Inorganic Chemistry
including bioinorganic chemistry
Inorg. Chem., 1998, 37(21), 5412-5413, DOI:10.1021/ic9806277

Structure Determination Supplementary Data

Empirical Formula	$C_{27}\ H_{29}\ Cl_3\ (I, Cl)_1\ Ir_1\ N_2\ O_1$
Crystal size (mm)	0.30 x 0.25 x 0.25
Crystal System	Triclinic
Space Group	$P\bar{1}$
Unit Cell Dimensions	$\underline{a}$ = 10.2890(8) Å
	$\underline{b}$ = 10.7647(10) Å
	$\underline{c}$ = 14.0078(12) Å
	α = 89.170(7)°
	β = 76.32(7)°
	γ = 64.516(8)°
Volume	1354.0(7) $Å^3$
Z	2
Formula weight	731.6 (Cloro), 823.0 (Iodo)
Density(calc.)	1.855 Mg/m^3
Absorption Coefficient	5.632 mm^{-1}
F(000)	735.58
Diffractometer Used	Enraf Nonius CAD4
Radiation	MoKα (λ = 0.71073 Å)
Temperature (K)	296
Monochromator	Highly oriented graphite crystal
Scan Type	ω-2θ
Standard Reflections	3 measured every 6000 reflections
Index Ranges	$-11 \le h \le 11$, $-11 \le k \le 11$
	$-15 \le l \le 15$
Reflections Collected	7038
Independent Reflections	3519 (R_{int} = 1.87%)
Observed Reflections	3089 ($F > 6.0\sigma(F)$)

Absorption Correction	Semi-empirical
Min./Max. Transmission	0.5091 / 0.7622
System Used	Siemens SHELXTL PLUS (PC Version)
Solution	Direct Methods
Refinement Method	Full-Matrix Least-Squares
Quantity Minimized	$\sum w(F_o - F_c)^2$
Hydrogen Atoms	Riding model, fixed isotropic U
Weighting Scheme	$w^{-1} = \sigma^2(F) + 0.0012F^2$
Number of Parameters Refined	313
Final R Indices (obs. data)	R = 3.25 %, wR = 3.78 %
R Indices (all data)	R = 3.87 %, wR = 5.23 %
Goodness-of-Fit	1.15
Largest and Mean Δ/σ	0.004, 0.001
Data-to-Parameter Ratio	11.2:1
Largest Difference Peak	2.11 $e\text{Å}^{-3}$ (associated with disorder)
Largest Difference Hole	-1.32 $e\text{Å}^{-3}$ (associated with disorder)

Supplementary Data. Atomic coordinates ($x10^4$) and equivalent isotropic displacement coefficients ($Å^2 x10^3$)

	x	y	z	U(eq)
Ir(1)	-1161(1)	3887(1)	2669(1)	38(1)
I(1)	-2154(2)	3418(1)	1311(1)	72(7)
Cl(1)	-2154(2)	3418(1)	1311(1)	44(5)
Cl(2)	-459(3)	-49(3)	2746(2)	72(1)
Cl(3)	3334(3)	-5355(2)	2710(2)	61(1)
Cl(4)	6603(4)	16(3)	-4452(2)	96(2)
O(8)	2919(6)	5(6)	79(4)	47(2)
N(2)	1089(7)	4463(6)	1101(5)	42(3)
N(5)	2013(7)	2345(6)	1393(4)	41(3)
C(1)	758(8)	3511(8)	1661(5)	39(3)
C(3)	2493(9)	3878(8)	508(6)	48(4)
C(4)	3137(9)	2508(9)	694(6)	47(4)
C(6)	2251(9)	998(8)	1717(6)	46(4)
C(7)	1949(9)	118(8)	1024(5)	45(4)
C(9)	2314(11)	44(11)	-726(6)	67(5)
C(10)	-29(10)	5942(8)	1137(6)	58(4)
C(11)	2228(8)	-1273(8)	1412(5)	39(3)
C(12)	1258(9)	-1434(8)	2212(6)	48(4)
C(13)	1599(9)	-2691(8)	2614(6)	45(4)
C(14)	2879(9)	-3764(8)	2215(6)	46(4)
C(15)	3884(11)	-3709(9)	1410(7)	64(4)
C(16)	3514(10)	-2404(9)	1008(7)	63(4)
C(21)	3372(9)	9(10)	-1629(6)	56(4)
C(22)	3676(10)	1181(10)	-1865(7)	63(4)
C(23)	4597(11)	1178(9)	-2691(7)	59(5)
C(24)	5342(11)	25(9)	-3370(6)	60(5)
C(25)	5099(12)	-1083(10)	-3212(7)	67(5)
C(26)	4063(10)	-1110(10)	-2331(6)	62(5)
C(31)	-2764(9)	3500(9)	3795(6)	52(4)
C(32)	-3368(9)	4949(10)	3697(6)	57(4)
C(33)	-3444(10)	6026(11)	4417(7)	69(5)
C(34)	-2006(10)	6202(11)	4221(8)	72(5)
C(35)	-682(10)	5023(9)	3611(6)	51(4)
C(36)	-112(12)	3593(14)	3829(6)	73(6)
C(37)	-901(13)	3208(13)	4757(7)	81(6)
C(38)	-2165(10)	2944(10)	4639(7)	66(5)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

Supplementary Data. Bond lengths (Å)

Ir(1)-I(1)	2.512 (2)	Ir(1)-Cl(1)	2.512 (2)
Ir(1)-C(1)	2.017 (7)	Ir(1)-C(31)	2.173 (9)
Ir(1)-C(32)	2.192 (7)	Ir(1)-C(35)	2.087 (11)
Ir(1)-C(36)	2.104 (11)	Cl(2)-C(12)	1.744 (7)
Cl(3)-C(14)	1.750 (9)	Cl(4)-C(24)	1.743 (10)
O(8)-C(7)	1.428 (9)	O(8)-C(9)	1.404 (12)
N(2)-C(1)	1.390 (11)	N(2)-C(3)	1.356 (9)
N(2)-C(10)	1.503 (9)	N(5)-C(1)	1.332 (8)
N(5)-C(4)	1.400 (11)	N(5)-C(6)	1.448 (11)
C(3)-C(4)	1.383 (12)	C(6)-C(7)	1.535 (14)
C(7)-C(11)	1.517 (12)	C(9)-C(21)	1.450 (12)
C(11)-C(12)	1.376 (11)	C(11)-C(16)	1.354 (9)
C(12)-C(13)	1.391 (12)	C(13)-C(14)	1.320 (9)
C(14)-C(15)	1.358 (13)	C(15)-C(16)	1.433 (14)
C(21)-C(22)	1.442 (16)	C(21)-C(26)	1.375 (12)
C(22)-C(23)	1.310 (13)	C(23)-C(24)	1.380 (12)
C(24)-C(25)	1.326 (17)	C(25)-C(26)	1.436 (13)
C(31)-C(32)	1.427 (14)	C(31)-C(38)	1.465 (14)
C(32)-C(33)	1.513 (16)	C(33)-C(34)	1.533 (17)
C(34)-C(35)	1.480 (11)	C(35)-C(36)	1.449 (16)
C(36)-C(37)	1.515 (16)	C(37)-C(38)	1.490 (20)

Supplementary Data. Bond angles (°)

I(1)-Ir(1)-Cl(1)	0.0(1)	I(1)-Ir(1)-C(1)	88.9(3)
Cl(1)-Ir(1)-C(1)	88.9(3)	I(1)-Ir(1)-C(31)	92.1(3)
Cl(1)-Ir(1)-C(31)	92.1(3)	C(1)-Ir(1)-C(31)	159.2(3)
I(1)-Ir(1)-C(32)	93.9(3)	Cl(1)-Ir(1)-C(32)	93.9(3)
C(1)-Ir(1)-C(32)	162.4(4)	C(31)-Ir(1)-C(32)	38.2(4)
I(1)-Ir(1)-C(35)	158.0(2)	Cl(1)-Ir(1)-C(35)	158.0(2)
C(1)-Ir(1)-C(35)	90.2(4)	C(31)-Ir(1)-C(35)	96.4(4)
C(32)-Ir(1)-C(35)	80.8(4)	I(1)-Ir(1)-C(36)	161.5(4)
Cl(1)-Ir(1)-C(36)	161.5(4)	C(1)-Ir(1)-C(36)	91.3(4)
C(31)-Ir(1)-C(36)	81.2(4)	C(32)-Ir(1)-C(36)	91.5(4)
C(35)-Ir(1)-C(36)	40.5(5)	C(7)-O(8)-C(9)	115.0(7)
C(1)-N(2)-C(3)	111.9(6)	C(1)-N(2)-C(10)	122.6(6)
C(3)-N(2)-C(10)	125.4(7)	C(1)-N(5)-C(4)	113.0(7)
C(1)-N(5)-C(6)	126.4(7)	C(4)-N(5)-C(6)	120.5(6)
Ir(1)-C(1)-N(2)	126.9(4)	Ir(1)-C(1)-N(5)	129.7(6)
N(2)-C(1)-N(5)	103.4(6)	N(2)-C(3)-C(4)	106.9(8)
N(5)-C(4)-C(3)	104.8(6)	N(5)-C(6)-C(7)	113.2(7)
O(8)-C(7)-C(6)	105.5(8)	O(8)-C(7)-C(11)	112.9(5)
C(6)-C(7)-C(11)	109.9(7)	O(8)-C(9)-C(21)	109.2(9)
C(7)-C(11)-C(12)	122.6(6)	C(7)-C(11)-C(16)	120.0(7)
C(12)-C(11)-C(16)	117.2(8)	Cl(2)-C(12)-C(11)	120.3(6)
Cl(2)-C(12)-C(13)	118.0(6)	C(11)-C(12)-C(13)	121.7(6)
C(12)-C(13)-C(14)	119.4(8)	Cl(3)-C(14)-C(13)	120.2(7)
Cl(3)-C(14)-C(15)	117.0(5)	C(13)-C(14)-C(15)	122.9(8)
C(14)-C(15)-C(16)	116.9(7)	C(11)-C(16)-C(15)	121.8(8)
C(9)-C(21)-C(22)	121.7(8)	C(9)-C(21)-C(26)	121.6(10)
C(22)-C(21)-C(26)	116.6(8)	C(21)-C(22)-C(23)	122.3(9)
C(22)-C(23)-C(24)	120.7(10)	Cl(4)-C(24)-C(23)	120.3(9)
Cl(4)-C(24)-C(25)	119.2(6)	C(23)-C(24)-C(25)	120.5(9)
C(24)-C(25)-C(26)	120.8(8)	C(21)-C(26)-C(25)	119.1(10)
Ir(1)-C(31)-C(32)	71.6(5)	Ir(1)-C(31)-C(38)	111.4(7)
C(32)-C(31)-C(38)	120.8(9)	Ir(1)-C(32)-C(31)	70.2(4)
Ir(1)-C(32)-C(33)	112.3(7)	C(31)-C(32)-C(33)	124.7(8)
C(32)-C(33)-C(34)	113.3(7)	C(33)-C(34)-C(35)	114.1(10)
Ir(1)-C(35)-C(34)	114.4(8)	Ir(1)-C(35)-C(36)	70.4(7)
C(34)-C(35)-C(36)	126.1(8)	Ir(1)-C(36)-C(35)	69.1(6)
Ir(1)-C(36)-C(37)	113.8(10)	C(35)-C(36)-C(37)	119.5(8)
C(36)-C(37)-C(38)	114.0(10)	C(31)-C(38)-C(37)	116.0(10)

Supplementary Data. Anisotropic displacement coefficients ($Å^2 x 10^3$)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ir(1)	33(1)	44(1)	34(1)	-17(1)	-2(1)	-1(1)
Cl(2)	45(1)	60(1)	77(2)	-5(1)	12(1)	8(1)
Cl(3)	66(1)	49(1)	64(1)	-22(1)	-14(1)	18(1)
Cl(4)	97(2)	95(2)	72(2)	-36(2)	7(2)	19(2)
O(8)	42(3)	53(3)	39(3)	-18(3)	-6(2)	7(2)
N(2)	36(4)	43(4)	41(4)	-13(3)	-7(3)	8(3)
N(5)	37(4)	44(4)	42(4)	-22(3)	-3(3)	5(3)
C(1)	40(4)	40(4)	36(4)	-15(4)	-9(3)	5(3)
C(3)	49(5)	53(5)	38(5)	-28(4)	9(4)	-1(4)
C(4)	33(4)	57(5)	49(5)	-24(4)	1(4)	2(4)
C(6)	43(5)	48(5)	42(5)	-16(4)	-9(4)	9(4)
C(7)	44(5)	49(5)	40(5)	-22(4)	-3(4)	1(4)
C(9)	65(6)	82(7)	46(5)	-25(5)	-14(5)	6(5)
C(10)	79(6)	48(5)	46(5)	-30(5)	-13(5)	11(4)
C(11)	35(4)	44(4)	38(4)	-17(4)	-8(3)	5(3)
C(12)	45(5)	46(5)	45(5)	-14(4)	-10(4)	-1(4)
C(13)	42(5)	51(5)	40(5)	-22(4)	-2(4)	11(4)
C(14)	48(5)	41(5)	48(5)	-18(4)	-13(4)	12(4)
C(15)	57(6)	45(5)	72(7)	-18(4)	8(5)	2(4)
C(16)	43(5)	54(6)	72(6)	-18(4)	13(5)	13(5)
C(21)	38(5)	63(6)	43(5)	0(4)	-13(4)	-4(4)
C(22)	53(6)	56(6)	54(6)	3(5)	-16(5)	-9(4)
C(23)	76(7)	41(5)	56(6)	-19(5)	-25(5)	20(4)
C(24)	91(7)	50(5)	34(5)	-28(5)	-18(5)	20(4)
C(25)	77(7)	57(6)	49(6)	-17(5)	-7(5)	-10(4)
C(26)	66(6)	63(6)	50(6)	-23(5)	-12(5)	4(5)
C(31)	44(5)	62(6)	49(5)	-32(4)	9(4)	0(4)
C(32)	31(4)	92(7)	45(5)	-29(5)	1(4)	-6(5)
C(33)	58(6)	72(6)	55(6)	-17(5)	0(5)	-20(5)
C(34)	66(6)	68(6)	75(7)	-27(5)	-8(5)	-23(5)
C(35)	53(5)	60(6)	42(5)	-31(5)	-6(4)	-9(4)
C(36)	66(6)	140(11)	38(5)	-68(7)	-13(4)	2(6)
C(37)	105(9)	100(8)	52(6)	-56(7)	-26(6)	26(6)
C(38)	57(6)	67(6)	60(6)	-26(5)	7(5)	5(5)

The anisotropic displacement exponent takes the form:

$-2\pi^2(h^2a^{*2}U_{11} + \ldots + 2hka^*b^*U_{12})$

Supplementary Data. H-Atom coordinates ($x10^4$) and isotropic displacement coefficients ($Å^2 x10^3$)

	x	y	z	U
H(3)	2956	4313	33	55
H(4)	4133	1817	411	42
H(6A)	1613	1124	2367	47
H(6B)	3262	491	1757	47
H(7)	935	608	988	42
H(9A)	1408	879	-647	63
H(9B)	2094	-732	-767	63
H(10A)	424	6420	698	55
H(10B)	-869	5969	936	55
H(10C)	-353	6382	1797	55
H(13)	918	-2761	3188	45
H(15)	4808	-4503	1132	63
H(16)	4190	-2328	433	62
H(22)	3192	1984	-1394	63
H(23)	4783	1973	-2824	82
H(25)	5607	-1869	-3699	68
H(26)	3876	-1907	-2220	60
H(31)	-3255	3038	3551	52
H(32)	-4164	5290	3374	54
H(33A)	-3627	5743	5069	69
H(33B)	-4272	6891	4398	69
H(34A)	-1832	6370	4840	71
H(34B)	-2157	7009	3875	71
H(35)	86	5275	3278	57
H(36)	953	3087	3638	48
H(37A)	-1286	3936	5281	86
H(37B)	-187	2393	4955	86
H(38A)	-2970	3313	5225	71
H(38B)	-1829	1960	4574	71