Synthesis, Structural Characterization, and Antimicrobial Activity of Zn(cloxyquin)$_2$: Towards Harnessing Zinc Intoxication and Immune Response Restoration to Combat *Staphylococcus aureus* and *Mycobacterium tuberculosis*

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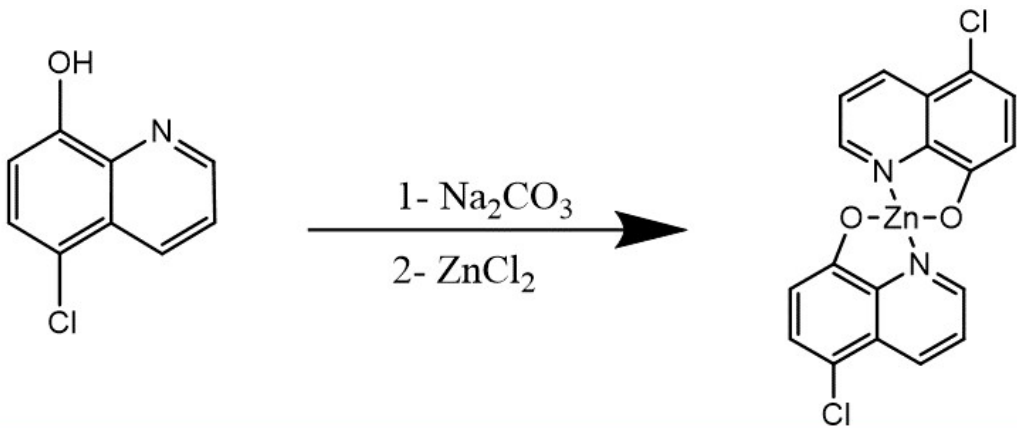


Scheme S1. Synthesis of Zn(cloxyquin)$_2$ in ethanol

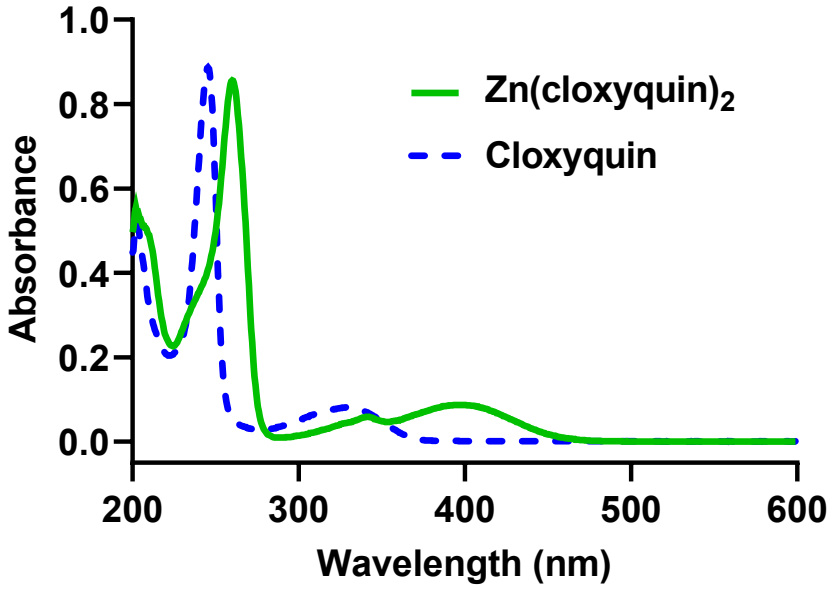


Figure S1. UV-vis spectra of Zn(cloxyquin)$_2$ in comparison with cloxyquin in DMSO/ethanol mixture.

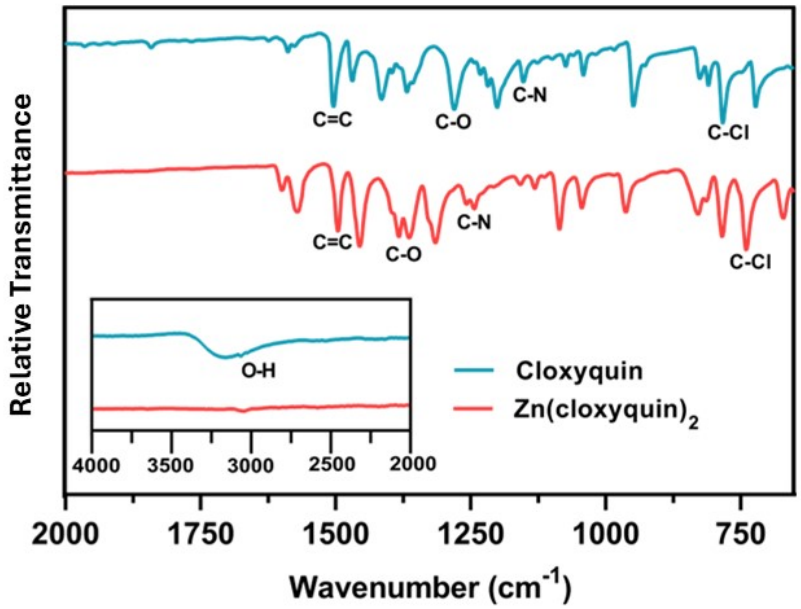
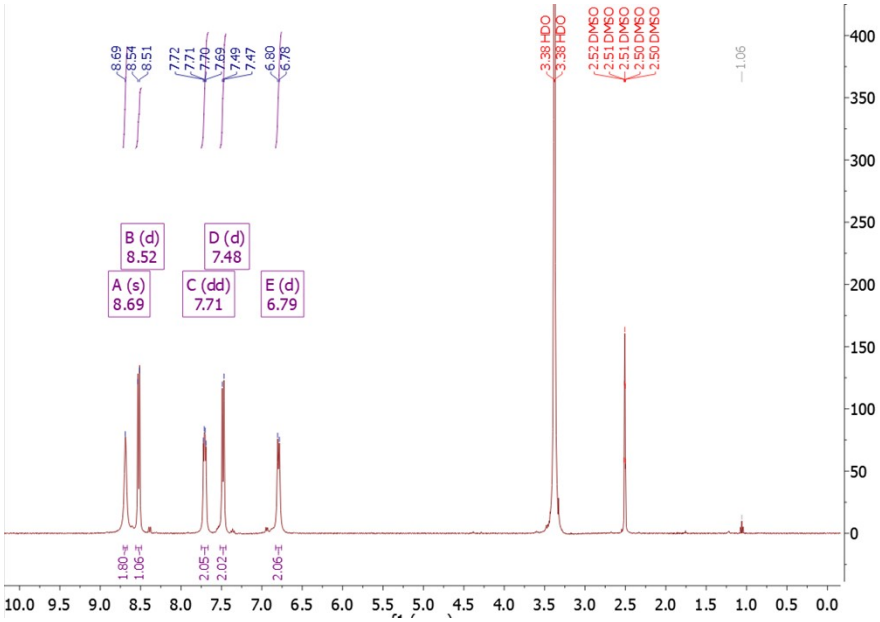


Figure S2. FT-IR spectra of $Zn(cloxyquin)_2$ in comparison with cloxyquin.



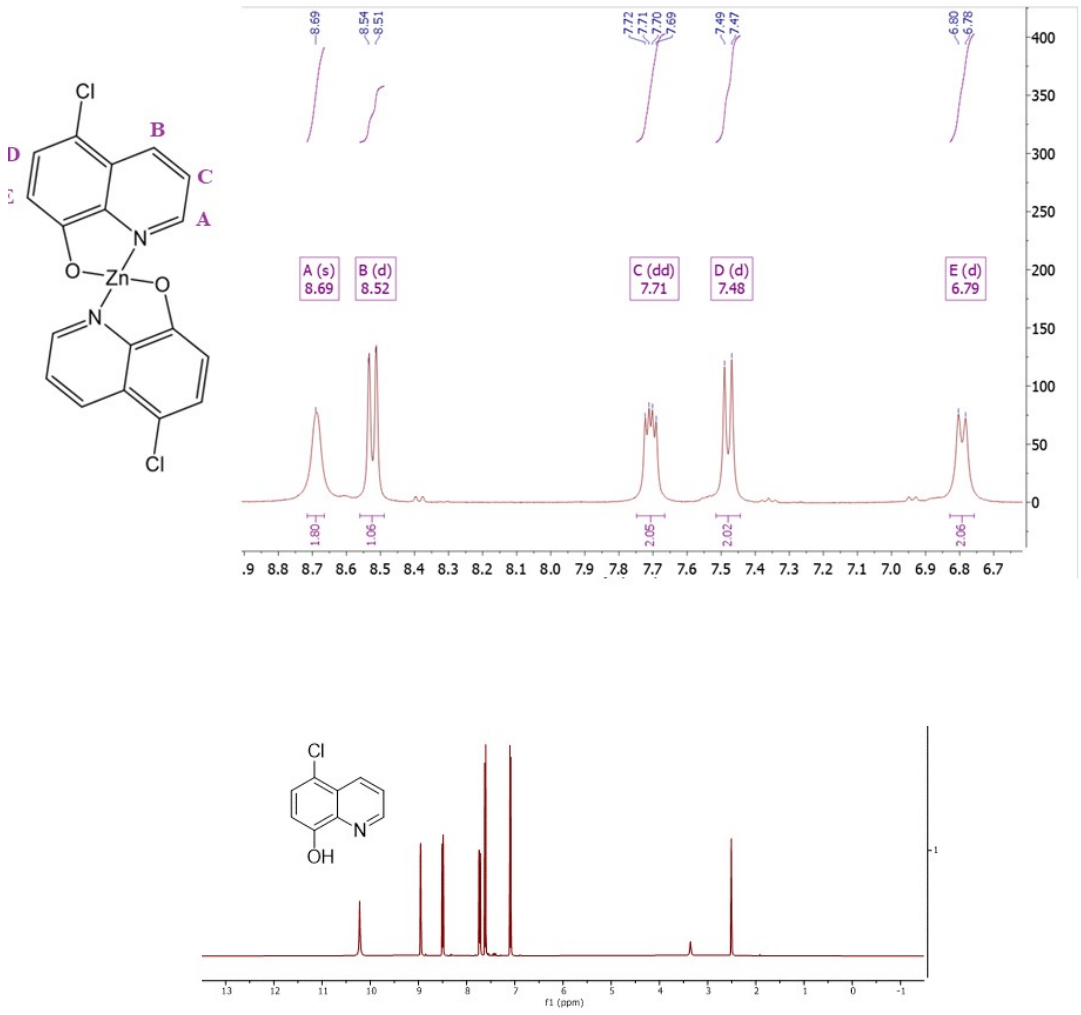
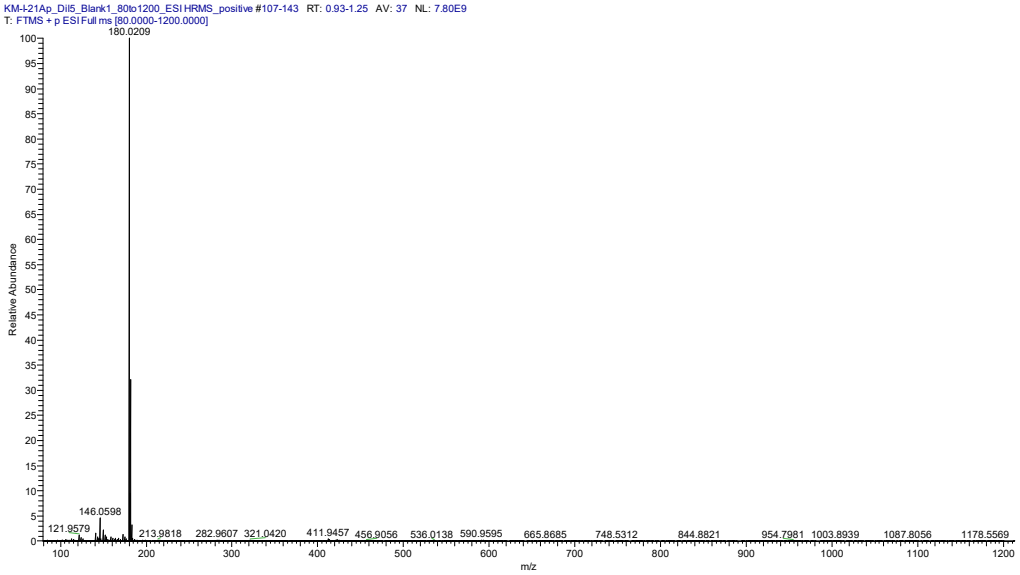
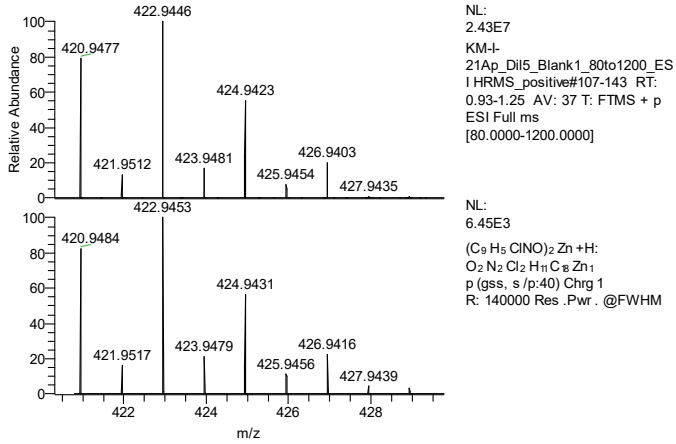
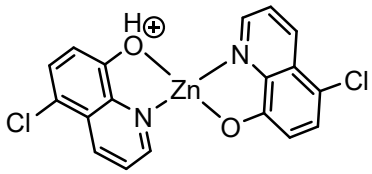


Figure S3. ^{1}H NMR spectrum of Zn(cloxyquin)$_2$ (top) with the expanded aromatic region (middle) in comparison with the ^{1}H NMR spectrum of cloxyquin in DMSO-d$_6$ (400 MHz)

[Note: δ = 8.69 (s, 1H, H_A), 8.52 (d, J = 7.6 Hz, 1H, H_B), 7.71 (dd, J = 7.5, 1.4 Hz, 1H, H_C), 7.48 (d, J = 8.1 Hz, 1H, H_D), 6.79 (d, J = 8.1 Hz, 1H, H_E), 3.38 (s, HDO), 2.50 (s, DMSO-d$_6$), 1.06 (s, residual/impurity)].







Chemical Formula: $C_{18}H_{11}Cl_2N_2O_2Zn^+$

Exact Mass: 420.9484

(High resolution electrospray ionization mass spectrometry (ESI-HRMS): m/z calculated for $[C_{18}H_{10}Cl_2N_2O_2Zn + H]^+$: 422.9446; found: 420.9484.)

Figure S4. ESI-HRMS: $[(C_9H_5ClNO)_2Zn+H]^+$ 422.9446.

Table S1. Elemental analysis results of Zn(cloxyquin)$_2$

Elements	$C_{18}H_{10}Cl_2N_2O_2Zn$	
	Calculated %	Experimental %
Carbon (C)	51.16	51.18
Hydrogen (H)	2.39	2.32

Nitrogen (N)	6.63	6.70

Experimental of single-crystal structure analysis:

Crystals grown from THF-hexane. For X-ray examination and data collection, a suitable orange-yellow, tablet-shaped crystal, approximate dimensions 0.116 x 0.098 x 0.065 mm, was mounted in a loop with Paratone-N oil and transferred to the goniostat bathed in a cold N_2 stream.

Intensity data were collected at 150K on a Bruker APEX-II CCD diffractometer using Mo Kα radiation, λ=0.71073Å (TRIUMPH curved-graphite monochromator). The data frames were processed using the program SAINT. The data were corrected for decay, Lorentz and polarization effects as well as absorption and beam corrections.

The structure was solved by a combination of direct methods and the difference Fourier technique as implemented in the SHELX suite of programs and refined by full-matrix least squares on F^2 for reflections out to 0.80Å resolution. Non-hydrogen atoms were refined with anisotropic displacement parameters. THF/m-THF crystallizes in the lattice. Disordered solvent atom occupancies were refined and then rounded. Anisotropic displacement parameter restraints were applied to the disordered atoms. H-atoms were calculated and treated with a riding model. The H-atom isotropic displacement parameters were defined as $a*U_{eq}$ of the adjacent atom (a=1.5 for methyl or 1.2 for all others). The refinement converged with crystallographic agreement factors of R1=3.07%, wR2=7.11% for 6799 reflections with I>2σ(I) (R1=4.49%, wR2=7.70% for all data) and 605 variable parameters.

References:

APEX3 v2018.1.0 and SAINT v8.38A, Bruker Analytical X-ray Instruments, Inc., Madison, WI; SADABS v2016/2, L. Krause, R. Herbst-Irmer, G.M. Sheldrick, D. Stalke, J. Appl. Cryst. (2015), 48, 3-10; SHELXTL v6.14, Bruker Analytical X-ray Instruments, Inc., Madison, WI; SHELX 2018/3, G.M. Sheldrick (2015) Acta Cryst., C71, 3-8.

Table S2. Crystal data and structure refinement for $C_{72}H_{40}N_8O_8Cl_8Zn_4.3.5(C_4H_8O).0.5(C_6H_{12})$.

CCDC Deposition Number	**CCDC-2383653**	
Formula	$C_{72}H_{40}N_8O_8Cl_8Zn_4.3.5(C_4H_8O).0.5(C_5H_{10}O)$	
Formula weight	1985.62	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 11.1480(9) Å	α = 89.2213(16)°
	b = 13.4176(11) Å	β = 81.6740(16)°
	c = 14.5317(12) Å	γ = 72.4901(15)°
Volume	2050.0(3) Å^3	
Z	1	
Density (calculated)	1.608 Mg/m^3	
Absorption coefficient	1.487 mm^{-1}	
F(000)	1012	
Crystal size	0.116 x 0.098 x 0.065 mm^3	
θ range for data collection	1.417 to 26.406°	
Index ranges	$-13 \leq h \leq 13$, $-16 \leq k \leq 16$, $-18 \leq l \leq 18$	
Reflections collected	40243	
Independent reflections	8403 [R_{int} = 0.0293]	
Completeness to θ = 25.242°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.862 and 0.834	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	8403 / 166 / 605	
Goodness-of-fit on F^2	1.011	
Final R indices [I>2σ(I)]	R1 = 0.0307, wR2 = 0.0711	
R indices (all data)	R1 = 0.0449, wR2 = 0.0770	
Largest diff. peak and hole	0.736 and -0.391 eÅ^{-3}	

Table S3. Atomic coordinates [x10^4] and equivalent isotropic displacement parameters [Å^{2}x10^3] for $C_{72}H_{40}N_8O_8Cl_8Zn_4.3.5(C_4H_8O).0.5(C_5H_{10}O)$. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
Zn(1)	5811(1)	5753(1)	4491(1)	21(1)
Zn(2)	5899(1)	6368(1)	2322(1)	24(1)
Cl(2)	6727(1)	761(1)	1713(1)	49(1)
Cl(4)	12300(1)	3176(1)	3257(1)	38(1)
Cl(6)	7365(1)	2528(1)	-1083(1)	54(1)
Cl(8)	1060(1)	10883(1)	3766(1)	46(1)
O(1)	5388(2)	4376(1)	4297(1)	24(1)
O(2)	7046(1)	5886(1)	3294(1)	24(1)
O(4)	7197(2)	6197(1)	1163(1)	28(1)
O(5)	4549(1)	6573(1)	3532(1)	24(1)
N(1)	4444(2)	2822(1)	4770(1)	22(1)
N(2)	7660(2)	4861(1)	4812(1)	23(1)
N(3)	5416(2)	5239(2)	1624(1)	26(1)
N(4)	4934(2)	7918(2)	2222(1)	25(1)
C(1)	5800(2)	3544(2)	3736(2)	21(1)
C(2)	6715(2)	3403(2)	2963(2)	25(1)
C(3)	7015(2)	2526(2)	2360(2)	30(1)
C(4)	6440(2)	1768(2)	2539(2)	30(1)
C(5)	5569(2)	1808(2)	3363(2)	26(1)
C(6)	5260(2)	2705(2)	3957(2)	21(1)
C(7)	4998(2)	1027(2)	3654(2)	33(1)
C(8)	4191(2)	1159(2)	4474(2)	33(1)
C(9)	3940(2)	2072(2)	5022(2)	27(1)
C(11)	8253(2)	5302(2)	3258(2)	22(1)
C(12)	9178(2)	5196(2)	2496(2)	26(1)
C(13)	10423(2)	4541(2)	2516(2)	27(1)
C(14)	10749(2)	3989(2)	3282(2)	26(1)
C(15)	9853(2)	4071(2)	4096(2)	22(1)
C(16)	8601(2)	4738(2)	4072(2)	21(1)
C(17)	10098(2)	3543(2)	4931(2)	27(1)
C(18)	9143(2)	3690(2)	5664(2)	31(1)
C(19)	7925(2)	4351(2)	5577(2)	28(1)
C(21)	7278(2)	5371(2)	665(2)	26(1)
C(22)	8242(2)	4946(2)	-67(2)	32(1)
C(23)	8256(2)	4074(2)	-589(2)	36(1)
C(24)	7328(2)	3598(2)	-400(2)	34(1)
C(25)	6314(2)	3969(2)	345(2)	29(1)
C(26)	6321(2)	4840(2)	876(2)	25(1)
C(27)	5306(2)	3543(2)	596(2)	34(1)
C(28)	4401(2)	3965(2)	1338(2)	35(1)

C(29)	4491(2)	4814(2)	1844(2)	33(1)
C(31)	3781(2)	7545(2)	3629(2)	22(1)
C(32)	2818(2)	7918(2)	4367(2)	25(1)
C(33)	2003(2)	8947(2)	4404(2)	28(1)
C(34)	2134(2)	9624(2)	3716(2)	29(1)
C(35)	3116(2)	9316(2)	2950(2)	28(1)
C(36)	3942(2)	8277(2)	2924(2)	23(1)
C(37)	3343(3)	9949(2)	2198(2)	35(1)
C(38)	4335(3)	9565(2)	1501(2)	38(1)
C(39)	5129(2)	8538(2)	1538(2)	31(1)
O(6)	2322(4)	11127(3)	209(3)	57(1)
C(40)	1371(5)	10597(4)	391(5)	53(1)
C(41)	135(5)	11409(4)	718(4)	58(1)
C(42)	476(8)	12311(5)	1011(5)	63(1)
C(43)	1787(5)	12201(4)	526(6)	60(1)
O(6B)	2406(14)	11386(11)	387(11)	56(2)
C(40B)	1580(18)	10762(13)	262(15)	55(2)
C(41B)	442(14)	11196(12)	1026(11)	56(2)
C(42B)	390(20)	12268(15)	1213(16)	58(2)
C(43B)	1489(17)	12413(12)	581(18)	58(2)
C(44B)	1203(9)	12869(8)	-282(7)	29(2)
O(7A)	1651(4)	1662(4)	6364(3)	67(1)
O(7B)	2258(11)	1180(9)	6623(7)	76(2)
C(45)	1136(4)	818(3)	6480(2)	66(1)
C(46)	326(4)	999(3)	7408(2)	67(1)
C(47)	696(4)	1836(4)	7855(3)	81(1)
C(48)	1886(3)	1857(3)	7316(3)	60(1)
H(2A)	7152	3910	2835	30
H(3A)	7629	2460	1820	35
H(7A)	5175	411	3279	40
H(8A)	3803	637	4673	39
H(9A)	3385	2152	5595	32
H(12A)	8973	5569	1952	31
H(13A)	11048	4482	1984	33
H(17A)	10923	3090	4981	33
H(18A)	9301	3348	6230	37
H(19A)	7265	4434	6088	34
H(22A)	8906	5255	-216	38
H(23A)	8928	3806	-1088	44
H(27A)	5256	2965	250	40
H(28A)	3715	3686	1510	42
H(29A)	3862	5095	2365	39
H(32A)	2705	7468	4860	30
H(33A)	1347	9177	4919	33
H(37A)	2802	10645	2178	43
H(38A)	4486	9990	996	45

H(39A)	5828	8279	1057	37
H(40A)	1588	10072	873	63
H(40B)	1315	10235	-183	63
H(41A)	-387	11592	209	69
H(41B)	-351	11155	1245	69
H(42A)	457	12325	1694	76
H(42B)	-130	12967	838	76
H(43A)	1755	12670	-8	71
H(43B)	2305	12381	958	71
H(40C)	2005	10011	346	66
H(40D)	1314	10851	-362	66
H(41C)	-353	11170	814	67
H(41D)	559	10784	1594	67
H(42C)	468	12376	1871	69
H(42D)	-419	12761	1073	69
H(43C)	1880	12857	911	70
H(44D)	1382	13541	-324	43
H(44E)	303	12979	-322	43
H(44F)	1729	12400	-796	43
H(45A)	1823	145	6457	79
H(45B)	620	801	5983	79
H(45C)	671	1231	6001	79
H(45D)	1415	68	6288	79
H(46A)	506	355	7772	80
H(46B)	-588	1234	7344	80
H(47A)	37	2521	7842	98
H(47B)	805	1675	8510	98
H(48A)	2617	1300	7496	72
H(48B)	2031	2545	7377	72
H(48C)	1787	2567	7074	72
H(48D)	2549	1711	7728	72

Table S4. Bond lengths [Å] and angles [°] for $C_{72}H_{40}N_8O_8Cl_8Zn_4.3.5(C_4H_8O).0.5(C_5H_{10}O)$.

Zn(1)-O(1)	2.0721(15)	Zn(1)-O(1)#1	2.0897(15)
Zn(1)-O(2)	2.0975(15)	Zn(1)-N(1)#1	2.1271(19)
Zn(1)-N(2)	2.1624(19)	Zn(1)-O(5)	2.1742(15)
Zn(2)-O(2)	2.0117(15)	Zn(2)-O(4)	2.0220(15)
Zn(2)-N(4)	2.0483(19)	Zn(2)-N(3)	2.074(2)
Zn(2)-O(5)	2.1044(15)	Cl(2)-C(4)	1.744(2)
Cl(4)-C(14)	1.740(2)	Cl(6)-C(24)	1.744(3)
Cl(8)-C(34)	1.748(2)	O(1)-C(1)	1.316(3)
O(2)-C(11)	1.332(3)	O(4)-C(21)	1.306(3)
O(5)-C(31)	1.324(3)	N(1)-C(9)	1.316(3)
N(1)-C(6)	1.363(3)	N(2)-C(19)	1.321(3)
N(2)-C(16)	1.363(3)	N(3)-C(29)	1.321(3)
N(3)-C(26)	1.364(3)	N(4)-C(39)	1.322(3)
N(4)-C(36)	1.364(3)	C(1)-C(2)	1.376(3)
C(1)-C(6)	1.441(3)	C(2)-C(3)	1.404(3)
C(3)-C(4)	1.360(4)	C(4)-C(5)	1.419(3)
C(5)-C(7)	1.413(3)	C(5)-C(6)	1.415(3)
C(7)-C(8)	1.363(4)	C(8)-C(9)	1.404(3)
C(11)-C(12)	1.376(3)	C(11)-C(16)	1.433(3)
C(12)-C(13)	1.405(3)	C(13)-C(14)	1.361(3)
C(14)-C(15)	1.415(3)	C(15)-C(17)	1.417(3)
C(15)-C(16)	1.419(3)	C(17)-C(18)	1.363(3)
C(18)-C(19)	1.402(4)	C(21)-C(22)	1.388(3)
C(21)-C(26)	1.447(3)	C(22)-C(23)	1.398(4)
C(23)-C(24)	1.366(4)	C(24)-C(25)	1.421(3)
C(25)-C(27)	1.409(4)	C(25)-C(26)	1.412(3)
C(27)-C(28)	1.363(4)	C(28)-C(29)	1.400(4)
C(31)-C(32)	1.383(3)	C(31)-C(36)	1.439(3)
C(32)-C(33)	1.402(3)	C(33)-C(34)	1.364(3)
C(34)-C(35)	1.412(3)	C(35)-C(37)	1.416(3)
C(35)-C(36)	1.418(3)	C(37)-C(38)	1.364(4)
C(38)-C(39)	1.401(4)	O(6)-C(43)	1.436(6)
O(6)-C(40)	1.438(6)	C(40)-C(41)	1.494(6)
C(41)-C(42)	1.458(7)	C(42)-C(43)	1.493(7)
O(6B)-C(40B)	1.448(15)	O(6B)-C(43B)	1.450(14)
C(40B)-C(41B)	1.531(15)	C(41B)-C(42B)	1.449(15)
C(42B)-C(43B)	1.482(15)	C(43B)-C(44B)	1.42(3)
O(7A)-C(45)	1.414(5)	O(7A)-C(48)	1.486(5)
O(7B)-C(48)	1.301(11)	O(7B)-C(45)	1.512(12)
C(45)-C(46)	1.488(5)	C(46)-C(47)	1.494(5)
C(47)-C(48)	1.448(5)		

O(1)-Zn(1)-O(1)#1	75.09(6)	O(1)-Zn(1)-O(2)	105.65(6)
O(1)#1-Zn(1)-O(2)	178.71(6)	O(1)-Zn(1)-N(1)#1	152.47(6)

O(1)#1-Zn(1)-N(1)#1	77.71(6)	O(2)-Zn(1)-N(1)#1	101.63(7)
O(1)-Zn(1)-N(2)	89.89(7)	O(1)#1-Zn(1)-N(2)	102.11(7)
O(2)-Zn(1)-N(2)	76.87(6)	N(1)#1-Zn(1)-N(2)	99.90(7)
O(1)-Zn(1)-O(5)	92.13(6)	O(1)#1-Zn(1)-O(5)	105.21(6)
O(2)-Zn(1)-O(5)	75.88(6)	N(1)#1-Zn(1)-O(5)	90.95(6)
N(2)-Zn(1)-O(5)	152.19(6)	O(2)-Zn(2)-O(4)	100.67(6)
O(2)-Zn(2)-N(4)	121.01(7)	O(4)-Zn(2)-N(4)	98.64(7)
O(2)-Zn(2)-N(3)	117.64(7)	O(4)-Zn(2)-N(3)	81.64(7)
N(4)-Zn(2)-N(3)	120.04(8)	O(2)-Zn(2)-O(5)	79.28(6)
O(4)-Zn(2)-O(5)	178.96(6)	N(4)-Zn(2)-O(5)	80.53(7)
N(3)-Zn(2)-O(5)	99.31(7)	C(1)-O(1)-Zn(1)	138.46(14)
C(1)-O(1)-Zn(1)#1	115.47(13)	Zn(1)-O(1)-Zn(1)#1	104.91(6)
C(11)-O(2)-Zn(2)	133.41(14)	C(11)-O(2)-Zn(1)	115.89(13)
Zn(2)-O(2)-Zn(1)	104.18(7)	C(21)-O(4)-Zn(2)	111.54(14)
C(31)-O(5)-Zn(2)	111.40(13)	C(31)-O(5)-Zn(1)	126.98(13)
Zn(2)-O(5)-Zn(1)	98.54(6)	C(9)-N(1)-C(6)	119.14(19)
C(9)-N(1)-Zn(1)#1	127.81(16)	C(6)-N(1)-Zn(1)#1	113.03(14)
C(19)-N(2)-C(16)	119.0(2)	C(19)-N(2)-Zn(1)	127.67(16)
C(16)-N(2)-Zn(1)	112.84(14)	C(29)-N(3)-C(26)	119.0(2)
C(29)-N(3)-Zn(2)	130.67(17)	C(26)-N(3)-Zn(2)	109.92(15)
C(39)-N(4)-C(36)	120.0(2)	C(39)-N(4)-Zn(2)	126.97(17)
C(36)-N(4)-Zn(2)	112.84(14)	O(1)-C(1)-C(2)	125.5(2)
O(1)-C(1)-C(6)	117.11(19)	C(2)-C(1)-C(6)	117.4(2)
C(1)-C(2)-C(3)	121.3(2)	C(4)-C(3)-C(2)	120.9(2)
C(3)-C(4)-C(5)	121.3(2)	C(3)-C(4)-Cl(2)	119.09(19)
C(5)-C(4)-Cl(2)	119.57(19)	C(7)-C(5)-C(6)	116.9(2)
C(7)-C(5)-C(4)	125.9(2)	C(6)-C(5)-C(4)	117.1(2)
N(1)-C(6)-C(5)	122.1(2)	N(1)-C(6)-C(1)	116.25(19)
C(5)-C(6)-C(1)	121.6(2)	C(8)-C(7)-C(5)	119.9(2)
C(7)-C(8)-C(9)	119.4(2)	N(1)-C(9)-C(8)	122.5(2)
O(2)-C(11)-C(12)	124.8(2)	O(2)-C(11)-C(16)	117.14(19)
C(12)-C(11)-C(16)	118.1(2)	C(11)-C(12)-C(13)	120.7(2)
C(14)-C(13)-C(12)	121.3(2)	C(13)-C(14)-C(15)	121.1(2)
C(13)-C(14)-Cl(4)	119.14(18)	C(15)-C(14)-Cl(4)	119.73(18)
C(14)-C(15)-C(17)	125.6(2)	C(14)-C(15)-C(16)	117.2(2)
C(17)-C(15)-C(16)	117.2(2)	N(2)-C(16)-C(15)	122.0(2)
N(2)-C(16)-C(11)	116.47(19)	C(15)-C(16)-C(11)	121.5(2)
C(18)-C(17)-C(15)	119.6(2)	C(17)-C(18)-C(19)	119.6(2)
N(2)-C(19)-C(18)	122.7(2)	O(4)-C(21)-C(22)	124.3(2)
O(4)-C(21)-C(26)	119.3(2)	C(22)-C(21)-C(26)	116.4(2)
C(21)-C(22)-C(23)	121.4(2)	C(24)-C(23)-C(22)	121.6(2)
C(23)-C(24)-C(25)	120.9(2)	C(23)-C(24)-Cl(6)	120.1(2)
C(25)-C(24)-Cl(6)	119.0(2)	C(27)-C(25)-C(26)	117.5(2)
C(27)-C(25)-C(24)	125.6(2)	C(26)-C(25)-C(24)	117.0(2)
N(3)-C(26)-C(25)	121.8(2)	N(3)-C(26)-C(21)	115.6(2)
C(25)-C(26)-C(21)	122.6(2)	C(28)-C(27)-C(25)	119.7(2)

C(27)-C(28)-C(29)	119.5(2)	N(3)-C(29)-C(28)	122.5(2)
O(5)-C(31)-C(32)	124.6(2)	O(5)-C(31)-C(36)	118.67(19)
C(32)-C(31)-C(36)	116.8(2)	C(31)-C(32)-C(33)	121.5(2)
C(34)-C(33)-C(32)	121.3(2)	C(33)-C(34)-C(35)	120.8(2)
C(33)-C(34)-Cl(8)	119.88(19)	C(35)-C(34)-Cl(8)	119.28(19)
C(34)-C(35)-C(37)	125.8(2)	C(34)-C(35)-C(36)	117.4(2)
C(37)-C(35)-C(36)	116.8(2)	N(4)-C(36)-C(35)	121.4(2)
N(4)-C(36)-C(31)	116.5(2)	C(35)-C(36)-C(31)	122.1(2)
C(38)-C(37)-C(35)	120.4(2)	C(37)-C(38)-C(39)	119.3(2)
N(4)-C(39)-C(38)	122.0(2)	C(43)-O(6)-C(40)	109.7(4)
O(6)-C(40)-C(41)	107.1(4)	C(42)-C(41)-C(40)	105.3(5)
C(41)-C(42)-C(43)	106.9(5)	O(6)-C(43)-C(42)	106.4(4)
C(40B)-O(6B)-C(43B)	101.1(13)	O(6B)-C(40B)-C(41B)	103.2(12)
C(42B)-C(41B)-C(40B)	106.2(12)	C(41B)-C(42B)-C(43B)	104.1(12)
C(44B)-C(43B)-O(6B)	108.2(16)	C(44B)-C(43B)-C(42B)	115.1(19)
O(6B)-C(43B)-C(42B)	107.0(14)	C(45)-O(7A)-C(48)	103.2(3)
C(48)-O(7B)-C(45)	107.6(7)	O(7A)-C(45)-C(46)	106.0(3)
C(46)-C(45)-O(7B)	104.0(4)	C(45)-C(46)-C(47)	103.5(3)
C(48)-C(47)-C(46)	105.8(3)	O(7B)-C(48)-C(47)	112.5(5)
C(47)-C(48)-O(7A)	100.4(3)		

Symmetry transformations used to generate equivalent atoms: #1 -x+1,-y+1,-z+1

Table S5. Anisotropic displacement parameters [$Å^2x10^3$] for $C_{72}H_{40}N_8O_8Cl_8Zn_4.3.5(C_4H_8O).0.5(C_5H_{10}O)$.
The anisotropic displacement factor exponent takes the form: $-2\pi^2[h\,^2a^{*2}U_{11}+... +2hka^*b^*U_{12}]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Zn(1)	22(1)	22(1)	18(1)	0(1)	2(1)	-7(1)
Zn(2)	25(1)	27(1)	18(1)	1(1)	-1(1)	-5(1)
Cl(2)	63(1)	37(1)	43(1)	-18(1)	10(1)	-15(1)
Cl(4)	23(1)	45(1)	39(1)	6(1)	-2(1)	-1(1)
Cl(6)	46(1)	55(1)	59(1)	-30(1)	0(1)	-12(1)
Cl(8)	44(1)	26(1)	56(1)	0(1)	4(1)	0(1)
O(1)	29(1)	21(1)	22(1)	-3(1)	4(1)	-10(1)
O(2)	21(1)	28(1)	20(1)	3(1)	-1(1)	-5(1)
O(4)	31(1)	32(1)	21(1)	0(1)	1(1)	-10(1)
O(5)	24(1)	24(1)	21(1)	2(1)	-2(1)	-4(1)
N(1)	21(1)	23(1)	21(1)	2(1)	-3(1)	-6(1)
N(2)	24(1)	24(1)	20(1)	1(1)	-1(1)	-10(1)
N(3)	25(1)	32(1)	21(1)	3(1)	-2(1)	-7(1)
N(4)	25(1)	28(1)	22(1)	3(1)	-4(1)	-9(1)
C(1)	21(1)	22(1)	20(1)	2(1)	-4(1)	-4(1)
C(2)	23(1)	27(1)	24(1)	1(1)	0(1)	-7(1)
C(3)	27(1)	31(1)	24(1)	-4(1)	3(1)	-3(1)
C(4)	32(1)	25(1)	28(1)	-9(1)	0(1)	-3(1)
C(5)	24(1)	23(1)	28(1)	-1(1)	-5(1)	-4(1)
C(6)	19(1)	22(1)	20(1)	2(1)	-5(1)	-3(1)
C(7)	36(1)	23(1)	40(1)	-4(1)	-6(1)	-8(1)
C(8)	34(1)	27(1)	40(1)	6(1)	-7(1)	-14(1)
C(9)	24(1)	30(1)	27(1)	4(1)	-3(1)	-9(1)
C(11)	22(1)	22(1)	23(1)	0(1)	-2(1)	-9(1)
C(12)	24(1)	32(1)	20(1)	4(1)	-2(1)	-8(1)
C(13)	23(1)	35(1)	23(1)	-1(1)	2(1)	-10(1)
C(14)	20(1)	26(1)	30(1)	-1(1)	-3(1)	-4(1)
C(15)	24(1)	22(1)	24(1)	1(1)	-5(1)	-10(1)
C(16)	23(1)	20(1)	21(1)	-1(1)	-2(1)	-9(1)
C(17)	27(1)	26(1)	29(1)	4(1)	-8(1)	-8(1)
C(18)	38(1)	33(1)	25(1)	8(1)	-8(1)	-14(1)
C(19)	32(1)	31(1)	22(1)	3(1)	-1(1)	-13(1)
C(21)	26(1)	28(1)	19(1)	6(1)	-5(1)	-3(1)
C(22)	26(1)	41(2)	27(1)	2(1)	-2(1)	-9(1)
C(23)	28(1)	44(2)	31(1)	-8(1)	3(1)	-4(1)
C(24)	33(1)	35(1)	30(1)	-11(1)	-5(1)	-2(1)
C(25)	28(1)	32(1)	26(1)	2(1)	-9(1)	-4(1)
C(26)	24(1)	30(1)	19(1)	3(1)	-6(1)	-3(1)
C(27)	34(1)	33(1)	35(1)	-1(1)	-13(1)	-9(1)
C(28)	29(1)	45(2)	34(1)	6(1)	-5(1)	-17(1)

C(29)	28(1)	43(2)	26(1)	2(1)	-2(1)	-9(1)
C(31)	21(1)	24(1)	24(1)	0(1)	-6(1)	-9(1)
C(32)	24(1)	27(1)	23(1)	1(1)	-2(1)	-9(1)
C(33)	24(1)	30(1)	29(1)	-5(1)	0(1)	-8(1)
C(34)	26(1)	21(1)	38(1)	-4(1)	-3(1)	-3(1)
C(35)	27(1)	26(1)	32(1)	2(1)	-7(1)	-10(1)
C(36)	21(1)	26(1)	23(1)	0(1)	-6(1)	-10(1)
C(37)	38(2)	26(1)	42(2)	8(1)	-8(1)	-9(1)
C(38)	44(2)	33(1)	36(1)	13(1)	-5(1)	-15(1)
C(39)	31(1)	36(1)	27(1)	6(1)	-2(1)	-13(1)
O(6)	54(2)	44(2)	62(2)	4(2)	8(2)	-6(2)
C(40)	54(2)	43(2)	57(2)	-5(2)	1(2)	-13(2)
C(41)	58(2)	44(2)	63(2)	-2(2)	9(2)	-10(2)
C(42)	62(2)	48(2)	70(3)	-4(2)	6(2)	-9(2)
C(43)	62(2)	44(2)	64(2)	3(2)	8(2)	-12(2)
O(6B)	57(2)	42(3)	60(3)	0(2)	5(2)	-10(2)
C(40B)	57(3)	42(3)	60(3)	-3(2)	3(2)	-10(2)
C(41B)	56(3)	44(3)	62(3)	-2(3)	4(3)	-12(2)
C(42B)	60(3)	45(2)	61(3)	0(2)	8(3)	-12(2)
C(43B)	60(3)	42(3)	63(3)	-1(2)	7(3)	-9(2)
C(44B)	21(5)	35(5)	33(5)	9(4)	-11(4)	-10(4)
O(7A)	70(3)	103(3)	45(2)	25(2)	-9(2)	-53(3)
O(7B)	86(5)	92(5)	51(4)	17(4)	15(4)	-39(4)
C(45)	73(2)	71(2)	55(2)	-16(2)	-2(2)	-26(2)
C(46)	75(2)	87(3)	57(2)	2(2)	-2(2)	-55(2)
C(47)	85(3)	111(3)	54(2)	-36(2)	21(2)	-50(3)
C(48)	62(2)	43(2)	80(3)	-1(2)	-8(2)	-25(2)

Table 6. Torsion angles [°] for $C_{72}H_{40}N_8O_8Cl_8Zn_4.3.5(C_4H_8O).0.5(C_5H_{10}O)$.

Zn(1)-O(1)-C(1)-C(2)	-6.1(4)	Zn(1)#1-O(1)-C(1)-C(2)	-171.48(18)
Zn(1)-O(1)-C(1)-C(6)	172.91(15)	Zn(1)#1-O(1)-C(1)-C(6)	7.5(2)
O(1)-C(1)-C(2)-C(3)	-174.5(2)	C(6)-C(1)-C(2)-C(3)	6.5(3)
C(1)-C(2)-C(3)-C(4)	-2.2(4)	C(2)-C(3)-C(4)-C(5)	-3.4(4)
C(2)-C(3)-C(4)-Cl(2)	173.69(19)	C(3)-C(4)-C(5)-C(7)	-174.6(2)
Cl(2)-C(4)-C(5)-C(7)	8.3(4)	C(3)-C(4)-C(5)-C(6)	4.3(4)
Cl(2)-C(4)-C(5)-C(6)	-172.83(17)	C(9)-N(1)-C(6)-C(5)	1.1(3)
Zn(1)#1-N(1)-C(6)-C(5)	179.52(17)	C(9)-N(1)-C(6)-C(1)	-178.6(2)
Zn(1)#1-N(1)-C(6)-C(1)	-0.1(2)	C(7)-C(5)-C(6)-N(1)	-0.4(3)
C(4)-C(5)-C(6)-N(1)	-179.4(2)	C(7)-C(5)-C(6)-C(1)	179.2(2)
C(4)-C(5)-C(6)-C(1)	0.2(3)	O(1)-C(1)-C(6)-N(1)	-4.9(3)
C(2)-C(1)-C(6)-N(1)	174.18(19)	O(1)-C(1)-C(6)-C(5)	175.4(2)
C(2)-C(1)-C(6)-C(5)	-5.5(3)	C(6)-C(5)-C(7)-C(8)	-0.1(4)
C(4)-C(5)-C(7)-C(8)	178.8(2)	C(5)-C(7)-C(8)-C(9)	-0.1(4)
C(6)-N(1)-C(9)-C(8)	-1.3(3)	Zn(1)#1-N(1)-C(9)-C(8)	-179.44(17)
C(7)-C(8)-C(9)-N(1)	0.8(4)	Zn(2)-O(2)-C(11)-C(12)	26.6(3)
Zn(1)-O(2)-C(11)-C(12)	173.08(18)	Zn(2)-O(2)-C(11)-C(16)	-152.89(15)
Zn(1)-O(2)-C(11)-C(16)	-6.4(2)	O(2)-C(11)-C(12)-C(13)	-178.5(2)
C(16)-C(11)-C(12)-C(13)	0.9(3)	C(11)-C(12)-C(13)-C(14)	0.4(4)
C(12)-C(13)-C(14)-C(15)	-1.4(4)	C(12)-C(13)-C(14)-Cl(4)	179.04(18)
C(13)-C(14)-C(15)-C(17)	-179.1(2)	Cl(4)-C(14)-C(15)-C(17)	0.5(3)
C(13)-C(14)-C(15)-C(16)	1.0(3)	Cl(4)-C(14)-C(15)-C(16)	-179.44(17)
C(19)-N(2)-C(16)-C(15)	0.6(3)	Zn(1)-N(2)-C(16)-C(15)	-171.93(16)
C(19)-N(2)-C(16)-C(11)	179.2(2)	Zn(1)-N(2)-C(16)-C(11)	6.7(2)
C(14)-C(15)-C(16)-N(2)	179.0(2)	C(17)-C(15)-C(16)-N(2)	-1.0(3)
C(14)-C(15)-C(16)-C(11)	0.4(3)	C(17)-C(15)-C(16)-C(11)	-179.5(2)
O(2)-C(11)-C(16)-N(2)	-0.5(3)	C(12)-C(11)-C(16)-N(2)	-180.0(2)
O(2)-C(11)-C(16)-C(15)	178.19(19)	C(12)-C(11)-C(16)-C(15)	-1.3(3)
C(14)-C(15)-C(17)-C(18)	-179.7(2)	C(16)-C(15)-C(17)-C(18)	0.2(3)
C(15)-C(17)-C(18)-C(19)	0.9(4)	C(16)-N(2)-C(19)-C(18)	0.6(3)
Zn(1)-N(2)-C(19)-C(18)	171.84(18)	C(17)-C(18)-C(19)-N(2)	-1.3(4)
Zn(2)-O(4)-C(21)-C(22)	-167.38(19)	Zn(2)-O(4)-C(21)-C(26)	12.2(2)
O(4)-C(21)-C(22)-C(23)	-178.3(2)	C(26)-C(21)-C(22)-C(23)	2.1(3)
C(21)-C(22)-C(23)-C(24)	-0.3(4)	C(22)-C(23)-C(24)-C(25)	-0.7(4)
C(22)-C(23)-C(24)-Cl(6)	178.8(2)	C(23)-C(24)-C(25)-C(27)	179.5(3)
Cl(6)-C(24)-C(25)-C(27)	0.1(4)	C(23)-C(24)-C(25)-C(26)	-0.3(4)
Cl(6)-C(24)-C(25)-C(26)	-179.71(18)	C(29)-N(3)-C(26)-C(25)	-1.7(3)
Zn(2)-N(3)-C(26)-C(25)	171.98(17)	C(29)-N(3)-C(26)-C(21)	178.2(2)
Zn(2)-N(3)-C(26)-C(21)	-8.2(2)	C(27)-C(25)-C(26)-N(3)	2.2(3)
C(24)-C(25)-C(26)-N(3)	-178.0(2)	C(27)-C(25)-C(26)-C(21)	-177.6(2)
C(24)-C(25)-C(26)-C(21)	2.2(3)	O(4)-C(21)-C(26)-N(3)	-2.5(3)
C(22)-C(21)-C(26)-N(3)	177.0(2)	O(4)-C(21)-C(26)-C(25)	177.3(2)
C(22)-C(21)-C(26)-C(25)	-3.1(3)	C(26)-C(25)-C(27)-C(28)	-1.2(4)
C(24)-C(25)-C(27)-C(28)	179.0(2)	C(25)-C(27)-C(28)-C(29)	-0.3(4)

C(26)-N(3)-C(29)-C(28)	0.1(4)	Zn(2)-N(3)-C(29)-C(28)	-172.09(18)
C(27)-C(28)-C(29)-N(3)	1.0(4)	Zn(2)-O(5)-C(31)-C(32)	-178.52(18)
Zn(1)-O(5)-C(31)-C(32)	-58.9(3)	Zn(2)-O(5)-C(31)-C(36)	1.5(2)
Zn(1)-O(5)-C(31)-C(36)	121.12(18)	O(5)-C(31)-C(32)-C(33)	-177.2(2)
C(36)-C(31)-C(32)-C(33)	2.8(3)	C(31)-C(32)-C(33)-C(34)	-0.5(4)
C(32)-C(33)-C(34)-C(35)	-1.4(4)	C(32)-C(33)-C(34)-Cl(8)	177.52(19)
C(33)-C(34)-C(35)-C(37)	180.0(2)	Cl(8)-C(34)-C(35)-C(37)	1.1(4)
C(33)-C(34)-C(35)-C(36)	0.8(4)	Cl(8)-C(34)-C(35)-C(36)	-178.15(18)
C(39)-N(4)-C(36)-C(35)	-1.2(3)	Zn(2)-N(4)-C(36)-C(35)	-176.59(17)
C(39)-N(4)-C(36)-C(31)	178.6(2)	Zn(2)-N(4)-C(36)-C(31)	3.2(2)
C(34)-C(35)-C(36)-N(4)	-178.5(2)	C(37)-C(35)-C(36)-N(4)	2.2(3)
C(34)-C(35)-C(36)-C(31)	1.7(3)	C(37)-C(35)-C(36)-C(31)	-177.6(2)
O(5)-C(31)-C(36)-N(4)	-3.2(3)	C(32)-C(31)-C(36)-N(4)	176.79(19)
O(5)-C(31)-C(36)-C(35)	176.6(2)	C(32)-C(31)-C(36)-C(35)	-3.4(3)
C(34)-C(35)-C(37)-C(38)	179.3(2)	C(36)-C(35)-C(37)-C(38)	-1.5(4)
C(35)-C(37)-C(38)-C(39)	-0.1(4)	C(36)-N(4)-C(39)-C(38)	-0.6(4)
Zn(2)-N(4)-C(39)-C(38)	174.14(19)	C(37)-C(38)-C(39)-N(4)	1.2(4)
C(43)-O(6)-C(40)-C(41)	7.8(7)	O(6)-C(40)-C(41)-C(42)	-18.5(7)
C(40)-C(41)-C(42)-C(43)	21.9(8)	C(40)-O(6)-C(43)-C(42)	5.8(8)
C(41)-C(42)-C(43)-O(6)	-17.5(9)	C(43B)-O(6B)-C(40B)-C(41B)	-40(2)
O(6B)-C(40B)-C(41B)-C(42B)	26(2)	C(40B)-C(41B)-C(42B)-C(43B)	-1(3)
C(40B)-O(6B)-C(43B)-C(44B)	-83.1(17)	C(40B)-O(6B)-C(43B)-C(42B)	41(2)
C(41B)-C(42B)-C(43B)-C(44B)	95.5(19)	C(41B)-C(42B)-C(43B)-O(6B)	-25(3)
C(48)-O(7A)-C(45)-C(46)	36.7(4)	C(48)-O(7B)-C(45)-C(46)	-24.9(8)
O(7A)-C(45)-C(46)-C(47)	-13.0(5)	O(7B)-C(45)-C(46)-C(47)	24.1(6)
C(45)-C(46)-C(47)-C(48)	-16.3(5)	C(45)-O(7B)-C(48)-C(47)	15.0(9)
C(46)-C(47)-C(48)-O(7B)	0.9(7)	C(46)-C(47)-C(48)-O(7A)	37.7(4)
C(45)-O(7A)-C(48)-C(47)	-46.2(4)		

Symmetry transformations used to generate equivalent atoms: #1 -x+1,-y+1,-z+1