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Crystal structure of *catena*-poly[*diaqua*-bis(μ_2 -2-(4-(2,4,4-trimethylpentan-2-yl)phenoxy)propanoato- $\kappa^2 O:O'$)-(2-(4-(2,4,4-trimethylpentan-2-yl)phenoxy)propanoato- $\kappa^2 O,O'$)yttrium(III)], $C_{51}H_{79}O_{11}Y$

<https://doi.org/10.1515/ncrs-2020-0424>

Received August 3, 2020; published online December 10, 2020

Abstract

$C_{51}H_{79}O_{11}Y$, monoclinic, $P2_1/c$ (no. 14), $a = 8.8682$ (3) Å, $b = 22.1328$ (8) Å, $c = 27.9848$ (10) Å, $\beta = 96.710$ (1)°, $V = 5455.2$ (3) Å³, $Z = 4$, $R_{gt}(F) = 0.0495$, $wR_{ref}(F2) = 0.1275$, $T = 296$ (2) K.

CCDC no.: 1863719.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.20 × 0.20 × 0.20 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.12 mm ⁻¹
Diffractometer, scan mode:	Bruker D8 venture, φ and ω
θ_{max} , completeness:	27.6°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	59,868, 12,548, 0.082
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 7777
$N(param)_{refined}$:	586
Programs:	SHELX [1], Bruker [2]

Source of material

All reagents were purchased from commercial sources and used without further purification. The organic ligand (0.278 g, 1 mmol), sodium hydroxide solution (5 mol/L, 0.20 mL) and yttrium chloride solution (1 mol/L, 0.34 mL) were sequentially dissolved in methanol (20 mL). Then, the resulting white solid was filtered off and added in polar solution (CH₃OH:CHCl₃ = 7:3). The mixture was set aside to crystallize for several days, giving colorless crystals.

Experimental details

The intensity data were recorded on a Bruker D8 QUEST by Bruker APX3 program. The structure was solved and refined using the Bruker Software Package [1, 2]. All hydrogen atoms of the organic ligands were generated onto the specific atoms with fixed thermal factors. H atoms of coordinated water molecules were identified in difference Fourier syntheses and refined with fixed thermal factors to oxygen atoms.

Comment

The recovery of rare earths from a variety of resources has drawn great attention due to the increasing global demand for rare earths [3, 4]. Various extractants have been developed for extraction and separation of rare earths, among organic acid proved to be efficient extractants. In our present project [5, 6], the *para*-(*tert*-octylphenoxy)*iso*-propanoic acid was used for the extraction of yttrium ions.

In this title compound, the Y(III) is nine-coordinated [7] by seven oxygen atoms from five carboxylate oxygen atoms and two oxygen atoms from coordination water molecules. The carboxyl extraction sites coordinate with Y(III). The organic ligand has three coordination modes, including monodentate-, bidentate-, and tridentate-fashion. The Y(III) ions were bridged by two of the three crystallographically independent ligands into one forming a chain structure in which the external edge was hydrophobic. The third independent ligand coordinates in a κ^2O,O' fashion. The work presented may shed some light on better understanding the structure of extracted complex and extraction mechanism.

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	U_{iso}^*/U_{eq}
Y001	0.23446 (3)	0.50454 (2)	0.50583 (2)	0.02671 (8)
O1	−0.0257 (2)	0.29041 (9)	0.45709 (7)	0.0387 (5)
O2	−0.17744 (19)	0.38963 (8)	0.48446 (7)	0.0374 (5)
O3	0.03320 (18)	0.44007 (8)	0.50050 (7)	0.0321 (5)
O4	0.4103 (3)	0.57655 (17)	0.35570 (10)	0.0938 (11)
O5	0.1306 (2)	0.52538 (10)	0.42170 (7)	0.0425 (5)
O6	0.3548 (2)	0.56283 (9)	0.44614 (7)	0.0370 (5)
O7	0.6267 (2)	0.67734 (9)	0.58811 (8)	0.0420 (5)
O8	0.6744 (2)	0.57140 (9)	0.54105 (7)	0.0388 (5)
O9	0.4407 (2)	0.54168 (9)	0.54869 (8)	0.0400 (5)
O10	0.3393 (2)	0.42279 (9)	0.55831 (7)	0.0414 (5)
H10A	0.332539	0.430773	0.587687	0.062*
H10B	0.434539	0.419883	0.557537	0.062*
O11	0.1579 (2)	0.51123 (9)	0.58727 (7)	0.0385 (5)
H11B	0.066341	0.499696	0.586853	0.058*
H11C	0.152041	0.545096	0.601253	0.058*
C1	1.0372 (5)	0.6239 (2)	0.1877 (2)	0.128 (2)
H1A	1.040680	0.642792	0.156913	0.193*
H1B	1.125664	0.599147	0.195148	0.193*
H1C	1.034695	0.654449	0.211914	0.193*
C2	0.8879 (6)	0.5598 (3)	0.2364 (2)	0.131 (2)
H2A	0.881905	0.592780	0.258392	0.196*
H2B	0.977798	0.536627	0.245976	0.196*
H2C	0.800102	0.534503	0.236629	0.196*
C3	0.8940 (5)	0.58448 (17)	0.18602 (17)	0.0729 (12)
C4	0.9159 (7)	0.5317 (3)	0.1531 (3)	0.159 (3)
H4A	0.834919	0.503220	0.154426	0.238*
H4B	1.011241	0.512432	0.163313	0.238*
H4C	0.915588	0.546038	0.120715	0.238*
C5	0.4953 (5)	0.6607 (2)	0.14619 (15)	0.0845 (14)
H5A	0.389438	0.653720	0.148234	0.127*
H5B	0.511432	0.665873	0.113108	0.127*
H5C	0.527310	0.696451	0.163978	0.127*
C6	0.7584 (4)	0.62511 (16)	0.16896 (14)	0.0646 (11)
H6A	0.772062	0.637486	0.136491	0.078*
H6B	0.770261	0.661267	0.188626	0.078*
C7	0.5510 (6)	0.5525 (2)	0.13405 (15)	0.0872 (14)
H7A	0.596570	0.516586	0.148587	0.131*
H7B	0.590109	0.559776	0.103995	0.131*
H7C	0.442931	0.547352	0.128478	0.131*
C8	0.5884 (4)	0.60604 (16)	0.16751 (13)	0.0575 (10)
C9	0.5408 (4)	0.59501 (15)	0.21758 (12)	0.0499 (9)
C10	0.4648 (4)	0.54397 (16)	0.22940 (13)	0.0559 (9)
H10	0.445453	0.513334	0.206768	0.067*
C11	0.5636 (6)	0.63961 (19)	0.25248 (15)	0.0843 (15)
H11A	0.611213	0.675171	0.244833	0.101*
C12	0.5198 (6)	0.6341 (2)	0.29756 (16)	0.0919 (16)
H12	0.540423	0.664558	0.320273	0.110*
C13	0.4165 (4)	0.53742 (19)	0.27436 (14)	0.0652 (11)
H13	0.365052	0.502682	0.281773	0.078*
C14	0.4453 (4)	0.5829 (2)	0.30842 (14)	0.0681 (12)
C15	0.2592 (4)	0.57431 (19)	0.36200 (13)	0.0628 (11)

Table 2: (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
H15	0.214289	0.542142	0.340861	0.075*
C16	0.1623 (7)	0.6288 (2)	0.35131 (18)	0.119 (2)
H16A	0.060509	0.620071	0.357686	0.178*
H16B	0.161965	0.639611	0.318087	0.178*
H16C	0.202069	0.661689	0.371282	0.178*
C17	0.2484 (3)	0.55340 (14)	0.41326 (11)	0.0386 (7)
C18	−0.0368 (3)	0.39034 (12)	0.49090 (10)	0.0290 (6)
C19	0.0548 (3)	0.33308 (12)	0.48858 (11)	0.0351 (7)
H19	0.152829	0.342338	0.477361	0.042*
C20	0.0797 (4)	0.30347 (14)	0.53763 (12)	0.0480 (8)
H20A	−0.016654	0.293937	0.548199	0.072*
H20B	0.134214	0.330646	0.560164	0.072*
H20C	0.137455	0.267048	0.535645	0.072*
C21	−0.0004 (3)	0.35656 (13)	0.38804 (12)	0.0401 (7)
H21	0.065821	0.382718	0.406068	0.048*
C22	−0.0585 (3)	0.30621 (13)	0.40966 (11)	0.0363 (7)
C23	−0.1566 (4)	0.26852 (14)	0.38163 (12)	0.0459 (8)
H23	−0.198768	0.235418	0.395654	0.055*
C24	−0.1926 (4)	0.27932 (15)	0.33332 (13)	0.0509 (9)
H24	−0.257336	0.252582	0.315373	0.061*
C25	−0.1363 (4)	0.32852 (13)	0.31011 (12)	0.0420 (8)
C26	−0.0418 (3)	0.36729 (13)	0.33963 (12)	0.0412 (8)
H26	−0.005160	0.401872	0.326032	0.049*
C27	−0.2545 (5)	0.2867 (2)	0.23012 (14)	0.0811 (13)
H27A	−0.192161	0.251630	0.237235	0.122*
H27B	−0.265283	0.294175	0.196104	0.122*
H27C	−0.352783	0.279929	0.240328	0.122*
C28	−0.1797 (4)	0.34169 (16)	0.25688 (13)	0.0527 (9)
C29	−0.2976 (5)	0.3932 (2)	0.25291 (15)	0.0792 (13)
H29A	−0.384033	0.381200	0.268352	0.119*
H29B	−0.329267	0.401678	0.219599	0.119*
H29C	−0.253064	0.428762	0.268290	0.119*
C30	−0.0454 (4)	0.36547 (16)	0.23106 (13)	0.0565 (9)
H30A	−0.089676	0.380238	0.199899	0.068*
H30B	−0.005644	0.400557	0.249102	0.068*
C31	0.0571 (7)	0.2830 (3)	0.17917 (18)	0.120 (2)
H31A	−0.010232	0.251975	0.187915	0.180*
H31B	0.149577	0.264898	0.171382	0.180*
H31C	0.009626	0.304656	0.151730	0.180*
C32	0.2144 (5)	0.3709 (2)	0.20844 (19)	0.1057 (18)
H32A	0.175603	0.393251	0.180252	0.158*
H32B	0.302792	0.348669	0.202064	0.158*
H32C	0.240953	0.398369	0.234621	0.158*
C33	0.0931 (5)	0.32705 (18)	0.22176 (14)	0.0656 (11)
C34	0.1614 (5)	0.29169 (19)	0.26589 (16)	0.0811 (13)
H34A	0.184528	0.318909	0.292441	0.122*
H34B	0.252693	0.271954	0.258926	0.122*
H34C	0.089804	0.261969	0.274053	0.122*
C35	0.5497 (3)	0.57709 (12)	0.55716 (10)	0.0288 (6)
C36	0.5299 (3)	0.62709 (13)	0.59286 (11)	0.0401 (7)
H36	0.423996	0.640676	0.588739	0.048*
C37	0.5688 (5)	0.60285 (17)	0.64324 (12)	0.0633 (11)
H37A	0.674449	0.592261	0.648167	0.095*
H37B	0.508432	0.567666	0.647468	0.095*
H37C	0.548227	0.633200	0.666113	0.095*
C38	0.5873 (3)	0.71613 (13)	0.55017 (11)	0.0354 (7)

Table 2: (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
C39	0.6733 (3)	0.76780 (13)	0.54922 (12)	0.0382 (7)
H39	0.755890	0.773648	0.572467	0.046*
C40	0.4697 (3)	0.70608 (14)	0.51413 (13)	0.0461 (8)
H40	0.412565	0.670813	0.513644	0.055*
C41	0.4388 (4)	0.74930 (16)	0.47892 (14)	0.0556 (9)
H41	0.361343	0.741697	0.454276	0.067*
C42	0.6384 (4)	0.81070 (14)	0.51435 (12)	0.0447 (8)
H42	0.697259	0.845523	0.514723	0.054*
C43	0.5169 (4)	0.80351 (15)	0.47825 (13)	0.0496 (8)
C44	0.5072 (6)	0.8210 (3)	0.39074 (17)	0.119 (2)
H44A	0.612850	0.810774	0.392390	0.179*
H44B	0.481449	0.849060	0.364918	0.179*
H44C	0.447027	0.785074	0.385087	0.179*
C45	0.4748 (5)	0.85042 (19)	0.43933 (15)	0.0713 (12)
C46	0.5730 (6)	0.9070 (2)	0.4459 (2)	0.120 (2)
H46A	0.567072	0.923638	0.477263	0.180*
H46B	0.537328	0.936252	0.421840	0.180*
H46C	0.676485	0.896736	0.442589	0.180*
C47	0.3037 (5)	0.8650 (2)	0.43260 (17)	0.0808 (14)
H47A	0.251508	0.827339	0.423920	0.097*
H47B	0.286264	0.891145	0.404694	0.097*
C48	0.2292 (5)	0.8576 (2)	0.51900 (17)	0.1002 (17)
H48A	0.199829	0.816628	0.511430	0.150*
H48B	0.162489	0.874520	0.540108	0.150*
H48C	0.331620	0.858276	0.534552	0.150*
C49	0.2194 (5)	0.89456 (18)	0.47288 (18)	0.0737 (12)
C50	0.0508 (6)	0.8960 (2)	0.4516 (2)	0.119 (2)
H50A	0.038704	0.922503	0.424239	0.179*
H50B	−0.009796	0.910377	0.475482	0.179*
H50C	0.018642	0.856018	0.441755	0.179*
C51	0.2654 (7)	0.9592 (2)	0.4843 (2)	0.144 (3)
H51A	0.365922	0.959958	0.501314	0.216*
H51B	0.195531	0.977015	0.503970	0.216*
H51C	0.264069	0.981699	0.454941	0.216*

Author contribution: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: This work was supported by Science and Technology Plan of Fujian Province, China (Grant No. 2017T3002).

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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