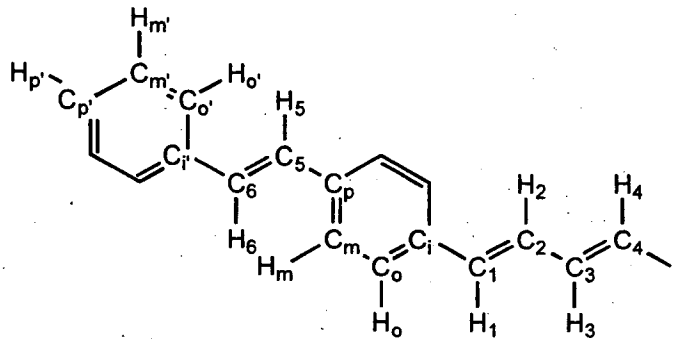
Experimental Section

Synthesis of $[Pd_3\{p\text{-}^tBuC_6H_4(CH{=}CH)_4C_6H_4{}^tBu\text{-}p\}_2][BF_4]_2$ (2'): A solution of $[Pd_2(CH_3CN)_6][BF_4]_2$ (327.9 mg, 518.2 μmol), $p\text{-}^tBuC_6H_4(CH{=}CH)_4C_6H_4{}^tBu\text{-}p$ (558.2 mg, 1.506 mmol) and $Pd_2(dba)_3{\cdot}CHCl_3$ (267.8 mg, 258.7 mmol) in CH_2Cl_2 was stirred for 23 h at room temperature. The solution was filtered off, and crystallization from CH_2Cl_2/benzene gave orange microcrystals of **2'** in 69% yield. For **2'-rac**: ^1H NMR (CD_2Cl_2, 25 °C) δ 7.31 (d, J = 7.8 Hz, 8H, *m*-Ar) 7.12 (d, J = 7.8 Hz, 8H, *o*-Ar) 5.70 (m, 8H, H1 and H2) 3.27 (m, 8H, H3 and H4) 1.45 (s, 36H, *t*-Bu). $^{13}C\{^1H\}$ NMR (CD_2Cl_2, 25 °C) δ 153.90 (s, *p*-Ar) 131.77 (s, *i*-Ar) 128.52 (s, *o*-Ar) 126.38 (s, *m*-Ar) 117.96 (s, C1) 95.77 (s, C2) 94.78 (s, C3 or C4) 91.51 (s, C3 or C4) 35.21 (s, $C(CH_3)_3$) 31.46 (s, $C(CH_3)_3$). For **2'-meso**: ^1H NMR (CD_2Cl_2, 25 °C) δ 7.25 (d, J = 8.7 Hz, 8H, *m*-Ph) 7.19 (d, J = 8.7 Hz, *o*-Ph) 6.66 (d, J = 13.7 Hz, 4H, H1) 5.33 (dd, J = 13.9 Hz, J = 10.8 Hz, 4H, H2) 3.61 (m, 4H, H3) 3.12 (m, 4H, H4) 1.34 (s, 36H, *t*-Bu). $^{13}C\{^1H\}$ NMR (CD_2Cl_2, 25 °C) δ 155.10 (s, *p*-Ar) 130.32 (s, *m*-Ar) 129.62 (s, *o*-Ar) 129.03 (s, *i*-Ph) 125.76 (s, C1) 95.09 (s, C2) 93.32 (s, C3) 87.14 (s, C4) 35.19 (s, $C(CH_3)_3$) 31.53 (s, $C(CH_3)_3$). Anal. Calcd. for $C_{56}H_{68}B_2F_8Pd_3$: C, 54.51; H, 5.55. Found: C, 54.12; H, 5.58.

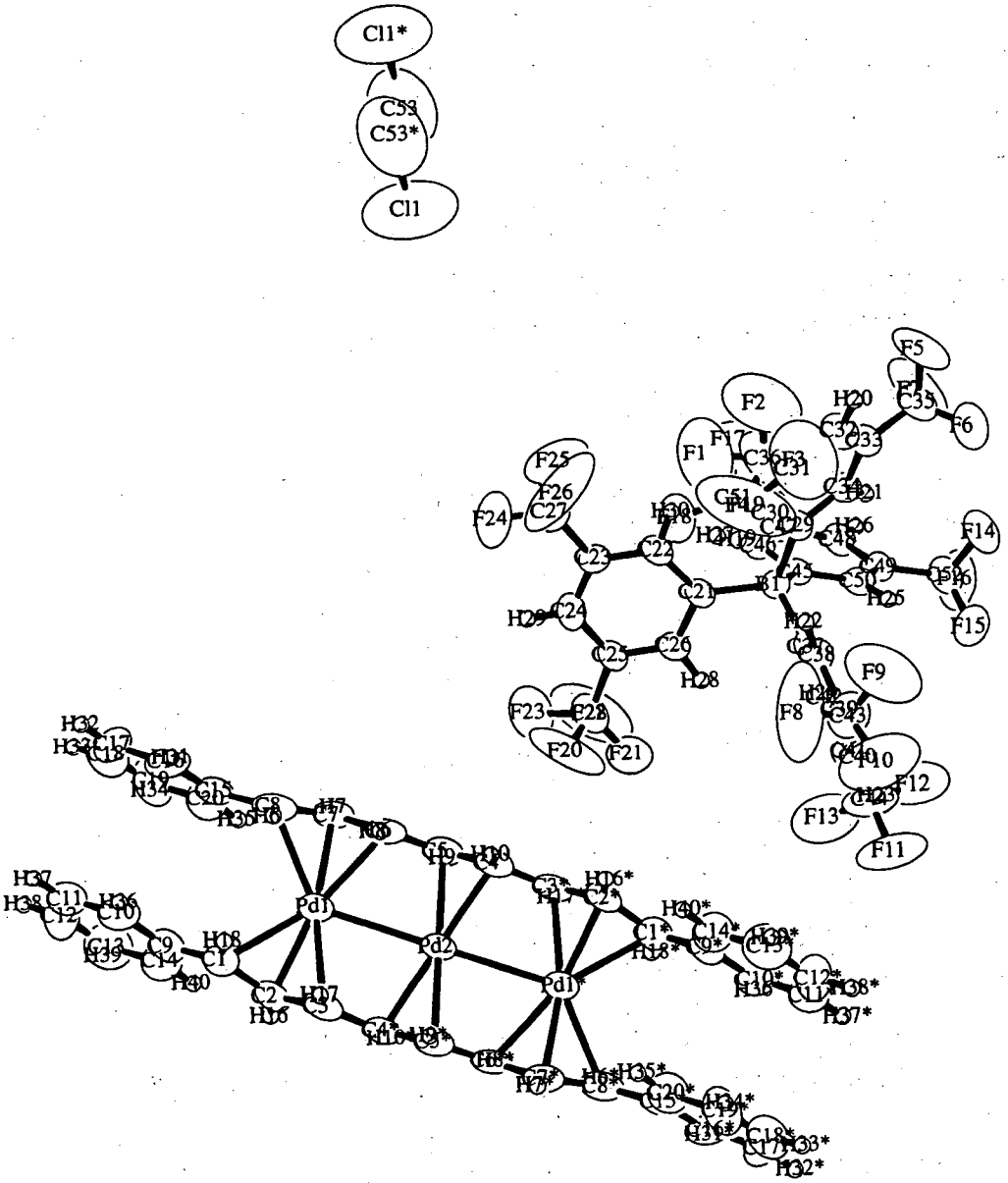
Synthesis of $[Pd_3\{p\text{-}^tBuC_6H_4(CH{=}CH)_4C_6H_4{}^tBu\text{-}p\}_2][BAr_f]_2$ (2): To a solution of **2'** (169.5 mg, 137.4 μmol) in CH_2Cl_2 was added $NaBAr_f$ (244.3 mg, 275.7 μmol). After the mixture was stirred for 20 min, it was filtered off, and crystallization from CH_2Cl_2/benzene afforded deep-red powders of **2** in 65% yield. For **2-rac**: ^1H NMR (CD_2Cl_2, 25 °C) δ 7.67 (br t, 16H, BAr_f) 7.52 (br s, 8H, BAr_f) 7.38 (d, J = 7.8 Hz, 8H, *m*-Ar) 7.05 (d, J = 7.8 Hz, 8H, *o*-Ar) 5.79 (d, J = 13.7 Hz, 4H, H1) 5.58 (dd, J = 13.7 Hz, J = 11.2 Hz, 4H, H2) 3.04 (m, 4H, H3) 2.92 (m, 4H, H4) 1.45 (s, *t*-Bu). $^{13}C\{^1H\}$ NMR (CD_2Cl_2, 25 °C) δ 156.92 (s, *p*-Ar) 129.64 (s, o-Ar) 129.01 (s, *i*-Ph) 127.09 (s, *m*-Ph) 123.38 (s, C1) 94.19 (s, C3) 92.85 (s, C2) 89.24 (s, C4) 35.56 (s, $C(CH_3)_3$) 31.36 (s, $C(CH_3)_3$). For **2-meso**: ^1H NMR (CD_2Cl_2, 25 °C) δ 7.67 (br t, 16H, BAr_f) 7.52 (br s, 8H, BAr_f) 7.31 (t, J = 8.5 Hz, 8H, *m*-Ar) 7.13 (d, J = 8.5 Hz, 8H, *o*-Ar) 6.60 (d, J = 13.8 Hz, 4H, H1) 5.23 (dd, J = 13.8 Hz, J = 11.1 Hz, 4H, H2) 3.30 (m, 4H, H3) 2.89 (m, 4H, H4) 1.35 (s, 36H, *t*-Bu). $^{13}C\{^1H\}$ NMR (CD_2Cl_2, 25 °C) δ 158.14 (s, *p*-Ar)

129.60 (s, *o*-Ar) 128.28 (s, *i*-Ar) 126.82 (s, *m*-Ar) 125.57 (s, C1) 93.43 (s, C2) 92.65 (s, C3) 87.32 (s, C4) 35.55 (s, $C(CH_3)_3$) 31.44 (s, $C(CH_3)_3$). Anal. Calcd. For $C_{120}H_{92}B_2F_{48}Pd_3$: C, 51.72; H, 3.33. Found C, 51.72; H, 3.36.

Synthesis of $[Pd_3\{PhCH=CHC_6H_4(CH=CH)_4C_6H_4CH=CHPh\}_2][BAr_f]_2$ (3): Treatment of $[Pd_2(CH_3CN)_6][BF_4]_2$ (14.8 mg, 23.4 μmol) and $Pd_2(dba)_3{\cdot}CHCl_3$ (12.1 mg, 11.7 μmol) with excess 1,4-diphenyl-1,3-butadiene (12.1 mg, 58.5 μmol), followed by addition of $NaBAr_f$ (41.5 mg, 46.8 μmol) in a manner similar to that for **2** described above afforded a supposedly Pd_3-diphenylbutadiene complex of the formula $[Pd_3\{Ph(CH=CH)_2Ph\}_2][BAr_f]_2$. This was used, without purification, for further reaction with *p*-styryl$C_6H_4(CH=CH)_4C_6H_4$sytyryl-*p* (28.5 mg, 61.6 μmol) in $C_2H_4Cl_2$ under reflux for 1h. The solution was filtered off, and the solvent was removed in vacuo. Recrystallization from CH_2Cl_2/n-hexane gave deep purple powders of **3** in 54% yield based on $[Pd_2(CH_3CN)_6][BF_4]_2$. For **3-rac**: ^{1}H NMR (CD_2Cl_2, 25 °C) δ 5.78 (dd, J = 10.7 Hz, J = 13.7 Hz, 4H, H2) 5.64 (d, J = 13.7 Hz, 4H, H1) 2.98 (m, 4H, H4) 2.91 (m, 4H, H3). The signals for *p*-styrylC_6H_4 protons could not be assigned. For **3-meso**: ^{1}H NMR (CD_2Cl_2, 25 °C) δ 7.68 (br s, BAr_f) 7.52 (br s, BAr_f) 7.32 (d, J = 7.2 Hz, 4H, Ho') 7.3-7.2 (m, H_m, H_o, H_m, $H_{6'}$, $H_{p'}$) 7.06 (d, J = 16.8 Hz, H5) 6.91 (d, J = 7.8 Hz, H_o) 6.46 (d, J = 14.2 Hz, 4H, H1) 5.18 (dd, J = 13.7 Hz, J = 11.2 Hz, 4H, H2) 3.45 (m, 4H, H3) 2.86 (m, 4H, H4). ^{13}C{^{1}H} NMR (CD_2Cl_2, 25 °C) for non-aromatic carbons δ 133.26 (s, C6) 127.01 (s, C5) 125.53 (s, C1) 93.84 (s, C2) 93.31 (s, C3) 86.57 (s, C4). Anal. Calcd. for $C_{136}H_{84}B_2F_{48}Pd_3$: C, 54.98; H, 2.85. Found: C, 54.57; H, 2.89.



X-ray Data for 1-meso



Experimental

Data Collection

A red prism crystal of $C_{106}H_{64}Pd_3B_2F_{48}Cl_2$ having approximate dimensions of 0.30 x 0.50 x 0.20 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS-RAPID Imaging Plate diffractometer with graphite monochromated Mo-Kα radiation.

Indexing was performed from 2 oscillations which were exposed for 3.3 minutes. The camera radius was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Cell constants and an orientation matrix for data collection corresponded to a primitive triclinic cell with dimensions:

a = 14.5246(7) $\AA$	α = 106.848(9)°
b = 16.0632(4) $\AA$	β = 100.968(5)°
c = 12.9564(9) $\AA$	γ = 70.382(8)°
V = 2710.6(3) $\AA^3$	

For Z = 1 and F.W. = 2661.32, the calculated density is 1.63 g/cm^3. Based on a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

$P\bar{1}$ (#2)

The data were collected at a temperature of 23 $\pm$ 1°C to a maximum 2θ value of 55.0°. A total of 44 images, corresponding to 220.0 ° oscillation angles, were collected with 2 different goniometer settings. Exposure time was 0.70 minutes per degree. The camera radius was 127.40 mm. Readout was performed in the 0.100 mm pixel mode. Data were processed by the PROCESS-AUTO program package.

Data Reduction

Of the 23545 reflections which were collected, 11969 were unique (R_{int} = 0.052); equivalent reflections were merged.

The linear absorption coefficient, μ, for Mo-Kα radiation is 6.7 cm^{-1}. A symmetry-related absorption correction using the program ABSCOR[1] was applied which resulted in transmission factors ranging from 0.55 to 0.87. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by heavy-atom Patterson methods[2] and expanded using Fourier techniques[3]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included but not refined. The final cycle of full-matrix least-squares refinement[4] was based on 6151 observed reflections (I > 3.00σ(I), 2θ

< 54.96) and 745 variable parameters and converged (largest parameter shift was 1.30 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma||Fo| - |Fc||/\Sigma|Fo| = 0.075$$

$$R_w = \sqrt{\Sigma w(|Fo| - |Fc|)^2/\Sigma wFo^2} = 0.063$$

$$R1 = \Sigma||Fo| - |Fc||/\Sigma|Fo| = 0.075 \quad for \;\; I > 3.0\sigma(I) \;\; data$$

The standard deviation of an observation of unit weight[5] was 3.71. The weighting scheme was based on counting statistics and included a factor (p = 0.010) to downweight the intense reflections. Plots of $\Sigma w(|Fo| - |Fc|)^2$ versus $|Fo|$, reflection order in data collection, $\sin\theta/\lambda$ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 1.96 and -0.71 $e^-/\AA^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[6] . Anomalous dispersion effects were included in Fcalc[7]; the values for Δf' and Δf" were those of Creagh and McAuley[8]. The values for the mass attenuation coefficients are those of Creagh and Hubbel[9]. All calculations were performed using the teXsan[10] crystallographic software package of Molecular Structure Corporation.

References

(1) ABSCOR: Higashi T. (1995). Program for Absorption Correction, Rigaku Corporation, Tokyo, Japan.

(2) PATTY: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M. (1994). The DIRDIF-94 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) DIRDIF94: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M. (1994). The DIRDIF-94 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(4) Least-Squares:

Function minimized: $\Sigma w(|Fo| - |Fc|)^2$

where w = $\frac{1}{\sigma^2(Fo)} = [\sigma_c^2(Fo) + \frac{p^2}{4}Fo^2]^{-1}$

$\sigma_c(Fo)$ = e.s.d. based on counting statistics

p = p-factor

(5) Standard deviation of an observation of unit weight:

$$\sqrt{\Sigma w(|Fo| - |Fc|)^2/(No - Nv)}$$

where: No = number of observations

Nv = number of variables

(6) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(7) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(8) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(9) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(10) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1999).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{106}H_{64}Pd_3B_2F_{48}Cl_2$
Formula Weight	2661.32
Crystal Color, Habit	red, prism
Crystal Dimensions	0.30 X 0.50 X 0.20 mm
Crystal System	triclinic
Lattice Type	Primitive
No. of Reflections Used for Unit Cell Determination (2θ range)	11970 (5.4 - 55.0°)
Indexing Images	2 oscillations at 3.3 minutes
Camera Radius	127.40 mm
Lattice Parameters	a = 14.5246(7) Å b = 16.0632(4) Å c = 12.9564(9) Å α = 106.848(9)° β = 100.968(5)° γ = 70.382(8)° V = 2710.6(3) $Å^3$
Space Group	$P\bar{1}$ (#2)
Z value	1
D_{calc}	1.630 g/cm^3
F_{000}	1314.00
μ(MoKα)	6.71 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID Imaging Plate

Radiation	MoKα (λ = 0.71069 Å) graphite monochromated
Temperature	23.0 °C
Voltage, Current	50 kV, 40 mA
Collimator Size	0.3 mm
Detector Aperture	270.0 mm x 256.0 mm
Data Images	44 exposures at 0.7 minutes per degree
Oscillation Range (ϕ=0.0°,χ=45.0°)	ω 130.0 - 190.0° with 5.0° step
Oscillation Range (ϕ=180.0°,χ=45.0°)	ω 0.0 - 160.0° with 5.0° step
Camera Radius	127.40 mm
Pixel Size	0.100 mm
$2\theta_{max}$	55.0°
No. of Reflections Measured	Total: 23545 Unique: 11969 (R_{int} = 0.052)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.5450 - 0.8745)

C. Structure Solution and Refinement

Structure Solution	Patterson Methods (DIRDIF94 PATTY)
Refinement	Full-matrix least-squares
Function Minimized	$\Sigma w(\|Fo\| - \|Fc\|)^2$
Least Squares Weights	$w = \frac{1}{\sigma^2(Fo)} = [\sigma_c^2(Fo) + \frac{p^2}{4}Fo^2]^{-1}$
p-factor	0.0100
Anomalous Dispersion	All non-hydrogen atoms
No. of Observations (I>3.00σ(I), 2θ<54.96°)	6151
No. Variables	745
Reflection/Parameter Ratio	8.26
Residuals: R; Rw	0.075 ; 0.063

Residuals: R1	0.075
No. of Reflections to calc R1	6151
Goodness of Fit Indicator	3.71
Max Shift/Error in Final Cycle	1.302
Maximum peak in Final Diff. Map	1.96 $e^-/\AA^3$
Minimum peak in Final Diff. Map	-0.71 $e^-/\AA^3$

Table 1. Atomic coordinates, B_{iso}/B_{eq} and occupancy

atom	x	y	z	B_{eq}	occ
Pd(1)	0.10389(5)	-0.02164(5)	-0.17159(6)	4.41(2)	1.0000
Pd(2)	0.0000	0.0000	0.0000	3.71(3)	0.5000
Cl(1)	0.9229(4)	0.4046(4)	0.4614(6)	20.9(3)	1.0000
F(1)	0.258(2)	0.705(1)	0.781(2)	15.3(7)	0.7500
F(2)	0.289(2)	0.767(1)	0.910(2)	16.6(7)	0.7500
F(3)	0.178(2)	0.782(1)	0.939(2)	16.5(7)	0.7500
F(4)	0.154(2)	0.725(1)	0.814(3)	20.4(8)	0.7500
F(5)	0.4467(6)	0.6142(6)	1.2023(5)	12.1(3)	1.0000
F(6)	0.3477(6)	0.5597(7)	1.2299(5)	12.5(3)	1.0000
F(7)	0.4763(6)	0.4767(6)	1.1673(5)	11.9(3)	1.0000
F(8)	-0.1144(8)	0.6096(6)	0.7591(8)	17.9(4)	1.0000
F(9)	-0.0857(6)	0.6341(5)	0.9210(8)	13.8(3)	1.0000
F(10)	-0.1918(6)	0.5814(5)	0.8540(9)	14.8(3)	1.0000
F(11)	-0.1192(5)	0.2590(5)	0.8068(7)	11.0(3)	1.0000
F(12)	0.0197(6)	0.2073(6)	0.8885(8)	12.5(3)	1.0000
F(13)	0.0033(6)	0.1772(5)	0.7201(8)	12.9(3)	1.0000
F(14)	0.3961(4)	0.2328(4)	1.1690(4)	7.9(2)	1.0000
F(15)	0.2743(5)	0.1894(4)	1.0866(4)	6.9(2)	1.0000
F(16)	0.4167(5)	0.0946(4)	1.0922(5)	8.6(2)	1.0000
F(17)	0.6538(4)	0.1554(4)	0.8224(5)	8.2(2)	1.0000
F(18)	0.5723(4)	0.1021(4)	0.6808(5)	7.7(2)	1.0000
F(19)	0.6250(4)	0.0325(4)	0.8056(5)	8.4(2)	1.0000
F(20)	0.131(1)	0.2296(8)	0.345(1)	13.9(5)	0.7500
F(21)	0.153(1)	0.169(1)	0.4430(9)	12.5(5)	0.7500

Table 1. Atomic coordinates, B_{iso}/B_{eq} and occupancy

atom	x	y	z	B_{eq}	occ
F(22)	0.262(2)	0.1203(9)	0.408(2)	15.9(6)	0.7500
F(23)	0.241(2)	0.172(1)	0.311(1)	14.3(6)	0.7500
F(24)	0.4071(8)	0.4004(7)	0.3870(6)	13.7(4)	1.0000
F(25)	0.4823(7)	0.4099(9)	0.5285(9)	16.3(5)	1.0000
F(26)	0.3639(9)	0.5073(6)	0.514(1)	18.6(5)	1.0000
C(1)	0.0620(7)	-0.0538(7)	-0.3607(8)	5.1(3)	1.0000
C(2)	0.0153(7)	-0.0869(6)	-0.3026(7)	4.1(2)	1.0000
C(3)	-0.0488(7)	-0.0319(7)	-0.2255(8)	5.1(3)	1.0000
C(4)	0.0812(7)	0.0608(7)	0.1486(8)	4.8(3)	1.0000
C(5)	0.1472(7)	0.0089(7)	0.0721(8)	5.1(3)	1.0000
C(6)	0.1716(7)	0.0382(7)	-0.0068(8)	4.7(3)	1.0000
C(7)	0.2425(6)	-0.0101(6)	-0.0804(8)	4.6(3)	1.0000
C(8)	0.2473(7)	0.0189(7)	-0.1698(9)	5.4(3)	1.0000
C(9)	0.1316(7)	-0.1055(8)	-0.4388(7)	4.8(3)	1.0000
C(10)	0.1624(8)	-0.0608(7)	-0.4955(9)	5.7(3)	1.0000
C(11)	0.2295(10)	-0.106(1)	-0.5714(10)	6.5(4)	1.0000
C(12)	0.2659(10)	-0.199(1)	-0.5934(9)	7.9(4)	1.0000
C(13)	0.2387(9)	-0.2468(9)	-0.539(1)	8.4(4)	1.0000
C(14)	0.1734(9)	-0.2022(8)	-0.4603(9)	6.5(3)	1.0000
C(15)	0.3163(7)	-0.0254(8)	-0.2558(8)	5.0(3)	1.0000
C(16)	0.3349(8)	0.0288(8)	-0.314(1)	6.9(4)	1.0000
C(17)	0.402(1)	-0.010(1)	-0.391(1)	8.9(5)	1.0000
C(18)	0.4502(9)	-0.104(1)	-0.409(1)	8.6(5)	1.0000
C(19)	0.4307(10)	-0.153(1)	-0.354(1)	8.5(4)	1.0000

Table 1. Atomic coordinates, B_{iso}/B_{eq} and occupancy

atom	x	y	z	B_{eq}	occ
C(20)	0.3660(8)	-0.1170(9)	-0.2771(9)	6.6(3)	1.0000
C(21)	0.2707(6)	0.3581(5)	0.6722(6)	3.0(2)	1.0000
C(22)	0.3238(6)	0.3973(6)	0.6327(6)	3.6(2)	1.0000
C(23)	0.3367(6)	0.3785(6)	0.5233(7)	3.9(2)	1.0000
C(24)	0.3002(7)	0.3122(7)	0.4478(6)	4.5(2)	1.0000
C(25)	0.2492(6)	0.2692(6)	0.4844(6)	3.5(2)	1.0000
C(26)	0.2348(6)	0.2928(5)	0.5929(6)	3.4(2)	1.0000
C(27)	0.392(1)	0.423(1)	0.484(1)	7.5(4)	1.0000
C(28)	0.209(1)	0.1980(9)	0.4077(9)	6.2(4)	1.0000
C(29)	0.2858(5)	0.4680(5)	0.8782(6)	3.2(2)	1.0000
C(30)	0.2532(7)	0.5501(6)	0.8468(6)	4.6(2)	1.0000
C(31)	0.2692(8)	0.6293(6)	0.9135(7)	5.3(3)	1.0000
C(32)	0.3196(8)	0.6299(6)	1.0146(7)	5.5(3)	1.0000
C(33)	0.3525(6)	0.5508(6)	1.0480(6)	4.0(2)	1.0000
C(34)	0.3344(5)	0.4721(5)	0.9809(6)	3.3(2)	1.0000
C(35)	0.4053(9)	0.5482(8)	1.1592(8)	6.2(3)	1.0000
C(36)	0.234(2)	0.714(1)	0.875(1)	9.6(6)	1.0000
C(37)	0.1397(6)	0.3879(5)	0.8113(6)	3.2(2)	1.0000
C(38)	0.0668(6)	0.4722(5)	0.8230(6)	3.4(2)	1.0000
C(39)	-0.0319(6)	0.4821(6)	0.8260(7)	4.4(2)	1.0000
C(40)	-0.0640(6)	0.4097(6)	0.8203(7)	4.5(2)	1.0000
C(41)	0.0059(7)	0.3243(6)	0.8099(7)	4.6(2)	1.0000
C(42)	0.1052(6)	0.3156(6)	0.8051(7)	4.1(2)	1.0000
C(43)	-0.1059(8)	0.5756(8)	0.835(1)	6.2(3)	1.0000

Table 1. Atomic coordinates, B_{iso}/B_{eq} and occupancy

atom	x	y	z	B_{eq}	occ
C(44)	-0.0246(9)	0.2463(9)	0.806(1)	7.6(4)	1.0000
C(45)	0.3342(5)	0.2867(5)	0.8441(6)	2.8(2)	1.0000
C(46)	0.4202(6)	0.2360(6)	0.7987(6)	3.3(2)	1.0000
C(47)	0.4905(6)	0.1646(5)	0.8414(7)	3.1(2)	1.0000
C(48)	0.4741(6)	0.1438(6)	0.9306(7)	3.5(2)	1.0000
C(49)	0.3888(6)	0.1936(5)	0.9787(6)	3.0(2)	1.0000
C(50)	0.3206(5)	0.2638(5)	0.9367(6)	3.1(2)	1.0000
C(51)	0.5832(7)	0.1152(7)	0.7890(8)	4.6(3)	1.0000
C(52)	0.3713(8)	0.1767(6)	1.0791(7)	4.6(3)	1.0000
C(53)	1.032(1)	0.484(2)	0.517(2)	18.2(9)	1.0000
B(1)	0.2575(6)	0.3754(6)	0.8010(7)	2.7(2)	1.0000
H(6)	0.1955	0.0750	-0.1764	4.0	1.0000
H(7)	0.2900	-0.0644	-0.0665	4.0	1.0000
H(8)	0.1326	0.0970	-0.0151	4.0	1.0000
H(9)	0.1789	-0.0553	0.0742	4.0	1.0000
H(10)	0.0529	0.1239	0.1490	4.0	1.0000
H(16)	0.0282	-0.1506	-0.3196	4.0	1.0000
H(17)	-0.0740	0.0309	-0.2226	4.0	1.0000
H(18)	0.0460	0.0132	-0.3418	4.0	1.0000
H(19)	0.2188	0.5505	0.7741	5.7	1.0000
H(20)	0.3299	0.6851	1.0614	7.3	1.0000
H(21)	0.3566	0.4174	1.0094	4.6	1.0000
H(22)	0.0870	0.5256	0.8268	5.1	1.0000
H(23)	-0.1352	0.4176	0.8233	6.4	1.0000

Table 1. Atomic coordinates, B_{iso}/B_{eq} and occupancy

atom	x	y	z	B_{eq}	occ
H(24)	0.1527	0.2560	0.7974	5.1	1.0000
H(25)	0.2613	0.2977	0.9714	4.2	1.0000
H(26)	0.5216	0.0987	0.9630	4.9	1.0000
H(27)	0.4359	0.2474	0.7365	4.3	1.0000
H(28)	0.1945	0.2628	0.6131	4.7	1.0000
H(29)	0.3081	0.2974	0.3710	5.6	1.0000
H(30)	0.3533	0.4406	0.6823	4.9	1.0000
H(31)	0.3009	0.0903	-0.3027	8.1	1.0000
H(32)	0.4196	0.0243	-0.4281	11.2	1.0000
H(33)	0.4976	-0.1432	-0.4591	10.3	1.0000
H(34)	0.4636	-0.2196	-0.3647	11.2	1.0000
H(35)	0.3535	-0.1553	-0.2407	8.1	1.0000
H(36)	0.1377	0.0032	-0.4856	6.5	1.0000
H(37)	0.2463	-0.0715	-0.6115	7.3	1.0000
H(38)	0.3107	-0.2276	-0.6481	10.3	1.0000
H(39)	0.2670	-0.3105	-0.5532	10.5	1.0000
H(40)	0.1538	-0.2361	-0.4260	7.6	1.0000

$$B_{eq} = \frac{8}{3}\pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}aa^*bb^*\cos\gamma + 2U_{13}aa^*cc^*\cos\beta + 2U_{23}bb^*cc^*\cos\alpha)$$

Table 2. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pd(1)	0.0486(5)	0.0614(6)	0.0574(5)	-0.0236(4)	-0.0058(4)	0.0128(4)
Pd(2)	0.0409(7)	0.0456(7)	0.0523(7)	-0.0173(5)	-0.0100(5)	0.0129(5)
Cl(1)	0.184(5)	0.158(5)	0.45(1)	-0.067(4)	0.013(6)	0.058(6)
F(1)	0.30(2)	0.13(1)	0.15(1)	-0.04(2)	0.02(2)	0.08(1)
F(2)	0.33(2)	0.10(1)	0.24(2)	-0.14(2)	-0.10(2)	0.10(1)
F(3)	0.29(2)	0.08(1)	0.22(2)	0.01(1)	0.04(2)	0.043(9)
F(4)	0.31(2)	0.08(1)	0.35(3)	-0.09(1)	-0.20(3)	0.12(2)
F(5)	0.210(8)	0.168(8)	0.102(5)	-0.131(7)	-0.071(5)	0.032(5)
F(6)	0.138(7)	0.30(1)	0.062(4)	-0.106(8)	0.001(4)	0.040(6)
F(7)	0.164(8)	0.120(7)	0.090(5)	0.007(6)	-0.058(5)	0.010(4)
F(8)	0.27(1)	0.159(9)	0.183(9)	0.147(8)	0.124(9)	0.126(8)
F(9)	0.138(7)	0.068(6)	0.211(9)	0.029(5)	-0.027(6)	-0.027(6)
F(10)	0.079(6)	0.090(6)	0.36(1)	0.026(5)	0.076(8)	0.030(7)
F(11)	0.064(5)	0.103(6)	0.264(9)	-0.039(4)	0.005(5)	0.056(6)
F(12)	0.137(7)	0.153(8)	0.243(10)	-0.087(6)	-0.053(7)	0.129(8)
F(13)	0.166(8)	0.082(6)	0.25(1)	-0.065(6)	0.057(8)	-0.009(6)
F(14)	0.135(6)	0.129(6)	0.049(3)	-0.068(5)	-0.010(3)	0.020(3)
F(15)	0.090(5)	0.121(5)	0.077(4)	-0.046(4)	0.008(3)	0.047(4)
F(16)	0.159(6)	0.073(5)	0.099(5)	0.006(4)	0.042(4)	0.055(4)
F(17)	0.054(4)	0.089(5)	0.148(6)	-0.023(3)	0.025(4)	-0.016(4)
F(18)	0.061(4)	0.132(6)	0.071(4)	0.009(4)	0.024(3)	0.016(4)
F(19)	0.082(4)	0.067(4)	0.161(6)	0.023(3)	0.057(4)	0.039(4)
F(20)	0.22(1)	0.116(10)	0.17(1)	-0.09(1)	-0.13(1)	0.040(9)
F(21)	0.22(1)	0.21(2)	0.098(8)	-0.18(1)	0.030(10)	-0.031(9)

Table 2. Anisotropic Displacement Parameters (continued)

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
F(22)	0.27(2)	0.057(9)	0.22(2)	-0.06(1)	-0.09(2)	0.011(9)
F(23)	0.29(2)	0.20(2)	0.084(8)	-0.17(2)	0.06(1)	-0.059(10)
F(24)	0.28(1)	0.23(1)	0.085(5)	-0.169(9)	0.053(7)	0.020(6)
F(25)	0.154(9)	0.33(2)	0.23(1)	-0.16(1)	-0.020(9)	0.15(1)
F(26)	0.30(1)	0.089(7)	0.39(2)	-0.055(8)	0.24(1)	0.041(8)
C(1)	0.056(7)	0.076(8)	0.059(7)	-0.026(6)	0.001(5)	0.010(6)
C(2)	0.054(6)	0.046(6)	0.046(6)	-0.017(5)	-0.010(5)	0.005(4)
C(3)	0.044(6)	0.087(8)	0.058(7)	-0.028(6)	-0.019(5)	0.015(6)
C(4)	0.045(6)	0.075(8)	0.055(7)	-0.026(6)	-0.011(5)	0.009(6)
C(5)	0.045(7)	0.083(8)	0.068(7)	-0.028(6)	-0.018(6)	0.026(6)
C(6)	0.039(6)	0.069(7)	0.071(7)	-0.026(5)	-0.017(5)	0.018(6)
C(7)	0.029(6)	0.063(7)	0.075(7)	-0.013(5)	-0.012(5)	0.018(6)
C(8)	0.045(6)	0.067(7)	0.085(8)	-0.021(5)	-0.016(6)	0.016(6)
C(9)	0.059(7)	0.072(8)	0.050(6)	-0.022(6)	-0.009(5)	0.017(6)
C(10)	0.068(8)	0.075(8)	0.083(8)	-0.035(6)	-0.005(6)	0.023(6)
C(11)	0.082(9)	0.11(1)	0.077(8)	-0.051(8)	-0.014(7)	0.037(7)
C(12)	0.10(1)	0.12(1)	0.073(9)	-0.029(10)	0.030(7)	0.011(8)
C(13)	0.10(1)	0.09(1)	0.11(1)	-0.019(8)	0.025(8)	-0.012(8)
C(14)	0.096(9)	0.078(9)	0.085(8)	-0.041(7)	0.007(7)	0.023(6)
C(15)	0.043(6)	0.080(8)	0.059(7)	-0.026(6)	-0.005(5)	0.004(6)
C(16)	0.082(9)	0.101(10)	0.100(9)	-0.062(8)	-0.003(7)	0.022(8)
C(17)	0.11(1)	0.19(2)	0.083(9)	-0.10(1)	0.020(8)	0.024(10)
C(18)	0.051(8)	0.17(2)	0.076(10)	-0.013(9)	0.001(7)	0.01(1)
C(19)	0.08(1)	0.15(1)	0.065(9)	-0.011(9)	0.002(7)	0.023(9)

Table 2. Anisotropic Displacement Parameters (continued)

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(20)	0.063(7)	0.096(10)	0.083(8)	-0.012(7)	-0.002(6)	0.029(7)
C(21)	0.041(5)	0.030(5)	0.038(5)	-0.005(4)	-0.005(4)	0.015(4)
C(22)	0.051(6)	0.045(6)	0.042(5)	-0.018(4)	-0.009(4)	0.015(4)
C(23)	0.056(6)	0.055(6)	0.052(6)	-0.030(5)	0.000(4)	0.021(5)
C(24)	0.060(6)	0.066(7)	0.042(5)	-0.011(5)	0.007(5)	0.017(5)
C(25)	0.052(5)	0.041(5)	0.037(5)	-0.014(4)	-0.001(4)	0.005(4)
C(26)	0.046(5)	0.038(5)	0.046(5)	-0.014(4)	-0.002(4)	0.014(4)
C(27)	0.13(1)	0.10(1)	0.079(9)	-0.06(1)	0.017(8)	0.010(8)
C(28)	0.11(1)	0.073(10)	0.054(7)	-0.046(9)	-0.002(8)	0.006(6)
C(29)	0.042(5)	0.033(5)	0.042(5)	-0.006(4)	0.002(4)	0.009(4)
C(30)	0.087(7)	0.038(6)	0.049(5)	-0.024(5)	-0.004(5)	0.009(4)
C(31)	0.106(8)	0.026(5)	0.067(6)	-0.024(5)	-0.007(6)	0.013(5)
C(32)	0.098(8)	0.050(7)	0.061(6)	-0.034(6)	0.002(6)	0.005(5)
C(33)	0.060(6)	0.040(6)	0.046(5)	-0.016(5)	0.009(4)	-0.001(4)
C(34)	0.043(5)	0.029(5)	0.051(5)	-0.007(4)	0.005(4)	0.009(4)
C(35)	0.093(9)	0.076(9)	0.059(7)	-0.038(7)	-0.008(7)	0.001(6)
C(36)	0.22(2)	0.059(10)	0.08(1)	-0.05(1)	-0.02(1)	0.015(8)
C(37)	0.049(5)	0.032(5)	0.034(5)	-0.005(4)	-0.002(4)	0.011(4)
C(38)	0.043(5)	0.032(5)	0.049(5)	0.002(4)	0.007(4)	0.015(4)
C(39)	0.049(6)	0.051(6)	0.059(6)	0.004(5)	0.017(5)	0.017(5)
C(40)	0.046(6)	0.053(6)	0.065(6)	-0.002(5)	0.008(5)	0.015(5)
C(41)	0.055(6)	0.041(6)	0.080(7)	-0.011(5)	0.009(5)	0.019(5)
C(42)	0.047(6)	0.041(6)	0.058(6)	0.000(4)	0.006(4)	0.014(4)
C(43)	0.056(7)	0.058(8)	0.098(9)	0.022(6)	0.032(7)	0.013(7)

Table 2. Anisotropic Displacement Parameters (continued)

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(44)	0.054(8)	0.083(10)	0.16(1)	-0.029(7)	-0.006(8)	0.043(9)
C(45)	0.038(5)	0.035(5)	0.033(4)	-0.010(4)	0.002(4)	0.007(3)
C(46)	0.041(5)	0.043(5)	0.043(5)	-0.016(4)	0.006(4)	0.008(4)
C(47)	0.035(5)	0.031(5)	0.049(6)	-0.011(4)	0.005(4)	0.003(4)
C(48)	0.043(6)	0.039(5)	0.044(5)	-0.004(4)	-0.003(4)	0.012(4)
C(49)	0.048(6)	0.035(5)	0.032(5)	-0.011(4)	0.000(4)	0.010(4)
C(50)	0.040(5)	0.025(4)	0.047(5)	-0.005(4)	0.005(4)	0.003(4)
C(51)	0.048(6)	0.050(7)	0.068(7)	-0.016(5)	0.000(5)	0.004(5)
C(52)	0.078(8)	0.043(6)	0.048(6)	-0.011(5)	0.004(6)	0.012(5)
C(53)	0.15(2)	0.24(3)	0.21(2)	0.07(2)	-0.02(2)	0.10(2)
B(1)	0.040(6)	0.022(5)	0.040(5)	-0.003(4)	0.006(4)	0.011(4)

The general temperature factor expression:

$$\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$$

Table 3. Bond Lengths($\AA$)

atom	atom	distance	atom	atom	distance
PD1	PD2	2.7959(7)	PD1	C1	2.356(9)
PD1	C2	2.163(8)	PD1	C3	2.241(9)
PD1	C6	2.257(9)	PD1	C7	2.168(8)
PD1	C8	2.38(1)	PD2	C4	2.207(8)
PD2	C4[1)]	2.207(8)	PD2	C5	2.198(9)
PD2	C5[1)]	2.198(9)	CL1	C53[2)]	2.04(4)
F1	F4	1.56(3)	F1	C36	1.28(2)
F2	F3	1.65(2)	F2	C36	1.29(2)
F3	F4	1.65(3)	F3	C36	1.30(2)
F4	C36	1.26(2)	F5	C35	1.32(1)
F6	C35	1.29(1)	F7	C35	1.28(1)
F8	C43	1.23(1)	F9	C43	1.29(1)
F10	C43	1.28(1)	F11	C44	1.32(1)
F12	C44	1.34(1)	F13	C44	1.34(1)
F14	C52	1.322(9)	F15	C52	1.37(1)
F16	C52	1.305(10)	F17	C51	1.331(10)
F18	C51	1.34(1)	F19	C51	1.32(1)
F20	F23	1.62(2)	F20	C28	1.31(2)
F21	F22	1.61(2)	F21	C28	1.28(1)
F22	F23	1.62(2)	F22	C28	1.23(2)
F23	C28	1.32(2)	F24	C27	1.25(1)
F25	C27	1.30(1)	F26	C27	1.24(1)
C1	C2	1.41(1)	C1	C9	1.44(1)
C2	C3	1.39(1)	C3	C4[1)]	1.42(1)

Table 3. Bond Lengths($\AA$) (continued)

atom	atom	distance	atom	atom	distance
C4	C5	1.39(1)	C5	C6	1.39(1)
C6	C7	1.41(1)	C7	C8	1.39(1)
C8	C15	1.50(1)	C9	C10	1.38(1)
C9	C14	1.43(1)	C10	C11	1.39(1)
C11	C12	1.36(2)	C12	C13	1.37(2)
C13	C14	1.39(1)	C15	C16	1.42(1)
C15	C20	1.38(1)	C16	C17	1.39(2)
C17	C18	1.42(2)	C18	C19	1.33(2)
C19	C20	1.36(1)	C21	C22	1.39(1)
C21	C26	1.402(10)	C21	B1	1.65(1)
C22	C23	1.40(1)	C23	C24	1.40(1)
C23	C27	1.47(1)	C24	C25	1.39(1)
C25	C26	1.383(10)	C25	C28	1.48(1)
C29	C30	1.39(1)	C29	C34	1.378(10)
C29	B1	1.66(1)	C30	C31	1.38(1)
C31	C32	1.37(1)	C31	C36	1.49(2)
C32	C33	1.37(1)	C33	C34	1.39(1)
C33	C35	1.50(1)	C37	C38	1.398(10)
C37	C42	1.39(1)	C37	B1	1.68(1)
C38	C39	1.40(1)	C39	C40	1.37(1)
C39	C43	1.51(1)	C40	C41	1.39(1)
C41	C42	1.41(1)	C41	C44	1.45(1)
C45	C46	1.374(9)	C45	C50	1.42(1)
C45	B1	1.66(1)	C46	C47	1.42(1)

Table 3. Bond Lengths($\AA$) (continued)

atom	atom	distance	atom	atom	distance
C47	C48	1.37(1)	C47	C51	1.49(1)
C48	C49	1.38(1)	C49	C50	1.40(1)
C49	C52	1.49(1)	C53	C53[2)]	1.00(3)

Symmetry operations

(1) -X,-Y,-Z (2) -X+2,-Y+1,-Z+1

Table 4. Bond Lengths($\AA$)

atom	atom	distance	atom	atom	distance
C1	H18	0.99	C2	H16	0.94
C3	H17	0.94	C4	H10	0.96
C5	H9	0.98	C6	H8	0.95
C7	H7	0.95	C8	H6	0.98
C10	H36	0.95	C11	H37	0.98
C12	H38	0.96	C13	H39	0.94
C14	H40	0.93	C16	H31	0.93
C17	H32	0.94	C18	H33	0.97
C19	H34	0.99	C20	H35	0.95
C22	H30	0.96	C24	H29	0.97
C26	H28	0.98	C30	H19	0.98
C32	H20	0.96	C34	H21	0.98
C38	H22	0.98	C40	H23	1.00
C42	H24	0.96	C46	H27	0.96
C48	H26	0.95	C50	H25	0.96

Table 5. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
PD2	PD1	C1	131.7(2)	PD2	PD1	C2	98.7(3)
PD2	PD1	C3	66.7(3)	PD2	PD1	C6	65.8(3)
PD2	PD1	C7	99.1(3)	PD2	PD1	C8	129.8(3)
C1	PD1	C2	35.9(3)	C1	PD1	C3	65.0(4)
C1	PD1	C6	159.6(4)	C1	PD1	C7	129.1(4)
C1	PD1	C8	97.4(4)	C2	PD1	C3	36.8(3)
C2	PD1	C6	164.1(4)	C2	PD1	C7	153.3(4)
C2	PD1	C8	131.6(4)	C3	PD1	C6	130.3(4)
C3	PD1	C7	165.7(4)	C3	PD1	C8	157.6(4)
C6	PD1	C7	37.0(3)	C6	PD1	C8	64.0(4)
C7	PD1	C8	35.2(3)	PD1	PD2	PD1[1)]	180.0
PD1	PD2	C4	106.1(3)	PD1	PD2	C4[1)]	73.9(3)
PD1	PD2	C5	73.5(3)	PD1	PD2	C5[1)]	106.5(3)
PD1[1)]	PD2	C4	73.9(3)	PD1[1)]	PD2	C4[1)]	106.1(3)
PD1[1)]	PD2	C5	106.5(3)	PD1[1)]	PD2	C5[1)]	73.5(3)
C4	PD2	C4[1)]	180.0	C4	PD2	C5	36.9(3)
C4	PD2	C5[1)]	143.1(3)	C4[1)]	PD2	C5	143.1(3)
C4[1)]	PD2	C5[1)]	36.9(3)	C5	PD2	C5[1)]	180.0
F4	F1	C36	51(1)	F3	F2	C36	50(1)
F2	F3	F4	88(1)	F2	F3	C36	50(1)
F4	F3	C36	48(1)	F1	F4	F3	94(1)
F1	F4	C36	52(1)	F3	F4	C36	50(1)
F23	F20	C28	52.1(9)	F22	F21	C28	48.6(8)
F21	F22	F23	92(1)	F21	F22	C28	51(1)

Table 5. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
F23	F22	C28	52(1)	F20	F23	F22	91(1)
F20	F23	C28	51.8(9)	F22	F23	C28	48.0(9)
PD1	C1	C2	64.6(5)	PD1	C1	C9	124.0(6)
C2	C1	C9	128.0(10)	PD1	C2	C1	79.5(6)
PD1	C2	C3	74.7(5)	C1	C2	C3	124.3(9)
PD1	C3	C2	68.6(5)	PD1	C3	C4[1)]	110.3(6)
C2	C3	C4[1)]	126.4(10)	PD2	C4	C3[1)]	99.2(6)
PD2	C4	C5	71.2(5)	C3[1)]	C4	C5	128(1)
PD2	C5	C4	71.9(5)	PD2	C5	C6	99.3(6)
C4	C5	C6	126(1)	PD1	C6	C5	110.1(7)
PD1	C6	C7	68.1(5)	C5	C6	C7	128.3(10)
PD1	C7	C6	74.9(5)	PD1	C7	C8	80.6(6)
C6	C7	C8	123.0(9)	PD1	C8	C7	64.2(6)
PD1	C8	C15	123.7(6)	C7	C8	C15	129(1)
C1	C9	C10	119(1)	C1	C9	C14	123(1)
C10	C9	C14	116.9(10)	C9	C10	C11	122(1)
C10	C11	C12	119(1)	C11	C12	C13	120(1)
C12	C13	C14	120(1)	C9	C14	C13	119(1)
C8	C15	C16	119(1)	C8	C15	C20	121(1)
C16	C15	C20	118(1)	C15	C16	C17	120(1)
C16	C17	C18	117(1)	C17	C18	C19	120(1)
C18	C19	C20	123(1)	C15	C20	C19	119(1)
C22	C21	C26	113.9(7)	C22	C21	B1	125.4(7)
C26	C21	B1	120.5(7)	C21	C22	C23	124.0(7)

Table 5. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C22	C23	C24	119.6(8)	C22	C23	C27	122.3(8)
C24	C23	C27	118.0(9)	C23	C24	C25	118.2(8)
C24	C25	C26	120.1(7)	C24	C25	C28	120.3(9)
C26	C25	C28	119.6(9)	C21	C26	C25	124.1(8)
F24	C27	F25	99(1)	F24	C27	F26	109(1)
F24	C27	C23	120(1)	F25	C27	F26	98(1)
F25	C27	C23	113(1)	F26	C27	C23	113(1)
F20	C28	F21	84(1)	F20	C28	F22	132(1)
F20	C28	F23	76(1)	F20	C28	C25	114(1)
F21	C28	F22	80(1)	F21	C28	F23	128(1)
F21	C28	C25	117(1)	F22	C28	F23	79(1)
F22	C28	C25	113(1)	F23	C28	C25	113(1)
C30	C29	C34	115.0(7)	C30	C29	B1	121.7(7)
C34	C29	B1	123.0(7)	C29	C30	C31	122.5(8)
C30	C31	C32	120.6(9)	C30	C31	C36	119.8(10)
C32	C31	C36	119.6(9)	C31	C32	C33	118.6(8)
C32	C33	C34	120.2(8)	C32	C33	C35	120.8(8)
C34	C33	C35	118.9(9)	C29	C34	C33	123.1(8)
F5	C35	F6	103.0(9)	F5	C35	F7	102(1)
F5	C35	C33	113(1)	F6	C35	F7	106(1)
F6	C35	C33	113.1(10)	F7	C35	C33	116.7(9)
F1	C36	F2	85(2)	F1	C36	F3	132(1)
F1	C36	F4	75(1)	F1	C36	C31	114(1)
F2	C36	F3	79(1)	F2	C36	F4	130(1)

Table 5. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
F2	C36	C31	114(1)	F3	C36	F4	80(2)
F3	C36	C31	112(1)	F4	C36	C31	115(1)
C38	C37	C42	114.0(8)	C38	C37	B1	122.6(7)
C42	C37	B1	123.3(7)	C37	C38	C39	122.7(8)
C38	C39	C40	122.1(8)	C38	C39	C43	119.0(10)
C40	C39	C43	118.9(9)	C39	C40	C41	117.3(8)
C40	C41	C42	119.6(9)	C40	C41	C44	119.1(9)
C42	C41	C44	121.2(9)	C37	C42	C41	124.1(8)
F8	C43	F9	105(1)	F8	C43	F10	108(1)
F8	C43	C39	114.9(10)	F9	C43	F10	98.4(10)
F9	C43	C39	112(1)	F10	C43	C39	114(1)
F11	C44	F12	104(1)	F11	C44	F13	105(1)
F11	C44	C41	117(1)	F12	C44	F13	101(1)
F12	C44	C41	112(1)	F13	C44	C41	112(1)
C46	C45	C50	115.6(7)	C46	C45	B1	121.9(7)
C50	C45	B1	122.2(6)	C45	C46	C47	122.1(8)
C46	C47	C48	120.6(7)	C46	C47	C51	118.4(8)
C48	C47	C51	121.0(8)	C47	C48	C49	118.9(8)
C48	C49	C50	120.2(7)	C48	C49	C52	120.3(8)
C50	C49	C52	119.4(8)	C45	C50	C49	122.6(7)
F17	C51	F18	104.1(9)	F17	C51	F19	104.3(8)
F17	C51	C47	115.1(7)	F18	C51	F19	104.6(8)
F18	C51	C47	114.5(8)	F19	C51	C47	113.1(9)
F14	C52	F15	102.7(8)	F14	C52	F16	105.9(8)

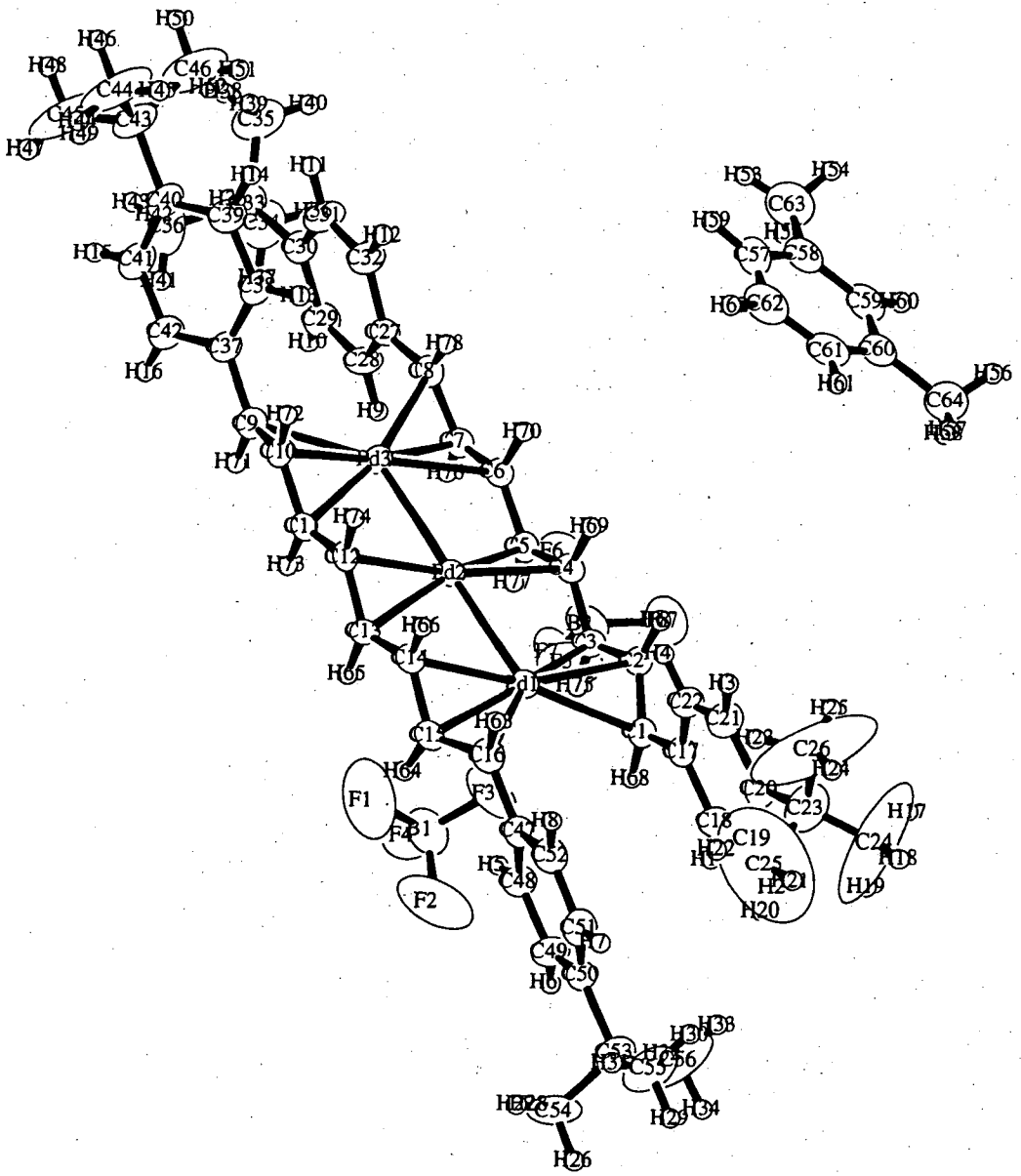
Table 5. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
F14	C52	C49	113.6(9)	F15	C52	F16	104.6(9)
F15	C52	C49	113.8(8)	F16	C52	C49	115.0(8)
CL1[2)]	C53	C53[2)]	88(4)	C21	B1	C29	112.6(7)
C21	B1	C37	108.4(6)	C21	B1	C45	107.6(6)
C29	B1	C37	108.7(6)	C29	B1	C45	107.5(6)
C37	B1	C45	112.1(6)				

Symmetry operations

(1)	-X,-Y,-Z	(2)	-X+2,-Y+1,-Z+1

X-ray data for 2'-rac



Experimental

Data Collection

A red prism crystal of $C_{64}H_{78}Pd_3B_2F_8$ having approximate dimensions of 0.48 x 0.13 x 0.10 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS-RAPID Imaging Plate diffractometer with graphite monochromated Mo-Kα radiation.

Indexing was performed from 2 oscillations which were exposed for 4.2 minutes. The camera radius was 127.40 mm. Readout was performed in the 0.200 mm pixel mode.

Cell constants and an orientation matrix for data collection corresponded to a primitive triclinic cell with dimensions:

a = 15.3395(7) $\AA$	α = 95.812(2)°
b = 21.4748(9) $\AA$	β = 96.587(1)°
c = 9.2123(3) $\AA$	γ = 95.1087(8)°
V = 2983.5(2) $\AA^3$	

For Z = 2 and F.W. = 1340.13, the calculated density is 1.49 g/cm^3. Based on a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

$P\bar{1}$ (#2)

The data were collected at a temperature of -50 $\pm$ 1°C to a maximum 2θ value of 55.0°. A total of 44 images, corresponding to 220.0 ° oscillation angles, were collected with 2 different goniometer settings. Exposure time was 1.70 minutes per degree. The camera radius was 127.40 mm. Readout was performed in the 0.200 mm pixel mode. Data were processed by the PROCESS-AUTO program package.

Data Reduction

Of the 28003 reflections which were collected, 13482 were unique (R_{int} = 0.026); equivalent reflections were merged.

The linear absorption coefficient, μ, for Mo-Kα radiation is 9.6 cm^{-1}. A symmetry-related absorption correction using the program ABSCOR[1] was applied which resulted in transmission factors ranging from 0.72 to 0.91. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by heavy-atom Patterson methods[2] and expanded using Fourier techniques[3]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included but not refined. The final cycle of full-matrix least-squares refinement[4] was based on 7971 observed reflections (I > 3.00σ(I), 2θ

< 54.96) and 694 variable parameters and converged (largest parameter shift was 6.23 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma||Fo| - |Fc||/\Sigma|Fo| = 0.031$$

$$R_w = \sqrt{\Sigma w(|Fo| - |Fc|)^2/\Sigma wFo^2} = 0.030$$

$$R1 = \Sigma||Fo| - |Fc||/\Sigma|Fo| = 0.030 \quad for \quad I > 3.0\sigma(I) \quad data$$

The standard deviation of an observation of unit weight[5] was 1.73. The weighting scheme was based on counting statistics and included a factor (p = 0.015) to downweight the intense reflections. Plots of $\Sigma w(|Fo| - |Fc|)^2$ versus $|Fo|$, reflection order in data collection, sin θ/λ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.45 and -0.39 $e^-/\AA^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[6] . Anomalous dispersion effects were included in Fcalc[7]; the values for Δf' and Δf" were those of Creagh and McAuley[8]. The values for the mass attenuation coefficients are those of Creagh and Hubbel[9]. All calculations were performed using the teXsan[10] crystallographic software package of Molecular Structure Corporation.

References

(1) ABSCOR: Higashi T. (1995). Program for Absorption Correction, Rigaku Corporation, Tokyo, Japan.

(2) PATTY: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M. (1994). The DIRDIF-94 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) DIRDIF94: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M. (1994). The DIRDIF-94 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(4) Least-Squares:

Function minimized: $\Sigma w(|Fo| - |Fc|)^2$

where w = $\frac{1}{\sigma^2(Fo)} = [\sigma_c^2(Fo) + \frac{p^2}{4}Fo^2]^{-1}$

$\sigma_c(Fo)$ = e.s.d. based on counting statistics

p = p-factor

(5) Standard deviation of an observation of unit weight:

$$\sqrt{\Sigma w(|Fo| - |Fc|)^2/(No - Nv)}$$

where: No = number of observations

Nv = number of variables

(6) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(7) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(8) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(9) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(10) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1999).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{64}H_{78}Pd_3B_2F_8$
Formula Weight	1340.13
Crystal Color, Habit	red, prism
Crystal Dimensions	0.48 X 0.13 X 0.10 mm
Crystal System	triclinic
Lattice Type	Primitive
No. of Reflections Used for Unit Cell Determination (2θ range)	11044 (4.4 - 54.9°)
Indexing Images	2 oscillations at 4.2 minutes
Camera Radius	127.40 mm
Lattice Parameters	a = 15.3395(7) Å b = 21.4748(9) Å c = 9.2123(3) Å α = 95.812(2)° β = 96.587(1)° γ = 95.1087(8)° V = 2983.5(2) Å^3
Space Group	$P\bar{1}$ (#2)
Z value	2
D_{calc}	1.492 g/cm^3
F_{000}	1364.00
μ(MoKα)	9.59 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID Imaging Plate

Radiation	MoKα (λ = 0.71069 Å) graphite monochromated
Temperature	-50.0 °C
Voltage, Current	50 kV, 40 mA
Collimator Size	0.8 mm
Detector Aperture	270.0 mm x 256.0 mm
Data Images	44 exposures at 1.7 minutes per degree
Oscillation Range (ϕ=0.0°,χ=45.0°)	ω 130.0 - 190.0° with 5.0° step
Oscillation Range (ϕ=180.0°,χ=45.0°)	ω 0.0 - 160.0° with 5.0° step
Camera Radius	127.40 mm
Pixel Size	0.200 mm
$2\theta_{max}$	55.0°
No. of Reflections Measured	Total: 28003 Unique: 13482 (R_{int} = 0.026)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.7178 - 0.9086)

C. Structure Solution and Refinement

Structure Solution	Patterson Methods (DIRDIF94 PATTY)
Refinement	Full-matrix least-squares
Function Minimized	$\Sigma w(\|Fo\| - \|Fc\|)^2$
Least Squares Weights	$w = \frac{1}{\sigma^2(Fo)} = [\sigma_c^2(Fo) + \frac{p^2}{4}Fo^2]^{-1}$
p-factor	0.0150
Anomalous Dispersion	All non-hydrogen atoms
No. of Observations (I>3.00σ(I), 2θ<54.96°)	7971
No. Variables	694
Reflection/Parameter Ratio	11.49
Residuals: R; Rw	0.031 ; 0.030

Residuals: R1	0.030
No. of Reflections to calc R1	7971
Goodness of Fit Indicator	1.73
Max Shift/Error in Final Cycle	6.234
Maximum peak in Final Diff. Map	0.45 $e^-/\AA^3$
Minimum peak in Final Diff. Map	-0.39 $e^-/\AA^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
Pd(1)	0.18211(2)	0.12055(1)	0.18888(4)	2.210(7)
Pd(2)	0.23444(2)	0.01062(2)	0.29066(4)	1.994(6)
Pd(3)	0.28729(2)	-0.10146(1)	0.38664(4)	2.218(7)
F(1)	0.0183(3)	0.0922(2)	0.4819(4)	11.0(1)
F(2)	0.0058(3)	0.1916(2)	0.5466(4)	10.7(1)
F(3)	0.1360(2)	0.1621(2)	0.5027(3)	6.32(9)
F(4)	0.0742(2)	0.1361(1)	0.7031(3)	5.52(8)
F(5)	0.3708(2)	0.1319(2)	0.7489(4)	7.6(1)
F(6)	0.4661(2)	0.0604(1)	0.7847(3)	5.06(8)
F(7)	0.4220(2)	0.1153(2)	0.9809(3)	6.22(9)
F(8)	0.5127(2)	0.1634(1)	0.8406(3)	5.39(8)
C(1)	0.2492(2)	0.2195(2)	0.2126(5)	2.40(9)
C(2)	0.3094(3)	0.1749(2)	0.2252(5)	2.32(9)
C(3)	0.3102(2)	0.1347(2)	0.3398(4)	2.31(9)
C(4)	0.3529(2)	0.0787(2)	0.3328(4)	2.23(9)
C(5)	0.3566(2)	0.0385(2)	0.4423(4)	2.24(9)
C(6)	0.3922(3)	-0.0201(2)	0.4211(5)	2.23(9)
C(7)	0.4067(2)	-0.0624(2)	0.5276(5)	2.28(9)
C(8)	0.4281(2)	-0.1220(2)	0.4766(5)	2.42(9)
C(9)	0.2050(3)	-0.1983(2)	0.3795(5)	2.77(10)
C(10)	0.1792(2)	-0.1636(2)	0.2653(5)	2.31(9)
C(11)	0.1441(2)	-0.1050(2)	0.2948(5)	2.38(9)
C(12)	0.1308(2)	-0.0628(2)	0.1883(4)	2.12(9)
C(13)	0.0911(2)	-0.0074(2)	0.2155(4)	2.16(9)

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
C(14)	0.0845(2)	0.0373(2)	0.1126(5)	2.31(9)
C(15)	0.0426(2)	0.0940(2)	0.1350(4)	2.28(9)
C(16)	0.0571(3)	0.1416(2)	0.0478(5)	2.58(9)
C(17)	0.2384(2)	0.2551(2)	0.0847(5)	2.37(9)
C(18)	0.2127(3)	0.3155(2)	0.1003(4)	2.71(9)
C(19)	0.2056(3)	0.3501(2)	-0.0174(5)	3.2(1)
C(20)	0.2212(3)	0.3254(2)	-0.1570(5)	3.2(1)
C(21)	0.2430(3)	0.2642(2)	-0.1724(5)	3.4(1)
C(22)	0.2516(3)	0.2305(2)	-0.0550(5)	3.05(10)
C(23)	0.2142(4)	0.3652(2)	-0.2873(6)	5.1(1)
C(24)	0.255(1)	0.4270(4)	-0.2502(9)	20.2(5)
C(25)	0.1215(9)	0.3740(8)	-0.328(1)	23.2(7)
C(26)	0.239(1)	0.3369(4)	-0.4172(10)	24.4(6)
C(27)	0.4438(2)	-0.1730(2)	0.5695(5)	2.28(9)
C(28)	0.4352(3)	-0.1699(2)	0.7177(5)	3.1(1)
C(29)	0.4516(3)	-0.2197(2)	0.7968(5)	3.05(10)
C(30)	0.4778(3)	-0.2749(2)	0.7319(5)	2.60(9)
C(31)	0.4895(3)	-0.2770(2)	0.5848(5)	3.4(1)
C(32)	0.4736(3)	-0.2271(2)	0.5036(5)	3.01(10)
C(33)	0.4885(3)	-0.3314(2)	0.8166(5)	3.3(1)
C(34)	0.5227(3)	-0.3116(2)	0.9784(5)	4.8(1)
C(35)	0.5514(4)	-0.3744(2)	0.7539(6)	5.5(2)
C(36)	0.3963(4)	-0.3673(3)	0.8088(6)	5.8(2)
C(37)	0.2416(3)	-0.2586(2)	0.3586(5)	2.65(9)

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
C(38)	0.2925(3)	-0.2740(2)	0.2485(5)	3.3(1)
C(39)	0.3210(3)	-0.3332(2)	0.2273(5)	3.6(1)
C(40)	0.2990(3)	-0.3797(2)	0.3152(5)	3.1(1)
C(41)	0.2512(3)	-0.3632(2)	0.4274(5)	3.8(1)
C(42)	0.2230(3)	-0.3038(2)	0.4495(5)	3.4(1)
C(43)	0.3319(4)	-0.4450(2)	0.2895(5)	4.0(1)
C(44)	0.3046(5)	-0.4730(3)	0.1358(7)	8.1(2)
C(45)	0.3022(7)	-0.4889(3)	0.3918(10)	13.3(3)
C(46)	0.4318(5)	-0.4384(3)	0.3119(9)	9.9(2)
C(47)	0.0260(2)	0.2041(2)	0.0733(4)	2.34(9)
C(48)	0.0184(3)	0.2324(2)	0.2131(5)	2.78(10)
C(49)	-0.0078(3)	0.2920(2)	0.2337(5)	3.3(1)
C(50)	-0.0291(3)	0.3254(2)	0.1142(5)	3.1(1)
C(51)	-0.0226(3)	0.2970(2)	-0.0240(5)	3.3(1)
C(52)	0.0061(3)	0.2379(2)	-0.0460(4)	2.86(10)
C(53)	-0.0584(3)	0.3913(2)	0.1424(6)	4.5(1)
C(54)	-0.1437(5)	0.3872(3)	0.205(1)	12.6(3)
C(55)	-0.0740(4)	0.4218(3)	0.0014(8)	7.7(2)
C(56)	0.0140(5)	0.4331(3)	0.2374(9)	10.2(2)
C(57)	0.7208(3)	0.0082(2)	0.0995(6)	4.4(1)
C(58)	0.7898(3)	0.0364(2)	0.2035(5)	3.9(1)
C(59)	0.8078(3)	0.1015(2)	0.2146(5)	3.8(1)
C(60)	0.7581(3)	0.1380(2)	0.1261(5)	3.7(1)
C(61)	0.6898(3)	0.1081(2)	0.0244(5)	4.1(1)

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
C(62)	0.6711(3)	0.0437(3)	0.0125(5)	4.5(1)
C(63)	0.8419(4)	-0.0017(3)	0.3019(6)	5.8(2)
C(64)	0.7781(3)	0.2078(2)	0.1437(6)	5.2(1)
B(1)	0.0609(4)	0.1453(3)	0.5605(6)	4.3(1)
B(2)	0.4418(4)	0.1175(3)	0.8394(6)	4.1(1)
H(1)	0.2008	0.3343	0.1959	3.3
H(2)	0.1889	0.3926	-0.0049	3.7
H(3)	0.2528	0.2456	-0.2692	4.1
H(4)	0.2679	0.1879	-0.0687	3.6
H(5)	0.0329	0.2097	0.2966	3.3
H(6)	-0.0116	0.3102	0.3332	4.0
H(7)	-0.0375	0.3187	-0.1085	3.9
H(8)	0.0127	0.2191	-0.1430	3.4
H(9)	0.4176	-0.1323	0.7673	3.7
H(10)	0.4465	-0.2156	0.9013	3.6
H(11)	0.5084	-0.3144	0.5363	4.1
H(12)	0.4828	-0.2301	0.4016	3.7
H(13)	0.3083	-0.2431	0.1844	4.0
H(14)	0.3566	-0.3429	0.1498	4.2
H(15)	0.2358	-0.3944	0.4920	4.4
H(16)	0.1900	-0.2937	0.5307	4.1
H(17)	0.3150	0.4276	-0.2247	21.5
H(18)	0.2426	0.4512	-0.3315	21.5
H(19)	0.2286	0.4469	-0.1686	21.5

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
H(20)	0.1006	0.3974	-0.2384	23.5
H(21)	0.1134	0.3999	-0.4024	23.5
H(22)	0.0857	0.3361	-0.3468	23.5
H(23)	0.1891	0.2958	-0.4469	19.9
H(24)	0.2176	0.3596	-0.5012	19.9
H(25)	0.2874	0.3231	-0.4202	19.9
H(26)	-0.1642	0.4280	0.2213	12.2
H(27)	-0.1885	0.3614	0.1363	12.2
H(28)	-0.1391	0.3690	0.2947	12.2
H(29)	-0.0937	0.4623	0.0167	8.4
H(30)	-0.0211	0.4257	-0.0462	8.4
H(31)	-0.1183	0.3959	-0.0691	8.4
H(32)	0.0278	0.4175	0.3302	10.8
H(33)	0.0672	0.4352	0.1904	10.8
H(34)	-0.0016	0.4750	0.2555	10.8
H(35)	0.5262	-0.3472	1.0325	5.6
H(36)	0.5802	-0.2889	0.9891	5.6
H(37)	0.4841	-0.2844	1.0234	5.6
H(38)	0.5557	-0.4108	0.8050	6.3
H(39)	0.5329	-0.3880	0.6520	6.3
H(40)	0.6101	-0.3527	0.7615	6.3
H(41)	0.3563	-0.3399	0.8516	6.6
H(42)	0.3720	-0.3804	0.7095	6.6
H(43)	0.3976	-0.4029	0.8621	6.6

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
H(44)	0.2413	-0.4780	0.1144	8.8
H(45)	0.3270	-0.4461	0.0678	8.8
H(46)	0.3254	-0.5132	0.1166	8.8
H(47)	0.2370	-0.4939	0.3814	14.4
H(48)	0.3209	-0.5289	0.3754	14.4
H(49)	0.3205	-0.4718	0.4928	14.4
H(50)	0.4547	-0.4783	0.2959	10.2
H(51)	0.4554	-0.4114	0.2460	10.2
H(52)	0.4540	-0.4203	0.4108	10.2
H(53)	0.8214	-0.0456	0.2806	6.6
H(54)	0.9033	0.0031	0.2889	6.6
H(55)	0.8371	0.0107	0.4032	6.6
H(56)	0.8380	0.2197	0.1277	6.0
H(57)	0.7399	0.2267	0.0757	6.0
H(58)	0.7711	0.2254	0.2414	6.0
H(59)	0.7082	-0.0373	0.0866	5.5
H(60)	0.8555	0.1218	0.2865	4.5
H(61)	0.6547	0.1333	-0.0383	5.1
H(62)	0.6225	0.0235	-0.0574	5.3
H(63)	0.0658	0.1268	-0.0626	3.9
H(64)	0.0129	0.0983	0.2274	3.9
H(65)	0.0611	0.0073	0.3035	3.9
H(66)	0.0982	0.0257	0.0117	3.9
H(67)	0.3409	0.1643	0.1311	3.9