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# Bis(3,5-dimethyl-1*H*-pyrazole-$\kappa N^2$)-bis(3,3″,5,5″-tetramethyl-[1,1′:3′,1″-terphenyl]-2′-carboxylato-$\kappa O$)iron(II) dichloromethane monosolvate

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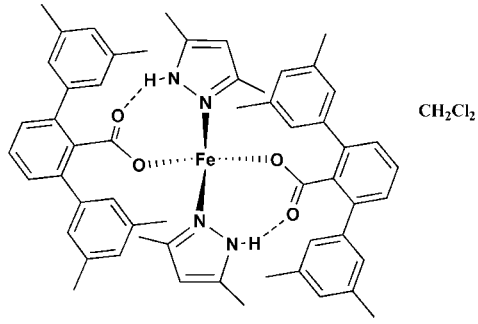
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.053; $wR$ factor = 0.149; data-to-parameter ratio = 15.1.

In the title compound, $[Fe(C_{23}H_{21}O_2)_2(C_5H_8N_2)_2]\cdot CH_2Cl_2$, the $Fe^{2+}$ cation is coordinated by the N atoms of two 3,5-dimethylpyrazole ligands and the carboxylate O atoms from two tetramethylterphenylcarboxylate ligands, forming an $FeN_2O_2$ polyhedron with a slightly distorted tetrahedral coordination geometry. Intramolecular N—H···O and C—H···O hydrogen-bonding interactions stabilize the molecular conformation. The dihedral angles formed by the central benzene ring with the outer benzene rings of the terphenyl groups are 47.92 (8), 59.38 (8), 48.24 (8) and 52.37 (8)°. The dichloromethane solvent molecule interacts with the complex molecule *via* a C—H···O hydrogen bond. In the crystal, centrosymmetrically related complex molecules are linked into dimers through pairs of C—H···O hydrogen bonds.

## Related literature

For the synthesis of substituted terphenyl-based carboxylate ligands, see: Saednya & Hart (1996); Du *et al.* (1986); Chen & Siegel (1994). For background to metal complexes with terphenyl-based carboxylate and 3,5-dimethylpyrazole ligands, see: Hagadorn *et al.* (1998); Chakravorty *et al.* (2011); Kannan *et al.* (2011); Tolman & Que (2002); Zhang *et al.* (2007); Cheng *et al.* (1990).




## Experimental

### Crystal data

$[Fe(C_{23}H_{21}O_2)_2(C_5H_8N_2)_2]\cdot CH_2Cl_2$
$M_r$ = 991.84
Triclinic, $P\overline{1}$
$a$ = 12.363 (3) Å
$b$ = 14.796 (3) Å
$c$ = 16.735 (3) Å
$\alpha$ = 111.11 (3)°
$\beta$ = 91.19 (3)°
$\gamma$ = 109.06 (3)°
$V$ = 2665.7 (13) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.43 mm$^{-1}$
$T$ = 293 K
0.32 × 0.12 × 0.10 mm

### Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min}$ = 0.839, $T_{max}$ = 1.0
20058 measured reflections
9561 independent reflections
8147 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.018

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.149
$S$ = 1.07
9561 reflections
633 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 1.13 e Å$^{-3}$
$\Delta\rho_{min}$ = −1.06 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N4—H4···O4 | 0.84 (3) | 1.95 (3) | 2.744 (3) | 158 (3) |
| N2—H2···O2 | 0.80 (3) | 2.14 (3) | 2.767 (3) | 135 (3) |
| C6—H6*C*···O1 | 0.96 | 2.40 | 3.256 (4) | 148 |
| C46—H46···O3 | 0.93 | 2.46 | 3.055 (4) | 122 |
| C54—H54···O3 | 0.93 | 2.51 | 3.037 (3) | 116 |
| C55—H55*B*···O1 | 0.96 | 2.54 | 3.481 (3) | 166 |
| C58—H58*A*···O4 | 0.97 | 2.48 | 3.326 (5) | 145 |
| C3—H3···O4$^{i}$ | 0.93 | 2.58 | 3.411 (5) | 149 |

Symmetry code: (i) $-x, -y+1, -z$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2736).

## References


Bruker (2000). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Chakravorty, S., Platts, J. A. & Das, B. K. (2011). *Dalton Trans.* **40**, 11605–11612.

Chen, C. T. & Siegel, J. S. (1994). *J. Am. Chem. Soc.* **116**, 5959–5960.

Cheng, C.-H., Lain, J.-S., Wu, Y.-J. & Wang, S.-L. (1990). *Acta Cryst.* C**46**, 208–210.

Du, C. J. F., Hart, H. & Ng, K. K. D. (1986). *J. Org. Chem.* **51**, 3162–3165.

Hagadorn, J. R., Que, L. Jr & Tolman, W. B. (1998). *J. Am. Chem. Soc.* **120**, 13531–13532.

Kannan, S., Venkatachalam, G., Lee, H. J., Min, B. K., Kim, W., Koo, E., Do, Y. R. & Yoon, S. (2011). *Polyhedron*, **30**, 340–346.

Saednya, A. & Hart, H. (1996). *Synthesis*, pp. 1455–1458.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Tolman, W. B. & Que, L. Jr (2002). *J. Chem. Soc. Dalton Trans.* pp. 653–660.

Zhang, X.-J., Han, J., Wang, C.-G. & Xing, Y.-H. (2007). *Acta Cryst.* E**63**, m2620–m2621.

# supporting information

*Acta Cryst.* (2012). E**68**, m631–m632 [doi:10.1107/S1600536812015553]

# Bis(3,5-dimethyl-1*H*-pyrazole-$\kappa N^2$)bis(3,3″,5,5″-tetramethyl-[1,1′:3′,1″-terphenyl]-2′-carboxylato-$\kappa O$)iron(II) dichloromethane monosolvate

**Yeojin Jeon, Dharmalingam Sivanesan and Sungho Yoon**

## S1. Comment

The synthetic routes for sterically hindered terphenyl-based carboxylate ligands have been investigated (Saednya & Hart, 1996; Du *et al.*, 1986; Chen & Siegel, 1994). Recently, $Fe^{2+}$ binuclear complexes of 3,3″,5,5″-tetramethyl-[1,1′:3′,1″-terphenyl]-2′-carboxylate have been synthesized for modeling dioxygen activation sites in diiron-containing proteins (Tolman & Que 2002). Four-coordinate $Fe^{2+}$ and $Co^{2+}$ metal complexes with a slightly distorted tetrahedral coordination geometry were reported with two 3,5-dimethylpyrazole and two benzoate ligands with less bulky substituents (Hagadorn *et al.*, 1998; Chakravorty *et al.*, 2011; Kannan *et al.*, 2011). Complexes with 3,5-dimethylpyrazole ligands have also been reported (Zhang *et al.*, 2007; Cheng *et al.*, 1990). Herein we report the structure of the tetrahedrally coordinated $Fe^{2+}$ title complex with one dichloromethane molecule in the lattice.

In the title complex (Fig. 1), the iron(II) metal is coordinated by the N atoms of two 3,5-dimethylpyrazole ligands and the carboxylate O atoms of two tetramethyl-terphenyl carboxylate ligands in a slightly distorted tetrahedral geometry. In the carboxylate ligands, the dihedral angles formed by the central benzene ring (C12–C17 and C35–C40) with the outer benzene rings (C18–C23 and C26–C28/C30/C31/C33; C41–C46 and C40–C54) of the terphenyl groups are 47.92 (8), 59.38 (8), 48.24 (8) and 52.37 (8)°, respectively. The conformation of the complex is stabilized by intramolecular N—H···O and C—H···O hydrogen bonds (Table 1). A space filling model (Fig. 2) conveys the steric wall imposed by two terphenyl-based carboxylate and two 3,5-dimethylpyrazole ligands. The combined influence of the sterically hindered carboxylate ligands and the intramolecular hydrogen bonding interactions determines the binding mode of the carboxylate ligands as monodentate and stabilizes the coordination number of four of the metal ion. The dichloromethane molecule of crystallization interacts with the complex *via* a C—H···O hydrogen bond. In the crystal, centrosymmetrically related complex molecules are linked into dimers through pairs of C—H···O hydrogen bonds, generating a ring of $R^2_2(16)$ motif.

## S2. Experimental

Sodium 3,3″,5,5″-tetramethyl-[1,1′:3′,1″-terphenyl]-2′-carboxylate (0.206 g, 0.552 mmol) was mixed with $Fe(BF_4)_2.6H_2O$ (0.0930 g, 0.276 mmol) in tetrahydrofuran (10 mL) at room temperature. After overnight stirring, the white fine precipitate was filtered off and 3,5-dimethylpyrazole (0.0530 g, 0.276 mmol) was added. After three hours, tetrahydrofuran was removed under reduced pressure and colourless block-like crystals were obtained by crystallization from a dichloromethane/pentane (2:3 *v*/*v*) solution (yield 75%, 0.187 g). Anal. Calc. for $FeC_{56}H_{58}O_4N_4$: C, 74.16; H, 6.45; N, 6.18. Found: C, 73.80; H, 6.51; N, 6.02.

### S3. Refinement

H atoms were placed at calculated positions and refined as riding with C–H(aromatic) = 0.95 Å, C–H($CH_3$) = 0.98 Å, and with $U_{iso}$(H) = 1.2 $U_{eq}$(C) or 1.5 $U_{eq}$(C) for methyl groups. The N-bound H atoms were located in a difference Fourier map and refined isotropically.

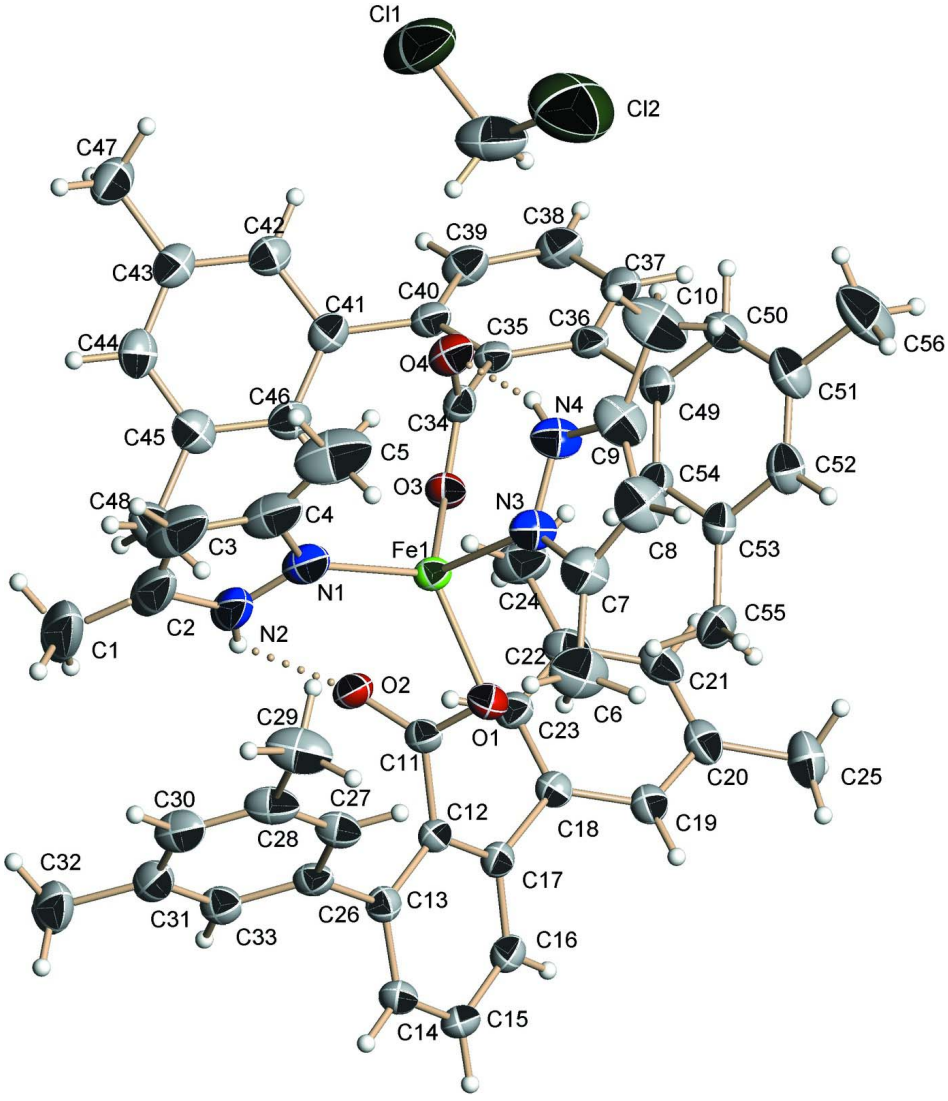


**Figure 1**

The molecular structure of the title compound, showing displacement ellipsoids drawn at the 50% probability level.

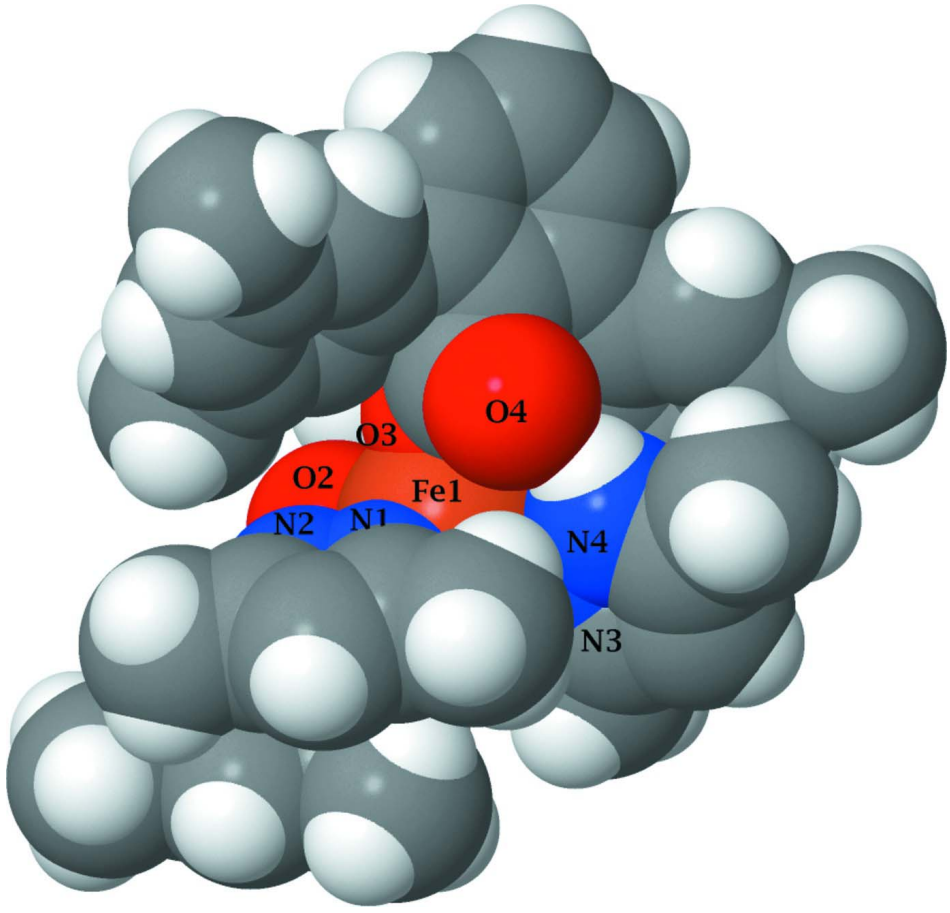


**Figure 2**

A space-filling representation of the title compound.

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*Crystal data*

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$c$ = 16.735 (3) Å
$\alpha$ = 111.11 (3)°
$\beta$ = 91.19 (3)°
$\gamma$ = 109.06 (3)°
$V$ = 2665.7 (13) Å$^3$
$Z$ = 2
$F(000)$ = 1044
$D_x$ = 1.236 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 1009 reflections
$\theta$ = 3.1–27.7°
$\mu$ = 0.43 mm$^{-1}$
$T$ = 293 K
Block, colorless
0.32 × 0.12 × 0.10 mm

*Data collection*

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min}$ = 0.839, $T_{max}$ = 1.0

20058 measured reflections
9561 independent reflections
8147 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$
$\theta_{max} = 25.3°$, $\theta_{min} = 1.9°$
$h = -14 \rightarrow 14$
$k = -17 \rightarrow 17$
$l = -20 \rightarrow 19$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.149$
$S = 1.07$
9561 reflections
633 parameters
0 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
$w = 1/[\sigma^2(F_o^2) + (0.0879P)^2 + 1.2662P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.13$ e Å$^{-3}$
$\Delta\rho_{min} = -1.06$ e Å$^{-3}$

*Special details*

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2$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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Fe1 | 0.20939 (3) | 0.51707 (2) | 0.21446 (2) | 0.02958 (12) |
| O3 | 0.30443 (15) | 0.66135 (12) | 0.23370 (11) | 0.0348 (4) |
| O1 | 0.29491 (14) | 0.45286 (13) | 0.26950 (11) | 0.0351 (4) |
| O2 | 0.36189 (15) | 0.45601 (13) | 0.14937 (11) | 0.0361 (4) |
| O4 | 0.16310 (14) | 0.72328 (13) | 0.23166 (11) | 0.0401 (4) |
| C34 | 0.2667 (2) | 0.73433 (17) | 0.24544 (14) | 0.0303 (5) |
| C11 | 0.36139 (19) | 0.43290 (16) | 0.21395 (15) | 0.0294 (5) |
| N1 | 0.1156 (2) | 0.43116 (16) | 0.09177 (15) | 0.0444 (5) |
| C17 | 0.5361 (2) | 0.42978 (17) | 0.29198 (14) | 0.0299 (5) |
| C18 | 0.57352 (19) | 0.54266 (18) | 0.34720 (15) | 0.0304 (5) |
| C12 | 0.43746 (19) | 0.37735 (17) | 0.22818 (14) | 0.0277 (5) |
| N3 | 0.08654 (18) | 0.52658 (17) | 0.30033 (15) | 0.0404 (5) |
| C27 | 0.1936 (2) | 0.21195 (18) | 0.12314 (17) | 0.0369 (5) |
| H27 | 0.1820 | 0.2437 | 0.1796 | 0.044* |
| C40 | 0.4078 (2) | 0.89183 (18) | 0.22863 (16) | 0.0343 (5) |
| C23 | 0.5838 (2) | 0.61643 (18) | 0.31164 (16) | 0.0342 (5) |
| H23 | 0.5639 | 0.5950 | 0.2521 | 0.041* |
| C49 | 0.3161 (2) | 0.84844 (18) | 0.43131 (15) | 0.0326 (5) |
| C35 | 0.35406 (19) | 0.84372 (17) | 0.28261 (15) | 0.0297 (5) |
| C54 | 0.3374 (2) | 0.76141 (18) | 0.43030 (14) | 0.0311 (5) |

| | | | | |
|---|---|---|---|---|
| H54 | 0.3837 | 0.7355 | 0.3922 | 0.037* |
| C20 | 0.6418 (2) | 0.6807 (2) | 0.49019 (16) | 0.0392 (6) |
| C36 | 0.3690 (2) | 0.90032 (18) | 0.37248 (15) | 0.0318 (5) |
| C13 | 0.4061 (2) | 0.27119 (17) | 0.17776 (14) | 0.0297 (5) |
| C16 | 0.6018 (2) | 0.37335 (19) | 0.30439 (15) | 0.0334 (5) |
| H16 | 0.6692 | 0.4076 | 0.3450 | 0.040* |
| N4 | 0.04781 (19) | 0.60714 (19) | 0.32059 (16) | 0.0433 (5) |
| C33 | 0.3208 (2) | 0.16675 (18) | 0.02217 (16) | 0.0363 (5) |
| H33 | 0.3945 | 0.1679 | 0.0110 | 0.044* |
| C19 | 0.6032 (2) | 0.57645 (19) | 0.43686 (15) | 0.0336 (5) |
| H19 | 0.5968 | 0.5278 | 0.4612 | 0.040* |
| C26 | 0.3044 (2) | 0.21535 (17) | 0.10658 (16) | 0.0320 (5) |
| C15 | 0.5684 (2) | 0.26825 (19) | 0.25767 (16) | 0.0356 (5) |
| H15 | 0.6112 | 0.2315 | 0.2687 | 0.043* |
| C41 | 0.3931 (2) | 0.83493 (18) | 0.13292 (16) | 0.0345 (5) |
| C37 | 0.4345 (2) | 1.00550 (19) | 0.40654 (16) | 0.0388 (6) |
| H37 | 0.4431 | 1.0438 | 0.4657 | 0.047* |
| C22 | 0.6234 (2) | 0.72150 (19) | 0.36390 (18) | 0.0392 (6) |
| C14 | 0.4715 (2) | 0.21733 (18) | 0.19445 (15) | 0.0338 (5) |
| H14 | 0.4497 | 0.1464 | 0.1627 | 0.041* |
| C45 | 0.3935 (2) | 0.6881 (2) | 0.00764 (18) | 0.0412 (6) |
| C53 | 0.2906 (2) | 0.71222 (19) | 0.48535 (15) | 0.0344 (5) |
| N2 | 0.1702 (3) | 0.39093 (19) | 0.02638 (15) | 0.0512 (6) |
| C21 | 0.6517 (2) | 0.7522 (2) | 0.45271 (17) | 0.0411 (6) |
| H21 | 0.6779 | 0.8225 | 0.4880 | 0.049* |
| C30 | 0.1192 (2) | 0.1149 (2) | −0.02760 (19) | 0.0471 (7) |
| H30 | 0.0570 | 0.0816 | −0.0727 | 0.057* |
| C55 | 0.3162 (2) | 0.6194 (2) | 0.48315 (17) | 0.0426 (6) |
| H55A | 0.3864 | 0.6415 | 0.5220 | 0.064* |
| H55B | 0.3250 | 0.5810 | 0.4253 | 0.064* |
| H55C | 0.2534 | 0.5760 | 0.5008 | 0.064* |
| C28 | 0.1002 (2) | 0.16175 (19) | 0.0564 (2) | 0.0438 (6) |
| C43 | 0.3497 (2) | 0.8277 (2) | −0.01151 (17) | 0.0403 (6) |
| C46 | 0.4087 (2) | 0.74075 (19) | 0.09713 (17) | 0.0375 (6) |
| H46 | 0.4297 | 0.7122 | 0.1335 | 0.045* |
| C39 | 0.4754 (2) | 0.99731 (19) | 0.26556 (18) | 0.0414 (6) |
| H39 | 0.5131 | 1.0298 | 0.2303 | 0.050* |
| C42 | 0.3634 (2) | 0.8778 (2) | 0.07789 (17) | 0.0380 (6) |
| H42 | 0.3527 | 0.9410 | 0.1015 | 0.046* |
| C31 | 0.2285 (2) | 0.11647 (19) | −0.04582 (17) | 0.0419 (6) |
| C50 | 0.2496 (2) | 0.8881 (2) | 0.49064 (17) | 0.0431 (6) |
| H50 | 0.2355 | 0.9466 | 0.4921 | 0.052* |
| C52 | 0.2241 (2) | 0.7537 (2) | 0.54303 (17) | 0.0432 (6) |
| H52 | 0.1921 | 0.7214 | 0.5799 | 0.052* |
| C7 | 0.0312 (2) | 0.4704 (2) | 0.34426 (19) | 0.0468 (6) |
| C38 | 0.4876 (2) | 1.05429 (19) | 0.35319 (18) | 0.0442 (6) |
| H38 | 0.5311 | 1.1250 | 0.3764 | 0.053* |
| C44 | 0.3637 (2) | 0.7328 (2) | −0.04499 (17) | 0.0418 (6) |

| | | | | |
|---|---|---|---|---|
| H44 | 0.3528 | 0.6978 | −0.1049 | 0.050* |
| C8 | −0.0422 (3) | 0.5161 (3) | 0.3913 (2) | 0.0557 (8) |
| H8 | −0.0904 | 0.4922 | 0.4269 | 0.067* |
| C2 | 0.1004 (4) | 0.3431 (2) | −0.0506 (2) | 0.0722 (11) |
| C47 | 0.3212 (3) | 0.8779 (3) | −0.06921 (19) | 0.0544 (8) |
| H47A | 0.3913 | 0.9153 | −0.0845 | 0.082* |
| H47B | 0.2838 | 0.9250 | −0.0389 | 0.082* |
| H47C | 0.2704 | 0.8253 | −0.1209 | 0.082* |
| C32 | 0.2471 (3) | 0.0662 (2) | −0.13746 (18) | 0.0581 (8) |
| H32A | 0.3238 | 0.1029 | −0.1444 | 0.087* |
| H32B | 0.1912 | 0.0681 | −0.1769 | 0.087* |
| H32C | 0.2381 | −0.0047 | −0.1496 | 0.087* |
| C29 | −0.0194 (2) | 0.1596 (2) | 0.0747 (3) | 0.0619 (9) |
| H29A | −0.0228 | 0.2274 | 0.0882 | 0.093* |
| H29B | −0.0359 | 0.1397 | 0.1230 | 0.093* |
| H29C | −0.0756 | 0.1105 | 0.0245 | 0.093* |
| C51 | 0.2041 (2) | 0.8426 (2) | 0.54731 (18) | 0.0483 (7) |
| C9 | −0.0298 (2) | 0.6024 (2) | 0.3749 (2) | 0.0525 (7) |
| C6 | 0.0509 (3) | 0.3745 (3) | 0.3385 (2) | 0.0633 (9) |
| H6A | −0.0016 | 0.3162 | 0.2907 | 0.095* |
| H6B | 0.0377 | 0.3639 | 0.3913 | 0.095* |
| H6C | 0.1292 | 0.3815 | 0.3298 | 0.095* |
| C4 | 0.0078 (3) | 0.4087 (2) | 0.0543 (2) | 0.0614 (9) |
| C3 | −0.0040 (4) | 0.3534 (3) | −0.0343 (3) | 0.0834 (14) |
| H3 | −0.0702 | 0.3281 | −0.0748 | 0.100* |
| C25 | 0.6735 (3) | 0.7153 (2) | 0.58695 (17) | 0.0545 (7) |
| H25A | 0.7539 | 0.7585 | 0.6046 | 0.082* |
| H25B | 0.6595 | 0.6556 | 0.6012 | 0.082* |
| H25C | 0.6270 | 0.7538 | 0.6165 | 0.082* |
| C1 | 0.1433 (5) | 0.2950 (3) | −0.1326 (2) | 0.1081 (19) |
| H1A | 0.2237 | 0.3060 | −0.1197 | 0.162* |
| H1B | 0.1342 | 0.3262 | −0.1723 | 0.162* |
| H1C | 0.0995 | 0.2218 | −0.1583 | 0.162* |
| C48 | 0.4112 (3) | 0.5862 (2) | −0.0304 (2) | 0.0589 (8) |
| H48A | 0.3459 | 0.5364 | −0.0737 | 0.088* |
| H48B | 0.4186 | 0.5618 | 0.0146 | 0.088* |
| H48C | 0.4804 | 0.5953 | −0.0564 | 0.088* |
| C24 | 0.6358 (3) | 0.8004 (2) | 0.3240 (2) | 0.0558 (8) |
| H24A | 0.6236 | 0.8600 | 0.3648 | 0.084* |
| H24B | 0.5793 | 0.7703 | 0.2726 | 0.084* |
| H24C | 0.7121 | 0.8212 | 0.3093 | 0.084* |
| C10 | −0.0849 (3) | 0.6826 (3) | 0.4048 (3) | 0.0726 (10) |
| H10A | −0.0290 | 0.7493 | 0.4132 | 0.109* |
| H10B | −0.1119 | 0.6847 | 0.4586 | 0.109* |
| H10C | −0.1491 | 0.6653 | 0.3620 | 0.109* |
| C56 | 0.1346 (4) | 0.8883 (3) | 0.6133 (3) | 0.0804 (12) |
| H56A | 0.1864 | 0.9430 | 0.6636 | 0.121* |
| H56B | 0.0845 | 0.8352 | 0.6298 | 0.121* |

| | | | | |
|---|---|---|---|---|
| H56C | 0.0888 | 0.9158 | 0.5884 | 0.121* |
| C5 | −0.0779 (3) | 0.4417 (3) | 0.1061 (3) | 0.0847 (13) |
| H5A | −0.1034 | 0.4004 | 0.1398 | 0.127* |
| H5B | −0.1431 | 0.4326 | 0.0679 | 0.127* |
| H5C | −0.0429 | 0.5135 | 0.1441 | 0.127* |
| Cl1 | 0.15706 (12) | 1.02798 (9) | 0.20616 (8) | 0.1043 (4) |
| Cl2 | 0.05816 (15) | 0.95928 (15) | 0.33896 (13) | 0.1414 (6) |
| C58 | 0.1625 (4) | 0.9602 (3) | 0.2718 (3) | 0.0840 (12) |
| H58A | 0.1522 | 0.8892 | 0.2352 | 0.101* |
| H58B | 0.2385 | 0.9915 | 0.3071 | 0.101* |
| H2 | 0.235 (3) | 0.393 (2) | 0.0360 (19) | 0.039 (8)* |
| H4 | 0.074 (3) | 0.653 (2) | 0.3006 (19) | 0.044 (8)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Fe1 | 0.02992 (19) | 0.02477 (18) | 0.0333 (2) | 0.00762 (14) | 0.00257 (14) | 0.01248 (14) |
| O3 | 0.0421 (9) | 0.0232 (8) | 0.0374 (9) | 0.0088 (7) | 0.0063 (7) | 0.0124 (7) |
| O1 | 0.0349 (9) | 0.0347 (9) | 0.0382 (9) | 0.0156 (7) | 0.0082 (7) | 0.0139 (7) |
| O2 | 0.0401 (9) | 0.0352 (9) | 0.0374 (9) | 0.0152 (8) | 0.0016 (7) | 0.0175 (7) |
| O4 | 0.0326 (9) | 0.0368 (9) | 0.0453 (10) | 0.0054 (7) | −0.0051 (8) | 0.0167 (8) |
| C34 | 0.0366 (13) | 0.0280 (12) | 0.0251 (11) | 0.0080 (10) | 0.0024 (9) | 0.0122 (9) |
| C11 | 0.0290 (11) | 0.0210 (11) | 0.0317 (12) | 0.0045 (9) | −0.0015 (9) | 0.0075 (9) |
| N1 | 0.0455 (13) | 0.0317 (11) | 0.0472 (13) | 0.0071 (10) | −0.0123 (10) | 0.0126 (10) |
| C17 | 0.0327 (12) | 0.0308 (12) | 0.0269 (11) | 0.0110 (10) | 0.0055 (9) | 0.0124 (9) |
| C18 | 0.0266 (11) | 0.0302 (12) | 0.0328 (12) | 0.0099 (9) | 0.0037 (9) | 0.0108 (10) |
| C12 | 0.0302 (11) | 0.0277 (11) | 0.0265 (11) | 0.0104 (9) | 0.0052 (9) | 0.0119 (9) |
| N3 | 0.0335 (11) | 0.0412 (12) | 0.0519 (13) | 0.0138 (9) | 0.0131 (10) | 0.0232 (10) |
| C27 | 0.0353 (13) | 0.0238 (11) | 0.0470 (14) | 0.0073 (10) | 0.0041 (11) | 0.0117 (10) |
| C40 | 0.0340 (12) | 0.0304 (12) | 0.0381 (13) | 0.0096 (10) | −0.0019 (10) | 0.0150 (10) |
| C23 | 0.0338 (12) | 0.0337 (12) | 0.0343 (12) | 0.0115 (10) | 0.0004 (10) | 0.0130 (10) |
| C49 | 0.0317 (12) | 0.0294 (12) | 0.0276 (11) | 0.0071 (10) | −0.0030 (9) | 0.0049 (9) |
| C35 | 0.0297 (11) | 0.0248 (11) | 0.0354 (12) | 0.0100 (9) | −0.0011 (9) | 0.0125 (9) |
| C54 | 0.0315 (12) | 0.0314 (12) | 0.0269 (11) | 0.0103 (10) | 0.0005 (9) | 0.0084 (9) |
| C20 | 0.0315 (12) | 0.0427 (14) | 0.0353 (13) | 0.0138 (11) | 0.0051 (10) | 0.0058 (11) |
| C36 | 0.0326 (12) | 0.0283 (12) | 0.0330 (12) | 0.0120 (10) | −0.0042 (9) | 0.0096 (10) |
| C13 | 0.0314 (12) | 0.0297 (11) | 0.0283 (11) | 0.0096 (10) | 0.0062 (9) | 0.0128 (9) |
| C16 | 0.0347 (12) | 0.0381 (13) | 0.0287 (12) | 0.0139 (11) | 0.0017 (10) | 0.0137 (10) |
| N4 | 0.0304 (11) | 0.0451 (13) | 0.0607 (15) | 0.0159 (10) | 0.0142 (10) | 0.0252 (12) |
| C33 | 0.0355 (13) | 0.0288 (12) | 0.0404 (13) | 0.0086 (10) | 0.0012 (10) | 0.0116 (10) |
| C19 | 0.0298 (12) | 0.0367 (13) | 0.0331 (12) | 0.0118 (10) | 0.0044 (10) | 0.0122 (10) |
| C26 | 0.0339 (12) | 0.0227 (11) | 0.0377 (13) | 0.0081 (9) | 0.0003 (10) | 0.0121 (10) |
| C15 | 0.0425 (14) | 0.0382 (13) | 0.0363 (13) | 0.0219 (11) | 0.0061 (11) | 0.0191 (11) |
| C41 | 0.0307 (12) | 0.0324 (12) | 0.0391 (13) | 0.0049 (10) | 0.0033 (10) | 0.0183 (10) |
| C37 | 0.0428 (14) | 0.0308 (13) | 0.0358 (13) | 0.0116 (11) | −0.0087 (11) | 0.0073 (10) |
| C22 | 0.0320 (13) | 0.0326 (13) | 0.0504 (15) | 0.0110 (10) | 0.0024 (11) | 0.0140 (11) |
| C14 | 0.0416 (13) | 0.0266 (11) | 0.0354 (13) | 0.0142 (10) | 0.0062 (10) | 0.0127 (10) |
| C45 | 0.0383 (14) | 0.0375 (14) | 0.0471 (15) | 0.0096 (11) | 0.0160 (12) | 0.0188 (12) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C53 | 0.0345 (12) | 0.0368 (13) | 0.0257 (11) | 0.0076 (10) | 0.0006 (10) | 0.0103 (10) |
| N2 | 0.0705 (19) | 0.0394 (13) | 0.0365 (13) | 0.0188 (13) | −0.0135 (12) | 0.0085 (10) |
| C21 | 0.0336 (13) | 0.0320 (13) | 0.0463 (15) | 0.0113 (11) | 0.0020 (11) | 0.0030 (11) |
| C30 | 0.0413 (15) | 0.0315 (13) | 0.0550 (17) | 0.0038 (11) | −0.0160 (12) | 0.0113 (12) |
| C55 | 0.0498 (16) | 0.0469 (15) | 0.0371 (14) | 0.0167 (13) | 0.0092 (12) | 0.0235 (12) |
| C28 | 0.0331 (13) | 0.0258 (12) | 0.0668 (18) | 0.0052 (10) | −0.0011 (12) | 0.0169 (12) |
| C43 | 0.0325 (13) | 0.0520 (16) | 0.0415 (14) | 0.0139 (12) | 0.0089 (11) | 0.0247 (12) |
| C46 | 0.0362 (13) | 0.0373 (13) | 0.0435 (14) | 0.0111 (11) | 0.0087 (11) | 0.0226 (11) |
| C39 | 0.0423 (14) | 0.0321 (13) | 0.0482 (15) | 0.0051 (11) | −0.0003 (12) | 0.0215 (12) |
| C42 | 0.0372 (13) | 0.0377 (13) | 0.0426 (14) | 0.0123 (11) | 0.0083 (11) | 0.0205 (11) |
| C31 | 0.0491 (15) | 0.0291 (12) | 0.0394 (14) | 0.0077 (11) | −0.0046 (12) | 0.0107 (11) |
| C50 | 0.0440 (15) | 0.0332 (13) | 0.0450 (15) | 0.0157 (12) | 0.0034 (12) | 0.0057 (11) |
| C52 | 0.0425 (15) | 0.0431 (15) | 0.0337 (13) | 0.0063 (12) | 0.0099 (11) | 0.0114 (11) |
| C7 | 0.0369 (14) | 0.0471 (16) | 0.0552 (17) | 0.0088 (12) | 0.0139 (12) | 0.0235 (13) |
| C38 | 0.0466 (15) | 0.0252 (12) | 0.0492 (16) | 0.0022 (11) | −0.0111 (12) | 0.0121 (11) |
| C44 | 0.0374 (14) | 0.0493 (15) | 0.0356 (13) | 0.0094 (12) | 0.0110 (11) | 0.0181 (12) |
| C8 | 0.0406 (16) | 0.0610 (19) | 0.0636 (19) | 0.0103 (14) | 0.0233 (14) | 0.0282 (16) |
| C2 | 0.118 (3) | 0.0394 (17) | 0.0440 (18) | 0.0205 (19) | −0.0268 (19) | 0.0077 (14) |
| C47 | 0.0555 (18) | 0.079 (2) | 0.0466 (16) | 0.0351 (17) | 0.0146 (14) | 0.0341 (16) |
| C32 | 0.073 (2) | 0.0507 (17) | 0.0374 (15) | 0.0153 (16) | −0.0071 (14) | 0.0089 (13) |
| C29 | 0.0342 (15) | 0.0421 (16) | 0.097 (3) | 0.0081 (13) | −0.0001 (15) | 0.0192 (17) |
| C51 | 0.0462 (16) | 0.0459 (16) | 0.0420 (15) | 0.0135 (13) | 0.0136 (12) | 0.0073 (12) |
| C9 | 0.0299 (14) | 0.0577 (18) | 0.0629 (19) | 0.0144 (13) | 0.0139 (13) | 0.0165 (15) |
| C6 | 0.064 (2) | 0.0564 (19) | 0.083 (2) | 0.0184 (16) | 0.0330 (18) | 0.0438 (18) |
| C4 | 0.0531 (18) | 0.0385 (15) | 0.076 (2) | 0.0004 (14) | −0.0293 (16) | 0.0205 (15) |
| C3 | 0.097 (3) | 0.0468 (19) | 0.077 (3) | 0.0054 (19) | −0.055 (2) | 0.0137 (18) |
| C25 | 0.0584 (18) | 0.0549 (18) | 0.0328 (14) | 0.0142 (15) | 0.0027 (13) | 0.0032 (13) |
| C1 | 0.208 (6) | 0.068 (3) | 0.0365 (19) | 0.056 (3) | −0.015 (3) | 0.0044 (17) |
| C48 | 0.075 (2) | 0.0495 (17) | 0.0571 (19) | 0.0268 (16) | 0.0313 (17) | 0.0215 (15) |
| C24 | 0.0583 (18) | 0.0365 (15) | 0.070 (2) | 0.0121 (13) | −0.0026 (15) | 0.0228 (14) |
| C10 | 0.0482 (18) | 0.074 (2) | 0.097 (3) | 0.0307 (17) | 0.0301 (18) | 0.026 (2) |
| C56 | 0.089 (3) | 0.067 (2) | 0.083 (3) | 0.035 (2) | 0.047 (2) | 0.017 (2) |
| C5 | 0.0388 (18) | 0.087 (3) | 0.126 (4) | 0.0121 (18) | −0.019 (2) | 0.049 (3) |
| Cl1 | 0.1176 (9) | 0.0743 (7) | 0.1066 (9) | 0.0258 (6) | −0.0435 (7) | 0.0302 (6) |
| Cl2 | 0.1312 (12) | 0.1640 (15) | 0.1702 (16) | 0.0944 (12) | 0.0593 (11) | 0.0716 (13) |
| C58 | 0.077 (3) | 0.067 (2) | 0.114 (3) | 0.036 (2) | −0.001 (2) | 0.034 (2) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Fe1—O3 | 1.9727 (17) | C30—H30 | 0.9300 |
| Fe1—O1 | 2.0286 (17) | C55—H55A | 0.9600 |
| Fe1—N1 | 2.058 (2) | C55—H55B | 0.9600 |
| Fe1—N3 | 2.116 (2) | C55—H55C | 0.9600 |
| O3—C34 | 1.266 (3) | C28—C29 | 1.509 (4) |
| O1—C11 | 1.273 (3) | C43—C44 | 1.383 (4) |
| O2—C11 | 1.245 (3) | C43—C42 | 1.387 (4) |
| O4—C34 | 1.242 (3) | C43—C47 | 1.511 (4) |
| C34—C35 | 1.509 (3) | C46—H46 | 0.9300 |

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|---|---|---|---|
| C11—C12 | 1.501 (3) | C39—C38 | 1.379 (4) |
| N1—C4 | 1.343 (4) | C39—H39 | 0.9300 |
| N1—N2 | 1.354 (4) | C42—H42 | 0.9300 |
| C17—C16 | 1.400 (3) | C31—C32 | 1.504 (4) |
| C17—C12 | 1.400 (3) | C50—C51 | 1.379 (4) |
| C17—C18 | 1.491 (3) | C50—H50 | 0.9300 |
| C18—C23 | 1.394 (3) | C52—C51 | 1.394 (4) |
| C18—C19 | 1.397 (3) | C52—H52 | 0.9300 |
| C12—C13 | 1.401 (3) | C7—C8 | 1.396 (4) |
| N3—C7 | 1.337 (3) | C7—C6 | 1.486 (4) |
| N3—N4 | 1.361 (3) | C38—H38 | 0.9300 |
| C27—C28 | 1.386 (4) | C44—H44 | 0.9300 |
| C27—C26 | 1.392 (3) | C8—C9 | 1.362 (5) |
| C27—H27 | 0.9300 | C8—H8 | 0.9300 |
| C40—C35 | 1.394 (3) | C2—C3 | 1.370 (6) |
| C40—C39 | 1.396 (4) | C2—C1 | 1.499 (6) |
| C40—C41 | 1.491 (3) | C47—H47A | 0.9600 |
| C23—C22 | 1.388 (3) | C47—H47B | 0.9600 |
| C23—H23 | 0.9300 | C47—H47C | 0.9600 |
| C49—C54 | 1.390 (3) | C32—H32A | 0.9600 |
| C49—C50 | 1.391 (4) | C32—H32B | 0.9600 |
| C49—C36 | 1.493 (3) | C32—H32C | 0.9600 |
| C35—C36 | 1.406 (3) | C29—H29A | 0.9600 |
| C54—C53 | 1.394 (3) | C29—H29B | 0.9600 |
| C54—H54 | 0.9300 | C29—H29C | 0.9600 |
| C20—C19 | 1.383 (4) | C51—C56 | 1.515 (4) |
| C20—C21 | 1.388 (4) | C9—C10 | 1.495 (4) |
| C20—C25 | 1.511 (4) | C6—H6A | 0.9600 |
| C36—C37 | 1.386 (3) | C6—H6B | 0.9600 |
| C13—C14 | 1.393 (3) | C6—H6C | 0.9600 |
| C13—C26 | 1.494 (3) | C4—C3 | 1.389 (5) |
| C16—C15 | 1.375 (3) | C4—C5 | 1.478 (6) |
| C16—H16 | 0.9300 | C3—H3 | 0.9300 |
| N4—C9 | 1.337 (4) | C25—H25A | 0.9600 |
| N4—H4 | 0.84 (3) | C25—H25B | 0.9600 |
| C33—C26 | 1.389 (3) | C25—H25C | 0.9600 |
| C33—C31 | 1.390 (4) | C1—H1A | 0.9600 |
| C33—H33 | 0.9300 | C1—H1B | 0.9600 |
| C19—H19 | 0.9300 | C1—H1C | 0.9600 |
| C15—C14 | 1.381 (4) | C48—H48A | 0.9600 |
| C15—H15 | 0.9300 | C48—H48B | 0.9600 |
| C41—C46 | 1.385 (4) | C48—H48C | 0.9600 |
| C41—C42 | 1.394 (3) | C24—H24A | 0.9600 |
| C37—C38 | 1.389 (4) | C24—H24B | 0.9600 |
| C37—H37 | 0.9300 | C24—H24C | 0.9600 |
| C22—C21 | 1.388 (4) | C10—H10A | 0.9600 |
| C22—C24 | 1.511 (4) | C10—H10B | 0.9600 |
| C14—H14 | 0.9300 | C10—H10C | 0.9600 |

| | | | |
|---|---|---|---|
| C45—C44 | 1.383 (4) | C56—H56A | 0.9600 |
| C45—C46 | 1.392 (4) | C56—H56B | 0.9600 |
| C45—C48 | 1.502 (4) | C56—H56C | 0.9600 |
| C53—C52 | 1.385 (4) | C5—H5A | 0.9600 |
| C53—C55 | 1.496 (4) | C5—H5B | 0.9600 |
| N2—C2 | 1.343 (4) | C5—H5C | 0.9600 |
| N2—H2 | 0.80 (3) | Cl1—C58 | 1.747 (4) |
| C21—H21 | 0.9300 | Cl2—C58 | 1.728 (5) |
| C30—C31 | 1.386 (4) | C58—H58A | 0.9700 |
| C30—C28 | 1.388 (4) | C58—H58B | 0.9700 |

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|---|---|---|---|
| O3—Fe1—O1 | 111.85 (7) | C45—C46—H46 | 119.5 |
| O3—Fe1—N1 | 114.09 (9) | C38—C39—C40 | 121.4 (2) |
| O1—Fe1—N1 | 119.33 (8) | C38—C39—H39 | 119.3 |
| O3—Fe1—N3 | 106.17 (8) | C40—C39—H39 | 119.3 |
| O1—Fe1—N3 | 96.59 (8) | C43—C42—C41 | 121.0 (2) |
| N1—Fe1—N3 | 106.18 (10) | C43—C42—H42 | 119.5 |
| C34—O3—Fe1 | 125.58 (15) | C41—C42—H42 | 119.5 |
| C11—O1—Fe1 | 99.69 (14) | C30—C31—C33 | 118.6 (3) |
| O4—C34—O3 | 125.2 (2) | C30—C31—C32 | 120.7 (3) |
| O4—C34—C35 | 117.4 (2) | C33—C31—C32 | 120.7 (3) |
| O3—C34—C35 | 117.3 (2) | C51—C50—C49 | 121.6 (3) |
| O2—C11—O1 | 121.8 (2) | C51—C50—H50 | 119.2 |
| O2—C11—C12 | 121.1 (2) | C49—C50—H50 | 119.2 |
| O1—C11—C12 | 117.09 (19) | C53—C52—C51 | 121.7 (2) |
| C4—N1—N2 | 105.6 (3) | C53—C52—H52 | 119.1 |
| C4—N1—Fe1 | 135.5 (2) | C51—C52—H52 | 119.1 |
| N2—N1—Fe1 | 118.65 (18) | N3—C7—C8 | 109.5 (3) |
| C16—C17—C12 | 118.3 (2) | N3—C7—C6 | 121.3 (3) |
| C16—C17—C18 | 118.9 (2) | C8—C7—C6 | 129.2 (3) |
| C12—C17—C18 | 122.8 (2) | C39—C38—C37 | 119.6 (2) |
| C23—C18—C19 | 118.7 (2) | C39—C38—H38 | 120.2 |
| C23—C18—C17 | 121.8 (2) | C37—C38—H38 | 120.2 |
| C19—C18—C17 | 119.5 (2) | C43—C44—C45 | 122.2 (2) |
| C17—C12—C13 | 120.6 (2) | C43—C44—H44 | 118.9 |
| C17—C12—C11 | 120.8 (2) | C45—C44—H44 | 118.9 |
| C13—C12—C11 | 118.6 (2) | C9—C8—C7 | 106.8 (3) |
| C7—N3—N4 | 105.3 (2) | C9—C8—H8 | 126.6 |
| C7—N3—Fe1 | 135.96 (19) | C7—C8—H8 | 126.6 |
| N4—N3—Fe1 | 118.77 (16) | N2—C2—C3 | 106.1 (3) |
| C28—C27—C26 | 120.7 (3) | N2—C2—C1 | 121.3 (4) |
| C28—C27—H27 | 119.6 | C3—C2—C1 | 132.6 (4) |
| C26—C27—H27 | 119.6 | C43—C47—H47A | 109.5 |
| C35—C40—C39 | 118.6 (2) | C43—C47—H47B | 109.5 |
| C35—C40—C41 | 122.2 (2) | H47A—C47—H47B | 109.5 |
| C39—C40—C41 | 119.2 (2) | C43—C47—H47C | 109.5 |
| C22—C23—C18 | 120.9 (2) | H47A—C47—H47C | 109.5 |
| C22—C23—H23 | 119.6 | H47B—C47—H47C | 109.5 |

| | | | |
|---|---|---|---|
| C18—C23—H23 | 119.6 | C31—C32—H32A | 109.5 |
| C54—C49—C50 | 118.6 (2) | C31—C32—H32B | 109.5 |
| C54—C49—C36 | 120.0 (2) | H32A—C32—H32B | 109.5 |
| C50—C49—C36 | 121.4 (2) | C31—C32—H32C | 109.5 |
| C40—C35—C36 | 120.5 (2) | H32A—C32—H32C | 109.5 |
| C40—C35—C34 | 121.0 (2) | H32B—C32—H32C | 109.5 |
| C36—C35—C34 | 118.0 (2) | C28—C29—H29A | 109.5 |
| C49—C54—C53 | 121.3 (2) | C28—C29—H29B | 109.5 |
| C49—C54—H54 | 119.4 | H29A—C29—H29B | 109.5 |
| C53—C54—H54 | 119.4 | C28—C29—H29C | 109.5 |
| C19—C20—C21 | 118.5 (2) | H29A—C29—H29C | 109.5 |
| C19—C20—C25 | 120.4 (3) | H29B—C29—H29C | 109.5 |
| C21—C20—C25 | 121.1 (2) | C50—C51—C52 | 118.4 (2) |
| C37—C36—C35 | 119.2 (2) | C50—C51—C56 | 121.0 (3) |
| C37—C36—C49 | 119.9 (2) | C52—C51—C56 | 120.5 (3) |
| C35—C36—C49 | 120.9 (2) | N4—C9—C8 | 106.5 (3) |
| C14—C13—C12 | 119.1 (2) | N4—C9—C10 | 121.0 (3) |
| C14—C13—C26 | 119.7 (2) | C8—C9—C10 | 132.6 (3) |
| C12—C13—C26 | 121.2 (2) | C7—C6—H6A | 109.5 |
| C15—C16—C17 | 121.2 (2) | C7—C6—H6B | 109.5 |
| C15—C16—H16 | 119.4 | H6A—C6—H6B | 109.5 |
| C17—C16—H16 | 119.4 | C7—C6—H6C | 109.5 |
| C9—N4—N3 | 112.0 (2) | H6A—C6—H6C | 109.5 |
| C9—N4—H4 | 128 (2) | H6B—C6—H6C | 109.5 |
| N3—N4—H4 | 120 (2) | N1—C4—C3 | 109.4 (4) |
| C26—C33—C31 | 120.8 (2) | N1—C4—C5 | 121.2 (3) |
| C26—C33—H33 | 119.6 | C3—C4—C5 | 129.4 (3) |
| C31—C33—H33 | 119.6 | C2—C3—C4 | 107.0 (3) |
| C20—C19—C18 | 121.4 (2) | C2—C3—H3 | 126.5 |
| C20—C19—H19 | 119.3 | C4—C3—H3 | 126.5 |
| C18—C19—H19 | 119.3 | C20—C25—H25A | 109.5 |
| C33—C26—C27 | 119.3 (2) | C20—C25—H25B | 109.5 |
| C33—C26—C13 | 119.4 (2) | H25A—C25—H25B | 109.5 |
| C27—C26—C13 | 121.2 (2) | C20—C25—H25C | 109.5 |
| C16—C15—C14 | 120.0 (2) | H25A—C25—H25C | 109.5 |
| C16—C15—H15 | 120.0 | H25B—C25—H25C | 109.5 |
| C14—C15—H15 | 120.0 | C2—C1—H1A | 109.5 |
| C46—C41—C42 | 119.0 (2) | C2—C1—H1B | 109.5 |
| C46—C41—C40 | 121.7 (2) | H1A—C1—H1B | 109.5 |
| C42—C41—C40 | 119.2 (2) | C2—C1—H1C | 109.5 |
| C36—C37—C38 | 120.6 (2) | H1A—C1—H1C | 109.5 |
| C36—C37—H37 | 119.7 | H1B—C1—H1C | 109.5 |
| C38—C37—H37 | 119.7 | C45—C48—H48A | 109.5 |
| C21—C22—C23 | 118.9 (2) | C45—C48—H48B | 109.5 |
| C21—C22—C24 | 121.0 (2) | H48A—C48—H48B | 109.5 |
| C23—C22—C24 | 120.1 (2) | C45—C48—H48C | 109.5 |
| C15—C14—C13 | 120.7 (2) | H48A—C48—H48C | 109.5 |
| C15—C14—H14 | 119.7 | H48B—C48—H48C | 109.5 |

| | | | |
|---|---|---|---|
| C13—C14—H14 | 119.7 | C22—C24—H24A | 109.5 |
| C44—C45—C46 | 118.4 (2) | C22—C24—H24B | 109.5 |
| C44—C45—C48 | 121.1 (3) | H24A—C24—H24B | 109.5 |
| C46—C45—C48 | 120.5 (3) | C22—C24—H24C | 109.5 |
| C52—C53—C54 | 118.3 (2) | H24A—C24—H24C | 109.5 |
| C52—C53—C55 | 121.8 (2) | H24B—C24—H24C | 109.5 |
| C54—C53—C55 | 119.9 (2) | C9—C10—H10A | 109.5 |
| C2—N2—N1 | 111.9 (3) | C9—C10—H10B | 109.5 |
| C2—N2—H2 | 127 (2) | H10A—C10—H10B | 109.5 |
| N1—N2—H2 | 121 (2) | C9—C10—H10C | 109.5 |
| C22—C21—C20 | 121.7 (2) | H10A—C10—H10C | 109.5 |
| C22—C21—H21 | 119.2 | H10B—C10—H10C | 109.5 |
| C20—C21—H21 | 119.2 | C51—C56—H56A | 109.5 |
| C31—C30—C28 | 121.6 (2) | C51—C56—H56B | 109.5 |
| C31—C30—H30 | 119.2 | H56A—C56—H56B | 109.5 |
| C28—C30—H30 | 119.2 | C51—C56—H56C | 109.5 |
| C53—C55—H55A | 109.5 | H56A—C56—H56C | 109.5 |
| C53—C55—H55B | 109.5 | H56B—C56—H56C | 109.5 |
| H55A—C55—H55B | 109.5 | C4—C5—H5A | 109.5 |
| C53—C55—H55C | 109.5 | C4—C5—H5B | 109.5 |
| H55A—C55—H55C | 109.5 | H5A—C5—H5B | 109.5 |
| H55B—C55—H55C | 109.5 | C4—C5—H5C | 109.5 |
| C27—C28—C30 | 118.8 (3) | H5A—C5—H5C | 109.5 |
| C27—C28—C29 | 120.4 (3) | H5B—C5—H5C | 109.5 |
| C30—C28—C29 | 120.8 (3) | Cl2—C58—Cl1 | 113.0 (2) |
| C44—C43—C42 | 118.4 (2) | Cl2—C58—H58A | 109.0 |
| C44—C43—C47 | 122.0 (2) | Cl1—C58—H58A | 109.0 |
| C42—C43—C47 | 119.6 (3) | Cl2—C58—H58B | 109.0 |
| C41—C46—C45 | 121.0 (2) | Cl1—C58—H58B | 109.0 |
| C41—C46—H46 | 119.5 | H58A—C58—H58B | 107.8 |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N4—H4···O4 | 0.84 (3) | 1.95 (3) | 2.744 (3) | 158 (3) |
| N2—H2···O2 | 0.80 (3) | 2.14 (3) | 2.767 (3) | 135 (3) |
| C6—H6*C*···O1 | 0.96 | 2.40 | 3.256 (4) | 148 |
| C46—H46···O3 | 0.93 | 2.46 | 3.055 (4) | 122 |
| C54—H54···O3 | 0.93 | 2.51 | 3.037 (3) | 116 |
| C55—H55*B*···O1 | 0.96 | 2.54 | 3.481 (3) | 166 |
| C58—H58*A*···O4 | 0.97 | 2.48 | 3.326 (5) | 145 |
| C3—H3···O4$^{i}$ | 0.93 | 2.58 | 3.411 (5) | 149 |

Symmetry code: (i) −*x*, −*y*+1, −*z*.