Appendix 2

X-Ray Crystallographic Data Relevant to Chapter 2

CALIFORNIA INSTITUTE OF TECHNOLOGY

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X-RAY CRYSTALLOGRAPHY LABORATORY

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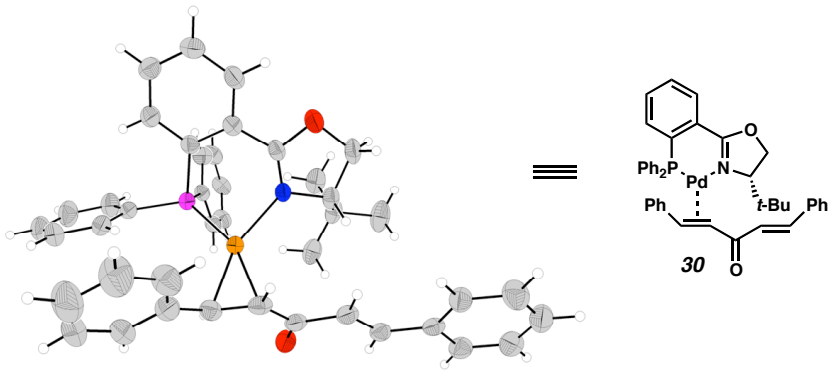
Crystal Structure Analysis of:

[{(*S*)-*t*-BuPHOX}Pd(0)(dba)] 30

(NHS03, 606912)

Contents

Figure A2.1. Representation of [{(S)-t-BuPHOX}Pd(0)(dba)] ***30***



Note: Crystallographic data have been deposited at the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK, and copies can be obtained on request, free of charge, by quoting the publication citation and the deposition number 606912.

Table A2.1. Crystal Data and Structure Refinement for NHS03 (CCDC 606912).

Empirical formula	$C_{42}H_{40}NO_2PPd \bullet C_4H_8O$
Formula weight	800.22
Crystallization solvent	THF
Crystal habit	Block
Crystal size	0.26 x 0.25 x 0.25 mm^3
Crystal color	Orange

Data Collection

Type of diffractometer	Bruker SMART 1000
Wavelength	0.71073 Å MoKα
Data collection temperature	100(2) K
θ range for 25286 reflections used in lattice determination	2.45 to 29.83°
Unit cell dimensions	a = 10.3986(4) Å b = 13.8720(5) Å c = 27.4970(10) Å
Volume	3966.4(3) $Å^3$
Z	4
Crystal system	Orthorhombic
Space group	$P2_12_12_1$
Density (calculated)	1.340 Mg/m^3
F(000)	1664
θ range for data collection	2.09 to 32.87°
Completeness to θ = 32.87°	94.5 %
Index ranges	$-15 \le h \le 15$, $-19 \le k \le 20$, $-35 \le l \le 41$
Data collection scan type	ω scans at 5 ϕ settings
Reflections collected	66411
Independent reflections	13693 [R_{int}= 0.0693]
Absorption coefficient	0.549 mm^{-1}
Absorption correction	None
Max. and min. transmission	0.8749 and 0.8704

Table A2.1. (cont.)

Structure Solution and Refinement

Structure solution program	Bruker XS v6.12
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	Bruker XL v6.12
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	13693 / 39 / 468
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	1.482
Final R indices [I>2σ(I), 10165 reflections]	R1 = 0.0400, *w*R2 = 0.0643
R indices (all data)	R1 = 0.0642, *w*R2 = 0.0672
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.005
Average shift/error	0.000
Absolute structure parameter	-0.048(16)
Largest diff. peak and hole	0.710 and -0.467 e.Å^{-3}

Special Refinement Details

The crystal contains solvent of crystallization, disordered at one site in the unit cell. The solvent was modeled as THF and included in least-squares refinement with geometric restraints on all of the THF atoms. The temperature factors were allowed to refine and the values reflect the diffuse nature of the electron density in the area.

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt), etc., and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles, and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Table A2.2. Atomic Coordinates (x 10^4) and Equivalent Isotropic Displacement Parameters ($Å^2$x 10^3) for NHS03 (CCDC 606912). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}	Occ
Pd(1)	9271(1)	8680(1)	9206(1)	30(1)	1
P(1)	9236(1)	9154(1)	8415(1)	32(1)	1
O(1)	11984(2)	6645(1)	8627(1)	46(1)	1
O(2)	7180(2)	7972(1)	10054(1)	45(1)	1
N(1)	10248(2)	7406(1)	8929(1)	31(1)	1
C(1)	10164(2)	6412(2)	9132(1)	37(1)	1
C(2)	11452(3)	5964(2)	8971(1)	44(1)	1
C(3)	11234(2)	7448(2)	8658(1)	32(1)	1
C(4)	11688(2)	8257(2)	8353(1)	31(1)	1
C(5)	12979(3)	8238(2)	8210(1)	39(1)	1
C(6)	13476(3)	8930(2)	7908(1)	42(1)	1
C(7)	12700(3)	9663(2)	7737(1)	41(1)	1
C(8)	11424(2)	9710(2)	7883(1)	38(1)	1
C(9)	10896(2)	9024(2)	8197(1)	33(1)	1
C(10)	8947(3)	5866(2)	8963(1)	44(1)	1
C(11)	7739(2)	6394(2)	9129(1)	51(1)	1
C(12)	8987(3)	4861(2)	9206(1)	66(1)	1
C(13)	8935(3)	5739(2)	8411(1)	55(1)	1
C(14)	8547(3)	11428(2)	9696(1)	58(1)	1
C(15)	9116(4)	12335(2)	9681(1)	70(1)	1
C(16)	10441(4)	12418(3)	9713(2)	84(1)	1
C(17)	11151(3)	11607(3)	9776(1)	77(1)	1
C(18)	10594(3)	10699(2)	9795(1)	58(1)	1
C(19)	9275(3)	10595(2)	9753(1)	43(1)	1
C(20)	8641(3)	9643(2)	9754(1)	38(1)	1
C(21)	9157(2)	8812(2)	9977(1)	37(1)	1
C(22)	8364(2)	8002(2)	10116(1)	37(1)	1
C(23)	9048(2)	7173(2)	10344(1)	36(1)	1
C(24)	8433(2)	6431(2)	10532(1)	36(1)	1
C(25)	9022(2)	5584(2)	10767(1)	35(1)	1
C(26)	8230(3)	4863(2)	10959(1)	45(1)	1
C(27)	8749(3)	4058(2)	11181(1)	51(1)	1
C(28)	10073(3)	3972(2)	11229(1)	53(1)	1
C(29)	10846(3)	4680(2)	11039(1)	53(1)	1
C(30)	10341(2)	5470(2)	10816(1)	41(1)	1
C(31)	8793(2)	10365(2)	8215(1)	37(1)	1
C(32)	8054(3)	10537(2)	7810(1)	52(1)	1
C(33)	7702(3)	11478(2)	7685(1)	62(1)	1
C(34)	8095(3)	12226(2)	7969(1)	59(1)	1
C(35)	8856(3)	12077(2)	8372(1)	58(1)	1
C(36)	9188(3)	11135(2)	8495(1)	43(1)	1
C(37)	8328(2)	8378(2)	8005(1)	32(1)	1
C(38)	8799(2)	8048(2)	7558(1)	40(1)	1
C(39)	8057(3)	7464(2)	7268(1)	50(1)	1
C(40)	6833(3)	7191(2)	7407(1)	46(1)	1
C(41)	6351(3)	7505(2)	7850(1)	46(1)	1
C(42)	7095(3)	8090(2)	8145(1)	39(1)	1

C11	3074(4)	3874(4)	8577(2)	72(2)	0.661(5)
C21	3790(5)	3787(5)	8994(2)	103(2)	0.661(5)
C31	5064(6)	3878(11)	8871(3)	355(12)	0.661(5)
C41	5137(8)	4356(9)	8435(3)	192(5)	0.661(5)
O11	3826(6)	4525(5)	8282(2)	163(3)	0.661(5)
C12	5073(7)	3895(5)	8910(2)	43(2)	0.339(5)
C22	4005(9)	4464(7)	8849(4)	148(7)	0.339(5)
C32	4284(11)	5145(7)	8502(3)	95(4)	0.339(5)
C42	5277(10)	4803(6)	8223(4)	79(4)	0.339(5)
O12	5633(8)	3881(5)	8428(3)	109(3)	0.339(5)

Table A2.3. Selected Bond Lengths [Å] and Angles [°] for NHS03 (CCDC 606912)

Pd(1)-C(20)	2.118(2)
Pd(1)-C(21)	2.131(2)
Pd(1)-N(1)	2.1753(19)
Pd(1)-P(1)	2.2730(6)
C(20)-Pd(1)-C(21)	38.81(9)
C(20)-Pd(1)-N(1)	154.96(9)
C(21)-Pd(1)-N(1)	116.38(9)
C(20)-Pd(1)-P(1)	119.55(7)
C(21)-Pd(1)-P(1)	157.84(7)
N(1)-Pd(1)-P(1)	84.69(5)

Table A2.4. Bond lengths [Å] and angles [°] for NHS03 (CCDC 606912).

Pd(1)-C(20)	2.118(2)
Pd(1)-C(21)	2.131(2)
Pd(1)-N(1)	2.1753(19)
Pd(1)-P(1)	2.2730(6)
P(1)-C(37)	1.822(3)
P(1)-C(31)	1.826(2)
P(1)-C(9)	1.836(3)
O(1)-C(3)	1.362(3)
O(1)-C(2)	1.447(3)
O(2)-C(22)	1.243(3)
N(1)-C(3)	1.269(3)
N(1)-C(1)	1.491(3)
C(1)-C(2)	1.542(4)
C(1)-C(10)	1.546(3)
C(3)-C(4)	1.479(3)
C(4)-C(5)	1.399(3)
C(4)-C(9)	1.413(3)
C(5)-C(6)	1.371(4)
C(6)-C(7)	1.380(4)
C(7)-C(8)	1.388(4)
C(8)-C(9)	1.398(3)
C(10)-C(11)	1.524(4)
C(10)-C(13)	1.528(4)
C(10)-C(12)	1.547(3)
C(14)-C(15)	1.391(4)
C(14)-C(19)	1.390(4)
C(15)-C(16)	1.386(5)
C(16)-C(17)	1.356(5)
C(17)-C(18)	1.387(4)
C(18)-C(19)	1.384(5)
C(19)-C(20)	1.476(4)
C(20)-C(21)	1.412(3)
C(21)-C(22)	1.446(3)
C(22)-C(23)	1.490(3)
C(23)-C(24)	1.317(3)
C(24)-C(25)	1.475(3)
C(25)-C(26)	1.399(3)
C(25)-C(30)	1.388(3)
C(26)-C(27)	1.382(4)
C(27)-C(28)	1.388(4)
C(28)-C(29)	1.371(4)
C(29)-C(30)	1.361(4)
C(31)-C(36)	1.378(3)
C(31)-C(32)	1.374(4)
C(32)-C(33)	1.399(4)
C(33)-C(34)	1.362(4)
C(34)-C(35)	1.376(4)
C(35)-C(36)	1.393(4)
C(37)-C(42)	1.396(3)
C(37)-C(38)	1.400(3)

C(38)-C(39)	1.374(4)
C(39)-C(40)	1.381(4)
C(40)-C(41)	1.390(4)
C(41)-C(42)	1.383(4)
C11-O11	1.445(6)
C11-C21	1.371(4)
C21-C31	1.373(5)
C31-C41	1.373(5)
C41-O11	1.446(6)
C12-C22	1.373(5)
C12-O12	1.446(6)
C22-C32	1.373(5)
C32-C42	1.372(5)
C42-O12	1.446(6)

C(20)-Pd(1)-C(21)	38.81(9)
C(20)-Pd(1)-N(1)	154.96(9)
C(21)-Pd(1)-N(1)	116.38(9)
C(20)-Pd(1)-P(1)	119.55(7)
C(21)-Pd(1)-P(1)	157.84(7)
N(1)-Pd(1)-P(1)	84.69(5)
C(37)-P(1)-C(31)	103.10(12)
C(37)-P(1)-C(9)	103.12(11)
C(31)-P(1)-C(9)	103.22(11)
C(37)-P(1)-Pd(1)	115.45(8)
C(31)-P(1)-Pd(1)	123.91(9)
C(9)-P(1)-Pd(1)	105.63(8)
C(3)-O(1)-C(2)	105.89(19)
C(3)-N(1)-C(1)	108.0(2)
C(3)-N(1)-Pd(1)	123.10(17)
C(1)-N(1)-Pd(1)	126.36(15)
N(1)-C(1)-C(2)	102.37(19)
N(1)-C(1)-C(10)	112.85(19)
C(2)-C(1)-C(10)	115.3(2)
O(1)-C(2)-C(1)	104.91(19)
N(1)-C(3)-O(1)	117.5(2)
N(1)-C(3)-C(4)	128.7(2)
O(1)-C(3)-C(4)	113.7(2)
C(5)-C(4)-C(9)	119.2(2)
C(5)-C(4)-C(3)	116.8(2)
C(9)-C(4)-C(3)	123.9(2)
C(6)-C(5)-C(4)	121.3(2)
C(7)-C(6)-C(5)	120.1(2)
C(6)-C(7)-C(8)	119.7(3)
C(7)-C(8)-C(9)	121.5(2)
C(8)-C(9)-C(4)	118.1(2)
C(8)-C(9)-P(1)	120.33(18)
C(4)-C(9)-P(1)	121.50(18)
C(11)-C(10)-C(13)	110.3(2)
C(11)-C(10)-C(1)	110.4(2)
C(13)-C(10)-C(1)	111.2(2)
C(11)-C(10)-C(12)	109.0(2)
C(13)-C(10)-C(12)	109.0(2)
C(1)-C(10)-C(12)	106.8(2)

C(15)-C(14)-C(19)	121.6(3)
C(14)-C(15)-C(16)	119.7(3)
C(17)-C(16)-C(15)	118.7(3)
C(16)-C(17)-C(18)	122.1(3)
C(19)-C(18)-C(17)	120.4(3)
C(18)-C(19)-C(14)	117.5(3)
C(18)-C(19)-C(20)	122.4(3)
C(14)-C(19)-C(20)	120.1(3)
C(21)-C(20)-C(19)	124.2(2)
C(21)-C(20)-Pd(1)	71.09(13)
C(19)-C(20)-Pd(1)	115.13(18)
C(20)-C(21)-C(22)	122.2(2)
C(20)-C(21)-Pd(1)	70.10(14)
C(22)-C(21)-Pd(1)	103.12(16)
O(2)-C(22)-C(21)	123.6(2)
O(2)-C(22)-C(23)	120.3(2)
C(21)-C(22)-C(23)	116.1(2)
C(24)-C(23)-C(22)	122.4(2)
C(23)-C(24)-C(25)	126.4(2)
C(26)-C(25)-C(30)	117.7(2)
C(26)-C(25)-C(24)	119.3(2)
C(30)-C(25)-C(24)	123.0(2)
C(27)-C(26)-C(25)	120.9(3)
C(26)-C(27)-C(28)	119.9(3)
C(29)-C(28)-C(27)	118.9(3)
C(30)-C(29)-C(28)	121.5(3)
C(29)-C(30)-C(25)	121.1(3)
C(36)-C(31)-C(32)	118.9(2)
C(36)-C(31)-P(1)	118.1(2)
C(32)-C(31)-P(1)	123.0(2)
C(31)-C(32)-C(33)	120.6(3)
C(34)-C(33)-C(32)	119.4(3)
C(33)-C(34)-C(35)	121.3(3)
C(34)-C(35)-C(36)	118.6(3)
C(31)-C(36)-C(35)	121.2(3)
C(42)-C(37)-C(38)	118.0(2)
C(42)-C(37)-P(1)	118.3(2)
C(38)-C(37)-P(1)	123.69(19)
C(39)-C(38)-C(37)	120.4(2)
C(40)-C(39)-C(38)	121.3(3)
C(39)-C(40)-C(41)	119.2(3)
C(42)-C(41)-C(40)	119.8(3)
C(41)-C(42)-C(37)	121.4(3)
O11-C11-C21	103.3(4)
C31-C21-C11	108.1(3)
C21-C31-C41	108.2(3)
O11-C41-C31	106.3(4)
C41-O11-C11	104.2(6)
C22-C12-O12	102.9(4)
C12-C22-C32	108.0(3)
C42-C32-C22	108.0(3)
C32-C42-O12	106.3(4)

Table A2.5. Anisotropic Displacement Parameters ($Å^2 x\ 10^4$) for NHS03 (CCDC 606912). The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$.

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Pd(1)	313(1)	282(1)	309(1)	15(1)	6(1)	5(1)
P(1)	328(3)	294(3)	330(3)	38(2)	14(3)	24(3)
O(1)	536(11)	376(11)	468(11)	33(8)	14(9)	137(9)
O(2)	299(10)	538(12)	527(12)	145(10)	34(9)	35(9)
N(1)	388(11)	244(10)	299(11)	13(9)	-22(9)	15(9)
C(1)	531(14)	261(12)	320(14)	2(12)	-76(11)	11(12)
C(2)	659(18)	255(13)	409(16)	33(12)	-69(14)	101(13)
C(3)	408(14)	259(13)	304(14)	-33(11)	-52(12)	70(11)
C(4)	333(13)	333(13)	276(13)	-34(11)	-32(11)	21(11)
C(5)	403(16)	349(14)	424(16)	-12(13)	4(13)	94(12)
C(6)	347(14)	457(17)	458(16)	-68(13)	41(12)	3(12)
C(7)	430(16)	423(15)	372(16)	10(12)	59(12)	-36(13)
C(8)	350(14)	380(15)	394(16)	48(12)	-12(12)	32(12)
C(9)	359(14)	319(11)	301(12)	-13(9)	5(11)	17(11)
C(10)	650(20)	307(13)	364(14)	39(11)	-98(13)	-81(13)
C(11)	540(16)	459(15)	518(18)	112(16)	-143(14)	-145(15)
C(12)	920(30)	358(14)	710(20)	117(16)	-260(20)	-181(15)
C(13)	800(30)	424(16)	409(16)	-31(13)	-168(16)	-81(15)
C(14)	620(18)	459(18)	670(20)	-15(17)	148(16)	101(16)
C(15)	860(30)	366(17)	860(30)	-44(16)	170(20)	13(19)
C(16)	990(30)	470(20)	1060(30)	-150(20)	10(30)	-160(20)
C(17)	630(20)	600(20)	1080(30)	-60(20)	-90(20)	-197(18)
C(18)	513(19)	516(18)	720(20)	-100(15)	-23(18)	-52(17)
C(19)	471(15)	405(14)	411(15)	-59(11)	-15(16)	-64(16)
C(20)	393(14)	406(15)	351(15)	-40(12)	3(12)	40(12)
C(21)	275(12)	529(15)	297(12)	-18(11)	26(11)	39(14)
C(22)	325(15)	495(16)	286(14)	13(12)	43(11)	35(12)
C(23)	319(15)	479(14)	288(13)	14(11)	15(11)	32(12)
C(24)	318(12)	475(16)	280(12)	1(12)	-1(10)	47(13)
C(25)	360(15)	423(13)	268(12)	-19(12)	29(12)	-4(10)
C(26)	356(15)	571(18)	412(17)	51(13)	42(12)	7(13)
C(27)	579(18)	479(18)	483(18)	93(14)	56(15)	2(15)
C(28)	630(20)	484(19)	479(18)	95(14)	-82(16)	66(16)
C(29)	395(16)	591(18)	608(18)	58(15)	-99(16)	24(16)
C(30)	382(14)	438(14)	410(15)	67(14)	-19(13)	1(11)
C(31)	346(14)	337(14)	421(16)	86(12)	101(12)	44(11)
C(32)	590(20)	433(17)	521(19)	82(14)	-87(15)	31(15)
C(33)	594(19)	490(20)	790(20)	235(18)	-49(17)	65(17)
C(34)	570(20)	404(18)	790(30)	231(17)	96(19)	106(15)
C(35)	690(20)	340(16)	700(20)	42(15)	180(18)	32(15)
C(36)	424(14)	361(15)	502(15)	97(11)	94(14)	10(14)
C(37)	297(13)	312(14)	362(15)	55(11)	-27(11)	63(10)
C(38)	359(14)	493(16)	357(15)	-11(13)	-25(11)	20(12)
C(39)	517(19)	541(18)	443(18)	-121(14)	-56(14)	100(15)
C(40)	394(16)	473(17)	522(19)	-108(14)	-126(14)	47(13)
C(41)	310(14)	421(16)	640(20)	-35(15)	-64(14)	36(12)
C(42)	409(16)	348(14)	421(16)	-4(12)	6(13)	71(12)

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Date 18 July 2008

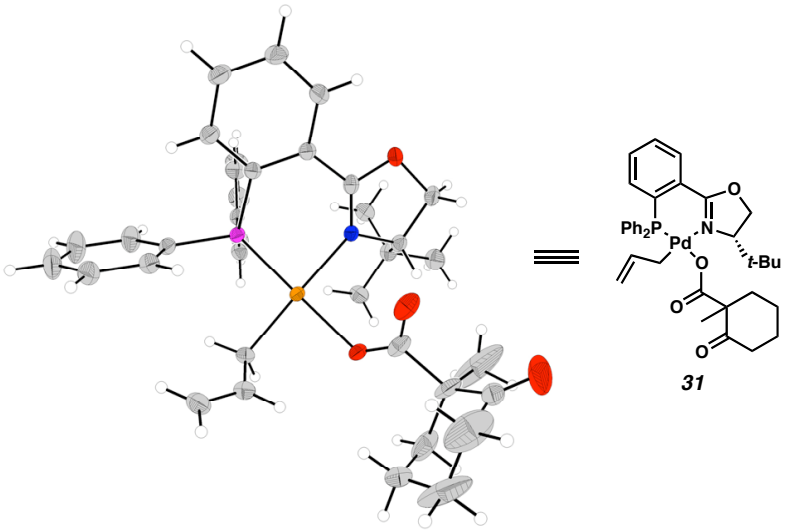
Crystal Structure Analysis of:

Palladium Allyl Carbonate 31

(NHS20, 695531)

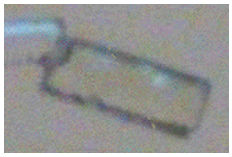
Contents

Figure A2.2. Representation of Palladium Allyl Carboxylate ***31***



Note: Crystallographic data have been deposited at the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK, and copies can be obtained on request, free of charge, by quoting the publication citation and the deposition number 695531.

Table A2.6. Crystal Data and Structure Refinement for NHS20 (CCDC 695531)



Empirical formula	$C_{36}H_{42}NO_4PPd$
Formula weight	690.08
Crystallization solvent	Ether/THF/hexanes
Crystal habit	Blade
Crystal size	0.17 x 0.10 x 0.06 mm^3
Crystal color	Pale yellow

Data Collection

Type of diffractometer	Bruker KAPPA APEX II
Wavelength	0.71073 Å MoKα
Data collection temperature	100(2) K
θ range for 9986 reflections used in lattice determination	2.50 to 33.63°
Unit cell dimensions	a = 10.7719(4) Å b = 12.4117(4) Å c = 24.7631(8) Å
Volume	3310.77(19) $Å^3$
Z	4
Crystal system	Orthorhombic
Space group	$P2_12_12_1$
Density (calculated)	1.384 Mg/m^3
F(000)	1432
θ range for data collection	2.06 to 34.15°
Completeness to θ = 34.15°	94.0 %
Index ranges	$-16 \le h \le 9$, $-18 \le k \le 17$, $-39 \le l \le 35$
Data collection scan type	ω scans; 11 settings
Reflections collected	62894
Independent reflections	12522 [R_{int}= 0.0472]
Absorption coefficient	0.648 mm^{-1}
Absorption correction	None
Max. and min. transmission	0.9622 and 0.8979

Table A2.6. (cont.)

Structure Solution and Refinement

Structure solution program	SHELXS-97 (Sheldrick, 2008)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	SHELXL-97 (Sheldrick, 2008)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	12522 / 0 / 402
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	1.778
Final R indices [I>2σ(I), 10824 reflections]	R1 = 0.0347, *w*R2 = 0.0482
R indices (all data)	R1 = 0.0454, *w*R2 = 0.0488
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.002
Average shift/error	0.000
Absolute structure parameter	-0.023(12)
Largest diff. peak and hole	1.696 and -1.113 e.Å^{-3}

Special Refinement Details

Crystals were mounted on a glass fiber using Paratone oil then placed on the diffractometer under a nitrogen stream at 100K.

The ketone oxygen (O4) is disordered over two positions, bonded to C28 (39.8%) or C32 (60.2%). The thermal ellipsoids and the difference Fourier in this area suggest there is some small rotational component to this disorder as well. The rotational component was not included in the model.

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt), etc., and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles, and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Table A2.7. Atomic Coordinates (x 10^4) and Equivalent Isotropic Displacement Parameters ($Å^2$x 10^3) for NHS20 (CCDC 695531). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}	Occ
Pd(1)	3632(1)	7917(1)	8446(1)	17(1)	1
P(1)	5054(1)	7679(1)	9067(1)	18(1)	1
O(1)	6459(1)	10035(1)	7822(1)	25(1)	1
O(2)	2273(1)	8140(1)	7828(1)	27(1)	1
O(3)	3711(1)	7986(1)	7181(1)	34(1)	1
O(4A)	2329(3)	8627(6)	6044(2)	206(5)	0.602(4)
O(4B)	-327(4)	8506(5)	7173(2)	73(2)	0.398(4)
N(1)	4786(1)	9182(1)	8149(1)	17(1)	1
C(1)	6611(2)	7734(2)	8768(1)	18(1)	1
C(2)	7547(2)	7083(2)	8966(1)	25(1)	1
C(3)	8770(2)	7172(2)	8776(1)	28(1)	1
C(4)	9051(2)	7913(2)	8384(1)	28(1)	1
C(5)	8125(2)	8571(2)	8179(1)	24(1)	1
C(6)	6898(2)	8498(2)	8358(1)	18(1)	1
C(7)	5979(2)	9225(2)	8117(1)	19(1)	1
C(8)	5407(2)	10555(2)	7558(1)	25(1)	1
C(9)	4276(2)	10150(2)	7871(1)	21(1)	1
C(10)	3687(2)	10973(2)	8264(1)	25(1)	1
C(11)	4604(2)	11384(2)	8687(1)	29(1)	1
C(12)	3221(2)	11928(2)	7921(1)	35(1)	1
C(13)	2575(2)	10459(2)	8548(1)	30(1)	1
C(14)	5105(2)	8772(2)	9554(1)	20(1)	1
C(15)	6212(2)	9262(2)	9713(1)	29(1)	1
C(16)	6191(2)	10115(2)	10076(1)	40(1)	1
C(17)	5077(2)	10470(2)	10288(1)	38(1)	1
C(18)	3976(2)	9991(2)	10138(1)	33(1)	1
C(19)	3981(2)	9144(2)	9769(1)	25(1)	1
C(20)	5098(2)	6447(2)	9464(1)	21(1)	1
C(21)	5154(2)	6451(2)	10023(1)	34(1)	1
C(22)	5217(3)	5480(2)	10303(1)	48(1)	1
C(23)	5212(2)	4513(2)	10025(1)	45(1)	1
C(24)	5147(2)	4506(2)	9471(1)	34(1)	1
C(25)	5085(2)	5473(2)	9192(1)	27(1)	1
C(26)	2636(2)	8148(2)	7339(1)	29(1)	1
C(27)	1653(2)	8437(2)	6908(1)	36(1)	1
C(28)	427(2)	7910(3)	7042(1)	54(1)	1
C(29)	418(3)	6739(3)	7002(1)	88(1)	1
C(30)	720(3)	6405(3)	6448(2)	85(1)	1
C(31)	1946(3)	6768(3)	6299(1)	81(1)	1
C(32)	2059(2)	7979(3)	6346(1)	65(1)	1
C(33)	1563(4)	9651(2)	6886(2)	147(2)	1
C(34)	2405(2)	6836(2)	8778(1)	25(1)	1
C(35)	2163(2)	5939(2)	8413(1)	30(1)	1
C(36)	2358(2)	4904(2)	8503(1)	33(1)	1

Table A2.8. Selected Bond Lengths [Å] and angles [°] for NHS20 (CCDC 695531)

Pd(1)-C(34)	2.0552(19)	C(34)-Pd(1)-N(1)	173.26(7)
Pd(1)-N(1)	2.1331(16)	C(34)-Pd(1)-O(2)	86.02(7)
Pd(1)-O(2)	2.1361(12)	N(1)-Pd(1)-O(2)	93.27(5)
Pd(1)-P(1)	2.1895(5)	C(34)-Pd(1)-P(1)	94.65(6)
		N(1)-Pd(1)-P(1)	86.19(4)
		O(2)-Pd(1)-P(1)	178.84(4)

Table A2.9. Bond Lengths [Å] and Angles [°] for NHS20 (CCDC 695531)

Pd(1)-C(34)	2.0552(19)	C(34)-Pd(1)-N(1)	173.26(7)
Pd(1)-N(1)	2.1331(16)	C(34)-Pd(1)-O(2)	86.02(7)
Pd(1)-O(2)	2.1361(12)	N(1)-Pd(1)-O(2)	93.27(5)
Pd(1)-P(1)	2.1895(5)	C(34)-Pd(1)-P(1)	94.65(6)
P(1)-C(14)	1.817(2)	N(1)-Pd(1)-P(1)	86.19(4)
P(1)-C(20)	1.819(2)	O(2)-Pd(1)-P(1)	178.84(4)
P(1)-C(1)	1.8348(17)	C(14)-P(1)-C(20)	105.51(9)
O(1)-C(7)	1.346(2)	C(14)-P(1)-C(1)	102.26(9)
O(1)-C(8)	1.458(2)	C(20)-P(1)-C(1)	103.02(9)
O(2)-C(26)	1.273(3)	C(14)-P(1)-Pd(1)	112.79(7)
O(3)-C(26)	1.238(3)	C(20)-P(1)-Pd(1)	120.74(7)
O(4A)-C(32)	1.136(5)	C(1)-P(1)-Pd(1)	110.57(5)
O(4B)-C(28)	1.144(5)	C(7)-O(1)-C(8)	105.99(14)
N(1)-C(7)	1.288(2)	C(26)-O(2)-Pd(1)	118.15(13)
N(1)-C(9)	1.490(2)	C(7)-N(1)-C(9)	107.74(16)
C(1)-C(2)	1.382(3)	C(7)-N(1)-Pd(1)	129.28(13)
C(1)-C(6)	1.424(2)	C(9)-N(1)-Pd(1)	122.58(12)
C(2)-C(3)	1.403(3)	C(2)-C(1)-C(6)	118.95(16)
C(3)-C(4)	1.372(3)	C(2)-C(1)-P(1)	120.17(13)
C(4)-C(5)	1.384(3)	C(6)-C(1)-P(1)	120.72(13)
C(5)-C(6)	1.397(2)	C(1)-C(2)-C(3)	121.31(19)
C(6)-C(7)	1.467(2)	C(4)-C(3)-C(2)	119.87(19)
C(8)-C(9)	1.529(3)	C(3)-C(4)-C(5)	119.68(17)
C(9)-C(10)	1.546(3)	C(4)-C(5)-C(6)	121.86(18)
C(10)-C(13)	1.528(3)	C(5)-C(6)-C(1)	118.32(17)
C(10)-C(11)	1.528(3)	C(5)-C(6)-C(7)	118.06(17)
C(10)-C(12)	1.542(3)	C(1)-C(6)-C(7)	123.61(16)
C(14)-C(15)	1.396(3)	N(1)-C(7)-O(1)	116.63(17)
C(14)-C(19)	1.400(3)	N(1)-C(7)-C(6)	128.47(17)
C(15)-C(16)	1.389(3)	O(1)-C(7)-C(6)	114.89(16)
C(16)-C(17)	1.382(3)	O(1)-C(8)-C(9)	104.24(14)
C(17)-C(18)	1.377(3)	N(1)-C(9)-C(8)	101.89(14)
C(18)-C(19)	1.395(3)	N(1)-C(9)-C(10)	113.19(15)
C(20)-C(25)	1.384(3)	C(8)-C(9)-C(10)	115.42(16)
C(20)-C(21)	1.386(3)	C(13)-C(10)-C(11)	109.33(16)
C(21)-C(22)	1.391(3)	C(13)-C(10)-C(12)	108.63(17)
C(22)-C(23)	1.384(3)	C(11)-C(10)-C(12)	109.32(16)
C(23)-C(24)	1.373(3)	C(13)-C(10)-C(9)	109.59(16)
C(24)-C(25)	1.387(3)	C(11)-C(10)-C(9)	112.69(18)
C(26)-C(27)	1.547(3)	C(12)-C(10)-C(9)	107.19(16)
C(27)-C(28)	1.511(4)	C(15)-C(14)-C(19)	119.26(18)
C(27)-C(33)	1.511(3)	C(15)-C(14)-P(1)	122.58(15)
C(27)-C(32)	1.564(4)	C(19)-C(14)-P(1)	118.15(15)
C(28)-C(29)	1.457(5)	C(16)-C(15)-C(14)	120.0(2)
C(29)-C(30)	1.469(5)	C(17)-C(16)-C(15)	120.2(2)
C(30)-C(31)	1.443(4)	C(18)-C(17)-C(16)	120.6(2)
C(31)-C(32)	1.513(5)	C(17)-C(18)-C(19)	119.9(2)
C(34)-C(35)	1.458(3)	C(18)-C(19)-C(14)	120.1(2)
C(35)-C(36)	1.320(3)	C(25)-C(20)-C(21)	119.36(19)
		C(25)-C(20)-P(1)	118.08(15)

C(21)-C(20)-P(1)	122.56(17)
C(20)-C(21)-C(22)	119.7(2)
C(23)-C(22)-C(21)	120.3(2)
C(24)-C(23)-C(22)	120.2(2)
C(23)-C(24)-C(25)	119.6(2)
C(20)-C(25)-C(24)	120.8(2)
O(3)-C(26)-O(2)	125.94(18)
O(3)-C(26)-C(27)	117.4(2)
O(2)-C(26)-C(27)	116.7(2)
C(28)-C(27)-C(33)	112.5(3)
C(28)-C(27)-C(26)	110.2(2)
C(33)-C(27)-C(26)	107.42(19)
C(28)-C(27)-C(32)	106.5(2)
C(33)-C(27)-C(32)	110.4(3)
C(26)-C(27)-C(32)	109.76(18)
O(4B)-C(28)-C(29)	131.2(4)
O(4B)-C(28)-C(27)	113.8(4)
C(29)-C(28)-C(27)	115.0(3)
C(28)-C(29)-C(30)	110.2(3)
C(31)-C(30)-C(29)	110.7(3)
C(30)-C(31)-C(32)	111.3(3)
O(4A)-C(32)-C(31)	132.3(6)
O(4A)-C(32)-C(27)	113.5(5)
C(31)-C(32)-C(27)	114.1(2)
C(35)-C(34)-Pd(1)	111.39(14)
C(36)-C(35)-C(34)	127.6(2)

Table A2.10. Anisotropic Displacement Parameters ($Å^2 x\ 10^4$) for NHS20 (CCDC 695531). The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$.

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Pd(1)	157(1)	174(1)	177(1)	5(1)	-26(1)	-1(1)
P(1)	156(2)	228(3)	144(2)	12(2)	3(2)	11(2)
O(1)	236(7)	281(7)	229(6)	92(6)	16(7)	15(7)
O(2)	232(7)	256(9)	312(8)	69(6)	-117(6)	-41(6)
O(3)	400(8)	326(8)	296(7)	-101(7)	-108(7)	112(9)
O(4A)	410(20)	4600(120)	1160(40)	2010(60)	-380(20)	-650(40)
O(4B)	340(30)	1510(60)	340(30)	-90(30)	20(20)	350(30)
N(1)	176(9)	157(9)	182(8)	17(6)	-10(6)	12(7)
C(1)	174(10)	214(11)	147(7)	-14(7)	-1(6)	-11(8)
C(2)	212(10)	292(11)	245(9)	21(10)	-13(7)	32(11)
C(3)	194(10)	357(11)	277(9)	11(9)	-22(8)	58(11)
C(4)	184(9)	371(11)	278(10)	3(12)	35(8)	59(11)
C(5)	214(10)	253(12)	244(10)	28(9)	35(8)	-1(9)
C(6)	184(9)	230(10)	140(9)	-18(7)	1(7)	-2(8)
C(7)	248(11)	217(11)	113(8)	-20(7)	5(7)	6(8)
C(8)	286(12)	240(11)	213(10)	63(8)	-19(9)	28(9)
C(9)	224(10)	179(10)	221(10)	29(8)	-34(8)	6(8)
C(10)	266(10)	181(10)	294(9)	13(7)	-5(10)	34(10)
C(11)	327(12)	225(12)	322(12)	-51(9)	35(9)	-21(10)
C(12)	406(13)	206(13)	436(13)	46(10)	-7(10)	45(10)
C(13)	241(11)	243(11)	419(15)	43(10)	44(10)	47(9)
C(14)	211(11)	245(11)	135(8)	16(7)	1(8)	3(9)
C(15)	276(12)	369(12)	228(10)	-65(9)	25(9)	-24(11)
C(16)	437(15)	472(14)	288(11)	-123(10)	-26(11)	-158(13)
C(17)	538(16)	336(14)	281(12)	-126(10)	41(12)	-22(13)
C(18)	429(15)	325(12)	240(11)	-15(9)	117(10)	40(10)
C(19)	264(12)	272(11)	225(10)	21(8)	26(8)	-13(9)
C(20)	160(10)	262(11)	200(9)	52(8)	5(8)	22(9)
C(21)	452(14)	365(13)	217(10)	46(9)	41(10)	62(12)
C(22)	675(19)	526(17)	226(12)	194(12)	3(13)	76(16)
C(23)	571(17)	352(15)	438(15)	207(12)	18(13)	77(13)
C(24)	296(13)	271(13)	460(14)	84(10)	10(11)	39(11)
C(25)	244(11)	291(12)	262(11)	46(9)	-2(9)	-7(10)
C(26)	382(13)	135(11)	357(12)	-37(8)	-201(10)	4(9)
C(27)	346(14)	283(12)	458(13)	-14(10)	-271(11)	10(10)
C(28)	330(14)	1110(30)	170(10)	-62(16)	-2(9)	100(20)
C(29)	940(30)	1040(30)	670(20)	400(20)	-520(20)	-740(20)
C(30)	780(20)	520(20)	1260(30)	-380(20)	-720(20)	109(18)
C(31)	690(20)	1110(40)	630(20)	-470(20)	-158(17)	220(20)
C(32)	212(13)	1260(30)	493(15)	490(20)	-165(11)	-218(18)
C(33)	2060(50)	277(18)	2070(50)	0(20)	-1750(40)	170(30)
C(34)	160(10)	296(13)	295(11)	62(9)	1(8)	-25(8)
C(35)	273(11)	332(12)	307(11)	40(11)	-55(10)	-101(9)
C(36)	370(13)	312(12)	320(12)	19(11)	-7(11)	-125(10)

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Date 09 September 2008

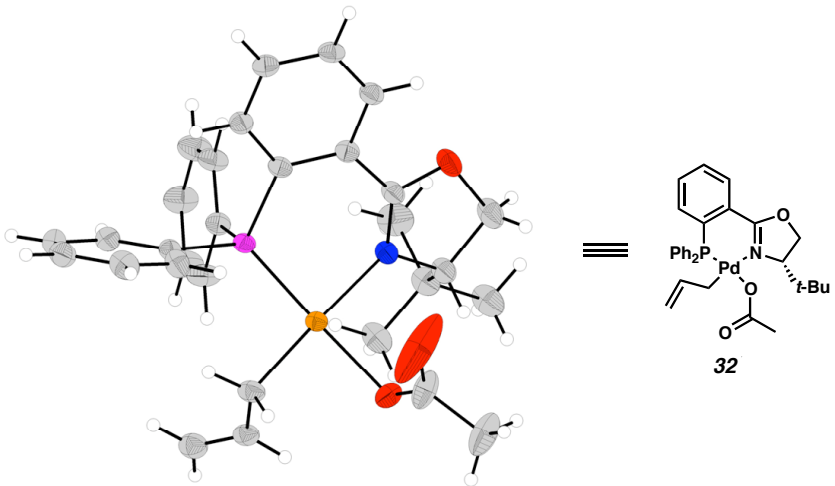
Crystal Structure Analysis of:

Palladium Allyl Acetate 32

(NHS22, 701671)

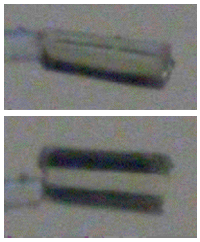
Contents

Figure A2.3. Representation of Palladium Allyl Acetate ***32***



Note: Crystallographic data have been deposited at the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK, and copies can be obtained on request, free of charge, by quoting the publication citation and the deposition number 701671.

Table A2.11. Crystal Data and Structure Refinement for NHS22 (CCDC 701671).



Empirical formula	C29 H32 N O5 P Pd, C6 H6
Formula weight	690.03
Crystallization solvent	Dichloromethane/ether
Crystal habit	Rod
Crystal size	0.33 x 0.14 x 0.10 mm^3
Crystal color	Pale yellow

Data Collection

Type of diffractometer	Bruker KAPPA APEX II
Wavelength	0.71073 Å MoKα
Data collection temperature	100(2) K
θ range for 9097 reflections used in lattice determination	2.50 to 34.73°
Unit cell dimensions	a = 28.2427(10) Å b = 28.2427(10) Å c = 10.4684(4) Å
Volume	7231.4(5) $Å^3$
Z	9
Crystal system	Rhombohedral
Space group	R3
Density (calculated)	1.426 Mg/m^3
F(000)	3204
θ range for data collection	2.12 to 34.96°
Completeness to θ = 34.96°	95.1 %
Index ranges	$-44 \le h \le 44$, $-44 \le k \le 44$, $-16 \le l \le 16$
Data collection scan type	ω scans; 20 settings
Reflections collected	117262
Independent reflections	13097 [R_{int}= 0.0305]
Absorption coefficient	0.669 mm^{-1}
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7469 and 0.6754

Table A2.11. (cont.)

Structure Solution and Refinement

Structure solution program	SHELXS-97 (Sheldrick, 2008)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	SHELXL-97 (Sheldrick, 2008)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	13097 / 1 / 353
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	3.223
Final R indices [I>2σ(I), 12405 reflections]	R1 = 0.0283, *w*R2 = 0.0620
R indices (all data)	R1 = 0.0321, *w*R2 = 0.0623
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.003
Average shift/error	0.000
Absolute structure parameter	-0.014(11)
Largest diff. peak and hole	0.926 and -0.561 e.Å^{-3}

Special Refinement Details

Crystals were mounted on a glass fiber using Paratone oil then placed on the diffractometer under a nitrogen stream at 100K.

A disordered benzene sits near the threefold rotation axis. Both components were constrained to ideal geometry with isotropic temperature factors; the minor component (44%) was assigned one overall temperature factor.

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt), etc., and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles, and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Table A2.12. Atomic Coordinates (x 10^4) and Equivalent Isotropic Displacement Parameters ($Å^2$x 10^3) for NHS22 (CCDC 701671). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}	Occ
Pd(1)	5618(1)	1009(1)	7615(1)	19(1)	1
P(1)	5171(1)	108(1)	7493(1)	18(1)	1
O(1)	5022(1)	689(1)	11364(1)	31(1)	1
O(2)	6063(1)	1851(1)	7815(2)	33(1)	1
O(3)	6590(1)	1744(1)	9230(2)	48(1)	1
O(4)	6005(1)	1107(1)	5946(1)	28(1)	1
O(5)	5242(1)	808(1)	4803(2)	41(1)	1
N(1)	5274(1)	943(1)	9349(1)	20(1)	1
C(1)	5242(1)	-151(1)	9029(2)	19(1)	1
C(2)	5276(1)	-626(1)	9092(2)	26(1)	1
C(3)	5274(1)	-864(1)	10250(2)	26(1)	1
C(4)	5240(1)	-634(1)	11370(2)	27(1)	1
C(5)	5214(1)	-154(1)	11345(2)	24(1)	1
C(6)	5212(1)	87(1)	10177(2)	20(1)	1
C(7)	5175(1)	589(1)	10230(2)	21(1)	1
C(8)	5111(1)	1243(1)	11280(2)	34(1)	1
C(9)	5146(1)	1360(1)	9848(2)	23(1)	1
C(10)	4629(1)	1321(1)	9247(2)	25(1)	1
C(11)	4129(1)	750(1)	9420(2)	31(1)	1
C(12)	4522(1)	1746(1)	9905(2)	35(1)	1
C(13)	4718(1)	1459(1)	7831(2)	29(1)	1
C(14)	4437(1)	-228(1)	7275(2)	19(1)	1
C(15)	4080(1)	-708(1)	7923(2)	24(1)	1
C(16)	3522(1)	-953(1)	7730(2)	27(1)	1
C(17)	3310(1)	-721(1)	6916(2)	25(1)	1
C(18)	3660(1)	-244(1)	6269(2)	24(1)	1
C(19)	4223(1)	2(1)	6434(2)	21(1)	1
C(20)	5427(1)	-191(1)	6348(2)	21(1)	1
C(21)	5080(1)	-637(1)	5595(2)	26(1)	1
C(22)	5292(1)	-870(1)	4778(2)	30(1)	1
C(23)	5856(1)	-654(1)	4700(2)	34(1)	1
C(24)	6205(1)	-211(1)	5451(2)	33(1)	1
C(25)	5994(1)	26(1)	6272(2)	27(1)	1
C(26)	6461(1)	2026(1)	8589(3)	39(1)	1
C(27)	6782(1)	2647(1)	8709(5)	92(2)	1
C(28)	5727(1)	976(1)	4889(2)	27(1)	1
C(29)	6075(1)	1044(1)	3734(2)	43(1)	1
C(41)	7391(2)	2775(1)	5211(4)	81(2)	0.560(7)
C(42)	7219(2)	2450(1)	4122(5)	132(4)	0.560(7)
C(43)	7255(2)	2686(2)	2934(4)	135(4)	0.560(7)
C(44)	7464(2)	3246(2)	2835(3)	87(2)	0.560(7)
C(45)	7637(2)	3571(1)	3924(4)	96(2)	0.560(7)
C(46)	7600(2)	3335(1)	5112(3)	42(1)	0.560(7)
C(51)	7230(2)	2547(2)	3398(5)	375(10)	0.440(7)
C(52)	7098(2)	2415(1)	4677(5)	375(10)	0.440(7)

C(53)	7168(2)	2818(2)	5543(4)	375(10)	0.440(7)
C(54)	7371(2)	3353(2)	5129(3)	375(10)	0.440(7)
C(55)	7503(2)	3485(1)	3850(4)	375(10)	0.440(7)
C(56)	7433(2)	3081(2)	2984(3)	375(10)	0.440(7)

Table A2.13. Selected Bond Lengths [Å] and Angles [°] for NHS22 (CCDC 701671).

Pd(1)-O(4)	2.0051(13)	O(4)-Pd(1)-N(1)	176.38(7)
Pd(1)-N(1)	2.0227(15)	O(4)-Pd(1)-O(2)	87.35(6)
Pd(1)-O(2)	2.0704(14)	N(1)-Pd(1)-O(2)	90.16(6)
Pd(1)-P(1)	2.2072(5)	O(4)-Pd(1)-P(1)	93.81(5)
		N(1)-Pd(1)-P(1)	88.53(4)
		O(2)-Pd(1)-P(1)	176.68(5)

Table A2.14. Bond Lengths [Å] and Angles [°] for NHS22 (CCDC 701671).

Pd(1)-O(4)	2.0051(13)	C(53)-C(54)	1.3899
Pd(1)-N(1)	2.0227(15)	C(54)-C(55)	1.3899
Pd(1)-O(2)	2.0704(14)	C(55)-C(56)	1.3899
Pd(1)-P(1)	2.2072(5)		
P(1)-C(20)	1.8116(18)	O(4)-Pd(1)-N(1)	176.38(7)
P(1)-C(14)	1.8130(17)	O(4)-Pd(1)-O(2)	87.35(6)
P(1)-C(1)	1.8187(18)	N(1)-Pd(1)-O(2)	90.16(6)
O(1)-C(7)	1.340(2)	O(4)-Pd(1)-P(1)	93.81(5)
O(1)-C(8)	1.459(2)	N(1)-Pd(1)-P(1)	88.53(4)
O(2)-C(26)	1.267(3)	O(2)-Pd(1)-P(1)	176.68(5)
O(3)-C(26)	1.230(3)	C(20)-P(1)-C(14)	106.98(8)
O(4)-C(28)	1.298(2)	C(20)-P(1)-C(1)	104.36(8)
O(5)-C(28)	1.210(2)	C(14)-P(1)-C(1)	103.18(8)
N(1)-C(7)	1.285(2)	C(20)-P(1)-Pd(1)	116.35(6)
N(1)-C(9)	1.488(2)	C(14)-P(1)-Pd(1)	117.09(6)
C(1)-C(2)	1.393(2)	C(1)-P(1)-Pd(1)	107.33(6)
C(1)-C(6)	1.399(3)	C(7)-O(1)-C(8)	105.75(15)
C(2)-C(3)	1.386(3)	C(26)-O(2)-Pd(1)	115.15(13)
C(3)-C(4)	1.366(3)	C(28)-O(4)-Pd(1)	119.55(12)
C(4)-C(5)	1.396(3)	C(7)-N(1)-C(9)	108.64(14)
C(5)-C(6)	1.399(3)	C(7)-N(1)-Pd(1)	128.56(12)
C(6)-C(7)	1.474(2)	C(9)-N(1)-Pd(1)	122.52(11)
C(8)-C(9)	1.528(3)	C(2)-C(1)-C(6)	118.10(17)
C(9)-C(10)	1.543(3)	C(2)-C(1)-P(1)	120.19(14)
C(10)-C(13)	1.522(3)	C(6)-C(1)-P(1)	121.44(13)
C(10)-C(11)	1.534(3)	C(3)-C(2)-C(1)	121.58(18)
C(10)-C(12)	1.538(3)	C(4)-C(3)-C(2)	120.27(17)
C(14)-C(15)	1.396(2)	C(3)-C(4)-C(5)	119.74(18)
C(14)-C(19)	1.398(2)	C(4)-C(5)-C(6)	120.18(18)
C(15)-C(16)	1.381(3)	C(1)-C(6)-C(5)	120.13(16)
C(16)-C(17)	1.383(3)	C(1)-C(6)-C(7)	122.95(16)
C(17)-C(18)	1.386(3)	C(5)-C(6)-C(7)	116.91(16)
C(18)-C(19)	1.391(2)	N(1)-C(7)-O(1)	116.13(16)
C(20)-C(21)	1.391(3)	N(1)-C(7)-C(6)	129.02(16)
C(20)-C(25)	1.401(3)	O(1)-C(7)-C(6)	114.85(16)
C(21)-C(22)	1.386(3)	O(1)-C(8)-C(9)	104.44(15)
C(22)-C(23)	1.395(3)	N(1)-C(9)-C(8)	100.86(14)
C(23)-C(24)	1.386(3)	N(1)-C(9)-C(10)	113.31(15)
C(24)-C(25)	1.391(3)	C(8)-C(9)-C(10)	115.30(17)
C(26)-C(27)	1.523(3)	C(13)-C(10)-C(11)	109.38(17)
C(28)-C(29)	1.508(3)	C(13)-C(10)-C(12)	107.97(17)
C(41)-C(42)	1.3900	C(11)-C(10)-C(12)	109.33(16)
C(41)-C(46)	1.3900	C(13)-C(10)-C(9)	110.43(15)
C(42)-C(43)	1.3900	C(11)-C(10)-C(9)	111.60(16)
C(43)-C(44)	1.3900	C(12)-C(10)-C(9)	108.05(16)
C(44)-C(45)	1.3900	C(15)-C(14)-C(19)	119.16(16)
C(45)-C(46)	1.3900	C(15)-C(14)-P(1)	121.94(13)
C(51)-C(56)	1.3899	C(19)-C(14)-P(1)	118.89(13)
C(51)-C(52)	1.3899	C(16)-C(15)-C(14)	120.31(17)
C(52)-C(53)	1.3899	C(15)-C(16)-C(17)	120.60(18)

C(16)-C(17)-C(18)	119.57(17)
C(17)-C(18)-C(19)	120.50(17)
C(18)-C(19)-C(14)	119.84(17)
C(21)-C(20)-C(25)	119.83(17)
C(21)-C(20)-P(1)	122.08(14)
C(25)-C(20)-P(1)	118.03(14)
C(22)-C(21)-C(20)	120.22(18)
C(21)-C(22)-C(23)	119.9(2)
C(24)-C(23)-C(22)	120.15(18)
C(23)-C(24)-C(25)	120.20(19)
C(24)-C(25)-C(20)	119.67(19)
O(3)-C(26)-O(2)	126.0(2)
O(3)-C(26)-C(27)	120.2(2)
O(2)-C(26)-C(27)	113.8(2)
O(5)-C(28)-O(4)	125.49(19)
O(5)-C(28)-C(29)	121.8(2)
O(4)-C(28)-C(29)	112.67(17)
C(42)-C(41)-C(46)	120.0
C(43)-C(42)-C(41)	120.0
C(42)-C(43)-C(44)	120.0
C(45)-C(44)-C(43)	120.0
C(44)-C(45)-C(46)	120.0
C(45)-C(46)-C(41)	120.0
C(56)-C(51)-C(52)	120.0
C(53)-C(52)-C(51)	120.0
C(54)-C(53)-C(52)	120.0
C(53)-C(54)-C(55)	120.0
C(56)-C(55)-C(54)	120.0
C(55)-C(56)-C(51)	120.

Table A2.15. Anisotropic Displacement Parameters ($Å^2 x\ 10^4$) for NHS22 (CCDC 701671). The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$.

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Pd(1)	154(1)	215(1)	198(1)	33(1)	10(1)	91(1)
P(1)	168(2)	235(2)	155(2)	-12(2)	0(2)	111(2)
O(1)	503(9)	363(7)	169(7)	-31(6)	1(6)	297(7)
O(2)	215(6)	241(6)	497(9)	81(6)	-1(6)	95(5)
O(3)	408(9)	455(9)	642(12)	-188(9)	-222(8)	274(8)
O(4)	211(6)	421(8)	204(7)	87(6)	44(5)	163(6)
O(5)	254(7)	723(12)	275(9)	70(8)	19(6)	263(8)
N(1)	206(7)	203(7)	201(8)	-22(6)	-14(5)	111(6)
C(1)	186(7)	247(8)	167(9)	5(6)	11(6)	127(6)
C(2)	292(9)	274(9)	253(10)	-41(7)	-12(8)	182(8)
C(3)	288(9)	261(9)	296(11)	8(8)	-14(8)	192(8)
C(4)	287(9)	298(9)	251(10)	70(8)	1(8)	165(8)
C(5)	298(9)	293(9)	177(9)	4(7)	-1(7)	173(8)
C(6)	206(7)	227(8)	177(9)	0(6)	-10(6)	125(6)
C(7)	242(8)	261(8)	147(9)	-24(7)	-21(7)	150(7)
C(8)	549(13)	324(10)	262(11)	-94(8)	-68(10)	295(10)
C(9)	264(8)	200(8)	257(10)	-40(7)	-28(7)	131(7)
C(10)	241(8)	251(8)	288(11)	-29(7)	-5(8)	146(7)
C(11)	234(9)	298(9)	376(12)	-30(8)	56(8)	123(7)
C(12)	380(11)	326(10)	420(13)	-38(9)	22(9)	238(9)
C(13)	300(9)	350(10)	305(11)	53(8)	5(8)	217(8)
C(14)	171(7)	236(8)	176(9)	-42(6)	-9(6)	110(6)
C(15)	207(8)	272(8)	217(10)	28(7)	3(7)	114(7)
C(16)	216(8)	305(9)	247(10)	25(8)	24(7)	100(7)
C(17)	178(7)	315(9)	240(10)	-50(7)	-4(7)	118(7)
C(18)	216(8)	303(9)	219(10)	0(7)	-12(7)	137(7)
C(19)	202(8)	235(8)	192(9)	1(7)	-7(7)	100(7)
C(20)	247(8)	282(8)	148(9)	10(7)	14(6)	172(7)
C(21)	305(9)	327(9)	180(10)	-13(7)	-21(7)	190(8)
C(22)	466(12)	355(10)	154(9)	-16(8)	-4(8)	272(10)
C(23)	585(14)	459(12)	187(10)	107(9)	125(9)	412(11)
C(24)	338(10)	488(12)	306(11)	118(10)	124(9)	310(10)
C(25)	256(8)	346(10)	245(10)	32(8)	28(7)	186(8)
C(26)	203(9)	279(10)	714(17)	-139(10)	-51(10)	145(8)
C(27)	405(15)	366(15)	1990(50)	-300(20)	-360(20)	195(13)
C(28)	242(8)	367(10)	259(11)	99(8)	61(7)	190(8)
C(29)	355(11)	785(18)	247(12)	114(11)	89(9)	361(12)