Electronic Supplementary Information

Rare example of a polynuclear heterometallic yttrium(III)-copper(I) iodide cluster with a $[Y_6(\mu_6\text{-}O)(\mu_3\text{-}OH)_8]^{8+}$ core structure showing single crystal-to-single crystal transformation

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Experimental

All manipulations were carried out under argon using Schlenk tubes and vacuum line techniques. Solvents were purified by standard methods. ^{1}H NMR spectra were registered on a Bruker AC-300 spectrometer. FT-IR spectra were recorded as Nujol or paratone mulls on a Perkin-Elmer Paragon 500 spectrometer. Crystals of **1** and **2** were obtained as mentioned above and mounted on a Nonius Kappa CCD diffractometer using MoK_α radiation (λ = 0.71073 Å). Intensities were collected at 150 K by means of the COLLECT software.[1] Reflection indexing, Lorentz-polarization correction, peak integration and background determination were carried out with DENZO.[2] Frame scaling and unit-cell parameters refinement were made with SCALEPACK.[2] An absorption correction was applied with the DIFABS program.[3] The structures were solved by direct methods with SIR97.[4] The remaining non-hydrogen atoms were located by successive difference Fourier map analyses. The hydrogen atoms were placed geometrically and included in the refinement using soft restraints on the bond lengths and angles to regularize their geometry (C-H in the range 0.93-0.98 Å and O-H = 0.82 Å) and isotropic atomic displacement parameters (U(H) in the range 1.2-1.5 times $U_{eq.}$ of the adjacent atom). The structure refinement was carried out with CRYSTALS.[5] In compound **1**, one of the highly disordered DMSO molecule (O7S7C13C14) was refined with restraints on the bond lengths and angles (the electronic density best explained with the given model). As a result of this, the methyl carbon C14 is close [2.81(5) Å] to inversion related C14 at (-x, -y, 1-z).

1 B. V. Nonius, *COLLECT*, Nonius, Delft, The Netherlands (1997-2001).

2 Z. Otwinowski and W. Minor, *Methods in Enzymology*, Eds.: C. W. Carter Jr and R. M. Sweet, Academic Press, New York, 1997, **Vol. 276**, pp. 307–326.

3 N. Walker and D. Stuart, *Acta Cryst*, 1983, **A39**, 158.

4 A. Altomare, M. C. Burla, M. Camalli, G. L. Cascarano, C. Giacovazzo, A. Guagliardi, A. G. G. Moliterni, G. Polidori and R. Spagna, *J. Appl. Cryst*., 1999, **32**, 115.

5 D. J. Watkin, C. K. Prout and L. J. Pearce, *CAMERON*, Chemical Crystallography Laboratory, OXFORD, UK (1996).

Table S1. Selected bond lengths (Å) and angles (°) for 1

Bond length			
Y1—O1	2.314(2)	Y3—O8	2.334(2)
Y1—O2	2.37(2)	Y3—O9	2.445(1)
Y1—O3	2.351(2)	Y3—O10	2.312(1)
Y1—O10	2.369(1)	Y3—O11^{i}	2.331(1)
Y1—O11	2.291(1)	Y3—O12	2.347(1)
Y1—O12^{i}	2.373(1)	Y3—O13	2.346(1)
Y1—O13	2.321(1)	Y3—O14	2.469(2)
Y1—O14	2.4957(2)	Y3···Y2	3.519(2)
Y2—O4	2.271(2)	Y3···Y1	3.515(3)
Y2—O5	2.324(2)	Y3···Y1^{i}	3.507(3)
Y2—O6	2.318(2)	Y2···Y1^{i}	3.523(2)
Y2—O10^{i}	2.314(1)	Y2···Y1	3.533(3)
Y2—O11	2.376(1)	Y3···Y2^{i}	3.499(3)
Y2—O12	2.302(1)	Cu1···Cu1^{ii}	2.618(8)
Y2—O13	2.324(1)	I2—Cu1^{ii}	2.582(4)
Y2—O14	2.493(2)	I2—Cu1	2.539(5)
Y3—O7	2.323(2)	I1—Cu1	2.531(5)
Bond angle			
O11^{i}—Y3—O13	129.6 (4)	O4—Y2—O6	77.3 (7)
O11^{i}—Y3—O12	79.8 (4)	O14—Y2—O6	133.6 (4)
O11^{i}—Y3—O9	77.4 (4)	O5—Y2—O6	74.5 (7)
O11^{i}—Y3—O10	80.7 (4)	O1—Y1—O14	137.8 (5)
O11^{i}—Y3—O7	98.7 (5)	O11—Y1—O3	83.4 (5)
O11^{i}—Y3—O14	65.2 (3)	O13—Y1—O3	78.7 (5)
O11^{i}—Y3—O8	153.8 (5)	O10—Y1—O3	135.5 (6)
O13—Y3—O12	78.3 (4)	O1—Y1—O3	74.3 (6)
O13—Y3—O9	140.6 (4)	O14—Y1—O3	134.5 (4)
O12—Y3—O9	140.2 (4)	O11—Y1—O2	87.0 (6)
O13—Y3—O10	80.3 (4)	O13—Y1—O2	152.3 (5)
O12—Y3—O10	130.7 (4)	O10—Y1—O2	126.7 (5)
O9—Y3—O10	76.7 (4)	O1—Y1—O2	79.3 (7)
O13—Y3—O7	118.5 (5)	O14—Y1—O2	130.6 (5)
O12—Y3—O7	75.5 (5)	O3—Y1—O2	75.8 (6)
O9—Y3—O7	76.2 (5)	O12^{i}—Y1—O11	80.0 (4)
O10—Y3—O7	152.4 (5)	O12^{i}—Y1—O13	128.8 (4)
O13—Y3—O14	64.4 (3)	O12^{i}—Y1—O10	78.4 (4)
O12—Y3—O14	65.3 (3)	O12^{i}—Y1—O1	113.3 (6)

O9—Y3—O14	129.7 (3)	O12^{i}—Y1—O14	64.5 (3)
O10—Y3—O14	65.5 (3)	O12^{i}—Y1—O3	143.6 (6)
O7—Y3—O14	139.3 (4)	O12^{i}—Y1—O2	71.2 (5)
O13—Y3—O8	72.0 (5)	O11—Y1—O13	79.5 (4)
O12—Y3—O8	123.4 (5)	O11—Y1—O10	129.6 (4)
O9—Y3—O8	76.5 (5)	O13—Y1—O10	79.6 (4)
O10—Y3—O8	90.3 (6)	O11—Y1—O1	156.1 (5)
O7—Y3—O8	78.1 (7)	O13—Y1—O1	104.0 (6)
O14—Y3—O8	132.6 (4)	O10—Y1—O1	73.9 (5)
O10^{i}—Y2—O11	79.7 (4)	O11—Y1—O14	65.3 (3)
O10^{i}—Y2—O13	129.4 (4)	O13—Y1—O14	64.3 (3)
O10^{i}—Y2—O12	81.0 (4)	O10—Y1—O14	64.2 (3)
O10^{i}—Y2—O4	99.1 (6)	Y1^{i}···Y2···Y3	59.74 (5)
O10^{i}—Y2—O14	65.0 (3)	Y1^{i}···Y2···Y3^{i}	60.08 (6)
O10^{i}—Y2—O5	81.5 (6)	Y3···Y2···Y3^{i}	89.45 (7)
O10^{i}—Y2—O6	156.0 (6)	Y1^{i}···Y2···Y1	90.06 (6)
O11—Y2—O13	77.7 (4)	Y3···Y2···Y1	59.79 (5)
O11—Y2—O12	129.7 (4)	Y3^{i}···Y2···Y1	59.82 (6)
O13—Y2—O12	79.7 (4)	Y2···Y1···Y2^{i}	89.94 (6)
O11—Y2—O4	71.4 (5)	Y2···Y1···Y3	59.91 (5)
O13—Y2—O4	115.3 (6)	Y2^{i}···Y1···Y3	59.63 (6)
O12—Y2—O4	157.9 (5)	Y2···Y1···Y3^{i}	59.61 (6)
O11—Y2—O14	64.2 (3)	Y2^{i}···Y1···Y3^{i}	60.08 (5)
O13—Y2—O14	64.3 (3)	Y3···Y1···Y3^{i}	89.39 (7)
O12—Y2—O14	65.5 (3)	Y1^{i}···Y3···Y2^{i}	60.57 (6)
O4—Y2—O14	134.7 (5)	Y1^{i}···Y3···Y2	60.18 (5)
O11—Y2—O5	140.3 (6)	Y2^{i}···Y3···Y2	90.55 (6)
O13—Y2—O5	139.4 (6)	Y1^{i}···Y3···Y1	90.61 (6)
O12—Y2—O5	80.7 (5)	Y2^{i}···Y3···Y1	60.29 (5)
O4—Y2—O5	77.4 (6)	Y2···Y3···Y1	60.30 (5)
O14—Y2—O5	134.9 (4)	I2^{ii}—Cu1—I2	118.52 (16)
O11—Y2—O6	120.2 (6)	I2^{ii}—Cu1—I1	118.67 (19)
O13—Y2—O6	71.6 (5)	I2—Cu1—I1	122.54 (15)
O12—Y2—O6	93.6 (6)		

Symmetry codes: (i) −*x*+1, −*y*, −*z*+1; (ii) −*x*, −*y*, −*z*+2.

Table S2. Selected bond lengths (Å) and angles (°) for 2

Bond lengths			
Y1—O1	2.339 (8)	Y3—O7	2.296 (8)
Y1—O2	2.331 (9)	Y3—O8	2.427 (8)
Y1—O3	2.339 (8)	Y3—O9	2.423 (8)
Y1—O10^{i}	2.307 (7)	Y3—O10	2.338 (7)
Y1—O11	2.379 (7)	Y3—O11	2.317 (8)
Y1—O12^{i}	2.352 (8)	Y3—O12^{i}	2.329 (7)
Y1—O13	2.354 (8)	Y3—O13^{i}	2.338 (7)

Y1—O14	2.5076 (1)	Y3—O14	2.4356 (1)
Y2—O4	2.358 (8)	Y2···Y1	3.5503 (2)
Y2—O5	2.309 (9)	Y2···Y1^{i}	3.5613 (2)
Y2—O6	2.323 (1)	Y2···Y3^{i}	3.5174 (2)
Y2—O10	2.386 (7)	Y2···Y3	3.4934 (2)
Y2—O11	2.304 (8)	I2—Cu1	2.5534 (2)
Y2—O12	2.356 (7)	I3—Cu1	2.4994 (2)
Y2—O13	2.333 (7)	I1—Cu1	2.581 (2)
Y2—O14	2.521(1)	I6—I7^{ii}	2.772 (4)
		I6—I7	2.772 (4)

Bond angle

O12^{i}—Y1—O10^{i}	79.6 (3)	O14—Y3—O9	131.1 (2)
O12^{i}—Y1—O14	64.79 (2)	O13^{i}—Y3—O8	130.1 (3)
O10^{i}—Y1—O14	65.21 (2)	O12^{i}—Y3—O8	146.7 (3)
O12^{i}—Y1—O11	80.6 (3)	O14—Y3—O8	134.1 (2)
O10^{i}—Y1—O11	129.5 (3)	O11—Y3—O7	153.4 (3)
O14—Y1—O11	64.31 (2)	O11—Y3—O10	80.8 (3)
O12^{i}—Y1—O13	129.0 (3)	O7—Y3—O10	116.1 (3)
O10^{i}—Y1—O13	79.6 (3)	O11—Y3—O9	78.1 (3)
O14—Y1—O13	64.24 (2)	O7—Y3—O9	76.1 (3)
O12^{i}—Y1—O1	155.9 (3)	O10—Y3—O9	140.8 (3)
O10^{i}—Y1—O1	93.8 (3)	O11—Y3—O8	84.8 (3)
O14—Y1—O1	133.2 (2)	O7—Y3—O8	80.5 (3)
O12^{i}—Y1—O2	80.0 (3)	O10—Y3—O8	75.1 (3)
O10^{i}—Y1—O2	82.1 (3)	O9—Y3—O8	70.5 (3)
O14—Y1—O2	134.8 (2)	O13^{i}—Y3—O12^{i}	79.7 (3)
O12^{i}—Y1—O3	98.5 (3)	O13^{i}—Y3—O14	65.63 (2)
O10^{i}—Y1—O3	160.5 (3)	O12^{i}—Y3—O14	66.30 (2)
O14—Y1—O3	131.7 (2)	O13^{i}—Y3—O11	132.0 (3)
O11—Y1—O13	78.0 (3)	O12^{i}—Y3—O11	82.4 (3)
O11—Y1—O1	120.0 (3)	O14—Y3—O11	66.36 (2)
O13—Y1—O1	71.3 (3)	O13^{i}—Y3—O7	73.6 (3)
O11—Y1—O2	138.4 (3)	O12^{i}—Y3—O7	98.0 (3)
O13—Y1—O2	141.1 (3)	O14—Y3—O7	138.1 (2)
O1—Y1—O2	76.2 (3)	O13^{i}—Y3—O10	79.3 (3)
O11—Y1—O3	68.5 (3)	O12^{i}—Y3—O10	132.2 (3)
O13—Y1—O3	115.1 (3)	O14—Y3—O10	65.96 (2)
O1—Y1—O3	79.9 (3)	O13^{i}—Y3—O9	138.3 (3)
O2—Y1—O3	78.5 (3)	O12^{i}—Y3—O9	76.9 (3)
O11—Y2—O10	80.1 (3)	Y2—O14—Y2^{i}	179.995
O13—Y2—O10	128.2 (3)	Y2—O14—Y1^{i}	90.18 (4)
O4—Y2—O10	144.2 (3)	Y2^{i}—O14—Y1^{i}	89.82 (4)
O5—Y2—O10	72.3 (3)	Y2—O14—Y1	89.82 (4)
O11—Y2—O12	129.6 (3)	Y2^{i}—O14—Y1	90.18 (4)
O13—Y2—O12	79.2 (3)	Y1^{i}—O14—Y1	179.995
O4—Y2—O12	136.4 (3)	Y2—O14—Y3^{i}	90.39 (4)

O5—Y2—O12	126.0 (3)	$Y2^{i}$—O14—$Y3^{i}$	89.61 (4)
O10—Y2—O12	78.0 (3)	$Y1^{i}$—O14—$Y3^{i}$	89.54 (4)
O11—Y2—O6	154.3 (3)	Y1—O14—$Y3^{i}$	90.46 (4)
O13—Y2—O6	101.1 (3)	Y2—O14—Y3	89.61 (4)
O4—Y2—O6	74.2 (4)	$Y2^{i}$—O14—Y3	90.39 (4)
O5—Y2—O6	79.5 (4)	$Y1^{i}$—O14—Y3	90.46 (4)
O10—Y2—O6	116.7 (3)	Y1—O14—Y3	89.54 (4)
O12—Y2—O6	75.0 (3)	$Y3^{i}$—O14—Y3	179.995
O14—Y2—O11	65.11 (2)	I1—Cu1—I2	109.83 (6)
O14—Y2—O13	64.31 (2)	I1—Cu1—I3	130.53 (7)
O14—Y2—O4	131.9 (2)	I2—Cu1—I3	119.65 (7)
O14—Y2—O5	131.5 (2)	$I7^{ii}$—I6—I7	179.72 (1)
O14—Y2—O10	63.92 (2)	$Y1^{i}$···Y2···$Y3^{i}$	58.92 (3)
O14—Y2—O12	64.52 (2)	$Y1^{i}$···Y2···Y3	59.66 (3)
O14—Y2—O6	138.6 (3)	$Y3^{i}$···Y2···Y3	88.02 (4)
O11—Y2—O13	79.9 (3)	$Y1^{i}$···Y2···Y1	89.69 (4)
O11—Y2—O4	81.2 (3)	$Y3^{i}$···Y2···Y1	59.55 (3)
O13—Y2—O4	77.3 (3)	Y3···Y2···Y1	59.24 (3)
O11—Y2—O5	88.3 (3)	$Y2^{i}$···Y1···Y2	90.31 (4)
O13—Y2—O5	153.0 (3)	$Y2^{i}$···Y3···Y2	91.98 (4)
O4—Y2—O5	76.9 (3)		

Symmetry codes: (i) −*x*+1/2, −*y*+3/2, −*z*+1; (ii) −*x*+1, *y*, −*z*+1/2.

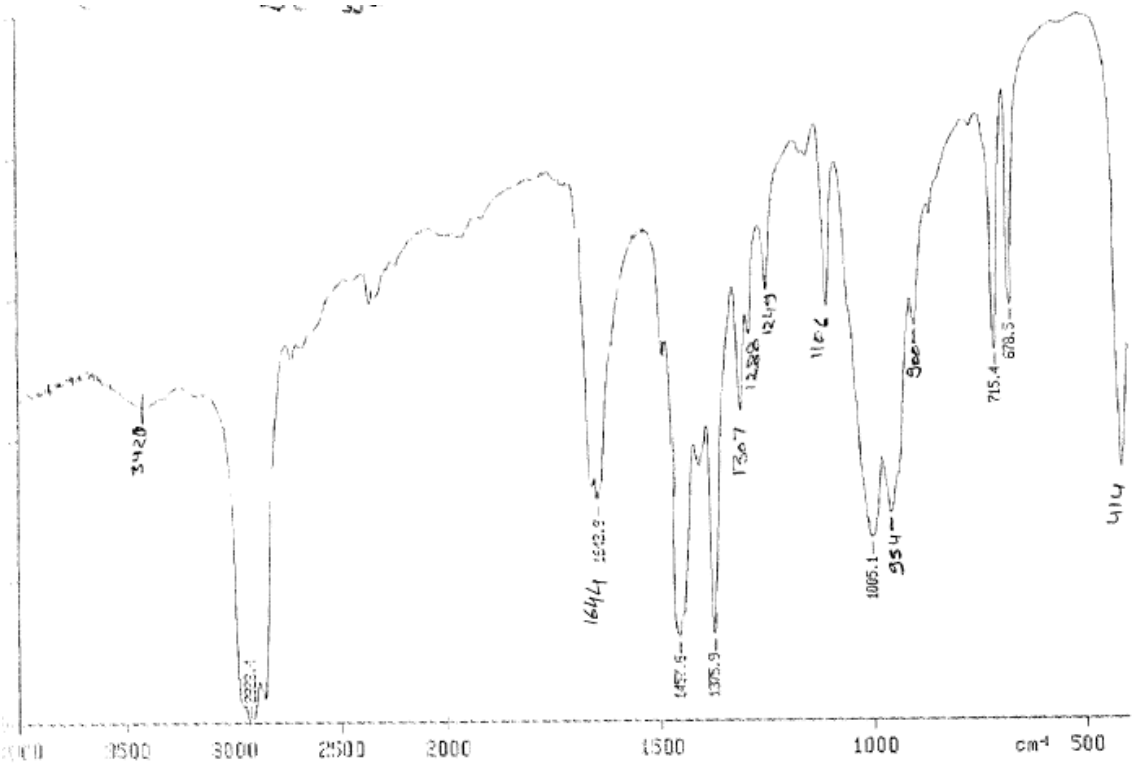

Fig. S1: FT-IR spectrum of **1** in nujol.

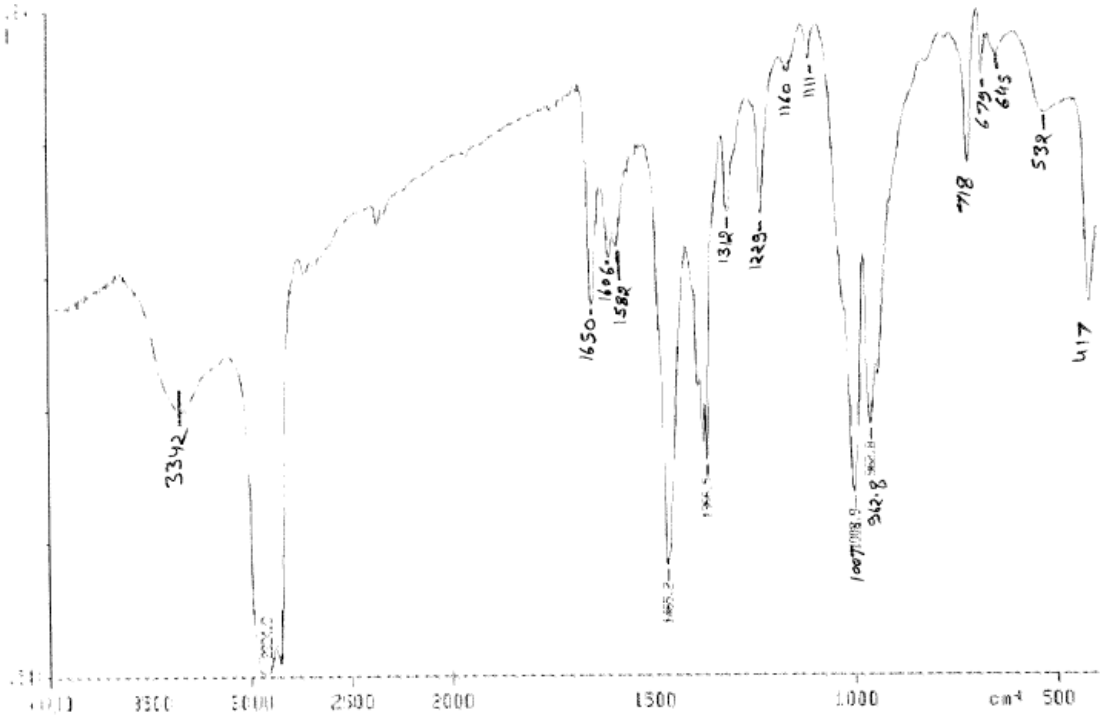

Fig. S2: FT-IR spectrum of **2** in paratone.