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[*N*,*N*′-Bis(2,6-dimethylphenyl)pentane-2,4-diiminato-$\kappa^2 N,N'$]trichlorido-(tetrahydrofuran-κO)zirconium

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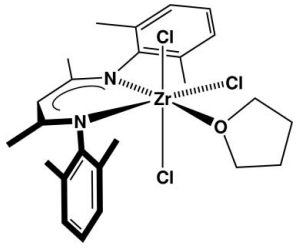
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.004 Å; disorder in main residue; R factor = 0.031; wR factor = 0.088; data-to-parameter ratio = 16.9.

The nacnac (pentane-2,4-diiminato) ligand in the title compound, $[Zr(C_{21}H_{25}N_2)Cl_3(C_4H_8O)]$, displays a κ^2-coordination to the Zr center. Three chlorido ligands and a coordinated tetrahydrofuran (THF) molecule complete the octahedral environment of the metal. Despite the C_2 symmetry observed in the ^{1}H NMR spectrum, the THF molecule is found *trans* to one of the N atoms of the nacnac ligand. The asymmetric unit contains two molecules. In one THF ligand one C atom and four H atoms are disordered over two sites in the ratio *ca* 0.58:0.42.

Related literature

For other zirconium complexes containing an κ^2-coordinated nacnac ligand, see: Rahim *et al.* (1998); Kakaliou *et al.* (1999); Qian *et al.* (1999); Jin & Novak (2000); Franceschini *et al.* (2003); Hamaki *et al.* (2006). For zirconium complexes containing nacnac ligands in other coordination modes, see: Rahim *et al.* (1998); Vollmerhaus *et al.* (2000); Basuli *et al.* (2004); Verguet, Fortuné *et al.* (2007); Verguet, Oguadinma & Schaper (2007). For related literature, see: Spek (2003).



Experimental

Crystal data

$[Zr(C_{21}H_{25}N_2)Cl_3(C_4H_8O)]$
M_r = 575.10
Triclinic, $P\overline{1}$
a = 13.6082 (5) Å
b = 14.5970 (5) Å
c = 16.3293 (6) Å
α = 114.948 (2)°
β = 98.898 (2)°
γ = 107.951 (2)°
V = 2641.04 (16) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 6.35 mm^{-1}
T = 150 (2) K
0.16 × 0.16 × 0.07 mm

Data collection

Bruker Smart 6000 diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.50, T_{max} = 0.64
36248 measured reflections
9929 independent reflections
9305 reflections with $I > 2\sigma(I)$
R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.088
S = 0.99
9929 reflections
589 parameters
11 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.61 e Å^{-3}
$\Delta\rho_{min}$ = −1.25 e Å^{-3}

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *UdMX* (Marris, 2004).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: OM2160).

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supplementary materials

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[*N*,*N*'-Bis(2,6-dimethylphenyl)pentane-2,4-diiminato-κ^2*N*,*N*']trichlorido(tetrahydrofuran-κ*O*)zirconium

R.-V. Fortuné, E. Verguet, P. O. Oguadinma and F. Schaper

Comment

The title compound was first synthesized by Jin & Novak (2000), although no experimental or spectroscopic data was provided. Our synthesis followed the same procedure described there and for the synthesis of other (nacnac)$ZrCl_3$(THF) complexes (Kakaliou *et al.*, 1999), *i.e.* reaction of the deprotonated nacnac ligand (nacnac = pentane-2,4-diiminato) with $ZrCl_4(THF)_2$.

Two identical, independent molecules are present in the asymmetric unit. Only the geometry of one of those is described in detail. Apart from the missing disorder of the THF molecule, no significant differences were observed in the second molecule.

The zirconium center has a slightly distorted octahedral coordination geometry with L–Zr–*L* bond angles ranging from 83° to 99°. The coordination of the nanac ligand is best described as an in-plane η^2-coordination *via* the nitrogen lone pairs. The ligand backbone is nearly planar, with a slight bending of the methyl groups and the central carbon atom C3 out of the ligand plane, probalby due to steric interaction with each other. Bond distances in the ligand backbone are in agreement with a strong electron delocalization. The zirconium center is slightly bend out of the ligand mean plane (⟨(Zr1,N1,N2),(N1,N2,C2–C4)=41°), a common feature for η^2-coordinated nacnac ligands in penta-coordinated or octahedral complexes (Rahim *et al.*, 1998; Qian *et al.*, 1999; Kakaliou *et al.*, 1999; Franceschini *et al.*, 2003; Hamaki *et al.*, 2006). Long Zr—C distances for C2–C4 (> 3.1 Å) indicate, however, that this is not due to coordination of the ligand π-system to the metal center, but rather to the steric strain introduced by the in-plane coordination of the metal center. This is further supported by a N–Zr–N angle smaller than 90° (⟨(N1–Zr1–N2)=83.11 (7)°) and slightly widened C3–C–N angles (⟨(N1–C2–C3)=123.5 (2)°, ⟨(N2–C4–C3)=123.6 (2)°).

One THF molecule, coordinated *trans* to N2, completes the octahedral coordination of the metal center. Although penta-coordinated (nacnac)$ZrCl_3$ complexes can be isolated in the absence of THF, coordination of THF to a (nacnac)$ZrCl_3$ fragment has been reported before (Kakaliou *et al.*, 1999; Jin & Novak, 2000). In all cases, THF preferred a coordination *trans* to nitrogen. As also observed in these complexes, the Zr—N2 distance in the title compound is slightly longer than Zr—N1 (2.218 (2) and 2.176 (2) Å, respectively), in agreement with a stronger *trans* effect of chlorine compared to oxygen.

Of the two possible coordination modes of the nacnac ligand (η^2–in plane or η^x–side on), the η^2-coordination seems to be preferred when an octahedral coordination of the metal center can be achieved (Franceschini *et al.*, 2003 (BEDLUS, BEDMAZ), Kakaliou *et al.*, 1999 (LIRDOF), Rahim *et al.*, 1998 (SEQXER, SEQXIV), Hamaki *et al.*, 2006 (DEGWIW), Jin & Novak, 2000). For penta-coordinated zirconium complexes both, η^2–coordination (LIRCIY, LIRCOE, LIRDUL: Kakaliou *et al.*, 1999; MAPJIW, MAPJUI: Qian *et al.*, 1999) and higher coordination modes (FAPBUU, FAPCEF, FAPCAB, FAPCIJ: Basuli *et al.*, 2004) have been observed. When a second, planar coordinated ligand such as cyclopentadienyl or indenyl is present, the nacnac ligand always prefers higher coordination modes (Vollmerhaus *et al.*, 2000 (QIPGUR); Rahim *et*

al., 1998 (SEQXOB); Verguet, Fortuné *et al.*, 2007; Verguet, Oguadinma & Schaper, 2007). (Codes refer to entries in the Cambridge Structural Database). It should be noted that a "higher coordination mode" does not necessarily correspond to a cyclopentadienyl–like η^5–coordination (Verguet, Fortuné *et al.*, 2007).

Experimental

To a solution of 7.8 g (25 mmol) nacnacH in THF, 11.5 ml n-BuLi in hexane (2.7 *M*, 27.5 mmol) were added at −78°C. The solution was allowed to warm to room temperature over night. Evaporation of the solvent yield a red-brown residue, which was washed two times with hexane to give 7.3 g nacnacLi(THF) (76%) as a yellow powder.

RMN ^{1}H (300 MHz, C_6D_6): 7.08–6.92 (m, 6H, CH [$C_6H_3Me_2$]), 4.97 (s, 1H, CH [nacnac]), 2.93 (br s, 4H, THF), 2.21 (s, 12H, CH_3 [$C_6H_3Me_2$]), 1.81 (s, 6H, CH_3 [nacnac]), 0.92 (br s, 4H, THF).

A solution of 2.94 g $ZrCl_4(THF)_2$ (7.8 mmol) in 20 ml toluene were added to a solution of 3.0 g nacnacLi(THF) (7.8 mmol) in 20 ml toluene. After stirring at room temperature for two hours the brown suspension is filtered and the precipitate washed with 4 ml toluene. The filtrates were combined and their volume reduced to 15 ml. 15 ml hexane were layered on top of the solution. After several days 4.0 g (89%) of the title compound were isolated as yellow-brown microcrystals.

RMN ^{1}H (300 MHz, C_6D_6): δ 6.94 (m, 6H, CH [$C_6H_3Me_2$]), 5.55 (s, 1H, CH [nacnac]), 3.80 (br s, 4H, THF), 2.44 (s, 12H, CH_3 [$C_6H_3Me_2$]), 1.45 (s, 6H, CH_3 [nacnac]), 1.01 (br s, 4H, THF).

Refinement

All non-H atoms were refined by full-matrix least-squares with anisotropic displacement parameters. The THF molecule in one of the two independent molecules in the asymmetric unit was found to be disordered. The disorder was resolved and refined isotropic with appropriate restraints. Relative occupancies refined to 0.57:0.43. Although the neighbouring atoms C24 and O1 show slightly enlarged thermal ellipsoids, no disorder was refined for those atoms. The atoms C25A and C25B were refined with isotropic thermal parameters. The H atoms were generated geometrically (C—H 0.93 to 0.98, N—H 0.86 and O—H 0.82 Å) and were included in the refinement in the riding model approximation; their temperature factors were set to 1.5 times those of the equivalent isotropic temperature factors of the parent site (methyl) and 1.2 times for others. A final verification of possible voids was performed using the VOID routine of the *PLATON* program (Spek, 2000).

Figures

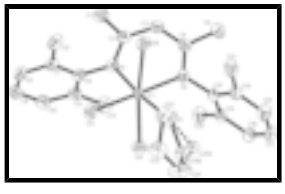


Fig. 1. Thermal ellipsoid drawing of the title compound. Thermal ellipsoids are shown at 50% probability levels. Hydrogen atoms are omitted for clarity. Only one of the two independent molecules is shown.

[*N*,*N*'-bis(2,6-dimethylphenyl)pentane-2,4-diiminato-κ^2*N*,*N*']\ trichlorido(tetrahydrofuran-κ*O*)zirconium

Crystal data

$[Zr(C_{21}H_{25}N_2)Cl_3(C_4H_8O)]$	$Z = 4$
$M_r = 575.10$	$F_{000} = 1184$
Triclinic, $P\overline{1}$	$D_x = 1.446$ Mg m^{-3}
Hall symbol: -P 1	Cu $K\alpha$ radiation $\lambda = 1.54178$ Å
$a = 13.6082$ (5) Å	Cell parameters from 16709 reflections
$b = 14.5970$ (5) Å	$\theta = 4.1$–71.5º
$c = 16.3293$ (6) Å	$\mu = 6.35$ mm^{-1}
$\alpha = 114.948$ (2)º	$T = 150$ (2) K
$\beta = 98.898$ (2)º	Plate, yellow
$\gamma = 107.951$ (2)º	0.16 × 0.16 × 0.07 mm
$V = 2641.04$ (16) Å^3	

Data collection

Bruker Smart 6000 diffractometer	9929 independent reflections
Radiation source: Rotating Anode	9305 reflections with $I > 2\sigma(I)$
Monochromator: Montel 200 optics	$R_{int} = 0.036$
Detector resolution: 5.5 pixels mm^{-1}	$\theta_{max} = 72.1$º
$T = 150(2)$ K	$\theta_{min} = 3.2$º
ω scans	$h = -16\rightarrow16$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$k = -14\rightarrow16$
$T_{min} = 0.50$, $T_{max} = 0.64$	$l = -20\rightarrow19$
36248 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.031$	H-atom parameters constrained
$wR(F^2) = 0.088$	$w = 1/[\sigma^2(F_o^2) + (0.0601P)^2 + 1.3844P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.99$	$(\Delta/\sigma)_{max} = 0.001$
9929 reflections	$\Delta\rho_{max} = 0.61$ e Å^{-3}
589 parameters	$\Delta\rho_{min} = -1.25$ e Å^{-3}
11 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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. Comments on remaining CHECKCIF errors:

All geometrically accessible data was collected. A data completeness of 0.96 was due to geometrical constraints of the instrument (particularly severe for triclinic space groups) and could not be improved. Errors in the T_{max}/T_{min} ratio are probably due to inaccurate determination of crystal dimensions.

Supplied e.s.d. values for the cell parameters are correct. Differences between calculated and supplied values arise most probably from the rounding of cell parameters in the CIF file. No additional electron density was found in solvent accessible voids.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.31188 (13)	0.27645 (15)	0.82802 (11)	0.0356 (4)	
O2	0.83397 (13)	0.29499 (14)	0.37605 (12)	0.0300 (3)	
Zr1	0.168238 (14)	0.257712 (14)	0.883656 (11)	0.02274 (6)	
Zr2	0.690471 (13)	0.266619 (14)	0.429264 (11)	0.02207 (6)	
Cl1	0.81986 (5)	0.40340 (5)	0.58818 (4)	0.04192 (15)	
Cl2	0.72529 (4)	0.11247 (5)	0.42947 (4)	0.03114 (12)	
Cl3	0.67650 (4)	0.41924 (4)	0.41025 (4)	0.02919 (11)	
Cl4	0.09555 (5)	0.06236 (5)	0.76288 (4)	0.03778 (13)	
Cl5	0.27106 (4)	0.22364 (5)	0.99607 (4)	0.03309 (12)	
Cl6	0.26375 (4)	0.45918 (4)	0.99344 (4)	0.03234 (12)	
N1	0.02755 (15)	0.25160 (15)	0.93244 (13)	0.0255 (4)	
N2	0.08358 (14)	0.29748 (15)	0.78438 (12)	0.0246 (4)	
N3	0.56673 (14)	0.14635 (14)	0.28439 (12)	0.0234 (3)	
N4	0.55027 (14)	0.22025 (14)	0.47524 (12)	0.0232 (3)	
C1	−0.1057 (2)	0.3230 (2)	0.9896 (2)	0.0385 (6)	
H1A	−0.0695	0.3429	1.0552	0.058*	
H1B	−0.1689	0.2510	0.9584	0.058*	
H1C	−0.1302	0.3799	0.9910	0.058*	
C2	−0.02606 (18)	0.31564 (18)	0.93455 (16)	0.0286 (4)	
C3	−0.01605 (19)	0.37371 (19)	0.88523 (17)	0.0315 (5)	
H3A	−0.0418	0.4305	0.9048	0.038*	
C4	0.02727 (19)	0.35953 (19)	0.80996 (16)	0.0306 (5)	
C5	0.0018 (3)	0.4131 (3)	0.7534 (2)	0.0503 (7)	
H5A	0.0702	0.4645	0.7561	0.075*	
H5B	−0.0408	0.4542	0.7806	0.075*	
H5C	−0.0407	0.3557	0.6867	0.075*	
C6	−0.00780 (18)	0.18171 (19)	0.97429 (16)	0.0276 (4)	
C7	0.04394 (19)	0.22239 (19)	1.07150 (16)	0.0304 (5)	
C8	0.0062 (2)	0.1533 (2)	1.10877 (18)	0.0353 (5)	
H8A	0.0417	0.1778	1.1738	0.042*	

C9	−0.0812 (2)	0.0505 (2)	1.05422 (19)	0.0377 (5)	
H9A	−0.1059	0.0056	1.0817	0.045*	
C10	−0.1324 (2)	0.0136 (2)	0.95944 (19)	0.0359 (5)	
H10A	−0.1931	−0.0568	0.9221	0.043*	
C11	−0.09697 (19)	0.07760 (19)	0.91753 (17)	0.0310 (5)	
C12	0.1335 (2)	0.3371 (2)	1.13648 (17)	0.0391 (6)	
H12A	0.2005	0.3316	1.1615	0.059*	
H12B	0.1113	0.3780	1.1898	0.059*	
H12C	0.1471	0.3764	1.1005	0.059*	
C13	−0.1544 (2)	0.0350 (2)	0.81371 (18)	0.0393 (6)	
H13A	−0.2242	−0.0288	0.7907	0.059*	
H13B	−0.1083	0.0122	0.7759	0.059*	
H13C	−0.1680	0.0941	0.8070	0.059*	
C14	0.09836 (18)	0.26843 (18)	0.69140 (15)	0.0267 (4)	
C15	0.01671 (18)	0.17145 (19)	0.61125 (16)	0.0282 (4)	
C16	0.0319 (2)	0.1401 (2)	0.52222 (16)	0.0330 (5)	
H16A	−0.0212	0.0734	0.4674	0.040*	
C17	0.1227 (2)	0.2045 (2)	0.51282 (17)	0.0380 (5)	
H17A	0.1324	0.1814	0.4520	0.046*	
C18	0.1996 (2)	0.3029 (2)	0.59173 (18)	0.0384 (5)	
H18A	0.2609	0.3477	0.5841	0.046*	
C19	0.18867 (19)	0.3376 (2)	0.68276 (16)	0.0316 (5)	
C20	−0.08737 (18)	0.1047 (2)	0.61822 (16)	0.0332 (5)	
H20A	−0.0697	0.0846	0.6670	0.050*	
H20B	−0.1305	0.0370	0.5562	0.050*	
H20C	−0.1298	0.1491	0.6359	0.050*	
C21	0.2722 (2)	0.4483 (2)	0.76503 (18)	0.0409 (6)	
H21A	0.2528	0.4625	0.8232	0.061*	
H21B	0.2736	0.5072	0.7505	0.061*	
H21C	0.3447	0.4472	0.7749	0.061*	
C22	0.42639 (17)	0.34392 (19)	0.89300 (16)	0.0337 (5)	
H22A	0.4299	0.3727	0.9607	0.040*	
H22B	0.4622	0.4074	0.8838	0.040*	
C23	0.4789 (2)	0.2648 (2)	0.8651 (2)	0.0418 (6)	
H23A	0.4721	0.2255	0.9022	0.050*	
H23B	0.5574	0.3041	0.8759	0.050*	
C24	0.4168 (2)	0.1845 (3)	0.7601 (2)	0.0582 (8)	
H24A	0.4654	0.1901	0.7215	0.070*	0.579 (15)
H24B	0.3858	0.1072	0.7476	0.070*	0.579 (15)
H24C	0.4544	0.2099	0.7214	0.070*	0.421 (15)
H24D	0.4137	0.1099	0.7435	0.070*	0.421 (15)
C25A	0.3286 (4)	0.2173 (5)	0.7364 (2)	0.0260 (13)*	0.579 (15)
H25A	0.3507	0.2666	0.7094	0.031*	0.579 (15)
H25B	0.2608	0.1511	0.6896	0.031*	0.579 (15)
C25B	0.3054 (4)	0.1789 (7)	0.7393 (4)	0.040 (2)*	0.421 (15)
H25C	0.2514	0.1081	0.7286	0.048*	0.421 (15)
H25D	0.2849	0.1861	0.6822	0.048*	0.421 (15)
C26	0.3907 (2)	0.0875 (2)	0.48075 (18)	0.0361 (5)	
H26A	0.4328	0.0964	0.5401	0.054*	

H26B	0.3376	0.0100	0.4393	0.054*
H26C	0.3520	0.1359	0.4958	0.054*
C27	0.46727 (17)	0.11865 (18)	0.43008 (15)	0.0257 (4)
C28	0.44382 (18)	0.03930 (17)	0.33490 (15)	0.0274 (4)
H28A	0.3950	−0.0344	0.3151	0.033*
C29	0.48297 (17)	0.05433 (17)	0.26492 (15)	0.0258 (4)
C30	0.4222 (2)	−0.03666 (19)	0.16210 (16)	0.0339 (5)
H30A	0.4749	−0.0524	0.1298	0.051*
H30B	0.3777	−0.0126	0.1296	0.051*
H30C	0.3747	−0.1040	0.1601	0.051*
C31	0.55240 (18)	0.30167 (17)	0.56685 (15)	0.0255 (4)
C32	0.49824 (18)	0.36925 (18)	0.56688 (16)	0.0277 (4)
C33	0.5087 (2)	0.45453 (19)	0.65476 (17)	0.0334 (5)
H33A	0.4732	0.5016	0.6562	0.040*
C34	0.5702 (2)	0.47140 (19)	0.73990 (17)	0.0370 (5)
H34A	0.5786	0.5312	0.7991	0.044*
C35	0.6193 (2)	0.4010 (2)	0.73850 (16)	0.0354 (5)
H35A	0.6598	0.4120	0.7973	0.043*
C36	0.61043 (19)	0.31394 (19)	0.65228 (16)	0.0300 (5)
C37	0.6616 (2)	0.2368 (2)	0.65460 (17)	0.0359 (5)
H37A	0.7303	0.2789	0.7085	0.054*
H37B	0.6766	0.2026	0.5947	0.054*
H37C	0.6113	0.1786	0.6621	0.054*
C38	0.42865 (19)	0.3507 (2)	0.47521 (16)	0.0324 (5)
H38A	0.3917	0.4007	0.4902	0.049*
H38B	0.3739	0.2734	0.4377	0.049*
H38C	0.4751	0.3658	0.4381	0.049*
C39	0.58412 (18)	0.16308 (17)	0.20537 (14)	0.0258 (4)
C40	0.65758 (19)	0.13058 (19)	0.16161 (15)	0.0298 (5)
C41	0.6829 (2)	0.1631 (2)	0.09567 (16)	0.0359 (5)
H41A	0.7336	0.1432	0.0662	0.043*
C42	0.6353 (2)	0.2239 (2)	0.07270 (16)	0.0369 (5)
H42A	0.6564	0.2490	0.0304	0.044*
C43	0.5570 (2)	0.2483 (2)	0.11125 (16)	0.0340 (5)
H43A	0.5217	0.2867	0.0924	0.041*
C44	0.52891 (18)	0.21755 (18)	0.17737 (15)	0.0286 (4)
C45	0.44014 (19)	0.2404 (2)	0.21513 (16)	0.0326 (5)
H45A	0.4094	0.2756	0.1850	0.049*
H45B	0.4707	0.2902	0.2846	0.049*
H45C	0.3823	0.1704	0.2004	0.049*
C46	0.7061 (2)	0.0582 (2)	0.17975 (18)	0.0365 (5)
H46A	0.7001	0.0611	0.2397	0.055*
H46B	0.7834	0.0852	0.1844	0.055*
H46C	0.6664	−0.0186	0.1269	0.055*
C47	0.9279 (2)	0.2706 (3)	0.4043 (2)	0.0419 (6)
H47A	0.9063	0.1894	0.3727	0.050*
H47B	0.9547	0.3021	0.4745	0.050*
C48	1.0156 (2)	0.3246 (2)	0.3719 (2)	0.0421 (6)
H48A	1.0529	0.2762	0.3446	0.050*

H48B	1.0708	0.3972	0.4258	0.050*
C49	0.9530 (2)	0.3393 (2)	0.29589 (17)	0.0350 (5)
H49A	1.0012	0.4001	0.2889	0.042*
H49B	0.9192	0.2699	0.2331	0.042*
C50	0.8673 (2)	0.3683 (2)	0.33600 (17)	0.0314 (5)
H50A	0.8983	0.4475	0.3863	0.038*
H50B	0.8046	0.3534	0.2851	0.038*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0294 (8)	0.0529 (10)	0.0224 (8)	0.0190 (8)	0.0109 (7)	0.0148 (7)
O2	0.0288 (8)	0.0374 (9)	0.0352 (8)	0.0165 (7)	0.0152 (7)	0.0242 (7)
Zr1	0.02394 (10)	0.02405 (10)	0.01852 (10)	0.01080 (7)	0.00542 (7)	0.00919 (8)
Zr2	0.02249 (10)	0.02244 (10)	0.01938 (10)	0.00790 (7)	0.00445 (7)	0.01071 (7)
Cl1	0.0381 (3)	0.0412 (3)	0.0247 (3)	0.0000 (2)	−0.0037 (2)	0.0145 (2)
Cl2	0.0351 (3)	0.0340 (3)	0.0347 (3)	0.0189 (2)	0.0122 (2)	0.0224 (2)
Cl3	0.0326 (3)	0.0247 (2)	0.0317 (3)	0.0131 (2)	0.0095 (2)	0.0149 (2)
Cl4	0.0485 (3)	0.0285 (3)	0.0281 (3)	0.0201 (2)	0.0034 (2)	0.0074 (2)
Cl5	0.0348 (3)	0.0435 (3)	0.0258 (2)	0.0196 (2)	0.0075 (2)	0.0198 (2)
Cl6	0.0344 (3)	0.0264 (3)	0.0240 (2)	0.0071 (2)	0.0067 (2)	0.0071 (2)
N1	0.0274 (9)	0.0251 (9)	0.0252 (9)	0.0114 (7)	0.0107 (7)	0.0126 (7)
N2	0.0253 (9)	0.0265 (9)	0.0220 (8)	0.0102 (7)	0.0066 (7)	0.0129 (7)
N3	0.0257 (9)	0.0229 (8)	0.0188 (8)	0.0103 (7)	0.0045 (7)	0.0089 (7)
N4	0.0255 (9)	0.0225 (9)	0.0202 (8)	0.0093 (7)	0.0074 (7)	0.0101 (7)
C1	0.0403 (13)	0.0370 (13)	0.0497 (15)	0.0226 (11)	0.0267 (12)	0.0228 (12)
C2	0.0291 (11)	0.0252 (10)	0.0288 (11)	0.0114 (9)	0.0108 (9)	0.0108 (9)
C3	0.0351 (12)	0.0286 (11)	0.0334 (12)	0.0184 (10)	0.0118 (10)	0.0140 (9)
C4	0.0346 (12)	0.0287 (11)	0.0287 (11)	0.0150 (9)	0.0072 (9)	0.0143 (9)
C5	0.077 (2)	0.0609 (18)	0.0466 (15)	0.0501 (17)	0.0298 (15)	0.0371 (14)
C6	0.0300 (11)	0.0282 (11)	0.0314 (11)	0.0150 (9)	0.0169 (9)	0.0161 (9)
C7	0.0350 (12)	0.0342 (12)	0.0292 (11)	0.0189 (10)	0.0177 (10)	0.0163 (10)
C8	0.0457 (14)	0.0459 (14)	0.0331 (12)	0.0293 (12)	0.0242 (11)	0.0245 (11)
C9	0.0468 (14)	0.0406 (13)	0.0506 (15)	0.0276 (12)	0.0320 (12)	0.0318 (12)
C10	0.0363 (13)	0.0315 (12)	0.0479 (14)	0.0167 (10)	0.0225 (11)	0.0218 (11)
C11	0.0311 (11)	0.0292 (11)	0.0360 (12)	0.0140 (9)	0.0151 (10)	0.0167 (10)
C12	0.0426 (14)	0.0407 (14)	0.0253 (11)	0.0138 (11)	0.0137 (10)	0.0110 (10)
C13	0.0325 (12)	0.0355 (13)	0.0378 (13)	0.0052 (10)	0.0080 (10)	0.0155 (11)
C14	0.0309 (11)	0.0297 (11)	0.0231 (10)	0.0138 (9)	0.0082 (9)	0.0157 (9)
C15	0.0302 (11)	0.0301 (11)	0.0263 (11)	0.0138 (9)	0.0061 (9)	0.0161 (9)
C16	0.0376 (12)	0.0325 (12)	0.0240 (11)	0.0136 (10)	0.0054 (9)	0.0123 (9)
C17	0.0501 (15)	0.0439 (14)	0.0282 (12)	0.0229 (12)	0.0172 (11)	0.0212 (11)
C18	0.0381 (13)	0.0447 (14)	0.0363 (13)	0.0128 (11)	0.0159 (11)	0.0252 (11)
C19	0.0339 (12)	0.0322 (12)	0.0286 (11)	0.0109 (10)	0.0089 (9)	0.0176 (10)
C20	0.0280 (11)	0.0351 (12)	0.0275 (11)	0.0069 (9)	0.0027 (9)	0.0145 (10)
C21	0.0423 (14)	0.0344 (13)	0.0362 (13)	0.0034 (11)	0.0092 (11)	0.0195 (11)
C22	0.0265 (11)	0.0371 (13)	0.0319 (12)	0.0096 (10)	0.0087 (9)	0.0153 (10)
C23	0.0308 (12)	0.0367 (13)	0.0516 (15)	0.0130 (10)	0.0101 (11)	0.0186 (12)

C24	0.0487 (17)	0.0466 (17)	0.0540 (18)	0.0241 (14)	0.0106 (14)	0.0031 (14)
C26	0.0375 (13)	0.0300 (12)	0.0345 (12)	0.0066 (10)	0.0164 (10)	0.0145 (10)
C27	0.0266 (10)	0.0244 (10)	0.0264 (10)	0.0105 (8)	0.0076 (8)	0.0135 (9)
C28	0.0285 (11)	0.0209 (10)	0.0261 (10)	0.0071 (8)	0.0055 (9)	0.0096 (8)
C29	0.0268 (10)	0.0216 (10)	0.0234 (10)	0.0102 (8)	0.0033 (8)	0.0082 (8)
C30	0.0374 (12)	0.0256 (11)	0.0234 (11)	0.0075 (9)	0.0043 (9)	0.0052 (9)
C31	0.0288 (11)	0.0211 (10)	0.0217 (10)	0.0070 (8)	0.0087 (8)	0.0090 (8)
C32	0.0285 (11)	0.0266 (11)	0.0288 (11)	0.0107 (9)	0.0113 (9)	0.0144 (9)
C33	0.0391 (13)	0.0253 (11)	0.0351 (12)	0.0148 (10)	0.0156 (10)	0.0123 (10)
C34	0.0475 (14)	0.0258 (11)	0.0275 (11)	0.0116 (10)	0.0139 (10)	0.0069 (9)
C35	0.0444 (14)	0.0314 (12)	0.0213 (10)	0.0113 (10)	0.0068 (10)	0.0099 (9)
C36	0.0344 (12)	0.0276 (11)	0.0248 (11)	0.0095 (9)	0.0093 (9)	0.0130 (9)
C37	0.0479 (14)	0.0386 (13)	0.0279 (11)	0.0204 (11)	0.0122 (10)	0.0206 (10)
C38	0.0343 (12)	0.0359 (12)	0.0313 (12)	0.0188 (10)	0.0121 (10)	0.0171 (10)
C39	0.0294 (11)	0.0242 (10)	0.0167 (9)	0.0093 (8)	0.0034 (8)	0.0070 (8)
C40	0.0348 (12)	0.0293 (11)	0.0197 (10)	0.0132 (9)	0.0075 (9)	0.0082 (9)
C41	0.0427 (13)	0.0381 (13)	0.0241 (11)	0.0167 (11)	0.0137 (10)	0.0125 (10)
C42	0.0478 (14)	0.0368 (13)	0.0201 (10)	0.0124 (11)	0.0088 (10)	0.0136 (10)
C43	0.0434 (13)	0.0299 (12)	0.0231 (10)	0.0149 (10)	0.0030 (9)	0.0116 (9)
C44	0.0310 (11)	0.0273 (11)	0.0186 (10)	0.0099 (9)	0.0019 (8)	0.0076 (8)
C45	0.0345 (12)	0.0331 (12)	0.0280 (11)	0.0169 (10)	0.0045 (9)	0.0134 (9)
C46	0.0459 (14)	0.0387 (13)	0.0337 (12)	0.0256 (11)	0.0189 (11)	0.0180 (11)
C47	0.0323 (13)	0.0599 (17)	0.0544 (16)	0.0260 (12)	0.0191 (12)	0.0397 (14)
C48	0.0327 (13)	0.0541 (16)	0.0454 (14)	0.0173 (11)	0.0170 (11)	0.0290 (13)
C49	0.0335 (12)	0.0381 (13)	0.0327 (12)	0.0126 (10)	0.0150 (10)	0.0175 (10)
C50	0.0373 (12)	0.0316 (12)	0.0313 (11)	0.0142 (10)	0.0167 (10)	0.0189 (10)

Geometric parameters (Å, °)

O1—C25a	1.473 (3)	C22—H22a	0.99
O1—C22	1.478 (2)	C22—H22b	0.99
O1—C25b	1.508 (4)	C23—C24	1.508 (4)
O1—Zr1	2.2694 (16)	C23—H23a	0.99
O2—C50	1.470 (3)	C23—H23b	0.99
O2—C47	1.487 (3)	C24—C25b	1.471 (4)
O2—Zr2	2.2517 (15)	C24—C25a	1.479 (4)
Zr1—N1	2.1759 (18)	C24—H24a	0.99
Zr1—N2	2.2182 (17)	C24—H24b	0.99
Zr1—Cl4	2.4316 (6)	C24—H24c	0.99
Zr1—Cl5	2.4453 (5)	C24—H24d	0.99
Zr1—Cl6	2.4551 (5)	C25a—H25a	0.99
Zr2—N4	2.1766 (17)	C25a—H25b	0.99
Zr2—N3	2.2262 (17)	C25b—H25c	0.99
Zr2—Cl1	2.4227 (6)	C25b—H25d	0.99
Zr2—Cl3	2.4336 (5)	C26—C27	1.506 (3)
Zr2—Cl2	2.4395 (5)	C26—H26a	0.98
N1—C2	1.345 (3)	C26—H26b	0.98
N1—C6	1.458 (3)	C26—H26c	0.98
N2—C4	1.338 (3)	C27—C28	1.400 (3)

N2—C14	1.459 (3)	C28—C29	1.402 (3)
N3—C29	1.337 (3)	C28—H28a	0.95
N3—C39	1.451 (3)	C29—C30	1.510 (3)
N4—C27	1.344 (3)	C30—H30a	0.98
N4—C31	1.455 (3)	C30—H30b	0.98
C1—C2	1.512 (3)	C30—H30c	0.98
C1—H1a	0.98	C31—C36	1.401 (3)
C1—H1b	0.98	C31—C32	1.401 (3)
C1—H1c	0.98	C32—C33	1.396 (3)
C2—C3	1.387 (3)	C32—C38	1.507 (3)
C3—C4	1.409 (3)	C33—C34	1.385 (4)
C3—H3a	0.95	C33—H33a	0.95
C4—C5	1.504 (3)	C34—C35	1.382 (4)
C5—H5a	0.98	C34—H34a	0.95
C5—H5b	0.98	C35—C36	1.399 (3)
C5—H5c	0.98	C35—H35a	0.95
C6—C11	1.403 (3)	C36—C37	1.505 (3)
C6—C7	1.403 (3)	C37—H37a	0.98
C7—C8	1.395 (3)	C37—H37b	0.98
C7—C12	1.503 (3)	C37—H37c	0.98
C8—C9	1.380 (4)	C38—H38a	0.98
C8—H8a	0.95	C38—H38b	0.98
C9—C10	1.379 (4)	C38—H38c	0.98
C9—H9a	0.95	C39—C40	1.401 (3)
C10—C11	1.392 (3)	C39—C44	1.405 (3)
C10—H10a	0.95	C40—C41	1.396 (3)
C11—C13	1.508 (3)	C40—C46	1.507 (3)
C12—H12a	0.98	C41—C42	1.380 (4)
C12—H12b	0.98	C41—H41a	0.95
C12—H12c	0.98	C42—C43	1.383 (4)
C13—H13a	0.98	C42—H42a	0.95
C13—H13b	0.98	C43—C44	1.395 (3)
C13—H13c	0.98	C43—H43a	0.95
C14—C19	1.400 (3)	C44—C45	1.502 (3)
C14—C15	1.403 (3)	C45—H45a	0.98
C15—C16	1.398 (3)	C45—H45b	0.98
C15—C20	1.503 (3)	C45—H45c	0.98
C16—C17	1.378 (4)	C46—H46a	0.98
C16—H16a	0.95	C46—H46b	0.98
C17—C18	1.383 (4)	C46—H46c	0.98
C17—H17a	0.95	C47—C48	1.514 (3)
C18—C19	1.403 (3)	C47—H47a	0.99
C18—H18a	0.95	C47—H47b	0.99
C19—C21	1.507 (3)	C48—C49	1.524 (4)
C20—H20a	0.98	C48—H48a	0.99
C20—H20b	0.98	C48—H48b	0.99
C20—H20c	0.98	C49—C50	1.508 (3)
C21—H21a	0.98	C49—H49a	0.99
C21—H21b	0.98	C49—H49b	0.99

C21—H21c	0.98	C50—H50a	0.99
C22—C23	1.490 (3)	C50—H50b	0.99
C25A—O1—C22	100.8 (2)	C22—C23—H23A	110.9
C22—O1—C25B	110.0 (2)	C24—C23—H23A	110.9
C25A—O1—ZR1	135.7 (2)	C22—C23—H23B	110.9
C22—O1—ZR1	122.05 (12)	C24—C23—H23B	110.9
C25B—O1—ZR1	119.1 (3)	H23A—C23—H23B	108.9
C50—O2—C47	108.24 (17)	C25B—C24—C23	108.9 (3)
C50—O2—ZR2	126.32 (13)	C25A—C24—C23	105.4 (2)
C47—O2—ZR2	122.94 (13)	C25B—C24—H24A	126.4
N1—ZR1—N2	83.11 (7)	C25A—C24—H24A	110.7
N1—ZR1—O1	174.03 (7)	C23—C24—H24A	110.7
N2—ZR1—O1	91.54 (6)	C25B—C24—H24B	89
N1—ZR1—CL4	97.72 (5)	C25A—C24—H24B	110.7
N2—ZR1—CL4	92.34 (5)	C23—C24—H24B	110.7
O1—ZR1—CL4	85.10 (5)	H24A—C24—H24B	108.8
N1—ZR1—CL5	98.86 (5)	C25A—C24—H24C	91.2
N2—ZR1—CL5	176.67 (5)	C23—C24—H24C	109.9
O1—ZR1—CL5	86.36 (4)	C25B—C24—H24D	109.9
CL4—ZR1—CL5	90.06 (2)	C25A—C24—H24D	129.9
N1—ZR1—CL6	91.09 (5)	C23—C24—H24D	109.9
N2—ZR1—CL6	86.50 (5)	H24A—C24—H24D	88.8
O1—ZR1—CL6	85.93 (5)	H24C—C24—H24D	108.3
CL4—ZR1—CL6	170.91 (2)	O1—C25A—C24	105.0 (2)
CL5—ZR1—CL6	90.77 (2)	O1—C25A—H25A	110.7
N4—ZR2—N3	82.65 (6)	C24—C25A—H25A	110.7
N4—ZR2—O2	173.55 (6)	O1—C25A—H25B	110.7
N3—ZR2—O2	94.92 (6)	C24—C25A—H25B	110.7
N4—ZR2—CL1	96.00 (5)	H25A—C25A—H25B	108.8
N3—ZR2—CL1	177.03 (5)	C24—C25B—O1	103.6 (3)
O2—ZR2—CL1	86.69 (5)	C24—C25B—H25C	111
N4—ZR2—CL3	100.57 (5)	O1—C25B—H25C	111
N3—ZR2—CL3	89.58 (5)	C24—C25B—H25D	111
O2—ZR2—CL3	85.35 (4)	O1—C25B—H25D	111
CL1—ZR2—CL3	88.06 (2)	H25C—C25B—H25D	109
N4—ZR2—CL2	89.39 (5)	C27—C26—H26A	109.5
N3—ZR2—CL2	88.26 (5)	C27—C26—H26B	109.5
O2—ZR2—CL2	84.55 (4)	H26A—C26—H26B	109.5
CL1—ZR2—CL2	94.37 (2)	C27—C26—H26C	109.5
CL3—ZR2—CL2	169.458 (19)	H26A—C26—H26C	109.5
C2—N1—C6	115.53 (18)	H26B—C26—H26C	109.5
C2—N1—ZR1	124.55 (15)	N4—C27—C28	123.38 (19)
C6—N1—ZR1	119.68 (13)	N4—C27—C26	119.86 (19)
C4—N2—C14	116.36 (17)	C28—C27—C26	116.70 (19)
C4—N2—ZR1	122.39 (14)	C27—C28—C29	128.5 (2)
C14—N2—ZR1	120.82 (13)	C27—C28—H28A	115.8
C29—N3—C39	118.24 (17)	C29—C28—H28A	115.8
C29—N3—ZR2	123.43 (14)	N3—C29—C28	123.48 (19)
C39—N3—ZR2	118.04 (13)	N3—C29—C30	119.20 (19)

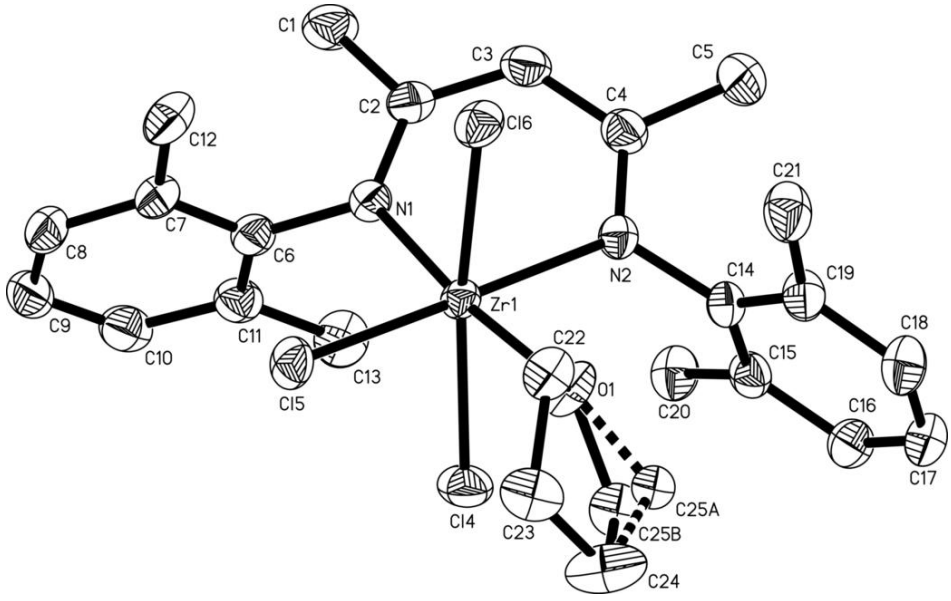
C27—N4—C31	117.46 (17)	C28—C29—C30	117.23 (19)
C27—N4—ZR2	124.81 (14)	C29—C30—H30A	109.5
C31—N4—ZR2	117.39 (13)	C29—C30—H30B	109.5
C2—C1—H1A	109.5	H30A—C30—H30B	109.5
C2—C1—H1B	109.5	C29—C30—H30C	109.5
H1A—C1—H1B	109.5	H30A—C30—H30C	109.5
C2—C1—H1C	109.5	H30B—C30—H30C	109.5
H1A—C1—H1C	109.5	C36—C31—C32	121.5 (2)
H1B—C1—H1C	109.5	C36—C31—N4	120.4 (2)
N1—C2—C3	123.5 (2)	C32—C31—N4	118.11 (19)
N1—C2—C1	119.8 (2)	C33—C32—C31	118.3 (2)
C3—C2—C1	116.7 (2)	C33—C32—C38	120.0 (2)
C2—C3—C4	128.5 (2)	C31—C32—C38	121.7 (2)
C2—C3—H3A	115.7	C34—C33—C32	120.9 (2)
C4—C3—H3A	115.7	C34—C33—H33A	119.5
N2—C4—C3	123.6 (2)	C32—C33—H33A	119.5
N2—C4—C5	119.8 (2)	C35—C34—C33	119.8 (2)
C3—C4—C5	116.5 (2)	C35—C34—H34A	120.1
C4—C5—H5A	109.5	C33—C34—H34A	120.1
C4—C5—H5B	109.5	C34—C35—C36	121.3 (2)
H5A—C5—H5B	109.5	C34—C35—H35A	119.4
C4—C5—H5C	109.5	C36—C35—H35A	119.4
H5A—C5—H5C	109.5	C35—C36—C31	117.9 (2)
H5B—C5—H5C	109.5	C35—C36—C37	119.3 (2)
C11—C6—C7	121.5 (2)	C31—C36—C37	122.8 (2)
C11—C6—N1	119.0 (2)	C36—C37—H37A	109.5
C7—C6—N1	119.4 (2)	C36—C37—H37B	109.5
C8—C7—C6	117.4 (2)	H37A—C37—H37B	109.5
C8—C7—C12	119.6 (2)	C36—C37—H37C	109.5
C6—C7—C12	123.0 (2)	H37A—C37—H37C	109.5
C9—C8—C7	122.1 (2)	H37B—C37—H37C	109.5
C9—C8—H8A	119	C32—C38—H38A	109.5
C7—C8—H8A	119	C32—C38—H38B	109.5
C10—C9—C8	119.4 (2)	H38A—C38—H38B	109.5
C10—C9—H9A	120.3	C32—C38—H38C	109.5
C8—C9—H9A	120.3	H38A—C38—H38C	109.5
C9—C10—C11	121.3 (2)	H38B—C38—H38C	109.5
C9—C10—H10A	119.4	C40—C39—C44	121.2 (2)
C11—C10—H10A	119.4	C40—C39—N3	120.88 (19)
C10—C11—C6	118.4 (2)	C44—C39—N3	117.81 (19)
C10—C11—C13	120.1 (2)	C41—C40—C39	118.2 (2)
C6—C11—C13	121.5 (2)	C41—C40—C46	119.1 (2)
C7—C12—H12A	109.5	C39—C40—C46	122.7 (2)
C7—C12—H12B	109.5	C42—C41—C40	121.0 (2)
H12A—C12—H12B	109.5	C42—C41—H41A	119.5
C7—C12—H12C	109.5	C40—C41—H41A	119.5
H12A—C12—H12C	109.5	C41—C42—C43	120.0 (2)
H12B—C12—H12C	109.5	C41—C42—H42A	120
C11—C13—H13A	109.5	C43—C42—H42A	120

C11—C13—H13B	109.5	C42—C43—C44	121.0 (2)
H13A—C13—H13B	109.5	C42—C43—H43A	119.5
C11—C13—H13C	109.5	C44—C43—H43A	119.5
H13A—C13—H13C	109.5	C43—C44—C39	118.2 (2)
H13B—C13—H13C	109.5	C43—C44—C45	120.1 (2)
C19—C14—C15	121.7 (2)	C39—C44—C45	121.7 (2)
C19—C14—N2	120.79 (19)	C44—C45—H45A	109.5
C15—C14—N2	117.50 (19)	C44—C45—H45B	109.5
C16—C15—C14	118.2 (2)	H45A—C45—H45B	109.5
C16—C15—C20	119.9 (2)	C44—C45—H45C	109.5
C14—C15—C20	121.9 (2)	H45A—C45—H45C	109.5
C17—C16—C15	121.0 (2)	H45B—C45—H45C	109.5
C17—C16—H16A	119.5	C40—C46—H46A	109.5
C15—C16—H16A	119.5	C40—C46—H46B	109.5
C16—C17—C18	120.1 (2)	H46A—C46—H46B	109.5
C16—C17—H17A	119.9	C40—C46—H46C	109.5
C18—C17—H17A	119.9	H46A—C46—H46C	109.5
C17—C18—C19	121.1 (2)	H46B—C46—H46C	109.5
C17—C18—H18A	119.4	O2—C47—C48	105.6 (2)
C19—C18—H18A	119.4	O2—C47—H47A	110.6
C14—C19—C18	117.8 (2)	C48—C47—H47A	110.6
C14—C19—C21	123.6 (2)	O2—C47—H47B	110.6
C18—C19—C21	118.6 (2)	C48—C47—H47B	110.6
C15—C20—H20A	109.5	H47A—C47—H47B	108.7
C15—C20—H20B	109.5	C47—C48—C49	104.0 (2)
H20A—C20—H20B	109.5	C47—C48—H48A	111
C15—C20—H20C	109.5	C49—C48—H48A	111
H20A—C20—H20C	109.5	C47—C48—H48B	111
H20B—C20—H20C	109.5	C49—C48—H48B	111
C19—C21—H21A	109.5	H48A—C48—H48B	109
C19—C21—H21B	109.5	C50—C49—C48	102.18 (19)
H21A—C21—H21B	109.5	C50—C49—H49A	111.3
C19—C21—H21C	109.5	C48—C49—H49A	111.3
H21A—C21—H21C	109.5	C50—C49—H49B	111.3
H21B—C21—H21C	109.5	C48—C49—H49B	111.3
O1—C22—C23	103.91 (18)	H49A—C49—H49B	109.2
O1—C22—H22A	111	O2—C50—C49	103.61 (18)
C23—C22—H22A	111	O2—C50—H50A	111
O1—C22—H22B	111	C49—C50—H50A	111
C23—C22—H22B	111	O2—C50—H50B	111
H22A—C22—H22B	109	C49—C50—H50B	111
C22—C23—C24	104.3 (2)	H50A—C50—H50B	109
C25A—O1—ZR1—N2	−65.5 (3)	C19—C14—C15—C16	5.1 (3)
C22—O1—ZR1—N2	131.26 (17)	N2—C14—C15—C16	−177.44 (19)
C25B—O1—ZR1—N2	−84.5 (4)	C19—C14—C15—C20	−172.0 (2)
C25A—O1—ZR1—CL4	26.7 (3)	N2—C14—C15—C20	5.5 (3)
C22—O1—ZR1—CL4	−136.52 (16)	C14—C15—C16—C17	−2.1 (3)
C25B—O1—ZR1—CL4	7.7 (4)	C20—C15—C16—C17	175.0 (2)
C25A—O1—ZR1—CL5	117.0 (3)	C15—C16—C17—C18	−1.2 (4)

C22—O1—ZR1—CL5	−46.14 (16)	C16—C17—C18—C19	1.7 (4)
C25B—O1—ZR1—CL5	98.1 (4)	C15—C14—C19—C18	−4.6 (3)
C25A—O1—ZR1—CL6	−151.9 (3)	N2—C14—C19—C18	178.0 (2)
C22—O1—ZR1—CL6	44.89 (16)	C15—C14—C19—C21	173.5 (2)
C25B—O1—ZR1—CL6	−170.9 (4)	N2—C14—C19—C21	−3.9 (4)
C50—O2—ZR2—N3	80.13 (17)	C17—C18—C19—C14	1.2 (4)
C47—O2—ZR2—N3	−119.98 (18)	C17—C18—C19—C21	−177.0 (2)
C50—O2—ZR2—CL1	−97.37 (16)	C25A—O1—C22—C23	−44.4 (3)
C47—O2—ZR2—CL1	62.52 (18)	C25B—O1—C22—C23	−23.3 (5)
C50—O2—ZR2—CL3	−9.05 (16)	ZR1—O1—C22—C23	123.79 (18)
C47—O2—ZR2—CL3	150.84 (18)	O1—C22—C23—C24	30.0 (3)
C50—O2—ZR2—CL2	167.91 (17)	C22—C23—C24—C25B	−27.4 (6)
C47—O2—ZR2—CL2	−32.20 (17)	C22—C23—C24—C25A	−4.0 (4)
N2—ZR1—N1—C2	−33.21 (18)	C22—O1—C25A—C24	41.9 (4)
CL4—ZR1—N1—C2	−124.64 (17)	C25B—O1—C25A—C24	−75.2 (5)
CL5—ZR1—N1—C2	144.08 (17)	ZR1—O1—C25A—C24	−123.6 (3)
CL6—ZR1—N1—C2	53.14 (17)	C25B—C24—C25A—O1	78.5 (5)
N2—ZR1—N1—C6	152.64 (16)	C23—C24—C25A—O1	−23.6 (5)
CL4—ZR1—N1—C6	61.20 (16)	C25A—C24—C25B—O1	−72.0 (5)
CL5—ZR1—N1—C6	−30.07 (16)	C23—C24—C25B—O1	12.9 (7)
CL6—ZR1—N1—C6	−121.02 (15)	C25A—O1—C25B—C24	75.1 (5)
N1—ZR1—N2—C4	35.98 (17)	C22—O1—C25B—C24	6.5 (7)
O1—ZR1—N2—C4	−141.36 (17)	ZR1—O1—C25B—C24	−141.6 (4)
CL4—ZR1—N2—C4	133.48 (17)	C31—N4—C27—C28	168.1 (2)
CL6—ZR1—N2—C4	−55.54 (17)	ZR2—N4—C27—C28	−18.8 (3)
N1—ZR1—N2—C14	−151.80 (16)	C31—N4—C27—C26	−9.0 (3)
O1—ZR1—N2—C14	30.85 (16)	ZR2—N4—C27—C26	164.13 (16)
CL4—ZR1—N2—C14	−54.30 (15)	N4—C27—C28—C29	−14.0 (4)
CL6—ZR1—N2—C14	116.67 (15)	C26—C27—C28—C29	163.1 (2)
N4—ZR2—N3—C29	−34.50 (16)	C39—N3—C29—C28	−166.3 (2)
O2—ZR2—N3—C29	139.49 (16)	ZR2—N3—C29—C28	20.0 (3)
CL3—ZR2—N3—C29	−135.21 (16)	C39—N3—C29—C30	10.2 (3)
CL2—ZR2—N3—C29	55.11 (16)	ZR2—N3—C29—C30	−163.49 (16)
N4—ZR2—N3—C39	151.81 (15)	C27—C28—C29—N3	12.9 (4)
O2—ZR2—N3—C39	−34.21 (15)	C27—C28—C29—C30	−163.7 (2)
CL3—ZR2—N3—C39	51.09 (14)	C27—N4—C31—C36	92.5 (2)
CL2—ZR2—N3—C39	−118.59 (14)	ZR2—N4—C31—C36	−81.2 (2)
N3—ZR2—N4—C27	34.00 (17)	C27—N4—C31—C32	−89.7 (2)
CL1—ZR2—N4—C27	−148.66 (16)	ZR2—N4—C31—C32	96.7 (2)
CL3—ZR2—N4—C27	122.20 (16)	C36—C31—C32—C33	3.9 (3)
CL2—ZR2—N4—C27	−54.32 (17)	N4—C31—C32—C33	−173.97 (19)
N3—ZR2—N4—C31	−152.88 (15)	C36—C31—C32—C38	−175.3 (2)
CL1—ZR2—N4—C31	24.46 (15)	N4—C31—C32—C38	6.9 (3)
CL3—ZR2—N4—C31	−64.68 (15)	C31—C32—C33—C34	−0.6 (3)
CL2—ZR2—N4—C31	118.80 (14)	C38—C32—C33—C34	178.5 (2)
C6—N1—C2—C3	−169.4 (2)	C32—C33—C34—C35	−2.0 (4)
ZR1—N1—C2—C3	16.2 (3)	C33—C34—C35—C36	1.6 (4)
C6—N1—C2—C1	8.0 (3)	C34—C35—C36—C31	1.6 (4)
ZR1—N1—C2—C1	−166.40 (17)	C34—C35—C36—C37	−177.6 (2)

N1—C2—C3—C4	15.9 (4)	C32—C31—C36—C35	−4.3 (3)
C1—C2—C3—C4	−161.6 (2)	N4—C31—C36—C35	173.5 (2)
C14—N2—C4—C3	164.9 (2)	C32—C31—C36—C37	174.8 (2)
ZR1—N2—C4—C3	−22.6 (3)	N4—C31—C36—C37	−7.4 (3)
C14—N2—C4—C5	−11.5 (3)	C29—N3—C39—C40	−94.8 (2)
ZR1—N2—C4—C5	161.0 (2)	ZR2—N3—C39—C40	79.2 (2)
C2—C3—C4—N2	−11.8 (4)	C29—N3—C39—C44	88.2 (2)
C2—C3—C4—C5	164.7 (3)	ZR2—N3—C39—C44	−97.80 (19)
C2—N1—C6—C11	85.2 (2)	C44—C39—C40—C41	6.1 (3)
ZR1—N1—C6—C11	−100.1 (2)	N3—C39—C40—C41	−170.8 (2)
C2—N1—C6—C7	−91.3 (3)	C44—C39—C40—C46	−171.2 (2)
ZR1—N1—C6—C7	83.4 (2)	N3—C39—C40—C46	11.9 (3)
C11—C6—C7—C8	2.6 (3)	C39—C40—C41—C42	−1.2 (3)
N1—C6—C7—C8	178.96 (19)	C46—C40—C41—C42	176.2 (2)
C11—C6—C7—C12	−174.8 (2)	C40—C41—C42—C43	−3.5 (4)
N1—C6—C7—C12	1.5 (3)	C41—C42—C43—C44	3.4 (4)
C6—C7—C8—C9	−2.4 (3)	C42—C43—C44—C39	1.3 (3)
C12—C7—C8—C9	175.1 (2)	C42—C43—C44—C45	−177.3 (2)
C7—C8—C9—C10	0.9 (4)	C40—C39—C44—C43	−6.2 (3)
C8—C9—C10—C11	0.6 (4)	N3—C39—C44—C43	170.87 (19)
C9—C10—C11—C6	−0.5 (3)	C40—C39—C44—C45	172.5 (2)
C9—C10—C11—C13	179.9 (2)	N3—C39—C44—C45	−10.5 (3)
C7—C6—C11—C10	−1.2 (3)	C50—O2—C47—C48	−4.6 (3)
N1—C6—C11—C10	−177.57 (19)	ZR2—O2—C47—C48	−167.62 (16)
C7—C6—C11—C13	178.5 (2)	O2—C47—C48—C49	−20.3 (3)
N1—C6—C11—C13	2.1 (3)	C47—C48—C49—C50	36.9 (3)
C4—N2—C14—C19	89.4 (3)	C47—O2—C50—C49	27.9 (2)
ZR1—N2—C14—C19	−83.3 (2)	ZR2—O2—C50—C49	−169.80 (14)
C4—N2—C14—C15	−88.1 (2)	C48—C49—C50—O2	−39.7 (2)
ZR1—N2—C14—C15	99.2 (2)		

Fig. 1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