metal-organic compounds

trans-Bis[1,2-bis(dimethylphosphino)-ethane]bromidonitrosyltungsten(0)

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The crystal structure of the title compound, [WBr(NO)-$(C_6H_{16}P_2)_2$], reveals a distorted octahedral geometry around the W centre. The W atom lies on a special position at an inversion centre (the Br and NO ligands are equally disordered). The bis(dimethylphosphino)ethane ligand is also severely disordered (site occupancy factors 0.52 and 0.48). This is the first structure of a tungsten species with nitrosyl and bromide ligands.

Related literature

For related nitrosyltetrakis(trimethylphosphine)tungsten complexes, see: Chen *et al.* (2005). For bis[1,2-bis(dimethylphosphino)ethane]bromidotungsten complexes, see: Manna *et al.* (1992); Cotton *et al.* (1989). For carbonylphosphine tungsten complexes, see: Drew *et al.* (1982); Cotton & Meadows (1984). For synthesis of the precursors of the title compound, see: Johnson (1967); Berg & Dehnicke (1985). For the Cambridge Structural Database (Release 2006, Version 5.28), see: Allen (2002).
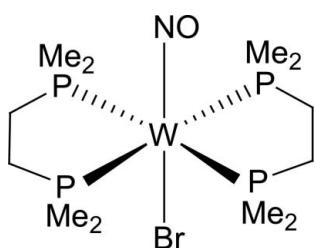
Experimental

Crystal data

[WBr(NO)($C_6H_{16}P_2$)$_2$]
M_r = 594.01
Monoclinic, $P2_1/c$
a = 8.8909 (14) Å
b = 12.5386 (16) Å
c = 12.823 (2) Å
β = 130.639 (14)°
V = 1084.8 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 7.46 mm^{-1}
T = 183 (2) K
$0.45 \times 0.39 \times 0.28$ mm

Data collection

Stoe IPDS diffractometer
Absorption correction: numerical (Coppens *et al.*, 1965)
T_{min} = 0.086, T_{max} = 0.213
19322 measured reflections
1901 independent reflections
1415 reflections with $I > 2\sigma(I)$
R_{int} = 0.076

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.089
S = 0.92
1901 reflections
133 parameters
45 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 2.37 e Å^{-3}
$\Delta\rho_{min}$ = −1.59 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

W1—N1	1.83 (2)	W1—Br1	2.555 (3)
W1—P1	2.424 (2)	N1—O1	1.267 (19)
W1—P2	2.4302 (18)		
N1—W1—Br1	176.5 (5)	P1^i—W1—P1	180
O1—N1—W1	177.9 (19)	P2^i—W1—P2	180
N1—W1—P1	90.8 (5)	P1^i—W1—Br1	87.62 (9)
N1^i—W1—P1	89.2 (5)	P1—W1—Br1	92.38 (9)
N1—W1—P2	91.3 (5)	P2^i—W1—Br1	89.28 (7)
N1^i—W1—P2	88.7 (5)	P2—W1—Br1	90.72 (7)

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

Data collection: *IPDS Software* (Stoe & Cie, 1999); cell refinement: *IPDS Software*; data reduction: *X-RED* in *IPDS Software*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: YA2063).

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supplementary materials

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Comment

According to the Cambridge Structural Database (Release 2006, Version 5.28; Allen, 2002), the title compound (I) is the first example of the structurally studied tungsten species with nitrosyl and bromide ligands. The tungsten atom lies on a special position in the inversion centre: the nitrosyl group and the halide ligand in *trans*-position to each other are disordered. The bidentate dmpe ligand (dmpe = 1,2-bis(dimethylphosphino)ethane) is also disordered over two positions which share common locations of the P atoms, but have different locations for all C atoms; the occupancy of the major component of the disorder was refined to 0.52 (1), which indicates, that both components are effectively equally represented in the structure.

The tungsten atom has a distorted octahedral coordination (Fig. 1). The W1—Br1 distance of 2.555 (3) Å is clearly shorter than in the related bromo-bis(1,2-bis(dimethylphosphino)ethane)tungsten complexes, with the bromide ligand in *trans* position to the triply bonded C—Ph ligand [2.744 (2) Å and 2.702 (2) Å in Manna *et al.* (1992)] or to an oxo ligand [2.678 (2) Å in Cotton *et al.* (1989)]. Nevertheless, the reported value is very similar to the bond distances observed in $(PPh_3)_2Br_2(CO)_2W$, where bromide is in *trans* position to the carbonyl ligand [2.55–2.57 Å in Drew *et al.* (1982), Cotton & Meadows (1984)].

Experimental

$[W(Br)_2(NO)(dmpe)_2]Br$ was prepared from the complex $[W(Br)_3(NO)(CH_3CN)_2]$, which is easily synthesized by the reaction of $W(Br)_5$ with gaseous NO in dibromomethane in the presence of acetonitrile at room temperature according to the literature procedure (Johnson, 1967; Berg & Dehnicke, 1985). 3.00 g (5.14 mmol) of $W(Br)_5$ and 0.54 ml (10.28 mmol) of acetonitrile were dissolved in 100 ml of dibromomethane in a 250 ml three-necked flask. Nitric oxide was passed through the solution, which was stirred at room temperature until the black colour of the solution turned to a light green precipitate after *ca* 1 h. The solution was concentrated to one third of its original volume and the addition of pentane (10 ml) afforded a green–yellow voluminous precipitate, which was filtered off, washed with pentane and dried in vacuum. Then $[W(Br)_2(NO)(dmpe)_2]Br$ (0.188 g, 0.25 mmol) was added to a stirred suspension of 1% sodium amalgam (0.026 g of Na, 1.12 mmol) in 20 ml of tetrahydrofurane. The mixture was then stirred overnight at room temperature. The solution was filtered off, separated from the mercury-containing residue, and the solvent was removed under vacuum. The residue was washed with pentane (10 ml *x* 2) and extracted with tetrahydrofurane (20 ml), concentrated and cooled to −30°C overnight yielding the title compound in the form of yellow crystals.

Yield: 0.126 g (85%).

IR (cm^{-1}, CH_2Cl_2): (NO) 1535.

^{1}H NMR (200.0 MHz, THF-d$_8$, 25°C): d 1.55 (m, 4H, P(C*H*$_2$)$_2$P); 1.49 (s, 24H, PC*H*$_3$) and 1.29 (m, 4H, P(C*H*$_2$)$_2$P).

supplementary materials

^{31}P{^{1}H} NMR (80.9 MHz, THF-d$_8$, 25°C): d 17.5 (s, ^{1}J$_{PW}$ = 380 Hz).

^{13}C{^{1}H} NMR (50.3 MHz, THF-d$_8$, 25°C): d 32.4 (m, P(CH$_2$)$_2$P); 15.1 (m, PCH$_3$); 14.4 (m, PCH$_3$).

Anal. Calcd for C$_{12}$H$_{32}$BrNOP$_4$W: C, 24.25; H, 5.39; N, 2.36. Found: C,24.60; H, 5.43; N, 2.28.

Refinement

The initial refinement of the structure produced very large thermal parameters for bromine, nitrogen, oxygen and all carbon atoms, especially for C2, C5 and C6 (> 0.175), and inadequate geometry of the dmpe ligand with coplanar P1, C1, C2 and P2 atoms. The location of the highest residual peaks showed unambigously that each of the Br, N, O and C atoms are distributed over two sites. The introduction of the disordered model, with respect to NO/Br and all carbon atoms of dmpe, yielded significantly lower discrepancy factors and ensured reasonable geometry of the dmpe ligand. Nevertheless, the highly disordered refinement model prompted us to consider possibility of alternative space groups (P2/c, P2 and Pc). However, none of them allowed to carry out reasonable refinement of the structure. Therefore we ended up with the P2$_1$/c refinement with significantly disordered model.

All hydrogen atoms were included at calculated positions and treated as riding atoms with C—H distances of 0.96–0.97 Å and U$_{iso}$(H) = 1.3U$_{eq}$(C). Positional disorders were refined with an occupancy factor of 0.5 for the *trans* NO and Br ligands since the metal atom occupies a special position in the inversion centre; the occupancy factors for two components of the dmpe disorder were deterimined by the refinement. The temperature factors of the C atoms of the dmpe ligand were refined with the SIMU, DELU and EADP restraints (Sheldrick, 1997). The largest positive and negative residual peaks are located at about 0.9 Å from P2 and W1, respectively; no chemical meaning could be attributed to these features.

Figures
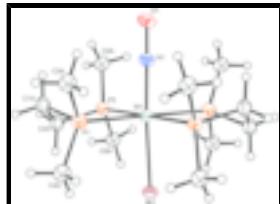
Fig. 1. The molecular structure of (I) with the atom-labeling scheme (displacement ellipsoids are drawn at the 20% probability level). The unlabeled atoms are derived from the corresponding labeled atoms by the 1 − x, −y, 1 − z symmetry transformation. Only one component of the disorder is shown for the nitrosyl/bromide groups as well as for the carbon atoms of the dmpe ligand.

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Crystal data

[WBr(NO)(C$_6$H$_{16}$P$_2$)$_2$]	F$_{000}$ = 576
M$_r$ = 594.01	D$_x$ = 1.819 Mg m^{-3}
Monoclinic, P2$_1$/c	Mo Kα radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 7998 reflections
a = 8.8909 (14) Å	θ = 3.1–30.3°
b = 12.5386 (16) Å	μ = 7.46 mm^{-1}
c = 12.823 (2) Å	T = 183 (2) K

$\beta = 130.639\,(14)°$

$V = 1084.8\,(4)$ Å^3

$Z = 2$

Block, yellow

$0.45 \times 0.39 \times 0.28$ mm

Data collection

Stoe IPDS diffractometer	1415 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.076$
$T = 183(2)$ K	$\theta_{max} = 25.0°$
φ rotation scan	$\theta_{min} = 3.4°$
Absorption correction: numerical (Coppens *et al.*, 1965)	$h = -10 \rightarrow 8$
$T_{min} = 0.086$, $T_{max} = 0.213$	$k = 0 \rightarrow 14$
19322 measured reflections	$l = 0 \rightarrow 15$
1901 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.034$	H-atom parameters constrained
$wR(F^2) = 0.089$	$w = 1/[\sigma^2(F_o^2) + (0.0711P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.92$	$(\Delta/\sigma)_{max} < 0.001$
1901 reflections	$\Delta\rho_{max} = 2.37$ e Å^{-3}
133 parameters	$\Delta\rho_{min} = -1.59$ e Å^{-3}
45 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 $F^2 > 2sigma(F^2)$ 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)
W1	0.5000	0.0000	0.5000	0.03782 (16)	

P1	0.4214 (3)	0.02436 (17)	0.2816 (2)	0.0576 (5)	
P2	0.2435 (3)	−0.13470 (15)	0.36543 (19)	0.0493 (4)	
Br1	0.2422 (4)	0.1376 (2)	0.4403 (3)	0.0554 (6)	0.50
N1	0.693 (3)	−0.0969 (14)	0.5538 (17)	0.056 (4)	0.50
O1	0.822 (3)	−0.1666 (17)	0.590 (2)	0.090 (8)	0.50
C1A	0.273 (3)	−0.0892 (15)	0.1748 (19)	0.065 (2)	0.483 (11)
H1A1	0.2038	−0.0732	0.0792	0.085*	0.483 (11)
H1A2	0.3570	−0.1507	0.2014	0.085*	0.483 (11)
C2A	0.119 (2)	−0.1137 (15)	0.1940 (15)	0.065 (2)	0.483 (11)
H2A1	0.0427	−0.1766	0.1415	0.085*	0.483 (11)
H2A2	0.0284	−0.0543	0.1606	0.085*	0.483 (11)
C3A	0.631 (3)	0.0151 (15)	0.286 (2)	0.063 (3)	0.483 (11)
H3A1	0.5828	0.0085	0.1941	0.082*	0.483 (11)
H3A2	0.7099	−0.0463	0.3387	0.082*	0.483 (11)
H3A3	0.7112	0.0782	0.3279	0.082*	0.483 (11)
C4A	0.292 (3)	0.1405 (15)	0.185 (3)	0.063 (3)	0.483 (11)
H4A1	0.3818	0.2000	0.2248	0.082*	0.483 (11)
H4A2	0.1867	0.1541	0.1856	0.082*	0.483 (11)
H4A3	0.2384	0.1304	0.0921	0.082*	0.483 (11)
C5A	0.322 (3)	−0.2708 (16)	0.407 (2)	0.063 (3)	0.483 (11)
H5A1	0.2086	−0.3167	0.3496	0.082*	0.483 (11)
H5A2	0.3872	−0.2837	0.5015	0.082*	0.483 (11)
H5A3	0.4120	−0.2851	0.3911	0.082*	0.483 (11)
C6A	0.034 (3)	−0.1313 (15)	0.3669 (19)	0.063 (3)	0.483 (11)
H6A1	0.0018	−0.0586	0.3680	0.082*	0.483 (11)
H6A2	0.0735	−0.1674	0.4473	0.082*	0.483 (11)
H6A3	−0.0792	−0.1663	0.2862	0.082*	0.483 (11)
C1B	0.188 (3)	−0.0582 (13)	0.1376 (16)	0.065 (2)	0.517 (11)
H1B1	0.0688	−0.0217	0.1056	0.085*	0.517 (11)
H1B2	0.1835	−0.0676	0.0603	0.085*	0.517 (11)
C2B	0.206 (3)	−0.1678 (12)	0.2005 (15)	0.065 (2)	0.517 (11)
H2B1	0.3174	−0.2078	0.2240	0.085*	0.517 (11)
H2B2	0.0858	−0.2094	0.1363	0.085*	0.517 (11)
C3B	0.574 (3)	−0.0126 (15)	0.245 (2)	0.075 (3)	0.517 (11)
H3B1	0.4959	−0.0165	0.1477	0.097*	0.517 (11)
H3B2	0.6320	−0.0810	0.2856	0.097*	0.517 (11)
H3B3	0.6767	0.0395	0.2832	0.097*	0.517 (11)
C4B	0.330 (3)	0.1570 (14)	0.196 (3)	0.075 (3)	0.517 (11)
H4B1	0.3094	0.1558	0.1129	0.097*	0.517 (11)
H4B2	0.4258	0.2107	0.2567	0.097*	0.517 (11)
H4B3	0.2068	0.1728	0.1750	0.097*	0.517 (11)
C5B	0.280 (3)	−0.2738 (16)	0.422 (2)	0.075 (3)	0.517 (11)
H5B1	0.1836	−0.3183	0.3446	0.097*	0.517 (11)
H5B2	0.2631	−0.2794	0.4889	0.097*	0.517 (11)
H5B3	0.4106	−0.2966	0.4625	0.097*	0.517 (11)
C6B	−0.007 (3)	−0.1078 (15)	0.282 (2)	0.075 (3)	0.517 (11)
H6B1	−0.0583	−0.0568	0.2102	0.097*	0.517 (11)
H6B2	−0.0158	−0.0794	0.3475	0.097*	0.517 (11)
H6B3	−0.0822	−0.1725	0.2438	0.097*	0.517 (11)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
W1	0.0415 (2)	0.0316 (2)	0.0478 (2)	−0.00644 (16)	0.03232 (17)	−0.00255 (17)
P1	0.0701 (12)	0.0545 (12)	0.0644 (11)	0.0032 (9)	0.0509 (11)	0.0073 (9)
P2	0.0425 (9)	0.0415 (10)	0.0570 (10)	−0.0105 (8)	0.0293 (8)	0.0000 (8)
Br1	0.0483 (14)	0.0447 (15)	0.0639 (14)	0.0138 (11)	0.0323 (13)	0.0059 (12)
N1	0.062 (11)	0.047 (10)	0.059 (8)	0.013 (8)	0.040 (8)	0.012 (8)
O1	0.095 (16)	0.086 (15)	0.075 (12)	0.049 (10)	0.049 (12)	0.029 (9)
C1A	0.066 (6)	0.064 (6)	0.048 (4)	−0.007 (4)	0.029 (4)	−0.007 (4)
C2A	0.066 (6)	0.064 (6)	0.048 (4)	−0.007 (4)	0.029 (4)	−0.007 (4)
C3A	0.068 (6)	0.059 (6)	0.070 (6)	−0.005 (5)	0.048 (5)	−0.002 (5)
C4A	0.068 (6)	0.059 (6)	0.070 (6)	−0.005 (5)	0.048 (5)	−0.002 (5)
C5A	0.068 (6)	0.059 (6)	0.070 (6)	−0.005 (5)	0.048 (5)	−0.002 (5)
C6A	0.068 (6)	0.059 (6)	0.070 (6)	−0.005 (5)	0.048 (5)	−0.002 (5)
C1B	0.066 (6)	0.064 (6)	0.048 (4)	−0.007 (4)	0.029 (4)	−0.007 (4)
C2B	0.066 (6)	0.064 (6)	0.048 (4)	−0.007 (4)	0.029 (4)	−0.008 (4)
C3B	0.079 (7)	0.068 (6)	0.077 (6)	−0.015 (5)	0.051 (5)	−0.007 (5)
C4B	0.079 (7)	0.068 (6)	0.077 (6)	−0.015 (5)	0.051 (5)	−0.007 (5)
C5B	0.079 (7)	0.068 (6)	0.077 (6)	−0.015 (5)	0.051 (5)	−0.007 (5)
C6B	0.079 (7)	0.068 (6)	0.077 (6)	−0.015 (5)	0.051 (5)	−0.007 (5)

Geometric parameters (Å, °)

W1—N1[i]	1.83 (2)	C3A—H3A3	0.9600
W1—N1	1.83 (2)	C4A—H4A1	0.9600
W1—P1[i]	2.424 (2)	C4A—H4A2	0.9600
W1—P1	2.424 (2)	C4A—H4A3	0.9600
W1—P2[i]	2.4302 (18)	C5A—H5A1	0.9600
W1—P2	2.4302 (18)	C5A—H5A2	0.9600
W1—Br1[i]	2.555 (3)	C5A—H5A3	0.9600
W1—Br1	2.555 (3)	C6A—H6A1	0.9600
P1—C3B	1.76 (2)	C6A—H6A2	0.9600
P1—C4A	1.77 (2)	C6A—H6A3	0.9600
P1—C1A	1.814 (16)	C1B—C2B	1.551 (17)
P1—C3A	1.833 (18)	C1B—H1B1	0.9700
P1—C4B	1.86 (2)	C1B—H1B2	0.9700
P1—C1B	1.946 (15)	C2B—H2B1	0.9700
P2—C2A	1.720 (15)	C2B—H2B2	0.9700
P2—C6B	1.763 (17)	C3B—H3B1	0.9600
P2—C5A	1.788 (19)	C3B—H3B2	0.9600
P2—C5B	1.836 (19)	C3B—H3B3	0.9600
P2—C6A	1.871 (16)	C4B—H4B1	0.9600
P2—C2B	1.957 (16)	C4B—H4B2	0.9600
N1—O1	1.267 (19)	C4B—H4B3	0.9600
C1A—C2A	1.566 (18)	C5B—H5B1	0.9600
C1A—H1A1	0.9700	C5B—H5B2	0.9600

C1A—H1A2	0.9700	C5B—H5B3	0.9600
C2A—H2A1	0.9700	C6B—H6B1	0.9600
C2A—H2A2	0.9700	C6B—H6B2	0.9600
C3A—H3A1	0.9600	C6B—H6B3	0.9600
C3A—H3A2	0.9600		
N1—W1—Br1	176.5 (5)	H1A1—C1A—H1A2	108.4
O1—N1—W1	177.9 (19)	C1A—C2A—P2	109.3 (12)
N1^i—W1—P1^i	90.8 (5)	C1A—C2A—H2A1	109.8
N1—W1—P1^i	89.2 (5)	P2—C2A—H2A1	109.8
N1—W1—P1	90.8 (5)	C1A—C2A—H2A2	109.8
N1^i—W1—P1	89.2 (5)	P2—C2A—H2A2	109.8
N1^i—W1—P2^i	91.3 (5)	H2A1—C2A—H2A2	108.3
N1—W1—P2^i	88.7 (5)	P1—C3A—H3A1	109.5
P1^i—W1—P2^i	80.57 (7)	P1—C3A—H3A2	109.5
P1—W1—P2^i	99.43 (7)	P1—C3A—H3A3	109.5
N1—W1—P2	91.3 (5)	P1—C4A—H4A1	109.5
N1^i—W1—P2	88.7 (5)	P1—C4A—H4A2	109.5
P1^i—W1—P2	99.43 (7)	P1—C4A—H4A3	109.5
P1—W1—P2	80.57 (7)	P2—C5A—H5A1	109.5
P1^i—W1—P1	180.0	P2—C5A—H5A2	109.5
P2^i—W1—P2	180.0	P2—C5A—H5A3	109.5
N1^i—W1—Br1^i	176.5 (5)	P2—C6A—H6A1	109.5
P1^i—W1—Br1^i	92.38 (9)	P2—C6A—H6A2	109.5
P1—W1—Br1^i	87.62 (9)	P2—C6A—H6A3	109.5
P2^i—W1—Br1^i	90.72 (7)	C2B—C1B—P1	107.1 (10)
P2—W1—Br1^i	89.28 (7)	C2B—C1B—H1B1	110.3
P1^i—W1—Br1	87.62 (9)	P1—C1B—H1B1	110.3
P1—W1—Br1	92.38 (9)	C2B—C1B—H1B2	110.3
P2^i—W1—Br1	89.28 (7)	P1—C1B—H1B2	110.3
P2—W1—Br1	90.72 (7)	H1B1—C1B—H1B2	108.5
C4A—P1—C1A	107.2 (10)	C1B—C2B—P2	105.3 (11)
C4A—P1—C3A	104.9 (9)	C1B—C2B—H2B1	110.7
C1A—P1—C3A	101.6 (9)	P2—C2B—H2B1	110.7
C3B—P1—C4B	101.4 (10)	C1B—C2B—H2B2	110.7
C3B—P1—C1B	97.8 (9)	P2—C2B—H2B2	110.7
C4B—P1—C1B	97.4 (9)	H2B1—C2B—H2B2	108.8
C3B—P1—W1	125.5 (8)	P1—C3B—H3B1	109.5
C4A—P1—W1	118.7 (10)	P1—C3B—H3B2	109.5
C1A—P1—W1	107.0 (6)	H3B1—C3B—H3B2	109.5
C3A—P1—W1	115.7 (7)	P1—C3B—H3B3	109.5
C4B—P1—W1	118.6 (10)	H3B1—C3B—H3B3	109.5
C1B—P1—W1	110.9 (5)	H3B2—C3B—H3B3	109.5
C2A—P2—C5A	109.0 (9)	P1—C4B—H4B1	109.5
C6B—P2—C5B	104.3 (9)	P1—C4B—H4B2	109.5
C2A—P2—C6A	101.2 (8)	H4B1—C4B—H4B2	109.5

C5A—P2—C6A	102.5 (8)	P1—C4B—H4B3	109.5
C6B—P2—C2B	97.2 (8)	H4B1—C4B—H4B3	109.5
C5B—P2—C2B	95.1 (8)	H4B2—C4B—H4B3	109.5
C2A—P2—W1	109.0 (6)	P2—C5B—H5B1	109.5
C6B—P2—W1	121.8 (6)	P2—C5B—H5B2	109.5
C5A—P2—W1	116.6 (7)	H5B1—C5B—H5B2	109.5
C5B—P2—W1	122.6 (7)	P2—C5B—H5B3	109.5
C6A—P2—W1	117.1 (6)	H5B1—C5B—H5B3	109.5
C2B—P2—W1	109.7 (5)	H5B2—C5B—H5B3	109.5
Br1[i]—N1—W1	167.8 (18)	P2—C6B—H6B1	109.5
C2A—C1A—P1	108.5 (12)	P2—C6B—H6B2	109.5
C2A—C1A—H1A1	110.0	H6B1—C6B—H6B2	109.5
P1—C1A—H1A1	110.0	P2—C6B—H6B3	109.5
C2A—C1A—H1A2	110.0	H6B1—C6B—H6B3	109.5
P1—C1A—H1A2	110.0	H6B2—C6B—H6B3	109.5
N1[i]—W1—P1—C3B	162.8 (9)	P1[i]—W1—P2—C5B	−52.2 (8)
N1—W1—P1—C3B	−17.2 (9)	P1—W1—P2—C5B	127.8 (8)
P2[i]—W1—P1—C3B	71.6 (8)	Br1[i]—W1—P2—C5B	40.1 (8)
P2—W1—P1—C3B	−108.4 (8)	Br1—W1—P2—C5B	−139.9 (8)
Br1[i]—W1—P1—C3B	−18.7 (8)	N1[i]—W1—P2—C6A	−38.8 (9)
Br1—W1—P1—C3B	161.3 (8)	N1—W1—P2—C6A	141.2 (9)
N1[i]—W1—P1—C4A	19.6 (9)	P1[i]—W1—P2—C6A	51.8 (7)
N1—W1—P1—C4A	−160.4 (9)	P1—W1—P2—C6A	−128.2 (7)
P2[i]—W1—P1—C4A	−71.6 (7)	Br1[i]—W1—P2—C6A	144.1 (7)
P2—W1—P1—C4A	108.4 (7)	Br1—W1—P2—C6A	−35.9 (7)
Br1[i]—W1—P1—C4A	−161.9 (7)	N1[i]—W1—P2—C2B	107.4 (7)
Br1—W1—P1—C4A	18.1 (7)	N1—W1—P2—C2B	−72.6 (7)
N1[i]—W1—P1—C1A	−101.9 (8)	P1[i]—W1—P2—C2B	−162.1 (5)
N1—W1—P1—C1A	78.1 (8)	P1—W1—P2—C2B	17.9 (5)
P2[i]—W1—P1—C1A	167.0 (7)	Br1[i]—W1—P2—C2B	−69.8 (5)
P2—W1—P1—C1A	−13.0 (7)	Br1—W1—P2—C2B	110.2 (5)
Br1[i]—W1—P1—C1A	76.7 (7)	N1[i]—W1—Br1—O1[i]	−14 (13)
Br1—W1—P1—C1A	−103.3 (7)	P1[i]—W1—Br1—O1[i]	−169 (8)
N1[i]—W1—P1—C3A	145.7 (8)	P1—W1—Br1—O1[i]	11 (8)
N1—W1—P1—C3A	−34.3 (8)	P2[i]—W1—Br1—O1[i]	111 (8)
P2[i]—W1—P1—C3A	54.6 (7)	P2—W1—Br1—O1[i]	−69 (8)
P2—W1—P1—C3A	−125.4 (7)	P1[i]—W1—Br1—N1[i]	−155 (8)
Br1[i]—W1—P1—C3A	−35.7 (7)	P1—W1—Br1—N1[i]	25 (8)
Br1—W1—P1—C3A	144.3 (7)	P2[i]—W1—Br1—N1[i]	125 (8)
N1[i]—W1—P1—C4B	31.1 (9)	P2—W1—Br1—N1[i]	−55 (8)
N1—W1—P1—C4B	−148.9 (9)	P1[i]—W1—N1—Br1[i]	155 (8)
P2[i]—W1—P1—C4B	−60.0 (8)	P1—W1—N1—Br1[i]	−25 (8)
P2—W1—P1—C4B	120.0 (8)	P2[i]—W1—N1—Br1[i]	−125 (8)
Br1[i]—W1—P1—C4B	−150.4 (8)	P2—W1—N1—Br1[i]	55 (8)

Br1—W1—P1—C4B	29.6 (8)	C3B—P1—C1A—C2A	167.4 (15)
N1ⁱ—W1—P1—C1B	−80.3 (8)	C4A—P1—C1A—C2A	−86.4 (16)
N1—W1—P1—C1B	99.7 (8)	C3A—P1—C1A—C2A	163.8 (13)
P2ⁱ—W1—P1—C1B	−171.5 (6)	C4B—P1—C1A—C2A	−92.6 (16)
P2—W1—P1—C1B	8.5 (6)	C1B—P1—C1A—C2A	−62 (2)
Br1ⁱ—W1—P1—C1B	98.2 (6)	W1—P1—C1A—C2A	42.0 (14)
Br1—W1—P1—C1B	−81.8 (6)	P1—C1A—C2A—P2	−56.4 (16)
N1ⁱ—W1—P2—C2A	75.3 (9)	C6B—P2—C2A—C1A	162.2 (16)
N1—W1—P2—C2A	−104.7 (9)	C5A—P2—C2A—C1A	−84.5 (15)
P1ⁱ—W1—P2—C2A	165.8 (7)	C5B—P2—C2A—C1A	−100.6 (15)
P1—W1—P2—C2A	−14.2 (7)	C6A—P2—C2A—C1A	167.9 (13)
Br1ⁱ—W1—P2—C2A	−101.9 (7)	C2B—P2—C2A—C1A	−52.8 (13)
Br1—W1—P2—C2A	78.1 (7)	W1—P2—C2A—C1A	43.9 (14)
N1ⁱ—W1—P2—C6B	−4.9 (9)	C3B—P1—C1B—C2B	92.0 (14)
N1—W1—P2—C6B	175.1 (9)	C4A—P1—C1B—C2B	−161.1 (16)
P1ⁱ—W1—P2—C6B	85.7 (8)	C1A—P1—C1B—C2B	41.9 (18)
P1—W1—P2—C6B	−94.3 (8)	C3A—P1—C1B—C2B	93.2 (14)
Br1ⁱ—W1—P2—C6B	178.0 (8)	C4B—P1—C1B—C2B	−165.4 (15)
Br1—W1—P2—C6B	−2.0 (8)	W1—P1—C1B—C2B	−40.9 (14)
N1ⁱ—W1—P2—C5A	−160.8 (9)	P1—C1B—C2B—P2	53.8 (14)
N1—W1—P2—C5A	19.2 (9)	C2A—P2—C2B—C1B	46.2 (13)
P1ⁱ—W1—P2—C5A	−70.2 (7)	C6B—P2—C2B—C1B	79.5 (13)
P1—W1—P2—C5A	109.8 (7)	C5A—P2—C2B—C1B	−163.9 (14)
Br1ⁱ—W1—P2—C5A	22.1 (7)	C5B—P2—C2B—C1B	−175.4 (13)
Br1—W1—P2—C5A	−157.9 (7)	C6A—P2—C2B—C1B	95.7 (14)
N1ⁱ—W1—P2—C5B	−142.7 (9)	W1—P2—C2B—C1B	−48.1 (12)
N1—W1—P2—C5B	37.3 (9)		

Symmetry codes: (i) −x+1, −y, −z+1.

Fig. 1