks are rarely reported [2,3]. On the other hand, it is well known that carboxylate and pyridine groups have good coordination capacities as well as the amide group, a fascinating functional group with two different types of hydrogen bonding sites: the –NH moiety which acts as an electron acceptor while the –C=O group acts as an electron donor [4-6].

In the title complex, the Pr(III) ion is eight-coordinated by two O atoms from two water molecules, two carboxylate O atoms from two HL^- ligands and four other O atoms from four different L^{2-} ligands, which forming a distorted bi-capped trigonal prismatic geometry. Furthermore, the HL^- anions adopt μ_2-η^1:η^1 bridging coordination mode, while two carboxylate groups of each L^{2-} ligand have different coordination modes, one is μ_2-η^1:η^1 bridging and the other one acts as μ_2-η^2:η^1-bridging coordination mode, whereas the pyridyl group is not participating in coordination. By such a coordination the complexes are interlinked into an infinite 2D network. The uncoordinated pyridyl group's nitrogen atoms and carboxylate group's oxygen atoms provide the hydrogen-bonding donor and acceptor, respectively, resulting in a 3D architecture through N–H···O and O–H···O hydrogen bonding interactions.

Table 1. Data collection and handling.

Crystal:	green block, size 0.10 × 0.14 × 0.20 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	17.77 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	50.04°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7189, 4972
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 4671
$N(param)_{refined}$:	416
Programs:	SHELXS-97, SHELXL-97 [7]

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

Atom	Site	x	y	z	U_{iso}
H(23)	2*i*	0.3666	1.1315	0.1904	0.032
H(21)	2*i*	0.5528	1.3563	0.0216	0.040
H(18)	2*i*	0.7081	1.2067	0.2481	0.034
H(26)	2*i*	0.1281	1.0589	–0.0214	0.055
H(27)	2*i*	0.0449	1.0720	–0.1503	0.067
H(28)	2*i*	0.2965	1.2617	–0.2686	0.064
H(29)	2*i*	0.3870	1.2595	–0.1446	0.057
H(8)	2*i*	1.3652	0.7203	0.6182	0.028
H(1)	2*i*	1.5635	0.3030	0.6114	0.030
H(5)	2*i*	1.1487	0.5298	0.5301	0.025
H(2)	2*i*	1.8404	0.5779	0.6485	0.049
H(14)	2*i*	2.0007	0.6359	0.7082	0.056
H(13)	2*i*	1.9237	0.4468	0.9280	0.047
H(12)	2*i*	1.7579	0.3852	0.8774	0.044
H(2A)	2*i*	0.4225	1.2573	–0.0216	0.041
H(3A)	2*i*	1.6554	0.5602	0.6263	0.037
H(3)	2*i*	0.8930	1.3834	0.1547	0.110

* Correspondence author (e-mail: hnkdz@yahoo.com.cn)

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(1WB)	2*i*	0.8904	0.7774	0.3686	0.061
H(1WA)	2*i*	0.9091	0.8880	0.3281	0.061
H(2WB)	2*i*	0.8768	1.0855	0.5974	0.041

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(2WA)	2*i*	0.8080	1.0190	0.6634	0.041
H(3WA)	2*i*	0.7408	0.1507	0.7684	0.163
H(3WB)	2*i*	0.7467	0.0260	0.8254	0.163

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

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pr(1)	2*i*	0.73167(2)	0.96089(2)	0.50337(1)	0.0192(1)	0.0151(1)	0.0237(1)	–0.00824(9)	–0.00771(8)	0.00068(8)
C(1)	2*i*	0.5111(4)	1.0992(4)	0.3252(2)	0.029(2)	0.021(2)	0.023(2)	–0.012(2)	–0.008(2)	0.001(1)
C(2)	2*i*	0.5318(4)	1.1619(4)	0.2339(2)	0.027(2)	0.023(2)	0.025(2)	–0.012(2)	–0.007(2)	0.000(1)
C(3)	2*i*	0.4387(5)	1.1701(4)	0.1752(2)	0.030(2)	0.031(2)	0.025(2)	–0.018(2)	–0.008(2)	0.001(2)
C(4)	2*i*	0.4533(5)	1.2361(4)	0.0935(2)	0.034(2)	0.031(2)	0.028(2)	–0.018(2)	–0.014(2)	0.001(2)
C(5)	2*i*	0.5502(5)	1.3045(4)	0.0742(3)	0.042(2)	0.038(2)	0.028(2)	–0.025(2)	–0.011(2)	0.007(2)
C(6)	2*i*	0.6428(5)	1.2960(4)	0.1325(2)	0.037(2)	0.035(2)	0.029(2)	–0.024(2)	–0.009(2)	0.002(2)
C(7)	2*i*	0.6388(5)	1.2194(4)	0.2109(2)	0.032(2)	0.031(2)	0.029(2)	–0.017(2)	–0.012(2)	0.001(2)
C(8)	2*i*	0.3251(5)	1.1434(5)	0.0155(3)	0.045(3)	0.043(3)	0.034(2)	–0.028(2)	–0.015(2)	0.004(2)
C(9)	2*i*	0.2671(5)	1.1588(4)	–0.0678(3)	0.044(2)	0.037(2)	0.029(2)	–0.021(2)	–0.014(2)	0.002(2)
C(10)	2*i*	0.1639(6)	1.1016(5)	–0.0708(3)	0.055(3)	0.057(3)	0.040(2)	–0.034(3)	–0.017(2)	0.000(2)
C(11)	2*i*	0.1152(7)	1.1094(6)	–0.1488(3)	0.059(3)	0.063(3)	0.058(3)	–0.026(3)	–0.026(3)	–0.016(3)
C(12)	2*i*	0.2622(7)	1.2209(6)	–0.2177(3)	0.067(4)	0.064(3)	0.030(2)	–0.025(3)	–0.015(2)	0.000(2)
C(13)	2*i*	0.3176(6)	1.2200(5)	–0.1437(3)	0.059(3)	0.054(3)	0.038(2)	–0.032(3)	–0.011(2)	0.001(2)
C(14)	2*i*	0.7432(5)	1.3752(5)	0.1127(3)	0.049(3)	0.050(3)	0.033(2)	–0.037(2)	–0.011(2)	0.004(2)
C(15)	2*i*	1.1065(4)	0.7884(4)	0.5569(2)	0.022(2)	0.021(2)	0.018(2)	–0.010(1)	–0.005(1)	–0.002(1)
C(16)	2*i*	1.2356(4)	0.6475(3)	0.5727(2)	0.019(2)	0.017(2)	0.024(2)	–0.009(1)	–0.007(1)	0.001(1)
C(17)	2*i*	1.3616(4)	0.6389(4)	0.6062(2)	0.026(2)	0.022(2)	0.029(2)	–0.015(2)	–0.008(2)	–0.001(1)
C(18)	2*i*	1.4815(4)	0.5109(4)	0.6219(2)	0.023(2)	0.026(2)	0.030(2)	–0.012(2)	–0.011(2)	0.001(2)
C(19)	2*i*	1.4818(4)	0.3892(4)	0.6011(2)	0.023(2)	0.020(2)	0.032(2)	–0.008(2)	–0.009(2)	0.001(2)
C(20)	2*i*	1.3581(4)	0.3972(4)	0.5643(2)	0.021(2)	0.020(2)	0.026(2)	–0.011(1)	–0.006(1)	0.001(1)
C(21)	2*i*	1.2334(4)	0.5254(4)	0.5520(2)	0.018(2)	0.019(2)	0.028(2)	–0.010(1)	–0.007(1)	–0.001(1)
C(22)	2*i*	1.6588(5)	0.4429(5)	0.7282(3)	0.038(2)	0.038(2)	0.044(2)	–0.022(2)	–0.019(2)	0.005(2)
C(23)	2*i*	1.7778(5)	0.4766(4)	0.7573(3)	0.028(2)	0.032(2)	0.040(2)	–0.014(2)	–0.015(2)	0.002(2)
C(24)	2*i*	1.8540(5)	0.5513(5)	0.7059(3)	0.042(3)	0.058(3)	0.035(2)	–0.030(2)	–0.013(2)	0.005(2)
C(25)	2*i*	1.9504(6)	0.5849(6)	0.7424(3)	0.044(3)	0.070(3)	0.045(3)	–0.040(3)	–0.015(2)	0.008(2)
C(26)	2*i*	1.9051(5)	0.4746(5)	0.8714(3)	0.038(2)	0.050(3)	0.034(2)	–0.022(2)	–0.012(2)	0.002(2)
C(27)	2*i*	1.8058(5)	0.4369(5)	0.8415(3)	0.036(2)	0.043(3)	0.040(2)	–0.023(2)	–0.016(2)	0.010(2)
C(28)	2*i*	1.3641(4)	0.2694(4)	0.5317(2)	0.026(2)	0.019(2)	0.032(2)	–0.012(2)	–0.003(2)	–0.001(1)
N(1)	2*i*	0.3671(4)	1.2432(4)	0.0285(2)	0.046(2)	0.042(2)	0.026(2)	–0.029(2)	–0.017(2)	0.007(1)
N(2)	2*i*	0.1635(5)	1.1671(5)	–0.2205(3)	0.065(3)	0.057(3)	0.040(2)	–0.013(2)	–0.026(2)	–0.012(2)
N(3)	2*i*	1.6083(4)	0.5116(3)	0.6555(2)	0.028(2)	0.030(2)	0.042(2)	–0.016(1)	–0.019(2)	0.005(1)
N(4)	2*i*	1.9758(4)	0.5487(4)	0.8236(2)	0.040(2)	0.062(3)	0.040(2)	–0.034(2)	–0.015(2)	0.003(2)
O(1)	2*i*	0.3837(3)	1.0937(3)	0.3524(2)	0.028(2)	0.044(2)	0.031(1)	–0.018(1)	–0.008(1)	0.011(1)
O(2)	2*i*	0.6200(3)	1.0636(3)	0.3681(2)	0.036(2)	0.043(2)	0.029(1)	–0.024(1)	–0.017(1)	0.005(1)
O(3)	2*i*	0.8520(5)	1.3287(5)	0.1604(3)	0.086(3)	0.099(3)	0.080(3)	–0.079(3)	–0.056(2)	0.047(2)
O(4)	2*i*	0.7212(4)	1.4717(4)	0.0589(2)	0.071(2)	0.058(2)	0.046(2)	–0.051(2)	–0.020(2)	0.021(2)
O(5)	2*i*	0.3354(5)	1.0418(4)	0.0661(2)	0.095(3)	0.057(2)	0.045(2)	–0.055(2)	–0.034(2)	0.018(2)
O(6)	2*i*	0.9821(3)	0.7910(3)	0.5428(2)	0.024(1)	0.020(1)	0.046(2)	–0.010(1)	–0.017(1)	–0.001(1)
O(7)	2*i*	1.1293(3)	0.8946(3)	0.5611(2)	0.029(1)	0.017(1)	0.044(2)	–0.014(1)	–0.016(1)	0.005(1)
O(8)	2*i*	1.4779(3)	0.1546(3)	0.5397(2)	0.040(2)	0.020(1)	0.041(2)	–0.005(1)	–0.011(1)	–0.003(1)
O(9)	2*i*	1.2552(4)	0.2827(3)	0.4946(3)	0.048(2)	0.027(2)	0.108(3)	–0.014(2)	–0.040(2)	–0.017(2)
O(10)	2*i*	1.6087(5)	0.3623(5)	0.7707(3)	0.086(3)	0.098(3)	0.072(2)	–0.075(3)	–0.050(2)	0.042(2)
O(1W)	2*i*	0.9236(4)	0.8370(3)	0.3750(2)	0.065(2)	0.045(2)	0.033(2)	–0.009(2)	–0.013(2)	–0.013(1)
O(2W)	2*i*	0.7982(3)	1.0672(3)	0.6154(2)	0.037(2)	0.039(2)	0.035(2)	–0.022(1)	–0.008(1)	–0.003(1)
O(3W)	2*i*	0.7920(9)	0.0677(7)	0.7885(3)	0.224(8)	0.150(6)	0.073(3)	–0.126(6)	–0.005(4)	0.008(4)

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