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Bromido(2,4,6-trimethylphenyl)mercury(II)

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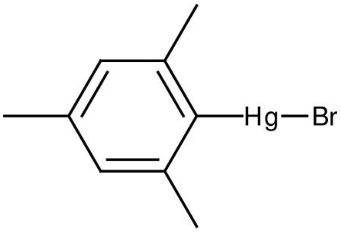
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.009 Å; R factor = 0.025; wR factor = 0.057; data-to-parameter ratio = 17.1.

Molecules of the title compound, [HgBr(C_9H_{11})], are located on a crystallographic twofold rotation axis. Due to the molecular symmetry, the Hg^{II} atom is linearly coordinated by the *ipso*-C of the mesityl group and the Br atom. In the crystal, molecules lie in planes parallel to (001).

Related literature

For dimesityl-mercury, see: Hayashi *et al.* (2011). For the synthesis of Hg[Mes]$_2$, see: Hübner *et al.* (2010).



Experimental

Crystal data

[HgBr(C_9H_{11})]
M_r = 399.68
Monoclinic, $C2/c$
a = 10.0459 (8) Å
b = 15.3072 (13) Å
c = 8.1517 (7) Å
β = 126.912 (5)°
V = 1002.27 (14) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 19.28 mm^{-1}
T = 173 K
0.21 × 0.10 × 0.03 mm

Data collection

Stoe IPDS II two-circle diffractometer
Absorption correction: multi-scan (*MULABS*; Spek, 2009; Blessing, 1995)
T_{min} = 0.107, T_{max} = 0.595
6756 measured reflections
942 independent reflections
892 reflections with $I > 2\sigma(I)$
R_{int} = 0.103

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.057
S = 1.05
942 reflections
55 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.71 e Å^{-3}

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-RED32*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK5065).

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supplementary materials

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Comment

Very recently we have shown that Hg[Mes]$_2$ could be synthesized when $HgCl_2$ was treated with two equivalents of Li[Mes] (Hübner *et al.*, 2010) in thf at ambient temperature (Hayashi *et al.*, 2011). In addition, we have investigated the reaction of Hg[Mes]$_2$ with BBr_3 (Hayashi *et al.*, 2011). In this paper we report the structure of the analogous Grignard compound Hg[Mes]Br which was obtained from the 1: 1 reaction of Hg[Mes]$_2$ with BBr_3.

Molecules of the title compound are located on a crystallographic twofold rotation axis with half a molecule in the asymmetric unit. Due to the molecular symmetry, the Hg centre is linearly coordinated by the *ipso*-C of the mesityl group and the Br atom. The Hg—C bond [2.053 (7) Å] is slightly shorter than that in dimesityl mercury [2.080 (6) Å] (Hübner *et al.*, 2010).

In the crystal, the molecules lie in planes parallel to (0 0 1). In a plane, the molecules are oriented parallel to each other with the Hg—Br vectors pointing in the same direction. The shortest intermolecular Hg···Br contact is 4.1270 (4) Å and the shortest intermolecular Hg···Hg contact is 5.1078 (4) Å (symmetry operator for equivalent atoms: 1 - *x*, 1 - *y*, 1 - *z*).

Experimental

In a round bottom flask Hg[Mes]$_2$ (0.21 g, 0.48 mmol) in 40 ml benzene was treated with one equivalent of BBr_3 (0.046 ml, 120 mg, 0.48 mmol) at ambient temperature. Single crystals of the title compound Hg[Mes]Br were obtained from a benzene solution after 2 days at 281 K. Yield 50 mg (26%). ^{1}H NMR (300.0 MHz, $CDCl_3$): δ = 7.00 (m, 2 H, *meta*-Ph), 2.47 (br., 6 H, *ortho*-Me), 2.34 (br., 3 H, *para*-Me). ^{13}C{1H} NMR (75.5 MHz, $CDCl_3$): δ = 154.1 (*ipso*-Mes), 141.6 (*ortho*-Mes), 139.3 (*para*-Mes), 128.0 (*meta*-Mes), 25.8 (*ortho*-Me), 20.9 (*para*-Me). EI$^+$ m/*z* (%): 396.1 (20.0) 397.1 (32.0) 398.1 (64.0) 399.1 (60.0) 400.1 (100.0) 401.1 (32.0) 402.1 (68.0) 403.1 (6.0) 404.1 (12.0) $[M]^+$, calcd. for $[M]^+$ 396.1 (30.8) 397.1 (54.4) 398.1 (100.0) 399.1 (93.1) 400.1 (95.5) 401.1 (45.8) 402.1 (25.5) 403.1 (11.0) 404.1 (20.9).

Refinement

H atoms were refined using a riding model, with methyl C—H = 0.98 Å and aromatic C—H = 0.95 Å and with $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl-H or $1.2U_{eq}(C)$ for aromatic-H. The methyl groups were allowed to rotate but not to tip. The methyl group in *para* position of the phenyl ring is disordered over two equally occupied positions.

Computing details

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA* (Stoe & Cie, 2001); data reduction: *X-RED32* (Stoe & Cie, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

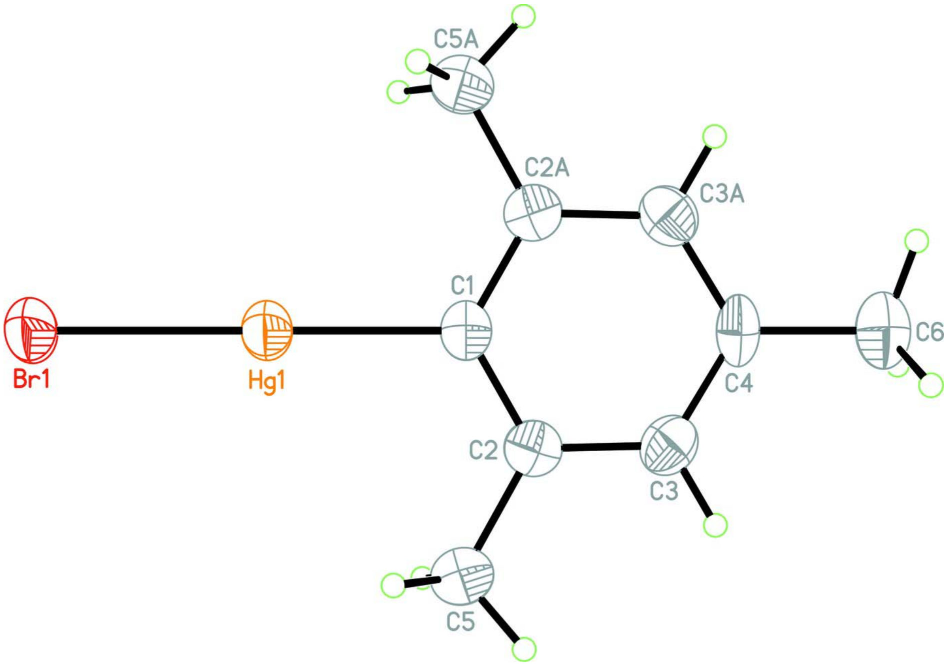

Figure 1

A perspective view of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. Only one set of the H atoms of the disordered methyl group is shown. Unlabelled atoms are related by the symmetry operation 1-*x*, *y*, 3/2-*z*.

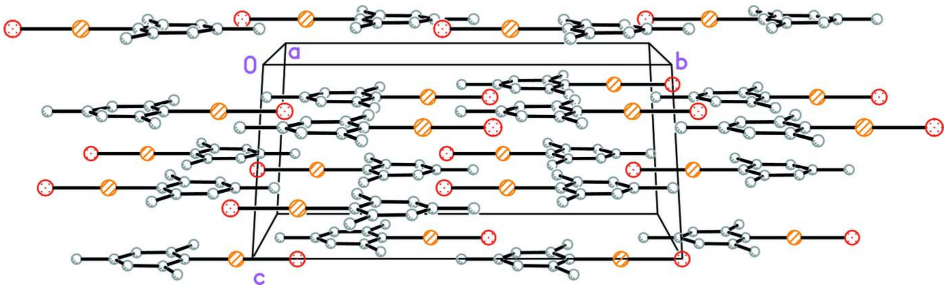

Figure 2

Packing diagram of the title compound viewed onto the *bc* plane. H atoms omitted for clarity.

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Crystal data

$[HgBr(C_9H_{11})]$
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Monoclinic, $C2/c$
Hall symbol: -C 2yc
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c = 8.1517 (7) Å
β = 126.912 (5)°
V = 1002.27 (14) Å^3
Z = 4

$F(000)$ = 720
D_x = 2.649 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 8005 reflections
θ = 4.1–25.9°
μ = 19.28 mm^{-1}
T = 173 K
Needle, colourless
0.21 × 0.10 × 0.03 mm

Data collection

Stoe IPDS II two-circle diffractometer	6756 measured reflections
Radiation source: Genix 3D IμS microfocus X-ray source	942 independent reflections
Genix 3D multilayer optics monochromator	892 reflections with $I > 2\sigma(I)$
ω scans	$R_{\text{int}} = 0.103$
Absorption correction: multi-scan (*MULABS*; Spek, 2009; Blessing, 1995)	$\theta_{\text{max}} = 25.6°$, $\theta_{\text{min}} = 4.1°$
$T_{\text{min}} = 0.107$, $T_{\text{max}} = 0.595$	$h = -12\rightarrow12$
	$k = -18\rightarrow18$
	$l = -9\rightarrow9$

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-atom parameters constrained
$wR(F^2) = 0.057$	$w = 1/[\sigma^2(F_o^2) + (0.024P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{\text{max}} = 0.001$
942 reflections	$\Delta\rho_{\text{max}} = 1.19$ e Å^{-3}
55 parameters	$\Delta\rho_{\text{min}} = -0.71$ e Å^{-3}
0 restraints	
Primary atom site location: structure-invariant direct methods	

Special details

Experimental. ;

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
Hg1	0.5000	0.600559 (17)	0.7500	0.03814 (13)	
Br1	0.5000	0.44177 (5)	0.7500	0.0531 (3)	
C1	0.5000	0.7347 (5)	0.7500	0.0336 (15)	
C2	0.6166 (6)	0.7798 (4)	0.7414 (7)	0.0331 (10)	
C3	0.6142 (7)	0.8712 (4)	0.7429 (8)	0.0375 (12)	
H3	0.6940	0.9024	0.7389	0.045*	
C4	0.5000	0.9178 (5)	0.7500	0.0369 (18)	
C5	0.7411 (7)	0.7320 (4)	0.7278 (9)	0.0422 (13)	
H5A	0.7982	0.6875	0.8359	0.063*	
H5B	0.8229	0.7736	0.7453	0.063*	
H5C	0.6831	0.7037	0.5933	0.063*	
C6	0.5000	1.0155 (5)	0.7500	0.048 (2)	
H6A	0.6116	1.0368	0.8599	0.073*	0.50
H6B	0.4202	1.0368	0.7726	0.073*	0.50
H6C	0.4682	1.0368	0.6175	0.073*	0.50

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Hg1	0.03770 (18)	0.02853 (18)	0.0464 (2)	0.000	0.02430 (15)	0.000
Br1	0.0503 (5)	0.0291 (5)	0.0834 (7)	0.000	0.0419 (5)	0.000
C1	0.038 (4)	0.029 (3)	0.032 (4)	0.000	0.020 (3)	0.000
C2	0.030 (2)	0.038 (3)	0.026 (2)	−0.001 (2)	0.014 (2)	−0.003 (2)
C3	0.037 (3)	0.038 (3)	0.034 (3)	−0.007 (2)	0.019 (2)	−0.004 (2)
C4	0.043 (4)	0.021 (3)	0.032 (4)	0.000	0.014 (3)	0.000
C5	0.038 (3)	0.045 (3)	0.044 (3)	−0.003 (2)	0.025 (3)	−0.001 (2)
C6	0.060 (6)	0.033 (4)	0.045 (5)	0.000	0.028 (4)	0.000

Geometric parameters (Å, º)

Hg1—C1	2.053 (7)	C4—C3^{i}	1.381 (7)
Hg1—Br1	2.4307 (8)	C4—C6	1.495 (10)
C1—C2^{i}	1.397 (6)	C5—H5A	0.9800
C1—C2	1.397 (6)	C5—H5B	0.9800
C2—C3	1.400 (8)	C5—H5C	0.9800
C2—C5	1.510 (7)	C6—H6A	0.9800
C3—C4	1.381 (7)	C6—H6B	0.9800
C3—H3	0.9500	C6—H6C	0.9800
C1—Hg1—Br1	180.000 (1)	C2—C5—H5A	109.5
C2^{i}—C1—C2	120.8 (7)	C2—C5—H5B	109.5
C2^{i}—C1—Hg1	119.6 (3)	H5A—C5—H5B	109.5
C2—C1—Hg1	119.6 (3)	C2—C5—H5C	109.5
C1—C2—C3	118.3 (5)	H5A—C5—H5C	109.5
C1—C2—C5	121.4 (5)	H5B—C5—H5C	109.5
C3—C2—C5	120.3 (5)	C4—C6—H6A	109.5
C4—C3—C2	122.4 (5)	C4—C6—H6B	109.5
C4—C3—H3	118.8	H6A—C6—H6B	109.5
C2—C3—H3	118.8	C4—C6—H6C	109.5
C3—C4—C3^{i}	117.8 (7)	H6A—C6—H6C	109.5
C3—C4—C6	121.1 (3)	H6B—C6—H6C	109.5
C3^{i}—C4—C6	121.1 (3)		
C2^{i}—C1—C2—C3	0.4 (3)	C1—C2—C3—C4	−0.8 (7)
Hg1—C1—C2—C3	−179.6 (3)	C5—C2—C3—C4	178.4 (4)
C2^{i}—C1—C2—C5	−178.8 (5)	C2—C3—C4—C3^{i}	0.4 (4)
Hg1—C1—C2—C5	1.2 (5)	C2—C3—C4—C6	−179.6 (4)

Symmetry code: (i) $-x+1, y, -z+3/2$.