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# Propane-1,2-diaminium tris(pyridine-2,6-dicarboxylato-$\kappa^3O^2,N,O^6$)zirconate(IV) trihydrate

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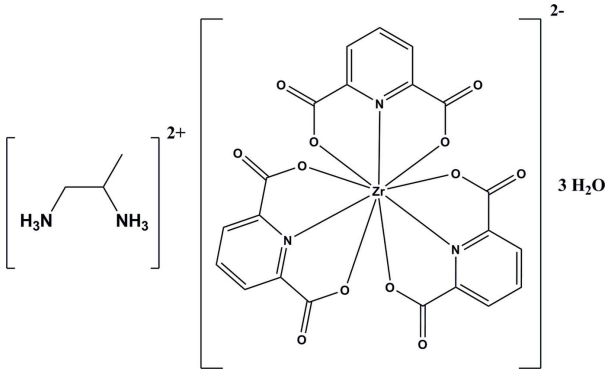
Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.009 Å; $R$ factor = 0.086; $wR$ factor = 0.206; data-to-parameter ratio = 17.5.

In the title compound, $(C_3H_{12}N_2)[Zr(C_7H_3NO_4)_3]\cdot 3H_2O$, the $Zr^{IV}$ cation is chelated by three pyridine-2,6-dicarboxylate anions in a distorted tricapped trigonal–prismatic environment. The crystal structure is stabilized by intermolecular N—H···O and O—H···O hydrogen bonds.

## Related literature

For the background to proton-transfer compounds, see: Aghabozorg *et al.* (2008). For related structures, see: Aghabozorg *et al.* (2005); Daneshvar *et al.* (2008); Pasdar *et al.* (2010*a*,*b*, 2011*a*,*b*).




## Experimental

### Crystal data

$(C_3H_{12}N_2)[Zr(C_7H_3NO_4)_3]\cdot 3H_2O$
$M_r$ = 716.73
Monoclinic, $P2_1/n$
$a$ = 10.515 (2) Å
$b$ = 19.195 (4) Å
$c$ = 14.149 (3) Å
$\beta$ = 103.39 (3)°
$V$ = 2778.1 (10) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.48 mm$^{-1}$
$T$ = 298 K
0.25 × 0.15 × 0.15 mm

### Data collection

Stoe IPDS II diffractometer
Absorption correction: numerical (*X-SHAPE* and *X-RED32*; Stoe & Cie, 2005)
$T_{min}$ = 0.915, $T_{max}$ = 0.926
21838 measured reflections
7481 independent reflections
5264 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.133

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.086
$wR(F^2)$ = 0.206
$S$ = 1.18
7481 reflections
427 parameters
7 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 1.09 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.77 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O13—H13$A$···O14 | 0.80 (8) | 2.10 (9) | 2.745 (11) | 138 (13) |
| O13—H13$B$···O2$^{i}$ | 0.80 (4) | 2.10 (8) | 2.793 (8) | 145 (12) |
| O14—H14$A$···O5$^{ii}$ | 0.91 (8) | 2.11 (9) | 2.978 (7) | 158 (12) |
| O14—H14$B$···O8 | 0.85 (4) | 1.89 (5) | 2.731 (9) | 171 (14) |
| O15—H15$A$···O12 | 0.98 (9) | 1.91 (10) | 2.857 (8) | 164 (12) |
| O15—H15$B$···O1$^{iii}$ | 0.83 (4) | 2.46 (7) | 3.227 (9) | 155 (13) |
| N4—H4$A$···O10$^{iv}$ | 0.89 | 1.92 | 2.791 (8) | 165 |
| N4—H4$B$···O6$^{ii}$ | 0.89 | 2.24 | 2.851 (7) | 125 |
| N4—H4$C$···O15$^{v}$ | 0.89 | 2.04 | 2.864 (10) | 154 |
| N5—H5$A$···O13 | 0.89 | 1.94 | 2.769 (9) | 154 |
| N5—H5$B$···O6$^{iv}$ | 0.89 | 2.18 | 2.844 (8) | 131 |
| N5—H5$C$···O4 | 0.89 | 1.91 | 2.776 (8) | 163 |

Symmetry codes: (i) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (ii) $x-\frac{1}{2}, -y+\frac{3}{2}, z+\frac{1}{2}$; (iii) $-x+2, -y+1, -z+1$; (iv) $x-1, y, z$; (v) $-x+1, -y+1, -z+1$.

Data collection: *X-AREA* (Stoe & Cie, 2005); cell refinement: *X-AREA* (Stoe & Cie, 2005); data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5157).

## References

Aghabozorg, H., Manteghi, F. & Sheshmani, S. (2008). *J. Iran Chem. Soc.* **5**, 184–227.
Aghabozorg, H., Moghimi, A., Manteghi, F. & Ranjbar, M. (2005). *Z. Anorg. Allg. Chem.* **631**, 909–913.
Daneshvar, S., Aghabozorg, H. & Manteghi, F. (2008). *Acta Cryst.* E**64**, m1308–m1309.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Pasdar, H., Ebdam, A., Aghabozorg, H. & Notash, B. (2011*a*). *Acta Cryst.* E**67**, m294.
Pasdar, H., Heidari, S., Aghabozorg, H. & Notash, B. (2010*a*). *Acta Cryst.* E**66**, m1581.
Pasdar, H., Majdolashrafi, M., Aghabozorg, H. & Khavasi, H. R. (2010*b*). *Acta Cryst.* E**66**, o3043.
Pasdar, H., Sadat Kashani, S., Aghabozorg, H. & Notash, B. (2011*b*). *Acta Cryst.* E**67**, m193–m194.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Stoe & Cie (2005). *X-AREA*, *X-SHAPE* and *X-RED32*. Stoe & Cie, Darmstadt, Germany.

# supporting information

*Acta Cryst.* (2011). E**67**, m587 [doi:10.1107/S1600536811008488]

# Propane-1,2-diaminium tris(pyridine-2,6-dicarboxylato-$\kappa^3O^2,N,O^6$)zirconate(IV) trihydrate

**Hoda Pasdar, Shahrzad Shakiba, Hossein Aghabozorg and Behrouz Notash**

## S1. Comment

Pyridine-2,6-dicarboxylic acid ($pydcH_2$) was commonly used by Aghabozorg and his co-workers as an acid in proton transfer systems (Aghabozorg *et al.* 2008). Our group have been focused on forming ion pairs between 2,6-$pydcH_2$ and various organic bases (Pasdar *et al.*, 2010*a*; Pasdar *et al.* 2011*a,b*). The structure of two proton transfer compound containing nine-coordinated $[Zr^{IV}(2,6\text{-}pydc)_3]^{2-}$ moiety were reported with the counter cationic part of 2,6-pyridinediamine(Aghabozorg *et al.* 2005) and 2,4,6-triamino-1,3,5-triazine (Daneshvar *et al.* 2008), respectively. Recently, we report the structure of $(2a6mpH)_2[Zr(2,6\text{-}pydc)_3].2H_2O$ (Pasdar *et al.* 2011*a*).

Herein, we report the synthesis and crystal structure of $(1,2\text{-}pdaH_2)[Zr(2,6\text{-}pydc)_3].3H_2O$. The title compound was prepared by the reaction of $ZrCl_4.3H_2O$, propane-1,2-diamine and 2,6-pyridinedicarboxylic acid in aqueous solution. Fig.1 present the molecular structure of the title compound. X-ray diffraction study shows that Zirconium(IV) ion is coordinated by three $pydc^{2-}$ moiety in a distorted tricapped trigonal prismatic geometry and $pydc^{2-}$ ligands act as tridentate ligand. Coordination environment around Zirconium(IV) ion in the $(1,2\text{-}pdaH_2)[Zr(2,6\text{-}pydc)_3].3H_2O$ is presented in Fig. 2. The Zr—N and Zr—O bond lengths and angles are in the normal ranges (Aghabozorg *et al.* 2005; Daneshvar *et al.* 2008). The crystal packing diagram of the title compound is shown in Fig. 3. In the crystal packing diagram of the title compound, there are several intermolecular N—H···O, O—H···O hydrogen bonds which play an important role in stabilization of crystal structure (Table 1 and Fig. 3).

## S2. Experimental

A solution of propane-1,2-diamine (0.074 g, 1 mmol) in water (8 ml) and 2,6-pyridinedicarboxylic acid (0.501 g, 3 mmol) in water (10 ml) were mixed and stirred until clear solution obtained. Then a solution of $ZrCl_4.3H_2O$ (0.116 g, 0.5 mmol) in water (5 ml) was added to the mixture of acid-base and stirrer for 5 h. Crystals of the title compound suitable for X-ray analysis were obtained by slow evaporation after one month at room temperature (m.p: 210 °C).

## S3. Refinement

Water H atoms were found in a difference Fourier map and positional parameters were refined, $U_{iso}$(H) = 0.115 Å$^2$. Ammonium H atoms were positioned geometrically and refined as riding atoms with N—H = 0.89 Å and $U_{iso}$(H) = 1.5$U_{eq}$(N). Other H atoms were positioned geometrically and refined as riding atoms with C—H = 0.93-0.98 Å, *U*iso(H) = 1.5*U*eq(C) for methyl H atoms and 1.2*Ueq*(C) for the others.

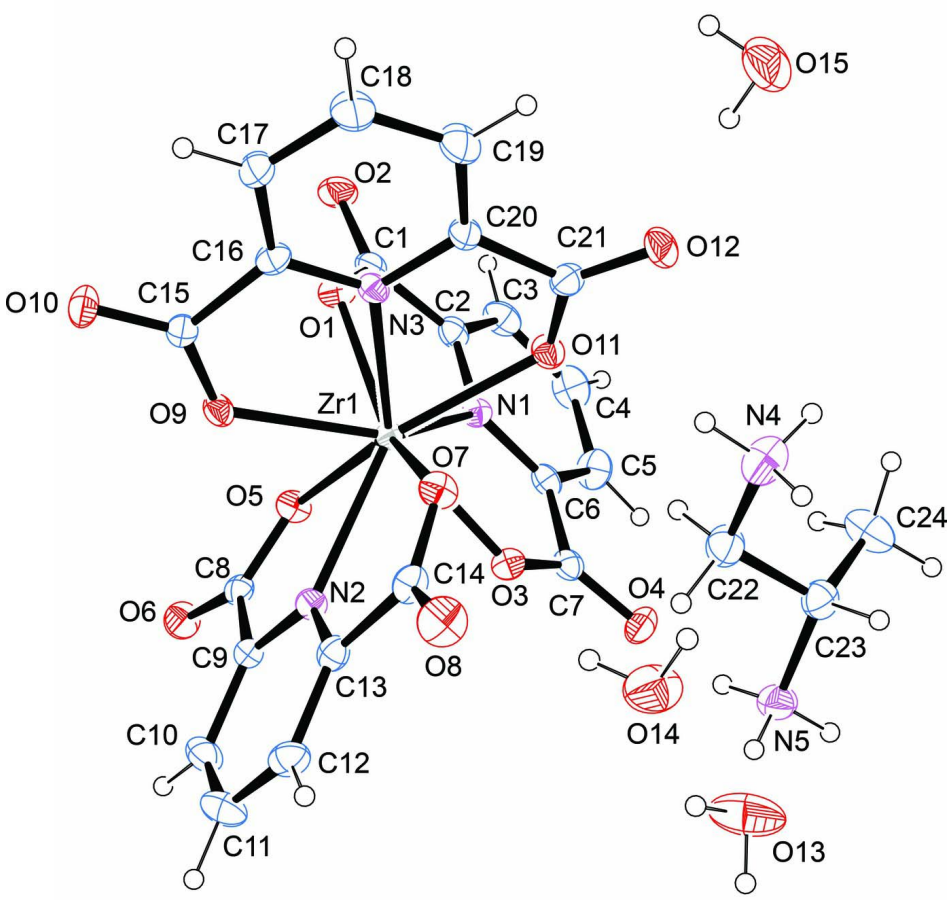


**Figure 1**

The molecular structure of the title compound with displacement ellipsoids drawn at 30% probability level.

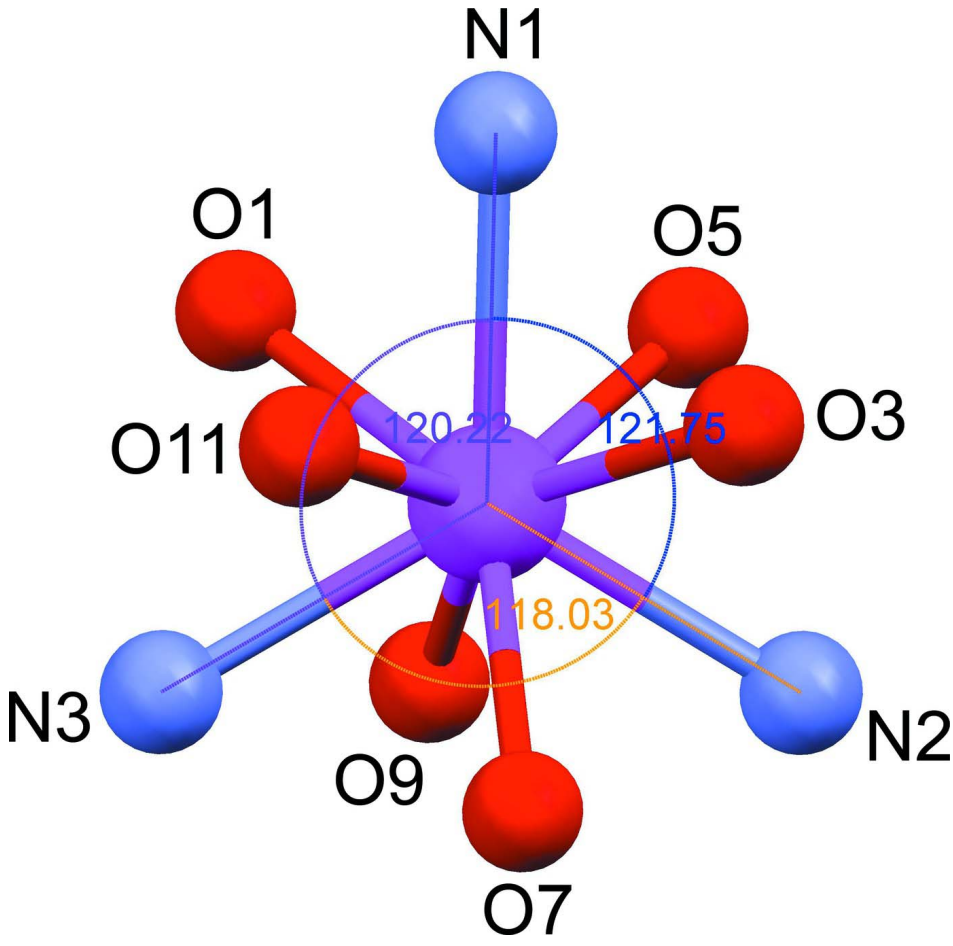


**Figure 2**

The coordination environment around $Zr^{IV}$ ion in the title compound.

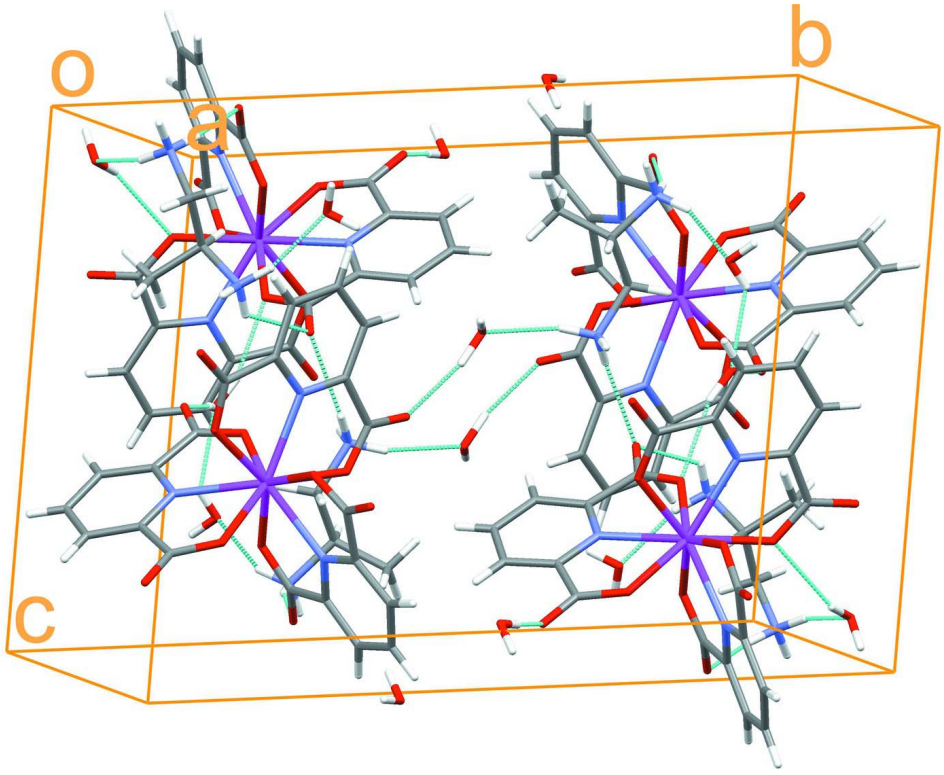


**Figure 3**

The packing diagram of the title compound. The intermolecular N—H···O, O—H···O hydrogen bonds are shown as dashed lines.

### Propane-1,2-diaminium tris(pyridine-2,6-dicarboxylato- $\kappa^3O^2,N,O^6$)zirconate(IV) trihydrate

*Crystal data*

$(C_3H_{12}N_2)[Zr(C_7H_3NO_4)_3]\cdot 3H_2O$
$M_r = 716.73$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 10.515$ (2) Å
$b = 19.195$ (4) Å
$c = 14.149$ (3) Å
$\beta = 103.39$ (3)°
$V = 2778.1$ (10) Å$^3$
$Z = 4$
$F(000) = 1464$
$D_x = 1.714$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7481 reflections
$\theta = 2.1$–29.2°
$\mu = 0.48$ mm$^{-1}$
$T = 298$ K
Prism, colorless
0.25 × 0.15 × 0.15 mm

*Data collection*

Stoe IPDS II diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
rotation method scans
Absorption correction: numerical (*X-SHAPE* and *X-RED32*; Stoe & Cie, 2005)
$T_{min} = 0.915$, $T_{max} = 0.926$
21838 measured reflections
7481 independent reflections
5264 reflections with $I > 2\sigma(I)$
$R_{int} = 0.133$
$\theta_{max} = 29.2°$, $\theta_{min} = 2.1°$
$h = -14\rightarrow11$
$k = -26\rightarrow26$
$l = -19\rightarrow19$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.086$
$wR(F^2) = 0.206$
$S = 1.18$
7481 reflections
427 parameters
7 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0753P)^2 + 3.9962P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.09$ e Å$^{-3}$
$\Delta\rho_{min} = -0.77$ e Å$^{-3}$

*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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C18 | 1.0667 (8) | 0.5828 (4) | 0.6397 (5) | 0.0486 (18) |
| H18 | 1.0902 | 0.5663 | 0.7032 | 0.058* |
| O13 | 0.3681 (10) | 0.8559 (3) | 0.2972 (5) | 0.084 (2) |
| O14 | 0.5141 (7) | 0.8422 (4) | 0.4841 (5) | 0.0666 (18) |
| Zr1 | 0.93403 (5) | 0.68252 (3) | 0.29848 (3) | 0.02015 (14) |
| O11 | 0.7886 (4) | 0.6046 (2) | 0.3278 (3) | 0.0299 (9) |
| O5 | 1.0314 (4) | 0.7169 (2) | 0.1812 (3) | 0.0285 (8) |
| O9 | 1.1276 (4) | 0.7203 (2) | 0.3786 (3) | 0.0281 (8) |
| O3 | 0.7472 (4) | 0.7187 (2) | 0.2025 (3) | 0.0281 (8) |
| N2 | 0.9428 (5) | 0.8044 (2) | 0.2844 (3) | 0.0250 (9) |
| O7 | 0.8366 (4) | 0.7374 (2) | 0.4015 (3) | 0.0318 (9) |
| O1 | 1.0691 (4) | 0.5933 (2) | 0.2947 (3) | 0.0311 (9) |
| N3 | 0.9968 (5) | 0.6338 (2) | 0.4535 (3) | 0.0242 (9) |
| N1 | 0.8636 (5) | 0.6103 (2) | 0.1615 (3) | 0.0242 (9) |
| O4 | 0.5741 (4) | 0.7014 (3) | 0.0797 (3) | 0.0364 (10) |
| C7 | 0.6845 (6) | 0.6870 (3) | 0.1263 (4) | 0.0279 (11) |
| C15 | 1.1900 (6) | 0.6989 (3) | 0.4611 (4) | 0.0254 (11) |
| O6 | 1.1211 (5) | 0.7982 (3) | 0.1045 (3) | 0.0385 (10) |
| O2 | 1.1365 (5) | 0.5005 (2) | 0.2250 (4) | 0.0421 (11) |
| O8 | 0.7646 (6) | 0.8342 (3) | 0.4614 (4) | 0.0499 (13) |
| C6 | 0.7542 (6) | 0.6251 (3) | 0.0975 (4) | 0.0278 (11) |
| C2 | 0.9348 (6) | 0.5542 (3) | 0.1484 (4) | 0.0282 (11) |
| C9 | 1.0020 (6) | 0.8333 (3) | 0.2204 (4) | 0.0267 (11) |
| C1 | 1.0562 (6) | 0.5475 (3) | 0.2271 (4) | 0.0276 (11) |

| | | | | |
|---|---|---|---|---|
| C8 | 1.0568 (5) | 0.7797 (3) | 0.1625 (4) | 0.0266 (11) |
| C14 | 0.8231 (6) | 0.8035 (3) | 0.4075 (4) | 0.0307 (12) |
| C13 | 0.8855 (6) | 0.8446 (3) | 0.3394 (4) | 0.0287 (12) |
| O10 | 1.3031 (4) | 0.7132 (3) | 0.5023 (3) | 0.0359 (10) |
| O12 | 0.6980 (5) | 0.5485 (3) | 0.4335 (3) | 0.0429 (11) |
| C16 | 1.1115 (6) | 0.6500 (3) | 0.5102 (4) | 0.0273 (11) |
| C4 | 0.7813 (8) | 0.5259 (4) | 0.0028 (5) | 0.0460 (17) |
| H4 | 0.7531 | 0.4974 | −0.0511 | 0.055* |
| C20 | 0.9149 (6) | 0.5919 (3) | 0.4871 (4) | 0.0283 (11) |
| C21 | 0.7902 (6) | 0.5797 (3) | 0.4117 (4) | 0.0308 (12) |
| C3 | 0.8944 (7) | 0.5103 (3) | 0.0707 (5) | 0.0375 (14) |
| H3 | 0.9426 | 0.4707 | 0.0639 | 0.045* |
| C11 | 0.9459 (8) | 0.9464 (3) | 0.2640 (5) | 0.0440 (17) |
| H11 | 0.9467 | 0.9946 | 0.2570 | 0.053* |
| C5 | 0.7101 (7) | 0.5843 (4) | 0.0153 (5) | 0.0406 (15) |
| H5 | 0.6342 | 0.5961 | −0.0303 | 0.049* |
| C19 | 0.9465 (8) | 0.5654 (4) | 0.5800 (5) | 0.0440 (16) |
| H19 | 0.8887 | 0.5365 | 0.6022 | 0.053* |
| C12 | 0.8857 (7) | 0.9168 (3) | 0.3310 (5) | 0.0378 (14) |
| H12 | 0.8460 | 0.9445 | 0.3698 | 0.045* |
| C17 | 1.1515 (6) | 0.6252 (4) | 0.6036 (4) | 0.0363 (14) |
| H17 | 1.2335 | 0.6365 | 0.6417 | 0.044* |
| C10 | 1.0048 (7) | 0.9048 (3) | 0.2074 (5) | 0.0380 (14) |
| H10 | 1.0453 | 0.9241 | 0.1617 | 0.046* |
| N5 | 0.3816 (6) | 0.7484 (3) | 0.1693 (4) | 0.0407 (13) |
| H5A | 0.3872 | 0.7903 | 0.1969 | 0.061* |
| H5B | 0.3091 | 0.7460 | 0.1222 | 0.061* |
| H5C | 0.4509 | 0.7413 | 0.1444 | 0.061* |
| C23 | 0.3774 (7) | 0.6935 (4) | 0.2445 (5) | 0.0379 (14) |
| H23 | 0.3049 | 0.7040 | 0.2751 | 0.045* |
| C22 | 0.5033 (7) | 0.6975 (4) | 0.3206 (5) | 0.0419 (16) |
| H22A | 0.5725 | 0.6760 | 0.2958 | 0.050* |
| H22B | 0.5263 | 0.7460 | 0.3342 | 0.050* |
| N4 | 0.4933 (6) | 0.6623 (4) | 0.4117 (4) | 0.0493 (16) |
| H4A | 0.4389 | 0.6860 | 0.4394 | 0.074* |
| H4B | 0.5720 | 0.6605 | 0.4520 | 0.074* |
| H4C | 0.4631 | 0.6192 | 0.3983 | 0.074* |
| C24 | 0.3509 (10) | 0.6242 (4) | 0.1930 (5) | 0.053 (2) |
| H24A | 0.4143 | 0.6165 | 0.1549 | 0.080* |
| H24B | 0.2648 | 0.6246 | 0.1512 | 0.080* |
| H24C | 0.3569 | 0.5877 | 0.2401 | 0.080* |
| O15 | 0.6503 (7) | 0.4645 (4) | 0.5884 (5) | 0.077 (2) |
| H15A | 0.672 (13) | 0.485 (7) | 0.531 (7) | 0.115* |
| H14A | 0.497 (14) | 0.820 (7) | 0.537 (8) | 0.115* |
| H14B | 0.592 (6) | 0.835 (7) | 0.479 (10) | 0.115* |
| H13A | 0.426 (11) | 0.870 (7) | 0.340 (8) | 0.115* |
| H13B | 0.342 (13) | 0.892 (4) | 0.271 (8) | 0.115* |
| H15B | 0.728 (6) | 0.453 (8) | 0.601 (10) | 0.115* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C18 | 0.053 (4) | 0.059 (4) | 0.029 (3) | −0.008 (4) | 0.000 (3) | 0.019 (3) |
| O13 | 0.141 (8) | 0.037 (3) | 0.070 (4) | 0.017 (4) | 0.017 (5) | −0.005 (3) |
| O14 | 0.072 (4) | 0.072 (4) | 0.066 (4) | 0.018 (4) | 0.037 (3) | 0.026 (3) |
| Zr1 | 0.0211 (2) | 0.0215 (2) | 0.0184 (2) | 0.0005 (2) | 0.00570 (16) | 0.0007 (2) |
| O11 | 0.032 (2) | 0.032 (2) | 0.0257 (18) | −0.0045 (17) | 0.0058 (16) | 0.0019 (16) |
| O5 | 0.035 (2) | 0.027 (2) | 0.0275 (19) | 0.0021 (17) | 0.0147 (17) | −0.0016 (15) |
| O9 | 0.029 (2) | 0.028 (2) | 0.0282 (19) | −0.0051 (16) | 0.0097 (16) | 0.0039 (15) |
| O3 | 0.030 (2) | 0.029 (2) | 0.0250 (18) | 0.0030 (16) | 0.0057 (16) | −0.0003 (15) |
| N2 | 0.026 (2) | 0.024 (2) | 0.027 (2) | 0.0010 (18) | 0.0093 (18) | −0.0020 (16) |
| O7 | 0.038 (2) | 0.034 (2) | 0.0275 (19) | 0.0006 (18) | 0.0161 (18) | 0.0001 (17) |
| O1 | 0.037 (2) | 0.031 (2) | 0.0264 (19) | 0.0058 (18) | 0.0080 (17) | 0.0005 (16) |
| N3 | 0.027 (2) | 0.024 (2) | 0.023 (2) | −0.0020 (18) | 0.0079 (18) | 0.0026 (17) |
| N1 | 0.026 (2) | 0.025 (2) | 0.023 (2) | 0.0015 (18) | 0.0070 (18) | 0.0011 (17) |
| O4 | 0.024 (2) | 0.047 (3) | 0.037 (2) | 0.0043 (18) | 0.0039 (18) | 0.0041 (19) |
| C7 | 0.030 (3) | 0.028 (3) | 0.027 (2) | −0.003 (2) | 0.010 (2) | 0.004 (2) |
| C15 | 0.025 (3) | 0.024 (3) | 0.027 (2) | 0.001 (2) | 0.007 (2) | −0.0047 (19) |
| O6 | 0.037 (2) | 0.049 (3) | 0.034 (2) | −0.001 (2) | 0.0153 (19) | 0.0118 (19) |
| O2 | 0.043 (3) | 0.026 (2) | 0.059 (3) | 0.010 (2) | 0.017 (2) | 0.004 (2) |
| O8 | 0.056 (3) | 0.053 (3) | 0.050 (3) | 0.007 (2) | 0.030 (3) | −0.011 (2) |
| C6 | 0.032 (3) | 0.028 (3) | 0.027 (2) | −0.006 (2) | 0.012 (2) | 0.002 (2) |
| C2 | 0.030 (3) | 0.027 (3) | 0.031 (3) | −0.002 (2) | 0.014 (2) | 0.000 (2) |
| C9 | 0.023 (3) | 0.026 (3) | 0.030 (3) | 0.000 (2) | 0.004 (2) | 0.003 (2) |
| C1 | 0.030 (3) | 0.020 (2) | 0.035 (3) | 0.001 (2) | 0.013 (2) | 0.003 (2) |
| C8 | 0.023 (3) | 0.033 (3) | 0.023 (2) | 0.001 (2) | 0.003 (2) | 0.006 (2) |
| C14 | 0.028 (3) | 0.037 (3) | 0.028 (3) | 0.003 (2) | 0.009 (2) | −0.006 (2) |
| C13 | 0.026 (3) | 0.031 (3) | 0.027 (3) | 0.000 (2) | 0.003 (2) | −0.005 (2) |
| O10 | 0.023 (2) | 0.049 (3) | 0.034 (2) | −0.0060 (19) | 0.0039 (17) | 0.001 (2) |
| O12 | 0.036 (2) | 0.050 (3) | 0.045 (2) | −0.011 (2) | 0.013 (2) | 0.016 (2) |
| C16 | 0.029 (3) | 0.029 (3) | 0.023 (2) | 0.004 (2) | 0.003 (2) | −0.001 (2) |
| C4 | 0.053 (4) | 0.048 (4) | 0.039 (3) | −0.011 (3) | 0.014 (3) | −0.014 (3) |
| C20 | 0.028 (3) | 0.030 (3) | 0.026 (2) | −0.002 (2) | 0.006 (2) | 0.004 (2) |
| C21 | 0.028 (3) | 0.033 (3) | 0.031 (3) | 0.004 (2) | 0.007 (2) | 0.003 (2) |
| C3 | 0.050 (4) | 0.028 (3) | 0.039 (3) | −0.002 (3) | 0.019 (3) | −0.005 (2) |
| C11 | 0.063 (5) | 0.024 (3) | 0.045 (3) | −0.002 (3) | 0.013 (3) | −0.003 (3) |
| C5 | 0.040 (4) | 0.049 (4) | 0.031 (3) | −0.004 (3) | 0.003 (3) | −0.008 (3) |
| C19 | 0.047 (4) | 0.050 (4) | 0.036 (3) | −0.008 (3) | 0.011 (3) | 0.013 (3) |
| C12 | 0.041 (4) | 0.029 (3) | 0.040 (3) | 0.007 (3) | 0.002 (3) | −0.008 (3) |
| C17 | 0.031 (3) | 0.041 (3) | 0.032 (3) | −0.003 (3) | 0.000 (2) | 0.007 (3) |
| C10 | 0.044 (4) | 0.032 (3) | 0.037 (3) | −0.008 (3) | 0.009 (3) | 0.008 (2) |
| N5 | 0.042 (3) | 0.038 (3) | 0.043 (3) | 0.004 (2) | 0.012 (3) | 0.009 (2) |
| C23 | 0.035 (3) | 0.045 (4) | 0.037 (3) | 0.002 (3) | 0.013 (3) | 0.004 (3) |
| C22 | 0.037 (4) | 0.049 (4) | 0.040 (3) | −0.002 (3) | 0.007 (3) | 0.004 (3) |
| N4 | 0.044 (3) | 0.072 (4) | 0.032 (3) | 0.008 (3) | 0.010 (3) | 0.001 (3) |
| C24 | 0.079 (6) | 0.041 (4) | 0.040 (4) | −0.013 (4) | 0.013 (4) | 0.001 (3) |
| O15 | 0.071 (4) | 0.091 (5) | 0.068 (4) | −0.024 (4) | 0.014 (4) | 0.031 (4) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C18—C17 | 1.388 (10) | C9—C10 | 1.387 (8) |
| C18—C19 | 1.389 (10) | C9—C8 | 1.509 (8) |
| C18—H18 | 0.9300 | C14—C13 | 1.508 (9) |
| O13—H13A | 0.80 (8) | C13—C12 | 1.392 (9) |
| O13—H13B | 0.80 (4) | O12—C21 | 1.239 (8) |
| O14—H14A | 0.91 (8) | C16—C17 | 1.376 (8) |
| O14—H14B | 0.85 (4) | C4—C3 | 1.378 (10) |
| Zr1—O9 | 2.210 (4) | C4—C5 | 1.382 (11) |
| Zr1—O3 | 2.225 (4) | C4—H4 | 0.9300 |
| Zr1—O7 | 2.230 (4) | C20—C19 | 1.377 (8) |
| Zr1—O1 | 2.233 (4) | C20—C21 | 1.504 (8) |
| Zr1—O5 | 2.241 (4) | C3—H3 | 0.9300 |
| Zr1—O11 | 2.245 (4) | C11—C10 | 1.375 (10) |
| Zr1—N3 | 2.334 (4) | C11—C12 | 1.379 (11) |
| Zr1—N2 | 2.352 (4) | C11—H11 | 0.9300 |
| Zr1—N1 | 2.358 (4) | C5—H5 | 0.9300 |
| O11—C21 | 1.277 (7) | C19—H19 | 0.9300 |
| O5—C8 | 1.275 (7) | C12—H12 | 0.9300 |
| O9—C15 | 1.267 (7) | C17—H17 | 0.9300 |
| O3—C7 | 1.280 (7) | C10—H10 | 0.9300 |
| N2—C9 | 1.333 (7) | N5—C23 | 1.506 (8) |
| N2—C13 | 1.334 (7) | N5—H5A | 0.8900 |
| O7—C14 | 1.283 (7) | N5—H5B | 0.8900 |
| O1—C1 | 1.283 (7) | N5—H5C | 0.8900 |
| N3—C16 | 1.321 (7) | C23—C22 | 1.502 (9) |
| N3—C20 | 1.343 (7) | C23—C24 | 1.510 (10) |
| N1—C6 | 1.320 (7) | C23—H23 | 0.9800 |
| N1—C2 | 1.350 (7) | C22—N4 | 1.480 (9) |
| O4—C7 | 1.226 (7) | C22—H22A | 0.9700 |
| C7—C6 | 1.502 (8) | C22—H22B | 0.9700 |
| C15—O10 | 1.227 (7) | N4—H4A | 0.8900 |
| C15—C16 | 1.521 (8) | N4—H4B | 0.8900 |
| O6—C8 | 1.230 (7) | N4—H4C | 0.8900 |
| O2—C1 | 1.241 (7) | C24—H24A | 0.9600 |
| O8—C14 | 1.233 (8) | C24—H24B | 0.9600 |
| C6—C5 | 1.390 (8) | C24—H24C | 0.9600 |
| C2—C3 | 1.372 (8) | O15—H15A | 0.98 (9) |
| C2—C1 | 1.492 (8) | O15—H15B | 0.83 (4) |
| | | | |
| C17—C18—C19 | 119.2 (6) | O6—C8—O5 | 125.9 (6) |
| C17—C18—H18 | 120.4 | O6—C8—C9 | 120.2 (5) |
| C19—C18—H18 | 120.4 | O5—C8—C9 | 114.0 (5) |
| H13A—O13—H13B | 100 (8) | O8—C14—O7 | 126.3 (6) |
| H14A—O14—H14B | 111 (10) | O8—C14—C13 | 119.9 (6) |
| O9—Zr1—O3 | 142.08 (15) | O7—C14—C13 | 113.7 (5) |
| O9—Zr1—O7 | 91.02 (16) | N2—C13—C12 | 121.0 (6) |

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|---|---|---|---|
| O3—Zr1—O7 | 77.11 (15) | N2—C13—C14 | 113.2 (5) |
| O9—Zr1—O1 | 75.43 (16) | C12—C13—C14 | 125.8 (6) |
| O3—Zr1—O1 | 134.53 (15) | N3—C16—C17 | 122.3 (6) |
| O7—Zr1—O1 | 140.25 (15) | N3—C16—C15 | 112.5 (5) |
| O9—Zr1—O5 | 76.03 (15) | C17—C16—C15 | 125.1 (5) |
| O3—Zr1—O5 | 86.94 (15) | C3—C4—C5 | 119.6 (6) |
| O7—Zr1—O5 | 134.60 (16) | C3—C4—H4 | 120.2 |
| O1—Zr1—O5 | 78.81 (15) | C5—C4—H4 | 120.2 |
| O9—Zr1—O11 | 135.19 (14) | N3—C20—C19 | 121.8 (6) |
| O3—Zr1—O11 | 77.16 (15) | N3—C20—C21 | 111.6 (5) |
| O7—Zr1—O11 | 76.04 (16) | C19—C20—C21 | 126.7 (6) |
| O1—Zr1—O11 | 87.57 (16) | O12—C21—O11 | 124.8 (6) |
| O5—Zr1—O11 | 141.28 (15) | O12—C21—C20 | 120.2 (5) |
| O9—Zr1—N3 | 67.84 (15) | O11—C21—C20 | 115.0 (5) |
| O3—Zr1—N3 | 135.72 (16) | C2—C3—C4 | 118.8 (6) |
| O7—Zr1—N3 | 69.39 (16) | C2—C3—H3 | 120.6 |
| O1—Zr1—N3 | 70.89 (15) | C4—C3—H3 | 120.6 |
| O5—Zr1—N3 | 137.31 (16) | C10—C11—C12 | 120.1 (6) |
| O11—Zr1—N3 | 67.44 (15) | C10—C11—H11 | 120.0 |
| O9—Zr1—N2 | 70.58 (16) | C12—C11—H11 | 120.0 |
| O3—Zr1—N2 | 71.60 (16) | C4—C5—C6 | 118.5 (6) |
| O7—Zr1—N2 | 67.56 (16) | C4—C5—H5 | 120.7 |
| O1—Zr1—N2 | 136.30 (17) | C6—C5—H5 | 120.7 |
| O5—Zr1—N2 | 67.09 (15) | C20—C19—C18 | 118.5 (6) |
| O11—Zr1—N2 | 136.09 (16) | C20—C19—H19 | 120.8 |
| N3—Zr1—N2 | 118.01 (16) | C18—C19—H19 | 120.8 |
| O9—Zr1—N1 | 133.70 (16) | C11—C12—C13 | 118.7 (6) |
| O3—Zr1—N1 | 67.48 (15) | C11—C12—H12 | 120.7 |
| O7—Zr1—N1 | 135.26 (16) | C13—C12—H12 | 120.7 |
| O1—Zr1—N1 | 67.05 (15) | C16—C17—C18 | 118.5 (6) |
| O5—Zr1—N1 | 71.04 (16) | C16—C17—H17 | 120.8 |
| O11—Zr1—N1 | 70.27 (16) | C18—C17—H17 | 120.8 |
| N3—Zr1—N1 | 120.25 (16) | C11—C10—C9 | 118.2 (6) |
| N2—Zr1—N1 | 121.74 (15) | C11—C10—H10 | 120.9 |
| C21—O11—Zr1 | 123.9 (4) | C9—C10—H10 | 120.9 |
| C8—O5—Zr1 | 125.9 (4) | C23—N5—H5A | 109.5 |
| C15—O9—Zr1 | 125.8 (4) | C23—N5—H5B | 109.5 |
| C7—O3—Zr1 | 125.1 (4) | H5A—N5—H5B | 109.5 |
| C9—N2—C13 | 120.1 (5) | C23—N5—H5C | 109.5 |
| C9—N2—Zr1 | 120.3 (4) | H5A—N5—H5C | 109.5 |
| C13—N2—Zr1 | 119.6 (4) | H5B—N5—H5C | 109.5 |
| C14—O7—Zr1 | 125.9 (4) | C22—C23—N5 | 107.8 (6) |
| C1—O1—Zr1 | 125.8 (4) | C22—C23—C24 | 115.1 (7) |
| C16—N3—C20 | 119.7 (5) | N5—C23—C24 | 107.9 (6) |
| C16—N3—Zr1 | 119.8 (4) | C22—C23—H23 | 108.6 |
| C20—N3—Zr1 | 120.5 (4) | N5—C23—H23 | 108.6 |
| C6—N1—C2 | 120.1 (5) | C24—C23—H23 | 108.6 |
| C6—N1—Zr1 | 119.7 (4) | N4—C22—C23 | 111.8 (6) |

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| C2—N1—Zr1 | 120.2 (4) | N4—C22—H22A | 109.3 |
| O4—C7—O3 | 125.8 (6) | C23—C22—H22A | 109.3 |
| O4—C7—C6 | 119.5 (5) | N4—C22—H22B | 109.3 |
| O3—C7—C6 | 114.6 (5) | C23—C22—H22B | 109.3 |
| O10—C15—O9 | 127.1 (6) | H22A—C22—H22B | 107.9 |
| O10—C15—C16 | 119.3 (5) | C22—N4—H4A | 109.5 |
| O9—C15—C16 | 113.6 (5) | C22—N4—H4B | 109.5 |
| N1—C6—C5 | 121.5 (6) | H4A—N4—H4B | 109.5 |
| N1—C6—C7 | 112.7 (5) | C22—N4—H4C | 109.5 |
| C5—C6—C7 | 125.7 (6) | H4A—N4—H4C | 109.5 |
| N1—C2—C3 | 121.4 (6) | H4B—N4—H4C | 109.5 |
| N1—C2—C1 | 111.8 (5) | C23—C24—H24A | 109.5 |
| C3—C2—C1 | 126.7 (6) | C23—C24—H24B | 109.5 |
| N2—C9—C10 | 121.9 (6) | H24A—C24—H24B | 109.5 |
| N2—C9—C8 | 112.5 (5) | C23—C24—H24C | 109.5 |
| C10—C9—C8 | 125.5 (6) | H24A—C24—H24C | 109.5 |
| O2—C1—O1 | 124.1 (6) | H24B—C24—H24C | 109.5 |
| O2—C1—C2 | 120.9 (5) | H15A—O15—H15B | 83 (10) |
| O1—C1—C2 | 115.0 (5) | | |

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|---|---|---|---|
| O9—Zr1—O11—C21 | 15.8 (6) | O9—Zr1—N1—C2 | 38.3 (5) |
| O3—Zr1—O11—C21 | −140.9 (5) | O3—Zr1—N1—C2 | −179.9 (5) |
| O7—Zr1—O11—C21 | −61.2 (5) | O7—Zr1—N1—C2 | −139.5 (4) |
| O1—Zr1—O11—C21 | 82.2 (5) | O1—Zr1—N1—C2 | −0.2 (4) |
| O5—Zr1—O11—C21 | 150.9 (4) | O5—Zr1—N1—C2 | 85.4 (4) |
| N3—Zr1—O11—C21 | 11.9 (4) | O11—Zr1—N1—C2 | −96.1 (4) |
| N2—Zr1—O11—C21 | −95.6 (5) | N3—Zr1—N1—C2 | −49.0 (5) |
| N1—Zr1—O11—C21 | 148.7 (5) | N2—Zr1—N1—C2 | 131.2 (4) |
| O9—Zr1—O5—C8 | −69.6 (4) | Zr1—O3—C7—O4 | 170.3 (4) |
| O3—Zr1—O5—C8 | 76.2 (4) | Zr1—O3—C7—C6 | −6.6 (7) |
| O7—Zr1—O5—C8 | 7.7 (5) | Zr1—O9—C15—O10 | 171.2 (5) |
| O1—Zr1—O5—C8 | −147.2 (5) | Zr1—O9—C15—C16 | −8.1 (7) |
| O11—Zr1—O5—C8 | 141.2 (4) | C2—N1—C6—C5 | −0.6 (9) |
| N3—Zr1—O5—C8 | −102.2 (5) | Zr1—N1—C6—C5 | 179.3 (5) |
| N2—Zr1—O5—C8 | 4.9 (4) | C2—N1—C6—C7 | 177.0 (5) |
| N1—Zr1—O5—C8 | 143.4 (5) | Zr1—N1—C6—C7 | −3.1 (6) |
| O3—Zr1—O9—C15 | 142.8 (4) | O4—C7—C6—N1 | −171.2 (5) |
| O7—Zr1—O9—C15 | 72.6 (4) | O3—C7—C6—N1 | 5.9 (7) |
| O1—Zr1—O9—C15 | −69.5 (4) | O4—C7—C6—C5 | 6.2 (9) |
| O5—Zr1—O9—C15 | −151.4 (5) | O3—C7—C6—C5 | −176.6 (6) |
| O11—Zr1—O9—C15 | 1.6 (5) | C6—N1—C2—C3 | −1.8 (8) |
| N3—Zr1—O9—C15 | 5.4 (4) | Zr1—N1—C2—C3 | 178.3 (4) |
| N2—Zr1—O9—C15 | 138.4 (5) | C6—N1—C2—C1 | 178.6 (5) |
| N1—Zr1—O9—C15 | −105.9 (5) | Zr1—N1—C2—C1 | −1.3 (6) |
| O9—Zr1—O3—C7 | 137.0 (4) | C13—N2—C9—C10 | −1.0 (9) |
| O7—Zr1—O3—C7 | −148.3 (5) | Zr1—N2—C9—C10 | 177.4 (5) |
| O1—Zr1—O3—C7 | 3.5 (5) | C13—N2—C9—C8 | −177.9 (5) |
| O5—Zr1—O3—C7 | 74.5 (4) | Zr1—N2—C9—C8 | 0.5 (6) |

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| O11—Zr1—O3—C7 | −70.0 (4) | Zr1—O1—C1—O2 | 176.4 (4) |
| N3—Zr1—O3—C7 | −107.1 (4) | Zr1—O1—C1—C2 | −3.6 (7) |
| N2—Zr1—O3—C7 | 141.3 (5) | N1—C2—C1—O2 | −177.0 (5) |
| N1—Zr1—O3—C7 | 3.8 (4) | C3—C2—C1—O2 | 3.4 (9) |
| O9—Zr1—N2—C9 | 80.1 (4) | N1—C2—C1—O1 | 2.9 (7) |
| O3—Zr1—N2—C9 | −97.0 (4) | C3—C2—C1—O1 | −176.7 (6) |
| O7—Zr1—N2—C9 | 179.7 (5) | Zr1—O5—C8—O6 | 172.6 (4) |
| O1—Zr1—N2—C9 | 39.2 (5) | Zr1—O5—C8—C9 | −6.3 (6) |
| O5—Zr1—N2—C9 | −2.4 (4) | N2—C9—C8—O6 | −175.6 (5) |
| O11—Zr1—N2—C9 | −143.9 (4) | C10—C9—C8—O6 | 7.6 (9) |
| N3—Zr1—N2—C9 | 130.3 (4) | N2—C9—C8—O5 | 3.3 (7) |
| N1—Zr1—N2—C9 | −49.9 (5) | C10—C9—C8—O5 | −173.5 (6) |
| O9—Zr1—N2—C13 | −101.4 (4) | Zr1—O7—C14—O8 | 176.7 (5) |
| O3—Zr1—N2—C13 | 81.4 (4) | Zr1—O7—C14—C13 | −2.5 (7) |
| O7—Zr1—N2—C13 | −1.9 (4) | C9—N2—C13—C12 | 0.3 (9) |
| O1—Zr1—N2—C13 | −142.4 (4) | Zr1—N2—C13—C12 | −178.1 (4) |
| O5—Zr1—N2—C13 | 176.0 (5) | C9—N2—C13—C14 | 179.8 (5) |
| O11—Zr1—N2—C13 | 34.5 (5) | Zr1—N2—C13—C14 | 1.4 (6) |
| N3—Zr1—N2—C13 | −51.2 (5) | O8—C14—C13—N2 | −178.7 (6) |
| N1—Zr1—N2—C13 | 128.5 (4) | O7—C14—C13—N2 | 0.5 (7) |
| O9—Zr1—O7—C14 | 70.8 (5) | O8—C14—C13—C12 | 0.8 (10) |
| O3—Zr1—O7—C14 | −72.8 (5) | O7—C14—C13—C12 | −180.0 (6) |
| O1—Zr1—O7—C14 | 139.0 (4) | C20—N3—C16—C17 | 0.3 (9) |
| O5—Zr1—O7—C14 | −0.4 (6) | Zr1—N3—C16—C17 | 177.4 (5) |
| O11—Zr1—O7—C14 | −152.5 (5) | C20—N3—C16—C15 | −178.9 (5) |
| N3—Zr1—O7—C14 | 136.7 (5) | Zr1—N3—C16—C15 | −1.8 (6) |
| N2—Zr1—O7—C14 | 2.4 (5) | O10—C15—C16—N3 | −173.5 (5) |
| N1—Zr1—O7—C14 | −110.7 (5) | O9—C15—C16—N3 | 5.9 (7) |
| O9—Zr1—O1—C1 | −150.1 (5) | O10—C15—C16—C17 | 7.4 (9) |
| O3—Zr1—O1—C1 | 2.4 (6) | O9—C15—C16—C17 | −173.2 (6) |
| O7—Zr1—O1—C1 | 136.3 (4) | C16—N3—C20—C19 | 0.4 (9) |
| O5—Zr1—O1—C1 | −71.8 (5) | Zr1—N3—C20—C19 | −176.6 (5) |
| O11—Zr1—O1—C1 | 71.7 (5) | C16—N3—C20—C21 | −179.9 (5) |
| N3—Zr1—O1—C1 | 138.7 (5) | Zr1—N3—C20—C21 | 3.1 (7) |
| N2—Zr1—O1—C1 | −110.4 (5) | Zr1—O11—C21—O12 | 164.3 (5) |
| N1—Zr1—O1—C1 | 2.1 (4) | Zr1—O11—C21—C20 | −14.5 (7) |
| O9—Zr1—N3—C16 | −1.3 (4) | N3—C20—C21—O12 | −172.3 (6) |
| O3—Zr1—N3—C16 | −144.6 (4) | C19—C20—C21—O12 | 7.4 (10) |
| O7—Zr1—N3—C16 | −101.3 (4) | N3—C20—C21—O11 | 6.7 (8) |
| O1—Zr1—N3—C16 | 80.3 (4) | C19—C20—C21—O11 | −173.7 (7) |
| O5—Zr1—N3—C16 | 33.0 (5) | N1—C2—C3—C4 | 2.7 (9) |
| O11—Zr1—N3—C16 | 175.8 (5) | C1—C2—C3—C4 | −177.8 (6) |
| N2—Zr1—N3—C16 | −52.7 (5) | C5—C4—C3—C2 | −1.2 (11) |
| N1—Zr1—N3—C16 | 127.5 (4) | C3—C4—C5—C6 | −1.0 (11) |
| O9—Zr1—N3—C20 | 175.7 (5) | N1—C6—C5—C4 | 1.9 (10) |
| O3—Zr1—N3—C20 | 32.4 (5) | C7—C6—C5—C4 | −175.3 (6) |
| O7—Zr1—N3—C20 | 75.7 (4) | N3—C20—C19—C18 | −0.1 (11) |
| O1—Zr1—N3—C20 | −102.7 (5) | C21—C20—C19—C18 | −179.7 (7) |

| | | | |
|---|---|---|---|
| O5—Zr1—N3—C20 | −150.0 (4) | C17—C18—C19—C20 | −1.0 (12) |
| O11—Zr1—N3—C20 | −7.2 (4) | C10—C11—C12—C13 | −0.3 (10) |
| N2—Zr1—N3—C20 | 124.3 (4) | N2—C13—C12—C11 | 0.4 (10) |
| N1—Zr1—N3—C20 | −55.5 (5) | C14—C13—C12—C11 | −179.1 (6) |
| O9—Zr1—N1—C6 | −141.6 (4) | N3—C16—C17—C18 | −1.4 (10) |
| O3—Zr1—N1—C6 | 0.2 (4) | C15—C16—C17—C18 | 177.6 (6) |
| O7—Zr1—N1—C6 | 40.6 (5) | C19—C18—C17—C16 | 1.8 (12) |
| O1—Zr1—N1—C6 | 179.9 (5) | C12—C11—C10—C9 | −0.3 (10) |
| O5—Zr1—N1—C6 | −94.5 (4) | N2—C9—C10—C11 | 1.0 (10) |
| O11—Zr1—N1—C6 | 84.0 (4) | C8—C9—C10—C11 | 177.5 (6) |
| N3—Zr1—N1—C6 | 131.1 (4) | N5—C23—C22—N4 | 162.0 (6) |
| N2—Zr1—N1—C6 | −48.7 (5) | C24—C23—C22—N4 | −77.5 (8) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O13—H13*A*···O14 | 0.80 (8) | 2.10 (9) | 2.745 (11) | 138 (13) |
| O13—H13*B*···O2$^{i}$ | 0.80 (4) | 2.10 (8) | 2.793 (8) | 145 (12) |
| O14—H14*A*···O5$^{ii}$ | 0.91 (8) | 2.11 (9) | 2.978 (7) | 158 (12) |
| O14—H14*B*···O8 | 0.85 (4) | 1.89 (5) | 2.731 (9) | 171 (14) |
| O15—H15*A*···O12 | 0.98 (9) | 1.91 (10) | 2.857 (8) | 164 (12) |
| O15—H15*B*···O1$^{iii}$ | 0.83 (4) | 2.46 (7) | 3.227 (9) | 155 (13) |
| N4—H4*A*···O10$^{iv}$ | 0.89 | 1.92 | 2.791 (8) | 165 |
| N4—H4*B*···O6$^{ii}$ | 0.89 | 2.24 | 2.851 (7) | 125 |
| N4—H4*C*···O15$^{v}$ | 0.89 | 2.04 | 2.864 (10) | 154 |
| N5—H5*A*···O13 | 0.89 | 1.94 | 2.769 (9) | 154 |
| N5—H5*B*···O6$^{iv}$ | 0.89 | 2.18 | 2.844 (8) | 131 |
| N5—H5*C*···O4 | 0.89 | 1.91 | 2.776 (8) | 163 |

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