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# Tris(1,10-phenanthroline-$\kappa^2N,N'$)iron(II) $\mu$-oxido-bis[trichloridoferrate(III)] ethanol hemisolvate

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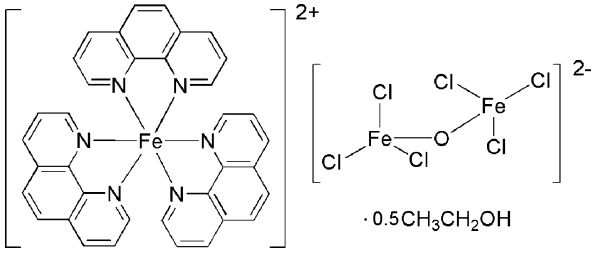
Key indicators: single-crystal X-ray study; $T$ = 294 K; mean $\sigma$(C–C) = 0.011 Å; disorder in solvent or counterion; $R$ factor = 0.064; $wR$ factor = 0.212; data-to-parameter ratio = 14.9.

The title compound, $[Fe(C_{12}H_8N_2)_3][Fe_2Cl_6O]\cdot0.5CH_3CH_2OH$, consists of one $[Fe(phen)_3]^{2+}$ cation (phen = 1,10-phenanthroline), one $[Fe_2Cl_6O]^{2-}$ anion and one half-molecule of ethanol. In the cation, the $Fe^{II}$ atom is coordinated by six N atoms from three phen ligands in a distorted octahedral geometry. In the bent anion, two $Fe^{III}$ atoms are connected by a bridging oxide O atom [bridging angle = 160.6 (4)°], and each $Fe^{III}$ atom is also coordinated by three Cl atoms, completing a distorted tetrahedral geometry.

## Related literature

For general background, see: Hwang & Ha (2006); Potočňák *et al.* (2002); Zhou & Guo (2007). For a related structure, see: Aparici Plaza *et al.* (2007).




## Experimental

### Crystal data

$[Fe(C_{12}H_8N_2)_3][Fe_2Cl_6O]\cdot0.5C_2H_6O$
$M_r$ = 959.89
Monoclinic, $P2_1/c$
$a$ = 15.3536 (18) Å
$b$ = 13.1857 (16) Å
$c$ = 20.897 (2) Å
$\beta$ = 94.701 (2)°
$V$ = 4216.4 (8) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 1.44 mm$^{-1}$
$T$ = 294 (2) K
0.55 × 0.47 × 0.42 mm

### Data collection

Bruker SMART 1K CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.452, $T_{max}$ = 0.544
20409 measured reflections
7434 independent reflections
4248 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.058

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.212
$S$ = 1.02
7434 reflections
498 parameters
3 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.84 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.39 e Å$^{-3}$

**Table 1**
Selected bond lengths (Å).

| | | | |
|---|---|---|---|
| Fe1—N4 | 1.958 (6) | Fe2—Cl4 | 2.204 (3) |
| Fe1—N2 | 1.970 (5) | Fe2—Cl6 | 2.223 (2) |
| Fe1—N6 | 1.979 (5) | Fe2—Cl5 | 2.231 (2) |
| Fe1—N1 | 1.980 (5) | Fe3—O1 | 1.756 (5) |
| Fe1—N3 | 1.982 (5) | Fe3—Cl2 | 2.213 (2) |
| Fe1—N5 | 1.985 (5) | Fe3—Cl3 | 2.227 (2) |
| Fe2—O1 | 1.735 (5) | Fe3—Cl1 | 2.227 (2) |

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; 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: HY2160).

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# supporting information

*Acta Cryst.* (2008). E**64**, m1514 [doi:10.1107/S1600536808035897]

# Tris(1,10-phenanthroline-$\kappa^2N,N'$)iron(II) $\mu$-oxido-bis[trichloridoferrate(III)] ethanol hemisolvate

**Zhan-Xian Li, Ming-Ming Yu, Yu-Na Zhang and Liu-He Wei**

## S1. Comment

1,10-Phenanthroline (phen) is a widely utilized chelating ligand in coordination chemistry and a lot of complexes with phen as a ligand have been reported (Hwang & Ha, 2006; Potocnak *et al.*, 2002; Zhou & Guo, 2007). Plaza *et al.* (2007) has reported the structure of a complex with $[Fe(phen)_3]^{2+}$ as cation and simple halide as counter-ion. We report here the synthesis and crystal structure of the title compound, in which an oxide-bridged iron(III) complex acts as counter-ion.

The title compound comprises one $[Fe(phen)_3]^{2+}$ cation, one $[Fe_2Cl_6O]^{2-}$ anion and a half of solvent ethanol molecule (Fig. 1). The $Fe^{II}$ atom in the cation is coordinated by six N atoms from three phen ligands, forming a distorted $FeN_6$ octahedral geometry. The Fe—N bond lengths are in the normal range (Table 1). In the anion, two $Fe^{III}$ atoms are bridged by an oxide O atom and is also coordinated by three Cl atoms in a distorted tetrahedral geometry. The Fe—Cl bond distances are in the range of 2.204 (3) to 2.231 (2) Å. The distance betweeen the $Fe^{III}$ atoms (Fe2 and Fe3) bridged by the O1 atom is 3.441 (5) Å [Fe2—O1 = 1.735 (5) and Fe3—O1 = 1.756 (5) Å].

## S2. Experimental

$Fe(ClO_4)_2.6H_2O$ (0.036 g, 0.1 mmol) and 1,10-phenanthroline (0.054 g, 0.3 mmol) are dissolved in 15 ml of water and ethanol (1:2 in volume), resulting in a pale green solution, which was transfered to the left hand of an H-shaped tube. An aqueous solution (15 ml) of $FeCl_3$ (0.032 g, 0.2 mmol) was placed in the right hand of the tube. An ethanol solution serving as the diffusion solvent was put in the connecting part between the left and right hands of the tube. The reaction container was kept at room temperature. Dark brown crystals suitable for X-ray diffraction analysis were obtained in the connecting part of the H-shaped tube after two months (yield 60% based on Fe).

## S3. Refinement

H atoms were positioned geometrically and refined as riding atoms, with C—H = 0.93 (aromatic), 0.97 ($CH_2$) and 0.96 ($CH_3$) Å and $U_{iso}$(H) = 1.2(1.5 for methyl)$U_{eq}$(C), and with O—H = 0.82 Å and $U_{iso}$(H) = 1.5$U_{eq}$(O).

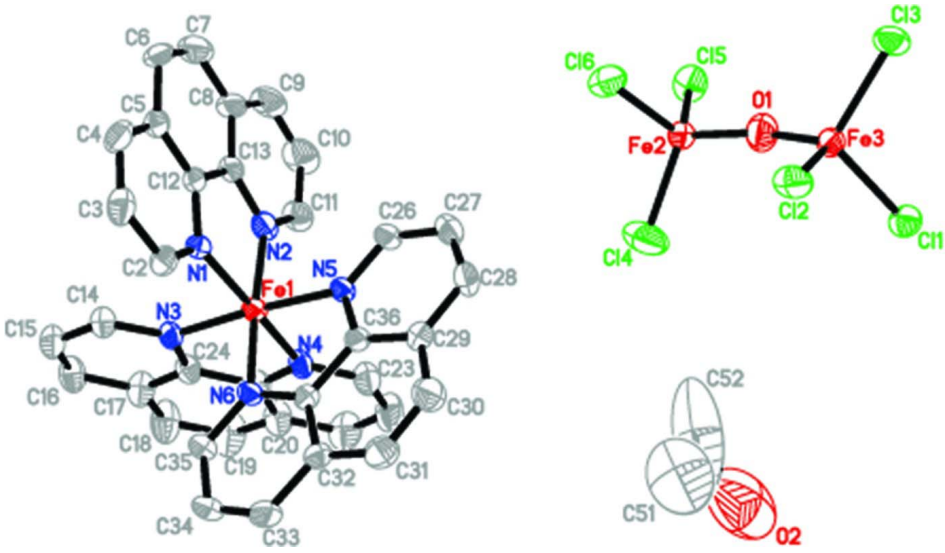


**Figure 1**

The structures of the cation, anion and solvate molecule in the title compoud. Displacement ellipsoids are drawn at the 30% probability level.

**Tris(1,10-phenanthroline-$\kappa^2N,N'$)iron(II) $\mu$-oxido-bis[trichloridoferrate(III)] ethanol semisolvate**

*Crystal data*

$[Fe(C_{12}H_8N_2)_3][Fe_2Cl_6O]\cdot 0.5C_2H_6O$
$M_r = 959.89$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 15.3536\ (18)$ Å
$b = 13.1857\ (16)$ Å
$c = 20.897\ (2)$ Å
$\beta = 94.701\ (2)°$
$V = 4216.4\ (8)$ Å$^3$
$Z = 4$
$F(000) = 1932$
$D_x = 1.512$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 20409 reflections
$\theta = 1.8–25.0°$
$\mu = 1.44$ mm$^{-1}$
$T = 294$ K
Block, brown
0.55 × 0.47 × 0.42 mm

*Data collection*

Bruker SMART 1K CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.452$, $T_{max} = 0.544$
20409 measured reflections
7434 independent reflections
4248 reflections with $I > 2\sigma(I)$
$R_{int} = 0.058$
$\theta_{max} = 25.0°$, $\theta_{min} = 1.8°$
$h = -18\rightarrow14$
$k = -15\rightarrow15$
$l = -17\rightarrow24$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.064$
$wR(F^2) = 0.212$
$S = 1.03$
7434 reflections
498 parameters
3 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1185P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.018$
$\Delta\rho_{max} = 0.84$ e Å$^{-3}$
$\Delta\rho_{min} = -0.39$ e Å$^{-3}$

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| Fe1 | 0.25729 (6) | 0.78424 (6) | 0.06260 (4) | 0.0388 (3) | |
| Fe2 | 0.21293 (7) | 0.12200 (8) | 0.71711 (5) | 0.0533 (3) | |
| Fe3 | 0.34590 (7) | 0.14054 (7) | 0.85736 (5) | 0.0505 (3) | |
| Cl1 | 0.27250 (13) | 0.18682 (15) | 0.94022 (9) | 0.0645 (5) | |
| Cl2 | 0.43389 (15) | 0.26757 (15) | 0.83747 (11) | 0.0765 (6) | |
| Cl3 | 0.42517 (14) | 0.00431 (14) | 0.88656 (10) | 0.0696 (6) | |
| Cl4 | 0.1307 (2) | 0.2590 (2) | 0.70798 (16) | 0.1203 (11) | |
| Cl5 | 0.12833 (15) | −0.01536 (17) | 0.70469 (11) | 0.0815 (7) | |
| Cl6 | 0.30925 (16) | 0.12534 (15) | 0.64332 (11) | 0.0811 (7) | |
| O1 | 0.2707 (4) | 0.1130 (5) | 0.7918 (3) | 0.0874 (18) | |
| N1 | 0.3671 (4) | 0.7608 (4) | 0.0213 (2) | 0.0440 (13) | |
| N2 | 0.2292 (4) | 0.6451 (4) | 0.0334 (3) | 0.0500 (15) | |
| N3 | 0.1978 (3) | 0.8384 (4) | −0.0180 (2) | 0.0431 (13) | |
| N4 | 0.1411 (4) | 0.7998 (4) | 0.0936 (3) | 0.0467 (14) | |
| N5 | 0.3138 (3) | 0.7370 (4) | 0.1461 (2) | 0.0393 (12) | |
| N6 | 0.2932 (3) | 0.9184 (4) | 0.0981 (2) | 0.0400 (12) | |
| C2 | 0.4338 (4) | 0.8226 (5) | 0.0168 (3) | 0.0475 (17) | |
| H27 | 0.4333 | 0.8864 | 0.0357 | 0.057* | |
| C3 | 0.5060 (5) | 0.7928 (7) | −0.0164 (3) | 0.063 (2) | |
| H36 | 0.5519 | 0.8378 | −0.0205 | 0.076* | |
| C4 | 0.5088 (6) | 0.6978 (7) | −0.0427 (3) | 0.068 (2) | |
| H33 | 0.5571 | 0.6775 | −0.0636 | 0.082* | |
| C5 | 0.4394 (6) | 0.6325 (6) | −0.0380 (3) | 0.057 (2) | |
| C6 | 0.4341 (7) | 0.5289 (7) | −0.0641 (3) | 0.076 (3) | |
| H38 | 0.4798 | 0.5029 | −0.0856 | 0.091* | |
| C7 | 0.3610 (8) | 0.4702 (7) | −0.0565 (4) | 0.085 (3) | |
| H47 | 0.3588 | 0.4049 | −0.0734 | 0.102* | |
| C8 | 0.2901 (6) | 0.5047 (6) | −0.0244 (4) | 0.064 (2) | |
| C9 | 0.2164 (8) | 0.4478 (6) | −0.0139 (4) | 0.085 (3) | |
| H32 | 0.2108 | 0.3821 | −0.0300 | 0.102* | |
| C10 | 0.1525 (7) | 0.4877 (7) | 0.0196 (5) | 0.088 (3) | |
| H49 | 0.1036 | 0.4496 | 0.0277 | 0.105* | |
| C11 | 0.1617 (5) | 0.5860 (6) | 0.0415 (3) | 0.062 (2) | |
| H37 | 0.1169 | 0.6127 | 0.0637 | 0.075* | |
| C12 | 0.3679 (5) | 0.6666 (5) | −0.0059 (3) | 0.0440 (16) | |
| C13 | 0.2948 (5) | 0.6026 (5) | 0.0009 (3) | 0.0490 (17) | |
| C14 | 0.2293 (5) | 0.8619 (5) | −0.0731 (3) | 0.0508 (18) | |
| H26 | 0.2890 | 0.8547 | −0.0765 | 0.061* | |
| C15 | 0.1775 (5) | 0.8967 (6) | −0.1261 (4) | 0.063 (2) | |
| H45 | 0.2029 | 0.9139 | −0.1635 | 0.076* | |
| C16 | 0.0899 (6) | 0.9056 (6) | −0.1234 (4) | 0.069 (2) | |

| | | | | | |
|---|---|---|---|---|---|
| H20 | 0.0548 | 0.9260 | −0.1594 | 0.083* | |
| C17 | 0.0521 (5) | 0.8837 (6) | −0.0653 (4) | 0.066 (2) | |
| C18 | −0.0379 (6) | 0.8918 (8) | −0.0552 (4) | 0.087 (3) | |
| H44 | −0.0775 | 0.9122 | −0.0889 | 0.105* | |
| C19 | −0.0667 (5) | 0.8707 (8) | 0.0018 (4) | 0.092 (3) | |
| H50 | −0.1263 | 0.8757 | 0.0067 | 0.111* | |
| C20 | −0.0097 (5) | 0.8408 (7) | 0.0554 (4) | 0.068 (2) | |
| C21 | −0.0344 (5) | 0.8189 (7) | 0.1172 (5) | 0.082 (3) | |
| H46 | −0.0928 | 0.8239 | 0.1257 | 0.099* | |
| C22 | 0.0263 (6) | 0.7905 (7) | 0.1645 (4) | 0.076 (2) | |
| H35 | 0.0095 | 0.7769 | 0.2054 | 0.092* | |
| C23 | 0.1141 (5) | 0.7815 (6) | 0.1520 (4) | 0.062 (2) | |
| H43 | 0.1549 | 0.7622 | 0.1851 | 0.075* | |
| C24 | 0.1109 (4) | 0.8498 (5) | −0.0134 (3) | 0.0482 (17) | |
| C25 | 0.0799 (4) | 0.8296 (5) | 0.0472 (3) | 0.0485 (17) | |
| C26 | 0.3254 (4) | 0.6437 (5) | 0.1694 (3) | 0.0484 (17) | |
| H28 | 0.3050 | 0.5893 | 0.1442 | 0.058* | |
| C27 | 0.3662 (5) | 0.6239 (5) | 0.2291 (4) | 0.0580 (19) | |
| H18 | 0.3717 | 0.5573 | 0.2434 | 0.070* | |
| C28 | 0.3980 (5) | 0.6995 (6) | 0.2667 (3) | 0.060 (2) | |
| H2 | 0.4250 | 0.6858 | 0.3072 | 0.072* | |
| C29 | 0.3903 (5) | 0.7982 (5) | 0.2448 (3) | 0.0528 (18) | |
| C30 | 0.4249 (6) | 0.8884 (6) | 0.2784 (4) | 0.076 (3) | |
| H42 | 0.4580 | 0.8807 | 0.3174 | 0.091* | |
| C31 | 0.4105 (6) | 0.9821 (6) | 0.2548 (4) | 0.076 (3) | |
| H48 | 0.4306 | 1.0379 | 0.2789 | 0.092* | |
| C32 | 0.3649 (5) | 0.9978 (5) | 0.1934 (3) | 0.0542 (19) | |
| C33 | 0.3478 (5) | 1.0945 (5) | 0.1650 (4) | 0.062 (2) | |
| H34 | 0.3652 | 1.1535 | 0.1868 | 0.075* | |
| C34 | 0.3054 (5) | 1.0992 (5) | 0.1050 (4) | 0.058 (2) | |
| H25 | 0.2939 | 1.1618 | 0.0857 | 0.070* | |
| C35 | 0.2796 (4) | 1.0107 (5) | 0.0728 (3) | 0.0489 (17) | |
| H19 | 0.2515 | 1.0159 | 0.0318 | 0.059* | |
| C36 | 0.3465 (4) | 0.8139 (4) | 0.1835 (3) | 0.0396 (15) | |
| C37 | 0.3342 (4) | 0.9128 (5) | 0.1575 (3) | 0.0405 (15) | |
| C51 | 0.2017 (13) | 0.6513 (18) | 0.8088 (10) | 0.128 (9) | 0.50 |
| H51A | 0.1715 | 0.7143 | 0.8008 | 0.191* | 0.50 |
| H51B | 0.2320 | 0.6333 | 0.7721 | 0.191* | 0.50 |
| H51C | 0.2429 | 0.6583 | 0.8456 | 0.191* | 0.50 |
| C52 | 0.1376 (18) | 0.570 (2) | 0.8211 (13) | 0.29 (4) | 0.50 |
| H52A | 0.1042 | 0.5525 | 0.7813 | 0.347* | 0.50 |
| H52B | 0.1682 | 0.5104 | 0.8376 | 0.347* | 0.50 |
| O2 | 0.0796 (14) | 0.6063 (17) | 0.8669 (11) | 0.183 (10) | 0.50 |
| H2A | 0.0895 | 0.6664 | 0.8746 | 0.275* | 0.50 |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Fe1 | 0.0416 (5) | 0.0331 (5) | 0.0421 (5) | 0.0006 (4) | 0.0060 (4) | 0.0023 (4) |
| Fe2 | 0.0609 (7) | 0.0507 (6) | 0.0489 (6) | 0.0022 (5) | 0.0082 (5) | 0.0018 (5) |
| Fe3 | 0.0615 (7) | 0.0439 (6) | 0.0467 (6) | −0.0040 (5) | 0.0082 (5) | 0.0014 (4) |
| Cl1 | 0.0630 (13) | 0.0672 (12) | 0.0658 (12) | −0.0007 (10) | 0.0218 (10) | 0.0013 (9) |
| Cl2 | 0.0889 (16) | 0.0510 (11) | 0.0956 (16) | −0.0046 (10) | 0.0435 (13) | 0.0125 (10) |
| Cl3 | 0.0819 (14) | 0.0420 (10) | 0.0844 (14) | 0.0027 (9) | 0.0029 (11) | −0.0028 (9) |
| Cl4 | 0.116 (2) | 0.0853 (18) | 0.166 (3) | 0.0400 (16) | 0.050 (2) | 0.0337 (17) |
| Cl5 | 0.0851 (16) | 0.0794 (15) | 0.0809 (15) | −0.0258 (12) | 0.0116 (12) | −0.0001 (11) |
| Cl6 | 0.1018 (17) | 0.0544 (12) | 0.0941 (16) | −0.0114 (11) | 0.0504 (13) | −0.0036 (11) |
| O1 | 0.106 (5) | 0.089 (4) | 0.064 (4) | −0.003 (3) | −0.006 (3) | −0.002 (3) |
| N1 | 0.051 (4) | 0.039 (3) | 0.043 (3) | 0.012 (3) | 0.010 (3) | 0.006 (2) |
| N2 | 0.062 (4) | 0.042 (3) | 0.045 (3) | −0.007 (3) | −0.003 (3) | 0.004 (3) |
| N3 | 0.045 (3) | 0.042 (3) | 0.044 (3) | −0.006 (2) | 0.009 (3) | 0.007 (2) |
| N4 | 0.048 (3) | 0.044 (3) | 0.049 (3) | −0.004 (3) | 0.008 (3) | 0.012 (3) |
| N5 | 0.045 (3) | 0.031 (3) | 0.043 (3) | −0.002 (2) | 0.007 (2) | 0.004 (2) |
| N6 | 0.043 (3) | 0.030 (3) | 0.049 (3) | 0.001 (2) | 0.012 (3) | 0.003 (2) |
| C2 | 0.040 (4) | 0.054 (4) | 0.051 (4) | −0.002 (3) | 0.015 (3) | 0.002 (3) |
| C3 | 0.051 (5) | 0.087 (6) | 0.052 (5) | 0.000 (4) | 0.009 (4) | 0.017 (4) |
| C4 | 0.069 (6) | 0.097 (7) | 0.041 (4) | 0.037 (5) | 0.014 (4) | 0.004 (4) |
| C5 | 0.075 (6) | 0.062 (5) | 0.036 (4) | 0.026 (4) | 0.006 (4) | 0.010 (3) |
| C6 | 0.116 (8) | 0.074 (6) | 0.038 (4) | 0.056 (6) | 0.014 (5) | −0.006 (4) |
| C7 | 0.130 (9) | 0.054 (5) | 0.067 (6) | 0.026 (6) | −0.008 (6) | −0.008 (4) |
| C8 | 0.097 (6) | 0.043 (4) | 0.051 (5) | 0.016 (4) | 0.005 (4) | −0.011 (3) |
| C9 | 0.131 (9) | 0.038 (5) | 0.082 (7) | −0.009 (5) | −0.015 (6) | −0.006 (4) |
| C10 | 0.107 (8) | 0.054 (5) | 0.101 (8) | −0.024 (5) | 0.003 (6) | −0.003 (5) |
| C11 | 0.073 (5) | 0.049 (4) | 0.064 (5) | −0.016 (4) | 0.005 (4) | 0.001 (4) |
| C12 | 0.056 (4) | 0.044 (4) | 0.033 (4) | 0.016 (3) | 0.006 (3) | 0.005 (3) |
| C13 | 0.066 (5) | 0.043 (4) | 0.038 (4) | 0.006 (3) | 0.000 (3) | 0.005 (3) |
| C14 | 0.052 (4) | 0.058 (4) | 0.043 (4) | −0.003 (3) | 0.002 (3) | 0.006 (3) |
| C15 | 0.068 (6) | 0.072 (5) | 0.052 (5) | −0.004 (4) | 0.014 (4) | 0.013 (4) |
| C16 | 0.073 (6) | 0.087 (6) | 0.045 (5) | −0.001 (5) | −0.010 (4) | 0.015 (4) |
| C17 | 0.055 (5) | 0.088 (6) | 0.053 (5) | −0.005 (4) | 0.002 (4) | 0.009 (4) |
| C18 | 0.053 (5) | 0.141 (9) | 0.066 (6) | 0.002 (5) | −0.010 (4) | 0.025 (6) |
| C19 | 0.039 (5) | 0.160 (10) | 0.078 (7) | 0.008 (5) | 0.003 (4) | 0.023 (6) |
| C20 | 0.041 (5) | 0.097 (6) | 0.068 (6) | 0.005 (4) | 0.009 (4) | 0.019 (4) |
| C21 | 0.044 (5) | 0.116 (8) | 0.089 (7) | 0.008 (5) | 0.020 (5) | 0.013 (6) |
| C22 | 0.060 (6) | 0.103 (7) | 0.070 (6) | −0.001 (5) | 0.025 (5) | 0.020 (5) |
| C23 | 0.065 (5) | 0.066 (5) | 0.056 (5) | 0.001 (4) | 0.008 (4) | 0.014 (4) |
| C24 | 0.042 (4) | 0.049 (4) | 0.053 (4) | −0.008 (3) | 0.000 (3) | 0.006 (3) |
| C25 | 0.042 (4) | 0.056 (4) | 0.047 (4) | −0.005 (3) | 0.005 (3) | 0.007 (3) |
| C26 | 0.057 (4) | 0.034 (4) | 0.055 (4) | 0.001 (3) | 0.010 (4) | 0.004 (3) |
| C27 | 0.069 (5) | 0.046 (4) | 0.059 (5) | −0.001 (4) | 0.004 (4) | 0.010 (4) |
| C28 | 0.072 (5) | 0.062 (5) | 0.046 (4) | 0.001 (4) | 0.003 (4) | 0.019 (4) |
| C29 | 0.063 (5) | 0.054 (4) | 0.041 (4) | 0.004 (4) | 0.007 (3) | −0.003 (3) |
| C30 | 0.104 (7) | 0.069 (6) | 0.051 (5) | 0.008 (5) | −0.019 (5) | −0.005 (4) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C31 | 0.125 (8) | 0.053 (5) | 0.048 (5) | −0.003 (5) | −0.010 (5) | −0.019 (4) |
| C32 | 0.079 (5) | 0.038 (4) | 0.048 (4) | 0.000 (4) | 0.020 (4) | −0.005 (3) |
| C33 | 0.085 (6) | 0.037 (4) | 0.067 (5) | −0.005 (4) | 0.018 (4) | −0.009 (3) |
| C34 | 0.076 (5) | 0.031 (4) | 0.068 (5) | 0.010 (3) | 0.009 (4) | 0.003 (3) |
| C35 | 0.058 (4) | 0.035 (4) | 0.054 (4) | 0.005 (3) | 0.008 (3) | 0.006 (3) |
| C36 | 0.045 (4) | 0.034 (3) | 0.041 (4) | 0.000 (3) | 0.011 (3) | 0.001 (3) |
| C37 | 0.046 (4) | 0.040 (4) | 0.036 (4) | 0.001 (3) | 0.006 (3) | −0.002 (3) |
| C51 | 0.12 (2) | 0.16 (2) | 0.105 (18) | 0.066 (18) | 0.018 (15) | −0.036 (16) |
| C52 | 0.12 (3) | 0.45 (8) | 0.28 (5) | −0.07 (4) | −0.10 (3) | 0.30 (6) |
| O2 | 0.18 (2) | 0.147 (18) | 0.21 (2) | 0.007 (16) | −0.078 (18) | −0.017 (16) |

*Geometric parameters (Å, º)*

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|---|---|---|---|
| Fe1—N4 | 1.958 (6) | C14—H26 | 0.9300 |
| Fe1—N2 | 1.970 (5) | C15—C16 | 1.356 (10) |
| Fe1—N6 | 1.979 (5) | C15—H45 | 0.9300 |
| Fe1—N1 | 1.980 (5) | C16—C17 | 1.418 (10) |
| Fe1—N3 | 1.982 (5) | C16—H20 | 0.9300 |
| Fe1—N5 | 1.985 (5) | C17—C18 | 1.419 (11) |
| Fe2—O1 | 1.735 (5) | C17—C24 | 1.425 (9) |
| Fe2—Cl4 | 2.204 (3) | C18—C19 | 1.335 (11) |
| Fe2—Cl6 | 2.223 (2) | C18—H44 | 0.9300 |
| Fe2—Cl5 | 2.231 (2) | C19—C20 | 1.419 (11) |
| Fe3—O1 | 1.756 (5) | C19—H50 | 0.9300 |
| Fe3—Cl2 | 2.213 (2) | C20—C21 | 1.405 (11) |
| Fe3—Cl3 | 2.227 (2) | C20—C25 | 1.408 (10) |
| Fe3—Cl1 | 2.227 (2) | C21—C22 | 1.355 (11) |
| N1—C2 | 1.318 (8) | C21—H46 | 0.9300 |
| N1—C12 | 1.367 (8) | C22—C23 | 1.399 (11) |
| N2—C11 | 1.319 (9) | C22—H35 | 0.9300 |
| N2—C13 | 1.379 (9) | C23—H43 | 0.9300 |
| N3—C14 | 1.323 (8) | C24—C25 | 1.415 (9) |
| N3—C24 | 1.353 (8) | C26—C27 | 1.373 (9) |
| N4—C23 | 1.343 (8) | C26—H28 | 0.9300 |
| N4—C25 | 1.353 (8) | C27—C28 | 1.337 (10) |
| N5—C26 | 1.330 (7) | C27—H18 | 0.9300 |
| N5—C36 | 1.351 (8) | C28—C29 | 1.381 (9) |
| N6—C35 | 1.337 (7) | C28—H2 | 0.9300 |
| N6—C37 | 1.346 (7) | C29—C36 | 1.412 (9) |
| C2—C3 | 1.411 (10) | C29—C30 | 1.458 (10) |
| C2—H27 | 0.9300 | C30—C31 | 1.342 (11) |
| C3—C4 | 1.370 (11) | C30—H42 | 0.9300 |
| C3—H36 | 0.9300 | C31—C32 | 1.425 (10) |
| C4—C5 | 1.380 (11) | C31—H48 | 0.9300 |
| C4—H33 | 0.9300 | C32—C37 | 1.408 (9) |
| C5—C12 | 1.406 (10) | C32—C33 | 1.421 (9) |
| C5—C6 | 1.471 (11) | C33—C34 | 1.365 (10) |
| C6—C7 | 1.383 (13) | C33—H34 | 0.9300 |

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|---|---|---|---|
| C6—H38 | 0.9300 | C34—C35 | 1.389 (9) |
| C7—C8 | 1.399 (12) | C34—H25 | 0.9300 |
| C7—H47 | 0.9300 | C35—H19 | 0.9300 |
| C8—C9 | 1.391 (12) | C36—C37 | 1.420 (8) |
| C8—C13 | 1.395 (10) | C51—C52 | 1.487 (18) |
| C9—C10 | 1.358 (13) | C51—H51A | 0.9600 |
| C9—H32 | 0.9300 | C51—H51B | 0.9600 |
| C10—C11 | 1.377 (11) | C51—H51C | 0.9600 |
| C10—H49 | 0.9300 | C52—O2 | 1.440 (19) |
| C11—H37 | 0.9300 | C52—H52A | 0.9700 |
| C12—C13 | 1.420 (10) | C52—H52B | 0.9700 |
| C14—C15 | 1.387 (9) | O2—H2A | 0.8200 |

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|---|---|---|---|
| N4—Fe1—N2 | 91.0 (2) | C15—C14—H26 | 118.4 |
| N4—Fe1—N6 | 90.9 (2) | C16—C15—C14 | 120.0 (7) |
| N2—Fe1—N6 | 174.7 (2) | C16—C15—H45 | 120.0 |
| N4—Fe1—N1 | 172.7 (2) | C14—C15—H45 | 120.0 |
| N2—Fe1—N1 | 83.8 (2) | C15—C16—C17 | 119.8 (7) |
| N6—Fe1—N1 | 94.7 (2) | C15—C16—H20 | 120.1 |
| N4—Fe1—N3 | 82.8 (2) | C17—C16—H20 | 120.1 |
| N2—Fe1—N3 | 89.9 (2) | C18—C17—C16 | 125.7 (7) |
| N6—Fe1—N3 | 95.3 (2) | C18—C17—C24 | 118.4 (7) |
| N1—Fe1—N3 | 92.1 (2) | C16—C17—C24 | 115.8 (7) |
| N4—Fe1—N5 | 95.0 (2) | C19—C18—C17 | 121.0 (7) |
| N2—Fe1—N5 | 92.8 (2) | C19—C18—H44 | 119.5 |
| N6—Fe1—N5 | 82.1 (2) | C17—C18—H44 | 119.5 |
| N1—Fe1—N5 | 90.4 (2) | C18—C19—C20 | 122.3 (8) |
| N3—Fe1—N5 | 176.5 (2) | C18—C19—H50 | 118.8 |
| O1—Fe2—Cl4 | 112.4 (2) | C20—C19—H50 | 118.8 |
| O1—Fe2—Cl6 | 107.8 (2) | C21—C20—C25 | 115.5 (7) |
| Cl4—Fe2—Cl6 | 109.44 (11) | C21—C20—C19 | 125.9 (7) |
| O1—Fe2—Cl5 | 107.4 (2) | C25—C20—C19 | 118.6 (7) |
| Cl4—Fe2—Cl5 | 109.35 (12) | C22—C21—C20 | 120.4 (7) |
| Cl6—Fe2—Cl5 | 110.45 (9) | C22—C21—H46 | 119.8 |
| O1—Fe3—Cl2 | 112.5 (2) | C20—C21—H46 | 119.8 |
| O1—Fe3—Cl3 | 110.9 (2) | C21—C22—C23 | 120.2 (8) |
| Cl2—Fe3—Cl3 | 109.50 (9) | C21—C22—H35 | 119.9 |
| O1—Fe3—Cl1 | 108.7 (2) | C23—C22—H35 | 119.9 |
| Cl2—Fe3—Cl1 | 107.12 (9) | N4—C23—C22 | 121.9 (7) |
| Cl3—Fe3—Cl1 | 107.94 (9) | N4—C23—H43 | 119.0 |
| Fe2—O1—Fe3 | 160.6 (4) | C22—C23—H43 | 119.0 |
| C2—N1—C12 | 119.9 (6) | N3—C24—C25 | 116.6 (6) |
| C2—N1—Fe1 | 129.0 (5) | N3—C24—C17 | 123.3 (7) |
| C12—N1—Fe1 | 111.1 (4) | C25—C24—C17 | 120.0 (6) |
| C11—N2—C13 | 115.8 (6) | N4—C25—C20 | 124.9 (7) |
| C11—N2—Fe1 | 131.7 (6) | N4—C25—C24 | 115.5 (6) |
| C13—N2—Fe1 | 112.4 (5) | C20—C25—C24 | 119.6 (6) |
| C14—N3—C24 | 117.9 (6) | N5—C26—C27 | 123.1 (6) |

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|---|---|---|---|
| C14—N3—Fe1 | 130.4 (5) | N5—C26—H28 | 118.4 |
| C24—N3—Fe1 | 111.7 (4) | C27—C26—H28 | 118.4 |
| C23—N4—C25 | 117.1 (6) | C28—C27—C26 | 120.6 (7) |
| C23—N4—Fe1 | 129.8 (5) | C28—C27—H18 | 119.7 |
| C25—N4—Fe1 | 113.1 (4) | C26—C27—H18 | 119.7 |
| C26—N5—C36 | 116.7 (5) | C27—C28—C29 | 119.4 (7) |
| C26—N5—Fe1 | 130.4 (4) | C27—C28—H2 | 120.3 |
| C36—N5—Fe1 | 112.8 (4) | C29—C28—H2 | 120.3 |
| C35—N6—C37 | 117.4 (5) | C28—C29—C36 | 117.3 (6) |
| C35—N6—Fe1 | 129.5 (4) | C28—C29—C30 | 126.3 (7) |
| C37—N6—Fe1 | 113.1 (4) | C36—C29—C30 | 116.4 (6) |
| N1—C2—C3 | 120.7 (7) | C31—C30—C29 | 122.1 (7) |
| N1—C2—H27 | 119.7 | C31—C30—H42 | 118.9 |
| C3—C2—H27 | 119.7 | C29—C30—H42 | 118.9 |
| C4—C3—C2 | 120.2 (8) | C30—C31—C32 | 121.2 (7) |
| C4—C3—H36 | 119.9 | C30—C31—H48 | 119.4 |
| C2—C3—H36 | 119.9 | C32—C31—H48 | 119.4 |
| C3—C4—C5 | 119.5 (7) | C37—C32—C33 | 116.6 (6) |
| C3—C4—H33 | 120.2 | C37—C32—C31 | 118.8 (6) |
| C5—C4—H33 | 120.3 | C33—C32—C31 | 124.5 (7) |
| C4—C5—C12 | 118.2 (7) | C34—C33—C32 | 118.8 (6) |
| C4—C5—C6 | 124.9 (8) | C34—C33—H34 | 120.6 |
| C12—C5—C6 | 116.9 (8) | C32—C33—H34 | 120.6 |
| C7—C6—C5 | 119.9 (8) | C33—C34—C35 | 120.2 (6) |
| C7—C6—H38 | 120.1 | C33—C34—H25 | 119.9 |
| C5—C6—H38 | 120.1 | C35—C34—H25 | 119.9 |
| C6—C7—C8 | 122.9 (8) | N6—C35—C34 | 123.0 (6) |
| C6—C7—H47 | 118.6 | N6—C35—H19 | 118.5 |
| C8—C7—H47 | 118.6 | C34—C35—H19 | 118.5 |
| C9—C8—C13 | 117.2 (8) | N5—C36—C29 | 122.8 (6) |
| C9—C8—C7 | 125.1 (8) | N5—C36—C37 | 115.9 (5) |
| C13—C8—C7 | 117.7 (9) | C29—C36—C37 | 121.3 (6) |
| C10—C9—C8 | 120.2 (8) | N6—C37—C32 | 124.0 (6) |
| C10—C9—H32 | 119.9 | N6—C37—C36 | 116.1 (5) |
| C8—C9—H32 | 119.9 | C32—C37—C36 | 119.9 (6) |
| C9—C10—C11 | 118.5 (9) | C52—C51—H51A | 109.3 |
| C9—C10—H49 | 120.7 | C52—C51—H51B | 109.6 |
| C11—C10—H49 | 120.7 | H51A—C51—H51B | 109.5 |
| N2—C11—C10 | 125.0 (9) | C52—C51—H51C | 109.5 |
| N2—C11—H37 | 117.5 | H51A—C51—H51C | 109.5 |
| C10—C11—H37 | 117.5 | H51B—C51—H51C | 109.5 |
| N1—C12—C5 | 121.5 (7) | O2—C52—C51 | 109.5 (17) |
| N1—C12—C13 | 117.7 (6) | O2—C52—H52A | 109.9 |
| C5—C12—C13 | 120.8 (7) | C51—C52—H52A | 109.7 |
| N2—C13—C8 | 123.1 (7) | O2—C52—H52B | 109.8 |
| N2—C13—C12 | 115.0 (6) | C51—C52—H52B | 109.7 |
| C8—C13—C12 | 121.9 (7) | H52A—C52—H52B | 108.2 |
| N3—C14—C15 | 123.2 (7) | C52—O2—H2A | 109.5 |

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| N3—C14—H26 | 118.4 |