Acta Crystallographica Section E
Structure Reports Online

ISSN 1600-5368

Bis(tetraphenylphosphonium) di-μ-iodido-bis[diiodidopalladate(II)]

Frank Mulligan,[a] Gary S. Nichol[b]* and Stephanie K. Hurst[a]

[a]Department of Chemistry and Biochemistry, Northern Arizona University, Box 5698, Flagstaff, AZ 86011, USA, and [b]Department of Chemistry, The University of Arizona, 1306 E University Boulevard, Tucson, AZ 85721, USA
Correspondence e-mail: gsnichol@email.arizona.edu

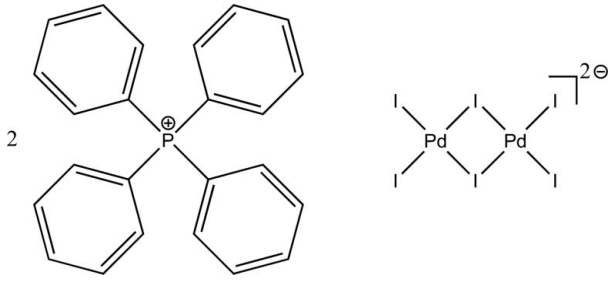
Received 13 September 2007; accepted 14 September 2007

Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.012 Å; R factor = 0.032; wR factor = 0.101; data-to-parameter ratio = 15.9.

The title compound, $(PPh_4)_2[Pd_2I_6]$, was obtained unintentionally as the product of an attempted synthesis of a tripalladium sandwich complex. The molecular dimensions are unexceptional and the Pd···Pd distance, at 3.8183 (12) Å, is much too long for any Pd—Pd interaction. Pd has a typical square-planar coordination geometry and the centrosymmetric anion is essentially planar.

Related literature

The PPh_3Me^+ salt has also been reported (Tonde *et al.*, 2005). For other examples of $[Pd_2I_6]^{2-}$, see Chan *et al.* (1996), Evans *et al.* (2002), Maassarani *et al.* (1987), Neve *et al.* (2000) and Neve & Crispini (2003). For the Cambridge Structural Database, see Allen (2002).



Experimental

Crystal data

$(C_{24}H_{20}P)_2[Pd_2I_6]$
M_r = 1652.94
Monoclinic, $P2_1/n$
a = 12.8951 (11) Å
b = 14.4427 (13) Å
c = 13.9409 (12) Å
β = 106.613 (1)°
V = 2488.0 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 4.54 mm^{-1}
T = 150 (2) K
0.39 × 0.24 × 0.05 mm

Data collection

Bruker SMART 1000 CCD diffractometer
Absorption correction: Gaussian (*SADABS*; Sheldrick, 2007)
T_{min} = 0.268, T_{max} = 0.805
15011 measured reflections
4154 independent reflections
2914 reflections with $I > 2\sigma(I)$
R_{int} = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.101
S = 1.14
4154 reflections
262 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.98 e Å^{-3}

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL* and local programs.

The work described in this paper was supported by the Northern Arizona University (NAU) Intramural Grant Program, the NAU College of Engineering and Natural Science start-up fund and the SABRE GBI program. The X-ray diffractometer was purchased with NSF grant CHE-9610374.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CF2142).

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  doi:10.1107/S160053680704528X

supplementary materials

Acta Cryst. (2007). E**63**, m2558 [**doi:10.1107/S160053680704528X**]

Bis(tetraphenylphosphonium) di-μ-iodido-bis[diiodidopalladate(II)]

F. Mulligan, G. S. Nichol and S. K. Hurst

Comment

The title compound, (I), was obtained unintentionally as the product of an attempted synthesis of a tripalladium sandwich complex. The $[Pd_2I_6]^{2-}$ ion lies on an inversion centre and thus the asymmetric unit is one-half of the complete chemical formula.

Both palladium centres have typical d^8 square-planar geometry and are bridged by two iodide ligands, with four terminal iodide ligands completing this discrete species. A search of the Cambridge Structural Database (Version 5.28 with two updates; Allen, 2002) shows that there are only 8 reported crystallographic examples of this unit. Two tetraphenylphosphonium cations balance the 2- charge. Molecular dimensions are unexceptional and the Pd···Pd distance, at 3.8183 (12) Å, is much too long for any Pd—Pd interaction.

The crystal packing consists mostly of coulombic and London forces, with the exception that the tetraphenylphosphonium cations show the typical phenyl embrace interactions usually encountered with this type of species.

Experimental

200 mg of $Pd_2(dba)_3$ (dba = dibenzylideneacetone) was stirred with 46 mg of $C_7H_7{\cdot}BF_4$ and 445 mg of PPh_4I in 30 ml of CH_2Cl_2 for 30 minutes at room temperature. The solvent was removed under vacuum and the residue recrystallized by slow evaporation of an acetonitrile solution. Yield = 12%. ^{1}H NMR: (400 MHz, $CDCl_3$): δ (p.p.m.) = 7.05 – 8.05 (40 H, m, Ph). ^{31}P NMR: (400 MHz, $CDCl_3$): δ (p.p.m.) = 24.2.

Refinement

All hydrogen atoms were initially located in a difference map and then refined using a riding model with U_{iso}(H)=1.2U_{eq}(C). The C–H distances were constrained to be 0.95 Å.

Figures

Fig. 1. Twice the asymmetric unit of (I) with displacement ellipsoids at the 50% probability level and hydrogen atoms shown as small spheres. Symmetry operation a: $-x+1,-y,-z+1$.

Bis(tetraphenylphosphonium) di-μ-iodidobis[diiodidodipalladate(II)]

Crystal data

$(C_{24}H_{20}P)_2[Pd_2I_6]$	F_{000} = 1536
M_r = 1652.94	D_x = 2.206 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 5989 reflections
a = 12.8951 (11) Å	θ = 2.4–27.3º
b = 14.4427 (13) Å	μ = 4.54 mm^{-1}
c = 13.9409 (12) Å	T = 150 (2) K
β = 106.613 (1)º	Plate, dark purple
V = 2488.0 (4) Å^3	0.39 × 0.24 × 0.05 mm
Z = 2	

Data collection

Bruker SMART 1000 CCD diffractometer	4154 independent reflections
Radiation source: sealed tube	2914 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.040
T = 150(2) K	θ_{max} = 25.0º
thin–slice ω scans	θ_{min} = 1.9º
Absorption correction: Gaussian (SADABS; Sheldrick, 2007)	h = −15→15
T_{min} = 0.268, T_{max} = 0.805	k = −17→17
15011 measured reflections	l = −16→16

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.032	H-atom parameters constrained
$wR(F^2)$ = 0.101	$w = 1/[\sigma^2(F_o^2) + (0.0253P)^2 + 19.5288P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.14	$(\Delta/\sigma)_{max}$ = 0.001
4154 reflections	$\Delta\rho_{max}$ = 1.19 e Å^{-3}
262 parameters	$\Delta\rho_{min}$ = −0.98 e Å^{-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 $F^2 > 2\text{sigma}(F^2)$ 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}
I1	0.59662 (4)	0.06678 (3)	0.59089 (4)	0.02764 (15)
I2	0.58538 (5)	−0.07752 (5)	0.24562 (5)	0.0469 (2)
I3	0.78898 (4)	0.05801 (4)	0.43871 (4)	0.03281 (16)
Pd	0.59643 (5)	−0.00576 (4)	0.41867 (4)	0.02353 (16)
P	0.81570 (15)	0.11432 (13)	0.04585 (14)	0.0193 (4)
C1	0.7167 (6)	0.1895 (5)	0.0730 (6)	0.0223 (17)
C2	0.6358 (6)	0.2255 (5)	−0.0063 (7)	0.0301 (19)
H2	0.6321	0.2096	−0.0733	0.036*
C3	0.5600 (7)	0.2853 (6)	0.0138 (7)	0.037 (2)
H3	0.5036	0.3098	−0.0398	0.044*
C4	0.5662 (7)	0.3091 (6)	0.1103 (7)	0.036 (2)
H4	0.5149	0.3510	0.1231	0.043*
C5	0.6462 (8)	0.2728 (7)	0.1890 (8)	0.047 (3)
H5	0.6500	0.2892	0.2559	0.056*
C6	0.7203 (7)	0.2128 (6)	0.1700 (6)	0.037 (2)
H6	0.7749	0.1870	0.2243	0.044*
C7	0.7644 (6)	−0.0016 (5)	0.0212 (5)	0.0220 (17)
C8	0.8366 (7)	−0.0763 (5)	0.0443 (6)	0.0265 (18)
H8	0.9119	−0.0664	0.0730	0.032*
C9	0.7950 (7)	−0.1646 (5)	0.0240 (6)	0.032 (2)
H9	0.8425	−0.2162	0.0406	0.038*
C10	0.6872 (7)	−0.1793 (6)	−0.0193 (6)	0.031 (2)
H10	0.6604	−0.2406	−0.0336	0.037*
C11	0.6170 (7)	−0.1048 (6)	−0.0423 (6)	0.034 (2)
H11	0.5420	−0.1150	−0.0726	0.040*
C12	0.6551 (6)	−0.0169 (5)	−0.0216 (6)	0.0264 (18)
H12	0.6065	0.0340	−0.0366	0.032*
C13	0.9359 (6)	0.1100 (5)	0.1492 (5)	0.0220 (16)
C14	1.0305 (7)	0.1553 (6)	0.1453 (6)	0.0313 (19)
H14	1.0316	0.1896	0.0875	0.038*
C15	1.1226 (7)	0.1500 (6)	0.2262 (6)	0.032 (2)
H15	1.1874	0.1794	0.2229	0.039*
C16	1.1208 (6)	0.1026 (5)	0.3110 (6)	0.0275 (18)

H16	1.1838	0.0997	0.3666	0.033*
C17	1.0267 (7)	0.0591 (6)	0.3149 (6)	0.0299 (19)
H17	1.0258	0.0264	0.3738	0.036*
C18	0.9347 (7)	0.0619 (6)	0.2357 (6)	0.0297 (19)
H18	0.8708	0.0314	0.2397	0.036*
C19	0.8461 (6)	0.1608 (5)	−0.0624 (6)	0.0218 (16)
C20	0.8435 (6)	0.1058 (5)	−0.1451 (6)	0.0251 (17)
H20	0.8225	0.0426	−0.1463	0.030*
C21	0.8716 (6)	0.1431 (5)	−0.2253 (6)	0.0275 (18)
H21	0.8693	0.1061	−0.2822	0.033*
C22	0.9033 (6)	0.2357 (5)	−0.2223 (6)	0.0245 (17)
H22	0.9238	0.2614	−0.2769	0.029*
C23	0.9050 (6)	0.2901 (5)	−0.1407 (6)	0.0250 (17)
H23	0.9264	0.3532	−0.1396	0.030*
C24	0.8760 (6)	0.2540 (5)	−0.0612 (6)	0.0255 (18)
H24	0.8763	0.2920	−0.0055	0.031*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.0230 (3)	0.0257 (3)	0.0314 (3)	−0.0031 (2)	0.0033 (2)	−0.0049 (2)
I2	0.0438 (4)	0.0617 (4)	0.0314 (3)	0.0086 (3)	0.0049 (3)	−0.0113 (3)
I3	0.0272 (3)	0.0357 (3)	0.0363 (3)	−0.0050 (2)	0.0104 (2)	0.0038 (3)
Pd	0.0214 (3)	0.0206 (3)	0.0261 (3)	0.0017 (2)	0.0028 (3)	0.0014 (3)
P	0.0183 (10)	0.0181 (9)	0.0213 (10)	−0.0010 (8)	0.0054 (8)	−0.0025 (8)
C1	0.017 (4)	0.022 (4)	0.030 (4)	−0.004 (3)	0.011 (3)	−0.003 (3)
C2	0.031 (5)	0.026 (4)	0.036 (5)	0.003 (4)	0.014 (4)	0.005 (4)
C3	0.032 (5)	0.033 (5)	0.051 (6)	0.016 (4)	0.022 (4)	0.025 (5)
C4	0.040 (5)	0.023 (4)	0.057 (6)	0.003 (4)	0.032 (5)	−0.002 (4)
C5	0.044 (6)	0.053 (6)	0.050 (6)	0.000 (5)	0.023 (5)	−0.025 (5)
C6	0.039 (5)	0.042 (5)	0.029 (5)	0.007 (4)	0.009 (4)	−0.003 (4)
C7	0.029 (4)	0.017 (4)	0.019 (4)	−0.003 (3)	0.005 (3)	0.003 (3)
C8	0.026 (4)	0.022 (4)	0.029 (4)	−0.001 (3)	0.005 (4)	−0.006 (4)
C9	0.045 (5)	0.022 (4)	0.031 (5)	0.007 (4)	0.013 (4)	0.000 (4)
C10	0.034 (5)	0.022 (4)	0.033 (5)	−0.009 (4)	0.002 (4)	−0.004 (4)
C11	0.028 (5)	0.028 (4)	0.038 (5)	−0.008 (4)	−0.002 (4)	0.000 (4)
C12	0.014 (4)	0.023 (4)	0.037 (5)	−0.003 (3)	−0.001 (3)	−0.002 (4)
C13	0.021 (4)	0.023 (4)	0.021 (4)	−0.004 (3)	0.005 (3)	−0.006 (3)
C14	0.031 (5)	0.027 (4)	0.034 (5)	−0.001 (4)	0.007 (4)	0.005 (4)
C15	0.023 (4)	0.038 (5)	0.034 (5)	−0.010 (4)	0.007 (4)	−0.007 (4)
C16	0.015 (4)	0.031 (4)	0.032 (4)	0.004 (3)	−0.002 (3)	−0.007 (4)
C17	0.038 (5)	0.028 (4)	0.023 (4)	0.005 (4)	0.008 (4)	−0.001 (4)
C18	0.029 (5)	0.034 (5)	0.027 (4)	−0.005 (4)	0.011 (4)	−0.010 (4)
C19	0.020 (4)	0.023 (4)	0.023 (4)	0.003 (3)	0.009 (3)	0.000 (3)
C20	0.022 (4)	0.020 (4)	0.031 (4)	−0.001 (3)	0.004 (3)	0.000 (4)
C21	0.029 (5)	0.029 (4)	0.024 (4)	0.004 (4)	0.007 (4)	0.001 (4)
C22	0.022 (4)	0.025 (4)	0.027 (4)	0.003 (3)	0.007 (3)	0.010 (4)
C23	0.025 (4)	0.023 (4)	0.026 (4)	0.000 (3)	0.007 (3)	0.003 (4)

C24	0.025 (4)	0.021 (4)	0.028 (4)	−0.003 (3)	0.004 (4)	−0.008 (4)

Geometric parameters (Å, °)

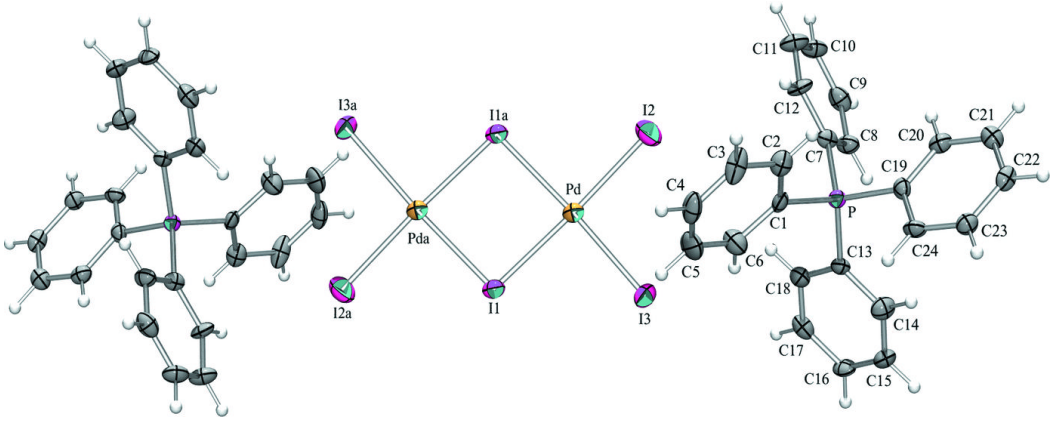
I1—Pd	2.6188 (8)	C10—C11	1.384 (11)
I1—Pd^{i}	2.6081 (8)	C11—H11	0.950
I2—Pd	2.5920 (9)	C11—C12	1.362 (11)
I3—Pd	2.5874 (8)	C12—H12	0.950
Pd—I1^{i}	2.6080 (8)	C13—C14	1.399 (11)
P—C1	1.796 (8)	C13—C18	1.396 (11)
P—C7	1.797 (7)	C14—H14	0.950
P—C13	1.790 (8)	C14—C15	1.387 (11)
P—C19	1.795 (8)	C15—H15	0.950
C1—C2	1.386 (11)	C15—C16	1.372 (11)
C1—C6	1.382 (11)	C16—H16	0.950
C2—H2	0.950	C16—C17	1.381 (11)
C2—C3	1.392 (11)	C17—H17	0.950
C3—H3	0.950	C17—C18	1.371 (11)
C3—C4	1.369 (12)	C18—H18	0.950
C4—H4	0.950	C19—C20	1.392 (10)
C4—C5	1.377 (13)	C19—C24	1.399 (10)
C5—H5	0.950	C20—H20	0.950
C5—C6	1.371 (12)	C20—C21	1.379 (11)
C6—H6	0.950	C21—H21	0.950
C7—C8	1.400 (10)	C21—C22	1.396 (11)
C7—C12	1.382 (10)	C22—H22	0.950
C8—H8	0.950	C22—C23	1.378 (11)
C8—C9	1.382 (11)	C23—H23	0.950
C9—H9	0.950	C23—C24	1.371 (11)
C9—C10	1.365 (11)	C24—H24	0.950
C10—H10	0.950		
Pd—I1—Pd^{i}	93.86 (2)	C10—C11—H11	119.9
I1—Pd—I1^{i}	86.14 (2)	C10—C11—C12	120.2 (8)
I1—Pd—I2	177.03 (3)	H11—C11—C12	119.9
I1^{i}—Pd—I2	91.12 (3)	C7—C12—C11	120.1 (7)
I1—Pd—I3	90.53 (3)	C7—C12—H12	119.9
I1^{i}—Pd—I3	176.65 (3)	C11—C12—H12	119.9
I2—Pd—I3	92.21 (3)	P—C13—C14	121.1 (6)
C1—P—C7	111.0 (3)	P—C13—C18	119.5 (6)
C1—P—C13	110.8 (3)	C14—C13—C18	119.4 (7)
C1—P—C19	106.9 (3)	C13—C14—H14	120.1
C7—P—C13	108.0 (3)	C13—C14—C15	119.8 (8)
C7—P—C19	110.1 (3)	H14—C14—C15	120.1
C13—P—C19	110.1 (3)	C14—C15—H15	119.8
P—C1—C2	118.5 (6)	C14—C15—C16	120.4 (8)
P—C1—C6	121.8 (6)	H15—C15—C16	119.8
C2—C1—C6	119.7 (7)	C15—C16—H16	120.2
C1—C2—H2	120.5	C15—C16—C17	119.5 (7)

C1—C2—C3	119.0 (8)	H16—C16—C17	120.2
H2—C2—C3	120.5	C16—C17—H17	119.2
C2—C3—H3	119.8	C16—C17—C18	121.5 (8)
C2—C3—C4	120.5 (8)	H17—C17—C18	119.2
H3—C3—C4	119.8	C13—C18—C17	119.3 (8)
C3—C4—H4	119.8	C13—C18—H18	120.4
C3—C4—C5	120.5 (8)	C17—C18—H18	120.4
H4—C4—C5	119.8	P—C19—C20	121.6 (6)
C4—C5—H5	120.3	P—C19—C24	118.4 (6)
C4—C5—C6	119.5 (9)	C20—C19—C24	120.0 (7)
H5—C5—C6	120.3	C19—C20—H20	120.1
C1—C6—C5	120.9 (9)	C19—C20—C21	119.9 (7)
C1—C6—H6	119.6	H20—C20—C21	120.1
C5—C6—H6	119.6	C20—C21—H21	120.2
P—C7—C8	119.3 (6)	C20—C21—C22	119.6 (7)
P—C7—C12	120.4 (6)	H21—C21—C22	120.2
C8—C7—C12	120.3 (7)	C21—C22—H22	119.8
C7—C8—H8	120.9	C21—C22—C23	120.4 (7)
C7—C8—C9	118.1 (7)	H22—C22—C23	119.8
H8—C8—C9	120.9	C22—C23—H23	119.8
C8—C9—H9	119.3	C22—C23—C24	120.4 (7)
C8—C9—C10	121.4 (8)	H23—C23—C24	119.8
H9—C9—C10	119.3	C19—C24—C23	119.7 (7)
C9—C10—H10	120.1	C19—C24—H24	120.2
C9—C10—C11	119.9 (7)	C23—C24—H24	120.2
H10—C10—C11	120.1		
Pd^{i}—I1—Pd—$I1^{i}$	0.0	C8—C7—C12—C11	−0.5 (12)
Pd^{i}—I1—Pd—I2	23.0 (6)	C1—P—C13—C14	−106.1 (7)
Pd^{i}—I1—Pd—I3	−179.59 (3)	C1—P—C13—C18	72.9 (7)
C7—P—C1—C2	−81.2 (7)	C7—P—C13—C14	132.1 (7)
C7—P—C1—C6	99.7 (7)	C7—P—C13—C18	−48.9 (7)
C13—P—C1—C2	158.9 (6)	C19—P—C13—C14	11.9 (8)
C13—P—C1—C6	−20.3 (8)	C19—P—C13—C18	−169.1 (6)
C19—P—C1—C2	39.0 (7)	P—C13—C14—C15	−179.4 (6)
C19—P—C1—C6	−140.2 (7)	C18—C13—C14—C15	1.6 (12)
P—C1—C2—C3	−178.7 (6)	C13—C14—C15—C16	−1.6 (12)
C6—C1—C2—C3	0.5 (12)	C14—C15—C16—C17	0.8 (12)
C1—C2—C3—C4	0.8 (12)	C15—C16—C17—C18	0.2 (12)
C2—C3—C4—C5	−1.3 (13)	C16—C17—C18—C13	−0.2 (12)
C3—C4—C5—C6	0.4 (14)	P—C13—C18—C17	−179.7 (6)
C4—C5—C6—C1	1.0 (14)	C14—C13—C18—C17	−0.7 (12)
P—C1—C6—C5	177.7 (7)	C1—P—C19—C20	−129.3 (6)
C2—C1—C6—C5	−1.4 (13)	C1—P—C19—C24	52.6 (7)
C1—P—C7—C8	−150.3 (6)	C7—P—C19—C20	−8.7 (7)
C1—P—C7—C12	30.7 (7)	C7—P—C19—C24	173.3 (6)
C13—P—C7—C8	−28.6 (7)	C13—P—C19—C20	110.2 (6)
C13—P—C7—C12	152.4 (6)	C13—P—C19—C24	−67.8 (7)
C19—P—C7—C8	91.6 (7)	P—C19—C20—C21	−177.4 (6)

C19—P—C7—C12	−87.4 (7)	C24—C19—C20—C21	0.6 (11)
P—C7—C8—C9	−179.7 (6)	C19—C20—C21—C22	0.6 (11)
C12—C7—C8—C9	−0.7 (12)	C20—C21—C22—C23	−1.1 (11)
C7—C8—C9—C10	1.5 (12)	C21—C22—C23—C24	0.3 (11)
C8—C9—C10—C11	−1.0 (13)	C22—C23—C24—C19	1.0 (11)
C9—C10—C11—C12	−0.2 (13)	P—C19—C24—C23	176.7 (6)
C10—C11—C12—C7	0.9 (13)	C20—C19—C24—C23	−1.4 (11)
P—C7—C12—C11	178.5 (7)		

Symmetry codes: (i) −*x*+1, −*y*, −*z*+1.

Fig. 1


I3a
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C14
C17
C16
C15