Crystallographic Results of 1

Experimental

Data Collection

A colorless prismatic crystal of $C_{18}H_{36}OPt$ having approximate dimensions of 0.70 X 0.40 X 0.15 mm was mounted in a glass capillary. All measurements were made on a Rigaku AFC5R diffractometer with graphite monochromated Mo-Ka radiation and a rotating anode generator.

Cell constants and an orientation matrix for data collection, obtained from a least-squares refinement using the setting angles of 20 carefully centered reflections in the range $18.75 < 2q < 21.84^{o}$ corresponded to a primitive monoclinic cell with dimensions:

a = 6.481(6) Å
b = 11.892(5) Å $\beta = 101.51(2)^{o}$
c = 10.026(4) Å
V = 757.2(6) $Å^3$

For Z = 2 and F.W. = 463.57, the calculated density is 2.03 g/cm^3. Based on the systematic absences of:

0k0: k ± 2n

packing considerations, a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

$P2_1$ (No. 4)

The data were collected at a temperature of 23 ± 1°C using the ω–2θ scan technique to a maximum 2θ value of 55.0°. Omega scans of several intense reflections, made prior to data collection, had an average width at half-height of 0.32° with a take-off angle of 6.0°. Scans of $(1.57 + 0.30 \tan \theta)^{o}$ were made at a speed of 16.0°/min (in ω). The weak reflections ($I < 10.0s(I)$) were rescanned (maximum of 3 scans) and the counts were accumulated to ensure good counting statistics. Stationary background counts were recorded on each side of the reflection. The ratio of peak counting time to background counting time was 2:1. The diameter of the incident beam collimator was 1.0 mm, the crystal to detector distance was 258 mm, and the detector aperture was 9.0 x 13.0 mm (horizontal x vertical).

Data Reduction

Of the 1977 reflections which were collected, 1825 were unique ($R_{int} = 0.122$); equivalent reflections were merged. The intensities of three representative reflections were measured after every 150 reflections. No decay correction was applied.

The linear absorption coefficient, μ, for Mo-Kα radiation is 92.2 cm^{-1}. An empirical absorption correction based on azimuthal scans of several reflections was applied which resulted in transmission factors ranging from 0.15 to 1.00. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically. The final cycle of full-matrix least-squares refinement[3] on F was based on 1580 observed reflections ($I > 3.00\sigma(I)$) and 171 variable parameters and converged (largest parameter shift was 0.79 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.062$$

$$R_W = [\, \Sigma \, w \, (|Fo| - |Fc|)^2 \, / \, \Sigma \, w \, Fo^2]^{1/2} = 0.058$$

The standard deviation of an observation of unit weight[4] was 5.18. The weighting scheme was based on counting statistics and included a factor ($p = 0.002$) to downweight the intense reflections. Plots of $\Sigma \, w \, (|Fo| - |Fc|)^2$ versus |Fo|, reflection order in data collection, sin q/l and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 3.55 and -4.54 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5]. Anomalous dispersion effects were included in Fcalc[6]; the values for Df' and Df" were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[8]. All calculations were performed using the teXsan[9] crystallographic software package of Molecular Structure Corporation.

References

(1) SIR97: Altomare, A., Cascarano, M., Giacovazzo, C., Guagliardi, A. (1993). J. Appl. Cryst., 26, 343.

(2) DIRDIF94: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1994). The DIRDIF-94 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least Squares function minimized:

$$\Sigma w(|F_o|-|F_c|)^2 \text{ where}$$

$$w = 1/[s^2(Fo)] = [s^2_c(Fo) + p^2Fo^2/4]^{-1}$$

$s_c(Fo)$ = e.s.d. based on counting statistics

p = p-factor

(4) Standard deviation of an observation of unit weight:

$[Sw(|F_O|-|F_C|)^2/(N_O-N_V)]^{1/2}$

where: N_O = number of observations

N_V = number of variables

(5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(8) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(9) teXsan for Windows: Crystal Structure Analysis Package, Molecular Structure Corporation (1997).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{18}H_{36}OPt$
Formula Weight	463.57
Crystal Color, Habit	colorless, prismatic
Crystal Dimensions	0.70 X 0.40 X 0.15 mm
Crystal System	monoclinic
Lattice Type	Primitive
No. of Reflections Used for Unit Cell Determination (2θ range)	20 (18.8 - 21.8°)
Omega Scan Peak Width at Half-height	0.32°
Lattice Parameters	a = 6.481(6) Å b = 11.892(5) Å c = 10.026(4) Å β = 101.51(2) Å V = 757.2(6) Å^3
Space Group	$P2_1$ (No. 4)
Z value	2
D_{calc}	2.033 g/cm^3
F_{000}	460.00
μ(MoKα)	92.24 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku AFC5R

Radiation	MoKα (λ = 0.71069 Å)
	graphite monochromated
Attenuator	Zr foil (factors = 1.00, 3.52, 11.54, 41.93)
Take-off Angle	6.0°
Detector Aperture	9.0 mm horizontal
	13.0 mm vertical
Crystal to Detector Distance	258 mm
Temperature	23.0°C
Scan Type	ω–2θ
Scan Rate	16.0°/min (in ω) (up to 3 scans)
Scan Width	(1.57 + 0.30 tan θ)°
$2\theta_{max}$	55.0°
No. of Reflections Measured	Total: 1977
	Unique: 1825 (R_{int} = 0.122)
Corrections	Lorentz-polarization
	Absorption
	(trans. factors: 0.1461 - 1.0000)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR97)
Refinement	Full-matrix least-squares on F
Function Minimized	Σ w (|Fo| - |Fc|)^2
Least Squares Weights	$1/\sigma^2(Fo) = 4Fo^2/\sigma^2(Fo^2)$
p-factor	0.0023
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>3.00σ(I))	1580
No. Variables	171
Reflection/Parameter Ratio	9.24
Residuals: R; Rw	0.062 ; 0.058
Goodness of Fit Indicator	5.18
Max Shift/Error in Final Cycle	0.79
Maximum peak in Final Diff. Map	3.55 $e^-/Å^3$
Minimum peak in Final Diff. Map	-4.54 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
Pt(1)	0.31451(6)	0.0521	0.21809(4)	2.919(9)

O(1)	0.039(2)	0.2362(9)	0.406(1)	5.5(3)
C(1)	0.252(4)	0.219(1)	0.230(3)	5.3(5)
C(2)	0.042(2)	0.242(1)	0.282(2)	4.7(4)
C(3)	-0.154(2)	0.250(2)	0.177(2)	5.9(5)
C(4)	0.181(2)	0.058(3)	0.016(1)	5.4(4)
C(5)	-0.023(2)	0.035(3)	-0.037(2)	13.1(7)
C(6)	-0.106(3)	0.027(3)	-0.180(2)	9.9(8)
C(7)	0.035(4)	0.063(6)	-0.266(2)	14(1)
C(8)	0.239(5)	0.116(2)	-0.208(2)	10.7(8)
C(9)	0.302(3)	0.106(1)	-0.073(2)	6.7(5)
C(10)	0.578(2)	0.054(2)	0.407(1)	5.9(4)
C(11)	0.394(2)	0.034(4)	0.450(1)	5.6(5)
C(12)	0.328(2)	-0.088(1)	0.478(2)	5.0(4)
C(13)	0.314(3)	-0.174(2)	0.368(2)	6.8(5)
C(14)	0.295(4)	-0.149(1)	0.232(3)	5.3(5)
C(15)	0.493(3)	-0.103(2)	0.186(1)	6.4(5)
C(16)	0.710(2)	-0.081(2)	0.262(2)	8.0(6)
C(17)	0.741(2)	-0.026(1)	0.387(1)	4.6(4)

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos g + 2U_{13}(aa^*cc^*)\cos b + 2U_{23}(bb^*cc^*)\cos a)$

Table 2. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pt(1)	0.0400(2)	0.0360(3)	0.0323(2)	-0.0103(6)	0.0011(2)	-0.0071(7)
O(1)	0.095(8)	0.054(8)	0.062(7)	-0.002(7)	0.024(6)	-0.024(6)
C(1)	0.11(2)	-0.008(8)	0.11(2)	-0.028(8)	0.05(1)	-0.022(8)
C(2)	0.07(1)	0.05(1)	0.07(1)	0.025(9)	0.023(9)	0.004(9)
C(3)	0.06(1)	0.08(1)	0.07(1)	0.01(1)	0.002(9)	0.03(1)
C(4)	0.086(9)	0.08(2)	0.039(7)	0.05(2)	0.001(7)	0.02(1)
C(5)	0.08(1)	0.32(3)	0.08(1)	0.10(2)	-0.044(9)	-0.15(2)
C(6)	0.10(1)	0.12(3)	0.13(2)	0.07(2)	-0.04(1)	-0.08(2)
C(7)	0.21(3)	0.26(5)	0.04(1)	0.11(5)	-0.04(1)	-0.04(3)
C(8)	0.26(3)	0.07(2)	0.08(2)	0.05(2)	0.04(2)	0.05(1)
C(9)	0.14(1)	0.07(1)	0.040(9)	-0.04(1)	0.01(1)	0.001(8)
C(10)	0.051(7)	0.12(1)	0.044(7)	0.04(1)	-0.015(6)	-0.08(1)
C(11)	0.057(7)	0.12(2)	0.027(6)	0.01(2)	-0.004(5)	-0.05(1)
C(12)	0.07(1)	0.04(1)	0.08(1)	0.005(8)	0.004(9)	0.038(9)
C(13)	0.11(1)	0.05(1)	0.09(1)	0.01(1)	0.01(1)	0.06(1)
C(14)	0.10(2)	0.001(9)	0.08(1)	0.00(1)	-0.03(1)	-0.005(9)

C(15)	0.10(1)	0.11(2)	0.043(9)	0.07(1)	0.023(9)	0.05(1)
C(16)	0.07(1)	0.11(2)	0.11(2)	0.05(1)	-0.02(1)	-0.06(1)
C(17)	0.044(7)	0.04(1)	0.08(1)	0.023(7)	-0.010(7)	0.015(8)

The general temperature factor expression:

$\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 3. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
Pt(1)	C(1)	2.03(4)	Pt(1)	C(4)	2.04(3)
Pt(1)	C(10)	2.28(2)	Pt(1)	C(11)	2.29(3)
Pt(1)	C(14)	2.40(4)	Pt(1)	C(15)	2.24(3)
O(1)	C(2)	1.25(3)	C(1)	C(2)	1.58(7)
C(2)	C(3)	1.48(5)	C(4)	C(5)	1.35(5)
C(4)	C(9)	1.42(5)	C(5)	C(6)	1.43(5)
C(6)	C(7)	1.44(8)	C(7)	C(8)	1.5(1)
C(8)	C(9)	1.34(6)	C(10)	C(11)	1.37(4)
C(10)	C(17)	1.46(5)	C(11)	C(12)	1.55(9)
C(12)	C(13)	1.49(5)	C(13)	C(14)	1.38(7)
C(14)	C(15)	1.55(7)	C(15)	C(16)	1.49(5)
C(16)	C(17)	1.40(4)			

Table 4. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
C(1)	Pt(1)	C(4)	89(3)	C(1)	Pt(1)	C(10)	94(2)
C(1)	Pt(1)	C(11)	92(3)	C(1)	Pt(1)	C(14)	163(1)
C(1)	Pt(1)	C(15)	158(2)	C(4)	Pt(1)	C(10)	157(1)
C(4)	Pt(1)	C(11)	168(2)	C(4)	Pt(1)	C(14)	94(3)
C(4)	Pt(1)	C(15)	91(2)	C(10)	Pt(1)	C(11)	35(1)
C(10)	Pt(1)	C(14)	90(2)	C(10)	Pt(1)	C(15)	79(2)
C(11)	Pt(1)	C(14)	81(3)	C(11)	Pt(1)	C(15)	93(2)
C(14)	Pt(1)	C(15)	39(2)	Pt(1)	C(1)	C(2)	113(3)
O(1)	C(2)	C(1)	120(4)	O(1)	C(2)	C(3)	122(3)
C(1)	C(2)	C(3)	117(3)	Pt(1)	C(4)	C(5)	125(3)
Pt(1)	C(4)	C(9)	117(3)	C(5)	C(4)	C(9)	117(4)
C(4)	C(5)	C(6)	123(4)	C(5)	C(6)	C(7)	115(5)
C(6)	C(7)	C(8)	121(4)	C(7)	C(8)	C(9)	116(5)
C(4)	C(9)	C(8)	125(5)	Pt(1)	C(10)	C(11)	73(1)
Pt(1)	C(10)	C(17)	109(2)	C(11)	C(10)	C(17)	129(6)
Pt(1)	C(11)	C(10)	72(2)	Pt(1)	C(11)	C(12)	105(3)

C(10)	C(11)	C(12)	121(6)	C(11)	C(12)	C(13)	119(3)
C(12)	C(13)	C(14)	125(3)	Pt(1)	C(14)	C(13)	106(3)
Pt(1)	C(14)	C(15)	65(2)	C(13)	C(14)	C(15)	117(4)
Pt(1)	C(15)	C(14)	76(2)	Pt(1)	C(15)	C(16)	104(3)
C(14)	C(15)	C(16)	131(3)	C(15)	C(16)	C(17)	120(3)
C(10)	C(17)	C(16)	117(3)				

Table 5. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
Pt(1)	C(1)	H(2)	108.6	Pt(1)	C(1)	H(1)	108.6
C(2)	C(1)	H(2)	108.6	C(2)	C(1)	H(1)	108.6
H(2)	C(1)	H(1)	109.5	C(2)	C(3)	H(3)	109.5
C(2)	C(3)	H(4)	109.5	C(2)	C(3)	H(5)	109.4
H(3)	C(3)	H(4)	109.5	H(3)	C(3)	H(5)	109.5
H(4)	C(3)	H(5)	109.5	C(4)	C(5)	H(6)	118.4
C(6)	C(5)	H(6)	118.3	C(5)	C(6)	H(7)	122.5
C(7)	C(6)	H(7)	122.4	C(6)	C(7)	H(8)	119.8
C(8)	C(7)	H(8)	119.3	C(7)	C(8)	H(9)	122.4
C(9)	C(8)	H(9)	122.1	C(4)	C(9)	H(10)	117.4
C(8)	C(9)	H(10)	117.4	Pt(1)	C(10)	H(11)	112.8
C(11)	C(10)	H(11)	112.8	C(17)	C(10)	H(11)	112.8
Pt(1)	C(11)	H(12)	116.5	C(10)	C(11)	H(12)	116.5
C(12)	C(11)	H(12)	116.4	C(11)	C(12)	H(13)	107.1
C(11)	C(12)	H(14)	107.1	C(13)	C(12)	H(13)	107.1
C(13)	C(12)	H(14)	107.1	H(13)	C(12)	H(14)	109.5
C(12)	C(13)	H(15)	105.5	C(12)	C(13)	H(16)	105.6
C(14)	C(13)	H(15)	105.6	C(14)	C(13)	H(16)	105.6
H(15)	C(13)	H(16)	109.5	Pt(1)	C(14)	H(17)	118.8
C(13)	C(14)	H(17)	118.8	C(15)	C(14)	H(17)	118.7
Pt(1)	C(15)	H(18)	112.2	C(14)	C(15)	H(18)	112.2
C(16)	C(15)	H(18)	112.2	C(15)	C(16)	H(19)	106.7
C(15)	C(16)	H(20)	106.8	C(17)	C(16)	H(19)	106.6
C(17)	C(16)	H(20)	106.8	H(19)	C(16)	H(20)	109.5
C(10)	C(17)	H(21)	107.6	C(10)	C(17)	H(22)	107.7
C(16)	C(17)	H(21)	107.6	C(16)	C(17)	H(22)	107.7
H(21)	C(17)	H(22)	109.5				