SUPPORTING INFORMATION

APPENDIX A

COMPLEX CRYSTAL STRUCTURES

A.1 – Copper(II) crystal structure of TzMorph2DO3A

Table 1. Crystal data and structure refinement details.

Identification code	**ros1**	
Empirical formula	$C_{27}H_{44}CuN_9O_{9.50}$	
Formula weight	710.25	
Temperature	100.15 K	
Wavelength	0.71075 Å	
Crystal system	Orthorhombic	
Space group	*Pbca*	
Unit cell dimensions	a = 12.5699(4) Å	α = 90°
	b = 12.9272(4) Å	β = 90°
	c = 39.0800(10) Å	γ = 90°
Volume	6350.2(3) Å^3	
Z	8	
Density (calculated)	1.486 Mg / m^3	
Absorption coefficient	0.756 mm^{-1}	
F(000)	2992	
Crystal	Needle; light blue	
Crystal size	0.2 × 0.01 × 0.01 mm^3	
θ range for data collection	3.075 – 25.026°	
Index ranges	$-13 \le h \le 14$, $-15 \le k \le 15$, $-46 \le l \le 46$	
Reflections collected	36862	
Independent reflections	5610 [R_{int} = 0.0917]	
Completeness to θ = 26.000°	89.9 %	
Absorption correction	Semi–empirical from equivalents	
Max. and min. transmission	1.00000 and 0.82442	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5610 / 0 / 430	
Goodness-of-fit on F^2	1.038	
Final R indices [$F^2 > 2\sigma(F^2)$]	*R1* = 0.0481, *wR2* = 0.1134	
R indices (all data)	*R1* = 0.0712, *wR2* = 0.1243	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.885 and –0.667 e Å^{-3}	

Diffractometer: Rigaku AFC12 goniometer equipped with an enhanced sensitivity (HG) Saturn724+ detector mounted at the window of an FR-E+ SuperBright molybdenum rotating anode generator with HF Varimax optics (100m focus). **Cell determination and data collection:** CrystalClear-SM Expert 3.1 b27 (Rigaku, 2012). **Data reduction, cell refinement and absorption correction:** CrysAlisPro, Agilent Technologies, Version 1.171.37.35).). **Structure solution:** SUPERFLIP (Palatinus, L. & Chapuis, G. (2007). J. Appl. Cryst. 40, 786-790). **Structure refinement:** SHELXL-2013 (Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122).

Special details:
In the crystal structure one half-occupied water molecule is disordered around centre of inversion.

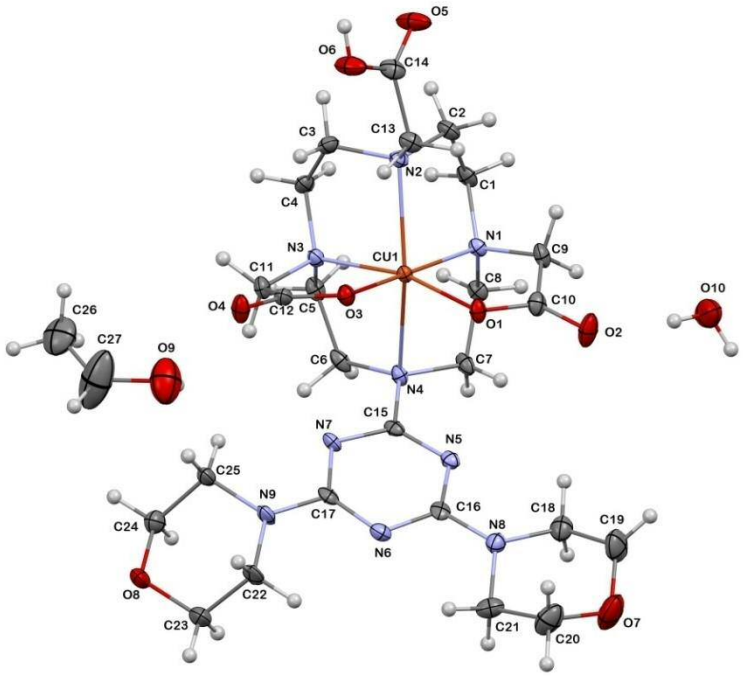

Figure 1. Molecular structure of 2014ncs0930 (MM158).
Displacement ellipsoids – 50% probability.

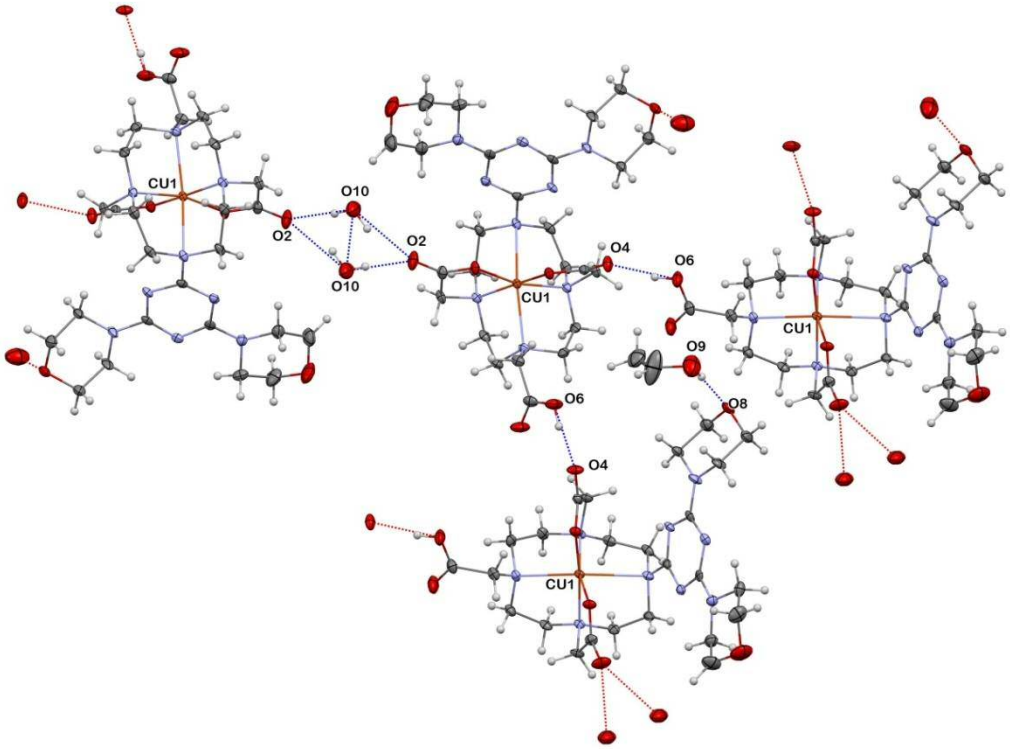

Figure 2. Hydrogen bond network in 2014ncs0930 (MM158)

Table 1. Atomic coordinates [$\times 10^4$], equivalent isotropic displacement parameters [$Å^2 \times 10^3$] and site occupancy factors. U_{eq} is defined as one third of the trace of the orthogonalized U^{ij} tensor.

Atom	x	y	z	U_{eq}	S.o.f.
C1	4288(3)	7390(3)	6381(1)	18(1)	1
C2	4704(3)	6734(3)	6677(1)	19(1)	1
C3	3239(3)	6378(3)	7088(1)	18(1)	1
C4	2418(3)	7146(3)	6962(1)	18(1)	1
C5	1275(3)	7587(2)	6489(1)	17(1)	1
C6	772(3)	7302(3)	6150(1)	18(1)	1
C7	2174(3)	6996(3)	5708(1)	18(1)	1
C8	3079(3)	7514(2)	5898(1)	16(1)	1
C9	4396(3)	6129(3)	5911(1)	18(1)	1
C10	3936(3)	5065(3)	5834(1)	18(1)	1
C11	918(3)	6014(3)	6819(1)	16(1)	1
C12	1207(3)	4880(3)	6799(1)	16(1)	1
C13	4385(3)	4996(3)	6913(1)	18(1)	1
C14	5008(3)	5055(3)	7249(1)	21(1)	1
C15	831(3)	5679(2)	5832(1)	14(1)	1
C16	582(3)	4384(2)	5458(1)	14(1)	1
C17	−524(3)	4569(3)	5906(1)	15(1)	1
C18	1748(3)	4273(3)	4959(1)	25(1)	1
C19	2287(3)	3407(3)	4765(1)	37(1)	1
C20	847(4)	2298(3)	4822(1)	37(1)	1
C21	205(3)	3090(3)	5016(1)	23(1)	1
C22	−2112(3)	3459(3)	5973(1)	23(1)	1
C23	−3244(3)	3864(3)	5974(1)	28(1)	1
C24	−2837(3)	5093(3)	6397(1)	24(1)	1
C25	−1693(3)	4756(3)	6409(1)	20(1)	1
C26	−2221(4)	8272(4)	7186(1)	50(1)	1
C27	−2182(6)	7394(4)	6959(1)	85(2)	1
N1	3677(2)	6788(2)	6122(1)	13(1)	1
N2	3923(2)	5989(2)	6809(1)	14(1)	1
N3	1759(2)	6712(2)	6679(1)	14(1)	1
N4	1372(2)	6554(2)	5941(1)	14(1)	1
N5	1156(2)	5223(2)	5546(1)	15(1)	1
N6	−283(2)	4032(2)	5623(1)	16(1)	1
N7	19(2)	5377(2)	6031(1)	14(1)	1
N8	909(2)	3851(2)	5178(1)	18(1)	1
N9	−1396(2)	4278(2)	6085(1)	20(1)	1
O1	3243(2)	4743(2)	6049(1)	16(1)	1
O2	4276(2)	4571(2)	5589(1)	31(1)	1
O3	1965(2)	4634(2)	6597(1)	16(1)	1
O4	685(2)	4239(2)	6963(1)	22(1)	1
O5	5704(2)	5683(2)	7301(1)	33(1)	1
O6	4682(2)	4364(2)	7471(1)	30(1)	1
O7	1551(3)	2790(2)	4586(1)	49(1)	1
O8	−3530(2)	4261(2)	6306(1)	27(1)	1
O9	−1423(3)	7369(3)	6704(1)	59(1)	1
O10	5683(5)	5484(5)	5139(1)	44(2)	0.5
Cu1	2702(1)	5758(1)	6371(1)	11(1)	1

Table 2. Bond lengths [Å] and angles [°].

C1–H1A	0.9900	C18–N8	1.464(4)
C1–H1B	0.9900	C19–H19	0.9500
C1–C2	1.526(4)	C19–O7	1.408(5)
C1–N1	1.492(4)	C20–H20A	0.9900
C2–H2A	0.9900	C20–H20B	0.9900
C2–H2B	0.9900	C20–C21	1.507(5)
C2–N2	1.469(4)	C20–O7	1.430(5)
C3–H3A	0.9900	C21–H21A	0.9900
C3–H3B	0.9900	C21–H21B	0.9900
C3–C4	1.514(5)	C21–N8	1.468(4)
C3–N2	1.476(4)	C22–H22A	0.9900
C4–H4A	0.9900	C22–H22B	0.9900
C4–H4B	0.9900	C22–C23	1.517(5)
C4–N3	1.493(4)	C22–N9	1.457(4)
C5–H5A	0.9900	C23–H23A	0.9900
C5–H5B	0.9900	C23–H23B	0.9900
C5–C6	1.513(4)	C23–O8	1.442(4)
C5–N3	1.483(4)	C24–H24A	0.9900
C6–H6A	0.9900	C24–H24B	0.9900
C6–H6B	0.9900	C24–C25	1.503(5)
C6–N4	1.474(4)	C24–O8	1.429(4)
C7–H7A	0.9900	C25–H25A	0.9900
C7–H7B	0.9900	C25–H25B	0.9900
C7–C8	1.515(4)	C25–N9	1.456(4)
C7–N4	1.475(4)	C26–H26A	0.9800
C8–H8A	0.9900	C26–H26B	0.9800
C8–H8B	0.9900	C26–H26C	0.9800
C8–N1	1.487(4)	C26–C27	1.441(7)
C9–H9A	0.9900	C27–H27	0.9500
C9–H9B	0.9900	C27–O9	1.381(6)
C9–C10	1.523(5)	N1–Cu1	2.056(3)
C9–N1	1.489(4)	N2–Cu1	2.319(3)
C10–O1	1.280(4)	N3–Cu1	2.091(3)
C10–O2	1.227(4)	O1–Cu1	1.942(2)
C11–H11A	0.9900	O3–Cu1	1.935(2)
C11–H11B	0.9900	O6–H6	0.8400
C11–C12	1.512(5)	O9–H9	0.8400
C11–N3	1.494(4)	O10–H10A	0.8503
C12–O3	1.277(4)	O10–H10B	0.8499
C12–O4	1.236(4)		
C13–H13A	0.9900	H1A–C1–H1B	107.7
C13–H13B	0.9900	C2–C1–H1A	108.8
C13–C14	1.532(5)	C2–C1–H1B	108.8
C13–N2	1.465(4)	N1–C1–H1A	108.8
C14–O5	1.210(4)	N1–C1–H1B	108.8
C14–O6	1.311(4)	N1–C1–C2	113.6(3)
C15–N4	1.387(4)	C1–C2–H2A	108.8
C15–N5	1.326(4)	C1–C2–H2B	108.8
C15–N7	1.343(4)	H2A–C2–H2B	107.7
C16–N5	1.347(4)	N2–C2–C1	113.8(3)
C16–N6	1.343(4)	N2–C2–H2A	108.8
C16–N8	1.357(4)	N2–C2–H2B	108.8
C17–N6	1.341(4)	H3A–C3–H3B	107.8
C17–N7	1.340(4)	C4–C3–H3A	109.1
C17–N9	1.354(4)	C4–C3–H3B	109.1
C18–H18A	0.9900	N2–C3–H3A	109.1
C18–H18B	0.9900	N2–C3–H3B	109.1
C18–C19	1.512(5)	N2–C3–C4	112.5(3)
C3–C4–H4A	109.2	C3–C4–H4B	109.2

H4A–C4–H4B	107.9
N3–C4–C3	111.8(3)
N3–C4–H4A	109.2
N3–C4–H4B	109.2
H5A–C5–H5B	107.5
C6–C5–H5A	108.5
C6–C5–H5B	108.5
N3–C5–H5A	108.5
N3–C5–H5B	108.5
N3–C5–C6	115.0(3)
C5–C6–H6A	108.4
C5–C6–H6B	108.4
H6A–C6–H6B	107.5
N4–C6–C5	115.5(3)
N4–C6–H6A	108.4
N4–C6–H6B	108.4
H7A–C7–H7B	107.9
C8–C7–H7A	109.1
C8–C7–H7B	109.1
N4–C7–H7A	109.1
N4–C7–H7B	109.1
N4–C7–C8	112.4(3)
C7–C8–H8A	109.0
C7–C8–H8B	109.0
H8A–C8–H8B	107.8
N1–C8–C7	112.9(3)
N1–C8–H8A	109.0
N1–C8–H8B	109.0
H9A–C9–H9B	107.7
C10–C9–H9A	108.9
C10–C9–H9B	108.9
N1–C9–H9A	108.9
N1–C9–H9B	108.9
N1–C9–C10	113.3(3)
O1–C10–C9	114.9(3)
O2–C10–C9	119.6(3)
O2–C10–O1	125.5(3)
H11A–C11–H11B	107.7
C12–C11–H11A	108.9
C12–C11–H11B	108.9
N3–C11–H11A	108.9
N3–C11–H11B	108.9
N3–C11–C12	113.4(3)
O3–C12–C11	116.9(3)
O4–C12–C11	119.7(3)
O4–C12–O3	123.4(3)
H13A–C13–H13B	107.7
C14–C13–H13A	108.9
C14–C13–H13B	108.9
N2–C13–H13A	108.9
N2–C13–H13B	108.9
N2–C13–C14	113.3(3)
O5–C14–C13	123.1(3)
O5–C14–O6	124.9(3)
O6–C14–C13	112.0(3)
N5–C15–N4	118.1(3)
N5–C15–N7	126.4(3)
N7–C15–N4	115.5(3)
N5–C16–N8	116.9(3)
C1–N1–Cu1	108.79(18)
N6–C16–N5	125.8(3)
N6–C16–N8	117.3(3)
N6–C17–N9	117.8(3)
N7–C17–N6	126.1(3)
N7–C17–N9	116.1(3)
H18A–C18–H18B	108.2
C19–C18–H18A	109.7
C19–C18–H18B	109.7
N8–C18–H18A	109.7
N8–C18–H18B	109.7
N8–C18–C19	109.9(3)
C18–C19–H19	124.0
O7–C19–C18	112.1(3)
O7–C19–H19	124.0
H20A–C20–H20B	108.1
C21–C20–H20A	109.5
C21–C20–H20B	109.5
O7–C20–H20A	109.5
O7–C20–H20B	109.5
O7–C20–C21	110.7(3)
C20–C21–H21A	109.6
C20–C21–H21B	109.6
H21A–C21–H21B	108.1
N8–C21–C20	110.5(3)
N8–C21–H21A	109.6
N8–C21–H21B	109.6
H22A–C22–H22B	108.3
C23–C22–H22A	109.9
C23–C22–H22B	109.9
N9–C22–H22A	109.9
N9–C22–H22B	109.9
N9–C22–C23	109.1(3)
C22–C23–H23A	109.4
C22–C23–H23B	109.4
H23A–C23–H23B	108.0
O8–C23–C22	111.1(3)
O8–C23–H23A	109.4
O8–C23–H23B	109.4
H24A–C24–H24B	107.9
C25–C24–H24A	109.2
C25–C24–H24B	109.2
O8–C24–H24A	109.2
O8–C24–H24B	109.2
O8–C24–C25	111.9(3)
C24–C25–H25A	109.7
C24–C25–H25B	109.7
H25A–C25–H25B	108.2
N9–C25–C24	109.9(3)
N9–C25–H25A	109.7
N9–C25–H25B	109.7
H26A–C26–H26B	109.5
H26A–C26–H26C	109.5
H26B–C26–H26C	109.5
C27–C26–H26A	109.5
C27–C26–H26B	109.5
C27–C26–H26C	109.5
C26–C27–H27	120.4
O9–C27–C26	119.1(5)
O9–C27–H27	120.4
C8–N1–C1	109.3(2)

C8–N1–C9	110.1(2)	C16–N8–C21	120.4(3)
C8–N1–Cu1	112.8(2)	C18–N8–C21	115.5(3)
C9–N1–C1	111.2(3)	C17–N9–C22	123.0(3)
C9–N1–Cu1	104.64(19)	C17–N9–C25	122.6(3)
C2–N2–C3	115.2(3)	C25–N9–C22	114.4(3)
C2–N2–Cu1	105.42(19)	C10–O1–Cu1	116.4(2)
C3–N2–Cu1	101.68(19)	C12–O3–Cu1	116.9(2)
C13–N2–C2	114.0(3)	C14–O6–H6	109.5
C13–N2–C3	109.0(3)	C19–O7–C20	109.6(3)
C13–N2–Cu1	110.68(19)	C24–O8–C23	109.8(3)
C4–N3–C11	110.4(2)	C27–O9–H9	109.5
C4–N3–Cu1	109.50(19)	H10A–O10–H10B	109.5
C5–N3–C4	108.2(2)	N1–Cu1–N2	82.68(10)
C5–N3–C11	110.7(3)	N1–Cu1–N3	103.18(10)
C5–N3–Cu1	113.34(19)	N3–Cu1–N2	82.83(10)
C11–N3–Cu1	104.75(19)	O1–Cu1–N1	85.46(10)
C6–N4–C7	115.9(3)	O1–Cu1–N2	109.54(10)
C15–N4–C6	117.1(3)	O1–Cu1–N3	165.95(10)
C15–N4–C7	117.4(3)	O3–Cu1–N1	171.01(10)
C15–N5–C16	114.0(3)	O3–Cu1–N2	94.45(9)
C17–N6–C16	113.8(3)	O3–Cu1–N3	84.84(10)
C17–N7–C15	113.7(3)	O3–Cu1–O1	87.51(9)
C16–N8–C18	120.0(3)		

Table 3. Anisotropic displacement parameters [$Å^2 \times 10^3$].

The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ...+ 2\ h\ k\ a^*\ b^*\ U^{12}]$.

Atom	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C1	14(2)	14(2)	26(2)	1(2)	1(1)	−6(1)
C2	13(2)	19(2)	24(2)	1(2)	−2(1)	−4(1)
C3	19(2)	21(2)	16(2)	−6(1)	−3(1)	0(2)
C4	17(2)	19(2)	19(2)	−9(1)	−1(1)	0(1)
C5	15(2)	12(2)	25(2)	1(1)	3(1)	1(1)
C6	12(2)	12(2)	29(2)	2(2)	1(1)	2(1)
C7	12(2)	20(2)	20(2)	8(2)	2(1)	−1(1)
C8	16(2)	12(2)	20(2)	2(1)	0(1)	−2(1)
C9	13(2)	17(2)	24(2)	0(2)	7(1)	1(1)
C10	18(2)	16(2)	19(2)	1(1)	3(2)	6(2)
C11	13(2)	17(2)	20(2)	0(1)	5(1)	2(1)
C12	13(2)	19(2)	16(2)	−4(1)	1(1)	−2(1)
C13	16(2)	13(2)	24(2)	−1(1)	−3(1)	1(1)
C14	18(2)	17(2)	27(2)	0(2)	−6(2)	4(2)
C15	11(2)	14(2)	16(2)	4(1)	−4(1)	0(1)
C16	12(2)	15(2)	15(2)	4(1)	−3(1)	7(1)
C17	9(2)	14(2)	22(2)	2(1)	−3(1)	3(1)
C18	25(2)	28(2)	22(2)	2(2)	2(2)	5(2)
C19	31(2)	38(3)	41(2)	−12(2)	12(2)	6(2)
C20	53(3)	29(2)	29(2)	−9(2)	4(2)	−2(2)
C21	28(2)	21(2)	21(2)	−2(2)	−8(2)	−1(2)
C22	13(2)	26(2)	30(2)	−9(2)	3(2)	−11(2)
C23	15(2)	44(2)	24(2)	−8(2)	0(2)	−8(2)
C24	20(2)	27(2)	24(2)	−6(2)	1(2)	2(2)
C25	11(2)	25(2)	23(2)	−8(2)	2(1)	−4(2)
C26	63(4)	51(3)	36(2)	14(2)	2(2)	9(3)
C27	143(6)	43(3)	69(4)	−7(3)	55(4)	−20(4)
N1	12(2)	10(1)	16(1)	0(1)	1(1)	−1(1)
N2	11(2)	15(2)	17(1)	−2(1)	−2(1)	1(1)
N3	11(2)	14(1)	17(1)	−3(1)	1(1)	−1(1)
N4	12(2)	13(1)	18(1)	1(1)	3(1)	−2(1)
N5	12(2)	16(2)	16(1)	2(1)	−2(1)	1(1)
N6	12(2)	15(2)	21(1)	−1(1)	−2(1)	0(1)
N7	8(1)	16(1)	20(1)	1(1)	1(1)	−2(1)
N8	18(2)	20(2)	16(1)	−1(1)	0(1)	2(1)
N9	10(2)	24(2)	27(2)	−9(1)	4(1)	−6(1)
O1	17(1)	12(1)	20(1)	−1(1)	7(1)	2(1)
O2	41(2)	23(1)	28(1)	−7(1)	17(1)	1(1)
O3	15(1)	14(1)	18(1)	−1(1)	2(1)	1(1)
O4	26(1)	15(1)	26(1)	−2(1)	12(1)	−6(1)
O5	33(2)	33(2)	33(1)	7(1)	−17(1)	−15(1)
O6	30(2)	34(2)	26(1)	10(1)	−14(1)	−12(1)
O7	64(2)	51(2)	31(2)	−18(2)	18(2)	−11(2)
O8	11(1)	41(2)	27(1)	−10(1)	2(1)	−4(1)
O9	60(2)	57(2)	60(2)	13(2)	17(2)	24(2)
O10	40(4)	61(4)	32(3)	−1(3)	7(3)	−23(3)
Cu1	10(1)	9(1)	14(1)	−1(1)	2(1)	0(1)

Table 4. Hydrogen coordinates [× 10^4] and isotropic displacement parameters [$Å^2$× 10^3].

Atom	x	y	z	Ueq	S.o.f.
H1A	3824	7941	6475	21	1
H1B	4898	7730	6267	21	1
H2A	5342	6351	6599	22	1
H2B	4925	7198	6865	22	1
H3A	3690	6713	7264	22	1
H3B	2871	5787	7197	22	1
H4A	1948	7343	7155	22	1
H4B	2783	7779	6881	22	1
H5A	1831	8114	6447	21	1
H5B	725	7908	6636	21	1
H6A	56	7014	6195	21	1
H6B	676	7943	6015	21	1
H7A	1826	7509	5556	21	1
H7B	2465	6438	5561	21	1
H8A	3576	7822	5729	19	1
H8B	2789	8083	6039	19	1
H9A	5079	6042	6034	21	1
H9B	4548	6488	5693	21	1
H11A	250	6129	6690	20	1
H11B	784	6199	7061	20	1
H13A	4867	4750	6730	21	1
H13B	3807	4482	6938	21	1
H18A	2278	4643	5101	30	1
H18B	1437	4773	4796	30	1
H19	3033	3293	4765	44	1
H20A	1265	1877	4986	45	1
H20B	361	1829	4697	45	1
H21A	−285	3447	4857	28	1
H21B	−228	2740	5193	28	1
H22A	−2053	2857	6128	28	1
H22B	−1915	3232	5739	28	1
H23A	−3314	4421	5801	33	1
H23B	−3737	3300	5910	33	1
H24A	−3046	5367	6624	29	1
H24B	−2914	5658	6228	29	1
H25A	−1232	5363	6453	23	1
H25B	−1591	4256	6598	23	1
H26A	−2158	8911	7052	75	1
H26B	−1632	8231	7350	75	1
H26C	−2898	8272	7310	75	1
H27	−2672	6840	6985	102	1
H6	5068	4389	7647	45	1
H9	−1419	7937	6600	89	1
H10A	5539	5378	4930	66	0.5
H10B	5261	5134	5264	66	0.5

Table 5. Torsion angles [°].

C1–C2–N2–C3	–89.8(3)	C24–C25–N9–C17	128.6(3)
C1–C2–N2–C13	143.0(3)	C24–C25–N9–C22	–51.7(4)
C1–C2–N2–Cu1	21.4(3)	C25–C24–O8–C23	–59.7(4)
C2–C1–N1–C8	165.6(3)	N1–C1–C2–N2	–43.6(4)
C2–C1–N1–C9	–72.7(3)	N1–C9–C10–O1	–24.8(4)
C2–C1–N1–Cu1	42.0(3)	N1–C9–C10–O2	158.6(3)
C3–C4–N3–C5	–159.5(3)	N2–C3–C4–N3	54.8(4)
C3–C4–N3–C11	79.3(3)	N2–C13–C14–O5	–52.4(5)
C3–C4–N3–Cu1	–35.5(3)	N2–C13–C14–O6	126.6(3)
C4–C3–N2–C2	72.4(4)	N3–C5–C6–N4	–41.0(4)
C4–C3–N2–C13	–157.9(3)	N3–C11–C12–O3	–17.0(4)
C4–C3–N2–Cu1	–41.0(3)	N3–C11–C12–O4	166.0(3)
C5–C6–N4–C7	–88.1(3)	N4–C7–C8–N1	61.1(4)
C5–C6–N4–C15	126.4(3)	N4–C15–N5–C16	–179.6(3)
C6–C5–N3–C4	167.7(3)	N4–C15–N7–C17	176.1(3)
C6–C5–N3–C11	–71.3(3)	N5–C15–N4–C6	153.1(3)
C6–C5–N3–Cu1	46.0(3)	N5–C15–N4–C7	8.1(4)
C7–C8–N1–C1	–161.0(3)	N5–C15–N7–C17	–3.9(5)
C7–C8–N1–C9	76.6(3)	N5–C16–N6–C17	–3.2(4)
C7–C8–N1–Cu1	–39.9(3)	N5–C16–N8–C18	–8.7(4)
C8–C7–N4–C6	65.5(4)	N5–C16–N8–C21	–165.2(3)
C8–C7–N4–C15	–149.1(3)	N6–C16–N5–C15	3.5(4)
C9–C10–O1–Cu1	12.2(4)	N6–C16–N8–C18	171.2(3)
C10–C9–N1–C1	140.4(3)	N6–C16–N8–C21	14.7(4)
C10–C9–N1–C8	–98.3(3)	N6–C17–N7–C15	4.2(5)
C10–C9–N1–Cu1	23.2(3)	N6–C17–N9–C22	–4.0(5)
C11–C12–O3–Cu1	4.4(4)	N6–C17–N9–C25	175.6(3)
C12–C11–N3–C4	–98.7(3)	N7–C15–N4–C6	–26.9(4)
C12–C11–N3–C5	141.5(3)	N7–C15–N4–C7	–171.9(3)
C12–C11–N3–Cu1	19.0(3)	N7–C15–N5–C16	0.4(5)
C14–C13–N2–C2	74.0(4)	N7–C17–N6–C16	–1.0(5)
C14–C13–N2–C3	–56.3(4)	N7–C17–N9–C22	175.9(3)
C14–C13–N2–Cu1	–167.4(2)	N7–C17–N9–C25	–4.5(5)
C18–C19–O7–C20	–62.9(4)	N8–C16–N5–C15	–176.5(3)
C19–C18–N8–C16	156.4(3)	N8–C16–N6–C17	176.8(3)
C19–C18–N8–C21	–46.0(4)	N8–C18–C19–O7	53.4(4)
C20–C21–N8–C16	–155.5(3)	N9–C17–N6–C16	178.9(3)
C20–C21–N8–C18	47.0(4)	N9–C17–N7–C15	–175.6(3)
C21–C20–O7–C19	62.8(4)	N9–C22–C23–O8	–55.9(4)
C22–C23–O8–C24	60.4(4)	O2–C10–O1–Cu1	–171.5(3)
C23–C22–N9–C17	–127.9(3)	O4–C12–O3–Cu1	–178.7(2)
C23–C22–N9–C25	52.4(4)	O7–C20–C21–N8	–54.0(4)
		O8–C24–C25–N9	54.5(4)

A.2 – Nickel(II) crystal structure of TzMorph2DO3A

Table 1. Crystal data and structure refinement details.

Identification code	**2014ncs0932 (MM160)**	
Empirical formula	$C_{56}H_{92}N_{18}Ni_2O_{21.51}$	
Formula weight	1479.01	
Temperature	100.15 K	
Wavelength	0.71075 Å	
Crystal system	Orthorhombic	
Space group	*Pbc*21	
Unit cell dimensions	a = 32.069(2) Å	α = 90°
	b = 16.3830(4) Å	β = 90°
	c = 12.8570(3) Å	γ = 90°
Volume	6754.9(5) Å^3	
Z	4	
Density (calculated)	1.454 Mg / m^3	
Absorption coefficient	0.645 mm^{-1}	
F(000)	3128	
Crystal	Long column; light purple	
Crystal size	0.45 × 0.05 × 0.035 mm^3	
θ range for data collection	3.016 – 27.483°	
Index ranges	$-41 \le h \le 41$, $-21 \le k \le 20$, $-16 \le l \le 16$	
Reflections collected	57826	
Independent reflections	14090 [R_{int} = 0.0509]	
Completeness to θ = 25.242°	99.8 %	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	14090 / 1 / 884	
Goodness-of-fit on F^2	1.170	
Final R indices [$F^2 > 2\sigma(F^2)$]	*R1* = 0.0436, *wR2* = 0.1059	
R indices (all data)	*R1* = 0.0551, *wR2* = 0.1099	
Absolute structure parameter	0.140(13)	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.889 and –0.347 e Å^{-3}	

Diffractometer: Rigaku AFC12 goniometer equipped with an enhanced sensitivity (HG) Saturn724+ detector mounted at the window of an FR-E+ SuperBright molybdenum rotating anode generator with HF Varimax optics (100m focus). **Cell determination and data collection:** CrystalClear-SM Expert 3.1 b27 (Rigaku, 2012). **Data reduction, cell refinement and absorption correction:**CrystalClear-SM Expert 3.1 b18 (Rigaku, 2012)**Structure solution:** SUPERFLIP (Palatinus, L. & Chapuis, G. (2007). J. Appl. Cryst. 40, 786-790). **Structure refinement:** SHELXL-2013 (Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122).

Special details:
In the crystal structure disordered and partially occupied water molecule has been modelled over two sites with 75:25 ratio. Asymmetric part contains two independent molecules (Z'=2).

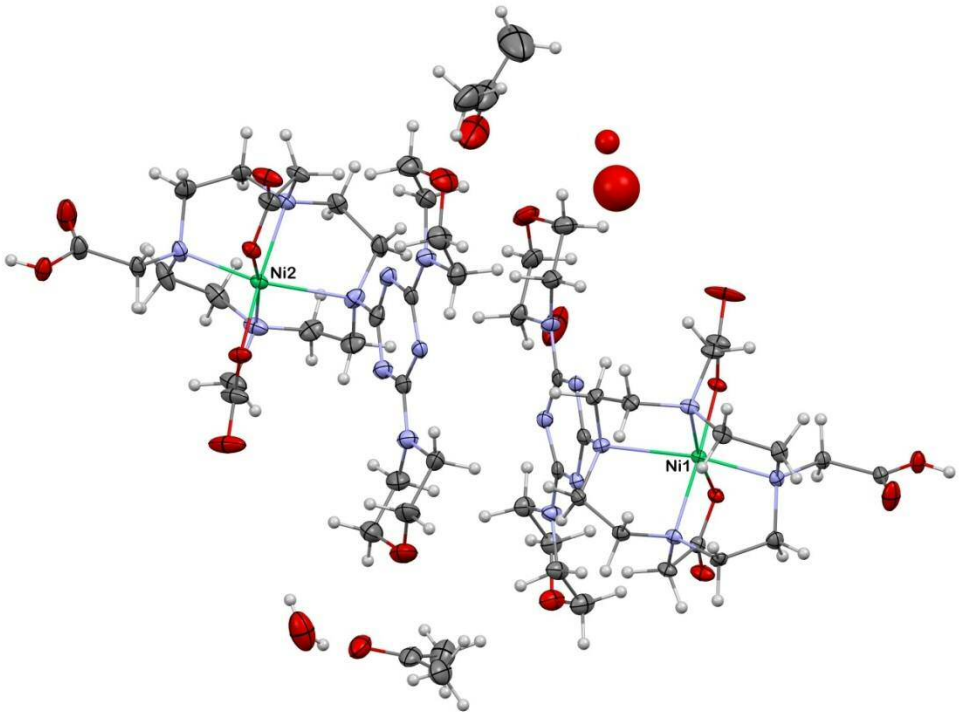

Figure 1. Molecular structure of 2014ncs0932 (MM160). Displacement ellipsoids 50% probability.

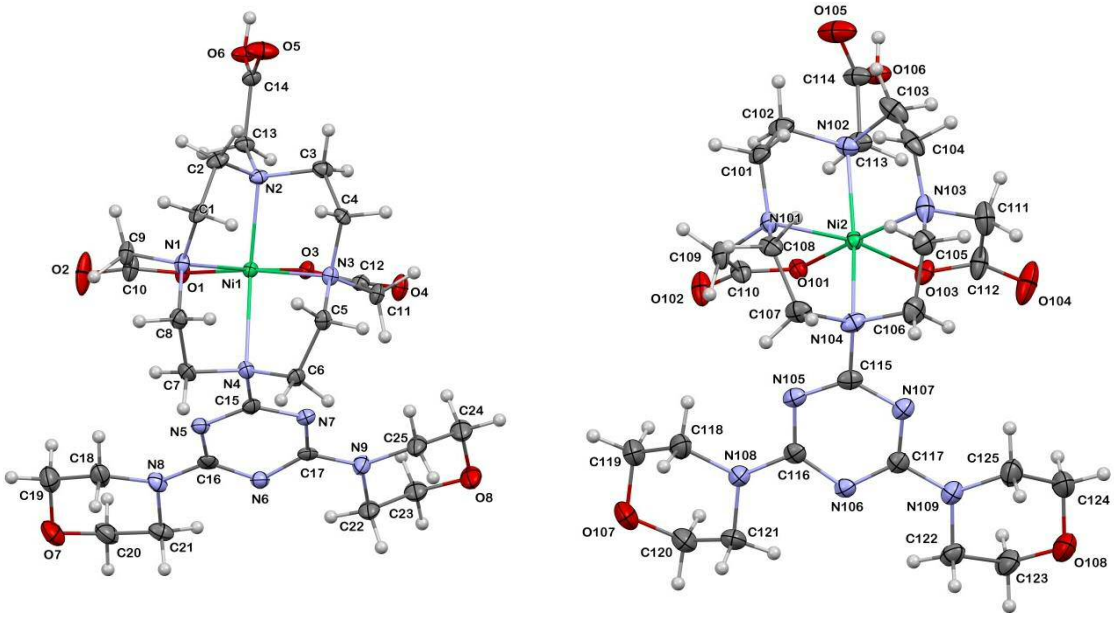

Figure 2. Two independent molecules present in the asymmetric part of the unit cell (Z'=2) and atom labelling scheme. Solvent molecules are omitted for clarity.

Table 1. Atomic coordinates [$\times 10^4$], equivalent isotropic displacement parameters [$Å^2 \times 10^3$] and site occupancy factors. U_{eq} is defined as one third of the trace of the orthogonalized U^{ij} tensor.

Atom	x	y	z	U_{eq}	S.o.f.
C1	3704(1)	1540(3)	3851(3)	18(1)	1
C2	4062(1)	1105(3)	4417(4)	20(1)	1
C3	4639(1)	2058(3)	4840(3)	19(1)	1
C4	4521(1)	2745(3)	4124(3)	16(1)	1
C5	3963(1)	3747(3)	3868(3)	17(1)	1
C6	3589(1)	4166(3)	4375(3)	18(1)	1
C7	3011(1)	3221(3)	4804(4)	20(1)	1
C8	3155(1)	2557(3)	4053(4)	19(1)	1
C9	3188(1)	1400(3)	5250(3)	20(1)	1
C10	3199(1)	1607(3)	6402(4)	23(1)	1
C11	4441(1)	3897(3)	5326(3)	18(1)	1
C12	4445(1)	3636(3)	6453(3)	15(1)	1
C13	4358(1)	1148(3)	6166(4)	16(1)	1
C14	4673(1)	465(2)	6038(3)	16(1)	1
C15	3289(1)	4128(2)	6129(3)	16(1)	1
C16	2864(1)	4403(2)	7466(4)	18(1)	1
C17	3457(1)	5121(2)	7257(3)	16(1)	1
C18	2202(1)	3671(3)	7607(4)	29(1)	1
C19	1937(2)	3308(3)	8455(4)	35(1)	1
C20	2080(1)	4379(3)	9587(4)	32(1)	1
C21	2363(1)	4802(3)	8824(4)	24(1)	1
C22	3620(1)	6251(3)	8442(4)	24(1)	1
C23	3993(1)	6423(3)	9120(4)	26(1)	1
C24	4451(1)	6112(3)	7790(4)	32(1)	1
C25	4103(1)	5921(3)	7053(4)	26(1)	1
C26	888(2)	4018(4)	10868(7)	71(2)	1
C27	697(3)	3380(5)	11584(8)	96(3)	1
C28	792(2)	3893(4)	9733(6)	68(2)	1
C101	376(1)	7300(3)	8853(4)	24(1)	1
C102	231(1)	7937(3)	8058(4)	27(1)	1
C103	738(2)	9057(3)	8409(4)	32(1)	1
C104	1095(2)	8758(3)	9106(4)	29(1)	1
C105	1683(2)	7828(3)	9197(4)	28(1)	1
C106	1899(2)	7176(3)	8565(4)	31(1)	1
C107	1377(1)	6067(3)	8813(4)	27(1)	1
C108	959(1)	6394(3)	9215(3)	22(1)	1
C109	544(1)	6171(3)	7653(4)	24(1)	1
C110	567(1)	6429(3)	6511(4)	21(1)	1
C111	1669(2)	8899(3)	7881(4)	35(1)	1
C112	1713(2)	8637(3)	6738(4)	32(1)	1
C113	493(1)	8797(3)	6654(4)	22(1)	1
C114	146(2)	9429(3)	6697(4)	28(1)	1
C115	1752(1)	6186(3)	7177(3)	21(1)	1
C116	1701(1)	5186(3)	5995(4)	22(1)	1
C117	2248(1)	6040(3)	5967(4)	22(1)	1
C118	1126(1)	4189(3)	6120(5)	32(1)	1
C119	818(2)	3874(3)	5309(5)	37(1)	1
C120	1346(2)	3703(3)	4073(4)	37(1)	1
C121	1683(2)	4006(3)	4804(4)	31(1)	1
C122	2814(1)	5865(3)	4696(4)	28(1)	1
C123	3016(2)	6445(3)	3934(5)	39(1)	1
C124	3055(2)	7482(3)	5186(5)	38(1)	1
C125	2860(2)	6955(3)	6019(4)	34(1)	1
C126	4143(2)	8908(3)	7953(5)	35(1)	1
C127	4320(2)	8378(3)	7116(5)	41(1)	1
C128	4301(2)	8798(4)	9014(5)	46(2)	1
N1	3431(1)	1972(2)	4593(3)	16(1)	1

N2	4260(1)	1623(2)	5228(3)	15(1)	1
N3	4222(1)	3314(2)	4639(3)	14(1)	1
N4	3373(1)	3679(2)	5206(3)	17(1)	1
N5	2938(1)	3936(2)	6628(3)	16(1)	1
N6	3099(1)	5025(2)	7799(3)	19(1)	1
N7	3565(1)	4693(2)	6403(3)	16(1)	1
N8	2517(1)	4216(2)	8042(3)	20(1)	1
N9	3731(1)	5685(2)	7624(3)	22(1)	1
N101	711(1)	6794(2)	8382(3)	19(1)	1
N102	597(1)	8425(2)	7677(3)	22(1)	1
N103	1416(1)	8320(2)	8521(3)	26(1)	1
N104	1592(1)	6611(3)	8052(3)	23(1)	1
N105	1523(1)	5580(2)	6806(3)	21(1)	1
N106	2063(1)	5387(2)	5545(3)	20(1)	1
N107	2111(1)	6451(2)	6809(3)	23(1)	1
N108	1492(1)	4534(2)	5596(3)	24(1)	1
N109	2606(1)	6315(2)	5525(3)	25(1)	1
Ni1	3837(1)	2596(1)	5595(1)	12(1)	1
Ni2	1093(1)	7566(1)	7495(1)	16(1)	1
O1	3487(1)	2081(2)	6702(2)	16(1)	1
O2	2934(1)	1285(2)	6978(3)	46(1)	1
O3	4204(1)	3056(2)	6725(2)	15(1)	1
O4	4676(1)	4011(2)	7084(2)	23(1)	1
O5	4813(1)	242(2)	5212(3)	34(1)	1
O6	4769(1)	140(2)	6941(2)	21(1)	1
O7	1755(1)	3936(2)	9093(3)	38(1)	1
O8	4339(1)	6716(2)	8529(3)	31(1)	1
O9	1097(2)	4591(3)	11195(6)	85(2)	1
O10	3468(2)	9474(3)	5845(3)	62(1)	1
O101	804(1)	7021(2)	6308(2)	19(1)	1
O102	371(1)	6038(2)	5844(2)	33(1)	1
O103	1453(1)	8112(2)	6423(2)	23(1)	1
O104	1977(1)	8982(3)	6204(3)	50(1)	1
O105	−82(1)	9546(2)	7435(3)	45(1)	1
O106	110(1)	9813(2)	5820(2)	25(1)	1
O107	1010(1)	3314(2)	4600(3)	46(1)	1
O108	3286(1)	7007(2)	4453(3)	45(1)	1
O109	3876(1)	9437(2)	7758(3)	40(1)	1
O110	2708(1)	5370(3)	11858(5)	83(2)	1
O11	2058(3)	3485(7)	12237(9)	135(5)	0.695(10)
O12	1672(4)	2867(7)	11147(10)	38(4)	0.305(10)

Table 2. Bond lengths [Å] and angles [°].

C1–H1A	0.9900	C19–H19A	0.9900
C1–H1B	0.9900	C19–H19B	0.9900
C1–C2	1.533(6)	C19–O7	1.440(6)
C1–N1	1.477(5)	C20–H20A	0.9900
C2–H2A	0.9900	C20–H20B	0.9900
C2–H2B	0.9900	C20–C21	1.506(6)
C2–N2	1.487(5)	C20–O7	1.422(6)
C3–H3A	0.9900	C21–H21A	0.9900
C3–H3B	0.9900	C21–H21B	0.9900
C3–C4	1.502(6)	C21–N8	1.475(6)
C3–N2	1.496(5)	C22–H22A	0.9900
C4–H4A	0.9900	C22–H22B	0.9900
C4–H4B	0.9900	C22–C23	1.508(6)
C4–N3	1.493(5)	C22–N9	1.446(6)
C5–H5A	0.9900	C23–H23A	0.9900
C5–H5B	0.9900	C23–H23B	0.9900
C5–C6	1.528(6)	C23–O8	1.426(6)
C5–N3	1.476(5)	C24–H24A	0.9900
C6–H6A	0.9900	C24–H24B	0.9900
C6–H6B	0.9900	C24–C25	1.498(7)
C6–N4	1.502(5)	C24–O8	1.418(6)
C7–H7A	0.9900	C25–H25A	0.9900
C7–H7B	0.9900	C25–H25B	0.9900
C7–C8	1.527(6)	C25–N9	1.451(5)
C7–N4	1.477(5)	C26–C27	1.521(11)
C8–H8A	0.9900	C26–C28	1.505(11)
C8–H8B	0.9900	C26–O9	1.229(7)
C8–N1	1.478(5)	C27–H27A	0.9800
C9–H9A	0.9900	C27–H27B	0.9800
C9–H9B	0.9900	C27–H27C	0.9800
C9–C10	1.521(7)	C28–H28A	0.9800
C9–N1	1.483(5)	C28–H28B	0.9800
C10–O1	1.267(5)	C28–H28C	0.9800
C10–O2	1.244(5)	C101–H10A	0.9900
C11–H11A	0.9900	C101–H10B	0.9900
C11–H11B	0.9900	C101–C102	1.533(7)
C11–C12	1.511(6)	C101–N101	1.486(6)
C11–N3	1.477(5)	C102–H10C	0.9900
C12–O3	1.272(5)	C102–H10D	0.9900
C12–O4	1.260(5)	C102–N102	1.503(6)
C13–H13A	0.9900	C103–H10E	0.9900
C13–H13B	0.9900	C103–H10F	0.9900
C13–C14	1.517(5)	C103–C104	1.534(7)
C13–N2	1.470(5)	C103–N102	1.471(6)
C14–O5	1.208(5)	C104–H10G	0.9900
C14–O6	1.315(5)	C104–H10H	0.9900
C15–N4	1.420(6)	C104–N103	1.463(6)
C15–N5	1.334(5)	C105–H10I	0.9900
C15–N7	1.329(5)	C105–H10J	0.9900
C16–N5	1.343(6)	C105–C106	1.510(7)
C16–N6	1.339(5)	C105–N103	1.461(6)
C16–N8	1.371(5)	C106–H10K	0.9900
C17–N6	1.351(5)	C106–H10L	0.9900
C17–N7	1.347(5)	C106–N104	1.505(6)
C17–N9	1.360(5)	C107–H10M	0.9900
C18–H18A	0.9900	C107–H10N	0.9900
C18–H18B	0.9900	C107–C108	1.534(6)
C18–C19	1.506(7)	C107–N104	1.492(6)
C18–N8	1.460(6)	C108–H10O	0.9900

C108–H10P	0.9900
C108–N101	1.486(6)
C109–H10Q	0.9900
C109–H10R	0.9900
C109–C110	1.529(7)
C109–N101	1.484(6)
C110–O101	1.259(5)
C110–O102	1.242(5)
C111–H11C	0.9900
C111–H11D	0.9900
C111–C112	1.538(8)
C111–N103	1.495(6)
C112–O103	1.264(6)
C112–O104	1.228(6)
C113–H11E	0.9900
C113–H11F	0.9900
C113–C114	1.520(6)
C113–N102	1.488(6)
C114–O105	1.214(6)
C114–O106	1.296(6)
C115–N104	1.420(6)
C115–N105	1.325(6)
C115–N107	1.318(6)
C116–N105	1.353(6)
C116–N106	1.337(5)
C116–N108	1.361(6)
C117–N106	1.338(6)
C117–N107	1.348(6)
C117–N109	1.357(6)
C118–H11G	0.9900
C118–H11H	0.9900
C118–C119	1.526(7)
C118–N108	1.466(6)
C119–H11I	0.9900
C119–H11J	0.9900
C119–O107	1.432(6)
C120–H12A	0.9900
C120–H12B	0.9900
C120–C121	1.515(7)
C120–O107	1.425(6)
C121–H12C	0.9900
C121–H12D	0.9900
C121–N108	1.469(6)
C122–H12E	0.9900
C122–H12F	0.9900
C122–C123	1.510(7)
C122–N109	1.458(6)
C123–H12G	0.9900
C123–H12H	0.9900
C123–O108	1.431(7)
C124–H12I	0.9900
C124–H12J	0.9900
C124–C125	1.512(7)
C124–O108	1.429(7)
C125–H12K	0.9900
C125–H12L	0.9900
C125–N109	1.472(6)
C126–C127	1.496(7)
C126–C128	1.467(8)
C126–O109	1.243(6)
C127–H12M	0.9800
C127–H12N	0.9800
C127–H12O	0.9800
C128–H12P	0.9800
C128–H12Q	0.9800
C128–H12R	0.9800
N1–Ni1	2.098(3)
N2–Ni1	2.144(3)
N3–Ni1	2.103(3)
N4–Ni1	2.370(3)
N101–Ni2	2.099(4)
N102–Ni2	2.137(3)
N103–Ni2	2.083(4)
N104–Ni2	2.349(4)
Ni1–O1	2.000(3)
Ni1–O3	2.016(3)
Ni2–O101	1.997(3)
Ni2–O103	2.007(3)
O6–H6	0.8400
O10–H10S	0.8498
O10–H10T	0.8503
O106–H106	0.8400
H1A–C1–H1B	108.0
C2–C1–H1A	109.4
C2–C1–H1B	109.4
N1–C1–H1A	109.4
N1–C1–H1B	109.4
N1–C1–C2	111.1(4)
C1–C2–H2A	109.0
C1–C2–H2B	109.0
H2A–C2–H2B	107.8
N2–C2–C1	112.8(3)
N2–C2–H2A	109.0
N2–C2–H2B	109.0
H3A–C3–H3B	108.1
C4–C3–H3A	109.5
C4–C3–H3B	109.5
N2–C3–H3A	109.5
N2–C3–H3B	109.5
N2–C3–C4	110.9(3)
C3–C4–H4A	109.4
C3–C4–H4B	109.4
H4A–C4–H4B	108.0
N3–C4–C3	111.0(3)
N3–C4–H4A	109.4
N3–C4–H4B	109.4
H5A–C5–H5B	107.9
C6–C5–H5A	109.2
C6–C5–H5B	109.2
N3–C5–H5A	109.2
N3–C5–H5B	109.2
N3–C5–C6	111.9(4)
C5–C6–H6A	108.5
C5–C6–H6B	108.5
H6A–C6–H6B	107.5
N4–C6–C5	115.2(3)
N4–C6–H6A	108.5
N4–C6–H6B	108.5
H7A–C7–H7B	108.1

C8–C7–H7A	109.6
C8–C7–H7B	109.6
N4–C7–H7A	109.6
N4–C7–H7B	109.6
N4–C7–C8	110.2(3)
C7–C8–H8A	109.6
C7–C8–H8B	109.6
H8A–C8–H8B	108.1
N1–C8–C7	110.2(4)
N1–C8–H8A	109.6
N1–C8–H8B	109.6
H9A–C9–H9B	107.7
C10–C9–H9A	108.8
C10–C9–H9B	108.8
N1–C9–H9A	108.8
N1–C9–H9B	108.8
N1–C9–C10	113.6(3)
O1–C10–C9	116.7(4)
O2–C10–C9	118.0(4)
O2–C10–O1	125.2(4)
H11A–C11–H11B	107.8
C12–C11–H11A	108.9
C12–C11–H11B	108.9
N3–C11–H11A	108.9
N3–C11–H11B	108.9
N3–C11–C12	113.2(3)
O3–C12–C11	118.1(4)
O4–C12–C11	119.0(4)
O4–C12–O3	123.0(4)
H13A–C13–H13B	107.4
C14–C13–H13A	108.2
C14–C13–H13B	108.2
N2–C13–H13A	108.2
N2–C13–H13B	108.2
N2–C13–C14	116.3(4)
O5–C14–C13	124.4(4)
O5–C14–O6	124.6(4)
O6–C14–C13	111.0(4)
N5–C15–N4	116.1(3)
N7–C15–N4	117.2(4)
N7–C15–N5	126.8(4)
N5–C16–N8	116.8(4)
N6–C16–N5	126.2(4)
N6–C16–N8	117.1(4)
N6–C17–N9	116.7(4)
N7–C17–N6	125.3(4)
N7–C17–N9	118.0(4)
H18A–C18–H18B	108.1
C19–C18–H18A	109.5
C19–C18–H18B	109.5
N8–C18–H18A	109.5
N8–C18–H18B	109.5
N8–C18–C19	110.8(4)
C18–C19–H19A	109.4
C18–C19–H19B	109.4
H19A–C19–H19B	108.0
O7–C19–C18	111.0(4)
O7–C19–H19A	109.4
O7–C19–H19B	109.4
H20A–C20–H20B	107.8
C21–C20–H20A	109.0
C21–C20–H20B	109.0
O7–C20–H20A	109.0
O7–C20–H20B	109.0
O7–C20–C21	112.8(4)
C20–C21–H21A	109.6
C20–C21–H21B	109.6
H21A–C21–H21B	108.1
N8–C21–C20	110.2(4)
N8–C21–H21A	109.6
N8–C21–H21B	109.6
H22A–C22–H22B	108.1
C23–C22–H22A	109.6
C23–C22–H22B	109.6
N9–C22–H22A	109.6
N9–C22–H22B	109.6
N9–C22–C23	110.2(4)
C22–C23–H23A	109.3
C22–C23–H23B	109.3
H23A–C23–H23B	107.9
O8–C23–C22	111.8(4)
O8–C23–H23A	109.3
O8–C23–H23B	109.3
H24A–C24–H24B	107.9
C25–C24–H24A	109.1
C25–C24–H24B	109.1
O8–C24–H24A	109.1
O8–C24–H24B	109.1
O8–C24–C25	112.4(4)
C24–C25–H25A	109.6
C24–C25–H25B	109.6
H25A–C25–H25B	108.1
N9–C25–C24	110.3(4)
N9–C25–H25A	109.6
N9–C25–H25B	109.6
C28–C26–C27	114.3(6)
O9–C26–C27	122.5(8)
O9–C26–C28	123.1(8)
C26–C27–H27A	109.5
C26–C27–H27B	109.5
C26–C27–H27C	109.5
H27A–C27–H27B	109.5
H27A–C27–H27C	109.5
H27B–C27–H27C	109.5
C26–C28–H28A	109.5
C26–C28–H28B	109.5
C26–C28–H28C	109.5
H28A–C28–H28B	109.5
H28A–C28–H28C	109.5
H28B–C28–H28C	109.5
H10A–C101–H10B	108.3
C102–C101–H10A	109.9
C102–C101–H10B	109.9
N101–C101–H10A	109.9
N101–C101–H10B	109.9
N101–C101–C102	109.1(4)
C101–C102–H10C	109.7
C101–C102–H10D	109.7
H10C–C102–H10D	108.2
N102–C102–C101	110.0(4)

N102–C102–H10C	109.7
N102–C102–H10D	109.7
H10E–C103–H10F	107.9
C104–C103–H10E	109.2
C104–C103–H10F	109.2
N102–C103–H10E	109.2
N102–C103–H10F	109.2
N102–C103–C104	112.2(4)
C103–C104–H10G	109.1
C103–C104–H10H	109.1
H10G–C104–H10H	107.9
N103–C104–C103	112.4(4)
N103–C104–H10G	109.1
N103–C104–H10H	109.1
H10I–C105–H10J	108.2
C106–C105–H10I	109.7
C106–C105–H10J	109.7
N103–C105–H10I	109.7
N103–C105–H10J	109.7
N103–C105–C106	109.8(4)
C105–C106–H10K	109.3
C105–C106–H10L	109.3
H10K–C106–H10L	107.9
N104–C106–C105	111.7(4)
N104–C106–H10K	109.3
N104–C106–H10L	109.3
H10M–C107–H10N	107.6
C108–C107–H10M	108.6
C108–C107–H10N	108.6
N104–C107–H10M	108.6
N104–C107–H10N	108.6
N104–C107–C108	114.5(4)
C107–C108–H10O	109.1
C107–C108–H10P	109.1
H10O–C108–H10P	107.9
N101–C108–C107	112.3(4)
N101–C108–H10O	109.1
N101–C108–H10P	109.1
H10Q–C109–H10R	107.7
C110–C109–H10Q	108.9
C110–C109–H10R	108.9
N101–C109–H10Q	108.9
N101–C109–H10R	108.9
N101–C109–C110	113.5(3)
O101–C110–C109	116.1(4)
O102–C110–C109	119.8(4)
O102–C110–O101	124.0(4)
H11C–C111–H11D	107.7
C112–C111–H11C	108.9
C112–C111–H11D	108.9
N103–C111–H11C	108.9
N103–C111–H11D	108.9
N103–C111–C112	113.4(4)
O103–C112–C111	115.8(4)
O104–C112–C111	117.9(5)
O104–C112–O103	126.1(5)
H11E–C113–H11F	107.6
C114–C113–H11E	108.7
C114–C113–H11F	108.7
N102–C113–H11E	108.7
N102–C113–H11F	108.7
N102–C113–C114	114.3(4)
O105–C114–C113	125.3(4)
O105–C114–O106	123.3(4)
O106–C114–C113	111.4(4)
N105–C115–N104	116.8(4)
N107–C115–N104	116.1(4)
N107–C115–N105	127.1(4)
N105–C116–N108	117.2(4)
N106–C116–N105	125.6(4)
N106–C116–N108	117.2(4)
N106–C117–N107	125.5(4)
N106–C117–N109	118.1(4)
N107–C117–N109	116.4(4)
H11G–C118–H11H	108.2
C119–C118–H11G	109.8
C119–C118–H11H	109.8
N108–C118–H11G	109.8
N108–C118–H11H	109.8
N108–C118–C119	109.6(4)
C118–C119–H11I	109.2
C118–C119–H11J	109.2
H11I–C119–H11J	107.9
O107–C119–C118	112.0(4)
O107–C119–H11I	109.2
O107–C119–H11J	109.2
H12A–C120–H12B	107.8
C121–C120–H12A	109.0
C121–C120–H12B	109.0
O107–C120–H12A	109.0
O107–C120–H12B	109.0
O107–C120–C121	113.1(5)
C120–C121–H12C	109.9
C120–C121–H12D	109.9
H12C–C121–H12D	108.3
N108–C121–C120	109.0(4)
N108–C121–H12C	109.9
N108–C121–H12D	109.9
H12E–C122–H12F	108.1
C123–C122–H12E	109.5
C123–C122–H12F	109.5
N109–C122–H12E	109.5
N109–C122–H12F	109.5
N109–C122–C123	110.7(4)
C122–C123–H12G	109.4
C122–C123–H12H	109.4
H12G–C123–H12H	108.0
O108–C123–C122	111.2(5)
O108–C123–H12G	109.4
O108–C123–H12H	109.4
H12I–C124–H12J	107.9
C125–C124–H12I	109.3
C125–C124–H12J	109.3
O108–C124–H12I	109.3
O108–C124–H12J	109.3
O108–C124–C125	111.8(4)
C124–C125–H12K	109.8
C124–C125–H12L	109.8
H12K–C125–H12L	108.3
N109–C125–C124	109.3(4)

N109–C125–H12K	109.8
N109–C125–H12L	109.8
C128–C126–C127	117.7(5)
O109–C126–C127	121.4(5)
O109–C126–C128	120.8(5)
C126–C127–H12M	109.5
C126–C127–H12N	109.5
C126–C127–H12O	109.5
H12M–C127–H12N	109.5
H12M–C127–H12O	109.5
H12N–C127–H12O	109.5
C126–C128–H12P	109.5
C126–C128–H12Q	109.5
C126–C128–H12R	109.5
H12P–C128–H12Q	109.5
H12P–C128–H12R	109.5
H12Q–C128–H12R	109.5
C1–N1–C8	111.3(3)
C1–N1–C9	112.1(3)
C1–N1–Ni1	105.1(2)
C8–N1–C9	111.3(3)
C8–N1–Ni1	110.2(3)
C9–N1–Ni1	106.6(2)
C2–N2–C3	112.7(3)
C2–N2–Ni1	107.9(2)
C3–N2–Ni1	103.5(2)
C13–N2–C2	111.4(3)
C13–N2–C3	110.6(3)
C13–N2–Ni1	110.4(2)
C4–N3–Ni1	106.7(2)
C5–N3–C4	111.4(3)
C5–N3–C11	111.0(3)
C5–N3–Ni1	109.3(2)
C11–N3–C4	111.3(3)
C11–N3–Ni1	107.0(2)
C6–N4–Ni1	105.0(2)
C7–N4–C6	112.5(3)
C7–N4–Ni1	100.8(2)
C15–N4–C6	114.0(3)
C15–N4–C7	113.9(3)
C15–N4–Ni1	109.3(2)
C15–N5–C16	113.7(4)
C16–N6–C17	113.8(4)
C15–N7–C17	114.1(4)
C16–N8–C18	119.4(4)
C16–N8–C21	119.6(4)
C18–N8–C21	115.4(3)
C17–N9–C22	121.9(3)
C17–N9–C25	122.4(4)
C22–N9–C25	113.5(3)
C101–N101–Ni2	107.9(3)
C108–N101–C101	109.8(4)
C108–N101–C109	110.1(3)
C108–N101–Ni2	110.1(3)
C109–N101–C101	112.4(4)
C109–N101–Ni2	106.4(3)
C102–N102–Ni2	105.5(3)
C103–N102–C102	113.9(4)
C103–N102–C113	110.3(3)
C103–N102–Ni2	107.8(3)
C113–N102–C102	109.3(4)
C113–N102–Ni2	109.9(3)
C104–N103–C105	112.1(4)
C104–N103–C111	110.7(4)
C104–N103–Ni2	105.5(3)
C105–N103–C111	111.1(4)
C105–N103–Ni2	109.9(3)
C111–N103–Ni2	107.3(3)
C106–N104–Ni2	99.7(3)
C107–N104–C106	112.5(4)
C107–N104–Ni2	106.5(3)
C115–N104–C106	114.3(4)
C115–N104–C107	113.2(4)
C115–N104–Ni2	109.4(3)
C115–N105–C116	113.6(4)
C116–N106–C117	114.0(4)
C115–N107–C117	114.1(4)
C116–N108–C118	121.6(4)
C116–N108–C121	121.2(4)
C121–N108–C118	115.1(4)
C117–N109–C122	121.7(4)
C117–N109–C125	121.6(4)
C122–N109–C125	114.9(4)
N1–Ni1–N2	84.05(14)
N1–Ni1–N3	106.20(14)
N1–Ni1–N4	81.08(13)
N2–Ni1–N4	155.11(14)
N3–Ni1–N2	85.18(12)
N3–Ni1–N4	79.98(13)
O1–Ni1–N1	83.25(12)
O1–Ni1–N2	101.50(12)
O1–Ni1–N3	169.09(13)
O1–Ni1–N4	96.46(12)
O1–Ni1–O3	88.47(12)
O3–Ni1–N1	170.86(13)
O3–Ni1–N2	93.86(12)
O3–Ni1–N3	82.43(13)
O3–Ni1–N4	103.78(12)
N101–Ni2–N102	84.39(14)
N101–Ni2–N104	80.23(14)
N102–Ni2–N104	155.78(14)
N103–Ni2–N101	107.71(16)
N103–Ni2–N102	84.85(15)
N103–Ni2–N104	82.17(15)
O101–Ni2–N101	82.71(14)
O101–Ni2–N102	91.81(14)
O101–Ni2–N103	168.62(15)
O101–Ni2–N104	104.58(13)
O101–Ni2–O103	86.69(13)
O103–Ni2–N101	167.78(15)
O103–Ni2–N102	102.06(13)
O103–Ni2–N103	83.39(14)
O103–Ni2–N104	96.66(13)
C10–O1–Ni1	116.9(3)
C12–O3–Ni1	115.8(3)
C14–O6–H6	109.5
C20–O7–C19	108.8(3)
C24–O8–C23	108.6(3)
H10S–O10–H10T	109.5
C110–O101–Ni2	117.8(3)

C112–O103–Ni2	117.5(3)	C120–O107–C119	109.9(4)
C114–O106–H106	109.5	C124–O108–C123	110.1(4)

Table 3. Anisotropic displacement parameters [$Å^2 \times 10^3$].

The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}]$.

Atom	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C1	24(2)	15(2)	15(2)	–7(2)	1(2)	–2(2)
C2	28(2)	16(2)	16(2)	–4(2)	1(2)	3(2)
C3	19(2)	21(2)	18(3)	1(2)	4(2)	3(2)
C4	13(2)	19(2)	16(2)	–1(2)	7(2)	1(2)
C5	18(2)	19(2)	13(2)	3(2)	0(2)	–2(2)
C6	19(2)	16(2)	18(2)	4(2)	–1(2)	0(2)
C7	13(2)	22(2)	23(3)	1(2)	–4(2)	0(2)
C8	17(2)	21(2)	18(2)	0(2)	–5(2)	–2(2)
C9	22(2)	21(2)	18(3)	–4(2)	3(2)	–10(2)
C10	26(2)	24(2)	19(3)	0(2)	0(2)	–13(2)
C11	19(2)	16(2)	18(3)	–1(2)	–1(2)	–6(2)
C12	17(2)	14(2)	14(2)	–3(2)	3(2)	1(2)
C13	19(2)	17(2)	14(2)	0(2)	3(2)	3(2)
C14	19(2)	14(2)	16(2)	1(2)	0(2)	2(2)
C15	18(2)	13(2)	18(2)	4(2)	–2(2)	1(2)
C16	18(2)	16(2)	19(2)	9(2)	–1(2)	7(2)
C17	18(2)	12(2)	16(2)	2(2)	0(2)	6(2)
C18	20(2)	32(2)	34(3)	10(2)	–2(2)	–3(2)
C19	25(2)	36(3)	43(3)	–1(3)	7(2)	–7(2)
C20	21(2)	37(3)	37(3)	3(2)	7(2)	6(2)
C21	23(2)	29(3)	19(2)	1(2)	3(2)	8(2)
C22	25(2)	20(2)	28(3)	–9(2)	6(2)	–1(2)
C23	29(2)	23(2)	27(3)	–8(2)	3(2)	1(2)
C24	28(2)	26(3)	42(3)	–14(2)	9(2)	–2(2)
C25	27(2)	20(2)	32(3)	–9(2)	11(2)	–4(2)
C26	43(4)	42(4)	126(8)	–23(4)	–20(4)	0(3)
C27	92(6)	66(5)	131(8)	13(5)	–45(6)	–27(5)
C28	35(3)	52(4)	117(7)	–21(4)	12(4)	6(3)
C101	21(2)	26(2)	23(3)	4(2)	10(2)	2(2)
C102	26(2)	32(3)	24(3)	2(2)	9(2)	5(2)
C103	57(3)	19(2)	19(3)	2(2)	–5(2)	4(2)
C104	51(3)	19(2)	17(3)	–6(2)	–2(2)	–6(2)
C105	26(2)	35(3)	24(3)	–7(2)	–6(2)	–3(2)
C106	25(2)	38(3)	29(3)	–3(2)	–8(2)	–5(2)
C107	30(2)	25(3)	25(3)	8(2)	2(2)	8(2)
C108	31(2)	22(2)	13(2)	0(2)	4(2)	2(2)
C109	30(2)	23(2)	19(3)	5(2)	0(2)	–11(2)
C110	29(2)	22(2)	13(2)	–2(2)	3(2)	–6(2)
C111	39(3)	34(3)	33(3)	7(3)	–4(2)	–17(2)
C112	30(3)	42(3)	23(3)	10(2)	–1(2)	–18(2)
C113	28(2)	21(2)	18(2)	6(2)	5(2)	7(2)
C114	34(3)	25(2)	25(3)	4(2)	8(2)	11(2)
C115	21(2)	26(2)	17(3)	0(2)	–5(2)	7(2)
C116	23(2)	18(2)	23(3)	9(2)	1(2)	5(2)
C117	21(2)	22(2)	22(3)	6(2)	–2(2)	2(2)
C118	29(3)	24(2)	43(3)	6(2)	6(2)	–2(2)
C119	32(3)	28(3)	51(4)	–8(3)	4(2)	–7(2)
C120	39(3)	27(3)	46(3)	–10(3)	7(2)	–3(2)
C121	29(2)	23(2)	40(3)	–4(2)	3(2)	5(2)
C122	22(2)	29(3)	34(3)	8(2)	4(2)	6(2)
C123	35(3)	36(3)	45(3)	8(3)	19(3)	–1(2)
C124	33(2)	26(2)	55(4)	2(2)	7(2)	–4(2)

C125	25(2)	32(3)	45(3)	3(3)	−2(2)	−5(2)
C126	23(2)	25(3)	55(4)	−7(3)	−3(2)	−5(2)
C127	43(3)	24(3)	57(4)	−8(3)	4(3)	1(2)
C128	42(3)	40(3)	57(4)	−16(3)	−17(3)	6(3)
N1	18(2)	18(2)	12(2)	−2(2)	0(1)	−5(1)
N2	18(2)	14(2)	13(2)	−1(2)	3(1)	2(1)
N3	17(2)	16(2)	10(2)	1(2)	0(1)	−2(1)
N4	14(2)	17(2)	19(2)	0(2)	0(1)	−2(1)
N5	17(2)	16(2)	17(2)	2(2)	3(1)	3(1)
N6	19(2)	17(2)	20(2)	1(2)	2(1)	2(1)
N7	18(2)	13(2)	16(2)	0(2)	0(1)	1(1)
N8	18(2)	25(2)	17(2)	3(2)	7(2)	2(2)
N9	22(2)	18(2)	26(2)	−4(2)	5(2)	−4(1)
N101	21(2)	18(2)	18(2)	2(2)	0(2)	−2(2)
N102	29(2)	18(2)	18(2)	4(2)	3(2)	5(2)
N103	30(2)	28(2)	20(2)	2(2)	0(2)	−12(2)
N104	21(2)	32(2)	17(2)	0(2)	2(2)	4(2)
N105	20(2)	24(2)	19(2)	2(2)	2(2)	4(2)
N106	20(2)	19(2)	20(2)	5(2)	2(2)	4(1)
N107	21(2)	26(2)	23(2)	2(2)	2(2)	0(2)
N108	22(2)	23(2)	27(2)	−2(2)	4(2)	−2(1)
N109	22(2)	28(2)	26(2)	2(2)	2(2)	−2(2)
Ni1	13(1)	12(1)	11(1)	−1(1)	1(1)	−1(1)
Ni2	17(1)	17(1)	15(1)	−1(1)	1(1)	−2(1)
O1	18(1)	16(2)	14(2)	0(1)	0(1)	−7(1)
O2	57(2)	62(3)	19(2)	−3(2)	12(2)	−45(2)
O3	17(1)	14(1)	15(2)	−1(1)	−1(1)	−2(1)
O4	27(2)	26(2)	18(2)	−1(1)	−2(1)	−12(1)
O5	42(2)	37(2)	22(2)	−2(2)	6(2)	21(2)
O6	26(2)	21(2)	17(2)	1(1)	3(1)	10(1)
O7	21(2)	47(2)	45(2)	−2(2)	13(2)	1(2)
O8	29(2)	26(2)	39(2)	−15(2)	7(2)	−5(1)
O9	57(3)	45(3)	152(6)	−27(3)	−21(3)	−6(2)
O10	100(4)	45(2)	40(3)	4(2)	2(2)	17(3)
O101	24(2)	17(2)	16(2)	0(1)	2(1)	−3(1)
O102	45(2)	39(2)	16(2)	1(2)	−1(2)	−23(2)
O103	22(2)	32(2)	16(2)	3(2)	3(1)	−6(1)
O104	38(2)	82(3)	30(2)	15(2)	−2(2)	−33(2)
O105	59(2)	43(2)	35(2)	11(2)	17(2)	26(2)
O106	27(2)	29(2)	20(2)	2(2)	3(1)	11(1)
O107	48(2)	26(2)	64(3)	−14(2)	7(2)	−11(2)
O108	32(2)	42(2)	60(3)	5(2)	16(2)	−6(2)
O109	26(2)	34(2)	61(3)	−7(2)	−5(2)	2(2)
O110	49(3)	59(3)	141(5)	−24(3)	−33(3)	23(2)

Table 4. Hydrogen coordinates [$\times 10^4$] and isotropic displacement parameters [$Å^2 \times 10^3$].

Atom	x	y	z	U_{eq}	S.o.f.
H1A	3821	1936	3347	22	1
H1B	3538	1134	3458	22	1
H2A	3953	602	4746	24	1
H2B	4276	943	3902	24	1
H3A	4821	1668	4465	23	1
H3B	4798	2278	5438	23	1
H4A	4775	3049	3919	19	1
H4B	4393	2519	3486	19	1
H5A	3862	3353	3341	20	1
H5B	4135	4161	3507	20	1
H6A	3683	4688	4681	21	1
H6B	3383	4297	3825	21	1
H7A	2819	3600	4441	23	1
H7B	2857	2970	5391	23	1
H8A	2909	2265	3771	22	1
H8B	3306	2809	3463	22	1
H9A	3299	841	5151	24	1
H9B	2894	1400	5012	24	1
H11A	4304	4437	5271	21	1
H11B	4732	3958	5082	21	1
H13A	4096	909	6434	20	1
H13B	4464	1529	6703	20	1
H18A	2341	3228	7213	34	1
H18B	2023	3979	7118	34	1
H19A	1713	2974	8141	42	1
H19B	2110	2945	8894	42	1
H20A	2247	4000	10017	38	1
H20B	1956	4792	10057	38	1
H21A	2209	5248	8472	29	1
H21B	2602	5046	9199	29	1
H22A	3393	6016	8869	29	1
H22B	3518	6767	8132	29	1
H23A	3917	6835	9651	32	1
H23B	4075	5917	9487	32	1
H24A	4533	5607	8161	38	1
H24B	4695	6304	7389	38	1
H25A	4041	6407	6621	31	1
H25B	4188	5471	6585	31	1
H27A	422	3220	11318	144	1
H27B	667	3608	12284	144	1
H27C	879	2900	11611	144	1
H28A	845	3322	9545	102	1
H28B	971	4250	9314	102	1
H28C	499	4025	9600	102	1
H10A	482	7576	9484	28	1
H10B	138	6949	9057	28	1
H10C	95	7661	7464	33	1
H10D	24	8307	8384	33	1
H10E	501	9226	8852	38	1
H10F	833	9541	8013	38	1
H10G	1224	9233	9458	35	1
H10H	981	8393	9649	35	1
H10I	1512	7570	9747	34	1
H10J	1893	8181	9536	34	1
H10K	2074	7439	8025	37	1
H10L	2085	6856	9025	37	1
H10M	1565	5978	9415	32	1
H10N	1329	5530	8481	32	1

H10O	795	5938	9512	26	1
H10P	1011	6793	9778	26	1
H10Q	250	6057	7834	29	1
H10R	704	5658	7744	29	1
H11C	1951	8947	8190	42	1
H11D	1537	9446	7906	42	1
H11E	409	8357	6168	27	1
H11F	746	9058	6367	27	1
H11G	991	4613	6552	38	1
H11H	1213	3736	6581	38	1
H11I	584	3598	5666	44	1
H11J	703	4341	4914	44	1
H12A	1236	4171	3670	44	1
H12B	1471	3313	3574	44	1
H12C	1823	3537	5142	37	1
H12D	1895	4317	4408	37	1
H12E	2608	5520	4327	34	1
H12F	3029	5503	4999	34	1
H12G	3178	6128	3416	47	1
H12H	2797	6751	3557	47	1
H12I	2834	7786	4817	46	1
H12J	3244	7885	5516	46	1
H12K	3081	6701	6449	41	1
H12L	2683	7294	6478	41	1
H12M	4625	8373	7170	62	1
H12N	4238	8592	6434	62	1
H12O	4213	7821	7195	62	1
H12P	4360	8219	9135	69	1
H12Q	4091	8985	9514	69	1
H12R	4558	9115	9103	69	1
H6	4952	−221	6857	32	1
H10S	3230	9456	6137	92	1
H10T	3657	9455	6307	92	1
H106	−68	10186	5881	38	1

Table 5. Torsion angles [°].

C1–C2–N2–C3	–95.3(4)	C105–C106–N104–C107	–70.7(5)
C1–C2–N2–C13	139.7(4)	C105–C106–N104–C115	158.4(4)
C1–C2–N2–Ni1	18.4(4)	C105–C106–N104–Ni2	41.9(4)
C2–C1–N1–C8	165.4(3)	C106–C105–N103–C104	160.0(4)
C2–C1–N1–C9	–69.2(4)	C106–C105–N103–C111	–75.5(5)
C2–C1–N1–Ni1	46.1(4)	C106–C105–N103–Ni2	43.1(4)
C3–C4–N3–C5	–154.2(3)	C107–C108–N101–C101	–163.8(4)
C3–C4–N3–C11	81.3(4)	C107–C108–N101–C109	71.9(5)
C3–C4–N3–Ni1	–35.1(4)	C107–C108–N101–Ni2	–45.1(4)
C4–C3–N2–C2	73.3(4)	C108–C107–N104–C106	93.5(5)
C4–C3–N2–C13	–161.2(3)	C108–C107–N104–C115	–135.1(4)
C4–C3–N2–Ni1	–43.1(4)	C108–C107–N104–Ni2	–14.8(5)
C5–C6–N4–C7	–95.3(4)	C109–C110–O101–Ni2	–2.6(5)
C5–C6–N4–C15	133.0(4)	C110–C109–N101–C101	98.1(4)
C5–C6–N4–Ni1	13.4(4)	C110–C109–N101–C108	–139.1(4)
C6–C5–N3–C4	166.0(3)	C110–C109–N101–Ni2	–19.8(4)
C6–C5–N3–C11	–69.3(4)	C111–C112–O103–Ni2	–10.3(6)
C6–C5–N3–Ni1	48.4(4)	C112–C111–N103–C104	–130.1(5)
C7–C8–N1–C1	–157.0(3)	C112–C111–N103–C105	104.7(5)
C7–C8–N1–C9	77.2(4)	C112–C111–N103–Ni2	–15.4(5)
C7–C8–N1–Ni1	–40.8(4)	C114–C113–N102–C102	65.5(5)
C8–C7–N4–C6	66.7(4)	C114–C113–N102–C103	–60.5(5)
C8–C7–N4–C15	–161.6(3)	C114–C113–N102–Ni2	–179.2(3)
C8–C7–N4–Ni1	–44.7(4)	C118–C119–O107–C120	–59.3(6)
C9–C10–O1–Ni1	7.5(5)	C119–C118–N108–C116	146.0(4)
C10–C9–N1–C1	132.4(4)	C119–C118–N108–C121	–50.2(5)
C10–C9–N1–C8	–102.3(4)	C120–C121–N108–C116	–146.3(4)
C10–C9–N1–Ni1	17.9(4)	C120–C121–N108–C118	49.9(5)
C11–C12–O3–Ni1	–2.0(5)	C121–C120–O107–C119	60.0(6)
C12–C11–N3–C4	–96.8(4)	C122–C123–O108–C124	–60.4(6)
C12–C11–N3–C5	138.5(4)	C123–C122–N109–C117	145.7(4)
C12–C11–N3–Ni1	19.4(4)	C123–C122–N109–C125	–49.2(5)
C14–C13–N2–C2	63.3(4)	C124–C125–N109–C117	–145.6(4)
C14–C13–N2–C3	–62.9(4)	C124–C125–N109–C122	49.2(5)
C14–C13–N2–Ni1	–176.8(3)	C125–C124–O108–C123	61.5(6)
C18–C19–O7–C20	–62.5(5)	N1–C1–C2–N2	–44.6(5)
C19–C18–N8–C16	159.6(4)	N1–C9–C10–O1	–17.9(6)
C19–C18–N8–C21	–47.0(5)	N1–C9–C10–O2	164.0(4)
C20–C21–N8–C16	–161.4(4)	N2–C3–C4–N3	55.0(5)
C20–C21–N8–C18	45.2(5)	N2–C13–C14–O5	–7.6(6)
C21–C20–O7–C19	61.9(5)	N2–C13–C14–O6	173.5(3)
C22–C23–O8–C24	–60.3(5)	N3–C5–C6–N4	–41.2(5)
C23–C22–N9–C17	145.8(4)	N3–C11–C12–O3	–12.7(5)
C23–C22–N9–C25	–50.6(5)	N3–C11–C12–O4	168.9(4)
C24–C25–N9–C17	–146.1(4)	N4–C7–C8–N1	61.0(4)
C24–C25–N9–C22	50.3(5)	N4–C15–N5–C16	–177.0(3)
C25–C24–O8–C23	60.3(5)	N4–C15–N7–C17	177.1(3)
C101–C102–N102–C103	–78.0(5)	N5–C15–N4–C6	145.7(4)
C101–C102–N102–C113	158.2(4)	N5–C15–N4–C7	14.7(5)
C101–C102–N102–Ni2	40.0(4)	N5–C15–N4–Ni1	–97.2(3)
C102–C101–N101–C108	159.3(4)	N5–C15–N7–C17	–2.7(6)
C102–C101–N101–C109	–77.7(5)	N5–C16–N6–C17	–5.3(6)
C102–C101–N101–Ni2	39.3(4)	N5–C16–N8–C18	–14.2(5)
C103–C104–N103–C105	–162.4(4)	N5–C16–N8–C21	–166.5(4)
C103–C104–N103–C111	72.9(5)	N6–C16–N5–C15	1.6(6)
C103–C104–N103–Ni2	–42.8(4)	N6–C16–N8–C18	166.8(4)
C104–C103–N102–C102	94.7(5)	N6–C16–N8–C21	14.5(5)
C104–C103–N102–C113	–142.0(4)	N6–C17–N7–C15	–1.8(6)
C104–C103–N102–Ni2	–22.0(5)	N6–C17–N9–C22	–10.1(6)

N6–C17–N9–C25	–172.4(4)	N105–C116–N106–C117	–0.2(6)
N7–C15–N4–C6	–34.1(5)	N105–C116–N108–C118	–9.6(6)
N7–C15–N4–C7	–165.1(4)	N105–C116–N108–C121	–172.4(4)
N7–C15–N4–Ni1	83.0(4)	N106–C116–N105–C115	–3.2(6)
N7–C15–N5–C16	2.8(6)	N106–C116–N108–C118	171.0(4)
N7–C17–N6–C16	5.4(6)	N106–C116–N108–C121	8.3(6)
N7–C17–N9–C22	171.2(4)	N106–C117–N107–C115	–3.8(6)
N7–C17–N9–C25	8.9(6)	N106–C117–N109–C122	–7.3(6)
N8–C16–N5–C15	–177.3(4)	N106–C117–N109–C125	–171.5(4)
N8–C16–N6–C17	173.6(3)	N107–C115–N104–C106	–12.7(6)
N8–C18–C19–O7	54.9(5)	N107–C115–N104–C107	–143.3(4)
N9–C17–N6–C16	–173.2(4)	N107–C115–N104–Ni2	98.1(4)
N9–C17–N7–C15	176.8(4)	N107–C115–N105–C116	3.5(6)
N9–C22–C23–O8	55.7(5)	N107–C117–N106–C116	4.0(6)
N101–C101–C102–N102	–54.6(5)	N107–C117–N109–C122	172.2(4)
N101–C109–C110–O101	16.1(6)	N107–C117–N109–C125	8.0(6)
N101–C109–C110–O102	–166.7(4)	N108–C116–N105–C115	177.5(4)
N102–C103–C104–N103	45.2(6)	N108–C116–N106–C117	179.1(4)
N102–C113–C114–O105	–11.9(7)	N108–C118–C119–O107	54.0(6)
N102–C113–C114–O106	170.8(4)	N109–C117–N106–C116	–176.6(4)
N103–C105–C106–N104	–60.6(5)	N109–C117–N107–C115	176.7(4)
N103–C111–C112–O103	17.7(7)	N109–C122–C123–O108	53.7(5)
N103–C111–C112–O104	–166.3(5)	O2–C10–O1–Ni1	–174.5(4)
N104–C107–C108–N101	39.8(6)	O4–C12–O3–Ni1	176.3(3)
N104–C115–N105–C116	–178.0(4)	O7–C20–C21–N8	–52.6(5)
N104–C115–N107–C117	–178.8(4)	O8–C24–C25–N9	–55.3(5)
N105–C115–N104–C106	168.6(4)	O102–C110–O101–Ni2	–179.7(4)
N105–C115–N104–C107	38.0(5)	O104–C112–O103–Ni2	174.1(5)
N105–C115–N104–Ni2	–80.6(4)	O107–C120–C121–N108	–54.2(6)
N105–C115–N107–C117	–0.3(7)	O108–C124–C125–N109	–54.6(6)

A.3 – MANGANESE(II) CRYSTAL STRUCTURE OF TZMORPH2DO3A ISOMER A

Table 1. Crystal data and structure refinement details.

Identificationcode	**2014ncs0931 (NS008)**	
Empirical formula	$C_{100}H_{200}Mn_9N_{36}O_{58}$	
Formula weight	3329.41	
Temperature	100.15 K	
Wavelength	0.71075 Å	
Crystal system	Tetragonal	
Space group	*I*–4	
Unit cell dimensions	*a* = 23.7262(18) Å	α = 90°
	b = 23.7262(18) Å	β = 90°
	c = 15.3454(10) Å	γ= 90°
Volume	8638.4(14) Å^3	
Z	2	
Density (calculated)	1.280 Mg / m^3	
Absorption coefficient	0.719 mm^{-1}	
F(000)	3482	
Crystal	Needle; colourless	
Crystal size	0.25 × 0.01 × 0.01 mm^3	
θrange for data collection	3.162 – 27.479°	
Index ranges	$-30 \le h \le 30$, $-30 \le k \le 30$, $-19 \le l \le 19$	
Reflections collected	52049	
Independent reflections	9864 [R_{int} = 0.0957]	
Completeness to θ = 25.242°	99.7 %	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	9864 / 0 / 467	
Goodness-of-fit on F^2	0.840	
Final *R*indices [$F^2 > 2\sigma(F^2)$]	*R1* = 0.0513, *wR2* = 0.1320	
*R*indices (all data)	*R1* = 0.0706, *wR2* = 0.1451	
Absolute structure parameter	0.086(12)	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.324 and –0.289 e Å^{-3}	

Diffractometer: Rigaku AFC12 goniometer equipped with an enhanced sensitivity (HG) Saturn724+ detector mounted at the window of an FR-E+ SuperBright molybdenum rotating anode generator with HF Varimax optics (100m focus). **Cell determination and data collection:**CrystalClear-SM Expert 3.1 b27 (Rigaku, 2012). **Data reduction, cell refinement and absorption correction:**CrystalClear-SM Expert 3.1 b27 (Rigaku, 2012**). Structure solution:** SUPERFLIP (Palatinus, L. & Chapuis, G. (2007). J. Appl. Cryst. 40, 786-790). **Structure refinement:** SHELXL-2013 (Sheldrick, G.M. (2008). ActaCryst. A64, 112-122).

Special details:
Crystal structure contains available voids filled with diffused solvent (water). This electron density has been masked out with SMTB routine within OLEX2 software.

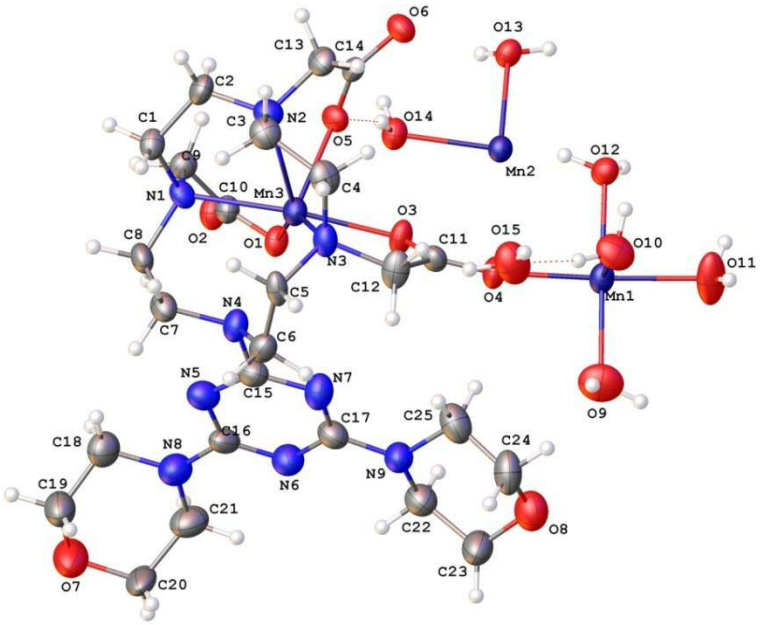

Figure 1. Molecular structure of 2014ncs0931 (NS008). Asymmetric part of the unit cell is shown. Displacement ellipsoids – 50% probability.

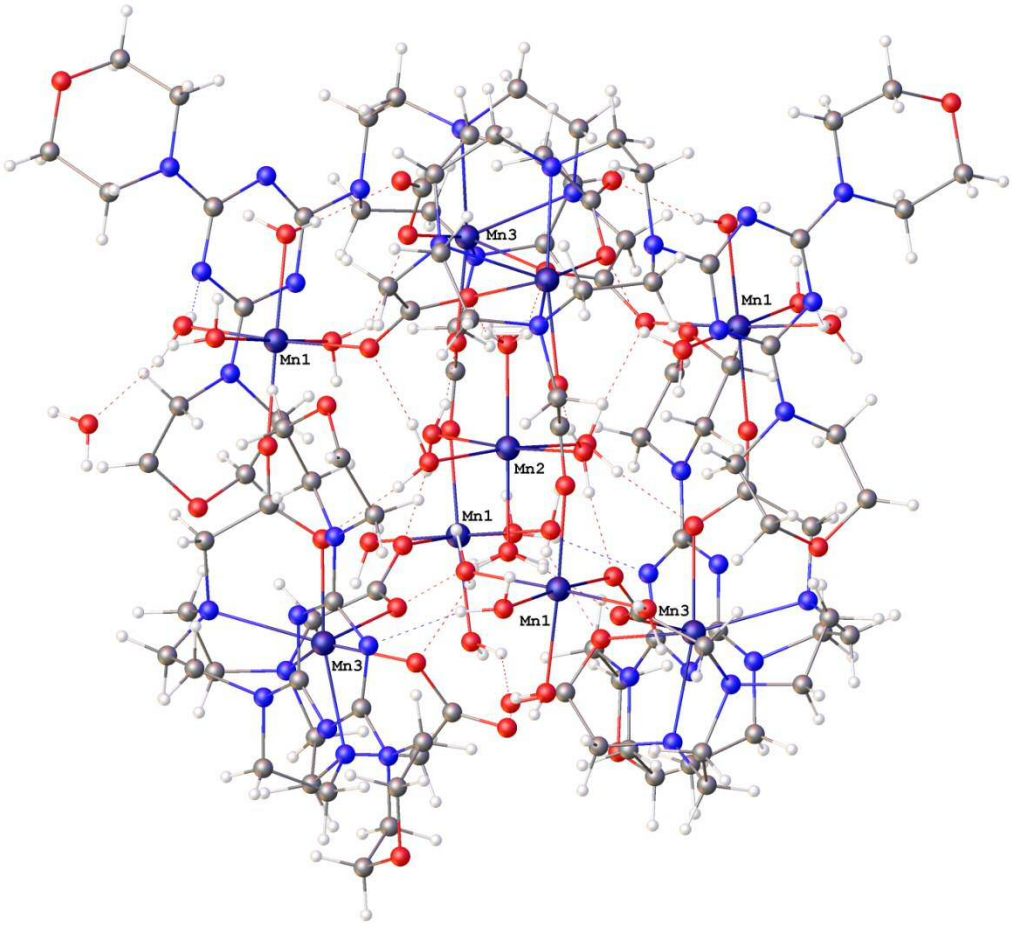

Figure 2. Crystal structure of 2014ncs0931 (NS008) – grown fragment with H-bonding network shown

Table 1. Atomic coordinates [$\times 10^4$], equivalent isotropic displacement parameters [$Å^2 \times 10^3$] and site occupancy factors. U_{eq} is defined as one third of the trace of the orthogonalized U^{ij} tensor.

Atom	x	y	z	U_{eq}	S.o.f.
Mn1	6448(1)	3614(1)	3493(1)	32(1)	1
Mn3	6158(1)	3891(1)	7503(1)	29(1)	1
O1	5228(2)	3875(2)	7675(2)	29(1)	1
O12	6454(2)	4529(2)	3647(3)	34(1)	1
O3	6115(2)	3811(2)	6113(3)	35(1)	1
O5	5976(2)	4787(2)	7158(3)	31(1)	1
O6	6372(2)	5528(2)	6489(3)	36(1)	1
O4	6478(2)	3521(2)	4872(3)	39(1)	1
O2	4545(2)	4232(2)	8529(3)	36(1)	1
N3	7021(2)	3443(2)	7076(3)	36(1)	1
N4	6119(2)	2854(2)	8050(3)	37(1)	1
N7	5578(2)	2541(3)	6869(4)	45(1)	1
O15	8055(2)	3187(2)	4671(4)	62(1)	1
O11	6427(3)	3751(3)	2107(3)	71(2)	1
N1	5982(2)	3950(2)	8961(3)	30(1)	1
O10	7360(2)	3598(2)	3378(4)	52(1)	1
O9	6493(2)	2704(2)	3318(4)	62(2)	1
N2	6899(2)	4495(2)	8027(4)	37(1)	1
N6	4657(2)	2175(2)	7130(4)	47(1)	1
N9	5021(2)	2230(2)	5733(4)	46(1)	1
C7	6161(3)	2913(3)	8998(4)	37(1)	1
C14	6379(2)	5116(2)	7021(4)	27(1)	1
C5	7127(2)	2940(2)	7602(4)	33(1)	1
O8	4931(2)	1960(2)	3955(4)	61(1)	1
C17	5093(3)	2326(3)	6609(5)	41(2)	1
C11	6493(2)	3565(2)	5654(4)	30(1)	1
N5	5246(2)	2460(2)	8325(4)	43(1)	1
C10	5034(3)	4140(2)	8355(4)	35(1)	1
C8	5821(3)	3401(3)	9355(4)	37(1)	1
C6	6620(3)	2555(3)	7667(4)	38(1)	1
O7	3814(3)	1306(3)	9699(4)	79(2)	1
C12	6969(3)	3293(3)	6129(4)	41(2)	1
N8	4377(3)	2033(2)	8540(4)	55(2)	1
C2	6714(3)	4664(3)	8916(4)	38(1)	1
C16	4763(3)	2230(3)	7972(5)	42(2)	1
C9	5483(2)	4337(2)	8995(4)	34(1)	1
C4	7483(3)	3849(3)	7200(4)	40(2)	1
C13	6940(2)	4990(2)	7439(4)	36(1)	1
C1	6476(2)	4204(3)	9438(4)	33(1)	1
C15	5614(3)	2607(3)	7738(4)	41(2)	1
C18	4413(3)	2110(3)	9486(5)	52(2)	1
C3	7414(3)	4175(3)	8034(5)	41(2)	1
C22	4505(3)	2036(3)	5370(5)	50(2)	1
C23	4613(4)	1668(4)	4567(5)	60(2)	1
C21	3857(4)	1751(3)	8263(6)	59(2)	1
C20	3782(4)	1207(4)	8766(5)	69(3)	1
C25	5406(3)	2484(3)	5078(5)	55(2)	1
C24	5452(3)	2115(4)	4325(5)	58(2)	1
C19	4341(4)	1567(4)	9931(6)	68(2)	1
Mn2	5000	5000	5000	29(1)	1
O14	5000	5000	6368(4)	34(1)	1
O13	4766(2)	5894(2)	5066(3)	31(1)	1

Table 2. Bond lengths [Å] and angles [°].

Mn1–O12	2.185(4)	N5–C15	1.303(9)
Mn1–O6i	2.183(4)	C10–C9	1.522(9)
Mn1–O4	2.128(4)	C8–H8A	0.9900
Mn1–O11	2.151(5)	C8–H8B	0.9900
Mn1–O10	2.173(5)	C6–H6A	0.9900
Mn1–O9	2.177(5)	C6–H6B	0.9900
Mn3–O1	2.225(4)	O7–C20	1.453(10)
Mn3–O3	2.143(4)	O7–C19	1.439(10)
Mn3–O5	2.233(4)	C12–H12C	0.9900
Mn3–N3	2.397(5)	C12–H12D	0.9900
Mn3–N1	2.281(5)	N8–C16	1.348(9)
Mn3–N2	2.407(5)	N8–C18	1.466(10)
O1–C10	1.301(7)	N8–C21	1.467(9)
O12–H12A	0.8540	C2–H2A	0.9900
O12–H12B	0.8535	C2–H2B	0.9900
O3–C11	1.280(7)	C2–C1	1.467(9)
O5–C14	1.253(7)	C9–H9C	0.9900
O6–Mn1ii	2.183(4)	C9–H9D	0.9900
O6–C14	1.274(7)	C4–H4A	0.9900
O4–C11	1.205(7)	C4–H4B	0.9900
O2–C10	1.210(7)	C4–C3	1.505(10)
N3–C5	1.463(7)	C13–H13A	0.9900
N3–C12	1.502(8)	C13–H13B	0.9900
N3–C4	1.470(8)	C1–H1A	0.9900
N4–C7	1.464(8)	C1–H1B	0.9900
N4–C6	1.504(8)	C18–H18A	0.9900
N4–C15	1.417(8)	C18–H18B	0.9900
N7–C17	1.321(8)	C18–C19	1.469(11)
N7–C15	1.346(9)	C3–H3A	0.9900
O15–H15A	0.8500	C3–H3B	0.9900
O15–H15B	0.8501	C22–H22A	0.9900
O11–H11A	0.8608	C22–H22B	0.9900
O11–H11B	0.8608	C22–C23	1.532(11)
N1–C8	1.487(7)	C23–H23	0.9500
N1–C9	1.499(7)	C21–H21A	0.9900
N1–C1	1.508(7)	C21–H21B	0.9900
O10–H10A	0.8709	C21–C20	1.514(12)
O10–H10B	0.8706	C20–H20A	0.9900
O9–H9A	0.8762	C20–H20B	0.9900
O9–H9B	0.8750	C25–H25A	0.9900
N2–C2	1.488(8)	C25–H25B	0.9900
N2–C13	1.483(8)	C25–C24	1.454(11)
N2–C3	1.440(8)	C24–H24A	0.9900
N6–C17	1.355(9)	C24–H24B	0.9900
N6–C16	1.322(9)	C19–H19A	0.9900
N9–C17	1.374(9)	C19–H19B	0.9900
N9–C22	1.421(9)	Mn2–O14i	2.099(6)
N9–C25	1.487(9)	Mn2–O14	2.099(6)
C7–H7A	0.9900	Mn2–O13	2.195(4)
C7–H7B	0.9900	Mn2–O13iii	2.195(4)
C7–C8	1.514(9)	Mn2–O13i	2.195(4)
C14–C13	1.508(8)	Mn2–O13ii	2.195(4)
C5–H5A	0.9900	O14–H14A	0.8617
C5–H5B	0.9900	O14–H14B	0.8618
C5–C6	1.514(9)	O13–H13C	0.8525
O8–C23	1.391(9)	O13–H13D	0.8522
O8–C24	1.409(10)		
C11–C12	1.492(8)	O6i–Mn1–O12	89.44(16)
N5–C16	1.379(9)	O4–Mn1–O12	89.72(16)

O4–Mn1–O6i	91.28(17)
O4–Mn1–O11	177.2(2)
O4–Mn1–O10	92.6(2)
O4–Mn1–O9	91.1(2)
O11–Mn1–O12	87.5(2)
O11–Mn1–O6i	89.3(2)
O11–Mn1–O10	86.8(2)
O11–Mn1–O9	91.7(2)
O10–Mn1–O12	91.09(17)
O10–Mn1–O6i	176.1(2)
O10–Mn1–O9	85.6(2)
O9–Mn1–O12	176.62(18)
O9–Mn1–O6i	93.83(18)
O1–Mn3–O5	81.42(15)
O1–Mn3–N3	150.83(17)
O1–Mn3–N1	72.65(15)
O1–Mn3–N2	134.00(16)
O3–Mn3–O1	94.00(15)
O3–Mn3–O5	80.73(15)
O3–Mn3–N3	74.34(16)
O3–Mn3–N1	166.60(16)
O3–Mn3–N2	114.81(18)
O5–Mn3–N3	121.51(16)
O5–Mn3–N1	97.91(16)
O5–Mn3–N2	69.72(16)
N3–Mn3–N2	74.44(19)
N1–Mn3–N3	116.89(17)
N1–Mn3–N2	76.68(17)
C10–O1–Mn3	116.0(4)
Mn1–O12–H12A	109.9
Mn1–O12–H12B	109.6
H12A–O12–H12B	109.3
C11–O3–Mn3	123.7(3)
C14–O5–Mn3	119.0(3)
C14–O6–Mn1ii	139.3(4)
C11–O4–Mn1	169.1(4)
C5–N3–Mn3	110.9(3)
C5–N3–C12	110.7(5)
C5–N3–C4	109.6(5)
C12–N3–Mn3	107.4(3)
C4–N3–Mn3	108.1(4)
C4–N3–C12	110.0(5)
C7–N4–C6	112.3(5)
C15–N4–C7	115.7(5)
C15–N4–C6	109.9(5)
C17–N7–C15	113.5(6)
H15A–O15–H15B	109.5
Mn1–O11–H11A	110.4
Mn1–O11–H11B	110.2
H11A–O11–H11B	108.8
C8–N1–Mn3	113.1(4)
C8–N1–C9	108.6(5)
C8–N1–C1	110.6(4)
C9–N1–Mn3	102.6(3)
C9–N1–C1	110.7(4)
C1–N1–Mn3	111.0(3)
Mn1–O10–H10A	110.8
Mn1–O10–H10B	110.6
H10A–O10–H10B	108.3
Mn1–O9–H9A	110.9
Mn1–O9–H9B	110.2
H9A–O9–H9B	107.7
C2–N2–Mn3	104.5(4)
C13–N2–Mn3	108.5(3)
C13–N2–C2	111.4(5)
C3–N2–Mn3	107.9(4)
C3–N2–C2	112.7(5)
C3–N2–C13	111.5(5)
C16–N6–C17	113.9(6)
C17–N9–C22	123.0(6)
C17–N9–C25	121.3(6)
C22–N9–C25	113.2(6)
N4–C7–H7A	108.9
N4–C7–H7B	108.9
N4–C7–C8	113.4(5)
H7A–C7–H7B	107.7
C8–C7–H7A	108.9
C8–C7–H7B	108.9
O5–C14–O6	125.1(5)
O5–C14–C13	118.7(5)
O6–C14–C13	115.9(5)
N3–C5–H5A	108.9
N3–C5–H5B	108.9
N3–C5–C6	113.1(5)
H5A–C5–H5B	107.8
C6–C5–H5A	108.9
C6–C5–H5B	108.9
C23–O8–C24	109.5(6)
N7–C17–N6	126.1(7)
N7–C17–N9	117.8(6)
N6–C17–N9	116.0(6)
O3–C11–C12	117.3(5)
O4–C11–O3	124.5(5)
O4–C11–C12	118.2(5)
C15–N5–C16	113.0(6)
O1–C10–C9	114.8(5)
O2–C10–O1	127.0(6)
O2–C10–C9	118.2(5)
N1–C8–C7	112.6(5)
N1–C8–H8A	109.1
N1–C8–H8B	109.1
C7–C8–H8A	109.1
C7–C8–H8B	109.1
H8A–C8–H8B	107.8
N4–C6–C5	111.6(5)
N4–C6–H6A	109.3
N4–C6–H6B	109.3
C5–C6–H6A	109.3
C5–C6–H6B	109.3
H6A–C6–H6B	108.0
C19–O7–C20	111.0(6)
N3–C12–H12C	108.4
N3–C12–H12D	108.4
C11–C12–N3	115.6(5)
C11–C12–H12C	108.4
C11–C12–H12D	108.4
H12C–C12–H12D	107.4
C16–N8–C18	123.8(6)
C16–N8–C21	122.9(7)
C21–N8–C18	113.1(6)

N2–C2–H2A	108.6	O8–C23–C22	110.4(6)
N2–C2–H2B	108.6	O8–C23–H23	124.8
H2A–C2–H2B	107.6	C22–C23–H23	124.8
C1–C2–N2	114.5(5)	N8–C21–H21A	109.7
C1–C2–H2A	108.6	N8–C21–H21B	109.7
C1–C2–H2B	108.6	N8–C21–C20	109.9(7)
N6–C16–N5	125.5(6)	H21A–C21–H21B	108.2
N6–C16–N8	117.9(6)	C20–C21–H21A	109.7
N8–C16–N5	116.6(6)	C20–C21–H21B	109.7
N1–C9–C10	110.0(5)	O7–C20–C21	111.0(6)
N1–C9–H9C	109.7	O7–C20–H20A	109.4
N1–C9–H9D	109.7	O7–C20–H20B	109.4
C10–C9–H9C	109.7	C21–C20–H20A	109.4
C10–C9–H9D	109.7	C21–C20–H20B	109.4
H9C–C9–H9D	108.2	H20A–C20–H20B	108.0
N3–C4–H4A	109.3	N9–C25–H25A	109.7
N3–C4–H4B	109.3	N9–C25–H25B	109.7
N3–C4–C3	111.4(5)	H25A–C25–H25B	108.2
H4A–C4–H4B	108.0	C24–C25–N9	109.8(7)
C3–C4–H4A	109.3	C24–C25–H25A	109.7
C3–C4–H4B	109.3	C24–C25–H25B	109.7
N2–C13–C14	110.9(5)	O8–C24–C25	114.3(7)
N2–C13–H13A	109.5	O8–C24–H24A	108.7
N2–C13–H13B	109.5	O8–C24–H24B	108.7
C14–C13–H13A	109.5	C25–C24–H24A	108.7
C14–C13–H13B	109.5	C25–C24–H24B	108.7
H13A–C13–H13B	108.0	H24A–C24–H24B	107.6
N1–C1–H1A	109.8	O7–C19–C18	111.4(8)
N1–C1–H1B	109.8	O7–C19–H19A	109.4
C2–C1–N1	109.3(5)	O7–C19–H19B	109.4
C2–C1–H1A	109.8	C18–C19–H19A	109.4
C2–C1–H1B	109.8	C18–C19–H19B	109.4
H1A–C1–H1B	108.3	H19A–C19–H19B	108.0
N7–C15–N4	115.9(6)	O14i–Mn2–O14	180.0
N5–C15–N4	116.4(6)	O14–Mn2–O13ii	92.65(11)
N5–C15–N7	127.7(6)	O14–Mn2–O13iii	87.35(11)
N8–C18–H18A	109.6	O14i–Mn2–O13ii	87.35(11)
N8–C18–H18B	109.6	O14–Mn2–O13i	92.65(11)
N8–C18–C19	110.1(7)	O14i–Mn2–O13	92.65(11)
H18A–C18–H18B	108.1	O14–Mn2–O13	87.35(11)
C19–C18–H18A	109.6	O14i–Mn2–O13iii	92.65(11)
C19–C18–H18B	109.6	O14i–Mn2–O13i	87.35(11)
N2–C3–C4	110.9(5)	O13iii–Mn2–O13	174.7(2)
N2–C3–H3A	109.5	O13i–Mn2–O13iii	90.122(11)
N2–C3–H3B	109.5	O13i–Mn2–O13	90.123(11)
C4–C3–H3A	109.5	O13ii–Mn2–O13iii	90.122(11)
C4–C3–H3B	109.5	O13ii–Mn2–O13	90.122(11)
H3A–C3–H3B	108.1	O13ii–Mn2–O13i	174.7(2)
N9–C22–H22A	109.4	Mn2–O14–H14A	110.2
N9–C22–H22B	109.4	Mn2–O14–H14B	110.2
N9–C22–C23	111.0(7)	H14A–O14–H14B	108.7
H22A–C22–H22B	108.0	Mn2–O13–H13C	109.4
C23–C22–H22A	109.4	Mn2–O13–H13D	109.1
C23–C22–H22B	109.4	H13C–O13–H13D	109.4

Symmetry transformations used to generate equivalent atoms:
(i) y,–x+1,–z+1 (ii) –y+1,x,–z+1 (iii) –x+1,–y+1,z

Table 3. Anisotropic displacement parameters [$Å^2 \times 10^3$].

The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}]$.

Atom	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Mn1	34(1)	37(1)	25(1)	−1(1)	0(1)	8(1)
Mn3	30(1)	34(1)	25(1)	1(1)	−3(1)	2(1)
O1	24(2)	41(2)	23(2)	−1(2)	2(2)	3(2)
O12	39(2)	33(2)	31(2)	−1(2)	−5(2)	2(2)
O3	36(2)	47(2)	21(2)	−4(2)	0(2)	15(2)
O5	34(2)	28(2)	30(2)	6(2)	−6(2)	3(2)
O6	40(2)	34(2)	35(2)	8(2)	−3(2)	−3(2)
O4	50(3)	44(2)	21(2)	0(2)	−1(2)	−1(2)
O2	32(2)	48(2)	28(2)	1(2)	2(2)	6(2)
N3	39(3)	44(3)	25(3)	4(2)	0(2)	8(2)
N4	41(3)	46(3)	26(3)	0(2)	−3(2)	3(2)
N7	45(3)	56(3)	34(3)	1(3)	−1(2)	5(3)
O15	64(3)	65(3)	56(4)	8(3)	15(3)	17(3)
O11	92(4)	86(4)	35(3)	5(3)	13(3)	57(4)
N1	25(2)	34(3)	30(3)	7(2)	2(2)	−2(2)
O10	39(2)	54(3)	63(4)	6(3)	13(2)	0(2)
O9	50(3)	51(3)	86(4)	−1(3)	2(3)	17(2)
N2	38(3)	34(3)	39(3)	2(2)	0(2)	−4(2)
N6	49(3)	47(3)	43(3)	1(3)	−3(3)	−8(3)
N9	59(3)	44(3)	34(3)	1(2)	−1(3)	−10(3)
C7	37(3)	43(3)	32(3)	7(3)	−7(3)	−10(3)
C14	34(3)	27(3)	20(3)	−2(2)	−3(2)	−2(2)
C5	34(3)	43(3)	22(3)	8(2)	5(2)	15(2)
O8	69(3)	65(3)	48(3)	6(3)	−4(3)	4(3)
C17	43(3)	37(3)	42(4)	6(3)	−1(3)	−8(3)
C11	35(3)	26(3)	28(3)	4(2)	0(2)	4(2)
N5	45(3)	38(3)	47(4)	0(3)	5(3)	−4(2)
C10	43(3)	36(3)	25(3)	2(2)	−2(3)	−4(2)
C8	47(3)	41(3)	24(3)	9(3)	−2(3)	−8(3)
C6	47(3)	34(3)	32(4)	−2(3)	−5(3)	5(3)
O7	99(5)	87(4)	52(4)	2(3)	2(3)	−45(4)
C12	34(3)	56(4)	33(3)	0(3)	−2(3)	23(3)
N8	67(4)	50(3)	47(4)	−4(3)	3(3)	−12(3)
C2	50(4)	41(3)	23(3)	−4(3)	−6(3)	−2(3)
C16	54(4)	30(3)	42(4)	−4(3)	11(3)	−10(3)
C9	37(3)	33(3)	32(3)	−2(2)	0(2)	1(2)
C4	31(3)	48(4)	40(4)	5(3)	8(3)	6(3)
C13	34(3)	41(3)	32(3)	1(3)	−9(3)	−5(2)
C1	28(3)	45(3)	26(3)	1(3)	1(2)	−3(2)
C15	49(4)	34(3)	41(4)	3(3)	−4(3)	−5(3)
C18	56(4)	56(4)	44(4)	−4(3)	8(3)	−2(3)
C3	34(3)	46(4)	43(4)	3(3)	−1(3)	0(3)
C22	58(4)	48(4)	45(4)	4(3)	−2(3)	−10(3)
C23	74(5)	65(5)	40(4)	−2(4)	0(4)	−24(4)
C21	63(5)	55(4)	60(5)	−12(4)	2(4)	−19(4)
C20	105(7)	66(5)	37(4)	−8(4)	−5(4)	−52(5)
C25	50(4)	66(5)	48(5)	17(4)	4(3)	−1(4)
C24	58(5)	81(5)	34(4)	10(4)	1(3)	8(4)
C19	92(6)	70(5)	41(5)	3(4)	−2(4)	−33(5)
Mn2	30(1)	30(1)	25(1)	0	0	0
O14	34(3)	42(3)	27(3)	0	0	4(3)
O13	31(2)	33(2)	27(2)	−4(2)	0(2)	5(2)

Table 4. Hydrogen coordinates [$\times 10^4$] and isotropic displacement parameters [$Å^2 \times 10^3$].

Atom	x	y	z	U_{eq}	S.o.f.
H12A	6465	4614	4188	51	1
H12B	6157	4669	3420	51	1
H15A	8014	3093	5202	93	1
H15B	8404	3192	4545	93	1
H11A	6749	3670	1879	107	1
H11B	6349	4098	1996	107	1
H10A	7513	3440	3833	78	1
H10B	7494	3938	3332	78	1
H9A	6834	2580	3425	93	1
H9B	6410	2615	2779	93	1
H7A	6562	2968	9157	44	1
H7B	6031	2560	9275	44	1
H5A	7240	3057	8196	39	1
H5B	7445	2728	7344	39	1
H8A	5416	3330	9244	45	1
H8B	5875	3421	9994	45	1
H6A	6522	2413	7080	45	1
H6B	6716	2226	8036	45	1
H12C	7325	3396	5833	49	1
H12D	6926	2879	6082	49	1
H2A	7041	4824	9231	46	1
H2B	6427	4965	8861	46	1
H9C	5601	4725	8847	41	1
H9D	5325	4340	9593	41	1
H4A	7490	4115	6702	48	1
H4B	7847	3645	7210	48	1
H13A	7224	4914	6980	43	1
H13B	7067	5322	7776	43	1
H1A	6351	4350	10011	40	1
H1B	6767	3912	9540	40	1
H18A	4117	2376	9680	62	1
H18B	4785	2273	9639	62	1
H3A	7410	3910	8533	49	1
H3B	7738	4433	8109	49	1
H22A	4270	2364	5202	60	1
H22B	4296	1816	5812	60	1
H23	4480	1292	4498	72	1
H21A	3875	1669	7631	71	1
H21B	3530	2002	8369	71	1
H20A	3412	1039	8621	83	1
H20B	4079	937	8593	83	1
H25A	5259	2856	4894	66	1
H25B	5783	2541	5339	66	1
H24A	5682	2305	3875	69	1
H24B	5655	1768	4501	69	1
H19A	4655	1313	9773	81	1
H19B	4353	1627	10570	81	1
H14A	4837	5299	6561	52	0.5
H14B	5341	4992	6561	52	0.5
H13C	4486	5934	5410	46	1
H13D	4673	6007	4559	46	1

Table 5.Torsion angles [°].

Mn1ii–O6–C14–O5	111.6(6)	C6–N4–C15–N5	128.3(6)
Mn1ii–O6–C14–C13	–75.0(7)	C12–N3–C5–C6	–70.7(6)
Mn1–O4–C11–O3	59(3)	C12–N3–C4–C3	158.2(5)
Mn1–O4–C11–C12	–124(2)	N8–C18–C19–O7	–57.1(10)
Mn3–O1–C10–O2	172.3(5)	N8–C21–C20–O7	53.3(10)
Mn3–O1–C10–C9	–9.3(6)	C2–N2–C13–C14	–86.4(6)
Mn3–O3–C11–O4	–179.4(4)	C2–N2–C3–C4	160.5(5)
Mn3–O3–C11–C12	3.6(7)	C16–N6–C17–N7	–3.6(10)
Mn3–O5–C14–O6	147.9(5)	C16–N6–C17–N9	174.5(6)
Mn3–O5–C14–C13	–25.4(7)	C16–N5–C15–N4	179.2(5)
Mn3–N3–C5–C6	48.5(6)	C16–N5–C15–N7	–2.6(10)
Mn3–N3–C12–C11	14.1(7)	C16–N8–C18–C19	–129.2(8)
Mn3–N3–C4–C3	41.2(6)	C16–N8–C21–C20	131.2(8)
Mn3–N1–C8–C7	39.8(6)	C9–N1–C8–C7	153.0(5)
Mn3–N1–C9–C10	49.3(5)	C9–N1–C1–C2	–73.6(6)
Mn3–N1–C1–C2	39.6(5)	C4–N3–C5–C6	167.8(5)
Mn3–N2–C2–C1	42.9(6)	C4–N3–C12–C11	–103.4(6)
Mn3–N2–C13–C14	28.1(6)	C13–N2–C2–C1	159.8(5)
Mn3–N2–C3–C4	45.7(6)	C13–N2–C3–C4	–73.4(6)
O1–C10–C9–N1	–29.1(7)	C1–N1–C8–C7	–85.4(6)
O3–C11–C12–N3	–12.8(8)	C1–N1–C9–C10	167.7(5)
O5–C14–C13–N2	–4.8(8)	C15–N4–C7–C8	–77.1(7)
O6–C14–C13–N2	–178.6(5)	C15–N4–C6–C5	152.2(5)
O4–C11–C12–N3	170.0(5)	C15–N7–C17–N6	–0.4(10)
O2–C10–C9–N1	149.5(5)	C15–N7–C17–N9	–178.5(6)
N3–C5–C6–N4	–58.2(7)	C15–N5–C16–N6	–2.3(10)
N3–C4–C3–N2	–61.3(7)	C15–N5–C16–N8	175.8(6)
N4–C7–C8–N1	–57.5(7)	C18–N8–C16–N6	–174.3(7)
N2–C2–C1–N1	–57.6(7)	C18–N8–C16–N5	7.5(10)
N9–C22–C23–O8	–56.1(9)	C18–N8–C21–C20	–52.6(9)
N9–C25–C24–O8	53.4(9)	C3–N2–C2–C1	–74.0(7)
C7–N4–C6–C5	–77.5(6)	C3–N2–C13–C14	146.8(5)
C7–N4–C15–N7	–178.6(6)	C22–N9–C17–N7	–175.7(6)
C7–N4–C15–N5	–0.2(8)	C22–N9–C17–N6	6.0(10)
C5–N3–C12–C11	135.4(6)	C22–N9–C25–C24	–48.2(8)
C5–N3–C4–C3	–79.9(6)	C23–O8–C24–C25	–61.2(9)
C17–N7–C15–N4	–177.9(6)	C21–N8–C16–N6	1.5(11)
C17–N7–C15–N5	3.8(10)	C21–N8–C16–N5	–176.7(6)
C17–N6–C16–N5	5.1(10)	C21–N8–C18–C19	54.6(9)
C17–N6–C16–N8	–173.0(6)	C20–O7–C19–C18	59.5(10)
C17–N9–C22–C23	–148.2(7)	C25–N9–C17–N7	–15.0(10)
C17–N9–C25–C24	149.3(7)	C25–N9–C17–N6	166.7(6)
C8–N1–C9–C10	–70.7(6)	C25–N9–C22–C23	49.7(8)
C8–N1–C1–C2	166.0(5)	C24–O8–C23–C22	59.9(9)
C6–N4–C7–C8	155.7(5)	C19–O7–C20–C21	–57.3(11)
C6–N4–C15–N7	–50.2(7)		
		–57.3(11)	

Symmetry transformations used to generate equivalent atoms:
(i) y,–x+1,–z+1 (ii) –y+1,x,–z+1 (iii) –x+1,–y+1,z

A.4 – MANGANESE(II) CRYSTAL STRUCTURE OF TZMORPH2DO3A ISOMER B

Table 1. Crystal data and structure refinement details.

Identification code	**2015ncs0424 (MM204)**	
Empirical formula	$C_{58}H_{98}F_{12}Mn_4N_{18}Na_2O_{35}$	
Formula weight	2101.28	
Temperature	100(2) K	
Wavelength	0.71075 Å	
Crystal system	Triclinic	
Space group	*P*–1	
Unit cell dimensions	*a* = 10.4386(3) Å	α = 84.678(2)°
	b = 12.7009(3) Å	β = 84.719(3)°
	c = 17.3418(6) Å	γ= 70.984(2)°
Volume	2159.66(11) Å^3	
Z	1	
Density (calculated)	1.616 Mg / m^3	
Absorption coefficient	0.701 mm^{-1}	
F(000)	1082	
Crystal	Plate; colourless	
Crystal size	0.120 × 0.050 × 0.010 mm^3	
θ range for data collection	1.700 – 29.812°	
Index ranges	$-14 \le h \le 13$, $-16 \le k \le 17$, $-22 \le l \le 23$	
Reflections collected	28898	
Independent reflections	11111 [R_{int} = 0.0341]	
Completeness to θ = 25.242°	100.0 %	
Absorption correction	Semi–empirical from equivalents	
Max. and min. transmission	1.00000 and 0.89866	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	11111 / 1416 / 649	
Goodness-of-fit on F^2	1.051	
Final *R* indices [$F^2 > 2\sigma(F^2)$]	*R1* = 0.0518, *wR2* = 0.1387	
R indices (all data)	*R1* = 0.0768, *wR2* = 0.1644	
Extinction coefficient	n/a	
Largest diff. peak and hole	1.152 and –0.933 e Å^{-3}	

Diffractometer: Rigaku AFC12 goniometer equipped with an enhanced sensitivity (HG) Saturn724+ detector mounted at the window of an FR-E+ SuperBright molybdenum rotating anode generator with HF Varimax optics (100m focus). **Cell determination and data collection:** CrystalClear-SM Expe. rt 3.1 b27 (Rigaku, 2012). **Data reduction, cell refinement and absorption correction:** CrysAlisPro, Agilent Technologies,, Version 1.171.37.35. **Structure solution:** SUPERFLIP (Palatinus, L. & Chapuis, G. (2007). J. Appl. Cryst. 40, 786-790). **Structure refinement:** SHELXL-2013 (Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122).

Special details:
Data were quite weak and also twinned. The final R-factor is quite high 12 %. In the crystal structure one –CF3 and one morpholine ring are disordered and both have been modelled over two positions with 47:53 and 68:32 ratio respectively. Water oxygen O21 disordered over symmetry element has been refined as 50% occupied.

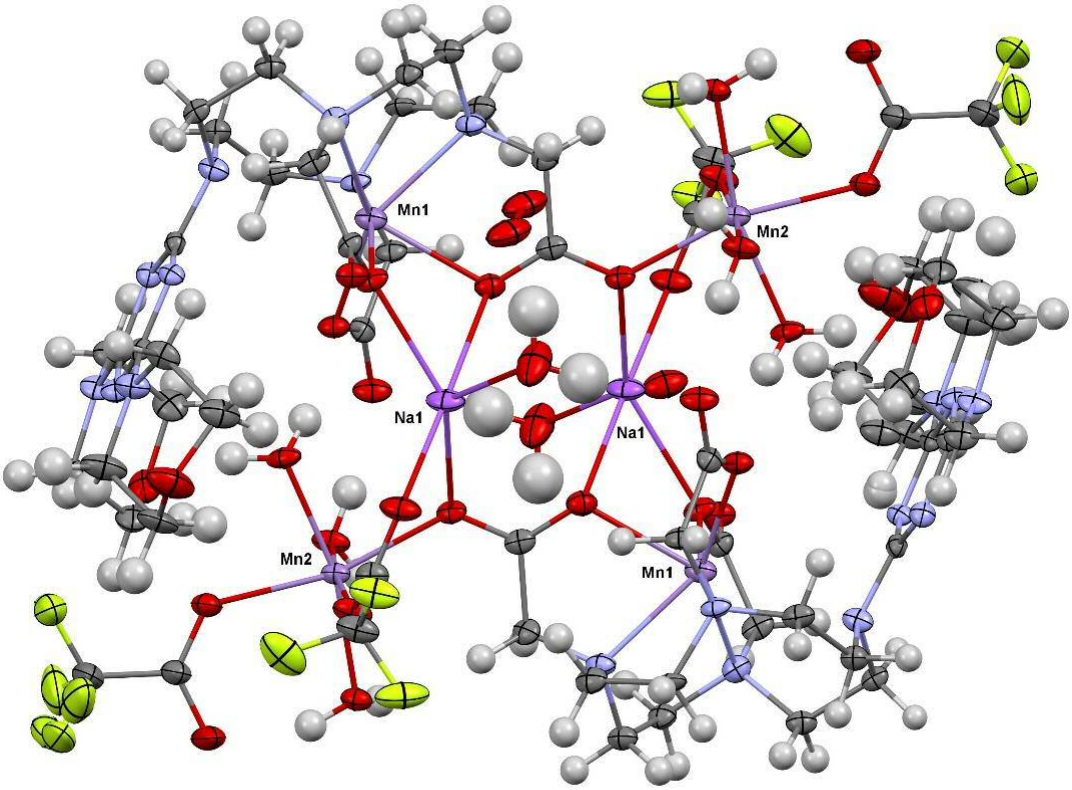

Figure 1. Molecular structure of 2015ncsd0424 (MM209) – grown fragment.
Displacement ellipsoids – 50% probability

Table 1. Atomic coordinates [$\times 10^4$], equivalent isotropic displacement parameters [$Å^2 \times 10^3$] and site occupancy factors. U_{eq} is defined as one third of the trace of the orthogonalized U^{ij} tensor.

Atom	x	y	z	U_{eq}	S.o.f.
Mn1	7221(1)	3256(1)	6777(1)	15(1)	1
Mn2	2167(1)	7224(1)	6763(1)	14(1)	1
Na1	5680(1)	5861(1)	5740(1)	24(1)	1
F1	3715(2)	10243(2)	6800(1)	44(1)	1
F2	3215(2)	10493(2)	5609(1)	41(1)	1
F3	5313(2)	9913(2)	5883(1)	34(1)	1
O15	3954(2)	6192(2)	7350(1)	21(1)	1
O2	563(2)	4589(2)	9068(2)	32(1)	1
O3	7548(2)	4833(2)	6535(1)	17(1)	1
O4	8949(2)	5836(2)	6541(1)	21(1)	1
O5	5045(2)	4046(2)	7105(1)	17(1)	1
O6	3072(2)	3857(2)	6835(1)	24(1)	1
O7	6318(2)	3747(2)	5575(1)	19(1)	1
O8	2902(2)	8565(2)	6382(1)	24(1)	1
O9	5102(2)	7875(2)	5920(2)	27(1)	1
O10	1681(2)	7853(2)	7960(1)	23(1)	1
O11	−187(3)	9340(2)	7811(2)	40(1)	1
O12	102(2)	8330(2)	6484(1)	20(1)	1
O13	1320(2)	5902(2)	7026(1)	20(1)	1
O14	7323(3)	6071(3)	4781(2)	51(1)	1
O35	3227(2)	6403(2)	5703(1)	17(1)	1
N1	6053(2)	6583(2)	8698(2)	21(1)	1
N2	6839(2)	4670(2)	8554(1)	17(1)	1
N3	5347(2)	3610(2)	8975(1)	18(1)	1
N4	4562(2)	5597(2)	9052(1)	17(1)	1
N5	3166(2)	4528(2)	9441(2)	22(1)	1
N6	7485(2)	2763(2)	8384(1)	16(1)	1
N7	9470(2)	2907(2)	7091(1)	16(1)	1
N8	6343(2)	1836(2)	7157(1)	16(1)	1
N9	8441(2)	1900(2)	5939(1)	17(1)	1
C5	5815(3)	5581(2)	8776(2)	17(1)	1
C6	4393(3)	4584(2)	9150(2)	16(1)	1
C7	2807(3)	3504(3)	9471(2)	24(1)	1
C8	1726(3)	3654(3)	8904(2)	29(1)	1
C9	916(3)	5594(3)	9035(2)	29(1)	1
C10	1988(3)	5506(3)	9599(2)	23(1)	1
C11	6508(3)	3722(2)	8660(2)	16(1)	1
C12	7173(3)	1702(2)	8503(2)	18(1)	1
C13	6063(3)	1646(2)	8008(2)	18(1)	1
C14	7261(3)	802(2)	6823(2)	19(1)	1
C15	7768(3)	1022(2)	5994(2)	18(1)	1
C16	9869(3)	1426(2)	6162(2)	19(1)	1
C17	10326(3)	2360(2)	6414(2)	19(1)	1
C18	9856(3)	2192(2)	7810(2)	16(1)	1
C19	8910(3)	2633(2)	8510(2)	16(1)	1
C20	9657(3)	4009(2)	7143(2)	18(1)	1
C21	8642(3)	4958(2)	6699(2)	16(1)	1
C22	5020(3)	2254(2)	6800(2)	18(1)	1
C23	4315(3)	3485(2)	6928(2)	15(1)	1
C24	8405(3)	2412(2)	5143(2)	20(1)	1
C25	2950(3)	6681(2)	5004(2)	15(1)	1
C26	4056(3)	8606(2)	6132(2)	20(1)	1
C27	4079(3)	9820(2)	6107(2)	26(1)	1
C28	753(3)	8684(2)	8179(2)	24(1)	1
C29	743(4)	8978(3)	9017(2)	42(1)	1
C1	4987(3)	7619(3)	8887(3)	36(1)	1

C2A	4724(5)	8462(4)	8257(3)	26(1)	0.679(6)
C3A	6904(5)	7624(4)	7665(3)	27(1)	0.679(6)
C4A	7280(8)	6744(7)	8339(4)	23(1)	0.679(6)
O1A	6120(13)	8671(13)	8016(10)	56(2)	0.679(6)
C2B	5407(12)	8599(8)	8785(8)	40(3)	0.321(6)
C3B	7529(12)	7718(8)	8194(7)	34(3)	0.321(6)
C4B	7180(20)	6699(15)	8095(10)	26(3)	0.321(6)
O1B	5880(30)	8630(30)	7900(20)	56(2)	0.321(6)
F4	−302(8)	8595(11)	9441(4)	70(3)	0.472(14)
F5	310(13)	9987(4)	9153(4)	65(3)	0.472(14)
F6	1799(12)	8395(14)	9396(12)	35(3)	0.472(14)
F4A	−411(6)	9396(11)	9368(4)	89(4)	0.528(14)
F5A	1278(13)	9900(6)	8957(4)	79(3)	0.528(14)
F6A	1596(11)	8254(12)	9467(11)	34(2)	0.528(14)
O16	8066(2)	7851(2)	5753(2)	36(1)	1
O21	10185(4)	5472(3)	5006(3)	31(1)	0.5

Table 2. Bond lengths [Å] and angles [°].

Mn1–O3	2.1381(19)	N9–C15	1.492(3)
Mn1–O5	2.2068(19)	C7–C8	1.520(4)
Mn1–N8	2.296(2)	C9–C10	1.524(4)
Mn1–O7	2.315(2)	C12–C13	1.528(4)
Mn1–N9	2.323(2)	C14–C15	1.518(4)
Mn1–N7	2.349(2)	C16–C17	1.526(4)
Mn1–Na1	3.5679(12)	C18–C19	1.519(4)
Mn2–O8	2.115(2)	C20–C21	1.521(4)
Mn2–O13	2.1357(19)	C22–C23	1.523(4)
Mn2–O15	2.179(2)	C24–C25i	1.530(4)
Mn2–O35	2.218(2)	C25–O7i	1.257(3)
Mn2–O12	2.224(2)	C25–C24i	1.530(4)
Mn2–O10	2.248(2)	C26–C27	1.547(4)
Na1–O14	2.338(3)	C28–C29	1.532(5)
Na1–O35	2.431(2)	C29–F5	1.250(5)
Na1–O3	2.431(2)	C29–F4A	1.270(6)
Na1–O9	2.473(2)	C29–F6	1.306(8)
Na1–O7	2.584(2)	C29–F6A	1.307(7)
Na1–C25	3.054(3)	C29–F5A	1.445(6)
Na1–Na1i	4.137(2)	C29–F4	1.450(6)
F1–C27	1.335(4)	C1–C2A	1.436(6)
F2–C27	1.339(4)	C1–C2B	1.439(10)
F3–C27	1.350(3)	C2A–O1A	1.577(14)
O2–C8	1.423(4)	C3A–O1A	1.469(16)
O2–C9	1.434(4)	C3A–C4A	1.525(9)
O3–C21	1.263(3)	C2B–O1B	1.57(4)
O4–C21	1.257(3)	C3B–C4B	1.483(19)
O5–C23	1.273(3)	C3B–O1B	1.83(3)
O6–C23	1.248(3)		
O7–C25i	1.257(3)	O3–Mn1–O5	92.46(7)
O8–C26	1.257(4)	O3–Mn1–N8	165.54(8)
O9–C26	1.232(4)	O5–Mn1–N8	73.24(7)
O10–C28	1.240(4)	O3–Mn1–O7	80.53(7)
O11–C28	1.244(4)	O5–Mn1–O7	78.30(7)
O35–C25	1.262(3)	N8–Mn1–O7	98.22(8)
N1–C5	1.367(4)	O3–Mn1–N9	114.55(8)
N1–C4A	1.439(8)	O5–Mn1–N9	134.81(8)
N1–C1	1.462(4)	N8–Mn1–N9	78.26(8)
N1–C4B	1.534(18)	O7–Mn1–N9	71.86(7)
N2–C11	1.350(4)	O3–Mn1–N7	76.00(7)
N2–C5	1.352(4)	O5–Mn1–N7	146.76(8)
N3–C11	1.328(4)	N8–Mn1–N7	114.82(8)
N3–C6	1.351(4)	O7–Mn1–N7	128.56(8)
N4–C5	1.345(4)	N9–Mn1–N7	77.45(8)
N4–C6	1.347(4)	O3–Mn1–Na1	41.71(5)
N5–C6	1.355(4)	O5–Mn1–Na1	65.43(5)
N5–C7	1.461(4)	N8–Mn1–Na1	129.07(6)
N5–C10	1.462(4)	O7–Mn1–Na1	46.30(5)
N6–C11	1.401(4)	N9–Mn1–Na1	111.30(6)
N6–C19	1.477(3)	N7–Mn1–Na1	116.08(6)
N6–C12	1.478(3)	O8–Mn2–O13	173.52(9)
N7–C18	1.478(3)	O8–Mn2–O15	97.06(8)
N7–C17	1.487(4)	O13–Mn2–O15	88.10(8)
N7–C20	1.488(3)	O8–Mn2–O35	87.32(8)
N8–C22	1.480(3)	O13–Mn2–O35	89.17(8)
N8–C14	1.482(3)	O15–Mn2–O35	85.67(8)
N8–C13	1.491(4)	O8–Mn2–O12	88.20(8)
N9–C24	1.469(4)	O13–Mn2–O12	87.79(8)
N9–C16	1.487(3)	O15–Mn2–O12	164.78(9)

O35–Mn2–O12	108.91(8)
O8–Mn2–O10	90.35(8)
O13–Mn2–O10	94.52(8)
O15–Mn2–O10	78.75(8)
O35–Mn2–O10	163.85(8)
O12–Mn2–O10	86.97(8)
O14–Na1–O35	130.14(10)
O14–Na1–O3	87.01(9)
O35–Na1–O3	142.60(9)
O14–Na1–O9	87.43(11)
O35–Na1–O9	80.87(7)
O3–Na1–O9	108.76(8)
O14–Na1–O7	92.69(11)
O35–Na1–O7	99.96(7)
O3–Na1–O7	70.05(7)
O9–Na1–O7	178.79(9)
O14–Na1–C25	107.22(9)
O35–Na1–C25	23.25(7)
O3–Na1–C25	163.17(8)
O9–Na1–C25	81.45(8)
O7–Na1–C25	99.66(8)
O14–Na1–Mn1	105.52(9)
O35–Na1–Mn1	115.18(6)
O3–Na1–Mn1	35.81(5)
O9–Na1–Mn1	138.47(7)
O7–Na1–Mn1	40.36(5)
C25–Na1–Mn1	128.62(6)
O14–Na1–Na1i	90.07(9)
O35–Na1–Na1i	64.92(6)
O3–Na1–Na1i	118.83(7)
O9–Na1–Na1i	132.13(8)
O7–Na1–Na1i	49.08(5)
C25–Na1–Na1i	53.91(6)
Mn1–Na1–Na1i	87.95(4)
C8–O2–C9	111.1(2)
C21–O3–Mn1	120.32(17)
C21–O3–Na1	135.98(17)
Mn1–O3–Na1	102.48(8)
C23–O5–Mn1	112.55(17)
C25i–O7–Mn1	117.56(17)
C25i–O7–Na1	120.53(17)
Mn1–O7–Na1	93.33(7)
C26–O8–Mn2	132.73(18)
C26–O9–Na1	134.05(19)
C28–O10–Mn2	128.2(2)
C25–O35–Mn2	128.59(18)
C25–O35–Na1	107.27(17)
Mn2–O35–Na1	111.77(8)
C5–N1–C4A	124.2(4)
C5–N1–C1	121.4(3)
C4A–N1–C1	113.9(4)
C5–N1–C4B	117.2(7)
C1–N1–C4B	116.1(7)
C11–N2–C5	113.1(2)
C11–N3–C6	113.9(2)
C5–N4–C6	114.3(2)
C6–N5–C7	122.6(2)
C6–N5–C10	123.8(2)
C7–N5–C10	112.4(2)
C11–N6–C19	115.9(2)
C11–N6–C12	117.7(2)
C19–N6–C12	113.2(2)
C18–N7–C17	110.0(2)
C18–N7–C20	110.6(2)
C17–N7–C20	108.8(2)
C18–N7–Mn1	114.94(16)
C17–N7–Mn1	105.15(16)
C20–N7–Mn1	107.10(15)
C22–N8–C14	112.5(2)
C22–N8–C13	107.5(2)
C14–N8–C13	110.9(2)
C22–N8–Mn1	101.17(15)
C14–N8–Mn1	108.14(16)
C13–N8–Mn1	116.27(16)
C24–N9–C16	110.1(2)
C24–N9–C15	109.4(2)
C16–N9–C15	111.2(2)
C24–N9–Mn1	108.87(17)
C16–N9–Mn1	109.46(17)
C15–N9–Mn1	107.77(17)
N4–C5–N2	125.8(2)
N4–C5–N1	116.6(2)
N2–C5–N1	117.6(2)
N4–C6–N3	125.5(3)
N4–C6–N5	117.9(2)
N3–C6–N5	116.6(2)
N5–C7–C8	109.1(3)
O2–C8–C7	111.2(3)
O2–C9–C10	111.8(3)
N5–C10–C9	108.8(3)
N3–C11–N2	127.1(3)
N3–C11–N6	116.9(2)
N2–C11–N6	116.0(2)
N6–C12–C13	114.1(2)
N8–C13–C12	114.4(2)
N8–C14–C15	111.9(2)
N9–C15–C14	111.4(2)
N9–C16–C17	109.2(2)
N7–C17–C16	111.9(2)
N7–C18–C19	112.7(2)
N6–C19–C18	111.7(2)
N7–C20–C21	113.2(2)
O4–C21–O3	124.6(2)
O4–C21–C20	116.3(2)
O3–C21–C20	119.0(2)
N8–C22–C23	111.1(2)
O6–C23–O5	125.9(2)
O6–C23–C22	116.7(2)
O5–C23–C22	117.4(2)
N9–C24–C25i	112.5(2)
O7i–C25–O35	125.2(3)
O7i–C25–C24i	117.7(2)
O35–C25–C24i	117.1(2)
O7i–C25–Na1	81.64(16)
O35–C25–Na1	49.48(13)
C24i–C25–Na1	150.78(19)
O9–C26–O8	131.8(3)
O9–C26–C27	118.2(3)
O8–C26–C27	110.0(2)
F1–C27–F2	106.8(3)

F1–C27–F3	107.1(3)	F4A–C29–C28	116.8(4)
F2–C27–F3	106.5(3)	F6–C29–C28	115.9(11)
F1–C27–C26	111.4(3)	F6A–C29–C28	117.2(10)
F2–C27–C26	111.6(3)	F5A–C29–C28	105.0(4)
F3–C27–C26	113.0(2)	F4–C29–C28	105.2(4)
O10–C28–O11	129.3(3)	C2A–C1–N1	113.9(3)
O10–C28–C29	116.9(3)	C2B–C1–N1	114.6(5)
O11–C28–C29	113.8(3)	C1–C2A–O1A	105.9(6)
F5–C29–F6	114.0(10)	O1A–C3A–C4A	105.9(8)
F4A–C29–F6A	111.9(9)	N1–C4A–C3A	108.4(6)
F4A–C29–F5A	101.7(6)	C3A–O1A–C2A	101.4(8)
F6A–C29–F5A	101.1(7)	C1–C2B–O1B	102.7(14)
F5–C29–F4	100.1(6)	C4B–C3B–O1B	92.2(14)
F6–C29–F4	100.4(8)	C3B–C4B–N1	111.0(12)
F5–C29–C28	117.6(4)	C2B–O1B–C3B	85.8(18)

Symmetry transformations used to generate equivalent atoms:
(i) –x+1,–y+1,–z+1

Table 3. Anisotropic displacement parameters [$Å^2 \times 10^3$].

The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}]$.

Atom	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Mn1	12(1)	13(1)	21(1)	−2(1)	−2(1)	−4(1)
Mn2	13(1)	11(1)	20(1)	−2(1)	−2(1)	−4(1)
Na1	17(1)	18(1)	35(1)	−2(1)	−5(1)	−3(1)
F1	51(1)	34(1)	57(1)	−23(1)	15(1)	−26(1)
F2	24(1)	22(1)	70(2)	15(1)	−3(1)	−4(1)
F3	21(1)	26(1)	60(1)	−5(1)	2(1)	−16(1)
O15	20(1)	15(1)	27(1)	−4(1)	−8(1)	−2(1)
O2	18(1)	46(1)	36(1)	−7(1)	−2(1)	−13(1)
O3	16(1)	15(1)	23(1)	0(1)	−4(1)	−7(1)
O4	19(1)	18(1)	30(1)	3(1)	−5(1)	−10(1)
O5	14(1)	14(1)	24(1)	−3(1)	−2(1)	−6(1)
O6	13(1)	17(1)	41(1)	−1(1)	−4(1)	−4(1)
O7	19(1)	16(1)	20(1)	−3(1)	−1(1)	−3(1)
O8	20(1)	16(1)	37(1)	−3(1)	2(1)	−9(1)
O9	19(1)	17(1)	44(1)	−4(1)	2(1)	−6(1)
O10	22(1)	19(1)	25(1)	−6(1)	−5(1)	1(1)
O11	41(1)	33(1)	31(1)	−13(1)	−15(1)	16(1)
O12	17(1)	17(1)	26(1)	−5(1)	−2(1)	−3(1)
O13	16(1)	15(1)	30(1)	−1(1)	−2(1)	−6(1)
O14	29(1)	98(2)	29(1)	10(1)	−7(1)	−27(2)
O35	17(1)	15(1)	18(1)	−1(1)	−3(1)	−4(1)
N1	18(1)	13(1)	31(1)	−2(1)	1(1)	−5(1)
N2	14(1)	16(1)	21(1)	−2(1)	−1(1)	−5(1)
N3	16(1)	18(1)	20(1)	−2(1)	−2(1)	−6(1)
N4	12(1)	17(1)	21(1)	−2(1)	−2(1)	−4(1)
N5	15(1)	22(1)	28(1)	−4(1)	0(1)	−7(1)
N6	13(1)	14(1)	23(1)	−3(1)	−1(1)	−4(1)
N7	15(1)	12(1)	20(1)	−1(1)	−3(1)	−4(1)
N8	15(1)	12(1)	21(1)	−2(1)	−2(1)	−3(1)
N9	15(1)	15(1)	21(1)	−2(1)	−4(1)	−4(1)
C5	16(1)	18(1)	17(1)	1(1)	−3(1)	−6(1)
C6	15(1)	19(1)	15(1)	−1(1)	−4(1)	−5(1)
C7	18(1)	26(1)	29(2)	−3(1)	−1(1)	−11(1)
C8	23(2)	39(2)	31(2)	−10(1)	1(1)	−16(1)
C9	18(1)	38(2)	30(2)	−2(1)	−3(1)	−7(1)
C10	14(1)	26(1)	27(2)	−6(1)	1(1)	−4(1)
C11	16(1)	18(1)	16(1)	0(1)	−3(1)	−4(1)
C12	19(1)	13(1)	23(1)	−1(1)	−2(1)	−6(1)
C13	19(1)	15(1)	21(1)	0(1)	−1(1)	−7(1)
C14	20(1)	12(1)	24(1)	−2(1)	−3(1)	−4(1)
C15	16(1)	14(1)	25(1)	−5(1)	−5(1)	−3(1)
C16	14(1)	20(1)	21(1)	−4(1)	−4(1)	−1(1)
C17	13(1)	22(1)	21(1)	−1(1)	−1(1)	−5(1)
C18	15(1)	14(1)	20(1)	−1(1)	−3(1)	−4(1)
C19	14(1)	17(1)	19(1)	−1(1)	−5(1)	−6(1)
C20	15(1)	16(1)	26(2)	0(1)	−5(1)	−8(1)
C21	13(1)	18(1)	18(1)	−1(1)	0(1)	−5(1)
C22	14(1)	14(1)	26(2)	−3(1)	−3(1)	−6(1)
C23	13(1)	15(1)	18(1)	−1(1)	−1(1)	−4(1)
C24	15(1)	23(1)	20(1)	−2(1)	−3(1)	−4(1)
C25	14(1)	13(1)	20(1)	−1(1)	−2(1)	−7(1)
C26	19(1)	15(1)	27(2)	−3(1)	−2(1)	−7(1)
C27	19(1)	16(1)	44(2)	−5(1)	4(1)	−8(1)
C28	24(1)	21(1)	26(2)	−6(1)	−6(1)	0(1)
C29	43(2)	35(2)	32(2)	−13(1)	−13(1)	17(1)
C1	23(2)	16(1)	65(2)	−3(1)	8(2)	−3(1)
C2A	21(2)	17(2)	35(3)	0(2)	−1(2)	−1(2)

C3A	27(2)	20(2)	33(3)	0(2)	2(2)	–9(2)
C4A	16(2)	21(2)	30(4)	–2(2)	2(2)	–6(2)
O1A	55(4)	18(2)	80(5)	7(2)	35(3)	–3(3)
C2B	38(5)	15(4)	66(6)	–5(4)	12(4)	–12(3)
C3B	38(5)	16(4)	49(6)	–6(4)	13(4)	–15(3)
C4B	27(5)	16(4)	37(7)	–2(5)	5(5)	–10(3)
O1B	55(4)	18(2)	80(5)	7(2)	35(3)	–3(3)
F4	52(4)	117(7)	40(3)	–23(4)	13(3)	–25(4)
F5	109(8)	25(2)	35(4)	–17(2)	–31(4)	26(3)
F6	32(4)	37(5)	28(5)	–5(3)	–11(4)	1(3)
F4A	46(3)	135(9)	43(3)	–45(5)	–8(2)	42(3)
F5A	149(8)	36(3)	55(4)	–6(2)	–47(4)	–25(4)
F6A	36(4)	33(3)	26(3)	–3(2)	–7(3)	2(3)
O16	21(1)	26(1)	56(2)	12(1)	–7(1)	–3(1)
O21	28(2)	37(3)	26(2)	1(2)	2(2)	–8(2)

Table 4. Hydrogen coordinates [$\times 10^4$] and isotropic displacement parameters [$Å^2 \times 10^3$].

Atom	x	y	z	U_{eq}	S.o.f.
H15A	3799	6211	7865	31	1
H15B	4174	5485	7228	31	1
H12A	−415	7932	6375	30	1
H12B	−303	8731	6887	30	1
H13A	590	6031	6757	30	1
H13B	1918	5252	6895	30	1
H14A	8108	5515	4828	76	1
H14B	7038	6063	4310	76	1
H7A	2459	3339	10004	28	1
H7B	3621	2870	9334	28	1
H8A	2104	3760	8369	35	1
H8B	1456	2972	8936	35	1
H9A	92	6226	9160	35	1
H9B	1265	5748	8501	35	1
H10A	2260	6188	9536	28	1
H10B	1613	5434	10140	28	1
H12C	6887	1594	9057	22	1
H12D	8012	1080	8385	22	1
H13C	5943	903	8110	22	1
H13D	5197	2211	8171	22	1
H14C	6772	251	6833	22	1
H14D	8048	478	7146	22	1
H15C	8421	322	5804	22	1
H15D	6993	1263	5657	22	1
H16A	10464	1075	5715	23	1
H16B	9934	847	6593	23	1
H17A	11283	2048	6552	23	1
H17B	10280	2925	5975	23	1
H18A	10793	2137	7913	20	1
H18B	9848	1431	7733	20	1
H19A	9197	2112	8969	20	1
H19B	8978	3364	8617	20	1
H20A	10588	3968	6937	22	1
H20B	9568	4176	7696	22	1
H22A	4431	1817	7030	21	1
H22B	5164	2150	6236	21	1
H24A	8565	1826	4773	24	1
H24B	9147	2743	5045	24	1
H1A	4138	7448	9051	44	1
H1B	5249	7920	9332	44	1
H2A1	4030	9156	8424	31	0.679(6)
H2A2	4397	8202	7815	31	0.679(6)
H3A1	7731	7701	7370	32	0.679(6)
H3A2	6351	7418	7307	32	0.679(6)
H4A1	7900	6033	8146	27	0.679(6)
H4A2	7748	6994	8721	27	0.679(6)
H2B1	6164	8526	9112	48	0.321(6)
H2B2	4643	9276	8911	48	0.321(6)
H3B1	8296	7794	7837	41	0.321(6)
H3B2	7691	7795	8736	41	0.321(6)
H4B1	7996	6035	8152	32	0.321(6)
H4B2	6872	6736	7567	32	0.321(6)
H16C	7196	8198	5763	55	1
H16D	8216	7191	5988	55	1
H21A	10033	5632	5490	47	0.5
H21B	10406	6003	4730	47	0.5

A.5 – VANADIUM(IV) CRYSTAL STRUCTURE OF TZMORPH2DO3A

Table 1. Crystal data and structure refinement details.

Identification code	**2015ncs0127_R2 (third type of crystal)**	
Empirical formula	$C_{75}H_{120}N_{27}O_{27}V_3$	
Formula weight	1984.79	
Temperature	293(2) K	
Wavelength	0.71075 Å	
Crystal system	Monoclinic	
Space group	*C*2/*c*	
Unit cell dimensions	*a* = 25.139(16) Å	α = 90°
	b = 19.357(12) Å	β = 98.828(8)°
	c = 46.62(3) Å	γ= 90°
Volume	22417(25) Å^3	
Z	8	
Density (calculated)	1.176 Mg / m^3	
Absorption coefficient	0.319 mm^{-1}	
F(000)	8352	
Crystal	Needle; light purple	
Crystal size	0.25 x0.02 x0.01 mm^3	
θ range for data collection	2.472 – 27.565°	
Index ranges	$-32 \le h \le 32$, $-25 \le k \le 25$, $-59 \le l \le 60$	
Reflections collected	166542	
Independent reflections	25813 [R_{int} = 0.4002]	
Completeness to θ = 25.242°	99.9 %	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	25813 / 2502 / 1180	
Goodness-of-fit on F^2	0.662	
Final *R* indices [$F^2 > 2\sigma(F^2)$]	*R1* = 0.0762, *wR2* = 0.1995	
R indices (all data)	*R1* = 0.2046, *wR2* = 0.2274	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.792 and –0.426 e Å^{-3}	

Diffractometer: Rigaku AFC12 goniometer equipped with an enhanced sensitivity (HG) Saturn724+ detector mounted at the window of an FR-E+ SuperBright molybdenum rotating anode generator with HF Varimax optics (100m focus). **Cell determination and data collection:** CrystalClear-SM Expe. rt 3.1 b27 (Rigaku, 2012). **Data reduction, cell refinement and absorption correction:** CrystalClear-SM Expert 3.1 b18 (Rigaku, 2012)**Structure solution:** SUPERFLIP (Palatinus, L. & Chapuis, G. (2007). J. Appl. Cryst. 40, 786-790). **Structure refinement:** SHELXL-2013 (Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122).

Special details:

Among other crystal morphologies, sample contains very small amount of crystals this type: small and thin light purple needles. The crystal found and examined gave poor quality diffraction pattern, which is reflected in the overall quality of the obtained crystal structure, which proves atomic connectivity, however data itself may not be publishable. Crystal structure contains three independent molecules present in the asymmetric part of the unit cell (Z'=3) . Despite of using number of restraints some atoms in the disordered parts are NPD (non positive definite). An attempt of modelling such disorder was made, however results were not satisfactory. EADP constraints had to be applied for NPD atoms. Additionally, highly diffused el.density of the solvent molecules have been masked out with SMTBX toolbox within OLEX2 software, which improves significantly overall modell.

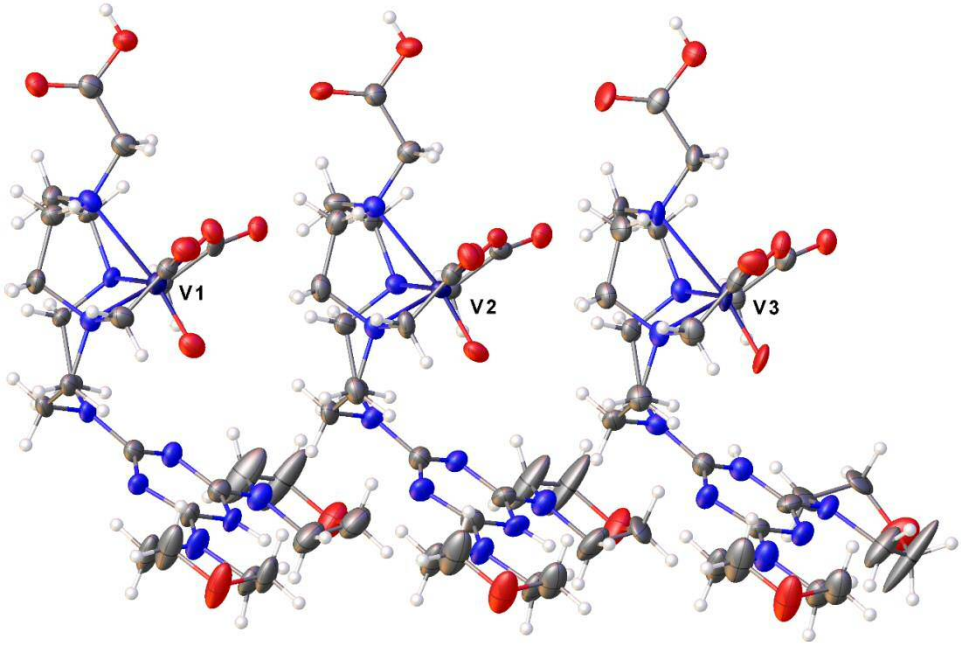

Figure 1. Asymmetric part of the unit cell containing three independent molecules (Z'=3). Atominc Displacement ellipsoids – 50% probability

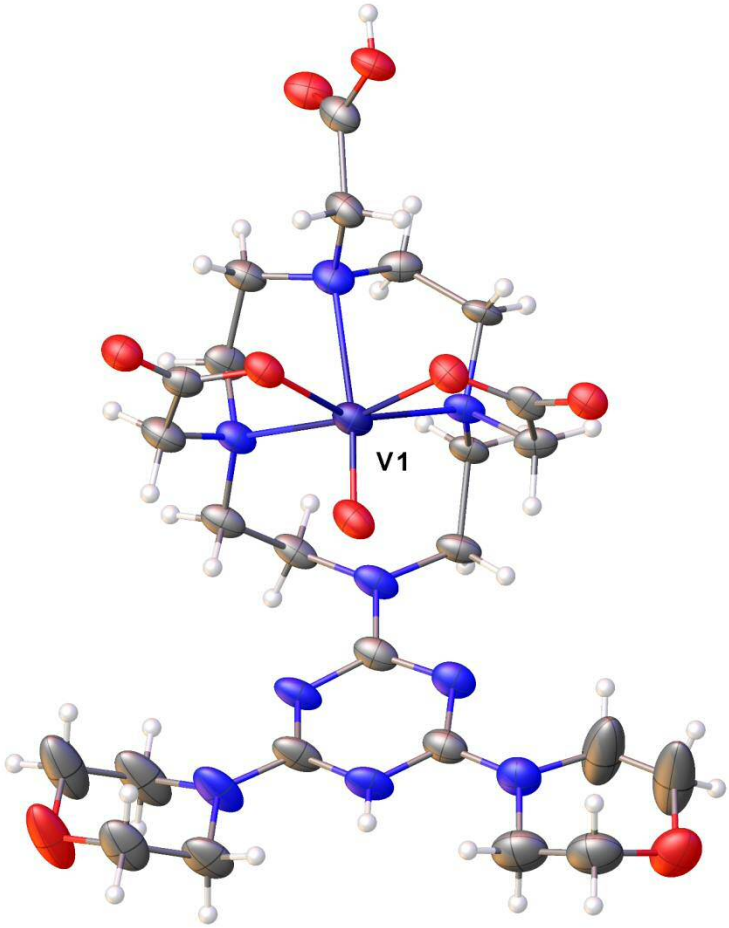

Figure 2. One independent molecule present in the asymmetric part of the unit cell. Atomic displacement ellipsoids – 50% probability.

Table 1. Atomic coordinates [$\times 10^4$], equivalent isotropic displacement parameters [$Å^2 \times 10^3$] and site occupancy factors. U_{eq} is defined as one third of the trace of the orthogonalized U^{ij} tensor.

Atom	x	y	z	U_{eq}	S.o.f.
V1	6606(1)	3850(1)	6498(1)	31(1)	1
O1	3545(3)	2705(4)	6097(2)	80(2)	1
O2	6145(3)	2289(3)	4794(1)	64(2)	1
O3	6037(2)	3184(3)	7213(1)	37(1)	1
O4	6534(2)	3329(3)	6860(1)	33(1)	1
O5	7290(2)	3335(3)	6466(1)	34(1)	1
O6	7788(2)	3004(3)	6134(1)	32(1)	1
O7	6197(2)	3442(3)	6260(1)	39(1)	1
O8	7776(2)	5176(3)	7360(1)	40(1)	1
O9	8297(2)	4227(3)	7381(1)	37(1)	1
N1	5087(3)	3008(3)	5552(1)	38(2)	1
N2	5075(3)	4005(3)	5854(1)	36(2)	1
N3	5813(3)	3836(3)	5583(1)	36(2)	1
N4	4402(3)	3198(4)	5833(2)	53(2)	1
N5	5761(3)	2904(4)	5270(1)	42(2)	1
N6	5829(3)	4709(3)	5932(1)	33(1)	1
N7	5951(2)	4462(3)	6635(1)	31(1)	1
N8	7031(2)	4545(3)	6232(1)	28(1)	1
N9	7087(3)	4596(3)	6854(1)	34(1)	1
C1	4870(3)	3398(4)	5746(2)	41(2)	1
C2	4121(4)	2565(5)	5726(2)	52(2)	1
C3	3873(4)	2243(5)	5961(2)	59(3)	1
C4	3847(4)	3302(5)	6202(3)	81(3)	1
C5	4076(4)	3668(5)	5977(2)	71(3)	1
C6	5544(3)	3269(4)	5472(2)	37(2)	1
C7	6193(5)	3211(6)	5138(2)	106(4)	1
C8	6409(5)	2853(7)	4949(3)	123(5)	1
C9	5853(4)	1899(5)	4994(2)	63(2)	1
C10	5491(4)	2331(5)	5118(2)	63(2)	1
C11	5566(3)	4167(4)	5786(2)	34(2)	1
C12	5583(3)	5098(4)	6147(2)	33(2)	1
C13	5475(3)	4696(4)	6415(2)	33(2)	1
C14	6293(3)	5006(4)	5824(2)	32(2)	1
C15	6779(3)	5144(4)	6058(2)	27(2)	1
C16	7237(3)	4030(4)	6037(2)	31(2)	1
C17	7462(3)	3407(4)	6221(2)	29(2)	1
C18	6119(3)	3472(4)	6984(2)	31(2)	1
C19	5723(3)	3984(4)	6836(2)	37(2)	1
C20	6175(3)	5083(4)	6803(2)	35(2)	1
C21	6685(3)	4908(4)	7014(2)	34(2)	1
C22	7346(3)	5143(4)	6697(1)	32(2)	1
C23	7515(3)	4830(4)	6430(1)	29(2)	1
C24	7504(3)	4207(4)	7047(2)	35(2)	1
C25	7875(3)	4588(5)	7282(2)	35(2)	1
V2	6609(1)	520(1)	6499(1)	29(1)	1
O10	3546(3)	−631(4)	6096(2)	78(2)	1
O11	6146(3)	−1041(3)	4792(1)	64(2)	1
O12	7786(2)	−324(3)	6133(1)	35(1)	1
O13	7288(2)	−6(3)	6467(1)	27(1)	1
O14	6528(2)	−1(3)	6859(1)	33(1)	1
O15	6032(2)	−153(3)	7210(1)	38(1)	1
O16	6199(2)	114(3)	6261(1)	38(1)	1
O17	7780(2)	1840(3)	7358(1)	37(1)	1
O18	8294(2)	899(3)	7381(1)	37(1)	1
N10	5087(3)	−325(4)	5556(2)	45(2)	1
N11	5081(3)	681(4)	5858(2)	44(2)	1
N12	5807(3)	500(4)	5586(1)	37(2)	1

N13	4405(3)	−136(4)	5827(2)	48(2)	1
N14	5764(3)	−424(4)	5270(2)	45(2)	1
N15	5834(3)	1367(4)	5933(1)	37(2)	1
N16	5946(3)	1132(3)	6635(1)	32(1)	1
N17	7030(3)	1208(3)	6230(1)	29(1)	1
N18	7091(2)	1267(3)	6858(1)	29(1)	1
C26	4872(4)	89(5)	5748(2)	45(2)	1
C27	4121(4)	−773(5)	5729(2)	57(2)	1
C28	3874(4)	−1105(5)	5967(2)	64(3)	1
C29	3855(4)	−51(6)	6207(3)	86(3)	1
C30	4080(4)	326(5)	5974(2)	69(3)	1
C31	5541(3)	−69(5)	5478(2)	38(2)	1
C32	6191(5)	−127(6)	5136(2)	106(5)	1
C33	6417(5)	−481(7)	4956(3)	123(5)	1
C34	5850(4)	−1447(4)	4989(2)	55(2)	1
C35	5493(4)	−998(5)	5117(2)	69(3)	1
C36	5563(3)	821(4)	5782(2)	38(2)	1
C37	5587(3)	1756(4)	6146(2)	35(2)	1
C38	5482(3)	1346(4)	6418(2)	36(2)	1
C39	6303(3)	1672(4)	5830(2)	36(2)	1
C40	6779(3)	1824(4)	6055(2)	35(2)	1
C41	7242(3)	687(4)	6039(1)	30(2)	1
C42	7469(3)	67(4)	6223(2)	30(2)	1
C43	6120(3)	138(4)	6989(2)	31(2)	1
C44	5717(3)	650(4)	6835(2)	34(2)	1
C45	6172(3)	1741(4)	6804(2)	37(2)	1
C46	6677(3)	1580(4)	7014(2)	34(2)	1
C47	7345(3)	1805(4)	6695(2)	35(2)	1
C48	7515(3)	1499(4)	6429(1)	28(2)	1
C49	7499(3)	869(4)	7045(2)	31(2)	1
C50	7868(4)	1257(4)	7270(2)	34(2)	1
V3	6602(1)	−2815(1)	6496(1)	28(1)	1
O19	3547(3)	−3968(4)	6097(2)	84(2)	1
O20	6129(2)	−4360(4)	4790(1)	73(2)	1
O21	7785(2)	−3662(3)	6135(1)	34(1)	1
O22	7281(2)	−3331(3)	6466(1)	34(1)	1
O23	6530(2)	−3335(3)	6857(1)	33(1)	1
O24	6029(2)	−3488(3)	7214(1)	41(1)	1
O25	6196(2)	−3225(3)	6256(1)	37(1)	1
O26	7784(2)	−1487(3)	7360(1)	44(2)	1
O27	8295(2)	−2440(3)	7379(1)	39(1)	1
N19	5081(3)	−3677(4)	5550(1)	43(2)	1
N20	5077(3)	−2662(4)	5857(2)	43(2)	1
N21	5787(3)	−2840(4)	5587(1)	41(2)	1
N22	4411(3)	−3464(4)	5832(2)	55(2)	1
N23	5779(3)	−3755(3)	5276(1)	43(2)	1
N24	5827(3)	−1975(4)	5934(1)	38(2)	1
N25	5950(3)	−2206(3)	6636(1)	34(1)	1
N26	7031(3)	−2129(3)	6230(1)	32(1)	1
N27	7089(2)	−2058(3)	6856(1)	29(1)	1
C51	4877(3)	−3259(5)	5734(2)	40(2)	1
C52	4083(4)	−3006(5)	5979(2)	66(3)	1
C53	3851(4)	−3379(6)	6202(3)	82(3)	1
C54	3879(4)	−4447(5)	5965(2)	63(3)	1
C55	4123(4)	−4095(5)	5725(2)	55(2)	1
C56	5541(4)	−3412(5)	5474(2)	41(2)	1
C57	6121(3)	−3371(4)	5095(2)	53(1)	1
C58	6483(3)	−3899(4)	4987(2)	53(1)	1
C59	5702(5)	−4618(7)	4921(3)	166(6)	1
C60	5515(4)	−4392(5)	5131(2)	72(3)	1

C61	5554(4)	−2509(5)	5789(2)	39(2)	1
C62	5577(3)	−1578(4)	6142(2)	38(2)	1
C63	5473(4)	−1984(5)	6417(2)	45(2)	1
C64	6294(3)	−1659(4)	5827(2)	35(2)	1
C65	6774(3)	−1533(4)	6058(2)	31(2)	1
C66	7233(3)	−2648(4)	6034(2)	32(2)	1
C67	7455(3)	−3269(4)	6218(2)	34(2)	1
C68	7504(3)	−1835(4)	6428(2)	35(2)	1
C69	7350(3)	−1522(4)	6696(2)	34(2)	1
C70	6674(3)	−1755(4)	7018(2)	36(2)	1
C71	6164(3)	−1599(4)	6803(2)	37(2)	1
C72	5712(3)	−2699(4)	6833(2)	40(2)	1
C73	6132(3)	−3206(4)	6992(2)	34(2)	1
C74	7498(3)	−2474(4)	7050(2)	34(2)	1
C75	7862(3)	−2060(4)	7277(1)	29(2)	1

Table 2. Bond lengths [Å] and angles [°].

V1–O4	2.000(5)	C9–C10	1.420(12)
V1–O5	2.012(5)	C10–H10A	0.9700
V1–O7	1.599(6)	C10–H10B	0.9700
V1–N7	2.201(6)	C12–H12A	0.9700
V1–N8	2.213(6)	C12–H12B	0.9700
V1–N9	2.385(7)	C12–C13	1.532(9)
O1–C3	1.429(9)	C13–H13A	0.9700
O1–C4	1.428(11)	C13–H13B	0.9700
O2–C8	1.416(11)	C14–H14A	0.9700
O2–C9	1.479(10)	C14–H14B	0.9700
O3–C18	1.250(8)	C14–C15	1.530(9)
O4–C18	1.296(9)	C15–H15A	0.9700
O5–C17	1.291(8)	C15–H15B	0.9700
O6–C17	1.242(8)	C16–H16A	0.9700
O8–C25	1.231(9)	C16–H16B	0.9700
O9–H9	0.8200	C16–C17	1.537(10)
O9–C25	1.297(9)	C18–C19	1.495(10)
N1–H1	0.8600	C19–H19A	0.9700
N1–C1	1.353(9)	C19–H19B	0.9700
N1–C6	1.359(9)	C20–H20A	0.9700
N2–C1	1.348(10)	C20–H20B	0.9700
N2–C11	1.357(10)	C20–C21	1.528(10)
N3–C6	1.351(9)	C21–H21A	0.9700
N3–C11	1.369(9)	C21–H21B	0.9700
N4–C1	1.358(10)	C22–H22A	0.9700
N4–C2	1.462(10)	C22–H22B	0.9700
N4–C5	1.456(11)	C22–C23	1.504(9)
N5–C6	1.357(9)	C23–H23A	0.9700
N5–C7	1.456(10)	C23–H23B	0.9700
N5–C10	1.430(10)	C24–H24A	0.9700
N6–C11	1.364(9)	C24–H24B	0.9700
N6–C12	1.467(9)	C24–C25	1.515(11)
N6–C14	1.455(9)	V2–O13	2.014(5)
N7–C13	1.521(9)	V2–O14	1.995(5)
N7–C19	1.491(9)	V2–O16	1.600(5)
N7–C20	1.496(9)	V2–N16	2.216(6)
N8–C15	1.498(9)	V2–N17	2.209(6)
N8–C16	1.494(9)	V2–N18	2.398(6)
N8–C23	1.512(8)	O10–C28	1.427(10)
N9–C21	1.474(9)	O10–C29	1.418(11)
N9–C22	1.492(9)	O11–C33	1.437(10)
N9–C24	1.481(9)	O11–C34	1.493(10)
C2–H2A	0.9700	O12–C42	1.221(9)
C2–H2B	0.9700	O13–C42	1.292(8)
C2–C3	1.479(11)	O14–C43	1.296(8)
C3–H3A	0.9700	O15–C43	1.226(8)
C3–H3B	0.9700	O17–C50	1.232(9)
C4–H4A	0.9700	O18–H18	0.8200
C4–H4B	0.9700	O18–C50	1.314(9)
C4–C5	1.459(13)	N10–H10	0.8600
C5–H5A	0.9700	N10–C26	1.372(10)
C5–H5B	0.9700	N10–C31	1.345(9)
C7–H7A	0.9700	N11–C26	1.332(11)
C7–H7B	0.9700	N11–C36	1.341(10)
C7–C8	1.301(12)	N12–C31	1.345(10)
C8–H8A	0.9700	N12–C36	1.328(9)
C8–H8B	0.9700	N13–C26	1.356(10)
C9–H9A	0.9700	N13–C27	1.460(10)
C9–H9B	0.9700	N13–C30	1.455(10)

N14–C31	1.376(10)
N14–C32	1.441(11)
N14–C35	1.434(10)
N15–C36	1.389(10)
N15–C37	1.457(9)
N15–C39	1.466(10)
N16–C38	1.483(9)
N16–C44	1.493(9)
N16–C45	1.481(9)
N17–C40	1.525(9)
N17–C41	1.495(9)
N17–C48	1.524(9)
N18–C46	1.487(9)
N18–C47	1.490(9)
N18–C49	1.459(9)
C27–H27A	0.9700
C27–H27B	0.9700
C27–C28	1.496(12)
C28–H28A	0.9700
C28–H28B	0.9700
C29–H29A	0.9700
C29–H29B	0.9700
C29–C30	1.491(14)
C30–H30A	0.9700
C30–H30B	0.9700
C32–H32A	0.9700
C32–H32B	0.9700
C32–C33	1.280(12)
C33–H33A	0.9700
C33–H33B	0.9700
C34–H34A	0.9700
C34–H34B	0.9700
C34–C35	1.442(12)
C35–H35A	0.9700
C35–H35B	0.9700
C37–H37A	0.9700
C37–H37B	0.9700
C37–C38	1.552(9)
C38–H38A	0.9700
C38–H38B	0.9700
C39–H39A	0.9700
C39–H39B	0.9700
C39–C40	1.496(10)
C40–H40A	0.9700
C40–H40B	0.9700
C41–H41A	0.9700
C41–H41B	0.9700
C41–C42	1.535(10)
C43–C44	1.518(10)
C44–H44A	0.9700
C44–H44B	0.9700
C45–H45A	0.9700
C45–H45B	0.9700
C45–C46	1.513(10)
C46–H46A	0.9700
C46–H46B	0.9700
C47–H47A	0.9700
C47–H47B	0.9700
C47–C48	1.491(9)
C48–H48A	0.9700
C48–H48B	0.9700
C49–H49A	0.9700
C49–H49B	0.9700
C49–C50	1.492(10)
V3–O22	2.002(5)
V3–O23	1.990(5)
V3–O25	1.605(5)
V3–N25	2.197(6)
V3–N26	2.210(6)
V3–N27	2.414(6)
O19–C53	1.417(11)
O19–C54	1.447(10)
O20–C58	1.478(10)
O20–C59	1.407(9)
O21–C67	1.232(8)
O22–C67	1.300(8)
O23–C73	1.285(8)
O24–C73	1.235(8)
O26–C75	1.201(9)
O27–H27	0.8200
O27–C75	1.340(9)
N19–C51	1.336(10)
N19–C56	1.361(10)
N20–C51	1.351(10)
N20–C61	1.321(10)
N21–H21	0.8600
N21–C56	1.338(10)
N21–C61	1.343(10)
N22–C51	1.380(11)
N22–C52	1.454(11)
N22–C55	1.466(11)
N23–C56	1.349(10)
N23–C57	1.491(10)
N23–C60	1.512(10)
N24–C61	1.363(10)
N24–C62	1.453(9)
N24–C64	1.476(9)
N25–C63	1.512(10)
N25–C71	1.466(9)
N25–C72	1.511(9)
N26–C65	1.494(9)
N26–C66	1.500(9)
N26–C68	1.501(9)
N27–C69	1.489(9)
N27–C70	1.498(9)
N27–C74	1.496(9)
C52–H52A	0.9700
C52–H52B	0.9700
C52–C53	1.458(13)
C53–H53A	0.9700
C53–H53B	0.9700
C54–H54A	0.9700
C54–H54B	0.9700
C54–C55	1.517(12)
C55–H55A	0.9700
C55–H55B	0.9700
C57–H57A	0.9700
C57–H57B	0.9700
C57–C58	1.506(10)
C58–H58A	0.9700

C58–H58B	0.9700
C59–H59A	0.9700
C59–H59B	0.9700
C59–C60	1.229(10)
C60–H60A	0.9700
C60–H60B	0.9700
C62–H62A	0.9700
C62–H62B	0.9700
C62–C63	1.559(10)
C63–H63A	0.9700
C63–H63B	0.9700
C64–H64A	0.9700
C64–H64B	0.9700
C64–C65	1.510(10)
C65–H65A	0.9700
C65–H65B	0.9700
C66–H66A	0.9700
C66–H66B	0.9700
C66–C67	1.532(11)
C68–H68A	0.9700
C68–H68B	0.9700
C68–C69	1.490(9)
C69–H69A	0.9700
C69–H69B	0.9700
C70–H70A	0.9700
C70–H70B	0.9700
C70–C71	1.531(10)
C71–H71A	0.9700
C71–H71B	0.9700
C72–H72A	0.9700
C72–H72B	0.9700
C72–C73	1.543(11)
C74–H74A	0.9700
C74–H74B	0.9700
C74–C75	1.517(10)

O4–V1–O5	90.1(2)
O4–V1–N7	81.8(2)
O4–V1–N8	153.4(2)
O4–V1–N9	79.6(2)
O5–V1–N7	166.7(2)
O5–V1–N8	76.6(2)
O5–V1–N9	90.2(2)
O7–V1–O4	101.9(2)
O7–V1–O5	99.8(3)
O7–V1–N7	92.2(3)
O7–V1–N8	103.1(2)
O7–V1–N9	169.9(3)
N7–V1–N8	106.4(2)
N7–V1–N9	78.1(2)
N8–V1–N9	77.5(2)
C3–O1–C4	110.3(7)
C8–O2–C9	108.1(7)
C18–O4–V1	116.8(5)
C17–O5–V1	114.8(5)
C25–O9–H9	109.5
C1–N1–H1	122.6
C6–N1–H1	122.6
C6–N1–C1	114.9(7)
C1–N2–C11	115.0(8)
C6–N3–C11	113.1(7)
C1–N4–C2	122.2(8)
C1–N4–C5	122.2(7)
C5–N4–C2	113.9(7)
C6–N5–C7	119.2(7)
C6–N5–C10	122.6(7)
C10–N5–C7	115.7(7)
C11–N6–C12	120.7(7)
C11–N6–C14	119.1(7)
C14–N6–C12	118.5(6)
C13–N7–V1	120.6(4)
C19–N7–V1	103.0(4)
C19–N7–C13	105.6(5)
C19–N7–C20	108.7(6)
C20–N7–V1	110.3(5)
C20–N7–C13	107.9(6)
C15–N8–V1	124.9(5)
C15–N8–C23	106.4(6)
C16–N8–V1	100.2(4)
C16–N8–C15	110.2(5)
C16–N8–C23	107.0(5)
C23–N8–V1	107.0(4)
C21–N9–V1	106.7(4)
C21–N9–C22	110.0(6)
C21–N9–C24	112.1(6)
C22–N9–V1	107.5(4)
C24–N9–V1	110.6(5)
C24–N9–C22	109.8(6)
N1–C1–N4	119.6(7)
N2–C1–N1	124.5(8)
N2–C1–N4	115.9(8)
N4–C2–H2A	109.7
N4–C2–H2B	109.7
N4–C2–C3	109.7(7)
H2A–C2–H2B	108.2
C3–C2–H2A	109.7
C3–C2–H2B	109.7
O1–C3–C2	113.8(8)
O1–C3–H3A	108.8
O1–C3–H3B	108.8
C2–C3–H3A	108.8
C2–C3–H3B	108.8
H3A–C3–H3B	107.7
O1–C4–H4A	109.0
O1–C4–H4B	109.0
O1–C4–C5	113.1(9)
H4A–C4–H4B	107.8
C5–C4–H4A	109.0
C5–C4–H4B	109.0
N4–C5–C4	110.0(8)
N4–C5–H5A	109.7
N4–C5–H5B	109.7
C4–C5–H5A	109.7
C4–C5–H5B	109.7
H5A–C5–H5B	108.2
N1–C6–N5	116.4(7)
N3–C6–N1	126.2(8)
N3–C6–N5	117.3(7)
N5–C7–H7A	107.5
N5–C7–H7B	107.5

H7A–C7–H7B	107.0
C8–C7–N5	119.1(10)
C8–C7–H7A	107.5
C8–C7–H7B	107.5
O2–C8–H8A	106.6
O2–C8–H8B	106.6
C7–C8–O2	122.9(9)
C7–C8–H8A	106.6
C7–C8–H8B	106.6
H8A–C8–H8B	106.6
O2–C9–H9A	109.3
O2–C9–H9B	109.3
H9A–C9–H9B	108.0
C10–C9–O2	111.5(8)
C10–C9–H9A	109.3
C10–C9–H9B	109.3
N5–C10–C9	112.0(8)
N5–C10–H10A	109.2
N5–C10–H10B	109.2
C9–C10–H10A	109.2
C9–C10–H10B	109.2
H10A–C10–H10B	107.9
N2–C11–N3	125.4(7)
N2–C11–N6	116.9(7)
N6–C11–N3	117.7(7)
N6–C12–H12A	108.2
N6–C12–H12B	108.2
N6–C12–C13	116.3(6)
H12A–C12–H12B	107.4
C13–C12–H12A	108.2
C13–C12–H12B	108.2
N7–C13–C12	118.9(6)
N7–C13–H13A	107.6
N7–C13–H13B	107.6
C12–C13–H13A	107.6
C12–C13–H13B	107.6
H13A–C13–H13B	107.0
N6–C14–H14A	108.6
N6–C14–H14B	108.6
N6–C14–C15	114.6(6)
H14A–C14–H14B	107.6
C15–C14–H14A	108.6
C15–C14–H14B	108.6
N8–C15–C14	118.4(6)
N8–C15–H15A	107.7
N8–C15–H15B	107.7
C14–C15–H15A	107.7
C14–C15–H15B	107.7
H15A–C15–H15B	107.1
N8–C16–H16A	110.0
N8–C16–H16B	110.0
N8–C16–C17	108.5(6)
H16A–C16–H16B	108.4
C17–C16–H16A	110.0
C17–C16–H16B	110.0
O5–C17–C16	115.6(7)
O6–C17–O5	123.3(7)
O6–C17–C16	121.1(7)
O3–C18–O4	122.2(8)
O3–C18–C19	120.3(7)
O4–C18–C19	117.5(7)
N7–C19–C18	114.0(6)
N7–C19–H19A	108.7
N7–C19–H19B	108.7
C18–C19–H19A	108.7
C18–C19–H19B	108.7
H19A–C19–H19B	107.6
N7–C20–H20A	109.3
N7–C20–H20B	109.3
N7–C20–C21	111.7(6)
H20A–C20–H20B	107.9
C21–C20–H20A	109.3
C21–C20–H20B	109.3
N9–C21–C20	109.8(6)
N9–C21–H21A	109.7
N9–C21–H21B	109.7
C20–C21–H21A	109.7
C20–C21–H21B	109.7
H21A–C21–H21B	108.2
N9–C22–H22A	110.0
N9–C22–H22B	110.0
N9–C22–C23	108.7(6)
H22A–C22–H22B	108.3
C23–C22–H22A	110.0
C23–C22–H22B	110.0
N8–C23–H23A	109.6
N8–C23–H23B	109.6
C22–C23–N8	110.4(6)
C22–C23–H23A	109.6
C22–C23–H23B	109.6
H23A–C23–H23B	108.1
N9–C24–H24A	107.5
N9–C24–H24B	107.5
N9–C24–C25	119.4(7)
H24A–C24–H24B	107.0
C25–C24–H24A	107.5
C25–C24–H24B	107.5
O8–C25–O9	125.7(8)
O8–C25–C24	122.3(8)
O9–C25–C24	112.0(8)
O13–V2–N16	167.2(2)
O13–V2–N17	77.1(2)
O13–V2–N18	90.7(2)
O14–V2–O13	90.2(2)
O14–V2–N16	81.3(2)
O14–V2–N17	154.5(2)
O14–V2–N18	79.8(2)
O16–V2–O13	99.4(2)
O16–V2–O14	101.4(3)
O16–V2–N16	91.7(3)
O16–V2–N17	102.5(3)
O16–V2–N18	169.8(3)
N16–V2–N18	78.4(2)
N17–V2–N16	106.8(2)
N17–V2–N18	78.3(2)
C29–O10–C28	110.2(7)
C33–O11–C34	107.9(7)
C42–O13–V2	115.2(5)
C43–O14–V2	118.6(5)
C50–O18–H18	109.5

C26–N10–H10	123.3
C31–N10–H10	123.3
C31–N10–C26	113.4(7)
C26–N11–C36	113.1(8)
C36–N12–C31	112.8(7)
C26–N13–C27	126.1(8)
C26–N13–C30	120.2(7)
C30–N13–C27	112.5(7)
C31–N14–C32	121.1(8)
C31–N14–C35	121.6(7)
C35–N14–C32	114.9(8)
C36–N15–C37	120.8(7)
C36–N15–C39	119.6(7)
C37–N15–C39	117.7(6)
C38–N16–V2	120.0(5)
C38–N16–C44	105.2(6)
C44–N16–V2	103.0(4)
C45–N16–V2	109.5(5)
C45–N16–C38	109.7(6)
C45–N16–C44	108.6(6)
C40–N17–V2	125.3(5)
C40–N17–C48	105.3(6)
C41–N17–V2	100.2(4)
C41–N17–C40	111.5(6)
C41–N17–C48	106.8(5)
C48–N17–V2	106.5(4)
C46–N18–V2	105.7(4)
C46–N18–C47	110.7(6)
C47–N18–V2	105.9(4)
C49–N18–V2	109.4(5)
C49–N18–C46	113.7(6)
C49–N18–C47	110.9(6)
N11–C26–N10	125.6(8)
N11–C26–N13	118.7(9)
N13–C26–N10	115.7(8)
N13–C27–H27A	109.3
N13–C27–H27B	109.3
N13–C27–C28	111.8(8)
H27A–C27–H27B	107.9
C28–C27–H27A	109.3
C28–C27–H27B	109.3
O10–C28–C27	111.6(8)
O10–C28–H28A	109.3
O10–C28–H28B	109.3
C27–C28–H28A	109.3
C27–C28–H28B	109.3
H28A–C28–H28B	108.0
O10–C29–H29A	109.3
O10–C29–H29B	109.3
O10–C29–C30	111.7(9)
H29A–C29–H29B	108.0
C30–C29–H29A	109.3
C30–C29–H29B	109.3
N13–C30–C29	110.3(8)
N13–C30–H30A	109.6
N13–C30–H30B	109.6
C29–C30–H30A	109.6
C29–C30–H30B	109.6
H30A–C30–H30B	108.1
N10–C31–N12	126.3(8)
N10–C31–N14	117.5(8)
N12–C31–N14	116.2(7)
N14–C32–H32A	107.3
N14–C32–H32B	107.3
H32A–C32–H32B	106.9
C33–C32–N14	120.3(10)
C33–C32–H32A	107.3
C33–C32–H32B	107.3
O11–C33–H33A	106.8
O11–C33–H33B	106.8
C32–C33–O11	122.1(10)
C32–C33–H33A	106.8
C32–C33–H33B	106.8
H33A–C33–H33B	106.6
O11–C34–H34A	109.7
O11–C34–H34B	109.7
H34A–C34–H34B	108.2
C35–C34–O11	109.7(7)
C35–C34–H34A	109.7
C35–C34–H34B	109.7
N14–C35–H35A	108.9
N14–C35–H35B	108.9
C34–C35–N14	113.2(8)
C34–C35–H35A	108.9
C34–C35–H35B	108.9
H35A–C35–H35B	107.7
N11–C36–N15	114.2(8)
N12–C36–N11	128.1(8)
N12–C36–N15	117.6(8)
N15–C37–H37A	108.3
N15–C37–H37B	108.3
N15–C37–C38	115.9(6)
H37A–C37–H37B	107.4
C38–C37–H37A	108.3
C38–C37–H37B	108.3
N16–C38–C37	119.1(6)
N16–C38–H38A	107.5
N16–C38–H38B	107.5
C37–C38–H38A	107.5
C37–C38–H38B	107.5
H38A–C38–H38B	107.0
N15–C39–H39A	108.2
N15–C39–H39B	108.2
N15–C39–C40	116.4(6)
H39A–C39–H39B	107.3
C40–C39–H39A	108.2
C40–C39–H39B	108.2
N17–C40–H40A	108.2
N17–C40–H40B	108.2
C39–C40–N17	116.2(7)
C39–C40–H40A	108.2
C39–C40–H40B	108.2
H40A–C40–H40B	107.4
N17–C41–H41A	109.8
N17–C41–H41B	109.8
N17–C41–C42	109.3(6)
H41A–C41–H41B	108.3
C42–C41–H41A	109.8
C42–C41–H41B	109.8
O12–C42–O13	124.9(7)

O12–C42–C41	119.8(7)
O13–C42–C41	115.3(7)
O14–C43–C44	115.6(7)
O15–C43–O14	123.8(8)
O15–C43–C44	120.3(7)
N16–C44–C43	114.2(6)
N16–C44–H44A	108.7
N16–C44–H44B	108.7
C43–C44–H44A	108.7
C43–C44–H44B	108.7
H44A–C44–H44B	107.6
N16–C45–H45A	108.9
N16–C45–H45B	108.9
N16–C45–C46	113.3(7)
H45A–C45–H45B	107.7
C46–C45–H45A	108.9
C46–C45–H45B	108.9
N18–C46–C45	110.3(6)
N18–C46–H46A	109.6
N18–C46–H46B	109.6
C45–C46–H46A	109.6
C45–C46–H46B	109.6
H46A–C46–H46B	108.1
N18–C47–H47A	109.6
N18–C47–H47B	109.6
N18–C47–C48	110.3(6)
H47A–C47–H47B	108.1
C48–C47–H47A	109.6
C48–C47–H47B	109.6
N17–C48–H48A	109.6
N17–C48–H48B	109.6
C47–C48–N17	110.5(6)
C47–C48–H48A	109.6
C47–C48–H48B	109.6
H48A–C48–H48B	108.1
N18–C49–H49A	108.0
N18–C49–H49B	108.0
N18–C49–C50	117.3(7)
H49A–C49–H49B	107.2
C50–C49–H49A	108.0
C50–C49–H49B	108.0
O17–C50–O18	122.0(8)
O17–C50–C49	124.8(8)
O18–C50–C49	113.1(7)
O22–V3–N25	166.5(2)
O22–V3–N26	76.3(2)
O22–V3–N27	90.3(2)
O23–V3–O22	89.9(2)
O23–V3–N25	81.5(2)
O23–V3–N26	153.4(2)
O23–V3–N27	79.8(2)
O25–V3–O22	99.3(2)
O25–V3–O23	102.1(2)
O25–V3–N25	92.8(3)
O25–V3–N26	102.5(3)
O25–V3–N27	170.2(2)
N25–V3–N26	107.1(2)
N25–V3–N27	77.9(2)
N26–V3–N27	77.7(2)
C53–O19–C54	110.2(7)
C59–O20–C58	111.8(7)
C67–O22–V3	115.1(5)
C73–O23–V3	119.6(5)
C75–O27–H27	109.5
C51–N19–C56	111.4(7)
C61–N20–C51	112.4(8)
C56–N21–H21	121.7
C56–N21–C61	116.6(8)
C61–N21–H21	121.7
C51–N22–C52	123.6(8)
C51–N22–C55	121.9(8)
C52–N22–C55	112.4(7)
C56–N23–C57	119.8(7)
C56–N23–C60	119.8(7)
C57–N23–C60	114.0(6)
C61–N24–C62	119.7(7)
C61–N24–C64	120.7(7)
C62–N24–C64	117.1(7)
C63–N25–V3	120.2(5)
C71–N25–V3	111.0(5)
C71–N25–C63	108.7(6)
C71–N25–C72	109.2(6)
C72–N25–V3	102.9(4)
C72–N25–C63	104.1(6)
C65–N26–V3	123.9(5)
C65–N26–C66	110.4(6)
C65–N26–C68	105.9(6)
C66–N26–V3	100.4(4)
C66–N26–C68	108.7(6)
C68–N26–V3	106.9(4)
C69–N27–V3	106.8(4)
C69–N27–C70	112.0(6)
C69–N27–C74	111.3(6)
C70–N27–V3	105.6(4)
C70–N27–C74	112.0(6)
C74–N27–V3	108.7(4)
N19–C51–N20	129.5(8)
N19–C51–N22	117.8(8)
N20–C51–N22	112.6(8)
N22–C52–H52A	109.5
N22–C52–H52B	109.5
N22–C52–C53	110.6(8)
H52A–C52–H52B	108.1
C53–C52–H52A	109.5
C53–C52–H52B	109.5
O19–C53–C52	113.9(10)
O19–C53–H53A	108.8
O19–C53–H53B	108.8
C52–C53–H53A	108.8
C52–C53–H53B	108.8
H53A–C53–H53B	107.7
O19–C54–H54A	109.6
O19–C54–H54B	109.6
O19–C54–C55	110.5(8)
H54A–C54–H54B	108.1
C55–C54–H54A	109.6
C55–C54–H54B	109.6
N22–C55–C54	111.0(8)
N22–C55–H55A	109.4
N22–C55–H55B	109.4

C54–C55–H55A	109.4	C64–C65–H65A	107.5
C54–C55–H55B	109.4	C64–C65–H65B	107.5
H55A–C55–H55B	108.0	H65A–C65–H65B	107.0
N21–C56–N19	124.6(8)	N26–C66–H66A	110.1
N21–C56–N23	116.8(8)	N26–C66–H66B	110.1
N23–C56–N19	118.6(8)	N26–C66–C67	108.1(6)
N23–C57–H57A	110.5	H66A–C66–H66B	108.4
N23–C57–H57B	110.5	C67–C66–H66A	110.1
N23–C57–C58	106.3(6)	C67–C66–H66B	110.1
H57A–C57–H57B	108.7	O21–C67–O22	123.7(8)
C58–C57–H57A	110.5	O21–C67–C66	120.9(7)
C58–C57–H57B	110.5	O22–C67–C66	115.3(7)
O20–C58–C57	106.3(6)	N26–C68–H68A	109.1
O20–C58–H58A	110.5	N26–C68–H68B	109.1
O20–C58–H58B	110.5	H68A–C68–H68B	107.8
C57–C58–H58A	110.5	C69–C68–N26	112.7(6)
C57–C58–H58B	110.5	C69–C68–H68A	109.1
H58A–C58–H58B	108.7	C69–C68–H68B	109.1
O20–C59–H59A	105.0	N27–C69–H69A	109.8
O20–C59–H59B	105.0	N27–C69–H69B	109.8
H59A–C59–H59B	105.9	C68–C69–N27	109.2(6)
C60–C59–O20	129.1(9)	C68–C69–H69A	109.8
C60–C59–H59A	105.0	C68–C69–H69B	109.8
C60–C59–H59B	105.0	H69A–C69–H69B	108.3
N23–C60–H60A	108.1	N27–C70–H70A	109.9
N23–C60–H60B	108.1	N27–C70–H70B	109.9
C59–C60–N23	116.8(8)	N27–C70–C71	109.0(6)
C59–C60–H60A	108.1	H70A–C70–H70B	108.3
C59–C60–H60B	108.1	C71–C70–H70A	109.9
H60A–C60–H60B	107.3	C71–C70–H70B	109.9
N20–C61–N21	124.7(8)	N25–C71–C70	113.1(7)
N20–C61–N24	117.2(8)	N25–C71–H71A	109.0
N21–C61–N24	118.1(8)	N25–C71–H71B	109.0
N24–C62–H62A	108.5	C70–C71–H71A	109.0
N24–C62–H62B	108.5	C70–C71–H71B	109.0
N24–C62–C63	115.1(7)	H71A–C71–H71B	107.8
H62A–C62–H62B	107.5	N25–C72–H72A	109.0
C63–C62–H62A	108.5	N25–C72–H72B	109.0
C63–C62–H62B	108.5	N25–C72–C73	112.9(6)
N25–C63–C62	118.9(7)	H72A–C72–H72B	107.8
N25–C63–H63A	107.6	C73–C72–H72A	109.0
N25–C63–H63B	107.6	C73–C72–H72B	109.0
C62–C63–H63A	107.6	O23–C73–C72	114.5(7)
C62–C63–H63B	107.6	O24–C73–O23	127.1(8)
H63A–C63–H63B	107.0	O24–C73–C72	118.0(7)
N24–C64–H64A	108.6	N27–C74–H74A	108.5
N24–C64–H64B	108.6	N27–C74–H74B	108.5
N24–C64–C65	114.5(6)	N27–C74–C75	114.9(6)
H64A–C64–H64B	107.6	H74A–C74–H74B	107.5
C65–C64–H64A	108.6	C75–C74–H74A	108.5
C65–C64–H64B	108.6	C75–C74–H74B	108.5
N26–C65–C64	119.4(7)	O26–C75–O27	123.7(8)
N26–C65–H65A	107.5	O26–C75–C74	127.2(8)
N26–C65–H65B	107.5	O27–C75–C74	109.1(7)

Table 3. Anisotropic displacement parameters [$Å^2 \times 10^3$].

The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}]$.

Atom	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
V1	28(1)	20(1)	42(1)	0(1)	−1(1)	−3(1)
O1	49(4)	46(4)	156(6)	−46(4)	47(4)	−20(3)
O2	84(5)	49(4)	60(4)	−15(3)	14(3)	−22(4)
O3	33(3)	33(3)	43(3)	4(2)	1(2)	−3(3)
O4	27(3)	25(3)	46(3)	2(2)	1(2)	−2(2)
O5	32(3)	24(3)	44(3)	7(2)	4(2)	2(2)
O6	33(3)	20(3)	43(3)	2(2)	3(2)	−2(3)
O7	29(3)	34(3)	51(3)	10(2)	3(2)	−3(3)
O8	40(3)	26(3)	49(3)	6(2)	−6(3)	3(3)
O9	27(3)	32(3)	48(3)	0(3)	−6(2)	−2(3)
N1	35(3)	18(3)	55(4)	−7(3)	−7(3)	−7(3)
N2	26(3)	21(3)	56(4)	−2(3)	−8(3)	−5(3)
N3	35(4)	21(3)	50(3)	−2(3)	0(3)	−5(3)
N4	33(4)	30(4)	94(5)	−17(3)	7(3)	−14(3)
N5	44(4)	33(4)	49(3)	−5(3)	4(3)	−11(3)
N6	26(3)	15(3)	53(3)	−1(2)	−4(2)	−2(3)
N7	24(3)	24(3)	44(3)	−1(2)	−2(2)	1(2)
N8	27(3)	15(2)	38(3)	−1(2)	−5(2)	−4(2)
N9	31(3)	27(3)	40(3)	7(2)	−3(2)	3(2)
C1	30(4)	20(4)	67(4)	−10(3)	−6(3)	−6(3)
C2	43(5)	31(4)	79(5)	−18(4)	1(4)	−16(4)
C3	47(6)	29(4)	101(6)	−24(4)	18(5)	−16(4)
C4	51(6)	50(5)	152(7)	−47(5)	45(6)	−18(5)
C5	41(5)	34(4)	140(7)	−35(4)	18(5)	−13(4)
C6	33(4)	27(4)	47(4)	2(3)	−4(3)	−6(3)
C7	131(8)	106(7)	99(7)	−62(6)	77(7)	−74(6)
C8	145(8)	118(7)	132(8)	−91(7)	101(7)	−107(7)
C9	68(4)	51(4)	64(3)	−13(3)	−3(3)	−19(3)
C10	68(4)	51(4)	64(3)	−13(3)	−3(3)	−19(3)
C11	30(4)	20(3)	48(4)	4(3)	−5(3)	−4(3)
C12	22(4)	17(4)	56(4)	3(3)	−5(3)	1(3)
C13	27(4)	17(4)	50(3)	1(3)	−5(3)	−3(3)
C14	28(3)	21(4)	43(4)	0(3)	−9(3)	−4(3)
C15	24(3)	18(3)	37(3)	−3(3)	−5(3)	−5(3)
C16	38(5)	15(3)	35(3)	−2(2)	−5(3)	−6(3)
C17	27(4)	13(3)	45(3)	0(3)	2(3)	−9(3)
C18	30(4)	23(4)	37(3)	−2(3)	−3(3)	−4(3)
C19	27(4)	31(4)	52(4)	4(3)	−4(3)	−1(3)
C20	33(4)	21(3)	50(4)	−9(3)	−1(3)	2(3)
C21	26(3)	29(4)	44(4)	0(3)	−4(3)	−3(3)
C22	37(4)	20(3)	36(3)	0(2)	−6(3)	−1(3)
C23	20(3)	24(4)	38(3)	−5(3)	−7(2)	−6(3)
C24	27(3)	33(4)	43(4)	8(3)	1(3)	−4(3)
C25	30(4)	29(4)	45(4)	10(3)	1(3)	−4(3)
V2	21(1)	21(1)	40(1)	4(1)	−9(1)	−2(1)
O10	37(4)	53(4)	152(6)	−38(4)	35(4)	−13(3)
O11	100(5)	44(4)	48(3)	−19(3)	15(3)	−18(4)
O12	36(3)	22(3)	47(3)	1(2)	2(2)	−6(3)
O13	23(2)	18(2)	37(2)	4(2)	−4(2)	−4(2)
O14	30(3)	24(3)	45(3)	4(2)	5(2)	0(2)
O15	35(3)	28(3)	50(3)	5(2)	6(3)	6(3)
O16	23(3)	33(3)	55(3)	8(2)	−7(2)	3(2)
O17	29(3)	30(3)	45(3)	−6(2)	−15(2)	1(3)
O18	28(3)	39(3)	39(3)	−6(2)	−8(2)	2(3)
N10	25(3)	36(4)	68(4)	−9(3)	−12(3)	−9(3)
N11	28(3)	32(4)	69(4)	−4(3)	−8(3)	−7(3)
N12	33(4)	27(3)	47(3)	−5(3)	−8(3)	−3(3)

N13	29(3)	31(4)	82(4)	−17(3)	−1(3)	−8(3)
N14	40(4)	34(4)	59(4)	−10(3)	−2(3)	−6(3)
N15	32(3)	28(3)	48(3)	−6(3)	−4(3)	−7(3)
N16	27(3)	21(3)	45(3)	3(2)	1(2)	0(2)
N17	27(3)	24(3)	35(3)	6(2)	−4(2)	−3(2)
N18	26(3)	26(3)	34(2)	1(2)	−1(2)	0(2)
C26	28(4)	31(4)	72(5)	−3(3)	−7(3)	−3(3)
C27	39(5)	37(4)	91(6)	−22(4)	0(4)	−15(4)
C28	35(5)	48(5)	111(7)	−23(4)	22(5)	−12(4)
C29	52(6)	65(6)	151(8)	−46(5)	44(6)	−19(5)
C30	31(5)	44(5)	130(7)	−37(5)	7(5)	−10(4)
C31	27(4)	33(4)	50(4)	−6(3)	−9(3)	−3(3)
C32	130(8)	96(7)	107(7)	−73(6)	70(7)	−75(6)
C33	157(9)	109(7)	124(8)	−95(6)	93(7)	−95(7)
C34	74(5)	24(3)	59(4)	14(3)	−22(3)	14(3)
C35	55(5)	57(5)	91(6)	−31(4)	2(4)	−25(4)
C36	32(4)	26(4)	53(4)	−3(3)	−5(3)	−2(3)
C37	35(5)	16(4)	50(3)	4(3)	−2(3)	−2(4)
C38	35(4)	24(4)	50(3)	1(3)	7(3)	5(3)
C39	37(4)	24(4)	44(4)	11(3)	−6(3)	−3(3)
C40	32(4)	23(4)	46(4)	5(3)	−6(3)	−5(3)
C41	24(4)	27(3)	35(3)	3(2)	−3(3)	−5(3)
C42	24(4)	25(3)	35(3)	5(3)	−8(3)	−8(3)
C43	28(4)	22(4)	42(3)	−2(3)	0(3)	−3(3)
C44	32(4)	17(3)	53(4)	0(3)	9(3)	2(3)
C45	39(4)	21(3)	51(4)	2(3)	6(3)	1(3)
C46	31(3)	26(4)	45(4)	−5(3)	6(3)	−3(3)
C47	28(4)	36(4)	38(3)	5(3)	−6(3)	−5(3)
C48	30(4)	14(3)	35(3)	10(3)	−4(2)	−10(3)
C49	28(3)	29(4)	35(3)	0(3)	2(2)	−4(3)
C50	34(4)	25(3)	41(4)	4(3)	−4(3)	−1(3)
V3	38(1)	11(1)	39(1)	−4(1)	16(1)	−2(1)
O19	47(4)	51(4)	164(7)	−39(4)	47(5)	−12(4)
O20	48(4)	92(5)	84(4)	−25(4)	23(3)	−23(4)
O21	29(3)	23(3)	48(3)	−5(2)	6(2)	0(3)
O22	40(3)	22(3)	42(3)	−6(2)	9(2)	−1(2)
O23	36(3)	18(3)	46(3)	−2(2)	8(2)	0(2)
O24	25(3)	49(4)	47(3)	4(3)	3(2)	−6(3)
O25	44(3)	18(3)	50(3)	−19(2)	9(2)	−6(2)
O26	64(4)	29(3)	38(3)	−17(2)	0(3)	3(3)
O27	41(3)	33(3)	41(3)	−6(3)	0(2)	2(3)
N19	39(4)	27(4)	59(4)	−7(3)	−9(3)	−6(3)
N20	27(3)	28(4)	69(4)	−13(3)	−5(3)	−4(3)
N21	32(4)	31(3)	55(4)	−3(3)	−5(3)	0(3)
N22	38(4)	36(4)	88(5)	−14(4)	5(3)	−4(3)
N23	57(4)	23(3)	42(3)	−6(2)	−14(3)	−6(3)
N24	30(3)	33(4)	48(3)	0(3)	−7(2)	−3(3)
N25	34(3)	24(3)	42(3)	−7(2)	6(2)	4(2)
N26	30(3)	23(3)	41(3)	3(2)	3(2)	−1(2)
N27	33(3)	10(3)	44(3)	−1(2)	2(2)	−1(2)
C51	32(4)	26(4)	55(4)	1(3)	−15(3)	−2(3)
C52	32(5)	37(4)	129(7)	−27(4)	10(5)	−6(4)
C53	51(6)	56(5)	148(7)	−34(5)	43(6)	−12(5)
C54	38(5)	46(5)	108(6)	−21(4)	18(5)	−4(4)
C55	37(5)	38(4)	86(5)	−12(4)	−2(4)	−4(4)
C56	40(4)	33(4)	42(4)	−1(3)	−18(3)	−3(3)
C57	50(3)	33(2)	66(3)	26(2)	−19(2)	9(2)
C58	50(3)	33(2)	66(3)	26(2)	−19(2)	9(2)
C59	199(9)	166(10)	172(8)	−151(8)	151(8)	−158(8)
C60	96(7)	66(5)	58(4)	−32(4)	25(4)	−47(5)

C61	32(4)	27(4)	54(4)	−2(3)	−5(3)	0(3)
C62	21(4)	29(4)	60(4)	−3(3)	−8(3)	−4(4)
C63	45(4)	34(5)	52(4)	−2(3)	−3(3)	3(4)
C64	36(4)	20(4)	46(4)	5(3)	−3(3)	0(3)
C65	28(4)	20(3)	44(4)	3(3)	3(3)	−5(3)
C66	28(4)	28(3)	43(3)	−1(3)	12(3)	1(3)
C67	31(4)	29(4)	42(4)	−10(3)	3(3)	1(3)
C68	32(4)	21(4)	47(3)	2(3)	−3(3)	0(3)
C69	42(5)	11(3)	45(3)	3(3)	−2(3)	−4(3)
C70	38(4)	29(4)	41(4)	−8(3)	4(3)	7(3)
C71	38(4)	27(4)	44(4)	−5(3)	4(3)	6(3)
C72	31(4)	35(4)	54(4)	0(3)	12(3)	0(3)
C73	39(4)	25(4)	39(3)	−11(3)	8(3)	−8(3)
C74	44(4)	17(3)	39(3)	−3(3)	4(3)	2(3)
C75	33(4)	29(3)	28(3)	−8(3)	12(2)	−6(3)

Table 4. Hydrogen coordinates [$\times 10^4$] and isotropic displacement parameters [$Å^2 \times 10^3$].

Atom	x	y	z	U_{eq}	S.o.f.
H9	8485	4443	7510	55	1
H1	4946	2626	5485	45	1
H2A	4374	2245	5660	63	1
H2B	3845	2672	5563	63	1
H3A	3655	1855	5882	70	1
H3B	4157	2066	6107	70	1
H4A	4135	3163	6354	97	1
H4B	3614	3615	6288	97	1
H5A	3789	3857	5836	85	1
H5B	4297	4048	6062	85	1
H7A	6057	3636	5044	127	1
H7B	6479	3337	5293	127	1
H8A	6748	2678	5049	148	1
H8B	6496	3179	4805	148	1
H9A	5653	1525	4889	75	1
H9B	6110	1698	5148	75	1
H10A	5217	2501	4965	75	1
H10B	5314	2061	5251	75	1
H12A	5244	5287	6052	39	1
H12B	5816	5486	6212	39	1
H13A	5247	4980	6517	39	1
H13B	5269	4288	6348	39	1
H14A	6185	5438	5727	39	1
H14B	6403	4695	5681	39	1
H15A	7055	5361	5965	32	1
H15B	6669	5478	6193	32	1
H16A	7517	4236	5944	37	1
H16B	6947	3883	5888	37	1
H19A	5424	3734	6726	45	1
H19B	5583	4255	6982	45	1
H20A	5906	5266	6912	42	1
H20B	6256	5437	6670	42	1
H21A	6831	5325	7111	41	1
H21B	6599	4588	7160	41	1
H22A	7658	5329	6822	39	1
H22B	7094	5518	6642	39	1
H23A	7772	4461	6485	34	1
H23B	7690	5178	6327	34	1
H24A	7728	3974	6926	42	1
H24B	7322	3850	7142	42	1
H18	8467	1115	7515	55	1
H10	4945	−710	5492	54	1
H27A	4371	−1093	5662	68	1
H27B	3841	−670	5568	68	1
H28A	3657	−1496	5889	76	1
H28B	4158	−1277	6115	76	1
H29A	4147	−203	6354	104	1
H29B	3629	260	6299	104	1
H30A	3788	513	5836	83	1
H30B	4299	709	6058	83	1
H32A	6049	288	5036	127	1
H32B	6471	18	5291	127	1
H33A	6745	−670	5064	147	1
H33B	6525	−157	4817	147	1
H34A	5643	−1811	4880	66	1
H34B	6104	−1660	5141	66	1
H35A	5313	−1264	5250	82	1
H35B	5219	−823	4964	82	1

H37A	5246	1939	6050	42	1
H37B	5817	2145	6210	42	1
H38A	5247	1625	6518	43	1
H38B	5283	933	6351	43	1
H39A	6191	2099	5729	44	1
H39B	6418	1360	5688	44	1
H40A	6670	2159	6190	41	1
H40B	7054	2040	5960	41	1
H41A	7523	893	5946	36	1
H41B	6955	536	5889	36	1
H44A	5422	397	6724	41	1
H44B	5570	921	6979	41	1
H45A	5902	1924	6912	45	1
H45B	6253	2096	6670	45	1
H46A	6818	2001	7110	41	1
H46B	6592	1262	7162	41	1
H47A	7656	1997	6818	42	1
H47B	7091	2176	6640	42	1
H48A	7775	1134	6485	33	1
H48B	7687	1851	6327	33	1
H49A	7717	629	6922	37	1
H49B	7316	521	7143	37	1
H27	8471	−2241	7517	59	1
H21	6079	−2692	5534	49	1
H52A	3796	−2813	5839	80	1
H52B	4302	−2627	6068	80	1
H53A	4140	−3524	6352	98	1
H53B	3621	−3067	6290	98	1
H54A	3664	−4837	5886	76	1
H54B	4164	−4619	6112	76	1
H55A	4371	−4410	5652	65	1
H55B	3840	−3980	5567	65	1
H57A	6333	−3021	5210	63	1
H57B	5899	−3146	4933	63	1
H58A	6744	−3675	4884	63	1
H58B	6676	−4159	5148	63	1
H59A	5398	−4643	4765	199	1
H59B	5799	−5093	4970	199	1
H60A	5534	−4756	5275	86	1
H60B	5137	−4290	5068	86	1
H62A	5235	−1401	6044	46	1
H62B	5804	−1184	6204	46	1
H63A	5243	−1702	6518	54	1
H63B	5271	−2397	6351	54	1
H64A	6182	−1223	5735	42	1
H64B	6403	−1959	5680	42	1
H65A	7050	−1312	5965	37	1
H65B	6666	−1202	6194	37	1
H66A	7514	−2446	5939	39	1
H66B	6942	−2794	5885	39	1
H68A	7765	−2199	6483	41	1
H68B	7675	−1485	6324	41	1
H69A	7102	−1142	6644	40	1
H69B	7667	−1344	6818	40	1
H70A	6811	−1334	7115	43	1
H70B	6592	−2079	7164	43	1
H71A	5890	−1420	6909	44	1
H71B	6244	−1242	6670	44	1
H72A	5426	−2961	6719	47	1
H72B	5555	−2435	6976	47	1

H74A	7721	–2717	6930	40	1
H74B	7312	–2819	7148	40	1

Table 5. Torsion angles [°].

V1–O4–C18–O3	–179.3(5)	C11–N2–C1–N4	174.5(7)
V1–O4–C18–C19	3.0(9)	C11–N3–C6–N1	–2.7(12)
V1–O5–C17–O6	–161.0(6)	C11–N3–C6–N5	–179.7(7)
V1–O5–C17–C16	18.7(8)	C11–N6–C12–C13	63.3(9)
V1–N7–C13–C12	–54.0(8)	C11–N6–C14–C15	–134.7(7)
V1–N7–C19–C18	27.7(7)	C12–N6–C11–N2	–1.0(11)
V1–N7–C20–C21	–41.6(7)	C12–N6–C11–N3	178.5(6)
V1–N8–C15–C14	–58.2(8)	C12–N6–C14–C15	59.7(9)
V1–N8–C16–C17	–43.5(6)	C13–N7–C19–C18	155.1(7)
V1–N8–C23–C22	–52.8(6)	C13–N7–C20–C21	–175.3(5)
V1–N9–C21–C20	–39.5(7)	C14–N6–C11–N2	–166.3(7)
V1–N9–C22–C23	–35.0(6)	C14–N6–C11–N3	13.3(11)
V1–N9–C24–C25	177.2(5)	C14–N6–C12–C13	–131.3(7)
O1–C4–C5–N4	–55.2(12)	C15–N8–C16–C17	–176.7(6)
O2–C9–C10–N5	–57.5(11)	C15–N8–C23–C22	82.8(7)
O3–C18–C19–N7	159.2(7)	C16–N8–C15–C14	60.8(8)
O4–C18–C19–N7	–23.0(10)	C16–N8–C23–C22	–159.4(6)
N4–C2–C3–O1	52.1(11)	C19–N7–C13–C12	–169.9(6)
N5–C7–C8–O2	21(2)	C19–N7–C20–C21	70.6(7)
N6–C12–C13–N7	69.4(9)	C20–N7–C13–C12	74.1(8)
N6–C14–C15–N8	60.5(9)	C20–N7–C19–C18	–89.3(8)
N7–C20–C21–N9	56.1(8)	C21–N9–C22–C23	–150.8(6)
N8–C16–C17–O5	20.9(9)	C21–N9–C24–C25	–63.9(9)
N8–C16–C17–O6	–159.4(7)	C22–N9–C21–C20	76.9(7)
N9–C22–C23–N8	60.2(8)	C22–N9–C24–C25	58.7(9)
N9–C24–C25–O8	15.9(11)	C23–N8–C15–C14	176.5(5)
N9–C24–C25–O9	–163.2(6)	C23–N8–C16–C17	68.0(7)
C1–N1–C6–N3	5.3(12)	C24–N9–C21–C20	–160.6(6)
C1–N1–C6–N5	–177.6(7)	C24–N9–C22–C23	85.3(7)
C1–N2–C11–N3	11.8(12)	V2–O13–C42–O12	–161.1(6)
C1–N2–C11–N6	–168.8(7)	V2–O13–C42–C41	17.1(8)
C1–N4–C2–C3	143.9(9)	V2–O14–C43–O15	179.7(6)
C1–N4–C5–C4	–142.1(9)	V2–O14–C43–C44	5.0(9)
C2–N4–C1–N1	0.8(14)	V2–N16–C38–C37	–54.8(8)
C2–N4–C1–N2	177.9(8)	V2–N16–C44–C43	28.4(7)
C2–N4–C5–C4	52.5(12)	V2–N16–C45–C46	–41.4(7)
C3–O1–C4–C5	56.9(12)	V2–N17–C40–C39	–59.0(8)
C4–O1–C3–C2	–55.5(12)	V2–N17–C41–C42	–43.0(6)
C5–N4–C1–N1	–163.5(9)	V2–N17–C48–C47	–51.8(6)
C5–N4–C1–N2	13.6(14)	V2–N18–C46–C45	–38.5(7)
C5–N4–C2–C3	–50.6(11)	V2–N18–C47–C48	–35.3(6)
C6–N1–C1–N2	0.8(12)	V2–N18–C49–C50	175.7(5)
C6–N1–C1–N4	177.6(8)	O10–C29–C30–N13	–57.1(12)
C6–N3–C11–N2	–6.5(11)	O11–C34–C35–N14	–56.5(11)
C6–N3–C11–N6	174.0(7)	O14–C43–C44–N16	–24.7(10)
C6–N5–C7–C8	178.4(13)	O15–C43–C44–N16	160.3(7)
C6–N5–C10–C9	–160.5(8)	N13–C27–C28–O10	52.7(11)
C7–N5–C6–N1	169.4(9)	N14–C32–C33–O11	26.0(2)
C7–N5–C6–N3	–13.3(13)	N15–C37–C38–N16	70.3(9)
C7–N5–C10–C9	37.7(13)	N15–C39–C40–N17	60.3(9)
C8–O2–C9–C10	55.9(11)	N16–C45–C46–N18	56.1(8)
C9–O2–C8–C7	–38.7(19)	N17–C41–C42–O12	–160.2(6)
C10–N5–C6–N1	8.3(12)	N17–C41–C42–O13	21.5(9)
C10–N5–C6–N3	–174.3(8)	N18–C47–C48–N17	60.5(7)
C10–N5–C7–C8	–19.2(18)	N18–C49–C50–O17	18.8(12)
C11–N2–C1–N1	–8.5(13)	N18–C49–C50–O18	–165.0(6)

C26–N10–C31–N12	5.6(13)
C26–N10–C31–N14	–175.5(7)
C26–N11–C36–N12	9.9(14)
C26–N11–C36–N15	–169.1(8)
C26–N13–C27–C28	142.5(9)
C26–N13–C30–C29	–140.1(9)
C27–N13–C26–N10	0.9(14)
C27–N13–C26–N11	–179.6(8)
C27–N13–C30–C29	51.3(11)
C28–O10–C29–C30	60.6(12)
C29–O10–C28–C27	–58.0(12)
C30–N13–C26–N10	–166.1(8)
C30–N13–C26–N11	13.5(14)
C30–N13–C27–C28	–49.7(11)
C31–N10–C26–N11	–1.4(13)
C31–N10–C26–N13	178.1(8)
C31–N12–C36–N11	–6.4(13)
C31–N12–C36–N15	172.5(8)
C31–N14–C32–C33	175.6(13)
C31–N14–C35–C34	–159.3(8)
C32–N14–C31–N10	168.7(9)
C32–N14–C31–N12	–12.2(13)
C32–N14–C35–C34	38.1(14)
C33–O11–C34–C35	55.6(11)
C34–O11–C33–C32	–42.1(18)
C35–N14–C31–N10	7.2(13)
C35–N14–C31–N12	–173.7(9)
C35–N14–C32–C33	–21.7(19)
C36–N11–C26–N10	–5.4(14)
C36–N11–C26–N13	175.1(8)
C36–N12–C31–N10	–2.1(13)
C36–N12–C31–N14	178.9(7)
C36–N15–C37–C38	64.9(10)
C36–N15–C39–C40	–136.4(8)
C37–N15–C36–N11	–2.4(12)
C37–N15–C36–N12	178.5(7)
C37–N15–C39–C40	59.0(9)
C38–N16–C44–C43	154.9(7)
C38–N16–C45–C46	–175.2(6)
C39–N15–C36–N11	–166.6(7)
C39–N15–C36–N12	14.3(12)
C39–N15–C37–C38	–130.6(7)
C40–N17–C41–C42	–177.7(6)
C40–N17–C48–C47	83.1(7)
C41–N17–C40–C39	61.8(8)
C41–N17–C48–C47	–158.2(6)
C44–N16–C38–C37	–170.0(6)
C44–N16–C45–C46	70.4(8)
C45–N16–C38–C37	73.3(8)
C45–N16–C44–C43	–87.7(8)
C46–N18–C47–C48	–149.5(6)
C46–N18–C49–C50	–66.4(8)
C47–N18–C46–C45	75.8(7)
C47–N18–C49–C50	59.2(8)
C48–N17–C40–C39	177.3(6)
C48–N17–C41–C42	67.8(7)
C49–N18–C46–C45	–158.5(6)
C49–N18–C47–C48	83.3(7)
V3–O22–C67–O21	–162.7(6)
V3–O22–C67–C66	19.3(8)
V3–O23–C73–O24	179.1(6)
V3–O23–C73–C72	6.4(9)
V3–N25–C63–C62	–54.4(9)
V3–N25–C71–C70	–41.8(7)
V3–N25–C72–C73	30.7(7)
V3–N26–C65–C64	–58.7(8)
V3–N26–C66–C67	–43.7(6)
V3–N26–C68–C69	–51.4(7)
V3–N27–C69–C68	–33.5(7)
V3–N27–C70–C71	–39.1(7)
V3–N27–C74–C75	176.3(5)
O19–C54–C55–N22	53.9(10)
O20–C59–C60–N23	–1(2)
N22–C52–C53–O19	–55.9(12)
N23–C57–C58–O20	66.2(7)
N24–C62–C63–N25	68.9(9)
N24–C64–C65–N26	59.4(9)
N25–C72–C73–O23	–27.1(10)
N25–C72–C73–O24	159.5(7)
N26–C66–C67–O21	–157.5(7)
N26–C66–C67–O22	20.6(9)
N26–C68–C69–N27	58.9(8)
N27–C70–C71–N25	56.3(8)
N27–C74–C75–O26	18.3(11)
N27–C74–C75–O27	–162.8(6)
C51–N19–C56–N21	5.7(12)
C51–N19–C56–N23	–176.2(7)
C51–N20–C61–N21	9.6(13)
C51–N20–C61–N24	–170.5(7)
C51–N22–C52–C53	–144.7(9)
C51–N22–C55–C54	144.4(8)
C52–N22–C51–N19	–166.1(9)
C52–N22–C51–N20	17.4(13)
C52–N22–C55–C54	–51.8(11)
C53–O19–C54–C55	–56.6(12)
C54–O19–C53–C52	58.9(12)
C55–N22–C51–N19	–4.1(13)
C55–N22–C51–N20	179.5(8)
C55–N22–C52–C53	51.7(12)
C56–N19–C51–N20	–3.7(13)
C56–N19–C51–N22	–179.5(8)
C56–N21–C61–N20	–8.0(13)
C56–N21–C61–N24	172.1(8)
C56–N23–C57–C58	158.9(7)
C56–N23–C60–C59	168.7(12)
C57–N23–C56–N19	153.9(7)
C57–N23–C56–N21	–27.9(10)
C57–N23–C60–C59	17.0(16)
C58–O20–C59–C60	20(2)
C59–O20–C58–C57	–51.9(11)
C60–N23–C56–N19	3.8(11)
C60–N23–C56–N21	–177.9(8)
C60–N23–C57–C58	–49.4(9)
C61–N20–C51–N19	–3.4(13)
C61–N20–C51–N22	172.6(7)
C61–N21–C56–N19	–0.5(13)
C61–N21–C56–N23	–178.6(7)
C61–N24–C62–C63	66.1(10)
C61–N24–C64–C65	–135.0(8)
C62–N24–C61–N20	–3.5(12)

C62–N24–C61–N21	176.4(7)	C68–N26–C66–C67	68.3(8)
C62–N24–C64–C65	62.9(9)	C69–N27–C70–C71	76.8(8)
C63–N25–C71–C70	–176.0(6)	C69–N27–C74–C75	58.9(8)
C63–N25–C72–C73	156.8(7)	C70–N27–C69–C68	–148.7(6)
C64–N24–C61–N20	–165.1(7)	C70–N27–C74–C75	–67.3(8)
C64–N24–C61–N21	14.8(12)	C71–N25–C63–C62	74.9(9)
C64–N24–C62–C63	–131.6(7)	C71–N25–C72–C73	–87.2(8)
C65–N26–C66–C67	–176.0(6)	C72–N25–C63–C62	–168.8(7)
C65–N26–C68–C69	82.4(7)	C72–N25–C71–C70	71.0(8)
C66–N26–C65–C64	60.1(8)	C74–N27–C69–C68	85.0(7)
C66–N26–C68–C69	–158.9(6)	C74–N27–C70–C71	–157.3(6)
C68–N26–C65–C64	177.5(6)		

A.6 – EUROPIUM(III) CRYSTAL STRUCTURE OF TZMORPH2DO3A

Table 1. Crystal data and structure refinement details.

Identification code	**2015ncs0605 (MM207)**	
Empirical formula	$C_{56}H_{114}Eu_2F_{12}N_{18}O_{42}S_2$	
Formula weight	2307.69	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	*P*–1	
Unit cell dimensions	*a* = 11.7661(3) Å	α = 98.7914(19)°
	b = 14.5390(3) Å	β = 112.313(2)°
	c = 14.6642(4) Å	γ= 104.6244(19)°
Volume	2157.14(10) Å^3	
Z	1	
Density (calculated)	1.776 Mg / m^3	
Absorption coefficient	1.619 mm^{-1}	
F(000)	1178	
Crystal	Plate; colourless	
Crystal size	0.15 × 0.11 × 0.07 mm^3	
θ range for data collection	1.563 – 29.706°	
Index ranges	$-16 \le h \le 15$, $-19 \le k \le 20$, $-19 \le l \le 19$	
Reflections collected	29486	
Independent reflections	10937 [R_{int} = 0.0361]	
Completeness to θ = 26.000°	100.0 %	
Absorption correction	Semi–empirical from equivalents	
Max. and min. transmission	1.00000 and 0.88317	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	10937 / 112 / 606	
Goodness-of-fit on F^2	1.100	
Final *R* indices [$F^2 > 2\sigma(F^2)$]	*R1* = 0.0346, *wR2* = 0.1123	
R indices (all data)	*R1* = 0.0424, *wR2* = 0.1617	
Extinction coefficient	n/a	
Largest diff. peak and hole	1.256 and –2.290 e Å^{-3}	

Diffractometer:*Rigaku AFC12* goniometer equipped with an enhanced sensitivity (HG) *Saturn724+* detector mounted at the window of an *FR-E+ SuperBright* molybdenum rotating anode generator with HF *Varimax* optics (100µm focus). **Cell determination anddata collection**:*CrystalClear-SM Expert 3.0 r27* (Rigaku, 2012). **Data reduction, cellrefinement andabsorption correction**: *CrystalClear-SM Expert 2.0 r13* (Rigaku, 2011). **Structure solution**: *ShelXT (Sheldrick, 2015)*. Acta Cryst. A64, 112-122). **Structure refinement**: *SHELXL-2014*(Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122).

Special details:

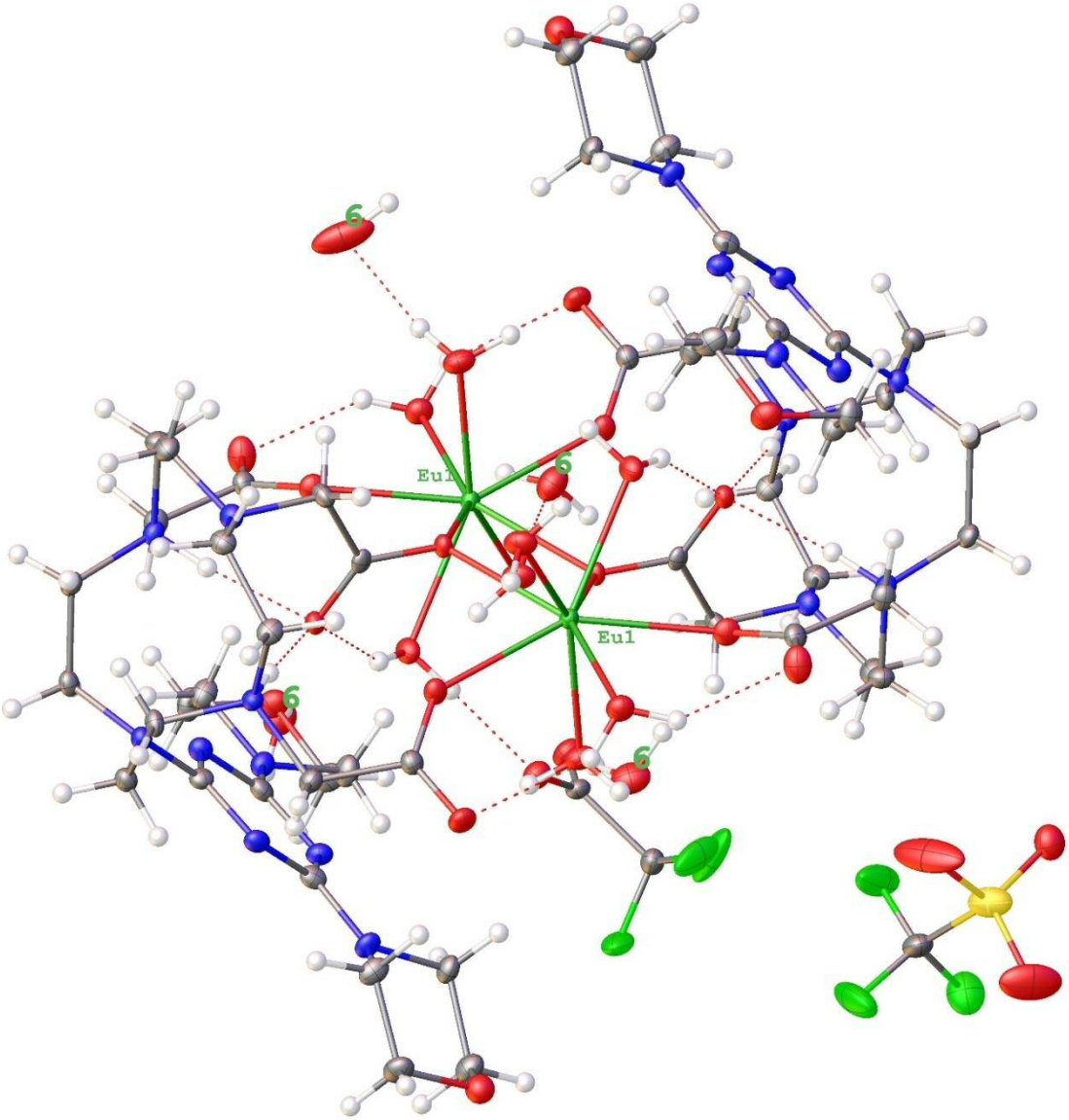

Figure 1. Molecular structure of 2015ncs0605 (MM207) – grown fragment.
Displacement ellipsoids- 50% probability

Table 1. Atomic coordinates [$\times 10^4$], equivalent isotropic displacement parameters [$Å^2 \times 10^3$] and site occupancy factors. U_{eq} is defined as one third of the trace of the orthogonalized U^{ij} tensor.

Atom	x	y	z	U_{eq}	S.o.f.
Eu1	6679(1)	5548(1)	4793(1)	10(1)	1
O1	−561(3)	7218(2)	2607(2)	20(1)	1
O2	7807(3)	8956(2)	2709(2)	20(1)	1
O3	1314(3)	5993(2)	5736(2)	19(1)	1
O4	3321(3)	5966(2)	6086(2)	15(1)	1
O5	9587(3)	7945(2)	5806(2)	22(1)	1
O6	7962(3)	7063(2)	6087(2)	16(1)	1
O7	5740(2)	7230(2)	6321(2)	12(1)	1
O8	5519(2)	5638(2)	5909(2)	11(1)	1
O9	7836(3)	4999(2)	6203(2)	19(1)	1
O10	8728(3)	6013(2)	4736(2)	18(1)	1
O11	5574(3)	6719(2)	4376(2)	15(1)	1
O12	6229(3)	5611(2)	3057(2)	20(1)	1
N1	4007(3)	8659(2)	5312(3)	15(1)	1
N2	2184(3)	7968(2)	3753(2)	15(1)	1
N3	4191(3)	8386(2)	3727(2)	14(1)	1
N4	6237(3)	8889(2)	3776(2)	14(1)	1
N5	6108(3)	9164(2)	5318(2)	14(1)	1
N6	5901(3)	9419(2)	6833(2)	13(1)	1
N7	4481(3)	7782(2)	7544(2)	12(1)	1
N8	6859(3)	7434(2)	8388(2)	14(1)	1
N9	8257(3)	8658(2)	7558(2)	12(1)	1
C1	3507(4)	8343(3)	4288(3)	14(1)	1
C2	1571(4)	7344(3)	2697(3)	16(1)	1
C3	261(4)	7445(3)	2112(3)	19(1)	1
C4	30(4)	7875(3)	3610(3)	20(1)	1
C5	1332(4)	7796(3)	4263(3)	17(1)	1
C6	5484(4)	8799(3)	4290(3)	14(1)	1
C7	5780(4)	8161(3)	2786(3)	19(1)	1
C8	6406(4)	8589(3)	2147(3)	20(1)	1
C9	8204(4)	9711(3)	3623(3)	19(1)	1
C10	7647(4)	9304(3)	4317(3)	18(1)	1
C11	5315(3)	9075(3)	5778(3)	13(1)	1
C12	5086(4)	9532(3)	7363(3)	14(1)	1
C13	5027(4)	8869(3)	8066(3)	13(1)	1
C14	3069(3)	7447(3)	6806(3)	14(1)	1
C15	2516(4)	6369(3)	6157(3)	14(1)	1
C16	4682(4)	7207(3)	8325(3)	15(1)	1
C17	6119(4)	7407(3)	9001(3)	15(1)	1
C18	6500(4)	6482(3)	7680(3)	14(1)	1
C19	5872(3)	6474(3)	6561(3)	11(1)	1
C20	8259(4)	7912(3)	8999(3)	16(1)	1
C21	8954(4)	8193(3)	8343(3)	16(1)	1
C22	8908(4)	8812(3)	6866(3)	15(1)	1
C23	8834(4)	7858(3)	6202(3)	14(1)	1
C24	8182(3)	9606(3)	8077(3)	13(1)	1
C25	7273(3)	10040(2)	7355(3)	13(1)	1
S1	−2786(1)	1282(1)	405(1)	28(1)	1
F4	−927(3)	2638(2)	322(4)	65(1)	1
F5	−2479(4)	3113(3)	390(3)	54(1)	1
F6	−2763(3)	2170(2)	−999(2)	34(1)	1
O15	−2125(6)	1612(5)	1501(3)	71(2)	1
O16	−4166(4)	1080(4)	−44(4)	50(1)	1
O17	−2344(3)	569(2)	−30(3)	25(1)	1
C28	−2200(4)	2362(3)	26(4)	27(1)	1
F1	1758(3)	4662(3)	131(3)	58(1)	1
F2	1204(3)	5799(2)	751(2)	35(1)	1

F3	1085(3)	4517(2)	1277(3)	46(1)	1
O13	3293(3)	5880(2)	2711(2)	19(1)	1
O14	4103(3)	5804(2)	1564(2)	19(1)	1
C26	1800(4)	5149(3)	976(3)	19(1)	1
C27	3210(3)	5663(3)	1835(3)	15(1)	1
O21	4288(3)	5624(3)	–296(3)	27(1)	1
O20	8642(3)	9340(2)	1202(2)	25(1)	1
O18	10454(4)	5870(4)	7222(3)	48(1)	1
O19	7837(3)	5278(2)	2181(2)	24(1)	1

Table 2. Bond lengths [Å] and angles [°].

Eu1–Eu1i	4.1766(3)	N6–C12	1.470(5)
Eu1–O4i	2.374(3)	N6–C25	1.457(5)
Eu1–O6	2.367(3)	N7–H7	0.9800
Eu1–O8i	2.429(2)	N7–C13	1.497(4)
Eu1–O8	2.505(2)	N7–C14	1.494(4)
Eu1–O9	2.383(3)	N7–C16	1.502(4)
Eu1–H9A	2.7887	N8–C17	1.468(5)
Eu1–H9B	2.7906	N8–C18	1.451(4)
Eu1–O10	2.372(3)	N8–C20	1.453(5)
Eu1–H10A	2.7906	N9–H9	0.9800
Eu1–H10B	2.7889	N9–C21	1.502(5)
Eu1–O11	2.408(3)	N9–C22	1.497(5)
Eu1–H11A	2.8172	N9–C24	1.510(5)
Eu1–O12	2.417(3)	C2–H2A	0.9700
Eu1–H12A	2.8166	C2–H2B	0.9700
Eu1–H12B	2.8182	C2–C3	1.511(5)
O1–C3	1.421(5)	C3–H3A	0.9700
O1–C4	1.421(5)	C3–H3B	0.9700
O2–C8	1.439(5)	C4–H4A	0.9700
O2–C9	1.426(5)	C4–H4B	0.9700
O3–C15	1.234(5)	C4–C5	1.511(5)
O4–Eu1i	2.374(3)	C5–H5A	0.9700
O4–C15	1.261(5)	C5–H5B	0.9700
O5–C23	1.223(5)	C7–H7A	0.9700
O6–C23	1.275(4)	C7–H7B	0.9700
O7–C19	1.234(4)	C7–C8	1.510(5)
O8–Eu1i	2.429(2)	C8–H8A	0.9700
O8–C19	1.286(4)	C8–H8B	0.9700
O9–H9A	0.8554	C9–H9C	0.9700
O9–H9B	0.8555	C9–H9D	0.9700
O10–H10A	0.8642	C9–C10	1.521(5)
O10–H10B	0.8635	C10–H10C	0.9700
O11–H11A	0.8598	C10–H10D	0.9700
O11–H11B	0.8602	C12–H12C	0.9700
O12–H12A	0.8508	C12–H12D	0.9700
O12–H12B	0.8508	C12–C13	1.524(5)
N1–C1	1.338(5)	C13–H13A	0.9700
N1–C11	1.344(5)	C13–H13B	0.9700
N2–C1	1.360(5)	C14–H14A	0.9700
N2–C2	1.460(5)	C14–H14B	0.9700
N2–C5	1.458(5)	C14–C15	1.531(5)
N3–C1	1.350(5)	C16–H16A	0.9700
N3–C6	1.338(5)	C16–H16B	0.9700
N4–C6	1.360(5)	C16–C17	1.522(5)
N4–C7	1.472(5)	C17–H17A	0.9700
N4–C10	1.448(5)	C17–H17B	0.9700
N5–C6	1.347(5)	C18–H18A	0.9700
N5–C11	1.336(5)	C18–H18B	0.9700
N6–C11	1.378(5)	C18–C19	1.518(5)

C20–H20A	0.9700
C20–H20B	0.9700
C20–C21	1.520(5)
C21–H21A	0.9700
C21–H21B	0.9700
C22–H22A	0.9700
C22–H22B	0.9700
C22–C23	1.531(5)
C24–H24A	0.9700
C24–H24B	0.9700
C24–C25	1.532(5)
C25–H25A	0.9700
C25–H25B	0.9700
S1–O15	1.430(4)
S1–O16	1.431(4)
S1–O17	1.439(3)
S1–C28	1.806(5)
F4–C28	1.320(5)
F5–C28	1.313(5)
F6–C28	1.341(5)
F1–C26	1.306(5)
F2–C26	1.323(5)
F3–C26	1.327(5)
O13–C27	1.236(5)
O14–C27	1.239(5)
C26–C27	1.548(5)
O21–H21C	0.8501
O21–H21D	0.8498
O20–H20C	0.8499
O20–H20D	0.8502
O18–H18C	0.8501
O18–H18D	0.8503
O19–H19A	0.8500
O19–H19B	0.8502

Eu1i–Eu1–H9A	103.9
Eu1i–Eu1–H9B	86.8
Eu1i–Eu1–H10A	154.6
Eu1i–Eu1–H10B	160.5
Eu1i–Eu1–H11A	67.6
Eu1i–Eu1–H12A	126.3
Eu1i–Eu1–H12B	114.7
O4i–Eu1–Eu1i	98.79(6)
O4i–Eu1–O8	123.09(8)
O4i–Eu1–O8i	72.53(8)
O4i–Eu1–O9	79.30(10)
O4i–Eu1–H9A	83.9
O4i–Eu1–H9B	63.2
O4i–Eu1–H10A	63.7
O4i–Eu1–H10B	92.6
O4i–Eu1–O11	137.11(9)
O4i–Eu1–H11A	149.2
O4i–Eu1–O12	72.90(10)
O4i–Eu1–H12A	62.9
O4i–Eu1–H12B	89.2
O6–Eu1–Eu1i	106.23(6)
O6–Eu1–O4i	145.73(9)
O6–Eu1–O8	75.37(8)
O6–Eu1–O8i	137.83(9)
O6–Eu1–O9	78.35(10)
O6–Eu1–H9A	67.8
O6–Eu1–H9B	94.8
O6–Eu1–O10	74.31(9)
O6–Eu1–H10A	84.5
O6–Eu1–H10B	57.5
O6–Eu1–O11	72.62(9)
O6–Eu1–H11A	64.2
O6–Eu1–O12	115.86(10)
O6–Eu1–H12A	115.8
O6–Eu1–H12B	100.9
O8–Eu1–Eu1i	31.62(6)
O8i–Eu1–Eu1i	32.74(6)
O8i–Eu1–O8	64.36(10)
O8i–Eu1–H9A	118.9
O8–Eu1–H9A	86.0
O8i–Eu1–H9B	92.1
O8–Eu1–H9B	82.6
O8i–Eu1–H10A	136.2
O8–Eu1–H10A	143.7
O8i–Eu1–H10B	164.6
O8–Eu1–H10B	129.6
O8i–Eu1–H11A	83.6
O8–Eu1–H11A	58.3
O8i–Eu1–H12A	96.1
O8–Eu1–H12A	152.1
O8–Eu1–H12B	128.2
O8i–Eu1–H12B	94.6
O9–Eu1–Eu1i	88.50(7)
O9–Eu1–O8	75.61(9)
O9–Eu1–O8i	102.20(10)
O9–Eu1–H9A	16.8
O9–Eu1–H9B	16.8
O9–Eu1–H10A	70.9
O9–Eu1–H10B	78.1
O9–Eu1–O11	141.91(10)
O9–Eu1–H11A	125.6
O9–Eu1–O12	146.50(10)
O9–Eu1–H12A	130.4
O9–Eu1–H12B	155.6
H9A–Eu1–H9B	28.9
H9A–Eu1–H10A	58.2
H9A–Eu1–H10B	61.6
H9A–Eu1–H11A	125.5
H9A–Eu1–H12A	121.7
H9A–Eu1–H12B	141.5
H9B–Eu1–H10A	69.1
H9B–Eu1–H10B	84.3
H9B–Eu1–H11A	138.5
H9B–Eu1–H12A	119.7
H9B–Eu1–H12B	148.1
O10–Eu1–Eu1i	170.57(7)
O10–Eu1–O4i	77.25(9)
O10–Eu1–O8i	147.86(9)
O10–Eu1–O8	145.44(9)
O10–Eu1–O9	82.37(10)
O10–Eu1–H9A	67.4
O10–Eu1–H9B	83.8
O10–Eu1–H10A	16.9
O10–Eu1–H10B	16.9
O10–Eu1–O11	112.00(9)
O10–Eu1–H11A	119.9
O10–Eu1–O12	73.67(10)

O10–Eu1–H12A	59.7
O10–Eu1–H12B	74.1
H10A–Eu1–H10B	29.2
H10A–Eu1–H11A	136.6
H10A–Eu1–H12A	64.2
H10A–Eu1–H12B	84.8
H10B–Eu1–H11A	108.9
H10B–Eu1–H12A	73.0
H10B–Eu1–H12B	81.1
O11–Eu1–Eu1i	76.78(7)
O11–Eu1–O8	73.62(9)
O11–Eu1–O8i	84.12(9)
O11–Eu1–H9A	138.9
O11–Eu1–H9B	155.2
O11–Eu1–H10A	128.6
O11–Eu1–H10B	105.2
O11–Eu1–H11A	16.7
O11–Eu1–O12	70.40(9)
O11–Eu1–H12A	85.2
O11–Eu1–H12B	56.7
H11A–Eu1–H12A	101.8
H11A–Eu1–H12B	73.3
O12–Eu1–Eu1i	113.63(7)
O12–Eu1–O8	135.84(9)
O12–Eu1–O8i	87.09(9)
O12–Eu1–H9A	138.1
O12–Eu1–H9B	134.0
O12–Eu1–H10A	80.1
O12–Eu1–H10B	84.8
O12–Eu1–H11A	87.1
O12–Eu1–H12A	16.6
O12–Eu1–H12B	16.5
H12A–Eu1–H12B	28.5
C4–O1–C3	109.9(3)
C9–O2–C8	110.2(3)
C15–O4–Eu1i	139.2(2)
C23–O6–Eu1	136.1(2)
Eu1i–O8–Eu1	115.64(10)
C19–O8–Eu1i	121.6(2)
C19–O8–Eu1	117.3(2)
Eu1–O9–H9A	109.6
Eu1–O9–H9B	109.8
H9A–O9–H9B	109.1
Eu1–O10–H10A	110.2
Eu1–O10–H10B	110.1
H10A–O10–H10B	108.8
Eu1–O11–H11A	109.7
Eu1–O11–H11B	110.0
H11A–O11–H11B	109.0
Eu1–O12–H12A	109.4
Eu1–O12–H12B	109.6
H12A–O12–H12B	109.4
C1–N1–C11	113.7(3)
C1–N2–C2	120.6(3)
C1–N2–C5	122.0(3)
C5–N2–C2	112.7(3)
C6–N3–C1	113.8(3)
C6–N4–C7	119.2(3)
C6–N4–C10	121.3(3)
C10–N4–C7	112.6(3)
C11–N5–C6	114.0(3)
C11–N6–C12	118.9(3)
C11–N6–C25	118.8(3)
C25–N6–C12	117.1(3)
C13–N7–H7	107.9
C13–N7–C16	110.2(3)
C14–N7–H7	107.9
C14–N7–C13	112.6(3)
C14–N7–C16	110.3(3)
C16–N7–H7	107.9
C18–N8–C17	113.1(3)
C18–N8–C20	113.7(3)
C20–N8–C17	113.2(3)
C21–N9–H9	108.0
C21–N9–C24	110.4(3)
C22–N9–H9	108.0
C22–N9–C21	110.0(3)
C22–N9–C24	112.3(3)
C24–N9–H9	108.0
N1–C1–N2	117.5(3)
N1–C1–N3	126.2(3)
N3–C1–N2	116.3(3)
N2–C2–H2A	109.6
N2–C2–H2B	109.6
N2–C2–C3	110.1(3)
H2A–C2–H2B	108.2
C3–C2–H2A	109.6
C3–C2–H2B	109.6
O1–C3–C2	111.2(3)
O1–C3–H3A	109.4
O1–C3–H3B	109.4
C2–C3–H3A	109.4
C2–C3–H3B	109.4
H3A–C3–H3B	108.0
O1–C4–H4A	109.3
O1–C4–H4B	109.3
O1–C4–C5	111.6(3)
H4A–C4–H4B	108.0
C5–C4–H4A	109.3
C5–C4–H4B	109.3
N2–C5–C4	109.8(3)
N2–C5–H5A	109.7
N2–C5–H5B	109.7
C4–C5–H5A	109.7
C4–C5–H5B	109.7
H5A–C5–H5B	108.2
N3–C6–N4	117.2(3)
N3–C6–N5	126.0(3)
N5–C6–N4	116.8(3)
N4–C7–H7A	109.5
N4–C7–H7B	109.5
N4–C7–C8	110.7(3)
H7A–C7–H7B	108.1
C8–C7–H7A	109.5
C8–C7–H7B	109.5
O2–C8–C7	110.7(3)
O2–C8–H8A	109.5
O2–C8–H8B	109.5
C7–C8–H8A	109.5
C7–C8–H8B	109.5
H8A–C8–H8B	108.1
O2–C9–H9C	109.5

O2–C9–H9D	109.5	N8–C20–H20B	109.2
O2–C9–C10	110.5(3)	N8–C20–C21	112.0(3)
H9C–C9–H9D	108.1	H20A–C20–H20B	107.9
C10–C9–H9C	109.5	C21–C20–H20A	109.2
C10–C9–H9D	109.5	C21–C20–H20B	109.2
N4–C10–C9	110.0(3)	N9–C21–C20	112.9(3)
N4–C10–H10C	109.7	N9–C21–H21A	109.0
N4–C10–H10D	109.7	N9–C21–H21B	109.0
C9–C10–H10C	109.7	C20–C21–H21A	109.0
C9–C10–H10D	109.7	C20–C21–H21B	109.0
H10C–C10–H10D	108.2	H21A–C21–H21B	107.8
N1–C11–N6	117.1(3)	N9–C22–H22A	108.6
N5–C11–N1	126.2(3)	N9–C22–H22B	108.6
N5–C11–N6	116.6(3)	N9–C22–C23	114.5(3)
N6–C12–H12C	108.4	H22A–C22–H22B	107.6
N6–C12–H12D	108.4	C23–C22–H22A	108.6
N6–C12–C13	115.5(3)	C23–C22–H22B	108.6
H12C–C12–H12D	107.5	O5–C23–O6	126.9(4)
C13–C12–H12C	108.4	O5–C23–C22	116.3(3)
C13–C12–H12D	108.4	O6–C23–C22	116.8(3)
N7–C13–C12	114.9(3)	N9–C24–H24A	108.6
N7–C13–H13A	108.5	N9–C24–H24B	108.6
N7–C13–H13B	108.5	N9–C24–C25	114.7(3)
C12–C13–H13A	108.5	H24A–C24–H24B	107.6
C12–C13–H13B	108.5	C25–C24–H24A	108.6
H13A–C13–H13B	107.5	C25–C24–H24B	108.6
N7–C14–H14A	108.7	N6–C25–C24	115.2(3)
N7–C14–H14B	108.7	N6–C25–H25A	108.5
N7–C14–C15	114.3(3)	N6–C25–H25B	108.5
H14A–C14–H14B	107.6	C24–C25–H25A	108.5
C15–C14–H14A	108.7	C24–C25–H25B	108.5
C15–C14–H14B	108.7	H25A–C25–H25B	107.5
O3–C15–O4	126.7(3)	O15–S1–O17	113.0(2)
O3–C15–C14	115.9(3)	O15–S1–C28	103.4(3)
O4–C15–C14	117.3(3)	O16–S1–O15	116.2(3)
N7–C16–H16A	109.1	O16–S1–O17	116.0(3)
N7–C16–H16B	109.1	O16–S1–C28	102.5(2)
N7–C16–C17	112.7(3)	O17–S1–C28	103.3(2)
H16A–C16–H16B	107.8	F4–C28–S1	111.9(3)
C17–C16–H16A	109.1	F4–C28–F6	106.7(5)
C17–C16–H16B	109.1	F5–C28–S1	112.0(4)
N8–C17–C16	110.8(3)	F5–C28–F4	108.7(4)
N8–C17–H17A	109.5	F5–C28–F6	106.9(4)
N8–C17–H17B	109.5	F6–C28–S1	110.4(3)
C16–C17–H17A	109.5	F1–C26–F2	107.9(4)
C16–C17–H17B	109.5	F1–C26–F3	107.8(4)
H17A–C17–H17B	108.1	F1–C26–C27	112.8(3)
N8–C18–H18A	108.9	F2–C26–F3	106.0(4)
N8–C18–H18B	108.9	F2–C26–C27	111.1(3)
N8–C18–C19	113.3(3)	F3–C26–C27	110.9(3)
H18A–C18–H18B	107.7	O13–C27–O14	128.4(4)
C19–C18–H18A	108.9	O13–C27–C26	114.8(3)
C19–C18–H18B	108.9	O14–C27–C26	116.8(3)
O7–C19–O8	123.9(3)	H21C–O21–H21D	109.5
O7–C19–C18	121.0(3)	H20C–O20–H20D	109.5
O8–C19–C18	115.1(3)	H18C–O18–H18D	109.4
N8–C20–H20A	109.2	H19A–O19–H19B	109.5

Symmetry transformations used to generate equivalent atoms:

(i) –x+1,–y+1,–z+1

Table 3. Anisotropic displacement parameters [$Å^2 \times 10^3$].

The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ...+ 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$.

Atom	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Eu1	9(1)	10(1)	10(1)	2(1)	4(1)	3(1)
O2	17(1)	26(1)	19(1)	5(1)	11(1)	9(1)
O3	10(1)	21(1)	23(2)	3(1)	6(1)	5(1)
O4	11(1)	14(1)	18(1)	2(1)	8(1)	2(1)
O5	25(2)	21(1)	24(2)	2(1)	18(1)	2(1)
O6	14(1)	18(1)	19(1)	5(1)	10(1)	5(1)
O7	11(1)	10(1)	14(1)	1(1)	6(1)	3(1)
O8	11(1)	10(1)	12(1)	0(1)	6(1)	2(1)
O9	14(1)	26(2)	18(1)	8(1)	7(1)	9(1)
O10	14(1)	20(1)	20(1)	4(1)	8(1)	6(1)
O11	19(1)	16(1)	12(1)	2(1)	7(1)	7(1)
O12	18(1)	27(1)	21(1)	8(1)	11(1)	13(1)
N1	11(1)	15(1)	20(2)	6(1)	8(1)	5(1)
N2	9(1)	18(1)	16(2)	2(1)	6(1)	3(1)
N3	13(1)	17(1)	16(2)	6(1)	7(1)	7(1)
N4	13(1)	15(1)	14(1)	4(1)	6(1)	3(1)
N5	13(1)	14(1)	16(2)	3(1)	8(1)	5(1)
N6	12(1)	11(1)	13(1)	2(1)	6(1)	4(1)
N7	12(1)	12(1)	11(1)	2(1)	4(1)	4(1)
N8	16(1)	15(1)	12(1)	2(1)	6(1)	6(1)
N9	11(1)	13(1)	11(1)	2(1)	5(1)	2(1)
C1	16(2)	14(2)	17(2)	8(1)	11(1)	7(1)
C2	14(2)	17(2)	13(2)	0(1)	4(1)	2(1)
C3	16(2)	23(2)	14(2)	3(1)	5(1)	6(1)
C4	14(2)	26(2)	21(2)	4(2)	8(2)	9(2)
C5	13(2)	24(2)	14(2)	7(1)	7(1)	4(1)
C6	16(2)	10(1)	19(2)	6(1)	10(1)	6(1)
C7	19(2)	19(2)	14(2)	0(1)	8(1)	3(1)
C8	18(2)	29(2)	15(2)	4(2)	10(2)	10(2)
C9	17(2)	17(2)	19(2)	1(1)	11(2)	−1(1)
C10	17(2)	19(2)	13(2)	5(1)	7(1)	2(1)
C11	13(2)	13(2)	12(2)	6(1)	6(1)	5(1)
C12	13(2)	13(2)	16(2)	2(1)	7(1)	5(1)
C13	18(2)	10(1)	11(2)	1(1)	7(1)	3(1)
C14	12(2)	16(2)	13(2)	2(1)	5(1)	4(1)
C15	15(2)	13(2)	15(2)	4(1)	7(1)	5(1)
C16	16(2)	19(2)	12(2)	6(1)	7(1)	5(1)
C17	18(2)	16(2)	13(2)	6(1)	7(1)	7(1)
C18	20(2)	11(1)	13(2)	3(1)	7(1)	7(1)
C19	7(1)	12(1)	13(2)	1(1)	5(1)	0(1)
C20	18(2)	18(2)	12(2)	3(1)	4(1)	6(1)
C21	15(2)	17(2)	16(2)	6(1)	5(1)	7(1)
C22	13(2)	16(2)	16(2)	2(1)	9(1)	3(1)
C23	15(2)	11(2)	16(2)	4(1)	7(1)	3(1)
C24	13(2)	14(2)	10(2)	0(1)	3(1)	4(1)
C25	13(2)	10(1)	16(2)	2(1)	8(1)	2(1)
S1	31(1)	47(1)	21(1)	16(1)	19(1)	24(1)
F4	17(1)	29(2)	122(4)	27(2)	4(2)	3(1)
F5	79(3)	44(2)	37(2)	2(1)	9(2)	46(2)
F6	45(2)	33(1)	27(1)	12(1)	21(1)	10(1)
O15	108(4)	125(4)	19(2)	27(2)	28(2)	97(4)
O16	34(2)	78(3)	71(3)	51(3)	40(2)	31(2)
O17	26(2)	22(1)	28(2)	5(1)	14(1)	5(1)
C28	19(2)	25(2)	28(2)	2(2)	1(2)	11(2)
F1	22(2)	79(3)	37(2)	−37(2)	3(1)	5(2)
F2	24(1)	25(1)	37(2)	9(1)	−7(1)	9(1)
F3	18(1)	47(2)	50(2)	30(2)	−2(1)	−9(1)

O13	16(1)	20(1)	18(1)	4(1)	7(1)	4(1)
O14	12(1)	24(1)	17(1)	2(1)	4(1)	3(1)
C26	15(2)	15(2)	21(2)	1(1)	4(1)	4(1)
C27	11(2)	15(2)	16(2)	4(1)	3(1)	5(1)
O21	26(2)	38(2)	23(2)	13(1)	12(1)	13(1)
O20	23(2)	29(2)	22(2)	6(1)	12(1)	5(1)
O18	24(2)	103(4)	32(2)	32(2)	18(2)	30(2)
O19	20(1)	28(2)	19(1)	5(1)	8(1)	2(1)

Table 4. Hydrogen coordinates [$\times 10^4$] and isotropic displacement parameters [$Å^2 \times 10^3$].

Atom	x	y	z	U_{eq}	S.o.f.
H9A	8651	5334	6467	28	1
H9B	7725	4386	5996	28	1
H10A	9046	5539	4790	27	1
H10B	9270	6533	5235	27	1
H11A	5422	6949	4878	23	1
H11B	4844	6436	3836	23	1
H12A	6798	5461	2901	30	1
H12B	6258	6193	3013	30	1
H7	4967	7643	7155	15	1
H9	7362	8197	7139	15	1
H2A	1455	6659	2696	20	1
H2B	2131	7539	2366	20	1
H3A	389	8117	2058	22	1
H3B	–157	7003	1423	22	1
H4A	–548	7724	3936	24	1
H4B	155	8549	3563	24	1
H5A	1735	8280	4927	20	1
H5B	1202	7143	4371	20	1
H7A	4838	7966	2416	22	1
H7B	5995	7575	2906	22	1
H8A	6148	8083	1523	24	1
H8B	6105	9124	1955	24	1
H9C	7902	10251	3445	22	1
H9D	9150	9970	3983	22	1
H10C	7997	8797	4535	21	1
H10D	7901	9830	4924	21	1
H12C	4204	9400	6851	17	1
H12D	5412	10216	7768	17	1
H13A	5903	9023	8599	16	1
H13B	4499	9025	8398	16	1
H14A	2954	7870	6351	17	1
H14B	2569	7530	7188	17	1
H16A	4259	6506	7974	18	1
H16B	4269	7377	8755	18	1
H17A	6476	8036	9520	18	1
H17B	6203	6894	9348	18	1
H18A	5896	5985	7805	17	1
H18B	7275	6302	7812	17	1
H20A	8611	7469	9361	20	1
H20B	8423	8503	9507	20	1
H21A	9829	8653	8786	19	1
H21B	9031	7605	7996	19	1
H22A	9820	9212	7283	18	1
H22B	8509	9182	6419	18	1
H24A	9054	10095	8423	16	1
H24B	7889	9485	8597	16	1
H25A	7345	10670	7748	16	1
H25B	7574	10169	6841	16	1
H21C	4946	6036	–295	40	1

H21D	4465	5111	–174	40	1
H20C	8405	9159	1645	37	1
H20D	8226	9704	934	37	1
H18C	10783	6109	6848	71	1
H18D	10408	5269	7151	71	1
H19A	8488	5791	2338	36	1
H19B	7377	5089	1535	36	1

Table 5. Torsion angles [°].

Eu1i–O4–C15–O3	3.3(6)	C10–N4–C6–N5	3.5(5)
Eu1i–O4–C15–C14	–175.0(2)	C10–N4–C7–C8	–52.1(4)
Eu1–O6–C23–O5	–27.5(6)	C11–N1–C1–N2	–176.0(3)
Eu1–O6–C23–C22	149.5(3)	C11–N1–C1–N3	2.0(5)
Eu1–O8–C19–O7	64.5(4)	C11–N5–C6–N3	0.7(5)
Eu1i–O8–C19–O7	–88.2(4)	C11–N5–C6–N4	178.5(3)
Eu1–O8–C19–C18	–114.6(3)	C11–N6–C12–C13	115.9(4)
Eu1i–O8–C19–C18	92.8(3)	C11–N6–C25–C24	–117.2(3)
O1–C4–C5–N2	55.9(4)	C12–N6–C11–N1	–14.3(5)
O2–C9–C10–N4	–57.4(4)	C12–N6–C11–N5	167.5(3)
N2–C2–C3–O1	–56.1(4)	C12–N6–C25–C24	88.4(4)
N4–C7–C8–O2	54.6(4)	C13–N7–C14–C15	172.7(3)
N6–C12–C13–N7	–60.8(4)	C13–N7–C16–C17	–63.5(4)
N7–C14–C15–O3	162.5(3)	C14–N7–C13–C12	–66.1(4)
N7–C14–C15–O4	–19.1(5)	C14–N7–C16–C17	171.6(3)
N7–C16–C17–N8	–42.7(4)	C16–N7–C13–C12	170.3(3)
N8–C18–C19–O7	2.1(5)	C16–N7–C14–C15	–63.7(4)
N8–C18–C19–O8	–178.8(3)	C17–N8–C18–C19	114.9(3)
N8–C20–C21–N9	44.1(4)	C17–N8–C20–C21	–163.6(3)
N9–C22–C23–O5	–164.1(3)	C18–N8–C17–C16	–66.6(4)
N9–C22–C23–O6	18.6(5)	C18–N8–C20–C21	65.4(4)
N9–C24–C25–N6	62.7(4)	C20–N8–C17–C16	162.1(3)
C1–N1–C11–N5	–2.1(5)	C20–N8–C18–C19	–114.1(3)
C1–N1–C11–N6	179.9(3)	C21–N9–C22–C23	65.0(4)
C1–N2–C2–C3	–151.4(3)	C21–N9–C24–C25	–169.0(3)
C1–N2–C5–C4	152.1(3)	C22–N9–C21–C20	–173.7(3)
C1–N3–C6–N4	–178.5(3)	C22–N9–C24–C25	67.8(4)
C1–N3–C6–N5	–0.8(5)	C24–N9–C21–C20	61.7(4)
C2–N2–C1–N1	–161.2(3)	C24–N9–C22–C23	–171.5(3)
C2–N2–C1–N3	20.5(5)	C25–N6–C11–N1	–168.4(3)
C2–N2–C5–C4	–52.3(4)	C25–N6–C11–N5	13.5(5)
C3–O1–C4–C5	–60.3(4)	C25–N6–C12–C13	–89.6(4)
C4–O1–C3–C2	60.3(4)	O15–S1–C28–F4	62.5(4)
C5–N2–C1–N1	–7.5(5)	O15–S1–C28–F5	–59.8(4)
C5–N2–C1–N3	174.3(3)	O15–S1–C28–F6	–178.8(3)
C5–N2–C2–C3	52.6(4)	O16–S1–C28–F4	–176.4(4)
C6–N3–C1–N1	–0.7(5)	O16–S1–C28–F5	61.3(4)
C6–N3–C1–N2	177.3(3)	O16–S1–C28–F6	–57.6(4)
C6–N4–C7–C8	156.6(3)	O17–S1–C28–F4	–55.5(4)
C6–N4–C10–C9	–156.5(3)	O17–S1–C28–F5	–177.8(3)
C6–N5–C11–N1	0.8(5)	O17–S1–C28–F6	63.2(3)
C6–N5–C11–N6	178.8(3)	F1–C26–C27–O13	–159.9(4)
C7–N4–C6–N3	–29.8(5)	F1–C26–C27–O14	20.0(5)
C7–N4–C6–N5	152.2(3)	F2–C26–C27–O13	78.8(4)
C7–N4–C10–C9	53.0(4)	F2–C26–C27–O14	–101.3(4)
C8–O2–C9–C10	61.2(4)	F3–C26–C27–O13	–38.8(5)
C9–O2–C8–C7	–60.0(4)	F3–C26–C27–O14	141.1(4)
C10–N4–C6–N3	–178.5(3)		

Symmetry transformations used to generate equivalent atoms:
(i) –x+1,–y+1,–z+1

A.7 – GADOLINIUM(III) CRYSTAL STRUCTURE OF TZMORPH2DO3A

Table 1. Crystal data and structure refinement details.

Identification code	**2015ncs0177 (MM166)**	
Empirical formula	$C_{56}H_{108}F_{12}Gd_2N_{18}O_{42}S_2$	
Formula weight	2312.22	
Temperature	100.15 K	
Wavelength	0.71075 Å	
Crystal system	Triclinic	
Space group	*P*–1	
Unit cell dimensions	*a* = 11.7628(2) Å	α = 99.074(7)°
	b = 14.5613(3) Å	β = 112.149(8)°
	c = 14.7079(10) Å	γ= 104.568(7)°
Volume	2166.8(2) Å^3	
Z	1	
Density (calculated)	1.772 Mg / m^3	
Absorption coefficient	1.694 mm^{-1}	
F(000)	1174	
Crystal	Block; Colorless	
Crystal size	0.27 × 0.17 × 0.07 mm^3	
θ range for data collection	3.013 – 27.484°	
Index ranges	$-15 \le h \le 14$, $-18 \le k \le 18$, $-19 \le l \le 18$	
Reflections collected	29871	
Independent reflections	9875 [R_{int} = 0.0392]	
Completeness to θ = 25.242°	99.8 %	
Absorption correction	Semi–empirical from equivalents	
Max. and min. transmission	1.000 and 0.793	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	9875 / 6 / 618	
Goodness-of-fit on F^2	0.865	
Final *R* indices [$F^2 > 2\sigma(F^2)$]	*R1* = 0.0267, *wR2* = 0.0960	
R indices (all data)	*R1* = 0.0277, *wR2* = 0.0973	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.806 and –1.007 e Å^{-3}	

Diffractometer: Rigaku AFC12 goniometer equipped with an enhanced sensitivity (HG) Saturn724+ detector mounted at the window of an FR-E+ SuperBright molybdenum rotating anode generator with HF Varimax optics (100m focus). **Cell determination and data collection:** CrystalClear-SM Expe. rt 3.1 b27 (Rigaku, 2012). **Data reduction, cell refinement and absorption correction:** CrystalClear-SM Expert 3.1 b18 (Rigaku, 2012)**Structure solution:** SUPERFLIP (Palatinus, L. & Chapuis, G. (2007). J. Appl. Cryst. 40, 786-790). **Structure refinement:** SHELXL-2013 (Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122).

Special details:
In the crystal structure one water molecule (O21a/O21B) is disordered and modelled over two sites with 64:36 ratio. The hydrogen atoms of two partially occupied Oxygen atoms have not been assigned.

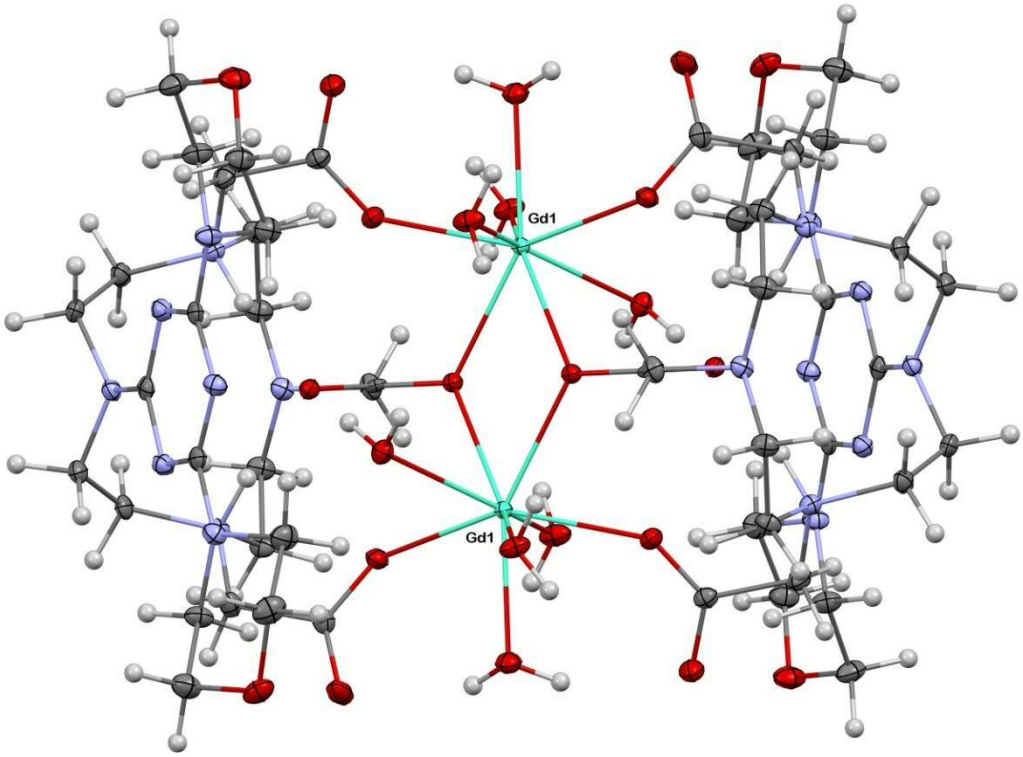

Figure 1. Molecular structure of 2015ncs0177 (MM166). Displacement ellipsoids – 50% probability. All counteranions and water molecules has been omitted for clarity.

Table 1. Atomic coordinates [$\times 10^4$], equivalent isotropic displacement parameters [$Å^2 \times 10^3$] and site occupancy factors. U_{eq} is defined as one third of the trace of the orthogonalized U^{ij} tensor.

Atom	x	y	z	U_{eq}	S.o.f.
Gd1	3327(1)	4456(1)	209(1)	8(1)	1
F1	1770(2)	4640(2)	−4858(2)	67(1)	1
F2	1220(2)	5795(1)	−4251(1)	37(1)	1
F3	1078(2)	4517(2)	−3731(2)	48(1)	1
O1	7841(2)	8964(1)	−2282(1)	19(1)	1
O2	−566(2)	7195(1)	−2393(1)	19(1)	1
O3	9608(2)	7929(1)	811(1)	20(1)	1
O4	5745(2)	7237(1)	1324(1)	11(1)	1
O5	5518(1)	5637(1)	905(1)	11(1)	1
O6	1310(2)	5994(1)	740(1)	17(1)	1
O7	3321(2)	5969(1)	1086(1)	14(1)	1
O8	2184(2)	5008(1)	−1185(1)	17(1)	1
O9	3760(2)	4386(1)	1932(1)	16(1)	1
O10	4421(2)	3286(1)	619(1)	14(1)	1
O11	1277(2)	4002(1)	266(1)	16(1)	1
O12	2039(2)	2941(1)	−1078(1)	13(1)	1
O13	4113(2)	5799(1)	−3438(1)	19(1)	1
O14	3298(2)	5870(1)	−2285(1)	17(1)	1
N1	6248(2)	8884(1)	−1231(1)	13(1)	1
N2	4203(2)	8382(2)	−1270(1)	13(1)	1
N3	2186(2)	7963(2)	−1252(1)	14(1)	1
N4	6122(2)	9158(1)	317(1)	12(1)	1
N5	4015(2)	8661(1)	308(1)	12(1)	1
N6	5908(2)	9422(1)	1828(1)	12(1)	1
N7	8266(2)	8664(1)	2556(1)	11(1)	1
N8	6859(2)	7451(1)	3390(1)	12(1)	1
N9	4479(2)	7793(1)	2543(1)	11(1)	1
C1	7672(2)	9309(2)	−674(2)	15(1)	1
C2	8223(3)	9711(2)	−1373(2)	20(1)	1
C3	6436(3)	8598(2)	−2845(2)	21(1)	1
C4	5801(2)	8163(2)	−2219(2)	19(1)	1
C5	5502(2)	8803(2)	−709(2)	12(1)	1
C6	3514(2)	8339(2)	−718(2)	12(1)	1
C7	1567(2)	7329(2)	−2307(2)	17(1)	1
C8	261(2)	7428(2)	−2897(2)	18(1)	1
C9	25(2)	7855(2)	−1388(2)	18(1)	1
C10	1342(2)	7789(2)	−735(2)	16(1)	1
C11	5327(2)	9077(2)	779(2)	11(1)	1
C12	7284(2)	10045(2)	2352(2)	13(1)	1
C13	8191(2)	9617(2)	3074(2)	13(1)	1
C14	8958(2)	8196(2)	3340(2)	14(1)	1
C15	8264(2)	7926(2)	4006(2)	14(1)	1
C16	6119(2)	7423(2)	4005(2)	15(1)	1
C17	4676(2)	7221(2)	3331(2)	14(1)	1
C18	5023(2)	8884(2)	3064(2)	14(1)	1
C19	5093(2)	9542(2)	2358(2)	13(1)	1
C20	8923(2)	8809(2)	1867(2)	14(1)	1
C21	8843(2)	7853(2)	1204(2)	13(1)	1
C22	6493(2)	6489(2)	2674(2)	14(1)	1
C23	5871(2)	6474(2)	1563(2)	10(1)	1
C24	3072(2)	7457(2)	1804(2)	14(1)	1
C25	2523(2)	6378(2)	1161(2)	12(1)	1
C26	1817(2)	5138(2)	−4027(2)	20(1)	1
C27	3228(2)	5659(2)	−3162(2)	14(1)	1
S1	2770(1)	8722(1)	4595(1)	26(1)	1
F4	2481(2)	6891(2)	4600(2)	50(1)	1
F5	923(2)	7355(2)	4682(2)	63(1)	1

F6	2770(2)	7831(1)	5995(1)	34(1)	1
O15	2102(3)	8381(3)	3493(2)	71(1)	1
O16	4162(2)	8930(2)	5043(2)	47(1)	1
O17	2319(2)	9430(1)	5030(1)	24(1)	1
C62	2197(3)	7642(2)	4984(2)	25(1)	1
O21A	–503(18)	4010(20)	–2260(11)	24(3)	0.65(8)
O21B	–296(19)	4380(40)	–2114(18)	24(4)	0.35(8)
O20	–2178(2)	5252(1)	–2825(1)	22(1)	1
O18	1357(2)	10664(2)	3808(2)	34(1)	1
O19	–4316(2)	4366(2)	–4709(1)	27(1)	1

Table 2. Bond lengths [Å] and angles [°].

Gd1–O5i	2.5002(15)	N7–C13	1.514(3)
Gd1–O5	2.4238(15)	N7–C14	1.508(3)
Gd1–O7	2.3747(16)	N7–C20	1.498(3)
Gd1–O8	2.3759(18)	N8–C15	1.459(3)
Gd1–O9	2.4131(16)	N8–C16	1.472(3)
Gd1–O10	2.4009(16)	N8–C22	1.465(3)
Gd1–O11	2.3718(16)	N9–H9	1.0000
Gd1–O12	2.3623(15)	N9–C17	1.513(3)
F1–C26	1.294(3)	N9–C18	1.502(3)
F2–C26	1.333(3)	N9–C24	1.493(3)
F3–C26	1.333(3)	C1–H1A	0.9900
O1–C2	1.421(3)	C1–H1B	0.9900
O1–C3	1.445(3)	C1–C2	1.524(3)
O2–C8	1.437(3)	C2–H2A	0.9900
O2–C9	1.426(3)	C2–H2B	0.9900
O3–C21	1.233(3)	C3–H3A	0.9900
O4–C23	1.243(3)	C3–H3B	0.9900
O5–Gd1i	2.5002(15)	C3–C4	1.507(3)
O5–C23	1.290(3)	C4–H4A	0.9900
O6–C25	1.247(3)	C4–H4B	0.9900
O7–C25	1.260(3)	C7–H7A	0.9900
O8–H8A	0.8797	C7–H7B	0.9900
O8–H8B	0.8794	C7–C8	1.510(3)
O9–H9A	0.8642	C8–H8C	0.9900
O9–H9B	0.8639	C8–H8D	0.9900
O10–H10A	0.8762	C9–H9C	0.9900
O10–H10B	0.8757	C9–H9D	0.9900
O11–H11A	0.8759	C9–C10	1.522(3)
O11–H11B	0.8768	C10–H10C	0.9900
O12–C21i	1.278(3)	C10–H10D	0.9900
O13–C27	1.236(3)	C12–H12A	0.9900
O14–C27	1.244(3)	C12–H12B	0.9900
N1–C1	1.464(3)	C12–C13	1.531(3)
N1–C4	1.468(3)	C13–H13A	0.9900
N1–C5	1.363(3)	C13–H13B	0.9900
N2–C5	1.345(3)	C14–H14A	0.9900
N2–C6	1.344(3)	C14–H14B	0.9900
N3–C6	1.366(3)	C14–C15	1.532(3)
N3–C7	1.465(3)	C15–H15A	0.9900
N3–C10	1.461(3)	C15–H15B	0.9900
N4–C5	1.346(3)	C16–H16A	0.9900
N4–C11	1.342(3)	C16–H16B	0.9900
N5–C6	1.347(3)	C16–C17	1.530(3)
N5–C11	1.349(3)	C17–H17A	0.9900
N6–C11	1.374(3)	C17–H17B	0.9900
N6–C12	1.462(3)	C18–H18A	0.9900
N6–C19	1.470(3)	C18–H18B	0.9900
N7–H7	1.0000	C18–C19	1.531(3)

C19–H19A	0.9900
C19–H19B	0.9900
C20–H20A	0.9900
C20–H20B	0.9900
C20–C21	1.528(3)
C21–O12i	1.278(3)
C22–H22A	0.9900
C22–H22B	0.9900
C22–C23	1.513(3)
C24–H24A	0.9900
C24–H24B	0.9900
C24–C25	1.529(3)
C26–C27	1.555(3)
S1–O15	1.443(2)
S1–O16	1.443(2)
S1–O17	1.4415(19)
S1–C62	1.815(3)
F4–C62	1.326(3)
F5–C62	1.319(3)
F6–C62	1.327(3)
O20–H20C	0.8702
O20–H20D	0.8692
O18–H18C	0.8498
O18–H18D	0.8509
O19–H19C	0.8701
O19–H19D	0.8690
O5–Gd1–O5i	64.33(6)
O7–Gd1–O5i	123.10(5)
O7–Gd1–O5	72.68(5)
O7–Gd1–O8	78.95(6)
O7–Gd1–O9	73.18(6)
O7–Gd1–O10	137.28(5)
O8–Gd1–O5i	75.49(6)
O8–Gd1–O5	101.83(6)
O8–Gd1–O9	146.39(6)
O8–Gd1–O10	142.10(6)
O9–Gd1–O5	87.58(5)
O9–Gd1–O5i	136.14(5)
O10–Gd1–O5	84.30(5)
O10–Gd1–O5i	73.80(5)
O10–Gd1–O9	70.30(6)
O11–Gd1–O5	147.70(5)
O11–Gd1–O5i	145.41(5)
O11–Gd1–O7	76.83(6)
O11–Gd1–O8	82.27(6)
O11–Gd1–O9	73.49(6)
O11–Gd1–O10	112.23(6)
O12–Gd1–O5	138.05(5)
O12–Gd1–O5i	75.62(5)
O12–Gd1–O7	145.38(6)
O12–Gd1–O8	78.69(6)
O12–Gd1–O9	115.32(6)
O12–Gd1–O10	72.63(6)
O12–Gd1–O11	74.26(6)
C2–O1–C3	109.57(18)
C9–O2–C8	110.04(17)
Gd1–O5–Gd1i	115.67(6)
C23–O5–Gd1	121.38(13)
C23–O5–Gd1i	117.55(13)
C25–O7–Gd1	139.66(15)

Gd1–O8–H8A	110.0
Gd1–O8–H8B	109.9
H8A–O8–H8B	109.0
Gd1–O9–H9A	110.1
Gd1–O9–H9B	109.9
H9A–O9–H9B	108.7
Gd1–O10–H10A	110.8
Gd1–O10–H10B	110.6
H10A–O10–H10B	108.1
Gd1–O11–H11A	110.6
Gd1–O11–H11B	111.0
H11A–O11–H11B	107.9
C21i–O12–Gd1	136.82(14)
C1–N1–C4	113.01(18)
C5–N1–C1	120.09(19)
C5–N1–C4	120.14(19)
C6–N2–C5	114.29(19)
C6–N3–C7	120.72(18)
C6–N3–C10	121.54(18)
C10–N3–C7	112.62(18)
C11–N4–C5	114.29(19)
C6–N5–C11	113.98(18)
C11–N6–C12	118.81(18)
C11–N6–C19	118.97(18)
C12–N6–C19	117.00(17)
C13–N7–H7	107.9
C14–N7–H7	107.9
C14–N7–C13	110.61(17)
C20–N7–H7	107.9
C20–N7–C13	112.60(18)
C20–N7–C14	109.76(17)
C15–N8–C16	112.95(17)
C15–N8–C22	113.69(18)
C22–N8–C16	112.85(18)
C17–N9–H9	107.9
C18–N9–H9	107.9
C18–N9–C17	110.12(17)
C24–N9–H9	107.9
C24–N9–C17	110.46(17)
C24–N9–C18	112.51(18)
N1–C1–H1A	109.9
N1–C1–H1B	109.9
N1–C1–C2	109.10(19)
H1A–C1–H1B	108.3
C2–C1–H1A	109.9
C2–C1–H1B	109.9
O1–C2–C1	111.4(2)
O1–C2–H2A	109.3
O1–C2–H2B	109.3
C1–C2–H2A	109.3
C1–C2–H2B	109.3
H2A–C2–H2B	108.0
O1–C3–H3A	109.4
O1–C3–H3B	109.4
O1–C3–C4	111.0(2)
H3A–C3–H3B	108.0
C4–C3–H3A	109.4
C4–C3–H3B	109.4
N1–C4–C3	110.6(2)
N1–C4–H4A	109.5
N1–C4–H4B	109.5

C3–C4–H4A	109.5
C3–C4–H4B	109.5
H4A–C4–H4B	108.1
N2–C5–N1	116.8(2)
N2–C5–N4	125.7(2)
N4–C5–N1	117.5(2)
N2–C6–N3	116.76(19)
N2–C6–N5	125.8(2)
N5–C6–N3	117.36(19)
N3–C7–H7A	109.6
N3–C7–H7B	109.6
N3–C7–C8	110.49(19)
H7A–C7–H7B	108.1
C8–C7–H7A	109.6
C8–C7–H7B	109.6
O2–C8–C7	110.34(19)
O2–C8–H8C	109.6
O2–C8–H8D	109.6
C7–C8–H8C	109.6
C7–C8–H8D	109.6
H8C–C8–H8D	108.1
O2–C9–H9C	109.2
O2–C9–H9D	109.2
O2–C9–C10	111.85(19)
H9C–C9–H9D	107.9
C10–C9–H9C	109.2
C10–C9–H9D	109.2
N3–C10–C9	109.51(19)
N3–C10–H10C	109.8
N3–C10–H10D	109.8
C9–C10–H10C	109.8
C9–C10–H10D	109.8
H10C–C10–H10D	108.2
N4–C11–N5	125.86(19)
N4–C11–N6	116.83(19)
N5–C11–N6	117.28(19)
N6–C12–H12A	108.4
N6–C12–H12B	108.4
N6–C12–C13	115.32(19)
H12A–C12–H12B	107.5
C13–C12–H12A	108.4
C13–C12–H12B	108.4
N7–C13–C12	114.55(17)
N7–C13–H13A	108.6
N7–C13–H13B	108.6
C12–C13–H13A	108.6
C12–C13–H13B	108.6
H13A–C13–H13B	107.6
N7–C14–H14A	109.0
N7–C14–H14B	109.0
N7–C14–C15	113.03(18)
H14A–C14–H14B	107.8
C15–C14–H14A	109.0
C15–C14–H14B	109.0
N8–C15–C14	111.26(17)
N8–C15–H15A	109.4
N8–C15–H15B	109.4
C14–C15–H15A	109.4
C14–C15–H15B	109.4
H15A–C15–H15B	108.0
N8–C16–H16A	109.5
N8–C16–H16B	109.5
N8–C16–C17	110.80(18)
H16A–C16–H16B	108.1
C17–C16–H16A	109.5
C17–C16–H16B	109.5
N9–C17–C16	112.48(19)
N9–C17–H17A	109.1
N9–C17–H17B	109.1
C16–C17–H17A	109.1
C16–C17–H17B	109.1
H17A–C17–H17B	107.8
N9–C18–H18A	108.6
N9–C18–H18B	108.6
N9–C18–C19	114.76(18)
H18A–C18–H18B	107.6
C19–C18–H18A	108.6
C19–C18–H18B	108.6
N6–C19–C18	115.88(19)
N6–C19–H19A	108.3
N6–C19–H19B	108.3
C18–C19–H19A	108.3
C18–C19–H19B	108.3
H19A–C19–H19B	107.4
N7–C20–H20A	108.6
N7–C20–H20B	108.6
N7–C20–C21	114.71(19)
H20A–C20–H20B	107.6
C21–C20–H20A	108.6
C21–C20–H20B	108.6
O3–C21–O12i	126.1(2)
O3–C21–C20	116.7(2)
O12i–C21–C20	117.1(2)
N8–C22–H22A	108.9
N8–C22–H22B	108.9
N8–C22–C23	113.48(19)
H22A–C22–H22B	107.7
C23–C22–H22A	108.9
C23–C22–H22B	108.9
O4–C23–O5	123.6(2)
O4–C23–C22	120.74(19)
O5–C23–C22	115.6(2)
N9–C24–H24A	108.7
N9–C24–H24B	108.7
N9–C24–C25	114.19(18)
H24A–C24–H24B	107.6
C25–C24–H24A	108.7
C25–C24–H24B	108.7
O6–C25–O7	126.2(2)
O6–C25–C24	116.0(2)
O7–C25–C24	117.8(2)
F1–C26–F2	108.7(2)
F1–C26–F3	107.4(2)
F1–C26–C27	113.4(2)
F2–C26–C27	110.7(2)
F3–C26–F2	104.8(2)
F3–C26–C27	111.5(2)
O13–C27–O14	129.4(2)
O13–C27–C26	116.1(2)
O14–C27–C26	114.5(2)
O15–S1–C62	103.53(18)
O16–S1–O15	116.06(17)

O16–S1–C62	102.27(13)	F5–C62–F4	108.2(2)
O17–S1–O15	113.09(13)	F5–C62–F6	108.0(3)
O17–S1–O16	116.28(16)	F6–C62–S1	110.73(18)
O17–S1–C62	103.01(12)	H20C–O20–H20D	109.4
F4–C62–S1	110.9(2)	H18C–O18–H18D	109.4
F4–C62–F6	107.1(2)	H19C–O19–H19D	109.5
F5–C62–S1	111.80(19)		

Symmetry transformations used to generate equivalent atoms:
(i) –x+1,–y+1,–z

Table 3. Anisotropic displacement parameters [$Å^2 \times 10^3$].
The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + \cdots + 2\,h\,k\,a^*\,b^*\,U^{12}]$.

Atom	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Gd1	8(1)	9(1)	9(1)	2(1)	4(1)	3(1)
F1	24(1)	90(2)	41(1)	–45(1)	1(1)	6(1)
F2	26(1)	24(1)	39(1)	11(1)	–9(1)	9(1)
F3	20(1)	48(1)	53(1)	33(1)	–3(1)	–10(1)
O1	20(1)	27(1)	18(1)	8(1)	12(1)	11(1)
O2	11(1)	25(1)	16(1)	1(1)	4(1)	2(1)
O3	23(1)	16(1)	22(1)	1(1)	16(1)	0(1)
O4	12(1)	11(1)	12(1)	4(1)	6(1)	5(1)
O5	9(1)	11(1)	13(1)	2(1)	5(1)	3(1)
O6	12(1)	16(1)	22(1)	3(1)	7(1)	5(1)
O7	12(1)	14(1)	16(1)	3(1)	7(1)	6(1)
O8	15(1)	25(1)	17(1)	12(1)	9(1)	10(1)
O9	12(1)	25(1)	14(1)	6(1)	7(1)	9(1)
O10	15(1)	14(1)	13(1)	4(1)	6(1)	7(1)
O11	11(1)	18(1)	18(1)	3(1)	6(1)	4(1)
O12	10(1)	13(1)	14(1)	1(1)	5(1)	1(1)
O13	15(1)	24(1)	15(1)	6(1)	5(1)	5(1)
O14	16(1)	18(1)	16(1)	6(1)	6(1)	5(1)
N1	12(1)	13(1)	13(1)	3(1)	5(1)	2(1)
N2	11(1)	14(1)	13(1)	4(1)	5(1)	4(1)
N3	10(1)	17(1)	11(1)	3(1)	4(1)	3(1)
N4	12(1)	12(1)	14(1)	5(1)	6(1)	3(1)
N5	12(1)	12(1)	14(1)	4(1)	6(1)	4(1)
N6	12(1)	9(1)	12(1)	1(1)	5(1)	2(1)
N7	10(1)	12(1)	10(1)	2(1)	4(1)	3(1)
N8	12(1)	13(1)	9(1)	2(1)	4(1)	2(1)
N9	10(1)	12(1)	11(1)	3(1)	5(1)	5(1)
C1	12(1)	20(1)	14(1)	5(1)	6(1)	3(1)
C2	16(1)	22(1)	19(1)	5(1)	9(1)	3(1)
C3	22(1)	30(1)	14(1)	6(1)	10(1)	11(1)
C4	19(1)	21(1)	15(1)	1(1)	9(1)	4(1)
C5	15(1)	9(1)	16(1)	6(1)	7(1)	6(1)
C6	14(1)	7(1)	15(1)	4(1)	7(1)	5(1)
C7	16(1)	19(1)	13(1)	2(1)	6(1)	4(1)
C8	16(1)	21(1)	16(1)	6(1)	7(1)	5(1)
C9	12(1)	25(1)	18(1)	3(1)	6(1)	7(1)
C10	13(1)	23(1)	15(1)	7(1)	7(1)	7(1)
C11	14(1)	8(1)	13(1)	6(1)	6(1)	6(1)
C12	13(1)	10(1)	15(1)	0(1)	5(1)	2(1)
C13	13(1)	9(1)	12(1)	–1(1)	4(1)	1(1)
C14	12(1)	16(1)	13(1)	6(1)	4(1)	5(1)
C15	13(1)	19(1)	9(1)	3(1)	4(1)	3(1)
C16	14(1)	18(1)	13(1)	6(1)	6(1)	5(1)
C17	14(1)	16(1)	12(1)	6(1)	7(1)	4(1)
C18	16(1)	11(1)	13(1)	1(1)	8(1)	3(1)
C19	14(1)	10(1)	16(1)	1(1)	8(1)	5(1)

C20	19(1)	11(1)	13(1)	2(1)	10(1)	3(1)
C21	14(1)	13(1)	11(1)	3(1)	4(1)	4(1)
C22	16(1)	12(1)	12(1)	5(1)	4(1)	4(1)
C23	7(1)	12(1)	13(1)	3(1)	7(1)	4(1)
C24	11(1)	16(1)	15(1)	3(1)	5(1)	7(1)
C25	13(1)	15(1)	12(1)	5(1)	6(1)	6(1)
C26	15(1)	18(1)	21(1)	3(1)	4(1)	4(1)
C27	14(1)	9(1)	17(1)	4(1)	3(1)	4(1)
S1	32(1)	40(1)	19(1)	13(1)	18(1)	21(1)
F4	72(2)	38(1)	38(1)	3(1)	11(1)	41(1)
F5	14(1)	28(1)	127(2)	31(1)	8(1)	4(1)
F6	45(1)	37(1)	28(1)	15(1)	22(1)	14(1)
O15	117(3)	127(3)	20(1)	31(1)	37(1)	105(2)
O16	34(1)	69(2)	67(2)	42(2)	38(1)	25(1)
O17	32(1)	19(1)	27(1)	5(1)	19(1)	8(1)
C62	18(1)	20(1)	28(1)	0(1)	2(1)	10(1)
O21A	14(3)	33(7)	21(3)	7(4)	5(2)	3(4)
O21B	4(4)	30(12)	23(5)	5(6)	–1(3)	–6(6)
O20	21(1)	25(1)	21(1)	7(1)	12(1)	6(1)
O18	37(1)	38(1)	28(1)	7(1)	16(1)	9(1)
O19	25(1)	40(1)	18(1)	11(1)	9(1)	13(1)

Table 4. Hydrogen coordinates [$\times 10^4$] and isotropic displacement parameters [$Å^2 \times 10^3$].

Atom	x	y	z	U_{eq}	S.o.f.
H8A	1371	4604	−1526	26	1
H8B	2561	5024	−1600	26	1
H9A	3255	4617	2127	24	1
H9B	4561	4736	2344	24	1
H10A	5176	3570	1157	21	1
H10B	4561	3031	108	21	1
H11A	681	3554	−299	24	1
H11B	1022	4514	334	24	1
H7	7353	8196	2128	13	1
H9	4979	7649	2152	13	1
H1A	8033	8796	−449	18	1
H1B	7923	9849	−59	18	1
H2A	7909	10258	−1553	23	1
H2B	9187	9982	−1005	23	1
H3A	6173	8086	−3485	26	1
H3B	6132	9147	−3032	26	1
H4A	4841	7963	−2599	23	1
H4B	6020	7565	−2102	23	1
H7A	1445	6632	−2304	20	1
H7B	2140	7518	−2645	20	1
H8C	390	8112	−2949	21	1
H8D	−162	6974	−3598	21	1
H9C	−562	7696	−1055	22	1
H9D	142	8541	−1435	22	1
H10C	1753	8288	−60	19	1
H10D	1222	7126	−621	19	1
H12A	7356	10689	2750	16	1
H12B	7594	10172	1829	16	1
H13A	9080	10116	3426	15	1
H13B	7890	9497	3603	15	1
H14A	9855	8660	3786	16	1
H14B	9026	7591	2983	16	1
H15A	8617	7474	4374	17	1
H15B	8435	8533	4522	17	1
H16A	6484	8064	4536	18	1
H16B	6206	6899	4354	18	1
H17A	4242	6505	2976	16	1
H17B	4257	7402	3769	16	1
H18A	5912	9044	3611	16	1
H18B	4477	9047	3395	16	1
H19A	4195	9408	1836	16	1
H19B	5429	10241	2768	16	1
H20A	9854	9213	2293	17	1
H20B	8525	9188	1414	17	1
H22A	5876	5985	2802	16	1
H22B	7283	6303	2808	16	1
H24A	2959	7884	1338	17	1
H24B	2557	7546	2189	17	1
H20C	−1826	5832	−2876	33	1
H20D	−1683	4899	−2829	33	1
H18C	1516	10737	3302	52	1
H18D	1817	10352	4134	52	1
H19C	−3558	4717	−4207	40	1
H19D	−4221	4173	−5257	40	1

Table 5. Torsion angles [°].

Gd1–O5–C23–O4	–88.9(2)	C7–N3–C6–N5	–161.0(2)
Gd1i–O5–C23–O4	64.0(2)	C7–N3–C10–C9	–52.2(3)
Gd1–O5–C23–C22	92.49(19)	C8–O2–C9–C10	–60.1(3)
Gd1i–O5–C23–C22	–114.57(17)	C9–O2–C8–C7	60.3(3)
Gd1–O7–C25–O6	3.7(4)	C10–N3–C6–N2	173.8(2)
Gd1–O7–C25–C24	–174.57(15)	C10–N3–C6–N5	–8.1(3)
F1–C26–C27–O13	21.3(3)	C10–N3–C7–C8	53.8(3)
F1–C26–C27–O14	–158.2(3)	C11–N4–C5–N1	178.34(19)
F2–C26–C27–O13	–101.1(3)	C11–N4–C5–N2	–0.8(3)
F2–C26–C27–O14	79.3(3)	C11–N5–C6–N2	1.6(3)
F3–C26–C27–O13	142.6(2)	C11–N5–C6–N3	–176.2(2)
F3–C26–C27–O14	–36.9(3)	C11–N6–C12–C13	–117.4(2)
O1–C3–C4–N1	55.0(3)	C11–N6–C19–C18	116.1(2)
O2–C9–C10–N3	55.5(3)	C12–N6–C11–N4	13.7(3)
N1–C1–C2–O1	–57.4(3)	C12–N6–C11–N5	–168.19(19)
N3–C7–C8–O2	–56.8(3)	C12–N6–C19–C18	–89.7(2)
N6–C12–C13–N7	62.7(2)	C13–N7–C14–C15	61.0(2)
N7–C14–C15–N8	44.4(3)	C13–N7–C20–C21	–171.22(18)
N7–C20–C21–O3	–163.7(2)	C14–N7–C13–C12	–168.77(18)
N7–C20–C21–O12i	18.9(3)	C14–N7–C20–C21	65.1(2)
N8–C16–C17–N9	–42.0(3)	C15–N8–C16–C17	162.6(2)
N8–C22–C23–O4	2.2(3)	C15–N8–C22–C23	–114.4(2)
N8–C22–C23–O5	–179.17(18)	C16–N8–C15–C14	–164.4(2)
N9–C18–C19–N6	–60.3(3)	C16–N8–C22–C23	115.3(2)
N9–C24–C25–O6	161.93(19)	C17–N9–C18–C19	169.96(18)
N9–C24–C25–O7	–19.7(3)	C17–N9–C24–C25	–63.6(2)
C1–N1–C4–C3	–52.1(3)	C18–N9–C17–C16	–63.9(2)
C1–N1–C5–N2	–178.3(2)	C18–N9–C24–C25	172.87(18)
C1–N1–C5–N4	2.4(3)	C19–N6–C11–N4	167.48(19)
C2–O1–C3–C4	–60.2(3)	C19–N6–C11–N5	–14.4(3)
C3–O1–C2–C1	61.6(3)	C19–N6–C12–C13	88.3(2)
C4–N1–C1–C2	52.4(3)	C20–N7–C13–C12	68.0(2)
C4–N1–C5–N2	–29.2(3)	C20–N7–C14–C15	–174.14(18)
C4–N1–C5–N4	151.6(2)	C22–N8–C15–C14	65.3(2)
C5–N1–C1–C2	–156.4(2)	C22–N8–C16–C17	–66.7(2)
C5–N1–C4–C3	156.7(2)	C24–N9–C17–C16	171.21(18)
C5–N2–C6–N3	176.9(2)	C24–N9–C18–C19	–66.3(2)
C5–N2–C6–N5	–0.9(3)	O15–S1–C62–F4	59.0(2)
C5–N4–C11–N5	1.7(3)	O15–S1–C62–F5	–61.8(2)
C5–N4–C11–N6	179.54(19)	O15–S1–C62–F6	177.68(19)
C6–N2–C5–N1	–178.68(19)	O16–S1–C62–F4	–62.0(2)
C6–N2–C5–N4	0.5(3)	O16–S1–C62–F5	177.2(2)
C6–N3–C7–C8	–151.2(2)	O16–S1–C62–F6	56.7(2)
C6–N3–C10–C9	153.0(2)	O17–S1–C62–F4	177.00(18)
C6–N5–C11–N4	–2.0(3)	O17–S1–C62–F5	56.2(2)
C6–N5–C11–N6	–179.91(19)	O17–S1–C62–F6	–64.3(2)
C7–N3–C6–N2	21.0(3)		

Symmetry transformations used to generate equivalent atoms:

(i) –x+1,–y+1,–z

Table 1. Crystal data and structure refinement details.

Identification code	**2015ncs0216 (MM161) Rev2**	
Empirical formula	$C_{58}H_{96}F_{18}La_2N_{18}Na_2O_{46}S_4$	
Formula weight	2575.56	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	*P*121/*n*1	
Unit cell dimensions	a = 14.3134(2) Å	α = 90°
	b = 13.6184(2) Å	β = 94.9153(16)°
	c = 24.5269(4) Å	γ= 90°
Volume	4763.31(13) Å^3	
Z	2	
Density (calculated)	1.796 Mg / m^3	
Absorption coefficient	1.119 mm^{-1}	
F(000)	2600	
Crystal	Plate; colourless	
Crystal size	0.24 × 0.11 × 0.02 mm^3	
θ range for data collection	1.712 – 29.682°	
Index ranges	$-19 \le h \le 12$, $-18 \le k \le 12$, $-32 \le l \le 33$	
Reflections collected	37098	
Independent reflections	12098 [R_{int} = 0.0304]	
Completeness to θ = 26.000°	100.0 %	
Absorption correction	Semi–empirical from equivalents	
Max. and min. transmission	1.00000 and 0.71738	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	12098 / 1309 / 692	
Goodness-of-fit on F^2	1.062	
Final R indices [$F^2 > 2\sigma(F^2)$]	*R1* = 0.0710, *wR2* = 0.1992	
R indices (all data)	*R1* = 0.0830, *wR2* = 0.2178	
Extinction coefficient	n/a	
Largest diff. peak and hole	4.540 and –2.676 e Å^{-3}	

Diffractometer:*Rigaku AFC12* goniometer equipped with an enhanced sensitivity (HG) *Saturn724+* detector mounted at the window of an *FR-E+ SuperBright* molybdenum rotating anode generator with HF *Varimax* optics (100µm focus). **Cell determination anddata collection**:*CrystalClear-SM Expert 3.0 r27* (Rigaku, 2012). **Data reduction, cellrefinement andabsorption correction**: *CrystalClear-SM Expert 2.0 r13* (Rigaku, 2011). **Structure solution**: *ShelXT (Sheldrick, 2015)*. Acta Cryst. A64, 112-122). **Structure refinement**: *SHELXL-2014*(Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122).

Special details:
Crystal structure contains two disordered CF3SO3- molecules. One was modelled over two sites with 58/42 ratio, whereas the second one was kept as isotropic.

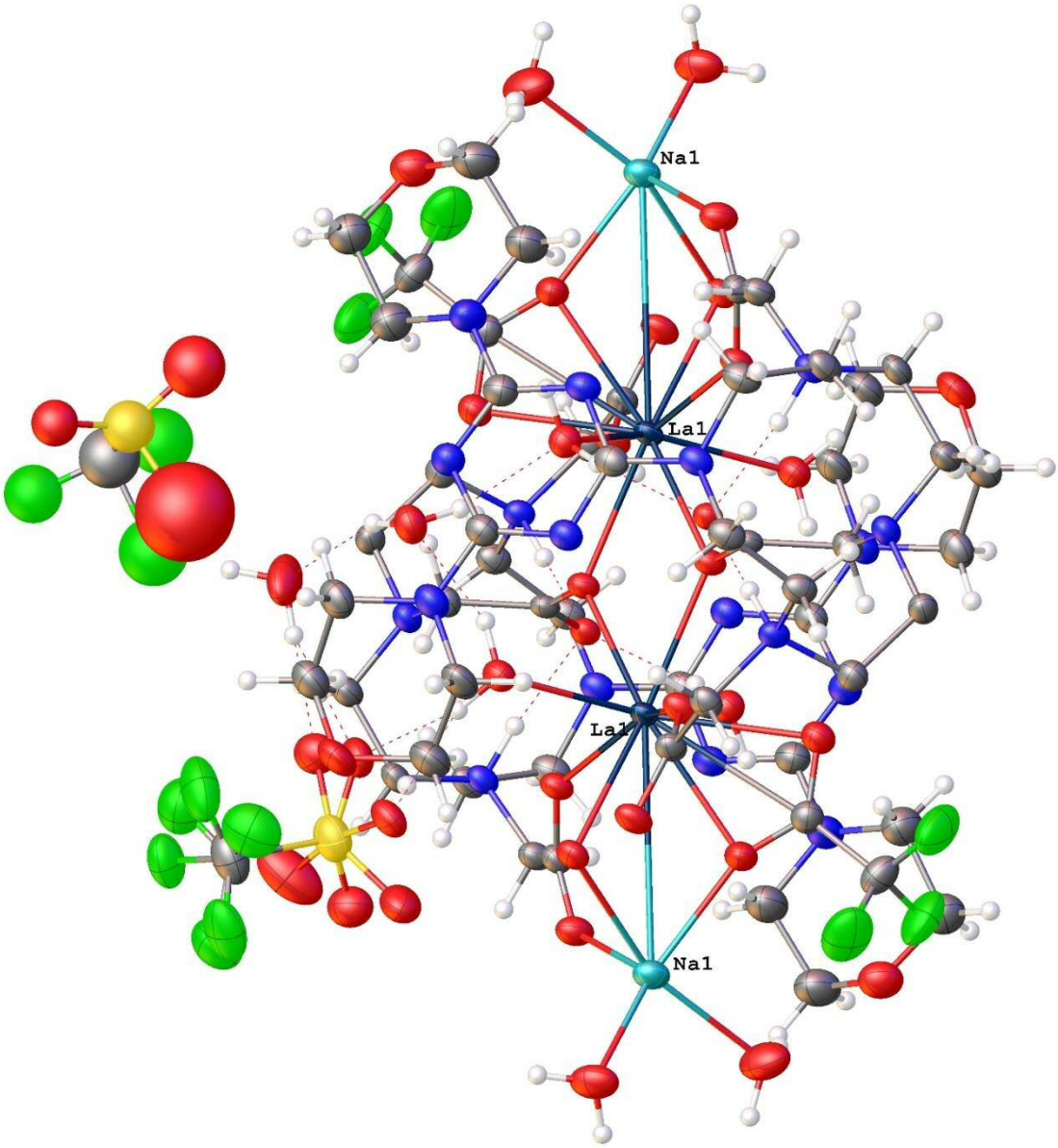

Figure 1. Molecular structure of 2015ncs0216 (MM161) Rev2 – grown fragment.
Displacement ellipsoids- 50% probability

Table 1. Atomic coordinates [$\times 10^4$], equivalent isotropic displacement parameters [$Å^2 \times 10^3$] and site occupancy factors. U_{eq} is defined as one third of the trace of the orthogonalized U^{ij} tensor.

Atom	x	y	z	U_{eq}	S.o.f.
La1	6045(1)	3854(1)	4881(1)	21(1)	1
Na1	8191(2)	2287(2)	4432(1)	30(1)	1
F1	4965(4)	1355(4)	3416(2)	68(1)	1
F2	6286(5)	883(4)	3764(2)	75(2)	1
F3	6215(4)	1810(4)	3072(2)	62(1)	1
O1	6834(3)	1729(4)	7283(2)	48(1)	1
O2	2221(4)	6654(4)	7329(2)	45(1)	1
O3	1261(3)	6156(3)	5407(2)	31(1)	1
O4	2416(3)	5325(3)	5056(2)	26(1)	1
O6	4315(3)	4363(3)	4835(2)	24(1)	1
O7	5984(3)	1090(3)	5422(2)	31(1)	1
O8	5158(3)	2396(3)	5108(2)	29(1)	1
O9	6110(3)	4946(3)	4062(2)	28(1)	1
O10	6235(3)	3887(3)	5907(2)	32(1)	1
O11	7288(3)	2510(3)	5258(2)	28(1)	1
O12	9031(4)	1001(4)	4809(2)	45(1)	1
O13	8504(5)	1903(5)	3548(2)	58(2)	1
O14	5189(3)	3004(3)	3975(2)	32(1)	1
O15	6697(3)	2712(3)	4144(2)	34(1)	1
N1	3883(4)	3534(4)	7057(2)	32(1)	1
N2	4948(4)	2259(4)	7060(2)	35(1)	1
N3	2446(3)	3380(4)	6505(2)	31(1)	1
N4	3591(3)	2129(4)	6485(2)	28(1)	1
N5	2753(4)	4735(5)	7059(3)	45(1)	1
N6	2178(3)	2028(3)	5955(2)	26(1)	1
N7	3479(3)	1368(3)	5082(2)	23(1)	1
N8	1372(3)	3559(3)	5096(2)	24(1)	1
N9	2458(3)	2681(4)	4344(2)	27(1)	1
O5	3379(3)	3413(3)	5282(2)	23(1)	1
C1	4120(4)	2646(4)	6870(2)	29(1)	1
C2	5499(5)	2662(5)	7540(3)	37(1)	1
C3	6516(5)	2681(6)	7426(3)	44(2)	1
C4	6304(5)	1383(6)	6803(3)	49(2)	1
C5	5284(5)	1310(5)	6901(3)	42(2)	1
C6	3040(5)	3848(5)	6868(3)	34(1)	1
C7	3464(6)	5446(6)	7304(3)	50(2)	1
C8	3000(6)	6180(6)	7641(4)	52(2)	1
C9	1546(6)	5941(7)	7173(4)	55(2)	1
C10	1934(6)	5220(6)	6790(4)	53(2)	1
C11	2760(4)	2532(4)	6331(2)	27(1)	1
C12	1196(4)	2323(5)	5860(3)	31(1)	1
C13	879(4)	2669(4)	5281(3)	29(1)	1
C14	1149(4)	4447(4)	5416(2)	28(1)	1
C15	1639(4)	5394(4)	5276(2)	26(1)	1
C16	1120(4)	3749(4)	4496(2)	29(1)	1
C17	1507(4)	2949(4)	4140(2)	30(1)	1
C18	3150(4)	3451(4)	4302(2)	27(1)	1
C19	3642(4)	3747(4)	4848(2)	22(1)	1
C20	2742(4)	1738(4)	4131(2)	29(1)	1
C21	3579(4)	1300(4)	4476(2)	28(1)	1
C22	4341(4)	983(4)	5393(2)	26(1)	1
C23	5236(4)	1528(4)	5298(2)	25(1)	1
C24	2621(4)	830(4)	5235(2)	27(1)	1
C25	2395(4)	1002(4)	5822(3)	29(1)	1
C26	5909(4)	2541(4)	3914(2)	29(1)	1
C27	5837(5)	1642(5)	3535(3)	39(1)	1
S1	1241(2)	6068(2)	3614(1)	48(1)	1

C28	756(6)	6414(7)	2940(4)	54(2)	1
F4	125(6)	5687(6)	2766(3)	55(2)	0.589(7)
F6	294(9)	7197(8)	2916(6)	68(3)	0.589(7)
F8	1353(7)	6324(9)	2549(4)	74(3)	0.589(7)
O17	564(7)	6086(7)	3949(4)	49(2)	0.589(7)
O19	2025(5)	6779(6)	3686(3)	40(2)	0.589(7)
O21	1667(7)	5093(7)	3515(4)	47(2)	0.589(7)
F5	−13(13)	6936(16)	2991(10)	78(5)	0.411(7)
F7	1465(11)	7101(11)	2776(6)	72(3)	0.411(7)
F9	846(12)	5790(12)	2649(6)	72(3)	0.411(7)
O16	349(12)	5386(15)	3672(9)	84(4)	0.411(7)
O18	1199(9)	6951(12)	3994(6)	57(3)	0.411(7)
O20	2061(9)	5669(12)	3614(5)	47(3)	0.411(7)
S2	4572(2)	5198(2)	1221(1)	68(1)	1
F10	3019(5)	4310(5)	1115(3)	84(2)	1
F11	3291(7)	5206(7)	1791(4)	119(3)	1
F12	3959(6)	3925(6)	1842(4)	108(3)	1
O24	4385(4)	5377(4)	694(2)	54(1)	1
O25	5438(7)	5042(7)	1474(4)	96(3)	1
O26	4246(17)	6046(17)	1604(10)	248(10)	1
C29	3800(10)	4440(11)	1430(6)	94(4)	1
O22	4665(3)	5211(3)	3299(2)	36(1)	1
O23	3097(4)	4282(6)	2979(3)	64(2)	1

Table 2. Bond lengths [Å] and angles [°].

La1–Na1	3.972(2)	N8–H8	0.9800
La1–O4i	2.463(4)	N8–C13	1.492(7)
La1–O6	2.566(4)	N8–C14	1.492(7)
La1–O6i	2.589(4)	N8–C16	1.507(7)
La1–O8	2.447(4)	N9–C17	1.456(7)
La1–O9	2.509(4)	N9–C18	1.453(8)
La1–O10	2.507(4)	N9–C20	1.457(7)
La1–O11	2.662(4)	O5–C19	1.246(7)
La1–O14	2.705(4)	C2–H2A	0.9700
La1–O15	2.617(4)	C2–H2B	0.9700
La1–C26	2.965(6)	C2–C3	1.506(10)
Na1–O3i	2.284(5)	C3–H3A	0.9700
Na1–O11	2.512(5)	C3–H3B	0.9700
Na1–O12	2.274(5)	C4–H4A	0.9700
Na1–O13	2.313(6)	C4–H4B	0.9700
Na1–O15	2.269(5)	C4–C5	1.505(11)
F1–C27	1.317(9)	C5–H5A	0.9700
F2–C27	1.316(9)	C5–H5B	0.9700
F3–C27	1.318(8)	C7–H7A	0.9700
O1–C3	1.427(9)	C7–H7B	0.9700
O1–C4	1.426(9)	C7–C8	1.489(11)
O2–C8	1.450(10)	C8–H8A	0.9700
O2–C9	1.400(11)	C8–H8B	0.9700
O3–Na1i	2.284(5)	C9–H9C	0.9700
O3–C15	1.226(7)	C9–H9D	0.9700
O4–La1i	2.463(4)	C9–C10	1.497(11)
O4–C15	1.281(7)	C10–H10C	0.9700
O6–La1i	2.589(4)	C10–H10D	0.9700
O6–C19	1.280(7)	C12–H12C	0.9700
O7–C23	1.241(7)	C12–H12D	0.9700
O8–C23	1.271(7)	C12–C13	1.527(8)
O9–H9A	0.8952	C13–H13C	0.9700
O9–H9B	0.8930	C13–H13D	0.9700
O10–H10A	0.8765	C14–H14A	0.9700
O10–H10B	0.8766	C14–H14B	0.9700
O12–H12A	0.8842	C14–C15	1.520(8)
O12–H12B	0.8830	C16–H16A	0.9700
O13–H13A	0.8751	C16–H16B	0.9700
O13–H13B	0.8733	C16–C17	1.530(8)
O14–C26	1.227(7)	C17–H17A	0.9700
O15–C26	1.240(7)	C17–H17B	0.9700
N1–C1	1.347(8)	C18–H18A	0.9700
N1–C6	1.326(8)	C18–H18B	0.9700
N2–C1	1.344(7)	C18–C19	1.513(7)
N2–C2	1.466(8)	C20–C21	1.527(8)
N2–C5	1.444(8)	C22–H22A	0.9700
N3–C6	1.339(8)	C22–H22B	0.9700
N3–C11	1.323(8)	C22–C23	1.516(8)
N4–C1	1.354(8)	C24–H24A	0.9700
N4–C11	1.334(7)	C24–H24B	0.9700
N5–C6	1.371(8)	C24–C25	1.519(8)
N5–C7	1.493(10)	C25–H25A	0.9700
N5–C10	1.453(9)	C25–H25B	0.9700
N6–C11	1.374(8)	C26–C27	1.535(8)
N6–C12	1.462(7)	S1–C28	1.801(8)
N6–C25	1.474(7)	S1–O17	1.322(10)
N7–H7	0.9800	S1–O19	1.481(9)
N7–C21	1.508(7)	S1–O21	1.489(9)
N7–C22	1.489(7)	S1–O16	1.596(18)
N7–C24	1.505(7)	S1–O18	1.526(15)

S1–O20	1.292(13)
C28–F4	1.383(12)
C28–F6	1.254(15)
C28–F8	1.344(14)
C28–F5	1.33(2)
C28–F7	1.461(17)
C28–F9	1.124(17)
S2–O24	1.319(6)
S2–O25	1.355(10)
S2–O26	1.58(2)
S2–C29	1.626(15)
F10–C29	1.314(15)
F11–C29	1.588(17)
F12–C29	1.235(15)
O22–H22C	0.8503
O22–H22D	0.8501
O23–H23A	0.8502
O23–H23B	0.8501

O4i–La1–Na1	63.43(9)
O4i–La1–O6i	75.83(12)
O4i–La1–O6	137.31(12)
O4i–La1–O9	71.75(13)
O4i–La1–O10	84.84(14)
O4i–La1–O11	73.91(13)
O4i–La1–O14	125.82(13)
O4i–La1–O15	86.73(14)
O4i–La1–C26	108.72(15)
O6–La1–Na1	154.09(9)
O6i–La1–Na1	138.99(9)
O6–La1–O6i	62.93(15)
O6–La1–O11	144.60(13)
O6i–La1–O11	133.94(12)
O6i–La1–O14	122.39(12)
O6–La1–O14	73.26(12)
O6i–La1–O15	146.48(14)
O6–La1–O15	122.01(13)
O6i–La1–C26	141.23(14)
O6–La1–C26	97.50(14)
O8–La1–Na1	93.19(10)
O8–La1–O4i	146.39(13)
O8–La1–O6i	125.50(13)
O8–La1–O6	73.23(13)
O8–La1–O9	135.98(13)
O8–La1–O10	78.43(14)
O8–La1–O11	72.99(13)
O8–La1–O14	68.43(14)
O8–La1–O15	84.13(15)
O8–La1–C26	72.12(16)
O9–La1–Na1	90.80(10)
O9–La1–O6	84.62(13)
O9–La1–O6i	71.41(13)
O9–La1–O11	128.04(13)
O9–La1–O14	68.93(13)
O9–La1–O15	76.04(14)
O9–La1–C26	73.77(15)
O10–La1–Na1	105.53(10)
O10–La1–O6i	73.79(13)
O10–La1–O6	93.47(13)
O10–La1–O9	141.74(14)
O10–La1–O11	69.66(13)
O10–La1–O14	146.56(14)
O10–La1–O15	133.59(13)
O10–La1–C26	143.89(15)
O11–La1–Na1	38.47(9)
O11–La1–O14	103.46(12)
O11–La1–C26	81.91(14)
O14–La1–Na1	81.29(9)
O14–La1–C26	24.45(14)
O15–La1–Na1	32.79(10)
O15–La1–O11	64.15(13)
O15–La1–O14	48.76(13)
O15–La1–C26	24.67(15)
C26–La1–Na1	56.87(12)
O3i–Na1–La1	73.47(12)
O3i–Na1–O11	86.63(17)
O3i–Na1–O13	106.3(2)
O11–Na1–La1	41.26(9)
O12–Na1–La1	134.07(16)
O12–Na1–O3i	118.9(2)
O12–Na1–O11	93.25(18)
O12–Na1–O13	93.9(2)
O13–Na1–La1	126.75(17)
O13–Na1–O11	159.7(2)
O15–Na1–La1	38.64(11)
O15–Na1–O3i	96.74(18)
O15–Na1–O11	71.60(15)
O15–Na1–O12	140.7(2)
O15–Na1–O13	91.1(2)
C4–O1–C3	110.2(5)
C9–O2–C8	108.4(6)
C15–O3–Na1i	132.8(4)
C15–O4–La1i	138.2(4)
La1–O6–La1i	117.07(14)
C19–O6–La1i	116.2(3)
C19–O6–La1	123.2(3)
C23–O8–La1	143.8(4)
La1–O9–H9A	111.9
La1–O9–H9B	111.2
H9A–O9–H9B	107.2
La1–O10–H10A	110.8
La1–O10–H10B	110.8
H10A–O10–H10B	108.3
Na1–O11–La1	100.27(14)
Na1–O12–H12A	111.4
Na1–O12–H12B	110.9
H12A–O12–H12B	107.5
Na1–O13–H13A	110.9
Na1–O13–H13B	110.2
H13A–O13–H13B	108.1
C26–O14–La1	89.7(3)
Na1–O15–La1	108.57(17)
C26–O15–La1	93.6(3)
C26–O15–Na1	152.9(4)
C6–N1–C1	114.8(5)
C1–N2–C2	122.0(5)
C1–N2–C5	124.1(5)
C5–N2–C2	112.6(5)
C11–N3–C6	114.6(5)
C11–N4–C1	114.6(5)
C6–N5–C7	119.7(6)
C6–N5–C10	119.9(6)

C10–N5–C7	112.5(6)
C11–N6–C12	119.3(5)
C11–N6–C25	119.7(5)
C12–N6–C25	116.4(5)
C21–N7–H7	108.2
C22–N7–H7	108.2
C22–N7–C21	109.8(4)
C22–N7–C24	111.0(4)
C24–N7–H7	108.2
C24–N7–C21	111.4(4)
C13–N8–H8	108.3
C13–N8–C14	111.7(4)
C13–N8–C16	110.9(4)
C14–N8–H8	108.3
C14–N8–C16	109.3(4)
C16–N8–H8	108.3
C17–N9–C20	112.4(4)
C18–N9–C17	114.7(5)
C18–N9–C20	113.5(5)
N1–C1–N4	124.1(5)
N2–C1–N1	118.3(6)
N2–C1–N4	117.6(5)
N2–C2–H2A	110.0
N2–C2–H2B	110.0
N2–C2–C3	108.6(6)
H2A–C2–H2B	108.4
C3–C2–H2A	110.0
C3–C2–H2B	110.0
O1–C3–C2	111.3(6)
O1–C3–H3A	109.4
O1–C3–H3B	109.4
C2–C3–H3A	109.4
C2–C3–H3B	109.4
H3A–C3–H3B	108.0
O1–C4–H4A	109.6
O1–C4–H4B	109.6
O1–C4–C5	110.1(6)
H4A–C4–H4B	108.2
C5–C4–H4A	109.6
C5–C4–H4B	109.6
N2–C5–C4	109.5(6)
N2–C5–H5A	109.8
N2–C5–H5B	109.8
C4–C5–H5A	109.8
C4–C5–H5B	109.8
H5A–C5–H5B	108.2
N1–C6–N3	125.8(6)
N1–C6–N5	117.1(6)
N3–C6–N5	117.1(6)
N5–C7–H7A	109.7
N5–C7–H7B	109.7
H7A–C7–H7B	108.2
C8–C7–N5	109.8(7)
C8–C7–H7A	109.7
C8–C7–H7B	109.7
O2–C8–C7	111.4(7)
O2–C8–H8A	109.4
O2–C8–H8B	109.4
C7–C8–H8A	109.4
C7–C8–H8B	109.4
H8A–C8–H8B	108.0
O2–C9–H9C	109.7
O2–C9–H9D	109.7
O2–C9–C10	110.0(7)
H9C–C9–H9D	108.2
C10–C9–H9C	109.7
C10–C9–H9D	109.7
N5–C10–C9	110.0(7)
N5–C10–H10C	109.7
N5–C10–H10D	109.7
C9–C10–H10C	109.7
C9–C10–H10D	109.7
H10C–C10–H10D	108.2
N3–C11–N4	126.0(6)
N3–C11–N6	116.7(5)
N4–C11–N6	117.4(5)
N6–C12–H12C	108.3
N6–C12–H12D	108.3
N6–C12–C13	115.9(5)
H12C–C12–H12D	107.4
C13–C12–H12C	108.3
C13–C12–H12D	108.3
N8–C13–C12	115.1(5)
N8–C13–H13C	108.5
N8–C13–H13D	108.5
C12–C13–H13C	108.5
C12–C13–H13D	108.5
H13C–C13–H13D	107.5
N8–C14–H14A	108.2
N8–C14–H14B	108.2
N8–C14–C15	116.4(5)
H14A–C14–H14B	107.3
C15–C14–H14A	108.2
C15–C14–H14B	108.2
O3–C15–O4	126.2(5)
O3–C15–C14	115.9(5)
O4–C15–C14	117.9(5)
N8–C16–H16A	109.3
N8–C16–H16B	109.3
N8–C16–C17	111.5(5)
H16A–C16–H16B	108.0
C17–C16–H16A	109.3
C17–C16–H16B	109.3
N9–C17–C16	110.9(5)
N9–C17–H17A	109.5
N9–C17–H17B	109.5
C16–C17–H17A	109.5
C16–C17–H17B	109.5
H17A–C17–H17B	108.0
N9–C18–H18A	108.9
N9–C18–H18B	108.9
N9–C18–C19	113.5(5)
H18A–C18–H18B	107.7
C19–C18–H18A	108.9
C19–C18–H18B	108.9
O6–C19–C18	116.5(5)
O5–C19–O6	122.9(5)
O5–C19–C18	120.5(5)
N9–C20–C21	112.1(5)
N7–C21–C20	112.6(5)
N7–C22–H22A	108.5
N7–C22–H22B	108.5

N7–C22–C23	115.0(4)	O19–S1–O21	106.6(5)
H22A–C22–H22B	107.5	O21–S1–C28	102.3(5)
C23–C22–H22A	108.5	O16–S1–C28	89.1(8)
C23–C22–H22B	108.5	O18–S1–C28	108.8(7)
O7–C23–O8	125.7(5)	O18–S1–O16	109.3(11)
O7–C23–C22	116.7(5)	O20–S1–C28	112.8(6)
O8–C23–C22	117.6(5)	O20–S1–O16	119.3(10)
N7–C24–H24A	108.8	O20–S1–O18	114.5(8)
N7–C24–H24B	108.8	F4–C28–S1	106.6(6)
N7–C24–C25	114.0(5)	F6–C28–S1	115.3(9)
H24A–C24–H24B	107.7	F6–C28–F4	105.4(9)
C25–C24–H24A	108.8	F6–C28–F8	114.2(11)
C25–C24–H24B	108.8	F8–C28–S1	114.4(7)
N6–C25–C24	115.0(5)	F8–C28–F4	98.8(9)
N6–C25–H25A	108.5	F5–C28–S1	108.2(13)
N6–C25–H25B	108.5	F5–C28–F7	106.5(13)
C24–C25–H25A	108.5	F7–C28–S1	101.4(8)
C24–C25–H25B	108.5	F9–C28–S1	109.2(10)
H25A–C25–H25B	107.5	F9–C28–F5	127.3(15)
O14–C26–La1	65.9(3)	F9–C28–F7	101.0(13)
O14–C26–O15	126.0(6)	O24–S2–O25	125.3(5)
O14–C26–C27	118.1(6)	O24–S2–O26	113.6(9)
O15–C26–La1	61.8(3)	O24–S2–C29	109.8(6)
O15–C26–C27	115.9(5)	O25–S2–O26	98.4(10)
C27–C26–La1	164.1(4)	O25–S2–C29	112.1(7)
F1–C27–F3	108.1(6)	O26–S2–C29	91.9(10)
F1–C27–C26	112.6(6)	F10–C29–S2	117.9(11)
F2–C27–F1	106.3(6)	F10–C29–F11	90.4(10)
F2–C27–F3	106.4(6)	F11–C29–S2	96.6(9)
F2–C27–C26	111.4(6)	F12–C29–S2	122.4(12)
F3–C27–C26	111.8(6)	F12–C29–F10	119.4(13)
O17–S1–C28	108.6(6)	F12–C29–F11	88.7(10)
O17–S1–O19	120.1(6)	H22C–O22–H22D	109.5
O17–S1–O21	116.8(6)	H23A–O23–H23B	109.5
O19–S1–C28	99.6(4)		

Symmetry transformations used to generate equivalent atoms:
(i) –x+1,–y+1,–z+1

Table 3. Anisotropic displacement parameters [$Å^2 \times 10^3$].

The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + \cdots + 2\,h\,k\,a^*\,b^*\,U^{12}]$.

Atom	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
La1	18(1)	18(1)	27(1)	−1(1)	−2(1)	5(1)
Na1	28(1)	25(1)	37(1)	−1(1)	2(1)	7(1)
F1	61(3)	74(3)	69(3)	−41(3)	8(2)	−17(2)
F2	113(4)	37(2)	71(3)	−10(2)	−8(3)	20(3)
F3	83(3)	70(3)	35(2)	−17(2)	18(2)	−13(3)
O1	33(2)	56(3)	53(3)	−3(2)	−14(2)	17(2)
O2	44(3)	38(2)	53(3)	−2(2)	5(2)	8(2)
O3	26(2)	26(2)	42(2)	−3(2)	2(2)	5(2)
O4	20(2)	24(2)	33(2)	−1(2)	−1(1)	6(1)
O6	20(2)	22(2)	30(2)	−2(1)	0(1)	5(1)
O7	25(2)	23(2)	43(2)	1(2)	−4(2)	7(1)
O8	24(2)	20(2)	40(2)	3(2)	−3(2)	4(1)
O9	29(2)	26(2)	29(2)	−2(2)	0(2)	1(2)
O10	29(2)	35(2)	31(2)	−2(2)	−3(2)	9(2)
O11	25(2)	27(2)	30(2)	2(2)	−4(1)	9(2)
O12	47(3)	38(2)	49(3)	9(2)	10(2)	18(2)
O13	67(4)	66(4)	45(3)	1(3)	16(3)	26(3)
O14	29(2)	31(2)	36(2)	−4(2)	−4(2)	6(2)
O15	29(2)	37(2)	34(2)	−8(2)	−4(2)	12(2)
N1	33(2)	31(2)	32(2)	−3(2)	−3(2)	8(2)
N2	31(2)	35(2)	36(2)	1(2)	−6(2)	7(2)
N3	28(2)	30(2)	34(2)	0(2)	1(2)	8(2)
N4	27(2)	26(2)	30(2)	1(2)	−1(2)	5(2)
N5	37(3)	46(3)	49(3)	−17(2)	−6(2)	14(2)
N6	23(2)	22(2)	31(2)	4(2)	1(2)	4(2)
N7	22(2)	17(2)	29(2)	−1(2)	0(2)	4(2)
N8	19(2)	21(2)	32(2)	0(2)	−2(2)	2(2)
N9	24(2)	26(2)	29(2)	−4(2)	−6(2)	5(2)
O5	22(2)	20(2)	27(2)	−2(1)	−2(1)	5(1)
C1	28(2)	28(2)	29(2)	2(2)	0(2)	5(2)
C2	37(3)	40(3)	33(3)	5(2)	−6(2)	5(2)
C3	37(3)	48(3)	44(3)	0(3)	−9(3)	8(3)
C4	38(3)	53(4)	53(4)	−4(3)	−9(3)	16(3)
C5	36(3)	38(3)	48(4)	−4(3)	−13(3)	15(2)
C6	32(3)	36(3)	34(3)	−3(2)	0(2)	8(2)
C7	45(3)	49(4)	54(4)	−13(3)	−2(3)	7(3)
C8	52(4)	50(4)	54(4)	−10(3)	−2(3)	11(3)
C9	46(4)	50(4)	68(5)	−14(4)	0(3)	13(3)
C10	45(3)	51(4)	60(4)	−13(3)	−6(3)	15(3)
C11	26(2)	27(2)	27(2)	6(2)	3(2)	3(2)
C12	25(2)	29(3)	40(3)	4(2)	4(2)	4(2)
C13	20(2)	24(2)	40(3)	−2(2)	−5(2)	0(2)
C14	22(2)	25(2)	36(3)	−3(2)	−1(2)	5(2)
C15	21(2)	26(2)	29(2)	−3(2)	−4(2)	4(2)
C16	22(2)	30(3)	34(2)	−3(2)	−8(2)	7(2)
C17	25(2)	30(3)	34(3)	−5(2)	−6(2)	7(2)
C18	25(2)	27(2)	27(2)	−1(2)	−4(2)	4(2)
C19	18(2)	20(2)	26(2)	−2(2)	0(2)	6(2)
C20	30(3)	26(2)	30(2)	−7(2)	−6(2)	8(2)
C21	28(3)	28(3)	27(2)	−1(2)	−2(2)	6(2)
C22	27(2)	20(2)	30(2)	4(2)	−3(2)	4(2)
C23	26(2)	18(2)	31(2)	−2(2)	−3(2)	6(2)
C24	25(2)	20(2)	35(3)	−1(2)	1(2)	1(2)
C25	26(3)	24(2)	37(3)	4(2)	3(2)	2(2)
C26	32(2)	26(2)	29(2)	−1(2)	−1(2)	5(2)
C27	50(3)	32(3)	35(3)	−8(2)	7(2)	1(2)
S1	50(1)	50(1)	41(1)	8(1)	−9(1)	1(1)

C28	52(3)	56(3)	50(3)	8(2)	−16(2)	−4(3)
F4	57(4)	48(4)	55(4)	−1(3)	−19(3)	−10(3)
F6	84(7)	45(4)	68(6)	16(4)	−33(5)	2(4)
F8	66(5)	109(8)	43(4)	10(4)	−12(3)	−22(4)
O17	49(4)	50(5)	49(4)	1(4)	6(3)	7(4)
O19	31(3)	48(4)	38(4)	4(3)	−8(3)	8(3)
O21	50(5)	46(4)	44(4)	4(3)	2(4)	10(3)
F5	58(6)	86(10)	87(10)	−8(8)	−13(6)	10(6)
F7	73(6)	62(6)	81(8)	20(5)	9(6)	2(5)
F9	82(9)	67(6)	65(6)	−6(5)	−4(6)	−1(5)
O16	50(6)	81(8)	119(11)	41(7)	−6(6)	−6(6)
O18	33(6)	85(7)	52(6)	−15(5)	−1(5)	12(5)
O20	42(4)	65(8)	33(5)	−6(5)	−10(4)	14(5)
O22	36(2)	37(2)	35(2)	−2(2)	−7(2)	2(2)
O23	38(3)	103(5)	50(3)	−29(3)	−1(2)	−17(3)

Table 4. Hydrogen coordinates [$\times 10^4$] and isotropic displacement parameters [$Å^2 \times 10^3$].

Atom	x	y	z	U_{eq}	S.o.f.
H9A	6521	4728	3834	42	1
H9B	6282	5556	4160	42	1
H10A	5907	4366	6034	48	1
H10B	6824	3970	6026	48	1
H12A	9248	1128	5150	67	1
H12B	9516	870	4622	67	1
H13A	9043	1599	3545	88	1
H13B	8529	2437	3353	88	1
H7	3412	2062	5176	27	1
H8	2049	3441	5151	29	1
H2A	5419	2258	7859	44	1
H2B	5287	3321	7614	44	1
H3A	6597	3134	7128	52	1
H3B	6892	2915	7748	52	1
H4A	6535	743	6704	58	1
H4B	6379	1830	6502	58	1
H5A	4924	1089	6571	50	1
H5B	5203	835	7188	50	1
H7A	3943	5097	7531	60	1
H7B	3763	5779	7016	60	1
H8A	2774	5855	7957	63	1
H8B	3454	6672	7773	63	1
H9C	993	6253	6994	66	1
H9D	1365	5600	7495	66	1
H10C	1460	4735	6678	63	1
H10D	2106	5559	6466	63	1
H12C	1081	2848	6113	37	1
H12D	807	1771	5947	37	1
H13C	972	2137	5029	35	1
H13D	212	2807	5262	35	1
H14A	1306	4309	5801	33	1
H14B	477	4557	5366	33	1
H16A	1370	4381	4399	35	1
H16B	443	3775	4426	35	1
H17A	1107	2375	4139	36	1
H17B	1506	3184	3767	36	1
H18A	2843	4023	4133	32	1
H18B	3615	3228	4065	32	1
H22A	4417	299	5295	31	1
H22B	4252	1006	5780	31	1
H24A	2712	132	5182	32	1
H24B	2088	1034	4991	32	1
H25A	1864	595	5894	35	1
H25B	2926	788	6065	35	1
H22C	4303	4734	3206	54	1
H22D	4984	5073	3598	54	1
H23A	2683	4632	3115	96	1
H23B	2862	4017	2685	96	1

Table 5. Torsion angles [°].

La1i–O4–C15–O3	37.3(9)	C6–N5–C10–C9	158.7(7)
La1i–O4–C15–C14	–139.6(4)	C7–N5–C6–N1	20.3(10)
La1–O6–C19–O5	84.0(5)	C7–N5–C6–N3	–159.3(7)
La1i–O6–C19–O5	–73.9(5)	C7–N5–C10–C9	–52.2(10)
La1–O6–C19–C18	–97.4(5)	C8–O2–C9–C10	–64.9(9)
La1i–O6–C19–C18	104.6(4)	C9–O2–C8–C7	62.9(9)
La1–O8–C23–O7	–4.6(11)	C10–N5–C6–N1	167.2(7)
La1–O8–C23–C22	174.1(4)	C10–N5–C6–N3	–12.4(10)
La1–O14–C26–O15	–14.7(7)	C10–N5–C7–C8	49.6(9)
La1–O14–C26–C27	162.8(5)	C11–N3–C6–N1	0.0(10)
La1–O15–C26–O14	15.3(7)	C11–N3–C6–N5	179.5(6)
La1–O15–C26–C27	–162.3(5)	C11–N4–C1–N1	–5.2(9)
La1–C26–C27–F1	86.3(17)	C11–N4–C1–N2	177.2(5)
La1–C26–C27–F2	–32.9(19)	C11–N6–C12–C13	118.2(6)
La1–C26–C27–F3	–151.8(13)	C11–N6–C25–C24	–115.4(6)
Na1i–O3–C15–O4	–19.5(9)	C12–N6–C11–N3	–13.4(8)
Na1i–O3–C15–C14	157.4(4)	C12–N6–C11–N4	167.1(5)
Na1–O15–C26–La1	145.4(10)	C12–N6–C25–C24	88.9(6)
Na1–O15–C26–O14	160.7(6)	C13–N8–C14–C15	178.0(4)
Na1–O15–C26–C27	–16.9(12)	C13–N8–C16–C17	–67.6(6)
O1–C4–C5–N2	58.1(8)	C14–N8–C13–C12	–66.5(6)
O2–C9–C10–N5	60.4(10)	C14–N8–C16–C17	168.8(5)
O14–C26–C27–F1	–14.0(9)	C16–N8–C13–C12	171.3(5)
O14–C26–C27–F2	–133.3(7)	C16–N8–C14–C15	–58.9(6)
O14–C26–C27–F3	107.9(7)	C17–N9–C18–C19	120.3(5)
O15–C26–C27–F1	163.8(6)	C17–N9–C20–C21	–161.2(5)
O15–C26–C27–F2	44.5(8)	C18–N9–C17–C16	–66.4(7)
O15–C26–C27–F3	–74.3(8)	C18–N9–C20–C21	66.7(6)
N2–C2–C3–O1	–56.1(7)	C20–N9–C17–C16	162.1(5)
N5–C7–C8–O2	–54.1(10)	C20–N9–C18–C19	–108.7(5)
N6–C12–C13–N8	–61.7(7)	C21–N7–C22–C23	60.7(6)
N7–C22–C23–O7	–160.8(5)	C21–N7–C24–C25	–170.9(4)
N7–C22–C23–O8	20.4(7)	C22–N7–C21–C20	–176.7(5)
N7–C24–C25–N6	62.3(6)	C22–N7–C24–C25	66.4(6)
N8–C14–C15–O3	158.5(5)	C24–N7–C21–C20	60.0(6)
N8–C14–C15–O4	–24.3(7)	C24–N7–C22–C23	–175.6(5)
N8–C16–C17–N9	–41.8(7)	C25–N6–C11–N3	–168.4(5)
N9–C18–C19–O6	174.3(4)	C25–N6–C11–N4	12.1(8)
N9–C18–C19–O5	–7.1(7)	C25–N6–C12–C13	–86.0(6)
N9–C20–C21–N7	45.0(7)	O17–S1–C28–F4	–65.2(9)
C1–N1–C6–N3	–2.4(10)	O17–S1–C28–F6	51.4(10)
C1–N1–C6–N5	178.1(6)	O17–S1–C28–F8	–173.3(9)
C1–N2–C2–C3	–138.0(6)	O19–S1–C28–F4	168.4(7)
C1–N2–C5–C4	136.9(7)	O19–S1–C28–F6	–75.1(10)
C1–N4–C11–N3	2.5(9)	O19–S1–C28–F8	60.3(9)
C1–N4–C11–N6	–178.0(5)	O21–S1–C28–F4	58.9(8)
C2–N2–C1–N1	13.2(9)	O21–S1–C28–F6	175.5(9)
C2–N2–C1–N4	–169.0(6)	O21–S1–C28–F8	–49.2(10)
C2–N2–C5–C4	–56.1(8)	O16–S1–C28–F5	67.2(14)
C3–O1–C4–C5	–60.6(8)	O16–S1–C28–F7	179.0(12)
C4–O1–C3–C2	60.5(8)	O16–S1–C28–F9	–75.0(15)
C5–N2–C1–N1	179.0(6)	O18–S1–C28–F5	–43.0(13)
C5–N2–C1–N4	–3.2(9)	O18–S1–C28–F7	68.8(10)
C5–N2–C2–C3	54.7(8)	O18–S1–C28–F9	174.8(13)
C6–N1–C1–N2	–177.2(6)	O20–S1–C28–F5	–171.2(13)
C6–N1–C1–N4	5.2(9)	O20–S1–C28–F7	–59.4(12)
C6–N3–C11–N4	0.0(9)	O20–S1–C28–F9	46.6(15)
C6–N3–C11–N6	–179.5(5)	O24–S2–C29–F10	–16.2(13)
C6–N5–C7–C8	–161.3(7)	O24–S2–C29–F11	–110.2(7)

O24–S2–C29–F12	157.2(11)	O26–S2–C29–F10	99.8(14)
O25–S2–C29–F10	–160.2(10)	O26–S2–C29–F11	5.8(12)
O25–S2–C29–F11	105.7(8)	O26–S2–C29–F12	–86.8(15)
O25–S2–C29–F12	13.1(15)		

Symmetry transformations used to generate equivalent atoms:
(i) –x+1,–y+1,–z+1

Table 1. Crystal data and structure refinement details.

Identification code	**2014ncs0044 (MM 24)**	
Empirical formula	$C_{86}H_{138}F_6La_6N_{24}NaO_{60}S_2$	
Formula weight	3502.77	
Temperature	100(2) K	
Wavelength	0.71075 Å	
Crystal system	Trigonal	
Space group	*R*–3*c*:*H*	
Unit cell dimensions	*a* = 16.667(2) Å	α = 90°
	b = 16.667(2) Å	β = 90°
	c = 98.668(12) Å	γ = 120°
Volume	23737(6) Å^3	
Z	6	
Density (calculated)	1.470 Mg / m^3	
Absorption coefficient	1.703 mm^{-1}	
F(000)	10446	
Crystal	Hexagonal prism; Colorless	
Crystal size	0.060 × 0.060 × 0.020 mm^3	
θ range for data collection	2.941 – 27.482°	
Index ranges	$-20 \le h \le 21$, $-18 \le k \le 21$, $-105 \le l \le 127$	
Reflections collected	51164	
Independent reflections	6060 [R_{int} = 0.0386]	
Completeness to θ = 25.242°	99.8 %	
Absorption correction	Semi–empirical from equivalents	
Max. and min. transmission	1.000 and 0.842	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6060 / 0 / 271	
Goodness-of-fit on F^2	1.080	
Final *R* indices [$F^2 > 2\sigma(F^2)$]	*R1* = 0.0647, *wR2* = 0.1617	
R indices (all data)	*R1* = 0.0656, *wR2* = 0.1623	
Extinction coefficient	n/a	
Largest diff. peak and hole	3.773 and –3.581 e Å^{-3}	

Diffractometer: Rigaku AFC12 goniometer equipped with an enhanced sensitivity (HG) Saturn724+ detector mounted at the window of an FR-E+ SuperBright molybdenum rotating anode generator with HF Varimax optics (100m focus). **Cell determination and data collection:** CrystalClear-SM Expert 3.0 r27 (Rigaku, 2012). **Data reduction, cell refinement and absorption correction:** CrystalClear-SM Expert 3.1 r27 (Rigaku, 2012). **Structure solution:** SUPERFLIP (Palatinus, L. & Chapuis, G. (2007). J. Appl. Cryst. 40, 786-790).. **Structure refinement:** SHELXL-2014 (Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122).

Special details:
The CF3CO3$^-$ is disordered over symmetry element (3-fold axis), however it was not modelled but all atoms were left as isotropic only. Modelling attempt has been made, but model was not satisfactory.
The H5N proton has been found and refined directly from Fourier difference map.

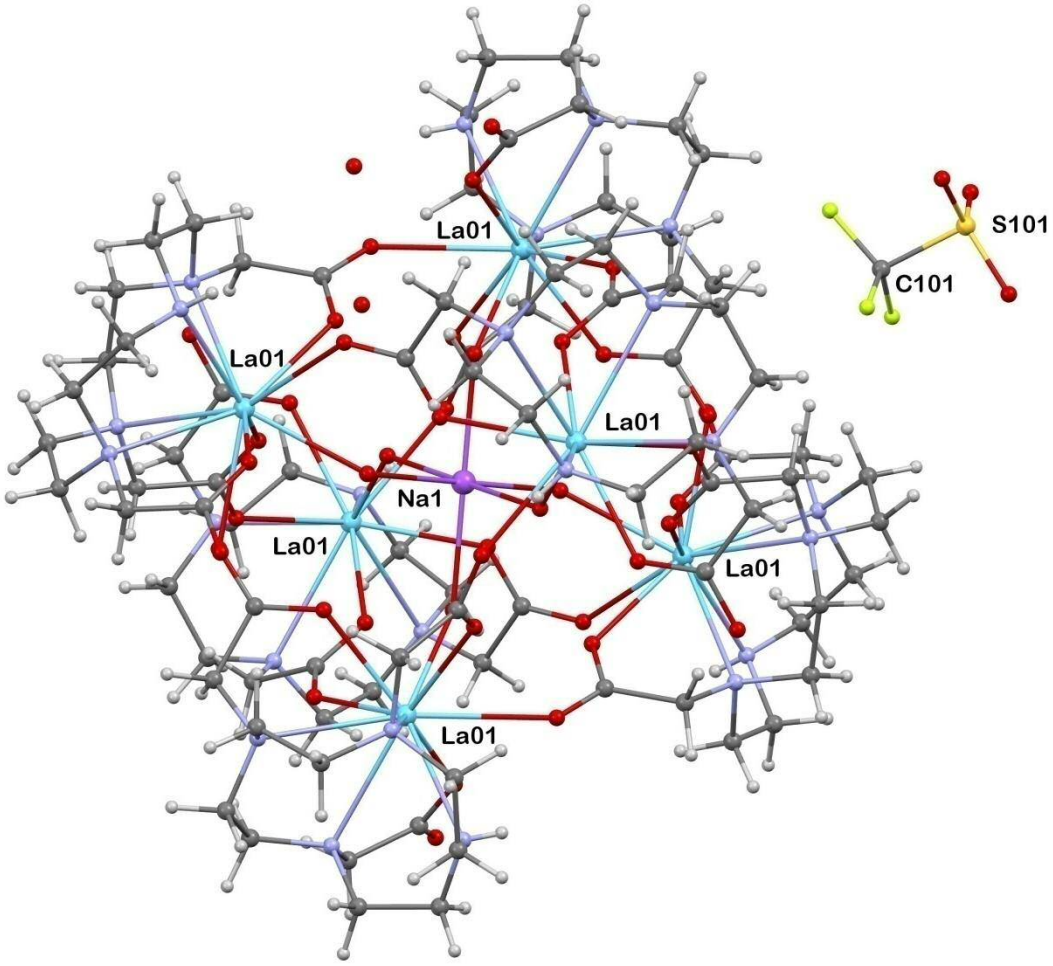

Figure 1. Molecular structure of 2014ncs0044 (MM 24) – grown fragment

Table 1. Atomic coordinates [$\times 10^4$], equivalent isotropic displacement parameters [$Å^2 \times 10^3$] and site occupancy factors. U_{eq} is defined as one third of the trace of the orthogonalized U^{ij} tensor.

Atom	x	y	z	U_{eq}	S.o.f.
C1	3451(5)	3420(5)	3519(1)	38(1)	1
C2	3952(4)	3339(4)	3396(1)	31(1)	1
C3	2138(4)	1872(5)	3534(1)	42(2)	1
C4	1677(4)	928(6)	3598(1)	46(2)	1
C5	2809(5)	2795(5)	3739(1)	41(1)	1
C6	3680(5)	3298(5)	3824(1)	41(1)	1
C7	3658(5)	2111(5)	3968(1)	40(2)	1
C8	3794(5)	1296(5)	3970(1)	44(2)	1
C9	5106(4)	3368(5)	3883(1)	35(1)	1
C10	5661(4)	3840(4)	3755(1)	30(1)	1
C11	3772(5)	36(5)	3851(1)	46(2)	1
C12	4026(6)	−201(5)	3714(1)	49(2)	1
C13	2421(5)	219(5)	3842(1)	46(2)	1
C14	2026(5)	−196(5)	3706(1)	43(2)	1
N1	3013(4)	2568(4)	3602(1)	36(1)	1
N2	4124(4)	2732(4)	3851(1)	33(1)	1
N3	3444(4)	710(4)	3846(1)	38(1)	1
N5	2311(4)	543(4)	3603(1)	40(1)	1
O1	4161(3)	3912(3)	3302(1)	37(1)	1
O2	4166(3)	2707(3)	3399(1)	32(1)	1
O3	6433(3)	4556(3)	3767(1)	33(1)	1
O4	5291(3)	3455(3)	3643(1)	29(1)	1
O5	4295(6)	−780(5)	3713(1)	81(2)	1
O6	4008(3)	226(3)	3611(1)	41(1)	1
O7	5539(3)	2074(3)	3461(1)	26(1)	1
O102	550(10)	1719(13)	3324(1)	190(7)	1
O103	2017(8)	3050(10)	3224(1)	135(4)	1
La01	4151(1)	1774(1)	3605(1)	26(1)	1
Na1	6667	3333	3333	23(1)	1
S101	6667	3333	4384(1)	90(1)	1
F101	5793(7)	2547(7)	4169(1)	131(3)	1
O101	5839(5)	3455(5)	4401(1)	80(2)	1
C101	6667	3333	4207(5)	211(19)	1

Table 2. Bond lengths [Å] and angles [°].

C1–N1	1.482(9)	Na1–O7iii	2.363(4)
C1–C2	1.516(8)	Na1–O7ii	2.363(4)
C1–H1A	0.9900	Na1–O7i	2.363(4)
C1–H1B	0.9900	Na1–O7v	2.363(4)
C2–O1	1.247(7)	Na1–O7iv	2.363(4)
C2–O2	1.269(7)	S101–O101iii	1.501(8)
C3–N1	1.494(9)	S101–O101ii	1.501(8)
C3–C4	1.503(10)	S101–O101	1.501(8)
C3–H3A	0.9900	S101–C101	1.74(5)
C3–H3B	0.9900	F101–C101	1.439(15)
C4–N5	1.485(10)	C101–F101iii	1.439(15)
C4–H4A	0.9900	C101–F101ii	1.439(15)
C4–H4B	0.9900		
C5–N1	1.482(8)	N1–C1–C2	112.9(5)
C5–C6	1.515(10)	N1–C1–H1A	109.0
C5–H5A	0.9900	C2–C1–H1A	109.0
C5–H5B	0.9900	N1–C1–H1B	109.0
C6–N2	1.487(9)	C2–C1–H1B	109.0
C6–H6A	0.9900	H1A–C1–H1B	107.8
C6–H6B	0.9900	O1–C2–O2	124.6(5)
C7–N2	1.482(7)	O1–C2–C1	118.5(6)
C7–C8	1.486(10)	O2–C2–C1	116.9(5)
C7–H7A	0.9900	N1–C3–C4	114.3(6)
C7–H7B	0.9900	N1–C3–H3A	108.7
C8–N3	1.487(9)	C4–C3–H3A	108.7
C8–H8A	0.9900	N1–C3–H3B	108.7
C8–H8B	0.9900	C4–C3–H3B	108.7
C9–N2	1.471(8)	H3A–C3–H3B	107.6
C9–C10	1.527(7)	N5–C4–C3	111.2(5)
C9–H9A	0.9900	N5–C4–H4A	109.4
C9–H9B	0.9900	C3–C4–H4A	109.4
C10–O3	1.249(7)	N5–C4–H4B	109.4
C10–O4	1.272(7)	C3–C4–H4B	109.4
C11–N3	1.476(9)	H4A–C4–H4B	108.0
C11–C12	1.527(10)	N1–C5–C6	111.3(5)
C11–H11A	0.9900	N1–C5–H5A	109.4
C11–H11B	0.9900	C6–C5–H5A	109.4
C12–O5	1.251(10)	N1–C5–H5B	109.4
C12–O6	1.251(9)	C6–C5–H5B	109.4
C13–N3	1.477(9)	H5A–C5–H5B	108.0
C13–C14	1.502(10)	N2–C6–C5	113.2(6)
C13–H13A	0.9900	N2–C6–H6A	108.9
C13–H13B	0.9900	C5–C6–H6A	108.9
C14–N5	1.485(9)	N2–C6–H6B	108.9
C14–H14A	0.9900	C5–C6–H6B	108.9
C14–H14B	0.9900	H6A–C6–H6B	107.8
N1–La01	2.804(5)	N2–C7–C8	112.5(5)
N2–La01	2.917(5)	N2–C7–H7A	109.1
N3–La01	2.845(5)	C8–C7–H7A	109.1
N5–La01	2.707(6)	N2–C7–H7B	109.1
N5–H5N	0.72(10)	C8–C7–H7B	109.1
O1–La01i	2.749(4)	H7A–C7–H7B	107.8
O2–La01	2.555(4)	C7–C8–N3	113.6(6)
O3–La01ii	2.617(4)	C7–C8–H8A	108.8
O4–La01	2.503(4)	N3–C8–H8A	108.8
O6–La01	2.471(5)	C7–C8–H8B	108.8
O7–Na1	2.363(4)	N3–C8–H8B	108.8
O7–La01	2.544(4)	H8A–C8–H8B	107.7
La01–O3iii	2.617(4)	N2–C9–C10	111.6(5)
La01–O1iv	2.749(4)	N2–C9–H9A	109.3

C10–C9–H9A	109.3
N2–C9–H9B	109.3
C10–C9–H9B	109.3
H9A–C9–H9B	108.0
O3–C10–O4	125.1(5)
O3–C10–C9	118.8(5)
O4–C10–C9	116.1(5)
N3–C11–C12	115.1(6)
N3–C11–H11A	108.5
C12–C11–H11A	108.5
N3–C11–H11B	108.5
C12–C11–H11B	108.5
H11A–C11–H11B	107.5
O5–C12–O6	123.7(8)
O5–C12–C11	117.1(7)
O6–C12–C11	119.0(7)
N3–C13–C14	113.6(5)
N3–C13–H13A	108.9
C14–C13–H13A	108.9
N3–C13–H13B	108.9
C14–C13–H13B	108.9
H13A–C13–H13B	107.7
N5–C14–C13	110.4(6)
N5–C14–H14A	109.6
C13–C14–H14A	109.6
N5–C14–H14B	109.6
C13–C14–H14B	109.6
H14A–C14–H14B	108.1
C1–N1–C5	109.1(5)
C1–N1–C3	108.3(5)
C5–N1–C3	110.4(5)
C1–N1–La01	109.4(3)
C5–N1–La01	113.9(4)
C3–N1–La01	105.5(4)
C9–N2–C7	110.3(5)
C9–N2–C6	108.0(5)
C7–N2–C6	109.5(5)
C9–N2–La01	104.6(3)
C7–N2–La01	114.2(4)
C6–N2–La01	110.0(3)
C11–N3–C13	110.1(6)
C11–N3–C8	107.5(6)
C13–N3–C8	110.6(5)
C11–N3–La01	107.8(4)
C13–N3–La01	109.0(4)
C8–N3–La01	111.8(4)
C4–N5–C14	112.0(5)
C4–N5–La01	117.0(4)
C14–N5–La01	113.1(4)
C4–N5–H5N	97(8)
C14–N5–H5N	114(9)
La01–N5–H5N	102(9)
C2–O1–La01i	136.4(4)
C2–O2–La01	127.0(4)
C10–O3–La01ii	136.3(4)
C10–O4–La01	126.8(4)
C12–O6–La01	126.5(5)
Na1–O7–La01	135.60(17)
O6–La01–O4	142.05(15)
O6–La01–O7	79.69(14)
O4–La01–O7	75.89(12)
O6–La01–O2	128.24(14)
O4–La01–O2	70.51(13)
O7–La01–O2	72.73(12)
O6–La01–O3iii	72.65(15)
O4–La01–O3iii	72.40(13)
O7–La01–O3iii	71.95(12)
O2–La01–O3iii	133.57(14)
O6–La01–N5	74.16(18)
O4–La01–N5	141.98(17)
O7–La01–N5	135.86(16)
O2–La01–N5	96.61(16)
O3iii–La01–N5	129.79(16)
O6–La01–O1iv	66.02(15)
O4–La01–O1iv	128.42(13)
O7–La01–O1iv	69.38(12)
O2–La01–O1iv	63.54(14)
O3iii–La01–O1iv	127.02(13)
N5–La01–O1iv	67.68(16)
O6–La01–N1	139.30(17)
O4–La01–N1	77.74(15)
O7–La01–N1	131.67(14)
O2–La01–N1	60.50(14)
O3iii–La01–N1	134.83(15)
N5–La01–N1	65.15(18)
O1iv–La01–N1	98.08(15)
O6–La01–N3	62.42(15)
O4–La01–N3	114.83(14)
O7–La01–N3	129.96(15)
O2–La01–N3	157.00(15)
O3iii–La01–N3	66.55(15)
N5–La01–N3	65.00(17)
O1iv–La01–N3	116.66(15)
N1–La01–N3	97.82(16)
O6–La01–N2	122.26(15)
O4–La01–N2	58.37(13)
O7–La01–N2	128.26(14)
O2–La01–N2	109.19(14)
O3iii–La01–N2	72.21(14)
N5–La01–N2	95.85(17)
O1iv–La01–N2	159.85(14)
N1–La01–N2	63.35(15)
N3–La01–N2	62.05(15)
O7iii–Na1–O7ii	94.14(12)
O7iii–Na1–O7	94.14(12)
O7ii–Na1–O7	94.14(12)
O7iii–Na1–O7i	180.0
O7ii–Na1–O7i	85.86(12)
O7–Na1–O7i	85.86(12)
O7iii–Na1–O7v	85.86(12)
O7ii–Na1–O7v	85.86(12)
O7–Na1–O7v	180.0
O7i–Na1–O7v	94.13(12)
O7iii–Na1–O7iv	85.86(12)
O7ii–Na1–O7iv	180.0
O7–Na1–O7iv	85.86(12)
O7i–Na1–O7iv	94.13(12)
O7v–Na1–O7iv	94.13(12)
O101iii–S101–O101ii	118.83(14)
O101iii–S101–O101	118.83(14)
O101ii–S101–O101	118.83(14)
O101iii–S101–C101	96.3(4)

O101ii–S101–C101	96.3(4)	F101iii–C101–F101ii	113.3(17)
O101–S101–C101	96.2(4)	F101–C101–S101	105(2)
F101–C101–F101iii	113.3(17)	F101iii–C101–S101	105(2)
F101–C101–F101ii	113.3(17)	F101ii–C101–S101	105(2)

Symmetry transformations used to generate equivalent atoms:
(i) y+1/3,–x+y+2/3,–z+2/3 (ii) –x+y+1,–x+1,z (iii) –y+1,x–y,z
(iv) x–y+1/3,x–1/3,–z+2/3 (v) –x+4/3,–y+2/3,–z+2/3

Table 3. Anisotropic displacement parameters [$Å^2 \times 10^3$].
The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + \cdots + 2\,h\,k\,a^*\,b^*\,U^{12}]$.

Atom	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C1	40(3)	50(4)	32(3)	2(3)	4(3)	30(3)
C2	28(3)	39(3)	29(3)	1(2)	–2(2)	18(2)
C3	31(3)	61(4)	35(3)	0(3)	1(2)	23(3)
C4	28(3)	58(4)	41(3)	1(3)	6(3)	13(3)
C5	35(3)	53(4)	36(3)	–2(3)	5(3)	25(3)
C6	43(4)	48(4)	33(3)	–2(3)	8(3)	24(3)
C7	39(3)	52(4)	22(3)	3(2)	10(2)	17(3)
C8	43(4)	55(4)	22(3)	10(3)	9(2)	17(3)
C9	37(3)	42(3)	21(2)	–2(2)	3(2)	17(3)
C10	35(3)	35(3)	21(2)	–1(2)	4(2)	19(3)
C11	50(4)	46(4)	39(3)	16(3)	10(3)	21(3)
C12	55(4)	45(4)	48(4)	12(3)	10(3)	27(4)
C13	36(3)	47(4)	40(3)	10(3)	12(3)	9(3)
C14	33(3)	40(4)	43(3)	4(3)	11(3)	8(3)
N1	29(2)	53(3)	31(2)	3(2)	6(2)	23(2)
N2	35(3)	40(3)	20(2)	2(2)	7(2)	15(2)
N3	41(3)	40(3)	29(2)	6(2)	11(2)	17(2)
N5	34(3)	43(3)	34(3)	0(2)	5(2)	13(2)
O1	39(2)	45(3)	33(2)	8(2)	4(2)	26(2)
O2	34(2)	37(2)	26(2)	1(2)	1(2)	21(2)
O3	34(2)	33(2)	26(2)	–5(2)	0(2)	14(2)
O4	29(2)	31(2)	22(2)	1(2)	4(2)	12(2)
O5	128(7)	81(5)	67(4)	21(3)	20(4)	77(5)
O6	49(3)	36(2)	35(2)	7(2)	10(2)	20(2)
O7	27(2)	30(2)	20(2)	2(1)	1(1)	14(2)
O102	138(11)	275(19)	153(10)	38(12)	–32(9)	99(12)
O103	115(8)	197(12)	111(7)	49(8)	15(6)	91(8)
La01	24(1)	30(1)	21(1)	2(1)	3(1)	12(1)
Na1	24(1)	24(1)	20(2)	0	0	12(1)

Table 4. Hydrogen coordinates [$\times 10^4$] and isotropic displacement parameters [$Å^2 \times 10^3$].

Atom	x	y	z	U_{eq}	S.o.f.
H1A	3897	3946	3575	45	1
H1B	2968	3559	3488	45	1
H3A	2278	1813	3438	51	1
H3B	1697	2107	3535	51	1
H4A	1485	971	3692	56	1
H4B	1115	505	3546	56	1
H5A	2360	2216	3785	49	1
H5B	2522	3188	3729	49	1
H6A	4129	3871	3776	49	1
H6B	3525	3481	3911	49	1
H7A	2986	1894	3964	48	1
H7B	3903	2465	4053	48	1
H8A	3473	911	4050	52	1
H8B	4463	1515	3980	52	1
H9A	5371	3019	3927	41	1
H9B	5157	3846	3947	41	1
H11A	3283	−543	3893	55	1
H11B	4323	286	3911	55	1
H13A	2209	658	3867	55	1
H13B	2176	−281	3911	55	1
H14A	2246	−625	3679	52	1
H14B	1341	−555	3712	52	1
H5N	2240(70)	380(70)	3534(10)	70(30)	1

Table 1. Crystal data and structure refinement details.

Identification code	**2015ncs0604**	
Empirical formula	$C_{25}H_{52}Cl_2LuN_9O_{20}$	
Formula weight	1044.62	
Temperature	100.15 K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	*P*–1	
Unit cell dimensions	*a* = 10.4652(3) Å	α = 101.275(2)°
	b = 13.4544(3) Å	β = 100.655(2)°
	c = 16.7831(4) Å	γ= 101.224(2)°
Volume	2211.27(10) Å^3	
Z	2	
Density (calculated)	1.569 Mg / m^3	
Absorption coefficient	2.433 mm^{-1}	
F(000)	1060	
Crystal	Fragment; colourless	
Crystal size	0.08 × 0.06 × 0.05 mm^3	
θ range for data collection	3.129 – 27.484°	
Index ranges	$-14 \le h \le 13$, $-16 \le k \le 18$, $-23 \le l \le 21$	
Reflections collected	30303	
Independent reflections	10087 [R_{int} = 0.0432]	
Completeness to θ = 26.000°	99.8 %	
Absorption correction	Semi–empirical from equivalents	
Max. and min. transmission	1.00000 and 0.62382	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	10087 / 3 / 522	
Goodness-of-fit on F^2	1.061	
Final *R* indices [$F^2 > 2\sigma(F^2)$]	*R1* = 0.0504, *wR2* = 0.1368	
R indices (all data)	*R1* = 0.0566, *wR2* = 0.1423	
Extinction coefficient	n/a	
Largest diff. peak and hole	3.123 and –1.894 e Å^{-3}	

Diffractometer: Rigaku AFC12 goniometer equipped with an enhanced sensitivity (HG) Saturn724+ detector mounted at the window of an FR-E+ SuperBright molybdenum rotating anode generator with HF Varimax optics (100m focus). **Cell determination and data collection:** CrystalClear-SM Expe. rt 3.1 b27 (Rigaku, 2012). **Data reduction, cell refinement and absorption correction:** CrysAlisPro, Agilent Technologies,, Version 1.171.37.35. **Structure solution:** SUPERFLIP (Palatinus, L. & Chapuis, G. (2007). J. Appl. Cryst. 40, 786-790). **Structure refinement:** SHELXL-2013 (Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122).

Special details:
One Cl- atom is fully occupied, whereas remaining two are refined as 50% occupied each (all together 2x Cl-). The free carboxylate group seems to be unprotonated COOO-. Both C=O bonds are similar which suggest charge delocalisation. Additionally differential map does not show any electron density peak which could ne a proton of –OH.

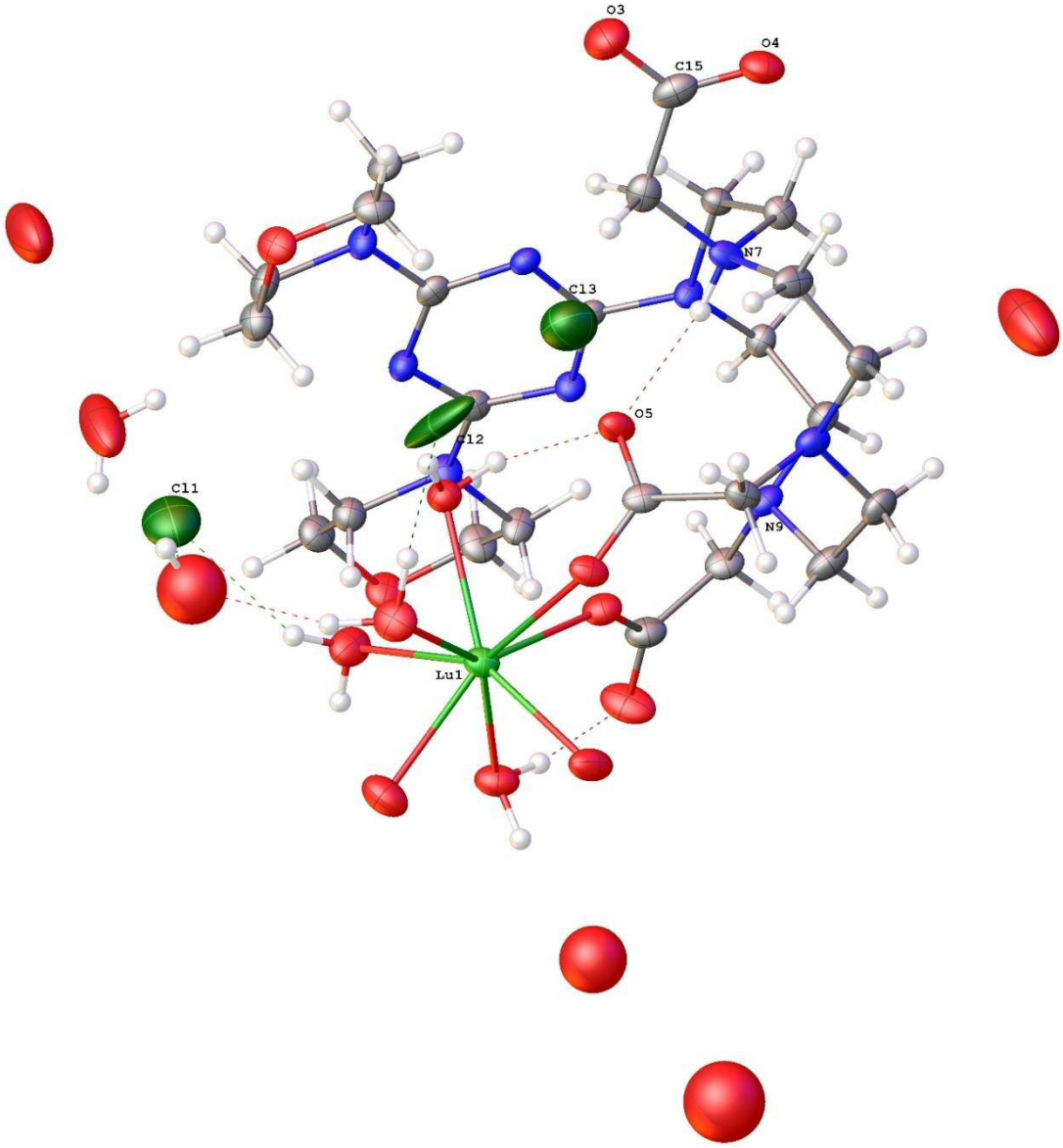

Figure 1. Molecular structure of 2015ncs0604. Displacement ellipsoids – 50% probability.

Table 1. Atomic coordinates [$\times 10^4$], equivalent isotropic displacement parameters [$Å^2 \times 10^3$] and site occupancy factors. U_{eq} is defined as one third of the trace of the orthogonalized U^{ij} tensor.

Atom	x	y	z	U_{eq}	S.o.f.
Lu1	−742(1)	2055(1)	2931(1)	20(1)	1
O1	985(4)	625(3)	−751(2)	27(1)	1
O2	−4719(4)	4514(3)	1134(2)	26(1)	1
O3	−1681(5)	8432(4)	2502(3)	40(1)	1
O4	449(5)	9048(3)	3151(3)	32(1)	1
O5	194(4)	4882(3)	2965(2)	24(1)	1
O6	−263(4)	3740(3)	3743(2)	22(1)	1
O7	2594(5)	1905(3)	2322(3)	39(1)	1
O8	1243(4)	2913(3)	2699(2)	25(1)	1
O9	459(4)	819(3)	2657(2)	28(1)	1
O10	316(5)	2097(3)	4268(2)	31(1)	1
O11	−1400(4)	3120(3)	2041(2)	23(1)	1
O12	−1699(5)	1071(3)	1593(2)	32(1)	1
O13	−2736(4)	2276(3)	3217(3)	30(1)	1
O14	−1877(5)	495(3)	3168(3)	36(1)	1
N1	−1079(4)	3424(3)	329(3)	18(1)	1
N2	−296(4)	5233(3)	1015(2)	16(1)	1
N3	1248(4)	4137(3)	940(2)	17(1)	1
N4	459(4)	2452(3)	124(3)	20(1)	1
N5	−2528(4)	4506(3)	388(3)	19(1)	1
N6	1954(4)	5854(3)	1699(2)	15(1)	1
N7	906(5)	7066(3)	3143(2)	18(1)	1
N8	2328(5)	6071(3)	4217(3)	21(1)	1
N9	3158(4)	4706(3)	2942(3)	20(1)	1
C1	203(5)	3368(4)	475(3)	18(1)	1
C2	−608(5)	1501(4)	−211(3)	22(1)	1
C3	−295(6)	837(4)	−954(3)	26(1)	1
C4	2005(6)	1573(4)	−472(3)	26(1)	1
C5	1800(5)	2262(4)	299(3)	23(1)	1
C6	−1255(5)	4391(4)	588(3)	18(1)	1
C7	−3684(5)	3615(4)	125(3)	23(1)	1
C8	−4358(5)	3560(4)	853(3)	24(1)	1
C9	−3558(6)	5381(4)	1400(3)	25(1)	1
C10	−2877(5)	5478(4)	686(3)	21(1)	1
C11	925(5)	5048(4)	1194(3)	15(1)	1
C12	1790(5)	6928(4)	1829(3)	17(1)	1
C13	1991(5)	7491(4)	2742(3)	19(1)	1
C14	−451(5)	7183(4)	2760(3)	23(1)	1
C15	−556(6)	8335(5)	2825(3)	27(1)	1
C16	1234(6)	7502(4)	4078(3)	25(1)	1
C17	2424(6)	7184(4)	4529(3)	24(1)	1
C18	1226(5)	5387(4)	4422(3)	20(1)	1
C19	321(5)	4611(4)	3654(3)	19(1)	1
C20	3593(6)	5789(5)	4437(3)	24(1)	1
C21	3614(6)	4769(5)	3866(3)	27(1)	1
C22	3318(5)	3699(4)	2447(3)	22(1)	1
C23	2309(6)	2757(4)	2508(3)	25(1)	1
C24	3913(5)	5624(4)	2700(3)	19(1)	1
C25	3320(5)	5726(4)	1830(3)	17(1)	1
O17	−4121(7)	896(5)	−1364(6)	85(3)	1
O15	−5134(9)	1246(7)	2013(6)	98(3)	1
Cl2	−3213(4)	4443(5)	3329(3)	75(2)	0.5
Cl1	−4212(5)	1057(3)	400(3)	61(1)	0.5
O16	1032(8)	393(7)	4677(5)	91(2)	1
Cl3	−2520(3)	6328(2)	4272(1)	63(1)	1
O18	−5595(6)	2114(5)	−2193(6)	84(2)	1
O19	5696(7)	7876(6)	3850(6)	91(3)	1
O20	3094(12)	−59(9)	5621(7)	133(4)	1

Table 2. Bond lengths [Å] and angles [°].

Lu1–O6	2.313(4)	C4–C5	1.513(7)
Lu1–O8	2.312(4)	C5–H5A	0.9900
Lu1–O9	2.299(4)	C5–H5B	0.9900
Lu1–O10	2.295(4)	C7–H7A	0.9900
Lu1–O11	2.366(4)	C7–H7B	0.9900
Lu1–O12	2.300(4)	C7–C8	1.526(7)
Lu1–O13	2.287(4)	C8–H8A	0.9900
Lu1–O14	2.346(4)	C8–H8B	0.9900
O1–C3	1.418(7)	C9–H9C	0.9900
O1–C4	1.427(7)	C9–H9D	0.9900
O2–C8	1.425(6)	C9–C10	1.518(7)
O2–C9	1.441(7)	C10–H10A	0.9900
O3–C15	1.245(7)	C10–H10B	0.9900
O4–C15	1.230(8)	C12–H12C	0.9900
O5–C19	1.271(6)	C12–H12D	0.9900
O6–C19	1.262(6)	C12–C13	1.526(6)
O7–C23	1.237(7)	C13–H13C	0.9900
O8–C23	1.260(7)	C13–H13D	0.9900
O9–H9A	0.9250	C14–H14A	0.9900
O9–H9B	0.9250	C14–H14B	0.9900
O11–H11A	0.9166	C14–C15	1.559(8)
O11–H11B	0.9142	C16–H16A	0.9900
O12–H12A	0.9063	C16–H16B	0.9900
O12–H12B	0.9082	C16–C17	1.515(8)
O13–H13A	0.9142	C17–H17A	0.9900
O13–H13B	0.9153	C17–H17B	0.9900
N1–C1	1.338(6)	C18–H18A	0.9900
N1–C6	1.349(6)	C18–H18B	0.9900
N2–C6	1.333(7)	C18–C19	1.511(7)
N2–C11	1.341(6)	C20–H20A	0.9900
N3–C1	1.343(7)	C20–H20B	0.9900
N3–C11	1.346(6)	C20–C21	1.519(8)
N4–C1	1.357(6)	C21–H21A	0.9900
N4–C2	1.460(7)	C21–H21B	0.9900
N4–C5	1.463(7)	C22–H22A	0.9900
N5–C6	1.359(6)	C22–H22B	0.9900
N5–C7	1.459(7)	C22–C23	1.517(8)
N5–C10	1.446(6)	C24–H24A	0.9900
N6–C11	1.379(6)	C24–H24B	0.9900
N6–C12	1.467(6)	C24–C25	1.521(6)
N6–C25	1.455(6)	C25–H25A	0.9900
N7–H7	0.89(7)	C25–H25B	0.9900
N7–C13	1.500(6)	O17–H17C	0.8496
N7–C14	1.498(7)	O17–H17D	0.8499
N7–C16	1.512(6)	O15–H15A	0.8497
N8–C17	1.464(7)	O15–H15B	0.8502
N8–C18	1.467(7)		
N8–C20	1.451(7)	O6–Lu1–O11	72.72(13)
N9–H9	1.0000	O6–Lu1–O14	129.35(14)
N9–C21	1.517(6)	O8–Lu1–O6	73.96(13)
N9–C22	1.502(6)	O8–Lu1–O11	75.67(13)
N9–C24	1.499(7)	O8–Lu1–O14	144.59(14)
C2–H2A	0.9900	O9–Lu1–O6	135.44(15)
C2–H2B	0.9900	O9–Lu1–O8	73.55(14)
C2–C3	1.513(7)	O9–Lu1–O11	126.05(13)
C3–H3A	0.9900	O9–Lu1–O12	72.81(15)
C3–H3B	0.9900	O9–Lu1–O14	71.75(15)
C4–H4A	0.9900	O10–Lu1–O6	70.76(13)
C4–H4B	0.9900	O10–Lu1–O8	90.69(15)

O10–Lu1–O9	80.00(14)
O10–Lu1–O11	143.28(13)
O10–Lu1–O12	147.38(14)
O10–Lu1–O14	76.61(16)
O12–Lu1–O6	141.86(14)
O12–Lu1–O8	98.38(15)
O12–Lu1–O11	69.21(14)
O12–Lu1–O14	78.00(16)
O13–Lu1–O6	75.05(15)
O13–Lu1–O8	143.49(14)
O13–Lu1–O9	142.92(15)
O13–Lu1–O10	96.90(15)
O13–Lu1–O11	77.10(14)
O13–Lu1–O12	94.17(16)
O13–Lu1–O14	71.61(16)
O14–Lu1–O11	132.26(15)
C3–O1–C4	110.6(4)
C8–O2–C9	111.0(4)
C19–O6–Lu1	133.0(3)
C23–O8–Lu1	141.9(4)
Lu1–O9–H9A	113.0
Lu1–O9–H9B	113.0
H9A–O9–H9B	105.5
Lu1–O11–H11A	112.7
Lu1–O11–H11B	112.4
H11A–O11–H11B	106.2
Lu1–O12–H12A	112.2
Lu1–O12–H12B	112.1
H12A–O12–H12B	106.6
Lu1–O13–H13A	112.2
Lu1–O13–H13B	112.7
H13A–O13–H13B	106.0
C1–N1–C6	114.0(4)
C6–N2–C11	113.9(4)
C1–N3–C11	113.7(4)
C1–N4–C2	121.4(4)
C1–N4–C5	121.9(4)
C2–N4–C5	113.7(4)
C6–N5–C7	122.0(4)
C6–N5–C10	122.0(4)
C10–N5–C7	112.6(4)
C11–N6–C12	119.6(4)
C11–N6–C25	119.4(4)
C25–N6–C12	116.7(4)
C13–N7–H7	107(4)
C13–N7–C16	112.9(4)
C14–N7–H7	107(4)
C14–N7–C13	114.0(4)
C14–N7–C16	110.0(4)
C16–N7–H7	105(4)
C17–N8–C18	113.6(4)
C20–N8–C17	112.8(4)
C20–N8–C18	112.4(4)
C21–N9–H9	108.3
C22–N9–H9	108.3
C22–N9–C21	109.2(4)
C24–N9–H9	108.3
C24–N9–C21	111.5(4)
C24–N9–C22	111.0(4)
N1–C1–N3	125.7(4)
N1–C1–N4	116.7(5)
N3–C1–N4	117.6(4)
N4–C2–H2A	109.8
N4–C2–H2B	109.8
N4–C2–C3	109.4(4)
H2A–C2–H2B	108.2
C3–C2–H2A	109.8
C3–C2–H2B	109.8
O1–C3–C2	111.8(4)
O1–C3–H3A	109.3
O1–C3–H3B	109.3
C2–C3–H3A	109.3
C2–C3–H3B	109.3
H3A–C3–H3B	107.9
O1–C4–H4A	109.4
O1–C4–H4B	109.4
O1–C4–C5	111.0(4)
H4A–C4–H4B	108.0
C5–C4–H4A	109.4
C5–C4–H4B	109.4
N4–C5–C4	109.4(4)
N4–C5–H5A	109.8
N4–C5–H5B	109.8
C4–C5–H5A	109.8
C4–C5–H5B	109.8
H5A–C5–H5B	108.2
N1–C6–N5	116.3(5)
N2–C6–N1	125.9(4)
N2–C6–N5	117.9(4)
N5–C7–H7A	109.9
N5–C7–H7B	109.9
N5–C7–C8	108.7(4)
H7A–C7–H7B	108.3
C8–C7–H7A	109.9
C8–C7–H7B	109.9
O2–C8–C7	110.7(4)
O2–C8–H8A	109.5
O2–C8–H8B	109.5
C7–C8–H8A	109.5
C7–C8–H8B	109.5
H8A–C8–H8B	108.1
O2–C9–H9C	109.6
O2–C9–H9D	109.6
O2–C9–C10	110.4(4)
H9C–C9–H9D	108.1
C10–C9–H9C	109.6
C10–C9–H9D	109.6
N5–C10–C9	109.0(4)
N5–C10–H10A	109.9
N5–C10–H10B	109.9
C9–C10–H10A	109.9
C9–C10–H10B	109.9
H10A–C10–H10B	108.3
N2–C11–N3	126.2(5)
N2–C11–N6	117.7(4)
N3–C11–N6	116.1(4)
N6–C12–H12C	108.6
N6–C12–H12D	108.6
N6–C12–C13	114.7(4)
H12C–C12–H12D	107.6

C13–C12–H12C	108.6	O6–C19–C18	117.7(4)
C13–C12–H12D	108.6	N8–C20–H20A	109.3
N7–C13–C12	113.6(4)	N8–C20–H20B	109.3
N7–C13–H13C	108.8	N8–C20–C21	111.6(4)
N7–C13–H13D	108.8	H20A–C20–H20B	108.0
C12–C13–H13C	108.8	C21–C20–H20A	109.3
C12–C13–H13D	108.8	C21–C20–H20B	109.3
H13C–C13–H13D	107.7	N9–C21–C20	114.5(4)
N7–C14–H14A	108.6	N9–C21–H21A	108.6
N7–C14–H14B	108.6	N9–C21–H21B	108.6
N7–C14–C15	114.5(5)	C20–C21–H21A	108.6
H14A–C14–H14B	107.6	C20–C21–H21B	108.6
C15–C14–H14A	108.6	H21A–C21–H21B	107.6
C15–C14–H14B	108.6	N9–C22–H22A	109.1
O3–C15–C14	114.5(6)	N9–C22–H22B	109.1
O4–C15–O3	126.2(6)	N9–C22–C23	112.5(4)
O4–C15–C14	119.2(5)	H22A–C22–H22B	107.8
N7–C16–H16A	109.0	C23–C22–H22A	109.1
N7–C16–H16B	109.0	C23–C22–H22B	109.1
N7–C16–C17	112.8(4)	O7–C23–O8	126.8(6)
H16A–C16–H16B	107.8	O7–C23–C22	116.0(5)
C17–C16–H16A	109.0	O8–C23–C22	117.2(5)
C17–C16–H16B	109.0	N9–C24–H24A	108.7
N8–C17–C16	110.7(4)	N9–C24–H24B	108.7
N8–C17–H17A	109.5	N9–C24–C25	114.1(4)
N8–C17–H17B	109.5	H24A–C24–H24B	107.6
C16–C17–H17A	109.5	C25–C24–H24A	108.7
C16–C17–H17B	109.5	C25–C24–H24B	108.7
H17A–C17–H17B	108.1	N6–C25–C24	115.0(4)
N8–C18–H18A	109.2	N6–C25–H25A	108.5
N8–C18–H18B	109.2	N6–C25–H25B	108.5
N8–C18–C19	112.0(4)	C24–C25–H25A	108.5
H18A–C18–H18B	107.9	C24–C25–H25B	108.5
C19–C18–H18A	109.2	H25A–C25–H25B	107.5
C19–C18–H18B	109.2	H17C–O17–H17D	109.5
O5–C19–C18	117.9(5)	H15A–O15–H15B	109.
O6–C19–O5	124.5(5)		

Table 3. Anisotropic displacement parameters [$Å^2 \times 10^3$].

The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + \cdots + 2\,h\,k\,a^*\,b^*\,U^{12}]$.

Atom	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Lu1	26(1)	18(1)	18(1)	3(1)	9(1)	9(1)
O1	31(2)	21(2)	29(2)	–2(2)	10(2)	13(2)
O2	21(2)	30(2)	32(2)	7(2)	14(2)	11(2)
O3	34(3)	42(3)	47(3)	13(2)	7(2)	17(2)
O4	43(3)	23(2)	34(2)	8(2)	11(2)	13(2)
O5	34(2)	20(2)	16(2)	5(1)	5(2)	6(2)
O6	34(2)	17(2)	18(2)	2(1)	12(2)	9(2)
O7	47(3)	28(2)	55(3)	11(2)	34(2)	21(2)
O8	27(2)	26(2)	26(2)	7(2)	9(2)	14(2)
O9	38(2)	23(2)	29(2)	8(2)	15(2)	15(2)
O10	51(3)	22(2)	19(2)	3(2)	6(2)	12(2)
O11	24(2)	22(2)	23(2)	5(1)	6(2)	8(2)
O12	40(2)	27(2)	27(2)	–2(2)	5(2)	15(2)
O13	29(2)	34(2)	35(2)	6(2)	19(2)	14(2)
O14	42(3)	23(2)	45(3)	6(2)	20(2)	6(2)
N1	16(2)	20(2)	18(2)	2(2)	3(2)	6(2)
N2	17(2)	19(2)	14(2)	5(2)	5(2)	8(2)
N3	17(2)	18(2)	17(2)	1(2)	6(2)	8(2)
N4	18(2)	18(2)	22(2)	–2(2)	7(2)	7(2)
N5	13(2)	22(2)	21(2)	2(2)	5(2)	7(2)
N6	16(2)	16(2)	14(2)	–1(1)	5(2)	9(2)
N7	28(2)	14(2)	13(2)	1(2)	9(2)	7(2)
N8	26(2)	23(2)	15(2)	4(2)	4(2)	10(2)
N9	20(2)	26(2)	18(2)	5(2)	5(2)	13(2)
C1	22(3)	21(2)	12(2)	2(2)	6(2)	9(2)
C2	23(3)	22(3)	23(2)	2(2)	6(2)	8(2)
C3	31(3)	22(3)	23(2)	–2(2)	6(2)	8(2)
C4	28(3)	21(3)	29(3)	–3(2)	12(2)	11(2)
C5	22(3)	22(3)	24(2)	–4(2)	5(2)	11(2)
C6	20(2)	25(3)	11(2)	5(2)	7(2)	10(2)
C7	15(2)	31(3)	21(2)	–1(2)	2(2)	7(2)
C8	18(3)	28(3)	27(3)	3(2)	8(2)	7(2)
C9	31(3)	25(3)	24(3)	4(2)	13(2)	16(2)
C10	18(2)	23(3)	25(2)	7(2)	7(2)	12(2)
C11	18(2)	19(2)	11(2)	3(2)	6(2)	9(2)
C12	21(2)	16(2)	16(2)	4(2)	7(2)	5(2)
C13	22(3)	20(2)	16(2)	1(2)	9(2)	6(2)
C14	24(3)	30(3)	18(2)	5(2)	10(2)	6(2)
C15	32(3)	38(3)	21(2)	13(2)	13(2)	20(3)
C16	38(3)	22(3)	15(2)	1(2)	6(2)	14(2)
C17	31(3)	22(3)	14(2)	0(2)	4(2)	4(2)
C18	29(3)	19(2)	14(2)	3(2)	9(2)	7(2)
C19	26(3)	22(2)	17(2)	5(2)	13(2)	15(2)
C20	25(3)	33(3)	14(2)	4(2)	2(2)	11(2)
C21	30(3)	39(3)	17(2)	8(2)	7(2)	21(3)
C22	26(3)	24(3)	21(2)	4(2)	11(2)	16(2)
C23	31(3)	25(3)	24(2)	8(2)	11(2)	16(2)
C24	17(2)	24(3)	18(2)	5(2)	4(2)	8(2)
C25	16(2)	22(2)	14(2)	3(2)	7(2)	6(2)
O17	38(3)	38(3)	156(7)	–2(4)	–4(4)	10(3)
Cl2	38(2)	150(5)	80(3)	87(3)	24(2)	52(3)
Cl1	58(2)	54(2)	78(3)	12(2)	19(2)	30(2)
Cl3	73(2)	60(1)	56(1)	19(1)	7(1)	18(1)
O18	41(4)	50(4)	145(7)	19(4)	–1(4)	–1(3)
O19	60(4)	52(4)	157(8)	13(4)	31(5)	8(3)

Table 4. Hydrogen coordinates [$\times 10^4$] and isotropic displacement parameters [$Å^2 \times 10^3$].

Atom	x	y	z	U_{eq}	S.o.f.
H9A	722	551	3111	41	1
H9B	1240	1074	2503	41	1
H11A	−699	3635	2022	34	1
H11B	−2028	3449	2205	34	1
H12A	−2606	917	1483	48	1
H12B	−1460	452	1495	48	1
H13A	−3453	1815	2841	46	1
H13B	−2862	2927	3201	46	1
H7	850(70)	6390(50)	3070(40)	26(16)	1
H9	2186	4707	2818	24	1
H2A	−1473	1682	−384	27	1
H2B	−683	1106	225	27	1
H3A	−985	170	−1155	31	1
H3B	−329	1203	−1412	31	1
H4A	1992	1951	−922	31	1
H4B	2892	1410	−344	31	1
H5A	1904	1916	769	28	1
H5B	2482	2933	461	28	1
H7A	−3394	2963	−45	28	1
H7B	−4323	3694	−359	28	1
H8A	−5170	2978	673	29	1
H8B	−3739	3422	1318	29	1
H9C	−2922	5275	1873	30	1
H9D	−3833	6033	1594	30	1
H10A	−3487	5636	228	25	1
H10B	−2060	6055	881	25	1
H12C	880	6916	1526	21	1
H12D	2438	7334	1582	21	1
H13C	2861	7437	3061	23	1
H13D	2033	8240	2777	23	1
H14A	−1112	6842	3035	28	1
H14B	−697	6810	2163	28	1
H16A	443	7256	4295	30	1
H16B	1425	8274	4197	30	1
H17A	3260	7597	4447	28	1
H17B	2459	7334	5135	28	1
H18A	693	5818	4698	24	1
H18B	1601	5005	4820	24	1
H20A	3747	5721	5022	29	1
H20B	4330	6352	4400	29	1
H21A	4537	4669	3977	32	1
H21B	3031	4188	4007	32	1
H22A	4234	3619	2652	26	1
H22B	3214	3725	1853	26	1
H24A	4848	5563	2730	23	1
H24B	3939	6269	3110	23	1
H25A	3896	6333	1716	21	1
H25B	3340	5095	1418	21	1
H17C	−3671	1491	−1067	127	1
H17D	−4185	462	−1059	127	1
H15A	−4939	1139	1539	147	1
H15B	−5784	1540	1993	147	1

Table 5. Torsion angles [°].

Lu1–O6–C19–O5	39.4(7)	C7–N5–C6–N1	16.7(6)
Lu1–O6–C19–C18	–141.2(4)	C7–N5–C6–N2	–163.6(4)
Lu1–O8–C23–O7	4.2(10)	C7–N5–C10–C9	57.1(5)
Lu1–O8–C23–C22	–179.8(4)	C8–O2–C9–C10	59.4(5)
O1–C4–C5–N4	–55.8(6)	C9–O2–C8–C7	–59.1(5)
O2–C9–C10–N5	–57.1(5)	C10–N5–C6–N1	174.7(4)
N4–C2–C3–O1	54.9(6)	C10–N5–C6–N2	–5.5(6)
N5–C7–C8–O2	56.5(5)	C10–N5–C7–C8	–56.6(5)
N6–C12–C13–N7	–69.2(5)	C11–N2–C6–N1	–1.1(6)
N7–C14–C15–O3	179.6(4)	C11–N2–C6–N5	179.2(4)
N7–C14–C15–O4	2.1(7)	C11–N3–C1–N1	–6.6(7)
N7–C16–C17–N8	–45.1(6)	C11–N3–C1–N4	173.7(4)
N8–C18–C19–O5	–30.4(6)	C11–N6–C12–C13	123.6(5)
N8–C18–C19–O6	150.1(4)	C11–N6–C25–C24	–109.2(5)
N8–C20–C21–N9	49.1(6)	C12–N6–C11–N2	–16.7(6)
N9–C22–C23–O7	–160.6(5)	C12–N6–C11–N3	164.4(4)
N9–C22–C23–O8	22.9(7)	C12–N6–C25–C24	94.0(5)
N9–C24–C25–N6	60.3(6)	C13–N7–C14–C15	–62.1(5)
C1–N1–C6–N2	–5.3(6)	C13–N7–C16–C17	–65.1(6)
C1–N1–C6–N5	174.4(4)	C14–N7–C13–C12	–63.3(5)
C1–N3–C11–N2	–1.0(6)	C14–N7–C16–C17	166.3(5)
C1–N3–C11–N6	177.8(4)	C16–N7–C13–C12	170.2(4)
C1–N4–C2–C3	147.1(5)	C16–N7–C14–C15	65.9(5)
C1–N4–C5–C4	–146.4(5)	C17–N8–C18–C19	126.1(4)
C2–N4–C1–N1	–15.2(7)	C17–N8–C20–C21	–159.1(4)
C2–N4–C1–N3	164.6(4)	C18–N8–C17–C16	–67.7(5)
C2–N4–C5–C4	52.9(6)	C18–N8–C20–C21	70.8(5)
C3–O1–C4–C5	60.2(6)	C20–N8–C17–C16	162.8(4)
C4–O1–C3–C2	–59.9(6)	C20–N8–C18–C19	–104.2(5)
C5–N4–C1–N1	–174.4(4)	C21–N9–C22–C23	71.1(5)
C5–N4–C1–N3	5.4(7)	C21–N9–C24–C25	–168.2(4)
C5–N4–C2–C3	–52.2(6)	C22–N9–C21–C20	176.0(5)
C6–N1–C1–N3	9.4(7)	C22–N9–C24–C25	69.8(5)
C6–N1–C1–N4	–170.8(4)	C24–N9–C21–C20	52.9(6)
C6–N2–C11–N3	4.5(6)	C24–N9–C22–C23	–165.5(4)
C6–N2–C11–N6	–174.3(4)	C25–N6–C11–N2	–172.9(4)
C6–N5–C7–C8	103.3(5)	C25–N6–C11–N3	8.2(6)
C6–N5–C10–C9	–102.8(5)	C25–N6–C12–C13	–79.7(5)

APPENDIX B

During the PhD it has been possible to obtain the crystal structure of an alternative compound or impurity. Unfortunately it has not been possible to obtain a full data set, but the structure will be reported alongside the crystallographer's comment about the sample:

"Sample contains clusters of crystalline material (highly twinned). I picked a small flat plate fragment which looked relatively clean. Again, this is not what you were expecting (me neither :)). Please see attached VERY interesting structure of bi (or maybe main :)) product. WELL DONE! Again I think I will complete it as it is. This compound is known...oh yeah."