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Poly[bis(*N*,*N*-dimethylacetamide)-1κO,2κO-bis(μ_4-thiophene-2,5-dicarboxylato-1:2:1′:2′$\kappa^4 O^2$:$O^{2'}$:O^5:$O^{5'}$)-dizinc]

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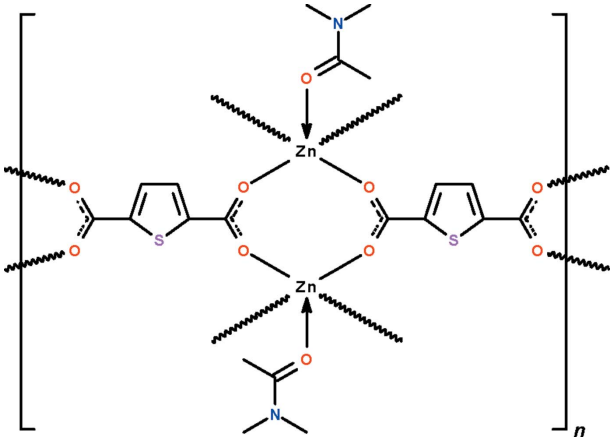
Key indicators: single-crystal X-ray study; T = 153 K; mean σ(C–C) = 0.005 Å; disorder in main residue; R factor = 0.034; wR factor = 0.089; data-to-parameter ratio = 15.6.

In the title polymeric complex, $[Zn_2(C_6H_2O_4S)_2(C_4H_9NO)_2]_n$, each carboxylate group of the thiophene-2,5-dicarboxylate dianion bridges a pair of inversion-related dimethylacetamide-coordinated Zn^{II} atoms, generating a layer motif parallel to (101). The Zn^{II} atom shows a distorted square-pyramidal coordination; the apical site is occupied by the O atom of the dimethylacetamide molecule, whereas the four basal sites are occupied by carboxylate O atoms. In the crystal, the dimethylacetamide molecule is disordered over two positions in a 0.72 (1):0.28 (1) ratio in respect of the C atoms.

Related literature

For the 1,10-phenanthroline adduct of zinc 2,5-thiophenedicarboxylate, see: Chen *et al.* (1999). For bond-length dimensions of the 2,5-thiophenedicarboxylate ion, see: Wu *et al.* (2006).



Experimental

Crystal data

$[Zn_2(C_6H_2O_4S)_2(C_4H_9NO)_2]$
M_r = 645.26
Monoclinic, $P2_1/n$
a = 8.4866 (2) Å
b = 14.8476 (4) Å
c = 10.1406 (3) Å
β = 100.734 (2)°
V = 1255.41 (6) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 2.13 mm^{-1}
T = 153 K
0.20 × 0.20 × 0.10 mm

Data collection

Gemini S Ultra diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
T_{min} = 0.675, T_{max} = 0.815
7660 measured reflections
2841 independent reflections
1990 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.089
S = 0.93
2841 reflections
182 parameters
5 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.55 e Å^{-3}

Data collection: *CrysAlis PRO* (Agilent, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

We thank the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5337).

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supporting information

Acta Cryst. (2011). E**67**, m1488 [doi:10.1107/S160053681103981X]

Poly[bis(*N*,*N*-dimethylacetamide)-1κO,2κO-bis(μ_4-thiophene-2,5-dicarboxylato-1:2:1′:2′κ^4O^2:$O^{2'}$:O^5:$O^{5'}$)dizinc]

Ming-Ming Du and Seik Weng Ng

S1. Comment

The dianions of rigid aromatic carboxylic acids such as phthalic, isophthalic and terephathalic acids furnish coordination polymers with divalent metal ions. The 2,5-thiophenedicarboxylate anion is less well studied; the only crystal structure study of a zinc(II) system is that of the zinc 2,5-thiophenedicarboxylate adduct with 1,10-phenanthroline (Chen *et al.*, 1999). In this study, the dimethylacetamide (DMA) used as solvent in the synthesis is incorporate into the crystal structure. Polymeric $[Zn_2(C_4H_9NO)_2(C_6H_2O_4S)_2]_n$ (Scheme I) has the $-CO_2$ parts of the thiophene-2,5-dicarboxylate dianion each bridging a pair of inversion-related, dimethylacetamide-coordinated zinc^{II} atoms to generate a layer motif (Fig. 1). The Zn^{II} atom shows square-pyramidal coordination; the apical site is occupied by the O atom of the DMA molecule. Bond dimensions involving the carboxylate unit are similar to those reported for ethylenediammonium thiophene-2,5-dicarboxylate dihydrate (Wu *et al.*, 2006).

S2. Experimental

2,5-Thiophenedicarboxylic acid (0.09 g, 0.5 mmol) and zinc nitrate hexahydrate (0.30 g,0.5 mmol) were dissolved in dimethylacetamide (10 ml). The solution was placed in a 25-ml flask, heated at 90 °C for 5 days. Colorless cystals separated from the solution upon cooling it to room temperature.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions [C—H 0.95 to 0.98 Å, U_{iso}(H) 1.2 to 1.5U_{eq}(C)] and were included in the refinement in the riding model approximation.

The DMA molecule is disordered over two positions with resect to the carbon atoms only. Pairs of distances (O–C, N–C) for the two components were restrained to within 0.01 Å of each other. The temperature factors of the primed atoms were set to those of the unprimed ones.

The final difference Fourier map had a peak at 1.04 Å from C3.

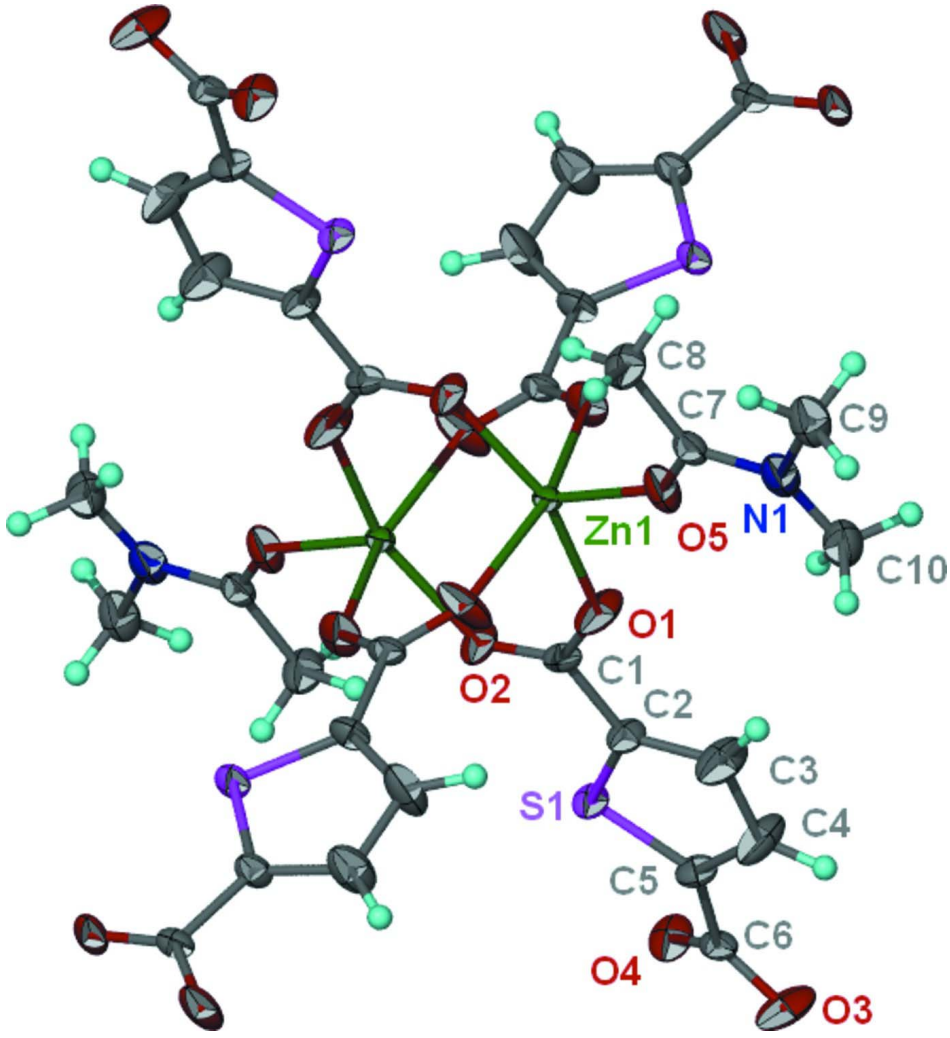

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of a portion of polymeric $Zn_2(C_4H_9NO)_2(C_6H_2O_4S)_2$ at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius. The disorder in the dimethylacetamide unit is not shown. Only the asymmetric unit is labeled.

Poly[bis(*N*,*N*-dimethylacetamide)-1κ*O*,2κ*O*- bis(μ_4-thiophene-2,5-dicarboxylato-1:2:1′:2′κ^4*O*^{2}:*O*$^{2'}$:*O*^{5}:*O*$^{5'}$)dizinc]

Crystal data

$[Zn_2(C_6H_2O_4S)_2(C_4H_9NO)_2]$
M_r = 645.26
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 8.4866 (2) Å
b = 14.8476 (4) Å
c = 10.1406 (3) Å
β = 100.734 (2)°
V = 1255.41 (6) Å^3
Z = 2

$F(000)$ = 656
D_x = 1.707 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3158 reflections
θ = 2.4–29.7°
μ = 2.13 mm^{-1}
T = 153 K
Block, colorless
0.20 × 0.20 × 0.10 mm

Data collection

Gemini S Ultra diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.1903 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
T_{min} = 0.675, T_{max} = 0.815
7660 measured reflections
2841 independent reflections
1990 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 27.5°, θ_{min} = 2.5°
h = −10→11
k = −19→17
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.089
S = 0.93
2841 reflections
182 parameters
5 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0535P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.55 e Å^{-3}

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

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
Zn1	0.64210 (4)	0.53115 (2)	0.60386 (3)	0.02026 (12)	
S1	0.70730 (9)	0.23894 (5)	0.23932 (7)	0.02835 (19)	
O1	0.7270 (3)	0.42738 (18)	0.5087 (2)	0.0482 (7)	
O2	0.5233 (3)	0.38003 (16)	0.3549 (2)	0.0392 (6)	
O3	1.0411 (4)	0.06503 (19)	0.2061 (2)	0.0544 (8)	
O4	0.8306 (3)	0.10976 (17)	0.0555 (2)	0.0432 (6)	
O5	0.8383 (3)	0.55742 (18)	0.7354 (2)	0.0428 (6)	
N1	1.0425 (3)	0.5890 (2)	0.8985 (3)	0.0387 (7)	
C1	0.6643 (4)	0.3774 (2)	0.4147 (3)	0.0262 (7)	
C2	0.7708 (4)	0.3090 (2)	0.3709 (3)	0.0284 (7)	
C3	0.9309 (5)	0.2970 (3)	0.4216 (4)	0.0507 (10)	
H3	0.9908	0.3328	0.4909	0.061*	
C4	0.9958 (5)	0.2253 (3)	0.3589 (4)	0.0550 (12)	
H4	1.1043	0.2064	0.3830	0.066*	
C5	0.8871 (4)	0.1853 (2)	0.2597 (3)	0.0312 (7)	
C6	0.9207 (4)	0.1141 (2)	0.1669 (3)	0.0299 (7)	
C7	0.8881 (5)	0.6019 (3)	0.8283 (4)	0.0298 (11)	0.72 (1)
C8	0.7793 (6)	0.6729 (4)	0.8697 (5)	0.0425 (13)	0.72 (1)
H8A	0.6843	0.6800	0.7990	0.064*	0.72 (1)
H8B	0.8368	0.7303	0.8839	0.064*	0.72 (1)
H8C	0.7461	0.6544	0.9532	0.064*	0.72 (1)
C9	1.0963 (7)	0.6473 (4)	1.0207 (5)	0.0471 (14)	0.72 (1)
H9A	1.1523	0.7004	0.9953	0.071*	0.72 (1)
H9B	1.1691	0.6128	1.0887	0.071*	0.72 (1)
H9C	1.0027	0.6663	1.0573	0.071*	0.72 (1)

C10	1.1457 (6)	0.5301 (4)	0.8593 (6)	0.0488 (14)	0.72 (1)
H10A	1.2457	0.5612	0.8526	0.073*	0.72 (1)
H10B	1.0980	0.5049	0.7716	0.073*	0.72 (1)
H10C	1.1683	0.4813	0.9253	0.073*	0.72 (1)
C7′	0.9677 (10)	0.5478 (7)	0.7833 (9)	0.0298 (11)	0.28
C8′	1.0369 (16)	0.4825 (8)	0.6974 (13)	0.0425 (13)	0.28
H8′1	0.9955	0.4220	0.7093	0.064*	0.279 (6)
H8′2	1.1541	0.4823	0.7234	0.064*	0.279 (6)
H8′3	1.0065	0.5004	0.6030	0.064*	0.279 (6)
C9′	1.2159 (12)	0.5540 (10)	0.9429 (15)	0.0471 (14)	0.28
H9′1	1.2414	0.5502	1.0411	0.071*	0.279 (6)
H9′2	1.2907	0.5955	0.9114	0.071*	0.279 (6)
H9′3	1.2254	0.4941	0.9044	0.071*	0.279 (6)
C10′	0.9967 (18)	0.6518 (8)	0.9728 (14)	0.0488 (14)	0.28
H10D	1.0135	0.6314	1.0663	0.073*	0.279 (6)
H10E	0.8827	0.6648	0.9412	0.073*	0.279 (6)
H10F	1.0597	0.7066	0.9669	0.073*	0.279 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.02204 (18)	0.01473 (18)	0.02585 (19)	−0.00035 (14)	0.00927 (13)	0.00035 (14)
S1	0.0292 (4)	0.0271 (4)	0.0297 (4)	0.0037 (3)	0.0078 (3)	−0.0091 (3)
O1	0.0437 (16)	0.0457 (16)	0.0504 (15)	0.0201 (12)	−0.0037 (12)	−0.0284 (13)
O2	0.0275 (13)	0.0387 (14)	0.0508 (14)	0.0083 (10)	0.0055 (10)	−0.0228 (12)
O3	0.071 (2)	0.0465 (16)	0.0451 (15)	0.0326 (15)	0.0095 (13)	−0.0162 (13)
O4	0.0420 (15)	0.0471 (16)	0.0420 (14)	0.0071 (12)	0.0123 (11)	−0.0218 (12)
O5	0.0324 (14)	0.0489 (16)	0.0444 (14)	−0.0157 (12)	−0.0001 (11)	0.0031 (13)
N1	0.0342 (17)	0.0409 (18)	0.0382 (16)	−0.0090 (14)	−0.0002 (13)	0.0064 (14)
C1	0.0343 (18)	0.0199 (16)	0.0261 (16)	0.0037 (13)	0.0102 (13)	0.0001 (13)
C2	0.0320 (18)	0.0264 (17)	0.0258 (15)	0.0058 (13)	0.0030 (13)	−0.0080 (13)
C3	0.052 (3)	0.052 (2)	0.043 (2)	0.018 (2)	−0.0052 (17)	−0.0188 (19)
C4	0.043 (2)	0.059 (3)	0.056 (2)	0.025 (2)	−0.0071 (19)	−0.021 (2)
C5	0.0386 (19)	0.0244 (17)	0.0318 (16)	0.0068 (14)	0.0094 (13)	−0.0052 (14)
C6	0.0369 (19)	0.0190 (17)	0.0393 (19)	0.0001 (14)	0.0215 (15)	−0.0049 (14)
C7	0.031 (2)	0.025 (2)	0.036 (2)	−0.0063 (17)	0.0121 (18)	0.0012 (18)
C8	0.036 (3)	0.044 (3)	0.047 (3)	0.003 (2)	0.007 (2)	−0.016 (2)
C9	0.040 (3)	0.057 (4)	0.042 (3)	−0.010 (2)	0.002 (2)	0.006 (2)
C10	0.036 (3)	0.052 (3)	0.059 (3)	−0.003 (2)	0.010 (2)	0.012 (3)
C7′	0.031 (2)	0.025 (2)	0.036 (2)	−0.0063 (17)	0.0121 (18)	0.0012 (18)
C8′	0.036 (3)	0.044 (3)	0.047 (3)	0.003 (2)	0.007 (2)	−0.016 (2)
C9′	0.040 (3)	0.057 (4)	0.042 (3)	−0.010 (2)	0.002 (2)	0.006 (2)
C10′	0.036 (3)	0.052 (3)	0.059 (3)	−0.003 (2)	0.010 (2)	0.012 (3)

Geometric parameters (Å, º)

Zn1—O5	1.969 (2)	C3—C4	1.404 (5)
Zn1—O1	2.021 (2)	C3—H3	0.9500

Zn1—O2^{i}	2.026 (2)	C4—C5	1.367 (5)
Zn1—O4^{ii}	2.041 (2)	C4—H4	0.9500
Zn1—O3^{iii}	2.044 (2)	C5—C6	1.479 (4)
Zn1—Zn1^{i}	3.0360 (6)	C7—C8	1.511 (6)
S1—C2	1.699 (3)	C8—H8A	0.9800
S1—C5	1.699 (3)	C8—H8B	0.9800
O1—C1	1.246 (4)	C8—H8C	0.9800
O2—C1	1.237 (4)	C9—H9A	0.9800
O2—Zn1^{i}	2.026 (2)	C9—H9B	0.9800
O3—C6	1.257 (4)	C9—H9C	0.9800
O3—Zn1^{iv}	2.044 (2)	C10—H10A	0.9800
O4—C6	1.242 (4)	C10—H10B	0.9800
O4—Zn1^{v}	2.041 (2)	C10—H10C	0.9800
O5—C7′	1.125 (8)	C7′—C8′	1.495 (10)
O5—C7	1.164 (4)	C8′—H8′1	0.9800
N1—C10′	1.303 (10)	C8′—H8′2	0.9800
N1—C10	1.349 (6)	C8′—H8′3	0.9800
N1—C7′	1.366 (8)	C9′—H9′1	0.9800
N1—C7	1.383 (5)	C9′—H9′2	0.9800
N1—C9	1.510 (5)	C9′—H9′3	0.9800
N1—C9′	1.547 (9)	C10′—H10D	0.9800
C1—C2	1.481 (4)	C10′—H10E	0.9800
C2—C3	1.372 (5)	C10′—H10F	0.9800

O5—Zn1—O1	98.22 (11)	C4—C3—H3	124.0
O5—Zn1—O2^{i}	105.20 (10)	C5—C4—C3	113.3 (3)
O1—Zn1—O2^{i}	156.55 (10)	C5—C4—H4	123.4
O5—Zn1—O4^{ii}	102.52 (10)	C3—C4—H4	123.4
O1—Zn1—O4^{ii}	87.40 (12)	C4—C5—C6	126.5 (3)
O2^{i}—Zn1—O4^{ii}	88.65 (11)	C4—C5—S1	110.7 (2)
O5—Zn1—O3^{iii}	100.14 (10)	C6—C5—S1	122.2 (2)
O1—Zn1—O3^{iii}	85.99 (13)	O4—C6—O3	125.8 (3)
O2^{i}—Zn1—O3^{iii}	88.75 (12)	O4—C6—C5	117.2 (3)
O4^{ii}—Zn1—O3^{iii}	157.07 (10)	O3—C6—C5	117.0 (3)
O5—Zn1—Zn1^{i}	173.08 (8)	O5—C7—N1	120.3 (4)
O1—Zn1—Zn1^{i}	75.18 (7)	O5—C7—C8	118.2 (4)
O2^{i}—Zn1—Zn1^{i}	81.37 (6)	N1—C7—C8	121.5 (4)
O4^{ii}—Zn1—Zn1^{i}	79.50 (7)	N1—C9—H9A	109.5
O3^{iii}—Zn1—Zn1^{i}	77.59 (7)	N1—C9—H9B	109.5
C2—S1—C5	92.59 (15)	N1—C9—H9C	109.5
C1—O1—Zn1	132.8 (2)	N1—C10—H10A	109.5
C1—O2—Zn1^{i}	124.3 (2)	N1—C10—H10B	109.5
C6—O3—Zn1^{iv}	129.6 (2)	N1—C10—H10C	109.5
C6—O4—Zn1^{v}	127.4 (2)	O5—C7′—N1	125.0 (7)
C7′—O5—C7	62.9 (5)	O5—C7′—C8′	106.8 (7)
C7′—O5—Zn1	154.4 (5)	N1—C7′—C8′	128.2 (8)
C7—O5—Zn1	142.7 (3)	C7′—C8′—H8′1	109.5
C10′—N1—C10	156.1 (8)	C7′—C8′—H8′2	109.5

C10′—N1—C7′	132.4 (8)	H8′1—C8′—H8′2	109.5
C10—N1—C7′	71.4 (5)	C7′—C8′—H8′3	109.5
C10′—N1—C7	81.0 (7)	H8′1—C8′—H8′3	109.5
C10—N1—C7	122.9 (4)	H8′2—C8′—H8′3	109.5
C10—N1—C9	119.9 (4)	N1—C9′—H9′1	109.5
C7—N1—C9	117.1 (4)	N1—C9′—H9′2	109.5
C10′—N1—C9′	116.2 (9)	H9′1—C9′—H9′2	109.5
C7′—N1—C9′	111.3 (7)	N1—C9′—H9′3	109.5
O2—C1—O1	126.3 (3)	H9′1—C9′—H9′3	109.5
O2—C1—C2	117.6 (3)	H9′2—C9′—H9′3	109.5
O1—C1—C2	116.1 (3)	N1—C10′—H10D	109.5
C3—C2—C1	126.5 (3)	N1—C10′—H10E	109.5
C3—C2—S1	111.2 (2)	H10D—C10′—H10E	109.5
C1—C2—S1	122.2 (2)	N1—C10′—H10F	109.5
C2—C3—C4	111.9 (3)	H10D—C10′—H10F	109.5
C2—C3—H3	124.0	H10E—C10′—H10F	109.5

O5—Zn1—O1—C1	178.6 (3)	C4—C5—C6—O4	−152.9 (4)
O2^{i}—Zn1—O1—C1	1.5 (5)	S1—C5—C6—O4	17.8 (4)
O4^{ii}—Zn1—O1—C1	−79.1 (3)	C4—C5—C6—O3	24.9 (5)
O3^{iii}—Zn1—O1—C1	78.9 (3)	S1—C5—C6—O3	−164.4 (3)
O1—Zn1—O5—C7′	2.8 (14)	C7′—O5—C7—N1	−5.0 (7)
O2^{i}—Zn1—O5—C7′	−178.3 (14)	Zn1—O5—C7—N1	173.8 (3)
O4^{ii}—Zn1—O5—C7′	−86.3 (14)	C7′—O5—C7—C8	175.6 (8)
O3^{iii}—Zn1—O5—C7′	90.2 (14)	Zn1—O5—C7—C8	−5.7 (7)
O1—Zn1—O5—C7	−174.5 (4)	C10′—N1—C7—O5	−176.9 (7)
O2^{i}—Zn1—O5—C7	4.3 (5)	C10—N1—C7—O5	4.7 (6)
O4^{ii}—Zn1—O5—C7	96.3 (4)	C7′—N1—C7—O5	4.7 (6)
O3^{iii}—Zn1—O5—C7	−87.2 (5)	C9—N1—C7—O5	−177.9 (4)
Zn1^{i}—O2—C1—O1	0.4 (5)	C9′—N1—C7—O5	4 (2)
Zn1^{i}—O2—C1—C2	−179.1 (2)	C10′—N1—C7—C8	2.5 (8)
Zn1—O1—C1—O2	−1.0 (5)	C10—N1—C7—C8	−175.9 (5)
Zn1—O1—C1—C2	178.6 (2)	C7′—N1—C7—C8	−175.9 (8)
O2—C1—C2—C3	178.1 (4)	C9—N1—C7—C8	1.5 (6)
O1—C1—C2—C3	−1.4 (5)	C9′—N1—C7—C8	−176 (2)
O2—C1—C2—S1	2.6 (4)	C7—O5—C7′—N1	5.3 (7)
O1—C1—C2—S1	−176.9 (3)	Zn1—O5—C7′—N1	−172.9 (4)
C5—S1—C2—C3	5.4 (3)	C7—O5—C7′—C8′	−174.9 (11)
C5—S1—C2—C1	−178.5 (3)	Zn1—O5—C7′—C8′	7 (2)
C1—C2—C3—C4	179.1 (4)	C10′—N1—C7′—O5	−7.2 (17)
S1—C2—C3—C4	−5.0 (5)	C10—N1—C7′—O5	175.0 (12)
C2—C3—C4—C5	1.7 (6)	C7—N1—C7′—O5	−5.1 (7)
C3—C4—C5—C6	174.0 (4)	C9—N1—C7′—O5	−16 (4)
C3—C4—C5—S1	2.4 (5)	C9′—N1—C7′—O5	174.8 (10)
C2—S1—C5—C4	−4.4 (3)	C10′—N1—C7′—C8′	173.1 (12)
C2—S1—C5—C6	−176.4 (3)	C10—N1—C7′—C8′	−4.8 (11)
Zn1^{v}—O4—C6—O3	−5.8 (5)	C7—N1—C7′—C8′	175.2 (15)
Zn1^{v}—O4—C6—C5	171.8 (2)	C9—N1—C7′—C8′	164 (2)

Zn1^{iv}—O3—C6—O4	4.4 (6)	C9′—N1—C7′—C8′	−5.0 (14)
Zn1^{iv}—O3—C6—C5	−173.1 (2)		

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