# organic compounds

# 3-Bromo-2-[4-(methylsulfanyl)phenyl]-5,6,7,8-tetrahydro-1,3-benzothiazolo-[3,2-a]imidazole

Alexander S. Bunev,[a]* Elena V. Sukhonosova,[b] Vladimir E. Statsyuk,[a] Gennady I. Ostapenko[a] and Victor N. Khrustalev[c]

[a]Department of Chemistry and Chemical Technology, Togliatti State University, 14 Belorusskaya St, Togliatti 445667, Russian Federation, [b]Department of Organic, Bioorganic and Medicinal Chemistry, Samara State University, 1 Akademician Pavlov St, Samara 443011, Russian Federation, and [c]X-Ray Structural Centre, A.N. Nesmeyanov Institute of Organoelement Compounds, Russian Academy of Sciences, 28 Vavilov Street, B-334, Moscow 119991, Russian Federation
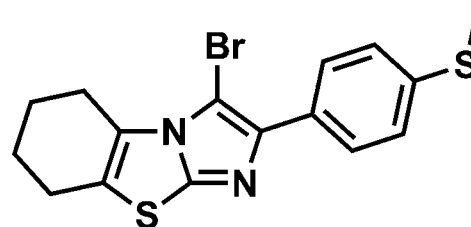Correspondence e-mail: a.s.bunev@gmail.com

In the title molecule, $C_{16}H_{15}BrN_2S_2$, the central imidazo[2,1-b]thiazole fragment is almost planar (r.m.s. deviation = 0.012 Å), and the fused 5,6,7,8-tetrahydrobenzene ring adopts an unsymmetrical half-chair conformation. The dihedral angle between the imidazo[2,1-b]thiazole and benzene planes is 18.25 (4)°. The terminal methylsulfanyl substituent lies practically within the benzene plane [the dihedral angle between the corresponding planes is 7.20 (10)°] and is turned toward the C—Br bond. In the crystal, molecules form infinite chains along [100] via secondary Br···N interactions [3.1861 (16) Å]. The chains are arranged at van der Waals distances.

## Related literature

For applications of imidazo[2,1-b][1,3]benzothiazoles, see: Ager *et al.* (1988); Sanfilippo *et al.* (1988); Barchéchath *et al.* (2005); Andreani *et al.* (2008); Chao *et al.* (2009); Kumbhare *et al.* (2011); Chandak *et al.* (2013). For the crystal structures of related compounds, see: Landreau *et al.* (2002); Adib *et al.* (2008); Fun, Asik *et al.* (2011); Fun, Hemamalini *et al.* (2011); Ghabbour *et al.* (2012); Bunev *et al.* (2013, 2014).

## Experimental

### Crystal data

| | |
|---|---|
| $C_{16}H_{15}BrN_2S_2$ | $\gamma = 101.938\ (1)°$ |
| $M_r = 379.34$ | $V = 771.03\ (6)$ Å$^3$ |
| Triclinic, $P\bar{1}$ | $Z = 2$ |
| $a = 7.3132\ (3)$ Å | Mo $K\alpha$ radiation |
| $b = 7.5663\ (3)$ Å | $\mu = 2.93$ mm$^{-1}$ |
| $c = 14.4543\ (7)$ Å | $T = 120$ K |
| $\alpha = 95.033\ (1)°$ | $0.15 \times 0.10 \times 0.10$ mm |
| $\beta = 97.188\ (1)°$ | |

### Data collection

| | |
|---|---|
| Bruker APEXII CCD diffractometer | 10352 measured reflections |
| Absorption correction: multi-scan (*SADABS*; Bruker, 2003) | 4508 independent reflections |
| $T_{min} = 0.668$, $T_{max} = 0.758$ | 3927 reflections with $I > 2\sigma(I)$ |
| | $R_{int} = 0.025$ |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)] = 0.030$ | 191 parameters |
| $wR(F^2) = 0.073$ | H-atom parameters constrained |
| $S = 1.05$ | $\Delta\rho_{max} = 0.67$ e Å$^{-3}$ |
| 4508 reflections | $\Delta\rho_{min} = -0.32$ e Å$^{-3}$ |

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2001); 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*.

Supporting information for this paper is available from the IUCr electronic archives (Reference: RK2426).

## References

Adib, M., Sheibani, E., Zhu, L.-G. & Bijanzadeh, H. R. (2008). *Synlett*, pp. 2941–2944.

Ager, I. R., Barnes, A. C., Danswan, G. W., Hairsine, P. W., Kay, D. P., Kennewell, P. D., Matharu, S. S., Miller, P. & Robson, P. (1988). *J. Med. Chem.* **31**, 1098–1115.

Andreani, A., Burnelli, S., Granaiola, M., Leoni, A., Locatelli, A., Morigi, R., Rambaldi, M., Varoli, L., Calonghi, N., Cappadone, C., Farruggia, G., Zini, M., Stefanelli, C., Masotti, L., Radin, N. S. & Shoemaker, R. H. (2008). *J. Med. Chem.* **51**, 809–816.

Barchéchath, S. D., Tawatao, R. I., Corr, V., Carson, D. I. & Cottam, H. B. (2005). *J. Med. Chem.* **48**, 6409–6422.

Bruker (2001). *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Bruker (2003). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Bruker (2005). *APEX2*. Bruker AXS Inc., Madison, Wisconsin, USA.

Bunev, A. S., Sukhonosova, E. V., Statsyuk, V. E., Ostapenko, G. I. & Khrustalev, V. N. (2014). *Acta Cryst.* E**70**, o143–o144.

Bunev, A. S., Sukhonosova, E. V., Syrazhetdinova, D. R., Statsyuk, V. E., Ostapenko, G. I. & Khrustalev, V. N. (2013). *Acta Cryst.* E**69**, o531.

Chandak, N., Bhardwaj, J. K., Sharma, R. K. & Sharma, P. K. (2013). *Eur. J. Med. Chem.* **59**, 203–208.

Chao, Q., Sprankle, K. G., Grotzfeld, R. M., Lai, A. G., Carter, T. A., Velasco, A. M., Gunawardane, R. N., Cramer, M. D., Gardner, M. F., James, J., Zarrinkar, P. P., Patel, H. K. & Bhagwat, S. S. (2009). *J. Med. Chem.* **52**, 7808–7816.

Fun, H.-K., Asik, S. I. J., Himaja, M., Munirajasekhar, D. & Sarojini, B. K. (2011). *Acta Cryst.* E**67**, o2810.

Fun, H.-K., Hemamalini, M., Umesha, K., Sarojini, B. K. & Narayana, B. (2011). *Acta Cryst.* E**67**, o3265–o3266.

Ghabbour, H. A., Chia, T. S. & Fun, H.-K. (2012). *Acta Cryst.* E**68**, o1631–o1632.

Kumbhare, R. M., Kumar, K. V., Ramaiah, M. J., Dadmal, T., Pushpavalli, S. N., Mukhopadhyay, D., Divya, B., Devi, T. A., Kosurkar, U. & Pal-Bhadra, M. (2011). *Eur. J. Med. Chem.* **46**, 4258–4266.

Landreau, C., Deniaud, D., Evain, M., Reliquet, A. & Meslin, J.-C. (2002). *J. Chem. Soc. Perkin Trans. 1*, pp. 741–745.

Sanfilippo, P. J., Urbanski, M., Press, J. B., Dubinsky, B. & Moore, J. B. Jr (1988). *J. Med. Chem.* **31**, 2221–2227.

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

# 3-Bromo-2-[4-(methylsulfanyl)phenyl]-5,6,7,8-tetrahydro-1,3-benzothiazolo[3,2-a]imidazole

## Alexander S. Bunev, Elena V. Sukhonosova, Vladimir E. Statsyuk, Gennady I. Ostapenko and Victor N. Khrustalev

## 1. Comment

Imidazo[2,1–*b*][1,3]benzothiazole are of great interest due to their biological properties. These compounds and their derivatives demonstrate the antitumor (Andreani *et al.*, 2008), antiallergic (Ager *et al.*, 1988), anesthetic (Sanfilippo *et al.*, 1988) and anti–cancer (Kumbhare *et al.*, 2011) activities as well as the inhibition activity of apoptosis in testiculargerm cells (Chandak *et al.*, 2013), lymphocytes (Barchéchath *et al.*, 2005), and FMS–like tyrosine kinase–3 (FLT3) (Chao *et al.*, 2009).

In this work, a new halogensubstituted 5,6,7,8–tetrahydrobenzo[d]imidazo[2,1–*b*]thiazole, $C_{16}H_{15}BrN_2S_2$, **I**, was prepared by the reaction of 5,6,7,8–tetrahydrobenzo[d]imidazo[2,1–*b*]thiazole with bromine at room temperature (Fig. 1), and its structure was unambiguously established by the X–ray diffraction study (Fig. 2).

In the title molecule (**I**) the central imidazo[2,1–*b*]thiazole fragment is almost planar (r.m.s. deviation = 0.012 Å), and the fused 5,6,7,8-tetrahydrobenzene ring adopts an unsymmetrical *half–chair* conformation (the C6 and C7 carbon atoms are out of the plane passed through the other atoms of the ring by -0.246 (2) and 0.508 (2) Å, respectively). The bond lengths and angles within the molecule of **I** are in a good agreement with those found in the related compounds (Landreau *et al.*, 2002; Adib *et al.*, 2008; Fun, Asik *et al.*, 2011; Fun, Hemamalini *et al.*, 2011; Ghabbour *et al.*, 2012; Bunev *et al.*, 2013, 2014). The dihedral angle between the imidazo[2,1–*b*]thiazole and benzene planes is 18.25 (4)°. The terminal methylthio substituent lies practically within the benzene plane (the dihedral angle between the corresponding planes is 7.20 (10)°) and is turned toward the C—Br bond.

In the crystal, the molecules of **I** form infinite chains along [100] by intermolecular secondary Br1···N1[i] interactions (3.1861 (16) Å) (Fig. 3). The chains are arranged at van der Waals distances. Symmetry code: (i) 1 + *x*, *y*, *z*.

## 2. Experimental

A solution of bromine (139 *µ*L, 430.4 mg, 2.69 mmol) in dry $CHCl_3$ (10 mL) was added to a solutions 2–(4–(methylthio)-phenyl)–5,6,7,8–tetrahydrobenzo[d]imidazo[2,1–b]thiazole (808.3 mg, 2.69 mmol) in dry $CHCl_3$ (30 mL). The reaction mixture was stirred at room temperature for 3 h. the solvent was evaporated from the reaction mixture on rotavapor. The crude product was diluted with 5% solution $Na_2CO_3$ in water (25 mL). The precipitate was filtered and crystallized from *DMF*. Yield is 75%. The single–crystal of the product **I** was obtained by slow crystallization from *DMF*. M.p. = 439–441 K. IR (KBr), *v*/cm⁻¹: 3131, 3073, 1580, 1523, 1501, 1337, 1144, 815, 714. ¹H NMR (600 MHz, *DMSO–d₆*, 304 K): 7.63 (d, 2H, *J* = 8.9), 7.54 (d, 2H, *J* = 8.9), 3.39–3.33 (m, 2H), 3.02–2.96 (m, 2H), 2.45 (s, 3H), 1.91–1.81 (m, 4H). Anal. Calcd for $C_{16}H_{15}BrN_2S$: C, 50.66; H, 3.99. Found: C, 50.57; H, 4.08.

## 3. Refinement

All hydrogen atoms were placed in the calculated positions with C—H = 0.95–0.99 Å and refined in the riding model with fixed isotropic displacement parameters: $U_{iso}(H) = 1.5U_{eq}(C)$ for the methyl group and $U_{iso}(H) = 1.2U_{eq}(C)$ for the other groups.
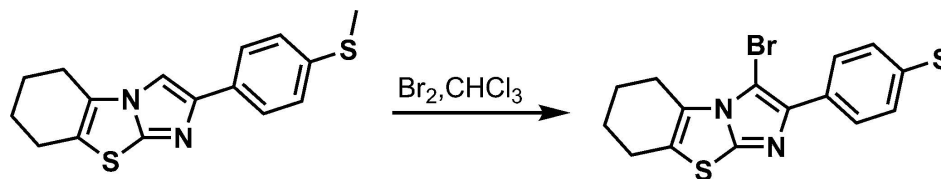


**Figure 1**

The synthesis of 3-bromo-2-[4-(methylsulfanyl)phenyl]-5,6,7,8-tetrahydro-1,3-benzothiazolo[3,2-*a*]imidazole.
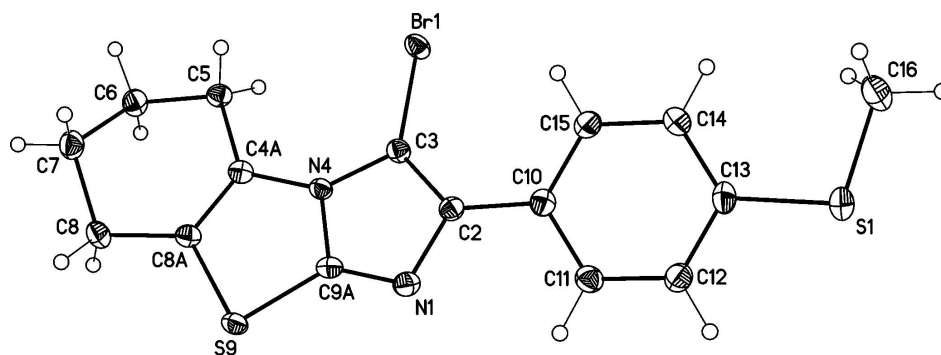


**Figure 2**

Molecular structure of **I**. Displacement ellipsoids are presented at the 50% probability level. H atoms are depicted as small spheres of arbitrary radius.
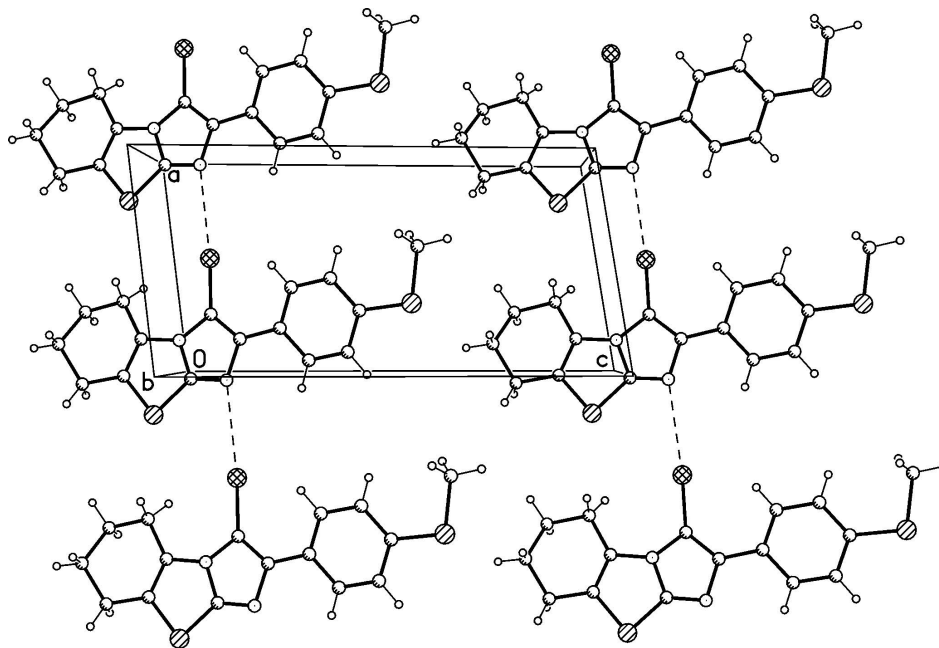
**Figure 3**

A portion of the crystal structure of **I** demonstrating the chains along [100]. The intermolecular secondary Br···N interactions are depicted by dashed lines.

## 3-Bromo-2-[4-(methylsulfanyl)phenyl]-5,6,7,8-tetrahydro-1,3-benzothiazolo[3,2-*a*]imidazole

*Crystal data*

| | |
|---|---|
| $C_{16}H_{15}BrN_2S_2$ | $Z = 2$ |
| $M_r = 379.34$ | $F(000) = 384$ |
| Triclinic, $P\overline{1}$ | $D_x = 1.634$ Mg m$^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $a = 7.3132\ (3)$ Å | Cell parameters from 4428 reflections |
| $b = 7.5663\ (3)$ Å | $\theta = 2.8–32.3°$ |
| $c = 14.4543\ (7)$ Å | $\mu = 2.93$ mm$^{-1}$ |
| $\alpha = 95.033\ (1)°$ | $T = 120$ K |
| $\beta = 97.188\ (1)°$ | Prism, colourless |
| $\gamma = 101.938\ (1)°$ | $0.15 \times 0.10 \times 0.10$ mm |
| $V = 771.03\ (6)$ Å$^3$ | |

*Data collection*

| | |
|---|---|
| Bruker APEXII CCD diffractometer | 10352 measured reflections |
| Radiation source: fine-focus sealed tube | 4508 independent reflections |
| Graphite monochromator | 3927 reflections with $I > 2\sigma(I)$ |
| $\varphi$ and $\omega$ scans | $R_{int} = 0.025$ |
| Absorption correction: multi-scan | $\theta_{max} = 30.0°$, $\theta_{min} = 2.8°$ |
| (*SADABS*; Bruker, 2003) | $h = -10\rightarrow10$ |
| $T_{min} = 0.668$, $T_{max} = 0.758$ | $k = -10\rightarrow10$ |
| | $l = -20\rightarrow20$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | |
| $R[F^2 > 2\sigma(F^2)] = 0.030$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.073$ | |
| $S = 1.05$ | H-atom parameters constrained |
| 4508 reflections | $w = 1/[\sigma^2(F_o^2) + (0.0377P)^2 + 0.1351P]$ |
| 191 parameters | where $P = (F_o^2 + 2F_c^2)/3$ |
| 0 restraints | $(\Delta/\sigma)_{max} = 0.001$ |
| Primary atom site location: structure-invariant direct methods | $\Delta\rho_{max} = 0.67$ e Å$^{-3}$ |
| | $\Delta\rho_{min} = -0.32$ e Å$^{-3}$ |

*Special details*

**Geometry**. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'s involving l.s. planes.

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted $R$–factor w$R$ 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* | $U_{iso}*/U_{eq}$ |
|---|---|---|---|---|
| Br1 | 0.52846 (2) | 0.33371 (2) | 0.103656 (13) | 0.01782 (6) |
| S1 | 0.31673 (8) | 0.75383 (8) | 0.55165 (4) | 0.02712 (12) |
| N1 | −0.0318 (2) | 0.3490 (2) | 0.10823 (11) | 0.0169 (3) |
| C2 | 0.1565 (3) | 0.3866 (2) | 0.15151 (13) | 0.0150 (3) |
| C3 | 0.2697 (2) | 0.3284 (2) | 0.09002 (13) | 0.0146 (3) |
| N4 | 0.1494 (2) | 0.2541 (2) | 0.00724 (10) | 0.0136 (3) |
| C4A | 0.1551 (3) | 0.1754 (2) | −0.08424 (13) | 0.0147 (3) |
| C5 | 0.3302 (3) | 0.1399 (2) | −0.11797 (13) | 0.0159 (3) |
| H5A | 0.3921 | 0.0696 | −0.0739 | 0.019* |
| H5B | 0.4195 | 0.2568 | −0.1199 | 0.019* |
| C6 | 0.2819 (3) | 0.0326 (3) | −0.21646 (14) | 0.0194 (4) |
| H6A | 0.3978 | 0.0462 | −0.2464 | 0.023* |
| H6B | 0.2372 | −0.0981 | −0.2106 | 0.023* |
| C7 | 0.1308 (3) | 0.0964 (3) | −0.27918 (13) | 0.0208 (4) |
| H7A | 0.1110 | 0.0295 | −0.3429 | 0.025* |
| H7B | 0.1735 | 0.2276 | −0.2844 | 0.025* |
| C8 | −0.0560 (3) | 0.0635 (3) | −0.23845 (13) | 0.0186 (4) |
| H8A | −0.1458 | 0.1253 | −0.2726 | 0.022* |
| H8B | −0.1133 | −0.0684 | −0.2457 | 0.022* |
| C8A | −0.0175 (3) | 0.1366 (2) | −0.13656 (13) | 0.0154 (3) |
| S9 | −0.19453 (6) | 0.18940 (6) | −0.07481 (3) | 0.01700 (10) |
| C9A | −0.0272 (2) | 0.2722 (2) | 0.02379 (13) | 0.0153 (3) |
| C10 | 0.2059 (3) | 0.4782 (2) | 0.24789 (13) | 0.0164 (3) |
| C11 | 0.0607 (3) | 0.4836 (3) | 0.30255 (14) | 0.0191 (4) |
| H11 | −0.0658 | 0.4267 | 0.2767 | 0.023* |
| C12 | 0.0991 (3) | 0.5705 (3) | 0.39362 (14) | 0.0204 (4) |

| | | | | |
|---|---|---|---|---|
| H12 | −0.0014 | 0.5733 | 0.4291 | 0.024* |
| C13 | 0.2850 (3) | 0.6542 (3) | 0.43388 (13) | 0.0199 (4) |
| C14 | 0.4292 (3) | 0.6527 (3) | 0.37994 (14) | 0.0232 (4) |
| H14 | 0.5554 | 0.7108 | 0.4058 | 0.028* |
| C15 | 0.3898 (3) | 0.5664 (3) | 0.28802 (14) | 0.0217 (4) |
| H15 | 0.4900 | 0.5676 | 0.2520 | 0.026* |
| C16 | 0.5684 (3) | 0.8198 (3) | 0.58283 (16) | 0.0302 (5) |
| H16A | 0.6001 | 0.8615 | 0.6502 | 0.045* |
| H16B | 0.6187 | 0.9185 | 0.5472 | 0.045* |
| H16C | 0.6242 | 0.7154 | 0.5682 | 0.045* |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Br1 | 0.01313 (9) | 0.02030 (10) | 0.01932 (10) | 0.00599 (7) | −0.00109 (6) | −0.00219 (7) |
| S1 | 0.0271 (3) | 0.0327 (3) | 0.0173 (2) | 0.0007 (2) | 0.0029 (2) | −0.0057 (2) |
| N1 | 0.0157 (7) | 0.0163 (7) | 0.0187 (8) | 0.0026 (6) | 0.0042 (6) | 0.0015 (6) |
| C2 | 0.0154 (8) | 0.0140 (8) | 0.0158 (8) | 0.0029 (7) | 0.0033 (7) | 0.0023 (6) |
| C3 | 0.0134 (8) | 0.0155 (8) | 0.0154 (8) | 0.0051 (7) | 0.0010 (6) | 0.0008 (6) |
| N4 | 0.0109 (7) | 0.0153 (7) | 0.0145 (7) | 0.0034 (6) | 0.0011 (5) | 0.0010 (6) |
| C4A | 0.0145 (8) | 0.0121 (8) | 0.0175 (9) | 0.0030 (6) | 0.0023 (7) | 0.0016 (6) |
| C5 | 0.0140 (8) | 0.0160 (8) | 0.0186 (9) | 0.0050 (7) | 0.0036 (7) | 0.0004 (7) |
| C6 | 0.0195 (9) | 0.0217 (9) | 0.0179 (9) | 0.0083 (8) | 0.0030 (7) | −0.0010 (7) |
| C7 | 0.0221 (9) | 0.0247 (10) | 0.0159 (9) | 0.0070 (8) | 0.0025 (7) | −0.0004 (7) |
| C8 | 0.0178 (9) | 0.0193 (9) | 0.0167 (9) | 0.0036 (7) | −0.0016 (7) | −0.0013 (7) |
| C8A | 0.0136 (8) | 0.0140 (8) | 0.0182 (8) | 0.0025 (7) | 0.0028 (7) | 0.0007 (6) |
| S9 | 0.01080 (19) | 0.0210 (2) | 0.0186 (2) | 0.00355 (17) | 0.00092 (16) | 0.00057 (17) |
| C9A | 0.0119 (8) | 0.0160 (8) | 0.0186 (8) | 0.0030 (7) | 0.0033 (7) | 0.0031 (7) |
| C10 | 0.0192 (9) | 0.0129 (8) | 0.0167 (8) | 0.0027 (7) | 0.0028 (7) | 0.0010 (6) |
| C11 | 0.0176 (9) | 0.0174 (9) | 0.0208 (9) | 0.0017 (7) | 0.0021 (7) | 0.0001 (7) |
| C12 | 0.0211 (9) | 0.0208 (9) | 0.0193 (9) | 0.0037 (8) | 0.0051 (7) | 0.0010 (7) |
| C13 | 0.0252 (10) | 0.0187 (9) | 0.0145 (8) | 0.0037 (8) | 0.0019 (7) | −0.0011 (7) |
| C14 | 0.0196 (9) | 0.0252 (10) | 0.0208 (10) | −0.0006 (8) | 0.0014 (7) | −0.0028 (8) |
| C15 | 0.0205 (9) | 0.0235 (10) | 0.0194 (9) | 0.0009 (8) | 0.0063 (7) | −0.0012 (8) |
| C16 | 0.0286 (11) | 0.0311 (12) | 0.0249 (11) | −0.0003 (9) | −0.0030 (9) | −0.0030 (9) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Br1—C3 | 1.8693 (18) | C7—H7B | 0.9900 |
| S1—C13 | 1.7673 (19) | C8—C8A | 1.498 (3) |
| S1—C16 | 1.793 (2) | C8—H8A | 0.9900 |
| N1—C9A | 1.312 (2) | C8—H8B | 0.9900 |
| N1—C2 | 1.400 (2) | C8A—S9 | 1.7529 (19) |
| C2—C3 | 1.391 (2) | S9—C9A | 1.7354 (19) |
| C2—C10 | 1.467 (3) | C10—C15 | 1.399 (3) |
| C3—N4 | 1.391 (2) | C10—C11 | 1.405 (3) |
| N4—C9A | 1.374 (2) | C11—C12 | 1.387 (3) |
| N4—C4A | 1.411 (2) | C11—H11 | 0.9500 |
| C4A—C8A | 1.349 (2) | C12—C13 | 1.403 (3) |
| C4A—C5 | 1.492 (2) | C12—H12 | 0.9500 |

| C5—C6 | 1.537 (3) | C13—C14 | 1.389 (3) |
|---|---|---|---|
| C5—H5A | 0.9900 | C14—C15 | 1.396 (3) |
| C5—H5B | 0.9900 | C14—H14 | 0.9500 |
| C6—C7 | 1.524 (3) | C15—H15 | 0.9500 |
| C6—H6A | 0.9900 | C16—H16A | 0.9800 |
| C6—H6B | 0.9900 | C16—H16B | 0.9800 |
| C7—C8 | 1.537 (3) | C16—H16C | 0.9800 |
| C7—H7A | 0.9900 | | |

| C13—S1—C16 | 103.71 (10) | C8A—C8—H8B | 109.9 |
|---|---|---|---|
| C9A—N1—C2 | 104.13 (15) | C7—C8—H8B | 109.9 |
| C3—C2—N1 | 110.13 (16) | H8A—C8—H8B | 108.3 |
| C3—C2—C10 | 130.54 (17) | C4A—C8A—C8 | 124.20 (17) |
| N1—C2—C10 | 119.33 (16) | C4A—C8A—S9 | 113.31 (14) |
| C2—C3—N4 | 106.03 (15) | C8—C8A—S9 | 122.44 (13) |
| C2—C3—Br1 | 131.99 (14) | C9A—S9—C8A | 89.99 (9) |
| N4—C3—Br1 | 121.98 (13) | N1—C9A—N4 | 114.32 (16) |
| C9A—N4—C3 | 105.38 (15) | N1—C9A—S9 | 134.58 (14) |
| C9A—N4—C4A | 114.32 (15) | N4—C9A—S9 | 111.08 (13) |
| C3—N4—C4A | 140.26 (16) | C15—C10—C11 | 117.62 (17) |
| C8A—C4A—N4 | 111.27 (16) | C15—C10—C2 | 123.61 (17) |
| C8A—C4A—C5 | 124.77 (17) | C11—C10—C2 | 118.73 (17) |
| N4—C4A—C5 | 123.97 (16) | C12—C11—C10 | 121.15 (18) |
| C4A—C5—C6 | 110.25 (15) | C12—C11—H11 | 119.4 |
| C4A—C5—H5A | 109.6 | C10—C11—H11 | 119.4 |
| C6—C5—H5A | 109.6 | C11—C12—C13 | 120.61 (18) |
| C4A—C5—H5B | 109.6 | C11—C12—H12 | 119.7 |
| C6—C5—H5B | 109.6 | C13—C12—H12 | 119.7 |
| H5A—C5—H5B | 108.1 | C14—C13—C12 | 118.75 (18) |
| C7—C6—C5 | 112.55 (15) | C14—C13—S1 | 124.86 (16) |
| C7—C6—H6A | 109.1 | C12—C13—S1 | 116.38 (15) |
| C5—C6—H6A | 109.1 | C13—C14—C15 | 120.45 (19) |
| C7—C6—H6B | 109.1 | C13—C14—H14 | 119.8 |
| C5—C6—H6B | 109.1 | C15—C14—H14 | 119.8 |
| H6A—C6—H6B | 107.8 | C14—C15—C10 | 121.38 (18) |
| C6—C7—C8 | 110.44 (16) | C14—C15—H15 | 119.3 |
| C6—C7—H7A | 109.6 | C10—C15—H15 | 119.3 |
| C8—C7—H7A | 109.6 | S1—C16—H16A | 109.5 |
| C6—C7—H7B | 109.6 | S1—C16—H16B | 109.5 |
| C8—C7—H7B | 109.6 | H16A—C16—H16B | 109.5 |
| H7A—C7—H7B | 108.1 | S1—C16—H16C | 109.5 |
| C8A—C8—C7 | 109.02 (15) | H16A—C16—H16C | 109.5 |
| C8A—C8—H8A | 109.9 | H16B—C16—H16C | 109.5 |
| C7—C8—H8A | 109.9 | | |

| C9A—N1—C2—C3 | −0.4 (2) | C8—C8A—S9—C9A | −175.86 (16) |
|---|---|---|---|
| C9A—N1—C2—C10 | 178.60 (16) | C2—N1—C9A—N4 | 0.5 (2) |
| N1—C2—C3—N4 | 0.2 (2) | C2—N1—C9A—S9 | −178.31 (16) |
| C10—C2—C3—N4 | −178.66 (17) | C3—N4—C9A—N1 | −0.4 (2) |

| | | | |
|---|---|---|---|
| N1—C2—C3—Br1 | −178.84 (14) | C4A—N4—C9A—N1 | −178.63 (15) |
| C10—C2—C3—Br1 | 2.3 (3) | C3—N4—C9A—S9 | 178.71 (12) |
| C2—C3—N4—C9A | 0.07 (19) | C4A—N4—C9A—S9 | 0.45 (19) |
| Br1—C3—N4—C9A | 179.24 (12) | C8A—S9—C9A—N1 | 177.7 (2) |
| C2—C3—N4—C4A | 177.6 (2) | C8A—S9—C9A—N4 | −1.11 (14) |
| Br1—C3—N4—C4A | −3.2 (3) | C3—C2—C10—C15 | 17.2 (3) |
| C9A—N4—C4A—C8A | 0.7 (2) | N1—C2—C10—C15 | −161.53 (18) |
| C3—N4—C4A—C8A | −176.6 (2) | C3—C2—C10—C11 | −164.91 (19) |
| C9A—N4—C4A—C5 | −179.12 (16) | N1—C2—C10—C11 | 16.3 (3) |
| C3—N4—C4A—C5 | 3.5 (3) | C15—C10—C11—C12 | −1.1 (3) |
| C8A—C4A—C5—C6 | −6.7 (3) | C2—C10—C11—C12 | −179.12 (17) |
| N4—C4A—C5—C6 | 173.18 (16) | C10—C11—C12—C13 | −0.5 (3) |
| C4A—C5—C6—C7 | 39.6 (2) | C11—C12—C13—C14 | 1.7 (3) |
| C5—C6—C7—C8 | −63.1 (2) | C11—C12—C13—S1 | −177.96 (15) |
| C6—C7—C8—C8A | 49.1 (2) | C16—S1—C13—C14 | −6.6 (2) |
| N4—C4A—C8A—C8 | 175.79 (16) | C16—S1—C13—C12 | 173.04 (16) |
| C5—C4A—C8A—C8 | −4.3 (3) | C12—C13—C14—C15 | −1.2 (3) |
| N4—C4A—C8A—S9 | −1.6 (2) | S1—C13—C14—C15 | 178.47 (16) |
| C5—C4A—C8A—S9 | 178.26 (14) | C13—C14—C15—C10 | −0.5 (3) |
| C7—C8—C8A—C4A | −17.4 (3) | C11—C10—C15—C14 | 1.7 (3) |
| C7—C8—C8A—S9 | 159.79 (14) | C2—C10—C15—C14 | 179.55 (18) |
| C4A—C8A—S9—C9A | 1.59 (15) | | |