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Palladium catalyzed Copolymerisation of Ethene with Acrolein Dimethyl Acetal: Catalyst Action and Deactivation

By W. Li, X. Zhang, A. Meetsma and B. Hessen

Part I: Experimental Section

Abbreviations: Ar = 2,6-diisopropylphenyl; DAD = ArN=CMe-CMe=NAr; Ar^f = 3,5-$(CF_3)_2C_6H_3$

General. All preparations were performed under an inert nitrogen atmosphere, using standard Schlenk or glovebox techniques, unless mentioned otherwise. Toluene, pentane, and hexane (Aldrich, anhydrous, 99.8%) were passed over columns of Al_2O_3 (Fluka), BASF R3-11-supported Cu oxygen scavenger, and molecular sieves (Aldrich, 4Å). Diethyl ether and THF (Aldrich, anhydrous, 99.8%) were dried over Al_2O_3 (Fluka). Dichloromethane and deuterated dichloromethane were distilled from CaH_2. All solvents were degassed and stored under nitrogen.

Reagents: (DAD)PdMeCl,[1] $NaBAr^f{}_4$[2] and [(DAD)PdMe(OEt_2)][$BAr^f{}_4$][1] were prepared following published procedures. Acrolein dimethyl acetal (ADMA, Aldrich 98%) and allyl ethyl ether (AEE, Acros 95%) were purified by distillation from CaH_2. NMR spectra were recorded on Varian Gemini VXR 300 or Varian Inova 500 spectrometers. The ^{1}H NMR spectra were referenced to resonances of residual protons in deuterated solvents. The ^{13}C NMR spectra were referenced to carbon resonances of deuterated solvents and reported in ppm relative to TMS (δ 0 ppm). Coupling constants are listed in Hz. Assignments of the resonances of the organometallic products were made using information from COSY and HSQC and/or HMBC spectra. GPC analysis of the polymers was performed by J. Vorenkamp at ambient

temperature on a Spectra Physics AS 1000 LC-system using a Viscotek H-502 viscometer and a Shodex RI-71 refractive index detector, using THF as eluent and universal calibration with polystyrene standards.

References: (1) Johnson, L.K.; Brookhart, M. *J. Am. Chem. Soc.* **1995**, *117*, 6414. (2) Nishida, H.; Takada, N.; Yoshimura, M. *Bull. Chem. Soc. Jpn* **1984**, *57*, 2600.

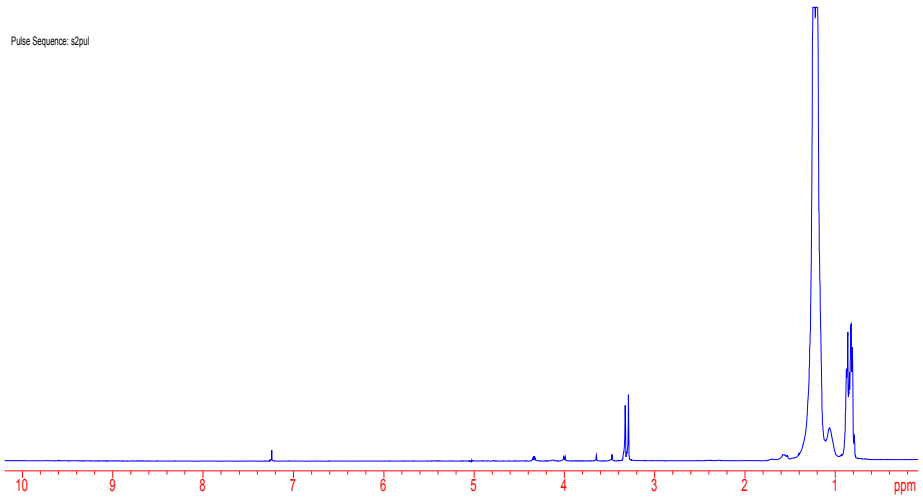
Synthesis of {(DAD)Pd[$CH_2CHMeCH(OMe)_2$]}[BAr^f_4] (2). Acrolein dimethyl acetal (0.5 mL, 4.2 mmol) was added to a solution of (DAD)PdMeCl (1.130 g, 2.01 mmol) and $NaBAr^f_4$ (1.752 g, 2.00 mmol) in 40 mL of Et_2O. The mixture was stirred at ambient temperature for 24 h. Filtration followed by evaporation of the solvent in vacuo yielded an orange solid. After rinsing with pentane (3 x 30 mL) and drying in vacuo, 2.72 g (3.8 mmol, 91%) of the title compound was obtained. ^{1}H NMR (CD_2Cl_2, 500MHz, 25 ^{o}C): δ 7.73 (s, 8H, Ar^f H_o), 7.57 (s, 4H, Ar^f H_p), 7.42 – 7.30 (m, 6H, Ar), 4.12 (d, J = 5.5, C*H*$(OMe)_2$), 3.12 (s, 3H, OMe), 3.02 – 2.90 (m, 4H, *i*Pr CH), 2.87 (m, 2H, PdC*H*$_2$), 2.84 (s, 3H, OMe), 2.20 and 2.16 (s, 3H each, DAD Me), 1.44, 1.42, 1.39, 1.39, 1.27, 1.25, 1.22 and 1.18 (d, 3H each, J = 6.8, *i*Pr Me), 1.3 (m, $PdCH_2C$*H*Me), 0.74 (d, 3H, J = 7.0, $PdCH_2CH$*Me*). ^{13}C NMR (CD_2Cl_2, 126MHz, 25 ^{o}C): δ178.03 and 172.18 (DAD N=C), 162.13 (q, J_{CB} = 50.6, Ar^f C_{ipso}), 141.74 and 141.71 (Ar C_{ipso}), 138.39, 138.30, 137.76 and 137.38 (Ar C_o), 135.20 (Ar^f C_o), 129.37 and 129.01 (Ar C_p), 129.31 (qq, J_{CF} = 31.5, J_{CB} = 2.9, Ar^f C_m), 125.24, 125.17, 125.02 and 124.93 (Ar C_m), 124.99 (q, J_{CF} = 273.1, Ar^f CF_3), 119.48 (d, J_{CH} = 172, *C*H$(OMe)_2$), 117.83 (septet, J_{CF} = 3.9, Ar^f C_p), 60.51 (q, J_{CH} = 147, OMe), 56.55 (q, J_{CH} = 145, OMe), 38.80 (t, J_{CH} = 140, Pd*C*H_2), 38.75 (d, J_{CH} = 112, $PdCH_2$*C*HMe), 29.61, 29.43, 29.18 and 29.13 (*i*Pr CH), 24.05, 23.88, 23.79, 23.70, 23.40, 23.19, 23.12 and 23.03 (*i*Pr Me), 21.60 and 20.13 (DAD Me), 14.65 (q, J_{CH} = 131,

$PdCH_2CH\mathit{Me}$). Anal. Calcd for ($C_{66}H_{65}O_2N_2BF_{24}Pd$): C, 53.15; H, 4.39; N, 1.88. Found: C, 52.9; H, 4.2; N, 1.8.

Copolymerization of ethene with ADMA by 2. The reactions were performed in a 50 mL glass mini-autoclave (Büchi) equipped with a Teflon coated magnetic stirrer. Before use, the reactor was dried in a vacuum over at 80°C for 2 h. In a nitrogen-filled drybox the reactor was charged with **2**, ADMA and dichloromethane solvent. (Nevertheless, the reaction is not adversely affected by charging the reactor under areobic conditions.) The reactor was then pressurized with 5 bar ethene and stirred at ambient temperature while the ethene pressure was kept constant. After the specified reaction time, the reactor was vented, and an excess of methanol was added while stirring. Subsequently the volatiles were removed in vacuo at 40°C. Further work-up was performed under areobic conditions. The polymer was recovered from the orange residue (the color resulting from the presence of **3**) by extraction with petroleum ether (leaving air-stable **3** to be recovered, see below). The polymer was precipitated by the addition of methanol. This sequence may be repeated to remove any remaining traces of **3**. The viscous polymer was subsequently dried in vacuo at 80°C overnight.

^1H and ^{13}C NMR spectra of a sample with 1.7 mol% comonomer incorporation are shown on the next page. (Note the two small singlets in the ^1H NMR spectrum at δ 3.47 and 3.65 ppm. The first is a trace of remaining methanol, the second is a resonance that appears to be associated with partial methanol loss from the acetal function upon warming the polymer when drying. When the polymer is treated with acid, this resonance increases together with resonances in the δ 9.5-9.8 region, probably associated with aldehyde groups)

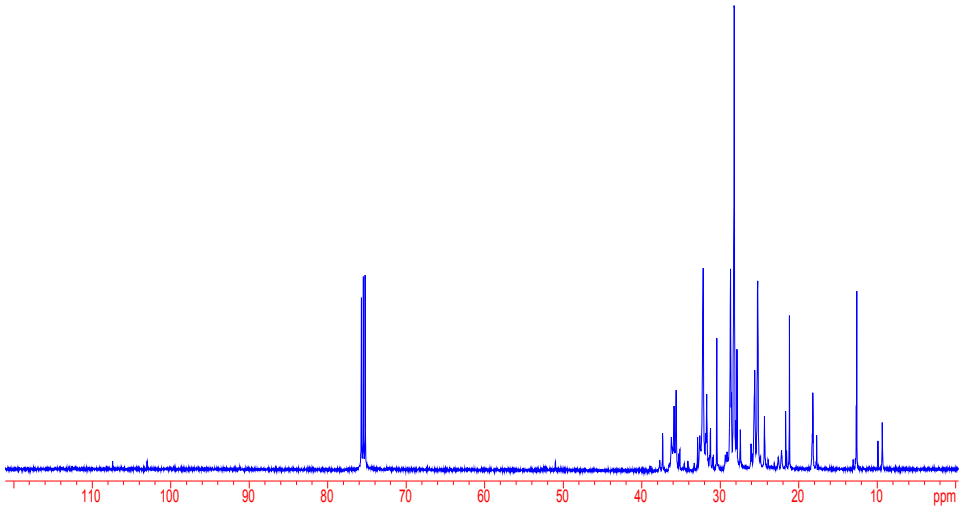
^{1}H NMR ($CDCl_3$, 500 MHz) spectrum of PE-*co*-ADMA (1.7 mol% comonomer)



I I II

C<u>*H*</u>(OMe)$_2$ O<u>*Me*</u>

^{13}C NMR ($CDCl_3$, 126 MHz) spectrum of PE-*co*-ADMA (1.7 mol% comonomer)



I I II

<u>*C*</u>H(OMe)$_2$ O<u>*Me*</u>

[(DAD)Pd(η^3-$CH_2CHCHOMe$)][BAr^f_4] (3). From a polymerization experiment (20 h run time) as described above, with 100 mg (67 μmol) of **2**, 1.0 mL (0.86 g, 8.44 mmol) of ADMA and 3 mL of dichloromethane, the crude complex was filtered off from the petroleum ether extract, and rinsed three times with petroleum ether to remove traces of remaining polymer. The orange powder was then recrystallized from a dichloromethane solution by the addition of petroleum ether. This yielded 80 mg (55 μmol, 82%) of **3** as deep red-orange crystals. ^{1}H NMR (CD_2Cl_2, 500MHz, 25 ^{o}C): δ 7.72 (s, 8H, Ar^f H_o), 7.56 (s, 4H, Ar^f H_p), 7.42 – 7.30 (m, 6H, Ar), 5.66 (d, J = 9.6, C*H*OMe), 5.27 (m, CH_2C*H*CHOMe), 2.95 (m, C*H*H'CHCHOMe), 2.91 and 2.76 (septet, 2H each, *i*Pr CH), 2.62 (s, 3H, OMe), 2.44 (dd, J = 12.8 and 2.2, CH*H'*CHCHOMe), 2.24 and 2.23 (s, 3H each, DAD Me), 1.43, 1.31, 1.31, 1.30, 1.27, 1.24, 1.22 and 1.11 (d, 3H each, J = 6.9, *i*Pr Me). ^{13}C NMR (CD_2Cl_2, 126MHz, 25 ^{o}C): δ175.06 and 174.93 (DAD C=N), 162.11 (q, J_{CB} = 50.7, Ar^f C_{ipso}), 144.14 and 143.07 (Ar C_{ipso}), 138.44 and 136.86 (Ar C_o), 135.16 (Ar^f C_o), 129.21 (q, J_{CF} = 30.7, Ar^f C_m), 128.88 and 128.69 (Ar C_p), 125.01 and 124.84 (Ar C_m), 125.01 (q, J_{CF} = 268.7, Ar^f CF_3), 120.33 (d, J_{CH} = 168, CH_2CH*C*HOMe), 117.83 (septet, J_{CF} = 3.9, Ar^f C_p), 98.67 (d, J_{CH} = 149, CH_2*C*HCHOMe), 60.18 (q, J_{CH} = 141, OMe), 54.50 (t, J_{CH} = 156, *C*H_2CHCHOMe), 29.85, 29.73, 29.45 and 29.35 (*i*Pr CH), 23.84, 23.81, 23.59, 23.37, 23.33, 23.30, 22.79 and 22.72 (*i*Pr Me), 20.33 and 20.02 (DAD Me). Anal. Calcd for ($C_{64}H_{59}ON_2BF_{24}Pd$): C, 53.18; H, 4.11; N, 1.94. Found: C, 53.28; H, 4.05; N, 1.88.

[(DAD)Pd($CH_2CHMeCH_2OEt$)][BAr^f_4] (5). Allyl ethyl ether (8.2 μL, 72 μmol) was added to a solution of [(DAD)PdMe(Et_2O)][BAr^f_4] (105 mg, 72 μmol) in 5 mL of CH_2Cl_2. The red solution was stirred for 2 hours at room temperature. Addition of 10 mL of hexane to the mixture and cooling to –30 ^{o}C yielded the title compound as oragne crystals. Yield 65 mg (44 μmol, 61%). ^{1}H NMR (CD_2Cl_2, 500MHz, 25 ^{o}C): δ

7.72 (s, 8H, Ar^f H_o), 7.56 (s, 4H, Ar^f H_p), 7.42 – 7.30 (m, 6H, Ar), 3.53 (dd, J = 7.7 and 6, C*H*H'OEt), 3.14 (t, J = 7.6, CH*H'*OEt), 2.97 (m, 4H,*i*Pr CH), 2.85 (m, 2H, OC*H*$_2$Me), 2.20 and 2.15 (s, DAD Me), 1.67 (m, 2H, PdCH$_2$), 1.29 (m, PdCH$_2$C*H*Me), 1.42, 1.41, 1.40, 1.39, 1.25, 1.24, 1.20 and 1.19 (d, 3H each, J = 7.0, *i*Pr Me), 0.82 (t, J = 7.0, OCH$_2$*Me*), 0.79 (d, J = 7.0, PdCH$_2$CH*Me*). ^{13}C NMR (CD_2Cl_2, 126MHz, 25 ^{o}C): δ 177.92 and 171.97 (DAD C=N), 162.10 (q, J_{CB} = 50.5, Ar^f C_{ipso}), 142.17 and 141.55 (Ar C_{ipso}), 138.27, 138.24, 137.74 and 137.55 (Ar C_o), 135.16 (Ar^f C_o), 129.11 (q, J_{CF} = 30.4, Ar^f C_m), 129.30 and 128.90 (Ar C_p), 125.20, 125.10, 125.03 and 124.94 (Ar C_m), 124.95 (q, J_{CF} = 272.1, Ar^f CF_3), 117.84 (septet, J_{CF} = 3.9, Ar^f C_p), 83.69 (t, J_{CH} = 145, *C*H$_2$OEt), 72.84 (t, J_{CH} = 146, O*C*H$_2$Me), 47.77 (t, J_{CH} = 150, Pd*C*H$_2$), 37.45 (d, J_{CH} = 161, PdCH$_2$*C*HMe), 29.56, 29.45, 29.13 and 29.13 (*i*Pr CH), 24.02, 23.88, 23.63, 23.59, 23.44, 23.43, 23.30 and 23.15 (*i*Pr Me), 21.59 and 20.35 (DAD Me), 14.62 (q, J_{CH} = 127, PdCH$_2$CH*Me*), 13.69 (q, J_{CH} = 127, OCH$_2$*Me*). Anal. Calcd for ($C_{66}H_{65}ON_2BF_{24}Pd$): C, 53.73; H, 4.44; N, 1.90. Found: C, 53.59; H, 4.41; N, 1.88.

[(DAD)Pd(η^3-CH$_2$CHCH$_2$)][BAr$^f_4$] (4).

A. Synthesized from (DAD)PdMeCl. Allyl ethyl ether (0.2 mL, 1.8 mmol) was added to a solution of (DAD)PdMeCl (204 mg, 0.36 mmol) and $NaBAr^f_4$ (341 mg, 0.39 mmol) in 20 mL Et_2O. The mixture was stirred at ambient temperature for 2 days. After removal of the volatiles in vacuo, the mixture was extracted with 10 mL of toluene. After addition of 30 mL of pentane and cooling to –80 ^{o}C an orange crystalline solid precipitated. After recrystallization from toluene/pentane, 330 mg of the title compound was obtained (0.23 mmol, 59%). ^{1}H NMR (CD_2Cl_2, 500MHz, 25 ^{o}C): δ 7.71 (s, 8H, Ar^f H_o), 7.56 (s, 4H, Ar^f H_p), 7.42 – 7.30 (m, 6H, Ar), 5.64 (m, allyl CH), 3.35 (d, 2H, J = 7.0, allyl C*H*H), 3.04 (d, 2H, J = 12.8, allyl CH*H*), 2.96 and 2.70 (septet, 2H each, J = 6.8, *i*Pr CH), 2.25 (s, 6H, DAD Me), 1.35, 1.26, 1.23

and 1.22 (d, 6H each, *i*Pr Me). ^{13}C NMR (CD_2Cl_2, 126MHz, 25 ^{o}C): δ 176.69 (DAD C=N), 162.10 (q, J_{CB} = 50.5, Ar^f C_{ipso}), 143.91 (Ar C_{ipso}), 136.95 and 136.91 (Ar C_o), 135.16 (Ar^f C_o), 129.25 (q, J_{CF} = 32.8, Ar^f C_m), 129.15 (Ar C_p), 125.03 and 124.98 (Ar C_m), 124.96 (q, J_{CF} = 273.0, Ar^f CF_3), 121.03 (d, J_{CH} = 170, allyl CH), 117.84 (septet, J_{CF} = 3.7, Ar^f C_p), 65.78 (t, J_{CH} = 185, allyl CH_2), 29.82 and 29.44 (*i*Pr CH), 23.65, 23.42, 23.33 and 23.30 (*i*Pr Me), 20.19 (DAD Me). Anal. Calcd for ($C_{63}H_{57}N_2BF_{24}Pd$): C, 53.46; H, 4.06; N, 1.98. Found: C, 53.1; H, 4.1; N, 1.8.

B. Recovered from ethene/AEE copolymerization. The same procedure was followed as described above for **3**. In this experiment, 100 mg of **2** (67.1 μmol) was used with 1.0 mL of AEE (0.76 g, 8.82 mmol) in 5 mL of dichloromethane. After 20 hours reaction time, work up similar to that described above yielded 87 mg (61 μmol, 87%) of complex **4**.

*Part II: Crystal structure determination of **2**.*

Abstract. $[C_{34}H_{53}N_2O_2Pd]^+.[C_{32}H_{12}BF_{24}]^-$, M_r = 1491.45, monoclinic, $P2_1/n$, a = 12.8098(7), b = 28.259(2), c = 19.154(1) Å, β = 103.450(1)°, V = 6743.4(7) Å^3, Z = 4, D_x = 1.469 gcm^{-3}, F(000) = 3032, μ = 3.86 cm^{-1}, λ(MoK$\bar{\alpha}$) = 0.71073 Å, T = 100(1) K, 48552 reflections measured, *GooF* = 1.024, *wR*(F^2) = 0.2107 for 11901 unique reflections and 950 parameters, 44 restraints and $R(F)$ = 0.0726 for 8207 reflections obeying $F_o \geq 4.0\ \sigma(F_o)$ criterion of observability.

The asymmetric unit consists of two moieties: a cationic Pd-complex, which is highly disordered, and a fluorinated-tetra-phenyl-borate anion.

Experimental

X-ray diffraction: Crystal and Molecular Structure.

A crystal with the dimensions of 0.27 x 0.17 x 0.15 mm was mounted on top of a glass fiber, by using inert-atmosphere handling techniques, and aligned on a *Bruker*[1] *SMART APEX CCD* diffractometer (Platform with full three-circle goniometer). The diffractometer was equipped with a 4K *CCD* detector set 60.0 mm from the crystal. The crystal was cooled to 100(1) K using the *Bruker KRYOFLEX* low-temperature device. Intensity measurements were performed using graphite monochromated Mo-K$\bar{\alpha}$ radiation from a sealed ceramic diffraction tube (*SIEMENS*). Generator settings were 50 KV/ 40 mA. *SMART* was used for preliminary determination of the unit cell constants and data collection control. The intensities of reflections of a hemisphere were collected by a combination of 3 sets of exposures (frames). Each set had a different ϕ angle for the crystal and each exposure covered a range of 0.3° in ω. A total of 1800 frames were collected with an exposure time of 20.0 seconds per frame. The overall data collection time was 13.3 h. Data integration and global cell refinement was performed with the program *SAINT*. The final unit cell was obtained from the *xyz* centroids of 7211 reflections after integration. Intensity data were corrected for Lorentz and polarization effects, scale variation, for decay and absorption: a multi-scan absorption correction was applied, based on the intensities of symmetry-related reflections measured at different angular settings (*SADABS*)[2], and reduced to F_o^2. The program suite *SHELXTL* was used for space group determination (*XPREP*).[1]

The unit cell[3] was identified as monoclinic; reduced cell calculations did not indicate any higher metric lattice symmetry.[4] The space group $P2_1/n$, was derived from the systematic extinctions. Examination of the final atomic coordinates of the structure did not yield extra crystallographic or metric symmetry elements.[5,6]

The structure was solved by Patterson methods and extension of the model was accomplished by direct methods applied to difference structure factors using the program *DIRDIF*.[7] The positional and anisotropic displacement parameters for the non-hydrogen atoms were refined. Refinement was complicated by a configurational-/conformational-disorder problem: from the

solution it was clear that the chelate moiety resulting from insertion of acrolein dimethyl acetal into the Pd-Me bond (O11-O12-C129—C134 positions) is highly disordered. A disorder model with two alternative conformations with bond restraints was used in the final refinement (in which the disorder is compensated by the large thermal displacement parameters). Although the disorder model was not satisfactory in all respects, it was the best of several models tested.

The disorder phenomena may also account for the observed unrealistic displacement parameters for some atoms when allowed to vary anisotropically, suggesting dynamic disorder (dynamic means that the smeared electron density is due to fluctuations of the atomic positions within each unit cell) as a consequence of the configurational- and rotational-disorder. This is in line with the weak scattering power of the crystals investigated.

The hydrogen atoms were included in the final refinement riding on their carrier atoms with their positions calculated by using sp^2 or sp^3 hybridization at the C-atom as appropriate with $U_{iso} = c \times U_{equiv}$ of their parent atom, where c = 1.2 for the non-methyl hydrogen atoms and c = 1.5 for the methyl hydrogen atoms and where values U_{equiv} are related to the atoms to which the hydrogen atoms are bonded. The methyl-groups were refined as rigid groups, which were allowed to rotate free.

Final refinement on F^2 carried out by full-matrix least-squares techniques converged at $wR(F^2)$ = 0.2107 for 11901 reflections and $R(F)$ = 0.0726 for 8207 reflections with $F_o \geq 4.0\ \sigma(F_o)$ and 950 parameters and 44 restraints. The final difference Fourier map was essentially featureless with one peak of 1.62 e/Å^3 in the neighborhood of F224. No other significant peaks (max. = 0.95(9) e/Å^3) having chemical meaning above the general background were observed in the final difference Fourier syntheses.

The positional and anisotropic displacement parameters for the non-hydrogen atoms and isotropic displacement parameters for hydrogen atoms were refined on F^2 with full-matrix least-squares procedures minimizing the function $Q = \sum_h [w(|(F_o^2) - k(F_c^2)|)^2]$, where $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$, $P = [\max(F_o^2,0) + 2F_c^2] / 3$, F_0 and F_c are the observed and calculated structure factor amplitudes, respectively; ultimately the suggested a (=0.1057) and b (= 15.8251) were used in the final refinement.

Crystal data and numerical details on data collection and refinement are given in Table 1. Final fractional atomic coordinates, equivalent displacement parameters and anisotropic displacement parameters for the non-hydrogen atoms are given in Table 2. Molecular geometry data are collected in Table 3. Neutral atom scattering factors and anomalous dispersion corrections were taken from *International Tables for Crystallography.*[10]

All refinement calculations and graphics were performed on a Pentium-III / Debian-Linux computer at the University of Groningen with the program packages *SHELXL*[11] (least-square refinements), a locally modified version of the program *PLUTO*[12] (preparation of illustrations) and *PLATON*[9] package (checking the final results for missed symmetry with the *MISSYM* option,

solvent accessible voids with the *SOLV* option, calculation of geometric data and the *ORTEP*[9] illustrations).

Each asymmetric unit contains one formula unit, consisting of two moieties: a cation Pd-complex and a fluorinated-tetraphenylborate anion separated by normal van der Waals distances[13].

No classic hydrogen bonds, no missed symmetry (*MISSYM*), but potential solvent-accessible area (voids of 49.2 $Å^3$ / unit cell) were detected by procedures implemented in *PLATON.*[14,15]

*) For disordered atoms, in the refinement described by several fractional s.o.f. atoms, only one of the fractional s.o.f. atoms is included with a dashed bonds / (displacement) ellipses. H-atoms have been omitted to improve clarity.

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Table 1.

a. Crystal data and details of the structure determination.

Moiety_Formula	$[C_{34}H_{53}N_2O_2Pd]^+.[C_{32}H_{12}BF_{24}]^-$
Formula_Weight, g.mol^{-1}	1491.45
Crystal system	monoclinic
Space group, no.[16]	$P2_1/n$, 14
a, Å	12.8098(7)
b, Å	28.259(2)
c, Å	19.154(1)
β, deg	103.450(1)
V, Å^3	6743.4(7)
Θ range unit cell: min.-max., deg; reflections	2.30 - 24.35 ; 7211
Formula_*Z*	4
SpaceGroup_*Z*	4
Z' (= Formula_Z / SpaceGroup_*Z)*	1
ρ_{calc}, g.cm^{-3}	1.469
F(000), electrons	3032
μ(Mo K$\bar{\alpha}$), cm^{-1}	3.86
Color, habit	red, block
Approx. crystal dimension, mm	0.27 x 0.17 x 0.15

b. Data collection.

λ(Mo K$\bar{\alpha}$), Å	0.71073
Monochromator	Graphite
Measurement device type	*CCD* area-detector diffractometer
Detector Area resolution (pixels / mm)	4096 x 4096 / 62 x 62 (binned 512)
Temperature, K	100(1)
Measurement method	φ- and ω-scans
θ range; min. max., deg	2.26, 25.03
Index ranges	h: -15→15; k: -33→33; l: -22→22
Min.- Max. absorption transmission factor	0.9037 – 0.9444
X-ray exposure time, h	13.3
Total data	48552
Unique data	11901
Data with criterion: ($F_o \geq 4.0\ \sigma\ (F_o)$)	8207
$R_{int} = \sum [\lvert F_o^2 - F_o^2\ (\text{mean})\rvert] / \sum [F_o^2]$	0.0534
$R_{sig} = \sum \sigma(F_o^2) / \sum [F_o^2]$	0.0611

c. Refinement.

Number of reflections	11901
Number of refined parameters	950
Number of restraints	44
Final agreement factors:	
$wR(F^2) = [\sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)^2]]^{1/2}$	0.2107
Weighting scheme: *a, b*	0.1057, 15.8251
$w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$	
And $P = [\max(F_o^2,0) + 2F_c^2] / 3$	
$R(F) = \sum (\|\|F_o\| - \|F_c\|\|) / \sum \|F_o\|$	0.0726
For $F_o > 4.0\ \sigma\ (F_o)$	
$\text{GooF} = S = [\sum [w(F_o^2 - F_c^2)^2] / (n-p)]^{1/2}$	1.024
n = number of reflections	
p = number of parameters refined	
Residual electron density in final	
Difference Fourier map, e/Å^3	-0.64, 0.95(9)
Max. (shift/σ) final cycle	0.004
Average (shift/σ) final cycle	0.000

Table 2. Final fractional atomic coordinates and equivalent isotropic displacement parameters with s.u.'s in parentheses.

Atoms of the Asymmetric Unit.

Non-Hydrogen parameters

Residue: 1.

Atom	*x*	*y*	*z*	*s.o.f.*	U_{eq} (Å^2)
Pd11	0.27869(4)	0.49174(2)	0.24755(2)	1.0(-) 0.04701(17)	
O112	0.1895(11)	0.5075(6)	0.3209(7)	0.507(8)	0.082(5)
O122	0.090(2)	0.5653(5)	0.2647(13)	0.507(8)	0.174(10)
N11	0.3615(3)	0.48154(14)	0.1659(2)	1.0(-)	0.0334(12)
N12	0.2909(3)	0.41870(14)	0.2421(2)	1.0(-)	0.0354(14)
C1292	0.115(2)	0.4816(7)	0.3465(15)	0.507(8)	0.160(15)
C1302	0.167(2)	0.5558(9)	0.3232(16)	0.507(8)	0.176(13)
C1312	0.255(3)	0.5861(8)	0.3060(14)	0.507(8)	0.156(10)
C1322	0.274(3)	0.5657(11)	0.2386(14)	0.507(8)	0.086(9)
C1332	-0.015(3)	0.578(3)	0.259(5)	0.507(8)	0.55(7)
C11	0.3986(4)	0.52034(17)	0.1289(3)	1.0(-)	0.0349(17)
C12	0.3242(4)	0.54185(18)	0.0727(3)	1.0(-)	0.0361(17)
C1342	0.320(4)	0.567(2)	0.3799(16)	0.507(8)	0.40(4)
C13	0.3586(5)	0.58252(18)	0.0430(3)	1.0(-)	0.0436(17)
C14	0.4609(5)	0.60012(19)	0.0686(3)	1.0(-)	0.0462(19)
C15	0.5321(5)	0.5775(2)	0.1224(3)	1.0(-)	0.0461(17)
C16	0.5041(4)	0.53676(19)	0.1548(3)	1.0(-)	0.0419(17)
C17	0.2132(4)	0.52203(19)	0.0433(3)	1.0(-)	0.0445(19)
C18	0.2015(5)	0.5053(2)	-0.0338(3)	1.0(-)	0.053(2)
C19	0.1249(5)	0.5574(3)	0.0470(5)	1.0(-)	0.079(3)
C110	0.5808(5)	0.5122(2)	0.2159(3)	1.0(-)	0.060(2)
C111	0.5657(6)	0.5288(3)	0.2884(3)	1.0(-)	0.082(3)
C112	0.6992(5)	0.5172(3)	0.2120(4)	1.0(-)	0.064(3)
C113	0.3760(4)	0.43826(17)	0.1499(2)	1.0(-)	0.0309(14)
C114	0.4272(4)	0.42325(18)	0.0911(3)	1.0(-)	0.0373(17)
C115	0.3372(4)	0.40209(17)	0.1949(3)	1.0(-)	0.0324(17)
C116	0.3550(4)	0.35090(17)	0.1838(3)	1.0(-)	0.0390(17)
C117	0.2467(5)	0.38844(18)	0.2885(3)	1.0(-)	0.0411(19)
C118	0.3041(6)	0.3830(3)	0.3587(3)	1.0(-)	0.066(3)
C119	0.2534(8)	0.3565(3)	0.4041(4)	1.0(-)	0.079(3)
C120	0.1519(7)	0.3388(2)	0.3793(4)	1.0(-)	0.074(3)
C121	0.0983(7)	0.3447(3)	0.3103(4)	1.0(-)	0.074(3)
C122	0.1442(5)	0.3687(2)	0.2620(3)	1.0(-)	0.056(2)
C123	0.4156(8)	0.4037(4)	0.3841(4)	1.0(-)	0.105(4)
C124	0.4995(9)	0.3695(7)	0.3772(6)	1.0(-)	0.202(11)
C125	0.4389(10)	0.4246(5)	0.4607(5)	1.0(-)	0.144(6)
C126	0.0831(6)	0.3752(3)	0.1850(4)	1.0(-)	0.083(3)
C127	0.0088(7)	0.3338(4)	0.1562(5)	1.0(-)	0.110(4)
C128	0.0227(7)	0.4216(3)	0.1770(5)	1.0(-)	0.105(4)
O111	0.2560(13)	0.5627(5)	0.2612(7)	0.493(8)	0.053(4)
O121	0.1609(10)	0.6181(3)	0.3050(7)	0.493(8)	0.094(5)

C1291	0.3246(14)	0.5993(4)	0.2468(8)	0.493(8)	0.075(6)
C1301	0.2014(14)	0.5727(4)	0.3152(8)	0.493(8)	0.054(5)
C1311	0.1309(18)	0.5300(7)	0.3188(11)	0.493(8)	0.097(8)
C1321	0.205(3)	0.4853(6)	0.3290(16)	0.493(8)	0.117(12)
C1331	0.0673(17)	0.6212(7)	0.2506(11)	0.493(8)	0.121(9)
C1341	0.080(2)	0.5399(6)	0.3831(13)	0.493(8)	0.139(13)

Residue: 2.

F21	0.7307(9)	0.3868(3)	0.1291(4)	1.0(-)	0.269(6)
F22	0.6584(5)	0.4028(2)	0.2193(4)	1.0(-)	0.163(3)
F23	0.8053(4)	0.3679(2)	0.2361(3)	1.0(-)	0.118(2)
F24	0.6364(9)	0.2582(3)	0.3606(3)	1.0(-)	0.199(5)
F25	0.6229(5)	0.1950(2)	0.3081(3)	1.0(-)	0.124(3)
F26	0.4852(5)	0.2333(4)	0.3096(4)	1.0(-)	0.202(5)
F27	0.7914(4)	0.12230(19)	0.0942(3)	1.0(-)	0.0993(19)
F28	0.7027(5)	0.0902(3)	0.1573(3)	1.0(-)	0.186(4)
F29	0.7295(5)	0.05607(17)	0.0641(4)	1.0(-)	0.133(3)
F210	0.3292(10)	0.0464(3)	-0.0607(5)	1.0(-)	0.283(6)
F211	0.3958(7)	0.0682(3)	-0.1433(4)	1.0(-)	0.195(4)
F212	0.2702(5)	0.10570(18)	-0.1213(3)	1.0(-)	0.110(2)
F213	0.1391(3)	0.20761(18)	0.1524(2)	1.0(-)	0.0888(19)
F214	0.1214(4)	0.16337(18)	0.0635(3)	1.0(-)	0.114(2)
F215	0.0057(3)	0.21851(19)	0.0654(3)	1.0(-)	0.104(2)
F216	0.0538(2)	0.36204(12)	-0.06720(19)	1.0(-)	0.0577(11)
F217	0.2118(3)	0.38273(11)	-0.07600(17)	1.0(-)	0.0500(11)
F218	0.1634(3)	0.39404(11)	0.02199(17)	1.0(-)	0.0532(11)
F219	0.3729(5)	0.25713(17)	-0.2698(3)	1.0(-)	0.113(2)
F220	0.3280(3)	0.32135(19)	-0.23130(19)	1.0(-)	0.0912(18)
F221	0.4543(3)	0.32024(17)	-0.28335(19)	1.0(-)	0.0824(18)
F222	0.8126(3)	0.3220(2)	-0.1285(3)	1.0(-)	0.121(2)
F223	0.8319(4)	0.3163(2)	-0.0224(3)	1.0(-)	0.123(3)
F224	0.8394(3)	0.25537(17)	-0.0763(5)	1.0(-)	0.145(3)
C21	0.5404(4)	0.26608(18)	0.1046(3)	1.0(-)	0.0360(17)
C22	0.5991(4)	0.3081(2)	0.1126(3)	1.0(-)	0.0457(19)
C23	0.6520(5)	0.3259(3)	0.1801(4)	1.0(-)	0.064(3)
C24	0.6470(6)	0.3031(3)	0.2410(4)	1.0(-)	0.078(3)
C25	0.5881(6)	0.2625(3)	0.2362(3)	1.0(-)	0.067(3)
C26	0.5349(5)	0.2439(2)	0.1685(3)	1.0(-)	0.0518(19)
C27	0.7164(7)	0.3712(3)	0.1800(7)	1.0(-)	0.112(4)
C28	0.5750(8)	0.2350(5)	0.3013(4)	1.0(-)	0.116(5)
C29	0.4905(4)	0.18799(18)	0.0220(3)	1.0(-)	0.0382(17)
C210	0.5836(5)	0.16548(19)	0.0593(3)	1.0(-)	0.0449(19)
C211	0.6041(6)	0.1179(2)	0.0490(3)	1.0(-)	0.057(2)
C212	0.5314(7)	0.0918(2)	0.0018(3)	1.0(-)	0.066(3)
C213	0.4368(6)	0.1123(2)	-0.0369(3)	1.0(-)	0.065(2)
C214	0.4173(5)	0.1603(2)	-0.0259(3)	1.0(-)	0.0527(19)
C215	0.7057(8)	0.0960(3)	0.0906(4)	1.0(-)	0.074(3)
C216	0.3539(10)	0.0838(3)	-0.0864(6)	1.0(-)	0.118(4)
C217	0.3472(4)	0.25924(16)	0.0218(2)	1.0(-)	0.0301(14)
C218	0.2787(4)	0.22991(19)	0.0494(3)	1.0(-)	0.0393(17)

C219	0.1748(4)	0.2423(2)	0.0498(3)	1.0(-)	0.0426(17)
C220	0.1326(4)	0.2849(2)	0.0212(3)	1.0(-)	0.0425(19)
C221	0.1988(4)	0.31547(17)	-0.0053(3)	1.0(-)	0.0315(16)
C222	0.3032(4)	0.30284(16)	-0.0041(2)	1.0(-)	0.0288(12)
C223	0.1088(5)	0.2079(3)	0.0819(4)	1.0(-)	0.064(3)
C224	0.1569(4)	0.36285(18)	-0.0323(3)	1.0(-)	0.0385(17)
C225	0.5173(4)	0.26584(15)	-0.0408(3)	1.0(-)	0.0303(16)
C226	0.4493(4)	0.27374(16)	-0.1090(3)	1.0(-)	0.0317(16)
C227	0.4895(4)	0.28778(17)	-0.1673(3)	1.0(-)	0.0359(17)
C228	0.5982(4)	0.29392(18)	-0.1607(3)	1.0(-)	0.0432(17)
C229	0.6676(4)	0.2861(2)	-0.0945(3)	1.0(-)	0.0446(17)
C230	0.6270(4)	0.27160(18)	-0.0362(3)	1.0(-)	0.0377(17)
C231	0.4121(5)	0.2963(2)	-0.2376(3)	1.0(-)	0.0466(19)
C232	0.7855(5)	0.2925(3)	-0.0857(4)	1.0(-)	0.062(2)
B2	0.4734(4)	0.24545(19)	0.0271(3)	1.0(-)	0.0319(17)

Hydrogen parameters

Residue: 1.

Atom	*x*	*y*	*z*	*s.o.f.*	U_{eq} (Å^2)
H13	0.31090(-)	0.59828(-)	0.00466(-)	1.0(-)	0.05236(-)
H14	0.48194(-)	0.62829(-)	0.04854(-)	1.0(-)	0.05528(-)
H15	0.60268(-)	0.58983(-)	0.13815(-)	1.0(-)	0.05529(-)
H17	0.20405(-)	0.49393(-)	0.07308(-)	1.0(-)	0.05315(-)
H18	0.21270(-)	0.53211(-)	-0.06369(-)	1.0(-)	0.07836(-)
H18'	0.12926(-)	0.49243(-)	-0.05201(-)	1.0(-)	0.07836(-)
H18"	0.25489(-)	0.48075(-)	-0.03512(-)	1.0(-)	0.07836(-)
H19	0.12777(-)	0.56514(-)	0.09735(-)	1.0(-)	0.11797(-)
H19'	0.05484(-)	0.54360(-)	0.02501(-)	1.0(-)	0.11797(-)
H19"	0.13534(-)	0.58632(-)	0.02119(-)	1.0(-)	0.11797(-)
H110	0.56311(-)	0.47768(-)	0.21201(-)	1.0(-)	0.07167(-)
H111	0.57459(-)	0.56321(-)	0.29201(-)	1.0(-)	0.12280(-)
H111'	0.61916(-)	0.51355(-)	0.32675(-)	1.0(-)	0.12280(-)
H111"	0.49346(-)	0.52031(-)	0.29316(-)	1.0(-)	0.12280(-)
H112	0.70696(-)	0.50752(-)	0.16438(-)	1.0(-)	0.09616(-)
H112'	0.74422(-)	0.49708(-)	0.24863(-)	1.0(-)	0.09616(-)
H112"	0.72162(-)	0.55029(-)	0.22058(-)	1.0(-)	0.09616(-)
H114	0.37551(-)	0.40506(-)	0.05536(-)	1.0(-)	0.05552(-)
H114'	0.48998(-)	0.40352(-)	0.11102(-)	1.0(-)	0.05552(-)
H114"	0.44969(-)	0.45129(-)	0.06832(-)	1.0(-)	0.05552(-)
H116	0.32408(-)	0.33218(-)	0.21707(-)	1.0(-)	0.05845(-)
H116'	0.43228(-)	0.34464(-)	0.19279(-)	1.0(-)	0.05845(-)
H116"	0.32060(-)	0.34215(-)	0.13431(-)	1.0(-)	0.05845(-)
H119	0.29014(-)	0.35089(-)	0.45248(-)	1.0(-)	0.09452(-)
H120	0.11862(-)	0.32210(-)	0.41124(-)	1.0(-)	0.08842(-)
H121	0.02796(-)	0.33210(-)	0.29465(-)	1.0(-)	0.08947(-)
H123	0.42063(-)	0.43052(-)	0.35104(-)	1.0(-)	0.12631(-)
H124	0.50082(-)	0.34346(-)	0.41123(-)	1.0(-)	0.30502(-)
H124'	0.56943(-)	0.38535(-)	0.38771(-)	1.0(-)	0.30502(-)
H124"	0.48398(-)	0.35695(-)	0.32821(-)	1.0(-)	0.30502(-)

H125	0.43859(-)	0.39916(-)	0.49541(-)	1.0(-)	0.21606(-)
H125'	0.38351(-)	0.44789(-)	0.46388(-)	1.0(-)	0.21606(-)
H125"	0.50935(-)	0.44001(-)	0.47135(-)	1.0(-)	0.21606(-)
H126	0.13728(-)	0.37732(-)	0.15503(-)	1.0(-)	0.09932(-)
H127	0.04582(-)	0.30395(-)	0.17160(-)	1.0(-)	0.16425(-)
H127'	-0.01098(-)	0.33514(-)	0.10361(-)	1.0(-)	0.16425(-)
H127"	-0.05611(-)	0.33590(-)	0.17480(-)	1.0(-)	0.16425(-)
H128	-0.02732(-)	0.42176(-)	0.20868(-)	1.0(-)	0.15669(-)
H128'	-0.01749(-)	0.42538(-)	0.12708(-)	1.0(-)	0.15669(-)
H128"	0.07372(-)	0.44778(-)	0.18994(-)	1.0(-)	0.15669(-)
H1291	0.28210(-)	0.62767(-)	0.23038(-)	0.493(8)	0.11201(-)
H1291'	0.36014(-)	0.58870(-)	0.20939(-)	0.493(8)	0.11201(-)
H1291"	0.37893(-)	0.60656(-)	0.29065(-)	0.493(8)	0.11201(-)
H1292	0.13086(-)	0.48405(-)	0.39890(-)	0.507(8)	0.24180(-)
H1292'	0.11900(-)	0.44835(-)	0.33266(-)	0.507(8)	0.24180(-)
H1292"	0.04312(-)	0.49391(-)	0.32599(-)	0.507(8)	0.24180(-)
H1301	0.25705(-)	0.57321(-)	0.36146(-)	0.493(8)	0.06478(-)
H1302	0.14777(-)	0.56545(-)	0.36876(-)	0.507(8)	0.20782(-)
H1311	0.07363(-)	0.52721(-)	0.27350(-)	0.493(8)	0.11627(-)
H1312	0.24091(-)	0.62092(-)	0.30433(-)	0.507(8)	0.18828(-)
H1321	0.16176(-)	0.45581(-)	0.32477(-)	0.493(8)	0.13703(-)
H1321'	0.25669(-)	0.48577(-)	0.37610(-)	0.493(8)	0.13703(-)
H1322	0.34315(-)	0.57747(-)	0.23028(-)	0.507(8)	0.10295(-)
H1322'	0.21599(-)	0.57534(-)	0.19744(-)	0.507(8)	0.10295(-)
H1331	0.08295(-)	0.61178(-)	0.20479(-)	0.493(8)	0.18187(-)
H1331'	0.04091(-)	0.65385(-)	0.24705(-)	0.493(8)	0.18187(-)
H1331"	0.01255(-)	0.60013(-)	0.26157(-)	0.493(8)	0.18187(-)
H1332	-0.01916(-)	0.59880(-)	0.29952(-)	0.507(8)	0.82602(-)
H1332'	-0.05881(-)	0.55014(-)	0.25929(-)	0.507(8)	0.82602(-)
H1332"	-0.04071(-)	0.59571(-)	0.21393(-)	0.507(8)	0.82602(-)
H1341	0.13732(-)	0.54603(-)	0.42603(-)	0.493(8)	0.20816(-)
H1341'	0.03844(-)	0.51230(-)	0.39144(-)	0.493(8)	0.20816(-)
H1341"	0.03323(-)	0.56755(-)	0.37257(-)	0.493(8)	0.20816(-)
H1342	0.28928(-)	0.53736(-)	0.39101(-)	0.507(8)	0.59520(-)
H1342'	0.31737(-)	0.59056(-)	0.41739(-)	0.507(8)	0.59520(-)
H1342"	0.39526(-)	0.56226(-)	0.37784(-)	0.507(8)	0.59520(-)

Residue: 2.

H22	0.60358(-)	0.32531(-)	0.07084(-)	1.0(-)	0.05506(-)
H24	0.68380(-)	0.31514(-)	0.28642(-)	1.0(-)	0.09317(-)
H26	0.49447(-)	0.21560(-)	0.16670(-)	1.0(-)	0.06228(-)
H210	0.63496(-)	0.18321(-)	0.09303(-)	1.0(-)	0.05439(-)
H212	0.54511(-)	0.05921(-)	-0.00481(-)	1.0(-)	0.07962(-)
H214	0.35234(-)	0.17420(-)	-0.05183(-)	1.0(-)	0.06297(-)
H218	0.30501(-)	0.20012(-)	0.06890(-)	1.0(-)	0.04724(-)
H220	0.06013(-)	0.29303(-)	0.01967(-)	1.0(-)	0.05076(-)
H222	0.34714(-)	0.32477(-)	-0.02168(-)	1.0(-)	0.03437(-)
H226	0.37408(-)	0.26940(-)	-0.11529(-)	1.0(-)	0.03807(-)
H228	0.62501(-)	0.30333(-)	-0.20087(-)	1.0(-)	0.05216(-)
H230	0.67601(-)	0.26542(-)	0.00836(-)	1.0(-)	0.04517(-)

*) $U_{eq} = 1/3 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a_i}.\mathbf{a_j}$ [17]

Anisotropic (displacement) parameters (Å^2)

Residue: 1.

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Pd11	0.0644(3)	0.0345(3)	0.0528(3)	-0.0094(2)	0.0353(2)	-0.0077(2)
O112	0.098(9)	0.090(9)	0.083(8)	-0.046(8)	0.070(7)	-0.017(7)
O122	0.256(19)	0.070(9)	0.27(2)	0.036(11)	0.210(17)	0.046(10)
N11	0.042(2)	0.031(2)	0.029(2)	-0.0025(17)	0.0117(18)	-0.0096(18)
N12	0.045(3)	0.029(2)	0.035(2)	0.0031(18)	0.015(2)	-0.0012(19)
C1292	0.24(3)	0.097(14)	0.21(3)	0.027(16)	0.19(3)	0.009(15)
C1302	0.32(3)	0.077(11)	0.20(2)	-0.03(2)	0.201(17)	-0.001(17)
C1312	0.30(2)	0.059(11)	0.162(19)	-0.017(14)	0.16(2)	0.003(15)
C1322	0.103(18)	0.073(16)	0.093(14)	-0.011(13)	0.047(12)	0.024(12)
C1332	0.30(5)	0.62(14)	0.83(14)	0.37(14)	0.34(5)	0.20(8)
C11	0.045(3)	0.032(3)	0.033(3)	-0.007(2)	0.020(2)	-0.006(2)
C12	0.040(3)	0.035(3)	0.039(3)	-0.005(2)	0.021(2)	-0.005(2)
C1342	0.45(5)	0.59(10)	0.108(15)	-0.16(3)	-0.02(3)	0.13(6)
C13	0.050(3)	0.034(3)	0.051(3)	0.002(2)	0.020(3)	-0.001(2)
C14	0.056(4)	0.035(3)	0.056(3)	-0.006(3)	0.030(3)	-0.011(3)
C15	0.045(3)	0.046(3)	0.052(3)	-0.013(3)	0.021(3)	-0.019(3)
C16	0.050(3)	0.039(3)	0.039(3)	-0.013(2)	0.015(3)	-0.013(3)
C17	0.041(3)	0.036(3)	0.060(4)	0.006(3)	0.019(3)	-0.003(2)
C18	0.052(4)	0.047(3)	0.054(4)	0.004(3)	0.003(3)	-0.007(3)
C19	0.050(4)	0.069(5)	0.123(7)	-0.017(4)	0.031(4)	0.000(3)
C110	0.061(4)	0.059(4)	0.051(4)	-0.003(3)	-0.003(3)	-0.023(3)
C111	0.065(5)	0.136(7)	0.042(4)	0.003(4)	0.008(3)	-0.032(5)
C112	0.053(4)	0.079(5)	0.056(4)	-0.017(3)	0.004(3)	-0.014(3)
C113	0.028(2)	0.035(3)	0.029(2)	-0.003(2)	0.005(2)	-0.004(2)
C114	0.035(3)	0.039(3)	0.039(3)	-0.004(2)	0.011(2)	-0.003(2)
C115	0.031(3)	0.033(3)	0.032(3)	-0.001(2)	0.005(2)	0.002(2)
C116	0.047(3)	0.032(3)	0.039(3)	0.003(2)	0.012(2)	0.006(2)
C117	0.058(4)	0.030(3)	0.041(3)	0.008(2)	0.023(3)	0.008(2)
C118	0.078(5)	0.077(5)	0.051(4)	0.011(3)	0.029(4)	0.015(4)
C119	0.109(7)	0.087(6)	0.048(4)	0.023(4)	0.035(4)	0.035(5)
C120	0.112(7)	0.042(4)	0.085(6)	0.021(4)	0.060(5)	0.006(4)
C121	0.099(6)	0.058(4)	0.081(5)	0.004(4)	0.052(5)	-0.014(4)
C122	0.065(4)	0.042(3)	0.071(4)	0.004(3)	0.038(3)	-0.005(3)
C123	0.098(7)	0.178(10)	0.039(4)	0.008(5)	0.015(4)	-0.004(7)
C124	0.089(8)	0.43(3)	0.081(7)	-0.095(11)	0.006(6)	0.050(12)
C125	0.135(9)	0.229(15)	0.066(6)	-0.033(7)	0.018(6)	-0.035(9)
C126	0.062(5)	0.126(7)	0.068(5)	-0.006(5)	0.030(4)	-0.026(5)
C127	0.079(6)	0.138(9)	0.122(8)	-0.059(7)	0.044(5)	-0.020(6)
C128	0.072(6)	0.104(7)	0.123(8)	0.028(6)	-0.007(5)	-0.020(5)
O111	0.091(8)	0.018(5)	0.069(8)	-0.007(5)	0.060(6)	-0.006(4)
O121	0.124(10)	0.057(6)	0.110(9)	-0.018(6)	0.043(7)	0.032(6)

C1291	0.153(15)	0.024(6)	0.079(9)	-0.031(6)	0.09(1)	-0.046(8)
C1301	0.092(10)	0.027(6)	0.059(8)	0.003(6)	0.050(7)	0.012(6)
C1311	0.149(17)	0.059(9)	0.124(16)	-0.025(10)	0.115(14)	-0.021(9)
C1321	0.22(3)	0.029(8)	0.15(2)	-0.009(10)	0.14(2)	0.002(10)
C1331	0.137(17)	0.088(14)	0.133(17)	0.010(12)	0.021(11)	0.069(13)
C1341	0.22(3)	0.065(11)	0.20(2)	-0.006(12)	0.19(2)	0.008(12)

Residue: 2.

F21	0.379(14)	0.274(11)	0.083(4)	0.065(5)	-0.093(6)	-0.278(11)
F22	0.127(5)	0.117(5)	0.202(7)	-0.062(5)	-0.052(5)	0.013(4)
F23	0.058(3)	0.192(6)	0.094(3)	-0.065(4)	-0.005(2)	-0.013(3)
F24	0.344(12)	0.197(8)	0.039(3)	0.017(4)	0.009(5)	0.054(7)
F25	0.137(5)	0.137(5)	0.118(4)	0.065(4)	0.069(4)	0.047(4)
F26	0.112(5)	0.398(13)	0.125(5)	0.140(7)	0.088(4)	0.090(6)
F27	0.097(4)	0.089(3)	0.097(3)	0.000(3)	-0.008(3)	0.042(3)
F28	0.151(5)	0.31(1)	0.110(4)	0.138(6)	0.057(4)	0.151(6)
F29	0.120(4)	0.062(3)	0.194(6)	-0.015(3)	-0.010(4)	0.048(3)
F210	0.415(15)	0.102(5)	0.213(9)	0.070(5)	-0.168(10)	-0.150(7)
F211	0.197(7)	0.168(7)	0.165(7)	-0.113(6)	-0.067(6)	0.089(6)
F212	0.115(4)	0.075(3)	0.119(4)	-0.031(3)	-0.015(3)	-0.010(3)
F213	0.073(3)	0.124(4)	0.077(3)	0.039(3)	0.033(2)	-0.017(2)
F214	0.129(4)	0.075(3)	0.162(5)	-0.011(3)	0.082(4)	-0.056(3)
F215	0.046(2)	0.147(4)	0.119(4)	0.058(3)	0.017(2)	-0.033(3)
F216	0.0311(17)	0.058(2)	0.076(2)	-0.0040(17)	-0.0041(16)	0.0146(15)
F217	0.0530(19)	0.0430(18)	0.0582(19)	0.0119(15)	0.0215(16)	0.0180(15)
F218	0.064(2)	0.0388(18)	0.057(2)	-0.0097(15)	0.0144(16)	0.0068(15)
F219	0.158(5)	0.073(3)	0.078(3)	-0.009(2)	-0.034(3)	-0.022(3)
F220	0.072(3)	0.156(4)	0.044(2)	0.023(2)	0.0101(19)	0.050(3)
F221	0.071(3)	0.132(4)	0.048(2)	0.033(2)	0.0213(19)	-0.007(2)
F222	0.041(2)	0.152(5)	0.171(5)	0.067(4)	0.025(3)	-0.007(3)
F223	0.052(3)	0.163(6)	0.155(5)	-0.034(4)	0.026(3)	-0.022(3)
F224	0.038(2)	0.069(3)	0.328(9)	0.008(4)	0.040(4)	0.013(2)
C21	0.035(3)	0.038(3)	0.036(3)	0.005(2)	0.010(2)	0.021(2)
C22	0.034(3)	0.051(4)	0.047(3)	-0.004(3)	-0.001(2)	0.015(3)
C23	0.042(4)	0.088(5)	0.052(4)	-0.015(4)	-0.010(3)	0.026(3)
C24	0.062(5)	0.096(6)	0.062(5)	-0.027(4)	-0.011(4)	0.035(5)
C25	0.061(4)	0.104(6)	0.034(3)	0.010(4)	0.008(3)	0.055(4)
C26	0.049(3)	0.066(4)	0.044(3)	0.009(3)	0.018(3)	0.025(3)
C27	0.052(5)	0.080(6)	0.169(11)	-0.058(7)	-0.046(6)	-0.004(4)
C28	0.109(8)	0.201(12)	0.043(4)	0.028(6)	0.029(5)	0.086(8)
C29	0.053(3)	0.032(3)	0.032(3)	0.006(2)	0.015(2)	0.008(2)
C210	0.057(4)	0.040(3)	0.040(3)	0.012(2)	0.016(3)	0.018(3)
C211	0.090(5)	0.037(3)	0.044(3)	0.010(3)	0.018(3)	0.026(3)
C212	0.109(6)	0.033(3)	0.058(4)	0.013(3)	0.021(4)	0.026(4)
C213	0.098(5)	0.033(3)	0.057(4)	-0.004(3)	0.006(4)	0.003(3)
C214	0.072(4)	0.036(3)	0.050(3)	0.012(3)	0.014(3)	0.016(3)
C215	0.096(6)	0.051(4)	0.069(5)	0.019(4)	0.009(4)	0.040(4)
C216	0.163(10)	0.034(4)	0.123(8)	-0.015(5)	-0.038(8)	0.013(5)
C217	0.037(3)	0.027(2)	0.028(2)	0.0008(19)	0.011(2)	0.000(2)
C218	0.046(3)	0.034(3)	0.038(3)	0.007(2)	0.010(2)	-0.002(2)

C219	0.039(3)	0.053(3)	0.037(3)	0.004(2)	0.011(2)	-0.013(3)
C220	0.028(3)	0.057(4)	0.043(3)	-0.005(3)	0.009(2)	-0.005(2)
C221	0.028(2)	0.033(3)	0.033(3)	-0.003(2)	0.006(2)	0.001(2)
C222	0.029(2)	0.029(2)	0.028(2)	-0.0041(19)	0.006(2)	-0.004(2)
C223	0.053(4)	0.068(5)	0.076(5)	0.003(4)	0.023(4)	-0.019(3)
C224	0.033(3)	0.041(3)	0.042(3)	-0.007(2)	0.010(2)	0.002(2)
C225	0.033(3)	0.020(2)	0.040(3)	0.002(2)	0.013(2)	0.0073(19)
C226	0.030(3)	0.027(2)	0.040(3)	-0.001(2)	0.012(2)	0.005(2)
C227	0.043(3)	0.031(3)	0.036(3)	0.000(2)	0.014(2)	0.003(2)
C228	0.050(3)	0.037(3)	0.052(3)	0.005(2)	0.031(3)	0.009(2)
C229	0.041(3)	0.043(3)	0.056(3)	0.007(3)	0.024(3)	0.014(2)
C230	0.037(3)	0.034(3)	0.044(3)	0.006(2)	0.013(2)	0.014(2)
C231	0.050(3)	0.054(4)	0.040(3)	0.003(3)	0.019(3)	0.003(3)
C232	0.037(3)	0.083(5)	0.072(4)	0.015(4)	0.025(3)	0.014(3)
B2	0.036(3)	0.027(3)	0.034(3)	0.003(2)	0.011(2)	0.005(2)

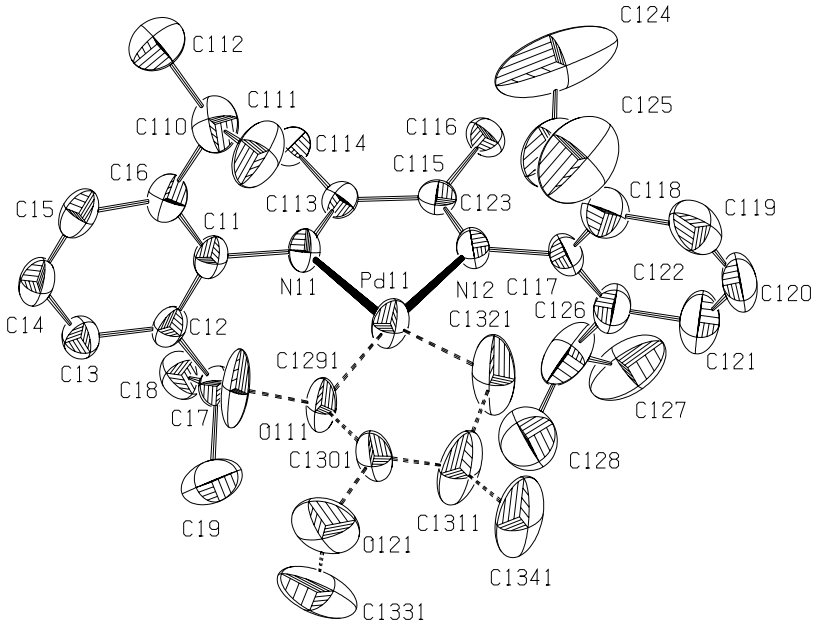
Thermal vibration amplitudes ($Å^2$)

$$F(\mathbf{h}) = F_o(\mathbf{h}) \exp\left(-2\pi^2 \sum_{i=1}^{3}\sum_{j=1}^{3} h_i h_l a_i^* a_j^* U_{ij}\right)$$

or

$$F(\mathbf{h}) = F_o(\mathbf{h}) \exp\left(-8\pi^2 U_{iso} (\sin(\theta)/\lambda)^2\right)$$

Cation of **2**:



Anion of **2**:

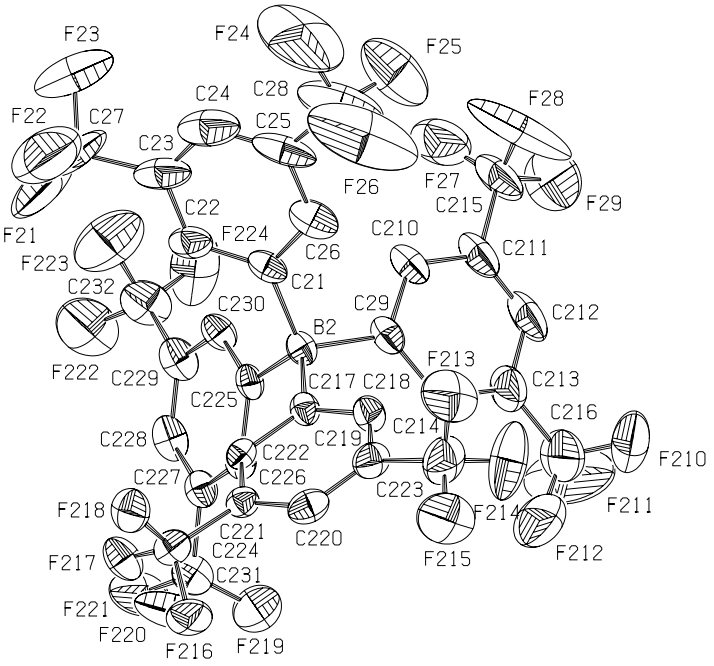


Table 3. Data on the geometry.

Standard deviations in the last decimal place are given in parentheses.

Residue: 1.

Interatomic Distances (Å)

Pd11	-O112	2.055(14)	C110	-C16	1.511(8)
Pd11	-N11	2.104(4)	C110	-C111	1.520(8)
Pd11	-N12	2.074(4)	C110	-C112	1.542(9)
Pd11	-C1322	2.10(3)	C113	-C114	1.491(7)
Pd11	-O111	2.052(14)	C113	-C115	1.495(7)
Pd11	-C1321	2.01(3)	C115	-C116	1.487(7)
O112	-C1292	1.38(3)	C117	-C118	1.383(8)
O112	-C1302	1.40(3)	C117	-C122	1.408(9)
O122	-C1302	1.34(4)	C118	-C119	1.415(12)
O122	-C1332	1.37(6)	C118	-C123	1.515(13)
N11	-C11	1.445(6)	C119	-C120	1.371(13)
N11	-C113	1.285(6)	C120	-C121	1.350(11)
N12	-C115	1.280(7)	C121	-C122	1.384(10)
N12	-C117	1.441(7)	C122	-C126	1.511(9)
C1302	-C1312	1.51(4)	C123	-C124	1.474(19)
C1312	-C1322	1.48(4)	C123	-C125	1.545(13)
C1312	-C1342	1.56(5)	C126	-C127	1.528(13)
C11	-C12	1.400(8)	C126	-C128	1.512(12)
C11	-C16	1.406(8)	O111	-C1291	1.43(2)
C12	-C13	1.398(8)	O111	-C1301	1.41(2)
C12	-C17	1.510(8)	O121	-C1301	1.381(16)
C13	-C14	1.381(9)	O121	-C1331	1.40(2)
C14	-C15	1.366(8)	C1301	-C1311	1.52(3)
C15	-C16	1.394(8)	C1311	-C1321	1.56(3)
C17	-C18	1.524(8)	C1311	-C1341	1.55(3)
C17	-C19	1.523(9)			

Bond Angles (deg.)

O112	-Pd11	-N11	173.9(4)	C12	-C17	-C19	112.6(5)
O112	-Pd11	-N12	108.1(5)	C18	-C17	-C19	110.0(5)
O112	-Pd11	-C1322	80.2(10)	C16	-C110	-C111	111.6(5)
N11	-Pd11	-N12	76.66(15)	C16	-C110	-C112	113.0(5)
N11	-Pd11	-C1322	94.9(9)	C111	-C110	-C112	110.6(5)
N11	-Pd11	-O111	109.8(4)	N11	-C113	-C114	124.4(4)
N11	-Pd11	-C1321	166.7(5)	N11	-C113	-C115	115.3(4)
N12	-Pd11	-C1322	171.4(9)	C114	-C113	-C115	120.3(4)
N12	-Pd11	-O111	173.5(4)	N12	-C115	-C113	115.3(4)
N12	-Pd11	-C1321	90.2(5)	N12	-C115	-C116	124.7(5)
O111	-Pd11	-C1321	83.3(6)	C113	-C115	-C116	120.0(5)

Pd11	-O112	-C1292	131.9(14)	N12	-C117	-C118	118.0(6)
Pd11	-O112	-C1302	112.6(14)	N12	-C117	-C122	118.7(5)
C1292	-O112	-C1302	110.0(17)	C118	-C117	-C122	123.1(6)
C1302	-O122	-C1332	130(4)	C117	-C118	-C119	116.1(7)
Pd11	-N11	-C11	122.8(3)	C117	-C118	-C123	121.2(6)
Pd11	-N11	-C113	115.7(3)	C119	-C118	-C123	122.7(6)
C11	-N11	-C113	121.5(4)	C118	-C119	-C120	120.9(7)
Pd11	-N12	-C115	116.9(3)	C119	-C120	-C121	121.3(8)
Pd11	-N12	-C117	121.0(3)	C120	-C121	-C122	121.1(8)
C115	-N12	-C117	122.1(4)	C117	-C122	-C121	117.3(6)
O112	-C1302	-O122	107(2)	C117	-C122	-C126	122.1(6)
O112	-C1302	-C1312	112(2)	C121	-C122	-C126	120.5(6)
O122	-C1302	-C1312	98(2)	C118	-C123	-C124	111.8(10)
C1302	-C1312	-C1322	105(2)	C118	-C123	-C125	114.3(8)
C1302	-C1312	-C1342	83(3)	C124	-C123	-C125	110.6(9)
C1322	-C1312	-C1342	120(3)	C122	-C126	-C127	113.4(7)
Pd11	-C1322	-C1312	108.9(18)	C122	-C126	-C128	109.9(6)
N11	-C11	-C12	117.6(4)	C127	-C126	-C128	111.5(7)
N11	-C11	-C16	118.4(5)	Pd11	-O111	-C1291	124.9(12)
C12	-C11	-C16	123.7(5)	Pd11	-O111	-C1301	113.6(10)
C11	-C12	-C13	116.6(5)	C1291	-O111	-C1301	115.6(12)
C11	-C12	-C17	122.6(5)	C1301	-O121	-C1331	113.1(13)
C13	-C12	-C17	120.8(5)	O111	-C1301	-O121	108.5(12)
C12	-C13	-C14	121.0(5)	O111	-C1301	-C1311	106.1(13)
C13	-C14	-C15	120.8(5)	O121	-C1301	-C1311	122.5(16)
C14	-C15	-C16	121.8(6)	C1301	-C1311	-C1321	107(2)
C11	-C16	-C15	116.2(5)	C1301	-C1311	-C1341	104.9(15)
C11	-C16	-C110	121.4(5)	C1321	-C1311	-C1341	113.7(19)
C15	-C16	-C110	122.4(5)	Pd11	-C1321	-C1311	102.2(15)
C12	-C17	-C18	110.4(5)				

Residue: 2.

Interatomic Distances (Å)

F21	-C27	1.123(15)	C24	-C25	1.364(12)
F22	-C27	1.475(12)	C25	-C26	1.418(8)
F23	-C27	1.375(13)	C25	-C28	1.511(12)
F24	-C28	1.387(12)	C29	-B2	1.644(7)
F25	-C28	1.278(15)	C210	-C29	1.393(8)
F26	-C28	1.198(12)	C210	-C211	1.393(8)
F27	-C215	1.314(11)	C211	-C212	1.355(9)
F28	-C215	1.297(10)	C211	-C215	1.493(12)
F29	-C215	1.302(10)	C212	-C213	1.391(10)
F210	-C216	1.238(13)	C213	-C214	1.404(8)
F211	-C216	1.394(14)	C213	-C216	1.486(13)
F212	-C216	1.283(13)	C214	-C29	1.390(8)
F213	-C223	1.315(8)	C217	-C218	1.397(7)
F214	-C223	1.327(10)	C217	-C222	1.397(6)
F215	-C223	1.319(8)	C217	-B2	1.643(7)
F216	-C224	1.335(6)	C218	-C219	1.378(8)

F217	-C224	1.336(6)	C219	-C220	1.380(8)
F218	-C224	1.351(6)	C219	-C223	1.510(9)
F219	-C231	1.309(8)	C220	-C221	1.387(8)
F220	-C231	1.318(8)	C221	-C222	1.379(7)
F221	-C231	1.318(7)	C221	-C224	1.490(7)
F222	-C232	1.273(9)	C225	-C226	1.409(8)
F223	-C232	1.393(9)	C225	-C230	1.397(7)
F224	-C232	1.246(9)	C225	-B2	1.637(8)
C21	-C22	1.395(8)	C226	-C227	1.392(8)
C21	-C26	1.391(8)	C227	-C228	1.380(7)
C21	-B2	1.639(8)	C227	-C231	1.494(8)
C22	-C23	1.406(9)	C228	-C229	1.387(8)
C23	-C24	1.347(11)	C229	-C230	1.398(8)
C23	-C27	1.523(12)	C229	-C232	1.491(9)

Bond Angles (deg.)

C22	-C21	-C26	115.0(5)	C218	-C217	-B2	122.1(4)
C22	-C21	-B2	123.9(5)	C222	-C217	-B2	122.8(4)
C26	-C21	-B2	120.9(5)	C217	-C218	-C219	122.9(5)
C21	-C22	-C23	122.5(6)	C218	-C219	-C220	120.6(5)
C22	-C23	-C24	121.0(7)	C218	-C219	-C223	118.1(5)
C22	-C23	-C27	116.4(7)	C220	-C219	-C223	121.2(5)
C24	-C23	-C27	122.5(8)	C219	-C220	-C221	118.2(5)
C23	-C24	-C25	118.7(7)	C220	-C221	-C222	120.4(5)
C24	-C25	-C26	120.9(6)	C220	-C221	-C224	119.0(5)
C24	-C25	-C28	122.9(7)	C222	-C221	-C224	120.5(5)
C26	-C25	-C28	116.2(7)	C217	-C222	-C221	122.9(5)
C21	-C26	-C25	121.7(6)	F213	-C223	-F214	104.0(6)
F21	-C27	-F22	114.1(9)	F213	-C223	-F215	106.7(6)
F21	-C27	-F23	115.6(10)	F213	-C223	-C219	111.3(6)
F21	-C27	-C23	121.9(11)	F214	-C223	-F215	109.2(6)
F22	-C27	-F23	93.8(8)	F214	-C223	-C219	112.8(6)
F22	-C27	-C23	99.8(7)	F215	-C223	-C219	112.4(6)
F23	-C27	-C23	107.0(8)	F216	-C224	-F217	107.3(4)
F24	-C28	-F25	99.6(8)	F216	-C224	-F218	105.9(4)
F24	-C28	-F26	106.8(9)	F216	-C224	-C221	113.2(4)
F24	-C28	-C25	106.4(9)	F217	-C224	-F218	105.1(4)
F25	-C28	-F26	113.8(12)	F217	-C224	-C221	113.3(4)
F25	-C28	-C25	113.3(8)	F218	-C224	-C221	111.5(4)
F26	-C28	-C25	115.1(9)	C226	-C225	-C230	115.8(5)
C210	-C29	-C214	116.5(5)	C226	-C225	-B2	122.7(5)
C210	-C29	-B2	122.0(5)	C230	-C225	-B2	121.2(5)
C214	-C29	-B2	121.1(5)	C225	-C226	-C227	121.6(5)
C29	-C210	-C211	122.4(5)	C226	-C227	-C228	121.1(5)
C210	-C211	-C212	119.8(6)	C226	-C227	-C231	118.5(5)
C210	-C211	-C215	119.6(6)	C228	-C227	-C231	120.4(5)
C212	-C211	-C215	120.6(6)	C227	-C228	-C229	118.9(5)
C211	-C212	-C213	120.5(6)	C228	-C229	-C230	119.9(5)
C212	-C213	-C214	119.1(6)	C228	-C229	-C232	120.1(5)
C212	-C213	-C216	121.3(6)	C230	-C229	-C232	120.0(5)

C214	-C213	-C216	119.6(7)	C225	-C230	-C229	122.6(5)
C29	-C214	-C213	121.8(6)	F219	-C231	-F220	105.4(6)
F27	-C215	-F28	103.6(7)	F219	-C231	-F221	107.0(5)
F27	-C215	-F29	104.0(8)	F219	-C231	-C227	112.9(5)
F27	-C215	-C211	114.0(7)	F220	-C231	-F221	104.8(5)
F28	-C215	-F29	109.7(8)	F220	-C231	-C227	112.9(5)
F28	-C215	-C211	110.9(8)	F221	-C231	-C227	113.1(5)
F29	-C215	-C211	113.9(7)	F222	-C232	-F223	96.9(6)
F210	-C216	-F211	102.8(9)	F222	-C232	-F224	114.8(7)
F210	-C216	-F212	111.0(11)	F222	-C232	-C229	115.3(6)
F210	-C216	-C213	114.8(10)	F223	-C232	-F224	99.8(7)
F211	-C216	-F212	99.9(8)	F223	-C232	-C229	111.9(6)
F211	-C216	-C213	108.8(10)	F224	-C232	-C229	115.3(7)
F212	-C216	-C213	117.2(7)	C21	-B2	-C29	110.9(4)
C218	-C217	-C222	114.9(5)	C21	-B2	-C217	105.8(4)
C21	-B2	-C225	113.0(4)	C29	-B2	-C225	103.0(4)
C29	-B2	-C217	111.9(4)	C217	-B2	-C225	112.5(4)

*Part III: Crystal structure determination of **3***

Abstract. $[C_{32}H_{47}N_2OPd]^+.[C_{32}H_{12}BF_{24}]^-$, M_r = 1445.37, monoclinic, *C*2/*c*, *a* = 21.9494(9), *b* = 12.8691(5), *c* = 25.207(1) Å, β = 115.522(1)°, *V* = 6425.4(4) Å^3, *Z* = 4, D_x = 1.494 gcm^{-3}, *F*(000) = 2928, μ = 4.01 cm^{-1}, λ(MoK$\bar{\alpha}$) = 0.71073 Å, T = 100(1) K, 29109 reflections measured, *GooF* = 1.036, *wR*(F^2) = 0.1874 for 7900 unique reflections and 597 parameters, 55 restraints and *R*(*F*) = 0.0675 for 6294 reflections obeying $F_o \geq 4.0\ \sigma(F_o)$ criterion of observability.

The asymmetric unit consists of two half moieties: a cationic Pd-complex and a fluorinated-tetraphenylborate anion. Both moieties have are a crystallographic imposed symmetry element: the Pd-complex has an inversion center, implying disorder, and the borate anion is located on a two fold axis.

Experimental

X-ray diffraction: Crystal and Molecular Structure.

A crystal with the dimensions of 0.34 x 0.33 x 0.16 mm was mounted on top of a glass fiber, by using inert-atmosphere handling techniques, and aligned on a *Bruker*[1] *SMART APEX CCD* diffractometer (Platform with full three-circle goniometer). The diffractometer was equipped with a 4K *CCD* detector set 60.0 mm from the crystal. The crystal was cooled to 100(1) K using the *Bruker KRYOFLEX* low-temperature device. Intensity measurements were performed using graphite monochromated Mo-K$\bar{\alpha}$ radiation from a sealed ceramic diffraction tube (*SIEMENS*). Generator settings were 50 KV/ 40 mA. *SMART* was used for preliminary determination of the unit cell constants and data collection control. The intensities of reflections of a hemisphere were collected by a combination of 3 sets of exposures (frames). Each set had a different ϕ angle for the crystal and each exposure covered a range of 0.3° in ω. A total of 1800 frames were collected with an exposure time of 10.0 seconds per frame. The overall data collection time was 8.0 h. Data integration and global cell refinement was performed with the program *SAINT*. The final unit cell was obtained from the *xyz* centroids of 7624 reflections after integration. Intensity data were corrected for Lorentz and polarization effects, scale variation, for decay and absorption: a multi-scan absorption correction was applied, based on the intensities of symmetry-related reflections measured at different angular settings (*SADABS*)[2], and reduced to F_o^2. The program suite *SHELXTL* was used for space group determination (*XPREP*).[1]

The unit cell[3] was identified as monoclinic; reduced cell calculations did not indicate any higher metric lattice symmetry.[4] The space group *C*2/*c*, was derived from the systematic extinctions. The $|E|$ distribution statistics were indicative of a centrosymmetric space group.[5] Examination of the final atomic coordinates of the structure did not yield extra crystallographic or metric symmetry elements.[5,6]

The structure was ultimately solved by Patterson methods, using the half scattering power of Pd, and extension of the model was accomplished by

direct methods applied to difference structure factors using the program *DIRDIF*[7] and several subsequent difference Fourier maps.

Refinement was complicated by a disorder problem: from the solution it was clear that the cation is located over an inversion center, implying disorder in which the parts of the cation (that has no inversion symmetry) are alternatively occupied. The anion is located over a twofold axis. The CF_3-groups all showed rotational disorder, so ultimately all the F-positions were split in two alternative positions.

Some atoms showed unrealistic displacement parameters when allowed to vary anisotropically, suggesting dynamic disorder (dynamic means that the smeared electron density is due to fluctuations of the atomic positions within each unit cell) as a consequence of the inversion-disorder. To improve the parameters chemical more reasonable, ultimately restrain instructions (*DFIX*) were applied in the refinement (the disorder is compensated by the large displacement parameters). The positional and anisotropic displacement parameters for the non-hydrogen atoms were refined. Although the disorder model was not satisfactory in all respects, it was the best of several models tested.

Hydrogen atoms were constrained to idealized geometries and allowed to ride on their carrier atoms with an isotropic displacement parameter related to the equivalent displacement parameter of their carrier atoms.

Final refinement on F^2 carried out by full-matrix least-squares techniques converged at $wR(F^2)$ = 0.1874 for 7900 reflections and $R(F)$ = 0.0675 for 6294 reflections with $F_o \geq 4.0\ \sigma(F_o)$ and 597 parameters and 55 restraints. The final difference Fourier map was essentially featureless: no significant peaks (0.87(8) e/Å^3) having chemical meaning above the general background were observed.

The positional and anisotropic displacement parameters for the non-hydrogen atoms and isotropic displacement parameters for hydrogen atoms were refined on F^2 with full-matrix least-squares procedures minimizing the function $Q = \sum_h [w(|(F_o^2) - k(F_c^2)|)^2]$, where $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$, $P = [\max(F_o^2,0) + 2F_c^2] / 3$, F_0 and F_c are the observed and calculated structure factor amplitudes, respectively; ultimately the suggested a (=0.0838) and b (= 17.3084) were used in the final refinement.

Crystal data and numerical details on data collection and refinement are given in Table 1. Final fractional atomic coordinates, equivalent displacement parameters and anisotropic displacement parameters for the non-hydrogen atoms are given in Table 2. Molecular geometry data are collected in Table 3. Neutral atom scattering factors and anomalous dispersion corrections were taken from *International Tables for Crystallography.*[10]

All refinement calculations and graphics were performed on a Pentium-III / Debian-Linux computer at the University of Groningen with the program packages *SHELXL*[11] (least-square refinements), a locally modified version of the program *PLUTO*[12] (preparation of illustrations) and *PLATON*[9] package (checking the final results for missed symmetry with the *MISSYM* option,

solvent accessible voids with the *SOLV* option, calculation of geometric data and the *ORTEP*[9] illustrations).

Each asymmetric unit contains one half formula unit, consisting of two moieties: a cation Pd-complex and fluorinated-tetraphenylborate anion; the B2 atom is located on a two fold axis (site symmetry .2.). The monoclinic unit cell contains four discrete (disordered) units, four cations and four anions molecules separated by normal van der Waals distances[13].

No classic hydrogen bonds, no missed symmetry (*MISSYM*), but potential solvent-accessible area (voids of 37.7 $Å^3$ / unit cell) were detected by procedures implemented in *PLATON.*[14,15]

*) For disordered atoms, in the refinement described by several fractional s.o.f. atoms, only one of the fractional (major) s.o.f. atoms is included in the plots.

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Table 1.

a. Crystal data and details of the structure determination.

Moiety_Formula	$[C_{32}H_{47}N_2OPd]^+.[C_{32}H_{12}BF_{24}]^-$
Formula_Weight, g.mol^{-1}	1445.37
Crystal system	Monoclinic
Space group, no.[16]	*C*2/*c*, 15
a, Å	21.9494(9)
b, Å	12.8691(5)
c, Å	25.207(1)
β, deg	115.522(1)
V, Å^3	6425.4(4)
Θ range unit cell: min.-max., deg; reflections	2.27 - 28.02 ; 7624
Formula_*Z*	4
SpaceGroup_*Z*	8
Z' (= Formula_Z / SpaceGroup_*Z)*	0.5
ρ_{calc}, g.cm^{-3}	1.494
F(000), electrons	2928
μ(Mo K$\bar{\alpha}$), cm^{-1}	4.01
Color, habit	red, block
Approx. crystal dimension, mm	0.34 x 0.33 x 0.16

b. Data collection.

λ(Mo K$\bar{\alpha}$), Å	0.71073
Monochromator	Graphite
Measurement device type	*CCD* area-detector diffractometer
Detector Area resolution (pixels / mm)	4096 x 4096 / 62 x 62 (binned 512)
Temperature, K	100(1)
Measurement method	φ- and ω-scans
θ range; min. max., deg	2.28, 28.28
Index ranges	h: -28→29; k: -17→17; l: -33→30
Min.- Max. absorption transmission factor	0.8912 – 0.9386
X-ray exposure time, h	8.0
Total data	29109
Unique data	7900
Data with criterion: ($F_o \geq 4.0\ \sigma\ (F_o)$)	6294
$R_{int} = \sum [\lvert F_o^2 - F_o^2\ (\text{mean})\rvert] / \sum [F_o^2]$	0.0255
$R_{sig} = \sum \sigma(F_o^2) / \sum [F_o^2]$	0.0454

c. Refinement.

Number of reflections	7900
Number of refined parameters	597
Number of restraints	55
Final agreement factors:	
$wR(F^2) = [\sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)^2]]^{1/2}$	0.1874
Weighting scheme: *a, b*	0.0838, 17.3084
$w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$	
And $P = [\max(F_o^2,0) + 2F_c^2] / 3$	
$R(F) = \sum (\|\|F_o\| - \|F_c\|\|) / \sum \|F_o\|$	0.0675
For $F_o > 4.0\ \sigma\ (F_o)$	
$GooF = S = [\sum [w(F_o^2 - F_c^2)^2] / (n-p)]^{1/2}$	1.036
n = number of reflections	
p = number of parameters refined	
Residual electron density in final	
Difference Fourier map, e/Å^3	-0.73, 0.87(8)
Max. (shift/σ) final cycle	0.857
Average (shift/σ) final cycle	0.013

Table 2. Final fractional atomic coordinates and equivalent isotropic displacement parameters with s.u.'s in parentheses.

Atoms of the Asymmetric Unit.

Non-Hydrogen parameters

Residue: 1.

Atom	*x*	*y*	*z*	*s.o.f.*	U_{eq} (Å^2)
Pd112	0.19815(3)	0.78428(5)	-0.04838(2)	0.5(-)	0.03211(16)
O112	0.0501(6)	0.7395(16)	-0.0876(7)	0.5(-)	0.224(11)
N111	0.3052(3)	0.7893(4)	-0.0071(2)	0.5(-)	0.0320(17)
N121	0.2248(2)	0.6872(4)	0.0267(2)	0.5(-)	0.0311(17)
C1131	0.3338(3)	0.7367(4)	0.0405(2)	0.5(-)	0.0324(17)
C1141	0.4093(3)	0.7347(10)	0.0779(6)	0.5(-)	0.048(4)
C1151	0.2878(3)	0.6746(5)	0.0598(3)	0.5(-)	0.044(2)
C1161	0.3250(5)	0.6113(8)	0.1150(3)	0.5(-)	0.044(3)
C1172	0.1721(11)	0.8885(15)	-0.1288(10)	0.5(-)	0.178(9)
C1182	0.1145(5)	0.8206(18)	-0.1288(6)	0.5(-)	0.156(7)
C1192	0.0904(6)	0.7966(12)	-0.0926(6)	0.5(-)	0.057(4)
C1202	0.0149(7)	0.7218(11)	-0.0617(7)	0.5(-)	0.111(6)
C11	0.3338(2)	0.8613(3)	-0.03420(16)	1.0(-)	0.0542(14)
C12	0.34396(18)	0.8271(3)	-0.08278(15)	1.0(-)	0.0477(11)
C13	0.38598(19)	0.8881(3)	-0.09809(15)	1.0(-)	0.0550(13)
C14	0.4140(2)	0.9779(4)	-0.06852(17)	1.0(-)	0.0627(14)
C15	0.4018(2)	1.0095(3)	-0.02166(16)	1.0(-)	0.0570(11)
C16	0.36196(19)	0.9521(3)	-0.00311(14)	1.0(-)	0.0466(10)
C17	0.3125(2)	0.7270(3)	-0.11518(18)	1.0(-)	0.0597(14)
C18	0.3484(3)	0.6322(4)	-0.0788(3)	1.0(-)	0.095(2)
C19	0.3114(3)	0.7195(5)	-0.1756(2)	1.0(-)	0.088(2)
C110	0.3477(2)	0.9877(3)	0.04785(16)	1.0(-)	0.0567(13)
C111	0.2892(3)	1.0641(5)	0.0265(3)	1.0(-)	0.106(2)
C112	0.4095(3)	1.0330(4)	0.09833(19)	1.0(-)	0.0834(18)

Residue: 2.

F2104	0.5820(6)	0.4357(8)	0.5078(5)	0.516(7)	0.113(4)
F2114	0.6214(6)	0.3103(11)	0.4814(3)	0.516(7)	0.125(5)
F2124	0.5282(6)	0.2982(12)	0.4845(5)	0.516(7)	0.177(7)
F214	0.4243(4)	-0.0564(9)	0.3050(3)	0.516(7)	0.084(3)
F224	0.3738(9)	-0.1104(9)	0.2185(5)	0.516(7)	0.220(11)
F234	0.3184(5)	-0.0710(14)	0.2656(7)	0.516(7)	0.258(10)
F244	0.1979(3)	0.1760(6)	0.1113(2)	0.516(7)	0.110(3)
F254	0.2179(3)	0.3023(6)	0.1666(2)	0.516(7)	0.068(2)
F264	0.2474(2)	0.2957(5)	0.0936(2)	0.516(7)	0.069(2)
F274	0.3474(3)	0.5937(8)	0.2962(4)	0.516(7)	0.080(3)
F284	0.4226(8)	0.6624(9)	0.2769(5)	0.516(7)	0.100(5)
F294	0.4289(3)	0.6761(4)	0.3629(3)	0.516(7)	0.0512(14)
C21	0.43126(14)	0.2211(2)	0.22971(12)	1.0(-)	0.0335(8)
C22	0.42953(18)	0.1227(3)	0.25189(13)	1.0(-)	0.0437(10)

C23	0.3693(2)	0.0683(4)	0.23552(16)	1.0(-)	0.0662(15)
C24	0.3083(2)	0.1112(5)	0.19742(17)	1.0(-)	0.0799(19)
C25	0.30851(17)	0.2088(4)	0.17668(15)	1.0(-)	0.0664(15)
C26	0.36858(15)	0.2634(3)	0.19181(13)	1.0(-)	0.0442(9)
C27	0.3697(3)	-0.0401(4)	0.2564(2)	1.0(-)	0.108(3)
C28	0.24302(19)	0.2614(5)	0.13758(18)	1.0(-)	0.098(3)
C29	0.49958(13)	0.3633(2)	0.30307(12)	1.0(-)	0.0305(7)
C210	0.46088(15)	0.4542(2)	0.29072(13)	1.0(-)	0.0375(8)
C211	0.45496(18)	0.5131(3)	0.33438(16)	1.0(-)	0.0456(10)
C212	0.48829(18)	0.4847(3)	0.39275(15)	1.0(-)	0.0448(10)
C213	0.52730(16)	0.3954(2)	0.40640(13)	1.0(-)	0.0384(9)
C214	0.53246(15)	0.3361(2)	0.36235(12)	1.0(-)	0.0336(8)
C215	0.4123(2)	0.6095(4)	0.31720(18)	1.0(-)	0.0714(17)
C216	0.5659(2)	0.3629(3)	0.46893(14)	1.0(-)	0.0570(13)
B2	0.5(-)	0.2906(3)	0.25000(-)	1.0(-)	0.0288(11)
F2103	0.5496(6)	0.4158(7)	0.5040(4)	0.484(7)	0.091(4)
F2113	0.6308(3)	0.3849(11)	0.4844(4)	0.484(7)	0.108(4)
F2123	0.5622(6)	0.2670(4)	0.4783(3)	0.484(7)	0.087(4)
F213	0.4244(5)	-0.0776(11)	0.2987(5)	0.484(7)	0.157(6)
F223	0.3514(4)	-0.1061(9)	0.2125(4)	0.484(7)	0.084(3)
F233	0.3209(5)	-0.0481(9)	0.2754(4)	0.484(7)	0.116(5)
F243	0.2095(6)	0.2303(13)	0.0870(4)	0.484(7)	0.342(11)
F253	0.2020(6)	0.2800(8)	0.1618(6)	0.484(7)	0.180(8)
F263	0.2506(3)	0.3729(7)	0.1271(5)	0.484(7)	0.158(4)
F273	0.3488(4)	0.5788(9)	0.2772(5)	0.484(7)	0.121(5)
F283	0.4287(8)	0.6789(10)	0.2889(6)	0.484(7)	0.107(4)
F293	0.4007(7)	0.6515(11)	0.3587(4)	0.484(7)	0.208(8)

Hydrogen parameters:

Residue: 1.

H13	0.39557(-)	0.86701(-)	-0.12981(-)	1.0(-)	0.06568(-)
H14	0.44201(-)	1.01882(-)	-0.08036(-)	1.0(-)	0.07491(-)
H15	0.42135(-)	1.07233(-)	-0.00185(-)	1.0(-)	0.06798(-)
H17	0.26468(-)	0.72506(-)	-0.12055(-)	1.0(-)	0.07200(-)
H18	0.39630(-)	0.63432(-)	-0.07058(-)	1.0(-)	0.14121(-)
H18'	0.32785(-)	0.56882(-)	-0.10067(-)	1.0(-)	0.14121(-)
H18"	0.34422(-)	0.63270(-)	-0.04159(-)	1.0(-)	0.14121(-)
H19	0.35781(-)	0.71631(-)	-0.17165(-)	1.0(-)	0.13145(-)
H19'	0.28868(-)	0.78063(-)	-0.19891(-)	1.0(-)	0.13145(-)
H19"	0.28704(-)	0.65656(-)	-0.19542(-)	1.0(-)	0.13145(-)
H110	0.33336(-)	0.92528(-)	0.06325(-)	1.0(-)	0.06797(-)
H111	0.27797(-)	1.08198(-)	0.05906(-)	1.0(-)	0.15859(-)
H111'	0.24978(-)	1.03244(-)	-0.00538(-)	1.0(-)	0.15859(-)
H111"	0.30200(-)	1.12723(-)	0.01203(-)	1.0(-)	0.15859(-)
H112	0.42216(-)	1.09834(-)	0.08575(-)	1.0(-)	0.12501(-)
H112'	0.44702(-)	0.98364(-)	0.10991(-)	1.0(-)	0.12501(-)
H112"	0.39915(-)	1.04627(-)	0.13186(-)	1.0(-)	0.12501(-)
H1172	0.16935(-)	0.96420(-)	-0.12330(-)	0.5(-)	0.21348(-)
H1172'	0.19129(-)	0.86934(-)	-0.15652(-)	0.5(-)	0.21348(-)
H1182	0.09037(-)	0.78766(-)	-0.16571(-)	0.5(-)	0.18581(-)

H1192	0.11127(-)	0.83774(-)	-0.05818(-)	0.5(-)	0.06895(-)
H1202	0.04210(-)	0.68729(-)	-0.02425(-)	0.5(-)	0.16661(-)
H1202'	-0.02278(-)	0.67655(-)	-0.08603(-)	0.5(-)	0.16661(-)
H1202"	-0.00284(-)	0.78733(-)	-0.05434(-)	0.5(-)	0.16661(-)
H11411	0.41655(-)	0.75681(-)	0.11630(-)	0.5(-)	0.021(14)
H11421	0.41961(-)	0.65788(-)	0.07695(-)	0.5(-)	0.06(3)
H11431	0.43586(-)	0.77423(-)	0.06154(-)	0.5(-)	3(3)
H11611	0.35478(-)	0.56183(-)	0.10654(-)	0.5(-)	0.037(18)
H11621	0.35382(-)	0.65942(-)	0.14758(-)	0.5(-)	0.032(17)
H11631	0.29662(-)	0.57086(-)	0.12902(-)	0.5(-)	0.10(4)

Residue: 2.

H22	0.47051(-)	0.09181(-)	0.27890(-)	1.0(-)	0.05249(-)
H24	0.26738(-)	0.07361(-)	0.18601(-)	1.0(-)	0.09591(-)
H26	0.36703(-)	0.33094(-)	0.17607(-)	1.0(-)	0.05307(-)
H210	0.43783(-)	0.47637(-)	0.25102(-)	1.0(-)	0.04498(-)
H212	0.48455(-)	0.52520(-)	0.42271(-)	1.0(-)	0.05371(-)
H214	0.55929(-)	0.27491(-)	0.37302(-)	1.0(-)	0.04030(-)

*) $U_{eq} = 1/3 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a_i}.\mathbf{a_j}$ [17]

Anisotropic (displacement) parameters (Å^2)

Residue: 1.

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Pd112	0.0274(3)	0.0445(3)	0.0244(2)	-0.00098(19)	0.01113(18)	0.0038(2)
O112	0.084(8)	0.42(3)	0.172(13)	0.130(15)	0.059(8)	-0.015(11)
N111	0.027(3)	0.042(3)	0.029(3)	-0.005(2)	0.014(2)	0.003(2)
N121	0.033(3)	0.034(3)	0.029(3)	-0.006(2)	0.016(2)	-0.001(2)
C1131	0.026(3)	0.046(3)	0.033(3)	0.002(2)	0.020(3)	0.001(2)
C1141	0.022(4)	0.063(7)	0.060(7)	0.007(4)	0.019(4)	0.001(4)
C1151	0.045(4)	0.047(4)	0.049(4)	0.006(3)	0.029(3)	-0.003(3)
C1161	0.060(6)	0.054(5)	0.036(4)	0.030(3)	0.038(4)	0.017(4)
C1172	0.131(16)	0.064(10)	0.182(19)	0.027(11)	-0.081(13)	0.011(10)
C1182	0.038(5)	0.32(2)	0.095(9)	0.125(13)	0.015(5)	0.022(9)
C1192	0.039(5)	0.072(9)	0.058(7)	-0.008(6)	0.018(4)	-0.013(4)
C1202	0.095(9)	0.107(9)	0.154(13)	0.011(9)	0.075(9)	-0.011(7)
C11	0.079(3)	0.0537(19)	0.051(2)	-0.0149(16)	0.048(2)	-0.0222(18)
C12	0.0521(19)	0.062(2)	0.0384(16)	-0.0143(15)	0.0283(15)	-0.0162(16)
C13	0.054(2)	0.081(3)	0.0379(17)	-0.0122(17)	0.0274(16)	-0.0201(19)
C14	0.065(2)	0.083(3)	0.048(2)	-0.0097(19)	0.0318(19)	-0.034(2)
C15	0.067(2)	0.060(2)	0.0423(19)	-0.0106(16)	0.0220(17)	-0.0245(18)
C16	0.062(2)	0.0469(17)	0.0340(16)	-0.0058(13)	0.0237(15)	-0.0088(15)
C17	0.068(2)	0.073(3)	0.059(2)	-0.031(2)	0.047(2)	-0.027(2)
C18	0.151(5)	0.066(3)	0.090(4)	-0.035(3)	0.074(4)	-0.025(3)
C19	0.108(4)	0.114(4)	0.059(3)	-0.048(3)	0.053(3)	-0.052(3)
C110	0.089(3)	0.0474(18)	0.0428(19)	-0.0121(15)	0.037(2)	-0.0065(18)

C111	0.139(5)	0.107(4)	0.067(3)	-0.019(3)	0.040(3)	0.045(4)
C112	0.129(4)	0.081(3)	0.044(2)	-0.026(2)	0.041(3)	-0.031(3)

Residue: 2.

F2104	0.155(10)	0.082(5)	0.044(4)	-0.026(3)	-0.013(5)	0.022(6)
F2114	0.146(9)	0.183(10)	0.026(3)	0.013(5)	0.017(4)	0.108(9)
F2124	0.147(10)	0.256(17)	0.078(7)	0.087(9)	0.001(5)	-0.078(10)
F214	0.141(8)	0.051(4)	0.066(4)	0.000(3)	0.049(5)	-0.045(4)
F224	0.46(3)	0.071(7)	0.128(11)	-0.004(7)	0.125(14)	-0.073(12)
F234	0.208(15)	0.29(2)	0.192(14)	0.103(13)	0.006(10)	-0.186(15)
F244	0.021(2)	0.252(9)	0.041(3)	-0.056(4)	-0.002(2)	-0.028(4)
F254	0.043(3)	0.111(5)	0.035(2)	-0.033(3)	0.0016(19)	0.004(3)
F264	0.023(2)	0.105(5)	0.060(3)	0.015(3)	-0.001(2)	-0.004(2)
F274	0.049(3)	0.079(4)	0.109(6)	-0.032(4)	0.031(3)	0.016(3)
F284	0.188(12)	0.051(5)	0.112(7)	0.032(5)	0.113(8)	0.056(5)
F294	0.046(2)	0.036(2)	0.073(3)	-0.017(2)	0.027(2)	0.002(2)
C21	0.0322(13)	0.0472(15)	0.0236(12)	-0.0078(11)	0.0143(11)	-0.0074(12)
C22	0.0536(18)	0.0544(18)	0.0260(14)	-0.0087(13)	0.0198(13)	-0.0192(15)
C23	0.082(3)	0.087(3)	0.0358(18)	-0.0168(18)	0.0313(19)	-0.051(2)
C24	0.061(2)	0.148(5)	0.0379(19)	-0.025(2)	0.0281(19)	-0.063(3)
C25	0.0310(16)	0.142(4)	0.0287(16)	-0.015(2)	0.0151(14)	-0.020(2)
C26	0.0287(14)	0.075(2)	0.0301(14)	-0.0058(14)	0.0138(12)	-0.0023(14)
C27	0.164(6)	0.101(4)	0.059(3)	-0.017(3)	0.047(4)	-0.093(4)
C28	0.0296(19)	0.217(7)	0.044(2)	-0.002(3)	0.0116(17)	-0.009(3)
C29	0.0274(12)	0.0374(13)	0.0271(12)	-0.0022(10)	0.0121(10)	-0.0041(10)
C210	0.0352(14)	0.0454(16)	0.0335(14)	0.0019(12)	0.0163(12)	0.0047(12)
C211	0.0510(18)	0.0464(17)	0.0482(18)	0.0008(14)	0.0298(16)	0.0104(14)
C212	0.0563(19)	0.0461(17)	0.0409(17)	-0.0090(13)	0.0295(15)	-0.0035(15)
C213	0.0468(16)	0.0420(15)	0.0289(14)	-0.0066(12)	0.0187(13)	-0.0071(13)
C214	0.0367(14)	0.0359(14)	0.0267(13)	-0.0029(10)	0.0122(11)	-0.0042(11)
C215	0.079(3)	0.079(3)	0.063(3)	0.003(2)	0.037(2)	0.040(3)
C216	0.079(3)	0.058(2)	0.0296(16)	-0.0085(15)	0.0193(17)	0.002(2)
B2	0.0266(18)	0.034(2)	0.0249(19)	0.00000(-)	0.0103(16)	0.00000(-)
F2103	0.152(10)	0.102(7)	0.025(4)	-0.008(3)	0.043(6)	0.039(6)
F2113	0.058(4)	0.185(10)	0.052(4)	0.026(6)	-0.004(3)	-0.008(5)
F2123	0.180(11)	0.040(3)	0.029(3)	0.0020(19)	0.033(5)	0.008(4)
F213	0.202(14)	0.062(7)	0.109(9)	0.007(5)	-0.025(8)	-0.063(6)
F223	0.101(5)	0.080(6)	0.070(5)	-0.041(4)	0.036(4)	-0.057(4)
F233	0.194(11)	0.110(6)	0.102(6)	-0.051(5)	0.119(8)	-0.108(7)
F243	0.194(14)	0.57(3)	0.098(9)	-0.195(15)	-0.093(9)	0.263(19)
F253	0.118(8)	0.097(7)	0.38(2)	0.099(9)	0.159(12)	0.040(6)
F263	0.041(3)	0.113(7)	0.224(11)	0.097(8)	-0.033(4)	-0.020(4)
F273	0.105(7)	0.133(9)	0.131(10)	0.026(7)	0.056(6)	0.091(6)
F283	0.131(9)	0.047(4)	0.126(9)	0.007(5)	0.040(7)	0.046(5)
F293	0.271(17)	0.273(17)	0.105(8)	0.027(9)	0.106(10)	0.213(14)

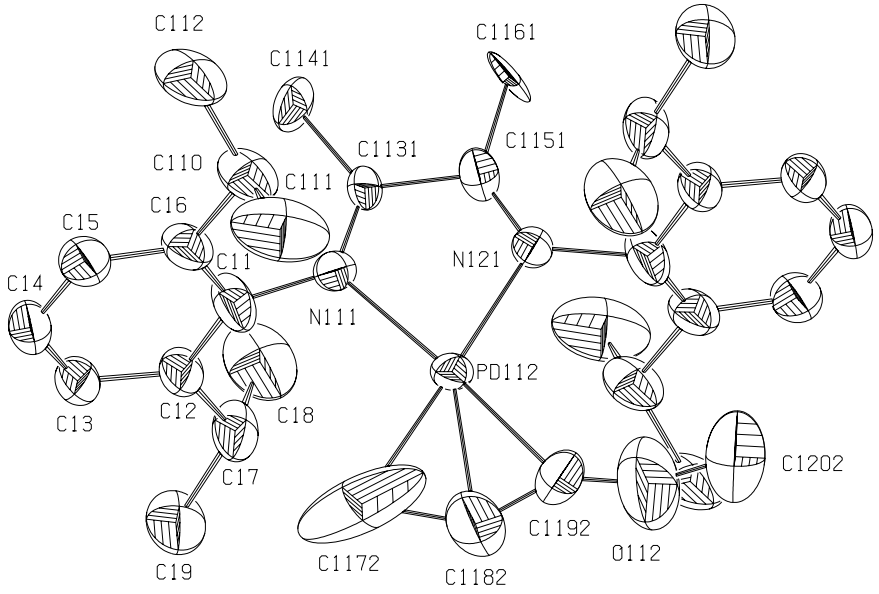
Thermal vibration amplitudes ($Å^2$)

$$F(\mathbf{h}) = F_o(\mathbf{h}) \exp\left(-2\pi^2 \sum_{i=1}^{3} \sum_{j=1}^{3} h_i h_j a_i^* a_j^* U_{ij}\right)$$

or

$$F(\mathbf{h}) = F_o(\mathbf{h}) \exp\left(-8\pi^2 U_{iso} (\sin(\theta)/\lambda)^2\right)$$

Cation of **3**:



Anion of **3**:

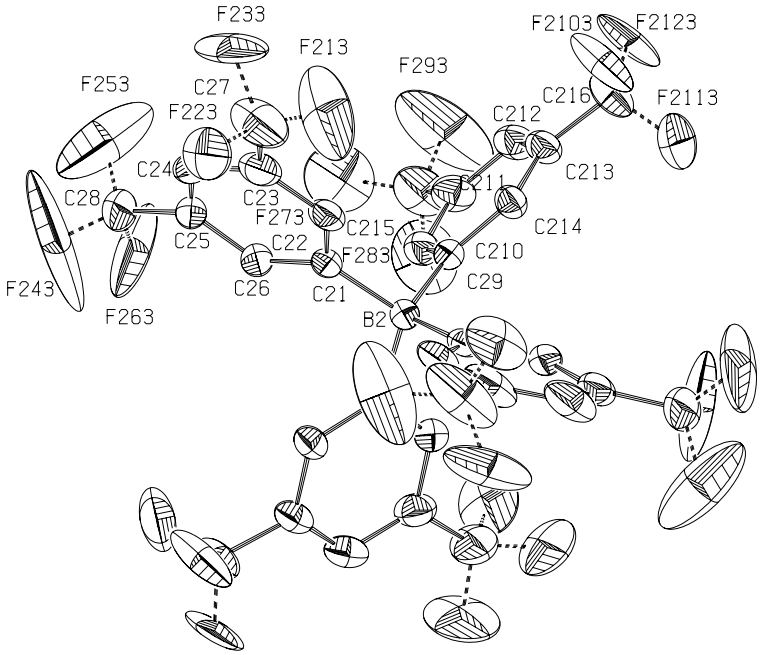


Table 3. Data on the geometry.

Standard deviations in the last decimal place are given in parentheses.

Residue: 1.

Interatomic Distances (Å)

Pd112	-N121	2.129(5)	C1182	-C1192	1.27(2)
Pd112	-C1172	2.29(2)	C11	-C12	1.407(5)
Pd112	-C1182	2.119(14)	C11	-C16	1.394(5)
Pd112	-C1192	2.142(14)	C12	-C13	1.387(6)
O112	-C1192	1.20(2)	C12	-C17	1.522(5)
O112	-C1202	1.23(2)	C13	-C14	1.369(6)
N111	-C1131	1.282(7)	C14	-C15	1.380(6)
N111	-C11	1.445(7)	C15	-C16	1.372(6)
N121	-C1151	1.281(9)	C17	-C18	1.526(7)
N121	-C11_b	1.513(7)	C17	-C19	1.516(7)
C1131	-C1141	1.513(13)	C110	-C16	1.518(5)
C1131	-C1151	1.523(9)	C110	-C111	1.520(8)
C1151	-C1161	1.512(11)	C110	-C112	1.520(7)
C1172	-C1182	1.54(3)			

Bond Angles (deg.)

N121	-Pd112	-C1172	178.6(7)	O112	-C1192	-C1182	141.0(17)
N121	-Pd112	-C1182	140.6(5)	N111	-C11	-C12	118.4(4)
N121	-Pd112	-C1192	109.0(4)	N111	-C11	-C16	116.7(4)
C1172	-Pd112	-C1182	40.6(8)	N111	-C11	-N121_b	27.2(3)
C1172	-Pd112	-C1192	72.4(8)	C12	-C11	-C16	123.6(4)
C1182	-Pd112	-C1192	34.8(5)	C12	-C11	-N121_b	116.2(4)
C1192	-O112	-C1202	146.3(19)	C16	-C11	-N121_b	118.1(4)
C1131	-N111	-C11	130.1(6)	C11	-C12	-C13	116.1(3)
Pd112	-N121	-C1151	117.4(4)	C11	-C12	-C17	121.8(4)
Pd112	-N121	-C11_b	115.5(3)	C13	-C12	-C17	122.2(3)
C1151	-N121	-C11_b	127.1(5)	C12	-C13	-C14	121.5(4)
N111	-C1131	-C1141	123.7(7)	C13	-C14	-C15	120.5(4)
N111	-C1131	-C1151	116.9(6)	C14	-C15	-C16	121.4(4)
C1141	-C1131	-C1151	119.4(6)	C11	-C16	-C15	116.9(4)
N121	-C1151	-C1131	113.8(5)	C11	-C16	-C110	121.7(4)
N121	-C1151	-C1161	132.1(7)	C15	-C16	-C110	121.3(3)
C1131	-C1151	-C1161	114.0(7)	C12	-C17	-C18	110.9(4)
Pd112	-C1172	-C1182	63.8(9)	C12	-C17	-C19	113.6(4)
Pd112	-C1182	-C1172	75.6(10)	C18	-C17	-C19	109.5(4)
Pd112	-C1182	-C1192	73.6(9)	C16	-C110	-C111	110.3(4)
C1172	-C1182	-C1192	137.2(16)	C16	-C110	-C112	112.9(4)
Pd112	-C1192	-O112	126.9(13)	C111	-C110	-C112	111.2(4)
Pd112	-C1192	-C1182	71.6(9)				

Residue: 2.

Interatomic Distances (Å)

F2104	-C216	1.290(11)	C29	-B2	1.636(4)
F2114	-C216	1.308(14)	C210	-C29	1.400(4)
F2124	-C216	1.346(15)	C210	-C211	1.388(5)
F214	-C27	1.310(9)	C211	-C212	1.381(5)
F224	-C27	1.347(14)	C211	-C215	1.501(6)
F234	-C27	1.306(15)	C212	-C213	1.385(5)
F244	-C28	1.436(9)	C213	-C214	1.392(4)
F254	-C28	1.210(8)	C213	-C216	1.492(4)
F264	-C28	1.235(7)	C214	-C29	1.395(4)
F274	-C215	1.304(9)	F2103	-C216	1.283(12)
F284	-C215	1.321(14)	F2113	-C216	1.335(10)
F294	-C215	1.354(8)	F2123	-C216	1.266(7)
C21	-C22	1.391(5)	F213	-C27	1.307(13)
C21	-C26	1.403(5)	F223	-C27	1.313(11)
C21	-B2	1.635(4)	F233	-C27	1.353(13)
C22	-C23	1.392(6)	F243	-C28	1.232(11)
C23	-C24	1.383(6)	F253	-C28	1.309(15)
C23	-C27	1.490(7)	F263	-C28	1.481(11)
C24	-C25	1.361(8)	F273	-C215	1.380(12)
C25	-C26	1.394(6)	F283	-C215	1.287(16)
C25	-C28	1.508(6)	F293	-C215	1.297(13)

Bond Angles (deg.)

C22	-C21	-C26	116.0(3)	C29	-C210	-C211	122.4(3)
C22	-C21	-B2	123.6(3)	C210	-C211	-C212	120.9(4)
C26	-C21	-B2	120.2(3)	C210	-C211	-C215	118.9(3)
C21	-C22	-C23	121.7(4)	C212	-C211	-C215	120.3(4)
C22	-C23	-C24	121.1(5)	C211	-C212	-C213	118.1(3)
C22	-C23	-C27	120.4(4)	C212	-C213	-C214	120.7(3)
C24	-C23	-C27	118.4(5)	C212	-C213	-C216	120.3(3)
C23	-C24	-C25	118.2(5)	C214	-C213	-C216	119.0(3)
C24	-C25	-C26	121.3(4)	C29	-C214	-C213	122.3(3)
C24	-C25	-C28	120.3(4)	F274	-C215	-F284	106.7(9)
C26	-C25	-C28	118.4(4)	F274	-C215	-F294	106.9(6)
C21	-C26	-C25	121.7(3)	F274	-C215	-C211	115.1(6)
F214	-C27	-F224	104.0(9)	F284	-C215	-F294	104.8(7)
F214	-C27	-F234	107.3(9)	F284	-C215	-C211	110.8(8)
F214	-C27	-C23	110.1(6)	F294	-C215	-C211	111.8(4)
F224	-C27	-F234	104.3(12)	C211	-C215	-F273	106.4(6)
F224	-C27	-C23	111.8(7)	C211	-C215	-F283	116.4(8)
F234	-C27	-C23	118.3(9)	C211	-C215	-F293	115.3(6)
C23	-C27	-F213	119.8(8)	F273	-C215	-F283	103.3(9)
C23	-C27	-F223	110.8(6)	F273	-C215	-F293	102.8(8)
C23	-C27	-F233	108.1(6)	F283	-C215	-F293	110.9(10)
F213	-C27	-F223	106.8(8)	F2104	-C216	-F2114	105.7(8)
F213	-C27	-F233	105.8(8)	F2104	-C216	-F2124	103.3(8)
F223	-C27	-F233	104.4(7)	F2104	-C216	-C213	116.4(6)
F244	-C28	-F254	102.0(5)	F2114	-C216	-F2124	103.6(9)
F244	-C28	-F264	99.5(4)	F2114	-C216	-C213	116.4(5)
F244	-C28	-C25	103.4(5)	F2124	-C216	-C213	109.9(6)

F254	-C28	-F264	128.3(7)	C213	-C216	-F2103	112.3(5)
F254	-C28	-C25	110.8(4)	C213	-C216	-F2113	107.3(5)
F264	-C28	-C25	109.0(4)	C213	-C216	-F2123	114.7(4)
C25	-C28	-F243	120.4(8)	F2103	-C216	-F2113	105.4(8)
C25	-C28	-F253	115.5(7)	F2103	-C216	-F2123	109.2(7)
C25	-C28	-F263	113.7(5)	F2113	-C216	-F2123	107.4(9)
F243	-C28	-F253	108.5(9)	C21	-B2	-C29	103.83(15)
F243	-C28	-F263	101.1(9)	C21	-B2	-C21_a	113.7(3)
F253	-C28	-F263	93.4(7)	C21	-B2	-C29_a	112.75(14)
C210	-C29	-C214	115.6(3)	C29	-B2	-C21_a	112.75(14)
C210	-C29	-B2	120.9(2)	C29	-B2	-C29_a	110.2(3)
C214	-C29	-B2	123.4(2)	C21_a	-B2	-C29_a	103.83(15)

*Part IV: Crystal structure determination of **4***

Abstract. $[C_{31}H_{45}N_2Pd]+.[C_{32}H_{12}BF_{24}]^-_{.0.5}(C_5H_{12})$, M_r = 1451.43, triclinic, *P*-1, *a* = 12.6191(5), *b* = 14.6417(6), *c* = 18.3813(8) Å, α = 108.590(1)°, β = 95.619(1)°, γ = 94.228(1)°, *V* = 3183.7(2) Å^3, *Z* = 2, D_x = 1.514 gcm^{-3}, *F*(000) = 1474, μ = 4.04 cm^{-1}, λ(MoK$\bar{\alpha}$) = 0.71073 Å, T = 100(1) K, 29404 reflections measured, *GooF* = 1.101, *wR*(*F*^{2}) = 0.1609 for 15198 unique reflections and 851 parameters and *R*(*F*) = 0.0554 for 11959 reflections obeying $F_o \geq 4.0\ \sigma(F_o)$ criterion of observability.

The asymmetric unit consists of three moieties: a cationic Pd-complex and a fluorinated-tetraphenylborate anion and half heavely disordered pentane solvent molecule, which is located over a inversion center.

Experimental

X-ray diffraction: Crystal and Molecular Structure.

A crystal with the dimensions of 0.52 x 0.39 x 0.11 mm was mounted on top of a glass fiber, by using inert-atmosphere handling techniques, and aligned on a *Bruker*[1] *SMART APEX CCD* diffractometer (Platform with full three-circle goniometer). The diffractometer was equipped with a 4K *CCD* detector set 60.0 mm from the crystal. The crystal was cooled to 100(1) K using the *Bruker KRYOFLEX* low-temperature device. Intensity measurements were performed using graphite monochromated Mo-K$\bar{\alpha}$ radiation from a sealed ceramic diffraction tube (*SIEMENS*). Generator settings were 50 KV/ 40 mA. *SMART* was used for preliminary determination of the unit cell constants and data collection control. The intensities of reflections of a hemisphere were collected by a combination of 3 sets of exposures (frames). Each set had a different ϕ angle for the crystal and each exposure covered a range of 0.3° in ω. A total of 1800 frames were collected with an exposure time of 10.0 seconds per frame. The overall data collection time was 8.0 h. Data integration and global cell refinement was performed with the program *SAINT*. The final unit cell was obtained from the *xyz* centroids of 9490 reflections after integration. Intensity data were corrected for Lorentz and polarization effects, scale variation, for decay and absorption: a multi-scan absorption correction was applied, based on the intensities of symmetry-related reflections measured at different angular settings (*SADABS*)[2], and reduced to F_o^2. The program suite *SHELXTL* was used for space group determination (*XPREP*).[1]

The unit cell[3] was identified as triclinic, space group *P*-1: the *E*-statistics were indicative of a centrosymmetric space group.[5] Reduced cell calculations did not indicate any higher metric lattice symmetry[4] and examination of the final atomic coordinates of the structure did not yield extra crystallographic or metric symmetry elements.[5,6]

The structure was solved by Patterson methods and extension of the model was accomplished by direct methods applied to difference structure factors using the program *DIRDIF*.[7] The positional and anisotropic displacement

parameters for the non-hydrogen atoms were refined. Refinement was complicated by a configurational-disorder problem: from the solution it was clear that C19 an C130 positions were disordered. A disorder model with two alternative positions was used in the refinement.

Some atoms showed unrealistic displacement parameters when allowed to vary anisotropically, suggesting dynamic disorder (dynamic means that the smeared electron density is due to fluctuations of the atomic positions within each unit cell) as a consequence of the rotational-disorder of some CF3 ligands.

From the solution it was clear that a pentane solvent molecule was highly disordered over an inversion center. The electron density appeared to be spread out, indicating transformational disorder. No satisfactory discrete model could be fitted in this density. The *BYPASS* procedure.[9] was used to take into account the electron density in the potential solvent area, which resulted in an electron count of 18 within a volume of 100.3 Å^3 in the unit cell (probably the cavities are partly occupied)

Hydrogen atoms were constrained to idealized geometries and allowed to ride on their carrier atoms with an isotropic displacement parameter related to the equivalent displacement parameter of their carrier atoms.

Final refinement on F^2 carried out by full-matrix least-squares techniques converged at $wR(F^2)$ = 0.1609 for 15198 reflections and $R(F)$ = 0.0554 for 11959 reflections with $F_o \geq 4.0\ \sigma(F_o)$ and 851 parameters. The final difference Fourier map was essentially featureless, except one free peak of 1.77(10) e/Å^3 between C113 and C115, but was neglected/rejected, being an disorder artifact (probably a disorder by a nearby 180° rotation about the line N11—N12: in other Pd structures we saw this disorder phenomena more heavily). No other significant peaks (max. = 1.09(10) e/Å^3) having chemical meaning above the general background were observed in the final difference Fourier syntheses.

The positional and anisotropic displacement parameters for the non-hydrogen atoms and isotropic displacement parameters for hydrogen atoms were refined on F^2 with full-matrix least-squares procedures minimizing the function $Q = \sum_h [w(\,|\,(F_o^2) - k(F_c^2)\,|\,)^2]$, where $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$, $P = [\max(F_o^2,0) + 2F_c^2]\ /\ 3$, F_0 and F_c are the observed and calculated structure factor amplitudes, respectively; ultimately the suggested a (=0.1015) and b (= 0.0) were used in the final refinement.

Crystal data and numerical details on data collection and refinement are given in Table 1. Final fractional atomic coordinates, equivalent displacement parameters and anisotropic displacement parameters for the non-hydrogen atoms are given in Table 2. Molecular geometry data are collected in Table 3. Neutral atom scattering factors and anomalous dispersion corrections were taken from *International Tables for Crystallography.*[10]

All refinement calculations and graphics were performed on a Pentium-III / Debian-Linux computer at the University of Groningen with the program packages *SHELXL*[11] (least-square refinements), a locally modified version of the program *PLUTO*[12] (preparation of illustrations) and *PLATON*[9] package

(checking the final results for missed symmetry with the *MISSYM* option, solvent accessible voids with the *SOLV* option, calculation of geometric data and the *ORTEP*[9] illustrations).

Each asymmetric unit contains one formula unit, consisting of three moieties: a cation Pd-complex and fluorinated-tetraphenylborate anion and a half, highly disordered, pentane solvent molecule, with no atom setting at special position. The monoclinic unit cell contains five discrete units, two cations and two anions molecules and one pentane solvent molecule, separated by normal van der Waals distances[13].

No classic hydrogen bonds or no missed symmetry (*MISSYM*) were detected by procedures implemented in *PLATON.*[14,15]

*) For disordered atoms, in the refinement described by several fractional s.o.f. atoms, only one of the fractional (major) s.o.f. atoms is included in the plots

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20. Derived values do contain the contribution of the distorted solvent.

Table 1.

a. Crystal data and details of the structure determination.

Moiety_Formula	$[C_{31}H_{45}N_2Pd]+.[C_{32}H_{12}BF_{24}]^-.0.5(C_5H_{12})$
Formula_Weight, g.mol^{-1}	1451.43
Crystal system	triclinic
Space group, no.[16]	*P*-1, 2
a, Å	12.6191(5)
b, Å	14.6417(6)
c, Å	18.3813(8)
α, deg	108.590(1)
β, deg	95.619(1)
γ, deg	94.228(1)
V, Å^3	3183.7(2)
Θ range unit cell: min.-max., deg; reflections	2.25 - 27.30 ; 9490
Formula_*Z*	2
SpaceGroup_*Z*	2
Z' (= Formula_Z / SpaceGroup_*Z)*	1
ρ_{calc}, g.cm^{-3}	1.514
F(000), electrons	1474
μ(Mo K$\bar{\alpha}$), cm^{-1}	4.04
Color, habit	yellow, platelet
Approx. crystal dimension, mm	0.52 x 0.39 x 0.11

b. Data collection.

λ(Mo K$\bar{\alpha}$), Å	0.71073
Monochromator	Graphite
Measurement device type	*CCD* area-detector diffractometer
Detector Area resolution (pixels / mm)	4096 x 4096 / 62 x 62 (binned 512)
Temperature, K	100(1)
Measurement method	φ- and ω-scans
θ range; min. max., deg	2.25, 28.28
Index ranges	h: -16→14; k: -17→19; l: -24→24
Min.- Max. absorption transmission factor	0.8814 – 0.9571
X-ray exposure time, h	8.0
Total data	29404
Unique data	15198
Data with criterion: ($F_o \geq 4.0\ \sigma\ (F_o)$)	11959
$R_{int} = \sum [\lvert F_o^2 - F_o^2\ (\text{mean})\rvert] / \sum [F_o^2]$	0.0218
$R_{sig} = \sum \sigma(F_o^2) / \sum [F_o^2]$	0.0417

c. Refinement.

Number of reflections	15198
Number of refined parameters	851
Final agreement factors:	
$wR(F^2) = [\sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)^2]]^{1/2}$	0.1609
Weighting scheme: *a, b*	0.1015, 0.0
$w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$	
And $P = [\max(F_o^2,0) + 2F_c^2] / 3$	
$R(F) = \sum (\|\|F_o\| - \|F_c\|\|) / \sum \|F_o\|$	0.0554
For $F_o > 4.0\ \sigma (F_o)$	
$GooF = S = [\sum [w(F_o^2 - F_c^2)^2] / (n-p)]^{1/2}$	1.101
n = number of reflections	
p = number of parameters refined	
Residual electron density in final	
Difference Fourier map, e/Å^3	-0.78, 1.77(10)
Max. (shift/σ) final cycle	0.015
Average (shift/σ) final cycle	0.001

Table 2. Final fractional atomic coordinates and equivalent isotropic displacement parameters with s.u.'s in parentheses.

Atoms of the Asymmetric Unit.

Non-Hydrogen parameters

Residue: 1.

Atom	*x*	*y*	*z*	*s.o.f.*	U_{eq} (Å^2)
Pd11	0.04459(2)	0.28205(2)	0.31278(1)	1.0(-)	0.02967(8)
N11	0.03673(19)	0.38764(16)	0.25853(13)	1.0(-)	0.0286(7)
N12	0.08520(19)	0.21088(16)	0.20244(13)	1.0(-)	0.0283(7)
C1302	-0.0082(12)	0.2505(7)	0.4092(5)	0.51(2)	0.041(3)
C11	-0.0097(2)	0.4763(2)	0.29314(16)	1.0(-)	0.0291(8)
C12	-0.1211(3)	0.4719(2)	0.2816(2)	1.0(-)	0.0454(10)
C13	-0.1680(3)	0.5537(2)	0.3214(2)	1.0(-)	0.0407(10)
C14	-0.1065(3)	0.6360(2)	0.37012(17)	1.0(-)	0.0361(9)
C15	0.0038(3)	0.6391(2)	0.38005(17)	1.0(-)	0.0360(9)
C16	0.0549(2)	0.5588(2)	0.34130(17)	1.0(-)	0.0322(8)
C17	-0.1917(3)	0.3829(3)	0.2292(4)	1.0(-)	0.097(2)
C18	-0.2618(5)	0.3380(4)	0.2708(4)	1.0(-)	0.118(3)
C110	0.1766(3)	0.5640(2)	0.3538(2)	1.0(-)	0.0417(10)
C111	0.2296(3)	0.6454(3)	0.3322(2)	1.0(-)	0.0564(13)
C112	0.2162(3)	0.5766(5)	0.4386(3)	1.0(-)	0.087(2)
C113	0.0696(2)	0.3644(2)	0.19187(15)	1.0(-)	0.0293(8)
C114	0.0725(3)	0.4302(2)	0.14301(17)	1.0(-)	0.0342(9)
C115	0.0974(2)	0.2638(2)	0.15952(16)	1.0(-)	0.0292(8)
C116	0.1304(3)	0.2297(2)	0.07975(16)	1.0(-)	0.0369(9)
C117	0.0944(3)	0.1072(2)	0.17577(16)	1.0(-)	0.0316(8)
C118	0.1945(3)	0.0728(2)	0.17643(16)	1.0(-)	0.0338(9)
C119	0.1952(3)	-0.0275(2)	0.1546(2)	1.0(-)	0.0484(11)
C120	0.1021(4)	-0.0893(3)	0.1339(3)	1.0(-)	0.0605(15)
C121	0.0032(3)	-0.0536(3)	0.1336(2)	1.0(-)	0.0512(11)
C122	-0.0028(3)	0.0466(2)	0.15617(18)	1.0(-)	0.0395(10)
C123	0.2982(2)	0.1407(2)	0.20169(16)	1.0(-)	0.0335(9)
C124	0.3978(3)	0.0932(3)	0.1723(2)	1.0(-)	0.0491(12)
C125	0.3179(3)	0.1852(3)	0.29052(18)	1.0(-)	0.0452(11)
C126	-0.1106(3)	0.0857(3)	0.1552(2)	1.0(-)	0.0484(11)
C127	-0.1975(4)	0.0271(4)	0.1801(3)	1.0(-)	0.0762(19)
C128	-0.1499(4)	0.0917(4)	0.0763(3)	1.0(-)	0.081(2)
C129	0.0076(4)	0.3460(3)	0.4259(2)	1.0(-)	0.0621(14)
C131	0.0555(4)	0.1848(3)	0.3765(2)	1.0(-)	0.0673(16)
C191	-0.2721(4)	0.4140(4)	0.1658(3)	0.666(11)	0.0519(19)
C1301	0.0571(19)	0.2727(9)	0.4233(6)	0.49(2)	0.066(5)
C192	-0.2009(16)	0.3428(15)	0.1632(10)	0.334(11)	0.103(8)

Residue: 2.

F21	0.77714(15)	0.62352(16)	0.03813(11)	1.0(-)	0.0514(7)
F22	0.93532(16)	0.69655(16)	0.07092(12)	1.0(-)	0.0531(7)

F23	0.8965(2)	0.57013(16)	0.10104(13)	1.0(-)	0.0715(9)
F24	1.06050(18)	0.8550(2)	0.35327(16)	1.0(-)	0.0798(10)
F25	0.9656(3)	0.97617(18)	0.37214(18)	1.0(-)	0.0952(11)
F26	0.94468(18)	0.87513(16)	0.43177(12)	1.0(-)	0.0613(8)
F27	0.6159(3)	1.1780(3)	0.3207(3)	1.0(-)	0.1456(19)
F28	0.73891(18)	1.14374(17)	0.38926(19)	1.0(-)	0.0791(12)
F29	0.6223(3)	1.22486(18)	0.4397(3)	1.0(-)	0.1266(16)
F210	0.32241(16)	1.05808(14)	0.50761(11)	1.0(-)	0.0510(6)
F211	0.27813(15)	0.90559(14)	0.45104(11)	1.0(-)	0.0449(6)
F212	0.23486(15)	1.00608(16)	0.39373(12)	1.0(-)	0.0515(7)
F213	0.6714(2)	0.57392(19)	0.48749(14)	1.0(-)	0.0759(10)
F214	0.6484(2)	0.72295(15)	0.52203(11)	1.0(-)	0.0604(8)
F215	0.5228(2)	0.6185(2)	0.52364(13)	1.0(-)	0.0709(9)
F216	0.47555(19)	0.35255(16)	0.21581(18)	1.0(-)	0.0856(9)
F217	0.3303(2)	0.39731(17)	0.2493(2)	1.0(-)	0.1008(12)
F218	0.3808(4)	0.4219(2)	0.1563(2)	1.0(-)	0.1589(19)
F219	0.15153(13)	0.68634(14)	0.14104(10)	1.0(-)	0.0391(6)
F220	0.14522(13)	0.67543(15)	0.02116(10)	1.0(-)	0.0450(6)
F221	0.21302(15)	0.56738(14)	0.06232(14)	1.0(-)	0.0569(7)
F222	0.43247(17)	0.84934(18)	-0.05248(12)	1.0(-)	0.0618(8)
F223	0.5773(2)	0.78992(17)	-0.03247(13)	1.0(-)	0.0627(8)
F224	0.55717(16)	0.93253(14)	0.03664(11)	1.0(-)	0.0495(6)
C21	0.6883(2)	0.77720(19)	0.25017(15)	1.0(-)	0.0256(7)
C22	0.7205(2)	0.71806(19)	0.18142(16)	1.0(-)	0.0264(8)
C23	0.8285(2)	0.7131(2)	0.17094(17)	1.0(-)	0.0305(8)
C24	0.9079(2)	0.7660(2)	0.22898(18)	1.0(-)	0.0360(9)
C25	0.8787(2)	0.8230(2)	0.29795(18)	1.0(-)	0.0355(9)
C26	0.7713(2)	0.8288(2)	0.30882(16)	1.0(-)	0.0301(8)
C27	0.8579(2)	0.6509(2)	0.09595(19)	1.0(-)	0.0374(10)
C28	0.9623(3)	0.8833(3)	0.3635(2)	1.0(-)	0.0516(11)
C29	0.5357(2)	0.88320(19)	0.32109(14)	1.0(-)	0.0243(7)
C210	0.5940(2)	0.9709(2)	0.32783(16)	1.0(-)	0.0302(8)
C211	0.5682(2)	1.0595(2)	0.37439(17)	1.0(-)	0.0340(9)
C212	0.4798(2)	1.0654(2)	0.41253(16)	1.0(-)	0.0335(8)
C213	0.4166(2)	0.9800(2)	0.40450(15)	1.0(-)	0.0283(8)
C214	0.4449(2)	0.89075(19)	0.36063(14)	1.0(-)	0.0252(7)
C215	0.6370(3)	1.1511(2)	0.3802(2)	1.0(-)	0.0487(11)
C216	0.3137(2)	0.9859(2)	0.43906(16)	1.0(-)	0.0347(9)
C217	0.5347(2)	0.68876(18)	0.29455(14)	1.0(-)	0.0237(7)
C218	0.5680(2)	0.69914(19)	0.37254(15)	1.0(-)	0.0258(7)
C219	0.5568(2)	0.6222(2)	0.40102(16)	1.0(-)	0.0288(8)
C220	0.5087(2)	0.5319(2)	0.35417(18)	1.0(-)	0.0347(9)
C221	0.4748(2)	0.5200(2)	0.27739(17)	1.0(-)	0.0332(8)
C222	0.4896(2)	0.59552(19)	0.24824(15)	1.0(-)	0.0281(8)
C223	0.5994(3)	0.6346(2)	0.48294(17)	1.0(-)	0.038(1)
C224	0.4192(3)	0.4225(2)	0.2262(2)	1.0(-)	0.0482(11)
C225	0.4862(2)	0.76816(18)	0.18107(14)	1.0(-)	0.0235(7)
C226	0.3797(2)	0.72538(19)	0.16445(14)	1.0(-)	0.0259(7)
C227	0.3153(2)	0.71955(19)	0.09685(15)	1.0(-)	0.0262(7)
C228	0.3534(2)	0.7588(2)	0.04330(15)	1.0(-)	0.0275(8)
C229	0.4580(2)	0.80400(18)	0.05979(15)	1.0(-)	0.0256(7)

C230	0.5221(2)	0.80847(18)	0.12701(14)	1.0(-)	0.0241(7)
C231	0.2067(2)	0.6626(2)	0.07990(17)	1.0(-)	0.0333(8)
C232	0.5048(2)	0.8441(2)	0.00327(16)	1.0(-)	0.0322(9)
B2	0.5604(2)	0.7790(2)	0.26228(16)	1.0(-)	0.0242(8)

Hydrogen parameters:

Residue: 1.

H13	-0.24377(-)	0.55255(-)	0.31468(-)	1.0(-)	0.04884(-)
H14	-0.13994(-)	0.69067(-)	0.39700(-)	1.0(-)	0.04326(-)
H15	0.04572(-)	0.69630(-)	0.41354(-)	1.0(-)	0.04315(-)
H18	-0.31134(-)	0.38363(-)	0.29489(-)	1.0(-)	0.17687(-)
H18'	-0.21806(-)	0.32148(-)	0.31090(-)	1.0(-)	0.17687(-)
H18"	-0.30275(-)	0.27896(-)	0.23429(-)	1.0(-)	0.17687(-)
H110	0.19816(-)	0.50158(-)	0.32033(-)	1.0(-)	0.04996(-)
H111	0.20614(-)	0.63625(-)	0.27770(-)	1.0(-)	0.08447(-)
H111'	0.30756(-)	0.64556(-)	0.34014(-)	1.0(-)	0.08447(-)
H111"	0.20967(-)	0.70732(-)	0.36471(-)	1.0(-)	0.08447(-)
H112	0.29098(-)	0.56285(-)	0.44293(-)	1.0(-)	0.13014(-)
H112'	0.17187(-)	0.53170(-)	0.45583(-)	1.0(-)	0.13014(-)
H112"	0.21116(-)	0.64341(-)	0.47102(-)	1.0(-)	0.13014(-)
H114	0.05500(-)	0.49439(-)	0.17298(-)	1.0(-)	0.05131(-)
H114'	0.02012(-)	0.40266(-)	0.09653(-)	1.0(-)	0.05131(-)
H114"	0.14439(-)	0.43615(-)	0.12787(-)	1.0(-)	0.05131(-)
H116	0.13689(-)	0.16001(-)	0.06449(-)	1.0(-)	0.05538(-)
H116'	0.19961(-)	0.26476(-)	0.07969(-)	1.0(-)	0.05538(-)
H116"	0.07631(-)	0.24205(-)	0.04303(-)	1.0(-)	0.05538(-)
H119	0.26193(-)	-0.05365(-)	0.15400(-)	1.0(-)	0.05795(-)
H120	0.10527(-)	-0.15727(-)	0.11950(-)	1.0(-)	0.07270(-)
H121	-0.06073(-)	-0.09729(-)	0.11813(-)	1.0(-)	0.06125(-)
H123	0.28950(-)	0.19495(-)	0.18036(-)	1.0(-)	0.04022(-)
H124	0.41141(-)	0.04258(-)	0.19525(-)	1.0(-)	0.07333(-)
H124'	0.45999(-)	0.14243(-)	0.18711(-)	1.0(-)	0.07333(-)
H124"	0.38522(-)	0.06428(-)	0.11577(-)	1.0(-)	0.07333(-)
H125	0.25774(-)	0.22093(-)	0.30896(-)	1.0(-)	0.06787(-)
H125'	0.38427(-)	0.22952(-)	0.30589(-)	1.0(-)	0.06787(-)
H125"	0.32410(-)	0.13336(-)	0.31324(-)	1.0(-)	0.06787(-)
H126	-0.10106(-)	0.15313(-)	0.19297(-)	1.0(-)	0.05781(-)
H127	-0.21495(-)	-0.03716(-)	0.14082(-)	1.0(-)	0.11401(-)
H127'	-0.26184(-)	0.06105(-)	0.18556(-)	1.0(-)	0.11401(-)
H127"	-0.17111(-)	0.02004(-)	0.22974(-)	1.0(-)	0.11401(-)
H128	-0.15915(-)	0.02664(-)	0.03777(-)	1.0(-)	0.12174(-)
H128'	-0.09720(-)	0.13341(-)	0.06200(-)	1.0(-)	0.12174(-)
H128"	-0.21857(-)	0.11899(-)	0.07823(-)	1.0(-)	0.12174(-)
H171	-0.14580(-)	0.33416(-)	0.20070(-)	1.0(-)	0.11568(-)
H191	-0.32445(-)	0.45386(-)	0.19272(-)	0.666(11)	0.07797(-)
H191'	-0.30972(-)	0.35534(-)	0.12652(-)	0.666(11)	0.07797(-)
H191"	-0.22942(-)	0.45095(-)	0.14069(-)	0.666(11)	0.07797(-)
H192	-0.14422(-)	0.36967(-)	0.14065(-)	0.334(11)	0.15445(-)
H192'	-0.27102(-)	0.35103(-)	0.13956(-)	0.334(11)	0.15445(-)

H192"	-0.19536(-)	0.27360(-)	0.15360(-)	0.334(11)	0.15445(-)
H1291	0.04661(-)	0.41031(-)	0.45584(-)	1.0(-)	0.07416(-)
H1291'	-0.06908(-)	0.34033(-)	0.43307(-)	1.0(-)	0.07416(-)
H1301	0.10902(-)	0.28575(-)	0.46742(-)	0.49(2)	0.07866(-)
H1302	-0.07298(-)	0.22604(-)	0.42217(-)	0.51(2)	0.04924(-)
H1311	-0.00936(-)	0.13981(-)	0.37156(-)	1.0(-)	0.08065(-)
H1311'	0.12304(-)	0.15454(-)	0.37749(-)	1.0(-)	0.08065(-)

Residue: 2.

H22	0.66719(-)	0.68018(-)	0.14064(-)	1.0(-)	0.03169(-)
H24	0.98130(-)	0.76331(-)	0.22170(-)	1.0(-)	0.04325(-)
H26	0.75381(-)	0.86857(-)	0.35707(-)	1.0(-)	0.03613(-)
H210	0.65334(-)	0.96998(-)	0.29961(-)	1.0(-)	0.03624(-)
H212	0.46210(-)	1.12625(-)	0.44372(-)	1.0(-)	0.04013(-)
H214	0.40164(-)	0.83326(-)	0.35726(-)	1.0(-)	0.03023(-)
H218	0.59921(-)	0.76080(-)	0.40675(-)	1.0(-)	0.03092(-)
H220	0.49912(-)	0.47977(-)	0.37399(-)	1.0(-)	0.04166(-)
H222	0.46831(-)	0.58372(-)	0.19473(-)	1.0(-)	0.03379(-)
H226	0.35065(-)	0.69953(-)	0.20044(-)	1.0(-)	0.03099(-)
H228	0.30942(-)	0.75489(-)	-0.00286(-)	1.0(-)	0.03293(-)
H230	0.59308(-)	0.84007(-)	0.13671(-)	1.0(-)	0.02890(-)

*) $U_{eq} = 1/3 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a_i}.\mathbf{a_j}$ [17]

Anisotropic (displacement) parameters (Å^2)

Residue: 1.

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Pd11	0.03810(14)	0.02394(13)	0.03069(12)	0.01227(9)	0.01216(9)	0.00161(9)
N11	0.0324(12)	0.0214(11)	0.0348(12)	0.0117(9)	0.0087(9)	0.0033(10)
N12	0.0339(12)	0.0196(11)	0.0316(11)	0.0080(9)	0.0076(9)	0.0021(9)
C1302	0.047(6)	0.054(5)	0.027(3)	0.021(3)	0.011(3)	-0.011(4)
C11	0.0310(14)	0.0246(14)	0.0358(14)	0.0131(11)	0.0117(11)	0.0053(11)
C12	0.0327(16)	0.0284(16)	0.070(2)	0.0065(15)	0.0192(15)	-0.0011(13)
C13	0.0301(15)	0.0345(17)	0.0588(19)	0.0127(14)	0.0173(14)	0.0086(13)
C14	0.0431(17)	0.0317(16)	0.0389(15)	0.0146(12)	0.0142(13)	0.0132(13)
C15	0.0416(17)	0.0254(15)	0.0396(15)	0.0085(12)	0.0046(13)	0.0066(13)
C16	0.0345(15)	0.0246(14)	0.0382(14)	0.0111(12)	0.0047(12)	0.0050(12)
C17	0.033(2)	0.043(2)	0.169(6)	-0.031(3)	0.032(3)	-0.0082(18)
C18	0.107(4)	0.089(4)	0.166(6)	0.084(5)	-0.037(4)	-0.057(4)
C110	0.0321(16)	0.0295(16)	0.0552(19)	0.0033(14)	0.0018(14)	0.0047(13)
C111	0.0348(18)	0.077(3)	0.071(2)	0.041(2)	0.0148(17)	0.0055(18)
C112	0.040(2)	0.146(5)	0.107(4)	0.092(4)	-0.006(2)	0.002(3)
C113	0.0217(13)	0.0369(16)	0.0289(13)	0.0109(11)	0.0049(10)	-0.0015(11)
C114	0.0439(17)	0.0266(15)	0.0386(15)	0.0176(12)	0.0099(12)	0.0070(13)
C115	0.0285(14)	0.0299(15)	0.0318(13)	0.0158(11)	0.0019(11)	-0.0039(11)
C116	0.0541(19)	0.0280(15)	0.0308(14)	0.0107(12)	0.0113(13)	0.0055(14)

C117	0.0463(17)	0.0187(13)	0.0301(13)	0.0087(11)	0.0081(12)	-0.0011(12)
C118	0.0518(18)	0.0205(13)	0.0321(14)	0.0099(11)	0.0141(12)	0.0064(13)
C119	0.065(2)	0.0246(16)	0.062(2)	0.0177(15)	0.0199(18)	0.0133(16)
C120	0.084(3)	0.0209(16)	0.076(3)	0.0114(16)	0.024(2)	0.0046(18)
C121	0.062(2)	0.0286(17)	0.060(2)	0.0114(15)	0.0119(18)	-0.0053(16)
C122	0.0510(19)	0.0263(15)	0.0418(16)	0.0127(13)	0.0082(14)	-0.0026(14)
C123	0.0418(16)	0.0287(15)	0.0346(14)	0.0142(12)	0.0089(12)	0.0097(13)
C124	0.052(2)	0.045(2)	0.058(2)	0.0213(16)	0.0236(17)	0.0116(16)
C125	0.0462(19)	0.054(2)	0.0359(16)	0.0156(15)	0.0045(14)	0.0067(16)
C126	0.0453(19)	0.0341(18)	0.063(2)	0.0149(16)	0.0045(16)	-0.0043(15)
C127	0.057(3)	0.075(3)	0.110(4)	0.045(3)	0.029(3)	0.001(2)
C128	0.058(3)	0.102(4)	0.094(4)	0.051(3)	0.000(2)	0.006(3)
C129	0.099(3)	0.055(2)	0.0440(19)	0.0206(18)	0.040(2)	0.022(2)
C131	0.110(4)	0.054(2)	0.064(2)	0.042(2)	0.044(2)	0.027(2)
C191	0.043(3)	0.047(4)	0.050(3)	-0.004(2)	0.014(2)	-0.015(3)
C1301	0.097(14)	0.070(7)	0.036(4)	0.028(4)	0.011(5)	-0.004(7)
C192	0.130(16)	0.106(16)	0.075(11)	0.061(11)	-0.046(10)	-0.036(13)

Residue: 2.

F21	0.0381(10)	0.0617(14)	0.0451(10)	0.0027(9)	0.0096(8)	0.0096(9)
F22	0.0426(11)	0.0680(14)	0.0526(11)	0.0224(10)	0.0197(9)	0.0018(10)
F23	0.116(2)	0.0518(13)	0.0626(13)	0.0276(11)	0.0252(13)	0.0509(14)
F24	0.0362(12)	0.095(2)	0.0918(19)	0.0199(15)	-0.0145(12)	-0.0164(12)
F25	0.109(2)	0.0438(14)	0.117(2)	0.0379(15)	-0.0616(18)	-0.0422(14)
F26	0.0651(14)	0.0509(13)	0.0521(12)	0.0089(10)	-0.0253(10)	-0.0124(11)
F27	0.148(3)	0.117(3)	0.189(4)	0.124(3)	-0.072(3)	-0.088(2)
F28	0.0453(13)	0.0402(13)	0.150(3)	0.0309(15)	0.0164(14)	-0.0106(10)
F29	0.111(2)	0.0248(13)	0.209(4)	-0.0199(17)	0.078(3)	-0.0247(14)
F210	0.0496(11)	0.0456(12)	0.0412(10)	-0.0094(8)	0.0106(8)	0.0015(9)
F211	0.0448(11)	0.0384(10)	0.0517(11)	0.0119(9)	0.0185(9)	0.0019(8)
F212	0.0319(10)	0.0659(14)	0.0578(12)	0.0236(10)	-0.0029(8)	0.0089(9)
F213	0.1042(19)	0.0733(17)	0.0576(13)	0.0254(12)	-0.0011(13)	0.0536(15)
F214	0.0985(17)	0.0448(12)	0.0356(10)	0.0191(9)	-0.0131(10)	-0.0052(12)
F215	0.0734(16)	0.103(2)	0.0509(12)	0.0438(13)	0.0196(11)	0.0020(14)
F216	0.0529(14)	0.0316(12)	0.135(2)	-0.0245(13)	0.0175(14)	-0.0017(10)
F217	0.0595(15)	0.0379(13)	0.164(3)	-0.0270(16)	0.0439(17)	-0.0195(11)
F218	0.284(5)	0.0570(19)	0.099(2)	0.0313(17)	-0.085(3)	-0.093(3)
F219	0.0275(9)	0.0524(11)	0.0395(9)	0.0188(8)	0.0059(7)	-0.0020(8)
F220	0.0278(9)	0.0696(14)	0.0319(9)	0.0134(9)	-0.0036(7)	-0.0066(9)
F221	0.0393(11)	0.0306(10)	0.0878(15)	0.0048(10)	0.0069(10)	-0.0089(8)
F222	0.0504(12)	0.0928(18)	0.0557(12)	0.0540(12)	-0.0114(10)	-0.0166(12)
F223	0.0881(16)	0.0607(14)	0.0646(13)	0.0393(11)	0.0485(12)	0.0317(12)
F224	0.0614(12)	0.0394(11)	0.0486(10)	0.0216(9)	0.0036(9)	-0.0165(9)
C21	0.0284(13)	0.0204(12)	0.0296(12)	0.0123(10)	0.0025(10)	-0.0033(10)
C22	0.0263(13)	0.0209(13)	0.0333(13)	0.0115(10)	0.0032(10)	-0.0005(10)
C23	0.0297(14)	0.0270(14)	0.0416(15)	0.0203(12)	0.0070(11)	0.0026(11)
C24	0.0245(14)	0.0397(17)	0.0519(17)	0.0271(14)	0.0043(12)	-0.0001(12)
C25	0.0325(15)	0.0307(16)	0.0429(16)	0.0168(13)	-0.0047(12)	-0.0086(12)
C26	0.0321(14)	0.0254(14)	0.0327(13)	0.0125(11)	-0.0007(11)	-0.0051(11)
C27	0.0307(15)	0.0416(18)	0.0485(17)	0.0238(14)	0.0095(13)	0.0122(13)

C28	0.0374(19)	0.044(2)	0.066(2)	0.0172(18)	-0.0111(16)	-0.0141(16)
C29	0.0280(13)	0.0213(13)	0.0224(11)	0.0082(9)	-0.0018(10)	-0.003(1)
C210	0.0305(14)	0.0243(14)	0.0355(14)	0.0108(11)	0.0029(11)	-0.0012(11)
C211	0.0365(16)	0.0202(14)	0.0422(15)	0.0102(12)	-0.0029(12)	-0.0047(12)
C212	0.0373(16)	0.0203(14)	0.0361(14)	0.0024(11)	-0.0043(12)	0.0038(12)
C213	0.0270(14)	0.0276(14)	0.0257(12)	0.0053(10)	-0.0057(10)	0.0010(11)
C214	0.0296(13)	0.0203(13)	0.0229(11)	0.0061(10)	-0.0022(10)	-0.003(1)
C215	0.050(2)	0.0227(16)	0.071(2)	0.0147(16)	0.0059(17)	-0.0055(14)
C216	0.0367(16)	0.0301(15)	0.0314(14)	0.0027(12)	0.0014(12)	0.0037(13)
C217	0.0247(13)	0.0185(12)	0.0268(12)	0.0058(10)	0.0055(10)	0.0001(10)
C218	0.0290(14)	0.0191(12)	0.0296(12)	0.0082(10)	0.0059(10)	0.0013(10)
C219	0.0311(14)	0.0255(14)	0.0342(13)	0.0138(11)	0.0092(11)	0.0057(11)
C220	0.0398(16)	0.0227(14)	0.0486(16)	0.0179(12)	0.0169(13)	0.0047(12)
C221	0.0338(15)	0.0182(13)	0.0442(16)	0.0057(12)	0.0100(12)	-0.0040(11)
C222	0.0299(14)	0.0225(13)	0.0293(13)	0.0046(10)	0.007(1)	-0.0005(11)
C223	0.0521(19)	0.0313(16)	0.0380(15)	0.0193(13)	0.0095(13)	0.0113(14)
C224	0.065(2)	0.0225(16)	0.0511(19)	0.0072(14)	0.0073(17)	-0.0111(16)
C225	0.0257(13)	0.0176(12)	0.0245(11)	0.0042(9)	0.0026(9)	-0.0013(10)
C226	0.0270(13)	0.0237(13)	0.0250(12)	0.0052(10)	0.0057(10)	0.0008(10)
C227	0.0246(13)	0.0237(13)	0.0270(12)	0.0044(10)	0.0034(10)	0.0004(11)
C228	0.0270(13)	0.0267(14)	0.0259(12)	0.0053(10)	0.0012(10)	0.0036(11)
C229	0.0280(13)	0.0196(13)	0.0295(12)	0.0081(10)	0.0043(10)	0.0035(10)
C230	0.0238(12)	0.0200(12)	0.0266(12)	0.0061(10)	0.0028(10)	-0.0019(10)
C231	0.0273(14)	0.0331(16)	0.0361(14)	0.0070(12)	0.0051(11)	0.0002(12)
C232	0.0363(16)	0.0294(15)	0.0333(14)	0.0147(12)	0.0021(11)	0.0023(12)
B2	0.0267(14)	0.0193(14)	0.0243(13)	0.0055(11)	0.0029(11)	-0.0029(11)

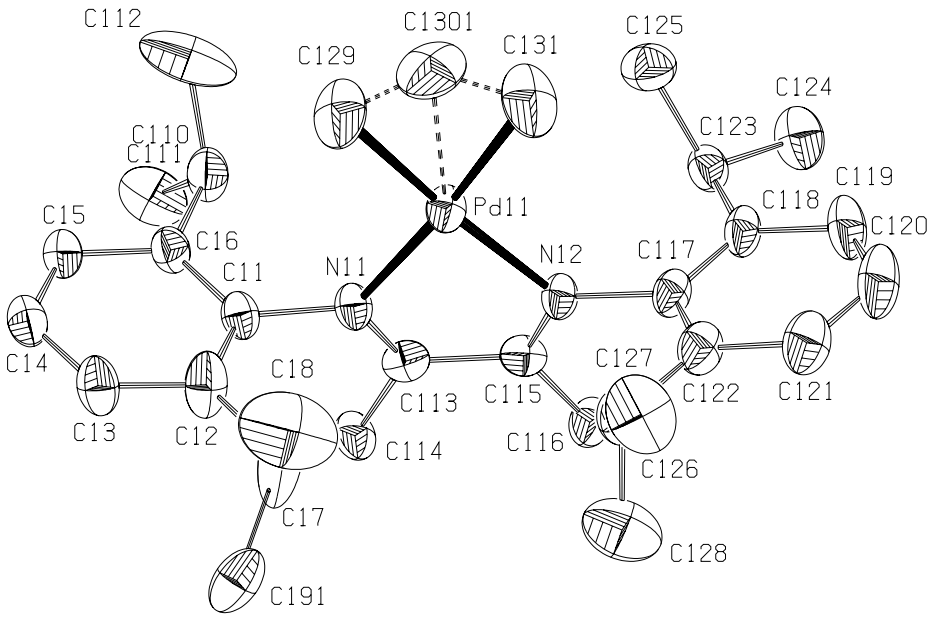
Thermal vibration amplitudes ($Å^2$)

$$F(\mathbf{h}) = F_o(\mathbf{h}) \exp\left(-2\pi^2 \sum_{i=1}^{3}\sum_{j=1}^{3} h_i h_j a_i^* a_j^* U_{ij}\right)$$

or

$$F(\mathbf{h}) = F_o(\mathbf{h}) \exp\left(-8\pi^2 U_{iso} (\sin(\theta)/\lambda)^2\right)$$

Cation of **4**:



Anion of **4**:

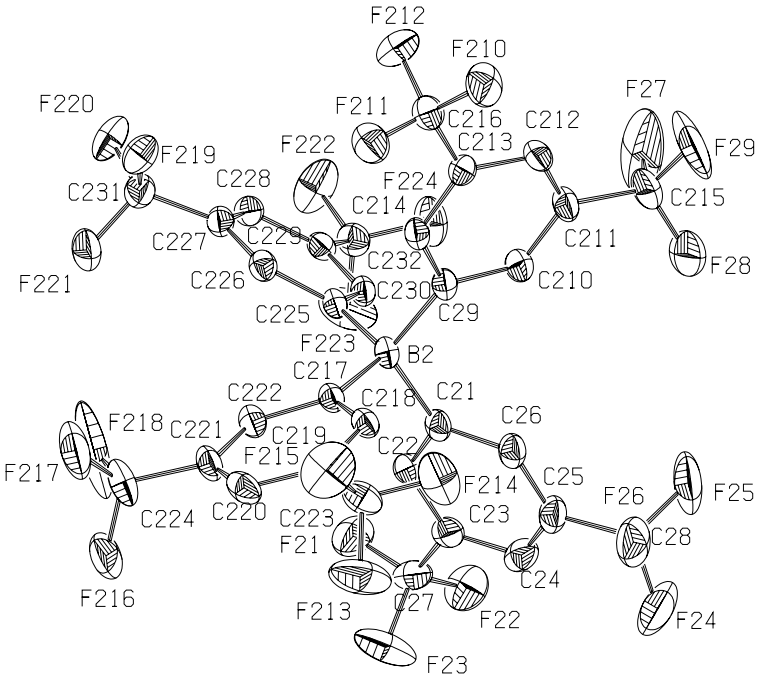


Table 3. Data on the geometry.

Standard deviations in the last decimal place are given in parentheses.

Residue: 1.

Interatomic Distances (Å)

Pd11	-N11	2.093(2)	C17	-C192	1.157(19)
Pd11	-N12	2.097(2)	C110	-C16	1.523(5)
Pd11	-C1302	2.123(10)	C110	-C111	1.504(5)
Pd11	-C129	2.105(4)	C110	-C112	1.538(6)
Pd11	-C131	2.116(4)	C113	-C114	1.513(4)
Pd11	-C1301	2.071(11)	C113	-C115	1.485(4)
N11	-C11	1.446(4)	C115	-C116	1.502(4)
N11	-C113	1.282(3)	C117	-C118	1.394(5)
N12	-C115	1.282(4)	C117	-C122	1.404(5)
N12	-C117	1.456(4)	C118	-C119	1.395(4)
C1302	-C129	1.328(11)	C118	-C123	1.520(4)
C1302	-C131	1.332(14)	C119	-C120	1.371(6)
C11	-C12	1.395(5)	C120	-C121	1.388(6)
C11	-C16	1.393(4)	C121	-C122	1.402(5)
C12	-C13	1.397(5)	C122	-C126	1.515(5)
C12	-C17	1.510(7)	C123	-C124	1.542(5)
C13	-C14	1.376(5)	C123	-C125	1.540(4)
C14	-C15	1.381(5)	C126	-C127	1.536(7)
C15	-C16	1.405(4)	C126	-C128	1.518(6)
C17	-C18	1.472(8)	C1301	-C129	1.271(18)
C17	-C191	1.664(8)	C1301	-C131	1.298(13)

Bond Angles (deg.)

N11	-Pd11	-N12	76.92(9)	C12	-C17	-C192	132.8(12)
N11	-Pd11	-C1302	140.2(3)	C18	-C17	-C191	106.4(4)
N11	-Pd11	-C129	106.78(14)	C18	-C17	-C192	113.9(12)
N11	-Pd11	-C131	175.24(12)	C16	-C110	-C111	111.9(3)
N11	-Pd11	-C1301	139.2(4)	C16	-C110	-C112	110.5(3)
N12	-Pd11	-C1302	140.2(3)	C111	-C110	-C112	109.4(4)
N12	-Pd11	-C129	176.19(14)	N11	-C113	-C114	123.8(3)
N12	-Pd11	-C131	107.30(13)	N11	-C113	-C115	116.7(3)
N12	-Pd11	-C1301	140.8(5)	C114	-C113	-C115	119.3(2)
C1302	-Pd11	-C129	36.6(3)	N12	-C115	-C113	115.2(2)
C1302	-Pd11	-C131	36.6(4)	N12	-C115	-C116	125.1(3)
C129	-Pd11	-C131	68.97(16)	C113	-C115	-C116	119.6(3)
C129	-Pd11	-C1301	35.4(5)	N12	-C117	-C118	120.8(3)
C131	-Pd11	-C1301	36.1(4)	N12	-C117	-C122	115.6(3)
Pd11	-N11	-C11	120.91(18)	C118	-C117	-C122	123.4(3)
Pd11	-N11	-C113	114.9(2)	C117	-C118	-C119	116.7(3)
C11	-N11	-C113	124.1(3)	C117	-C118	-C123	122.1(3)
Pd11	-N12	-C115	115.7(2)	C119	-C118	-C123	121.2(3)
Pd11	-N12	-C117	121.71(18)	C118	-C119	-C120	121.7(4)
C115	-N12	-C117	122.6(2)	C119	-C120	-C121	120.7(4)

Pd11	-C1302	-C129	71.0(5)	C120	-C121	-C122	120.3(4)
Pd11	-C1302	-C131	71.4(5)	C117	-C122	-C121	117.1(3)
C129	-C1302	-C131	128.0(11)	C117	-C122	-C126	122.4(3)
N11	-C11	-C12	116.9(3)	C121	-C122	-C126	120.4(3)
N11	-C11	-C16	120.3(2)	C118	-C123	-C124	114.3(3)
C12	-C11	-C16	122.5(3)	C118	-C123	-C125	110.4(2)
C11	-C12	-C13	117.7(3)	C124	-C123	-C125	109.3(3)
C11	-C12	-C17	122.9(3)	C122	-C126	-C127	113.4(4)
C13	-C12	-C17	119.4(3)	C122	-C126	-C128	111.7(3)
C12	-C13	-C14	121.2(3)	C127	-C126	-C128	109.1(4)
C13	-C14	-C15	120.1(3)	Pd11	-C129	-C1302	72.4(5)
C14	-C15	-C16	120.9(3)	Pd11	-C129	-C1301	70.8(5)
C11	-C16	-C15	117.6(3)	Pd11	-C131	-C1302	72.0(5)
C11	-C16	-C110	122.7(3)	Pd11	-C131	-C1301	70.1(6)
C15	-C16	-C110	119.8(3)	Pd11	-C1301	-C129	73.8(6)
C12	-C17	-C18	113.3(5)	Pd11	-C1301	-C131	73.9(5)
C12	-C17	-C191	109.1(4)	C129	-C1301	-C131	137.0(14)

Residue: 2.

Interatomic Distances (Å)

F21	-C27	1.336(4)	C24	-C25	1.379(4)
F22	-C27	1.342(4)	C25	-C26	1.394(4)
F23	-C27	1.338(4)	C25	-C28	1.510(5)
F24	-C28	1.347(5)	C29	-B2	1.636(4)
F25	-C28	1.315(5)	C210	-C29	1.395(4)
F26	-C28	1.336(4)	C210	-C211	1.389(4)
F27	-C215	1.287(6)	C211	-C212	1.368(4)
F28	-C215	1.297(4)	C211	-C215	1.511(4)
F29	-C215	1.309(6)	C212	-C213	1.391(4)
F210	-C216	1.350(3)	C213	-C214	1.392(4)
F211	-C216	1.319(4)	C213	-C216	1.497(4)
F212	-C216	1.345(3)	C214	-C29	1.409(4)
F213	-C223	1.332(4)	C217	-C218	1.410(4)
F214	-C223	1.332(4)	C217	-C222	1.401(4)
F215	-C223	1.330(4)	C217	-B2	1.636(4)
F216	-C224	1.265(4)	C218	-C219	1.389(4)
F217	-C224	1.307(5)	C219	-C220	1.383(4)
F218	-C224	1.325(5)	C219	-C223	1.497(4)
F219	-C231	1.345(3)	C220	-C221	1.385(4)
F220	-C231	1.338(3)	C221	-C222	1.383(4)
F221	-C231	1.337(4)	C221	-C224	1.511(4)
F222	-C232	1.329(3)	C225	-C226	1.402(4)
F223	-C232	1.338(4)	C225	-C230	1.401(4)
F224	-C232	1.333(4)	C225	-B2	1.638(4)
C21	-C22	1.402(4)	C226	-C227	1.391(4)
C21	-C26	1.405(4)	C227	-C228	1.394(4)
C21	-B2	1.652(4)	C227	-C231	1.501(4)
C22	-C23	1.399(4)	C228	-C229	1.392(4)
C23	-C24	1.375(4)	C229	-C230	1.389(4)

C23	-C27	1.489(4)		C229	-C232	1.494(4)	

Bond Angles (deg.)

C22	-C21	-C26	115.8(2)	C218	-C217	-B2	120.0(2)
C22	-C21	-B2	121.2(2)	C222	-C217	-B2	124.6(2)
C26	-C21	-B2	122.8(2)	C217	-C218	-C219	122.2(3)
C21	-C22	-C23	122.1(3)	C218	-C219	-C220	121.1(3)
C22	-C23	-C24	120.6(3)	C218	-C219	-C223	120.6(3)
C22	-C23	-C27	119.8(2)	C220	-C219	-C223	118.4(3)
C24	-C23	-C27	119.6(2)	C219	-C220	-C221	117.8(3)
C23	-C24	-C25	118.6(2)	C220	-C221	-C222	121.2(3)
C24	-C25	-C26	121.3(3)	C220	-C221	-C224	118.3(3)
C24	-C25	-C28	120.9(3)	C222	-C221	-C224	120.5(3)
C26	-C25	-C28	117.8(3)	C217	-C222	-C221	122.6(2)
C21	-C26	-C25	121.6(3)	F213	-C223	-F214	105.5(3)
F21	-C27	-F22	105.3(3)	F213	-C223	-F215	106.2(3)
F21	-C27	-F23	107.1(3)	F213	-C223	-C219	112.4(3)
F21	-C27	-C23	114.1(2)	F214	-C223	-F215	106.2(3)
F22	-C27	-F23	105.4(2)	F214	-C223	-C219	113.6(3)
F22	-C27	-C23	111.9(3)	F215	-C223	-C219	112.4(3)
F23	-C27	-C23	112.5(3)	F216	-C224	-F217	107.6(3)
F24	-C28	-F25	109.1(4)	F216	-C224	-F218	106.1(3)
F24	-C28	-F26	104.7(3)	F216	-C224	-C221	115.5(3)
F24	-C28	-C25	111.7(3)	F217	-C224	-F218	99.4(4)
F25	-C28	-F26	106.2(3)	F217	-C224	-C221	113.5(3)
F25	-C28	-C25	111.9(3)	F218	-C224	-C221	113.2(3)
F26	-C28	-C25	112.9(3)	C226	-C225	-C230	115.8(2)
C210	-C29	-C214	115.6(2)	C226	-C225	-B2	122.0(2)
C210	-C29	-B2	122.7(2)	C230	-C225	-B2	122.0(2)
C214	-C29	-B2	121.2(2)	C225	-C226	-C227	122.1(2)
C29	-C210	-C211	122.2(2)	C226	-C227	-C228	121.1(2)
C210	-C211	-C212	121.3(3)	C226	-C227	-C231	118.1(2)
C210	-C211	-C215	119.0(3)	C228	-C227	-C231	120.6(2)
C212	-C211	-C215	119.7(3)	C227	-C228	-C229	117.7(2)
C211	-C212	-C213	118.3(3)	C228	-C229	-C230	120.8(2)
C212	-C213	-C214	120.5(2)	C228	-C229	-C232	120.2(2)
C212	-C213	-C216	119.0(3)	C230	-C229	-C232	118.9(2)
C214	-C213	-C216	120.4(3)	C225	-C230	-C229	122.6(2)
C29	-C214	-C213	122.0(3)	F219	-C231	-F220	106.6(2)
F27	-C215	-F28	108.3(3)	F219	-C231	-F221	105.2(2)
F27	-C215	-F29	105.4(4)	F219	-C231	-C227	112.4(2)
F27	-C215	-C211	112.1(3)	F220	-C231	-F221	107.3(2)
F28	-C215	-F29	104.7(3)	F220	-C231	-C227	112.8(2)
F28	-C215	-C211	113.3(3)	F221	-C231	-C227	112.0(2)
F29	-C215	-C211	112.5(3)	F222	-C232	-F223	106.0(2)
F210	-C216	-F211	106.9(2)	F222	-C232	-F224	107.1(3)
F210	-C216	-F212	105.3(2)	F222	-C232	-C229	113.5(2)
F210	-C216	-C213	111.9(2)	F223	-C232	-F224	105.0(2)
F211	-C216	-F212	106.7(2)	F223	-C232	-C229	111.8(3)
F211	-C216	-C213	113.9(2)	F224	-C232	-C229	112.9(2)

F212	-C216	-C213	111.6(2)	C21	-B2	-C29	112.3(2)
C218	-C217	-C222	115.1(2)	C21	-B2	-C217	104.0(2)
C21	-B2	-C225	110.9(2)	C29	-B2	-C225	103.6(2)
C29	-B2	-C217	113.1(2)	C217	-B2	-C225	113.3(2)

*Part V: Crystal structure determination of **5***

Abstract. $[C_{34}H_{53}N_2OPd]^+.[C_{32}H_{12}BF_{24}]^-$, M_r = 1475.45, triclinic, *P*-1, *a* = 12.5293(8), *b* = 15.592(1), *c* = 18.468(1) Å, α = 77.686(1)°, β = 70.452(1)°, γ = 82.915(1)°, *V* = 3316.4(4) Å^3, *Z* = 2, D_x = 1.478 gcm^{-3}, *F*(000) = 1500, *μ* = 3.9 cm^{-1}, λ(MoK$\bar{\alpha}$) = 0.71073 Å, T = 100(1) K, 24071 reflections measured, *GooF* = 1.041, *wR*(F^2) = 0.2318 for 11523 unique reflections and 962 parameters, 112 restraints and *R*(*F*) = 0.0786 for 5617 reflections obeying $F_o \geq 4.0\ \sigma(F_o)$ criterion of observability.

The asymmetric unit consists of two moieties: a cationic Pd-complex and a fluorinated-tetraphenylborate anion.

Comment

The scattering power of the crystals investigated was very weak: nearby half of the unique (till Θ = 25 °) merged reflections obey the $F_o \geq 4.0\ \sigma(F_o)$ criterion of observability. This implies that the mean *s.u*. is large compared to the mean magnitude of the (even more than double of the squared) structure factor. The weak scattering power might be the result of a disorder problem.

Experimental

X-ray diffraction: Crystal and Molecular Structure.

A crystal with the dimensions of 0.20 x 0.11 x 0.035 mm was mounted on top of a glass fiber, by using inert-atmosphere handling techniques, and aligned on a *Bruker*[1] *SMART APEX CCD* diffractometer (Platform with full three-circle goniometer). The diffractometer was equipped with a 4K *CCD* detector set 60.0 mm from the crystal. The crystal was cooled to 100(1) K using the *Bruker KRYOFLEX* low-temperature device. Intensity measurements were performed using graphite monochromated Mo-K$\bar{\alpha}$ radiation from a sealed ceramic diffraction tube (*SIEMENS*). Generator settings were 50 KV/ 40 mA. *SMART* was used for preliminary determination of the unit cell constants and data collection control. The intensities of reflections of a hemisphere were collected by a combination of 3 sets of exposures (frames). Each set had a different φ angle for the crystal and each exposure covered a range of 0.3° in ω. A total of 1800 frames were collected with an exposure time of 30.0 seconds per frame. The overall data collection time was 18.8 h. Data integration and global cell refinement was performed with the program *SAINT*. The final unit cell was obtained from the *xyz* centroids of 5277 reflections after integration. Intensity data were corrected for Lorentz and polarization effects, scale variation, for decay and absorption: a multi-scan absorption correction was applied, based on the intensities of symmetry-related reflections measured at different angular settings (*SADABS*)[2], and reduced to F_o^2. The program suite *SHELXTL* was used for space group determination (*XPREP*).[1]

The unit cell[3] was identified as triclinic. Reduced cell calculations did not indicate any higher metric lattice symmetry.[4] Space group, *P-1*, was determined from considerations of the unit cell parameters, statistical

analyses of intensity distributions: the *E*-statistics[5] were indicative of a centrosymmetric space group. Examination of the final atomic coordinates of the structure did not yield extra crystallographic or metric symmetry elements.[5,6]

The structure was solved by Patterson methods and extension of the model was accomplished by direct methods applied to difference structure factors using the program *DIRDIF*.[7] The positional and anisotropic displacement parameters for the non-hydrogen atoms were refined. Some atoms showed unrealistic displacement parameters when allowed to vary anisotropically, suggesting dynamic disorder (dynamic means that the smeared electron density is due to fluctuations of the atomic positions within each unit cell) as a consequence of the rotational-disorder (especially for the CF_3 ligands). The observed disorder is in line with the weak scattering power of the crystals investigated.

The most extreme disorder CF_3-ligand (F27...F29) has been described by two site occupancy factors with separately refined displacement parameters. The s.o.f. of the major fraction of the component of the disorder model refined to a value of 0.59(2).

Also the chelate fragment derived from insertion of allyl ethyl ether into the Pd-Me bond showed disorder: inspection of the remaining electron density in a subsequent difference Fourier map lead to the suggestion that the ligand might be disordered by a twofold rotation about the vector Pd1--C132. The coordinates of the alternative setting were introduced in the refinement and the site occupancy factors refined. To improve the parameters chemical more reasonable, ultimately restrain instructions (*DFIX, DELU, SIMU*) for the disordered part were applied in the refinement (the disorder is compensated by the large displacement parameters). The s.o.f. of the major fraction of this component of the disorder model refined to a value of 0.60(1).

The hydrogen atoms were generated by geometrical considerations, which coordinates and isotropic displacement parameters were refined.

Final refinement on F^2 carried out by full-matrix least-squares techniques converged at $wR(F^2)$ = 0.2318 for 11523 reflections and $R(F)$ = 0.0786 for 5617 reflections with $F_o \geq 4.0\ \sigma(F_o)$ and 962 parameters and 112 restraints. The final difference Fourier map was essentially featureless: no significant peaks (0.80(9) e/Å^3) having chemical meaning above the general background were observed.

The positional and anisotropic displacement parameters for the non-hydrogen atoms and isotropic displacement parameters for hydrogen atoms were refined on F^2 with full-matrix least-squares procedures minimizing the function $Q = \sum_h[w(|(F_o^2) - k(F_c^2)|)^2]$, where $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$, $P = [\max(F_o^2,0) + 2F_c^2] / 3$, F_0 and F_c are the observed and calculated structure factor amplitudes, respectively; ultimately the suggested *a* (=0.1180) and *b* (= 0.) were used in the final refinement.

Crystal data and numerical details on data collection and refinement are given in Table 1. Final fractional atomic coordinates, equivalent displacement parameters and anisotropic displacement parameters for the non-hydrogen

atoms are given in Table 2. Molecular geometry data are collected in Table 3. Neutral atom scattering factors and anomalous dispersion corrections were taken from *International Tables for Crystallography.*[10]

All refinement calculations and graphics were performed on a Pentium-III / Debian-Linux computer at the University of Groningen with the program packages *SHELXL*[11] (least-square refinements), a locally modified version of the program *PLUTO*[12] (preparation of illustrations) and *PLATON*[9] package (checking the final results for missed symmetry with the *MISSYM* option, solvent accessible voids with the *SOLV* option, calculation of geometric data and the *ORTEP*[9] illustrations).

Each asymmetric unit contains one formula unit, consisting of two moieties: a cationic Pd-complex and fluorinated-tetraphenylborate anion, with no atom setting at special position. The triclinic unit cell contains four discrete units, two cations and two anions moieties separated by normal van der Waals distances[13].

No classic hydrogen bonds, no missed symmetry (*MISSYM*), but a small solvent-accessible area (voids of 14.20 $Å^3$ / unit cell) were detected by procedures implemented in *PLATON.*[14,15]

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Table 1.

a. Crystal data and details of the structure determination.

Moiety_Formula	$[C_{34}H_{53}N_2OPd]^+.[C_{32}H_{12}BF_{24}]^-$
Formula_Weight, g.mol^{-1}	1475.45
Crystal system	triclinic
Space group, no.[16]	*P*-1, 2
a, Å	12.5293(8)
b, Å	15.592(1)
c, Å	18.468(1)
α, deg	77.686(1)
β, deg	70.452(1)
γ, deg	82.915(1)
V, Å^3	3316.4(4)
Θ range unit cell: min.-max., deg; reflections	2.38 - 21.67 ; 5277
Formula_*Z*	2
SpaceGroup_*Z*	2
Z' (= Formula_Z / SpaceGroup_*Z)*	1
ρ_{calc}, g.cm^{-3}	1.478
F(000), electrons	1500
μ(Mo K$\bar{\alpha}$), cm^{-1}	3.9
Color, habit	reddish, platelet
Approx. crystal dimension, mm	0.20 x 0.11 x 0.035

b. Data collection.

λ(Mo K$\bar{\alpha}$), Å	0.71073
Monochromator	Graphite
Measurement device type	*CCD* area-detector diffractometer
Detector Area resolution (pixels / mm)	4096 x 4096 / 62 x 62 (binned 512)
Temperature, K	100(1)
Measurement method	φ- and ω-scans
θ range; min. max., deg	2.38, 25.03
Index ranges	h: -14→14; k: -18→18; l: -21→21
Min.- Max. absorption transmission factor	0.8962 – 0.9865
X-ray exposure time, h	18.8
Total data	24071
Unique data	11523
Data with criterion: ($F_o \geq 4.0\ \sigma\ (F_o)$)	5617
$R_{int} = \sum [\lvert F_o^2 - F_o^2\ (\text{mean})\rvert] / \sum [F_o^2]$	0.0556
$R_{sig} = \sum \sigma(F_o^2) / \sum [F_o^2]$	0.1149

c. Refinement.

Number of reflections	11523
Number of refined parameters	962
Number of restraints	112
Final agreement factors:	
$wR(F^2) = [\sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)^2]]^{1/2}$	0.2318
Weighting scheme: *a, b*	0.1180, 0.
$w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$	
And $P = [\max(F_o^2,0) + 2F_c^2] / 3$	
$R(F) = \sum (\|\|F_o\| - \|F_c\|\|) / \sum \|F_o\|$	0.0786
For $F_o > 4.0\ \sigma\ (F_o)$	
$GooF = S = [\sum [w(F_o^2 - F_c^2)^2] / (n-p)]^{1/2}$	1.041
n = number of reflections	
p = number of parameters refined	
Residual electron density in final	
Difference Fourier map, e/Å^3	-1.64, 0.80(9)
Max. (shift/σ) final cycle	0.096
Average (shift/σ) final cycle	0.002

Table 2. Final fractional atomic coordinates and equivalent isotropic displacement parameters with s.u.'s in parentheses.

Atoms of the Asymmetric Unit.

Non-Hydrogen parameters

Residue: 1.

Atom	*x*	*y*	*z*	*s.o.f.*	U_{eq} (Å^2)
Pd1	0.24370(5)	0.27480(5)	0.18411(3)	1.0(-)	0.0664(3)
O11a	0.3706(7)	0.3108(5)	0.0849(5)	0.603(10)	0.061(3)
N11	0.1075(4)	0.2200(3)	0.2750(3)	1.0(-)	0.0394(17)
N12	0.2014(4)	0.3655(3)	0.2610(3)	1.0(-)	0.0384(17)
C129a	0.2562(12)	0.1632(9)	0.1266(5)	0.603(10)	0.062(5)
C130a	0.2951(10)	0.2070(7)	0.0404(5)	0.603(10)	0.059(4)
C131a	0.3216(15)	0.1385(12)	-0.0139(9)	0.603(10)	0.063(6)
C132a	0.3991(12)	0.2591(13)	0.0238(9)	0.603(10)	0.059(5)
C133a	0.4481(13)	0.3732(11)	0.0729(10)	0.603(10)	0.081(6)
C134a	0.5618(17)	0.3414(16)	0.0648(14)	0.603(10)	0.119(9)
C11	0.0548(6)	0.1429(4)	0.2733(4)	1.0(-)	0.044(3)
C12	-0.0239(6)	0.1548(5)	0.2330(4)	1.0(-)	0.053(3)
C13	-0.0722(7)	0.0811(6)	0.2307(5)	1.0(-)	0.070(3)
C14	-0.0418(7)	-0.0005(6)	0.2654(6)	1.0(-)	0.078(4)
C15	0.0344(7)	-0.0121(5)	0.3036(5)	1.0(-)	0.072(3)
C16	0.0852(6)	0.0610(5)	0.3098(4)	1.0(-)	0.057(3)
C17	-0.0574(8)	0.2458(6)	0.1936(5)	1.0(-)	0.074(4)
C18	-0.1806(11)	0.2680(7)	0.2259(7)	1.0(-)	0.135(6)
C19	-0.0344(11)	0.2479(10)	0.1073(6)	1.0(-)	0.179(8)
C110	0.1688(7)	0.0479(5)	0.3557(5)	1.0(-)	0.068(3)
C111	0.2895(8)	0.0438(8)	0.3051(7)	1.0(-)	0.120(5)
C112	0.1470(11)	-0.0356(7)	0.4213(8)	1.0(-)	0.142(7)
C113	0.0663(5)	0.2605(4)	0.3326(3)	1.0(-)	0.033(2)
C114	-0.0307(5)	0.2323(4)	0.4043(3)	1.0(-)	0.041(2)
C115	0.1197(5)	0.3449(4)	0.3247(3)	1.0(-)	0.033(2)
C116	0.0729(5)	0.3995(4)	0.3865(4)	1.0(-)	0.040(2)
C117	0.2556(5)	0.4474(4)	0.2433(4)	1.0(-)	0.042(3)
C118	0.3478(5)	0.4497(4)	0.2704(4)	1.0(-)	0.043(2)
C119	0.4072(6)	0.5265(5)	0.2437(4)	1.0(-)	0.049(3)
C120	0.3779(6)	0.5958(5)	0.1935(4)	1.0(-)	0.056(3)
C121	0.2843(6)	0.5924(5)	0.1699(4)	1.0(-)	0.059(3)
C122	0.2224(6)	0.5176(5)	0.1935(4)	1.0(-)	0.050(3)
C123	0.3829(6)	0.3749(4)	0.3265(4)	1.0(-)	0.050(3)
C124	0.4969(7)	0.3309(6)	0.2864(5)	1.0(-)	0.086(3)
C125	0.3900(8)	0.4059(5)	0.3987(5)	1.0(-)	0.078(4)
C126	0.1203(7)	0.5150(5)	0.1685(5)	1.0(-)	0.065(3)
C127	0.0249(7)	0.5747(6)	0.2058(6)	1.0(-)	0.090(4)
C128	0.1473(9)	0.5361(11)	0.0805(6)	1.0(-)	0.164(7)
O11b	0.3166(11)	0.2160(8)	0.0960(8)	0.397(10)	0.057(5)
C129b	0.378(2)	0.3702(15)	0.1060(15)	0.397(10)	0.095(8)
C130b	0.4710(16)	0.3029(15)	0.0749(13)	0.397(10)	0.090(8)

C131b	0.574(3)	0.352(3)	0.017(2)	0.397(10)	0.163(16)
C132b	0.422(2)	0.245(2)	0.0367(16)	0.397(10)	0.086(8)
C133b	0.285(2)	0.1395(12)	0.0852(12)	0.397(10)	0.064(7)
C134b	0.266(3)	0.137(2)	0.0138(15)	0.397(10)	0.087(13)

Residue: 2.

F21	0.1541(4)	0.1292(4)	0.5547(3)	1.0(-)	0.111(2)
F22	0.1610(4)	0.2674(4)	0.5338(3)	1.0(-)	0.103(2)
F23	0.2312(3)	0.1999(3)	0.4411(2)	1.0(-)	0.0707(19)
F24	0.6257(4)	0.1071(4)	0.3751(3)	1.0(-)	0.095(2)
F25	0.7215(3)	0.1602(3)	0.4296(3)	1.0(-)	0.0773(17)
F26	0.6667(4)	0.0319(3)	0.4725(3)	1.0(-)	0.0808(19)
F27a	0.8667(15)	0.3741(10)	0.6460(8)	0.60(2)	0.086(6)
F28a	0.8144(8)	0.3511(10)	0.5523(6)	0.60(2)	0.071(5)
F29a	0.9291(8)	0.2614(7)	0.5931(7)	0.60(2)	0.069(4)
F210	0.7789(4)	0.0657(3)	0.8641(2)	1.0(-)	0.0751(17)
F211	0.7030(4)	0.1747(3)	0.9181(2)	1.0(-)	0.0829(19)
F212	0.6016(4)	0.0734(3)	0.9222(3)	1.0(-)	0.0769(17)
F213	0.3935(5)	-0.1269(3)	0.7960(3)	1.0(-)	0.104(2)
F214	0.4379(5)	-0.0920(3)	0.8852(4)	1.0(-)	0.115(3)
F215	0.2817(4)	-0.1441(3)	0.9108(3)	1.0(-)	0.094(2)
F216	-0.0334(4)	0.0697(4)	0.9355(3)	1.0(-)	0.107(2)
F217	0.0178(4)	0.1924(6)	0.9423(4)	1.0(-)	0.179(4)
F218	-0.0195(3)	0.1787(3)	0.8429(3)	1.0(-)	0.0730(17)
F219	0.3273(5)	0.4756(7)	0.8965(4)	1.0(-)	0.223(6)
F220	0.2059(8)	0.3901(4)	0.9307(3)	1.0(-)	0.169(4)
F221	0.1694(6)	0.5101(5)	0.8895(3)	1.0(-)	0.141(3)
F222	0.2709(7)	0.6177(3)	0.6055(3)	1.0(-)	0.148(4)
F223	0.2137(4)	0.5154(4)	0.5708(3)	1.0(-)	0.111(2)
F224	0.3857(4)	0.5416(3)	0.5285(3)	1.0(-)	0.0759(17)
C21	0.4329(5)	0.2002(4)	0.6207(3)	1.0(-)	0.0303(17)
C22	0.3336(5)	0.2064(4)	0.6007(3)	1.0(-)	0.036(2)
C23	0.3315(5)	0.1845(4)	0.5324(4)	1.0(-)	0.039(2)
C24	0.4280(6)	0.1549(4)	0.4798(4)	1.0(-)	0.042(2)
C25	0.5276(5)	0.1467(4)	0.4975(3)	1.0(-)	0.036(2)
C26	0.5288(5)	0.1691(4)	0.5667(3)	1.0(-)	0.034(2)
C27	0.2208(6)	0.1925(5)	0.5158(4)	1.0(-)	0.053(3)
C28	0.6340(6)	0.1121(5)	0.4435(4)	1.0(-)	0.051(3)
C29	0.5473(5)	0.2169(4)	0.7196(3)	1.0(-)	0.0306(19)
C210	0.6374(5)	0.2650(4)	0.6660(3)	1.0(-)	0.033(2)
C211	0.7406(5)	0.2641(4)	0.6781(3)	1.0(-)	0.035(2)
C212	0.7591(5)	0.2161(4)	0.7457(4)	1.0(-)	0.039(2)
C213	0.6709(5)	0.1700(4)	0.8015(3)	1.0(-)	0.034(2)
C214	0.5680(5)	0.1703(4)	0.7883(3)	1.0(-)	0.0321(19)
C215	0.8341(6)	0.3169(5)	0.6192(4)	1.0(-)	0.048(3)
C216	0.6871(6)	0.1220(5)	0.8758(4)	1.0(-)	0.046(3)
C217	0.3382(5)	0.1520(4)	0.7695(3)	1.0(-)	0.035(2)
C218	0.3734(5)	0.0626(4)	0.7867(3)	1.0(-)	0.036(2)
C219	0.3055(6)	0.0007(4)	0.8394(4)	1.0(-)	0.041(2)
C220	0.1933(6)	0.0241(6)	0.8768(4)	1.0(-)	0.052(3)

C221	0.1528(5)	0.1084(6)	0.8602(4)	1.0(-)	0.047(3)
C222	0.2236(5)	0.1720(4)	0.8076(4)	1.0(-)	0.042(2)
C223	0.3535(7)	-0.0902(5)	0.8559(5)	1.0(-)	0.056(3)
C224	0.0290(7)	0.1401(7)	0.8971(4)	1.0(-)	0.071(3)
C225	0.3729(5)	0.3242(4)	0.7118(4)	1.0(-)	0.033(2)
C226	0.3364(5)	0.3496(4)	0.7864(4)	1.0(-)	0.043(3)
C227	0.2898(5)	0.4329(4)	0.7964(4)	1.0(-)	0.043(2)
C228	0.2776(6)	0.4952(5)	0.7336(4)	1.0(-)	0.048(3)
C229	0.3134(5)	0.4731(4)	0.6609(4)	1.0(-)	0.042(2)
C230	0.3596(5)	0.3894(4)	0.6499(4)	1.0(-)	0.035(2)
C231	0.2560(8)	0.4537(5)	0.8745(5)	1.0(-)	0.061(3)
C232	0.2988(7)	0.5385(5)	0.5928(5)	1.0(-)	0.061(3)
B2	0.4248(6)	0.2230(5)	0.7048(4)	1.0(-)	0.035(2)
F27b	0.7958(11)	0.4045(11)	0.6022(14)	0.41(2)	0.085(7)
F28b	0.881(2)	0.2915(17)	0.5576(11)	0.41(2)	0.099(10)
F29b	0.9167(15)	0.3296(18)	0.6449(11)	0.41(2)	0.077(8)

Hydrogen parameters:

Residue: 1.

H13	-0.12729(-)	0.08707(-)	0.20476(-)	1.0(-)	0.08385(-)
H14	-0.07566(-)	-0.05028(-)	0.26227(-)	1.0(-)	0.09323(-)
H15	0.05433(-)	-0.06968(-)	0.32669(-)	1.0(-)	0.08609(-)
H17	-0.01329(-)	0.29112(-)	0.20041(-)	1.0(-)	0.08915(-)
H18	-0.22293(-)	0.23228(-)	0.20835(-)	1.0(-)	0.20174(-)
H18'	-0.19664(-)	0.33042(-)	0.20748(-)	1.0(-)	0.20174(-)
H18"	-0.20367(-)	0.25584(-)	0.28311(-)	1.0(-)	0.20174(-)
H19	-0.10118(-)	0.22950(-)	0.09952(-)	1.0(-)	0.26853(-)
H19'	0.03098(-)	0.20786(-)	0.08830(-)	1.0(-)	0.26853(-)
H19"	-0.01828(-)	0.30784(-)	0.07835(-)	1.0(-)	0.26853(-)
H110	0.15745(-)	0.09988(-)	0.38172(-)	1.0(-)	0.08101(-)
H111	0.30172(-)	-0.00317(-)	0.27482(-)	1.0(-)	0.17942(-)
H111'	0.33907(-)	0.03159(-)	0.33780(-)	1.0(-)	0.17942(-)
H111"	0.30703(-)	0.10014(-)	0.26930(-)	1.0(-)	0.17942(-)
H112	0.16167(-)	-0.08820(-)	0.39762(-)	1.0(-)	0.21311(-)
H112'	0.06794(-)	-0.03285(-)	0.45521(-)	1.0(-)	0.21311(-)
H112"	0.19762(-)	-0.03821(-)	0.45239(-)	1.0(-)	0.21311(-)
H114	-0.05397(-)	0.17520(-)	0.40257(-)	1.0(-)	0.06211(-)
H114'	-0.09456(-)	0.27597(-)	0.40664(-)	1.0(-)	0.06211(-)
H114"	-0.00733(-)	0.22721(-)	0.45069(-)	1.0(-)	0.06211(-)
H116	0.11550(-)	0.45261(-)	0.37165(-)	1.0(-)	0.05901(-)
H116'	0.07949(-)	0.36570(-)	0.43614(-)	1.0(-)	0.05901(-)
H116"	-0.00716(-)	0.41618(-)	0.39225(-)	1.0(-)	0.05901(-)
H119	0.46964(-)	0.53055(-)	0.26081(-)	1.0(-)	0.05888(-)
H120	0.42140(-)	0.64632(-)	0.17468(-)	1.0(-)	0.06683(-)
H121	0.26228(-)	0.64207(-)	0.13701(-)	1.0(-)	0.07008(-)
H123	0.32439(-)	0.33008(-)	0.34493(-)	1.0(-)	0.06023(-)
H124	0.48701(-)	0.29535(-)	0.25148(-)	1.0(-)	0.12817(-)
H124'	0.52623(-)	0.29303(-)	0.32595(-)	1.0(-)	0.12817(-)
H124"	0.55068(-)	0.37604(-)	0.25591(-)	1.0(-)	0.12817(-)
H125	0.44936(-)	0.44802(-)	0.38210(-)	1.0(-)	0.11640(-)

H125'	0.40812(-)	0.35519(-)	0.43507(-)	1.0(-)	0.11640(-)
H125"	0.31691(-)	0.43427(-)	0.42471(-)	1.0(-)	0.11640(-)
H126	0.09409(-)	0.45368(-)	0.18667(-)	1.0(-)	0.07773(-)
H127	0.01012(-)	0.56220(-)	0.26244(-)	1.0(-)	0.13402(-)
H127'	-0.04325(-)	0.56540(-)	0.19431(-)	1.0(-)	0.13402(-)
H127"	0.04522(-)	0.63593(-)	0.18505(-)	1.0(-)	0.13402(-)
H128	0.17277(-)	0.59613(-)	0.06078(-)	1.0(-)	0.24604(-)
H128'	0.07923(-)	0.53129(-)	0.06704(-)	1.0(-)	0.24604(-)
H128"	0.20752(-)	0.49460(-)	0.05662(-)	1.0(-)	0.24604(-)
H1291	0.31271(-)	0.11712(-)	0.13890(-)	0.603(10)	0.07361(-)
H1291'	0.18198(-)	0.13712(-)	0.14160(-)	0.603(10)	0.07361(-)
H1292	0.35390(-)	0.40920(-)	0.06391(-)	0.397(10)	0.11395(-)
H1292'	0.40096(-)	0.40598(-)	0.13621(-)	0.397(10)	0.11395(-)
H1301	0.23258(-)	0.24900(-)	0.02989(-)	0.603(10)	0.07120(-)
H1302	0.49334(-)	0.26538(-)	0.11930(-)	0.397(10)	0.10854(-)
H1311	0.38268(-)	0.09674(-)	-0.00431(-)	0.603(10)	0.09421(-)
H1311'	0.34525(-)	0.16866(-)	-0.06858(-)	0.603(10)	0.09421(-)
H1311"	0.25346(-)	0.10678(-)	-0.00311(-)	0.603(10)	0.09421(-)
H1312	0.55119(-)	0.41415(-)	0.00237(-)	0.397(10)	0.24700(-)
H1312'	0.60403(-)	0.32571(-)	-0.03008(-)	0.397(10)	0.24700(-)
H1312"	0.63350(-)	0.34824(-)	0.04145(-)	0.397(10)	0.24700(-)
H1321	0.46628(-)	0.21872(-)	0.02504(-)	0.603(10)	0.07144(-)
H1321'	0.41601(-)	0.29778(-)	-0.02820(-)	0.603(10)	0.07144(-)
H1322	0.40778(-)	0.27882(-)	-0.01122(-)	0.397(10)	0.10278(-)
H1322'	0.47501(-)	0.19360(-)	0.02293(-)	0.397(10)	0.10278(-)
H1331	0.42112(-)	0.40500(-)	0.11734(-)	0.603(10)	0.09721(-)
H1331'	0.44783(-)	0.41655(-)	0.02518(-)	0.603(10)	0.09721(-)
H1332	0.21465(-)	0.12266(-)	0.12825(-)	0.397(10)	0.07610(-)
H1332'	0.34462(-)	0.09309(-)	0.09158(-)	0.397(10)	0.07610(-)
H1341	0.56699(-)	0.31052(-)	0.11568(-)	0.603(10)	0.17814(-)
H1341'	0.61130(-)	0.39088(-)	0.04532(-)	0.603(10)	0.17814(-)
H1341"	0.58554(-)	0.30078(-)	0.02769(-)	0.603(10)	0.17814(-)
H1342	0.19811(-)	0.17407(-)	0.01076(-)	0.397(10)	0.12972(-)
H1342'	0.25622(-)	0.07667(-)	0.01176(-)	0.397(10)	0.12972(-)
H1342"	0.33182(-)	0.15978(-)	-0.03029(-)	0.397(10)	0.12972(-)

Residue: 2.

H22	0.26475(-)	0.22642(-)	0.63549(-)	1.0(-)	0.04284(-)
H24	0.42615(-)	0.14052(-)	0.43279(-)	1.0(-)	0.04974(-)
H26	0.59872(-)	0.16261(-)	0.57732(-)	1.0(-)	0.04035(-)
H210	0.62724(-)	0.29959(-)	0.61952(-)	1.0(-)	0.03858(-)
H212	0.83053(-)	0.21479(-)	0.75347(-)	1.0(-)	0.04642(-)
H214	0.50904(-)	0.13764(-)	0.82717(-)	1.0(-)	0.03869(-)
H218	0.44906(-)	0.04462(-)	0.75984(-)	1.0(-)	0.04325(-)
H220	0.14497(-)	-0.01851(-)	0.91375(-)	1.0(-)	0.06157(-)
H222	0.19331(-)	0.23056(-)	0.79740(-)	1.0(-)	0.04953(-)
H226	0.34435(-)	0.30807(-)	0.83038(-)	1.0(-)	0.05177(-)
H228	0.24492(-)	0.55228(-)	0.74074(-)	1.0(-)	0.05757(-)
H230	0.38279(-)	0.37596(-)	0.59888(-)	1.0(-)	0.04235(-)

*) $U_{eq} = 1/3 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a_i}.\mathbf{a_j}$ [17]

Anisotropic (displacement) parameters (Å^2)

Residue: 1.

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Pd1	0.0454(4)	0.1112(6)	0.0380(4)	-0.0267(3)	-0.0056(3)	0.0141(3)
O11a	0.063(6)	0.059(6)	0.054(5)	-0.027(4)	0.008(4)	-0.020(4)
N11	0.033(3)	0.052(3)	0.035(3)	-0.018(3)	-0.003(2)	-0.014(3)
N12	0.032(3)	0.051(3)	0.035(3)	-0.013(3)	-0.008(3)	-0.012(3)
C129a	0.067(9)	0.061(9)	0.045(7)	-0.015(7)	0.010(7)	-0.031(7)
C130a	0.071(8)	0.055(8)	0.049(7)	-0.008(6)	-0.011(6)	-0.021(6)
C131a	0.077(12)	0.074(11)	0.043(9)	-0.012(8)	-0.018(8)	-0.028(10)
C132a	0.076(9)	0.053(8)	0.048(8)	-0.017(6)	-0.007(7)	-0.022(7)
C133a	0.068(9)	0.08(1)	0.098(10)	-0.068(8)	0.000(9)	0.002(8)
C134a	0.090(13)	0.132(16)	0.142(19)	-0.016(15)	-0.052(14)	-0.010(13)
C11	0.046(4)	0.052(5)	0.036(4)	-0.019(3)	-0.009(3)	-0.008(4)
C12	0.054(5)	0.072(5)	0.045(4)	-0.017(4)	-0.025(4)	-0.014(4)
C13	0.065(6)	0.081(6)	0.091(6)	-0.044(5)	-0.044(5)	-0.003(5)
C14	0.075(6)	0.072(6)	0.112(7)	-0.053(6)	-0.043(6)	0.000(5)
C15	0.075(6)	0.065(5)	0.099(7)	-0.047(5)	-0.046(5)	0.016(4)
C16	0.053(5)	0.060(5)	0.069(5)	-0.037(4)	-0.020(4)	0.002(4)
C17	0.090(7)	0.092(7)	0.061(5)	0.002(5)	-0.049(5)	-0.036(6)
C18	0.138(12)	0.071(7)	0.151(11)	-0.017(7)	-0.005(9)	0.034(7)
C19	0.152(12)	0.277(18)	0.053(7)	0.002(9)	-0.019(7)	0.101(12)
C110	0.075(6)	0.054(5)	0.102(7)	-0.045(5)	-0.058(5)	0.029(4)
C111	0.064(7)	0.181(11)	0.153(10)	-0.100(9)	-0.059(7)	0.039(7)
C112	0.176(12)	0.105(9)	0.198(13)	0.015(9)	-0.151(12)	-0.014(8)
C113	0.031(4)	0.037(4)	0.035(4)	-0.010(3)	-0.013(3)	-0.003(3)
C114	0.040(4)	0.044(4)	0.043(4)	-0.015(3)	-0.012(3)	-0.004(3)
C115	0.027(3)	0.041(4)	0.035(4)	-0.014(3)	-0.011(3)	-0.002(3)
C116	0.036(4)	0.041(4)	0.041(4)	-0.009(3)	-0.009(3)	-0.004(3)
C117	0.023(4)	0.061(5)	0.038(4)	-0.011(4)	0.001(3)	-0.013(3)
C118	0.037(4)	0.049(4)	0.040(4)	-0.004(3)	-0.008(3)	-0.010(3)
C119	0.038(4)	0.056(5)	0.050(4)	-0.002(4)	-0.012(4)	-0.012(4)
C120	0.042(5)	0.058(5)	0.063(5)	-0.005(4)	-0.012(4)	-0.014(4)
C121	0.049(5)	0.058(5)	0.059(5)	0.005(4)	-0.012(4)	-0.010(4)
C122	0.038(4)	0.056(5)	0.052(4)	-0.004(4)	-0.014(4)	-0.004(4)
C123	0.048(5)	0.054(5)	0.056(5)	-0.008(4)	-0.022(4)	-0.020(4)
C124	0.072(6)	0.086(6)	0.080(6)	0.000(5)	-0.020(5)	0.026(5)
C125	0.088(7)	0.085(6)	0.076(6)	0.002(5)	-0.046(5)	-0.035(5)
C126	0.057(5)	0.071(5)	0.074(6)	0.000(4)	-0.037(5)	-0.006(4)
C127	0.054(6)	0.114(8)	0.106(7)	-0.004(6)	-0.042(6)	-0.003(5)
C128	0.061(7)	0.38(2)	0.068(7)	-0.073(10)	-0.023(6)	-0.016(9)
O11b	0.067(8)	0.051(8)	0.052(8)	-0.032(7)	-0.004(7)	0.002(6)
C129b	0.081(15)	0.093(15)	0.090(14)	-0.070(12)	0.037(12)	-0.019(13)
C130b	0.058(13)	0.094(14)	0.097(13)	-0.048(11)	0.03(1)	-0.026(10)
C131b	0.10(2)	0.21(3)	0.14(3)	-0.04(2)	0.05(2)	-0.098(18)
C132b	0.081(13)	0.077(13)	0.076(13)	-0.038(11)	0.020(11)	-0.004(10)

C133b	0.091(13)	0.065(11)	0.045(11)	-0.026(10)	-0.029(10)	0.005(10)
C134b	0.13(3)	0.093(19)	0.050(17)	-0.022(14)	-0.047(16)	0.022(17)

Residue: 2.

F21	0.062(3)	0.158(5)	0.111(4)	0.061(4)	-0.056(3)	-0.067(4)
F22	0.075(3)	0.151(5)	0.120(4)	-0.071(4)	-0.072(3)	0.048(3)
F23	0.059(3)	0.114(4)	0.057(3)	-0.013(2)	-0.040(2)	-0.015(2)
F24	0.071(3)	0.164(5)	0.061(3)	-0.058(3)	-0.023(3)	0.018(3)
F25	0.048(3)	0.083(3)	0.091(3)	-0.043(3)	0.012(2)	-0.017(2)
F26	0.079(3)	0.055(3)	0.096(4)	-0.021(3)	-0.016(3)	0.021(2)
F27a	0.084(13)	0.087(10)	0.073(8)	-0.047(9)	0.036(9)	-0.067(9)
F28a	0.040(6)	0.107(11)	0.045(7)	0.034(8)	-0.008(5)	-0.026(6)
F29a	0.032(5)	0.088(7)	0.054(7)	0.010(6)	0.010(4)	0.007(4)
F210	0.065(3)	0.091(3)	0.064(3)	0.006(2)	-0.035(2)	0.021(3)
F211	0.131(4)	0.084(3)	0.050(3)	-0.008(2)	-0.049(3)	-0.017(3)
F212	0.065(3)	0.099(3)	0.062(3)	0.038(3)	-0.037(2)	-0.032(3)
F213	0.181(6)	0.056(3)	0.065(3)	-0.009(3)	-0.037(4)	0.023(3)
F214	0.121(5)	0.075(3)	0.183(6)	0.014(3)	-0.113(5)	-0.018(3)
F215	0.101(4)	0.067(3)	0.093(4)	0.024(3)	-0.017(3)	-0.032(3)
F216	0.041(3)	0.161(5)	0.078(3)	0.055(4)	-0.006(2)	-0.023(3)
F217	0.046(3)	0.372(11)	0.170(6)	-0.214(8)	-0.006(4)	0.005(5)
F218	0.039(3)	0.080(3)	0.084(3)	0.001(3)	-0.008(2)	-0.005(2)
F219	0.075(4)	0.516(16)	0.137(6)	-0.232(9)	0.022(4)	-0.095(7)
F220	0.299(10)	0.108(5)	0.058(4)	-0.035(4)	0.016(5)	-0.026(6)
F221	0.171(6)	0.168(6)	0.076(4)	-0.071(4)	-0.033(4)	0.088(5)
F222	0.277(9)	0.070(4)	0.082(4)	-0.026(3)	-0.069(5)	0.096(5)
F223	0.067(3)	0.156(5)	0.099(4)	0.035(4)	-0.048(3)	-0.009(3)
F224	0.073(3)	0.075(3)	0.059(3)	0.012(2)	-0.015(3)	0.013(2)
C21	0.028(3)	0.032(3)	0.027(3)	0.003(3)	-0.008(3)	-0.004(3)
C22	0.030(4)	0.041(4)	0.037(4)	-0.006(3)	-0.011(3)	-0.006(3)
C23	0.040(4)	0.041(4)	0.043(4)	-0.001(3)	-0.025(3)	-0.008(3)
C24	0.047(4)	0.046(4)	0.036(4)	-0.006(3)	-0.017(3)	-0.010(3)
C25	0.035(4)	0.035(4)	0.036(4)	-0.004(3)	-0.012(3)	-0.001(3)
C26	0.036(4)	0.035(4)	0.032(3)	-0.001(3)	-0.016(3)	-0.004(3)
C27	0.036(4)	0.077(6)	0.055(5)	-0.016(4)	-0.024(4)	0.000(4)
C28	0.049(5)	0.068(5)	0.045(4)	-0.020(4)	-0.017(4)	-0.010(4)
C29	0.024(3)	0.037(4)	0.029(3)	-0.008(3)	-0.007(3)	0.004(3)
C210	0.030(4)	0.035(4)	0.035(3)	-0.004(3)	-0.015(3)	-0.002(3)
C211	0.028(4)	0.039(4)	0.033(4)	-0.005(3)	-0.004(3)	-0.008(3)
C212	0.031(4)	0.047(4)	0.041(4)	-0.011(3)	-0.014(3)	-0.002(3)
C213	0.033(4)	0.037(4)	0.031(3)	-0.005(3)	-0.010(3)	0.000(3)
C214	0.025(3)	0.037(4)	0.032(3)	-0.001(3)	-0.007(3)	-0.009(3)
C215	0.032(4)	0.066(6)	0.047(5)	0.000(4)	-0.016(4)	-0.013(4)
C216	0.039(4)	0.056(5)	0.046(4)	0.005(4)	-0.023(4)	-0.007(4)
C217	0.028(4)	0.049(4)	0.031(3)	-0.010(3)	-0.010(3)	-0.009(3)
C218	0.027(3)	0.051(4)	0.035(4)	-0.006(3)	-0.015(3)	-0.010(3)
C219	0.045(4)	0.048(4)	0.037(4)	0.001(3)	-0.020(4)	-0.022(4)
C220	0.041(5)	0.087(6)	0.030(4)	-0.009(4)	-0.008(3)	-0.028(4)
C221	0.026(4)	0.085(6)	0.034(4)	-0.018(4)	-0.008(3)	-0.007(4)
C222	0.036(4)	0.054(4)	0.038(4)	-0.012(3)	-0.012(3)	-0.009(3)

C223	0.058(5)	0.053(5)	0.064(5)	0.012(4)	-0.031(5)	-0.031(4)
C224	0.048(5)	0.134(8)	0.029(4)	-0.014(5)	-0.002(4)	-0.034(5)
C225	0.018(3)	0.041(4)	0.040(4)	-0.008(3)	-0.010(3)	0.001(3)
C226	0.033(4)	0.059(5)	0.037(4)	-0.006(3)	-0.013(3)	0.000(3)
C227	0.035(4)	0.050(4)	0.051(4)	-0.028(4)	-0.016(3)	0.005(3)
C228	0.048(5)	0.049(4)	0.053(5)	-0.015(4)	-0.026(4)	0.015(3)
C229	0.031(4)	0.044(4)	0.048(4)	-0.005(3)	-0.013(3)	0.007(3)
C230	0.027(3)	0.044(4)	0.040(4)	-0.013(3)	-0.015(3)	0.002(3)
C231	0.068(6)	0.054(5)	0.057(5)	-0.016(4)	-0.011(5)	-0.006(5)
C232	0.062(6)	0.055(5)	0.066(6)	-0.009(4)	-0.032(5)	0.025(4)
B2	0.024(4)	0.045(4)	0.037(4)	-0.012(4)	-0.010(3)	-0.001(3)
F27b	0.055(9)	0.082(12)	0.089(15)	0.03(1)	-0.006(9)	-0.027(7)
F28b	0.095(19)	0.13(2)	0.058(13)	-0.055(15)	0.034(14)	-0.058(17)
F29b	0.045(10)	0.131(19)	0.054(10)	-0.005(12)	-0.013(8)	-0.029(9)

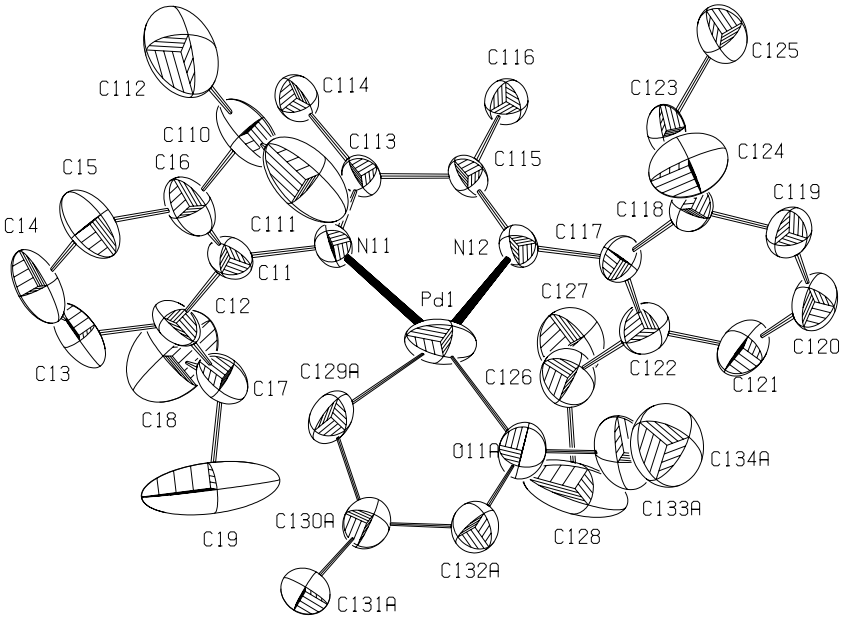
Thermal vibration amplitudes ($Å^2$)

$$F(\mathbf{h}) = F_o(\mathbf{h}) \exp\left(-2\pi^2 \sum_{i=1}^{3}\sum_{j=1}^{3} h_i h_l a_i^* a_j^* U_{ij}\right)$$

or

$$F(\mathbf{h}) = F_o(\mathbf{h}) \exp\left(-8\pi^2 U_{iso} (\sin(\theta)/\lambda)^2\right)$$

Cation of **5**:



Anion of **5**:

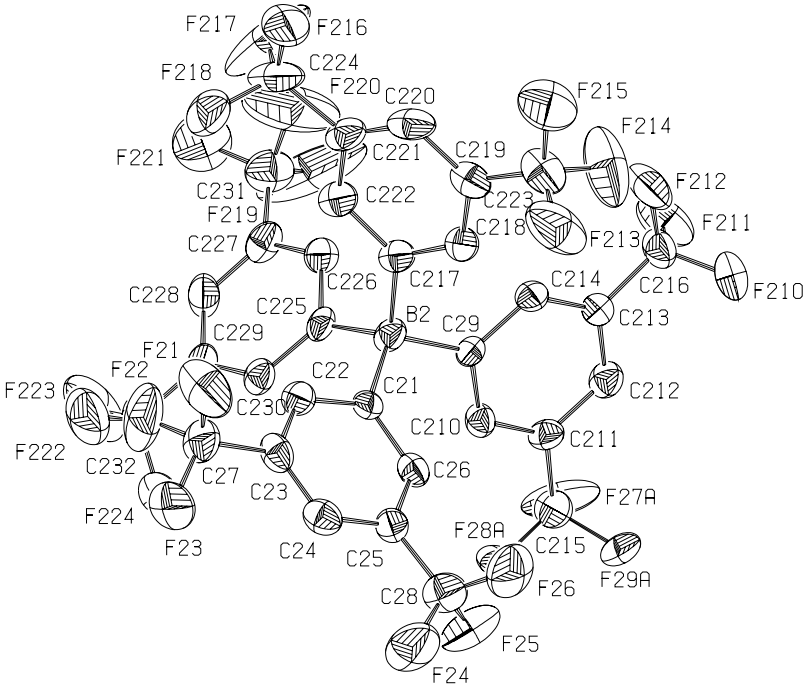


Table 3. Data on the geometry.

Standard deviations in the last decimal place are given in parentheses.

Residue: 1.

Interatomic Distances (Å)

Pd1	-O11a	2.008(9)	C110	-C16	1.525(12)
Pd1	-N11	2.071(5)	C110	-C111	1.490(15)
Pd1	-N12	2.113(5)	C110	-C112	1.559(15)
Pd1	-C129a	2.189(13)	C113	-C114	1.493(8)
Pd1	-O11b	1.939(13)	C113	-C115	1.507(9)
Pd1	-C129b	2.28(3)	C115	-C116	1.488(9)
O11a	-C132a	1.45(2)	C117	-C118	1.411(10)
O11a	-C133a	1.39(2)	C117	-C122	1.39(1)
N11	-C11	1.452(8)	C118	-C119	1.398(10)
N11	-C113	1.279(8)	C118	-C123	1.513(10)
N12	-C115	1.284(8)	C119	-C120	1.365(11)
N12	-C117	1.444(8)	C120	-C121	1.391(11)
C129a	-C130a	1.529(13)	C121	-C122	1.390(11)
C130a	-C131a	1.55(2)	C122	-C126	1.506(12)
C130a	-C132a	1.53(2)	C123	-C124	1.525(12)
C133a	-C134a	1.42(3)	C123	-C125	1.544(11)
C11	-C12	1.396(11)	C126	-C127	1.489(13)
C11	-C16	1.384(10)	C126	-C128	1.515(13)
C12	-C13	1.378(12)	O11b	-C132b	1.46(3)
C12	-C17	1.528(12)	O11b	-C133b	1.38(2)
C13	-C14	1.368(13)	C129b	-C130b	1.52(3)
C14	-C15	1.340(13)	C130b	-C131b	1.54(4)
C15	-C16	1.415(12)	C130b	-C132b	1.55(4)
C17	-C18	1.483(17)	C133b	-C134b	1.43(4)
C17	-C19	1.516(14)			

Bond Angles (deg.)

O11a	-Pd1	-N11	168.8(3)	C12	-C17	-C19	110.2(9)
O11a	-Pd1	-N12	113.8(3)	C18	-C17	-C19	105.8(10)
O11a	-Pd1	-C129a	79.8(4)	C16	-C110	-C111	113.0(8)
N11	-Pd1	-N12	77.11(19)	C16	-C110	-C112	112.4(8)
N11	-Pd1	-C129a	89.4(3)	C111	-C110	-C112	109.1(9)
N11	-Pd1	-O11b	119.9(4)	N11	-C113	-C114	125.5(6)
N11	-Pd1	-C129b	161.9(6)	N11	-C113	-C115	115.9(5)
N12	-Pd1	-C129a	166.4(3)	C114	-C113	-C115	118.6(5)
N12	-Pd1	-O11b	163.0(4)	N12	-C115	-C113	115.0(5)
N12	-Pd1	-C129b	84.9(6)	N12	-C115	-C116	125.1(6)
O11b	-Pd1	-C129b	78.2(7)	C113	-C115	-C116	119.8(5)
Pd1	-O11a	-C132a	119.0(8)	N12	-C117	-C118	118.5(6)
Pd1	-O11a	-C133a	125.8(9)	N12	-C117	-C122	118.3(6)
C132a	-O11a	-C133a	114.3(11)	C118	-C117	-C122	122.8(6)

Pd1	-N11	-C11	123.2(4)	C117	-C118	-C119	116.8(6)
Pd1	-N11	-C113	116.5(4)	C117	-C118	-C123	123.5(6)
C11	-N11	-C113	120.2(5)	C119	-C118	-C123	119.8(6)
Pd1	-N12	-C115	115.4(4)	C118	-C119	-C120	121.7(7)
Pd1	-N12	-C117	123.5(4)	C119	-C120	-C121	120.0(7)
C115	-N12	-C117	121.0(5)	C120	-C121	-C122	121.2(7)
Pd1	-C129a	-C130a	101.5(8)	C117	-C122	-C121	117.5(7)
C129a	-C130a	-C131a	111.5(10)	C117	-C122	-C126	121.7(7)
C129a	-C130a	-C132a	109.3(11)	C121	-C122	-C126	120.8(7)
C131a	-C130a	-C132a	110.8(12)	C118	-C123	-C124	111.3(6)
O11a	-C132a	-C130a	106.0(11)	C118	-C123	-C125	111.9(6)
O11a	-C133a	-C134a	116.6(16)	C124	-C123	-C125	109.1(7)
N11	-C11	-C12	117.9(6)	C122	-C126	-C127	111.6(7)
N11	-C11	-C16	119.6(7)	C122	-C126	-C128	112.6(8)
C12	-C11	-C16	122.5(7)	C127	-C126	-C128	110.1(9)
C11	-C12	-C13	117.5(7)	Pd1	-O11b	-C132b	121.6(14)
C11	-C12	-C17	121.9(7)	Pd1	-O11b	-C133b	126.2(13)
C13	-C12	-C17	120.6(8)	C132b	-O11b	-C133b	111.8(18)
C12	-C13	-C14	120.8(8)	Pd1	-C129b	-C130b	98.0(14)
C13	-C14	-C15	121.8(9)	C129b	-C130b	-C131b	109(2)
C14	-C15	-C16	120.3(8)	C129b	-C130b	-C132b	107(2)
C11	-C16	-C15	117.1(7)	C131b	-C130b	-C132b	113(2)
C11	-C16	-C110	122.7(7)	O11b	-C132b	-C130b	105(2)
C15	-C16	-C110	120.2(7)	O11b	-C133b	-C134b	120(2)
C12	-C17	-C18	111.6(8)				

Residue: 2.

Interatomic Distances (Å)

F21	-C27	1.299(10)	C25	-C28	1.492(10)
F22	-C27	1.349(10)	C29	-B2	1.635(10)
F23	-C27	1.323(8)	C210	-C29	1.400(8)
F24	-C28	1.321(9)	C210	-C211	1.382(9)
F25	-C28	1.328(9)	C211	-C212	1.387(9)
F26	-C28	1.324(9)	C211	-C215	1.496(10)
F27a	-C215	1.273(18)	C212	-C213	1.385(9)
F28a	-C215	1.328(13)	C213	-C214	1.389(9)
F29a	-C215	1.392(14)	C213	-C216	1.482(9)
F210	-C216	1.344(9)	C214	-C29	1.409(8)
F211	-C216	1.320(9)	C217	-C218	1.411(9)
F212	-C216	1.329(9)	C217	-C222	1.400(9)
F213	-C223	1.277(10)	C217	-B2	1.642(9)
F214	-C223	1.335(11)	C218	-C219	1.360(9)
F215	-C223	1.325(10)	C219	-C220	1.385(11)
F216	-C224	1.352(11)	C219	-C223	1.48(1)
F217	-C224	1.251(13)	C220	-C221	1.359(13)
F218	-C224	1.339(10)	C221	-C222	1.394(10)
F219	-C231	1.207(13)	C221	-C224	1.537(12)
F220	-C231	1.317(11)	C225	-C226	1.425(10)
F221	-C231	1.302(12)	C225	-C230	1.399(9)

F222	-C232	1.290(9)	C225	-B2	1.645(10)
F223	-C232	1.371(11)	C226	-C227	1.380(9)
F224	-C232	1.312(10)	C227	-C228	1.383(10)
C21	-C22	1.399(9)	C227	-C231	1.457(11)
C21	-C26	1.391(8)	C228	-C229	1.371(10)
C21	-B2	1.634(9)	C229	-C230	1.388(9)
C22	-C23	1.384(9)	C229	-C232	1.489(11)
C23	-C24	1.373(10)	F27b	-C215	1.397(19)
C23	-C27	1.503(10)	F28b	-C215	1.22(2)
C24	-C25	1.377(10)	F29b	-C215	1.33(2)
C25	-C26	1.400(8)			

Bond Angles (deg.)

C22	-C21	-C26	114.3(5)	F210	-C216	-F212	105.0(6)
C22	-C21	-B2	118.7(5)	F210	-C216	-C213	112.3(5)
C26	-C21	-B2	126.8(6)	F211	-C216	-F212	106.3(6)
C21	-C22	-C23	122.7(6)	F211	-C216	-C213	112.9(6)
C22	-C23	-C24	121.6(6)	F212	-C216	-C213	114.4(6)
C22	-C23	-C27	119.2(6)	C218	-C217	-C222	114.4(5)
C24	-C23	-C27	119.2(6)	C218	-C217	-B2	121.2(5)
C23	-C24	-C25	117.8(6)	C222	-C217	-B2	124.2(6)
C24	-C25	-C26	120.2(6)	C217	-C218	-C219	123.9(6)
C24	-C25	-C28	119.8(6)	C218	-C219	-C220	119.4(7)
C26	-C25	-C28	120.0(6)	C218	-C219	-C223	119.2(7)
C21	-C26	-C25	123.4(6)	C220	-C219	-C223	121.4(7)
F21	-C27	-F22	105.9(6)	C219	-C220	-C221	119.5(7)
F21	-C27	-F23	107.5(6)	C220	-C221	-C222	120.8(7)
F21	-C27	-C23	114.4(6)	C220	-C221	-C224	123.3(7)
F22	-C27	-F23	102.6(6)	C222	-C221	-C224	115.9(8)
F22	-C27	-C23	111.0(6)	C217	-C222	-C221	121.8(6)
F23	-C27	-C23	114.4(6)	F213	-C223	-F214	106.7(7)
F24	-C28	-F25	107.3(6)	F213	-C223	-F215	107.3(7)
F24	-C28	-F26	105.7(6)	F213	-C223	-C219	114.1(7)
F24	-C28	-C25	113.7(6)	F214	-C223	-F215	102.8(7)
F25	-C28	-F26	104.6(6)	F214	-C223	-C219	110.9(7)
F25	-C28	-C25	112.4(6)	F215	-C223	-C219	114.2(7)
F26	-C28	-C25	112.6(6)	F216	-C224	-F217	109.9(7)
C210	-C29	-C214	115.1(6)	F216	-C224	-F218	103.7(7)
C210	-C29	-B2	121.0(5)	F216	-C224	-C221	109.2(8)
C214	-C29	-B2	123.8(5)	F217	-C224	-F218	108.1(9)
C29	-C210	-C211	122.5(5)	F217	-C224	-C221	113.5(8)
C210	-C211	-C212	121.0(6)	F218	-C224	-C221	111.9(6)
C210	-C211	-C215	120.5(5)	C226	-C225	-C230	115.6(6)
C212	-C211	-C215	118.5(6)	C226	-C225	-B2	118.9(6)
C211	-C212	-C213	118.4(6)	C230	-C225	-B2	125.5(6)
C212	-C213	-C214	120.2(5)	C225	-C226	-C227	121.7(6)
C212	-C213	-C216	119.2(6)	C226	-C227	-C228	120.8(6)
C214	-C213	-C216	120.6(6)	C226	-C227	-C231	118.1(6)
C29	-C214	-C213	122.8(5)	C228	-C227	-C231	121.1(6)
F27a	-C215	-F28a	111.7(11)	C227	-C228	-C229	118.9(7)

F27a	-C215	-F29a	106.0(11)	C228	-C229	-C230	121.1(6)
F27a	-C215	-C211	113.9(9)	C228	-C229	-C232	119.8(6)
F28a	-C215	-F29a	100.4(9)	C230	-C229	-C232	119.1(6)
F28a	-C215	-C211	114.1(8)	C225	-C230	-C229	121.9(6)
F29a	-C215	-C211	109.5(7)	F219	-C231	-F220	104.5(9)
C211	-C215	-F27b	110.5(10)	F219	-C231	-F221	107.5(8)
C211	-C215	-F28b	117.3(14)	F219	-C231	-C227	118.7(9)
C211	-C215	-F29b	115.1(10)	F220	-C231	-F221	94.6(8)
F27b	-C215	-F28b	108.1(16)	F220	-C231	-C227	114.2(7)
F27b	-C215	-F29b	98.5(15)	F221	-C231	-C227	114.3(8)
F28b	-C215	-F29b	105.7(16)	F222	-C232	-F223	105.6(8)
F210	-C216	-F211	105.2(6)	F222	-C232	-F224	108.0(7)
F222	-C232	-C229	114.9(7)	C21	-B2	-C217	104.2(5)
F223	-C232	-F224	102.5(7)	C21	-B2	-C225	111.1(5)
F223	-C232	-C229	109.9(7)	C29	-B2	-C217	112.0(5)
F224	-C232	-C229	114.9(7)	C29	-B2	-C225	105.3(5)
C21	-B2	-C29	113.9(5)	C217	-B2	-C225	110.5(5)