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CRYSTAL AND MOLECULAR STRUCTURE OF HYDRIDOIODOBIS(TRIPHENYLPHOSPHINE)-*p*-METHOXYPHENYLDIAZENE-C^2, N^1-IRIDIUM/CHLOROFORM 1/1

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Summary

The crystal and molecular structure of the title compound has been determined from three-dimensional X-ray data collected by counter methods. The crystals are monoclinic (space group $P2_1/n$) with cell dimensions: a 19.012(15), b 11.682(10), c 19.803(16) Å, β 92.73(9)°, U 4396.0 Å^3. The positions of the hydrogen atoms bonded to Ir and to N(1) atoms could not be determined. The title formulation is consistent with the bond distances and angles observed. Relevant parameters within the metallocycle are: the Ir—N(1) distance of 2.076(8) Å, which is longer than that usually observed in diazene complexes because of the *trans* influence of the hydrido ligand, and the distance N(1)—N(2) of 1.265(12) Å, typical of a double bond interaction.

Introduction

Transition metal complexes of aryldiazenato ($ArNN^-$) and of aryldiazene (ArNNH) ligands represent valuable models of the catalytic activation of dinitrogen and for its preliminary reduction to diazene (N_2H_2). A comprehensive review of these compounds was published by Sutton in 1975 [1].

Species containing an aryldiazenato ligand can react with solvents [2] or anions [3] to yield aryldiazene complexes of unexpected compositions; on the other hand, the protonation of these ligands to aryldiazenes is an intriguing reaction (of great importance in the model chemistry of nitrogen fixation), which has been shown to involve a donor molecule of solvent [4]. Despite the many reports dealing with the synthesis and the structure of this class of compounds, only two structures of orthometallated aryldiazene complexes have been fully elucidated [2,5].

We are at present investigating the structures of other orthometallated species, obtained by treating aryldiazonium salts with $IrH_3(PPh_3)_3$ [6] and $IrH(CO)(PPh_3)_3$ [7]. In some cases the complexes obtained contain an hydrido ligand *trans* to N(1) and therefore offer an opportunity of investigating the thermodynamic lability of the metal—nitrogen bond. This report describes the structure of the complex $Ir(p\text{-}CH_3OC_6H_3NNH)HI(PPh_3)_2$ (I), whose formulation was previously suggested on the basis of IR and NMR spectra [8].

Experimental

Crystal data

$C_{43}H_{38}IIrN_2OP_2 \cdot CHCl_3$, mol. wt. 1099.2, prepared as in ref. 8, gives orange crystals of polyhedral habit. The reduced cell dimensions are: a 19.012(15), b 11.692(10), c 19.803(16) Å, β 92.73(9)°, U 4396.0 Å^3; space group $P2_1/n$ (No. 14), D_m 1.67 g cm^{-3} (by flotation), $Z = 4$, D_c 1.66 g cm^{-3} $F(000)$ 2144. Cell dimensions determined from precession photographs using Mo-K_α radiation (λ 0.7107 Å).

Structure solution and refinement

The integrated intensities were collected on a PAILRED linear diffractometer with Mo-K_α radiation, monochromatized with a graphite crystal. The specimen, a multifaced polyhedron of approximate dimensions 0.43 × 0.41 × 0.36 mm, was mounted approximately along the direction of its maximum elongation (c axis). 4196 independent reflections, belonging to 11 reciprocal lattice layers, from $hk0$ to $hk10$, and having $\sigma(I)/I \leqslant 0.25$ were measured by the stationary counter-moving crystal technique within the limit angle $2\theta \leqslant 50°$ since no significant reflections were detected outside this range. No crystal decay was noticed during the collection of data. The integrated intensities were corrected for Lorentz and polarisation effects, the latter allowing for the presence of the monochromator [9]. Transmission factors (μ 42.4 cm^{-1}) were sampled for each reflection in 8^3 points of the crystal with the method described in ref. 10.

The structure was solved by conventional Patterson and Fourier methods. The model obtained was refined by full matrix least squares, the minimized function being $\Sigma w(F_0 - k|F_c|)^2$. The six independent phenyl rings belonging to the phosphine ligands were treated as rigid bodies possessing D_{6h} symmetry (C—C 1.392 Å). All non hydrogen atoms were assigned anisotropical behaviour, apart from the phenyl carbon atoms which were treated isotropically. In structure factor computations the contribution of all hydrogen atoms was included. Their coordinates, updated after each refinement cycle, were generated by assuming (i) C—H 1.08 Å, C—C—H 120° and 109.5° in phenyl and methyl groups. No allowance was made for the H—N(1) and the hydrido ligand at this stage (see discussion). Including a scale factor, a total of 235 parameters were refined.

In least squares and structure factor computations the atomic scattering factors of ref. 11 and 12 were adopted. Real and imaginary correction for the anomalous dispersion of Ir and I atoms were taken into account [13]. Each independent observation was assigned a weight according to the formula w =

TABLE 1

POSITIONAL AND THERMAL PARAMETERS IN $Ir(p\text{-}CH_3C_6H_3NNH)HI(PPh_3)_2$ [a], [b]

Atom	x	y	z	b_{11}	b_{12}	b_{13}	b_{22}	b_{23}	b_{33}
Ir	303(0)	1216(0)	1664(0)	15(0)	2(0)	2(0)	63(0)	7(0)	12(0)
I	−217(0)	1755(0)	364(0)	23(0)	9(0)	0(0)	82(0)	8(0)	14(0)
P(1)	−831(1)	884(2)	1993(1)	17(0)	3(2)	5(1)	103(3)	5(2)	15(0)
P(2)	1384(1)	2022(2)	1434(1)	18(0)	−1(2)	1(1)	57(2)	7(1)	15(0)
N(1)	527(3)	−466(7)	1420(3)	21(2)	0(6)	8(3)	55(8)	0(6)	19(2)
N(2)	785(4)	−1147(8)	1863(4)	25(2)	14(8)	3(4)	80(9)	17(9)	25(2)
C(1)	892(5)	−597(10)	2475(5)	21(3)	12(9)	0(4)	65(11)	30(9)	27(3)
C(2)	706(4)	545(9)	2534(4)	15(2)	0(7)	3(4)	79(11)	23(7)	16(2)
C(3)	807(5)	1092(12)	3172(4)	23(3)	−17(11)	0(4)	147(15)	12(10)	13(2)
C(4)	1100(5)	438(13)	3709(4)	24(3)	−26(12)	2(4)	147(16)	24(9)	13(2)
C(5)	1292(6)	−699(15)	3636(6)	34(4)	24(14)	−1(6)	150(20)	58(13)	25(3)
C(6)	1178(6)	−1232(12)	3028(5)	34(3)	30(12)	−7(5)	111(13)	48(12)	30(3)
O	1224(4)	884(11)	4337(4)	44(3)	−19(11)	−6(4)	210(15)	25(9)	16(2)
C(7)	1059(8)	2043(18)	4453(5)	59(6)	−53(20)	−5(6)	194(26)	−14(13)	15(3)

Atom	x	y	z	B (Å^2)	Atom	x	y	z	B (Å^2)
C(111)	−901(4)	888(7)	2915(2)	3.6(2)	C(211)	1979(3)	1196(6)	920(3)	2.8(1)
C(112)	−695(4)	−77(6)	3284(4)	4.8(2)	C(212)	2703(3)	1386(5)	961(3)	3.6(2)
C(113)	−706(4)	−64(6)	3987(4)	5.7(3)	C(213)	3141(2)	758(6)	557(3)	4.9(2)
C(114)	−922(4)	913(7)	4320(2)	6.7(3)	C(214)	2856(3)	−59(6)	112(3)	5.0(2)
C(115)	−1129(4)	1878(6)	3950(4)	7.4(3)	C(215)	2133(3)	−249(5)	71(3)	4.3(2)
C(116)	−1119(4)	1866(6)	3247(4)	5.1(2)	C(216)	1694(2)	378(6)	475(3)	3.2(1)
C(121)	−1470(4)	1993(6)	1702(4)	3.6(2)	C(221)	1930(3)	2283(6)	2207(2)	2.8(1)
C(122)	−1254(3)	3128(7)	1671(4)	5.4(2)	C(222)	2311(3)	1372(4)	2491(3)	3.5(2)
C(123)	−1732(4)	3973(5)	1460(4)	6.9(3)	C(223)	2682(3)	1507(5)	3109(3)	4.1(2)
C(124)	−2426(4)	3682(6)	1282(4)	6.6(3)	C(224)	2671(3)	2554(6)	3444(2)	4.4(2)
C(125)	−2642(3)	2547(7)	1313(4)	6.3(3)	C(225)	2290(3)	3464(4)	3160(3)	4.9(2)
C(126)	−2164(4)	1702(5)	1522(4)	5.4(2)	C(226)	1919(3)	3329(5)	2542(3)	3.9(2)
C(131)	−1252(6)	−458(7)	1739(4)	3.4(2)	C(231)	1344(4)	3417(5)	1004(3)	3.2(2)
C(132)	−1055(4)	−996(8)	1150(3)	3.8(2)	C(232)	1893(3)	3753(6)	607(4)	4.5(2)
C(133)	−1379(5)	−2013(6)	943(3)	5.1(2)	C(233)	1867(3)	4807(6)	281(3)	5.7(2)
C(134)	−1900(6)	−2492(7)	1325(4)	6.8(3)	C(234)	1291(4)	5526(5)	351(3)	6.0(2)
C(135)	−2097(4)	−1952(8)	1914(3)	6.9(3)	C(235)	742(3)	5190(6)	747(4)	6.2(3)
C(136)	−1773(5)	−936(6)	2120(3)	5.4(2)	C(236)	769(3)	4136(6)	1073(3)	5.2(2)

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[a] All values × 10^4, apart from isotropic temperature factors. [b] The b_{ij} values are the coefficients of $\exp -(h^2b_{11} + k^2b_{22} + l^2b_{33} + hkb_{12} + hlb_{13} + klb_{23})$.

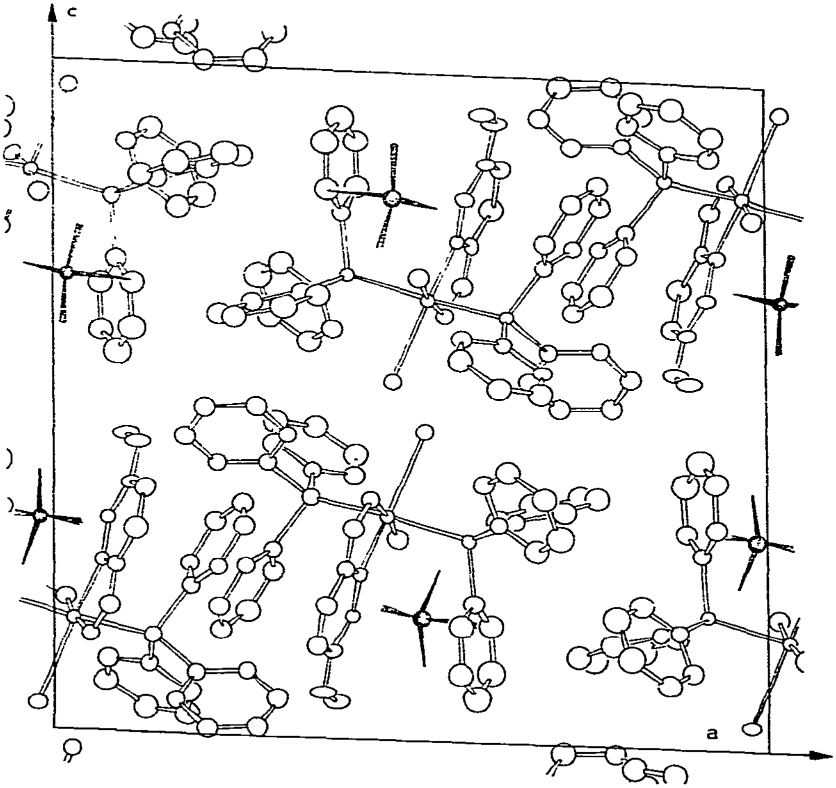

Fig. 1. ORTEP view of the molecular packing in $Ir(p\text{-}CH_3C_6H_3NNH)HI(PPh_3) \cdot CHCl_3$ (b axis downward).

$(25 - 0.22F_0 + 0.0012\,F_0^2)^{-1}$. The final reliability indices were: $R = 0.034$ and R', $[\Sigma w(F_0 - k|F_c|)^2/wF_0^2]^{1/2}$, 0.043 *. The atomic parameters obtained in the last cycle of refinement are listed in Table 1.

Computations

All computations were carried out on a UNIVAC 1108 computer. All the programmes used were well checked items of our own library, partly derived from entries 7528, 7531, 7535 of the '1966 World List of Crystallographic Programs'. Johnson's ORTEP has been used in preparing the drawings.

Description of the structure and discussion

The crystal structure consists of discrete and orderly packed complex molecules; there are cavities in the packing, approximately at ($\frac{1}{2}$, 0, $\frac{1}{4}$), which are occupied by disordered solvent molecules $CHCl_3$. The packing, which is shown in Fig. 1, is dictated by a number of H···H contacts, none of which is shorter than 2.2 Å.

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* A list of computed and observed structure factor moduli can be obtained on application to the authors.

TABLE 2

SELECTED BOND DISTANCES (Å) AND ANGLES (deg) WITH esd s ON LAST SIGNIFICANT DIGITS FOR Ir(p-$CH_3OC_6H_3NNH$)HI(PPh_3)$_2$

(a) within the coordination sphere				*(c) within the phosphine ligands*			
Ir—I	2.786(1)	I—Ir—N(1)	93.8(2)	P(1)—C(111)	1.837(6)	Ir—P(2)—C(211)	118.0(3)
Ir—P(1)	2.316(2)	I—Ir—C(2)	169.7(3)	P(1)—C(121)	1.849(8)	Ir—P(2)—C(221)	111.9(2)
Ir—P(2)	2.326(2)	P(1)—Ir—P(2)	165.2(1)	P(1)—C(131)	1.822(10)	Ir—P(2)—C(231)	115.7(3)
Ir—N(1)	2.076(8)	P(1)—Ir—N(1)	96.4(2)	P(2)—C(211)	1.832(7)	P(1)—C(111)—C(114)	176.6(5)
Ir—C(2)	2.012(9)	P(1)—Ir—C(2)	90.9(3)	P(2)—C(221)	1.834(6)	P(1)—C(121)—C(124)	179.1(5)
I—Ir—P(1)	90.1(1)	P(2)—Ir—N(1)	98.3(2)	P(2)—C(231)	1.839(7)	P(1)—C(131)—C(134)	178.9(5)
I—Ir—P(2)	90.3(1)	P(2)—Ir—C(2)	91.2(3)	Ir—P(1)—C(111)	113.1(3)	P(2)—C(211)—C(214)	178.5(3)
		N(1)—Ir—C(2)	75.9(4)	Ir—P(1)—C(121)	113.7(3)	P(2)—C(221)—C(224)	174.7(4)
				Ir—P(1)—C(131)	118.0(4)	P(2)—C(231)—C(234)	179.7(5)
(b) within the diazene ligand				*(d) within the chloroform molecule*			
N(1)—N(2)	1.265(12)	N(1)—N(2)—C(1)	110.6(9)	C(8)—Cl(1)	1.49(7)	Cl(1)—C(8)—C(13)	111(3)
N(2)—C(1)	1.379(14)	N(2)—C(1)—C(2)	119.7(9)	C(8)—Cl(2)	2.04(6)	Cl(1)—C(8)—Cl(4)	127(2)
C(1)—C(2)	1.388(16)	N(2)—C(1)—C(6)	118.2(11)	C(8)—Cl(3)	1.58(5)	Cl(2)—C(8)—Cl(3)	86(2)
C(1)—C(6)	1.411(16)	C(2)—C(1)—C(6)	122.1(10)	C(8)—Cl(4)	1.62(6)	Cl(2)—C(8)—Cl(4)	93(3)
C(2)—C(3)	1.420(14)	C(1)—C(2)—C(3)	119.0(9)	Cl(1)—C(8)—Cl(2)	100(4)	Cl(3)—C(8)—Cl(4)	121(4)
C(3)—C(4)	1.403(16)	C(2)—C(3)—C(4)	117.4(12)				
C(4)—C(5)	1.389(23)	C(3)—C(4)—C(5)	122.7(10)				
C(5)—C(6)	1.364(18)	C(4)—C(5)—C(6)	119.9(12)				
C(4)—O	1.358(14)	C(3)—C(4)—O	121.9(13)				
O—C(7)	1.412(25)	C(5)—C(4)—O	115.4(11)				
Ir—N(1)—N(2)	120.7(7)	C(4)—O—C(7)	119.2(10)				
Ir—C(2)—C(1)	113.0(7)						

TABLE 3

COMPARISON OF RELEVANT BOND LENGTHS IN SOME DOUBLY BENT ARYLDIAZENE AND ARYLDIAZENATO COMPLEXES OF Ir, Rh AND Pt

	I [a]	II [b]	III [c]	IV [d]	V [e]	VI [f]
M—N(1)	2.076(8)	1.990(7)	2.04(3)	1.973(7)	1.961(7)	1.975(28)
M—C(2)	2.012(9)	2.011(8)	2.01(3)			
N(1)—N(2)	1.265(12)	1.272(9)	1.28(3)	1.235(10)	1.172(9)	1.17(3)
N(2)—C(1)	1.379(14)	1.376(12)	1.39(3)	1.410(12)	1.445(11)	1.49(3)
M—P	2.316(2)	2.376(2)	2.399(8)	2.314(2)	2.368(3)	2.302(4)
	2.326(2)	2.364(2)	2.373(9)	2.326(2)	2.352(3)	
					2.274(3)	

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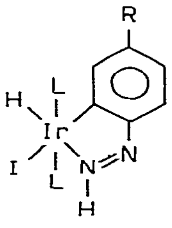
[a] Title compound. [b] $Ir(p\text{-}CH_3OC_6H_3NNH)Cl_2(PPh_3)_2$ (see ref. 2). [c] $[Ir(p\text{-}FC_6H_3NNH)(CO)Cl(PPh_3)_2]^+$ (see ref. 5). [d] $[Pt(p\text{-}FC_6H_4NNH)Cl(PEt_3)_2]^+$ (see ref. 17). [e] $[Rh(C_6H_5N_2)ClPhP((CH_2)_3PPh_2)_2]^+$ (see ref. 21). [f] $Pt(p\text{-}FC_6H_4N_2)Cl(PEt_3)_2$ (see ref. 22).

This disordered arrangement of the trichloromethane molecules is interpreted in terms of disordered tetrahedra centred by a carbon atom and having as apices four chlorine atoms of weight $\frac{3}{4}$. Distances and angles within this mean molecule display quite a large dispersion (see Table 2); moreover, the mean C—Cl distance of 1.69 Å is too short compared with the expected value of 1.767(2) Å in gaseous $CHCl_3$ [14]. These facts show that the model of disorder is not completely adequate.

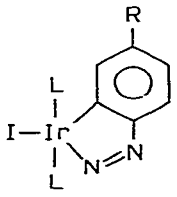
Three Fourier difference syntheses have been computed at the end of the refinement, based upon different ranges of $\sin^2\theta$, in order to establish the position of the hydrogen atoms bonded to IR and to N(1) (NMR spectroscopy [15] and structural results [2] indicate that the protonation of aryldiazenato ligands occur at this position). The syntheses show only spurious peaks whose height and position vary impredictably; the maximum peak (height 1.1 e Å^{-3} in the widest synthesis) is found at the position of Ir atom. The failure to localise the two H atoms is due to a poor signal-to-noise ratio, caused by the presence of two heavy atoms in the complex and by the disorder.

The title formula, suggested on the basis of spectroscopic results [8] cannot therefore be confirmed unambiguously by the X-ray analysis. However, the structural parameters listed in Table 2 and comparison of these with the corresponding parameters reported in Table 3 for a series of closely related compounds of Rh, Ir and Pt, fully support the view that the present compound is a hydrido diazene complex, as discussed below.

Since I is diamagnetic, both hydrogen atoms must be simultaneously present or absent; in other words, two formulae are possible: Ia, a hexacoordinated aryldiazene or Ib, a pentacoordinated aryldiazenato species:



(Ia)



(Ib)

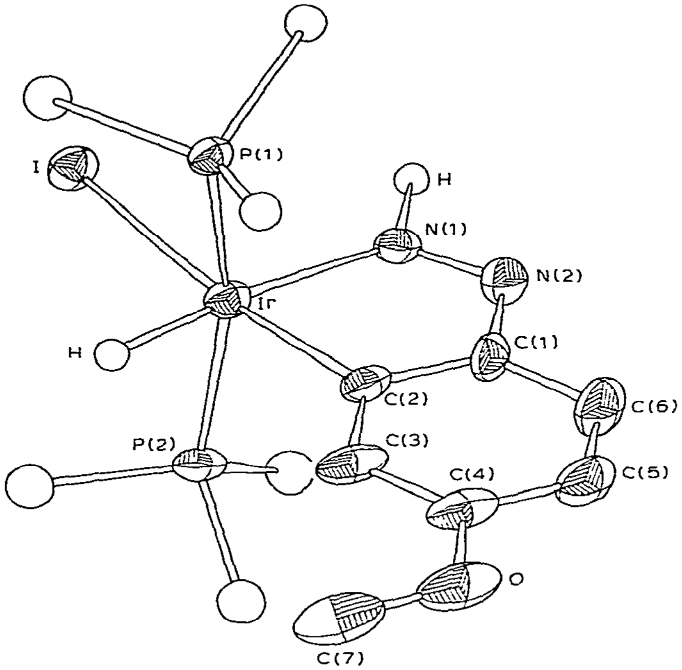


Fig. 2. ORTEP perspective view of the Ir(p-$CH_3C_6H_3NNH$)HI(PC_3)$_2$ moiety. The hydrogen atoms bonded to Ir and N(1) are shown in their postulated positions.

Formula Ib would imply either a trigonal bipyramidal or a square pyramidal coordination with a pattern of bond angles around the metal atom widely different, in both cases, from that actually observed. The experimental angles of Table 2 strongly suggest the octahedral coordination required by formula Ia with a hydrido ligand *trans* to N(1) as depicted in Fig. 2. Major departures from the ideal geometry are: the angle N(1)—Ir—C(2), 75.9(4)°, which is constrained to this value by the chelate ring geometry and the angle P(1)—Ir—P(2), 165.2(1)°, which will be discussed later.

In the series of diazene and diazenato complexes referred to in Table 4, except for compounds I and III, the distances M—N(1) fall into the narrow range 1.961(7)—1.990(7) Å; this shows that protonation at N(1) has little if any effect on the length of that bond. In compound I however, a longer interaction is found (2.076(8) Å), which can only be due to the *trans* influence of the hydrido ligands. Since many experimental results exclude the presence of two labilising ligands mutually *trans* to each other [16], the labilisation of the M—N(1) bond confirms also that N(1) has been protonated: the resulting diazene has no *trans* influence [17] whereas a diazenato ligand has an effect comparable to that of hydrogen or of a σ-bonded carbon atom. Compare for instance, the Pt—Cl bond *trans* to N(1) in compound IV, a diazene, and VI, a diazenato complex; the values are 2.291(2) and 2.413(6) Å respectively. The latter length agrees well with that found in *trans*-PtHCl(PPh_3)$_2$, 2.422(9) Å [18]. The difference between a diazene and a highly *trans* influent species can also be appreciated by comparing the two Ir—Cl distances in the orthometallated species II in which

TABLE 4

BEST PLANES WITHIN THE IrI(p-$CH_3OC_6H_3NNH$) MOIETY

Equations of best planes through specified atoms, in the form $Ax + By + Cz + D = 0$

	A	B	C	D
Plane (1): Ir, I, N(1), C(2)	−17.676	−3.192	5.765	−0.035
Plane (2): Ir, I, N(1), N(2), C(1), C(2)	−17.697	−3.158	5.747	−0.036
Plane (3): C(1), C(2), C(3), C(4), C(5), C(6)	−17.690	−3.202	5.695	−0.023

Distances (Å × 10^3) of atoms from the best planes:

	1	2	3
Ir	0(0)	0(0)	1(0)
I	0(1)	5(1)	8(1)
N(1)	0(7)	6(7)	1(7)
N(2)	18(8)	8(8)	17(9)
C(1)	5(10)	4(10)	1(10)
C(2)	3(9)	2(9)	4(9)
C(3)	18(10)	14(10)	6(10)
C(4)	18(11)	10(11)	2(11)
C(5)	0(13)	11(13)	4(13)
C(6)	21(12)	8(12)	11(12)
O	19(9)	11(9)	2(9)

the chlorine atoms are *trans* to N(1) and C(2), respectively (2.393(2) and 2.493(2) Å).

Protonation at N(1) makes the N(1)—N(2) distance closely similar to those found in compounds II and III; the three values agree within one esd and indicate a typical double bond. In the diazenato species V and VI a significantly shorter bond is observed. Chelate rings in II and III also agree well with the ring observed here.

The *trans* influence of the C(2) atom is apparent if the present Ir—I distance of 2.786(2) Å is compared with the values found in IrI_2(COOMe)(CO)(dipy) [19], where the two iodine atoms are *trans* each to the other (2.672(2) and 2.684(2) Å), and in the cation $[IrI(CO)(NO)(PPh_3)_2]^+$, (2.666(3) Å) [20].

In complex I, the equatorial plane containing the chelate ring involves two thermodynamically labilized metal—ligand interactions, a pure σ bond (Ir—H), and a Ir—C bond which is also predominantly σ in character. This situation increases the availability of the donor and acceptor orbitals on the metal atom; understandably then, the two Ir—P bonds are shorter than those in II and III.

As can be seen from the angular parameters reported in Table 2 and from Fig. 2, both the Ir—P bonds are bent away from the chelate ring, toward the position postulated for the hydrido ligand. This happens because of the thickness of the aryldiazene and of the short non-bonding radius of hydrogen. As a result of this bending, the planarity of the diazene iridium moiety is verified very accurately, as can be seen in Table 4. In compound II, in which a much less significant bending of the angle P(1)—Ir—P(2) is found (176.2(1)°), the deviation from planarity of that part of the molecule are more pronounced.

In neutral iridium(III) orthometallated diazene complexes two different distances M—N(1) have been found: a "normal" one, present in compound II, and a labilised one, found in the title compound. In the cationic complex III, which has a carbonyl group *trans* to N(1), the Ir—N(1) distance is not statistically distinguishable from either the normal and the labilised interaction. Outline elucidations of the structures of fluorocarbonyl diazene cations have been reported by us and by Sutton [3]. The structure of one of these compounds is being accurately refined in our laboratory in order to provide precise information on the type of metal—nitrogen interaction involved.

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