Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis(2-chlorobenzoato-κ^2O,O')bis[methyl *N*-(3-pyridyl)carbamato-κN]zinc(II)

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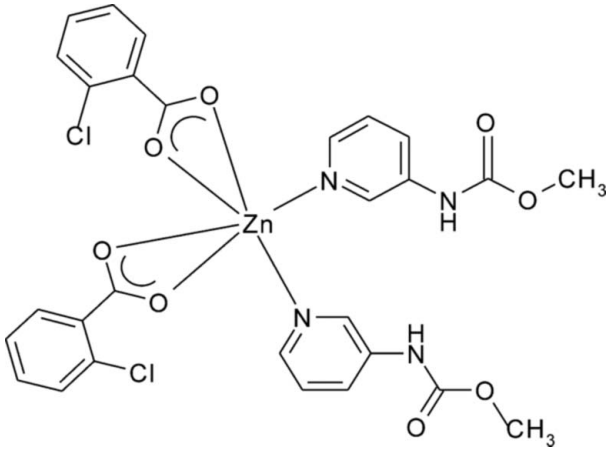
Received 16 April 2007; accepted 20 April 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.037; wR factor = 0.096; data-to-parameter ratio = 17.5.

In the title compound, $[Zn(C_7H_4ClO_2)_2(C_7H_8N_2O_2)_2]$, the Zn^{II} ion is coordinated by two N atoms from two methyl *N*-(3-pyridyl)carbamate ligands and four O atoms from two bidentate 2-chlorobenzoate anions in a pseudo-octahedral geometry. The Zn—O distances are in the range 2.0484 (16)–2.380 (2) Å, and the Zn—N distance is 2.1012 (18) Å. The molecules are linked into a chain along the *c* axis by N—H···O and C—H···Cl hydrogen bonds.

Related literature

The Zn^{II} atom adopts a tetrahedral geometry in related complexes with methyl *N*-(3-pyridyl)carbamate ligands (Zeleňák *et al.*, 2004, 2007).



Experimental

Crystal data

$[Zn(C_7H_4ClO_2)_2(C_7H_8N_2O_2)_2]$
M_r = 680.78
Orthorhombic, *Pccn*
a = 15.444 (3) Å
b = 13.650 (3) Å
c = 13.627 (3) Å
V = 2872.7 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.10 mm^{-1}
T = 293 (2) K
0.70 × 0.50 × 0.40 mm

Data collection

Siemens P4 diffractometer
Absorption correction: ψ scan (*XEMP*; Siemens, 1994)
T_{min} = 0.304, T_{max} = 0.339
(expected range = 0.578–0.644)
4275 measured reflections
3429 independent reflections
2566 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
3 standard reflections
every 97 reflections
intensity decay: 4.3%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.096
S = 1.02
3429 reflections
196 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O2^{i}	0.86	2.02	2.812 (3)	153
C8—H8···Cl^{i}	0.93	2.82	3.723 (2)	165

Symmetry code: (i) $x, -y+\frac{1}{2}, z+\frac{1}{2}$.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *enCIFer* (Allen *et al.*, 2004).

The authors thank the Slovak Ministry of Education (VEGA Project Nos. 1/2474/05 and 1/2452/05) and the Research and Development Support Agency (APVT-20–005504) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2367).

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  doi:10.1107/S1600536807019836

supplementary materials

Acta Cryst. (2007). E**63**, m1520 [**doi:10.1107/S1600536807019836**]

Bis(2-chlorobenzoato-κ^2O,O')bis[methyl *N*-(3-pyridyl)carbamato-κN]zinc(II)

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Comment

Compound (I) is a mononuclear zinc(II) compound (Fig. 1). The Zn^{II} atom exists in a pseudo-octahedral coordination environment, created by two pyridine N atoms from two methyl-3-pyridylcarbamate (mpc) ligands and four O atoms from two bidentate 2-chlorobenzoate anions. But a tetrahedral arrangement is found for the Zn^{II} atoms in related structures, *viz*. [Zn(benzoato)$_2$(mpc)$_2$] (Zeleňák *et al.*, 2004) and [Zn(cinnamato)$_2$(mpc)] (Zeleňák *et al.*, 2007). The mean Zn–N distances of 2.032 Å (in the former) and 2.021 Å (in the latter) are shorter than that in compound (I) (2.181 Å), as expected for a tetrahedral arrangement. The Zn—O distances are in the range 2.0484 (16)–2.380 (2) Å, and the Zn—N and Zn—Cl distances are 2.1012 (18) and 2.546 (2) Å, respectively.

In the crystal structure of (I), the molecules are linked by N2–H2···O2^{ii} and C8–H8···Cl^{ii} [symmetry code: (ii) x, 1/2 − y, 1/2 + z] hydrogen bonds (Table 1), forming a chain along the c axis (Fig. 2).

Experimental

A mixture of $ZnCO_3$ (0.4180 g, 3.33 mmol) and 2-chlorobenzoic acid (1.0438 g, 3.33 mmol) in ethanol (50 ml) was stirred at room temperature for 1 h and then filtered. An ethanol solution (50 ml) of methyl-3-pyridylcarbamate (1.0143 g, 3.33 mmol) was added to the filtrate and the mixture was stirred for 3 h. The resulting clear solution was allowed to stand in air at room temperature for two weeks, yielding colourless crystals of (I). The crystals were separated and dried at ambient temperature.

Refinement

H atoms were placed in calculated positions [N–H = 0.86 Å and C–H = 0.93 (aromatic) or 0.96 Å (methyl)] and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Figures

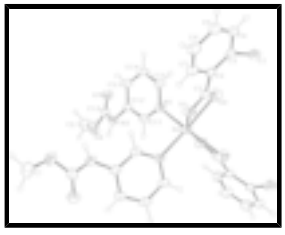

Fig. 1. The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Unlabelled atoms are related to labelled atoms by (−x + 1/2, −y + 1/2, z).

Fig. 2. The crystal packing of (I), viewed along the *a* axis. [symmetry code: (ii) *x*, 1/2 − *y*, 1/2 + *z*.]

Bis(2-chlorobenzoato-κ^2*O*,*O*')bis[methyl N-(3-pyridyl)carbamato-κ*N*]zinc(II)

Crystal data

$[Zn(C_7H_4ClO_2)_2(C_7H_8N_2O_2)_2]$	F_{000} = 1392
M_r = 680.78	D_x = 1.574 Mg m^{-3}
Orthorhombic, *Pccn*	Mo *K*α radiation λ = 0.71069 Å
Hall symbol: -P 2ab 2ac	Cell parameters from 25 reflections
a = 15.444 (3) Å	θ = 4.5–8.6º
b = 13.650 (3) Å	μ = 1.10 mm^{-1}
c = 13.627 (3) Å	*T* = 293 (2) K
V = 2872.7 (10) Å^3	Prism, colourless
Z = 4	0.70 × 0.50 × 0.40 mm

Data collection

Siemens P4 diffractometer	R_{int} = 0.021
Radiation source: fine-focus sealed tube	θ_{max} = 28.0º
Monochromator: graphite	θ_{min} = 2.5º
T = 293(2) K	*h* = −1→20
2θ/ω scans	*k* = −1→18
Absorption correction: ψ scan (XEMP; Siemens, 1994)	*l* = −1→18
T_{min} = 0.304, T_{max} = 0.339	3 standard reflections
4275 measured reflections	every 97 reflections
3429 independent reflections	intensity decay: 4.3%
2566 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.037	H-atom parameters constrained
$wR(F^2)$ = 0.096	$w = 1/[\sigma^2(F_o^2) + (0.0406P)^2 + 0.9964P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.02	$(\Delta/\sigma)_{max}$ = 0.001
3429 reflections	$\Delta\rho_{max}$ = 0.27 e Å^{-3}
196 parameters	$\Delta\rho_{min}$ = −0.36 e Å^{-3}

Primary atom site location: structure-invariant direct methods

Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Zn	0.2500	0.2500	0.00830 (3)	0.04173 (11)
Cl	0.02319 (4)	0.17755 (6)	−0.27367 (6)	0.0697 (2)
N1	0.30551 (11)	0.34776 (13)	0.10944 (13)	0.0420 (4)
N2	0.22484 (13)	0.47062 (13)	0.32673 (15)	0.0503 (5)
H2	0.1861	0.4281	0.3423	0.060*
O1	0.14614 (11)	0.33727 (13)	−0.02481 (14)	0.0577 (4)
O2	0.14705 (12)	0.20595 (14)	−0.11392 (15)	0.0677 (5)
O3	0.27541 (12)	0.62242 (13)	0.36654 (14)	0.0632 (5)
O4	0.15579 (14)	0.55887 (13)	0.43625 (14)	0.0683 (5)
C1	0.11059 (14)	0.28121 (17)	−0.08633 (17)	0.0458 (5)
C2	0.02036 (13)	0.30729 (14)	−0.11921 (16)	0.0402 (4)
C3	−0.02401 (14)	0.26336 (16)	−0.19616 (17)	0.0450 (5)
C4	−0.10918 (16)	0.2880 (2)	−0.21635 (19)	0.0567 (6)
H4	−0.1382	0.2570	−0.2675	0.068*
C5	−0.15084 (17)	0.3575 (2)	−0.1616 (2)	0.0647 (7)
H5	−0.2082	0.3732	−0.1752	0.078*
C6	−0.10790 (17)	0.4042 (2)	−0.0863 (2)	0.0643 (7)
H6	−0.1356	0.4525	−0.0499	0.077*
C7	−0.02320 (15)	0.37853 (18)	−0.06538 (18)	0.0519 (5)
H7	0.0053	0.4097	−0.0140	0.062*
C8	0.25616 (14)	0.37803 (15)	0.18363 (16)	0.0423 (4)
H8	0.2005	0.3525	0.1891	0.051*
C9	0.28322 (14)	0.44532 (15)	0.25299 (15)	0.0411 (4)
C10	0.36653 (15)	0.48203 (17)	0.24529 (17)	0.0494 (5)
H10	0.3877	0.5266	0.2910	0.059*
C11	0.41724 (15)	0.45099 (18)	0.16834 (19)	0.0539 (6)
H11	0.4732	0.4751	0.1612	0.065*
C12	0.38551 (14)	0.38477 (18)	0.10245 (17)	0.0502 (5)
H12	0.4207	0.3647	0.0509	0.060*
C13	0.22424 (16)	0.55699 (17)	0.37586 (17)	0.0492 (5)
C14	0.1445 (2)	0.6486 (2)	0.4903 (2)	0.0798 (9)

H14A	0.1280	0.7000	0.4461	0.120*
H14B	0.1979	0.6656	0.5220	0.120*
H14C	0.1001	0.6398	0.5388	0.120*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn	0.04048 (17)	0.04280 (18)	0.0419 (2)	0.00580 (14)	0.000	0.000
Cl	0.0637 (4)	0.0734 (4)	0.0720 (4)	−0.0131 (3)	0.0083 (3)	−0.0261 (4)
N1	0.0427 (8)	0.0407 (9)	0.0427 (10)	0.0003 (7)	−0.0003 (8)	−0.0004 (7)
N2	0.0636 (11)	0.0395 (9)	0.0477 (11)	−0.0067 (8)	0.0129 (9)	−0.0020 (8)
O1	0.0516 (9)	0.0575 (10)	0.0642 (11)	0.0012 (8)	−0.0142 (8)	0.0015 (8)
O2	0.0678 (11)	0.0697 (12)	0.0656 (12)	0.0302 (10)	−0.0050 (9)	−0.0042 (10)
O3	0.0788 (12)	0.0477 (9)	0.0632 (11)	−0.0113 (9)	0.0033 (9)	−0.0093 (8)
O4	0.0933 (13)	0.0544 (10)	0.0572 (11)	−0.0041 (10)	0.0267 (10)	−0.0112 (9)
C1	0.0470 (11)	0.0482 (11)	0.0422 (12)	0.0033 (10)	0.0020 (9)	0.0115 (9)
C2	0.0443 (10)	0.0386 (10)	0.0378 (11)	0.0021 (8)	0.0008 (8)	0.0102 (8)
C3	0.0458 (11)	0.0465 (12)	0.0428 (11)	−0.0061 (9)	0.0028 (9)	0.0082 (9)
C4	0.0503 (12)	0.0734 (16)	0.0463 (13)	−0.0089 (12)	−0.0075 (11)	0.0143 (12)
C5	0.0470 (12)	0.0817 (19)	0.0653 (17)	0.0114 (13)	−0.0039 (12)	0.0221 (15)
C6	0.0609 (15)	0.0689 (16)	0.0633 (16)	0.0260 (13)	0.0045 (13)	0.0061 (13)
C7	0.0551 (13)	0.0530 (13)	0.0477 (13)	0.0085 (11)	−0.0024 (10)	0.0003 (10)
C8	0.0417 (10)	0.0370 (9)	0.0481 (11)	−0.0023 (8)	0.0017 (10)	0.0004 (9)
C9	0.0497 (10)	0.0329 (9)	0.0406 (11)	0.0005 (8)	0.0009 (9)	0.0072 (9)
C10	0.0540 (12)	0.0459 (12)	0.0484 (13)	−0.0060 (10)	−0.0070 (10)	−0.0027 (10)
C11	0.0428 (11)	0.0586 (14)	0.0601 (15)	−0.0090 (10)	−0.0009 (10)	−0.0018 (12)
C12	0.0437 (11)	0.0585 (13)	0.0484 (13)	−0.0012 (10)	0.0032 (10)	0.0003 (11)
C13	0.0665 (13)	0.0432 (11)	0.0379 (11)	0.0005 (10)	0.0013 (10)	0.0025 (9)
C14	0.104 (2)	0.0697 (18)	0.0658 (19)	0.0088 (17)	0.0153 (17)	−0.0246 (15)

Geometric parameters (Å, °)

Zn—O1	2.0484 (16)	C2—C7	1.391 (3)
Zn—O1^{i}	2.0484 (16)	C3—C4	1.385 (3)
Zn—N1	2.1012 (18)	C4—C5	1.368 (4)
Zn—N1^{i}	2.1012 (18)	C4—H4	0.93
Zn—O2	2.380 (2)	C5—C6	1.378 (4)
Zn—O2^{i}	2.380 (2)	C5—H5	0.93
Zn—C1^{i}	2.546 (2)	C6—C7	1.384 (3)
Cl—C3	1.737 (2)	C6—H6	0.93
N1—C8	1.332 (3)	C7—H7	0.93
N1—C12	1.338 (3)	C8—C9	1.383 (3)
N2—C13	1.356 (3)	C8—H8	0.93
N2—C9	1.394 (3)	C9—C10	1.385 (3)
N2—H2	0.86	C10—C11	1.376 (3)
O1—C1	1.261 (3)	C10—H10	0.93
O2—C1	1.230 (3)	C11—C12	1.365 (3)
O3—C13	1.199 (3)	C11—H11	0.93

O4—C13	1.340 (3)	C12—H12	0.93
O4—C14	1.439 (3)	C14—H14A	0.96
C1—C2	1.506 (3)	C14—H14B	0.96
C2—C3	1.389 (3)	C14—H14C	0.96
O1—Zn—O1^{i}	154.55 (11)	C2—C3—Cl	122.89 (17)
O1—Zn—N1	95.43 (7)	C5—C4—C3	120.4 (2)
O1^{i}—Zn—N1	101.20 (7)	C5—C4—H4	119.8
O1—Zn—N1^{i}	101.20 (7)	C3—C4—H4	119.8
O1^{i}—Zn—N1^{i}	95.43 (7)	C4—C5—C6	120.0 (2)
N1—Zn—N1^{i}	98.02 (10)	C4—C5—H5	120.0
O1—Zn—O2	57.97 (6)	C6—C5—H5	120.0
O1^{i}—Zn—O2	102.83 (7)	C5—C6—C7	119.4 (3)
N1—Zn—O2	153.14 (6)	C5—C6—H6	120.3
N1^{i}—Zn—O2	91.49 (7)	C7—C6—H6	120.3
O1—Zn—O2^{i}	102.83 (7)	C6—C7—C2	121.7 (2)
O1^{i}—Zn—O2^{i}	57.97 (6)	C6—C7—H7	119.2
N1—Zn—O2^{i}	91.49 (7)	C2—C7—H7	119.2
N1^{i}—Zn—O2^{i}	153.14 (6)	N1—C8—C9	123.51 (19)
O2—Zn—O2^{i}	91.17 (10)	N1—C8—H8	118.2
O1—Zn—C1^{i}	130.40 (8)	C9—C8—H8	118.2
O1^{i}—Zn—C1^{i}	29.39 (7)	C8—C9—C10	118.0 (2)
N1—Zn—C1^{i}	95.36 (7)	C8—C9—N2	117.5 (2)
N1^{i}—Zn—C1^{i}	124.83 (7)	C10—C9—N2	124.5 (2)
O2—Zn—C1^{i}	99.69 (7)	C11—C10—C9	118.4 (2)
O2^{i}—Zn—C1^{i}	28.68 (7)	C11—C10—H10	120.8
C8—N1—C12	117.73 (19)	C9—C10—H10	120.8
C8—N1—Zn	117.48 (14)	C12—C11—C10	120.1 (2)
C12—N1—Zn	124.73 (15)	C12—C11—H11	120.0
C13—N2—C9	125.2 (2)	C10—C11—H11	120.0
C13—N2—H2	117.4	N1—C12—C11	122.3 (2)
C9—N2—H2	117.4	N1—C12—H12	118.8
C1—O1—Zn	97.73 (14)	C11—C12—H12	118.8
C1—O2—Zn	83.16 (15)	O3—C13—O4	124.8 (2)
C13—O4—C14	115.2 (2)	O3—C13—N2	126.2 (2)
O2—C1—O1	120.7 (2)	O4—C13—N2	109.0 (2)
O2—C1—C2	122.0 (2)	O4—C14—H14A	109.5
O1—C1—C2	117.2 (2)	O4—C14—H14B	109.5
C3—C2—C7	117.46 (19)	H14A—C14—H14B	109.5
C3—C2—C1	125.4 (2)	O4—C14—H14C	109.5
C7—C2—C1	117.1 (2)	H14A—C14—H14C	109.5
C4—C3—C2	120.9 (2)	H14B—C14—H14C	109.5
C4—C3—Cl	116.18 (19)		
O1—Zn—N1—C8	55.12 (16)	O2—C1—C2—C7	163.7 (2)
O1^{i}—Zn—N1—C8	−144.24 (15)	O1—C1—C2—C7	−12.7 (3)

N1^{i}—Zn—N1—C8	−47.03 (13)	C7—C2—C3—C4	−1.8 (3)
O2—Zn—N1—C8	62.6 (2)	C1—C2—C3—C4	175.5 (2)
O2^{i}—Zn—N1—C8	158.16 (15)	C7—C2—C3—Cl	176.85 (17)
C1^{i}—Zn—N1—C8	−173.37 (15)	C1—C2—C3—Cl	−5.9 (3)
O1—Zn—N1—C12	−122.03 (18)	C2—C3—C4—C5	1.1 (3)
O1^{i}—Zn—N1—C12	38.62 (19)	Cl—C3—C4—C5	−177.63 (19)
N1^{i}—Zn—N1—C12	135.8 (2)	C3—C4—C5—C6	0.6 (4)
O2—Zn—N1—C12	−114.5 (2)	C4—C5—C6—C7	−1.5 (4)
O2^{i}—Zn—N1—C12	−18.99 (18)	C5—C6—C7—C2	0.7 (4)
C1^{i}—Zn—N1—C12	9.49 (19)	C3—C2—C7—C6	0.9 (3)
O1^{i}—Zn—O1—C1	48.91 (13)	C1—C2—C7—C6	−176.6 (2)
N1—Zn—O1—C1	179.75 (14)	C12—N1—C8—C9	0.1 (3)
N1^{i}—Zn—O1—C1	−80.94 (15)	Zn—N1—C8—C9	−177.22 (16)
O2—Zn—O1—C1	3.74 (13)	N1—C8—C9—C10	−0.9 (3)
O2^{i}—Zn—O1—C1	86.97 (15)	N1—C8—C9—N2	179.92 (19)
C1^{i}—Zn—O1—C1	77.97 (19)	C13—N2—C9—C8	−153.2 (2)
O1—Zn—O2—C1	−3.83 (13)	C13—N2—C9—C10	27.6 (4)
O1^{i}—Zn—O2—C1	−165.62 (14)	C8—C9—C10—C11	1.1 (3)
N1—Zn—O2—C1	−12.6 (2)	N2—C9—C10—C11	−179.7 (2)
N1^{i}—Zn—O2—C1	98.47 (15)	C9—C10—C11—C12	−0.7 (4)
O2^{i}—Zn—O2—C1	−108.26 (15)	C8—N1—C12—C11	0.3 (3)
C1^{i}—Zn—O2—C1	−135.80 (13)	Zn—N1—C12—C11	177.46 (18)
Zn—O2—C1—O1	6.1 (2)	C10—C11—C12—N1	0.0 (4)
Zn—O2—C1—C2	−170.09 (19)	C14—O4—C13—O3	1.7 (4)
Zn—O1—C1—O2	−7.2 (2)	C14—O4—C13—N2	−177.3 (2)
Zn—O1—C1—C2	169.25 (16)	C9—N2—C13—O3	−3.4 (4)
O2—C1—C2—C3	−13.6 (3)	C9—N2—C13—O4	175.6 (2)
O1—C1—C2—C3	170.0 (2)		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O2^{ii}	0.86	2.02	2.812 (3)	153
C8—H8···Cl^{ii}	0.93	2.82	3.723 (2)	165

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

Fig. 1

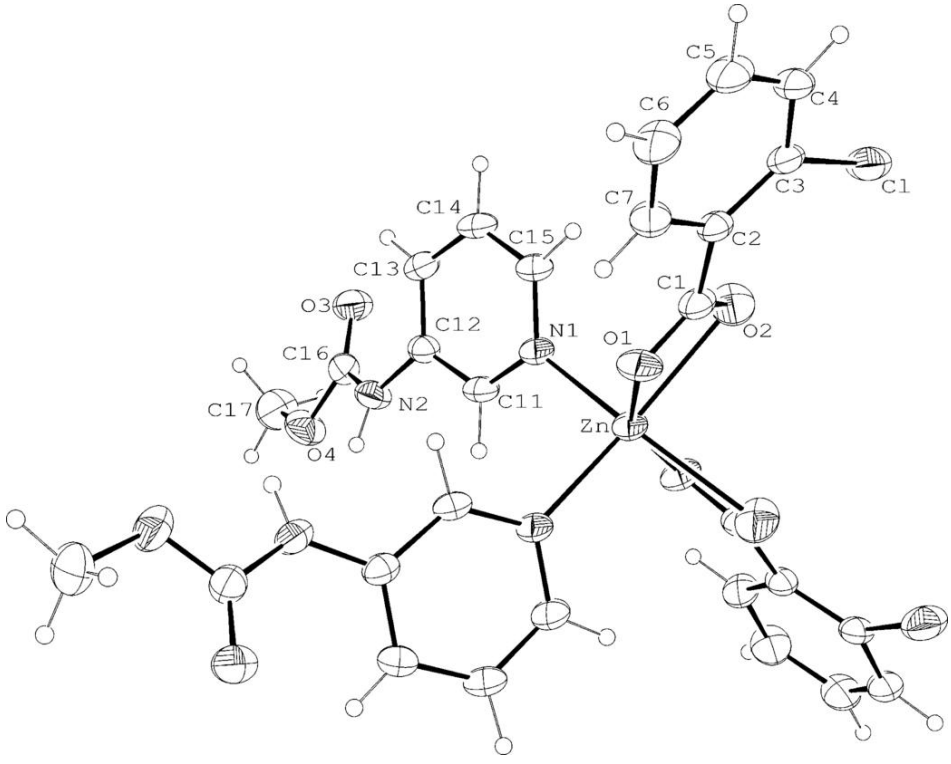
C5
C4
C6
C3
C1
C7
C2
C14
C15
C13
C1
O3
C12
N1
O1
O2
C16
N2
C11
C17
Zn
O4

Fig. 2