

Dimethyl[2-(phenyliminomethyl)-phenolato- κ^2 N,O]gold(III)

Alexander A. Bessonov,* Anton I. Smolentsev, Iraida A. Baidina and Natalia B. Morozova

Nikolaev Institute of Inorganic Chemistry, SB Russian Academy of Sciences, Akademian Lavrentiev Prospekt 3, Novosibirsk 90, 630090, Russian Federation
Correspondence e-mail: bessonov@che.nsk.su

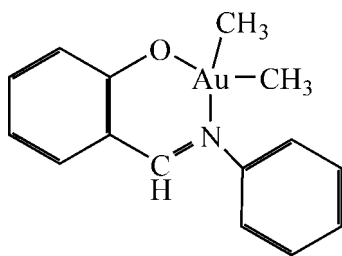
Received 14 November 2007; accepted 19 November 2007

Key indicators: single-crystal X-ray study; $T = 150$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; disorder in main residue; R factor = 0.012; wR factor = 0.027; data-to-parameter ratio = 12.6.

The Au atom in the title compound, $[\text{Au}(\text{CH}_3)_2(\text{C}_{13}\text{H}_{10}\text{NO})]$, has a square-planar coordination geometry. The molecule is located on a crystallographic mirror plane. The phenolato ring is disordered over two equally occupied positions. The molecules are packed in infinite stacks parallel to the b axis, with an $\text{Au} \cdots \text{Au}$ distance of 4.2106 (4) Å.

Related literature

Dimethylgold(III) derivatives are used in metal-organic chemical vapor deposition processes because of their volatility and thermal stability (Larson *et al.*, 1987; Semyannikov *et al.*, 2006; Bessonov, Morozova *et al.*, 2007; Bessonov, Baidina *et al.*, 2007). For a similar structure, see: Shibata *et al.* (1990). For literature related to the synthesis, see: Bessonov *et al.* (2008); Murray *et al.* (1973).



Experimental

Crystal data

$[\text{Au}(\text{CH}_3)_2(\text{C}_{13}\text{H}_{10}\text{NO})]$
 $M_r = 423.25$

Monoclinic, $P2_1/m$
 $a = 8.5316$ (10) Å

$b = 7.3356$ (7) Å
 $c = 11.1180$ (13) Å
 $\beta = 101.006$ (3)°
 $V = 683.02$ (13) Å³
 $Z = 2$

Mo $K\alpha$ radiation
 $\mu = 10.76$ mm⁻¹
 $T = 150$ (2) K
 $0.25 \times 0.06 \times 0.06$ mm

Data collection

Bruker–Nonius X8 APEXII diffractometer
Absorption correction: multi-scan (SADABS; Bruker, 2004)
 $T_{\min} = 0.454$, $T_{\max} = 0.565$
(expected range = 0.422–0.524)

4954 measured reflections
1506 independent reflections
1423 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.018$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.012$
 $wR(F^2) = 0.027$
 $S = 1.11$
1506 reflections

120 parameters
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.43$ e Å⁻³
 $\Delta\rho_{\min} = -0.52$ e Å⁻³

Table 1

Selected bond lengths (Å).

Au1—C12	2.030 (4)	Au1—O1	2.077 (2)
Au1—C13	2.031 (3)	Au1—N1	2.104 (3)

Data collection: APEX2 (Bruker, 2004); cell refinement: SAINT (Bruker, 2004); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Bruker, 2004); program(s) used to refine structure: SHELXTL; molecular graphics: POV-RAY (Cason, 2002); software used to prepare material for publication: SHELXTL.

The authors thank the Russian Science Support Foundation for financial support and Professor Igor K. Igumenov for helpful comments.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2619).

References

- Bessonov, A. A., Baidina, I. A., Morozova, N. B., Semyannikov, P. P., Trubin, S. V., Gelfond, N. V. & Igumenov, I. K. (2007). *J. Struct. Chem.* **48**, 282–287.
Bessonov, A. A., Morozova, N. B., Gelfond, N. V., Semyannikov, P. P., Trubin, S. V., Shevtsov, Yu. V., Shubin, Yu. V. & Igumenov, I. K. (2007). *Surf. Coat. Technol.* **201**, 9099–9103.
Bessonov, A. A., Morozova, N. B., Kuratieva, N. V., Baidina, I. A., Gelfond, N. V. & Igumenov, I. K. (2008). *Koord. Khim.* **34**, 73–80.
Bruker (2004). APEX2 (Version 1.08), SAINT (Version 7.03), SADABS (Version 2.11) and SHELXTL (Version 6.12). Bruker AXS Inc., Madison, Wisconsin, USA.
Cason, C. J. (2002). *POV-RAY for Windows*. Version 3.5. <http://www.pov-ray.org>.
Larson, C. E., Baum, T. H. & Jackson, R. L. (1987). *J. Electrochem. Soc.* **134**, 266.
Murray, K. S., Reichert, B. E. & West, B. O. (1973). *J. Organomet. Chem.* **61**, 451–456.
Semyannikov, P. P., Moroz, B. L., Trubin, S. V., Zharkova, G. I., Pyryaev, P. A., Smirnov, M. Yu. & Bukhtiyarov, V. I. (2006). *J. Struct. Chem.* **47**, 458–462.
Shibata, S., Lijima, K. & Baum, T. H. (1990). *J. Chem. Soc. Dalton Trans.* pp. 1519–1520.

supplementary materials

Acta Cryst. (2007). E63, m3154 [doi:10.1107/S1600536807060709]

Dimethyl[2-(phenyliminomethyl)phenolato- κ^2N,O]gold(III)

A. A. Bessonov, A. I. Smolentsev, I. A. Baidina and N. B. Morozova

Comment

The title compound has molecular structure constructed from neutral molecules $(\text{CH}_3)_2\text{Au}(\text{C}_{13}\text{H}_{10}\text{NO})$. The gold atom in the molecule has a slightly distorted square planar configuration formed by oxygen and nitrogen atoms of salicylaldehyde ligand, and two carbon atoms of methyl groups (Fig. 1). In the coordination square, Au—O and Au—N distances differ slightly, and O—Au—N angle is very close to 90° . The values of Au—CH₃ bonds are typical for dimethylgold(III) derivatives (Shibata *et al.*, 1990; Bessonov, Baidina *et al.*, 2007). The salicylaldehyde fragment is located on a mirror plane, however carbon atoms C5, C6, C7, C8 and C9 are disordered. The planes of salicylaldehyde and phenyl ring are nearly perpendicular (the angle is about 91°). In the crystal, molecules are packed in infinite stacks along the monoclinic axis with the shortest Au...Au distance of 4.2106 (4) Å and Au—Au—Au angle of 121.173 (8) $^\circ$ (Fig. 2). The oxygen atom is involved in intermolecular O...H—C interactions with O...C distances of 3.291 (3) Å. The shortest intermolecular H...H distance in the crystal is about 2.5 Å.

Experimental

Sodium bicarbonate, NaHCO_3 , and dimethylgold(III) iodide, $[(\text{CH}_3)_2\text{AuI}]_2$, (prepared as described in Bessonov *et al.*, 2008) were added to a solution of *N*-phenylsalicylaldehyde in methanol. The mixture was stirred for 6 h in an argon flow. Then, the methanol was removed in vacuum and the residue was redissolved in hexane. The hexane solution was filtered and the volatile components were removed in vacuum. The complex was purified by recrystallization from hexane at -10°C . Pale yellow crystals suitable for X-ray analysis were formed in yield of 80%. The melting point of the complex was 147–150 $^\circ\text{C}$ (154 $^\circ\text{C}$ from Murray *et al.*, 1973). The product is stable to air and moisture and soluble in most common organic solvents.

Refinement

H atoms were refined with fixed individual displacement parameters [$U(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$] using a riding model with $C_{\text{aromatic}}\text{—H} = 0.95$ Å and $C_{\text{methyl}}\text{—H} = 0.98$ Å. The phenolic ring and the methyl H atoms are disordered over two equally occupied positions.

Figures

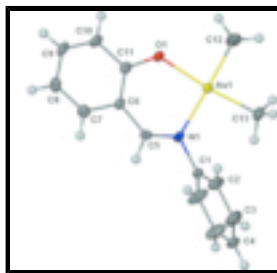


Fig. 1. Plot of $\text{Me}_2\text{Au}(\text{Sal}=\text{N}-\text{Ph})$ molecule showing the atom numbering for the core atoms. Displacement ellipsoids are drawn at the 50% probability level. Only one set of the two disordered atoms is shown.

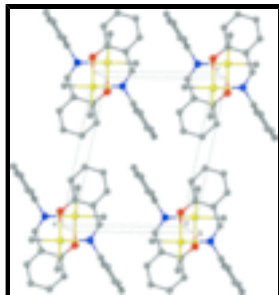


Fig. 2. A packing diagram of the $\text{Me}_2\text{Au}(\text{Sal}=\text{N}-\text{Ph})$ structure, viewed along the b axis. H atoms omitted for clarity. Only one set of the two disordered atoms is shown.

Dimethyl[2-(phenyliminomethyl)phenolato- $\kappa^2\text{N},\text{O}$]gold(III)

Crystal data

$[\text{Au}(\text{CH}_3)_2(\text{C}_{13}\text{H}_{10}\text{NO})]$

$M_r = 423.25$

Monoclinic, $P2_1/m$

Hall symbol: $-P\ 2yb$

$a = 8.5316\ (10)\ \text{\AA}$

$b = 7.3356\ (7)\ \text{\AA}$

$c = 11.1180\ (13)\ \text{\AA}$

$\beta = 101.006\ (3)^\circ$

$V = 683.02\ (13)\ \text{\AA}^3$

$Z = 2$

$F_{000} = 400$

$D_x = 2.058\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073\ \text{\AA}$

Cell parameters from 3995 reflections

$\theta = 2.4\text{--}28.3^\circ$

$\mu = 10.76\ \text{mm}^{-1}$

$T = 150\ (2)\ \text{K}$

Needle, pale yellow

$0.25 \times 0.06 \times 0.06\ \text{mm}$

Data collection

Bruker–Nonius X8 APEX CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

Detector resolution: $25\ \text{pixels mm}^{-1}$

$T = 150\ (2)\ \text{K}$

φ scans

Absorption correction: multi-scan (SADABS; Bruker, 2004)

$T_{\min} = 0.454$, $T_{\max} = 0.565$

4954 measured reflections

1506 independent reflections

1423 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.018$

$\theta_{\max} = 26.4^\circ$

$\theta_{\min} = 1.9^\circ$

$h = -10 \rightarrow 10$

$k = -6 \rightarrow 9$

$l = -13 \rightarrow 13$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.012$

$wR(F^2) = 0.027$

Secondary atom site location: difference Fourier map

Hydrogen site location: difference Fourier map

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0113P)^2 + 0.2414P]$

where $P = (F_o^2 + 2F_c^2)/3$

$S = 1.11$ $(\Delta/\sigma)_{\max} = 0.001$
 1506 reflections $\Delta\rho_{\max} = 0.43 \text{ e } \text{\AA}^{-3}$
 120 parameters $\Delta\rho_{\min} = -0.51 \text{ e } \text{\AA}^{-3}$
 Primary atom site location: structure-invariant direct methods Extinction correction: none

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR 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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Au1	0.935702 (14)	0.2500	0.069963 (11)	0.02283 (5)	
N1	1.1812 (3)	0.2500	0.0660 (2)	0.0256 (6)	
O1	0.8825 (3)	0.2500	-0.12028 (19)	0.0255 (5)	
C13	0.9623 (4)	0.2500	0.2555 (3)	0.0393 (10)	
H13A	0.8683	0.3061	0.2790	0.059*	0.50
H13B	1.0579	0.3196	0.2911	0.059*	0.50
H13C	0.9733	0.1243	0.2857	0.059*	0.50
C11	0.9822 (4)	0.2500	-0.1954 (3)	0.0265 (7)	
C5	1.2359 (5)	0.2098 (5)	-0.0345 (4)	0.0203 (14)	0.50
H5	1.3454	0.1770	-0.0232	0.024*	0.50
C12	0.6969 (5)	0.2500	0.0668 (4)	0.086 (2)	
H12A	0.6630	0.3726	0.0857	0.129*	0.50
H12B	0.6725	0.1636	0.1278	0.129*	0.50
H12C	0.6402	0.2138	-0.0148	0.129*	0.50
C1	1.3007 (4)	0.2500	0.1778 (3)	0.0263 (7)	
C4	1.5304 (4)	0.2500	0.3890 (3)	0.0367 (9)	
H4	1.6108	0.2500	0.4610	0.044*	
C2	1.3560 (3)	0.0877 (4)	0.2304 (2)	0.0438 (7)	
H2	1.3156	-0.0246	0.1947	0.053*	
C3	1.4724 (4)	0.0896 (4)	0.3371 (3)	0.0483 (8)	
H3	1.5117	-0.0224	0.3740	0.058*	
C6	1.1492 (5)	0.2099 (6)	-0.1589 (4)	0.0217 (15)	0.50
C10	0.9187 (4)	0.2500	-0.3223 (3)	0.0338 (8)	
H10	0.8088	0.2767	-0.3498	0.041*	0.50
C9	1.0136 (6)	0.2117 (7)	-0.4081 (4)	0.0292 (18)	0.50
H9	0.9654	0.2010	-0.4922	0.035*	0.50
C8	1.1776 (6)	0.1888 (7)	-0.3728 (4)	0.0379 (15)	0.50

supplementary materials

H8	1.2433	0.1738	-0.4321	0.045*	0.50
C7	1.2419 (6)	0.1884 (6)	-0.2503 (4)	0.0320 (13)	0.50
H7	1.3540	0.1730	-0.2257	0.038*	0.50

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Au1	0.01707 (7)	0.03630 (8)	0.01489 (7)	0.000	0.00249 (5)	0.000
N1	0.0208 (14)	0.0411 (17)	0.0138 (13)	0.000	0.0006 (11)	0.000
O1	0.0201 (11)	0.0402 (13)	0.0153 (11)	0.000	0.0011 (9)	0.000
C13	0.030 (2)	0.073 (3)	0.0160 (18)	0.000	0.0060 (15)	0.000
C11	0.0248 (17)	0.0351 (19)	0.0192 (17)	0.000	0.0032 (14)	0.000
C5	0.0171 (18)	0.020 (4)	0.023 (2)	0.0005 (15)	0.0014 (15)	-0.0012 (16)
C12	0.018 (2)	0.213 (7)	0.027 (2)	0.000	0.0045 (17)	0.000
C1	0.0156 (15)	0.049 (2)	0.0143 (16)	0.000	0.0021 (13)	0.000
C4	0.0229 (18)	0.067 (3)	0.0172 (17)	0.000	-0.0029 (14)	0.000
C2	0.0474 (17)	0.0474 (17)	0.0285 (14)	-0.0204 (13)	-0.0134 (12)	0.0121 (12)
C3	0.0539 (18)	0.0532 (18)	0.0302 (15)	-0.0070 (15)	-0.0111 (13)	0.0173 (14)
C6	0.025 (2)	0.024 (4)	0.0152 (18)	-0.0032 (17)	0.0019 (15)	0.0001 (17)
C10	0.0287 (19)	0.053 (2)	0.0172 (17)	0.000	-0.0018 (14)	0.000
C9	0.039 (2)	0.031 (6)	0.0158 (19)	-0.001 (2)	0.0012 (17)	-0.0008 (19)
C8	0.036 (3)	0.060 (4)	0.021 (2)	-0.003 (2)	0.014 (2)	-0.004 (2)
C7	0.024 (2)	0.047 (3)	0.026 (2)	0.0008 (19)	0.0071 (19)	-0.0023 (19)

Geometric parameters (\AA , $^\circ$)

Au1—C12	2.030 (4)	C8—C7	1.368 (7)
Au1—C13	2.031 (3)	C13—H13A	0.9800
Au1—O1	2.077 (2)	C13—H13B	0.9800
Au1—N1	2.104 (3)	C13—H13C	0.9800
N1—C5	1.324 (5)	C12—H12A	0.9800
N1—C1	1.449 (4)	C12—H12B	0.9800
O1—C11	1.301 (4)	C12—H12C	0.9800
C11—C10	1.411 (5)	C5—H5	0.9500
C11—C6	1.436 (5)	C4—H4	0.9500
C5—C6	1.438 (6)	C2—H2	0.9500
C1—C2	1.369 (3)	C3—H3	0.9500
C4—C3	1.361 (4)	C10—H10	0.9500
C2—C3	1.392 (4)	C9—H9	0.9500
C6—C7	1.412 (6)	C8—H8	0.9500
C10—C9	1.393 (6)	C7—H7	0.9500
C9—C8	1.389 (7)		
C12—Au1—C13	86.27 (16)	H13A—C13—H13B	109.5
C12—Au1—O1	87.62 (14)	Au1—C13—H13C	109.5
C13—Au1—O1	173.89 (11)	H13A—C13—H13C	109.5
C12—Au1—N1	177.84 (14)	H13B—C13—H13C	109.5
C13—Au1—N1	95.89 (13)	N1—C5—H5	116.1
O1—Au1—N1	90.22 (9)	C6—C5—H5	116.1

C5—N1—C1	114.9 (3)	Au1—C12—H12A	109.5
C5—N1—Au1	122.0 (2)	Au1—C12—H12B	109.5
C1—N1—Au1	121.5 (2)	H12A—C12—H12B	109.5
C11—O1—Au1	127.7 (2)	Au1—C12—H12C	109.5
O1—C11—C10	117.9 (3)	H12A—C12—H12C	109.5
C10—C11—C6	117.0 (3)	H12B—C12—H12C	109.5
O1—C11—C6	123.7 (3)	C3—C4—H4	120.2
N1—C5—C6	127.8 (4)	C1—C2—H2	120.5
C2—C1—N1	119.70 (16)	C3—C2—H2	120.5
C1—C2—C3	119.2 (3)	C4—C3—H3	119.6
C4—C3—C2	120.8 (3)	C2—C3—H3	119.6
C7—C6—C11	118.9 (4)	C9—C10—H10	119.2
C7—C6—C5	115.8 (4)	C11—C10—H10	119.2
C11—C6—C5	124.9 (4)	C8—C9—H9	119.5
C9—C10—C11	121.4 (4)	C10—C9—H9	119.5
C10—C9—C8	121.2 (4)	C7—C8—H8	120.9
C7—C8—C9	118.3 (4)	C9—C8—H8	120.9
C8—C7—C6	122.8 (4)	C8—C7—H7	118.5
Au1—C13—H13A	109.5	C6—C7—H7	118.5
Au1—C13—H13B	109.5		

Fig. 1

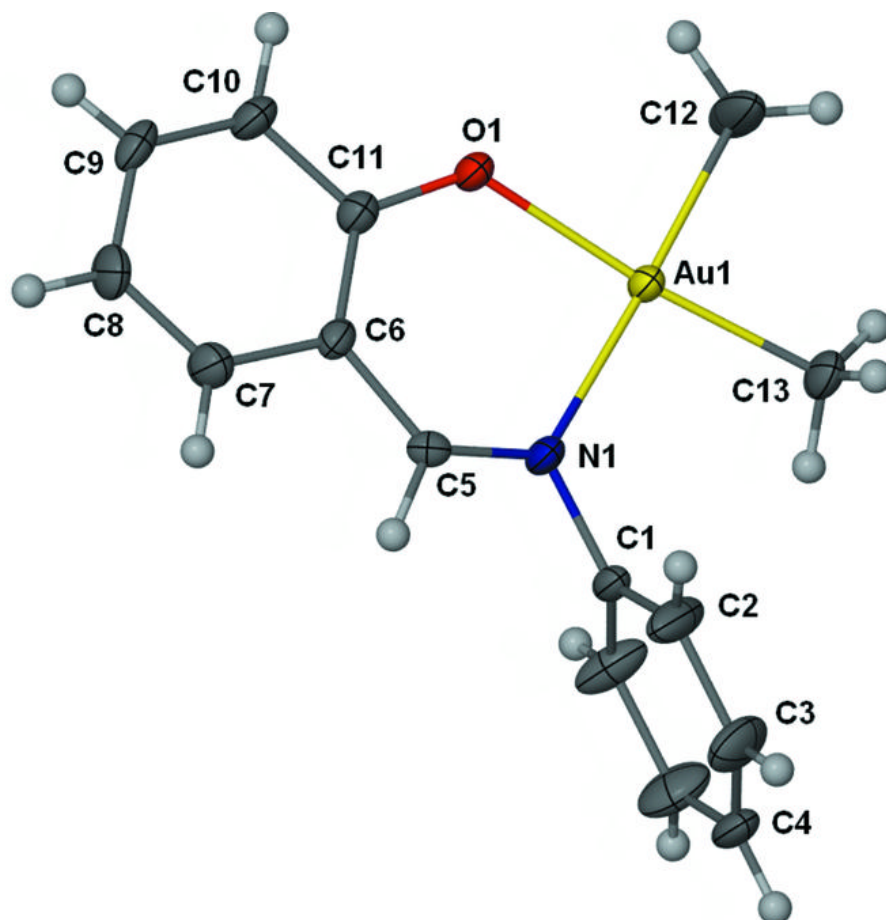


Fig. 2

