A Homoleptic Mononuclear Iridium(II) Organometallic Complex: Synthesis and X-ray Structure of $[Ir(C_6Cl_5)_4]^{2-}$

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Summary: The homoleptic iridium(II) dianionic complex $[Ir(C_6Cl_5)_4]^{2-}$ *has been prepared from* $[\{Ir(\mu\text{-}Cl)(cyclooctene)_2\}_2]$ *and* $Li(C_6Cl_5)$ *in* Et_2O *at −50 °C. It reacts with carbon monoxide affording the iridium(I) complex* $[Ir(C_6Cl_5)_3(CO)]^{2-}$*. Both dianionic complexes have been fully characterized by elemental analyses, by MS, and by several physical properties. The crystal structure of* $[PBzPh_3]_2[Ir(C_6Cl_5)_4]\cdot CH_2Cl_2$ *(1) has been established by an X-ray diffraction study. 1 crystallizes in the monoclinic space group C2/c with* $a = 19.355(8)$ Å, $b = 18.188(6)$ Å, $c = 22.691(9)$ Å, $\beta = 102.82(2)°$, $V = 7789(5)$ Å^3, $Z = 4$, $\mu = 25.53$ cm^{-1}, *and* $d_{calc} = 1.690$ g·cm^{-3}*. The anionic iridium(II) complex has an imposed* C_2 *symmetry with the 2-fold axis bisecting two pentachlorophenyl groups. The coordination around the Ir atom is almost perfectly square planar. The dispositions of the aryl rings with respect to the metal coordination plane are all inclined in the same way conforming a propeller-like shape for the iridium(II) complex.*

Introduction

Mononuclear rhodium and iridium(II) complexes are a rarity,[1] although in the last few years some work has been done on the subject, either on the coordination chemistry or in the organometallic chemistry area.[2–5] One of our current research interests is the chemistry of pentachlorophenyl derivatives of rhodium and iridium, and we have reported that the reaction of $[\{Rh(\mu\text{-}Cl)(L_2)\}_2]$ [L_2 = 1,5-cyclooctadiene (cod) or $\{P(OPh)_3\}_2$] with LiC_6Cl_5 gives compounds of the type $[NBu_4][Rh(C_6Cl_5)_2(L_2)]$ in which anionic rhodium(I) complexes are present. Oxidation of these anionic complexes leads to monomeric, neutral, paramagnetic rhodium(II) compounds.[6] In the same way, the reaction of $[\{Ir(\mu\text{-}Cl)(cod)\}_2]$ with LiC_6Cl_5 gives the anionic iridium(I) complex $[Ir(C_6Cl_5)_2(cod)]^-$ which by oxidation with chlorine, iodine, or Ag^+ yields the iridium(II) complex $[Ir(C_6Cl_5)_2(cod)]$ previously structurally characterized.[7] By contrast, when the iridium dimer $[\{Ir(\mu\text{-}Cl)(C_8H_{14})_2\}_2]$ (C_8H_{14} = cyclooctene) is used as the starting product, we have obtained the homoleptic dianion iridium(II) $[Ir(C_6Cl_5)_4]^{2-}$ complex. In this note we wish to report the preparation and characterization of this dianion as well as its reduction to an iridium(I) complex by reaction with carbon monoxide.

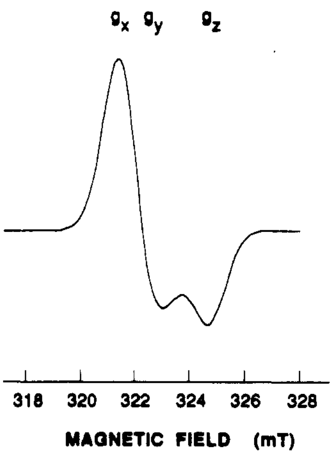

Figure 1. EPR spectrum of a frozen solution of 1 in THF, measured at 77 K.

Results and Discussion

The reaction of an excess of $Li(C_6Cl_5)$ with $[\{Ir(\mu\text{-}Cl)(cyclooctene)_2\}_2]$ in Et_2O, at −50 °C, leads to the formation of $[Ir(C_6Cl_5)_4]^{2-}$; the anionic complex is isolated as the yellow microcrystalline solids $[PBzPh_3]_2[Ir(C_6Cl_5)_4]$ (1) and $[NBu_4]_2$ $[Ir(C_6Cl_5)_4]$ (2) after the addition of bulky cations. These compounds are not stable to the air, but they can be kept under nitrogen for short periods without decomposition being observed; in deoxygenated acetone solutions (approximately 5×10^{-4} M), they behave as 2:1 electrolytes.[8] The analytical data and IR and NMR spectra indicate that the complexes contain C_6Cl_5 groups and that the olefin ligand has been displaced. The mass spectrum (FAB) of compound 2 shows the parent peak of the anion (*m/z* 1189, 21%). The EPR spectra of polycrystalline powdered samples as well as of frozen THF solutions of 1 and 2 have been measured in the X band at 77 K. The spectra are very similar, the only difference being the bigger

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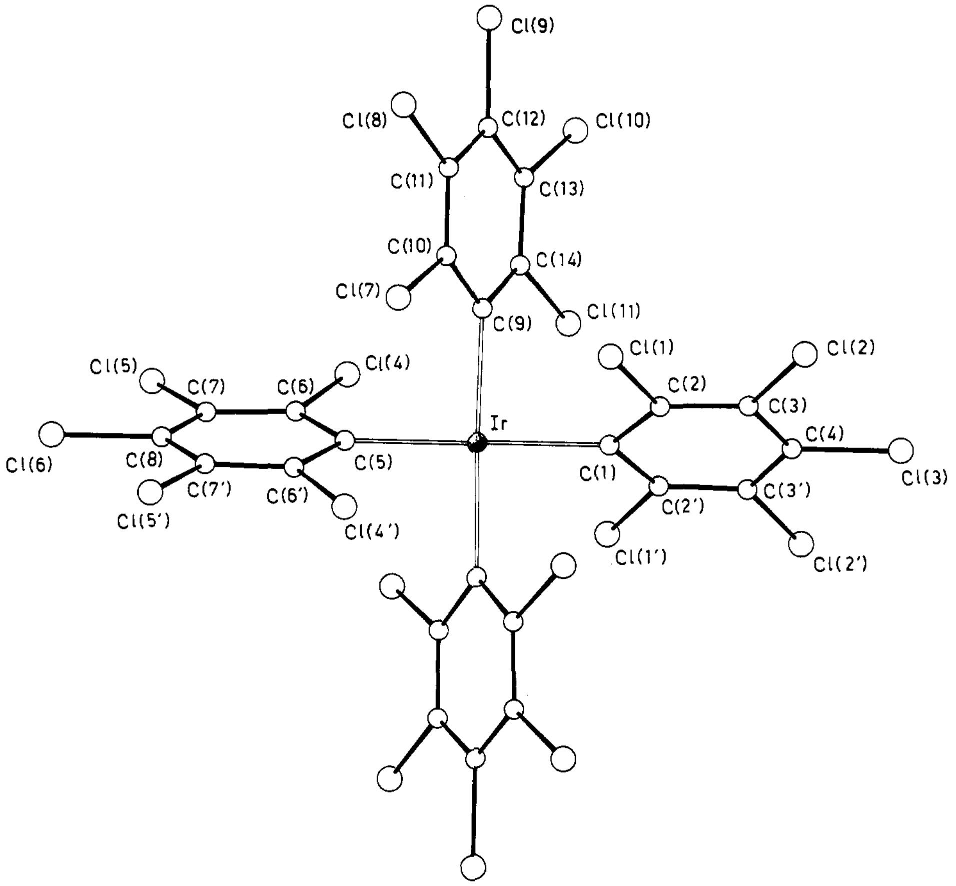

Figure 2. View of the structure of the anionic complex $[Ir(C_6Cl_5)_4]^{2-}$ together with the atomic numbering scheme.

Table I. Bond Distances (Å) and Selected Angles (deg) for the Anionic Complex $[Ir(C_6Cl_5)_4]^{2-}$ [a]

Ir–C(1)	2.090(10)	C(12)–C(13)	1.371(10)
Ir–C(5)	2.087(10)	C(13)–C(14)	1.399(11)
Ir–C(9)	2.116(7)	C(2)–Cl(1)	1.739(6)
C(1)–C(2)	1.412(8)	C(3)–Cl(2)	1.725(7)
C(2)–C(3)	1.397(9)	C(4)–Cl(3)	1.711(10)
C(3)–C(4)	1.390(8)	C(6)–Cl(4)	1.737(7)
C(5)–C(6)	1.417(9)	C(7)–Cl(5)	1.731(7)
C(6)–C(7)	1.397(10)	C(8)–Cl(6)	1.735(11)
C(7)–C(8)	1.377(9)	C(10)–Cl(7)	1.726(7)
C(9)–C(10)	1.399(9)	C(11)–Cl(8)	1.725(7)
C(9)–C(14)	1.403(9)	C(12)–Cl(9)	1.724(8)
C(10)–C(11)	1.393(11)	C(13)–Cl(10)	1.722(7)
C(11)–C(12)	1.395(10)	C(14)–Cl(11)	1.739(7)
C(1)–Ir–C(9)	89.2(2)	C(6)–C(5)–C(6′)	112.2(4)
Ir–C(1)–C(2)	123.8(3)	Ir–C(9)–C(10)	123.7(5)
C(2)–C(1)–C(2′)	112.5(4)	Ir–C(9)–C(14)	123.0(5)
Ir–C(5)–C(6)	123.9(3)	C(10)–C(9)–C(14)	113.3(6)

[a] Primed atoms are related to the unprimed ones by the symmetry transformation $^1/_2 - x, y, ^1/_2 - z$.

width of the polycrystalline signals. In Figure 1 is shown the spectrum of the frozen solution of 1. No resolved hyperfine structure is observed, and the EPR spectra can be understood with a standard Hamiltonian including only a electronic Zeeman contribution with $g_x = 2.131$, $g_y = 2.126$, and $g_z = 2.110$. Compound 2 has also been studied by cyclic voltammetry. When an increasing voltage (−1.2 to +1.2 V with sweep rates of 0.05, 0.1, or 0.2 V s^{-1}) is applied to CH_2Cl_2 solutions (0.5 mM) of 2, only an irreversible oxidation wave at 0.20 V is observed.

When carbon monoxide is bubbled through a dichloromethane solution of 2, an immediate reaction takes place. The IR spectrum of the solution shows the presence of coordinated CO; it presents an absorption ν(CO) at 1965 cm^{-1}; the position of this band is indicative of a reduction of the metal to iridium(I). The complex is crystallized as a yellow solid by the addition of diethyl ether under a CO atmosphere. The analytical, MS (FAB, parent peak, M^-, *m/z* 969, 42%), and conductivity (in approximately 5×10^{-4} M acetone solution, 2:1) data agree with the formulation of the complex as $(NBu_4)_2[Ir(C_6Cl_5)_3(CO)]$ (**3**). The ^{13}C NMR spectrum, in HDA, indicates that there are two different C_6Cl_5 groups, as expected for the substitution of a C_6Cl_5 group by a CO ligand.

The complete characterization of the anionic iridium(II) complexes comes from an X-ray diffraction study of the dichloromethane solvate of 1. In the crystals of 1, $[Ir(C_6Cl_5)_4]^{2-}$ anions, $[PBzPh_3]^+$ cations, and dichloromethane molecules of solvation are present. The structure of the anion is shown in Figure 2, together with the atomic labeling system; the most significant bond distances and angles are given in Table I. The anionic complex has a crystallographically imposed C_2 symmetry with the 2-fold axis bisecting two opposed pentachlorophenyl rings. The Ir atom, bonded to four C atoms from the C_6Cl_5 ligands, is in an almost perfect square planar arrangement [the C(1)–Ir–C(9) angle is 89.2(2)°]. The Ir–C bond distances, in the range 2.087(10)–2.116(7) Å, compare well with those found for related iridium(II) complexes containing coordinated aryl groups: $[Ir(C_6Cl_5)_2(cod)]$ (2.073(5) Å),[7] $[Ir(mes)_2(SEt_2)_2]$ (2.092(11) Å),[3] and $[Ir(mes)_2(PPh_3)_2]$ (2.112(9) and 2.097(9) Å).[3] The values of these distances are also comparable with the Pt–C ones found in the isoelectronic and isostructural platinum(III) complex $[Pt(C_6Cl_5)_4]^-$ (mean value 2.093(4) Å).[9] All the pentachlorophenyl groups are roughly planar with the Cl atoms slightly deviating from the mean planes [maximum

Table II. Summary of Crystallographic Data for Complex 1

mol formula	$[C_{25}H_{22}P]_2[C_{24}Cl_{20}Ir]\cdot CH_2Cl_2$
mol wt	1981.32
cryst syst	monoclinic
space group	$C2/c$
radiation (λ, Å)	Nb-filtered Mo Kα (0.710 73)
a, Å	19.355(8)
b, Å	18.188(6)
c, Å	22.691(9)
β, deg	102.82(2)
V, Å^3	7789(5)
Z	4
D_{calcd}, g cm^{-3}	1.690
$F(000)$	3908
cryst dimens, mm	0.17 × 0.24 × 030
μ, cm^{-1}	25.53
$R(F_o)^a$	0.0351
$R_w(F_o)^b$	0.0402

^{a} $R = \sum||F_o| - |F_c||/\sum|F_o|$. ^{b} $R_w = [\sum\omega(|F_o| - |F_c|)^2/\sum\omega(F_o)^2]^{1/2}$.

deviation 0.098(2) Å for the Cl(5) atom]. The aryl rings are not coplanar with the metal coordination plane and are all rotated in the same way, conforming a propeller-like shape for the whole complex [dihedral angles in the range 59.5(1)-65.8(2)°]. No short Ir–Cl contacts (minimum value for the Ir–Cl separation: 3.399(2) Å] are observed, in contrast to those of $[Ir(C_6Cl_5)_2(cod)]$ [shortest Ir–Cl separation: 3.077(1) Å] in which the two pentachlorophenyl rings were disposed almost perpendicular to the coordination plane and slightly twisted in order to interact with the metal.[7] The disposition of the pentachlorophenyl rings in 1 is very similar to that observed in the closely related $[Pt(C_6Cl_5)_4]^-$ or $[Pt(C_6Cl_5)_4]^{2-}$ complexes (values for the dihedral angles of 63°).[9]

Experimental Section

General Data. C, H, and N analyses, conductance determinations, and IR and NMR spectra were performed as described elsewhere.[10] MS were measured in a VG Autospec double focusing mass spectrometer operating in the negative mode; ions were produced with the standard Cs^+ gun at ca. 30 kV; 3-nitrobenzyl alcohol (NBA) was used as the matrix; high resolution FAB spectra are in accordance with the simulated isotopic pattern distribution. Cyclic voltammetric experiments were performed by employing an EG&G PARC Model 273 potentiostat. A three electrode system was used, which consists of a platinum disk working electrode, a platinum wire auxiliary electrode and a saturated calomel reference electrode. The measurements were carried out in CH_2Cl_2 solutions with 0.1 M Bu_4NPF_6 as supporting electrolyte. Under the present experimental conditions the ferrocenium/ferrocene couple was located at 0.47 V. EPR data were taken in a Varian E-112 spectrometer working in the X band. The samples were introduced in a standard EPR quartz tube (707-SQ from Wilmad), and their spectra were taken at 77 K. The magnetic field was measured with a NMR gaussmeter Bruker ER035M and the diphenylpicrylhydrazyl (DPPH) resonance signal (g = 2.0037 ± 0.0002) was used for determining the microwave frequencies. The starting iridium(I) dimer $[\{Ir(\mu\text{-}Cl)(cyclooctene)_2\}_2]$ was prepared as described in the literature.[11] LiC_6Cl_5 was obtained as described in ref 12 and was directly used without further isolation. Diethyl ether, CH_2Cl_2, and methanol were distilled under nitrogen from the appropriate drying agents.

Table III. Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Thermal Parameters (Å^2 × 10^4) with Esd's in Parentheses for the Non-Hydrogen Atoms of Complex 1

	x/a	y/b	z/c	$U_{eq}{}^a$
Ir	5000	−1552(1)	2500	259(1)
Cl(1)	4247(1)	−408(1)	1336(1)	461(6)
Cl(2)	4319(1)	1276(1)	1332(1)	553(7)
Cl(3)	5000	2127(1)	2500	523(11)
Cl(4)	6436(1)	−2695(1)	2875(1)	508(7)
Cl(5)	6410(1)	−4375(1)	2941(1)	600(8)
Cl(6)	5000	−5237(2)	2500	729(15)
Cl(7)	4498(1)	−2264(1)	1060(1)	465(6)
Cl(8)	5363(1)	−2253(1)	85(1)	584(8)
Cl(9)	6840(1)	−1517(2)	319(1)	791(10)
Cl(10)	7438(1)	−749(1)	1552(1)	668(9)
Cl(11)	6614(1)	−782(1)	2555(1)	510(7)
P1	3754(1)	3565(1)	3777(1)	354(6)
C(1)	5000	−402(5)	2500	303(28)
C(2)	4687(3)	29(4)	1994(3)	334(21)
C(3)	4692(4)	797(4)	1981(3)	371(20)
C(4)	5000	1186(5)	2500	362(34)
C(5)	5000	−2699(5)	2500	349(33)
C(6)	5621(3)	−3134(4)	2666(3)	345(21)
C(7)	5625(4)	−3901(4)	2686(3)	405(26)
C(8)	5000	−4283(6)	2500	437(39)
C(9)	5570(3)	−1536(4)	1805(3)	321(19)
C(10)	5324(3)	−1857(4)	1237(3)	347(22)
C(11)	5699(4)	−1852(4)	779(3)	402(25)
C(12)	6362(4)	−1516(5)	876(3)	441(22)
C(13)	6630(4)	−1188(4)	1424(3)	400(24)
C(14)	6236(3)	−1201(4)	1871(3)	350(23)
C(15)	4697(3)	3687(4)	4005(3)	375(22)
C(16)	4983(4)	4280(5)	4368(4)	579(32)
C(17)	5701(4)	4403(5)	4491(5)	677(36)
C(18)	6141(4)	3942(5)	4267(4)	629(35)
C(19)	5862(4)	3355(5)	3917(4)	611(35)
C(20)	5141(4)	3227(4)	3784(3)	447(24)
C(21)	3559(4)	2728(4)	3351(4)	418(26)
C(22)	3250(4)	2739(5)	2745(4)	587(31)
C(23)	3093(5)	2086(6)	2428(5)	784(42)
C(24)	3232(5)	1432(6)	2724(6)	840(50)
C(25)	3536(5)	1411(5)	3314(6)	785(44)
C(26)	3684(5)	2070(5)	3649(4)	600(35)
C(27)	3344(3)	3541(4)	4408(3)	390(21)
C(28)	3730(4)	3599(4)	5000(3)	505(29)
C(29)	3374(5)	3622(5)	5468(4)	653(34)
C(30)	2645(5)	3587(5)	5344(4)	707(42)
C(31)	2268(5)	3508(6)	4761(5)	763(38)
C(32)	2610(4)	3481(6)	4292(4)	604(30)
C(33)	3386(4)	4323(4)	3290(3)	435(24)
C(34)	3351(4)	5052(4)	3598(3)	438(24)
C(35)	3900(5)	5564(5)	3638(4)	662(36)
C(36)	3838(7)	6243(6)	3884(5)	865(50)
C(37)	3272(7)	6423(5)	4085(5)	856(53)
C(38)	2739(6)	5938(6)	4061(4)	748(39)
C(39)	2769(4)	5246(5)	3809(4)	588(34)
Cl(12)	4315(3)	331(3)	4720(3)	2481(22)^b

^{a} Equivalent isotropic u_{eq} defined as one-third of the trace of the orthogonalized u_{ij} tensor. ^{b} Isotropic thermal parameter.

Synthesis of $Q_2[Ir(C_6Cl_5)_4]$ (Q = $PBzPh_3$, 1, or NBu_4, 2). Solid iridium(I) dimer $[\{Ir(\mu\text{-}Cl)(cyclooctene)_2\}_2]$ (200 mg, 0.22 mmol) was added to diethyl ether solutions of LiC_6Cl_5 (2.1 mmol) at −50 °C. The mixture was allowed to warm slowly to 0 °C while the stirring was continued for about 3 h; during this time the starting solid was dissolved and a brown solution was formed. The solution was treated with NBu_4Br (284 mg, 0.88 mmol) or $PBzPh_3Cl$ (350 mg, 0.90 mmol) together with 20 mL of aqueous diethyl ether, yielding an oily yellow solid. The mixture was concentrated till 20 mL and the solid was separated by decantation; after being washed with four fractions of diethyl ether/methanol (20 mL, 1:1) the complex was dried under vacuo and kept under nitrogen at −5 °C. Yield: 1, 34%; 2, 28%. Anal. Calcd for $[NBu_4]_2[Ir(C_6Cl_5)_4]$, 2: C, 40.17; H, 4.33; N, 1.67. Found: C, 39.96; H, 4.35; N, 1.60. IR (Nujol suspension between polyethylene sheets); $\nu(C_6Cl_5)$ (cm^{-1}): 1315 (sh), 1300 (s), 1290

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(s), 880 (s), and 580 (s). Mass spectrum, *m/z* (%): 1189 (21) [M$^-$, Ir(C_6Cl_5)$_4$], 941 (100) [Ir(C_6Cl_5)$_3$], 691 (100) [Ir(C_6Cl_5)$_2$]. Conductivity (in 3.6 × 10^{-4} M acetone solution): 191 Ω^{-1} cm^2 mol^{-1}.

Synthesis of [NBu_4]$_2$[Ir(C_6Cl_5)$_3$(CO)] (3). Carbon monoxide was bubbled through a solution of complex [NBu_4]$_2$[Ir(C_6Cl_5)$_4$] (100 mg, 0.06 mmol) in dichloromethane (10 mL) for 30 min. Diethyl ether (20 mL) was added to the solution, and a yellow solid was formed while CO was passed through for another 5 min. The solid was filtered off, washed with diethyl ether/methanol (1:1), and dried (80% yield). Anal. Calcd: C, 42.15; H, 4.99; N, 1.93. Found: C, 41.83; H, 4.55; N, 1.80. IR (CH_2Cl_2), ν(CO) (cm^{-1}): 1965; (Nujol suspension between polyethylene sheets), ν(C_6Cl_5) 1315 (s), 1310 (s), 1305 (s), 1295 (s), 885 (m), 665 (s), 585 (s). ^{13}C NMR (HDA, 20 °C), δ: 184.6 (s, CO), 172.7, 163.7 (s, C_{ipso}), 145.2, 144.5, 130.0, 127.4, 126.8, 121.1 (s, C_{ortho}, C_{para}, C_{meta}), 59.1, 24.2, 20.1, and 13.6 (s, NBu_4^+). Mass spectrum, *m/z* (%): 969 (42) [M$^-$, Ir(C_6Cl_5)$_3$(CO)], 941 (28) [Ir(C_6Cl_5)$_3$], 691 (94) [Ir(C_6Cl_5)$_2$]. Conductivity (in 4.3 × 10^{-4} M acetone solution): 187 Ω^{-1} cm^2 mol^{-1}.

Crystal Structure Determination of [$PBzPh_3$]$_2$[Ir(C_6Cl_5)$_4$]·CH_2Cl_2 (1). Suitable crystals for X-ray diffraction were obtained by slow diffusion of diethyl ether, through a methanol interphase (1 mL), into a dichloromethane solution of 1. Table II collects some relevant crystallographic data. Data were collected at room temperature on a Siemens AED diffractometer, using the niobium-filtered Mo Kα radiation (λ = 0.710 73 Å) and the $\theta/2\theta$ scan type. Of 7749 unique reflections, with θ in the range 3–26°, 5398 with $I > 2\sigma(I)$ were used for the analysis. One standard reflection was monitored every 50 measurements; no significant decay was noticed over the time of data collection. The individual profiles have been analyzed following Lehmann and Larsen.[13] Intensities were corrected for Lorentz and polarization effects. No correction for absorption was applied.

The structure was solved by Patterson and Fourier methods and refined by full-matrix least squares first with isotropic thermal parameters and then with anisotropic thermal parameters for all non-hydrogen atoms of the cationic and anionic complexes. A molecule of chloroform was found in the final ΔF map with the carbon atom disordered and distributed in all positions equidistant from the Cl atoms, so no position for it could be given. All hydrogen atoms, excepting those of the solvent molecule, were placed at their geometrically calculated positions (C–H = 0.96 Å) and refined "riding" on the corresponding carbon atoms. The final cycles of refinement were carried out on the basis of 451 variables. The biggest remaining peak in the final difference map was equivalent to about 1.1 e/Å^3. Unit weights were used in all stages of the refinement. The analytical scattering factors, corrected for the real and imaginary parts of anomalous dispersions, were taken from ref 14. All calculations were carried out on the GOULD POWERNODE 6040 of the "Centro di Studio per la Strutturistica Diffrattometrica" del CNR, Parma, using the SHELX-76 and SHELXS-86 systems of crystallographic computer programs.[15] The final atomic coordinates for the non-hydrogen atoms are given in Table III.

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Supplementary Material Available: Tables of hydrogen atom coordinates (Table SI), anisotropic thermal parameters for the non-hydrogen atoms (Table SII), and crystallographic data (Table SIII) (4 pages). Ordering information is given on any current masthead page.

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