# Side-on coordination mode of a pyrazolyl group in the structure of a divalent [Sm{B(3-Mepz)$_4$}$_2$] complex (3-Mepz is 3-methylpyrazol-1-yl)

**Yasuhiro Arikawa, Kei-ichiro Inada and Masayoshi Onishi**

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# Side-on coordination mode of a pyrazolyl group in the structure of a divalent [Sm{B(3-Mepz)₄}₂] complex (3-Mepz is 3-methylpyrazol-1-yl)

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The discovery of polypyrazolylborate ligands allowed the development of various chemical fields and these ligands are an alternative to cyclopentadienyl, because both ligands have the same charge and donate the same number of electrons, as well as adopting the same facial geometry. Easy control of the bulkiness of polypyrazolylborate ligands is possible by modification of the substituents in the 3- and 5-positions of the pyrazolyl rings. The title complex, bis[tetrakis(3-methyl-1$H$-pyrazol-1-yl)borato]samarium(II), [Sm(C₁₆H₂₀BN₈)₂], was synthesized from the reaction of SmI₂ with potassium tetrakis(3-methyl-1$H$-pyrazol-1-yl)borate, denoted K[B(3-Mepz)₄], in tetrahydrofuran. The X-ray structure analysis revealed an unusual side-on coordination mode of a 3-methylpyrazolyl group through an N=N group in the B(3-Mepz)₄ ligand. The distortion is defined by the B—N—N—Sm torsion angle [85.5 (4)°]. This is in contrast to the structure of the similar divalent samarium complex [Sm(Tp^Me₂)₂] [Tp^Me₂ is tris(3,5-dimethylpyrazol-1-yl)borate], which displays normal $\kappa^3$-bonding modes of the Tp^Me₂ ligands.

## 1. Introduction

Thanks to Trofimenko's discovery of polypyrazolylborate (Trofimenko, 1966), marvelous chemical fields have been opened up. Many interesting results have been reported and developments still continue (Trofimenko, 1999; Pettinari, 2008). The polypyrazolylborate ligands have been an alternative for cyclopentadienyl derivatives which are one of the most useful types of ligands, because both ligands have the same charge and donate the same number of electrons, as well as adopting the same facial geometry. The cone angle of polypyrazolylborate ligands is usually wider than that of the corresponding cyclopentadienyl derivatives; thus, in the lanthanide and actinide complexes, which have a larger metal ionic radius, the polypyrazolylborate ligands have often been used (Marques *et al.*, 2002; Santos & Marques, 1995). Easy control of the bulkiness by modification of the substituents on the 3- and 5-positions of the pyrazolyl rings is also an attractive feature of these ligands. We are interested in samarium complexes having polypyrazolylborate ligands (Onishi *et al.*, 1998, 2004). In connection with interesting reduction reactions of divalent lanthanides complexes, five divalent hydrido-tris(pyrazolyl)borate complexes of samarium have been structurally characterized, namely [Sm(Tp^{*t*Bu,Me*t*Bu,Me})₂] [Tp^{*t*Bu,Me} is tris(3-*tert*-butyl-5-methylpyrazol-1-yl)borate] (Zhang *et al.*, 1995), [Sm(Tp^Me₂)₂] [Tp^Me₂ is tris(3,5-dimethyl-pyrazol-1-yl)-

electronic reprint
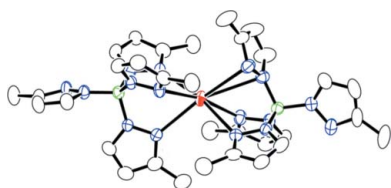
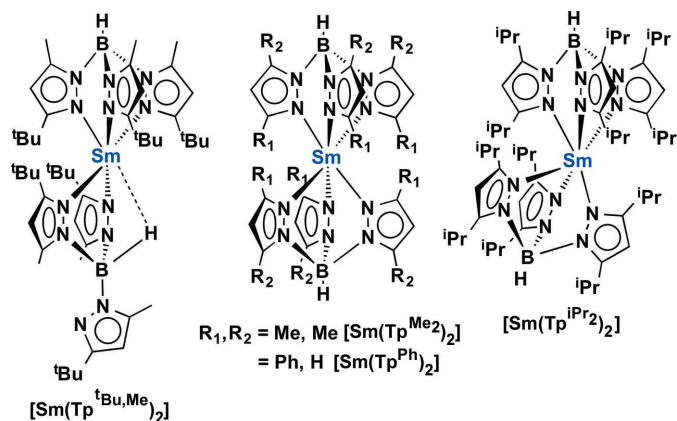**Figure 1**
Schematic illustration of the divalent hydridotris(pyrazolyl)borate samarium complexes.

borate], [Sm(Tp$^{Me_2,4-Et}$)$_2$] [Tp$^{Me_2,4-Et}$ is tris(4-ethyl-3,5-di-methylpyrazol-1-yl)borate] and [Sm(Tp$^{Ph}$)$_2$] [Tp$^{Ph}$ is tris(3-phenylpyrazol-1-yl)borate] (Hillier *et al.*, 2001), and [Sm-(Tp$^{iPr_2}$)$_2$] [Tp$^{iPr_2}$ is tris(3,5-diisopropylpyrazol-1-yl)borate] (Momin *et al.*, 2014; Kuhling *et al.*, 2015) (Fig. 1). The coordination of the substituted Tp ligands in these complexes is in the normal $\kappa^3$-bonding mode, except for [Sm(Tp$^{tBu,Me}$)$_2$],
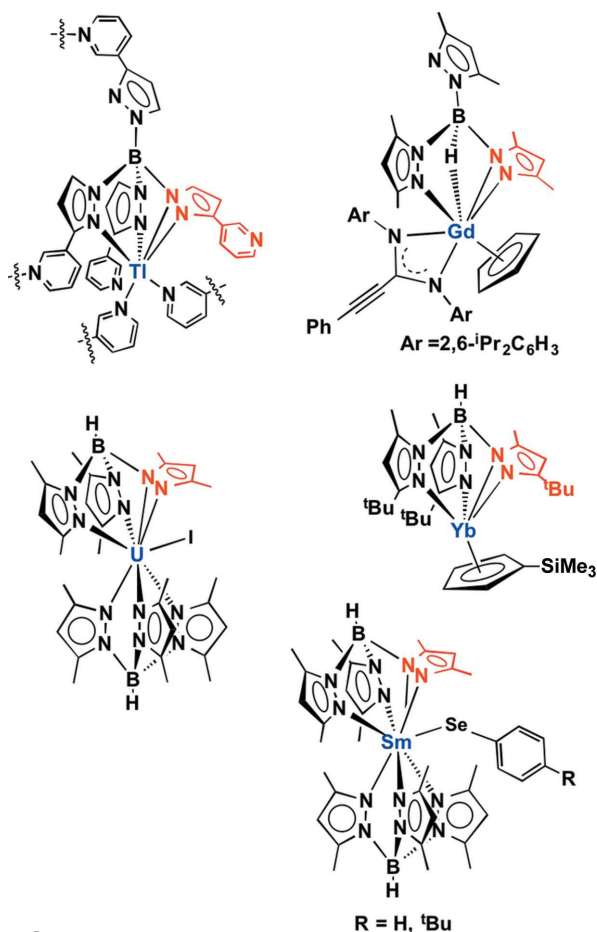
which has an agostic B—H$\cdots$Sm interaction as a result of the steric demand of the *tert*-butyl groups. Also, X-ray diffraction analysis has revealed that the structure of [Sm(Tp$^{iPr_2}$)$_2$] has a 'bent sandwich-like' geometry. The lack of the tetrakis-(pyrazolyl)borate congeners motivated us to prepare them. Although the divalent tetrakis(pyrazolyl)borate [B(pz)$_4$] complex [Sm{B(pz)$_4$}$_2$(thf)$_2$] (thf is tetrahydrofuran) has been prepared, the solid-state structure has not been reported (Domingos *et al.*, 1995). In this paper, we report the synthesis of [Sm{B(3-Mepz)$_4$}$_2$], (1), which was determined by X-ray diffraction. Interestingly, an unusual side-on interaction through the N═N group in one of the 3-methylpyrazolyl groups was found. To the best of our knowledge, six similar side-on coordination modes of pyrazolylborate, not including coordination to alkali metal ions, have been observed, *i.e.* in [Tl{B(3-Pypz)$_4$}] [3-Pypz is 3-(pyridin-3-yl)-1*H*-pyrazol-1-yl; Adams *et al.*, 2005], [Gd(Cp)(Tp$^{Me_2}$)(amidinato)] (Cp is cyclopentadienyl; Zhang *et al.*, 2014), [UI(Tp$^{Me_2}$)$_2$] (Sun *et al.*, 1994), [Yb(C$_5$H$_4$SiMe$_3$)(Tp$^{tBu,Me}$)] (Ferrence *et al.*, 2000), and [Sm(Tp$^{Me_2}$)(SePh)] and [Sm(Tp$^{Me_2}$)(SeC$_6$H$_4$$^t$Bu-4)] (Hillier *et al.*, 2000) (Fig. 2).



**(1)**

## 2. Experimental

### 2.1. General

The title divalent samarium complex is extremely air sensitive and, therefore, the preparation and subsequent manipulation were carried out in a glove-box under an argon atmosphere. Anhydrous tetrahydrofuran (THF) and ether were commercially available and used without further purification. A THF solution of SmI$_2$ (0.1 *M*) was purchased from Aldrich and used as received.

### 2.2. Synthesis and crystallization

Potassium tetrakis(3-methylpyrazolyl)borate (K[B(3-Mepz)$_4$]) was prepared according to a literature method with a slight modification (Niedenzu *et al.*, 1985). A mixture of potassium

**Figure 2**
Schematic illustration of the side-on coordination mode of pyrazolyl-borate.

**Table 1**
Experimental details.

Crystal data

| | |
|---|---|
| Chemical formula | $[Sm(C_{16}H_{20}BN_8)_2]$ |
| $M_r$ | 820.80 |
| Crystal system, space group | Monoclinic, $P2_1/c$ |
| Temperature (K) | 293 |
| $a, b, c$ (Å) | 11.2742 (10), 22.304 (3), 14.4624 (3) |
| $\beta$ (°) | 95.3065 (6) |
| $V$ (Å$^3$) | 3621.2 (6) |
| $Z$ | 4 |
| Radiation type | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 1.67 |
| Crystal size (mm) | $0.60 \times 0.50 \times 0.30$ |

Data collection

| | |
|---|---|
| Diffractometer | Rigaku Quantum |
| Absorption correction | Multi-scan (*REQAB*; Rigaku, 1998) |
| $T_{min}$, $T_{max}$ | 0.391, 0.605 |
| No. of measured, independent and observed [$F^2 > 2.0\sigma(F^2)$] reflections | 26604, 6237, 5131 |
| $R_{int}$ | 0.029 |
| $(\sin\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.595 |

Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.051, 0.116, 1.14 |
| No. of reflections | 6237 |
| No. of parameters | 468 |
| H-atom treatment | H-atom parameters constrained |
| | $w = 1/[\sigma^2(F_o^2) + (0.0391P)^2 + 12.5843P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 3.07, −1.41 |

Computer programs: *d\*TREK* (Rigaku, 2014), *SIR2004* (Burla *et al.*, 2005), *SHELXL2014* (Sheldrick, 2015) and *CrystalStructure* (Rigaku, 2016).

tetrahydroborate (KBH$_4$) and 3-methylpyrazole (3-MepzH) in a 1:5 molar ratio was heated to maintain gentle reflux of the excess 3-MepzH until evolution of H$_2$ was complete (for 10 h). After cooling to room temperature, the solid was crushed under benzene, filtered and washed with a small amount of benzene. The solid was extracted with methanol and dried. After washing with dichloromethane, the resulting solid was dried under vacuum at 373 K to remove unreacted 3-MepzH.

To a THF solution of SmI$_2$ (3.0 ml, 0.3 mmol) was added a THF (10 ml) suspension of K[B(3-Mepz)$_4$] (227 mg, 0.61 mmol). The mixture immediately turned from a dark-blue to a dark-green suspension. After stirring for 2 h, the mixture was filtered and the resulting white solid was washed with a small amount of THF. The dark-green filtrate and washings were dried to give [Sm{B(3-Mepz)$_4$}$_2$], (1), quantitatively. X-ray-quality single crystals were obtained from THF/diethyl ether.

### 2.3. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. Seven preliminary data frames were measured at 0.5° increments of $\omega$ in order to assess the crystal quality and preliminary unit-cell parameters. The intensity images were obtained with $\omega$ scans of 0.5° interval per frame for a duration of 35 s. The frame data were
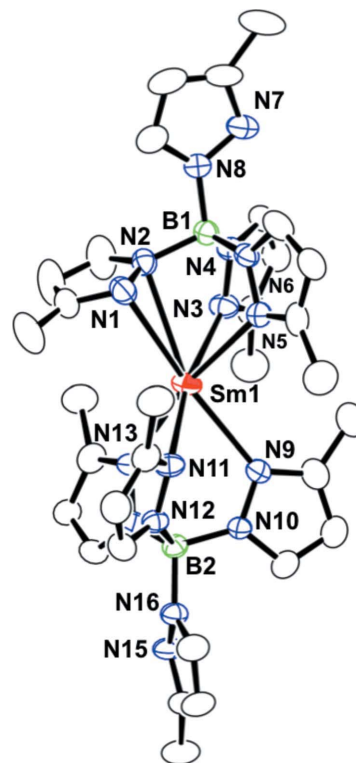


**Figure 3**
The molecular structure of [Sm{B(3-Mepz)$_4$}$_2$], (1), with displacement ellipsoids drawn at the 50% probability level. H atoms have been omitted for clarity.

integrated using the *d\*TREK* program package (Rigaku, 2014). H atoms were placed in calculated positions (0.96 and 0.93 Å), treated as riding and constrained with $U_{iso}$(H) = 1.2$U_{eq}$(parent). The high value of the maximum residual electron density may be accounted for by the heavy Sm atom or the data collection at room temperature.

### 3. Results and discussion

The title complex was prepared by the reaction of SmI$_2$ with potassium tetrakis(3-methylpyrazolyl)borate (K[B(3-Mepz)$_4$]). Addition of a THF suspension of two equivalents of K[B(3-Mepz)$_4$] to a THF solution of SmI$_2$ resulted in an immediate colour change from dark blue to dark green and the precipitation of KI. After work-up, [Sm{B(3-Mepz)$_4$}$_2$], (1), was obtained quantitatively. Use of Na[B(3-Mepz)$_4$] instead of K[B(3-Mepz)$_4$] failed to produce complex (1).

Complex (1) crystallized from a THF/ether mixture in the space group $P2_1/c$. There are no crystallization solvents in the lattice. The molecular structure of (1) is shown in Fig. 3, and selected bond lengths and angles are listed in Table 2. The complex consists of two [B(3-Mepz)$_4$] ligands without coordination of the crystallization solvents. On the other hand, in the 'parent' {B(pz)$_4$} ligand, *i.e.* [Sm{B(pz)$_4$}$_2$(thf)$_2$], containing two thf ligands has been reported. One of the [B(3-Mepz)$_4$] ligands in (1) is bonded to samarium through three N atoms (N9, N11 and N13), while the other ligand shows an unusual distortion. Two of the 3-methylpyrazolyl groups are aligned

**Table 2**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Sm1—N1 | 2.835 (5) | N13—N14 | 1.375 (6) |
| Sm1—N2 | 2.926 (4) | N15—N16 | 1.378 (6) |
| Sm1—N3 | 2.647 (4) | N2—B1 | 1.563 (7) |
| Sm1—N5 | 2.616 (4) | N4—B1 | 1.547 (7) |
| Sm1—N9 | 2.597 (4) | N6—B1 | 1.531 (7) |
| Sm1—N11 | 2.614 (4) | N8—B1 | 1.525 (7) |
| Sm1—N13 | 2.602 (4) | N10—B2 | 1.557 (7) |
| N1—N2 | 1.351 (6) | N12—B2 | 1.559 (7) |
| N3—N4 | 1.376 (6) | N14—B2 | 1.544 (7) |
| N5—N6 | 1.385 (6) | N16—B2 | 1.533 (6) |
| N7—N8 | 1.369 (6) | Sm1—B1 | 3.463 (6) |
| N9—N10 | 1.370 (6) | Sm1—B2 | 3.695 (6) |
| N11—N12 | 1.375 (6) | | |
| | | | |
| N2—N1—Sm1 | 80.3 (3) | N3—N4—B1 | 118.1 (4) |
| N4—N3—Sm1 | 117.0 (3) | N5—N6—B1 | 121.8 (4) |
| N6—N5—Sm1 | 113.8 (3) | N7—N8—B1 | 121.8 (4) |
| N10—N9—Sm1 | 120.2 (3) | N9—N10—B2 | 124.8 (4) |
| N12—N11—Sm1 | 119.6 (3) | N11—N12—B2 | 121.2 (4) |
| N14—N13—Sm1 | 123.9 (3) | N13—N14—B2 | 119.9 (4) |
| N1—N2—B1 | 124.8 (4) | N15—N16—B2 | 121.4 (4) |
| | | | |
| Sm1—N1—N2—B1 | −85.5 (4) | Sm1—N9—N10—B2 | 5.0 (6) |
| Sm1—N3—N4—B1 | −10.0 (5) | Sm1—N11—N12—B2 | 27.6 (5) |
| Sm1—N5—N6—B1 | −16.3 (5) | Sm1—N13—N14—B2 | −7.4 (6) |

approximately parallel to the B—Sm vector, while the third is twisted sideways such that the N—N bond is brought into side-on bonding interaction. The average Sm—N bond length for a normal pyrazolyl coordination is 2.615 Å, which is similar to those of [Sm(Tp$^{Me_2}$)$_2$]. The Sm1—N1 bond [2.835 (5) Å] is shorter than Sm1—N2 [2.926 (4) Å]. This tendency is observed in other complexes having a similar side-on coordination mode, except for [Tl{B(3-Pypz)$_4$}] and [Sm(Tp$^{Me_2}$)(Se-C$_6$H$_4^t$Bu-4)]. The B1—N2—N1—Sm1 torsion angle is 85.5 (4)°, which is comparable to the average value of the six compounds displaying a similar side-on coordination mode of pyrazolylborate (average 83.9°) (Table 3). The N1—N2 [1.351 (6) Å] and N2—B1 [1.563 (7) Å] bond lengths are not different from the other N—N and N—B bond lengths, respectively, except for N8—B1 [1.525 (7) Å] and N16—B2 [1.533 (6) Å]. The side-on interaction shortens the Sm1···B1 distance [3.463 (6) Å] compared to the other Sm1···B2 distance [3.695 (6) Å]. The former is the shortest distance among divalent hydridotris(pyrazolyl)borate samarium com-

plexes. The B1···Sm1···B2 angle is almost linear [176.54 (13)°], although the 'bent sandwich-like' divalent Sm complex [Sm(Tp$^{iPr_2}$)$_2$] [B1···Sm1···B2 = 151.19 (5)°] have been reported. In contrast to this side-on interaction, the pyrazolyl groups of [Sm(Tp$^{Me_2}$)$_2$] display the normal $\kappa^3$-bonding mode. This may be accounted for by the electron-withdrawing character of the uncoordinated 3-methyl-pyrazolyl group (Onishi *et al.*, 2001).

In summary, we succeeded in the preparation and structural characterization of a divalent samarium complex having two tetrakis(3-methylpyrazolyl)borate ligands. From the X-ray structure analysis, an unusual side-on interaction of the pyrazolylborate was revealed. This is the first structurally characterized example in divalent samarium complexes.

**Table 3**
B—N–N–Sm torsion angles (°) of the side-on coordination mode of pyrazolylborate.

3-Mepz is 3-methyl-1*H*-pyrazol-1-yl, 3-Pypz is 3-(pyridin-3-yl)-1*H*-pyrazol-1-yl, Tp$^{Me_2}$ is tris(3,5-dimethylpyrazol-1-yl)borate, Tp$^{tBu,Me}$ is tris(3-*tert*-butyl-5-methylpyrazol-1-yl)borate and Cp is cyclopentadienyl (see Fig. 2).

| Complex | B—N–N–Sm | Reference |
|---|---|---|
| [Sm{B(3-Mepz)$_4$}$_2$], (1) | 85.5 (4) | This work |
| [Tl{B(3-Pypz)$_4$}] | 95.6 | Adams *et al.* (2005) |
| [Gd(Cp)(Tp$^{Me_2}$)(amidinato)] | 70.0 | Zhang *et al.* (2014) |
| [UI(Tp$^{Me_2}$)$_2$] | 87.4 | Sun *et al.* (1994) |
| [Yb(C$_5$H$_4$SiMe$_3$)(Tp$^{tBu,Me}$)] | 75.3 | Ferrence *et al.* (2000) |
| [Sm(Tp$^{Me_2}$)(SePh)] | 83.7 | Hillier *et al.* (2000) |
| [Sm(Tp$^{Me_2}$)(SeC$_6$H$_4^t$Bu-4)] | 91.3 | Hillier *et al.* (2000) |

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# supporting information

## Side-on coordination mode of a pyrazolyl group in the structure of a divalent [Sm{B(3-Mepz)₄}₂] complex (3-Mepz is 3-methylpyrazol-1-yl)

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**Computing details**

Data collection: *D\*TREK* (Rigaku, 2014); cell refinement: *D\*TREK* (Rigaku, 2014); data reduction: *D\*TREK* (Rigaku, 2014); program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *CrystalStructure* (Rigaku, 2016); software used to prepare material for publication: *CrystalStructure* (Rigaku, 2016).

**Bis[tetrakis(3-methyl-1*H*-pyrazol-1-yl)borato]samarium(II)**

*Crystal data*

| | |
|---|---|
| $[Sm(C_{16}H_{20}BN_8)_2]$ | $F(000) = 1664.00$ |
| $M_r = 820.80$ | $D_x = 1.505$ Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å |
| $a = 11.2742$ (10) Å | Cell parameters from 17171 reflections |
| $b = 22.304$ (3) Å | $\theta = 3.0–27.6°$ |
| $c = 14.4624$ (3) Å | $\mu = 1.67$ mm$^{-1}$ |
| $\beta = 95.3065$ (6)° | $T = 293$ K |
| $V = 3621.2$ (6) Å$^3$ | Block, dark-green |
| $Z = 4$ | $0.60 \times 0.50 \times 0.30$ mm |

*Data collection*

| | |
|---|---|
| Rigaku Quantum diffractometer | 6237 independent reflections |
| Detector resolution: 10.000 pixels mm$^{-1}$ | 5131 reflections with $F^2 > 2.0\sigma(F^2)$ |
| $\omega$ scans | $R_{int} = 0.029$ |
| Absorption correction: multi-scan (REQAB; Rigaku, 1998) | $\theta_{max} = 25.0°$, $\theta_{min} = 3.0°$ |
| $T_{min} = 0.391$, $T_{max} = 0.605$ | $h = -13 \rightarrow 13$ |
| 26604 measured reflections | $k = -25 \rightarrow 26$ |
| | $l = -17 \rightarrow 17$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.051$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.116$ | H-atom parameters constrained |
| $S = 1.14$ | $w = 1/[\sigma^2(F_o^2) + (0.0391P)^2 + 12.5843P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| 6237 reflections | |
| 468 parameters | |
| 0 restraints | $(\Delta/\sigma)_{max} < 0.001$ |
| Primary atom site location: structure-invariant direct methods | $\Delta\rho_{max} = 3.07$ e Å$^{-3}$ |
| | $\Delta\rho_{min} = -1.41$ 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 was performed using all reflections. The weighted R-factor (wR) and goodness of fit (S) are based on $F^2$. R-factor (gt) are based on F. The threshold expression of $F^2 > 2.0$ sigma($F^2$) is used only for calculating R-factor (gt).

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

|  | $x$ | $y$ | $z$ | $U_{iso}*/U_{eq}$ |
|---|---|---|---|---|
| Sm1 | 0.74099 (2) | −0.00402 (2) | 0.26183 (2) | 0.03783 (12) |
| N1 | 0.6111 (4) | −0.0883 (2) | 0.1460 (3) | 0.0429 (11) |
| N2 | 0.7113 (4) | −0.12031 (19) | 0.1689 (3) | 0.0363 (9) |
| N3 | 0.9218 (4) | −0.08012 (19) | 0.2706 (3) | 0.0391 (10) |
| N4 | 0.9317 (4) | −0.11596 (18) | 0.1943 (3) | 0.0335 (9) |
| N5 | 0.8174 (4) | −0.00323 (18) | 0.0965 (3) | 0.0386 (10) |
| N6 | 0.8268 (3) | −0.05977 (18) | 0.0582 (3) | 0.0323 (9) |
| N7 | 0.9275 (4) | −0.1858 (2) | 0.0100 (3) | 0.0487 (12) |
| N8 | 0.8294 (4) | −0.17157 (18) | 0.0547 (3) | 0.0387 (10) |
| N9 | 0.8460 (4) | 0.09306 (19) | 0.3259 (3) | 0.0398 (10) |
| N10 | 0.7864 (4) | 0.13287 (17) | 0.3770 (3) | 0.0318 (9) |
| N11 | 0.5904 (4) | 0.08526 (18) | 0.2444 (3) | 0.0367 (10) |
| N12 | 0.5660 (4) | 0.11727 (17) | 0.3217 (3) | 0.0316 (9) |
| N13 | 0.6910 (4) | 0.01137 (18) | 0.4323 (3) | 0.0374 (9) |
| N14 | 0.6561 (4) | 0.06526 (18) | 0.4672 (3) | 0.0329 (9) |
| N15 | 0.6637 (4) | 0.18440 (19) | 0.5557 (3) | 0.0415 (10) |
| N16 | 0.6282 (4) | 0.17856 (17) | 0.4624 (3) | 0.0345 (9) |
| C1 | 0.4060 (6) | −0.0852 (4) | 0.1895 (6) | 0.080 (2) |
| H1A | 0.3887 | −0.0682 | 0.1288 | 0.096* |
| H1B | 0.3984 | −0.0548 | 0.2356 | 0.096* |
| H1C | 0.3510 | −0.1171 | 0.1981 | 0.096* |
| C2 | 0.5294 (5) | −0.1091 (3) | 0.1987 (4) | 0.0448 (13) |
| C3 | 0.5764 (6) | −0.1550 (4) | 0.2550 (5) | 0.071 (2) |
| H3 | 0.5378 | −0.1773 | 0.2974 | 0.086* |
| C4 | 0.6914 (6) | −0.1603 (3) | 0.2349 (5) | 0.0650 (19) |
| H4 | 0.7469 | −0.1872 | 0.2624 | 0.078* |
| C5 | 1.0410 (6) | −0.0540 (3) | 0.4146 (4) | 0.0624 (17) |
| H5A | 1.0371 | −0.0829 | 0.4634 | 0.075* |
| H5B | 0.9788 | −0.0249 | 0.4181 | 0.075* |
| H5C | 1.1170 | −0.0343 | 0.4213 | 0.075* |
| C6 | 1.0255 (5) | −0.0847 (2) | 0.3237 (4) | 0.0413 (12) |
| C7 | 1.1016 (5) | −0.1229 (3) | 0.2814 (4) | 0.0448 (13) |
| H7 | 1.1787 | −0.1337 | 0.3038 | 0.054* |
| C8 | 1.0410 (5) | −0.1414 (2) | 0.2008 (4) | 0.0399 (12) |
| H8 | 1.0698 | −0.1672 | 0.1575 | 0.048* |
| C9 | 0.8216 (7) | 0.1009 (3) | 0.0477 (5) | 0.0656 (18) |

| | | | |
|---|---|---|---|
| H9A | 0.8248 | 0.1083 | 0.1133 | 0.079* |
| H9B | 0.7484 | 0.1166 | 0.0180 | 0.079* |
| H9C | 0.8877 | 0.1202 | 0.0228 | 0.079* |
| C10 | 0.8273 (5) | 0.0351 (2) | 0.0307 (4) | 0.0417 (12) |
| C11 | 0.8451 (5) | 0.0062 (3) | −0.0526 (4) | 0.0444 (13) |
| H11 | 0.8563 | 0.0237 | −0.1096 | 0.053* |
| C12 | 0.8426 (5) | −0.0530 (3) | −0.0319 (4) | 0.0468 (14) |
| H12 | 0.8506 | −0.0841 | −0.0737 | 0.056* |
| C13 | 0.9776 (8) | −0.2540 (3) | −0.1129 (5) | 0.078 (2) |
| H13A | 1.0573 | −0.2425 | −0.0908 | 0.094* |
| H13B | 0.9594 | −0.2394 | −0.1750 | 0.094* |
| H13C | 0.9713 | −0.2969 | −0.1126 | 0.094* |
| C14 | 0.8923 (7) | −0.2280 (2) | −0.0513 (4) | 0.0541 (16) |
| C15 | 0.7739 (7) | −0.2410 (3) | −0.0458 (5) | 0.070 (2) |
| H15 | 0.7286 | −0.2693 | −0.0808 | 0.084* |
| C16 | 0.7356 (6) | −0.2043 (3) | 0.0213 (5) | 0.0599 (17) |
| H16 | 0.6590 | −0.2025 | 0.0401 | 0.072* |
| C17 | 1.0420 (6) | 0.0855 (3) | 0.2649 (5) | 0.0682 (19) |
| H17A | 1.0031 | 0.0552 | 0.2260 | 0.082* |
| H17B | 1.1015 | 0.0673 | 0.3077 | 0.082* |
| H17C | 1.0791 | 0.1141 | 0.2271 | 0.082* |
| C18 | 0.9522 (5) | 0.1167 (3) | 0.3177 (4) | 0.0454 (13) |
| C19 | 0.9640 (5) | 0.1716 (3) | 0.3624 (5) | 0.0548 (15) |
| H19 | 1.0295 | 0.1971 | 0.3663 | 0.066* |
| C20 | 0.8582 (5) | 0.1800 (2) | 0.3995 (4) | 0.0461 (13) |
| H20 | 0.8388 | 0.2129 | 0.4346 | 0.055* |
| C21 | 0.4913 (6) | 0.0609 (3) | 0.0904 (4) | 0.0535 (15) |
| H21A | 0.5589 | 0.0349 | 0.0884 | 0.064* |
| H21B | 0.4942 | 0.0917 | 0.0442 | 0.064* |
| H21C | 0.4194 | 0.0380 | 0.0781 | 0.064* |
| C22 | 0.4934 (5) | 0.0888 (2) | 0.1840 (4) | 0.0382 (12) |
| C23 | 0.4054 (5) | 0.1223 (3) | 0.2215 (4) | 0.0460 (13) |
| H23 | 0.3300 | 0.1316 | 0.1936 | 0.055* |
| C24 | 0.4540 (5) | 0.1389 (2) | 0.3087 (4) | 0.0418 (12) |
| H24 | 0.4159 | 0.1612 | 0.3515 | 0.050* |
| C25 | 0.7131 (7) | −0.0931 (3) | 0.4839 (5) | 0.0618 (18) |
| H25A | 0.7355 | −0.0978 | 0.4218 | 0.074* |
| H25B | 0.6460 | −0.1184 | 0.4923 | 0.074* |
| H25C | 0.7787 | −0.1041 | 0.5276 | 0.074* |
| C26 | 0.6805 (5) | −0.0296 (2) | 0.4991 (4) | 0.0432 (13) |
| C27 | 0.6376 (5) | −0.0028 (2) | 0.5757 (3) | 0.0400 (12) |
| H27 | 0.6221 | −0.0213 | 0.6310 | 0.048* |
| C28 | 0.6228 (5) | 0.0560 (2) | 0.5533 (3) | 0.0395 (12) |
| H28 | 0.5942 | 0.0853 | 0.5913 | 0.047* |
| C29 | 0.6756 (7) | 0.2636 (3) | 0.6737 (4) | 0.067 (2) |
| H29A | 0.7507 | 0.2842 | 0.6781 | 0.080* |
| H29B | 0.6803 | 0.2301 | 0.7155 | 0.080* |
| H29C | 0.6144 | 0.2905 | 0.6898 | 0.080* |

| | | | |
|---|---|---|---|
| C30 | 0.6467 (5) | 0.2417 (2) | 0.5765 (4) | 0.0437 (13) |
| C31 | 0.6026 (6) | 0.2735 (2) | 0.4978 (4) | 0.0514 (15) |
| H31 | 0.5837 | 0.3141 | 0.4940 | 0.062* |
| C32 | 0.5930 (5) | 0.2328 (2) | 0.4280 (4) | 0.0455 (13) |
| H32 | 0.5666 | 0.2408 | 0.3664 | 0.055* |
| B1 | 0.8269 (5) | −0.1167 (3) | 0.1173 (4) | 0.0330 (12) |
| B2 | 0.6593 (5) | 0.1227 (2) | 0.4078 (4) | 0.0318 (12) |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Sm1 | 0.04262 (17) | 0.03988 (17) | 0.03085 (17) | 0.00649 (12) | 0.00262 (11) | −0.01103 (11) |
| N1 | 0.040 (2) | 0.044 (2) | 0.046 (3) | 0.004 (2) | 0.013 (2) | 0.001 (2) |
| N2 | 0.035 (2) | 0.042 (2) | 0.032 (2) | −0.0026 (18) | 0.0047 (19) | 0.0006 (17) |
| N3 | 0.036 (2) | 0.043 (2) | 0.037 (2) | 0.0040 (18) | −0.004 (2) | −0.0096 (18) |
| N4 | 0.035 (2) | 0.035 (2) | 0.030 (2) | 0.0036 (17) | −0.0009 (18) | −0.0041 (16) |
| N5 | 0.044 (2) | 0.035 (2) | 0.039 (2) | −0.0049 (19) | 0.0118 (19) | −0.0022 (18) |
| N6 | 0.029 (2) | 0.039 (2) | 0.029 (2) | 0.0004 (17) | 0.0032 (17) | −0.0061 (17) |
| N7 | 0.054 (3) | 0.045 (3) | 0.048 (3) | 0.003 (2) | 0.010 (2) | −0.015 (2) |
| N8 | 0.041 (2) | 0.035 (2) | 0.041 (2) | −0.0012 (18) | 0.007 (2) | −0.0062 (18) |
| N9 | 0.038 (2) | 0.043 (2) | 0.040 (2) | −0.0053 (19) | 0.013 (2) | −0.0119 (19) |
| N10 | 0.036 (2) | 0.031 (2) | 0.028 (2) | −0.0027 (17) | 0.0060 (18) | −0.0052 (16) |
| N11 | 0.043 (2) | 0.035 (2) | 0.031 (2) | 0.0056 (18) | −0.004 (2) | −0.0045 (17) |
| N12 | 0.039 (2) | 0.030 (2) | 0.025 (2) | 0.0017 (17) | 0.0014 (18) | −0.0010 (15) |
| N13 | 0.046 (2) | 0.033 (2) | 0.034 (2) | 0.0053 (18) | 0.0051 (19) | −0.0066 (17) |
| N14 | 0.035 (2) | 0.034 (2) | 0.030 (2) | −0.0001 (17) | 0.0036 (18) | −0.0014 (16) |
| N15 | 0.060 (3) | 0.035 (2) | 0.030 (2) | 0.003 (2) | 0.005 (2) | −0.0088 (17) |
| N16 | 0.046 (2) | 0.030 (2) | 0.028 (2) | 0.0023 (18) | 0.0055 (19) | −0.0014 (16) |
| C1 | 0.051 (4) | 0.107 (6) | 0.088 (6) | 0.002 (4) | 0.032 (4) | 0.001 (5) |
| C2 | 0.040 (3) | 0.061 (4) | 0.034 (3) | −0.012 (3) | 0.010 (2) | −0.008 (2) |
| C3 | 0.053 (4) | 0.102 (6) | 0.060 (4) | −0.011 (4) | 0.014 (3) | 0.031 (4) |
| C4 | 0.050 (4) | 0.080 (5) | 0.065 (4) | 0.005 (3) | 0.007 (3) | 0.037 (4) |
| C5 | 0.059 (4) | 0.079 (5) | 0.046 (4) | 0.001 (3) | −0.011 (3) | −0.011 (3) |
| C6 | 0.041 (3) | 0.042 (3) | 0.039 (3) | −0.004 (2) | −0.004 (2) | 0.002 (2) |
| C7 | 0.033 (3) | 0.054 (3) | 0.046 (3) | 0.004 (2) | −0.002 (3) | 0.006 (3) |
| C8 | 0.037 (3) | 0.041 (3) | 0.043 (3) | 0.007 (2) | 0.009 (2) | −0.001 (2) |
| C9 | 0.078 (5) | 0.049 (4) | 0.072 (5) | 0.002 (3) | 0.014 (4) | 0.008 (3) |
| C10 | 0.033 (3) | 0.045 (3) | 0.047 (3) | −0.001 (2) | 0.006 (2) | 0.017 (2) |
| C11 | 0.045 (3) | 0.055 (3) | 0.035 (3) | 0.006 (3) | 0.009 (2) | 0.011 (2) |
| C12 | 0.047 (3) | 0.066 (4) | 0.028 (3) | 0.006 (3) | 0.007 (3) | −0.002 (2) |
| C13 | 0.106 (6) | 0.067 (5) | 0.063 (5) | 0.028 (4) | 0.017 (4) | −0.024 (4) |
| C14 | 0.085 (5) | 0.036 (3) | 0.041 (3) | 0.011 (3) | 0.010 (3) | −0.010 (2) |
| C15 | 0.083 (5) | 0.054 (4) | 0.072 (5) | −0.012 (4) | −0.001 (4) | −0.033 (3) |
| C16 | 0.056 (4) | 0.050 (3) | 0.073 (5) | −0.012 (3) | 0.009 (3) | −0.021 (3) |
| C17 | 0.053 (4) | 0.078 (5) | 0.077 (5) | −0.010 (3) | 0.022 (4) | −0.015 (4) |
| C18 | 0.041 (3) | 0.061 (4) | 0.034 (3) | −0.004 (3) | 0.004 (2) | −0.002 (2) |
| C19 | 0.047 (3) | 0.057 (4) | 0.061 (4) | −0.017 (3) | 0.009 (3) | −0.010 (3) |
| C20 | 0.047 (3) | 0.043 (3) | 0.048 (3) | −0.011 (2) | 0.004 (3) | −0.009 (2) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C21 | 0.066 (4) | 0.056 (4) | 0.035 (3) | 0.002 (3) | −0.011 (3) | −0.004 (2) |
| C22 | 0.040 (3) | 0.041 (3) | 0.031 (3) | −0.002 (2) | −0.009 (2) | 0.004 (2) |
| C23 | 0.034 (3) | 0.059 (3) | 0.043 (3) | 0.001 (2) | −0.011 (2) | 0.003 (3) |
| C24 | 0.036 (3) | 0.050 (3) | 0.039 (3) | 0.003 (2) | 0.002 (2) | −0.003 (2) |
| C25 | 0.089 (5) | 0.035 (3) | 0.064 (4) | 0.008 (3) | 0.021 (4) | 0.003 (3) |
| C26 | 0.049 (3) | 0.037 (3) | 0.044 (3) | 0.000 (2) | 0.000 (3) | 0.009 (2) |
| C27 | 0.058 (3) | 0.034 (3) | 0.029 (3) | −0.005 (2) | 0.008 (2) | 0.002 (2) |
| C28 | 0.057 (3) | 0.039 (3) | 0.025 (2) | 0.002 (2) | 0.012 (2) | −0.001 (2) |
| C29 | 0.106 (6) | 0.051 (4) | 0.046 (4) | −0.007 (4) | 0.021 (4) | −0.021 (3) |
| C30 | 0.057 (3) | 0.035 (3) | 0.041 (3) | −0.004 (2) | 0.015 (3) | −0.011 (2) |
| C31 | 0.064 (4) | 0.028 (3) | 0.063 (4) | 0.005 (2) | 0.008 (3) | −0.007 (2) |
| C32 | 0.057 (3) | 0.035 (3) | 0.045 (3) | 0.007 (2) | 0.002 (3) | 0.002 (2) |
| B1 | 0.028 (3) | 0.037 (3) | 0.034 (3) | 0.001 (2) | 0.001 (2) | −0.003 (2) |
| B2 | 0.034 (3) | 0.034 (3) | 0.027 (3) | −0.002 (2) | −0.001 (2) | −0.004 (2) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Sm1—N1 | 2.835 (5) | C6—C7 | 1.392 (8) |
| Sm1—N2 | 2.926 (4) | C7—C8 | 1.360 (8) |
| Sm1—N3 | 2.647 (4) | C7—H7 | 0.9300 |
| Sm1—N5 | 2.616 (4) | C8—H8 | 0.9300 |
| Sm1—N9 | 2.597 (4) | C9—C10 | 1.490 (8) |
| Sm1—N11 | 2.614 (4) | C9—H9A | 0.9600 |
| Sm1—N13 | 2.602 (4) | C9—H9B | 0.9600 |
| N1—N2 | 1.351 (6) | C9—H9C | 0.9600 |
| N3—N4 | 1.376 (6) | C10—C11 | 1.398 (8) |
| N5—N6 | 1.385 (6) | C11—C12 | 1.356 (8) |
| N7—N8 | 1.369 (6) | C11—H11 | 0.9300 |
| N9—N10 | 1.370 (6) | C12—H12 | 0.9300 |
| N11—N12 | 1.375 (6) | C13—C14 | 1.489 (9) |
| N13—N14 | 1.375 (6) | C13—H13A | 0.9600 |
| N15—N16 | 1.378 (6) | C13—H13B | 0.9600 |
| N2—B1 | 1.563 (7) | C13—H13C | 0.9600 |
| N4—B1 | 1.547 (7) | C14—C15 | 1.376 (10) |
| N6—B1 | 1.531 (7) | C15—C16 | 1.370 (9) |
| N8—B1 | 1.525 (7) | C15—H15 | 0.9300 |
| N10—B2 | 1.557 (7) | C16—H16 | 0.9300 |
| N12—B2 | 1.559 (7) | C17—C18 | 1.495 (9) |
| N14—B2 | 1.544 (7) | C17—H17A | 0.9600 |
| N16—B2 | 1.533 (6) | C17—H17B | 0.9600 |
| Sm1—B1 | 3.463 (6) | C17—H17C | 0.9600 |
| Sm1—B2 | 3.695 (6) | C18—C19 | 1.386 (8) |
| N1—C2 | 1.332 (7) | C19—C20 | 1.365 (8) |
| N2—C4 | 1.341 (7) | C19—H19 | 0.9300 |
| N3—C6 | 1.342 (7) | C20—H20 | 0.9300 |
| N4—C8 | 1.352 (6) | C21—C22 | 1.489 (7) |
| N5—C10 | 1.291 (6) | C21—H21A | 0.9600 |
| N6—C12 | 1.339 (7) | C21—H21B | 0.9600 |

| | | | |
|---|---|---|---|
| N7—C14 | 1.328 (7) | C21—H21C | 0.9600 |
| N8—C16 | 1.338 (7) | C22—C23 | 1.391 (8) |
| N9—C18 | 1.324 (7) | C23—C24 | 1.378 (8) |
| N10—C20 | 1.349 (6) | C23—H23 | 0.9300 |
| N11—C22 | 1.338 (6) | C24—H24 | 0.9300 |
| N12—C24 | 1.348 (7) | C25—C26 | 1.484 (8) |
| N13—C26 | 1.343 (7) | C25—H25A | 0.9600 |
| N14—C28 | 1.350 (6) | C25—H25B | 0.9600 |
| N15—C30 | 1.331 (7) | C25—H25C | 0.9600 |
| N16—C32 | 1.353 (7) | C26—C27 | 1.386 (8) |
| C1—C2 | 1.484 (9) | C27—C28 | 1.359 (7) |
| C1—H1A | 0.9600 | C27—H27 | 0.9300 |
| C1—H1B | 0.9600 | C28—H28 | 0.9300 |
| C1—H1C | 0.9600 | C29—C30 | 1.495 (8) |
| C2—C3 | 1.382 (9) | C29—H29A | 0.9600 |
| C3—C4 | 1.359 (9) | C29—H29B | 0.9600 |
| C3—H3 | 0.9300 | C29—H29C | 0.9600 |
| C4—H4 | 0.9300 | C30—C31 | 1.393 (8) |
| C5—C6 | 1.478 (8) | C31—C32 | 1.355 (8) |
| C5—H5A | 0.9600 | C31—H31 | 0.9300 |
| C5—H5B | 0.9600 | C32—H32 | 0.9300 |
| C5—H5C | 0.9600 | | |
| | | | |
| N2—N1—Sm1 | 80.3 (3) | H9A—C9—H9B | 109.5 |
| N4—N3—Sm1 | 117.0 (3) | C10—C9—H9C | 109.5 |
| N6—N5—Sm1 | 113.8 (3) | H9A—C9—H9C | 109.5 |
| N10—N9—Sm1 | 120.2 (3) | H9B—C9—H9C | 109.5 |
| N12—N11—Sm1 | 119.6 (3) | N5—C10—C11 | 111.0 (5) |
| N14—N13—Sm1 | 123.9 (3) | N5—C10—C9 | 121.5 (6) |
| N1—N2—B1 | 124.8 (4) | C11—C10—C9 | 127.4 (5) |
| N3—N4—B1 | 118.1 (4) | C12—C11—C10 | 104.5 (5) |
| N5—N6—B1 | 121.8 (4) | C12—C11—H11 | 127.7 |
| N7—N8—B1 | 121.8 (4) | C10—C11—H11 | 127.7 |
| N9—N10—B2 | 124.8 (4) | N6—C12—C11 | 109.4 (5) |
| N11—N12—B2 | 121.2 (4) | N6—C12—H12 | 125.3 |
| N13—N14—B2 | 119.9 (4) | C11—C12—H12 | 125.3 |
| N15—N16—B2 | 121.4 (4) | C14—C13—H13A | 109.5 |
| N9—Sm1—N13 | 71.54 (13) | C14—C13—H13B | 109.5 |
| N9—Sm1—N11 | 70.72 (14) | H13A—C13—H13B | 109.5 |
| N13—Sm1—N11 | 78.18 (13) | C14—C13—H13C | 109.5 |
| N9—Sm1—N5 | 98.22 (13) | H13A—C13—H13C | 109.5 |
| N13—Sm1—N5 | 169.68 (13) | H13B—C13—H13C | 109.5 |
| N11—Sm1—N5 | 99.95 (13) | N7—C14—C15 | 110.1 (5) |
| N9—Sm1—N3 | 101.19 (13) | N7—C14—C13 | 120.4 (7) |
| N13—Sm1—N3 | 105.80 (13) | C15—C14—C13 | 129.5 (6) |
| N11—Sm1—N3 | 169.66 (14) | C16—C15—C14 | 106.6 (6) |
| N5—Sm1—N3 | 74.39 (14) | C16—C15—H15 | 126.7 |
| N9—Sm1—N1 | 163.25 (14) | C14—C15—H15 | 126.7 |

| | | | |
|---|---|---|---|
| N13—Sm1—N1 | 120.15 (13) | N8—C16—C15 | 106.9 (6) |
| N11—Sm1—N1 | 98.81 (13) | N8—C16—H16 | 126.5 |
| N5—Sm1—N1 | 70.12 (13) | C15—C16—H16 | 126.5 |
| N3—Sm1—N1 | 87.49 (13) | C18—C17—H17A | 109.5 |
| N9—Sm1—N2 | 158.73 (12) | C18—C17—H17B | 109.5 |
| N13—Sm1—N2 | 121.77 (12) | H17A—C17—H17B | 109.5 |
| N11—Sm1—N2 | 125.83 (12) | C18—C17—H17C | 109.5 |
| N5—Sm1—N2 | 67.63 (12) | H17A—C17—H17C | 109.5 |
| N3—Sm1—N2 | 60.55 (12) | H17B—C17—H17C | 109.5 |
| N1—Sm1—N2 | 27.07 (12) | N9—C18—C19 | 111.0 (5) |
| C2—N1—N2 | 106.4 (5) | N9—C18—C17 | 121.4 (5) |
| C2—N1—Sm1 | 103.8 (3) | C19—C18—C17 | 127.5 (6) |
| C4—N2—N1 | 109.6 (5) | C20—C19—C18 | 104.8 (5) |
| C4—N2—B1 | 125.2 (5) | C20—C19—H19 | 127.6 |
| C4—N2—Sm1 | 106.4 (4) | C18—C19—H19 | 127.6 |
| N1—N2—Sm1 | 72.7 (3) | N10—C20—C19 | 109.0 (5) |
| B1—N2—Sm1 | 96.2 (3) | N10—C20—H20 | 125.5 |
| C6—N3—N4 | 106.5 (4) | C19—C20—H20 | 125.5 |
| C6—N3—Sm1 | 134.6 (3) | C22—C21—H21A | 109.5 |
| C8—N4—N3 | 109.2 (4) | C22—C21—H21B | 109.5 |
| C8—N4—B1 | 132.5 (4) | H21A—C21—H21B | 109.5 |
| C10—N5—N6 | 107.1 (4) | C22—C21—H21C | 109.5 |
| C10—N5—Sm1 | 137.4 (4) | H21A—C21—H21C | 109.5 |
| C12—N6—N5 | 108.0 (4) | H21B—C21—H21C | 109.5 |
| C12—N6—B1 | 130.0 (4) | N11—C22—C23 | 110.3 (5) |
| C14—N7—N8 | 105.9 (5) | N11—C22—C21 | 120.9 (5) |
| C16—N8—N7 | 110.5 (5) | C23—C22—C21 | 128.9 (5) |
| C16—N8—B1 | 126.5 (5) | C24—C23—C22 | 105.3 (5) |
| C18—N9—N10 | 106.4 (4) | C24—C23—H23 | 127.4 |
| C18—N9—Sm1 | 133.3 (4) | C22—C23—H23 | 127.4 |
| C20—N10—N9 | 108.8 (4) | N12—C24—C23 | 108.6 (5) |
| C20—N10—B2 | 126.4 (4) | N12—C24—H24 | 125.7 |
| C22—N11—N12 | 106.7 (4) | C23—C24—H24 | 125.7 |
| C22—N11—Sm1 | 126.4 (3) | C26—C25—H25A | 109.5 |
| C24—N12—N11 | 109.1 (4) | C26—C25—H25B | 109.5 |
| C24—N12—B2 | 129.6 (4) | H25A—C25—H25B | 109.5 |
| C26—N13—N14 | 106.5 (4) | C26—C25—H25C | 109.5 |
| C26—N13—Sm1 | 129.2 (3) | H25A—C25—H25C | 109.5 |
| C28—N14—N13 | 108.6 (4) | H25B—C25—H25C | 109.5 |
| C28—N14—B2 | 131.5 (4) | N13—C26—C27 | 110.0 (5) |
| C30—N15—N16 | 106.1 (4) | N13—C26—C25 | 120.2 (5) |
| C32—N16—N15 | 109.0 (4) | C27—C26—C25 | 129.8 (5) |
| C32—N16—B2 | 127.6 (4) | C28—C27—C26 | 105.7 (4) |
| C2—C1—H1A | 109.5 | C28—C27—H27 | 127.1 |
| C2—C1—H1B | 109.5 | C26—C27—H27 | 127.1 |
| H1A—C1—H1B | 109.5 | N14—C28—C27 | 109.2 (4) |
| C2—C1—H1C | 109.5 | N14—C28—H28 | 125.4 |
| H1A—C1—H1C | 109.5 | C27—C28—H28 | 125.4 |

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| H1B—C1—H1C | 109.5 | C30—C29—H29A | 109.5 |
| N1—C2—C3 | 110.3 (5) | C30—C29—H29B | 109.5 |
| N1—C2—C1 | 121.1 (6) | H29A—C29—H29B | 109.5 |
| C3—C2—C1 | 128.6 (6) | C30—C29—H29C | 109.5 |
| C4—C3—C2 | 105.1 (6) | H29A—C29—H29C | 109.5 |
| C4—C3—H3 | 127.4 | H29B—C29—H29C | 109.5 |
| C2—C3—H3 | 127.4 | N15—C30—C31 | 110.7 (5) |
| N2—C4—C3 | 108.6 (6) | N15—C30—C29 | 120.1 (5) |
| N2—C4—H4 | 125.7 | C31—C30—C29 | 129.2 (5) |
| C3—C4—H4 | 125.7 | C32—C31—C30 | 105.3 (5) |
| C6—C5—H5A | 109.5 | C32—C31—H31 | 127.3 |
| C6—C5—H5B | 109.5 | C30—C31—H31 | 127.3 |
| H5A—C5—H5B | 109.5 | N16—C32—C31 | 109.0 (5) |
| C6—C5—H5C | 109.5 | N16—C32—H32 | 125.5 |
| H5A—C5—H5C | 109.5 | C31—C32—H32 | 125.5 |
| H5B—C5—H5C | 109.5 | N8—B1—N6 | 109.4 (4) |
| N3—C6—C7 | 109.5 (5) | N8—B1—N4 | 112.4 (4) |
| N3—C6—C5 | 120.0 (5) | N6—B1—N4 | 110.7 (4) |
| C7—C6—C5 | 130.4 (5) | N8—B1—N2 | 107.7 (4) |
| C8—C7—C6 | 106.3 (5) | N6—B1—N2 | 110.6 (4) |
| C8—C7—H7 | 126.8 | N4—B1—N2 | 105.8 (4) |
| C6—C7—H7 | 126.8 | N16—B2—N14 | 111.7 (4) |
| N4—C8—C7 | 108.4 (5) | N16—B2—N10 | 106.8 (4) |
| N4—C8—H8 | 125.8 | N14—B2—N10 | 110.5 (4) |
| C7—C8—H8 | 125.8 | N16—B2—N12 | 107.8 (4) |
| C10—C9—H9A | 109.5 | N14—B2—N12 | 109.4 (4) |
| C10—C9—H9B | 109.5 | N10—B2—N12 | 110.7 (4) |
| | | | |
| C2—N1—N2—C4 | 0.1 (6) | N12—N11—C22—C21 | 177.5 (5) |
| Sm1—N1—N2—C4 | 101.7 (5) | Sm1—N11—C22—C21 | −33.0 (7) |
| C2—N1—N2—B1 | 172.8 (5) | N11—C22—C23—C24 | −0.2 (6) |
| Sm1—N1—N2—B1 | −85.5 (4) | C21—C22—C23—C24 | −178.3 (6) |
| C2—N1—N2—Sm1 | −101.7 (4) | N11—N12—C24—C23 | −1.5 (6) |
| C6—N3—N4—C8 | −0.7 (6) | B2—N12—C24—C23 | −179.5 (5) |
| Sm1—N3—N4—C8 | 165.9 (3) | C22—C23—C24—N12 | 1.1 (6) |
| C6—N3—N4—B1 | −176.5 (4) | N14—N13—C26—C27 | 0.9 (6) |
| Sm1—N3—N4—B1 | −10.0 (5) | Sm1—N13—C26—C27 | −172.1 (4) |
| C10—N5—N6—C12 | 0.1 (6) | N14—N13—C26—C25 | −179.2 (5) |
| Sm1—N5—N6—C12 | 167.6 (3) | Sm1—N13—C26—C25 | 7.8 (8) |
| C10—N5—N6—B1 | 176.2 (4) | N13—C26—C27—C28 | −0.2 (7) |
| Sm1—N5—N6—B1 | −16.3 (5) | C25—C26—C27—C28 | 179.9 (6) |
| C14—N7—N8—C16 | −0.2 (7) | N13—N14—C28—C27 | 1.1 (6) |
| C14—N7—N8—B1 | −168.2 (5) | B2—N14—C28—C27 | −179.3 (5) |
| C18—N9—N10—C20 | −0.4 (6) | C26—C27—C28—N14 | −0.6 (7) |
| Sm1—N9—N10—C20 | −178.1 (3) | N16—N15—C30—C31 | −1.1 (7) |
| C18—N9—N10—B2 | −177.3 (5) | N16—N15—C30—C29 | 179.5 (5) |
| Sm1—N9—N10—B2 | 5.0 (6) | N15—C30—C31—C32 | 0.3 (7) |
| C22—N11—N12—C24 | 1.4 (5) | C29—C30—C31—C32 | 179.7 (6) |

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| Sm1—N11—N12—C24 | −150.6 (3) | N15—N16—C32—C31 | −1.4 (7) |
| C22—N11—N12—B2 | 179.6 (4) | B2—N16—C32—C31 | −165.2 (5) |
| Sm1—N11—N12—B2 | 27.6 (5) | C30—C31—C32—N16 | 0.7 (7) |
| C26—N13—N14—C28 | −1.2 (6) | C16—N8—B1—N6 | −97.1 (6) |
| Sm1—N13—N14—C28 | 172.2 (3) | N7—N8—B1—N6 | 68.9 (6) |
| C26—N13—N14—B2 | 179.1 (4) | C16—N8—B1—N4 | 139.4 (6) |
| Sm1—N13—N14—B2 | −7.4 (6) | N7—N8—B1—N4 | −54.6 (6) |
| C30—N15—N16—C32 | 1.5 (6) | C16—N8—B1—N2 | 23.2 (7) |
| C30—N15—N16—B2 | 166.5 (5) | N7—N8—B1—N2 | −170.8 (4) |
| N2—N1—C2—C3 | −0.7 (7) | C12—N6—B1—N8 | −8.9 (7) |
| Sm1—N1—C2—C3 | −84.4 (5) | N5—N6—B1—N8 | 176.0 (4) |
| N2—N1—C2—C1 | −178.0 (6) | C12—N6—B1—N4 | 115.6 (6) |
| Sm1—N1—C2—C1 | 98.3 (6) | N5—N6—B1—N4 | −59.5 (6) |
| N1—C2—C3—C4 | 1.0 (8) | C12—N6—B1—N2 | −127.4 (5) |
| C1—C2—C3—C4 | 178.0 (7) | N5—N6—B1—N2 | 57.5 (6) |
| N1—N2—C4—C3 | 0.6 (8) | C8—N4—B1—N8 | 24.3 (8) |
| B1—N2—C4—C3 | −172.2 (6) | N3—N4—B1—N8 | −161.0 (4) |
| Sm1—N2—C4—C3 | 77.6 (7) | C8—N4—B1—N6 | −98.4 (6) |
| C2—C3—C4—N2 | −0.9 (9) | N3—N4—B1—N6 | 76.3 (5) |
| N4—N3—C6—C7 | 0.4 (6) | C8—N4—B1—N2 | 141.7 (5) |
| Sm1—N3—C6—C7 | −162.7 (4) | N3—N4—B1—N2 | −43.6 (6) |
| N4—N3—C6—C5 | −176.4 (5) | C4—N2—B1—N8 | 69.5 (7) |
| Sm1—N3—C6—C5 | 20.5 (8) | N1—N2—B1—N8 | −102.2 (5) |
| N3—C6—C7—C8 | 0.1 (6) | Sm1—N2—B1—N8 | −175.4 (3) |
| C5—C6—C7—C8 | 176.4 (6) | C4—N2—B1—N6 | −171.0 (5) |
| N3—N4—C8—C7 | 0.7 (6) | N1—N2—B1—N6 | 17.4 (7) |
| B1—N4—C8—C7 | 175.8 (5) | Sm1—N2—B1—N6 | −55.8 (4) |
| C6—C7—C8—N4 | −0.5 (6) | C4—N2—B1—N4 | −51.0 (7) |
| N6—N5—C10—C11 | −0.8 (6) | N1—N2—B1—N4 | 137.4 (5) |
| Sm1—N5—C10—C11 | −163.9 (4) | Sm1—N2—B1—N4 | 64.2 (3) |
| N6—N5—C10—C9 | −179.8 (5) | C32—N16—B2—N14 | −161.8 (5) |
| Sm1—N5—C10—C9 | 17.2 (9) | N15—N16—B2—N14 | 36.2 (6) |
| N5—C10—C11—C12 | 1.3 (7) | C32—N16—B2—N10 | 77.4 (6) |
| C9—C10—C11—C12 | −179.8 (6) | N15—N16—B2—N10 | −84.6 (5) |
| N5—N6—C12—C11 | 0.7 (6) | C32—N16—B2—N12 | −41.6 (7) |
| B1—N6—C12—C11 | −174.9 (5) | N15—N16—B2—N12 | 156.4 (4) |
| C10—C11—C12—N6 | −1.2 (7) | C28—N14—B2—N16 | 5.8 (8) |
| N8—N7—C14—C15 | −0.4 (7) | N13—N14—B2—N16 | −174.7 (4) |
| N8—N7—C14—C13 | 179.8 (6) | C28—N14—B2—N10 | 124.5 (5) |
| N7—C14—C15—C16 | 0.8 (8) | N13—N14—B2—N10 | −56.0 (6) |
| C13—C14—C15—C16 | −179.4 (7) | C28—N14—B2—N12 | −113.4 (6) |
| N7—N8—C16—C15 | 0.6 (8) | N13—N14—B2—N12 | 66.1 (5) |
| B1—N8—C16—C15 | 168.0 (6) | C20—N10—B2—N16 | 4.1 (7) |
| C14—C15—C16—N8 | −0.8 (8) | N9—N10—B2—N16 | −179.6 (4) |
| N10—N9—C18—C19 | −0.1 (7) | C20—N10—B2—N14 | −117.6 (5) |
| Sm1—N9—C18—C19 | 177.2 (4) | N9—N10—B2—N14 | 58.8 (6) |
| N10—N9—C18—C17 | −179.5 (6) | C20—N10—B2—N12 | 121.1 (5) |
| Sm1—N9—C18—C17 | −2.2 (9) | N9—N10—B2—N12 | −62.5 (6) |

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| N9—C18—C19—C20 | 0.5 (7) | C24—N12—B2—N16 | −24.3 (7) |
| C17—C18—C19—C20 | 179.8 (7) | N11—N12—B2—N16 | 158.0 (4) |
| N9—N10—C20—C19 | 0.7 (6) | C24—N12—B2—N14 | 97.4 (6) |
| B2—N10—C20—C19 | 177.6 (5) | N11—N12—B2—N14 | −80.4 (5) |
| C18—C19—C20—N10 | −0.8 (7) | C24—N12—B2—N10 | −140.7 (5) |
| N12—N11—C22—C23 | −0.7 (6) | N11—N12—B2—N10 | 41.5 (6) |
| Sm1—N11—C22—C23 | 148.8 (4) | | |