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# Tetrabutylammonium tetrakis(trimethylsilanolato-$\kappa O$)ferrate(III)

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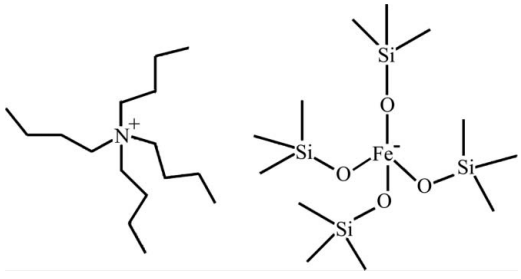
Key indicators: single-crystal X-ray study; $T$ = 173 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.041; $wR$ factor = 0.088; data-to-parameter ratio = 26.3.

In the title salt, $(C_{16}H_{36}N)[Fe(C_3H_9OSi)_4]$, the cation contains a central N atom bonded to four *n*-butyl alkyl groups in a tetrahedral arrangement, while the anion contains a central $Fe^{III}$ atom tetrahedrally coordinated by four trimethylsilanolate ligands.

## Related literature

For general background to the structural characterization of silsesquioxane compounds containing tetrabutylammonium iron(III), see: Hay & Geib (2007); Hay *et al.* (2003, 2009). For details of the synthesis, see: Shapley *et al.* (2003).




## Experimental

### Crystal data

$(C_{16}H_{36}N)[Fe(C_3H_9OSi)_4]$
$M_r$ = 655.08
Triclinic, $P\overline{1}$
$a$ = 10.4952 (5) Å
$b$ = 10.5143 (5) Å
$c$ = 19.3506 (9) Å
$\alpha$ = 82.722 (1)°
$\beta$ = 82.834 (1)°
$\gamma$ = 81.658 (1)°
$V$ = 2083.37 (17) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.50 mm$^{-1}$
$T$ = 173 K
0.56 × 0.35 × 0.28 mm

### Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
$T_{min}$ = 0.767, $T_{max}$ = 0.872
18068 measured reflections
9424 independent reflections
6223 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.071

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.088
$S$ = 0.93
9424 reflections
359 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.36 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.30 e Å$^{-3}$

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

| | | | |
|---|---|---|---|
| Fe1—O1 | 1.8608 (13) | Fe1—O3 | 1.8515 (14) |
| Fe1—O2 | 1.8591 (13) | Fe1—O4 | 1.8583 (13) |

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); 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*.

We thank the University College of the Pennsylvania State University and the Air Force Office of Scientific Research (FA9550–08-1–0213 F A9550–08-1–0213) for their financial support. The CCD-based X-ray diffractometer at Michigan State University was upgraded and/or replaced by departmental funds.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: VN2048).

## References

Bruker (2008). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
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Hay, M. T., Geib, S. J. & Pettner, D. A. (2009). *Polyhedron*, **28**, 2183–2186.
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# supporting information

*Acta Cryst.* (2012). E**68**, m1186 [doi:10.1107/S1600536812035337]

## Tetrabutylammonium tetrakis(trimethylsilanolato-$\kappa O$)ferrate(III)

**Michael Hay, Richard Staples and Andre Lee**

### S1. Comment

Over the past decade, Hay *et al.* reported on the structural characterization of numerous tetrabutylammonium iron (III) containing silsesquioxane compounds (Hay *et al.*, 2003; Hay & Geib, 2007, Hay *et al.*, 2009). In order to make a more complete structural study of these compounds, it was useful to have structural data on an analogous tetrabutylammonium iron (III) silanolato compound – the title compound (Fig. 1). Its structural arrangement contains a pair of tetrabutylammonium cations and tetrakistrimethylsilanolato ferrate (III) anions in a triclinic unit cell (Fig. 2). The tetrabutylammonium cation, $C_{16}H_{36}N^{+}$, consists of a tetrahedrally arranged central nitrogen atom, with N—C bond lengths in the range of 1.515 (2)–1.520 (2) Å and C—N—C bond angles in the range of 105.74 (13)–111.41 (14)°. The complex anion, $C_{12}H_{36}FeO_4Si_4^{-}$, contains a four coordinate $Fe^{III}$ atom with a tetrahedral arrangement of four trimethylsilanolato ligands. The O—Fe—O bond angles are 105.17 (6)–112.58 (6)°. The Fe—O bond lengths are in the range of 1.8515 (14)–1.8608 (13) Å.

### S2. Experimental

The following synthetic protocol is adapted from the previously reported procedure (Shapley, *et al.*, 2003). A yellow solution of $[C_{16}H_{36}N][FeCl_4]$ (1.136 mmol, 0.5000 g) in dichloromethane (10 ml) was treated with four equivalents of solid sodium trimethlysilanate (4.544 mmol, 0.5098 g). Immediately, the yellow solution turned red due to the formation of a dark red precipitate. The reaction mixture was stirred for 40 minutes before the precipitate was removed by filtration through celite. The resulting pale green filtrate was concentrated under reduced pressure to give a pale green powder. The powder was extracted with diethyl ether and filtered to remove any insoluble material. Hexanes were added to the diethyl ether filtrate and the sample was stored at 243 K for about 30 minutes before colorless block crystals formed which were analyzed.

### S3. Refinement

All H atoms were placed in calculated positions and refined using a riding model. C—H(aromatic) = 0.94 Å and $U_{iso}$(H) = 1.2$U_{eq}$(C); C—H (alaphatic) = 0.99 Å and $U_{iso}$(H) = 1.2$U_{eq}$(C); $CH_2$ = 0.98 Å and $U_{iso}$(H) = 1.2$U_{eq}$(C); $CH_3$ = 0.97 Å and $U_{iso}$(H) = 1.5$U_{eq}$(C); N—H = 0.86 (0.92) Å and $U_{iso}$(H) = 1.2$U_{eq}$(N); O—H(alcohol) = 0.85 Å and $U_{iso}$(H) = 1.2$U_{eq}$(O); O—H(acid) = 0.82 Å and $U_{iso}$(H) = 1.5$U_{eq}$(O). ?

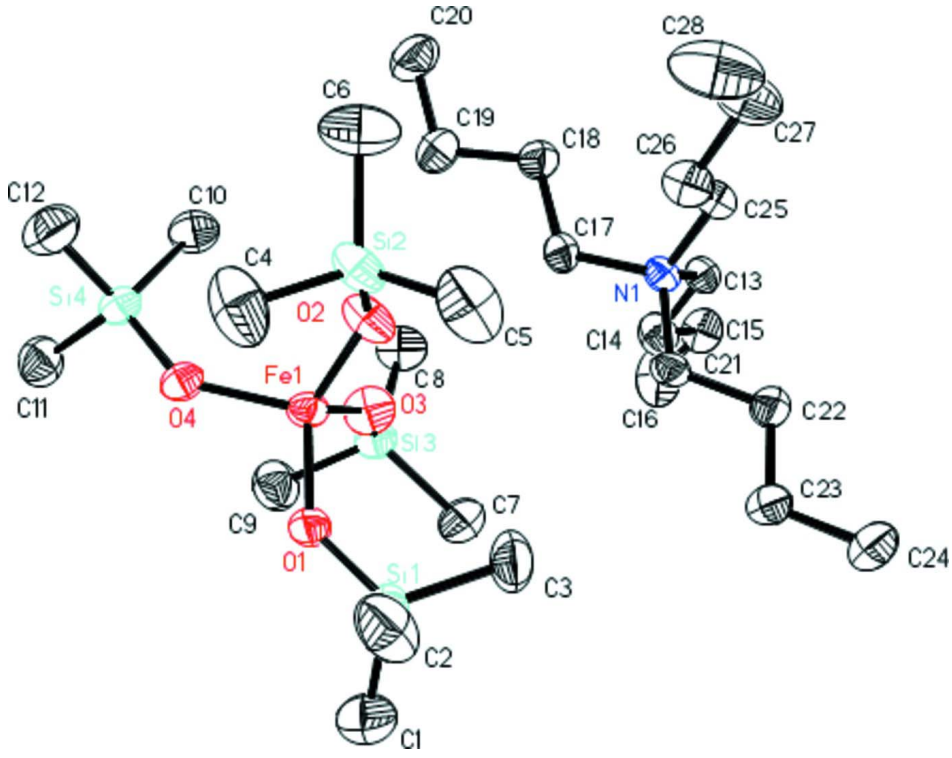


**Figure 1**

A 50% thermal ellipsoidal drawing of the asymmetric cell.

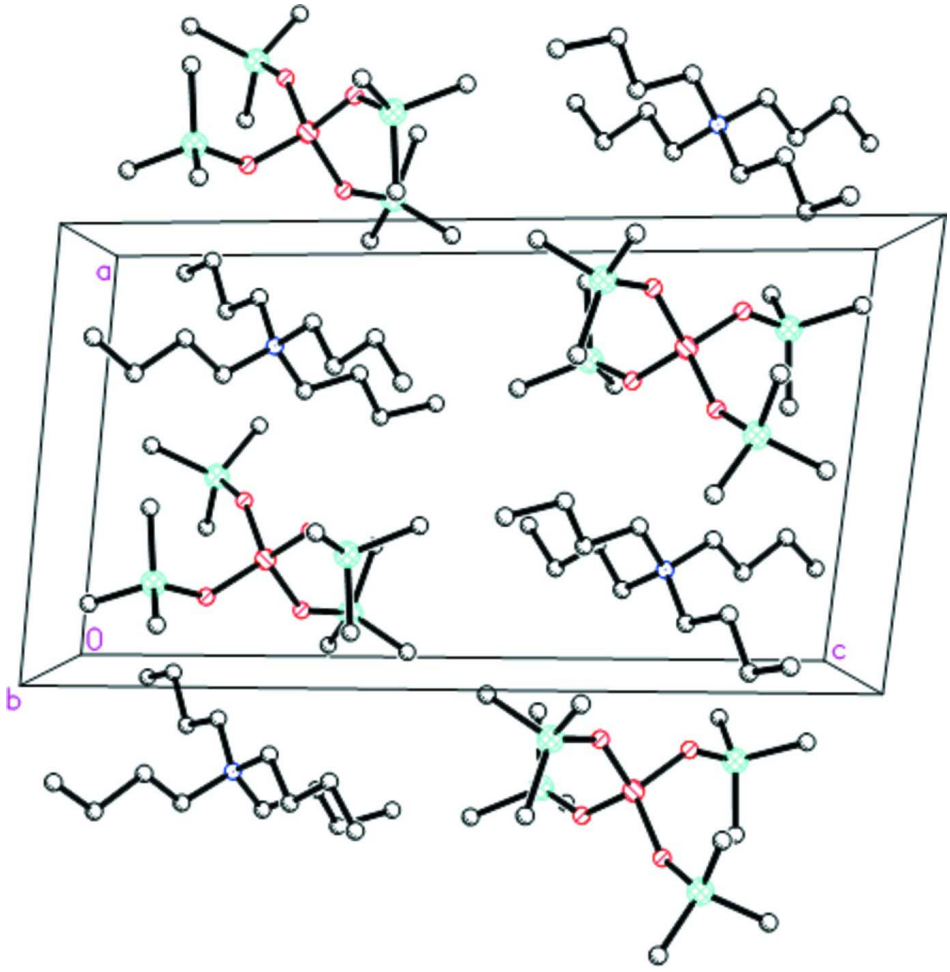


**Figure 2**

Drawing of the packing along the *b* axis.

**Tetrabutylammonium tetrakis(trimethylsilanolato-*κO*)ferrate(III)**

*Crystal data*

$(C_{16}H_{36}N)[Fe(C_3H_9OSi)_4]$
$M_r = 655.08$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 10.4952\ (5)$ Å
$b = 10.5143\ (5)$ Å
$c = 19.3506\ (9)$ Å
$\alpha = 82.722\ (1)°$
$\beta = 82.834\ (1)°$
$\gamma = 81.658\ (1)°$
$V = 2083.37\ (17)$ Å$^3$
$Z = 2$
$F(000) = 722$
$D_x = 1.044$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7900 reflections
$\theta = 2.3–27.4°$
$\mu = 0.50$ mm$^{-1}$
$T = 173$ K
Block, colourless
0.56 × 0.35 × 0.28 mm

*Data collection*

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 836.6 pixels mm$^{-1}$
$\omega$ and $\varphi$ 0.5 deg scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)

$T_{min}$ = 0.767, $T_{max}$ = 0.872
18068 measured reflections
9424 independent reflections
6223 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.071

$\theta_{max}$ = 28.6°, $\theta_{min}$ = 2.0°
$h$ = −14→13
$k$ = −13→14
$l$ = −25→25

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.088
$S$ = 0.93
9424 reflections
359 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0118P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.36 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.30 e Å$^{-3}$

*Special details*

**Experimental**. Data was collected using a BRUKER CCD (charge coupled device) based diffractometer equipped with an Oxford low-temperature apparatus operating at 173 K. A suitable crystal was chosen and mounted on a glass fiber or nylon loop using Paratone oil for Mo radiation and Mineral oil for Copper radiation. Data were measured using omega and phi scans of 0.5° per frame for 30 s. The total number of images were based on results from the program *COSMO* where redundancy was expected to be 4 and completeness to 0.83 Å to 100%. Cell parameters were retrieved using *APEX* II software and refined using *SAINT* on all observed reflections. Data reduction was performed using the *SAINT* software which corrects for Lp. Scaling and absorption corrections were applied using *SADABS6* multi-scan technique (Sheldrick, 2008). The structures are solved by the direct method using the *SHELXS97* program and refined by least squares method on $F^2$, *SHELXL97*, incorporated in *SHELXTL*-PC.

**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**. _michigan_contact_Crystallographer_name ′Richard Staples′ _michigan_contact_Crystallographer_email xraystaples@chemistry.msu.com

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Fe1 | 0.74285 (3) | 0.64074 (3) | 0.746004 (14) | 0.03091 (9) |
| Si1 | 0.79239 (6) | 0.42170 (6) | 0.87610 (3) | 0.04012 (16) |
| Si2 | 0.72382 (6) | 0.43686 (6) | 0.63710 (3) | 0.04463 (17) |
| Si3 | 0.54754 (6) | 0.86616 (6) | 0.82993 (3) | 0.03772 (15) |
| Si4 | 0.88113 (6) | 0.84989 (6) | 0.63377 (3) | 0.03860 (16) |
| O1 | 0.82805 (13) | 0.52860 (13) | 0.81292 (7) | 0.0461 (4) |
| O2 | 0.67258 (13) | 0.54525 (13) | 0.68921 (7) | 0.0456 (4) |
| O3 | 0.60452 (14) | 0.74786 (13) | 0.78552 (7) | 0.0483 (4) |
| O4 | 0.86943 (13) | 0.73219 (13) | 0.69549 (7) | 0.0416 (4) |
| C1 | 0.8425 (3) | 0.4626 (3) | 0.95884 (12) | 0.0724 (8) |
| H1A | 0.9368 | 0.4608 | 0.9541 | 0.109* |
| H1B | 0.8164 | 0.3994 | 0.9978 | 0.109* |
| H1C | 0.8007 | 0.5493 | 0.9681 | 0.109* |
| C2 | 0.8795 (2) | 0.2593 (2) | 0.85816 (15) | 0.0737 (9) |

| | | | | |
|---|---|---|---|---|
| H2A | 0.8566 | 0.2371 | 0.8140 | 0.111* |
| H2B | 0.8546 | 0.1942 | 0.8965 | 0.111* |
| H2C | 0.9732 | 0.2612 | 0.8546 | 0.111* |
| C3 | 0.6159 (2) | 0.4104 (3) | 0.88869 (14) | 0.0755 (9) |
| H3A | 0.5684 | 0.4925 | 0.9020 | 0.113* |
| H3B | 0.5979 | 0.3407 | 0.9258 | 0.113* |
| H3C | 0.5881 | 0.3919 | 0.8449 | 0.113* |
| C4 | 0.9026 (2) | 0.4104 (3) | 0.62274 (16) | 0.0805 (9) |
| H4A | 0.9343 | 0.4884 | 0.5974 | 0.121* |
| H4B | 0.9306 | 0.3374 | 0.5951 | 0.121* |
| H4C | 0.9377 | 0.3913 | 0.6681 | 0.121* |
| C5 | 0.6691 (2) | 0.2787 (2) | 0.67500 (16) | 0.0762 (9) |
| H5A | 0.7098 | 0.2473 | 0.7179 | 0.114* |
| H5B | 0.6941 | 0.2155 | 0.6410 | 0.114* |
| H5C | 0.5747 | 0.2903 | 0.6859 | 0.114* |
| C6 | 0.6578 (3) | 0.4858 (3) | 0.55180 (13) | 0.0842 (9) |
| H6A | 0.5632 | 0.4910 | 0.5587 | 0.126* |
| H6B | 0.6930 | 0.4217 | 0.5194 | 0.126* |
| H6C | 0.6824 | 0.5705 | 0.5322 | 0.126* |
| C7 | 0.4713 (2) | 0.8052 (2) | 0.91776 (11) | 0.0580 (7) |
| H7A | 0.3996 | 0.7586 | 0.9120 | 0.087* |
| H7B | 0.4384 | 0.8783 | 0.9446 | 0.087* |
| H7C | 0.5360 | 0.7465 | 0.9428 | 0.087* |
| C8 | 0.4215 (2) | 0.9769 (2) | 0.78437 (12) | 0.0525 (6) |
| H8A | 0.4607 | 1.0150 | 0.7393 | 0.079* |
| H8B | 0.3846 | 1.0459 | 0.8135 | 0.079* |
| H8C | 0.3528 | 0.9282 | 0.7764 | 0.079* |
| C9 | 0.6750 (2) | 0.9638 (2) | 0.84233 (13) | 0.0593 (7) |
| H9A | 0.7423 | 0.9088 | 0.8672 | 0.089* |
| H9B | 0.6366 | 1.0346 | 0.8699 | 0.089* |
| H9C | 0.7134 | 0.9996 | 0.7965 | 0.089* |
| C10 | 0.7182 (2) | 0.9279 (2) | 0.61219 (12) | 0.0544 (7) |
| H10A | 0.6715 | 0.8637 | 0.5972 | 0.082* |
| H10B | 0.7277 | 0.9982 | 0.5743 | 0.082* |
| H10C | 0.6695 | 0.9629 | 0.6537 | 0.082* |
| C11 | 0.9695 (2) | 0.9731 (2) | 0.66132 (12) | 0.0529 (6) |
| H11A | 0.9220 | 1.0071 | 0.7034 | 0.079* |
| H11B | 0.9766 | 1.0440 | 0.6235 | 0.079* |
| H11C | 1.0564 | 0.9331 | 0.6715 | 0.079* |
| C12 | 0.9728 (2) | 0.7892 (2) | 0.55258 (12) | 0.0647 (7) |
| H12A | 1.0562 | 0.7410 | 0.5638 | 0.097* |
| H12B | 0.9878 | 0.8627 | 0.5174 | 0.097* |
| H12C | 0.9224 | 0.7322 | 0.5341 | 0.097* |
| N1 | 0.24290 (14) | 0.52480 (15) | 0.75675 (8) | 0.0290 (4) |
| C13 | 0.12540 (17) | 0.61155 (18) | 0.78604 (10) | 0.0324 (5) |
| H13A | 0.0806 | 0.6579 | 0.7462 | 0.039* |
| H13B | 0.0651 | 0.5559 | 0.8142 | 0.039* |
| C14 | 0.15129 (19) | 0.7106 (2) | 0.83111 (11) | 0.0417 (5) |

| | | | | |
|---|---|---|---|---|
| H14A | 0.1976 | 0.6660 | 0.8707 | 0.050* |
| H14B | 0.2078 | 0.7701 | 0.8030 | 0.050* |
| C15 | 0.0275 (2) | 0.7877 (2) | 0.85923 (11) | 0.0438 (6) |
| H15A | −0.0253 | 0.7287 | 0.8906 | 0.053* |
| H15B | −0.0224 | 0.8250 | 0.8196 | 0.053* |
| C16 | 0.0493 (2) | 0.8961 (2) | 0.89928 (14) | 0.0655 (7) |
| H16A | 0.0958 | 0.8598 | 0.9397 | 0.098* |
| H16B | −0.0345 | 0.9430 | 0.9156 | 0.098* |
| H16C | 0.1007 | 0.9556 | 0.8685 | 0.098* |
| C17 | 0.34387 (18) | 0.60341 (19) | 0.71623 (10) | 0.0326 (5) |
| H17A | 0.4220 | 0.5432 | 0.7025 | 0.039* |
| H17B | 0.3688 | 0.6603 | 0.7479 | 0.039* |
| C18 | 0.30222 (19) | 0.6867 (2) | 0.65075 (10) | 0.0391 (5) |
| H18A | 0.2888 | 0.6304 | 0.6157 | 0.047* |
| H18B | 0.2189 | 0.7409 | 0.6627 | 0.047* |
| C19 | 0.4030 (2) | 0.7727 (2) | 0.61963 (11) | 0.0444 (6) |
| H19A | 0.4883 | 0.7191 | 0.6125 | 0.053* |
| H19B | 0.4098 | 0.8349 | 0.6530 | 0.053* |
| C20 | 0.3703 (2) | 0.8464 (2) | 0.55050 (11) | 0.0601 (7) |
| H20A | 0.3713 | 0.7853 | 0.5161 | 0.090* |
| H20B | 0.4345 | 0.9055 | 0.5337 | 0.090* |
| H20C | 0.2840 | 0.8961 | 0.5568 | 0.090* |
| C21 | 0.31012 (18) | 0.44134 (18) | 0.81477 (10) | 0.0329 (5) |
| H21A | 0.3380 | 0.4990 | 0.8450 | 0.039* |
| H21B | 0.3893 | 0.3916 | 0.7932 | 0.039* |
| C22 | 0.23097 (19) | 0.3468 (2) | 0.86101 (10) | 0.0400 (5) |
| H22A | 0.1541 | 0.3951 | 0.8854 | 0.048* |
| H22B | 0.2004 | 0.2894 | 0.8316 | 0.048* |
| C23 | 0.3114 (2) | 0.2658 (2) | 0.91458 (10) | 0.0420 (5) |
| H23A | 0.3423 | 0.3236 | 0.9437 | 0.050* |
| H23B | 0.3882 | 0.2178 | 0.8899 | 0.050* |
| C24 | 0.2344 (2) | 0.1701 (2) | 0.96193 (12) | 0.0609 (7) |
| H24A | 0.1604 | 0.2175 | 0.9881 | 0.091* |
| H24B | 0.2902 | 0.1181 | 0.9949 | 0.091* |
| H24C | 0.2030 | 0.1132 | 0.9333 | 0.091* |
| C25 | 0.19311 (19) | 0.44218 (19) | 0.70961 (10) | 0.0344 (5) |
| H25A | 0.1265 | 0.3941 | 0.7378 | 0.041* |
| H25B | 0.1502 | 0.5003 | 0.6726 | 0.041* |
| C26 | 0.2951 (2) | 0.3457 (2) | 0.67477 (11) | 0.0448 (6) |
| H26A | 0.3357 | 0.2839 | 0.7112 | 0.054* |
| H26B | 0.3635 | 0.3922 | 0.6471 | 0.054* |
| C27 | 0.2357 (2) | 0.2720 (2) | 0.62695 (13) | 0.0622 (7) |
| H27A | 0.1715 | 0.2212 | 0.6554 | 0.075* |
| H27B | 0.1895 | 0.3346 | 0.5929 | 0.075* |
| C28 | 0.3361 (3) | 0.1820 (3) | 0.58759 (15) | 0.0907 (10) |
| H28A | 0.3972 | 0.2323 | 0.5574 | 0.136* |
| H28B | 0.2933 | 0.1345 | 0.5588 | 0.136* |
| H28C | 0.3830 | 0.1206 | 0.6211 | 0.136* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Fe1 | 0.03597 (17) | 0.02678 (16) | 0.03030 (17) | −0.00261 (13) | −0.00944 (13) | −0.00036 (12) |
| Si1 | 0.0344 (3) | 0.0395 (4) | 0.0461 (4) | −0.0115 (3) | −0.0156 (3) | 0.0149 (3) |
| Si2 | 0.0378 (4) | 0.0445 (4) | 0.0565 (4) | 0.0014 (3) | −0.0152 (3) | −0.0219 (3) |
| Si3 | 0.0411 (4) | 0.0376 (3) | 0.0337 (3) | −0.0011 (3) | −0.0025 (3) | −0.0072 (3) |
| Si4 | 0.0440 (4) | 0.0387 (3) | 0.0314 (3) | −0.0072 (3) | −0.0058 (3) | 0.0063 (3) |
| O1 | 0.0402 (8) | 0.0498 (9) | 0.0476 (9) | −0.0145 (7) | −0.0188 (7) | 0.0210 (7) |
| O2 | 0.0356 (8) | 0.0480 (9) | 0.0580 (10) | 0.0021 (7) | −0.0150 (8) | −0.0235 (8) |
| O3 | 0.0533 (9) | 0.0416 (9) | 0.0477 (9) | 0.0050 (8) | −0.0012 (8) | −0.0130 (7) |
| O4 | 0.0465 (9) | 0.0388 (8) | 0.0377 (8) | −0.0081 (7) | −0.0100 (7) | 0.0114 (7) |
| C1 | 0.082 (2) | 0.087 (2) | 0.0508 (16) | −0.0161 (17) | −0.0132 (15) | −0.0040 (15) |
| C2 | 0.0672 (18) | 0.0410 (15) | 0.119 (2) | −0.0111 (14) | −0.0364 (18) | 0.0005 (15) |
| C3 | 0.0402 (15) | 0.083 (2) | 0.097 (2) | −0.0209 (15) | −0.0119 (15) | 0.0309 (17) |
| C4 | 0.0470 (16) | 0.086 (2) | 0.114 (2) | −0.0026 (16) | 0.0061 (17) | −0.0519 (19) |
| C5 | 0.0668 (18) | 0.0441 (15) | 0.120 (3) | 0.0008 (14) | −0.0237 (18) | −0.0148 (16) |
| C6 | 0.111 (2) | 0.084 (2) | 0.0618 (19) | 0.0044 (19) | −0.0300 (18) | −0.0245 (16) |
| C7 | 0.0594 (16) | 0.0718 (17) | 0.0410 (14) | −0.0125 (14) | 0.0010 (12) | −0.0021 (12) |
| C8 | 0.0564 (15) | 0.0482 (14) | 0.0511 (15) | 0.0045 (12) | −0.0079 (12) | −0.0102 (12) |
| C9 | 0.0579 (16) | 0.0612 (16) | 0.0626 (17) | −0.0139 (14) | −0.0074 (14) | −0.0136 (13) |
| C10 | 0.0578 (15) | 0.0532 (15) | 0.0488 (15) | −0.0031 (13) | −0.0166 (13) | 0.0130 (12) |
| C11 | 0.0542 (15) | 0.0461 (14) | 0.0578 (15) | −0.0126 (12) | −0.0058 (13) | 0.0032 (12) |
| C12 | 0.0726 (18) | 0.0747 (18) | 0.0430 (15) | −0.0062 (15) | 0.0006 (14) | −0.0025 (13) |
| N1 | 0.0224 (8) | 0.0352 (9) | 0.0299 (9) | −0.0038 (7) | −0.0066 (7) | −0.0014 (7) |
| C13 | 0.0224 (10) | 0.0390 (12) | 0.0350 (12) | −0.0030 (9) | −0.0043 (9) | −0.0013 (9) |
| C14 | 0.0330 (12) | 0.0459 (13) | 0.0467 (13) | −0.0041 (11) | −0.0023 (11) | −0.0106 (11) |
| C15 | 0.0413 (13) | 0.0398 (13) | 0.0477 (14) | −0.0014 (11) | 0.0012 (11) | −0.0050 (11) |
| C16 | 0.0629 (17) | 0.0500 (15) | 0.0823 (19) | −0.0071 (14) | 0.0130 (15) | −0.0228 (14) |
| C17 | 0.0239 (10) | 0.0388 (12) | 0.0344 (11) | −0.0050 (9) | −0.0025 (9) | −0.0015 (9) |
| C18 | 0.0357 (12) | 0.0439 (13) | 0.0361 (12) | −0.0050 (10) | −0.0057 (10) | 0.0028 (10) |
| C19 | 0.0500 (14) | 0.0397 (13) | 0.0412 (13) | −0.0080 (11) | −0.0017 (11) | 0.0032 (10) |
| C20 | 0.0754 (18) | 0.0564 (16) | 0.0459 (15) | −0.0174 (14) | −0.0037 (14) | 0.0116 (12) |
| C21 | 0.0273 (11) | 0.0377 (12) | 0.0327 (11) | 0.0011 (9) | −0.0103 (9) | 0.0000 (9) |
| C22 | 0.0341 (12) | 0.0475 (13) | 0.0370 (12) | −0.0077 (11) | −0.0039 (10) | 0.0035 (10) |
| C23 | 0.0481 (13) | 0.0433 (13) | 0.0341 (12) | −0.0077 (11) | −0.0071 (11) | 0.0017 (10) |
| C24 | 0.0762 (18) | 0.0606 (16) | 0.0462 (15) | −0.0233 (15) | −0.0104 (14) | 0.0130 (13) |
| C25 | 0.0332 (12) | 0.0395 (12) | 0.0326 (11) | −0.0081 (10) | −0.0082 (9) | −0.0041 (9) |
| C26 | 0.0433 (13) | 0.0457 (14) | 0.0456 (14) | −0.0005 (11) | −0.0070 (11) | −0.0096 (11) |
| C27 | 0.0691 (18) | 0.0645 (17) | 0.0581 (16) | 0.0015 (15) | −0.0169 (14) | −0.0282 (14) |
| C28 | 0.112 (3) | 0.081 (2) | 0.082 (2) | 0.0331 (19) | −0.040 (2) | −0.0439 (17) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Fe1—O1 | 1.8608 (13) | C12—H12B | 0.9800 |
| Fe1—O2 | 1.8591 (13) | C12—H12C | 0.9800 |
| Fe1—O3 | 1.8515 (14) | N1—C21 | 1.515 (2) |
| Fe1—O4 | 1.8583 (13) | N1—C17 | 1.518 (2) |

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|---|---|---|---|
| Si1—O1 | 1.5993 (14) | N1—C13 | 1.518 (2) |
| Si1—C3 | 1.857 (2) | N1—C25 | 1.520 (2) |
| Si1—C1 | 1.864 (2) | C13—C14 | 1.514 (3) |
| Si1—C2 | 1.869 (2) | C13—H13A | 0.9900 |
| Si2—O2 | 1.6071 (14) | C13—H13B | 0.9900 |
| Si2—C4 | 1.847 (2) | C14—C15 | 1.507 (3) |
| Si2—C6 | 1.853 (2) | C14—H14A | 0.9900 |
| Si2—C5 | 1.870 (2) | C14—H14B | 0.9900 |
| Si3—O3 | 1.6031 (15) | C15—C16 | 1.515 (3) |
| Si3—C9 | 1.855 (2) | C15—H15A | 0.9900 |
| Si3—C8 | 1.862 (2) | C15—H15B | 0.9900 |
| Si3—C7 | 1.864 (2) | C16—H16A | 0.9800 |
| Si4—O4 | 1.6143 (13) | C16—H16B | 0.9800 |
| Si4—C10 | 1.859 (2) | C16—H16C | 0.9800 |
| Si4—C11 | 1.863 (2) | C17—C18 | 1.520 (2) |
| Si4—C12 | 1.871 (2) | C17—H17A | 0.9900 |
| C1—H1A | 0.9800 | C17—H17B | 0.9900 |
| C1—H1B | 0.9800 | C18—C19 | 1.510 (3) |
| C1—H1C | 0.9800 | C18—H18A | 0.9900 |
| C2—H2A | 0.9800 | C18—H18B | 0.9900 |
| C2—H2B | 0.9800 | C19—C20 | 1.511 (3) |
| C2—H2C | 0.9800 | C19—H19A | 0.9900 |
| C3—H3A | 0.9800 | C19—H19B | 0.9900 |
| C3—H3B | 0.9800 | C20—H20A | 0.9800 |
| C3—H3C | 0.9800 | C20—H20B | 0.9800 |
| C4—H4A | 0.9800 | C20—H20C | 0.9800 |
| C4—H4B | 0.9800 | C21—C22 | 1.519 (2) |
| C4—H4C | 0.9800 | C21—H21A | 0.9900 |
| C5—H5A | 0.9800 | C21—H21B | 0.9900 |
| C5—H5B | 0.9800 | C22—C23 | 1.516 (2) |
| C5—H5C | 0.9800 | C22—H22A | 0.9900 |
| C6—H6A | 0.9800 | C22—H22B | 0.9900 |
| C6—H6B | 0.9800 | C23—C24 | 1.524 (3) |
| C6—H6C | 0.9800 | C23—H23A | 0.9900 |
| C7—H7A | 0.9800 | C23—H23B | 0.9900 |
| C7—H7B | 0.9800 | C24—H24A | 0.9800 |
| C7—H7C | 0.9800 | C24—H24B | 0.9800 |
| C8—H8A | 0.9800 | C24—H24C | 0.9800 |
| C8—H8B | 0.9800 | C25—C26 | 1.519 (3) |
| C8—H8C | 0.9800 | C25—H25A | 0.9900 |
| C9—H9A | 0.9800 | C25—H25B | 0.9900 |
| C9—H9B | 0.9800 | C26—C27 | 1.520 (3) |
| C9—H9C | 0.9800 | C26—H26A | 0.9900 |
| C10—H10A | 0.9800 | C26—H26B | 0.9900 |
| C10—H10B | 0.9800 | C27—C28 | 1.510 (3) |
| C10—H10C | 0.9800 | C27—H27A | 0.9900 |
| C11—H11A | 0.9800 | C27—H27B | 0.9900 |
| C11—H11B | 0.9800 | C28—H28A | 0.9800 |

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|---|---|---|---|
| C11—H11C | 0.9800 | C28—H28B | 0.9800 |
| C12—H12A | 0.9800 | C28—H28C | 0.9800 |
| | | | |
| O3—Fe1—O4 | 112.58 (6) | H12A—C12—H12C | 109.5 |
| O3—Fe1—O2 | 105.76 (6) | H12B—C12—H12C | 109.5 |
| O4—Fe1—O2 | 111.55 (6) | C21—N1—C17 | 105.74 (13) |
| O3—Fe1—O1 | 112.53 (7) | C21—N1—C13 | 111.41 (14) |
| O4—Fe1—O1 | 105.17 (6) | C17—N1—C13 | 111.40 (14) |
| O2—Fe1—O1 | 109.33 (6) | C21—N1—C25 | 111.07 (14) |
| O1—Si1—C3 | 111.45 (9) | C17—N1—C25 | 111.13 (15) |
| O1—Si1—C1 | 109.84 (10) | C13—N1—C25 | 106.18 (13) |
| C3—Si1—C1 | 108.97 (13) | C14—C13—N1 | 116.35 (15) |
| O1—Si1—C2 | 110.30 (11) | C14—C13—H13A | 108.2 |
| C3—Si1—C2 | 107.99 (12) | N1—C13—H13A | 108.2 |
| C1—Si1—C2 | 108.20 (12) | C14—C13—H13B | 108.2 |
| O2—Si2—C4 | 111.65 (10) | N1—C13—H13B | 108.2 |
| O2—Si2—C6 | 109.81 (10) | H13A—C13—H13B | 107.4 |
| C4—Si2—C6 | 109.42 (14) | C15—C14—C13 | 111.60 (16) |
| O2—Si2—C5 | 110.26 (11) | C15—C14—H14A | 109.3 |
| C4—Si2—C5 | 107.23 (12) | C13—C14—H14A | 109.3 |
| C6—Si2—C5 | 108.38 (13) | C15—C14—H14B | 109.3 |
| O3—Si3—C9 | 111.92 (10) | C13—C14—H14B | 109.3 |
| O3—Si3—C8 | 110.69 (9) | H14A—C14—H14B | 108.0 |
| C9—Si3—C8 | 107.22 (11) | C14—C15—C16 | 113.43 (18) |
| O3—Si3—C7 | 110.32 (10) | C14—C15—H15A | 108.9 |
| C9—Si3—C7 | 108.60 (11) | C16—C15—H15A | 108.9 |
| C8—Si3—C7 | 107.95 (11) | C14—C15—H15B | 108.9 |
| O4—Si4—C10 | 110.89 (9) | C16—C15—H15B | 108.9 |
| O4—Si4—C11 | 109.98 (9) | H15A—C15—H15B | 107.7 |
| C10—Si4—C11 | 109.09 (10) | C15—C16—H16A | 109.5 |
| O4—Si4—C12 | 110.34 (9) | C15—C16—H16B | 109.5 |
| C10—Si4—C12 | 108.16 (11) | H16A—C16—H16B | 109.5 |
| C11—Si4—C12 | 108.32 (11) | C15—C16—H16C | 109.5 |
| Si1—O1—Fe1 | 137.69 (9) | H16A—C16—H16C | 109.5 |
| Si2—O2—Fe1 | 137.57 (8) | H16B—C16—H16C | 109.5 |
| Si3—O3—Fe1 | 151.05 (10) | N1—C17—C18 | 115.47 (14) |
| Si4—O4—Fe1 | 139.01 (8) | N1—C17—H17A | 108.4 |
| Si1—C1—H1A | 109.5 | C18—C17—H17A | 108.4 |
| Si1—C1—H1B | 109.5 | N1—C17—H17B | 108.4 |
| H1A—C1—H1B | 109.5 | C18—C17—H17B | 108.4 |
| Si1—C1—H1C | 109.5 | H17A—C17—H17B | 107.5 |
| H1A—C1—H1C | 109.5 | C19—C18—C17 | 111.02 (15) |
| H1B—C1—H1C | 109.5 | C19—C18—H18A | 109.4 |
| Si1—C2—H2A | 109.5 | C17—C18—H18A | 109.4 |
| Si1—C2—H2B | 109.5 | C19—C18—H18B | 109.4 |
| H2A—C2—H2B | 109.5 | C17—C18—H18B | 109.4 |
| Si1—C2—H2C | 109.5 | H18A—C18—H18B | 108.0 |
| H2A—C2—H2C | 109.5 | C18—C19—C20 | 111.97 (17) |

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|---|---|---|---|
| H2B—C2—H2C | 109.5 | C18—C19—H19A | 109.2 |
| Si1—C3—H3A | 109.5 | C20—C19—H19A | 109.2 |
| Si1—C3—H3B | 109.5 | C18—C19—H19B | 109.2 |
| H3A—C3—H3B | 109.5 | C20—C19—H19B | 109.2 |
| Si1—C3—H3C | 109.5 | H19A—C19—H19B | 107.9 |
| H3A—C3—H3C | 109.5 | C19—C20—H20A | 109.5 |
| H3B—C3—H3C | 109.5 | C19—C20—H20B | 109.5 |
| Si2—C4—H4A | 109.5 | H20A—C20—H20B | 109.5 |
| Si2—C4—H4B | 109.5 | C19—C20—H20C | 109.5 |
| H4A—C4—H4B | 109.5 | H20A—C20—H20C | 109.5 |
| Si2—C4—H4C | 109.5 | H20B—C20—H20C | 109.5 |
| H4A—C4—H4C | 109.5 | N1—C21—C22 | 116.20 (14) |
| H4B—C4—H4C | 109.5 | N1—C21—H21A | 108.2 |
| Si2—C5—H5A | 109.5 | C22—C21—H21A | 108.2 |
| Si2—C5—H5B | 109.5 | N1—C21—H21B | 108.2 |
| H5A—C5—H5B | 109.5 | C22—C21—H21B | 108.2 |
| Si2—C5—H5C | 109.5 | H21A—C21—H21B | 107.4 |
| H5A—C5—H5C | 109.5 | C23—C22—C21 | 110.69 (15) |
| H5B—C5—H5C | 109.5 | C23—C22—H22A | 109.5 |
| Si2—C6—H6A | 109.5 | C21—C22—H22A | 109.5 |
| Si2—C6—H6B | 109.5 | C23—C22—H22B | 109.5 |
| H6A—C6—H6B | 109.5 | C21—C22—H22B | 109.5 |
| Si2—C6—H6C | 109.5 | H22A—C22—H22B | 108.1 |
| H6A—C6—H6C | 109.5 | C22—C23—C24 | 111.91 (17) |
| H6B—C6—H6C | 109.5 | C22—C23—H23A | 109.2 |
| Si3—C7—H7A | 109.5 | C24—C23—H23A | 109.2 |
| Si3—C7—H7B | 109.5 | C22—C23—H23B | 109.2 |
| H7A—C7—H7B | 109.5 | C24—C23—H23B | 109.2 |
| Si3—C7—H7C | 109.5 | H23A—C23—H23B | 107.9 |
| H7A—C7—H7C | 109.5 | C23—C24—H24A | 109.5 |
| H7B—C7—H7C | 109.5 | C23—C24—H24B | 109.5 |
| Si3—C8—H8A | 109.5 | H24A—C24—H24B | 109.5 |
| Si3—C8—H8B | 109.5 | C23—C24—H24C | 109.5 |
| H8A—C8—H8B | 109.5 | H24A—C24—H24C | 109.5 |
| Si3—C8—H8C | 109.5 | H24B—C24—H24C | 109.5 |
| H8A—C8—H8C | 109.5 | C26—C25—N1 | 115.46 (15) |
| H8B—C8—H8C | 109.5 | C26—C25—H25A | 108.4 |
| Si3—C9—H9A | 109.5 | N1—C25—H25A | 108.4 |
| Si3—C9—H9B | 109.5 | C26—C25—H25B | 108.4 |
| H9A—C9—H9B | 109.5 | N1—C25—H25B | 108.4 |
| Si3—C9—H9C | 109.5 | H25A—C25—H25B | 107.5 |
| H9A—C9—H9C | 109.5 | C25—C26—C27 | 111.05 (17) |
| H9B—C9—H9C | 109.5 | C25—C26—H26A | 109.4 |
| Si4—C10—H10A | 109.5 | C27—C26—H26A | 109.4 |
| Si4—C10—H10B | 109.5 | C25—C26—H26B | 109.4 |
| H10A—C10—H10B | 109.5 | C27—C26—H26B | 109.4 |
| Si4—C10—H10C | 109.5 | H26A—C26—H26B | 108.0 |
| H10A—C10—H10C | 109.5 | C28—C27—C26 | 112.4 (2) |

| | | | |
|---|---|---|---|
| H10B—C10—H10C | 109.5 | C28—C27—H27A | 109.1 |
| Si4—C11—H11A | 109.5 | C26—C27—H27A | 109.1 |
| Si4—C11—H11B | 109.5 | C28—C27—H27B | 109.1 |
| H11A—C11—H11B | 109.5 | C26—C27—H27B | 109.1 |
| Si4—C11—H11C | 109.5 | H27A—C27—H27B | 107.9 |
| H11A—C11—H11C | 109.5 | C27—C28—H28A | 109.5 |
| H11B—C11—H11C | 109.5 | C27—C28—H28B | 109.5 |
| Si4—C12—H12A | 109.5 | H28A—C28—H28B | 109.5 |
| Si4—C12—H12B | 109.5 | C27—C28—H28C | 109.5 |
| H12A—C12—H12B | 109.5 | H28A—C28—H28C | 109.5 |
| Si4—C12—H12C | 109.5 | H28B—C28—H28C | 109.5 |

| | | | |
|---|---|---|---|
| C3—Si1—O1—Fe1 | −3.07 (19) | O2—Fe1—O4—Si4 | 66.36 (15) |
| C1—Si1—O1—Fe1 | −123.94 (15) | O1—Fe1—O4—Si4 | −175.23 (13) |
| C2—Si1—O1—Fe1 | 116.88 (16) | C21—N1—C13—C14 | 63.1 (2) |
| O3—Fe1—O1—Si1 | 55.77 (16) | C17—N1—C13—C14 | −54.7 (2) |
| O4—Fe1—O1—Si1 | 178.67 (13) | C25—N1—C13—C14 | −175.85 (16) |
| O2—Fe1—O1—Si1 | −61.44 (16) | N1—C13—C14—C15 | −177.98 (16) |
| C4—Si2—O2—Fe1 | −7.49 (19) | C13—C14—C15—C16 | −174.84 (18) |
| C6—Si2—O2—Fe1 | −129.04 (15) | C21—N1—C17—C18 | 174.73 (16) |
| C5—Si2—O2—Fe1 | 111.60 (15) | C13—N1—C17—C18 | −64.1 (2) |
| O3—Fe1—O2—Si2 | 178.46 (13) | C25—N1—C17—C18 | 54.1 (2) |
| O4—Fe1—O2—Si2 | 55.75 (15) | N1—C17—C18—C19 | 173.01 (17) |
| O1—Fe1—O2—Si2 | −60.14 (15) | C17—C18—C19—C20 | 174.14 (18) |
| C9—Si3—O3—Fe1 | 12.8 (2) | C17—N1—C21—C22 | −176.67 (16) |
| C8—Si3—O3—Fe1 | 132.34 (18) | C13—N1—C21—C22 | 62.1 (2) |
| C7—Si3—O3—Fe1 | −108.2 (2) | C25—N1—C21—C22 | −56.0 (2) |
| O4—Fe1—O3—Si3 | −48.4 (2) | N1—C21—C22—C23 | 177.55 (16) |
| O2—Fe1—O3—Si3 | −170.41 (18) | C21—C22—C23—C24 | 179.85 (18) |
| O1—Fe1—O3—Si3 | 70.3 (2) | C21—N1—C25—C26 | −58.1 (2) |
| C10—Si4—O4—Fe1 | 7.56 (17) | C17—N1—C25—C26 | 59.3 (2) |
| C11—Si4—O4—Fe1 | 128.31 (14) | C13—N1—C25—C26 | −179.38 (16) |
| C12—Si4—O4—Fe1 | −112.26 (15) | N1—C25—C26—C27 | −177.93 (17) |
| O3—Fe1—O4—Si4 | −52.36 (15) | C25—C26—C27—C28 | 176.0 (2) |