

[*N'*-(5-Bromo-2-oxidobenzylidene- κ O)-3-hydroxy-2-naphthohydrazidato- κ^2 *N',O*]-dibutyltin(IV)

See Mun Lee, Kong Mun Lo, Hapipah Mohd Ali and Seik Weng Ng*

Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: seikweng@um.edu.my

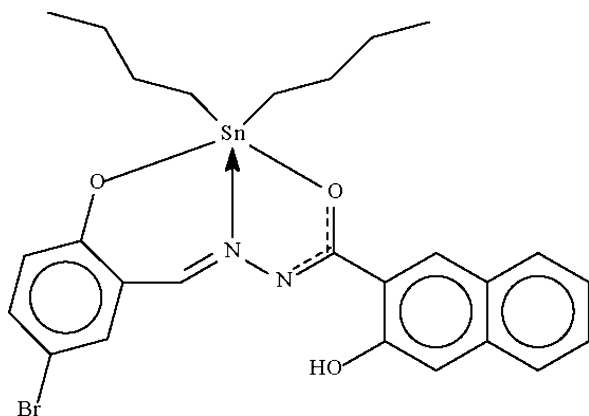
Received 24 June 2009; accepted 25 June 2009

Key indicators: single-crystal X-ray study; $T = 140$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; disorder in main residue; R factor = 0.025; wR factor = 0.060; data-to-parameter ratio = 16.8.

The Sn^{IV} atom in the title compound, $[\text{Sn}(\text{C}_4\text{H}_9)_2(\text{C}_{18}\text{H}_{11}\text{BrN}_2\text{O}_3)]$, shows a distorted *cis*- $\text{C}_2\text{NO}_2\text{Sn}$ trigonal-bipyramidal coordination. One of the butyl chains is disordered over two sites in a 0.60 (1):0.40 (1) ratio.

Related literature

The dianions of similar *N'*-(2-hydroxybenzylidene)benzohydrazones *O,N,O'*-chelate to tin in organotin compounds; see: Labib *et al.* (1996); Samanta *et al.* (2007).



Experimental

Crystal data

$[\text{Sn}(\text{C}_4\text{H}_9)_2(\text{C}_{18}\text{H}_{11}\text{BrN}_2\text{O}_3)]$
 $M_r = 616.11$
Triclinic, $P\bar{1}$
 $a = 10.1626$ (2) Å
 $b = 12.2534$ (2) Å
 $c = 12.5583$ (2) Å
 $\alpha = 62.309$ (1)°
 $\beta = 83.809$ (1)°

$\gamma = 65.802$ (1)°
 $V = 1256.44$ (4) Å³
 $Z = 2$
Mo $K\alpha$ radiation
 $\mu = 2.64$ mm⁻¹
 $T = 140$ K
 $0.29 \times 0.26 \times 0.20$ mm

Data collection

Bruker SMART APEX
diffractometer
Absorption correction: multi-scan
(*SADABS*; Sheldrick, 1996)
 $T_{\text{min}} = 0.515$, $T_{\text{max}} = 0.621$

12053 measured reflections
5740 independent reflections
4886 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.020$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.025$
 $wR(F^2) = 0.060$
 $S = 1.01$
5740 reflections
342 parameters
47 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.53$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.59$ e Å⁻³

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *pubCIF* (Westrip, 2009).

We thank the University of Malaya (PS320/2008C, RG020/09AFR) for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2544).

References

- Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Labib, L., Khalil, T. E., Iskander, M. F. & Refaat, L. S. (1996). *Polyhedron*, **21**, 3697–3707.
Samanta, B., Chakraborty, J., Dey, D. K. & Mitra, S. (2007). *Struct. Chem.* **18**, 287–297.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
Westrip, S. P. (2009). *pubCIF*. In preparation.

supplementary materials

Acta Cryst. (2009). E65, m862 [doi:10.1107/S1600536809024477]

[N'-(5-Bromo-2-oxidobenzylidene- κ O)-3-hydroxy-2-naphthohydrazidato- κ^2 N',O]dibutyltin(IV)

S. M. Lee, K. M. Lo, H. M. Ali and S. W. Ng

Experimental

The Schiff base (0.39 g, 1 mmol) prepared from the condensation of 5-bromosalicylaldehyde and 3-hydroxy-2-naphthoic hydrazide was heated with dibutyltin oxide (0.25 g, 1 mmol) in ethanol (100 ml) until the oxide dissolved completely. Slow cooling of the filtrate gave the product as yellow crystals.

Refinement

One of the two butyl chains is disordered over two positions in all four carbon atoms. The C–C distances were restrained to 1.54±0.01 Å; the anisotropic temperature factors of the carbon atoms were restrained to be nearly isotropic. The disorder refined to a 60 (1):40 (1) ratio.

Hydrogen atoms were placed at calculated positions (C–H 0.95–0.98 Å) and were treated as riding on their parent atoms, with $U_{\text{iso}}(\text{H})$ set to 1.2–1.5 $U_{\text{eq}}(\text{C})$. The hydroxy H-atom was refined with a distance restraint of 0.84±0.01 Å.

Figures

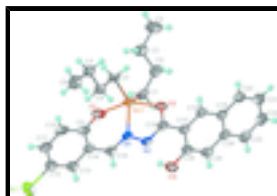


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $\text{Sn}(\text{C}_4\text{H}_9)_2(\text{C}_{18}\text{H}_{11}\text{BrN}_2\text{O}_3)$ at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius. The disorder in one of the two butyl chains is not shown.

[N'-(5-Bromo-2-oxidobenzylidene- κ O)-3-hydroxy-2-naphthohydrazidato- κ^2 N',O]dibutyltin(IV)

Crystal data

$[\text{Sn}(\text{C}_4\text{H}_9)_2(\text{C}_{18}\text{H}_{11}\text{BrN}_2\text{O}_3)]$

$M_r = 616.11$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 10.1626$ (2) Å

$b = 12.2534$ (2) Å

$c = 12.5583$ (2) Å

$\alpha = 62.309$ (1)°

$\beta = 83.809$ (1)°

$\gamma = 65.802$ (1)°

$V = 1256.44$ (4) Å³

$Z = 2$

$F_{000} = 616$

$D_x = 1.629$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 5075 reflections

$\theta = 2.5$ – 29.6 °

$\mu = 2.64$ mm⁻¹

$T = 140$ K

Block, yellow

$0.29 \times 0.26 \times 0.20$ mm

supplementary materials

Data collection

| | |
|---|--|
| Bruker SMART APEX diffractometer | 5740 independent reflections |
| Radiation source: fine-focus sealed tube | 4886 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{\text{int}} = 0.020$ |
| $T = 140$ K | $\theta_{\text{max}} = 27.5^\circ$ |
| ω scans | $\theta_{\text{min}} = 1.8^\circ$ |
| Absorption correction: Multi-scan (SADABS; Sheldrick, 1996) | $h = -13 \rightarrow 13$ |
| $T_{\text{min}} = 0.515$, $T_{\text{max}} = 0.621$ | $k = -15 \rightarrow 15$ |
| 12053 measured reflections | $l = -16 \rightarrow 16$ |

Refinement

| | |
|--|--|
| Refinement on F^2 | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.025$ | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2) = 0.060$ | $w = 1/[\sigma^2(F_o^2) + (0.0287P)^2 + 0.3063P]$ |
| $S = 1.01$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 5740 reflections | $(\Delta/\sigma)_{\text{max}} = 0.001$ |
| 342 parameters | $\Delta\rho_{\text{max}} = 0.53 \text{ e } \text{\AA}^{-3}$ |
| 47 restraints | $\Delta\rho_{\text{min}} = -0.59 \text{ e } \text{\AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | Extinction correction: none |

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|-----|---------------|---------------|---------------|----------------------------------|-----------|
| Sn1 | 0.828380 (16) | 0.685444 (15) | 0.242560 (14) | 0.02643 (5) | |
| Br1 | 0.66105 (3) | 1.14077 (3) | 0.54233 (3) | 0.04563 (8) | |
| N1 | 0.62849 (19) | 0.86055 (18) | 0.22108 (16) | 0.0253 (4) | |
| N2 | 0.51208 (19) | 0.88674 (18) | 0.15093 (16) | 0.0269 (4) | |
| O1 | 0.91148 (17) | 0.75767 (16) | 0.32829 (17) | 0.0373 (4) | |
| O2 | 0.67073 (17) | 0.70268 (15) | 0.12755 (14) | 0.0312 (4) | |
| O3 | 0.24653 (18) | 1.01189 (17) | 0.04941 (17) | 0.0377 (4) | |
| H3 | 0.316 (2) | 0.997 (3) | 0.091 (2) | 0.043 (8)* | |
| C1 | 0.9977 (13) | 0.6790 (7) | 0.1322 (11) | 0.033 (2) | 0.604 (8) |
| H1A | 0.9610 | 0.7589 | 0.0518 | 0.040* | 0.604 (8) |
| H1B | 1.0748 | 0.6859 | 0.1674 | 0.040* | 0.604 (8) |
| C2 | 1.0651 (6) | 0.5515 (4) | 0.1148 (4) | 0.0393 (14) | 0.604 (8) |
| H2A | 0.9899 | 0.5474 | 0.0747 | 0.047* | 0.604 (8) |
| H2B | 1.1428 | 0.5585 | 0.0605 | 0.047* | 0.604 (8) |
| C3 | 1.1279 (6) | 0.4224 (5) | 0.2312 (5) | 0.0407 (14) | 0.604 (8) |

supplementary materials

| | | | | | |
|-----|-------------|------------|---------------|------------|-----------|
| H3A | 1.0489 | 0.4069 | 0.2811 | 0.049* | 0.604 (8) |
| H3B | 1.1940 | 0.4299 | 0.2774 | 0.049* | 0.604 (8) |
| C4 | 1.2132 (13) | 0.3006 (7) | 0.2037 (10) | 0.053 (3) | 0.604 (8) |
| H4A | 1.2446 | 0.2161 | 0.2796 | 0.079* | 0.604 (8) |
| H4B | 1.2983 | 0.3108 | 0.1628 | 0.079* | 0.604 (8) |
| H4C | 1.1501 | 0.2987 | 0.1513 | 0.079* | 0.604 (8) |
| C1' | 0.9820 (16) | 0.6826 (9) | 0.1057 (16) | 0.027 (3) | 0.396 (8) |
| H1C | 0.9347 | 0.6928 | 0.0347 | 0.032* | 0.396 (8) |
| H1D | 1.0088 | 0.7599 | 0.0786 | 0.032* | 0.396 (8) |
| C2' | 1.1199 (7) | 0.5504 (6) | 0.1557 (8) | 0.037 (2) | 0.396 (8) |
| H2C | 1.1921 | 0.5585 | 0.0961 | 0.044* | 0.396 (8) |
| H2D | 1.1619 | 0.5366 | 0.2307 | 0.044* | 0.396 (8) |
| C3' | 1.0915 (9) | 0.4283 (7) | 0.1826 (10) | 0.047 (2) | 0.396 (8) |
| H3C | 1.0529 | 0.4391 | 0.1076 | 0.056* | 0.396 (8) |
| H3D | 1.0187 | 0.4194 | 0.2416 | 0.056* | 0.396 (8) |
| C4' | 1.2367 (15) | 0.2992 (9) | 0.2356 (14) | 0.045 (3) | 0.396 (8) |
| H4D | 1.2299 | 0.2276 | 0.2245 | 0.068* | 0.396 (8) |
| H4E | 1.2546 | 0.2687 | 0.3221 | 0.068* | 0.396 (8) |
| H4F | 1.3167 | 0.3204 | 0.1936 | 0.068* | 0.396 (8) |
| C5 | 0.8159 (2) | 0.5186 (2) | 0.4004 (2) | 0.0270 (5) | |
| H5A | 0.7944 | 0.4611 | 0.3760 | 0.032* | |
| H5B | 0.9116 | 0.4638 | 0.4490 | 0.032* | |
| C6 | 0.7010 (2) | 0.5593 (2) | 0.4793 (2) | 0.0284 (5) | |
| H6A | 0.7220 | 0.6169 | 0.5039 | 0.034* | |
| H6B | 0.6049 | 0.6132 | 0.4315 | 0.034* | |
| C7 | 0.6958 (3) | 0.4372 (2) | 0.5921 (2) | 0.0334 (5) | |
| H7A | 0.6758 | 0.3792 | 0.5673 | 0.040* | |
| H7B | 0.7918 | 0.3837 | 0.6400 | 0.040* | |
| C8 | 0.5807 (3) | 0.4764 (3) | 0.6711 (2) | 0.0438 (6) | |
| H8A | 0.5856 | 0.3950 | 0.7443 | 0.066* | |
| H8B | 0.4845 | 0.5231 | 0.6263 | 0.066* | |
| H8C | 0.5978 | 0.5365 | 0.6935 | 0.066* | |
| C9 | 0.8505 (2) | 0.8415 (2) | 0.3749 (2) | 0.0300 (5) | |
| C10 | 0.9369 (3) | 0.8446 (2) | 0.4528 (2) | 0.0366 (6) | |
| H10 | 1.0369 | 0.7856 | 0.4712 | 0.044* | |
| C11 | 0.8800 (3) | 0.9309 (2) | 0.5027 (2) | 0.0352 (6) | |
| H11 | 0.9402 | 0.9297 | 0.5565 | 0.042* | |
| C12 | 0.7347 (3) | 1.0201 (2) | 0.4748 (2) | 0.0323 (5) | |
| C13 | 0.6465 (2) | 1.0216 (2) | 0.3985 (2) | 0.0298 (5) | |
| H13 | 0.5475 | 1.0835 | 0.3794 | 0.036* | |
| C14 | 0.7019 (2) | 0.9314 (2) | 0.3481 (2) | 0.0268 (5) | |
| C15 | 0.6026 (2) | 0.9392 (2) | 0.2698 (2) | 0.0265 (5) | |
| H15 | 0.5080 | 1.0093 | 0.2516 | 0.032* | |
| C16 | 0.5445 (2) | 0.8009 (2) | 0.10719 (19) | 0.0265 (5) | |
| C17 | 0.4293 (2) | 0.8171 (2) | 0.03210 (19) | 0.0267 (5) | |
| C18 | 0.4605 (2) | 0.7287 (2) | -0.01503 (19) | 0.0265 (5) | |
| H18 | 0.5566 | 0.6618 | -0.0006 | 0.032* | |
| C19 | 0.3555 (3) | 0.7338 (2) | -0.08373 (19) | 0.0290 (5) | |
| C20 | 0.3883 (3) | 0.6414 (2) | -0.1309 (2) | 0.0339 (5) | |

supplementary materials

| | | | | |
|-----|------------|------------|-------------|------------|
| H20 | 0.4847 | 0.5757 | -0.1188 | 0.041* |
| C21 | 0.2825 (3) | 0.6461 (3) | -0.1935 (2) | 0.0408 (6) |
| H21 | 0.3047 | 0.5829 | -0.2234 | 0.049* |
| C22 | 0.1397 (3) | 0.7457 (3) | -0.2134 (2) | 0.0397 (6) |
| H22 | 0.0665 | 0.7484 | -0.2568 | 0.048* |
| C23 | 0.1049 (3) | 0.8378 (3) | -0.1720 (2) | 0.0348 (5) |
| H23 | 0.0086 | 0.9047 | -0.1879 | 0.042* |
| C24 | 0.2118 (2) | 0.8345 (2) | -0.1049 (2) | 0.0292 (5) |
| C25 | 0.1806 (2) | 0.9263 (2) | -0.0580 (2) | 0.0304 (5) |
| H25 | 0.0851 | 0.9945 | -0.0731 | 0.036* |
| C26 | 0.2844 (2) | 0.9195 (2) | 0.0086 (2) | 0.0290 (5) |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|--------------|--------------|--------------|---------------|--------------|---------------|
| Sn1 | 0.01985 (8) | 0.01819 (8) | 0.03093 (9) | -0.00410 (6) | 0.00688 (6) | -0.00751 (6) |
| Br1 | 0.04928 (17) | 0.05103 (18) | 0.05338 (18) | -0.02710 (14) | 0.01566 (14) | -0.03359 (15) |
| N1 | 0.0219 (9) | 0.0205 (9) | 0.0239 (9) | -0.0055 (7) | 0.0040 (7) | -0.0059 (7) |
| N2 | 0.0236 (9) | 0.0225 (9) | 0.0251 (9) | -0.0051 (8) | 0.0031 (8) | -0.0076 (8) |
| O1 | 0.0220 (8) | 0.0282 (9) | 0.0573 (11) | -0.0044 (7) | 0.0013 (8) | -0.0209 (8) |
| O2 | 0.0257 (8) | 0.0239 (8) | 0.0356 (9) | -0.0017 (7) | 0.0028 (7) | -0.0144 (7) |
| O3 | 0.0257 (9) | 0.0317 (9) | 0.0517 (11) | -0.0017 (7) | -0.0001 (8) | -0.0241 (9) |
| C1 | 0.026 (3) | 0.035 (3) | 0.017 (5) | -0.003 (2) | 0.003 (2) | -0.005 (2) |
| C2 | 0.035 (3) | 0.042 (3) | 0.028 (2) | -0.008 (2) | 0.009 (2) | -0.015 (2) |
| C3 | 0.033 (3) | 0.034 (3) | 0.043 (3) | -0.009 (2) | 0.007 (2) | -0.013 (2) |
| C4 | 0.044 (4) | 0.036 (3) | 0.062 (6) | -0.007 (3) | 0.017 (3) | -0.020 (3) |
| C1' | 0.026 (5) | 0.021 (4) | 0.019 (7) | -0.007 (3) | 0.010 (4) | -0.002 (3) |
| C2' | 0.022 (3) | 0.036 (4) | 0.041 (4) | -0.008 (3) | 0.008 (3) | -0.014 (3) |
| C3' | 0.044 (5) | 0.036 (4) | 0.057 (6) | -0.016 (3) | 0.016 (4) | -0.022 (4) |
| C4' | 0.043 (6) | 0.031 (4) | 0.050 (6) | -0.011 (4) | 0.023 (5) | -0.016 (4) |
| C5 | 0.0246 (11) | 0.0182 (10) | 0.0282 (11) | -0.0058 (9) | 0.0018 (9) | -0.0055 (9) |
| C6 | 0.0291 (12) | 0.0237 (11) | 0.0279 (12) | -0.0096 (9) | 0.0047 (9) | -0.0098 (9) |
| C7 | 0.0362 (13) | 0.0282 (12) | 0.0276 (12) | -0.0137 (11) | 0.0043 (10) | -0.0065 (10) |
| C8 | 0.0462 (16) | 0.0477 (16) | 0.0332 (14) | -0.0236 (13) | 0.0140 (12) | -0.0138 (12) |
| C9 | 0.0262 (11) | 0.0193 (11) | 0.0358 (13) | -0.0098 (9) | 0.0044 (10) | -0.0058 (9) |
| C10 | 0.0287 (12) | 0.0214 (12) | 0.0467 (15) | -0.0096 (10) | -0.0042 (11) | -0.0048 (11) |
| C11 | 0.0391 (14) | 0.0257 (12) | 0.0362 (13) | -0.0196 (11) | -0.0023 (11) | -0.0040 (10) |
| C12 | 0.0376 (13) | 0.0255 (12) | 0.0331 (13) | -0.0176 (10) | 0.0083 (10) | -0.0101 (10) |
| C13 | 0.0289 (12) | 0.0236 (11) | 0.0304 (12) | -0.0112 (10) | 0.0088 (10) | -0.0085 (10) |
| C14 | 0.0257 (11) | 0.0199 (11) | 0.0253 (11) | -0.0087 (9) | 0.0052 (9) | -0.0041 (9) |
| C15 | 0.0208 (10) | 0.0189 (11) | 0.0286 (11) | -0.0047 (9) | 0.0054 (9) | -0.0058 (9) |
| C16 | 0.0252 (11) | 0.0211 (11) | 0.0235 (11) | -0.0081 (9) | 0.0075 (9) | -0.0051 (9) |
| C17 | 0.0272 (11) | 0.0206 (11) | 0.0224 (11) | -0.0081 (9) | 0.0070 (9) | -0.0048 (9) |
| C18 | 0.0249 (11) | 0.0217 (11) | 0.0214 (11) | -0.0056 (9) | 0.0054 (9) | -0.0050 (9) |
| C19 | 0.0331 (12) | 0.0248 (11) | 0.0183 (10) | -0.0100 (10) | 0.0064 (9) | -0.0039 (9) |
| C20 | 0.0360 (13) | 0.0290 (13) | 0.0267 (12) | -0.0074 (10) | 0.0035 (10) | -0.0103 (10) |
| C21 | 0.0510 (16) | 0.0361 (14) | 0.0293 (13) | -0.0142 (12) | 0.0016 (12) | -0.0130 (11) |
| C22 | 0.0425 (15) | 0.0428 (15) | 0.0271 (13) | -0.0187 (12) | 0.0000 (11) | -0.0092 (11) |

| | | | | | | |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C23 | 0.0318 (13) | 0.0340 (13) | 0.0252 (12) | -0.0129 (11) | 0.0025 (10) | -0.0038 (10) |
| C24 | 0.0297 (12) | 0.0242 (11) | 0.0217 (11) | -0.0108 (10) | 0.0058 (9) | -0.0022 (9) |
| C25 | 0.0241 (11) | 0.0237 (11) | 0.0302 (12) | -0.0069 (9) | 0.0066 (9) | -0.0058 (9) |
| C26 | 0.0290 (12) | 0.0220 (11) | 0.0281 (12) | -0.0087 (9) | 0.0094 (10) | -0.0084 (9) |

Geometric parameters (Å, °)

| | | | |
|-----------|-------------|-----------|-----------|
| Sn1—O1 | 2.0857 (17) | C5—H5B | 0.9900 |
| Sn1—C1 | 2.089 (15) | C6—C7 | 1.525 (3) |
| Sn1—C5 | 2.126 (2) | C6—H6A | 0.9900 |
| Sn1—O2 | 2.1531 (16) | C6—H6B | 0.9900 |
| Sn1—C1' | 2.20 (2) | C7—C8 | 1.522 (3) |
| Sn1—N1 | 2.1932 (18) | C7—H7A | 0.9900 |
| Br1—C12 | 1.895 (2) | C7—H7B | 0.9900 |
| N1—C15 | 1.296 (3) | C8—H8A | 0.9800 |
| N1—N2 | 1.389 (2) | C8—H8B | 0.9800 |
| N2—C16 | 1.316 (3) | C8—H8C | 0.9800 |
| O1—C9 | 1.319 (3) | C9—C10 | 1.405 (3) |
| O2—C16 | 1.295 (3) | C9—C14 | 1.417 (3) |
| O3—C26 | 1.353 (3) | C10—C11 | 1.373 (4) |
| O3—H3 | 0.830 (10) | C10—H10 | 0.9500 |
| C1—C2 | 1.538 (7) | C11—C12 | 1.389 (3) |
| C1—H1A | 0.9900 | C11—H11 | 0.9500 |
| C1—H1B | 0.9900 | C12—C13 | 1.369 (3) |
| C2—C3 | 1.507 (6) | C13—C14 | 1.415 (3) |
| C2—H2A | 0.9900 | C13—H13 | 0.9500 |
| C2—H2B | 0.9900 | C14—C15 | 1.429 (3) |
| C3—C4 | 1.566 (7) | C15—H15 | 0.9500 |
| C3—H3A | 0.9900 | C16—C17 | 1.474 (3) |
| C3—H3B | 0.9900 | C17—C18 | 1.377 (3) |
| C4—H4A | 0.9800 | C17—C26 | 1.437 (3) |
| C4—H4B | 0.9800 | C18—C19 | 1.408 (3) |
| C4—H4C | 0.9800 | C18—H18 | 0.9500 |
| C1'—C2' | 1.538 (9) | C19—C20 | 1.419 (3) |
| C1'—H1C | 0.9900 | C19—C24 | 1.424 (3) |
| C1'—H1D | 0.9900 | C20—C21 | 1.367 (3) |
| C2'—C3' | 1.516 (8) | C20—H20 | 0.9500 |
| C2'—H2C | 0.9900 | C21—C22 | 1.414 (4) |
| C2'—H2D | 0.9900 | C21—H21 | 0.9500 |
| C3'—C4' | 1.563 (9) | C22—C23 | 1.359 (4) |
| C3'—H3C | 0.9900 | C22—H22 | 0.9500 |
| C3'—H3D | 0.9900 | C23—C24 | 1.422 (3) |
| C4'—H4D | 0.9800 | C23—H23 | 0.9500 |
| C4'—H4E | 0.9800 | C24—C25 | 1.412 (3) |
| C4'—H4F | 0.9800 | C25—C26 | 1.369 (3) |
| C5—C6 | 1.525 (3) | C25—H25 | 0.9500 |
| C5—H5A | 0.9900 | | |
| O1—Sn1—C1 | 90.6 (3) | C7—C6—H6A | 109.2 |
| O1—Sn1—C5 | 97.48 (8) | C5—C6—H6A | 109.2 |

supplementary materials

| | | | |
|-------------|-------------|-------------|-------------|
| C1—Sn1—C5 | 126.0 (2) | C7—C6—H6B | 109.2 |
| O1—Sn1—O2 | 153.97 (6) | C5—C6—H6B | 109.2 |
| C1—Sn1—O2 | 98.7 (3) | H6A—C6—H6B | 107.9 |
| C5—Sn1—O2 | 96.49 (8) | C8—C7—C6 | 112.7 (2) |
| O1—Sn1—C1' | 98.9 (4) | C8—C7—H7A | 109.0 |
| C1—Sn1—C1' | 9.2 (6) | C6—C7—H7A | 109.0 |
| C5—Sn1—C1' | 128.1 (2) | C8—C7—H7B | 109.0 |
| O2—Sn1—C1' | 89.5 (4) | C6—C7—H7B | 109.0 |
| O1—Sn1—N1 | 82.45 (6) | H7A—C7—H7B | 107.8 |
| C1—Sn1—N1 | 128.2 (2) | C7—C8—H8A | 109.5 |
| C5—Sn1—N1 | 105.82 (7) | C7—C8—H8B | 109.5 |
| O2—Sn1—N1 | 72.64 (6) | H8A—C8—H8B | 109.5 |
| C1'—Sn1—N1 | 124.9 (2) | C7—C8—H8C | 109.5 |
| C15—N1—N2 | 115.59 (18) | H8A—C8—H8C | 109.5 |
| C15—N1—Sn1 | 128.43 (15) | H8B—C8—H8C | 109.5 |
| N2—N1—Sn1 | 115.90 (13) | O1—C9—C10 | 118.8 (2) |
| C16—N2—N1 | 112.44 (17) | O1—C9—C14 | 123.1 (2) |
| C9—O1—Sn1 | 133.15 (15) | C10—C9—C14 | 118.1 (2) |
| C16—O2—Sn1 | 115.23 (14) | C11—C10—C9 | 121.5 (2) |
| C26—O3—H3 | 110 (2) | C11—C10—H10 | 119.3 |
| C2—C1—Sn1 | 114.9 (8) | C9—C10—H10 | 119.3 |
| C2—C1—H1A | 108.5 | C10—C11—C12 | 120.1 (2) |
| Sn1—C1—H1A | 108.5 | C10—C11—H11 | 119.9 |
| C2—C1—H1B | 108.5 | C12—C11—H11 | 119.9 |
| Sn1—C1—H1B | 108.5 | C13—C12—C11 | 120.5 (2) |
| H1A—C1—H1B | 107.5 | C13—C12—Br1 | 120.52 (18) |
| C3—C2—C1 | 113.6 (6) | C11—C12—Br1 | 118.98 (18) |
| C3—C2—H2A | 108.8 | C12—C13—C14 | 120.4 (2) |
| C1—C2—H2A | 108.8 | C12—C13—H13 | 119.8 |
| C3—C2—H2B | 108.8 | C14—C13—H13 | 119.8 |
| C1—C2—H2B | 108.8 | C13—C14—C9 | 119.4 (2) |
| H2A—C2—H2B | 107.7 | C13—C14—C15 | 116.9 (2) |
| C2—C3—C4 | 110.1 (6) | C9—C14—C15 | 123.7 (2) |
| C2—C3—H3A | 109.6 | N1—C15—C14 | 126.7 (2) |
| C4—C3—H3A | 109.6 | N1—C15—H15 | 116.7 |
| C2—C3—H3B | 109.6 | C14—C15—H15 | 116.7 |
| C4—C3—H3B | 109.6 | O2—C16—N2 | 123.8 (2) |
| H3A—C3—H3B | 108.2 | O2—C16—C17 | 118.5 (2) |
| C2'—C1'—Sn1 | 111.5 (10) | N2—C16—C17 | 117.68 (19) |
| C2'—C1'—H1C | 109.3 | C18—C17—C26 | 118.6 (2) |
| Sn1—C1'—H1C | 109.3 | C18—C17—C16 | 118.82 (19) |
| C2'—C1'—H1D | 109.3 | C26—C17—C16 | 122.6 (2) |
| Sn1—C1'—H1D | 109.3 | C17—C18—C19 | 122.5 (2) |
| H1C—C1'—H1D | 108.0 | C17—C18—H18 | 118.7 |
| C3'—C2'—C1' | 112.8 (8) | C19—C18—H18 | 118.7 |
| C3'—C2'—H2C | 109.0 | C18—C19—C20 | 122.1 (2) |
| C1'—C2'—H2C | 109.0 | C18—C19—C24 | 118.3 (2) |
| C3'—C2'—H2D | 109.0 | C20—C19—C24 | 119.6 (2) |
| C1'—C2'—H2D | 109.0 | C21—C20—C19 | 120.5 (2) |

| | | | |
|----------------|--------------|-----------------|--------------|
| H2C—C2'—H2D | 107.8 | C21—C20—H20 | 119.7 |
| C2'—C3'—C4' | 109.0 (7) | C19—C20—H20 | 119.7 |
| C2'—C3'—H3C | 109.9 | C20—C21—C22 | 119.6 (2) |
| C4'—C3'—H3C | 109.9 | C20—C21—H21 | 120.2 |
| C2'—C3'—H3D | 109.9 | C22—C21—H21 | 120.2 |
| C4'—C3'—H3D | 109.9 | C23—C22—C21 | 121.5 (2) |
| H3C—C3'—H3D | 108.3 | C23—C22—H22 | 119.3 |
| C3'—C4'—H4D | 109.5 | C21—C22—H22 | 119.3 |
| C3'—C4'—H4E | 109.5 | C22—C23—C24 | 120.4 (2) |
| H4D—C4'—H4E | 109.5 | C22—C23—H23 | 119.8 |
| C3'—C4'—H4F | 109.5 | C24—C23—H23 | 119.8 |
| H4D—C4'—H4F | 109.5 | C25—C24—C23 | 122.6 (2) |
| H4E—C4'—H4F | 109.5 | C25—C24—C19 | 119.1 (2) |
| C6—C5—Sn1 | 113.65 (14) | C23—C24—C19 | 118.3 (2) |
| C6—C5—H5A | 108.8 | C26—C25—C24 | 121.6 (2) |
| Sn1—C5—H5A | 108.8 | C26—C25—H25 | 119.2 |
| C6—C5—H5B | 108.8 | C24—C25—H25 | 119.2 |
| Sn1—C5—H5B | 108.8 | O3—C26—C25 | 118.2 (2) |
| H5A—C5—H5B | 107.7 | O3—C26—C17 | 121.9 (2) |
| C7—C6—C5 | 112.22 (19) | C25—C26—C17 | 119.9 (2) |
| O1—Sn1—N1—C15 | -9.94 (19) | C9—C10—C11—C12 | -1.4 (4) |
| C1—Sn1—N1—C15 | -94.8 (4) | C10—C11—C12—C13 | 0.8 (4) |
| C5—Sn1—N1—C15 | 85.7 (2) | C10—C11—C12—Br1 | -178.50 (18) |
| O2—Sn1—N1—C15 | 177.7 (2) | C11—C12—C13—C14 | 0.6 (3) |
| C1'—Sn1—N1—C15 | -105.6 (5) | Br1—C12—C13—C14 | 179.89 (16) |
| O1—Sn1—N1—N2 | 173.53 (15) | C12—C13—C14—C9 | -1.4 (3) |
| C1—Sn1—N1—N2 | 88.7 (4) | C12—C13—C14—C15 | 179.8 (2) |
| C5—Sn1—N1—N2 | -90.79 (15) | O1—C9—C14—C13 | -177.6 (2) |
| O2—Sn1—N1—N2 | 1.18 (13) | C10—C9—C14—C13 | 0.8 (3) |
| C1'—Sn1—N1—N2 | 77.9 (5) | O1—C9—C14—C15 | 1.1 (4) |
| C15—N1—N2—C16 | -178.12 (19) | C10—C9—C14—C15 | 179.5 (2) |
| Sn1—N1—N2—C16 | -1.1 (2) | N2—N1—C15—C14 | 178.17 (19) |
| C1—Sn1—O1—C9 | 146.5 (3) | Sn1—N1—C15—C14 | 1.6 (3) |
| C5—Sn1—O1—C9 | -87.0 (2) | C13—C14—C15—N1 | -175.1 (2) |
| O2—Sn1—O1—C9 | 34.9 (3) | C9—C14—C15—N1 | 6.1 (4) |
| C1'—Sn1—O1—C9 | 142.4 (3) | Sn1—O2—C16—N2 | 0.9 (3) |
| N1—Sn1—O1—C9 | 18.1 (2) | Sn1—O2—C16—C17 | -178.28 (14) |
| O1—Sn1—O2—C16 | -18.6 (2) | N1—N2—C16—O2 | 0.2 (3) |
| C1—Sn1—O2—C16 | -128.4 (3) | N1—N2—C16—C17 | 179.36 (17) |
| C5—Sn1—O2—C16 | 103.52 (16) | O2—C16—C17—C18 | -0.9 (3) |
| C1'—Sn1—O2—C16 | -128.1 (3) | N2—C16—C17—C18 | 179.84 (19) |
| N1—Sn1—O2—C16 | -1.07 (14) | O2—C16—C17—C26 | 177.4 (2) |
| O1—Sn1—C1—C2 | 137.0 (6) | N2—C16—C17—C26 | -1.8 (3) |
| C5—Sn1—C1—C2 | 37.3 (8) | C26—C17—C18—C19 | -0.9 (3) |
| O2—Sn1—C1—C2 | -67.4 (7) | C16—C17—C18—C19 | 177.48 (19) |
| C1'—Sn1—C1—C2 | -69 (3) | C17—C18—C19—C20 | -179.3 (2) |
| N1—Sn1—C1—C2 | -142.1 (5) | C17—C18—C19—C24 | 0.1 (3) |
| Sn1—C1—C2—C3 | -59.5 (10) | C18—C19—C20—C21 | 177.6 (2) |
| C1—C2—C3—C4 | -171.9 (8) | C24—C19—C20—C21 | -1.8 (3) |

supplementary materials

| | | | |
|-----------------|--------------|-----------------|------------|
| O1—Sn1—C1'—C2' | 85.7 (8) | C19—C20—C21—C22 | 1.3 (4) |
| C1—Sn1—C1'—C2' | 59 (2) | C20—C21—C22—C23 | 0.2 (4) |
| C5—Sn1—C1'—C2' | -21.2 (11) | C21—C22—C23—C24 | -1.2 (4) |
| O2—Sn1—C1'—C2' | -119.1 (8) | C22—C23—C24—C25 | -178.8 (2) |
| N1—Sn1—C1'—C2' | 172.7 (6) | C22—C23—C24—C19 | 0.6 (3) |
| Sn1—C1'—C2'—C3' | 67.2 (12) | C18—C19—C24—C25 | 0.9 (3) |
| C1'—C2'—C3'—C4' | -178.6 (11) | C20—C19—C24—C25 | -179.7 (2) |
| O1—Sn1—C5—C6 | 68.73 (16) | C18—C19—C24—C23 | -178.6 (2) |
| C1—Sn1—C5—C6 | 165.0 (4) | C20—C19—C24—C23 | 0.8 (3) |
| O2—Sn1—C5—C6 | -89.24 (16) | C23—C24—C25—C26 | 178.5 (2) |
| C1'—Sn1—C5—C6 | 176.3 (5) | C19—C24—C25—C26 | -1.0 (3) |
| N1—Sn1—C5—C6 | -15.50 (18) | C24—C25—C26—O3 | 179.3 (2) |
| Sn1—C5—C6—C7 | -179.63 (15) | C24—C25—C26—C17 | 0.1 (3) |
| C5—C6—C7—C8 | -179.5 (2) | C18—C17—C26—O3 | -178.3 (2) |
| Sn1—O1—C9—C10 | 164.47 (17) | C16—C17—C26—O3 | 3.3 (3) |
| Sn1—O1—C9—C14 | -17.1 (3) | C18—C17—C26—C25 | 0.8 (3) |
| O1—C9—C10—C11 | 179.1 (2) | C16—C17—C26—C25 | -177.5 (2) |
| C14—C9—C10—C11 | 0.6 (4) | | |

Hydrogen-bond geometry (\AA , $^\circ$)

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-------------------|----------|-------------|-------------|---------------|
| O3—H3 \cdots N2 | 0.83 (1) | 1.88 (2) | 2.606 (2) | 146 (3) |

Fig. 1

