Electronic Supplementary Material (ESI) for Dalton Transactions

Electronic Supplemental Information

A Bisphosphonite Calix[5]arene Ligand that Stabilizes η^6 Arene Coordination to Palladium

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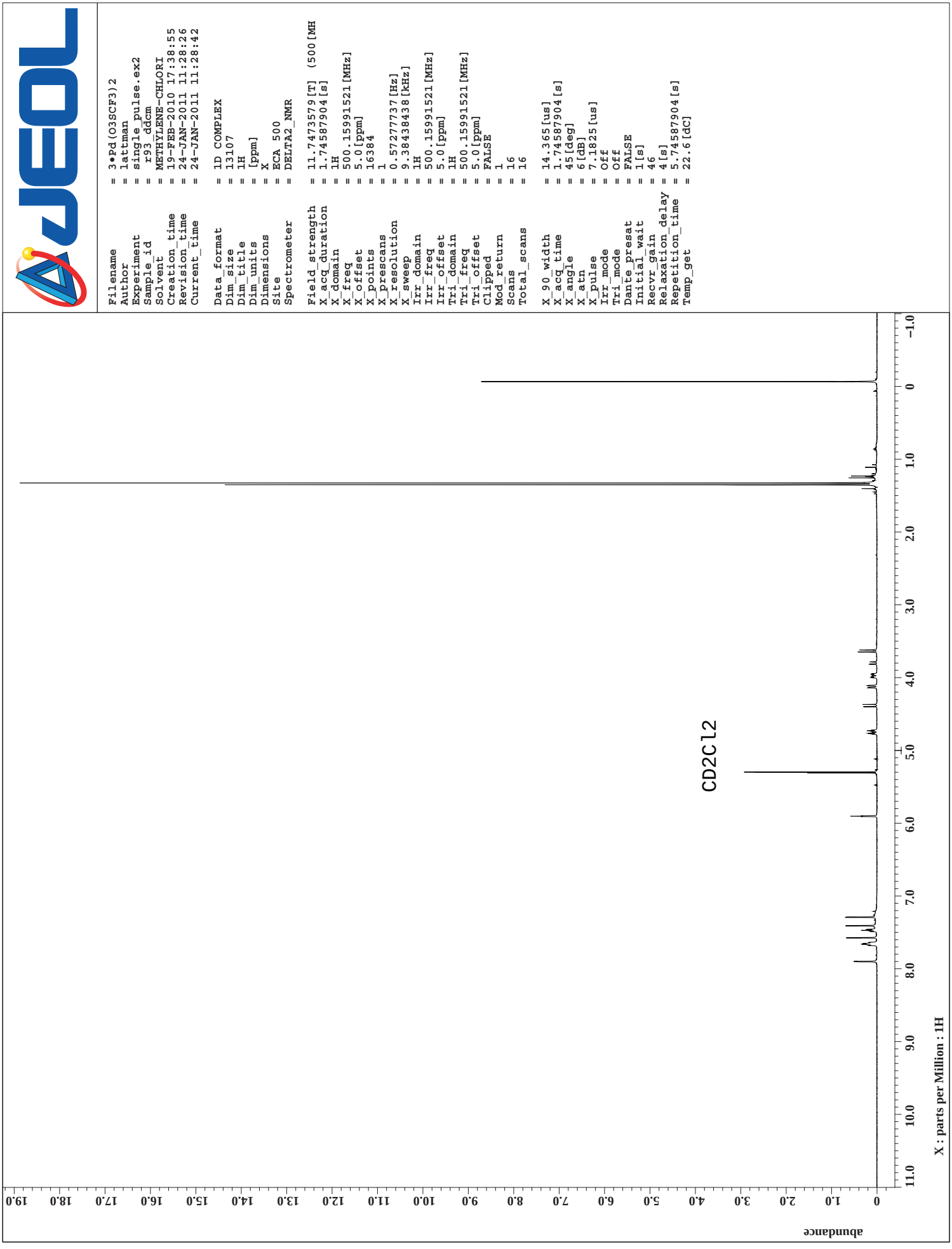
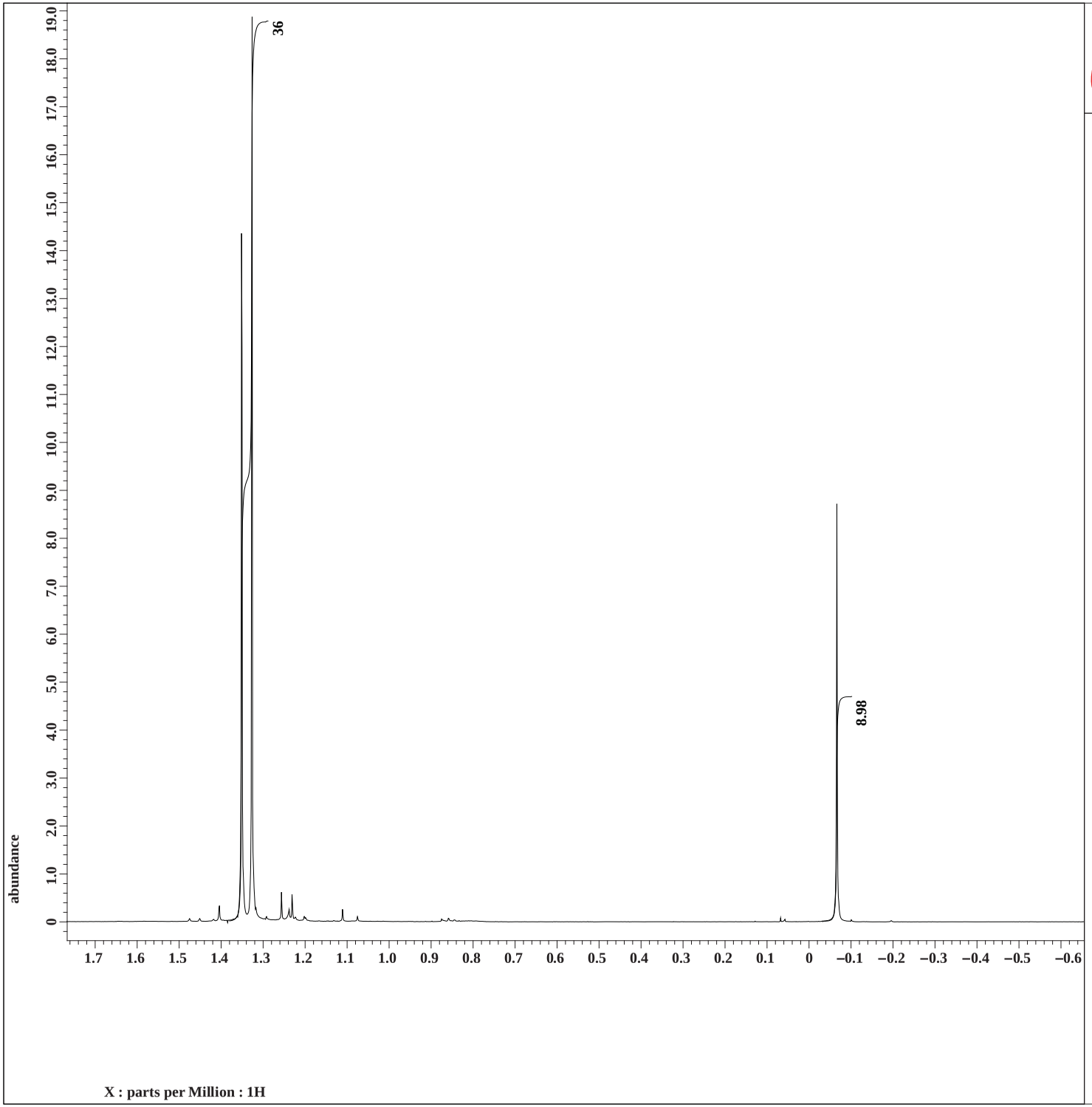
NMR spectra for 3•Pd(O3SCF3)2

Crystallographic details for compounds, including a complete table of bond distances and angles for 3•Pd(O3SCF3)2





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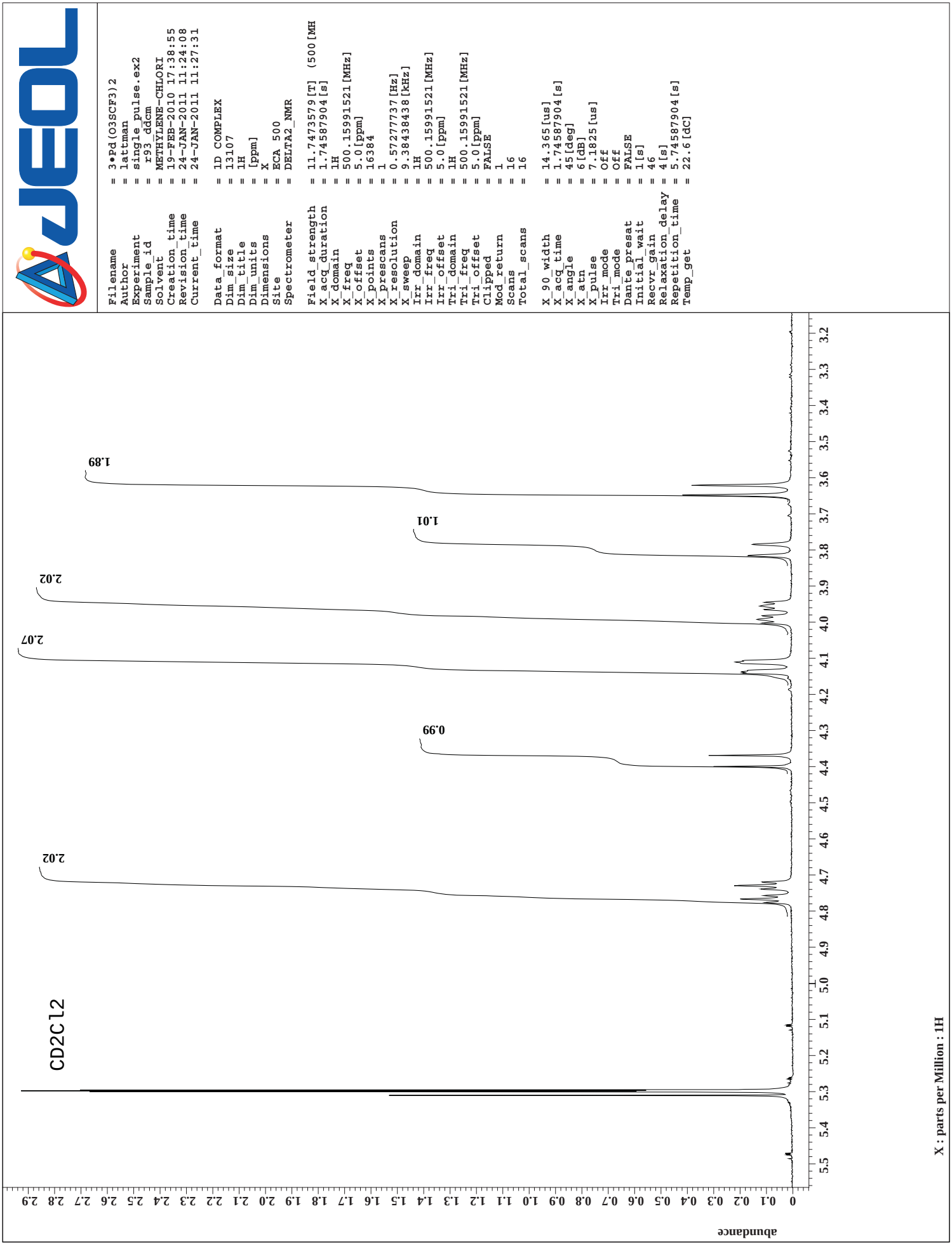
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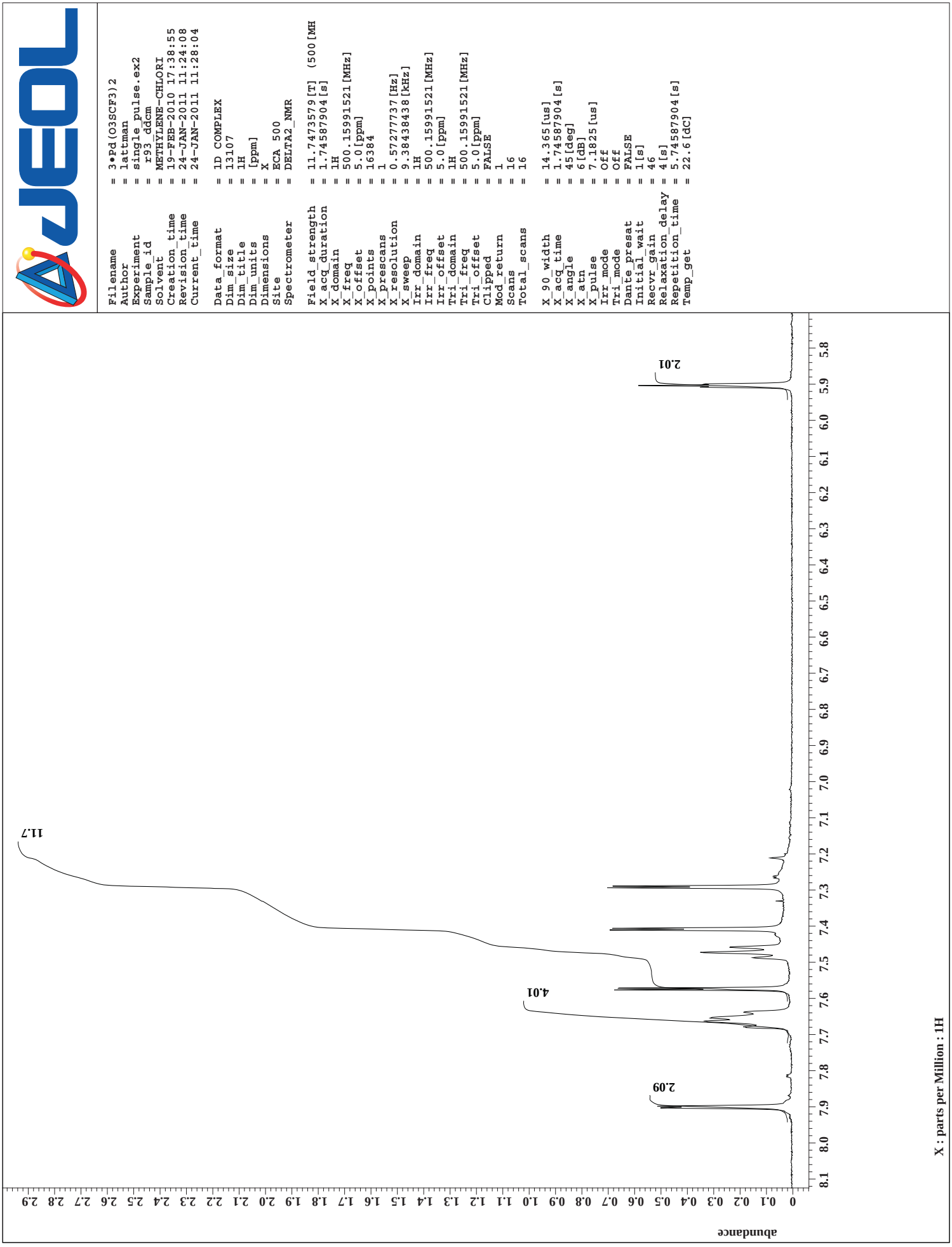
2.02
0.99
1.01
1.89
abundance
X : parts per Million : 1H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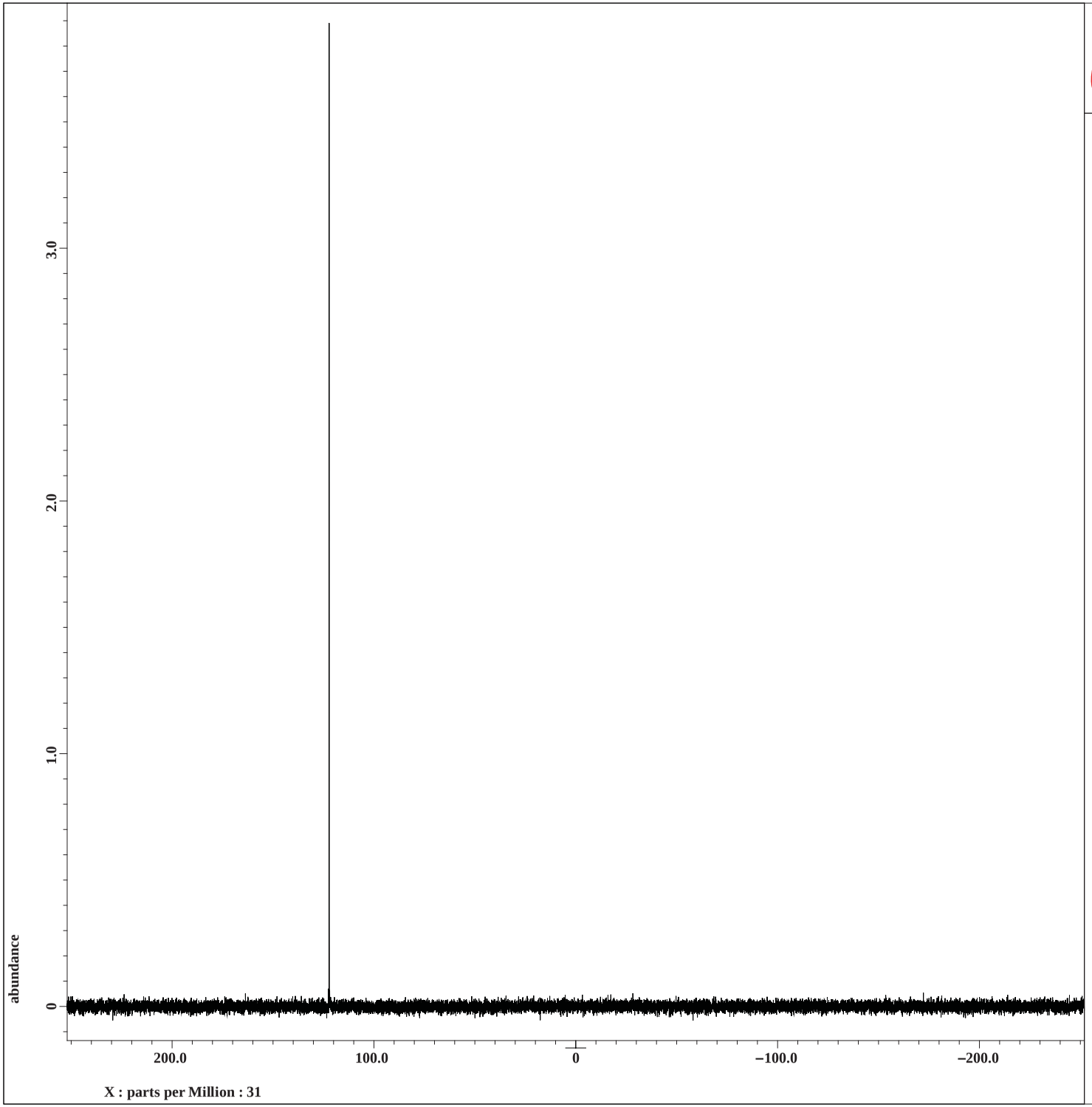


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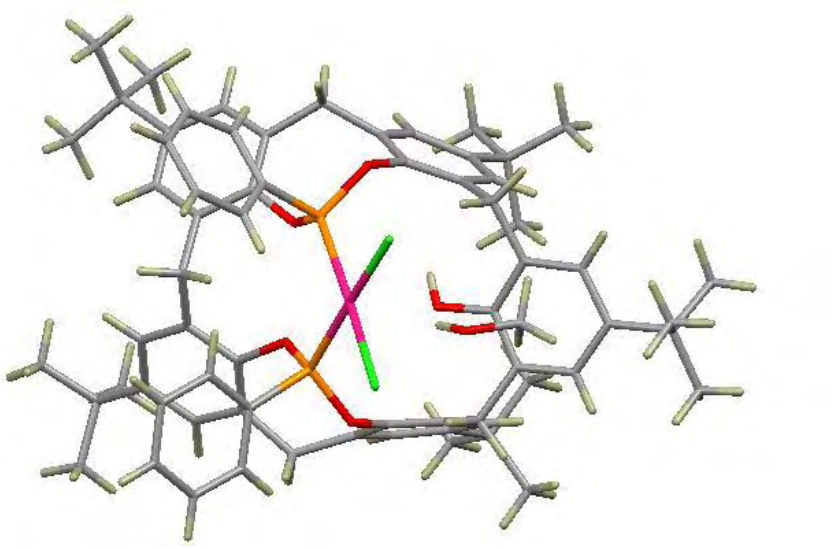
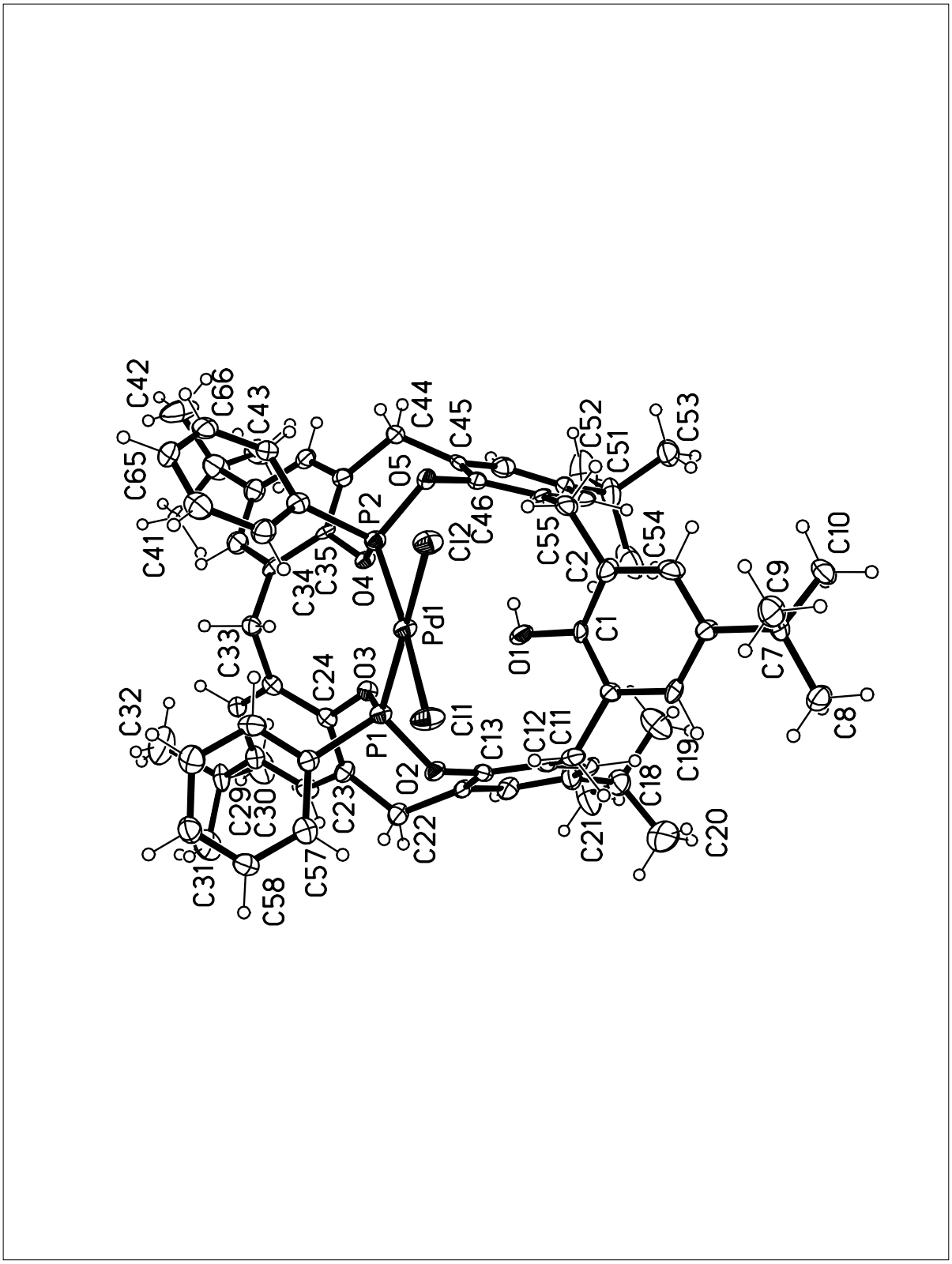
3•PdCl2





Comment

The metal complex contained two *t*-butyl groups that were disordered. The occupancies of C19, C20, and C21 refined to 0.796(12) and 0.204(12) for the unprimed and primed atoms; the occupancies of atoms C41, C42, and C43 refined to 0.565(10) and 0.435(10) for the unprimed and primed atoms. Restraints on the positional and displacement parameters of the disordered atoms were required. Two methanol solvent sites were severely disordered and were best modeled using the Squeeze program.(4) The displacement ellipsoids were drawn at the 50% probability level.

Experimental

A yellow prism-shaped crystal of dimensions 0.37 x 0.19 x 0.17 mm was selected for structural analysis. Intensity data for this compound were collected using a diffractometer with a Bruker APEX ccd area detector (1) and graphite-monochromated Mo Kα radiation (λ = 0.71073 Å). The sample was cooled to 100(2) K. Cell parameters were determined from a non-linear least squares fit of 5606 peaks in the range 2.35 < θ < 25.13°. A total of 36340 data were measured in the range 2.35 < θ < 25.50° using ϕ and ω oscillation frames. The data were corrected for absorption by the empirical method (2) giving minimum and maximum transmission factors of 0.8468 and 0.9251. The data were merged to form a set of 12001 independent data with R(int) = 0.1090 and a coverage of 98.6 %.

The triclinic space group $P\bar{1}$ was determined by statistical tests and verified by subsequent refinement. The structure was solved by direct methods and refined by full-matrix least-squares methods on F^2 (3). Hydrogen atom positions of hydrogens bonded to carbons were initially determined by geometry and refined by a riding model. Hydrogens bonded to oxygens were located on a difference map. The position of H1 was refined independently, and the position of H1A was refined with a riding model. Non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atom displacement parameters were set to 1.2 (1.5 for methyl) times the isotropic equivalent displacement parameters of the bonded atoms. A total of 771 parameters were refined against 266 restraints and 12001 data to give wR(F^2) = 0.1509 and S = 0.989 for weights of w = 1/[σ^2 (F^2) + (0.0560 P)^2], where P = [F_o^2 + 2F_c^2] / 3. The final R(F) was 0.0674 for the 7189 observed, [F > 4σ(F)], data. The largest shift/s.u. was 0.005 in the final refinement cycle. The final difference map had maxima and minima of 0.755 and -0.811 e/Å^3,

respectively.

Acknowledgment

The authors thank the National Science Foundation (grant CHE-0130835) and the University of Oklahoma for funds to purchase of the X-ray instrument and computers. This structure was determined by Douglas R. Powell.

References

(1) (a) Data Collection: SMART Software Reference Manual (2007). Bruker-AXS, 5465 E. Cheryl Parkway, Madison, WI 53711-5373 USA. (b) Data Reduction: SAINT Software Reference Manual (2007). Bruker-AXS, 5465 E. Cheryl Parkway, Madison, WI 53711-5373 USA.

(2) G. M. Sheldrick (2001). SADABS. Program for Empirical Absorption Correction of Area Detector Data. University of Göttingen, Germany.

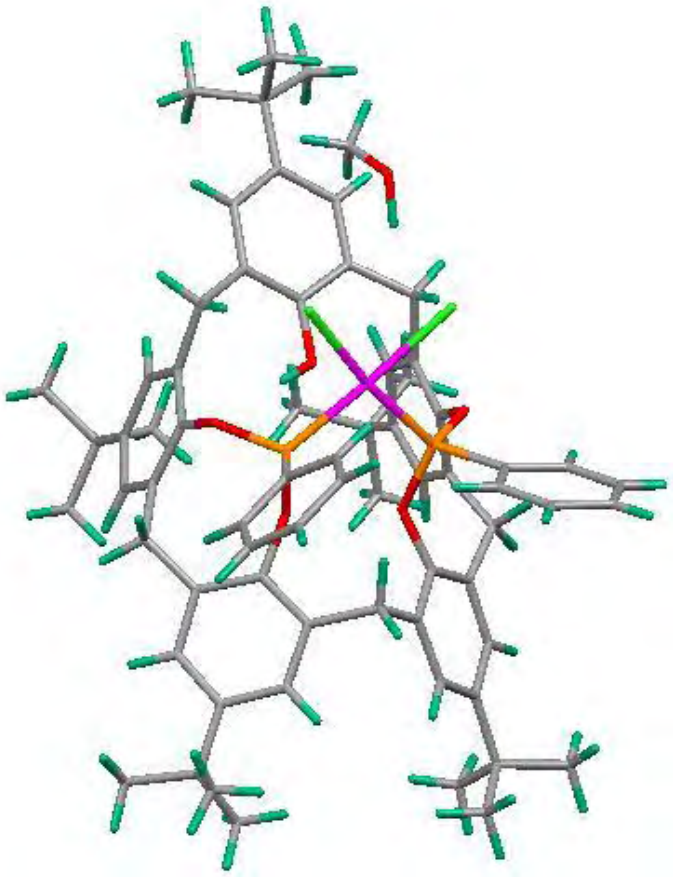
(3) G. M. Sheldrick (2008). *Acta Cryst., A64*, 112-122.

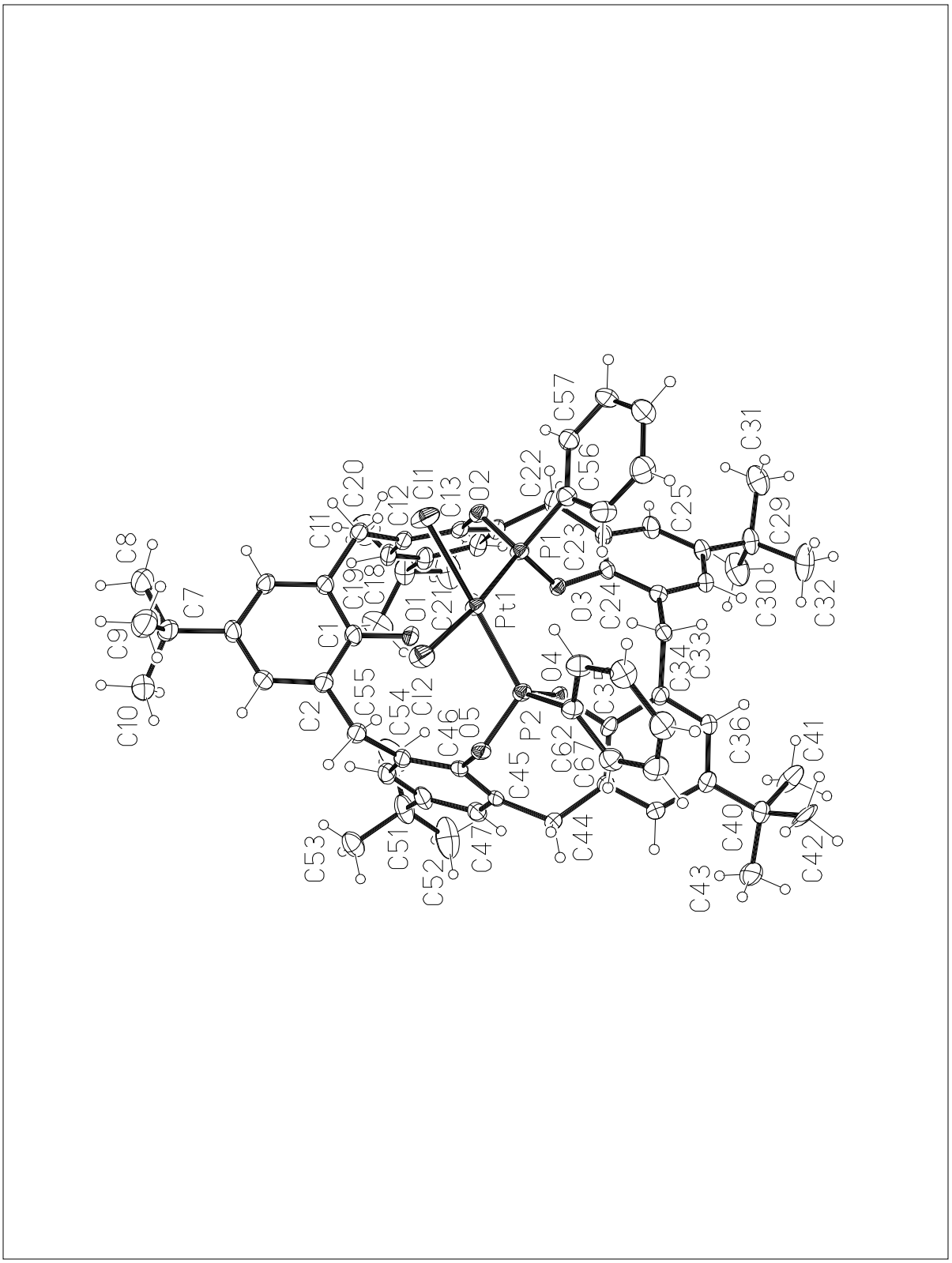
(4) P. van der Sluis and A. L. Spek (1990). *Acta Cryst. A46*, 194-201.

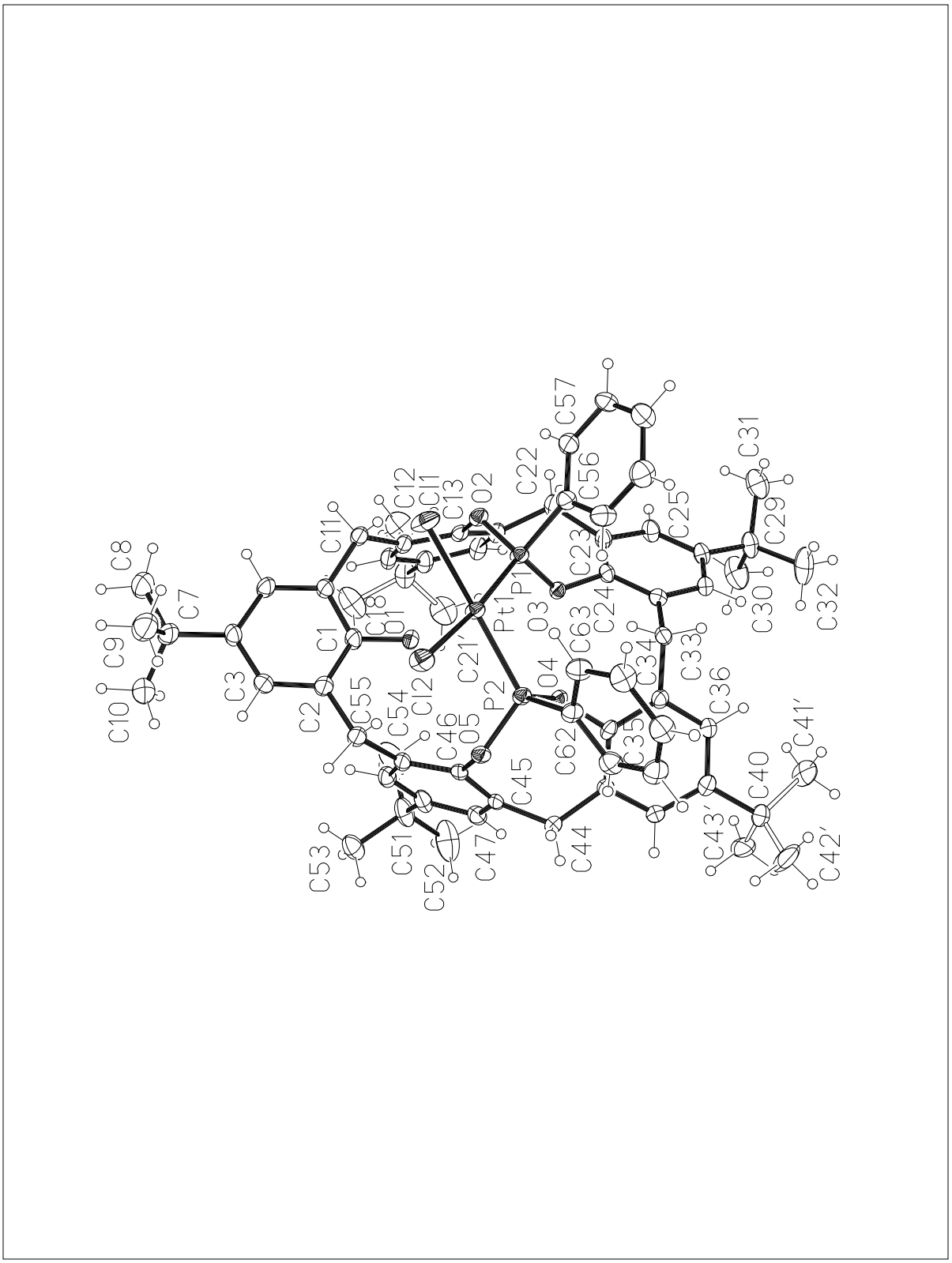
Table 7. Hydrogen bonds for 10061[Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(1)-H(1)...C(46)	0.75(6)	2.67(6)	3.377(7)	159(6)
O(1)-H(1)...C(50)	0.75(6)	2.28(6)	2.910(7)	142(6)
O(1A)-H(1A)...Cl(1)	0.90	2.58	3.319(5)	139.8
O(1A)-H(1A)...Cl(2)	0.90	2.54	3.267(5)	138.0

3•PtCl2







Comment

The asymmetric unit of the cell appeared to contain one molecule of the metal complex and three methanol solvent sites. Two of the methanol sites were severely disordered and were best modeled using the Squeeze program(4). Two of the *t*-butyl groups were disordered. The occupancies for atoms C19, C20, and C21 refined to 0.799(9) and 0.201(9) for the unprimed and primed atoms. The occupancies for atoms C41, C42, and C43 refined to 0.567(8) and 0.433(8) for the unprimed and primed atoms, respectively. Restraints on the positional and displacement parameters of the disordered *t*-butyl groups were required. The displacement ellipsoids were drawn at the 50% probability level.

Experimental

A light brown prism-shaped crystal of dimensions 0.46 x 0.30 x 0.29 mm was selected for structural analysis. Intensity data for this compound were collected using a diffractometer with a Bruker APEX ccd area detector (1) and graphite-monochromated Mo Kα radiation (λ = 0.71073 Å). The sample was cooled to 100(2) K. Cell parameters were determined from a non-linear least squares fit of 8323 peaks in the range 2.26 < θ < 28.29°. A total of 46574 data were measured in the range 1.64 < θ < 28.31° using ϕ and ω oscillation frames. The data were corrected for absorption by the semi-empirical method (2) giving minimum and maximum transmission factors of 0.4162 and 0.5538. The data were merged to form a set of 16310 independent data with R(int) = 0.0389 and a coverage of 100.0 %.

The triclinic space group $P\bar{1}$ was determined by systematic absences and statistical tests and verified by subsequent refinement. The structure was solved by direct methods and refined by full-matrix least-squares methods on F^2 (3). Hydrogen atom positions of hydrogens bonded to carbons were initially determined by geometry and refined by a riding model. Hydrogens bonded to the oxygens were located on a difference map, and their positions were refined independently. Non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atom displacement parameters were set to 1.2 (1.5 for methyl) times the isotropic equivalent displacement parameters of the bonded atoms. A total of 774 parameters were refined against 254 restraints and 16310 data to give wR(F^2) = 0.0961 and S = 1.000 for weights of w = 1/[σ^2 (F^2) + (0.0480 P)^2 + 4.8000 P], where P = [F_o^2 + 2F_c^2] / 3. The final R(F) was 0.0387 for the 14365 observed, [F >

$4\sigma(F)$], data. The largest shift/s.u. was 0.002 in the final refinement cycle. The final difference map had maxima and minima of 2.671 and -1.181 e/Å^3, respectively.

Acknowledgment

The authors thank the National Science Foundation (grant CHE-0130835) and the University of Oklahoma for funds to purchase of the X-ray instrument and computers. This structure was determined by Douglas R. Powell.

References

(1) (a) Data Collection: SMART Software Reference Manual (2007). Bruker-AXS, 5465 E. Cheryl Parkway, Madison, WI 53711-5373 USA. (b) Data Reduction: SAINT Software Reference Manual (2007). Bruker-AXS, 5465 E. Cheryl Parkway, Madison, WI 53711-5373 USA.

(2) G. M. Sheldrick (2001). SADABS. Program for Empirical Absorption Correction of Area Detector Data. University of Göttingen, Germany.

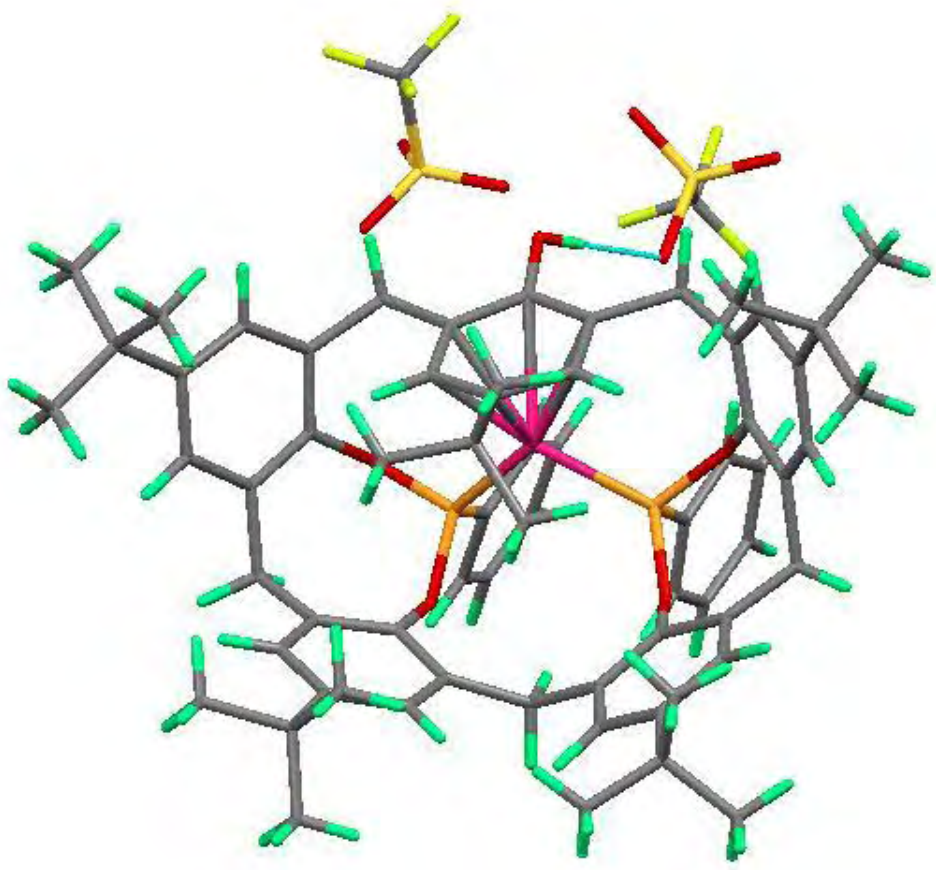
(3) G. M. Sheldrick (2008). *Acta Cryst., A64*, 112-122.

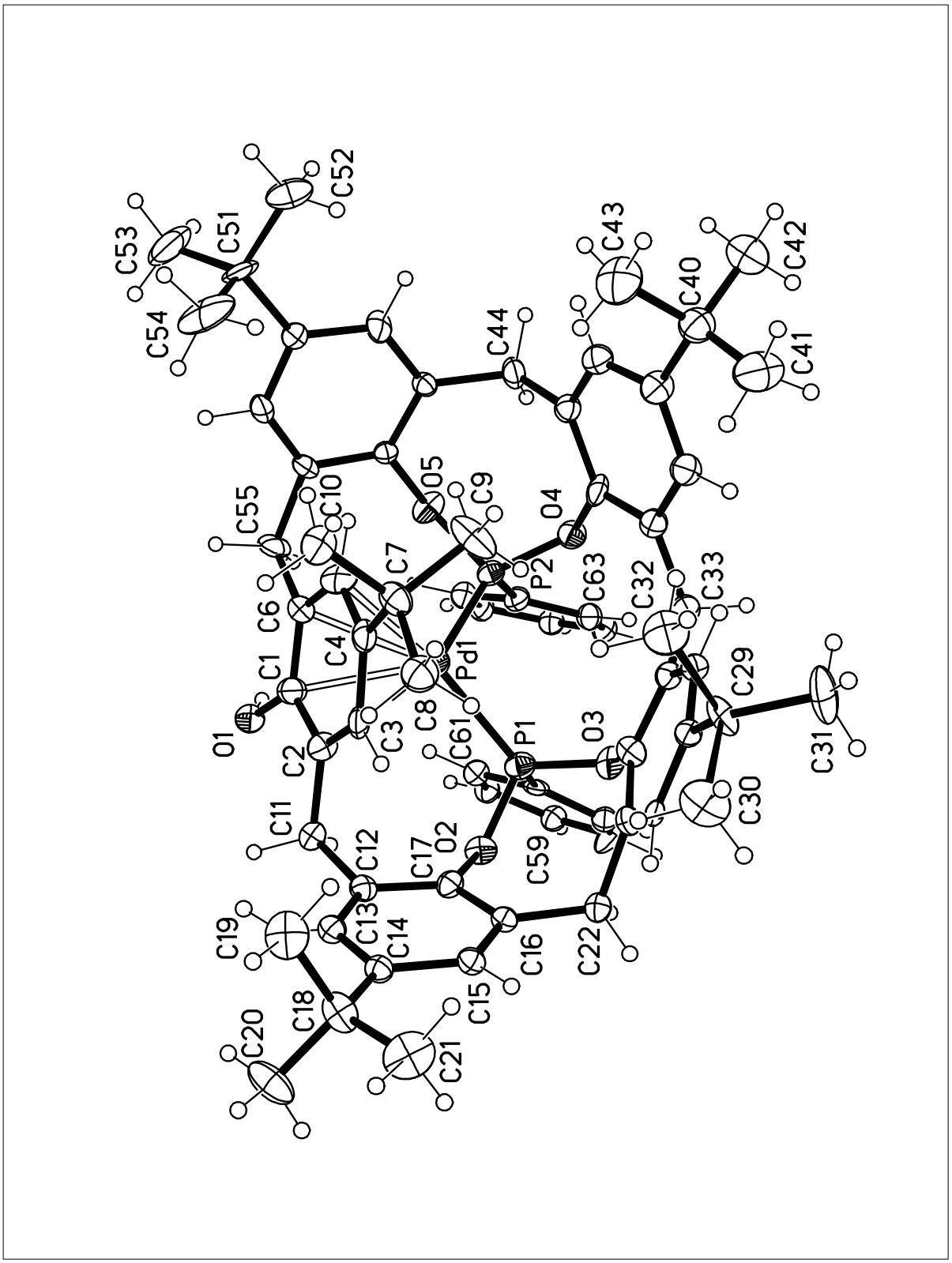
(4) P. van der Sluis and A. L. Spek (1990). *Acta Cryst. A46*, 194-201.

Table 7. Hydrogen bonds for 11005[Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(1)-H(1)...C(46)	0.71(5)	2.71(5)	3.382(4)	159(5)
O(1)-H(1)...C(50)	0.71(5)	2.28(5)	2.894(5)	145(5)
O(1A)-H(1A)...Cl(1)	1.06(7)	2.36(7)	3.301(5)	147(5)
O(1A)-H(1A)...Cl(2)	1.06(7)	2.54(7)	3.289(5)	127(5)

3•Pd(O3SCF3)2





Comment

The intensity data were truncated to 1.42 Å resolution because data in higher resolution shells all had R_{int} > 0.25. The two methylene chloride groups were disordered. The occupancies for the S molecule refined to 0.668(6) and 0.332(6) for the unprimed and primed atoms. The occupancies for the T molecule refined to 0.619(10) and 0.381(10) for the unprimed and primed atoms. Restraints on the positional and displacement parameters of the disordered atoms and the displacement parameters of all atoms were required. The displacement ellipsoids were drawn at the 50% probability level.

Experimental

A yellow plate-shaped crystal of dimensions 0.32 x 0.27 x 0.06 mm was selected for structural analysis. Intensity data for this compound were collected using a diffractometer with a Bruker APEX ccd area detector (1) and graphite-monochromated Cu Kα radiation (λ = 1.54178 Å). The sample was cooled to 100(2) K. Cell parameters were determined from a non-linear least squares fit of 2105 peaks in the range 2.53 < θ < 36.62°. A total of 11891 data were measured in the range 5.04 < θ < 32.88° using ϕ and ω oscillation frames. The data were corrected for absorption by the empirical method (2) giving minimum and maximum transmission factors of 0.2949 and 0.7516. The data were merged to form a set of 2560 independent data with R(int) = 0.1040 and a coverage of 97.3 %.

The monoclinic space group $P2_1/c$ was determined by systematic absences and statistical tests and verified by subsequent refinement. The structure was solved by direct methods and refined by full-matrix least-squares methods on F^2 (3). Hydrogen atom positions of hydrogens bonded to carbons were initially determined by geometry and refined by a riding model. The hydrogen bonded to O1 was located on a difference map, and its position was refined with a riding model. Non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atom displacement parameters were set to 1.2 (1.5 for methyl) times the isotropic equivalent displacement parameters of the bonded atoms. A total of 945 parameters were refined against 638 restraints and 2560 data to give wR(F^2) = 0.1680 and S = 1.069 for weights of w = 1/[σ^2 (F^2) + (0.1200 P)^2 + 7.0000 P], where P = [F_o^2 + 2F_c^2] / 3. The final R(F) was 0.0602 for the 1865 observed, [$F > 4\sigma(F)$], data. The largest shift/s.u. was 0.000 in the final refinement cycle. The final difference map had maxima and minima of 0.592 and -0.429 e/Å^3, respectively.

Acknowledgment

The authors thank the National Science Foundation (grant CHE-0130835) and the University of Oklahoma for funds to purchase of the X-ray instrument and computers. This structure was determined by Douglas R. Powell.

References

(1) (a) Data Collection: SMART Software Reference Manual (2007). Bruker-AXS, 5465 E. Cheryl Parkway, Madison, WI 53711-5373 USA. (b) Data Reduction: SAINT Software Reference Manual (2007). Bruker-AXS, 5465 E. Cheryl Parkway, Madison, WI 53711-5373 USA.

(2) G. M. Sheldrick (2001). SADABS. Program for Empirical Absorption Correction of Area Detector Data. University of Göttingen, Germany.

(3) G. M. Sheldrick (2008). *Acta Cryst., A64*, 112-122.

Table 3. Bond lengths [Å] and angles [°] for 09088a.

Pd(1)-P(2)	2.262(6)	C(10)-H(10C)	0.9800
Pd(1)-P(1)	2.263(6)	C(11)-C(12)	1.491(19)
Pd(1)-C(5)	2.367(18)	C(11)-H(11A)	0.9900
Pd(1)-C(2)	2.381(18)	C(11)-H(11B)	0.9900
Pd(1)-C(6)	2.396(18)	C(12)-C(13)	1.381(19)
Pd(1)-C(3)	2.396(18)	C(12)-C(17)	1.399(19)
Pd(1)-C(1)	2.433(19)	C(13)-C(14)	1.379(19)
Pd(1)-C(4)	2.43(2)	C(13)-H(13)	0.9500
P(1)-O(2)	1.595(12)	C(14)-C(15)	1.432(19)
P(1)-O(3)	1.598(12)	C(14)-C(18)	1.49(2)
P(1)-C(56)	1.793(19)	C(15)-C(16)	1.41(2)
P(2)-O(4)	1.570(12)	C(15)-H(15)	0.9500
P(2)-O(5)	1.591(12)	C(16)-C(17)	1.36(2)
P(2)-C(62)	1.795(19)	C(16)-C(22)	1.503(19)
O(1)-C(1)	1.342(18)	C(18)-C(21)	1.51(2)
O(1)-H(1)	0.8498	C(18)-C(20)	1.52(2)
O(2)-C(17)	1.393(18)	C(18)-C(19)	1.55(2)
O(3)-C(28)	1.425(19)	C(19)-H(19A)	0.9800
O(4)-C(39)	1.414(19)	C(19)-H(19B)	0.9800
O(5)-C(50)	1.404(19)	C(19)-H(19C)	0.9800
C(1)-C(6)	1.37(2)	C(20)-H(20A)	0.9800
C(1)-C(2)	1.38(2)	C(20)-H(20B)	0.9800
C(2)-C(3)	1.39(2)	C(20)-H(20C)	0.9800
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C(3)-C(4)	1.40(2)	C(21)-H(21B)	0.9800
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C(4)-C(5)	1.41(2)	C(22)-C(23)	1.52(2)
C(4)-C(7)	1.57(2)	C(22)-H(22A)	0.9900
C(5)-C(6)	1.44(2)	C(22)-H(22B)	0.9900
C(5)-H(5)	0.9500	C(23)-C(28)	1.36(2)
C(6)-C(55)	1.54(2)	C(23)-C(24)	1.39(2)
C(7)-C(8)	1.52(2)	C(24)-C(25)	1.39(2)
C(7)-C(10)	1.53(2)	C(24)-H(24)	0.9500
C(7)-C(9)	1.54(2)	C(25)-C(26)	1.39(2)
C(8)-H(8A)	0.9800	C(25)-C(29)	1.53(2)
C(8)-H(8B)	0.9800	C(26)-C(27)	1.38(2)
C(8)-H(8C)	0.9800	C(26)-H(26)	0.9500
C(9)-H(9A)	0.9800	C(27)-C(28)	1.39(2)
C(9)-H(9B)	0.9800	C(27)-C(33)	1.49(2)
C(9)-H(9C)	0.9800	C(29)-C(31)	1.49(2)
C(10)-H(10A)	0.9800	C(29)-C(30)	1.56(2)
C(10)-H(10B)	0.9800	C(29)-C(32)	1.57(2)

C(30)-H(30A)	0.9800	C(49)-C(50)	1.38(2)
C(30)-H(30B)	0.9800	C(49)-C(55)	1.47(2)
C(30)-H(30C)	0.9800	C(51)-C(52)	1.50(2)
C(31)-H(31A)	0.9800	C(51)-C(53)	1.53(2)
C(31)-H(31B)	0.9800	C(51)-C(54)	1.54(2)
C(31)-H(31C)	0.9800	C(52)-H(52A)	0.9800
C(32)-H(32A)	0.9800	C(52)-H(52B)	0.9800
C(32)-H(32B)	0.9800	C(52)-H(52C)	0.9800
C(32)-H(32C)	0.9800	C(53)-H(53A)	0.9800
C(33)-C(34)	1.50(2)	C(53)-H(53B)	0.9800
C(33)-H(33A)	0.9900	C(53)-H(53C)	0.9800
C(33)-H(33B)	0.9900	C(54)-H(54A)	0.9800
C(34)-C(39)	1.34(2)	C(54)-H(54B)	0.9800
C(34)-C(35)	1.44(2)	C(54)-H(54C)	0.9800
C(35)-C(36)	1.38(2)	C(55)-H(55A)	0.9900
C(35)-H(35)	0.9500	C(55)-H(55B)	0.9900
C(36)-C(37)	1.38(2)	C(56)-C(57)	1.40(2)
C(36)-C(40)	1.55(2)	C(56)-C(61)	1.414(19)
C(37)-C(38)	1.38(2)	C(57)-C(58)	1.36(2)
C(37)-H(37)	0.9500	C(57)-H(57)	0.9500
C(38)-C(39)	1.43(2)	C(58)-C(59)	1.35(2)
C(38)-C(44)	1.47(2)	C(58)-H(58)	0.9500
C(40)-C(41)	1.50(2)	C(59)-C(60)	1.36(2)
C(40)-C(42)	1.54(2)	C(59)-H(59)	0.9500
C(40)-C(43)	1.56(2)	C(60)-C(61)	1.42(2)
C(41)-H(41A)	0.9800	C(60)-H(60)	0.9500
C(41)-H(41B)	0.9800	C(61)-H(61)	0.9500
C(41)-H(41C)	0.9800	C(62)-C(63)	1.38(2)
C(42)-H(42A)	0.9800	C(62)-C(67)	1.39(2)
C(42)-H(42B)	0.9800	C(63)-C(64)	1.382(19)
C(42)-H(42C)	0.9800	C(63)-H(63)	0.9500
C(43)-H(43A)	0.9800	C(64)-C(65)	1.347(19)
C(43)-H(43B)	0.9800	C(64)-H(64)	0.9500
C(43)-H(43C)	0.9800	C(65)-C(66)	1.351(19)
C(44)-C(45)	1.53(2)	C(65)-H(65)	0.9500
C(44)-H(44A)	0.9900	C(66)-C(67)	1.42(2)
C(44)-H(44B)	0.9900	C(66)-H(66)	0.9500
C(45)-C(50)	1.39(2)	C(67)-H(67)	0.9500
C(45)-C(46)	1.39(2)	S(1M)-O(2M)	1.416(13)
C(46)-C(47)	1.39(2)	S(1M)-O(3M)	1.435(12)
C(46)-H(46)	0.9500	S(1M)-O(1M)	1.478(12)
C(47)-C(48)	1.39(2)	S(1M)-C(1M)	1.88(3)
C(47)-C(51)	1.55(2)	C(1M)-F(2M)	1.27(2)
C(48)-C(49)	1.37(2)	C(1M)-F(1M)	1.33(2)
C(48)-H(48)	0.9500	C(1M)-F(3M)	1.34(2)

S(1N)-O(1N)	1.387(13)	C(2S')-Cl(3')	1.736(10)
S(1N)-O(3N)	1.407(15)	C(2S')-H(2SA)	0.9900
S(1N)-O(2N)	1.426(16)	C(2S')-H(2SB)	0.9900
S(1N)-C(1N)	1.81(2)	Cl(1T)-C(2T)	1.736(10)
C(1N)-F(3N)	1.27(2)	C(2T)-Cl(3T)	1.736(10)
C(1N)-F(1N)	1.27(2)	C(2T)-H(2TA)	0.9900
C(1N)-F(2N)	1.36(2)	C(2T)-H(2TB)	0.9900
Cl(1S)-C(2S)	1.736(10)	Cl(1")-C(2T")	1.737(10)
C(2S)-Cl(3S)	1.737(10)	C(2T")-Cl(3")	1.737(10)
C(2S)-H(2S1)	0.9900	C(2T")-H(2TC)	0.9900
C(2S)-H(2S2)	0.9900	C(2T")-H(2TD)	0.9900
Cl(1')-C(2S')	1.736(10)		

P(2)-Pd(1)-P(1)	97.7(2)	O(3)-P(1)-Pd(1)	117.6(5)
P(2)-Pd(1)-C(5)	99.9(5)	C(56)-P(1)-Pd(1)	119.3(7)
P(1)-Pd(1)-C(5)	160.5(5)	O(4)-P(2)-O(5)	110.5(7)
P(2)-Pd(1)-C(2)	151.8(5)	O(4)-P(2)-C(62)	97.2(8)
P(1)-Pd(1)-C(2)	96.5(5)	O(5)-P(2)-C(62)	98.7(8)
C(5)-Pd(1)-C(2)	71.4(6)	O(4)-P(2)-Pd(1)	117.7(5)
P(2)-Pd(1)-C(6)	97.0(5)	O(5)-P(2)-Pd(1)	109.3(5)
P(1)-Pd(1)-C(6)	149.3(5)	C(62)-P(2)-Pd(1)	121.5(6)
C(5)-Pd(1)-C(6)	35.2(5)	C(1)-O(1)-H(1)	115.9
C(2)-Pd(1)-C(6)	60.1(7)	C(17)-O(2)-P(1)	133.4(12)
P(2)-Pd(1)-C(3)	159.7(5)	C(28)-O(3)-P(1)	123.1(12)
P(1)-Pd(1)-C(3)	100.2(5)	C(39)-O(4)-P(2)	131.2(12)
C(5)-Pd(1)-C(3)	61.1(6)	C(50)-O(5)-P(2)	131.9(11)
C(2)-Pd(1)-C(3)	33.8(5)	O(1)-C(1)-C(6)	121(2)
C(6)-Pd(1)-C(3)	71.9(6)	O(1)-C(1)-C(2)	118(2)
P(2)-Pd(1)-C(1)	118.8(5)	C(6)-C(1)-C(2)	121(2)
P(1)-Pd(1)-C(1)	117.0(5)	O(1)-C(1)-Pd(1)	133.7(13)
C(5)-Pd(1)-C(1)	60.6(6)	C(6)-C(1)-Pd(1)	72.0(12)
C(2)-Pd(1)-C(1)	33.3(5)	C(2)-C(1)-Pd(1)	71.2(11)
C(6)-Pd(1)-C(1)	33.0(5)	C(1)-C(2)-C(3)	122(2)
C(3)-Pd(1)-C(1)	60.2(6)	C(1)-C(2)-C(11)	118.5(19)
P(2)-Pd(1)-C(4)	126.2(5)	C(3)-C(2)-C(11)	119.6(19)
P(1)-Pd(1)-C(4)	126.7(5)	C(1)-C(2)-Pd(1)	75.4(12)
C(5)-Pd(1)-C(4)	34.1(5)	C(3)-C(2)-Pd(1)	73.7(11)
C(2)-Pd(1)-C(4)	59.8(6)	C(11)-C(2)-Pd(1)	126.3(13)
C(6)-Pd(1)-C(4)	61.3(6)	C(2)-C(3)-C(4)	119(2)
C(3)-Pd(1)-C(4)	33.6(5)	C(2)-C(3)-Pd(1)	72.5(11)
C(1)-Pd(1)-C(4)	70.2(6)	C(4)-C(3)-Pd(1)	74.7(12)
O(2)-P(1)-O(3)	108.1(7)	C(2)-C(3)-H(3)	120.4
O(2)-P(1)-C(56)	97.9(7)	C(4)-C(3)-H(3)	120.4
O(3)-P(1)-C(56)	102.2(8)	Pd(1)-C(3)-H(3)	123.9
O(2)-P(1)-Pd(1)	109.7(5)	C(3)-C(4)-C(5)	120(2)

C(3)-C(4)-C(7)	122.3(19)	C(2)-C(11)-H(11B)	109.7
C(5)-C(4)-C(7)	118.0(19)	H(11A)-C(11)-H(11B)	108.2
C(3)-C(4)-Pd(1)	71.7(12)	C(13)-C(12)-C(17)	115.7(19)
C(5)-C(4)-Pd(1)	70.4(11)	C(13)-C(12)-C(11)	123.7(19)
C(7)-C(4)-Pd(1)	132.4(12)	C(17)-C(12)-C(11)	119.1(19)
C(4)-C(5)-C(6)	119.6(19)	C(14)-C(13)-C(12)	127.5(19)
C(4)-C(5)-Pd(1)	75.6(12)	C(14)-C(13)-H(13)	116.3
C(6)-C(5)-Pd(1)	73.5(11)	C(12)-C(13)-H(13)	116.3
C(4)-C(5)-H(5)	120.2	C(13)-C(14)-C(15)	113.0(19)
C(6)-C(5)-H(5)	120.2	C(13)-C(14)-C(18)	125.1(19)
Pd(1)-C(5)-H(5)	122.1	C(15)-C(14)-C(18)	121.7(19)
C(1)-C(6)-C(5)	119(2)	C(16)-C(15)-C(14)	122.5(18)
C(1)-C(6)-C(55)	125(2)	C(16)-C(15)-H(15)	118.8
C(5)-C(6)-C(55)	116.1(19)	C(14)-C(15)-H(15)	118.8
C(1)-C(6)-Pd(1)	75.0(12)	C(17)-C(16)-C(15)	119(2)
C(5)-C(6)-Pd(1)	71.3(11)	C(17)-C(16)-C(22)	121(2)
C(55)-C(6)-Pd(1)	125.3(12)	C(15)-C(16)-C(22)	120.2(18)
C(8)-C(7)-C(10)	108.4(15)	C(16)-C(17)-O(2)	119.0(19)
C(8)-C(7)-C(9)	110.2(15)	C(16)-C(17)-C(12)	123(2)
C(10)-C(7)-C(9)	108.1(16)	O(2)-C(17)-C(12)	118.3(19)
C(8)-C(7)-C(4)	111.1(17)	C(14)-C(18)-C(21)	115.7(17)
C(10)-C(7)-C(4)	107.2(16)	C(14)-C(18)-C(20)	109.1(17)
C(9)-C(7)-C(4)	111.7(15)	C(21)-C(18)-C(20)	110.6(18)
C(7)-C(8)-H(8A)	109.5	C(14)-C(18)-C(19)	105.8(17)
C(7)-C(8)-H(8B)	109.5	C(21)-C(18)-C(19)	108.3(17)
H(8A)-C(8)-H(8B)	109.5	C(20)-C(18)-C(19)	106.9(16)
C(7)-C(8)-H(8C)	109.5	C(18)-C(19)-H(19A)	109.5
H(8A)-C(8)-H(8C)	109.5	C(18)-C(19)-H(19B)	109.5
H(8B)-C(8)-H(8C)	109.5	H(19A)-C(19)-H(19B)	109.5
C(7)-C(9)-H(9A)	109.5	C(18)-C(19)-H(19C)	109.5
C(7)-C(9)-H(9B)	109.5	H(19A)-C(19)-H(19C)	109.5
H(9A)-C(9)-H(9B)	109.5	H(19B)-C(19)-H(19C)	109.5
C(7)-C(9)-H(9C)	109.5	C(18)-C(20)-H(20A)	109.5
H(9A)-C(9)-H(9C)	109.5	C(18)-C(20)-H(20B)	109.5
H(9B)-C(9)-H(9C)	109.5	H(20A)-C(20)-H(20B)	109.5
C(7)-C(10)-H(10A)	109.5	C(18)-C(20)-H(20C)	109.5
C(7)-C(10)-H(10B)	109.5	H(20A)-C(20)-H(20C)	109.5
H(10A)-C(10)-H(10B)	109.5	H(20B)-C(20)-H(20C)	109.5
C(7)-C(10)-H(10C)	109.5	C(18)-C(21)-H(21A)	109.5
H(10A)-C(10)-H(10C)	109.5	C(18)-C(21)-H(21B)	109.5
H(10B)-C(10)-H(10C)	109.5	H(21A)-C(21)-H(21B)	109.5
C(12)-C(11)-C(2)	109.6(16)	C(18)-C(21)-H(21C)	109.5
C(12)-C(11)-H(11A)	109.7	H(21A)-C(21)-H(21C)	109.5
C(2)-C(11)-H(11A)	109.7	H(21B)-C(21)-H(21C)	109.5
C(12)-C(11)-H(11B)	109.7	C(16)-C(22)-C(23)	112.0(16)

C(16)-C(22)-H(22A)	109.2	H(32A)-C(32)-H(32C)	109.5
C(23)-C(22)-H(22A)	109.2	H(32B)-C(32)-H(32C)	109.5
C(16)-C(22)-H(22B)	109.2	C(27)-C(33)-C(34)	118.8(16)
C(23)-C(22)-H(22B)	109.2	C(27)-C(33)-H(33A)	107.6
H(22A)-C(22)-H(22B)	107.9	C(34)-C(33)-H(33A)	107.6
C(28)-C(23)-C(24)	119(2)	C(27)-C(33)-H(33B)	107.6
C(28)-C(23)-C(22)	125(2)	C(34)-C(33)-H(33B)	107.6
C(24)-C(23)-C(22)	117(2)	H(33A)-C(33)-H(33B)	107.1
C(23)-C(24)-C(25)	121(2)	C(39)-C(34)-C(35)	117(2)
C(23)-C(24)-H(24)	119.5	C(39)-C(34)-C(33)	123(2)
C(25)-C(24)-H(24)	119.5	C(35)-C(34)-C(33)	120.6(19)
C(26)-C(25)-C(24)	116(2)	C(36)-C(35)-C(34)	122.6(19)
C(26)-C(25)-C(29)	119.8(19)	C(36)-C(35)-H(35)	118.7
C(24)-C(25)-C(29)	124.2(19)	C(34)-C(35)-H(35)	118.7
C(27)-C(26)-C(25)	125(2)	C(35)-C(36)-C(37)	118(2)
C(27)-C(26)-H(26)	117.3	C(35)-C(36)-C(40)	121.0(19)
C(25)-C(26)-H(26)	117.3	C(37)-C(36)-C(40)	121(2)
C(26)-C(27)-C(28)	114(2)	C(36)-C(37)-C(38)	123(2)
C(26)-C(27)-C(33)	120.9(19)	C(36)-C(37)-H(37)	118.4
C(28)-C(27)-C(33)	125(2)	C(38)-C(37)-H(37)	118.4
C(23)-C(28)-C(27)	124(2)	C(37)-C(38)-C(39)	116(2)
C(23)-C(28)-O(3)	118(2)	C(37)-C(38)-C(44)	120(2)
C(27)-C(28)-O(3)	117(2)	C(39)-C(38)-C(44)	124(2)
C(31)-C(29)-C(25)	110.2(16)	C(34)-C(39)-O(4)	119(2)
C(31)-C(29)-C(30)	109.3(17)	C(34)-C(39)-C(38)	124(2)
C(25)-C(29)-C(30)	110.8(16)	O(4)-C(39)-C(38)	117.0(19)
C(31)-C(29)-C(32)	110.8(16)	C(41)-C(40)-C(42)	109.9(16)
C(25)-C(29)-C(32)	109.3(17)	C(41)-C(40)-C(36)	113.0(17)
C(30)-C(29)-C(32)	106.2(16)	C(42)-C(40)-C(36)	107.6(16)
C(29)-C(30)-H(30A)	109.5	C(41)-C(40)-C(43)	108.7(16)
C(29)-C(30)-H(30B)	109.5	C(42)-C(40)-C(43)	109.4(16)
H(30A)-C(30)-H(30B)	109.5	C(36)-C(40)-C(43)	108.2(16)
C(29)-C(30)-H(30C)	109.5	C(40)-C(41)-H(41A)	109.5
H(30A)-C(30)-H(30C)	109.5	C(40)-C(41)-H(41B)	109.5
H(30B)-C(30)-H(30C)	109.5	H(41A)-C(41)-H(41B)	109.5
C(29)-C(31)-H(31A)	109.5	C(40)-C(41)-H(41C)	109.5
C(29)-C(31)-H(31B)	109.5	H(41A)-C(41)-H(41C)	109.5
H(31A)-C(31)-H(31B)	109.5	H(41B)-C(41)-H(41C)	109.5
C(29)-C(31)-H(31C)	109.5	C(40)-C(42)-H(42A)	109.5
H(31A)-C(31)-H(31C)	109.5	C(40)-C(42)-H(42B)	109.5
H(31B)-C(31)-H(31C)	109.5	H(42A)-C(42)-H(42B)	109.5
C(29)-C(32)-H(32A)	109.5	C(40)-C(42)-H(42C)	109.5
C(29)-C(32)-H(32B)	109.5	H(42A)-C(42)-H(42C)	109.5
H(32A)-C(32)-H(32B)	109.5	H(42B)-C(42)-H(42C)	109.5
C(29)-C(32)-H(32C)	109.5	C(40)-C(43)-H(43A)	109.5

C(40)-C(43)-H(43B)	109.5	H(53A)-C(53)-H(53C)	109.5
H(43A)-C(43)-H(43B)	109.5	H(53B)-C(53)-H(53C)	109.5
C(40)-C(43)-H(43C)	109.5	C(51)-C(54)-H(54A)	109.5
H(43A)-C(43)-H(43C)	109.5	C(51)-C(54)-H(54B)	109.5
H(43B)-C(43)-H(43C)	109.5	H(54A)-C(54)-H(54B)	109.5
C(38)-C(44)-C(45)	114.3(15)	C(51)-C(54)-H(54C)	109.5
C(38)-C(44)-H(44A)	108.7	H(54A)-C(54)-H(54C)	109.5
C(45)-C(44)-H(44A)	108.7	H(54B)-C(54)-H(54C)	109.5
C(38)-C(44)-H(44B)	108.7	C(49)-C(55)-C(6)	111.4(16)
C(45)-C(44)-H(44B)	108.7	C(49)-C(55)-H(55A)	109.4
H(44A)-C(44)-H(44B)	107.6	C(6)-C(55)-H(55A)	109.4
C(50)-C(45)-C(46)	118(2)	C(49)-C(55)-H(55B)	109.4
C(50)-C(45)-C(44)	121(2)	C(6)-C(55)-H(55B)	109.4
C(46)-C(45)-C(44)	121.3(19)	H(55A)-C(55)-H(55B)	108.0
C(47)-C(46)-C(45)	122(2)	C(57)-C(56)-C(61)	120(2)
C(47)-C(46)-H(46)	119.1	C(57)-C(56)-P(1)	122.0(16)
C(45)-C(46)-H(46)	119.1	C(61)-C(56)-P(1)	118.1(15)
C(46)-C(47)-C(48)	117(2)	C(58)-C(57)-C(56)	120(2)
C(46)-C(47)-C(51)	122.1(19)	C(58)-C(57)-H(57)	120.1
C(48)-C(47)-C(51)	121(2)	C(56)-C(57)-H(57)	120.1
C(49)-C(48)-C(47)	124(2)	C(59)-C(58)-C(57)	119(2)
C(49)-C(48)-H(48)	117.9	C(59)-C(58)-H(58)	120.6
C(47)-C(48)-H(48)	117.9	C(57)-C(58)-H(58)	120.6
C(48)-C(49)-C(50)	117(2)	C(58)-C(59)-C(60)	126(2)
C(48)-C(49)-C(55)	123(2)	C(58)-C(59)-H(59)	116.8
C(50)-C(49)-C(55)	119(2)	C(60)-C(59)-H(59)	116.8
C(49)-C(50)-C(45)	123(2)	C(59)-C(60)-C(61)	115.7(19)
C(49)-C(50)-O(5)	118(2)	C(59)-C(60)-H(60)	122.2
C(45)-C(50)-O(5)	119(2)	C(61)-C(60)-H(60)	122.2
C(52)-C(51)-C(53)	109.5(18)	C(56)-C(61)-C(60)	119.6(19)
C(52)-C(51)-C(54)	107.6(19)	C(56)-C(61)-H(61)	120.2
C(53)-C(51)-C(54)	111.5(18)	C(60)-C(61)-H(61)	120.2
C(52)-C(51)-C(47)	112.2(17)	C(63)-C(62)-C(67)	118(2)
C(53)-C(51)-C(47)	107.9(18)	C(63)-C(62)-P(2)	122.0(17)
C(54)-C(51)-C(47)	108.1(16)	C(67)-C(62)-P(2)	119.8(16)
C(51)-C(52)-H(52A)	109.5	C(62)-C(63)-C(64)	119.9(19)
C(51)-C(52)-H(52B)	109.5	C(62)-C(63)-H(63)	120.1
H(52A)-C(52)-H(52B)	109.5	C(64)-C(63)-H(63)	120.1
C(51)-C(52)-H(52C)	109.5	C(65)-C(64)-C(63)	120(2)
H(52A)-C(52)-H(52C)	109.5	C(65)-C(64)-H(64)	120.2
H(52B)-C(52)-H(52C)	109.5	C(63)-C(64)-H(64)	120.2
C(51)-C(53)-H(53A)	109.5	C(64)-C(65)-C(66)	125(2)
C(51)-C(53)-H(53B)	109.5	C(64)-C(65)-H(65)	117.5
H(53A)-C(53)-H(53B)	109.5	C(66)-C(65)-H(65)	117.5
C(51)-C(53)-H(53C)	109.5	C(65)-C(66)-C(67)	114.2(19)

C(65)-C(66)-H(66)	122.9	F(1N)-C(1N)-S(1N)	115.3(17)
C(67)-C(66)-H(66)	122.9	F(2N)-C(1N)-S(1N)	107.2(16)
C(62)-C(67)-C(66)	123.1(19)	Cl(1S)-C(2S)-Cl(3S)	112.3(11)
C(62)-C(67)-H(67)	118.4	Cl(1S)-C(2S)-H(2S1)	109.1
C(66)-C(67)-H(67)	118.4	Cl(3S)-C(2S)-H(2S1)	109.1
O(2M)-S(1M)-O(3M)	116.4(10)	Cl(1S)-C(2S)-H(2S2)	109.1
O(2M)-S(1M)-O(1M)	116.6(9)	Cl(3S)-C(2S)-H(2S2)	109.1
O(3M)-S(1M)-O(1M)	113.8(9)	H(2S1)-C(2S)-H(2S2)	107.9
O(2M)-S(1M)-C(1M)	104.6(11)	Cl(1')-C(2S')-Cl(3')	112.4(12)
O(3M)-S(1M)-C(1M)	102.4(11)	Cl(1')-C(2S')-H(2SA)	109.1
O(1M)-S(1M)-C(1M)	99.9(11)	Cl(3')-C(2S')-H(2SA)	109.1
F(2M)-C(1M)-F(1M)	110(3)	Cl(1')-C(2S')-H(2SB)	109.1
F(2M)-C(1M)-F(3M)	110(3)	Cl(3')-C(2S')-H(2SB)	109.1
F(1M)-C(1M)-F(3M)	108(2)	H(2SA)-C(2S')-H(2SB)	107.8
F(2M)-C(1M)-S(1M)	112.3(19)	Cl(3T)-C(2T)-Cl(1T)	112.8(12)
F(1M)-C(1M)-S(1M)	108.2(18)	Cl(3T)-C(2T)-H(2TA)	109.0
F(3M)-C(1M)-S(1M)	107.5(19)	Cl(1T)-C(2T)-H(2TA)	109.0
O(1N)-S(1N)-O(3N)	116.2(11)	Cl(3T)-C(2T)-H(2TB)	109.0
O(1N)-S(1N)-O(2N)	112.5(11)	Cl(1T)-C(2T)-H(2TB)	109.0
O(3N)-S(1N)-O(2N)	113.4(13)	H(2TA)-C(2T)-H(2TB)	107.8
O(1N)-S(1N)-C(1N)	108.0(11)	Cl(1")-C(2T")-Cl(3")	107.5(17)
O(3N)-S(1N)-C(1N)	104.0(11)	Cl(1")-C(2T")-H(2TC)	110.2
O(2N)-S(1N)-C(1N)	100.9(11)	Cl(3")-C(2T")-H(2TC)	110.2
F(3N)-C(1N)-F(1N)	113(2)	Cl(1")-C(2T")-H(2TD)	110.2
F(3N)-C(1N)-F(2N)	104(2)	Cl(3")-C(2T")-H(2TD)	110.2
F(1N)-C(1N)-F(2N)	104(2)	H(2TC)-C(2T")-H(2TD)	108.5
F(3N)-C(1N)-S(1N)	112.0(18)		

Table 7. Hydrogen bonds for 09088a[Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(1)-H(1)...O(1M)	0.85	1.77	2.622(16)	179.0