Supporting Information for
Palladium-Catalyzed Aerobic Oxidative Kinetic Resolution of Alcohols Using an Achiral Exogenous Base

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General Information. All reagents, unless specified, were bought from commercial sources and used without further purification. *tert*-butanol was filtered through freshly activated alumina. Crushed 3Å molecular sieves were freshly activated by heating under vacuum. Methods and characterization of all alcohols and *meso*-diols has been previously reported.[1]

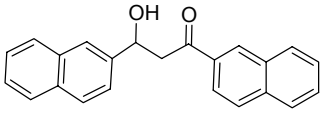
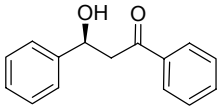
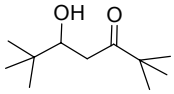
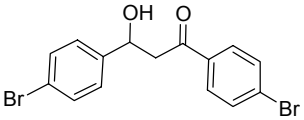
Preperation of [(-)-sparteine]$PdCl_2$]: In a 250 ml round bottom flask, 2.35 g of (-)-sparteine (10.0 mmol) and 2.59 g (9.99 mmol) of $Pd(MeCN)_2Cl_2$ were added to 100 mL of 1,2-dichloroethane. The reaction was heated to 65 ^oC for one hour and then the solvent was removed *in vacuo.* The resulting red oil was dissolved in CH_2Cl_2 and hexanes. Concentration and removal of the solvent *in vacuo* gave an orange-red powdery solid, which was rinsed with hexanes. Yield: 4.01 g, 97.4 %.[2]

Procedure for the gram scale oxidative kinetic resolution of sec-phenethyl alcohol: In a 100 mL two neck round bottom flask fitted with a reflux condenser, 10 mmol (1.22g) of the *sec*-phenethyl alcohol, 530 mg (5 mmol) of Na_2CO_3, 205 mg (0.5 mmol) of Pd[(-)sparteine]Cl_2, 1.0 g of 3Å molecular sieves (freshly activated), and 40 mL *ter*t-butanol were combined. A balloon filled with oxygen gas was attached to the top of the condenser via a three way joint. The apparatus was evacuated (water aspirator) and refilled with oxygen from the balloon three times. The reaction mixture was allowed to stir magnetically, while heating in an oil bath at 65^oC. The conversion was monitored by GC. After 32h and cooling to room temperature, the mixtured was filtered through a pad of silica, washed with ether, and concentrated under vacuum. Ketone by-product and optically active alcohol were then separated by column chromatography (5% ethyl acetate/hexane to 15% ethyl acetate/hexane). This gave optically enriched alcohol (yield: 31.4%, ee: 99.3%) and the ketone byproduct (62% isolated yield, 64.1% conversion by GC).

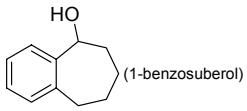
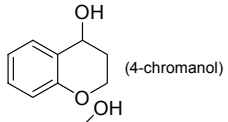
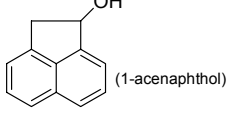
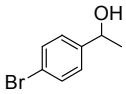
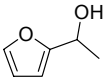
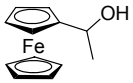
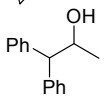
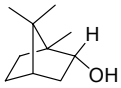
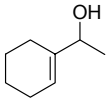
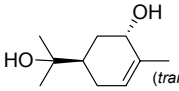
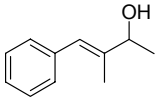
Isolation of [(-)-sparteine]Pd(CO_3) Complex:

In a 10 ml round bottom flask, 41.2 mg (0.1 mmol) of [(-)-sparteine]$PdCl_2$ and 12.72 mg (0.12 mmol) of Na_2CO_3 were combined in 3 ml of *tert*-Butyl alcohol. The mixture was heated at 65 ^oC on an oil bath for 4 h and then filtered to remove excess Na_2CO_3. After evaporation of solvent, the resulting solid (38.02 mg, 95%) was taken up in 3 ml dichloroethane and 1ml of hexanes in a vial. The vial was then placed in a jar containing hexanes and was allowed to sit undisturbed for 2 days while hexanes diffused in to solution. The mother liquor was then removed and the resulting orange crystals were washed three times with hexanes. Orange crystal**,** IR (KBr): 3272, 2931, 1640, 1445, 1207, 985, 829, 732, 488 cm^{-1}; ^1H NMR (300 MHz, CD_2Cl_2): 1.30-1.72 (m, 8H), 1.75-2.10 (m, 8H), 2.15-2.35 (m, 2H), 2.61 (dd, 1H, J = 2.7, 13.0 Hz), 2.89-3.05 (m, 3H), 3.05-3.32 (m, 2H), 3.90-4.05 (m, 1H), 4.05-4.20 (m, 1H); ^{13}C NMR (75 MHz, CD_2Cl_2): 20.2, 23.7, 23.8, 23.8, 25.4, 27.5, 30.2, 34.9, 44.3, 49.1, 60.8, 62.8, 65.7, 66.0, 68.6, 166.7.

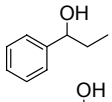
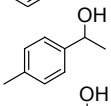
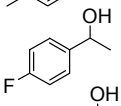
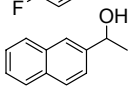
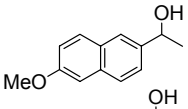
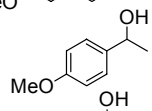
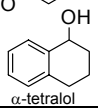
GC and HPLC separation conditions :

Alcohol	Retention Times (in minutes)	Method	Conditions[a]
	43.68 major 52.21 minor	HPLC	1ml / min 95%hexanes/IPA Chiralpak AS column
	14.2 major 16.3 minor	HPLC	1ml / min 95%hexanes/IPA Chiralcel OD column
	17.64 minor 17.78 major	GC	50^o C hold 2min, ramp 5^{o}C/min to 150^{o}C
	14.8 minor 25.6 major	HPLC	1ml / min 90%hexanes/IPA Chiralpak AS column

[a]All GC separations were performed using an autosampling HP 6890 GC fitted with a Hewlett Packard HP Chiral 20% Permethylated β–cyclodextrin column (0.25μm film thickness, 30m length, phase ratio 320) using a 2.0 mL/min carrier gas (H_2) flow rate

Alcohol	Retention Times (in minutes)	Method	Conditions[a]
 (1-benzosuberol)	32.58 minor 32.81 major	GC	50° C hold 2min, ramp 4°C/min to 160°C
 (4-chromanol)	20.71 major 24.23 minor	HPLC	1ml / min 98%hexanes/IPA Chiralcel OD column
 (1-acenaphthol)	19.28 major 21.90 minor	HPLC	1ml / min 96.5%hexanes/IPA Chiralcel OD column
	37.71 minor[2] 38.12 major	GC	50° C hold 5min, ramp 3.5°C/min to 220°C
	14.17 minor 14.40 major	GC	50° C hold 2min, ramp 4°C/min to 160°C
	7.60 major 14.15 minor	HPLC	1ml / min 90%hexanes/IPA Chiralpak AS column
	9.32 minor 9.58 major	GC	50° C hold 5min, ramp 5°C/min to 100°C
	6.13 minor 6.27 major	GC	50° C hold 2min, ramp 4°C/min to 160°C
	10.18 minor 11.62 major	HPLC	1ml / min 99%hexanes/IPA Chiralcel OD column
 (*endo*-borneol)	23.33 minor 23.62 major	GC	50° C hold 2min, ramp 4°C/min to 160°C
	47.81 major 48.31 minor	GC	50° C hold 2min, ramp 1°C/min to 160°C
 (*trans*-sobrerol)	16.61 major 16.73 minor	GC	50° C hold 2min, ramp 10°C/min to 200°C
	10.63 minor 12.36 major	HPLC	1ml / min 96%hexanes/IPA Chiralcel OD column

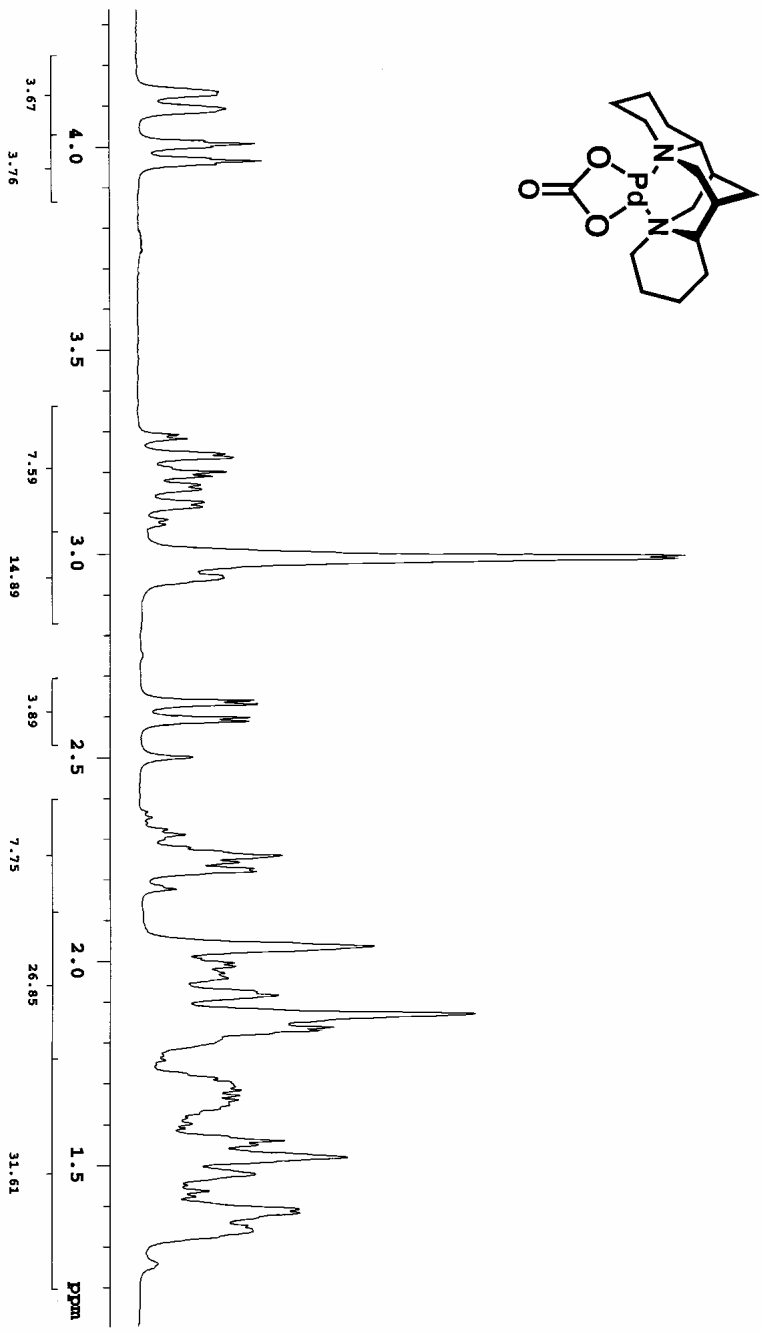
[a]All GC separations were performed using an autosampling HP 6890 GC fitted with a Hewlett Packard HP Chiral 20% Permethylated β–cyclodextrin column (0.25μm film thickness, 30m length, phase ratio 320) using a 2.0 mL/min carrier gas (H_2) flow rate

Alcohol	Retention Times (in minutes)	Method	Conditions[c]
	15.08 minor R-(+)[a] 15.23 major S-(-)	GC	50° C hold 5min, ramp 10°C/min to 150°C
	24.14 minor R-(+)[a] 24.38 major S-(-)	GC	50° C hold 2min, ramp 4°C/min to 160°C
	25.74 minor R-(+)[b] 26.30 major S-(-)	GC	50° C hold 2min, ramp 3.5°C/min
	12.39 minor R-(+)[b] 12.56 major S-(-)	GC	50° C hold 2min, ramp 10°C/min
	45.48 minor R-(+)[b] 45.76 major S-(-)	GC	50° C hold 5min, ramp 3.5°C/min to 220°C
	16.23 major 23.55 minor	HPLC	1ml / min 95%hexanes/IPA Chiralcel OD column
	25.00 minor R-(+)[b] 25.17 major S-(-)	GC	50° C hold 2min, ramp 5°C/min to 175°C
 α-tetralol	50.35 major 50.65 minor	GC	50° C hold 2min, ramp 2°C/min to 160°C

[a] Enantiomers assigned by comparison to authentic samples. [b] Enantiomers assigned by analogy [c]All GC separations were performed using an autosampling HP 6890 GC fitted with a Hewlett Packard HP Chiral 20% Permethylated β–cyclodextrin column (.25μm film thickness, 30m length, phase ratio 320) using a 2.0 mL/min carrier gas (H_2) flow rate

References

1. Mandal, S. K.; Jensen, D. R.; Pugsley, J. S.; Sigman, M. S. *J. Org. Chem.* **2003**, *68*, 4600.
2. For analytical data, see: Mueller, J. A.; Jensen, D. R.; Sigman, M. S. *J. Am. Chem. Soc.* **2002**, *124*, 8202-8203.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3.5
3.0
2.5
2.0
1.5
ppm
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3.76
7.59
14.89
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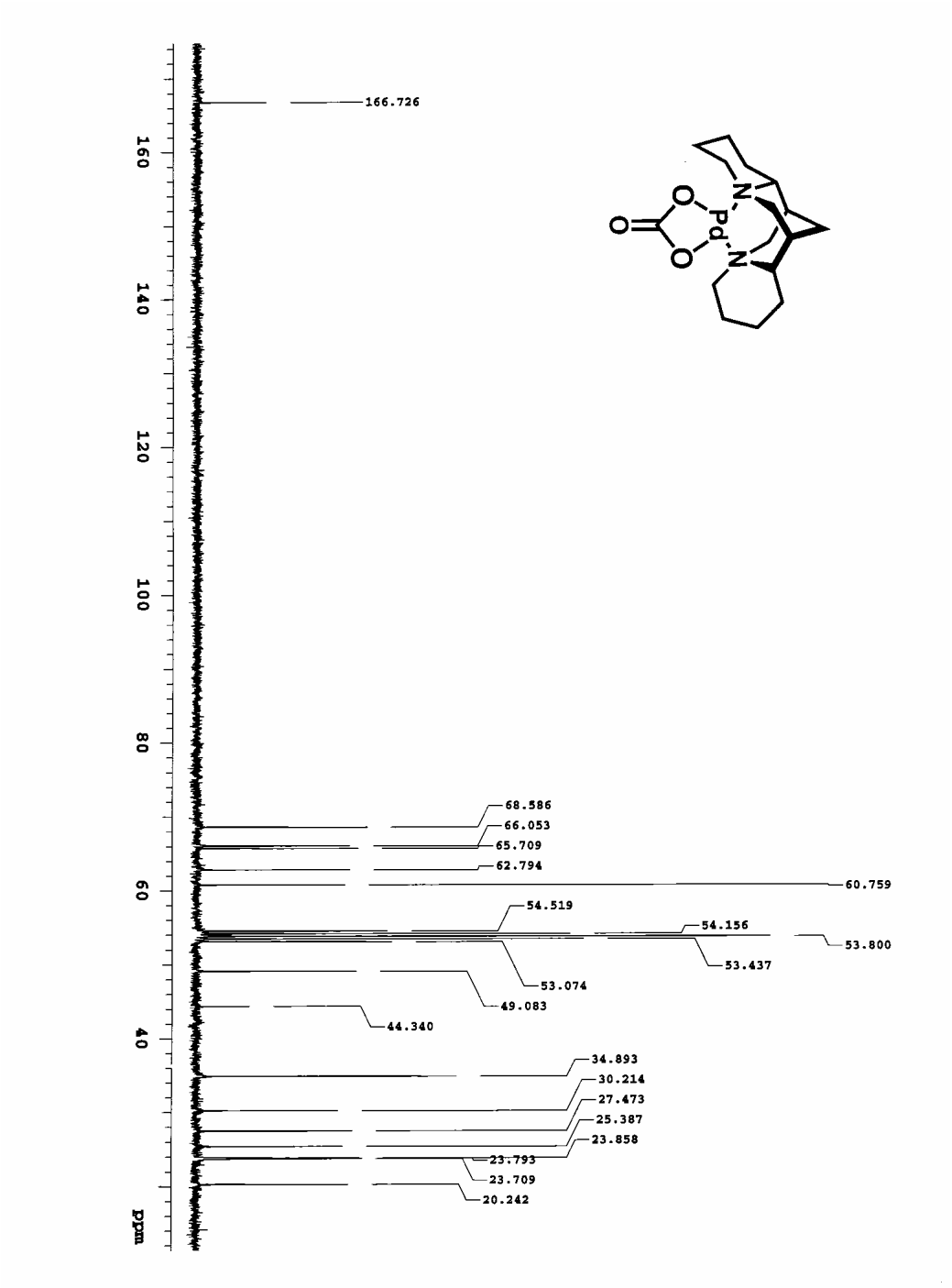
26.85
31.61
N
Pd
N
O
O
O


166.726
68.586
66.053
65.709
62.794
60.759
54.519
54.156
53.800
53.437
53.074
49.083
44.340
34.893
30.214
27.473
25.387
23.858
23.793
23.709
20.242
160
140
120
100
80
60
40
ppm
O
O
Pd
N
N

Crystal Structure Report for [(-)-sparteine]$PdCO_3$: An orange prism shaped crystal 0.33 x 0.30 x 0.20 mm in size was mounted on a glass fiber with traces of viscous oil and then transferred to a Nonius KappaCCD diffractometer equipped with Mo Kα radiation (λ = 0.71073 Å). Ten frames of data were collected at 150(1)K with an oscillation range of *1* deg/frame and an exposure time of *20* sec/frame.[1] Indexing and unit cell refinement based on all observed reflection from those ten frames, indicated an orthorhombic *P* lattice. A total of 3559 reflections (Θ_{max} = 27.49°) were indexed, integrated and corrected for Lorentz, polarization and absorption effects using DENZO-SMN and SCALEPAC.[2] Post refinement of the unit cell gave *a* = 9.1577(3) Å, b = 11.0566(4) Å, c = 15.6253(6) Å, and V = 1582.11(10) $Å^3$. Axial photographs and systematic absences were consistent with the compound having crystallized in the orthorhombic space group P212121.

The structure was solved by a combination of direct methods and heavy atom using SIR 97.[3] All of the non-hydrogen atoms were refined with anisotropic displacement coefficients. Hydrogen atoms were located and refined isotropically using SHELXL97.[4] The weighting scheme employed was $w = 1/[\sigma^2(F_o^2) + (0.0232P)^2 + 1.5848P]$ where $P = (F_o^2 + 2F_c^2)/3$. The refinement converged to R1 = 0.0272, wR2 = 0.0624, and S = 1.107 for 3403 reflections with $1 > 2\sigma(I)$, and R1 = 0.0297, wR2 = 0.064, and S = 1.107 for 3559 unique reflections and 305 parameters. [5] The maximum Δ/σ in the final cycle of the least-squares was 0.001, and the residual peaks on the final difference-Fourier map ranged from -1.076 to 0.898 e/$Å^3$. Scattering factors were taken from the International Tables for Crystallography, Volume C.[6,7]

(1) COLLECT Data Collection Software. Nonius B.V. 1998.

(2) Otwinowski, Z.; Minor, W., "Processing of X-ray Diffraction Data Collected in Oscillation Mode", Methods Enzymol. 1997, 276, 307-326.

(3) SIR97 (Release 1.02) - A program for automatic solution and refinement of crystal structure. A. Altomare, M.C. Burla, M. Camalli, G. Cascarano, C. Giacovazzo, A. Guagliardi, A.G. G. Moliteni, G. Polidori, and R. Spagna.

(4) SHELX97 [Includes SHELXS97, SHELXL97, CIFTAB] - Sheldrick, G. M. (1997). Programs for Crystal Structure Analysis (Release 97-2). University of Göttingen, Germany.

(5) R1 = Σ(|| F_o | – |F_c ||) / Σ |F_o|, wR2 = [Σ(w(F_o^2 – F_c^2)2) / Σ(F_o^2)^2]$^{1/2}$, and S = Goodness-of-fit on F^2 = [Σ(w(F_o^2 – F_c^2)^2 / (n-p)]$^{1/2}$, where n is the number of reflections and p is the number of parameters refined.

(6) Maslen, E. N.; Fox, A. G.; O'Keefe, M. A., International Tables for Crystallography: Mathemetical, Physical and Chemical Tables, Vol. C, Chapter 6, Wilson, A. J. C., Ed.; Kluwer, Dordrecht, The Netherlands, 1992; pp. 476-516.

(7) Creagh, D. C.; McdAuley, W. J., International Tables for Crystallography: mathematical, Physical and Chemical tables, Vol. C, Chapter 4 Wilson, A. J. C., Ed.; Kluwer, Dordrecht, The Netherlands, 1992; pp. 206-222.

(8) ORTEP3 for Windows - L. J. Farrugia, *J. Appl. Crystallogr*. **1997**, *30*, 565.

(9) WinGX A Windows Program for Crystal Structure Analysis. L. J. Farrugia, University of Glasgow, Glasgow, 1998.

Table 1. Crystal data and structure refinement for [(-)-sparteine]$PdCO_3$.

Identification code	mss015	
Empirical formula	C16 H26 N2 O3 Pd	
Formula weight	400.79	
Temperature	150(1) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$	
Unit cell dimensions	a = 9.1577(3) Å	α= 90°.
	b = 11.0566(4) Å	β= 90°.
	c = 15.6253(6) Å	γ = 90°.
Volume	1582.11(10) Å^3	
Z	4	
Density (calculated)	1.683 Mg/m^3	
Absorption coefficient	1.188 mm^{-1}	
F(000)	824	
Crystal size	0.33 x 0.30 x 0.20 mm^3	
Theta range for data collection	3.89 to 27.49°.	
Index ranges	-11<=h<=11, -14<=k<=14, -20<=l<=20	
Reflections collected	3559	
Independent reflections	3559 [R(int) = 0.0000]	
Completeness to theta = 27.49°	98.6 %	
Absorption correction	Multi-scan	
Max. and min. transmission	0.7971 and 0.6953	
Refinement method	Full-matrix least-squares on F^2	

Data / restraints / parameters	3559 / 0 / 305
Goodness-of-fit on F^2	1.107
Final R indices [I>2sigma(I)]	R1 = 0.0272, wR2 = 0.0624
R indices (all data)	R1 = 0.0297, wR2 = 0.0640
Absolute structure parameter	0.07(4)
Extinction coefficient	0.0058(9)
Largest diff. peak and hole	0.898 and -1.076 e.Å^{-3}

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for mss015. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Pd(1)	7136(1)	1730(1)	23(1)	21(1)
O(1)	6925(3)	2748(2)	-1039(2)	24(1)
O(2)	7397(3)	816(2)	-1089(2)	29(1)
O(3)	7168(3)	1855(2)	-2328(2)	34(1)
N(1)	7432(3)	447(3)	972(2)	27(1)
N(2)	6880(3)	3001(3)	984(2)	23(1)
C(1)	7738(5)	-772(3)	580(2)	35(1)
C(2)	6421(4)	-1281(3)	102(4)	42(1)
C(3)	5076(5)	-1316(4)	677(3)	47(1)
C(4)	4804(4)	-56(4)	1061(3)	39(1)
C(5)	6139(4)	348(3)	1558(2)	32(1)
C(6)	5929(4)	1520(4)	2070(2)	34(1)
C(7)	5614(4)	2635(3)	1536(2)	30(1)
C(8)	6538(4)	4161(3)	533(2)	30(1)
C(9)	6574(5)	5267(4)	1098(3)	41(1)
C(10)	8056(6)	5379(4)	1527(3)	50(1)
C(11)	8384(5)	4245(4)	2027(3)	45(1)
C(12)	8299(4)	3091(3)	1475(2)	30(1)
C(13)	8546(4)	1951(4)	2002(2)	36(1)
C(14)	8756(4)	806(4)	1478(2)	34(1)
C(15)	7267(5)	1732(5)	2624(2)	40(1)
C(16)	7163(3)	1811(3)	-1540(2)	24(1)

Table 3. Bond lengths [Å] and angles [°] for mss015.

Pd(1)-O(1)	2.015(3)
Pd(1)-O(2)	2.025(2)
Pd(1)-N(2)	2.069(3)
Pd(1)-N(1)	2.069(3)
Pd(1)-C(16)	2.445(3)
O(1)-C(16)	1.317(4)
O(2)-C(16)	1.324(4)
O(3)-C(16)	1.231(4)
N(1)-C(14)	1.501(5)
N(1)-C(5)	1.501(4)
N(1)-C(1)	1.506(4)
N(2)-C(8)	1.497(4)
N(2)-C(7)	1.501(5)
N(2)-C(12)	1.512(4)
C(1)-C(2)	1.527(6)
C(1)-H(1A)	0.99(4)
C(1)-H(1B)	0.96(4)
C(2)-C(3)	1.526(6)
C(2)-H(2A)	0.87(5)
C(2)-H(2B)	1.01(4)
C(3)-C(4)	1.537(6)
C(3)-H(3A)	0.86(5)
C(3)-H(3B)	0.94(4)
C(4)-C(5)	1.516(5)
C(4)-H(4A)	1.00(4)
C(4)-H(4B)	1.01(4)
C(5)-C(6)	1.535(5)
C(5)-H(5)	0.97(4)
C(6)-C(7)	1.517(5)
C(6)-C(15)	1.518(6)
C(6)-H(6)	0.99(5)
C(7)-H(7A)	1.02(4)
C(7)-H(7B)	0.93(4)
C(8)-C(9)	1.509(5)
C(8)-H(8A)	1.00(4)
C(8)-H(8B)	0.85(4)
C(9)-C(10)	1.519(7)
C(9)-H(9A)	1.06(4)
C(9)-H(9B)	0.91(5)
C(10)-C(11)	1.508(7)
C(10)-H(10A)	0.84(6)
C(10)-H(10B)	1.03(5)
C(11)-C(12)	1.542(5)
C(11)-H(11A)	1.01(5)
C(11)-H(11B)	1.06(5)
C(12)-C(13)	1.523(5)
C(12)-H(12)	1.02(4)
C(13)-C(14)	1.520(5)
C(13)-C(15)	1.541(6)
C(13)-H(13)	0.93(4)
C(14)-H(14A)	0.97(4)
C(14)-H(14B)	0.96(4)
C(15)-H(15A)	0.74(5)
C(15)-H(15B)	0.96(4)
O(1)-Pd(1)-O(2)	65.34(10)
O(1)-Pd(1)-N(2)	101.96(10)
O(2)-Pd(1)-N(2)	167.13(10)
O(1)-Pd(1)-N(1)	170.25(10)
O(2)-Pd(1)-N(1)	104.92(12)
N(2)-Pd(1)-N(1)	87.76(11)
O(1)-Pd(1)-C(16)	32.57(10)
O(2)-Pd(1)-C(16)	32.76(10)
N(2)-Pd(1)-C(16)	134.51(11)
N(1)-Pd(1)-C(16)	137.68(11)
C(16)-O(1)-Pd(1)	91.98(19)

C(16)-O(2)-Pd(1)	91.37(18)	N(1)-C(5)-C(4)	110.2(3)
C(14)-N(1)-C(5)	109.6(3)	N(1)-C(5)-C(6)	110.8(3)
C(14)-N(1)-C(1)	107.5(3)	C(4)-C(5)-C(6)	114.5(3)
C(5)-N(1)-C(1)	109.2(3)	N(1)-C(5)-H(5)	102(2)
C(14)-N(1)-Pd(1)	107.6(2)	C(4)-C(5)-H(5)	113(2)
C(5)-N(1)-Pd(1)	112.6(2)	C(6)-C(5)-H(5)	106(2)
C(1)-N(1)-Pd(1)	110.3(2)	C(7)-C(6)-C(15)	110.0(3)
C(8)-N(2)-C(7)	109.8(3)	C(7)-C(6)-C(5)	115.1(3)
C(8)-N(2)-C(12)	111.2(3)	C(15)-C(6)-C(5)	109.0(3)
C(7)-N(2)-C(12)	113.0(3)	C(7)-C(6)-H(6)	104(3)
C(8)-N(2)-Pd(1)	105.3(2)	C(15)-C(6)-H(6)	112(3)
C(7)-N(2)-Pd(1)	108.8(2)	C(5)-C(6)-H(6)	107(3)
C(12)-N(2)-Pd(1)	108.4(2)	N(2)-C(7)-C(6)	112.8(3)
N(1)-C(1)-C(2)	112.4(3)	N(2)-C(7)-H(7A)	109(2)
N(1)-C(1)-H(1A)	108(2)	C(6)-C(7)-H(7A)	114.3(18)
C(2)-C(1)-H(1A)	112(2)	N(2)-C(7)-H(7B)	105(2)
N(1)-C(1)-H(1B)	107(2)	C(6)-C(7)-H(7B)	112(2)
C(2)-C(1)-H(1B)	111(3)	H(7A)-C(7)-H(7B)	103(3)
H(1A)-C(1)-H(1B)	106(3)	N(2)-C(8)-C(9)	114.5(3)
C(3)-C(2)-C(1)	111.0(4)	N(2)-C(8)-H(8A)	107(2)
C(3)-C(2)-H(2A)	106(4)	C(9)-C(8)-H(8A)	111(2)
C(1)-C(2)-H(2A)	103(3)	N(2)-C(8)-H(8B)	114(2)
C(3)-C(2)-H(2B)	112(3)	C(9)-C(8)-H(8B)	101(2)
C(1)-C(2)-H(2B)	113(3)	H(8A)-C(8)-H(8B)	110(3)
H(2A)-C(2)-H(2B)	112(5)	C(8)-C(9)-C(10)	110.1(3)
C(2)-C(3)-C(4)	109.7(3)	C(8)-C(9)-H(9A)	110(2)
C(2)-C(3)-H(3A)	109(3)	C(10)-C(9)-H(9A)	112(2)
C(4)-C(3)-H(3A)	109(3)	C(8)-C(9)-H(9B)	108(3)
C(2)-C(3)-H(3B)	112(3)	C(10)-C(9)-H(9B)	114(3)
C(4)-C(3)-H(3B)	108(3)	H(9A)-C(9)-H(9B)	102(4)
H(3A)-C(3)-H(3B)	109(4)	C(11)-C(10)-C(9)	109.8(4)
C(5)-C(4)-C(3)	109.7(3)	C(11)-C(10)-H(10A)	103(4)
C(5)-C(4)-H(4A)	105(2)	C(9)-C(10)-H(10A)	115(4)
C(3)-C(4)-H(4A)	119(2)	C(11)-C(10)-H(10B)	100(3)
C(5)-C(4)-H(4B)	111(3)	C(9)-C(10)-H(10B)	121(3)
C(3)-C(4)-H(4B)	109(2)	H(10A)-C(10)-H(10B)	106(5)
H(4A)-C(4)-H(4B)	103(3)	C(10)-C(11)-C(12)	112.8(3)

C(10)-C(11)-H(11A)	113(3)	N(1)-C(14)-H(14A)	109(3)
C(12)-C(11)-H(11A)	109(3)	C(13)-C(14)-H(14A)	109(2)
C(10)-C(11)-H(11B)	116(3)	N(1)-C(14)-H(14B)	106(2)
C(12)-C(11)-H(11B)	102(3)	C(13)-C(14)-H(14B)	115(2)
H(11A)-C(11)-H(11B)	103(4)	H(14A)-C(14)-H(14B)	104(3)
N(2)-C(12)-C(13)	110.3(3)	C(6)-C(15)-C(13)	106.2(3)
N(2)-C(12)-C(11)	112.5(3)	C(6)-C(15)-H(15A)	114(4)
C(13)-C(12)-C(11)	112.0(3)	C(13)-C(15)-H(15A)	111(4)
N(2)-C(12)-H(12)	105.6(19)	C(6)-C(15)-H(15B)	112(3)
C(13)-C(12)-H(12)	113(2)	C(13)-C(15)-H(15B)	109(3)
C(11)-C(12)-H(12)	103(2)	H(15A)-C(15)-H(15B)	105(4)
C(14)-C(13)-C(12)	114.6(3)	O(3)-C(16)-O(1)	124.3(3)
C(14)-C(13)-C(15)	107.8(3)	O(3)-C(16)-O(2)	124.4(3)
C(12)-C(13)-C(15)	111.0(3)	O(1)-C(16)-O(2)	111.3(3)
C(14)-C(13)-H(13)	106(2)	O(3)-C(16)-Pd(1)	179.6(3)
C(12)-C(13)-H(13)	108(2)	O(1)-C(16)-Pd(1)	55.45(16)
C(15)-C(13)-H(13)	109(2)	O(2)-C(16)-Pd(1)	55.87(15)
N(1)-C(14)-C(13)	113.7(3)		

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2$x 10^3)for mss015. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Pd(1)	22(1)	20(1)	20(1)	1(1)	2(1)	1(1)
O(1)	31(1)	19(1)	23(1)	0(1)	0(1)	2(1)
O(2)	42(2)	19(1)	26(1)	1(1)	4(1)	0(1)
O(3)	43(1)	34(1)	25(1)	-2(1)	1(1)	3(1)
N(1)	27(2)	26(1)	28(2)	8(1)	3(1)	3(1)
N(2)	24(2)	23(1)	22(1)	-2(1)	1(1)	-1(1)
C(1)	45(2)	23(2)	36(2)	10(1)	6(2)	8(2)
C(2)	49(2)	24(1)	52(3)	8(2)	8(2)	-3(1)
C(3)	51(2)	33(2)	55(3)	14(2)	-1(2)	-13(2)
C(4)	33(2)	38(2)	46(2)	11(2)	6(2)	-6(2)
C(5)	33(2)	35(2)	29(2)	13(2)	7(1)	1(1)
C(6)	27(2)	46(2)	29(2)	9(2)	9(1)	4(2)
C(7)	29(2)	34(2)	27(2)	2(1)	6(1)	5(1)
C(8)	41(2)	21(2)	29(2)	1(1)	2(2)	3(1)
C(9)	52(2)	28(2)	43(2)	-9(2)	5(2)	4(2)
C(10)	63(3)	35(2)	52(3)	-19(2)	3(2)	-8(2)
C(11)	42(2)	53(2)	39(2)	-15(2)	-5(2)	-3(2)
C(12)	29(2)	38(2)	24(2)	-5(1)	-1(1)	-1(1)
C(13)	31(2)	51(2)	24(2)	1(2)	-4(1)	7(2)
C(14)	31(2)	41(2)	31(2)	7(2)	-3(2)	7(2)
C(15)	44(2)	50(2)	25(2)	9(2)	1(2)	7(2)
C(16)	24(1)	20(1)	27(2)	0(1)	2(1)	-1(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for mss015.

	x	y	z	U(eq)
H(1A)	8070(40)	-1320(40)	1040(20)	31(9)
H(1B)	8560(50)	-680(40)	210(30)	46(12)
H(2A)	6670(50)	-2030(40)	20(40)	65(13)
H(2B)	6220(50)	-850(40)	-460(30)	41(12)
H(3A)	5220(50)	-1820(50)	1090(30)	52(13)
H(3B)	4230(50)	-1550(40)	370(30)	42(11)
H(4A)	3960(40)	60(40)	1460(20)	31(10)
H(4B)	4570(50)	530(40)	580(30)	47(12)
H(5)	6470(40)	-260(40)	1970(20)	35(10)
H(6)	5030(60)	1410(40)	2420(30)	57(14)
H(7A)	4700(40)	2570(30)	1160(20)	22(9)
H(7B)	5420(40)	3300(40)	1880(20)	28(9)
H(8A)	5550(50)	4060(40)	260(30)	45(12)
H(8B)	7160(40)	4350(30)	150(30)	29(10)
H(9A)	5720(50)	5230(40)	1550(30)	45(11)
H(9B)	6320(50)	5920(40)	770(30)	48(13)
H(10A)	8110(60)	5920(50)	1900(40)	70(17)
H(10B)	9000(60)	5440(40)	1170(30)	50(13)
H(11A)	7740(60)	4140(40)	2550(30)	58(14)
H(11B)	9450(60)	4190(40)	2290(30)	55(13)
H(12)	9090(40)	3230(30)	1020(20)	23(8)
H(13)	9400(40)	2050(30)	2320(20)	27(9)
H(14A)	9010(50)	150(40)	1860(30)	44(11)
H(14B)	9560(40)	840(30)	1080(30)	25(9)
H(15A)	7430(50)	1230(40)	2920(30)	36(12)
H(15B)	7160(50)	2430(40)	2990(30)	36(11)

Table 6. Torsion angles [°] for mss015.

O(2)-Pd(1)-O(1)-C(16)	0.26(18)
N(2)-Pd(1)-O(1)-C(16)	178.1(2)
N(1)-Pd(1)-O(1)-C(16)	2.7(8)
O(1)-Pd(1)-O(2)-C(16)	-0.26(18)
N(2)-Pd(1)-O(2)-C(16)	-10.0(6)
N(1)-Pd(1)-O(2)-C(16)	-179.8(2)
O(1)-Pd(1)-N(1)-C(14)	114.2(7)
O(2)-Pd(1)-N(1)-C(14)	116.5(2)
N(2)-Pd(1)-N(1)-C(14)	-61.2(2)
C(16)-Pd(1)-N(1)-C(14)	116.4(2)
O(1)-Pd(1)-N(1)-C(5)	-125.0(6)
O(2)-Pd(1)-N(1)-C(5)	-122.7(2)
N(2)-Pd(1)-N(1)-C(5)	59.6(2)
C(16)-Pd(1)-N(1)-C(5)	-122.8(2)
O(1)-Pd(1)-N(1)-C(1)	-2.7(8)
O(2)-Pd(1)-N(1)-C(1)	-0.4(2)
N(2)-Pd(1)-N(1)-C(1)	-178.1(3)
C(16)-Pd(1)-N(1)-C(1)	-0.5(3)
O(1)-Pd(1)-N(2)-C(8)	3.7(2)
O(2)-Pd(1)-N(2)-C(8)	12.8(7)
N(1)-Pd(1)-N(2)-C(8)	-177.0(2)
C(16)-Pd(1)-N(2)-C(8)	5.2(3)
O(1)-Pd(1)-N(2)-C(7)	121.4(2)
O(2)-Pd(1)-N(2)-C(7)	130.5(5)
N(1)-Pd(1)-N(2)-C(7)	-59.3(2)
C(16)-Pd(1)-N(2)-C(7)	122.9(2)
O(1)-Pd(1)-N(2)-C(12)	-115.4(2)
O(2)-Pd(1)-N(2)-C(12)	-106.4(5)
N(1)-Pd(1)-N(2)-C(12)	63.8(2)
C(16)-Pd(1)-N(2)-C(12)	-113.9(2)
C(14)-N(1)-C(1)-C(2)	175.7(3)
C(5)-N(1)-C(1)-C(2)	56.9(4)
Pd(1)-N(1)-C(1)-C(2)	-67.3(3)
N(1)-C(1)-C(2)-C(3)	-54.3(4)
C(1)-C(2)-C(3)-C(4)	53.9(5)

C(2)-C(3)-C(4)-C(5)	-58.0(5)
C(14)-N(1)-C(5)-C(4)	-178.1(3)
C(1)-N(1)-C(5)-C(4)	-60.6(4)
Pd(1)-N(1)-C(5)-C(4)	62.3(3)
C(14)-N(1)-C(5)-C(6)	54.2(4)
C(1)-N(1)-C(5)-C(6)	171.6(3)
Pd(1)-N(1)-C(5)-C(6)	-65.5(3)
C(3)-C(4)-C(5)-N(1)	62.0(4)
C(3)-C(4)-C(5)-C(6)	-172.4(3)
N(1)-C(5)-C(6)-C(7)	62.1(4)
C(4)-C(5)-C(6)-C(7)	-63.2(4)
N(1)-C(5)-C(6)-C(15)	-62.0(4)
C(4)-C(5)-C(6)-C(15)	172.7(3)
C(8)-N(2)-C(7)-C(6)	-175.3(3)
C(12)-N(2)-C(7)-C(6)	-50.5(4)
Pd(1)-N(2)-C(7)-C(6)	69.9(3)
C(15)-C(6)-C(7)-N(2)	57.1(4)
C(5)-C(6)-C(7)-N(2)	-66.5(4)
C(7)-N(2)-C(8)-C(9)	73.6(4)
C(12)-N(2)-C(8)-C(9)	-52.2(4)
Pd(1)-N(2)-C(8)-C(9)	-169.5(3)
N(2)-C(8)-C(9)-C(10)	56.8(5)
C(8)-C(9)-C(10)-C(11)	-56.9(5)
C(9)-C(10)-C(11)-C(12)	55.5(5)
C(8)-N(2)-C(12)-C(13)	174.1(3)
C(7)-N(2)-C(12)-C(13)	50.1(4)
Pd(1)-N(2)-C(12)-C(13)	-70.6(3)
C(8)-N(2)-C(12)-C(11)	48.3(4)
C(7)-N(2)-C(12)-C(11)	-75.8(4)
Pd(1)-N(2)-C(12)-C(11)	163.6(3)
C(10)-C(11)-C(12)-N(2)	-51.7(5)
C(10)-C(11)-C(12)-C(13)	-176.7(4)
N(2)-C(12)-C(13)-C(14)	64.7(4)
C(11)-C(12)-C(13)-C(14)	-169.2(3)
N(2)-C(12)-C(13)-C(15)	-57.7(4)
C(11)-C(12)-C(13)-C(15)	68.4(4)
C(5)-N(1)-C(14)-C(13)	-54.4(4)

C(1)-N(1)-C(14)-C(13)	-172.9(3)
Pd(1)-N(1)-C(14)-C(13)	68.3(3)
C(12)-C(13)-C(14)-N(1)	-64.9(4)
C(15)-C(13)-C(14)-N(1)	59.2(4)
C(7)-C(6)-C(15)-C(13)	-61.9(4)
C(5)-C(6)-C(15)-C(13)	65.2(4)
C(14)-C(13)-C(15)-C(6)	-62.8(4)
C(12)-C(13)-C(15)-C(6)	63.5(4)
Pd(1)-O(1)-C(16)-O(3)	179.6(3)
Pd(1)-O(1)-C(16)-O(2)	-0.4(3)
Pd(1)-O(2)-C(16)-O(3)	-179.6(3)
Pd(1)-O(2)-C(16)-O(1)	0.4(3)
O(1)-Pd(1)-C(16)-O(3)	-53(39)
O(2)-Pd(1)-C(16)-O(3)	128(39)
N(2)-Pd(1)-C(16)-O(3)	-56(39)
N(1)-Pd(1)-C(16)-O(3)	128(39)
O(2)-Pd(1)-C(16)-O(1)	-179.6(3)
N(2)-Pd(1)-C(16)-O(1)	-2.7(3)
N(1)-Pd(1)-C(16)-O(1)	-179.3(2)
O(1)-Pd(1)-C(16)-O(2)	179.6(3)
N(2)-Pd(1)-C(16)-O(2)	176.90(19)
N(1)-Pd(1)-C(16)-O(2)	0.2(3)

Symmetry transformations used to generate equivalent atoms: