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Crystal structure of triaqua(μ_2-pyrazine-2,3-dicarboxylato)-(hydrogen μ_2-pyrazine-2,3-dicarboxylato)cerium(III), $Ce(H_2O)_3(C_6H_2O_4N_2)(C_6H_3O_4N_2)$

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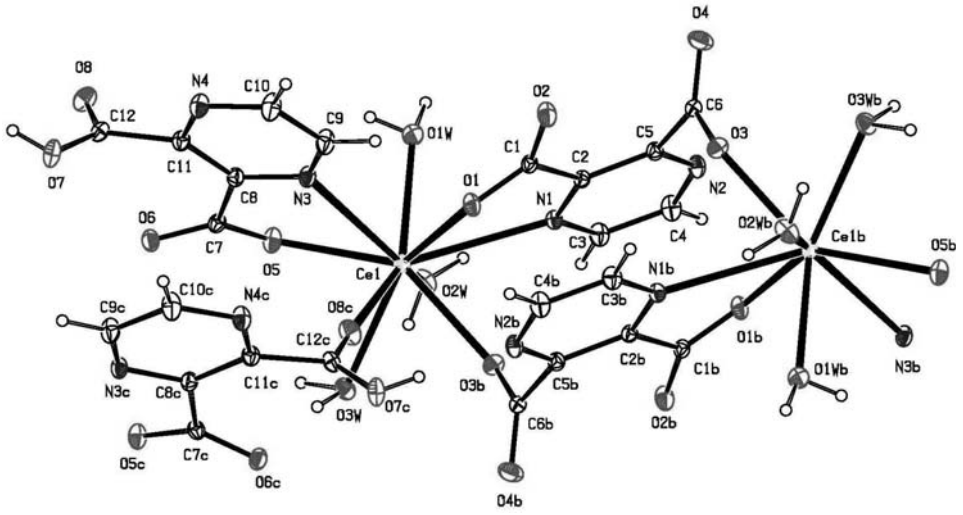

Abstract

$C_{12}H_{11}CeN_4O_{11}$, orthorhombic, *Pbca* (no. 61),
a = 7.9362(4) Å, b = 14.7422(6) Å, c = 27.840(2) Å,
V = 3257.2 Å^3, Z = 8, $R_{gt}(F)$ = 0.049, $wR_{ref}(F^2)$ = 0.118,
T = 298 K.

Source of material

A solution of pyrazine-2,3-dicarboxlic acid (0.51 g, 0.30 mmol) in H_2O (15 ml) was added to a solution of $CeCl_3 \cdot 7H_2O$ (0.56 g, 0.15 mmol) in water (15 ml) and the resulting pale yellow solution was stirred for 40 min at 50 °C. This solution was left to evaporate slowly at room temperature. After one week, yellow block-shaped crystals of the title compound were isolated (yield 0.57 g, 71.3 %).

Experimental details

H atoms of water molecules were located from difference Fourier maps and refined. The other hydrogen atoms were placed in calculated positions with d(C—H) in the range of 0.82 - 0.93 Å and U_{iso}(H) = 1.2 U_{eq}(C).

Discussion

Takusagawa & Shimada first determined the structure of pyrazine-2,3-dicarboxlic acid (pzdc) by single crystal X-ray analysis [1]. Almost at the same time, the first metal-organic compound of pyrazine-2,3-dicarboxylic acid was reported [2]. Among many reported compounds containing pyrazine-2,3-dicarboxylic acid, most are complexes of transition metal ions and main group metals including manganese [3], copper [4], iron [5], calcium [6] and strontium [7]. Also, there are many reported compounds of pyrazine-2,3-dicarboxylic acid with lanthanide(III) metals such as praseodymium, europium and lanthanum [8].

The asymmetric unit of the title compound consists of one cerium(III) ion, one mono- and one di-deprotonated 2,3-pyrazinedicarboxylic acid group and three coordinating water molecules. The Ce—OW, Ce—$O_{carboxylate}$ and Ce—N bond lengths are in the ranges 2.498(5) - 2.608(4) Å, 2.430(4) - 2.807(4) Å and 2.807(4) - 2.858(4) Å, respectively. The ligand pzdc binds as a bridging tri-coordinating ligand. Ce(III) ion is nine-coordinated and the coordination sphere is formed by two pzdc as a bidentate ligand (via N and O atoms), three water molecules and two pzdc as a bridging ligand (via O atoms). Each cerium ion is connected to the three adjacent Ce ions via bridging pzdc ligands to produce a two-dimensional polymer in (010). In the crystal packing, the structure consists of two different 32-membered and 12-membered metallocycle rings. The first one, 32-memebered metallocycle, consists of six cerium ions and six bridging pzdc ligands while the small 12-memebered ring is generated from two cerium ions and two bridging pzdc ligands.

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Table 1. Data collection and handling.

Crystal:	yellow block, size 0.10 × 0.14 × 0.20 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	28.69 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	58.36°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	33609, 4387
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3695
$N(param)_{refined}$:	279
Program:	SHELXTL [9]

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

Atom	Site	x	y	z	U_{iso}
H(3)	8*c*	0.1419	0.7638	0.4872	0.038
H(4)	8*c*	0.2615	0.7845	0.5614	0.042
H(9)	8*c*	0.1337	1.1313	0.3450	0.045
H(10)	8*c*	0.1796	1.2325	0.2837	0.051
H(1WA)	8*c*	0.36(1)	0.886(6)	0.343(3)	0.07(3)
H(1WB)	8*c*	0.43(1)	0.849(9)	0.378(5)	0.14(5)
H(2WA)	8*c*	0.01(1)	0.691(7)	0.381(3)	0.07(3)
H(2WB)	8*c*	0.17(1)	0.704(6)	0.412(3)	0.09(3)
H(3WA)	8*c*	–0.186(9)	0.736(5)	0.315(3)	0.05(2)
H(3WB)	8*c*	–0.29(1)	0.808(7)	0.326(4)	0.09(3)
H(7)	8*c*	0.1194	1.0725	0.1135	0.056

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}
C(1)	8*c*	0.1689(6)	1.0513(3)	0.4457(2)	0.031(2)	0.021(2)	0.019(2)	–0.005(2)	0.002(2)	–0.002(2)
C(2)	8*c*	0.1981(6)	0.9741(3)	0.4808(2)	0.024(2)	0.022(2)	0.019(2)	–0.000(2)	0.001(2)	–0.001(2)
C(3)	8*c*	0.1752(7)	0.8217(4)	0.4965(2)	0.043(3)	0.023(2)	0.030(3)	0.001(2)	–0.001(2)	–0.004(2)
C(4)	8*c*	0.2464(8)	0.8336(4)	0.5409(2)	0.050(3)	0.023(2)	0.031(3)	0.006(2)	–0.005(2)	0.001(2)
C(5)	8*c*	0.2708(6)	0.9890(4)	0.5258(2)	0.023(2)	0.029(3)	0.021(2)	0.003(2)	0.000(2)	–0.002(2)
C(6)	8*c*	0.3182(6)	1.0805(3)	0.5472(2)	0.022(2)	0.030(2)	0.021(2)	–0.001(2)	0.001(2)	–0.001(2)
C(7)	8*c*	0.0656(6)	0.8966(3)	0.2510(2)	0.030(2)	0.021(2)	0.023(2)	–0.001(2)	0.004(2)	0.001(2)
C(8)	8*c*	0.1107(6)	0.9955(3)	0.2591(2)	0.027(2)	0.027(2)	0.021(2)	0.001(2)	–0.001(2)	0.002(2)
C(9)	8*c*	0.1348(9)	1.1105(4)	0.3135(2)	0.062(4)	0.025(3)	0.026(3)	–0.011(2)	0.003(2)	–0.006(2)
C(10)	8*c*	0.1637(9)	1.1716(4)	0.2764(2)	0.075(4)	0.026(3)	0.028(3)	–0.009(3)	0.005(3)	–0.001(2)
C(11)	8*c*	0.1436(6)	1.0563(3)	0.2219(2)	0.033(2)	0.023(2)	0.024(2)	–0.001(2)	0.001(2)	0.001(2)
C(12)	8*c*	0.1647(6)	1.0311(3)	0.1696(2)	0.032(2)	0.022(2)	0.025(2)	0.001(2)	0.008(2)	–0.000(2)
N(1)	8*c*	0.1523(5)	0.8909(3)	0.4662(1)	0.028(2)	0.022(2)	0.021(2)	0.000(2)	–0.001(1)	–0.001(2)
N(2)	8*c*	0.2936(6)	0.9168(3)	0.5542(2)	0.036(2)	0.026(2)	0.022(2)	0.007(2)	–0.003(2)	–0.001(2)
N(3)	8*c*	0.1088(6)	1.0230(3)	0.3049(1)	0.040(2)	0.024(2)	0.020(2)	–0.005(2)	0.000(2)	0.002(2)
N(4)	8*c*	0.1691(7)	1.1452(3)	0.2307(2)	0.052(3)	0.020(2)	0.026(2)	–0.003(2)	0.003(2)	0.002(2)
O(1)	8*c*	0.0710(5)	1.0328(2)	0.4113(1)	0.043(2)	0.027(2)	0.022(2)	–0.001(2)	–0.009(2)	0.003(1)
O(1W)	8*c*	0.3294(6)	0.8841(4)	0.3713(2)	0.031(2)	0.072(3)	0.033(2)	–0.003(2)	0.002(2)	–0.005(2)
O(2)	8*c*	0.2405(6)	1.1238(3)	0.4534(1)	0.064(3)	0.027(2)	0.027(2)	–0.019(2)	–0.007(2)	0.002(2)
O(2W)	8*c*	0.0907(5)	0.7265(3)	0.3884(2)	0.043(2)	0.025(2)	0.045(2)	0.002(2)	–0.013(2)	–0.001(2)
O(3)	8*c*	0.1977(5)	1.1245(2)	0.5643(1)	0.033(2)	0.024(2)	0.032(2)	0.001(1)	0.006(1)	–0.003(1)
O(3W)	8*c*	–0.2202(5)	0.7775(3)	0.3431(1)	0.035(2)	0.031(2)	0.033(2)	0.001(2)	–0.004(2)	–0.008(2)
O(4)	8*c*	0.4686(5)	1.0999(3)	0.5477(2)	0.028(2)	0.051(3)	0.051(3)	–0.007(2)	0.000(2)	–0.019(2)
O(5)	8*c*	0.0715(6)	0.8468(2)	0.2877(1)	0.052(2)	0.022(2)	0.025(2)	0.003(2)	0.002(2)	0.001(1)
O(6)	8*c*	0.0262(5)	0.8725(3)	0.2096(1)	0.043(2)	0.032(2)	0.023(2)	–0.007(2)	–0.003(2)	–0.001(1)
O(7)	8*c*	0.0734(6)	1.0725(3)	0.1399(1)	0.054(3)	0.038(2)	0.021(2)	0.015(2)	0.005(2)	0.003(2)
O(8)	8*c*	0.2775(5)	0.9763(3)	0.1588(1)	0.041(2)	0.032(2)	0.039(2)	0.011(2)	0.011(2)	0.003(2)
Ce(1)	8*c*	0.01478(3)	0.88909(2)	0.371325(9)	0.0244(2)	0.0198(2)	0.0185(2)	–0.00013(9)	–0.00004(8)	–0.00001(9)

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