Crystal structure of tetrakis(1,10-phenanthroline)-tetrakis(2-hydroxy-3,5-dinitrobenzoato)tetrazinc(II), $Zn_4(C_{12}H_8N_2)_4(C_7H_2N_2O_7)_4$

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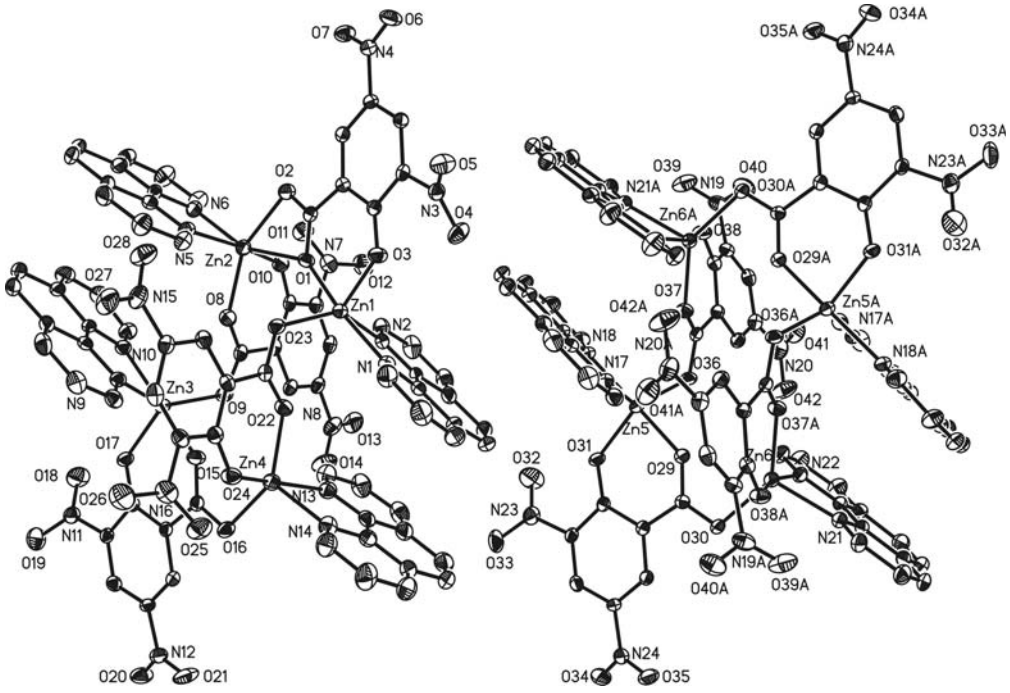

Abstract

$C_{76}H_{40}N_{16}O_{28}Zn_4$, monoclinic, $P12_1/c1$ (no. 14), a = 14.605(1) Å, b = 21.402(2) Å, c = 34.967(3) Å, β = 94.763(1)°, V = 10892 Å^3, Z = 6, $R_{gt}(F)$ = 0.041, $wR_{ref}(F^2)$ = 0.094, T = 293 K.

Source of material

The title complex was prepared by reaction of 1,10-phenanthroline (phen), 2-hydroxy-3,5-dinitrobenzoic acid and $ZnSO_4$ at room temperature using interface diffusion technique. In a clean tube three kinds of solutions were carefully loaded using injector S, forming two clear interfaces. From the bottom to the top the three layers are 0.5 mmol $ZnSO_4$ in 5 mL water; the mixture of 2.5 ml ethanol with 2.5 ml water and 0.5 mmol 1,10-phenanthroline with 0.5 mmol 2-hydroxy-3,5-dinitrobenzoic acid in 5 ml ethanol which pH value was adjusted to be approx. 7 in advance. The tube was then air-proofed and kept untouched in the air. After one week crystals of the title compound were obtained.

Experimental details

All H atoms were located at calculated positions and refined as riding on their parent atoms with d(C—H) = 0.93 Å and U_{iso}(H) = 1.2 U_{eq}(C). Search with PLATON [1] did not reveal any additional symmetry elements.

Discussion

The title zinc compound features isolated tetra-nuclear cluster units. All zinc(II) ions in the complex adopt a distorted trigonal bipyramidal coordination by one hydroxyl oxygen atom (d(Zn—O) = 1.958(2) - 1.998(2) Å), two carboxylate oxygen atoms (d(Zn—O) = 1.963(2) - 2.090(2) Å) from two 2-hydroxy-3,5-dinitrobenzoate anions and two N atoms (d(Zn—N) = 2.080(3) - 2.172(3) Å) from the phen molecule. The carboxyl group of 2-hydroxy-3,5-dinitrobenzoate is deprotonated and is bidentate ligand in this compound. Adjacent Zn(II) ions are linked by a 2-hydroxy-3,5-dinitrobenzoate anion with the two carboxylate oxygen atoms bonding different Zn(II) ions. At the same time, each Zn(II) ion is connected to two adjacent Zn(II) ions through two 2-hydroxy-3,5-dinitrobenzoate anions to form a cage-like tetra-nuclear cluster. These tetra-nuclear molecules are stacked via π-π interactions between the phen ligands in an offset fashion (the shortest centroid to centroid distance being 3.638 Å), and form a 3D network.

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Table 1. Data collection and handling.

Crystal:	yellow block, size 0.250 × 0.310 × 0.322 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	14.08 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	67511, 19906
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 10709
$N(param)_{refined}$:	1694
Programs:	PLATON [1], DIAMOND [2], SHELXS-97, SHELXL-97 [3]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	0.2657	0.6800	0.2573	0.086
H(2)	4*e*	0.4050	0.7147	0.2401	0.111
H(3)	4*e*	0.5051	0.6478	0.2151	0.103
H(6)	4*e*	0.5370	0.5360	0.1928	0.102
H(7)	4*e*	0.4913	0.4365	0.1850	0.095
H(10)	4*e*	0.3685	0.3529	0.1945	0.093
H(11)	4*e*	0.2266	0.3274	0.2137	0.091
H(12)	4*e*	0.1383	0.4049	0.2395	0.067
H(17)	4*e*	–0.1323	0.6611	0.1338	0.052
H(19)	4*e*	–0.1903	0.5192	0.2025	0.048
H(20)	4*e*	–0.0166	0.5705	0.3289	0.070
H(21)	4*e*	–0.0932	0.6432	0.3637	0.084
H(22)	4*e*	–0.2240	0.6136	0.3916	0.081
H(25)	4*e*	–0.3406	0.5288	0.4035	0.076
H(26)	4*e*	–0.3856	0.4284	0.3959	0.080
H(29)	4*e*	–0.3599	0.3199	0.3681	0.088
H(30)	4*e*	–0.2728	0.2567	0.3326	0.092
H(31)	4*e*	–0.1468	0.2972	0.3055	0.080
H(36)	4*e*	0.1775	0.1884	0.2625	0.061
H(38)	4*e*	0.2765	0.3170	0.3376	0.057
H(39)	4*e*	0.2163	0.5925	0.4316	0.064
H(40)	4*e*	0.1408	0.6736	0.4586	0.080

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(41)	4*e*	–0.0059	0.6596	0.4753	0.077
H(44)	4*e*	–0.1390	0.5837	0.4828	0.080
H(45)	4*e*	–0.2004	0.4877	0.4736	0.082
H(48)	4*e*	–0.1855	0.3752	0.4478	0.086
H(49)	4*e*	–0.0971	0.3032	0.4203	0.079
H(50)	4*e*	0.0517	0.3280	0.4077	0.071
H(55)	4*e*	0.4811	0.3424	0.5371	0.054
H(57)	4*e*	0.5396	0.4645	0.4558	0.050
H(58)	4*e*	0.3683	0.4204	0.3353	0.083
H(59)	4*e*	0.4404	0.3457	0.3018	0.100
H(60)	4*e*	0.5690	0.3716	0.2713	0.098
H(63)	4*e*	0.6878	0.4546	0.2572	0.097
H(64)	4*e*	0.7365	0.5537	0.2633	0.096
H(67)	4*e*	0.7187	0.6648	0.2932	0.101
H(68)	4*e*	0.6374	0.7293	0.3299	0.110
H(69)	4*e*	0.5106	0.6908	0.3571	0.090
H(74)	4*e*	0.1938	0.7976	0.4158	0.063
H(76)	4*e*	0.0809	0.6753	0.3393	0.056
H(77)	4*e*	0.5467	0.5732	0.0913	0.065
H(78)	4*e*	0.4655	0.6556	0.1147	0.082
H(79)	4*e*	0.3196	0.6395	0.1321	0.084
H(82)	4*e*	0.1892	0.5633	0.1425	0.080
H(83)	4*e*	0.1334	0.4654	0.1365	0.086
H(86)	4*e*	0.1540	0.3515	0.1171	0.083
H(87)	4*e*	0.2467	0.2769	0.0937	0.087
H(88)	4*e*	0.3942	0.3024	0.0806	0.072
H(93)	4*e*	0.3433	0.2076	–0.0846	0.058
H(95)	4*e*	0.5301	0.3060	–0.0197	0.056
H(96)	4*e*	0.8717	0.6379	–0.0029	0.068
H(97)	4*e*	1.0060	0.6494	–0.0327	0.075
H(98)	4*e*	1.0658	0.5644	–0.0623	0.075
H(101)	4*e*	1.0532	0.4486	–0.0791	0.075
H(102)	4*e*	0.9763	0.3574	–0.0775	0.075
H(105)	4*e*	0.8348	0.2980	–0.0569	0.082
H(106)	4*e*	0.7039	0.2986	–0.0257	0.086
H(107)	4*e*	0.6536	0.3910	0.0003	0.069
H(112)	4*e*	0.8146	0.3314	0.2035	0.054
H(114)	4*e*	0.8718	0.4508	0.1208	0.047

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	4*e*		0.3057(3)	0.6518(2)	0.2471(1)	0.075(4)	0.071(3)	0.069(3)	–0.030(3)	0.013(3)	–0.010(2)
C(2)	4*e*		0.3894(3)	0.6728(2)	0.2368(1)	0.101(5)	0.102(4)	0.077(4)	–0.052(3)	0.012(3)	–0.003(3)
C(3)	4*e*		0.4489(3)	0.6333(2)	0.2221(1)	0.057(3)	0.132(5)	0.068(3)	–0.048(3)	0.004(3)	0.008(3)
C(4)	4*e*		0.4253(3)	0.5697(2)	0.2174(1)	0.033(3)	0.112(4)	0.045(2)	–0.010(3)	0.004(2)	0.012(2)
C(5)	4*e*		0.3398(2)	0.5519(2)	0.22914(9)	0.039(3)	0.070(3)	0.034(2)	–0.006(2)	–0.002(2)	0.007(2)
C(6)	4*e*		0.4803(3)	0.5241(3)	0.2008(1)	0.034(3)	0.164(5)	0.056(3)	0.010(3)	0.005(2)	0.021(3)
C(7)	4*e*		0.4535(3)	0.4648(2)	0.1963(1)	0.058(3)	0.134(5)	0.047(3)	0.039(3)	0.012(2)	0.013(3)
C(8)	4*e*		0.3667(3)	0.4438(2)	0.2087(1)	0.057(3)	0.086(3)	0.039(2)	0.026(3)	0.006(2)	0.011(2)
C(9)	4*e*		0.3106(2)	0.4880(2)	0.22526(9)	0.041(3)	0.071(3)	0.030(2)	0.009(2)	0.005(2)	0.004(2)
C(10)	4*e*		0.3330(3)	0.3836(2)	0.2050(1)	0.102(4)	0.078(3)	0.056(3)	0.042(3)	0.017(3)	0.002(3)
C(11)	4*e*		0.2487(3)	0.3681(2)	0.2164(1)	0.108(4)	0.053(3)	0.069(3)	0.012(3)	0.019(3)	0.003(2)
C(12)	4*e*		0.1964(3)	0.4149(2)	0.2322(1)	0.065(3)	0.051(3)	0.052(3)	0.002(2)	0.008(2)	0.002(2)
C(13)	4*e*		–0.0408(2)	0.5015(2)	0.24349(9)	0.039(2)	0.037(2)	0.040(2)	0.000(2)	0.009(2)	–0.002(2)
C(14)	4*e*		–0.0608(2)	0.5473(1)	0.21140(9)	0.039(2)	0.029(2)	0.036(2)	0.000(2)	0.003(2)	–0.000(2)
C(15)	4*e*		0.0075(2)	0.5895(2)	0.19883(9)	0.040(2)	0.037(2)	0.038(2)	0.004(2)	0.006(2)	–0.006(2)
C(16)	4*e*		–0.0250(2)	0.6314(2)	0.16877(9)	0.039(2)	0.036(2)	0.040(2)	–0.001(2)	0.013(2)	0.002(2)
C(17)	4*e*		–0.1139(2)	0.6321(2)	0.15271(9)	0.043(3)	0.049(2)	0.038(2)	0.011(2)	0.004(2)	0.006(2)
C(18)	4*e*		–0.1758(2)	0.5891(2)	0.16504(9)	0.032(2)	0.044(2)	0.039(2)	0.002(2)	0.002(2)	–0.002(2)
C(19)	4*e*		–0.1480(2)	0.5477(2)	0.19432(9)	0.039(2)	0.041(2)	0.040(2)	–0.003(2)	0.007(2)	–0.003(2)
C(20)	4*e*		–0.0695(3)	0.5583(2)	0.3400(1)	0.054(3)	0.060(3)	0.063(3)	0.003(2)	0.015(2)	0.000(2)
C(21)	4*e*		–0.1152(3)	0.6026(2)	0.3610(1)	0.076(4)	0.056(3)	0.080(3)	0.003(3)	0.015(3)	–0.011(2)
C(22)	4*e*		–0.1930(3)	0.5847(2)	0.3775(1)	0.071(3)	0.077(3)	0.054(3)	0.019(3)	0.007(2)	–0.012(2)
C(23)	4*e*		–0.2262(3)	0.5239(2)	0.3735(1)	0.054(3)	0.062(3)	0.040(2)	0.012(2)	0.010(2)	0.001(2)
C(24)	4*e*		–0.1757(2)	0.4830(2)	0.35179(9)	0.043(3)	0.057(3)	0.040(2)	0.008(2)	–0.002(2)	0.010(2)
C(25)	4*e*		–0.3064(3)	0.5017(2)	0.3895(1)	0.053(3)	0.089(3)	0.049(3)	0.022(3)	0.017(2)	–0.003(2)
C(26)	4*e*		–0.3334(3)	0.4419(2)	0.3848(1)	0.044(3)	0.100(4)	0.058(3)	0.010(3)	0.016(2)	0.013(3)

Table 3. Continued.

Atom	Site	Occ.	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(27)	4*e*		–0.2841(3)	0.3985(2)	0.3631(1)	0.044(3)	0.072(3)	0.050(3)	0.006(2)	0.006(2)	0.017(2)
C(28)	4*e*		–0.2055(3)	0.4198(2)	0.3465(1)	0.044(3)	0.061(3)	0.043(2)	0.011(2)	0.011(2)	0.014(2)
C(29)	4*e*		–0.3085(3)	0.3359(2)	0.3574(1)	0.054(3)	0.079(3)	0.090(4)	–0.008(3)	0.023(3)	0.027(3)
C(30)	4*e*		–0.2572(3)	0.2985(2)	0.3363(1)	0.071(4)	0.058(3)	0.106(4)	–0.010(3)	0.027(3)	0.018(3)
C(31)	4*e*		–0.1811(3)	0.3231(2)	0.3202(1)	0.066(3)	0.049(3)	0.086(3)	0.003(2)	0.017(3)	0.009(2)
C(32)	4*e*		0.1396(3)	0.3919(2)	0.3426(1)	0.048(3)	0.047(2)	0.043(2)	–0.002(2)	0.015(2)	0.005(2)
C(33)	4*e*		0.1474(2)	0.3365(2)	0.31661(9)	0.046(3)	0.042(2)	0.040(2)	0.000(2)	0.016(2)	0.002(2)
C(34)	4*e*		0.0734(2)	0.3161(2)	0.29001(9)	0.049(3)	0.039(2)	0.039(2)	–0.001(2)	0.016(2)	0.005(2)
C(35)	4*e*		0.0888(3)	0.2572(2)	0.2713(1)	0.054(3)	0.041(2)	0.045(2)	–0.001(2)	0.011(2)	0.006(2)
C(36)	4*e*		0.1698(3)	0.2256(2)	0.2757(1)	0.063(3)	0.040(2)	0.052(2)	0.006(2)	0.023(2)	0.001(2)
C(37)	4*e*		0.2403(3)	0.2493(2)	0.3000(1)	0.046(3)	0.047(2)	0.053(2)	0.011(2)	0.017(2)	0.002(2)
C(38)	4*e*		0.2287(2)	0.3029(2)	0.3206(1)	0.045(3)	0.049(2)	0.051(2)	–0.005(2)	0.015(2)	0.005(2)
C(39)	4*e*		0.1569(3)	0.5860(2)	0.43844(9)	0.062(3)	0.054(3)	0.044(2)	–0.008(2)	0.000(2)	0.008(2)
C(40)	4*e*		0.1118(3)	0.6352(2)	0.4547(1)	0.089(4)	0.051(3)	0.061(3)	–0.004(3)	0.009(3)	0.007(2)
C(41)	4*e*		0.0254(3)	0.6267(2)	0.4649(1)	0.090(4)	0.057(3)	0.047(3)	0.020(3)	0.009(2)	0.000(2)
C(42)	4*e*		–0.0168(3)	0.5684(2)	0.45981(9)	0.053(3)	0.064(3)	0.037(2)	0.015(2)	0.004(2)	0.004(2)
C(43)	4*e*		0.0335(2)	0.5210(2)	0.44311(9)	0.039(2)	0.057(3)	0.033(2)	0.002(2)	–0.004(2)	0.007(2)
C(44)	4*e*		–0.1055(3)	0.5531(2)	0.4712(1)	0.059(3)	0.100(4)	0.042(2)	0.028(3)	0.009(2)	0.003(2)
C(45)	4*e*		–0.1422(3)	0.4957(2)	0.4657(1)	0.046(3)	0.116(4)	0.045(3)	0.007(3)	0.010(2)	0.015(3)
C(46)	4*e*		–0.0939(3)	0.4473(2)	0.4482(1)	0.037(3)	0.082(3)	0.041(2)	0.002(2)	0.003(2)	0.015(2)
C(47)	4*e*		–0.0046(2)	0.4592(2)	0.43820(9)	0.031(2)	0.063(3)	0.035(2)	0.006(2)	0.001(2)	0.009(2)
C(48)	4*e*		–0.1270(3)	0.3860(2)	0.4414(1)	0.049(3)	0.098(4)	0.068(3)	–0.023(3)	0.001(2)	0.024(3)
C(49)	4*e*		–0.0742(3)	0.3431(2)	0.4258(1)	0.063(3)	0.065(3)	0.069(3)	–0.020(2)	0.000(2)	0.015(2)
C(50)	4*e*		0.0153(3)	0.3586(2)	0.4177(1)	0.052(3)	0.060(3)	0.066(3)	–0.002(2)	0.007(2)	0.003(2)
C(51)	4*e*		0.3830(2)	0.4906(2)	0.42210(9)	0.041(3)	0.041(2)	0.038(2)	–0.002(2)	0.009(2)	–0.004(2)
C(52)	4*e*		0.4047(2)	0.4468(2)	0.45511(9)	0.039(2)	0.039(2)	0.033(2)	0.004(2)	0.007(2)	–0.002(2)
C(53)	4*e*		0.3360(2)	0.4111(2)	0.47206(9)	0.035(2)	0.044(2)	0.036(2)	–0.004(2)	0.008(2)	–0.001(2)
C(54)	4*e*		0.3710(2)	0.3734(2)	0.50425(9)	0.039(2)	0.041(2)	0.038(2)	0.000(2)	0.008(2)	0.005(2)
C(55)	4*e*		0.4620(2)	0.3681(2)	0.51649(9)	0.050(3)	0.045(2)	0.040(2)	0.006(2)	0.000(2)	0.006(2)
C(56)	4*e*		0.5247(2)	0.4015(2)	0.49754(9)	0.030(2)	0.050(2)	0.044(2)	–0.004(2)	0.001(2)	0.003(2)
C(57)	4*e*		0.4962(2)	0.4412(2)	0.46765(9)	0.036(2)	0.049(2)	0.041(2)	–0.002(2)	0.009(2)	–0.001(2)
C(58)	4*e*		0.4207(3)	0.4314(2)	0.3235(1)	0.075(3)	0.059(3)	0.074(3)	0.003(3)	0.011(3)	–0.003(2)
C(59)	4*e*		0.4634(3)	0.3862(2)	0.3032(1)	0.110(5)	0.066(3)	0.074(3)	0.011(3)	0.013(3)	–0.006(3)
C(60)	4*e*		0.5398(3)	0.4015(2)	0.2852(1)	0.106(4)	0.081(4)	0.059(3)	0.038(3)	0.004(3)	–0.008(3)
C(61)	4*e*		0.5741(3)	0.4631(2)	0.2880(1)	0.063(3)	0.079(3)	0.052(3)	0.018(3)	0.005(2)	0.004(2)
C(62)	4*e*		0.5268(3)	0.5052(2)	0.3093(1)	0.055(3)	0.067(3)	0.039(2)	0.020(2)	–0.003(2)	0.003(2)
C(63)	4*e*		0.6553(3)	0.4829(2)	0.2711(1)	0.074(4)	0.109(4)	0.060(3)	0.032(3)	0.002(3)	0.000(3)
C(64)	4*e*		0.6845(3)	0.5418(2)	0.2751(1)	0.048(3)	0.140(5)	0.054(3)	0.010(3)	0.012(2)	0.009(3)
C(65)	4*e*		0.6385(3)	0.5881(2)	0.2972(1)	0.055(3)	0.098(4)	0.045(3)	0.004(3)	0.005(2)	0.015(2)
C(66)	4*e*		0.5601(3)	0.5684(2)	0.3143(1)	0.052(3)	0.069(3)	0.047(2)	0.011(2)	0.000(2)	0.015(2)
C(67)	4*e*		0.6667(3)	0.6499(2)	0.3039(1)	0.077(4)	0.101(4)	0.076(3)	–0.024(3)	0.020(3)	0.021(3)
C(68)	4*e*		0.6192(3)	0.6881(2)	0.3257(1)	0.106(5)	0.081(4)	0.091(4)	–0.028(3)	0.030(3)	0.014(3)
C(69)	4*e*		0.5427(3)	0.6645(2)	0.3418(1)	0.082(4)	0.065(3)	0.079(3)	–0.007(3)	0.021(3)	0.003(3)
C(70)	4*e*		0.2156(3)	0.6019(2)	0.32770(9)	0.046(3)	0.046(2)	0.037(2)	0.000(2)	0.009(2)	–0.000(2)
C(71)	4*e*		0.2133(2)	0.6562(2)	0.35462(9)	0.051(3)	0.040(2)	0.034(2)	0.001(2)	0.002(2)	–0.003(2)
C(72)	4*e*		0.2925(2)	0.6754(2)	0.37869(9)	0.045(3)	0.041(2)	0.042(2)	–0.005(2)	0.007(2)	0.004(2)
C(73)	4*e*		0.2806(3)	0.7324(2)	0.39966(9)	0.056(3)	0.042(2)	0.042(2)	–0.006(2)	0.000(2)	–0.001(2)
C(74)	4*e*		0.1987(3)	0.7622(2)	0.4006(1)	0.074(3)	0.038(2)	0.047(2)	–0.006(2)	0.017(2)	–0.000(2)
C(75)	4*e*		0.1232(3)	0.7397(2)	0.3789(1)	0.050(3)	0.043(2)	0.045(2)	0.003(2)	0.011(2)	–0.000(2)
C(76)	4*e*		0.1315(3)	0.6883(2)	0.35517(9)	0.055(3)	0.044(2)	0.042(2)	0.003(2)	0.006(2)	0.002(2)
C(77)	4*e*		0.4878(3)	0.5662(2)	0.0986(1)	0.056(3)	0.059(3)	0.048(2)	–0.007(2)	0.001(2)	0.009(2)
C(78)	4*e*		0.4392(3)	0.6161(2)	0.1126(1)	0.091(4)	0.046(3)	0.068(3)	–0.002(3)	0.008(3)	0.000(2)
C(79)	4*e*		0.3528(3)	0.6062(2)	0.1232(1)	0.093(4)	0.059(3)	0.059(3)	0.028(3)	0.008(3)	0.000(2)
C(80)	4*e*		0.3136(3)	0.5471(2)	0.1210(1)	0.049(3)	0.060(3)	0.045(2)	0.018(2)	0.003(2)	0.006(2)
C(81)	4*e*		0.3673(2)	0.4993(2)	0.10654(9)	0.035(2)	0.057(2)	0.034(2)	0.005(2)	–0.001(2)	0.006(2)
C(82)	4*e*		0.2247(3)	0.5322(2)	0.1325(1)	0.053(3)	0.096(4)	0.051(3)	0.035(3)	0.013(2)	0.000(3)
C(83)	4*e*		0.1919(3)	0.4737(2)	0.1291(1)	0.039(3)	0.123(4)	0.056(3)	0.020(3)	0.014(2)	0.014(3)
C(84)	4*e*		0.2434(3)	0.4240(2)	0.1145(1)	0.036(3)	0.082(3)	0.044(2)	–0.003(2)	0.003(2)	0.010(2)
C(85)	4*e*		0.3321(2)	0.4362(2)	0.10398(9)	0.028(2)	0.061(3)	0.036(2)	0.004(2)	0.000(2)	0.002(2)
C(86)	4*e*		0.2125(3)	0.3620(2)	0.1106(1)	0.040(3)	0.101(4)	0.067(3)	–0.018(3)	0.008(2)	0.018(3)
C(87)	4*e*		0.2677(3)	0.3177(2)	0.0972(1)	0.052(3)	0.083(3)	0.083(3)	–0.023(3)	0.002(3)	0.002(3)
C(88)	4*e*		0.3560(3)	0.3335(2)	0.0888(1)	0.054(3)	0.056(3)	0.068(3)	–0.006(2)	0.001(2)	0.000(2)
C(89)	4*e*		0.4313(2)	0.3955(2)	0.00601(9)	0.033(2)	0.046(2)	0.039(2)	0.001(2)	0.009(2)	0.004(2)
C(90)	4*e*		0.4041(2)	0.3453(2)	–0.02234(9)	0.037(2)	0.039(2)	0.037(2)	0.002(2)	0.004(2)	–0.002(2)
C(91)	4*e*		0.3120(2)	0.3393(2)	–0.03878(9)	0.047(3)	0.041(2)	0.037(2)	0.002(2)	0.003(2)	0.000(2)
C(92)	4*e*		0.2924(2)	0.2819(2)	–0.05890(9)	0.043(3)	0.042(2)	0.041(2)	–0.002(2)	–0.004(2)	0.002(2)
C(93)	4*e*		0.3585(3)	0.2418(2)	–0.06901(9)	0.067(3)	0.035(2)	0.042(2)	0.005(2)	0.008(2)	0.003(2)
C(94)	4*e*		0.4485(3)	0.2530(2)	–0.0556(1)	0.053(3)	0.039(2)	0.049(2)	0.004(2)	0.010(2)	0.002(2)

Table 3. Continued.

Atom	Site	Occ.	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(95)	4*e*		0.4703(2)	0.3020(2)	–0.03083(9)	0.042(3)	0.049(2)	0.050(2)	0.000(2)	0.008(2)	0.005(2)
C(96)	4*e*		0.8953(3)	0.6036(2)	–0.0151(1)	0.055(3)	0.062(3)	0.053(3)	–0.005(2)	0.012(2)	0.012(2)
C(97)	4*e*		0.9763(3)	0.6109(2)	–0.0328(1)	0.062(3)	0.065(3)	0.062(3)	–0.016(2)	0.012(2)	0.005(2)
C(98)	4*e*		1.0115(3)	0.5604(2)	–0.0504(1)	0.046(3)	0.095(4)	0.048(3)	–0.017(2)	0.013(2)	0.017(2)
C(99)	4*e*		0.9665(2)	0.5030(2)	–0.05056(9)	0.040(3)	0.073(3)	0.038(2)	0.009(2)	0.007(2)	–0.001(2)
C(100)	4*e*		0.8843(2)	0.5005(2)	–0.03280(9)	0.038(2)	0.053(2)	0.036(2)	0.005(2)	0.005(2)	0.001(2)
C(101)	4*e*		0.9985(3)	0.4473(2)	–0.0672(1)	0.041(3)	0.100(4)	0.045(2)	0.005(2)	0.008(2)	0.002(2)
C(102)	4*e*		0.9527(3)	0.3929(2)	–0.0665(1)	0.052(3)	0.086(3)	0.051(3)	0.018(2)	0.008(2)	–0.012(2)
C(103)	4*e*		0.8664(3)	0.3893(2)	–0.0487(1)	0.041(3)	0.067(3)	0.045(2)	0.011(2)	0.002(2)	–0.005(2)
C(104)	4*e*		0.8337(2)	0.4432(2)	–0.03163(9)	0.036(2)	0.060(3)	0.036(2)	0.008(2)	0.005(2)	–0.003(2)
C(105)	4*e*		0.8153(3)	0.3348(2)	–0.0460(1)	0.066(3)	0.060(3)	0.081(3)	0.008(2)	0.021(3)	–0.026(2)
C(106)	4*e*		0.7376(3)	0.3351(2)	–0.0277(1)	0.075(3)	0.053(3)	0.091(3)	–0.006(2)	0.023(3)	–0.023(2)
C(107)	4*e*		0.7080(3)	0.3911(2)	–0.0118(1)	0.048(3)	0.063(3)	0.064(3)	–0.004(2)	0.015(2)	–0.012(2)
C(108)	4*e*		0.7176(2)	0.4661(1)	0.08172(9)	0.035(2)	0.033(2)	0.038(2)	0.005(2)	0.002(2)	–0.006(2)
C(109)	4*e*		0.7382(2)	0.4277(1)	0.11706(9)	0.038(2)	0.032(2)	0.037(2)	–0.000(2)	0.006(2)	–0.003(2)
C(110)	4*e*		0.6698(2)	0.3920(1)	0.13518(9)	0.037(2)	0.034(2)	0.038(2)	0.000(2)	0.008(2)	–0.003(2)
C(111)	4*e*		0.7046(2)	0.3574(2)	0.16829(9)	0.039(2)	0.042(2)	0.041(2)	–0.003(2)	0.011(2)	0.005(2)
C(112)	4*e*		0.7955(2)	0.3556(2)	0.18227(9)	0.051(3)	0.050(2)	0.034(2)	0.009(2)	0.006(2)	0.003(2)
C(113)	4*e*		0.8571(2)	0.3909(2)	0.16386(9)	0.028(2)	0.052(2)	0.040(2)	0.002(2)	0.001(2)	–0.000(2)
C(114)	4*e*		0.8287(2)	0.4267(2)	0.13223(9)	0.038(2)	0.041(2)	0.038(2)	–0.002(2)	0.007(2)	–0.001(2)
N(1)	4*e*		0.2809(2)	0.5926(1)	0.24293(8)	0.048(2)	0.061(2)	0.046(2)	–0.007(2)	0.002(2)	–0.002(2)
N(2)	4*e*		0.2269(2)	0.4726(1)	0.23715(7)	0.045(2)	0.053(2)	0.034(2)	0.001(2)	0.005(2)	0.004(2)
N(3)	4*e*		0.0373(2)	0.6778(1)	0.15447(8)	0.045(2)	0.048(2)	0.046(2)	–0.002(2)	0.008(2)	0.006(2)
N(4)	4*e*		–0.2697(2)	0.5884(1)	0.14810(8)	0.041(2)	0.058(2)	0.047(2)	0.005(2)	0.003(2)	0.001(2)
N(5)	4*e*		–0.0980(2)	0.4999(1)	0.33514(8)	0.045(2)	0.050(2)	0.052(2)	–0.001(2)	0.005(2)	0.011(2)
N(6)	4*e*		–0.1556(2)	0.3822(1)	0.32502(8)	0.049(2)	0.053(2)	0.053(2)	0.007(2)	0.011(2)	0.010(2)
N(7)	4*e*		0.0150(2)	0.2281(1)	0.24596(9)	0.075(3)	0.052(2)	0.057(2)	–0.001(2)	0.003(2)	–0.002(2)
N(8)	4*e*		0.3261(2)	0.2158(2)	0.3045(1)	0.056(3)	0.061(2)	0.091(3)	0.013(2)	0.023(2)	0.004(2)
N(9)	4*e*		0.1195(2)	0.5305(1)	0.43228(7)	0.040(2)	0.049(2)	0.043(2)	–0.002(2)	0.001(2)	0.007(2)
N(10)	4*e*		0.0500(2)	0.4150(1)	0.42387(8)	0.039(2)	0.052(2)	0.046(2)	–0.005(2)	0.001(2)	0.005(2)
N(11)	4*e*		0.3068(2)	0.3370(1)	0.52524(9)	0.049(2)	0.064(2)	0.053(2)	–0.006(2)	0.010(2)	0.016(2)
N(12)	4*e*		0.6218(2)	0.3937(2)	0.50774(9)	0.041(2)	0.079(3)	0.056(2)	–0.002(2)	0.000(2)	0.013(2)
N(13)	4*e*		0.4503(2)	0.4897(1)	0.32689(8)	0.051(2)	0.054(2)	0.055(2)	0.006(2)	0.003(2)	0.006(2)
N(14)	4*e*		0.5134(2)	0.6066(1)	0.33646(9)	0.066(3)	0.050(2)	0.058(2)	0.005(2)	0.011(2)	0.011(2)
N(15)	4*e*		0.0349(2)	0.7694(1)	0.38064(9)	0.065(3)	0.047(2)	0.075(3)	0.004(2)	0.026(2)	–0.006(2)
N(16)	4*e*		0.3590(3)	0.7590(1)	0.42372(9)	0.080(3)	0.049(2)	0.071(3)	–0.016(2)	0.000(2)	–0.007(2)
N(17)	4*e*		0.4528(2)	0.5092(1)	0.09524(7)	0.036(2)	0.051(2)	0.039(2)	–0.004(2)	0.001(2)	0.000(2)
N(18)	4*e*		0.3878(2)	0.3915(1)	0.09202(8)	0.032(2)	0.055(2)	0.049(2)	–0.002(2)	0.001(2)	–0.002(2)
N(19)	4*e*		0.1973(2)	0.2668(1)	–0.07173(9)	0.062(3)	0.046(2)	0.065(2)	–0.000(2)	–0.016(2)	–0.004(2)
N(20)	4*e*		0.5191(2)	0.2122(1)	–0.06815(9)	0.068(3)	0.046(2)	0.072(2)	0.009(2)	0.026(2)	–0.005(2)
N(21)	4*e*		0.8504(2)	0.5500(1)	–0.01489(8)	0.041(2)	0.052(2)	0.045(2)	–0.000(2)	0.006(2)	0.007(2)
N(22)	4*e*		0.7546(2)	0.4440(1)	–0.01332(8)	0.040(2)	0.048(2)	0.046(2)	0.000(2)	0.008(2)	0.001(2)
N(23)	4*e*	0.629(7)	0.6415(2)	0.3208(2)	0.18955(9)	0.061(5)	0.056(5)	0.065(4)	–0.002(4)	0.013(4)	0.024(3)
N(23A)	4*e*	0.371	0.6415(2)	0.3208(2)	0.18955(9)	0.061(3)	0.056(4)	0.065(4)	–0.002(2)	0.013(2)	0.024(2)
N(24)	4*e*		0.9541(2)	0.3890(2)	0.17705(8)	0.047(2)	0.066(2)	0.044(2)	0.001(2)	0.005(2)	–0.006(2)
O(1)	4*e*		0.0366(2)	0.4992(1)	0.26239(6)	0.040(2)	0.053(2)	0.043(1)	–0.003(1)	–0.004(1)	0.010(1)
O(2)	4*e*		–0.1039(2)	0.4658(1)	0.25189(6)	0.043(2)	0.051(2)	0.054(2)	–0.005(1)	0.005(1)	0.016(1)
O(3)	4*e*		0.0908(2)	0.5919(1)	0.21251(7)	0.041(2)	0.063(2)	0.059(2)	–0.009(1)	–0.005(1)	0.018(1)
O(4)	4*e*		0.1182(2)	0.6659(1)	0.15326(8)	0.052(2)	0.066(2)	0.096(2)	–0.002(2)	0.026(2)	0.022(2)
O(5)	4*e*		0.0051(2)	0.7279(1)	0.14360(8)	0.061(2)	0.055(2)	0.103(2)	–0.004(2)	0.000(2)	0.034(2)
O(6)	4*e*		–0.2940(2)	0.6290(1)	0.12480(7)	0.052(2)	0.090(2)	0.070(2)	–0.004(2)	–0.016(2)	0.034(2)
O(7)	4*e*		–0.3214(2)	0.5473(1)	0.15796(7)	0.042(2)	0.068(2)	0.077(2)	–0.010(1)	–0.002(2)	0.010(2)
O(8)	4*e*		0.0725(2)	0.4284(1)	0.33853(6)	0.056(2)	0.047(2)	0.050(2)	0.012(1)	0.001(1)	–0.003(1)
O(9)	4*e*		0.2035(2)	0.3985(1)	0.36892(7)	0.041(2)	0.059(2)	0.059(2)	0.001(1)	0.001(1)	–0.012(1)
O(10)	4*e*		–0.0003(2)	0.3461(1)	0.28223(6)	0.051(2)	0.050(2)	0.055(2)	0.010(1)	0.002(1)	–0.004(1)
O(11)	4*e*		–0.0643(2)	0.2356(1)	0.25325(8)	0.069(2)	0.071(2)	0.097(2)	–0.017(2)	0.006(2)	–0.015(2)
O(12)	4*e*		0.0387(2)	0.1957(2)	0.21978(8)	0.108(3)	0.108(3)	0.078(2)	0.007(2)	–0.003(2)	–0.047(2)
O(13)	4*e*		0.3315(2)	0.1647(1)	0.28785(8)	0.084(2)	0.070(2)	0.100(2)	0.034(2)	0.022(2)	–0.014(2)
O(14)	4*e*		0.3908(2)	0.2385(1)	0.3244(1)	0.056(2)	0.077(2)	0.148(3)	0.017(2)	0.007(2)	–0.011(2)
O(15)	4*e*		0.3036(2)	0.4982(1)	0.40670(6)	0.039(2)	0.066(2)	0.053(2)	–0.006(1)	–0.005(1)	0.018(1)
O(16)	4*e*		0.4491(2)	0.5201(1)	0.40944(6)	0.038(2)	0.063(2)	0.058(2)	–0.001(1)	0.008(1)	0.020(1)
O(17)	4*e*		0.2503(2)	0.4120(1)	0.46165(6)	0.033(2)	0.065(2)	0.055(2)	–0.005(1)	0.005(1)	0.011(1)
O(18)	4*e*		0.2402(2)	0.3134(1)	0.50907(9)	0.074(2)	0.097(3)	0.102(3)	–0.033(2)	0.008(2)	0.024(2)
O(19)	4*e*		0.3241(2)	0.3330(2)	0.55999(8)	0.094(3)	0.134(3)	0.064(2)	–0.019(2)	0.022(2)	0.029(2)
O(20)	4*e*		0.6469(2)	0.3524(1)	0.53038(8)	0.050(2)	0.106(2)	0.085(2)	0.001(2)	–0.011(2)	0.042(2)
O(21)	4*e*		0.6757(2)	0.4275(2)	0.49315(9)	0.035(2)	0.118(3)	0.119(3)	–0.010(2)	–0.001(2)	0.051(2)
O(22)	4*e*		0.2830(2)	0.5659(1)	0.32747(6)	0.053(2)	0.048(2)	0.048(2)	0.012(1)	–0.005(1)	–0.006(1)
O(23)	4*e*		0.1447(2)	0.5951(1)	0.30453(6)	0.038(2)	0.069(2)	0.052(2)	0.005(1)	0.000(1)	–0.021(1)

Table 3. Continued.

Atom	Site	Occ.	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(24)	4*e*		0.3683(2)	0.6462(1)	0.38263(7)	0.049(2)	0.054(2)	0.064(2)	0.002(1)	–0.006(1)	–0.015(1)
O(25)	4*e*		0.4368(2)	0.7499(1)	0.41493(9)	0.060(2)	0.074(2)	0.107(2)	–0.024(2)	0.009(2)	–0.016(2)
O(26)	4*e*		0.3409(2)	0.7913(2)	0.45095(9)	0.103(3)	0.108(3)	0.107(3)	–0.012(2)	–0.001(2)	–0.067(2)
O(27)	4*e*		0.0323(2)	0.8188(1)	0.39887(8)	0.083(2)	0.074(2)	0.105(2)	0.010(2)	0.026(2)	–0.038(2)
O(28)	4*e*		–0.0335(2)	0.7452(1)	0.36490(9)	0.059(2)	0.076(2)	0.125(3)	0.007(2)	0.007(2)	–0.024(2)
O(29)	4*e*		0.6371(2)	0.4717(1)	0.06617(6)	0.036(2)	0.065(2)	0.046(2)	–0.001(1)	0.003(1)	0.014(1)
O(30)	4*e*		0.7833(2)	0.4926(1)	0.06740(6)	0.033(2)	0.057(2)	0.047(2)	–0.006(1)	0.007(1)	0.011(1)
O(31)	4*e*		0.5848(2)	0.3906(1)	0.12352(6)	0.030(2)	0.062(2)	0.058(2)	–0.007(1)	0.002(1)	0.014(1)
O(32)	4*e*	0.629	0.5735(4)	0.3000(3)	0.1750(2)	0.110(6)	0.148(7)	0.110(5)	–0.088(5)	–0.020(4)	0.049(5)
O(33)	4*e*	0.629	0.6623(3)	0.3161(3)	0.2250(1)	0.102(5)	0.126(5)	0.052(3)	–0.031(4)	0.024(3)	0.019(3)
O(34)	4*e*		0.9820(2)	0.3465(1)	0.19811(8)	0.061(2)	0.097(2)	0.069(2)	0.014(2)	–0.013(2)	0.026(2)
O(35)	4*e*		1.0039(2)	0.4301(1)	0.16629(8)	0.040(2)	0.085(2)	0.085(2)	–0.010(2)	0.000(2)	0.007(2)
O(36)	4*e*		0.5055(2)	0.3868(1)	0.02713(6)	0.032(2)	0.065(2)	0.047(2)	0.012(1)	–0.002(1)	–0.012(1)
O(37)	4*e*		0.3828(2)	0.4433(1)	0.01006(6)	0.044(2)	0.043(2)	0.050(2)	0.009(1)	–0.003(1)	–0.006(1)
O(38)	4*e*		0.2502(2)	0.3807(1)	–0.03703(7)	0.044(2)	0.054(2)	0.064(2)	0.012(1)	–0.007(1)	–0.010(1)
O(39)	4*e*		0.1357(2)	0.2834(1)	–0.05229(9)	0.049(2)	0.090(2)	0.125(3)	0.001(2)	–0.001(2)	–0.045(2)
O(40)	4*e*		0.1835(2)	0.2358(1)	–0.10104(8)	0.092(3)	0.079(2)	0.075(2)	–0.017(2)	–0.019(2)	–0.026(2)
O(41)	4*e*		0.4970(2)	0.1708(1)	–0.09149(8)	0.087(2)	0.061(2)	0.092(2)	0.009(2)	0.027(2)	–0.024(2)
O(42)	4*e*		0.5985(2)	0.2202(1)	–0.0549(1)	0.053(2)	0.083(2)	0.151(3)	0.009(2)	0.013(2)	–0.043(2)
O(32A)	4*e*	0.371	0.5672(5)	0.3353(4)	0.1930(2)	0.045(7)	0.080(7)	0.112(9)	0.015(5)	0.052(6)	0.044(6)
O(33A)	4*e*	0.371	0.6671(6)	0.2662(4)	0.1975(3)	0.081(7)	0.072(7)	0.18(1)	0.022(5)	0.057(7)	0.076(7)
Zn(1)	4*e*		0.15452(3)	0.54911(2)	0.25650(1)	0.0356(3)	0.0493(3)	0.0402(2)	0.0001(2)	0.0009(2)	–0.0034(2)
Zn(2)	4*e*		–0.04227(3)	0.42740(2)	0.30267(1)	0.0455(3)	0.0471(3)	0.0451(3)	0.0076(2)	0.0085(2)	0.0082(2)
Zn(3)	4*e*		0.18573(3)	0.44970(2)	0.41464(1)	0.0338(3)	0.0543(3)	0.0483(3)	–0.0001(2)	0.0014(2)	0.0013(2)
Zn(4)	4*e*		0.40049(3)	0.56394(2)	0.36067(1)	0.0481(3)	0.0498(3)	0.0482(3)	0.0056(2)	0.0008(2)	0.0060(2)
Zn(5)	4*e*		0.51811(3)	0.42902(2)	0.07773(1)	0.0299(3)	0.0562(3)	0.0439(3)	0.0003(2)	0.0036(2)	–0.0030(2)
Zn(6)	4*e*		0.73644(3)	0.53167(2)	0.01639(1)	0.0389(3)	0.0445(3)	0.0416(2)	0.0060(2)	0.0054(2)	0.0031(2)

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