1) Physical and NMR data of the complexes, 1b, 1c, 1d, 2b, 3b, and 4b

1b: Nitromethane (14 μl, 0.25 mmol) in CH_2Cl_2 (0.5 ml) was added to the Cp*Ir[(*R*,*R*)-Tscydn] (**1a**) (150 mg, 0.25 mmol) in CH_2Cl_2 (1 ml). The purple color of the solution changed to yellow immediately. Evaporation of the solvent under reduced pressure at room temperature gave a pale yellow complex, **1b**, quantitatively. Crystals suitable for X-ray single crystal structural analysis were obtained by recrystallization from CH_3NO_2. More conveniently, a mixture of Cp*IrCl[(*R*,*R*)-Tscydn] (187 mg, 0.259 mmol) and 4 ml of nitromethane in CH_2Cl_2 (6 ml) was treated with 1.0 equiv of NaOH aq (0.1 M) at room temperature for 30 min. The solvent was then removed under reduced pressure at room temperature to give a yellow compound, which was extracted with CH_2Cl_2. Evaporation of the solvent gave **1b** in 90% yield.

Decomp >170 °C. ^{1}H NMR (300 MHz, CD_2Cl_2); δ 0.80–2.1 (9H, cyclohexane ring protons), 1.59 (s, 15H, $C_5(CH_3)_5$), 2.38 (s, 3H, CH_3 of Ts), 2.84 (m, 1H, C*H*-NTs), 3.57 (d, 1H, N*H*H), 4.62 (t, 1H, NH*H*), 5.58 (d, ^3J (H,H) = 5.6 Hz, 1H, IrC*H*HNO$_2$), 5.66 (d, ^3J (H,H) = 5.6 Hz, 1H, IrCH*H*NO$_2$), 7.20, 7.69 (d, ^3J (H,H) = 8.3 Hz, 4H, aromatic ring protons). The signal of one of two NH_2 protons was overlapped with signals of cyclohexane ring protons. ^{13}C NMR (75 MHz, CD_2Cl_2); δ 8.6, 21.4 (CH_3), 25.1, 25.3, 34.1, 36.7 (CH_2), 64.4, 68.1, 126.7, 129.1 (*C*H) 86.7, 140.8, 146.0 (*C*). Anal calcd. for $C_{24}H_{36}N_3O_4SIr$: C 44.02, H 5.54, N 6.42, S 4.90; found: C 43.90, H 5.60, N 6.33, S 4.70.

2b, **3b**, and **4b**: Similarly, **3b** and **4b** was obtained by the reaction of **3a** or **4a** with nitromethane in CH_2Cl_2. **2b** was obtained by the reaction of RuCl[(*R*,*R*)-Tscydn](*p*-cymene) with nitromethane in the presence of 1.0 equiv of NaOHaq in CH_2Cl_2.

2b: Decomp >145 °C. ^{1}H NMR (400 MHz, CD_2Cl_2); δ 0.7-1.5(9H, cyclohexane ring protons), 1.26, 1.35 (d, ^3J (H,H) = 6.8 Hz, 6H, $(CH_3)_2CH$), 2.33 (s, 3H, CH_3 of *p*-cymene), 2.39 (s, 3H, CH_3 of Ts), 2.86 (m, 1H, $CH(CH_3)_2$), 3.95 (t, 1H, C*H*-NTs), 4.08 (d, ^3J (H,H) = 4.1 Hz, 1H, N*H*H), 4.08, 5.67 (d, ^3J (H,H) = 5.9 Hz, 2H, aromatic ring protons of *p*-cymene), 4.91 (m, 1H, Ru-C*H*HNO$_2$), 5.18, 5.39 (d, ^3J (H,H) = 4.3 Hz, 2H, aromatic ring protons of *p*-cymene), 7.22, 7.65 (d, ^3J (H,H) = 7.8 Hz, 4H, aromatic ring protons of Ts). The signals for *C*H-NH_2 and one of the two NH_2 protons was overlapped with signals of cyclohexane ring protons. Anal calcd. for $C_{24}H_{36}N_3O_4SRu$: C 51.14, H 6.44, N 7.45, S 5.69; found: C 50.98, H 6.40, N 7.16, S 5.51.

3b: Decomp >135 °C. ^{1}H NMR (400 MHz, $CDCl_3$); δ 1.75 (s, 15H, $C_5(CH_3)_5$), 2.16(s, 3H, CH_3 of *p*-

cymene), 3.65 (d, ^3J (H,H) = 9 Hz, 1H, N*H*H), 3.71 (m, 1H, C*H*NH$_2$), 4.58 (d, ^3J (H,H) = 11 Hz, 1H, C*H*NTs), 5.90 (t, ^3J (H,H) = 9Hz, 1H, NH*H*), 5.97 (d, ^3J (H,H) = 5.9 Hz, 1H, IrC*H*H), 6.26 (d, ^3J (H,H) = 5.9 Hz, 1H, IrCH*H*), 6.6-7.3 (aromatic ring protons, 14H). ^{13}C NMR (75 MHz, CD_3NO_2): δ 18.9, 24.3 (*C*H$_3$) 73.7, 73.8, 125.4, 126.0, 126.3, 126.4, 126.9, 127.4, 127.7, 128.1 (*C*H) 86.1, 137.0, 138.8, 138.8, 141.4 (C). The signal of Ir-*C*D$_2$NO$_2$ was overlapped with signals of solvent. Anal calcd. for $C_{32}H_{38}N_3O_4SIr$: C 51.05, H 5.09, N 5.58, S 4.26; found: C 50.67, H 5.11, N 5.54, S 4.06.

4b: ^{1}H NMR (400 MHz, CD_2Cl_2); δ 1.32, 1.44 (d, ^3J (H,H) = 6.8 Hz, 6H, (C*H*$_3$)$_2$CH), 2.22 (s, 3H, C*H*$_3$ of *p*-cymene), 2.46 (s, 3H, C*H*$_3$ of Ts), 2.96 (m, 1H, C*H*(CH$_3$)$_2$), 3.93(m, 1H, N*H*H), 4.15(d, ^3J (H,H) = 11 Hz, 1H, C*H*NTs), 4.45(m, 1H, C*H*NH$_2$), 5.02 (d, *J* = 6 Hz, RuC*H*H, 1H), 5.06 (d, ^3J (H,H) = 6 Hz, RuCH*H*, 1H), 4.57, 5.89 (d, *J* = 4.3 Hz, 2H, aromatic ring protons of *p*-cymene), 5.39, 5.50 (d, ^3J (H,H) = 5.9 Hz, 2H, aromatic ring protons of *p*-cymene), 6.6–7.2 (14 H, aromatic ring protons). The signal of one of two N*H*$_2$ protons was overlapped with signals of the aromatic ring protons.

^{13}C NMR (75 MHz, CD_3NO_2); δ 16.5, 19.2, 20.6, 21.9 (*C*H$_3$) 29.4, 67.7, 72.4, 79.8, 83.3, 84.0, 84.7, 125.6, 126.4, 126.6, 127.3, 127.5, 127.9, 128.6 (*C*H) 98.3, 106.3, 138.2, 138.9, 139.2, 142.8 (*C*). The signal of Ru–*C*D$_2$NO$_2$ was overlapped with signals of solvent.

1c and **1d**: **1c** and **1d** were also obtained quantitatively by the reaction of Cp*IrCl[(*R*,*R*)-Tscydn] with acetone or phenylacetylene in the presence of 1.0 equiv of NaOHaq in CH_2Cl_2.

1c: Decomp >130 °C. IR (KBr): υ [cm^{-1}]: 1608 (CO). ^{1}H NMR (300 MHz, CD_2Cl_2); δ 0.70–2.01 (9H, cyclohexane ring protons), 1.64 (s, 15H, C$_5$(C*H*$_3$)$_5$), 1.96 (s, 3H, COC*H*$_3$), 2.31 (s, 3H, C*H*$_3$ of Ts), 2.68 (m, 1H, C*H*-NTs), 3.12 (d, ^3J (H,H) = 6.6 Hz, 1H, IrC*H*H), 3.20 (br, 1H, N*H*H), 3.53 (d, ^3J (H,H) = 6.6 Hz, 1H, IrCH*H*), 5.56 (t, 1H, NH*H*), 5.66 (d, ^3J (H,H) = 5.6 Hz, 1H, CH*H*-NO$_2$), 7.19, 7.71 (d, ^3J (H,H) = 8.0 Hz, 4H, aromatic ring protons). ^{13}C NMR (75 MHz, CD_2Cl_2); δ 9.4, 20.7, 21.3 (*C*H$_3$), 25.2, 25.4, 34.2, 36.6, 85.2 (*C*H$_2$), 63.7, 67.5, 127.0, 129.0 (*C*H)140.3, 146.7, 216.8 (*C*). Signal of *C*$_5$(CH$_3$)$_5$ was overlapped with other signals. Anal calcd. for $C_{26}H_{39}N_2O_3SIr{\cdot}H_2O$: C 46.62, H 6.17, N 4.18, S 4.79; found: C 46.61, H 5.93, N 3.90, S 4.85.

1d: Decomp >125 °C. ^{1}H NMR (300 MHz, CD2Cl2); δ 0.50–2.55 (10H, cyclohexane ring protons), 1.79 (s, 15H, C$_5$(C*H*$_3$)$_5$), 2.24 (s, 3H, C*H*$_3$ of Ts), 3.42 (br, 2H, N*HH*), 7.01, 7.93 (d, ^3J (H,H) = 7.8 Hz, 4H, aromatic ring protons of Ts), 7.0–7.3 (m, 5H, aromatic ring protons of phenylethyl group). ^{13}C NMR (75 MHz, CD_2Cl_2); δ 9.1, 20.8 (*C*H$_3$), 24.7, 24.8, 31.4, 36.1(*C*H$_2$), 64.1, 67.2, 124.4 , 127.6, 127.7, 129.1,

131.5 (*C*H), 88.1, 103.2, 139.7, 140.9 (*C*). Anal calcd. for $C_{31}H_{39}N_2O_2SIr \cdot C_6H_5CCH$: C 58.69, H 5.68, N 3.51, S 4.02; found: C 59.03, H 5.75, N 3.26, S 3.80.

2) Single-crystal X-ray analysis data of (η^5-$C_5(CH_3)_5$)Ir(CH_2NO_2)[(1*R*,2*R*)-*N*-*p*-toluenesulfonyl-1,2-cyclohexanediamine] (1b)

Experimental

Data Collection

An yellow prismatic crystal of $C_{24}H_{36}N_3O_4SIr \cdot CH_3NO_2$ having approximate dimensions of 0.20 x 0.20 x 0.20 mm was mounted on a glass fiber. All measurements were made on a Rigaku AFC7R diffractometer with graphite monochromated Mo-Kα 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 21 carefully centered reflections in the range $29.73 < 2\theta < 30.04°$ corresponded to a primitive orthorhombic cell with dimensions:

a = 13.198(10) Å
b = 22.501(10) Å
c = 9.39(1) Å
V = 2787(3) $Å^3$

For Z = 4 and F.W. = 715.89, the calculated density is 1.71 g/cm^3. The systematic absences of:
h00: h # 2n
0k0: k # 2n
00l: l # 2n
uniquely determine the space group to be:
$P2_12_12_1$ (#19)

The data were collected at a temperature of –20 ± 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 0.42° with a take-off angle of 6.0°. Scans of (1.84 + 0.30 tan θ)° were made at a speed of 16.0°/min (in ω). The weak reflections ($I < 10.0\sigma(I)$) 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 and the crystal to detector distance was 235 mm. The computer-controlled slits were set to 9.0 mm (horizontal) and 13.0 mm (vertical).

Data Reduction

Of the 3627 reflections which were collected, 3618 were unique (*R*int = 0.082). The intensities

of three representative reflection were measured after every 150 reflections. No decay correction was applied.

The linear absorption coefficient, μ, for Mo-Kα radiation is 49.2 cm^{-1}. An emoirical absoption correction based on azimuthal scans of several reflections was applied which resulted in transmission factors ranging from 0.53 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. Hydrogen atoms were included but not refined. The final cycle of full-matrix least-squares refinement[3] was based on 2938 observed reflections ($I > 2.00\sigma(I)$) and 298 variable parameters and converged (largest parameter shift was 0.04 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma||Fo|-|Fc||/\Sigma|Fo| = 0.050$$

$$Rw = [(\Sigma w(|Fo|-|Fc|)^2 /\Sigma\ wFo^2)]^{1/2} = 0.059$$

The standard deviation of an observation of unit weight[4] was 2.55. The weighting scheme was based on counting statistics and included a factor ($p = 0.007$) to downweight the intense reflections. Plots of $\Sigma w(|Fo|-|Fc|)^2$ versus |Fo|, reflection order in data collection, $\sin\theta/\lambda$ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 1.60 and –3.40 $e^-/Å^3$, respectively.

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

References

(1) SHELXS86: Sheldrick, G. M. In *Crystallographic Computing 3* ; Sheldrick, G. M.; Kruger, C.; Goddard, R. Eds.; Oxford University Press, 1985, pp 175-189.

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

(3) Least-Squares:

Function minimized: $\Sigma w(|Fo|-|Fc|)^2$

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

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

p = p-factor

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

$$[\Sigma w(|Fo|-|Fc|)^2/(No-Nv)]^{1/2}$$

where: No = number of observations

Nv = number of variables

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

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

(7) Creagh, D. C.; McAuley, W. J. *International Tables for Crystallography,Vol C*; Wilson, A. J. C. Ed.; Kluwer Academic Publishers: Boston, 1992, Table 4.2.6.8, pp 219–222 .

(8) Creagh, D. C.; Hubbell, J. H. *International Tables for Crystallography*, *Vol C*; Wilson, A. J. C. Ed.; Kluwer Academic Publishers: Boston, 1992, Table 4.2.4.3, pp 200–206.

(9) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation, 1985 & 1992.

EXPERIMENTAL DETAILS

A. Crystal Data	
Empirical Formula	$C_{24}H_{36}N_3O_4SIr \cdot CH_3NO_2$
Formula Weight	715.89
Crystal Color, Habit	yellow, prismatic
Crystal Dimensions	0.20 x 0.20 x 0.20 mm
Crystal System	orthortombic
Lattice Type	Primitive
No. of Reflections Used for Unit	
Cell Determination (2θ range)	21 (29.7–30.0°)
Omega Scan Peak Width at Half-height	0.42°
Lattice Parameters	a = 13.198(10)Å
	b = 22.501(10) Å
	c = 9.39 (1) Ä
	V = 2787(3) $Å^3$
Space Group	$P2_12_12_1$(#19)
Z value	4
D_{calc}	1.706 g/cm^3
F_{000}	1432.00
μ(MoKα)	49.23cm^{-1}
B. Intensity Measurements	
Diffractometer	Rigaku AFC7R
Radiation	MoKα (λ= 0.71069 Å)
	graphite monochromated
Attenuator	Zr foil (factor = 7.02)
Take-off Angle	6.0°
Detector Aperture	9.0 mm horizontal
	13.0 mm vertical
Crystal to Detector Distance	235 mm
Temperature	–20.0 °C
Scan Type	ω–2θ
Scan Rate	16.0°/min (in ω) (up to 3 scans)
Scan Width	(1.84 + 0.30 tan θ)°
2θ*max*	55.0°
No. of Reflections Measured	Total: 3627
	Unique: 3618 (R*int* = 0.082)

Corrections	Lorentz-polarization Absorption (trans. Factors: 0.5325–1.000)
C. Structure Solution and Refinement	
Structure Solution	Direct Methods (SHELXS86)
Refinement	Full-matrix least-squares
Function Minimized	$\Sigma w(\|Fo\|-\|Fc\|)^2$
Least Squares Weights	$w = 1/[\sigma^2(Fo)]$ $= [\sigma_c^2(Fo) + (p^2/4)Fo^2]^{-1}$
p-factor	0.0070
Anomalous Dispersion	All non-hydrogen atoms
No. Observations ($I > 3.00\sigma(I)$)	2938
No. Variables	298
Reflection/Parameter Ratio	9.86
Residuals: R; Rw	0.050; 0.059
Goodness of Fit Indicator	2.55
Max Shift/Error in Final Cycle	0.04
Maximum peak in Final Diff. Map	1.60 e–/Å^3 Minimum peak in Final Diff. Map	–3.40 e–/Å^3

Table 1. Atomic coordinates and Biso/Beq

atom	x	y	z	Beq
Ir(1)	0.53798(4)	0.19479(3)	0.30655(6)	2.335(8)
S(1)	0.3633(2)	0.2935(2)	0.2372(4)	2.73(7)
O(1)	0.3431(8)	0.2475(5)	0.138(1)	3.6(2)
O(2)	0.2876(7)	0.3039(6)	0.345(1)	3.9(2)
O(3)	0.437(1)	0.1514(8)	0.641(1)	6.9(4)
O(4)	0.404(1)	0.0735(8)	0.511(2)	7.9(5)
O(5)	0.2560	0.0363	0.7869	9.8424
O(6)	0.2093	0.1102	0.8599	9.4630
N(1)	0.5858(10)	0.2279(6)	0.507(1)	2.8(3)
N(2)	0.4727(8)	0.2836(5)	0.305(1)	2.4(2)
N(3)	0.414(1)	0.1293(9)	0.529(2)	5.6(5)
N(4)	0.2287	0.0694	0.8706	3.3896
C(1)	0.5910(10)	0.2950(6)	0.501(1)	2.4(3)
C(2)	0.492(1)	0.3159(7)	0.442(1)	2.7(3)
C(3)	0.496(1)	0.3844(8)	0.426(2)	3.9(4)
C(4)	0.526(1)	0.4137(8)	0.567(2)	4.1(4)
C(5)	0.622(1)	0.3893(9)	0.629(2)	4.1(4)
C(6)	0.614(1)	0.3199(8)	0.646(2)	3.3(3)
C(7)	0.683(1)	0.1923(6)	0.194(2)	3.0(2)
C(8)	0.604(1)	0.1987(9)	0.090(2)	3.6(3)
C(9)	0.537(2)	0.1509(9)	0.104(2)	4.2(4)
C(10)	0.578(1)	0.1096(6)	0.208(2)	3.3(3)
C(11)	0.665(1)	0.1378(8)	0.268(1)	3.2(3)
C(12)	0.775(1)	0.2287(8)	0.216(2)	4.6(4)
C(13)	0.596(2)	0.247(1)	-0.017(2)	5.0(5)
C(14)	0.451(2)	0.136(1)	0.001(2)	5.6(5)
C(15)	0.533(2)	0.0489(7)	0.241(2)	5.2(5)
C(16)	0.735(1)	0.111(1)	0.373(2)	4.9(5)
C(17)	0.366(1)	0.3594(7)	0.136(2)	3.2(3)
C(18)	0.457(1)	0.3798(7)	0.066(2)	3.7(3)
C(19)	0.453(2)	0.4253(8)	-0.023(2)	4.5(4)
C(20)	0.364(2)	0.4559(9)	-0.052(2)	4.3(4)
C(21)	0.275(2)	0.4368(8)	0.013(2)	4.0(4)
C(22)	0.278(1)	0.3894(8)	0.097(2)	4.1(4)
C(23)	0.367(2)	0.5094(9)	-0.155(2)	5.5(5)
C(24)	0.399(1)	0.1652(8)	0.404(2)	4.0(4)
C(25)	0.2633	0.0383	0.9890	10.9763
H(1)	0.6438	0.3059	0.4371	2.8424
H(2)	0.4397	0.3057	0.5076	3.2354
H(3)	0.5438	0.3947	0.3548	4.6636
H(4)	0.4307	0.3987	0.3990	4.6636
H(5)	0.5354	0.4549	0.5511	4.7930
H(6)	0.4735	0.4075	0.6339	4.7930
H(7)	0.6332	0.4069	0.7197	4.9518
H(8)	0.6771	0.3987	0.5676	4.9518
H(9)	0.5603	0.3102	0.7096	3.9967
H(10)	0.6755	0.3042	0.6804	3.9967

Table 1. Atomic coordinates and Biso/Beq (continued)

H(11)	0.5386	0.2158	0.5795	3.3686
H(12)	0.6507	0.2117	0.5308	3.3686
H(13)	0.7787	0.2402	0.3128	5.5690
H(14)	0.8327	0.2060	0.1912	5.5690
H(15)	0.7715	0.2631	0.1572	5.5690
H(16)	0.6500	0.2735	-0.0058	5.9200
H(17)	0.5331	0.2663	-0.0058	5.9200
H(18)	0.5988	0.2291	-0.1098	5.9200
H(19)	0.4053	0.1686	-0.0033	6.6368
H(20)	0.4782	0.1287	-0.0907	6.6368
H(21)	0.4163	0.1018	0.0341	6.6368
H(22)	0.4660	0.0535	0.2748	6.3106
H(23)	0.5732	0.0297	0.3112	6.3106
H(24)	0.5326	0.0254	0.1565	6.3106
H(25)	0.7645	0.0758	0.3323	5.8704
H(26)	0.6984	0.1001	0.4558	5.8704
H(27)	0.7867	0.1381	0.3959	5.8704
H(28)	0.5194	0.3606	0.0844	4.4482
H(29)	0.5134	0.4377	-0.0692	5.3424
H(30)	0.2133	0.4577	-0.0012	4.8760
H(31)	0.2148	0.3748	0.1308	4.9295
H(32)	0.4113	0.5388	-0.1168	6.4426
H(33)	0.3013	0.5254	-0.1652	6.4426
H(34)	0.3919	0.4967	-0.2448	6.4426
H(35)	0.3605	0.1991	0.4303	4.8961
H(36)	0.3621	0.1425	0.3359	4.8961
H(37)	0.2322	0.0002	0.9917	13.1716
H(38)	0.3348	0.0337	0.9830	13.1716
H(39)	0.2467	0.0596	1.0732	13.1716

$Beq = 8/3\ PI^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos GAMMA + 2U_{13}(aa^*cc^*)\cos BETA + 2U_{23}(bb^*cc^*)\cos ALPHA)$

Table 2. Anisotropic Displacement Parameters

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ir(1)	0.0254(2)	0.0386(2)	0.0247(2)	0.0003(3)	-0.0005(2)	0.0026(3)
S(1)	0.023(1)	0.048(2)	0.033(2)	0.004(1)	-0.006(1)	0.000(2)
O(1)	0.042(6)	0.044(7)	0.050(6)	-0.006(5)	-0.031(5)	0.000(5)
O(2)	0.034(5)	0.054(6)	0.059(7)	0.001(6)	0.009(5)	0.008(7)
O(3)	0.09(1)	0.13(1)	0.043(7)	-0.01(1)	0.004(8)	0.028(9)
O(4)	0.10(1)	0.08(1)	0.12(1)	-0.010(10)	0.03(1)	0.05(1)
O(5)	0.1247	0.1247	0.1247	0.0000	0.0000	0.0000
O(6)	0.1199	0.1199	0.1199	0.0000	0.0000	0.0000
N(1)	0.039(7)	0.042(8)	0.025(6)	0.012(6)	-0.005(5)	0.000(5)
N(2)	0.026(5)	0.043(6)	0.023(4)	0.005(5)	-0.001(6)	0.000(5)
N(3)	0.06(1)	0.08(1)	0.08(1)	0.004(9)	0.017(10)	0.04(1)
N(4)	0.0429	0.0429	0.0429	0.0000	0.0000	0.0000
C(1)	0.025(6)	0.030(8)	0.036(7)	0.008(6)	0.001(5)	0.001(6)
C(2)	0.033(7)	0.043(9)	0.026(6)	-0.003(6)	0.000(5)	-0.003(6)
C(3)	0.046(9)	0.05(1)	0.051(10)	-0.007(8)	0.002(8)	-0.002(9)
C(4)	0.05(1)	0.046(9)	0.054(9)	0.005(9)	0.003(10)	-0.009(8)
C(5)	0.06(1)	0.06(1)	0.041(9)	-0.007(9)	-0.008(9)	-0.017(9)
C(6)	0.029(7)	0.06(1)	0.040(8)	0.003(7)	0.003(6)	-0.004(7)
C(7)	0.051(7)	0.016(5)	0.045(7)	0.011(6)	0.027(7)	0.001(8)
C(8)	0.040(8)	0.06(1)	0.036(7)	0.021(9)	0.010(7)	0.007(9)
C(9)	0.051(9)	0.08(1)	0.027(7)	0.02(1)	-0.002(9)	0.004(8)
C(10)	0.050(8)	0.032(7)	0.042(8)	-0.012(6)	0.015(8)	-0.015(7)
C(11)	0.039(8)	0.06(1)	0.023(7)	-0.003(8)	0.003(6)	0.006(7)
C(12)	0.045(9)	0.05(1)	0.08(1)	-0.017(8)	0.00(1)	-0.01(1)
C(13)	0.06(1)	0.09(1)	0.038(9)	0.02(1)	0.014(9)	0.012(10)
C(14)	0.06(1)	0.09(2)	0.06(1)	-0.01(1)	0.00(1)	-0.02(1)
C(15)	0.09(1)	0.029(8)	0.08(1)	0.01(1)	0.01(1)	-0.004(8)
C(16)	0.05(1)	0.09(2)	0.038(9)	0.03(1)	-0.011(8)	-0.01(1)
C(17)	0.031(8)	0.039(9)	0.051(9)	0.003(7)	-0.003(7)	0.000(8)
C(18)	0.044(8)	0.050(9)	0.046(8)	0.002(9)	0.005(9)	-0.002(7)
C(19)	0.09(1)	0.042(9)	0.042(8)	0.00(1)	0.00(1)	-0.001(7)
C(20)	0.08(1)	0.06(1)	0.032(8)	0.018(10)	-0.007(9)	-0.012(8)
C(21)	0.07(1)	0.040(10)	0.040(9)	0.023(9)	-0.021(9)	-0.009(8)
C(22)	0.040(9)	0.05(1)	0.06(1)	0.002(8)	-0.006(9)	-0.029(9)
C(23)	0.11(2)	0.06(1)	0.040(10)	0.03(1)	-0.01(1)	-0.004(9)
C(24)	0.031(8)	0.06(1)	0.07(1)	-0.003(8)	0.000(8)	0.026(9)
C(25)	0.1390	0.1390	0.1390	0.0000	0.0000	0.0000

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)

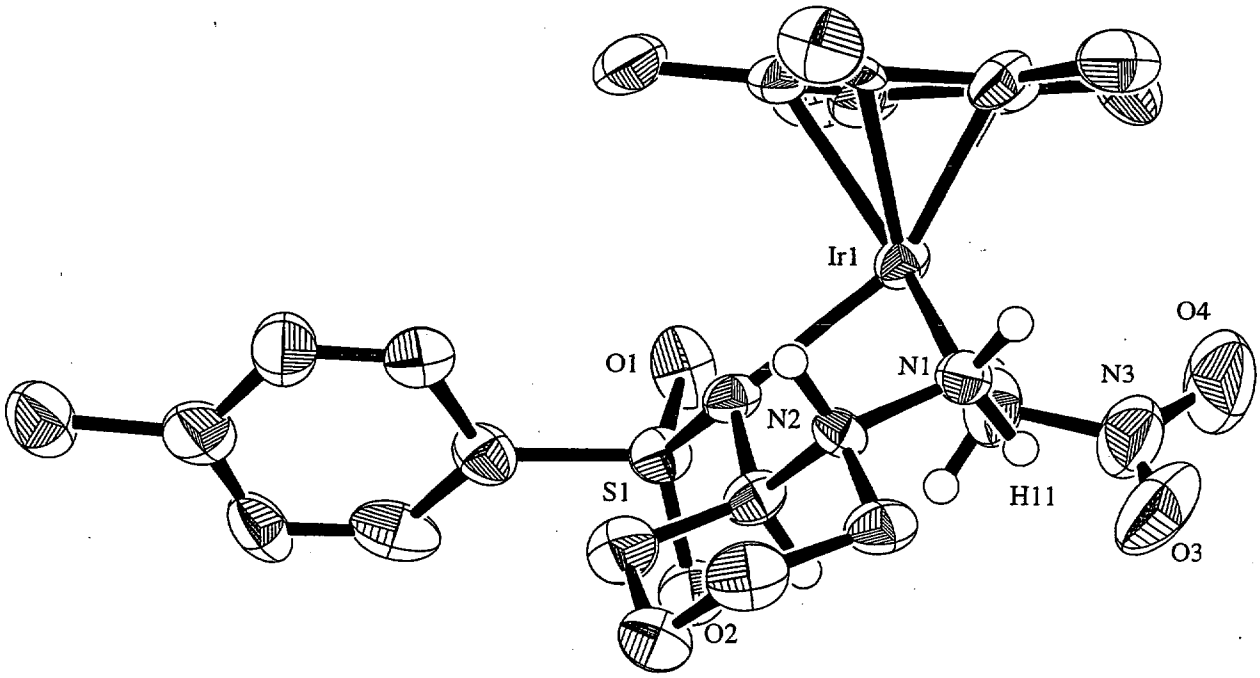
atom	atom	distance	atom	atom	distance
IR1	N1	2.12(1)	IR1	N2	2.18(1)
IR1	C7	2.18(1)	IR1	C8	2.21(1)
IR1	C9	2.14(2)	IR1	C10	2.19(1)
IR1	C11	2.14(2)	IR1	C24	2.16(2)
S1	O1	1.42(1)	S1	O2	1.44(1)
S1	N2	1.59(1)	S1	C17	1.76(2)
O3	N3	1.20(2)	O4	N3	1.27(2)
O5	N4	1.1423(1)	O6	N4	0.96
N1	C1	1.51(2)	N1	H11	0.96
N1	H12	0.96	N2	C2	1.50(2)
N3	C24	1.43(2)	N4	C25	1.3918(1)
C1	C2	1.50(2)	C1	C6	1.50(2)
C1	H1	0.95	C2	C3	1.55(2)
C2	H2	0.95	C3	C4	1.53(2)
C3	H3	0.95	C3	H4	0.95
C4	C5	1.49(3)	C4	H5	0.95
C4	H6	0.95	C5	C6	1.57(3)
C5	H7	0.95	C5	H8	0.95
C6	H9	0.95	C6	H10	0.95
C7	C8	1.43(2)	C7	C11	1.43(2)
C7	C12	1.48(2)	C8	C9	1.40(3)
C8	C13	1.48(3)	C9	C10	1.45(2)
C9	C14	1.53(3)	C10	C11	1.42(2)
C10	C15	1.52(2)	C11	C16	1.49(2)
C12	H13	0.95	C12	H14	0.95
C12	H15	0.95	C13	H16	0.94
C13	H17	0.95	C13	H18	0.96
C14	H19	0.95	C14	H20	0.95
C14	H21	0.95	C15	H22	0.95
C15	H23	0.95	C15	H24	0.95
C16	H25	0.95	C16	H26	0.95
C16	H27	0.95	C17	C18	1.44(2)
C17	C22	1.40(2)	C18	C19	1.32(2)
C18	H28	0.95	C19	C20	1.39(3)
C19	H29	0.95	C20	C21	1.39(3)
C20	C23	1.54(3)	C21	C22	1.32(3)
C21	H30	0.95	C22	H31	0.95
C23	H32	0.95	C23	H33	0.95
C23	H34	0.95	C24	H35	0.95
C24	H36	0.95	C25	H37	0.95
C25	H38	0.95	C25	H39	0.95

Table 4. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
N1	IR1	N2	78.6(5)	N1	IR1	C7	100.2(6)
N1	IR1	C8	133.2(6)	N1	IR1	C9	162.0(6)
N1	IR1	C10	127.5(6)	N1	IR1	C11	97.4(5)
N1	IR1	C24	89.2(6)	N2	IR1	C7	111.5(5)
N2	IR1	C8	96.5(6)	N2	IR1	C9	114.6(6)
N2	IR1	C10	153.5(6)	N2	IR1	C11	149.2(5)
N2	IR1	C24	87.1(6)	C7	IR1	C8	38.0(6)
C7	IR1	C9	64.1(7)	C7	IR1	C10	64.0(5)
C7	IR1	C11	38.5(5)	C7	IR1	C24	160.4(6)
C8	IR1	C9	37.4(7)	C8	IR1	C10	63.3(7)
C8	IR1	C11	63.8(6)	C8	IR1	C24	137.5(7)
C9	IR1	C10	39.0(6)	C9	IR1	C11	64.9(6)
C9	IR1	C24	103.2(8)	C10	IR1	C11	38.3(6)
C10	IR1	C24	96.7(7)	C11	IR1	C24	123.6(6)
O1	S1	O2	116.6(7)	O1	S1	N2	109.3(6)
O1	S1	C17	105.5(7)	O2	S1	N2	111.8(6)
O2	S1	C17	104.7(8)	N2	S1	C17	108.3(7)
IR1	N1	C1	109.3(8)	IR1	N1	H11	109.6
IR1	N1	H12	109.9	C1	N1	H11	109.7
C1	N1	H12	110.3	H11	N1	H12	107.9
IR1	N2	S1	119.3(6)	IR1	N2	C2	111.9(8)
S1	N2	C2	115.3(9)	O3	N3	O4	123(1)
O3	N3	C24	121(1)	O4	N3	C24	115(2)
O5	N4	O6	129.691(3)	O5	N4	C25	96.681(10)
O6	N4	C25	130.863(3)	N1	C1	C2	106(1)
N1	C1	C6	110(1)	N1	C1	H1	108.3
C2	C1	C6	113(1)	C2	C1	H1	109.0
C6	C1	H1	109.1	N2	C2	C1	108(1)
N2	C2	C3	113(1)	N2	C2	H2	108.6
C1	C2	C3	108(1)	C1	C2	H2	108.4
C3	C2	H2	109.0	C2	C3	C4	110(1)
C2	C3	H3	109.4	C2	C3	H4	109.4
C4	C3	H3	109.0	C4	C3	H4	108.9
H3	C3	H4	109.4	C3	C4	C5	113(1)
C3	C4	H5	108.6	C3	C4	H6	108.2
C5	C4	H5	108.5	C5	C4	H6	107.9
H5	C4	H6	109.9	C4	C5	C6	110(1)
C4	C5	H7	109.2	C4	C5	H8	108.9
C6	C5	H7	109.8	C6	C5	H8	109.4
H7	C5	H8	109.4	C1	C6	C5	107(1)
C1	C6	H9	109.7	C1	C6	H10	110.2
C5	C6	H9	109.9	C5	C6	H10	110.2
H9	C6	H10	109.5	IR1	C7	C8	71.9(8)
IR1	C7	C11	69.0(8)	IR1	C7	C12	129(1)
C8	C7	C11	107(1)	C8	C7	C12	129(1)
C11	C7	C12	122(1)	IR1	C8	C7	70.1(8)
IR1	C8	C9	68.6(9)	IR1	C8	C13	128(1)

Table 4. Bond Angles(°) (continued)

C7	C8	C9	108(1)	C7	C8	C13	125(1)
C9	C8	C13	125(1)	IR1	C9	C8	74(1)
IR1	C9	C10	72.5(9)	IR1	C9	C14	131(1)
C8	C9	C10	108(1)	C8	C9	C14	125(1)
C10	C9	C14	124(1)	IR1	C10	C9	68.4(9)
IR1	C10	C11	68.8(9)	IR1	C10	C15	127(1)
C9	C10	C11	106(1)	C9	C10	C15	124(1)
C11	C10	C15	129(1)	IR1	C11	C7	72.4(9)
IR1	C11	C10	72.9(9)	IR1	C11	C16	128(1)
C7	C11	C10	109(1)	C7	C11	C16	124(1)
C10	C11	C16	125(1)	C7	C12	H13	109.4
C7	C12	H14	109.2	C7	C12	H15	109.3
H13	C12	H14	109.9	H13	C12	H15	109.6
H14	C12	H15	109.3	C8	C13	H16	109.8
C8	C13	H17	108.7	C8	C13	H18	108.3
H16	C13	H17	110.7	H16	C13	H18	110.1
H17	C13	H18	109.1	C9	C14	H19	109.6
C9	C14	H20	109.7	C9	C14	H21	109.4
H19	C14	H20	109.4	H19	C14	H21	109.3
H20	C14	H21	109.4	C10	C15	H22	109.6
C10	C15	H23	109.3	C10	C15	H24	109.4
H22	C15	H23	109.6	H22	C15	H24	109.6
H23	C15	H24	109.2	C11	C16	H25	109.4
C11	C16	H26	109.4	C11	C16	H27	109.3
H25	C16	H26	109.5	H25	C16	H27	109.5
H26	C16	H27	109.8	S1	C17	C18	122(1)
S1	C17	C22	122(1)	C18	C17	C22	114(1)
C17	C18	C19	120(1)	C17	C18	H28	119.8
C19	C18	H28	119.8	C18	C19	C20	122(2)
C18	C19	H29	119.0	C20	C19	H29	118.4
C19	C20	C21	118(1)	C19	C20	C23	118(1)
C21	C20	C23	122(1)	C20	C21	C22	119(1)
C20	C21	H30	120.3	C22	C21	H30	120.5
C17	C22	C21	124(1)	C17	C22	H31	118.2
C21	C22	H31	117.2	C20	C23	H32	109.1
C20	C23	H33	109.3	C20	C23	H34	109.2
H32	C23	H33	109.6	H32	C23	H34	109.7
H33	C23	H34	109.9	IR1	C24	N3	113(1)
IR1	C24	H35	108.3	IR1	C24	H36	108.2
N3	C24	H35	108.5	N3	C24	H36	108.6
H35	C24	H36	109.7	N4	C25	H37	109.5
N4	C25	H38	109.5	N4	C25	H39	109.5
H37	C25	H38	109.5	H37	C25	H39	109.5
H38	C25	H39	109.5				



$(\eta^5\text{-}C_5(CH_3)_5)Ir(CH_2NO_2)$[(1*R*,2*R*)-*N*-*p*-toluenesulfonyl-1,2-cyclohexanediamine] (1b)

3) Single-crystal X-ray analysis data of (η^5-$C_5(CH_3)_5$)Ir(CH_2COCH_3)[(1*R*,2*R*)-*N*-*p*-toluenesulfonyl-1,2-cyclohexanediamine] (1c)

Experimental

Data Collection

An yellow prismatic crystal of $C_{26}H_{39}N_2O_3SIr \cdot C_6H_5CH_3$ having approximate dimensions of 0.20 x 0.20 x 0.20 mm was mounted on a glass fiber. All measurements were made on a Rigaku AFC7R diffractometer with graphite monochromated Mo-Kα 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 11 carefully centered reflections in the range $29.56 < 2\theta < 29.94°$ corresponded to a C-centered monoclinic cell with dimensions:

a = 24.426(8) Å
b = 13.461(9) Å β = 102.71(5)°
c = 9.418(6) Å
V = 3020(2) Å^3

For Z = 4 and F.W. = 744.03, the calculated density is 1.64 g/cm^3. The systematic absences of:

hkl: h+k # 2n

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

C2 (#5)

The data were collected at a temperature of –20 ± 1 °C using the ω–2θ scan technique to a maximum 2θ value of 55.1°. Omega scans of several intense reflections, made prior to data collection, had an average width at half-height of 0.63° with a take-off angle of 6.0°. Scans of (1.63 + 0.30 tan θ)° were made at a speed of 16.0°/min (in ω). The weak reflections ($I < 10.0\sigma(I)$) were rescanned (maximum of 5 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 and the crystal to detector distance was 235 mm. The computer-controlled slits were set to 9.0 mm (horizontal) and 13.0 mm (vertical).

Data Reduction

Of the 3700 reflections which were collected, 3617 were unique (*R*int = 0.111). The intensities of three representative reflection were measured after every 150 reflections. No decay correction was applied.

The linear absorption coefficient, μ, for Mo-Kα radiation is 45.3 cm^{-1}. An emoirical absoption correction based on azimuthal scans of several reflections was applied which resulted in transmission factors ranging from 0.58 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. Hydrogen atoms were included but not refined. The final cycle of full-matrix least-squares refinement[3] was based on 2141 observed reflections ($I > 2.00\sigma(I)$) and 311 variable parameters and converged (largest parameter shift was 1.04 times its esd) with unweighted and weighted agreement factors of:

$R = \Sigma||Fo|-|Fc||/\Sigma|Fo| = 0.052$

$Rw = [(\Sigma w(|Fo|-|Fc|)^2 / \Sigma\, wFo^2)]^{1/2} = 0.056$

The standard deviation of an observation of unit weight[4] was 1.43. The weighting scheme was based on counting statistics and included a factor (p = 0.050) to downweight the intense reflections. Plots of $\Sigma w(|Fo|-|Fc|)^2$ versus |Fo|, reflection order in data collection, sin θ/λ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 1.47 and –0.98 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber.[5] The values for the mass attenuation coefficients are those of Creagh and Hubbel.[6] All calculations were performed using the teXsan[7] crystallographic software package of Molecular Structure Corporation.

References

(1) SHELXS86: Sheldrick, G. M. In *Crystallographic Computing 3* ; Sheldrick, G. M.; Kruger, C.; Goddard, R. Eds.; Oxford University Press, 1985, pp 175-189.

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

(3) Least-Squares:

Function minimized: $\Sigma w(|Fo|-|Fc|)^2$

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

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

p = p-factor

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

$[\Sigma w(|Fo|-|Fc|)^2/(No-Nv)]^{1/2}$

where: No = number of observations

Nv = number of variables

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

(6) Creagh, D. C.; McAuley, W. J. *International Tables for Crystallography,Vol C*; Wilson, A. J. C. Ed.; Kluwer Academic Publishers: Boston, 1992, Table 4.2.6.8, pp 219–222 .

(7) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation, 1985 & 1992.

EXPERIMENTAL DETAILS

A. Crystal Data	
Empirical Formula	$C_{26}H_{39}N_2O_3SIr \cdot C_6H_5CH_3$
Formula Weight	744.03
Crystal Color, Habit	yellow, prismatic
Crystal Dimensions	0.20 x 0.20 x 0.20 mm
Crystal System	monoclinic
Lattice Type	C-centered
No. of Reflections Used for Unit	
Cell Determination (2θ range)	11 (29.6–29.9°)
Omega Scan Peak Width at Half-height	0.63°
Lattice Parameters	a = 24.426(8)Å
	b = 13.461(9) Å
	c = 9.418 (6) Å
	β = 102.71(5)°
	V = 3020(2) Å^3
Space Group	C2(#5)
Z value	4
D_{calc}	1.636 g/cm^3
F_{000}	1420.00
μ(MoKα)	45.33cm^{-1}
B. Intensity Measurements	
Diffractometer	Rigaku AFC7R
Radiation	MoKα (λ= 0.71069 Å)
	graphite monochromated
Attenuator	Zr foil (factor = 7.00)
Take-off Angle	6.0°
Detector Aperture	9.0 mm horizontal
	13.0 mm vertical
Crystal to Detector Distance	235 mm
Temperature	–20.0 °C
Scan Type	ω–2θ
Scan Rate	16.0°/min (in ω) (up to 5 scans)
Scan Width	(1.63 + 0.30 tan θ)°
2θ*max*	55.1°
No. of Reflections Measured	Total: 3700

	Unique: 3617 (R*int* = 0.111)
Corrections	Lorentz-polarization
	Absorption
	(trans. Factors: 0.5831–0.9978)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SHELXS86)
Refinement	Full-matrix least-squares
Function Minimized	$\Sigma w(\|Fo\|-\|Fc\|)^2$
Least Squares Weights	$w = 1/[\sigma^2(Fo)]$
	$= [\sigma_c^2(Fo) + (p^2/4)Fo^2]^{-1}$
p-factor	0.0500
No. Observations ($I > 2.00\sigma(I)$)	2141
No. Variables	311
Reflection/Parameter Ratio	6.88
Residuals: R; Rw	0.052; 0.056
Goodness of Fit Indicator	1.43
Max Shift/Error in Final Cycle	1.04
Maximum peak in Final Diff. Map	1.47 e–/Å^3
Minimum peak in Final Diff. Map	–0.98 e–/Å^3

Table 1. Atomic coordinates, B*iso*/B*eq*

atom	x	y	z	B*eq*
Ir(1)	0.70210(3)	0.504(3)	0.65376(6)	4.33(1)
S(1)	0.7886(2)	0.323(3)	0.7549(5)	4.3(1)
O(1)	0.7977(6)	0.257(4)	0.642(1)	6.0(4)
O(2)	0.7435(6)	0.303(4)	0.823(2)	4.8(4)
O(3)	0.6782(9)	0.451(4)	0.276(2)	11.0(7)
N(1)	0.7383(7)	0.566(4)	0.490(2)	5.1(4)
N(2)	0.7836(6)	0.436(4)	0.701(1)	3.6(2)
C(1)	0.803(1)	0.560(4)	0.530(2)	5.0(4)
C(2)	0.8153(10)	0.460(4)	0.589(2)	4.9(5)
C(3)	0.8804(10)	0.457(4)	0.635(3)	7.0(5)
C(4)	0.911(1)	0.487(5)	0.515(4)	11(1)
C(5)	0.891(2)	0.583(4)	0.461(4)	9.6(9)
C(6)	0.826(1)	0.589(4)	0.407(2)	6.7(6)
C(7)	0.8504(8)	0.308(4)	0.895(2)	5.0(4)
C(8)	0.8735(10)	0.393(4)	0.979(2)	5.9(5)
C(9)	0.922(1)	0.373(4)	1.088(3)	7.6(6)
C(10)	0.9455(10)	0.283(4)	1.116(2)	7.1(6)
C(11)	0.921(1)	0.207(4)	1.037(3)	7.1(6)
C(12)	0.8739(9)	0.217(4)	0.917(2)	5.0(4)
C(13)	0.9962(9)	0.272(5)	1.236(3)	9.8(9)
C(14)	0.6433(9)	0.623(4)	0.671(3)	6.1(5)
C(15)	0.6261(9)	0.525(4)	0.719(2)	6.0(5)
C(16)	0.660(1)	0.500(5)	0.838(2)	7.0(5)
C(17)	0.707(1)	0.565(4)	0.871(2)	6.8(5)
C(18)	0.6963(9)	0.649(4)	0.770(2)	5.7(4)
C(19)	0.611(1)	0.686(4)	0.561(2)	7.7(7)
C(20)	0.569(1)	0.491(6)	0.673(3)	13(1)
C(21)	0.657(2)	0.409(4)	0.943(4)	15(1)
C(22)	0.757(1)	0.567(5)	0.998(2)	12.3(10)
C(23)	0.724(1)	0.742(4)	0.761(3)	7.4(8)
C(24)	0.6725(8)	0.382(4)	0.496(2)	5.5(5)
C(25)	0.650(1)	0.427(4)	0.367(3)	8.8(7)
C(26)	0.588(2)	0.445(5)	0.318(3)	12(1)
H(1)	0.8157	0.6036	0.6061	5.9956
H(2)	0.8039	0.4118	0.5113	5.9346
H(3)	0.8920	0.4981	0.7146	8.3770
H(4)	0.8913	0.3884	0.6635	8.3770
H(5)	0.9497	0.4870	0.5536	14.0212
H(6)	0.9024	0.4377	0.4378	14.0212
H(7)	0.9076	0.5975	0.3818	11.5540
H(8)	0.9021	0.6284	0.5367	11.5540
H(9)	0.8135	0.5421	0.3278	8.0142
H(10)	0.8143	0.6521	0.3771	8.0142
H(11)	0.7250	0.5286	0.4017	6.1205
H(12)	0.7276	0.6316	0.4769	6.1205
H(13)	0.8575	0.4556	0.9632	7.0667
H(14)	0.9391	0.4246	1.1448	9.2150
H(15)	0.9351	0.1397	1.0621	8.4792

Table 1. Atomic coordinates, B*iso*/*Beq* (continued)

atom	x	y	z	B*eq*
H(16)	0.8600	0.1603	0.8544	5.8531
H(17)	1.0254	0.3113	1.2183	11.7155
H(18)	1.0081	0.2029	1.2432	11.7155
H(19)	0.9871	0.2895	1.3261	11.7155
H(20)	0.6338	0.7036	0.4959	9.1366
H(21)	0.5794	0.6488	0.5099	9.1366
H(22)	0.5997	0.7408	0.6063	9.1366
H(23)	0.5515	0.5214	0.5855	16.1424
H(24)	0.5685	0.4196	0.6593	16.1424
H(25)	0.5493	0.5053	0.7472	16.1424
H(26)	0.6245	0.3683	0.9021	18.5800
H(27)	0.6545	0.4303	1.0355	18.5800
H(28)	0.6894	0.3665	0.9504	18.5800
H(29)	0.7535	0.6212	1.0574	14.7139
H(30)	0.7575	0.5062	1.0528	14.7139
H(31)	0.7899	0.5708	0.9621	14.7139
H(32)	0.7574	0.7430	0.8368	8.8013
H(33)	0.7340	0.7452	0.6697	8.8013
H(34)	0.7001	0.7933	0.7731	8.8013
H(35)	0.6449	0.3414	0.5285	6.5987
H(36)	0.7030	0.3389	0.4875	6.5987
H(37)	0.5738	0.4063	0.2316	15.4874
H(38)	0.5811	0.5109	0.3025	15.4874
H(39)	0.5697	0.4197	0.3925	15.4874

$$Beq = 8/3\pi^2(U_{11}aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}aa^*bb^*\cos\gamma + 2U_{13}aa^*cc^*\cos\beta + 2U_{23}bb^*cc^*\cos\alpha)$$

Table 2. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ir(1)	0.0675(4)	0.0535(3)	0.0423(3)	0.0143(9)	0.0093(2)	0.0077(8)
S(1)	0.062(3)	0.039(2)	0.057(3)	-0.001(2)	0.001(2)	0.002(2)
O(1)	0.11(1)	0.049(8)	0.062(8)	0.009(8)	0.000(7)	-0.017(5)
O(2)	0.055(8)	0.047(9)	0.07(1)	-0.006(7)	-0.003(6)	0.012(7)
O(3)	0.16(2)	0.18(2)	0.06(1)	0.01(2)	0.01(1)	0.01(1)
N(1)	0.092(10)	0.07(1)	0.041(7)	0.021(9)	0.034(7)	0.002(7)
N(2)	0.060(5)	0.035(4)	0.037(6)	-0.008(5)	-0.001(5)	-0.014(5)
C(1)	0.092(10)	0.048(7)	0.05(1)	0.01(1)	0.008(10)	-0.023(8)
C(2)	0.08(1)	0.07(1)	0.04(1)	0.01(1)	0.013(8)	0.03(1)
C(3)	0.08(1)	0.07(1)	0.11(2)	0.02(1)	-0.01(1)	0.00(1)
C(4)	0.13(2)	0.16(4)	0.19(3)	0.08(3)	0.12(2)	0.13(3)
C(5)	0.15(2)	0.11(2)	0.13(3)	-0.01(2)	0.08(2)	0.03(2)
C(6)	0.16(2)	0.05(1)	0.07(1)	0.04(1)	0.07(1)	0.029(10)
C(7)	0.06(1)	0.08(1)	0.047(9)	0.007(9)	0.009(5)	0.010(8)
C(8)	0.08(1)	0.08(1)	0.06(1)	0.00(1)	0.005(9)	-0.001(10)
C(9)	0.06(1)	0.15(2)	0.08(1)	-0.01(1)	0.003(9)	-0.03(2)
C(10)	0.06(1)	0.16(2)	0.05(1)	-0.02(1)	0.006(8)	0.03(1)
C(11)	0.07(2)	0.11(2)	0.08(1)	0.02(1)	-0.003(10)	0.06(1)
C(12)	0.07(1)	0.07(1)	0.054(10)	-0.021(9)	0.014(7)	0.009(10)
C(13)	0.04(1)	0.23(4)	0.09(2)	0.00(2)	-0.006(10)	0.04(2)
C(14)	0.08(1)	0.06(1)	0.10(1)	0.027(7)	0.010(9)	-0.001(10)
C(15)	0.082(8)	0.08(2)	0.08(1)	0.02(1)	0.056(9)	0.02(1)
C(16)	0.12(1)	0.11(1)	0.057(8)	0.03(2)	0.062(7)	0.00(1)
C(17)	0.11(1)	0.11(2)	0.043(6)	0.05(1)	0.013(9)	-0.005(7)
C(18)	0.08(1)	0.072(8)	0.07(1)	0.04(1)	0.022(9)	-0.026(6)
C(19)	0.12(2)	0.10(2)	0.06(1)	0.02(2)	0.00(1)	0.02(1)
C(20)	0.11(2)	0.25(5)	0.17(3)	-0.11(3)	0.07(2)	-0.05(4)
C(21)	0.36(6)	0.10(2)	0.18(3)	0.05(3)	0.15(4)	0.09(2)
C(22)	0.15(2)	0.25(4)	0.04(1)	0.10(3)	-0.03(1)	-0.07(2)
C(23)	0.10(2)	0.08(2)	0.09(2)	0.00(2)	0.02(2)	-0.04(2)
C(24)	0.06(1)	0.06(1)	0.08(1)	-0.02(1)	0.00(1)	-0.015(7)
C(25)	0.15(2)	0.11(2)	0.06(1)	-0.01(2)	-0.02(1)	-0.03(1)
C(26)	0.15(2)	0.23(5)	0.08(2)	0.04(3)	-0.02(2)	0.01(2)

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
Ir(1)	N(1)	2.11(3)	Ir(1)	N(2)	2.15(3)
Ir(1)	C(14)	2.18(5)	Ir(1)	C(15)	2.10(3)
Ir(1)	C(16)	2.20(2)	Ir(1)	C(17)	2.19(3)
Ir(1)	C(18)	2.26(6)	Ir(1)	C(24)	2.22(5)
S(1)	O(1)	1.44(4)	S(1)	O(2)	1.41(2)
S(1)	N(2)	1.60(6)	S(1)	C(7)	1.79(2)
O(3)	C(25)	1.26(4)	N(1)	C(1)	1.54(3)
N(1)	H(11)	0.964	N(1)	H(12)	0.925
N(2)	C(2)	1.47(3)	C(1)	C(2)	1.46(7)
C(1)	C(6)	1.45(4)	C(1)	H(1)	0.932
C(2)	C(3)	1.55(3)	C(2)	H(2)	0.969
C(3)	C(4)	1.54(5)	C(3)	H(3)	0.925
C(3)	H(4)	0.977	C(4)	C(5)	1.43(8)
C(4)	H(5)	0.943	C(4)	H(6)	0.975
C(5)	C(6)	1.56(5)	C(5)	H(7)	0.949
C(5)	H(8)	0.934	C(6)	H(9)	0.967
C(6)	H(10)	0.924	C(7)	C(8)	1.43(6)
C(7)	C(12)	1.36(7)	C(8)	C(9)	1.41(3)
C(8)	H(13)	0.928	C(9)	C(10)	1.34(7)
C(9)	H(14)	0.920	C(10)	C(11)	1.34(6)
C(10)	C(13)	1.49(3)	C(11)	C(12)	1.42(3)
C(11)	H(15)	0.979	C(12)	H(16)	0.975
C(13)	H(17)	0.931	C(13)	H(18)	0.976
C(13)	H(19)	0.948	C(14)	C(15)	1.48(7)
C(14)	C(18)	1.47(3)	C(14)	C(19)	1.43(5)
C(15)	C(16)	1.29(3)	C(15)	C(20)	1.45(5)
C(16)	C(17)	1.41(6)	C(16)	C(21)	1.59(8)
C(17)	C(18)	1.47(6)	C(17)	C(22)	1.51(3)
C(18)	C(23)	1.44(7)	C(19)	H(20)	0.943
C(19)	H(21)	0.963	C(19)	H(22)	0.923
C(20)	H(23)	0.932	C(20)	H(24)	0.974
C(20)	H(25)	0.943	C(21)	H(26)	0.977
C(21)	H(27)	0.935	C(21)	H(28)	0.956
C(22)	H(29)	0.935	C(22)	H(30)	0.962
C(22)	H(31)	0.946	C(23)	H(32)	0.948
C(23)	H(33)	0.944	C(23)	H(34)	0.930
C(24)	C(25)	1.36(4)	C(24)	H(35)	0.967
C(24)	H(36)	0.966	C(25)	C(26)	1.50(5)
C(26)	H(37)	0.963	C(26)	H(38)	0.910
C(26)	H(39)	0.967			

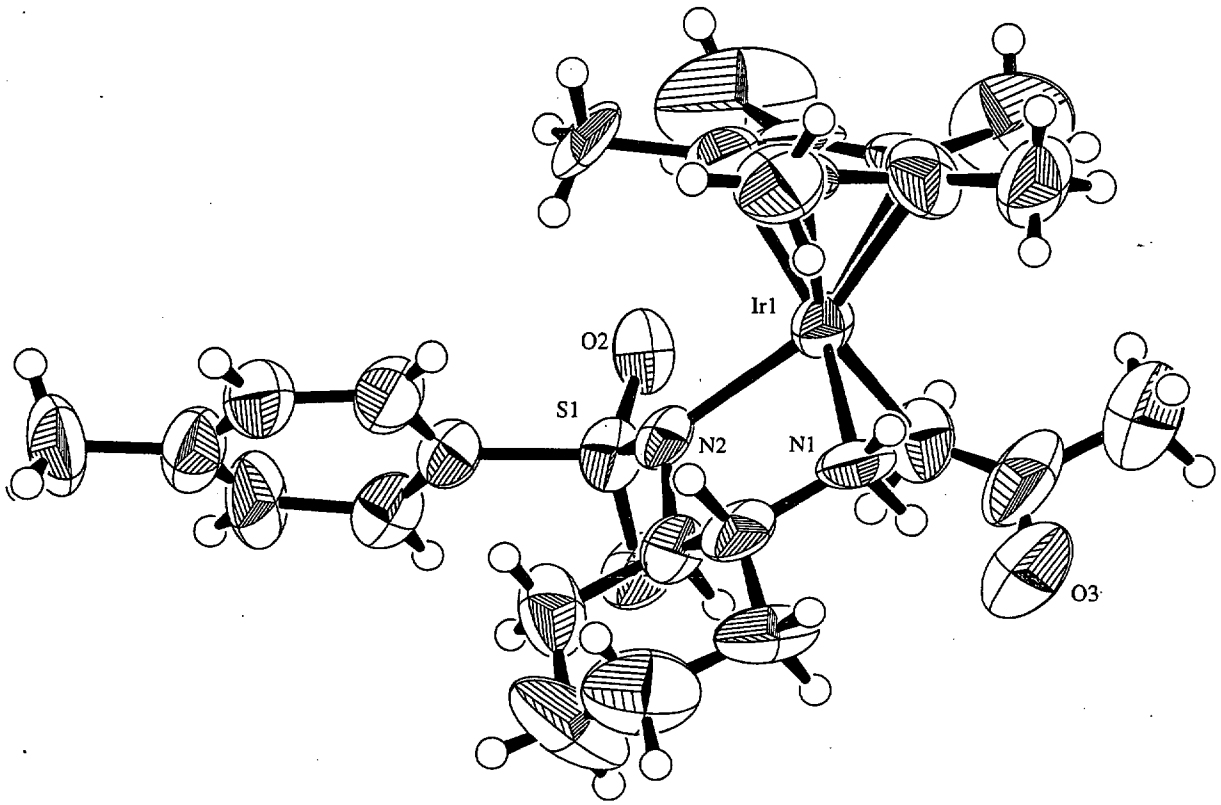
Table 4. Bond Angles(°)

atom	atom	atom	angle
N(1)	Ir(1)	N(2)	78(1)
N(1)	Ir(1)	C(15)	135(2)
N(1)	Ir(1)	C(17)	127(2)
N(1)	Ir(1)	C(24)	86(1)
N(2)	Ir(1)	C(15)	147(2)
N(2)	Ir(1)	C(17)	96(1)
N(2)	Ir(1)	C(24)	89(2)
C(14)	Ir(1)	C(16)	62(2)
C(14)	Ir(1)	C(18)	39(1)
C(15)	Ir(1)	C(16)	35(1)
C(15)	Ir(1)	C(18)	66(2)
C(16)	Ir(1)	C(17)	37(2)
C(16)	Ir(1)	C(24)	112(2)
C(17)	Ir(1)	C(24)	147(2)
O(1)	S(1)	O(2)	119(3)
O(1)	S(1)	C(7)	104(2)
O(2)	S(1)	C(7)	105(1)
Ir(1)	N(1)	C(1)	112(2)
Ir(1)	N(1)	H(12)	108.84
C(1)	N(1)	H(12)	109.14
Ir(1)	N(2)	S(1)	118(2)
S(1)	N(2)	C(2)	115(3)
N(1)	C(1)	C(6)	110(2)
C(2)	C(1)	C(6)	118(3)
C(6)	C(1)	H(1)	108.58
N(2)	C(2)	C(3)	118(2)
C(1)	C(2)	C(3)	104(3)
C(3)	C(2)	H(2)	107.47
C(2)	C(3)	H(3)	108.81
C(4)	C(3)	H(3)	108.85
H(3)	C(3)	H(4)	109.28
C(3)	C(4)	H(5)	108.96
C(5)	C(4)	H(5)	112.15
H(5)	C(4)	H(6)	107.98
C(4)	C(5)	H(7)	106.88
C(6)	C(5)	H(7)	108.83
H(7)	C(5)	H(8)	110.95
C(1)	C(6)	H(9)	109.49
C(5)	C(6)	H(9)	109.88
H(9)	C(6)	H(10)	110.18
S(1)	C(7)	C(12)	118(3)
C(7)	C(8)	C(9)	115(4)
C(9)	C(8)	H(13)	123.29
C(8)	C(9)	H(14)	118.61
C(9)	C(10)	C(11)	117(3)
C(11)	C(10)	C(13)	123(5)
C(10)	C(11)	H(15)	118.46
C(7)	C(12)	C(11)	116(4)

atom	atom	atom	angle
N(1)	Ir(1)	C(14)	99(2)
N(1)	Ir(1)	C(16)	158(3)
N(1)	Ir(1)	C(18)	96(2)
N(2)	Ir(1)	C(14)	153(2)
N(2)	Ir(1)	C(16)	113(1)
N(2)	Ir(1)	C(18)	115(1)
C(14)	Ir(1)	C(15)	41(2)
C(14)	Ir(1)	C(17)	64(1)
C(14)	Ir(1)	C(24)	118(1)
C(15)	Ir(1)	C(17)	63(1)
C(15)	Ir(1)	C(24)	96(2)
C(16)	Ir(1)	C(18)	63(2)
C(17)	Ir(1)	C(18)	39(2)
C(18)	Ir(1)	C(24)	156(1)
O(1)	S(1)	N(2)	111(2)
O(2)	S(1)	N(2)	108(3)
N(2)	S(1)	C(7)	109(2)
Ir(1)	N(1)	H(11)	108.13
C(1)	N(1)	H(11)	108.45
H(11)	N(1)	H(12)	110.39
Ir(1)	N(2)	C(2)	112(2)
N(1)	C(1)	C(2)	105(3)
N(1)	C(1)	H(1)	108.18
C(2)	C(1)	H(1)	106.37
N(2)	C(2)	C(1)	112(3)
N(2)	C(2)	H(2)	106.84
C(1)	C(2)	H(2)	108.69
C(2)	C(3)	C(4)	115(2)
C(2)	C(3)	H(4)	107.44
C(4)	C(3)	H(4)	107.85
C(3)	C(4)	C(5)	109(4)
C(3)	C(4)	H(6)	108.29
C(5)	C(4)	H(6)	110.61
C(4)	C(5)	C(6)	114(4)
C(4)	C(5)	H(8)	107.19
C(6)	C(5)	H(8)	109.30
C(1)	C(6)	C(5)	106(2)
C(1)	C(6)	H(10)	110.39
C(5)	C(6)	H(10)	111.01
S(1)	C(7)	C(8)	119(3)
C(8)	C(7)	C(12)	123(2)
C(7)	C(8)	H(13)	122.12
C(8)	C(9)	C(10)	125(4)
C(10)	C(9)	H(14)	116.43
C(9)	C(10)	C(13)	119(4)
C(10)	C(11)	C(12)	124(4)
C(12)	C(11)	H(15)	117.78
C(7)	C(12)	H(16)	122.62

Table 4. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C(11)	C(12)	H(16)	120.94	C(10)	C(13)	H(17)	110.36
C(10)	C(13)	H(18)	109.32	C(10)	C(13)	H(19)	109.39
H(17)	C(13)	H(18)	108.93	H(17)	C(13)	H(19)	111.31
H(18)	C(13)	H(19)	107.46	Ir(1)	C(14)	C(15)	67(2)
Ir(1)	C(14)	C(18)	74(2)	Ir(1)	C(14)	C(19)	131(2)
C(15)	C(14)	C(18)	107(3)	C(15)	C(14)	C(19)	127(3)
C(18)	C(14)	C(19)	126(4)	Ir(1)	C(15)	C(14)	73(2)
Ir(1)	C(15)	C(16)	77(2)	Ir(1)	C(15)	C(20)	137(3)
C(14)	C(15)	C(16)	109(4)	C(14)	C(15)	C(20)	121(4)
C(16)	C(15)	C(20)	125(4)	Ir(1)	C(16)	C(15)	68(1)
Ir(1)	C(16)	C(17)	71(2)	Ir(1)	C(16)	C(21)	127(4)
C(15)	C(16)	C(17)	112(5)	C(15)	C(16)	C(21)	129(4)
C(17)	C(16)	C(21)	119(3)	Ir(1)	C(17)	C(16)	72(2)
Ir(1)	C(17)	C(18)	73(2)	Ir(1)	C(17)	C(22)	128(3)
C(16)	C(17)	C(18)	108(3)	C(16)	C(17)	C(22)	131(4)
C(18)	C(17)	C(22)	120(4)	Ir(1)	C(18)	C(14)	68(2)
Ir(1)	C(18)	C(17)	68(2)	Ir(1)	C(18)	C(23)	130(2)
C(14)	C(18)	C(17)	103(3)	C(14)	C(18)	C(23)	123(3)
C(17)	C(18)	C(23)	134(2)	C(14)	C(19)	H(20)	108.23
C(14)	C(19)	H(21)	107.76	C(14)	C(19)	H(22)	108.67
H(20)	C(19)	H(21)	108.93	H(20)	C(19)	H(22)	112.47
H(21)	C(19)	H(22)	110.65	C(15)	C(20)	H(23)	110.01
C(15)	C(20)	H(24)	108.84	C(15)	C(20)	H(25)	109.26
H(23)	C(20)	H(24)	108.97	H(23)	C(20)	H(25)	111.67
H(24)	C(20)	H(25)	108.03	C(16)	C(21)	H(26)	109.40
C(16)	C(21)	H(27)	111.12	C(16)	C(21)	H(28)	110.83
H(26)	C(21)	H(27)	108.44	H(26)	C(21)	H(28)	106.70
H(27)	C(21)	H(28)	110.22	C(17)	C(22)	H(29)	109.05
C(17)	C(22)	H(30)	109.07	C(17)	C(22)	H(31)	108.94
H(29)	C(22)	H(30)	109.76	H(29)	C(22)	H(31)	111.14
H(30)	C(22)	H(31)	108.84	C(18)	C(23)	H(32)	107.62
C(18)	C(23)	H(33)	107.89	C(18)	C(23)	H(34)	107.95
H(32)	C(23)	H(33)	110.15	H(32)	C(23)	H(34)	111.32
H(33)	C(23)	H(34)	111.74	Ir(1)	C(24)	C(25)	106(4)
Ir(1)	C(24)	H(35)	111.02	Ir(1)	C(24)	H(36)	111.10
C(25)	C(24)	H(35)	110.64	C(25)	C(24)	H(36)	111.01
H(35)	C(24)	H(36)	106.76	O(3)	C(25)	C(24)	123(3)
O(3)	C(25)	C(26)	115(3)	C(24)	C(25)	C(26)	122(3)
C(25)	C(26)	H(37)	108.39	C(25)	C(26)	H(38)	109.96
C(25)	C(26)	H(39)	108.00	H(37)	C(26)	H(38)	111.82
H(37)	C(26)	H(39)	107.05	H(38)	C(26)	H(39)	111.47



(η^5-$C_5(CH_3)_5$)Ir(CH_2COCH_3)[(1*R*,2*R*)-*N*-*p*-toluenesulfonyl-1,2-cyclohexanediamine] (1c)

4) Single-crystal X-ray analysis data of (η^5-$C_5(CH_3)_5$)Ir(CCC_6H_5)[(1*R*,2*R*)-*N*-*p*-toluenesulfonyl-1,2-cyclohexanediamine] (1d)

Experimental

Data Collection

An yellow prismatic crystal of $C_{31}H_{39}N_2O_2SIr \cdot C_6H_5CH_3$ having approximate dimensions of 0.20 x 0.20 x 0.20 mm was mounted on a glass fiber. All measurements were made on a Rigaku AFC7R diffractometer with graphite monochromated Mo-Kα 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 24q carefully centered reflections in the range $29.53 < 2\theta < 29.92°$ corresponded to a primitive monoclinic cell with dimensions:

a = 10.705(3) Å

b = 13.313(5) Å

c = 13.190(3) Å $\qquad \beta = 107.98(2)^\circ$

V = 1787.9(9) $Å^3$

For Z = 2 and F.W. = 788.08, the calculated density is 1.46 g/cm^3. The systematic absences of:

0k0: k # 2n

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

$P2_1$ (#4)

The data were collected at a temperature of –20 ± 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 0.29° with a take-off angle of 6.0°. Scans of (1.63 + 0.30 tan θ)° were made at a speed of 16.0°/min (in ω). The weak reflections ($I < 10.0\sigma(I)$) were rescanned (maximum of 5 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 and the crystal to detector distance was 235 mm. The computer-controlled slits were set to 9.0 mm (horizontal) and 13.0 mm (vertical).

Data Reduction

Of the 4514 reflections which were collected, 4294 were unique (*R*int = 0.051). The intensities of three representative reflection were measured after every 150 reflections. No decay correction was applied.

The linear absorption coefficient, μ, for Mo-Kα radiation is 38.4 cm^{-1}. An emoirical absoption correction based on azimuthal scans of several reflections was applied which resulted in transmission factors ranging from 0.79 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. Hydrogen atoms were included but not refined. The final cycle of full-matrix least-squares refinement[3] was based on 2389 observed reflections ($I > 3.00\sigma(I)$) and 341 variable parameters and converged (largest parameter shift was 1.45 times its esd) with unweighted and weighted agreement factors of:

$R = \Sigma||Fo|-|Fc||/\Sigma|Fo| = 0.041$

$Rw = [(\Sigma w(|Fo|-|Fc|)^2/\Sigma\ wFo^2)]^{1/2} = 0.035$

The standard deviation of an observation of unit weight[4] was 1.57. The weighting scheme was based on counting statistics and included a factor ($p = 0.009$) to downweight the intense reflections. Plots of $\Sigma w(|Fo|-|Fc|)^2$ versus |Fo|, reflection order in data collection, sin θ/λ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.83 and –0.57 $e^-/Å^3$, respectively.

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

References

(1)SIR92: Altomare, A.; Burla, M. C.; Camalli, M.; Cascarano, M.; Giacovazzo, C.; Guagliardi, A.; Polidori, G. *J. Appl. Cryst.* **1994**, *27*, 435.

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

(3) Least-Squares:

Function minimized: $\Sigma w(|Fo|-|Fc|)^2$

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

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

p = p-factor

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

$[\Sigma w(|Fo|-|Fc|)^2/(No-Nv)]^{1/2}$

where: No = number of observations

Nv = number of variables

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

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

(7) Creagh, D. C.; McAuley, W. J. *International Tables for Crystallography,Vol C*; Wilson, A. J. C. Ed.; Kluwer Academic Publishers: Boston, 1992, Table 4.2.6.8, pp 219–222 .

(8) Creagh, D. C.; Hubbell, J. H. *International Tables for Crystallography, Vol C*; Wilson, A. J. C. Ed.; Kluwer Academic Publishers: Boston, 1992, Table 4.2.4.3, pp 200–206.

(9) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation, 1985 & 1992.

EXPERIMENTAL DETAILS

A. Crystal Data	
Empirical Formula	$C_{31}H_{39}N_2O_2SIr \cdot C_6H_5CH_3$
Formula Weight	788.08
Crystal Color, Habit	yellow, prismatic
Crystal Dimensions	0.20 x 0.20 x 0.20 mm
Crystal System	monoclinic
Lattice Type	Primitive
No. of Reflections Used for Unit	
Cell Determination (2θ range)	24 (29.5–29.9°)
Omega Scan Peak Width at Half-height	0.29°
Lattice Parameters	a = 10.705(3)Å
	b = 13.313(5) Å
	c = 13.190 (3) Å
	β = 107.98(2)°
	V = 1787.9(9) Å^3
Space Group	$P2_1$ (#4)
Z value	2
D_{calc}	1.464 g/cm^3
F_{000}	796.00
μ(MoKα)	38.36cm^{-1}
B. Intensity Measurements	
Diffractometer	Rigaku AFC7R
Radiation	MoKα (λ= 0.71069 Å)
	graphite monochromated
Attenuator	Zr foil (factor = 6.92)
Take-off Angle	6.0°
Detector Aperture	9.0 mm horizontal
	13.0 mm vertical
Crystal to Detector Distance	235 mm
Temperature	–20.0 °C
Scan Type	ω–2θ
Scan Rate	16.0°/min (in ω) (up to 5 scans)
Scan Width	(1.63 + 0.30 tan θ)°
2θ*max*	55.0°
No. of Reflections Measured	Total: 4514

	Unique: 4294 (*Rint* = 0.051)
Corrections	Lorentz-polarization
	Absorption
	(trans. Factors: 0.7890–1.000)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares
Function Minimized	$\Sigma w(\|Fo\|-\|Fc\|)^2$
Least Squares Weights	$w = 1/[\sigma^2(Fo)]$
	$= [\sigma_c^2(Fo) + (p^2/4)Fo^2]^{-1}$
p-factor	0.0090
Anomalous Dispersion	All non-hydrogen atoms
No. Observations ($I > 3.00\sigma(I)$)	2389
No. Variables	341
Reflection/Parameter Ratio	7.01
Residuals: R; Rw	0.041; 0.035
Goodness of Fit Indicator	1.57
Max Shift/Error in Final Cycle	1.45
Maximum peak in Final Diff. Map	0.83 e–/Å^3
Minimum peak in Final Diff. Map	–0.57 e–/Å^3

Table 1. Atomic coodinates and B*iso*/B*eq*

atom	x	y	z	B*eq*
Ir(1)	0.61251(4)	0.230(2)	0.64345(3)	4.218(9)
S(1)	0.4184(3)	0.424(2)	0.6048(3)	4.63(8)
O(1)	0.3455(8)	0.458(2)	0.4999(6)	5.6(2)
O(2)	0.5475(8)	0.470(2)	0.6485(7)	6.4(2)
N(1)	0.4836(9)	0.129(2)	0.5442(7)	4.5(2)
N(2)	0.4256(9)	0.308(2)	0.6113(7)	3.7(1)
C(1)	0.345(1)	0.140(2)	0.546(1)	4.1(3)
C(2)	0.3163(9)	0.254(2)	0.5353(8)	3.8(2)
C(3)	0.177(1)	0.266(2)	0.5420(10)	5.1(3)
C(4)	0.078(1)	0.208(2)	0.457(1)	6.3(3)
C(5)	0.109(1)	0.096(2)	0.467(1)	6.5(4)
C(6)	0.250(1)	0.077(2)	0.466(1)	4.9(3)
C(7)	0.727(1)	0.125(2)	0.763(1)	6.1(4)
C(8)	0.793(2)	0.177(2)	0.716(2)	5.4(3)
C(9)	0.817(2)	0.283(2)	0.741(2)	6.0(3)
C(10)	0.715(1)	0.282(2)	0.810(1)	7.4(3)
C(11)	0.662(1)	0.193(2)	0.809(1)	6.3(3)
C(12)	0.698(1)	0.008(2)	0.756(1)	8.6(5)
C(13)	0.896(1)	0.120(2)	0.659(1)	7.7(5)
C(14)	0.880(2)	0.353(2)	0.721(1)	9.3(6)
C(15)	0.700(2)	0.379(2)	0.858(1)	14.6(7)
C(16)	0.571(2)	0.168(3)	0.878(1)	13.8(8)
C(17)	0.329(1)	0.465(2)	0.6909(9)	4.5(3)
C(18)	0.279(1)	0.566(2)	0.679(1)	5.1(4)
C(19)	0.213(2)	0.594(2)	0.746(2)	8.3(6)
C(20)	0.202(2)	0.534(2)	0.831(2)	7.1(5)
C(21)	0.253(1)	0.442(2)	0.8424(10)	5.7(4)
C(22)	0.317(1)	0.407(2)	0.7732(10)	4.9(3)
C(23)	0.131(1)	0.575(2)	0.902(1)	10.0(6)
C(24)	0.6318(10)	0.281(2)	0.5118(7)	4.2(2)
C(25)	0.648(1)	0.313(2)	0.4279(9)	4.8(3)
C(26)	0.675(1)	0.348(2)	0.3322(10)	4.9(3)
C(27)	0.586(1)	0.348(2)	0.232(1)	8.1(4)
C(28)	0.613(3)	0.378(3)	0.147(2)	13(1)
C(29)	0.745(3)	0.415(3)	0.157(2)	12.9(9)
C(30)	0.829(2)	0.411(2)	0.250(2)	10.8(7)
C(31)	0.803(2)	0.386(2)	0.343(1)	7.3(5)
H(1)	0.4875	0.1385	0.4722	5.5072
H(2)	0.5129	0.0630	0.5660	5.5072
H(3)	0.3363	0.1190	0.6088	5.1866
H(4)	0.3117	0.2773	0.4636	4.8913
H(5)	0.1743	0.2437	0.6103	5.9987
H(6)	0.1543	0.3393	0.5398	5.9987
H(7)	-0.0089	0.2278	0.4575	7.8533
H(8)	0.0844	0.2357	0.3883	7.8533
H(9)	0.1082	0.0780	0.5419	7.9714
H(10)	0.0476	0.0595	0.4204	7.9714
H(11)	0.2510	0.0942	0.3945	5.5636

Table 1. Atomic coodinates and B*iso*/B*eq* (continued)

H(12)	0.2720	0.0090	0.4784	5.5636
H(13)	0.6024	-0.0033	0.7176	9.9319
H(14)	0.7455	-0.0271	0.7210	9.9319
H(15)	0.7097	-0.0203	0.8262	9.9319
H(16)	0.8427	0.0764	0.6003	7.9896
H(17)	0.9330	0.1702	0.6214	7.9896
H(18)	0.9593	0.0843	0.7048	7.9896
H(19)	0.9831	0.3352	0.7396	9.8052
H(20)	0.8670	0.3647	0.6404	9.8052
H(21)	0.8872	0.4199	0.7479	9.8052
H(22)	0.7154	0.3754	0.9360	19.1887
H(23)	0.7470	0.4345	0.8456	19.1887
H(24)	0.6037	0.4024	0.8328	19.1887
H(25)	0.4891	0.2088	0.8537	16.1273
H(26)	0.5384	0.0985	0.8647	16.1273
H(27)	0.6073	0.1773	0.9504	16.1273
H(28)	0.2857	0.6147	0.6211	5.8651
H(29)	0.1689	0.6592	0.7421	9.1781
H(30)	0.2453	0.4005	0.9015	7.2266
H(31)	0.3604	0.3408	0.7849	6.0203
H(32)	0.1716	0.6421	0.9365	10.0135
H(33)	0.0432	0.5925	0.8657	10.0135
H(34)	0.1334	0.5353	0.9634	10.0135
H(35)	0.4904	0.3253	0.2218	10.6729
H(36)	0.5417	0.3745	0.0721	17.4404
H(37)	0.7529	0.4398	0.0938	16.2246
H(38)	0.9294	0.4303	0.2569	12.3118
H(39)	0.8711	0.3940	0.4137	9.9307

$$Beq=8/3\pi^2(U_{11}aa^*)^2+U_{22}(bb^*)^2+U_{33}(cc^*)^2+2U_{12}aa^*bb^*\cos\gamma+2U_{13}aa^*cc^*\cos\beta+2U_{23}bb^*cc^*\cos\alpha)$$

Table 2. Abisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ir(1)	0.0479(2)	0.0662(3)	0.0496(2)	0.0124(6)	0.0201(2)	0.0027(7)
S(1)	0.060(2)	0.052(2)	0.071(2)	0.004(2)	0.029(2)	0.010(2)
O(1)	0.091(6)	0.055(6)	0.068(4)	-0.002(5)	0.027(4)	0.012(4)
O(2)	0.064(4)	0.082(7)	0.102(7)	-0.014(4)	0.033(5)	-0.006(5)
N(1)	0.057(5)	0.050(6)	0.076(7)	0.006(4)	0.036(5)	-0.002(4)
N(2)	0.050(4)	0.050(2)	0.042(5)	0.005(3)	0.018(4)	-0.006(4)
C(1)	0.058(6)	0.041(8)	0.067(8)	0.019(6)	0.033(7)	0.015(6)
C(2)	0.046(5)	0.037(7)	0.063(6)	-0.002(5)	0.020(4)	0.004(6)
C(3)	0.052(5)	0.042(9)	0.108(9)	0.017(5)	0.040(6)	0.003(6)
C(4)	0.043(7)	0.084(9)	0.111(10)	0.004(7)	0.021(6)	-0.004(8)
C(5)	0.055(7)	0.082(9)	0.11(1)	0.006(8)	0.033(8)	0.013(10)
C(6)	0.067(7)	0.038(8)	0.090(9)	0.006(6)	0.037(7)	-0.001(6)
C(7)	0.07(1)	0.089(9)	0.07(1)	0.034(7)	0.025(8)	0.041(6)
C(8)	0.054(6)	0.074(6)	0.06(1)	0.016(7)	-0.003(6)	0.015(7)
C(9)	0.048(5)	0.079(7)	0.07(1)	0.014(6)	-0.021(5)	-0.014(8)
C(10)	0.030(9)	0.14(1)	0.078(7)	-0.014(9)	-0.028(6)	-0.046(9)
C(11)	0.043(9)	0.13(1)	0.041(4)	0.016(6)	-0.027(5)	-0.015(6)
C(12)	0.10(1)	0.091(9)	0.12(1)	0.019(10)	0.01(1)	0.05(1)
C(13)	0.055(9)	0.15(2)	0.09(1)	0.035(10)	0.021(9)	0.00(1)
C(14)	0.11(2)	0.10(1)	0.11(1)	-0.05(1)	-0.02(1)	0.02(1)
C(15)	0.25(2)	0.15(2)	0.09(1)	0.11(2)	-0.05(1)	-0.06(1)
C(16)	0.10(1)	0.38(4)	0.057(10)	0.05(2)	0.048(9)	0.07(1)
C(17)	0.046(7)	0.053(7)	0.072(8)	0.003(6)	0.016(5)	-0.003(5)
C(18)	0.055(9)	0.051(8)	0.08(1)	-0.005(7)	0.019(8)	0.015(8)
C(19)	0.08(1)	0.11(2)	0.14(2)	0.03(1)	0.06(1)	-0.02(1)
C(20)	0.055(9)	0.09(1)	0.12(2)	0.008(9)	0.021(10)	-0.04(1)
C(21)	0.084(10)	0.077(9)	0.072(9)	0.000(7)	0.047(7)	-0.007(8)
C(22)	0.062(8)	0.055(8)	0.077(8)	0.015(7)	0.030(6)	0.004(6)
C(23)	0.08(1)	0.17(2)	0.13(1)	0.02(1)	0.05(1)	-0.08(1)
C(24)	0.048(6)	0.069(8)	0.040(5)	0.000(6)	0.008(5)	-0.026(4)
C(25)	0.060(7)	0.070(9)	0.058(6)	0.008(7)	0.026(6)	-0.001(6)
C(26)	0.092(8)	0.048(9)	0.059(6)	0.010(8)	0.040(5)	0.008(6)
C(27)	0.10(1)	0.13(1)	0.068(7)	0.06(1)	0.018(6)	0.005(10)
C(28)	0.34(4)	0.16(2)	0.07(1)	0.05(3)	0.12(2)	0.03(2)
C(29)	0.36(4)	0.10(2)	0.07(1)	0.02(3)	0.13(2)	0.00(1)
C(30)	0.20(2)	0.13(2)	0.14(2)	-0.01(2)	0.14(2)	0.02(1)
C(31)	0.11(1)	0.08(1)	0.12(1)	-0.04(1)	0.08(1)	-0.03(1)

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)

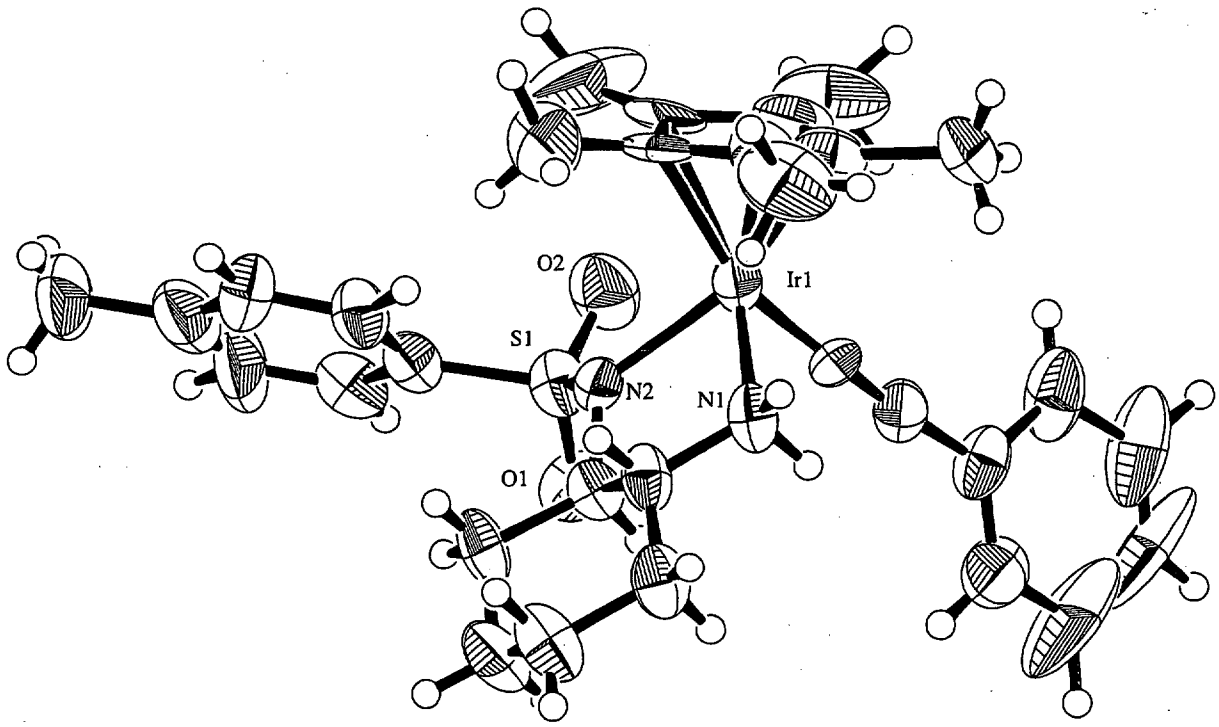
atom	atom	distance	atom	atom	distance
Ir(1)	N(1)	2.07(2)	Ir(1)	N(2)	2.17(2)
Ir(1)	C(7)	2.18(2)	Ir(1)	C(8)	2.00(2)
Ir(1)	C(9)	2.29(2)	Ir(1)	C(10)	2.25(2)
Ir(1)	C(11)	2.14(2)	Ir(1)	C(24)	1.93(2)
S(1)	O(1)	1.44(1)	S(1)	O(2)	1.46(2)
S(1)	N(2)	1.55(3)	S(1)	C(17)	1.78(2)
N(1)	C(1)	1.50(2)	N(1)	H(1)	0.972
N(1)	H(2)	0.947	N(2)	C(2)	1.47(2)
C(1)	C(2)	1.54(4)	C(1)	C(6)	1.48(2)
C(1)	H(3)	0.909	C(2)	C(3)	1.53(2)
C(2)	H(4)	0.984	C(3)	C(4)	1.50(2)
C(3)	H(5)	0.958	C(3)	H(6)	1.003
C(4)	C(5)	1.53(4)	C(4)	H(7)	0.964
C(4)	H(8)	1.003	C(5)	C(6)	1.54(2)
C(5)	H(9)	1.011	C(5)	H(10)	0.892
C(6)	H(11)	0.969	C(6)	H(12)	0.933
C(7)	C(8)	1.28(3)	C(7)	C(11)	1.38(3)
C(7)	C(12)	1.59(4)	C(8)	C(9)	1.46(4)
C(8)	C(13)	1.69(3)	C(9)	C(10)	1.63(3)
C(9)	C(14)	1.22(4)	C(10)	C(11)	1.32(4)
C(10)	C(15)	1.47(4)	C(11)	C(16)	1.57(3)
C(12)	H(13)	1.000	C(12)	H(14)	0.912
C(12)	H(15)	0.971	C(13)	H(16)	0.997
C(13)	H(17)	0.979	C(13)	H(18)	0.897
C(14)	H(19)	1.081	(14)	H(20)	1.038
C(14)	H(21)	0.960	C(15)	H(22)	0.987
C(15)	H(23)	0.939	C(15)	H(24)	1.029
C(16)	H(25)	0.998	C(16)	H(26)	0.982
C(16)	H(27)	0.919	C(17)	C(18)	1.44(3)
C(17)	C(22)	1.37(2)	C(18)	C(19)	1.35(3)
C(18)	H(28)	1.019	C(19)	C(20)	1.41(4)
C(19)	H(29)	0.985	C(20)	C(21)	1.32(4)
C(20)	C(23)	1.48(3)	C(21)	C(22)	1.38(2)
C(21)	H(30)	0.981	C(22)	H(31)	0.987
C(23)	H(32)	1.041	C(23)	H(33)	0.940
C(23)	H(34)	0.959	C(24)	C(25)	1.25(2)
C(25)	C(26)	1.45(2)	C(26)	C(27)	1.37(2)
C(26)	C(31)	1.42(3)	C(27)	C(28)	1.31(3)
C(27)	H(35)	1.038	C(28)	C(29)	1.46(5)
C(28)	H(36)	1.042	C(29)	C(30)	1.27(3)
C(29)	H(37)	0.926	C(30)	C(31)	1.39(3)
C(30)	H(38)	1.077	C(31)	H(39)	0.994

Table 4. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
N(1)	Ir(1)	N(2)	78.0(7)	N(1)	Ir(1)	C(7)	99(1)
N(1)	Ir(1)	C(8)	115(1)	N(1)	Ir(1)	C(9)	153(1)
N(1)	Ir(1)	C(10)	148(1)	N(1)	Ir(1)	C(11)	114(1)
N(1)	Ir(1)	C(24)	83.9(7)	N(2)	Ir(1)	C(7)	136.4(6)
N(2)	Ir(1)	C(8)	162.8(9)	N(2)	Ir(1)	C(9)	129(1)
N(2)	Ir(1)	C(10)	100.7(8)	N(2)	Ir(1)	C(11)	103.5(6)
N(2)	Ir(1)	C(24)	90.3(7)	C(7)	Ir(1)	C(8)	35.2(9)
C(7)	Ir(1)	C(9)	64.0(9)	C(7)	Ir(1)	C(10)	59.5(9)
C(7)	Ir(1)	C(11)	37.4(8)	C(7)	Ir(1)	C(24)	133.1(8)
C(8)	Ir(1)	C(9)	39(1)	C(8)	Ir(1)	C(10)	62.2(9)
C(8)	Ir(1)	C(11)	62.0(8)	C(8)	Ir(1)	C(24)	101.5(8)
C(9)	Ir(1)	C(10)	42.0(8)	C(9)	Ir(1)	C(11)	66.6(8)
C(9)	Ir(1)	C(24)	92.8(8)	C(10)	Ir(1)	C(11)	35(1)
C(10)	Ir(1)	C(24)	128(1)	C(11)	Ir(1)	C(24)	159.3(7)
O(1)	S(1)	O(2)	114(1)	O(1)	S(1)	N(2)	112(1)
O(1)	S(1)	C(17)	106.1(9)	O(2)	S(1)	N(2)	112(1)
O(2)	S(1)	C(17)	105(1)	N(2)	S(1)	C(17)	107(1)
Ir(1)	N(1)	C(1)	114(1)	Ir(1)	N(1)	H(1)	108.19
Ir(1)	N(1)	H(2)	108.37	C(1)	N(1)	H(1)	110.01
C(1)	N(1)	H(2)	108.73	H(1)	N(1)	H(2)	107.86
Ir(1)	N(2)	S(1)	120.9(9)	Ir(1)	N(2)	C(2)	113(1)
S(1)	N(2)	C(2)	116(1)	N(1)	C(1)	C(2)	105(2)
N(1)	C(1)	C(6)	114(2)	N(1)	C(1)	H(3)	111.43
C(2)	C(1)	C(6)	115(1)	C(2)	C(1)	H(3)	107.74
C(6)	C(1)	H(3)	103.88	N(2)	C(2)	C(1)	109(1)
N(2)	C(2)	C(3)	119(1)	N(2)	C(2)	H(4)	106.89
C(1)	C(2)	C(3)	106(2)	C(1)	C(2)	H(4)	110.45
C(3)	C(2)	H(4)	105.13	C(2)	C(3)	C(4)	112(1)
C(2)	C(3)	H(5)	109.49	C(2)	C(3)	H(6)	109.67
C(4)	C(3)	H(5)	108.74	C(4)	C(3)	H(6)	111.71
H(5)	C(3)	H(6)	104.55	C(3)	C(4)	C(5)	111(1)
C(3)	C(4)	H(7)	108.45	C(3)	C(4)	H(8)	104.66
C(5)	C(4)	H(7)	116.73	C(5)	C(4)	H(8)	111.42
H(7)	C(4)	H(8)	104.11	C(4)	C(5)	C(6)	110(2)
C(4)	C(5)	H(9)	104.34	C(4)	C(5)	H(10)	111.85
C(6)	C(5)	H(9)	106.08	C(6)	C(5)	H(10)	114.50
H(9)	C(5)	H(10)	109.09	C(1)	C(6)	C(5)	111(2)
C(1)	C(6)	H(11)	110.18	C(1)	C(6)	H(12)	109.99
C(5)	C(6)	H(11)	106.11	C(5)	C(6)	H(12)	110.31
H(11)	C(6)	H(12)	109.30	Ir(1)	C(7)	C(8)	65(1)
Ir(1)	C(7)	C(11)	70(1)	Ir(1)	C(7)	C(12)	122(1)
C(8)	C(7)	C(11)	107(2)	C(8)	C(7)	C(12)	129(2)
C(11)	C(7)	C(12)	123(2)	Ir(1)	C(8)	C(7)	80(1)
Ir(1)	C(8)	C(9)	81(1)	Ir(1)	C(8)	C(13)	128(1)
C(7)	C(8)	C(9)	120(2)	C(7)	C(8)	C(13)	121(3)
C(9)	C(8)	C(13)	116(2)	Ir(1)	C(9)	C(8)	60(1)
Ir(1)	C(9)	C(10)	67.7(9)	Ir(1)	C(9)	C(14)	127(2)
C(8)	C(9)	C(10)	91(2)	C(8)	C(9)	C(14)	139(3)

Table 4. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C(10)	C(9)	C(14)	130(3)	Ir(1)	C(10)	C(9)	70(1)
Ir(1)	C(10)	C(11)	68(1)	Ir(1)	C(10)	C(15)	128(1)
C(9)	C(10)	C(11)	111(2)	C(9)	C(10)	C(15)	115(2)
C(11)	C(10)	C(15)	134(2)	Ir(1)	C(11)	C(7)	73(1)
Ir(1)	C(11)	C(10)	77(1)	Ir(1)	C(11)	C(16)	129.9(9)
C(7)	C(11)	C(10)	109(2)	C(7)	C(11)	C(16)	127(3)
C(10)	C(11)	C(16)	122(2)	C(7)	C(12)	H(13)	109.10
C(7)	C(12)	H(14)	113.56	C(7)	C(12)	H(15)	110.65
H(13)	C(12)	H(14)	108.37	H(13)	C(12)	H(15)	103.77
H(14)	C(12)	H(15)	110.89	C(8)	C(13)	H(16)	107.89
C(8)	C(13)	H(17)	110.21	C(8)	C(13)	H(18)	113.38
H(16)	C(13)	H(17)	103.43	H(16)	C(13)	H(18)	109.91
H(17)	C(13)	H(18)	111.50	C(9)	C(14)	H(19)	112.21
C(9)	C(14)	H(20)	115.91	C(9)	C(14)	H(21)	127.77
H(19)	C(14)	H(20)	94.15	H(19)	C(14)	H(21)	98.82
H(20)	C(14)	H(21)	101.86	C(10)	C(15)	H(22)	113.55
C(10)	C(15)	H(23)	118.57	C(10)	C(15)	H(24)	110.99
H(22)	C(15)	H(23)	107.27	H(22)	C(15)	H(24)	100.61
H(23)	C(15)	H(24)	103.97	C(11)	C(16)	H(25)	109.79
C(11)	C(16)	H(26)	110.01	C(11)	C(16)	H(27)	115.74
H(25)	C(16)	H(26)	103.16	H(25)	C(16)	H(27)	108.00
H(26)	C(16)	H(27)	109.38	S(1)	C(17)	C(18)	118(1)
S(1)	C(17)	C(22)	122(2)	C(18)	C(17)	C(22)	120(2)
C(17)	C(18)	C(19)	116(2)	C(17)	C(18)	H(28)	124.67
C(19)	C(18)	H(28)	119.49	C(18)	C(19)	C(20)	124(3)
C(18)	C(19)	H(29)	122.96	C(20)	C(19)	H(29)	113.10
C(19)	C(20)	C(21)	119(2)	C(19)	C(20)	C(23)	118(2)
C(21)	C(20)	C(23)	123(2)	C(20)	C(21)	C(22)	121(2)
C(20)	C(21)	H(30)	118.65	C(22)	C(21)	H(30)	120.74
C(17)	C(22)	C(21)	121(2)	C(17)	C(22)	H(31)	118.25
C(21)	C(22)	H(31)	120.36	C(20)	C(23)	H(32)	111.29
C(20)	C(23)	H(33)	113.64	C(20)	C(23)	H(34)	116.28
H(32)	C(23)	H(33)	102.94	H(32)	C(23)	H(34)	101.65
H(33)	C(23)	H(34)	109.58	Ir(1)	C(24)	C(25)	178.2(9)
C(24)	C(25)	C(26)	176(2)	C(25)	C(26)	C(27)	125(2)
C(25)	C(26)	C(31)	118(1)	C(27)	C(26)	C(31)	117(2)
C(26)	C(27)	C(28)	124(2)	C(26)	C(27)	H(35)	118.98
C(28)	C(27)	H(35)	116.87	C(27)	C(28)	C(29)	119(2)
C(27)	C(28)	H(36)	120.27	C(29)	C(28)	H(36)	120.73
C(28)	C(29)	C(30)	117(3)	C(28)	C(29)	H(37)	112.83
C(30)	C(29)	H(37)	130.02	C(29)	C(30)	C(31)	126(3)
C(29)	C(30)	H(38)	117.79	C(31)	C(30)	H(38)	116.53
C(26)	C(31)	C(30)	116(1)	C(26)	C(31)	H(39)	122.73
C(30)	C(31)	H(39)	120.78				



(η^5-$C_5(CH_3)_5$)Ir(CCC_6H_5)[(1*R*,2*R*)-*N*-*p*-toluenesulfonyl-1,2-cyclohexanediamine] (1d)