Supporting Information: Supplementary Information for Self-Assembly Motifs of Water in Crystals of Palladium β-Amino Acid Complexes Influenced by Methyl Substitution on the Amino Acid β-Carbon.

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Contents

1 Table of Results from DFT Calculations

Compound	Solvent	ΔG (hartrees)	ΔΔG (trans-cis) (hartrees)	ΔΔG (kJ/mol)
cis-compound 1	None	-772.827162	0	0
cis-compound 2	None	-851.398244	0	0
cis-compound 3	None	-929.963847	0	0
trans-compound 1	None	-772.86408	-0.03692	-19.31472
trans-compound 2	None	-851.433915	-0.035671	-21.02685
trans-compound 3	None	-930.000513	-0.036666	-21.81848
cis-compound 1	water	-772.902187	0	0
cis-compound 2	water	-851.472101	0	0
cis-compound 3	water	-930.034208	0	0
trans-compound 1	water	-772.909531	-0.007344	-97.0996
trans-compound 2	water	-851.480096	-0.007995	-93.81473
trans-compound 3	water	-930.042504	-0.008296	-96.43158

DFT calculations using the WebMO interface[S1] to Gaussian 09[S2] at the B3LYP level of theory[S3,S4] using the lanl2dz basis set[S5] were carried out on the two isomers of all compounds.

References

(S1) Schmidt, J.; Polik, W. WebMO Enterprise. `https://www.webmo.net/`.

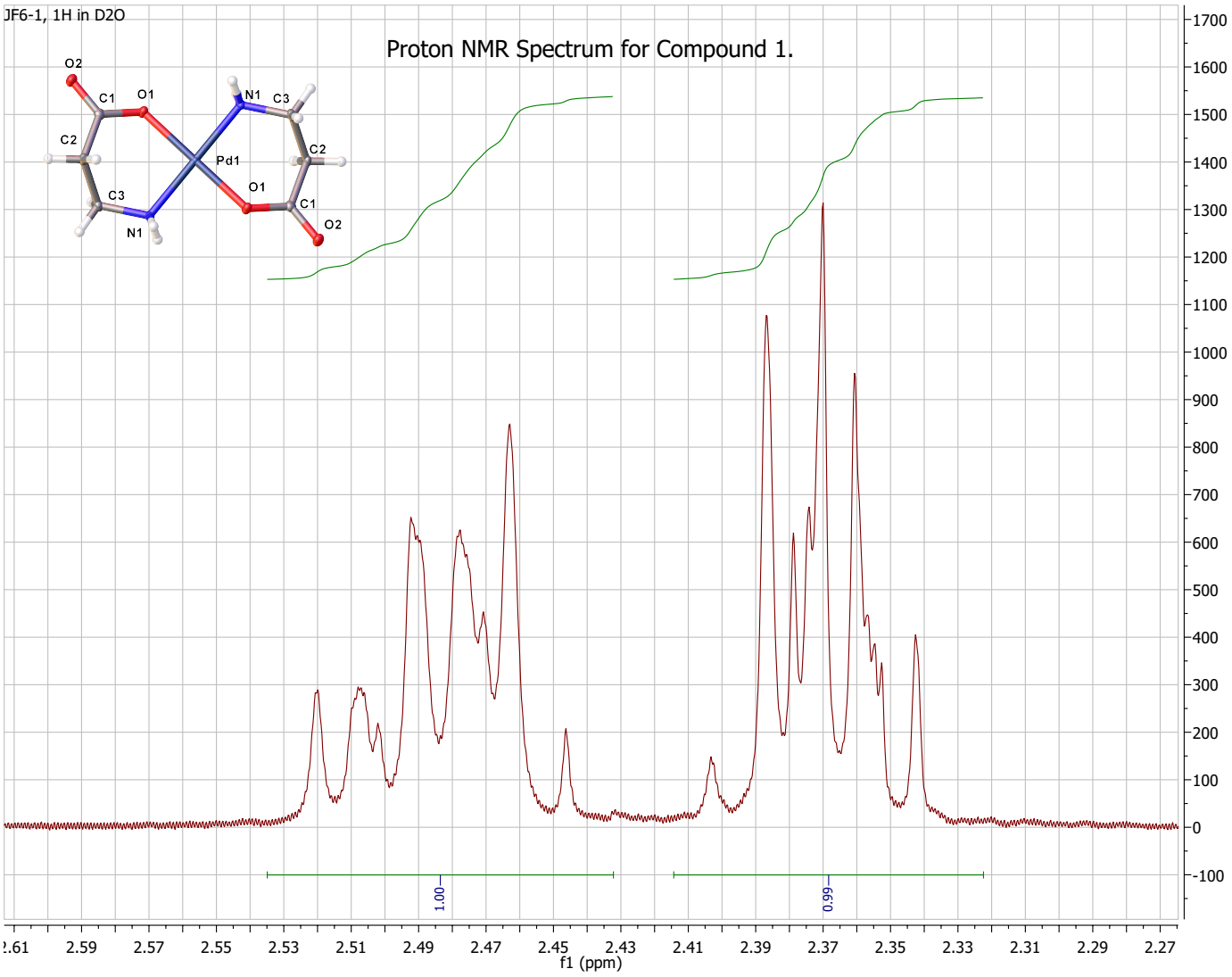
(S2) Frisch, M. J.; Trucks, G. W.; Schlegel, H. B.; Scuseria, G. E.; Robb, M. A.; Cheeseman, J. R.; Scalmani, G.; Barone, V.; Petersson, G. A.; Nakatsuji, H.; Li, X.; Caricato, M.; Marenich, A.; Bloino, J.; Janesko, B. G.; Gomperts, R.; Mennucci, B.; Hratchian, H. P.; Ortiz, J. V.; Izmaylov, A. F.; Sonnenberg, J. L.; Williams-Young, D.; Ding, F.; Lipparini, F.; Egidi, F.; Goings, J.; Peng, B.; Petrone, A.; Henderson, T.; Ranasinghe, D.; Zakrzewski, V. G.; Gao, J.; Rega, N.; Zheng, G.; Liang, W.; Hada, M.; Ehara, M.; Toyota, K.; Fukuda, R.; Hasegawa, J.; Ishida, M.; Nakajima, T.; Honda, Y.; Kitao, O.; Nakai, H.; Vreven, T.; Throssell, K.; J. A. Montgomery, J.; Peralta, J. E.; Ogliaro, F.; Bearpark, M.; Heyd, J. J.; Brothers, E.; Kudin, K. N.; Staroverov, V. N.; Keith, T.; Kobayashi, R.; Normand, J.; Raghavachari, K.; Rendell, A.; Burant, J. C.; Iyengar, S. S.; Tomasi, J.; Cossi, M.; Millam, J. M.; Klene, M.; Adamo, C.; Cammi, R.; Ochterski, J. W.; Martin, R. L.; Morokuma, K.; Farkas, O.; Foresman, J. B.; Fox, D. J. *Gaussian09*; 2016.

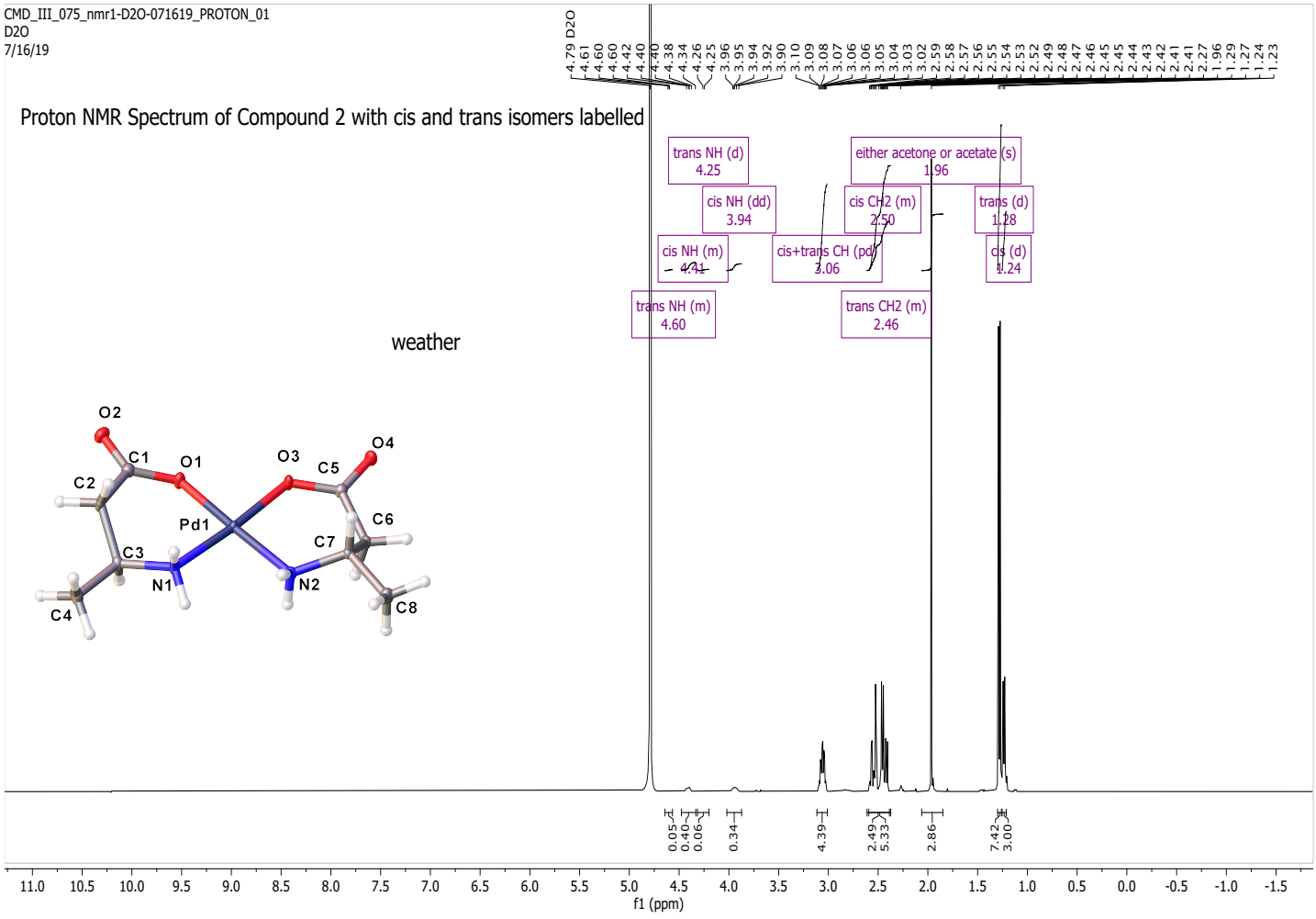
(S3) Becke, A. D. Density-functional thermochemistry. III. The role of exact exchange. *J. Chem. Phys.* **1993**, *98*, 5648–5652.

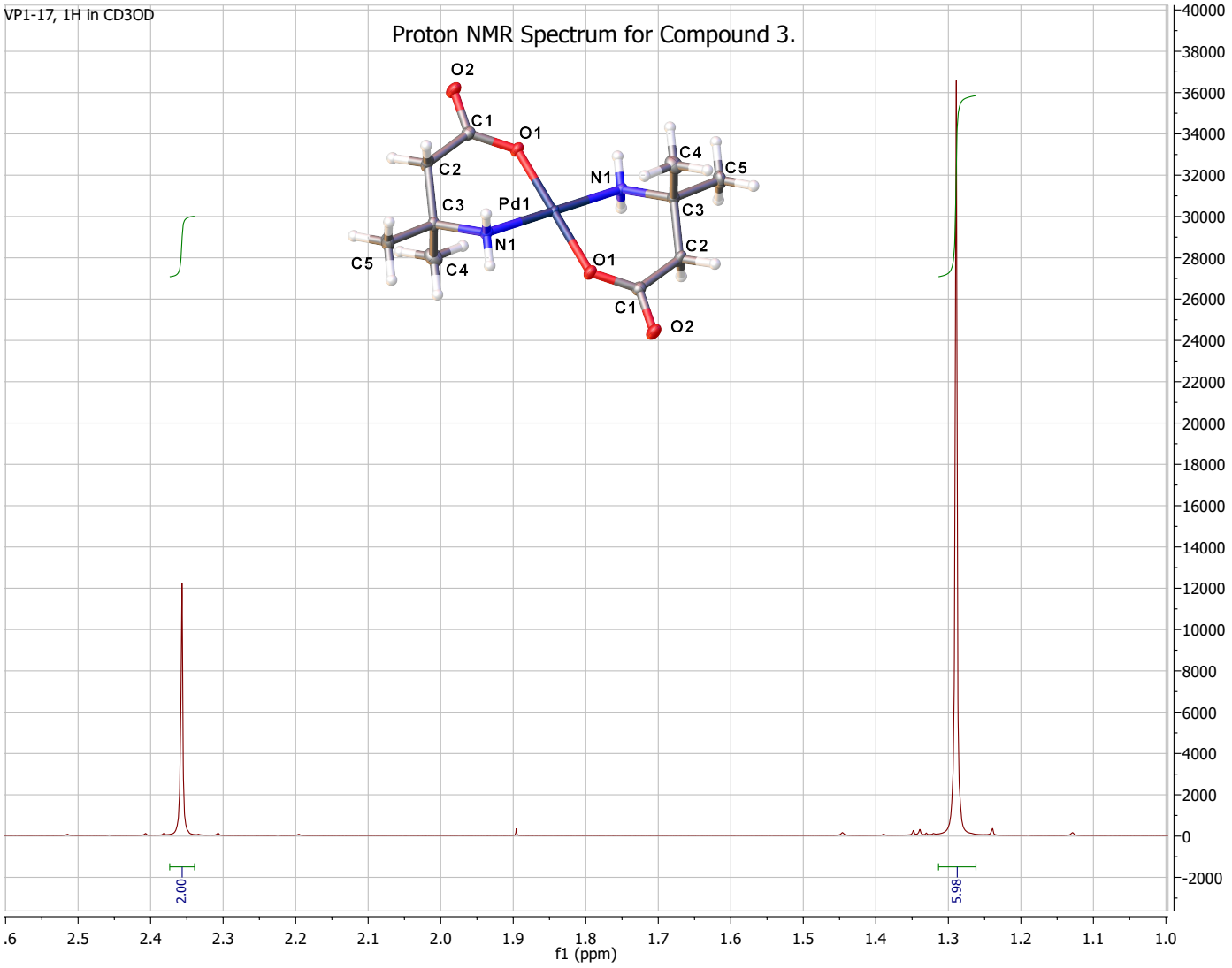
(S4) Lee, C.; Yang, W.; Parr, R. G. Development of the Colle-Salvetti correlation-energy formula into a functional of the electron density. *Phys. Rev. B* **1988**, *37*, 785–789.

(S5) Hay, P. J.; Wadt, W. R. Ab initio effective core potentials for molecular calculations. Potentials for K to Au including the outermost core orbitals. *J. Chem. Phys.* **1985**, *82*, 299–310.

2 Proton NMR Spectra



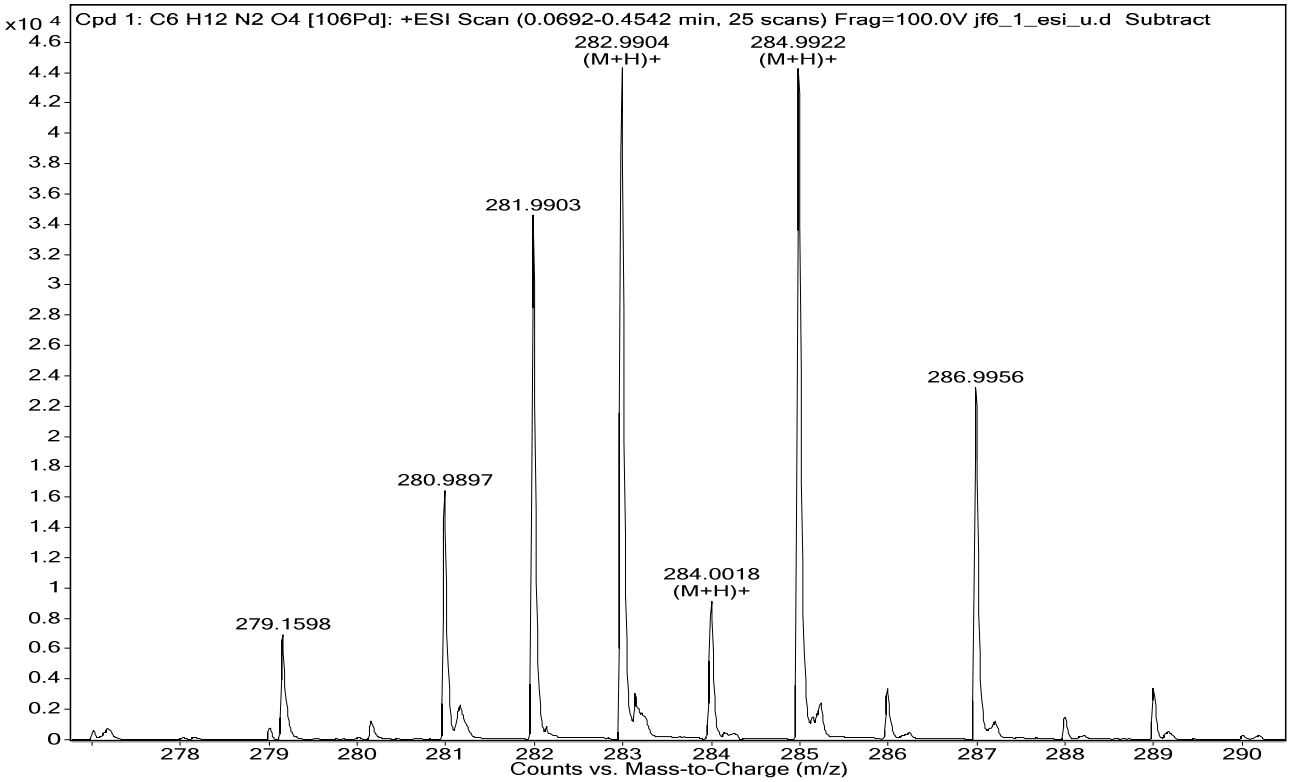

CMD_III_075_nmr1-D2O-071619_PROTON_01
D2O
7/16/19
Proton NMR Spectrum of Compound 2 with cis and trans isomers labelled
trans NH (d) 4.25
either acetone or acetate (s) 1.96
cis NH (dd) 3.94
cis CH2 (m) 2.50
trans (d) 1.28
cis NH (m) 4.41
cis+trans CH (pd) 3.06
cis (d) 1.24
trans NH (m) 4.60
trans CH2 (m) 2.46
weather
O2
C1
O1
O3
C5
O4
C2
Pd1
C6
C7
C3
N1
N2
C4
C8
f1 (ppm)


VP1-17, 1H in CD3OD
Proton NMR Spectrum for Compound 3.
O2
C1
O1
C2
N1
C4
C5
C3
Pd1
C3
N1
C2
C5
C4
O1
C1
O2
2.00
5.98
f1 (ppm)

3 High Resolution Mass Spectra

High Resolution Mass Spectrum for Compound 1, bis-(3-aminopropionato)palladium(II)

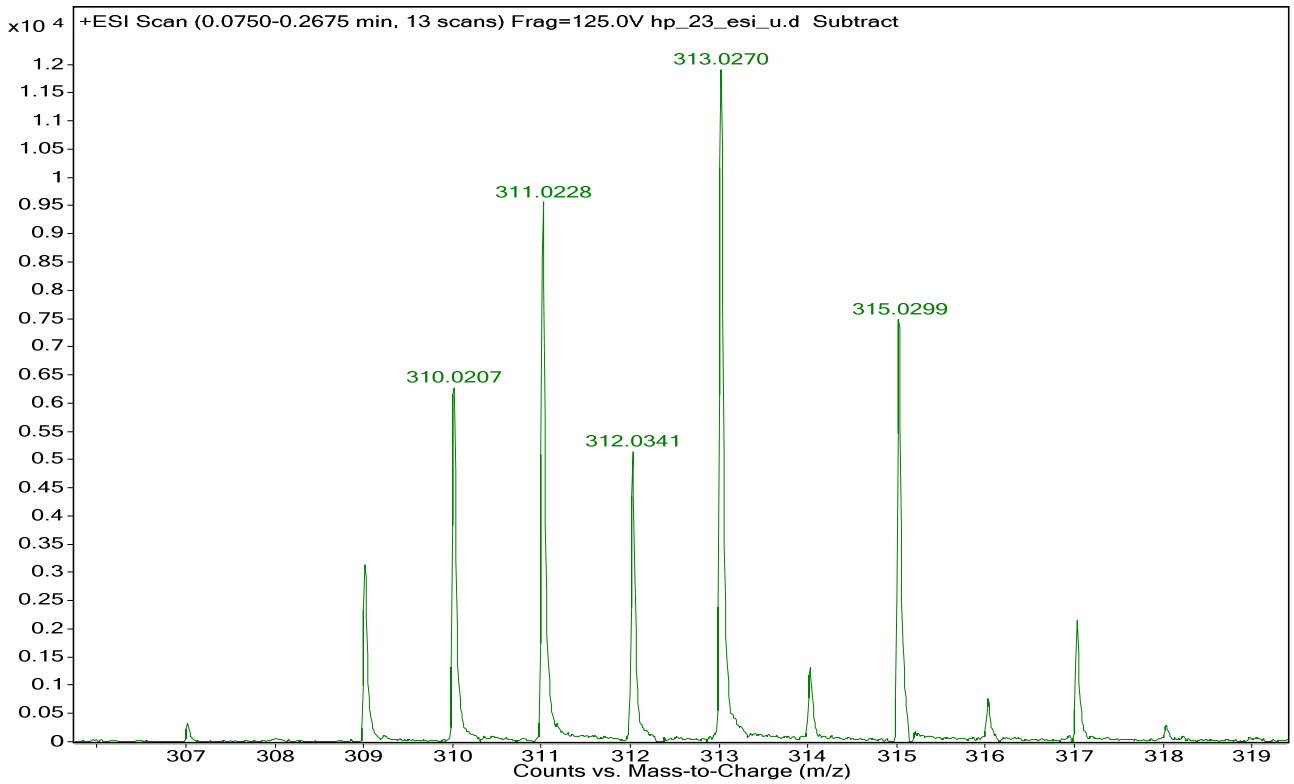
Sample Name	jf6_1	**Position**	P1-B7	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	-1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	All Ions Missed
Data Filename	jf6_1_esi_u.d	**ACQ Method**	MMI_esi_union.m	**Comment**		441382 **Acquired Time**	2/1/2014 12:38:14 PM



The peaks are fully consistent with the Pd isotope distribution and with all peaks being (M+H)+ ions

High Resolution Mass Spectrum for Compound 2, bis-(3-aminobutanato)palladium(II)

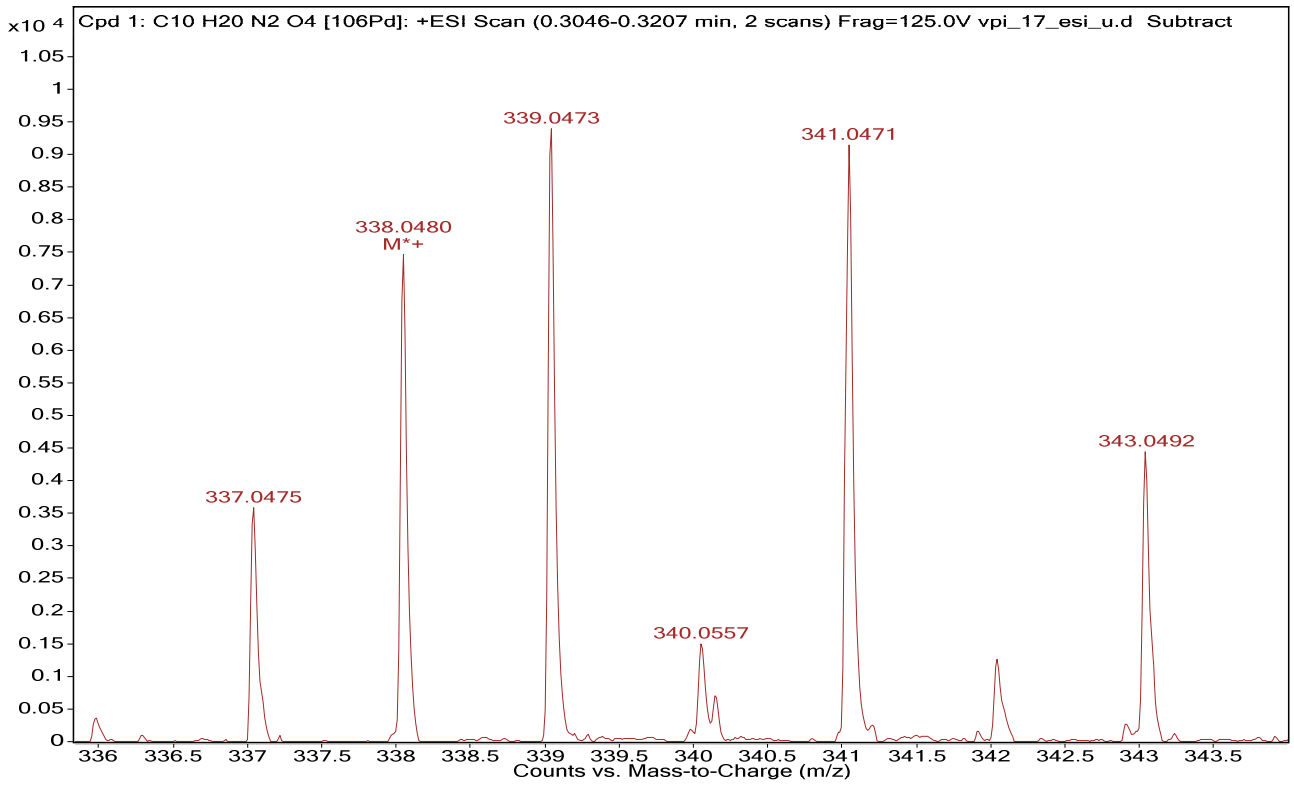
Sample Name	hp_23	**Position**	P1-D8	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	-1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	All Ions Missed
Data Filename	hp_23_esi_u.d	**ACQ Method**	MMI_esi_union.m	**Comment**	441382	**Acquired Time**	8/15/2014 8:45:25 AM



The peaks are fully consistent with the Pd isotope distribution and with all peaks being (M+H)+ ions

High Resolution Mass Spectrum for Compound 3, bis-(3-amino-3-methylbutanato)palladium(II)

Sample Name	vpi_17	**Position**	P1-A1	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	-1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	All Ions Missed
Data Filename	vpi_17_esi_u.d	**ACQ Method**	MMI_esi_union.m	**Comment**	234226	**Acquired Time**	11/18/2013 3:24:51 PM



The peaks are fully consistent with the Pd isotope distribution and with all peaks being (M+H)+ ions

4 XRD Full Experimental Results

Compound 1 Full X-Ray Results

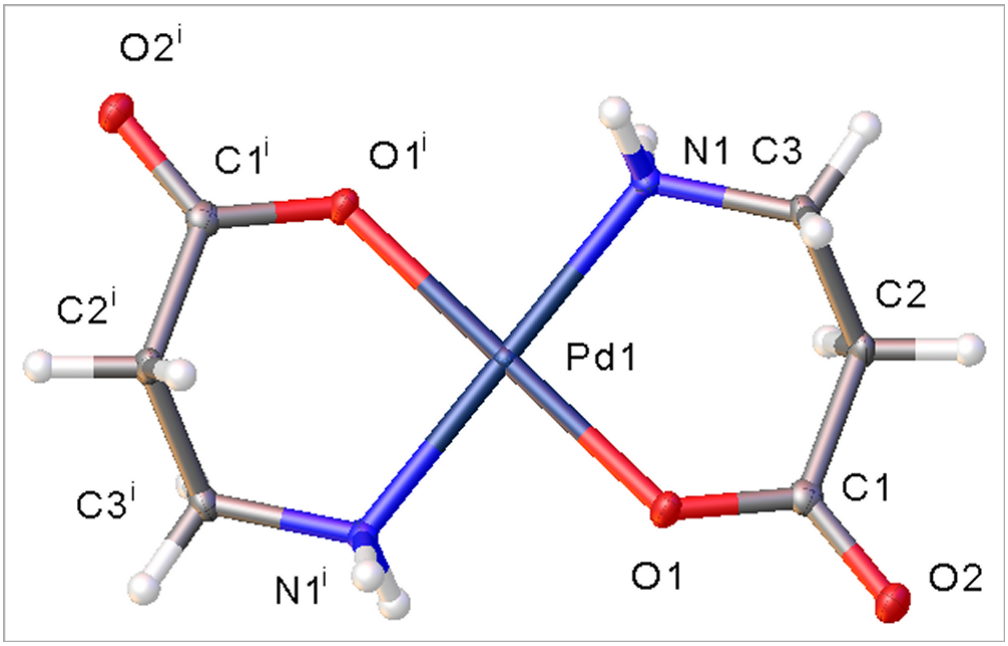


Table 1 Crystal data and structure refinement for Compound 1

Identification code	JF6-1
Empirical formula	$C_6H_{12}N_2O_4Pd$
Formula weight	282.58
Temperature/K	100.00(10)
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	5.6938(3)
b/Å	8.8497(4)
c/Å	9.0421(5)
α/°	90
β/°	104.686(6)
γ/°	90
Volume/Å^3	440.73(4)
Z	2
ρ_{calc}g/cm^3	2.129
μ/mm^{-1}	2.090
F(000)	280.0
Crystal size/mm^3	0.39 × 0.12 × 0.1
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	7.68 to 64.158
Index ranges	-8 ≤ h ≤ 8, -13 ≤ k ≤ 13, -12 ≤ l ≤ 10
Reflections collected	4467
Independent reflections	1451 [R_{int} = 0.0315, R_{sigma} = 0.0356]
Data/restraints/parameters	1451/0/62
Goodness-of-fit on F^2	1.041
Final R indexes [I>=2σ (I)]	R_1 = 0.0215, wR_2 = 0.0450

Final R indexes [all data]	$R_1 = 0.0328$, $wR_2 = 0.0512$
Largest diff. peak/hole / e Å^{-3}	0.76/-0.78

Table 2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for JF6-1. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
Pd1	5000	5000	5000	6.88(8)
O1	7169(3)	5394.3(17)	3601.2(18)	12.9(3)
O2	7978(3)	6080.3(17)	1441.5(18)	15.5(3)
N1	2602(3)	6659.7(19)	4100.0(19)	10.4(3)
C3	3226(4)	7604(2)	2907(3)	12.6(4)
C1	6524(4)	6003(2)	2250(2)	11.2(4)
C2	3989(4)	6653(2)	1705(2)	12.0(4)

Table 3 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for JF6-1. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Pd1	7.40(11)	7.10(11)	6.24(11)	0.31(8)	1.90(8)	0.66(8)
O1	10.9(7)	17.6(7)	11.1(7)	4.3(6)	4.7(6)	1.9(6)
O2	14.9(8)	18.0(8)	15.7(8)	4.6(6)	7.8(7)	1.1(6)
N1	12.5(8)	10.2(8)	9.3(8)	0.3(7)	3.9(7)	2.2(7)
C3	14.4(9)	10.9(9)	12.0(10)	3.4(8)	2.5(8)	2.1(8)
C1	12.8(9)	8.6(9)	12.2(10)	-0.4(8)	3.2(8)	-0.8(7)
C2	11.1(9)	15.4(10)	9.4(9)	4.0(8)	2.3(8)	1.7(8)

Table 4 Bond Lengths for JF6-1.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Pd1	O1[1]	2.0097(16)	O2	C1	1.238(3)
Pd1	O1	2.0097(16)	N1	C3	1.477(3)
Pd1	N1	2.0300(16)	C3	C2	1.522(3)
Pd1	N1[1]	2.0300(16)	C1	C2	1.516(3)
O1	C1	1.300(3)			

[1]1-X,1-Y,1-Z

Table 5 Bond Angles for JF6-1.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
O1[1]	Pd1	O1	180.00(6)	C3	N1	Pd1	116.11(13)
O1	Pd1	N1[1]	85.39(7)	N1	C3	C2	111.95(17)
O1[1]	Pd1	N1	85.39(7)	O1	C1	C2	118.82(19)
O1[1]	Pd1	N1[1]	94.61(7)	O2	C1	O1	120.41(19)
O1	Pd1	N1	94.61(7)	O2	C1	C2	120.75(19)
N1[1]	Pd1	N1	180.0	C1	C2	C3	113.47(18)
C1	O1	Pd1	126.23(14)				

[1]1-X,1-Y,1-Z

Table 6 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for JF6-1.

Atom	*x*	*y*	*z*	U(eq)
H1A	1162	6236	3705	13
H1B	2444	7262	4858	13
H3A	4540	8282	3380	15
H3B	1831	8215	2416	15
H2A	2843	5830	1402	14
H2B	3913	7273	809	14

Experimental

Single crystals of $C_6H_{12}N_2O_4Pd$ **[JF6-1]** were **[From water/acetone]**. A suitable crystal was selected and **[On fiber]** on a **Xcalibur, Eos, Gemini ultra** diffractometer. The crystal was kept at 100.00(10) K during data collection. Using Olex2 [1], the structure was solved with the ShelXT [2] structure solution program using Intrinsic Phasing and refined with the ShelXL [3] refinement package using Least Squares minimisation.

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.
2. Sheldrick, G.M. (2015). Acta Cryst. A71, 3-8.
3. Sheldrick, G.M. (2015). Acta Cryst. C71, 3-8.

Crystal structure determination of [JF6-1]

Crystal Data for $C_6H_{12}N_2O_4Pd$ (M =282.58 g/mol): monoclinic, space group $P2_1/n$ (no. 14), a = 5.6938(3) Å, b = 8.8497(4) Å, c = 9.0421(5) Å, β = 104.686(6)°, V = 440.73(4) Å^3, Z = 2, T = 100.00(10) K, μ(MoKα) = 2.090 mm^{-1}, *Dcalc* = 2.129 g/cm^3, 4467 reflections measured (7.68° ≤ 2Θ ≤ 64.158°), 1451 unique (R_{int} = 0.0315, R_{sigma} = 0.0356) which were used in all calculations. The final R_1 was 0.0215 (I > 2σ(I)) and wR_2 was 0.0512 (all data).

Refinement model description

Number of restraints - 0, number of constraints - unknown.
Details:

```
1. Fixed Uiso
 At 1.2 times of:
  All C(H,H) groups, All N(H,H) groups
2.a Secondary CH2 refined with riding coordinates:
 N1(H1A,H1B), C3(H3A,H3B), C2(H2A,H2B)
```

This report has been created with Olex2, compiled on 2018.05.29 svn.r3508 for OlexSys. Please let us know if there are any errors or if you would like to have additional features.

Compound 2 Full X-Ray Results

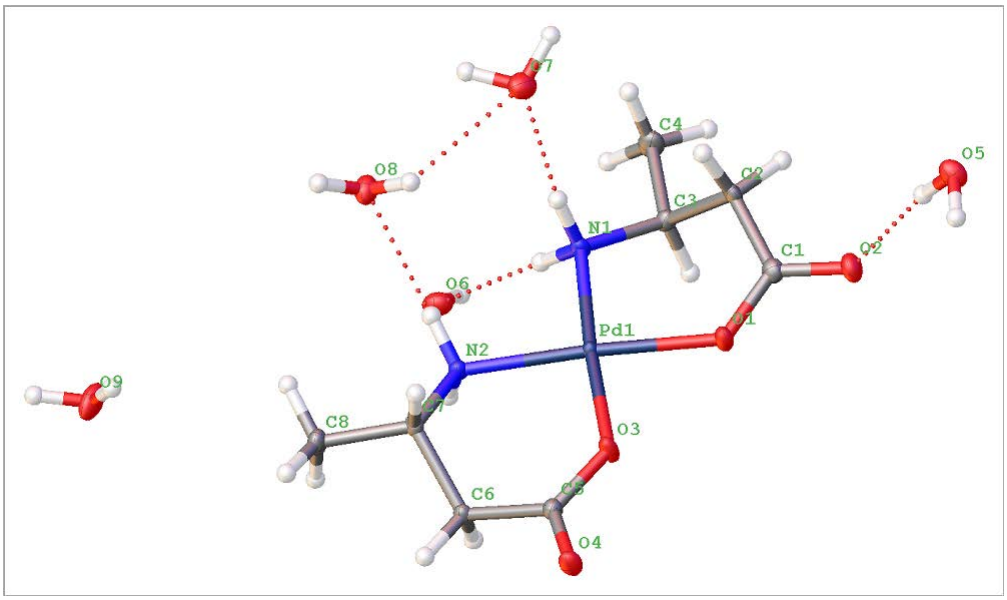


Table 1 Crystal data and structure refinement for compound 2

Identification code	HP-23_abs
Empirical formula	$C_{16}H_{44}N_4O_{15}Pd_2$
Formula weight	745.35
Temperature/K	100.15
Crystal system	monoclinic
Space group	I2
a/Å	10.7593(2)
b/Å	7.38120(10)
c/Å	17.8370(3)
α/°	90
β/°	97.217(2)
γ/°	90
Volume/Å^3	1405.33(4)
Z	2
ρ_{calc}g/cm^3	1.761
μ/mm^{-1}	1.352
F(000)	760.0
Crystal size/mm^3	0.2973 × 0.2328 × 0.198
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	7.462 to 64.882
Index ranges	$-15 \le h \le 15$, $-10 \le k \le 11$, $-26 \le l \le 26$
Reflections collected	14648
Independent reflections	4716 [R_{int} = 0.0381, R_{sigma} = 0.0425]
Data/restraints/parameters	4716/1/187
Goodness-of-fit on F^2	1.087
Final R indexes [I>=2σ (I)]	R_1 = 0.0254, wR_2 = 0.0528
Final R indexes [all data]	R_1 = 0.0292, wR_2 = 0.0556
Largest diff. peak/hole / e Å^{-3}	0.89/-0.68
Flack parameter	0.003(18)

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for HP-23_abs. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
Pd1	4008.0(2)	9044.1(5)	2914.2(2)	9.20(6)
O1	2472(2)	8537(3)	2172.2(13)	14.9(6)
O2	461(2)	8531(3)	1764.5(14)	18.1(6)
O3	4787.0(19)	9343(5)	1950.3(12)	14.6(8)
O4	6310(2)	9782(4)	1252.7(13)	15.6(5)
N1	3110(2)	8794(6)	3833.6(14)	11.4(8)
N2	5608(2)	9483(4)	3608.2(14)	10.9(6)
C1	1314(3)	8630(4)	2293(2)	14.2(7)
C2	1016(3)	8832(8)	3097.3(17)	15.4(10)
C3	1890(3)	7828(5)	3695.4(19)	12.4(6)
C4	1296(3)	7663(6)	4423(2)	19.3(8)
C5	5958(3)	9488(4)	1876.7(18)	12.3(7)
C6	6916(3)	9239(8)	2568.9(16)	11.5(8)
C7	6665(3)	10319(5)	3265.4(18)	10.6(6)
C8	7831(3)	10350(5)	3848.6(19)	15.7(7)
O5	-1335(2)	10685(4)	1067.3(15)	17.2(5)
O6	5000	7012(6)	5000	18.2(10)
O7	2453(3)	12517(4)	4339.4(15)	19.7(6)
O8	5000	11572(6)	5000	18.0(10)
O9	10000	13034(6)	5000	23.8(9)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for HP-23_abs. The Anisotropic displacement factor exponent takes the form: $-2π^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+…]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Pd1	7.32(9)	12.25(10)	8.02(10)	-0.27(13)	0.91(6)	-0.63(13)
O1	9.3(10)	23.9(16)	11.2(11)	-1.1(9)	0.8(8)	-3.1(9)
O2	12.6(11)	22.5(15)	18.0(12)	1.0(9)	-2.7(9)	-0.8(9)
O3	7.6(9)	26(2)	10.2(9)	2.6(10)	1.7(7)	-2.9(10)
O4	11.6(11)	25.1(13)	10.5(11)	2.2(9)	3.2(9)	-1.3(10)
N1	8.6(10)	17(2)	9.4(11)	0.7(12)	2.7(8)	-0.8(12)
N2	9.5(11)	15.0(17)	8.4(11)	-0.4(9)	2.0(9)	-0.6(9)
C1	12.7(14)	14(2)	15.8(15)	0.0(11)	0.8(11)	-1.8(11)
C2	7.0(11)	22(3)	16.8(14)	-2.5(16)	1.0(10)	-1.1(16)
C3	11.4(15)	14.9(16)	11.2(15)	-1.4(12)	2.8(12)	-3.1(12)
C4	15.8(17)	28(2)	15.7(17)	-1.6(15)	7.0(13)	-6.4(15)
C5	11.1(14)	14.5(19)	11.2(14)	-1.6(11)	0.8(11)	-0.1(11)
C6	8.6(11)	17(2)	9.4(11)	0.2(15)	2.1(9)	-0.7(15)
C7	8.8(14)	10.8(15)	12.1(15)	0.1(12)	1.2(11)	-1.8(12)
C8	13.1(15)	21.9(19)	11.3(16)	-1.9(13)	-0.8(12)	-2.9(13)
O5	14.6(12)	18.4(14)	19.2(14)	5.8(10)	3.9(10)	-0.5(10)
O6	20(2)	19(2)	17(2)	0	7.9(17)	0
O7	20.2(13)	21.2(14)	17.3(13)	0.9(11)	0.8(10)	4.7(11)
O8	19(2)	16(2)	17(2)	0	-3.3(17)	0
O9	33(2)	21(2)	14.6(19)	0	-8.8(16)	0

Table 4 Bond Lengths for HP-23_abs.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Pd1	O1	2.018(2)	N1	C3	1.487(4)
Pd1	O3	2.017(2)	N2	C7	1.491(4)
Pd1	N1	2.013(2)	C1	C2	1.516(5)
Pd1	N2	2.016(2)	C2	C3	1.523(5)
O1	C1	1.293(4)	C3	C4	1.522(5)
O2	C1	1.232(4)	C5	C6	1.517(4)
O3	C5	1.287(4)	C6	C7	1.529(5)
O4	C5	1.239(4)	C7	C8	1.526(5)

Table 5 Bond Angles for HP-23_abs.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
O3	Pd1	O1	81.67(9)	O2	C1	C2	120.3(3)
N1	Pd1	O1	94.59(10)	C1	C2	C3	115.5(3)
N1	Pd1	O3	175.85(10)	N1	C3	C2	109.7(3)
N1	Pd1	N2	88.55(10)	N1	C3	C4	110.7(3)
N2	Pd1	O1	176.40(10)	C4	C3	C2	110.4(3)
N2	Pd1	O3	95.26(9)	O3	C5	C6	118.7(3)
C1	O1	Pd1	127.4(2)	O4	C5	O3	121.3(3)
C5	O3	Pd1	127.8(2)	O4	C5	C6	119.9(3)
C3	N1	Pd1	114.4(2)	C5	C6	C7	115.3(3)
C7	N2	Pd1	116.83(19)	N2	C7	C6	109.8(3)
O1	C1	C2	119.0(3)	N2	C7	C8	109.3(3)
O2	C1	O1	120.7(3)	C8	C7	C6	110.3(3)

Table 6 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for HP-23_abs.

Atom	*x*	*y*	*z*	U(eq)
H1A	3616.83	8189.3	4197.23	14
H1B	2975.61	9919.87	4014.96	14
H2A	5427.48	10210.18	3992.2	13
H2B	5875.31	8403.33	3815.4	13
H2C	150.93	8397.14	3118.63	19
H2D	1036.82	10136.83	3227.04	19
H3	2039.92	6583.12	3504.4	15
H4A	1872.4	7029.56	4804.49	29
H4B	511.91	6979.23	4324.97	29
H4C	1121.41	8875.55	4608.09	29
H6A	7749.39	9589.16	2435.75	14
H6B	6953.72	7936.76	2702.54	14
H7	6435.91	11590.14	3110.5	13
H8A	7634.77	10949.05	4309.8	23
H8B	8500.88	11016.29	3643.61	23
H8C	8107.1	9106.14	3966.8	23
H5A	-874.37	9936.96	1359.25	26
H5B	-934.35	10820.35	676.6	26
H6	4602.46	6112.58	5176.76	27
H7A	2930.32	12922.18	4733.89	30
H7B	1695.23	12763.19	4429.8	30
H8D	4497.19	11965.16	4614.04	27
H8E	5502.39	12476.8	5124.31	27
H9A	9727.87	13901.4	4692.02	36
H9B	10575.93	13541.54	5317.79	36

Experimental

Single crystals of $C_{16}H_{44}N_4O_{15}Pd_2$ **[HP-23_abs]** were **[From methanol/acetone/water]**. A suitable crystal was selected and **[on kryton fiber]** on a **Xcalibur, Eos, Gemini ultra** diffractometer. The crystal was kept at 100.15 K during data collection. Using Olex2 [1], the structure was solved with the ShelXS [2] structure solution program using Direct Methods and refined with the ShelXL [3] refinement package using Least Squares minimisation.

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.
2. Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122.
3. Sheldrick, G.M. (2015). Acta Cryst. C71, 3-8.

Crystal structure determination of [HP-23_abs]

Crystal Data for $C_{16}H_{44}N_4O_{15}Pd_2$ (*M* =745.35 g/mol): monoclinic, space group I2 (no. 5), *a* = 10.7593(2) Å, *b* = 7.38120(10) Å, *c* = 17.8370(3) Å, *β* = 97.217(2)°, *V* = 1405.33(4) Å^3, *Z* = 2, *T* = 100.15 K, μ(MoKα) = 1.352 mm^{-1}, *Dcalc* = 1.761 g/cm^3, 14648 reflections measured (7.462° ≤ 2Θ ≤ 64.882°), 4716 unique (R_{int} = 0.0381, R_{sigma} = 0.0425) which were used in all calculations. The final R_1 was 0.0254 (I > 2σ(I)) and wR_2 was 0.0556 (all data).

Table 7 Atomic Occupancy for HP-23_abs.

Atom	*Occupancy*	Atom	*Occupancy*	Atom	*Occupancy*
H6	0.5	H8D	0.5	H8E	0.5
H9A	0.5	H9B	0.5		

Experimental

Single crystals of $C_{16}H_{44}N_4O_{15}Pd_2$ **[HP-23_abs]** were **[From methanol/acetone/water]**. A suitable crystal was selected and **[on kryton fiber]** on a **Xcalibur, Eos, Gemini ultra** diffractometer. The crystal was kept at 100.15 K during data collection. Using Olex2 [1], the structure was solved with the ShelXS [2] structure solution program using Direct Methods and refined with the ShelXL [3] refinement package using Least Squares minimisation.

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Crystal structure determination of [HP-23_abs]

Crystal Data for $C_{16}H_{44}N_4O_{15}Pd_2$ (M =745.35 g/mol): monoclinic, space group I2 (no. 5), a = 10.7593(2) Å, b = 7.38120(10) Å, c = 17.8370(3) Å, β = 97.217(2)°, V = 1405.33(4) Å^3, Z = 2, T = 100.15 K, μ(MoKα) = 1.352 mm^{-1}, *Dcalc* = 1.761 g/cm^3, 14648 reflections measured (7.462° ≤ 2Θ ≤ 64.882°), 4716 unique (R_{int} = 0.0381, R_{sigma} = 0.0425) which were used in all calculations. The final R_1 was 0.0254 (I > 2σ(I)) and wR_2 was 0.0556 (all data).

Refinement model description

Number of restraints - 1, number of constraints - unknown.

Details:

```
1. Fixed Uiso
 At 1.2 times of:
  All C(H) groups, All C(H,H) groups, All N(H,H) groups
 At 1.5 times of:
  All C(H,H,H) groups, All O(H) groups, All O(H,H) groups
2. Others
 Fixed Sof: H6(0.5) H8D(0.5) H8E(0.5) H9A(0.5) H9B(0.5)
3.a Free rotating group:
 O5(H5A,H5B), O6(H6), O7(H7A,H7B), O8(H8D,H8E), O9(H9A,H9B)
3.b Ternary CH refined with riding coordinates:
 C3(H3), C7(H7)
3.c Secondary CH2 refined with riding coordinates:
 N1(H1A,H1B), N2(H2A,H2B), C2(H2C,H2D), C6(H6A,H6B)
3.d Idealised Me refined as rotating group:
 C4(H4A,H4B,H4C), C8(H8A,H8B,H8C)
```

This report has been created with Olex2, compiled on May 18 2018 14:05:52 for OlexSys. Please let us know if there are any errors or if you would like to have additional features.

Complex 3 Full X-ray Data

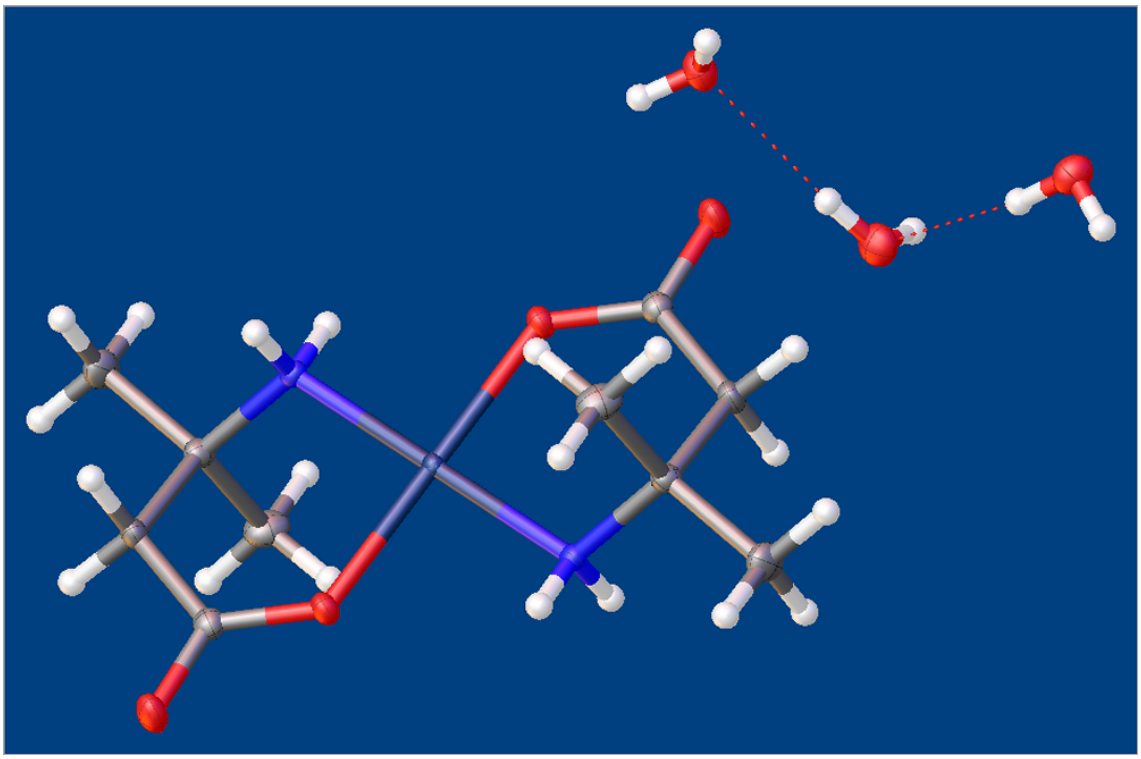

Table 1 Crystal data and structure refinement for Compound 3

Identification code	VGP-beta1
Empirical formula	$C_{10}H_{32}N_2O_{10}Pd$
Formula weight	446.77
Temperature/K	97(5)
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	12.4228(5)
b/Å	7.0497(2)
c/Å	12.5509(5)
α/°	90
β/°	117.080(5)
γ/°	90
Volume/Å^3	978.67(7)
Z	2
ϱ_{calc}g/cm^3	1.516
μ/mm^{-1}	0.993
F(000)	464.0
Crystal size/mm^3	0.4 × 0.214 × 0.114
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	6.932 to 64.77
Index ranges	-18 ≤ h ≤ 18, -10 ≤ k ≤ 10, -17 ≤ l ≤ 18
Reflections collected	9102

Independent reflections	3258 [R_{int} = 0.0379, R_{sigma} = 0.0474]
Data/restraints/parameters	3258/0/140
Goodness-of-fit on F^2	1.055
Final R indexes [I>=2σ (I)]	R_1 = 0.0280, wR_2 = 0.0553
Final R indexes [all data]	R_1 = 0.0448, wR_2 = 0.0636
Largest diff. peak/hole / e $Å^{-3}$	0.62/-0.75

Table 2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for VGP-beta1. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
Pd1	5000	5000	5000	8.91(6)
O1	5999.0(13)	3255.9(19)	4567.4(12)	14.2(3)
O2	6435.7(14)	1362(2)	3434.6(13)	19.5(3)
N1	3442.1(16)	4353(2)	3508.3(15)	11.0(3)
C1	5663.0(18)	2213(3)	3634.7(17)	13.6(4)
C2	4347.4(18)	2010(3)	2712.0(17)	15.6(4)
C3	3332.7(18)	2360(3)	3063.4(17)	12.5(4)
C4	2113.4(19)	2124(3)	1938.2(18)	19.8(4)
C5	3396(2)	1024(3)	4047(2)	21.1(5)
O5	11081.8(14)	5053(3)	3562.9(14)	18.9(3)
O3	8803.6(16)	3128(2)	-7.1(15)	19.4(3)
O4	9847.6(16)	6496(3)	1261.4(17)	21.1(3)

Table 3 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for VGP-beta1. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Pd1	10.09(9)	9.21(9)	7.68(9)	-0.33(8)	4.26(7)	1.03(8)
O1	14.5(7)	15.0(7)	13.4(7)	-3.6(5)	6.5(6)	1.6(5)
O2	17.5(8)	24.2(8)	17.4(7)	-6.8(6)	8.5(6)	2.5(6)
N1	11.7(8)	11.6(7)	9.2(8)	0.6(6)	4.4(6)	1.5(6)
C1	15.8(10)	12.3(9)	13.0(9)	1.0(7)	7.0(8)	0.7(7)
C2	15.8(10)	15.6(10)	13.8(9)	-4.5(8)	5.4(8)	2.7(7)
C3	12.4(9)	10.3(9)	13.5(9)	-0.6(7)	4.6(7)	-0.1(7)
C4	15.8(10)	19.6(10)	18.8(10)	-5.2(9)	3.4(8)	-2.1(8)
C5	20.6(11)	17.1(11)	23.7(11)	5.2(9)	8.4(9)	-0.3(8)
O5	20.9(8)	17.9(8)	19.4(8)	-4.8(7)	10.5(6)	-1.6(7)
O3	16.1(8)	22.1(9)	18.6(8)	1.1(7)	6.6(7)	-2.0(7)
O4	21.9(9)	24.8(9)	20.6(9)	-0.3(7)	13.1(7)	-0.2(7)

Table 4 Bond Lengths for VGP-beta1.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Pd1	O1[1]	1.9900(14)	N1	C3	1.495(2)
Pd1	O1	1.9900(14)	C1	C2	1.520(3)
Pd1	N1[1]	2.0396(17)	C2	C3	1.533(3)
Pd1	N1	2.0396(17)	C3	C4	1.539(3)
O1	C1	1.281(2)	C3	C5	1.526(3)
O2	C1	1.251(2)			

[1]1-X,1-Y,1-Z

Table 5 Bond Angles for VGP-beta1.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
O1[1]	Pd1	O1	180.0	O2	C1	O1	119.89(18)
O1	Pd1	N1	93.46(6)	O2	C1	C2	117.35(17)
O1	Pd1	N1[1]	86.54(6)	C1	C2	C3	120.37(17)
O1[1]	Pd1	N1	86.54(6)	N1	C3	C2	108.17(16)
O1[1]	Pd1	N1[1]	93.46(6)	N1	C3	C4	109.20(15)
N1	Pd1	N1[1]	180.0	N1	C3	C5	108.40(16)
C1	O1	Pd1	128.30(13)	C2	C3	C4	108.25(16)
C3	N1	Pd1	116.11(12)	C5	C3	C2	112.74(17)
O1	C1	C2	122.72(18)	C5	C3	C4	110.02(17)

[1]1-X,1-Y,1-Z

Table 6 Hydrogen Bonds for VGP-beta1.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/°
N1	H1A	O2[1]	0.84(2)	2.06(3)	2.883(2)	167(2)
O5	H5D	O3[2]	0.72(3)	2.07(3)	2.788(2)	173(3)
O5	H5E	O4[3]	0.84(4)	1.98(4)	2.811(3)	170(3)
O3	H3A	O2[4]	0.75(3)	1.96(3)	2.715(2)	175(3)
O3	H3B	O4	0.78(4)	2.05(4)	2.823(3)	170(5)
O4	H4D	O3[5]	0.70(3)	2.09(3)	2.785(3)	176(3)
O4	H4E	O5	0.74(5)	2.03(5)	2.774(2)	175(6)

[1]1-X,1/2+Y,1/2-Z; [2]2-X,1/2+Y,1/2-Z; [3]2-X,-1/2+Y,1/2-Z; [4]+X,1/2-Y,-1/2+Z; [5]2-X,1-Y,-Z

Table 7 Torsion Angles for VGP-beta1.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
Pd1	O1	C1	O2	171.77(13)	O1	C1	C2	C3	-24.6(3)
Pd1	O1	C1	C2	-5.8(3)	O2	C1	C2	C3	157.79(18)
Pd1	N1	C3	C2	-62.19(18)	C1	C2	C3	N1	59.7(2)
Pd1	N1	C3	C4	-179.79(13)	C1	C2	C3	C4	177.87(18)
Pd1	N1	C3	C5	60.36(19)	C1	C2	C3	C5	-60.2(2)

Table 8 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for VGP-beta1.

Atom	*x*	*y*	*z*	U(eq)
H1A	3420(20)	5080(30)	2970(20)	16(6)
H1B	2860(20)	4590(30)	3670(20)	19(6)
H2A	4244	733	2393	19
H2B	4214	2867	2059	19
H4A	1466	2369	2137	30
H4B	2046	852	1641	30
H4C	2068	3003	1335	30
H5A	4152	1200	4751	32
H5B	3336	-265	3778	32
H5C	2742	1295	4230	32
H5D	11110(30)	5790(40)	3980(30)	36(9)
H5E	10790(30)	4040(50)	3690(30)	68(12)
H3A	8140(30)	3200(40)	-440(30)	33(8)
H3B	9010(40)	4090(60)	320(40)	103(17)
H4D	10200(30)	6640(40)	960(30)	34(9)
H4E	10160(50)	6150(70)	1890(50)	140(20)

Experimental

Single crystals of $C_{10}H_{32}N_2O_{10}Pd$ **[VGP-beta1]** were **[From methanol/acetone/water]**. A suitable crystal was selected and **[On a fiber with kryton fluid.]** on a **Xcalibur, Eos, Gemini ultra** diffractometer. The crystal was kept at 97(5) K during data collection. Using Olex2 [1], the structure was solved with the ShelXT [2] structure solution program using Direct Methods and refined with the ShelXL [3] refinement package using Least Squares minimisation.

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.
2. Sheldrick, G.M. (2015). Acta Cryst. A71, 3-8.
3. Sheldrick, G.M. (2015). Acta Cryst. C71, 3-8.

Crystal structure determination of [VGP-beta1]

Crystal Data for $C_{10}H_{32}N_2O_{10}Pd$ (M =446.77 g/mol): monoclinic, space group $P2_1/c$ (no. 14), a = 12.4228(5) Å, b = 7.0497(2) Å, c = 12.5509(5) Å, β = 117.080(5)°, V = 978.67(7) Å^3, Z = 2, T = 97(5) K, μ(MoKα) = 0.993 mm^{-1}, *Dcalc* = 1.516 g/cm^3, 9102 reflections measured (6.932° ≤ 2Θ ≤ 64.77°), 3258 unique (R_{int} = 0.0379, R_{sigma} = 0.0474) which were used in all calculations. The final R_1 was 0.0280 (I > 2σ(I)) and wR_2 was 0.0636 (all data).

Refinement model description

Number of restraints - 0, number of constraints - unknown.

Details:

```
1. Fixed Uiso
 At 1.2 times of:
  All C(H,H) groups
 At 1.5 times of:
  All C(H,H,H) groups
2.a Secondary CH2 refined with riding coordinates:
 C2(H2A,H2B)
2.b Idealised Me refined as rotating group:
 C4(H4A,H4B,H4C), C5(H5A,H5B,H5C)
```