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## Structure Reports

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## \{2-[(Dimethylamino)methyl]phen-yl\}bis(4-methylphenyl)bismuthane

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Received 4 October 2010; accepted 26 October 2010
Key indicators: single-crystal X-ray study; $T=100 \mathrm{~K}$; mean $\sigma(\mathrm{C}-\mathrm{C})=0.008 \AA$; $R$ factor $=0.019 ; w R$ factor $=0.037$; data-to-parameter ratio $=15.7$.

The title compound, $\left[\mathrm{Bi}\left(\mathrm{C}_{7} \mathrm{H}_{7}\right)_{2}\left(\mathrm{C}_{9} \mathrm{H}_{12} \mathrm{~N}\right)\right]$, was obtained by treating chlorodi( $p$-tolyl)bismuthane with o-lithio- $\mathrm{N}, \mathrm{N}$ dimethylbenzylamine. An intramolecular $\mathrm{Bi} \cdots \mathrm{N}$ nonbonding interaction is observed in the distorted trigonal triarylbismuth coordination of the title compound.

## Related literature

For a review of the applications and structural chemistry of organobismuth compounds, see: Matano \& Ikegami (2001); Silvestru et al. (1999). For related structural reports, see: Suzuki et al. (1993); Tokunaga et al. (2000a,b); Okajima et al. (2002).


## Experimental

## Crystal data

$\left[\mathrm{Bi}\left(\mathrm{C}_{7} \mathrm{H}_{7}\right)_{2}\left(\mathrm{C}_{9} \mathrm{H}_{12} \mathrm{~N}\right)\right]$
$M_{r}=525.43$
Monoclinic, $P 2_{1}$
$a=6.0991$ (12) £
$b=19.630$ (4) $\AA$
$c=8.3699(16) \AA$
$\beta=93.073(2)^{\circ}$
$V=1000.6(3) \AA^{3}$
$Z=2$
Mo $K \alpha$ radiation
$\mu=8.81 \mathrm{~mm}^{-1}$
$T=100 \mathrm{~K}$
$0.20 \times 0.08 \times 0.01 \mathrm{~mm}$

## Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
$T_{\text {min }}=0.272, T_{\text {max }}=0.917$

4908 measured reflections 3626 independent reflections 3410 reflections with $I>2 \sigma(I)$ $R_{\text {int }}=0.016$

## Refinement

$R\left[F^{2}>2 \sigma\left(F^{2}\right)\right]=0.019$
H -atom parameters constrained
$w R\left(F^{2}\right)=0.037$
$\Delta \rho_{\text {max }}=0.86$ e $\AA^{-3}$
$S=1.00$
3626 reflections
231 parameters
Absolute structure: Flack (1983),
with 1544 Friedel pairs
1 restraint

Flack parameter: 0.412 (7)

Table 1
Selected geometric parameters ( $\left(\mathrm{A},{ }^{\circ}\right)$.

| C1-Bi1 | $2.291(5)$ | C15-Bi1 | $2.267(5)$ |
| :--- | ---: | :--- | :--- |
| C8-Bi1 | $2.265(5)$ | N1-Bi1 | $2.902(4)$ |
|  |  |  |  |
| C1-Bi1-C8 | $96.07(16)$ | C8-Bi1-C15 | $94.85(17)$ |
| C1-Bi1-C15 | $90.74(16)$ | C8-Bi1-N1 | $81.26(14)$ |
| C1-Bi1-N1 | $157.55(14)$ | C15-Bi1-N1 | $67.43(13)$ |

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT (Bruker, 2005); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: WinGX (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the

Okajima, S., Yasuike, S., Kakuswa, N., Osada, A., Yamaguchi, K., Seki, H. \&

[^0]IUCr electronic archives (Reference: SI2298).

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## supporting information

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\{2-[(Dimethylamino)methyl]phenyl\}bis(4-methylphenyl)bismuthane

Masatoshi Kawahata, Shuji Yasuike, Izumi Kinebuchi, Kentaro Yamaguchi and Jyoji Kurita

## S1. Comment

Interest in the chemistry of organobisumuth(III) compounds has increased in recent years, due to the potential reagents and catalysts in organic synthesis as well as biological activity (Matano \& Ikegami, 2001). Among these, the structural chemistry of bismuth compounds, including intramolecular interaction between bismuth and heteroatoms, has been widely reported in a review (Silvestru et al. 1999). On the other hand, we have recently reported the synthesis and structure of various organoantimony(III) compounds, such as 1-[8-( $N, N$-dimethylaminomethyl)naphthyl]bis(4-methylphenyl)stibane (Tokunaga et al., 2000a), [2-(N,N-dimethylaminomethyl)phenyl]bis(4-methylphenyl)stibane (Tokunaga et al., 2000b), and $\operatorname{Sb}(S)-[2-(S)-(N, N$-dimethylaminomethyl)phenyl](1-naphthyl)(4-methylphenyl)stibane (Okajima et al., 2002), bearing the $\mathrm{CH}_{2} \mathrm{NMe}_{2}$ moiety adjacent to the Sb atom as a pendant arm. X-ray crystal analyses of these compounds revealed the presence of intramolecular coordination between the Sb and N atoms. Here we report the synthesis and structure of the title compound, in which the central Sb atom of the [2-( $N, N$-dimethylaminomethyl)phenyl]-bis(4-methylphenyl)stibane is replaced with Bi atom. The molecular structure and atom-numbering of the title compound are shown in Fig. 1. Selected geometric parameters are presented in Table 1. The analysis revealed that the Bi and three C ( $\mathrm{C} 1, \mathrm{C} 8$, and C 15 ) atoms exhibit a distorted trigonal-pyramidal arrangement with the Bi atom being far from the basal three-carbon plane (1.220 (3) $\AA$ ). In addition, an intramolecular coordination between the Bi and N atoms is observed; the distance between the Bi and N atoms is 2.902 (4) $\AA$, which corresponds to $74 \%$ of the sum of the van der Waals radii of both elements ( $3.94 \AA$ ) and accords with $131 \%$ of the covalent bond length ( $2.22 \AA$ ). It should be noted that the bond angle for $\mathrm{C} 1 — \mathrm{Bi} 1 — \mathrm{~N} 1\left[157.55(14)^{\circ}\right]$ is significantly larger than those for $\mathrm{C} 8 — \mathrm{Bi} 1 — \mathrm{~N} 1\left[81.26(14)^{\circ}\right]$ and $\mathrm{C} 15 — \mathrm{Bi} 1-$ $\mathrm{N} 1\left[67.43(13)^{\circ}\right]$, and the bond distance between Bi1 and $\mathrm{C} 1[2.291(5) \AA$ ] is obviously longer than those for Bi1-C8 [2.265 (5) $\AA$ ] and C15 [2.267 (5) $\AA$ ]. The results imply that the central Bi atom is distorted equatorial vacant trigonal bipyramidal configuration with the N 1 of the pendant arm and the C 1 of the tolyl group being apical positions, similar to the geometry of chloro[2-(N,N-dimethylaminomethyl)phenyl](4-methylphenyl)bismuthane (Suzuki et al., 1993). These results showed that the title compound is a hypervalent compound with $10-\mathrm{Bi}-4$ system, by analogy with the $10-\mathrm{Sb}-4$ system of the organoantimony compounds (Tokunaga et al., 2000a,b; Okajima et al., 2002).

## S2. Experimental

The title compound was synthesized as follows: To a solution of $N, N$-dimethylbenzylamine ( $1.89 \mathrm{~g}, 14.0 \mathrm{mmol}$ ) in ether $(25 \mathrm{ml})$ was added n-butyllithium ( 1.65 M in hexane, $10.2 \mathrm{ml}, 16.8 \mathrm{mmol}$ ) at 273 K under an argon atmosphere, and the mixture was stirred for 24 h at room temperature. To this solution was added a suspension of chlorobis(4-methylphenyl)bismuthane [prepared by the redistribution reaction on the treatment of tris(4-methylphenyl)bismuthane ( $2.31 \mathrm{~g}, 4.8$ $\mathrm{mmol})$ and trichlorobismuthane ( $756 \mathrm{mg}, 2.4 \mathrm{mmol}$ ) in ether $(20 \mathrm{ml})$ at room temperature for 2 h ] over 10 min at 273 K , and the mixture was stirred for 24 h at the same temperature. The mixture was quenched with water $(100 \mathrm{ml})$ and diluted with $\mathrm{CH}_{2} \mathrm{Cl}_{2}(100 \mathrm{ml})$, and insoluble substances were removed by filtration. The organic layer was separated and the
aqueous layer was extracted with $\mathrm{CH}_{2} \mathrm{Cl}_{2}(50 \mathrm{ml})$. The combined organic layer was washed with brine, dried and evaporated in vacuo. Purification of the residue by recrystallization from $\mathrm{CH}_{3} \mathrm{CN}$ gave 2-( $N, N$-dimethylaminomethyl)-phenylbis(4-methylphenyl)bismuthane as colourless prisms $\left(2.0 \mathrm{~g}, 53 \%\right.$ yield; m.p. $372-374 \mathrm{~K} ;{ }^{1} \mathrm{H}$ NMR $\left(\mathrm{CDCl}_{3}\right): \delta 1.98$ $(\mathrm{s}, 6 \mathrm{H}), 2.30(\mathrm{~s}, 6 \mathrm{H}), 3.40(\mathrm{~s}, 2 \mathrm{H}), 7.14(\mathrm{~d}, J=7.3 \mathrm{~Hz}, 4 \mathrm{H}), 7.16(\mathrm{~m}, 1 \mathrm{H}), 7.25(\mathrm{~m}, 2 \mathrm{H}), 7.62(\mathrm{~d}, J=7.3 \mathrm{~Hz}, 4 \mathrm{H}), 7.80(\mathrm{~d}$, $J=6.9 \mathrm{~Hz}, 1 \mathrm{H}) ;{ }^{13} \mathrm{C}$ NMR $\left(\mathrm{CDCl}_{3}\right): \delta 21.5(q), 44.5(q), 67.1(t), 127.2(d), 129.3(d), 129.6(d), 130.8(d), 136.5(s)$, $137.7(d), 139.6(d), 145.1(s), 155.9(s), 158.5(s)$; analysis calculated for $\mathrm{C}_{23} \mathrm{H}_{26} \mathrm{BiN}$ : C 52.58, H 4.99, N 2.67\%; found: C 52.57, H 4.92, N $2.63 \%$.

## S3. Refinement

The H atoms were positioned geometrically $(\mathrm{C}-\mathrm{H}=0.93-0.96 \AA)$ and were included in the refinement in the riding model approximation, with $U_{\text {iso }}(\mathrm{H})=x U_{\mathrm{eq}}(\mathrm{C}, \mathrm{N})$, where $x=1.5$ for methyl and $x=1.2$ for all other H atoms. The crystal studied was a twin with the refined BASF ratio of 0.412 (7)/0.588 (7). The Flack parameter $=0.412$ (7) was refined in the full matrix least-squares process using the TWIN/BASF option.


Figure 1
View of the title compound with the atom numbering scheme. The thermal ellipsoids are drawn at the $50 \%$ probability level. H atoms are shown as small spheres of arbitrary radii.

## \{2-[(Dimethylamino)methyl]phenyl\}bis(4-methylphenyl)bismuthane

## Crystal data

$\left[\mathrm{Bi}\left(\mathrm{C}_{7} \mathrm{H}_{7}\right)_{2}\left(\mathrm{C}_{9} \mathrm{H}_{12} \mathrm{~N}\right)\right]$
$M_{r}=525.43$
Monoclinic, $P 2_{1}$
Hall symbol: P2yb
$a=6.0991$ (12) $\AA$
$b=19.630(4) \AA$

$$
\begin{aligned}
& c=8.3699(16) \AA \\
& \beta=93.073(2)^{\circ} \\
& V=1000.6(3) \AA^{3} \\
& Z=2 \\
& F(000)=508 \\
& D_{\mathrm{x}}=1.744 \mathrm{Mg} \mathrm{~m}^{-3}
\end{aligned}
$$

Mo $K \alpha$ radiation, $\lambda=0.71073 \AA$
Cell parameters from 3137 reflections
$\theta=2.4-26.9^{\circ}$
$\mu=8.81 \mathrm{~mm}^{-1}$

## Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan
(SADABS; Sheldrick, 1996)
$T_{\text {min }}=0.272, T_{\text {max }}=0.917$

## Refinement

Refinement on $F^{2}$
Least-squares matrix: full
$R\left[F^{2}>2 \sigma\left(F^{2}\right)\right]=0.019$
$w R\left(F^{2}\right)=0.037$
$S=1.00$
3626 reflections
231 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
$T=100 \mathrm{~K}$
Prismatic, colourless
$0.20 \times 0.08 \times 0.01 \mathrm{~mm}$

4908 measured reflections
3626 independent reflections
3410 reflections with $I>2 \sigma(I)$
$R_{\text {int }}=0.016$
$\theta_{\text {max }}=27.0^{\circ}, \theta_{\text {min }}=2.1^{\circ}$
$h=-7 \rightarrow 7$
$k=-22 \rightarrow 24$
$l=-10 \rightarrow 5$

Hydrogen site location: inferred from neighbouring sites
H -atom parameters constrained
$w=1 /\left[\sigma^{2}\left(F_{0}^{2}\right)+(0.0116 P)^{2}\right]$
where $P=\left(F_{0}^{2}+2 F_{\mathrm{c}}{ }^{2}\right) / 3$
$(\Delta / \sigma)_{\max }=0.001$
$\Delta \rho_{\text {max }}=0.86$ e $\AA^{-3}$
$\Delta \rho_{\text {min }}=-0.59$ e $\AA^{-3}$
Absolute structure: Flack (1983), with 1544
Friedel pairs
Absolute structure parameter: 0.412 (7)

## 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 $F^{2}>2 \sigma\left(F^{2}\right)$ 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 ( $\hat{A}^{2}$ )

|  | $x$ | $y$ | $z$ | $U_{\text {iso }} * / U_{\text {eq }}$ |
| :--- | :--- | :--- | :--- | :--- |
| C1 | $0.6751(8)$ | $0.6439(2)$ | $0.4839(6)$ | $0.0163(10)$ |
| C2 | $0.8412(8)$ | $0.6254(3)$ | $0.5966(6)$ | $0.0230(12)$ |
| C3 | $0.8193(8)$ | $0.5693(3)$ | $0.6955(6)$ | $0.0232(12)$ |
| C4 | $0.6268(8)$ | $0.5298(2)$ | $0.6864(6)$ | $0.0204(11)$ |
| C5 | $0.4595(8)$ | $0.5495(2)$ | $0.5785(6)$ | $0.0216(11)$ |
| C6 | $0.4845(8)$ | $0.6055(2)$ | $0.4787(6)$ | $0.0199(11)$ |
| C7 | $0.6102(9)$ | $0.4657(2)$ | $0.7843(6)$ | $0.0261(12)$ |
| C8 | $0.9474(8)$ | $0.7916(2)$ | $0.4269(6)$ | $0.0169(11)$ |
| C9 | $0.8880(8)$ | $0.8577(2)$ | $0.4634(6)$ | $0.0181(11)$ |
| C10 | $1.0307(8)$ | $0.9002(2)$ | $0.5535(6)$ | $0.0185(11)$ |
| C11 | $1.2387(8)$ | $0.8775(2)$ | $0.6077(6)$ | $0.0158(10)$ |


| C12 | 1.2973 (8) | 0.8115 (2) | 0.5681 (6) | 0.0190 (11) |
| :---: | :---: | :---: | :---: | :---: |
| C13 | 1.1561 (8) | 0.7696 (2) | 0.4794 (6) | 0.0176 (11) |
| C14 | 1.3902 (8) | 0.9223 (2) | 0.7087 (6) | 0.0240 (12) |
| C15 | 0.9686 (7) | 0.6605 (2) | 0.1743 (5) | 0.0134 (10) |
| C16 | 0.9732 (8) | 0.5895 (2) | 0.2016 (6) | 0.0197 (11) |
| C17 | 1.1305 (9) | 0.5485 (3) | 0.1324 (6) | 0.0248 (12) |
| C18 | 1.2779 (9) | 0.5775 (3) | 0.0349 (7) | 0.0286 (13) |
| C19 | 1.2694 (8) | 0.6474 (3) | 0.0021 (6) | 0.0220 (11) |
| C20 | 1.1185 (18) | 0.6885 (5) | 0.0734 (12) | 0.019 (2) |
| C21 | 1.1160 (17) | 0.7642 (5) | 0.0378 (11) | 0.0148 (19) |
| C22 | 0.9016 (9) | 0.8666 (2) | 0.0186 (6) | 0.0234 (12) |
| C23 | 0.7809 (8) | 0.7669 (2) | -0.1263 (6) | 0.0248 (12) |
| N1 | 0.8969 (6) | 0.79253 (18) | 0.0204 (5) | 0.0154 (9) |
| Bil | 0.70642 (2) | 0.72364 (2) | 0.286635 (16) | 0.01465 (4) |
| H2 | 0.9692 | 0.6512 | 0.6057 | 0.028* |
| H3 | 0.9333 | 0.5577 | 0.7687 | 0.028* |
| H5 | 0.3285 | 0.5251 | 0.5724 | 0.026* |
| H6 | 0.3698 | 0.6174 | 0.4065 | 0.024* |
| H7A | 0.6739 | 0.4284 | 0.7290 | 0.039* |
| H7B | 0.4586 | 0.4560 | 0.8001 | 0.039* |
| H7C | 0.6874 | 0.4719 | 0.8863 | 0.039* |
| H9 | 0.7511 | 0.8741 | 0.4273 | 0.022* |
| H10 | 0.9866 | 0.9442 | 0.5778 | 0.022* |
| H12 | 1.4351 | 0.7952 | 0.6022 | 0.023* |
| H13 | 1.2011 | 0.7258 | 0.4542 | 0.021* |
| H14A | 1.3614 | 0.9164 | 0.8195 | 0.036* |
| H14B | 1.3662 | 0.9691 | 0.6790 | 0.036* |
| H14C | 1.5398 | 0.9102 | 0.6922 | 0.036* |
| H16 | 0.8711 | 0.5698 | 0.2661 | 0.024* |
| H17 | 1.1347 | 0.5019 | 0.1524 | 0.030* |
| H18 | 1.3842 | 0.5506 | -0.0097 | 0.034* |
| H19 | 1.3657 | 0.6663 | -0.0680 | 0.026* |
| H21A | 1.1975 | 0.7878 | 0.1237 | 0.018* |
| H21B | 1.1904 | 0.7721 | -0.0600 | 0.018* |
| H22A | 0.9813 | 0.8819 | -0.0705 | 0.035* |
| H22B | 0.9724 | 0.8829 | 0.1163 | 0.035* |
| H22C | 0.7541 | 0.8837 | 0.0086 | 0.035* |
| H23A | 0.8557 | 0.7820 | -0.2180 | 0.037* |
| H23B | 0.6332 | 0.7840 | -0.1326 | 0.037* |
| H23C | 0.7782 | 0.7180 | -0.1241 | 0.037* |

Atomic displacement parameters $\left(\hat{A}^{2}\right)$

|  | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
| :--- | :--- | :--- | :--- | :--- | :--- | :--- |
| C1 | $0.019(3)$ | $0.015(2)$ | $0.015(3)$ | $0.001(2)$ | $0.003(2)$ | $0.000(2)$ |
| C2 | $0.019(3)$ | $0.030(3)$ | $0.019(3)$ | $-0.007(2)$ | $0.002(2)$ | $-0.002(2)$ |
| C3 | $0.024(3)$ | $0.028(3)$ | $0.018(3)$ | $0.005(2)$ | $-0.002(2)$ | $0.004(2)$ |
| C4 | $0.021(3)$ | $0.022(3)$ | $0.018(3)$ | $0.004(2)$ | $0.007(2)$ | $0.001(2)$ |


| C5 | $0.018(3)$ | $0.021(3)$ | $0.027(3)$ | $-0.006(2)$ | $0.007(2)$ | $0.001(2)$ |
| :--- | :--- | :--- | :--- | :--- | :--- | :--- |
| C6 | $0.017(3)$ | $0.019(3)$ | $0.024(3)$ | $0.004(2)$ | $-0.001(2)$ | $0.002(2)$ |
| C7 | $0.034(3)$ | $0.018(3)$ | $0.026(3)$ | $0.002(2)$ | $0.009(3)$ | $0.002(2)$ |
| C8 | $0.020(3)$ | $0.014(2)$ | $0.017(3)$ | $-0.0034(19)$ | $0.004(2)$ | $-0.0005(19)$ |
| C9 | $0.020(3)$ | $0.015(2)$ | $0.019(3)$ | $0.002(2)$ | $-0.001(2)$ | $0.001(2)$ |
| C10 | $0.027(3)$ | $0.014(2)$ | $0.016(3)$ | $0.001(2)$ | $0.007(2)$ | $0.000(2)$ |
| C11 | $0.020(3)$ | $0.016(2)$ | $0.011(3)$ | $-0.005(2)$ | $0.002(2)$ | $0.0015(19)$ |
| C12 | $0.017(3)$ | $0.021(3)$ | $0.019(3)$ | $0.003(2)$ | $-0.001(2)$ | $0.002(2)$ |
| C13 | $0.022(3)$ | $0.015(2)$ | $0.016(3)$ | $0.000(2)$ | $0.004(2)$ | $-0.0033(19)$ |
| C14 | $0.025(3)$ | $0.023(3)$ | $0.024(3)$ | $-0.005(2)$ | $0.004(2)$ | $-0.006(2)$ |
| C15 | $0.013(2)$ | $0.015(2)$ | $0.013(3)$ | $0.0007(19)$ | $0.001(2)$ | $-0.0001(19)$ |
| C16 | $0.026(3)$ | $0.017(3)$ | $0.015(3)$ | $-0.004(2)$ | $-0.004(2)$ | $-0.002(2)$ |
| C17 | $0.031(3)$ | $0.017(3)$ | $0.026(3)$ | $0.003(2)$ | $-0.005(3)$ | $-0.003(2)$ |
| C18 | $0.025(3)$ | $0.031(3)$ | $0.030(3)$ | $0.006(2)$ | $0.001(3)$ | $-0.011(2)$ |
| C19 | $0.021(3)$ | $0.026(3)$ | $0.019(3)$ | $-0.006(2)$ | $0.002(2)$ | $-0.002(2)$ |
| C20 | $0.017(4)$ | $0.020(4)$ | $0.020(5)$ | $0.000(3)$ | $-0.012(3)$ | $-0.004(3)$ |
| C21 | $0.013(4)$ | $0.017(4)$ | $0.015(4)$ | $0.000(3)$ | $0.007(3)$ | $-0.001(3)$ |
| C22 | $0.032(3)$ | $0.019(3)$ | $0.019(3)$ | $-0.001(2)$ | $0.004(2)$ | $0.006(2)$ |
| C23 | $0.027(3)$ | $0.025(3)$ | $0.022(3)$ | $0.004(2)$ | $0.001(2)$ | $0.006(2)$ |
| N1 | $0.013(2)$ | $0.016(2)$ | $0.017(2)$ | $-0.0011(16)$ | $0.0037(17)$ | $0.0023(16)$ |
| Bi1 | $0.01336(7)$ | $0.01541(7)$ | $0.01517(7)$ | $0.0000(2)$ | $0.00051(5)$ | $-0.0003(2)$ |

Geometric parameters ( $\mathrm{A},{ }^{\circ}$ )

| C1-C6 | $1.384(6)$ | $\mathrm{C} 14-\mathrm{H} 14 \mathrm{~A}$ | 0.9600 |
| :--- | :--- | :--- | :--- |
| C1-C2 | $1.394(6)$ | $\mathrm{C} 14-\mathrm{H} 14 \mathrm{~B}$ | 0.9600 |
| C1-Bi1 | $2.291(5)$ | $\mathrm{C} 14-\mathrm{H} 14 \mathrm{C}$ | 0.9600 |
| C2-C3 | $1.389(7)$ | $\mathrm{C} 15-\mathrm{C} 20$ | $1.391(12)$ |
| C2-H2 | 0.9300 | $\mathrm{C} 15-\mathrm{C} 16$ | $1.412(6)$ |
| C3-C4 | $1.405(7)$ | $\mathrm{C} 15-\mathrm{Bi1}$ | $2.267(5)$ |
| C3-H3 | 0.9300 | $\mathrm{C} 16-\mathrm{C} 17$ | $1.401(7)$ |
| C4-C5 | $1.381(7)$ | $\mathrm{C} 16-\mathrm{H} 16$ | 0.9300 |
| C4-C7 | $1.509(7)$ | C17-C18 | $1.370(7)$ |
| C5-C6 | $1.394(7)$ | C17-H17 | 0.9300 |
| C5-H5 | 0.9300 | C18-C19 | $1.400(7)$ |
| C6-H6 | 0.9300 | C18-H18 | 0.9300 |
| C7-H7A | 0.9600 | C19-C20 | $1.383(11)$ |
| C7-H7B | 0.9600 | C19-H19 | 0.9300 |
| C7-H7C | 0.9600 | C20-C21 | $1.515(7)$ |
| C8-C9 | $1.386(6)$ | C21-N1 | $1.448(10)$ |
| C8-C13 | $1.393(6)$ | C21-H21A | 0.9700 |
| C8-Bi1 | $2.265(5)$ | C21-H21B | 0.9700 |
| C9-C10 | $1.397(6)$ | C22-N1 | $1.454(6)$ |
| C9-H9 | 0.9300 | C22-H22A | 0.9600 |
| C10-C11 | $1.397(6)$ | C22-H22B | 0.9600 |
| C10-H10 | 0.9300 | C22-H22C | 0.9600 |
| C11-C12 | $1.388(6)$ | C23-N1 | $1.473(6)$ |
| C11-C14 | $1.503(6)$ | C23-H23A | 0.9600 |


| C12-C13 | 1.379 (6) | C23-H23B | 0.9600 |
| :---: | :---: | :---: | :---: |
| C12-H12 | 0.9300 | C23-H23C | 0.9600 |
| C13-H13 | 0.9300 | N1—Bil | 2.902 (4) |
| C6- $\mathrm{C} 1-\mathrm{C} 2$ | 117.3 (4) | C20-C15-C16 | 118.7 (6) |
| C6-C1-Bi1 | 116.8 (3) | C20-C15-Bil | 122.5 (5) |
| C2-C1-Bi1 | 125.4 (4) | C16-C15-Bi1 | 118.8 (4) |
| $\mathrm{C} 3-\mathrm{C} 2-\mathrm{C} 1$ | 121.3 (5) | C17-C16-C15 | 120.5 (5) |
| C3-C2-H2 | 119.4 | C17-C16-H16 | 119.7 |
| $\mathrm{C} 1-\mathrm{C} 2-\mathrm{H} 2$ | 119.4 | C15-C16-H16 | 119.7 |
| C2-C3-C4 | 120.8 (5) | C18-C17-C16 | 119.6 (5) |
| C2-C3-H3 | 119.6 | C18-C17-H17 | 120.2 |
| $\mathrm{C} 4-\mathrm{C} 3-\mathrm{H} 3$ | 119.6 | C16-C17-H17 | 120.2 |
| C5-C4-C3 | 117.9 (5) | C17-C18-C19 | 120.3 (5) |
| C5-C4-C7 | 121.3 (5) | C17-C18-H18 | 119.8 |
| C3-C4-C7 | 120.7 (4) | C19-C18-H18 | 119.8 |
| C4-C5-C6 | 120.7 (5) | C20-C19-C18 | 120.4 (6) |
| C4-C5-H5 | 119.6 | C20-C19-H19 | 119.8 |
| C6-C5-H5 | 119.6 | C18-C19-H19 | 119.8 |
| C1-C6-C5 | 122.0 (4) | C19-C20-C15 | 120.4 (8) |
| C1-C6-H6 | 119.0 | C19-C20-C21 | 119.1 (11) |
| C5-C6-H6 | 119.0 | C15-C20-C21 | 120.5 (10) |
| C4-C7-H7A | 109.5 | N1-C21-C20 | 113.4 (10) |
| C4-C7-H7B | 109.5 | N1-C21-H21A | 108.9 |
| H7A-C7-H7B | 109.5 | $\mathrm{C} 20-\mathrm{C} 21-\mathrm{H} 21 \mathrm{~A}$ | 108.9 |
| C4-C7- H 7 C | 109.5 | N1-C21-H21B | 108.9 |
| H7A-C7-H7C | 109.5 | C20-C21-H21B | 108.9 |
| H7B-C7-H7C | 109.5 | $\mathrm{H} 21 \mathrm{~A}-\mathrm{C} 21-\mathrm{H} 21 \mathrm{~B}$ | 107.7 |
| C9-C8-C13 | 117.8 (4) | N1-C22-H22A | 109.5 |
| C9-C8-Bi1 | 119.7 (3) | N1-C22-H22B | 109.5 |
| C13-C8-Bi1 | 122.5 (3) | $\mathrm{H} 22 \mathrm{~A}-\mathrm{C} 22-\mathrm{H} 22 \mathrm{~B}$ | 109.5 |
| C8-C9-C10 | 121.0 (4) | N1-C22-H22C | 109.5 |
| C8-C9-H9 | 119.5 | $\mathrm{H} 22 \mathrm{~A}-\mathrm{C} 22-\mathrm{H} 22 \mathrm{C}$ | 109.5 |
| C10-C9-H9 | 119.5 | $\mathrm{H} 22 \mathrm{~B}-\mathrm{C} 22-\mathrm{H} 22 \mathrm{C}$ | 109.5 |
| C9-C10-C11 | 120.9 (4) | N1-C23-H23A | 109.5 |
| C9-C10-H10 | 119.6 | N1-C23-H23B | 109.5 |
| C11-C10-H10 | 119.6 | $\mathrm{H} 23 \mathrm{~A}-\mathrm{C} 23-\mathrm{H} 23 \mathrm{~B}$ | 109.5 |
| C12-C11-C10 | 117.4 (4) | N1-C23-H23C | 109.5 |
| C12-C11-C14 | 121.5 (4) | $\mathrm{H} 23 \mathrm{~A}-\mathrm{C} 23-\mathrm{H} 23 \mathrm{C}$ | 109.5 |
| C10-C11-C14 | 121.0 (4) | H23B-C23-H23C | 109.5 |
| C13-C12-C11 | 121.6 (4) | C21-N1-C22 | 111.6 (5) |
| $\mathrm{C} 13-\mathrm{C} 12-\mathrm{H} 12$ | 119.2 | $\mathrm{C} 21-\mathrm{N} 1-\mathrm{C} 23$ | 110.6 (5) |
| C11-C12-H12 | 119.2 | C22-N1-C23 | 110.0 (4) |
| C12-C13-C8 | 121.3 (4) | C21-N1-Bil | 98.6 (4) |
| C12-C13-H13 | 119.4 | C22-N1-Bil | 118.9 (3) |
| C8-C13-H13 | 119.4 | C23-N1-Bil | 106.7 (3) |
| C11-C14-H14A | 109.5 | C1-Bil-C8 | 96.07 (16) |
| C11-C14-H14B | 109.5 | C1-Bil-C15 | 90.74 (16) |


| $\mathrm{H} 14 \mathrm{~A}-\mathrm{C} 14-\mathrm{H} 14 \mathrm{~B}$ | 109.5 | $\mathrm{C} 1-\mathrm{Bi} 1-\mathrm{N} 1$ | $157.55(14)$ |
| :--- | :--- | :--- | :--- |
| $\mathrm{C} 11-\mathrm{C} 14-\mathrm{H} 14 \mathrm{C}$ | 109.5 | $\mathrm{C} 8-\mathrm{Bi} 1-\mathrm{C} 15$ | $94.85(17)$ |
| $\mathrm{H} 14 \mathrm{~A}-\mathrm{C} 14-\mathrm{H} 14 \mathrm{C}$ | 109.5 | $\mathrm{C} 8-\mathrm{Bi} 1-\mathrm{N} 1$ | $81.26(14)$ |
| H14B-C14-H14C | 109.5 | C15-Bi1-N1 | $67.43(13)$ |


[^0]:    $\ddagger$ Also at: Organization for Frontier Research in Preventive Pharmaceutical Sciences, Hokuriku University.

