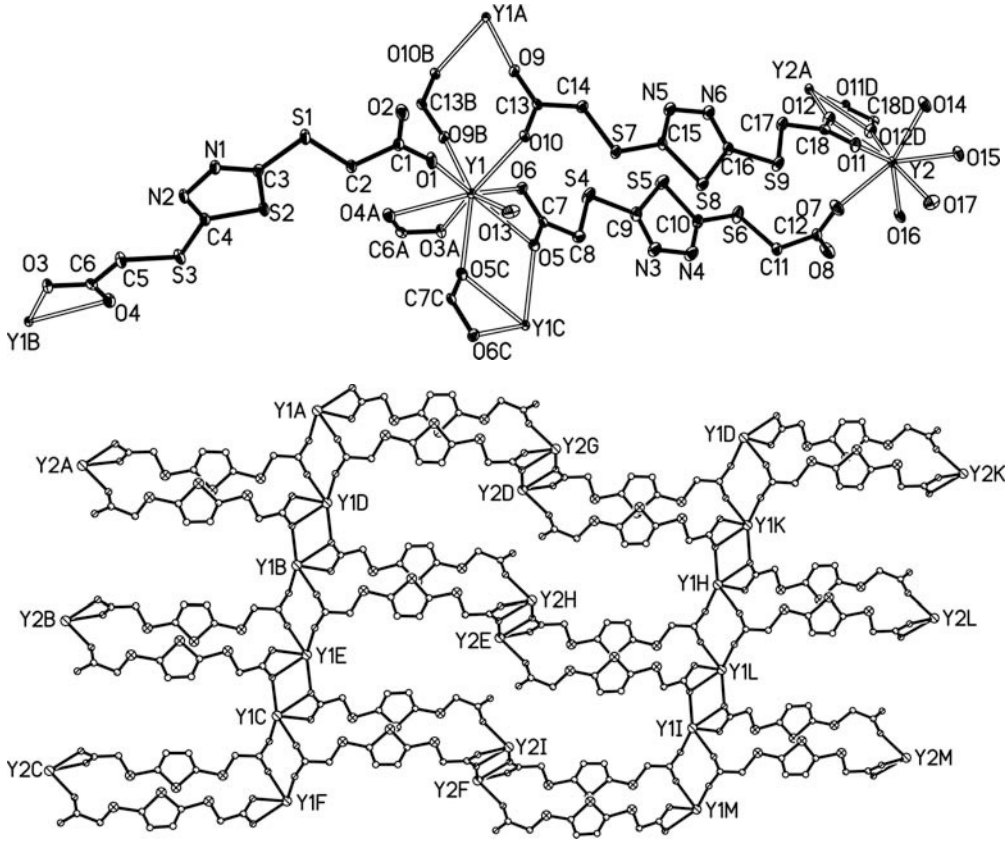
Crystal structure of pentaaqua-tris[(1,3,4-thiadiazole-2,5-diyldithio)diacetato]diyttrium(III), $Y_2(H_2O)_5(C_6H_{12}NO_4S_3)_3$

Yu-Ting Wang*,[I], Hui-Ling Li[I] and Jian-Ge Wang[II]

[I] Henan Institute of Education, Department of Chemistry, Zhengzhou, 450046 Henan, P. R. China
[II] Luoyang Normal University, College of Chemistry and Chemical Engineering, Luoyang, 471022 Henan, P. R. China

Received September 12, 2009, accepted and available on-line September 26, 2009; CCDC no. 1267/2763



Abstract

$C_{18}H_{22}N_6O_{17}S_9Y_2$, triclinic, $P\bar{1}$ (no. 2), a = 9.5713(9) Å, b = 13.700(1) Å, c = 14.932(1) Å, α = 64.646(1)°, β = 82.282(1)°, γ = 81.089(1)°, V = 1742.9 Å^3, Z = 2, $R_{gt}(F)$ = 0.024, $wR_{ref}(F^2)$ = 0.057, T = 291 K.

Source of material

The title compound was prepared by hydrothermal method. A mixture of (1,3,4-thiadiazole-2,5-diyldithio)diacetic acid (H_2tzda, 0.6 mmol), NaOH (1.2 mmol), $Y(NO_3)_3 \cdot 6H_2O$ (0.4 mmol), and water (15 mL) was sealed in a 25 mL Teflon-lined stainless steel vessel and then heated at 120 °C for 96 h. After being cooled slowly to room temperature, the title complex as colorless needle-like crystals was collected by filtration, washed with water, and dried in air.

Discussion

In past decades, the design and synthesis of metal-organic frameworks has been a field of rapid growth in supramolecular and material chemistry due to their fascinating topologies and useful properties [1-5]. An effective approach in building such materials is to employ suitable organic bridging ligands [6,7]. Carboxylate-containing ligands have attract considerable interest in reported multidentate ligands because of their diverse coordination modes [8-12]. Furthermore, it is found flexible ligands may induce novel topologies owing to their flexibility and conformation freedoms [13].

The title crystal structure exhibits an infinite three-dimensional network. In each independent crystallographic unit, there are two Y(III) ions, three $tzda^{2-}$ anions and five aqua ligands (figure, top). Y1(III) ion is surrounded by nine oxygen atoms, eight of them are from carboxylate groups of six $tzda^{2-}$ anions, while the last oxygen atom is from a water molecule. The local environment of the

* Correspondence author (e-mail: yutingwangyt@163.com)

Y1(III) ion can be best described as a strongly distorted tricapped trigonal prism. Y2(III) ion is coordinated by four oxygen atoms from three $tzda^{2-}$ anions and four coordinated water molecules in a distorted square-antiprismatic manner (figure, top). The bond lengths of Y—O range from 2.279(2) to 2.483(2) Å, comparable with those in the polymer $\{Y_2(tzda)_3(H_2O)_{10}] \cdot 5H_2O\}_n$ [14]. Noticeably, three $tzda^{2-}$ anions present three kinds of coordination modes linking the Y(III) ions: (a) one carboxylate group adopts a monodentate mode, and the other one adopts a bidentate chelating mode (μ_2-η^1:η^1:η^1mode); (b) one carboxylate group adopts a *syn-syn* bidentate bridging mode, and one adopts a chelating-bridging mode (μ_4-η^1:η^1:η^1:η^2 mode); (c) one carboxylate group adopts a monodentate mode, while another adopts a chelating-bridging mode (μ_3-η^1:η^1:η^2 mode). Two types of $tzda^{2-}$ anion ligands in μ_4-η^1:η^1:η^1:η^2 and μ_3-η^1:η^1:η^2 coordination modes link Y(III) centers to form a 2D network consisting of the $[Y1_2Y2_2(tzda)_4]$ repeating unit (figure, bottom), in which the distance between two neighboring Y1 ions is 5.713 Å and the distance between two adjacent Y1 and Y2 ions is about 14.755 Å. Within this 2D network, the repeating units $[Y1_2Y2_2(tzda)_4]$ are connected by carboxylate groups of $tzda^{2-}$ ligands in chelating-bridging mode forming an infinite wave-like chain, and in this chain the two nearest Y2 ions form dimeric centers are separated by 4.029 Å. Among two adjacent chains, two Y1 ions are connected also through chelating-bridging carboxylate groups with a distance of 4.250 Å. Then these two-dimensional networks are further linked by $tzda^{2-}$ ligands *via* μ_2-η^1:η^1:η^1 coordination mode along [010], leading to the final three-dimensional network. The crystal structure is stabilized through extensive hydrogen bonding involving the $tzda^{2-}$ ligands and coordinated water molecules.

Table 1. Data collection and handling.

Crystal:	colorless needle, size 0.18 × 0.33 × 0.45 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	39.34 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	13372, 6448
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 5719
$N(param)_{refined}$:	470
Programs:	SHELXS-97 [15], SHELXL-97 [16], SHELXTL [17]

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

Atom	Site	x	y	z	U_{iso}
H(1W)	2*i*	0.5744	–0.1835	1.0002	0.050
H(2W)	2*i*	0.6853	–0.2492	1.0512	0.050
H(3W)	2*i*	1.0707	0.3153	–0.1273	0.051
H(4W)	2*i*	1.0492	0.2531	–0.0273	0.051
H(5W)	2*i*	0.8511	0.5316	–0.2632	0.045
H(6W)	2*i*	0.9416	0.4379	–0.2387	0.045
H(7W)	2*i*	0.6780	0.6251	–0.1733	0.044
H(8W)	2*i*	0.6424	0.5930	–0.0735	0.044
H(9W)	2*i*	0.6280	0.3375	–0.0465	0.054
H(10W)	2*i*	0.7403	0.2617	–0.0438	0.054
H(2A)	2*i*	0.6725	–0.2404	1.3552	0.027
H(2B)	2*i*	0.7855	–0.1608	1.3372	0.027
H(5A)	2*i*	0.4303	–0.2298	1.8157	0.032
H(5B)	2*i*	0.5780	–0.1964	1.8209	0.032
H(8A)	2*i*	0.4998	0.2475	0.7581	0.031
H(8B)	2*i*	0.5651	0.1626	0.7148	0.031
H(11A)	2*i*	0.5599	0.4012	0.2458	0.036
H(11B)	2*i*	0.4653	0.5136	0.2180	0.036
H(14A)	2*i*	1.0824	–0.0147	0.7582	0.030
H(14B)	2*i*	0.9920	0.0883	0.7644	0.030
H(17A)	2*i*	0.9686	0.3418	0.2751	0.031
H(17B)	2*i*	1.0659	0.2387	0.2732	0.031

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}
Y(1)	2*i*	0.70726(2)	–0.03349(2)	1.04961(2)	0.0149(1)	0.0174(1)	0.0107(1)	–0.00129(9)	–0.00051(9)	–0.00461(9)
Y(2)	2*i*	0.86338(2)	0.43664(2)	–0.04651(2)	0.0209(1)	0.0158(1)	0.0104(1)	–0.00139(9)	–0.00135(9)	–0.00319(9)
S(1)	2*i*	0.85320(7)	–0.32989(5)	1.45946(5)	0.0361(4)	0.0268(3)	0.0142(3)	0.0131(3)	–0.0058(3)	–0.0093(3)
S(2)	2*i*	0.64601(7)	–0.16081(5)	1.50532(4)	0.0319(4)	0.0212(3)	0.0113(3)	0.0088(3)	–0.0018(3)	–0.0044(3)
S(3)	2*i*	0.49825(7)	–0.09433(5)	1.66343(5)	0.0351(4)	0.0207(3)	0.0157(3)	0.0057(3)	0.0043(3)	–0.0047(3)
S(4)	2*i*	0.72848(8)	0.28458(6)	0.68647(5)	0.0424(4)	0.0423(4)	0.0169(3)	–0.0224(3)	–0.0086(3)	0.0036(3)
S(5)	2*i*	0.78641(8)	0.41738(6)	0.46896(5)	0.0357(4)	0.0461(4)	0.0198(4)	–0.0198(3)	–0.0045(3)	0.0007(3)
S(6)	2*i*	0.68312(9)	0.52396(6)	0.26574(5)	0.0554(5)	0.0435(4)	0.0151(3)	–0.0269(4)	–0.0024(3)	–0.0032(3)
S(7)	2*i*	0.84503(7)	–0.01953(5)	0.74385(4)	0.0255(3)	0.0328(4)	0.0114(3)	–0.0107(3)	–0.0005(2)	–0.0049(3)
S(8)	2*i*	0.75177(7)	0.08065(6)	0.53457(5)	0.0248(3)	0.0413(4)	0.0136(3)	–0.0125(3)	–0.0012(3)	–0.0031(3)
S(9)	2*i*	0.83109(7)	0.19963(6)	0.31699(5)	0.0333(4)	0.0421(4)	0.0123(3)	–0.0185(3)	–0.0034(3)	–0.0011(3)
O(1)	2*i*	0.7974(2)	–0.1875(2)	1.1752(1)	0.044(1)	0.031(1)	0.0132(9)	0.0070(9)	–0.0041(8)	–0.0074(8)
O(2)	2*i*	0.9283(2)	–0.3346(2)	1.2709(1)	0.041(1)	0.035(1)	0.026(1)	0.0156(9)	–0.0048(9)	–0.0168(9)
O(3)	2*i*	0.4034(2)	–0.1309(1)	1.9374(1)	0.030(1)	0.0224(9)	0.0155(9)	–0.0041(7)	0.0013(7)	–0.0092(7)
O(4)	2*i*	0.3615(2)	0.0013(1)	1.7905(1)	0.031(1)	0.024(1)	0.022(1)	0.0049(8)	–0.0013(8)	–0.0097(8)
O(5)	2*i*	0.5482(2)	0.0486(1)	0.9061(1)	0.0222(9)	0.0217(9)	0.0152(9)	–0.0054(7)	0.0021(7)	–0.0041(7)
O(6)	2*i*	0.7392(2)	0.1286(1)	0.8857(1)	0.0231(9)	0.029(1)	0.0157(9)	–0.0062(7)	–0.0026(7)	–0.0049(8)
O(7)	2*i*	0.7045(2)	0.4498(2)	0.0825(1)	0.047(1)	0.048(1)	0.019(1)	0.018(1)	0.0000(9)	–0.008(1)
O(8)	2*i*	0.5636(2)	0.6043(2)	0.0407(2)	0.051(1)	0.033(1)	0.030(1)	0.005(1)	0.008(1)	–0.009(1)
O(9)	2*i*	1.1036(2)	–0.0410(2)	0.9353(1)	0.0208(9)	0.042(1)	0.0200(9)	–0.0076(8)	–0.0028(7)	–0.0158(9)
O(10)	2*i*	0.8864(2)	–0.0882(1)	0.9498(1)	0.0191(9)	0.0261(9)	0.0139(9)	–0.0050(7)	0.0034(7)	–0.0077(7)
O(11)	2*i*	0.9046(2)	0.2752(1)	0.1105(1)	0.037(1)	0.0208(9)	0.0161(9)	–0.0080(8)	–0.0035(8)	–0.0045(8)
O(12)	2*i*	1.0103(2)	0.4157(1)	0.0869(1)	0.035(1)	0.0210(9)	0.0172(9)	–0.0114(8)	–0.0029(8)	0.0004(8)
O(13)	2*i*	0.6403(2)	–0.1877(2)	1.0338(2)	0.025(1)	0.027(1)	0.051(1)	0.0007(8)	–0.0106(9)	–0.018(1)
O(14)	2*i*	1.0496(2)	0.3159(2)	–0.0717(1)	0.046(1)	0.028(1)	0.017(1)	0.0111(9)	0.0013(9)	–0.0063(8)
O(15)	2*i*	0.8986(2)	0.4837(1)	–0.2187(1)	0.044(1)	0.024(1)	0.0166(9)	0.0114(8)	–0.0046(8)	–0.0076(8)
O(16)	2*i*	0.6865(2)	0.5753(1)	–0.1172(1)	0.037(1)	0.030(1)	0.0119(9)	0.0110(8)	–0.0002(8)	–0.0046(8)

Table 3. Continued.

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(17)	2*i*	0.7155(2)	0.3267(2)	–0.0580(2)	0.037(1)	0.027(1)	0.046(1)	–0.0090(9)	–0.0075(9)	–0.012(1)
N(1)	2*i*	0.7585(2)	–0.3358(2)	1.6385(2)	0.028(1)	0.023(1)	0.014(1)	0.0052(9)	–0.0033(9)	–0.0075(9)
N(2)	2*i*	0.6752(2)	–0.2799(2)	1.6892(2)	0.029(1)	0.024(1)	0.012(1)	0.0026(9)	0.0000(9)	–0.0074(9)
N(3)	2*i*	0.5504(2)	0.3376(2)	0.5398(2)	0.034(1)	0.048(2)	0.016(1)	–0.016(1)	–0.003(1)	–0.001(1)
N(4)	2*i*	0.5397(3)	0.3955(2)	0.4372(2)	0.036(1)	0.055(2)	0.019(1)	–0.016(1)	–0.006(1)	–0.002(1)
N(5)	2*i*	0.9993(2)	0.0953(2)	0.5727(2)	0.028(1)	0.038(1)	0.013(1)	–0.010(1)	–0.0018(9)	–0.002(1)
N(6)	2*i*	0.9958(2)	0.1492(2)	0.4692(2)	0.030(1)	0.037(1)	0.013(1)	–0.012(1)	–0.0004(9)	–0.002(1)
C(1)	2*i*	0.8398(3)	–0.2542(2)	1.2582(2)	0.023(1)	0.023(1)	0.016(1)	–0.005(1)	–0.001(1)	–0.008(1)
C(2)	2*i*	0.7734(3)	–0.2341(2)	1.3477(2)	0.025(1)	0.025(1)	0.013(1)	0.006(1)	–0.004(1)	–0.006(1)
C(3)	2*i*	0.7545(3)	–0.2831(2)	1.5423(2)	0.023(1)	0.017(1)	0.016(1)	0.002(1)	–0.004(1)	–0.007(1)
C(4)	2*i*	0.6107(3)	–0.1886(2)	1.6300(2)	0.023(1)	0.018(1)	0.013(1)	–0.001(1)	0.000(1)	–0.005(1)
C(5)	2*i*	0.4843(3)	–0.1687(2)	1.7970(2)	0.039(2)	0.023(1)	0.016(1)	0.004(1)	0.001(1)	–0.008(1)
C(6)	2*i*	0.4107(2)	–0.0929(2)	1.8426(2)	0.018(1)	0.022(1)	0.021(1)	–0.003(1)	0.002(1)	–0.010(1)
C(7)	2*i*	0.6287(2)	0.1226(2)	0.8532(2)	0.019(1)	0.019(1)	0.012(1)	0.001(1)	0.002(1)	–0.007(1)
C(8)	2*i*	0.5851(3)	0.2017(2)	0.7518(2)	0.023(1)	0.029(1)	0.018(1)	–0.005(1)	–0.002(1)	–0.002(1)
C(9)	2*i*	0.6711(3)	0.3436(2)	0.5659(2)	0.035(2)	0.023(1)	0.016(1)	–0.009(1)	–0.002(1)	–0.002(1)
C(10)	2*i*	0.6530(3)	0.4415(2)	0.3929(2)	0.034(2)	0.027(1)	0.016(1)	–0.008(1)	–0.003(1)	–0.006(1)
C(11)	2*i*	0.5607(3)	0.4794(2)	0.2122(2)	0.028(1)	0.044(2)	0.018(1)	–0.008(1)	0.001(1)	–0.014(1)
C(12)	2*i*	0.6133(3)	0.5131(2)	0.1038(2)	0.028(1)	0.037(2)	0.019(1)	–0.008(1)	0.003(1)	–0.015(1)
C(13)	2*i*	0.9951(2)	–0.0436(2)	0.8989(2)	0.019(1)	0.021(1)	0.018(1)	–0.002(1)	0.002(1)	–0.011(1)
C(14)	2*i*	0.9951(3)	0.0103(2)	0.7870(2)	0.022(1)	0.035(2)	0.017(1)	–0.010(1)	–0.001(1)	–0.008(1)
C(15)	2*i*	0.8809(3)	0.0550(2)	0.6156(2)	0.026(1)	0.024(1)	0.012(1)	–0.004(1)	0.000(1)	–0.006(1)
C(16)	2*i*	0.8756(3)	0.1472(2)	0.4402(2)	0.026(1)	0.021(1)	0.012(1)	–0.008(1)	0.001(1)	–0.003(1)
C(17)	2*i*	0.9754(3)	0.2821(2)	0.2556(2)	0.032(1)	0.028(1)	0.015(1)	–0.013(1)	–0.002(1)	–0.003(1)
C(18)	2*i*	0.9624(2)	0.3247(2)	0.1454(2)	0.019(1)	0.016(1)	0.016(1)	0.0010(9)	–0.001(1)	–0.003(1)

Acknowledgments. This work was supported by the Foundation for Young Backbone Teacher of Henan Colleges and Universities in 2008 (grant nos. 102, 198), the Natural Science Research Assistance Program from the Education Department of Henan Province (grant nos. 2009A150007, 2009A430024), and the Base and Cutting-edge Technology Research Project of Henan Province (grant no. 092300410033), which are gratefully acknowledged.

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