Studies on Olefin-co-ordinating Transition-metal Carbene Complexes. Part 14.* An Unusual Reaction of Tricarbonyl(η^4-cyclopenta-1,3-diene)iron: A New Approach to Acyldicarbonyl(η-cyclopentadienyl)iron Complexes, and the *X*-Ray Crystal Structure of [Fe(COC$_6$H$_4$CF$_3$-*p*)(η-C$_5$H$_5$)(CO)$_2$]†

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Reaction of tricarbonyl(η^4-cyclopenta-1,3-diene)iron (**1**) with aryl-lithium reagents, LiR (R = Ph, C_6H_4Me-*o* or -*p*, C_6H_4OMe-*p*, or $C_6H_4CF_3$-*p*), in diethyl ether at low temperature, followed by the reaction of the acylmetalate intermediates formed with Et_3OBF_4 in CH_2Cl_2 at −60 °C or in aqueous solution at 0 °C, afforded yellow crystalline compounds formulated as [Fe(COR)(η-C$_5$H$_5$)(CO)$_2$] (**2**)—(**6**) based on elemental analyses, i.r., ^{1}H n.m.r., and mass spectra. Complex (**6**) has been studied by *X*-ray diffraction: triclinic, space group $P\bar{1}$ with the unit-cell constants $a = 7.670(7)$, $b = 9.008(4)$, $c = 10.662(1)$ Å, $\alpha = 103.70(2)$, $\beta = 96.496(8)$, $\gamma = 100.03(2)°$, $Z = 2$, $R = 0.039$, and $R' = 0.045$ for 2 834 reflections with $I > 3\sigma(I)$.

In extension of our research in the field of olefin-co-ordinating metal carbene complexes, we discovered that different cyclopolyene-ligated carbonyliron compounds reacted with aryl-lithium reagents at low temperature, followed by alkylation of the acylmetalate intermediates formed with Et_3OBF_4 to give different products. For example, the reaction of 1,3-cyclohexadiene- and cyclo-octatetraene-tricarbonyliron gave a series of diallyldicarbonyliron complexes.[1,2] In the case of tricarbonyl(cycloheptatriene)iron and tricarbonyl(5,6,7,8-tetrafluoro-1,4-dihydro-1,4-ethenonaphthalene)iron a ring-opening reaction occurred to afford novel ring-opened products.[3]

In this study we chose tricarbonyl(cyclopenta-1,3-diene)iron (**1**)[4] as starting material in order to investigate further the reaction of different cyclo-olefin-ligated compounds. The results show that compound (**1**) gives unusual products, instead of the analogous diallyldicarbonyliron complexes or ring-opened products mentioned above.

Results and Discussion

Preparation of Complexes (**2**)—(**6**).—Equimolar quantities of compound (**1**) and aryl-lithium, LiR (R = Ph, C_6H_4Me-*o* or -*p*, C_6H_4OMe-*p*, or $C_6H_4CF_3$-*p*), were used for the reaction in diethyl ether at low temperature, and the acylmetalates formed were subsequently treated with Et_3OBF_4 in CH_2Cl_2 at −60 °C or in aqueous solution at 0 °C. The reaction mixtures were chromatographed on alumina at low temperature and the crude products were recrystallized from light petroleum (b.p. 30—60 °C) at −80 °C to give yellow crystalline compounds (**2**)—(**6**) with the composition [Fe(COR)(η-C$_5$H$_5$)(CO)$_2$] in moderate yields (Table 1).

The diamagnetic compounds (**2**)—(**6**) are soluble both in polar and non-polar organic solvents, and rather sensitive to air. On the basis of elemental analyses, i.r., ^{1}H n.m.r., and mass spectra (Table 2) as well as the *X*-ray crystal structure of (**6**), they are formulated as acyldicarbonyl(η-cyclopentadienyl)iron complexes, of which (**2**) is known.[5,6]

The i.r. spectra of complexes (**2**)—(**6**) in hexane solution showed two strong bands in the ν(CO) region, but as potassium chloride pellets they showed a strong band at 1 575—1 587 cm^{-1} assigned to the formyl vibration band besides the two CO bands. The i.r. data show that different aryl substituents exerted certain effects on the CO vibration frequency. In accordance with the order of increasing electron-withdrawing ability of the aryl substituents, *p*-MeOC$_6$H$_4$ < *p*-MeC$_6$H$_4$ ≈ *o*-MeC$_6$H$_4$ < Ph < *p*-CF$_3$C$_6$H$_4$, the CO absorption bands shifted to higher frequencies. The ^{1}H n.m.r. spectra showed the expected proton signals attributed to the aryl substituents and the cyclopentadienyl groups. The strong electron-withdrawing ability of the trifluoromethylphenyl group is responsible for a slight downfield shift of the cyclopentadienyl protons of complex (**6**), compared with those of (**2**)—(**5**). In the mass spectra all of the complexes showed, besides their molecular ion peaks, characteristic fragments generated by successive loss of CO ligands and other ion peaks produced by further cleavage of these fragments.

A possible reaction mechanism for the formation of complexes (**2**)—(**6**) is proposed in the Scheme. Acylmetalate intermediates (**A**) were formed when (**1**) reacted with aryl-lithiums, and were observed as orange precipitates and separated in the case of crystalline compound [Fe{C(OLi)C$_6$H$_4$-Me-*o*}(η^4-C$_5$H$_6$)(CO)$_2$]·*x*Et$_2$O formed by the reaction of (**1**) with *o*-tolyl-lithium. The i.r. spectrum of this compound showed the two expected absorption bands at 1 962vs and 1 808vs cm^{-1} (CH$_2$Cl$_2$) similar to those observed for [Fe{C(OLi)Ph}(C$_4$H$_6$)(CO)$_2$]·2Et$_2$O.[7] The η^4-cyclopentadiene ligand has a tendency to transform into η^5-cyclopentadienyl under appropriate conditions.[8,9] Since one of the hydrogen atoms at the saturated carbon of the cyclopentadiene ring in (**A**) must be relatively hydridic, such a conversion can occur in the presence of an appropriate Lewis acid which can abstract a hydridic ion from the η^4-cyclopentadiene ligand, resulting in the formation of the neutral compounds (**2**)—(**6**). We believe that the hydridic ion was abstracted not by a proton but by Et_3OBF_4, because of the following facts. (1) The gas which evolved from the resulting solution during the alkylation with Et_3OBF_4 was shown to be ethane by gas chromatography, in comparison with the gas generated by the hydrolysis of MgBr(Et). (2) When an organic acid such as acetic acid and an inorganic acid such as hydrochloric acid or dry hydrogen chloride instead of Et_3OBF_4

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* For Part 13 see J-B. Chen, J-G. Yin, G-X. Lei, Y-Y. Wang, and G-D. Lin, *Acta Chim. Sin.*, 1988, **46**, 305.

† Dicarbonyl(η-cyclopentadienyl)(*p*-trifluoromethylbenzoyl)iron.

Supplementary data available: see Instructions for Authors, *J. Chem. Soc., Dalton Trans.*, 1989, Issue 1, pp. xvii—xx.

Table 1. Physical and analytical data for acyl complexes (**2**)—(**6**)

Complex	Yield (%)	Analysis[a] (%)			M.p. (°C)
		C	H	Fe	
(**2**) $[Fe(COPh)(\eta\text{-}C_5H_5)(CO)_2]$	51	59.70 (59.60)	3.60 (3.55)	19.45 (19.80)	62—63[b]
(**3**) $[Fe(COC_6H_4Me\text{-}o)(\eta\text{-}C_5H_5)(CO)_2]$	53	61.00 (60.85)	4.25 (4.10)	18.70 (18.85)	78—79 (decomp.)
(**4**) $[Fe(COC_6H_4Me\text{-}p)(\eta\text{-}C_5H_5)(CO)_2]$	56	60.90 (60.85)	4.10 (4.10)	18.65 (18.85)	88—89 (decomp.)
(**5**) $[Fe(COC_6H_4OMe\text{-}p)(\eta\text{-}C_5H_5)(CO)_2]$	46	57.80 (57.75)	4.00 (3.90)	17.90 (17.90)	115—116 (decomp.)
(**6**) $[Fe(COC_6H_4CF_3\text{-}p)(\eta\text{-}C_5H_5)(CO)_2]$	62	51.55[c] (51.45)	2.55 (2.60)	15.75 (15.95)	86—87 (decomp.)

[a] Calculated values in parentheses. [b] Lit., 59—62[5] and 63—65 °C.[6] [c] F, 16.30 (16.30)%.

Table 2. Spectroscopic data for complexes (**2**)—(**6**)

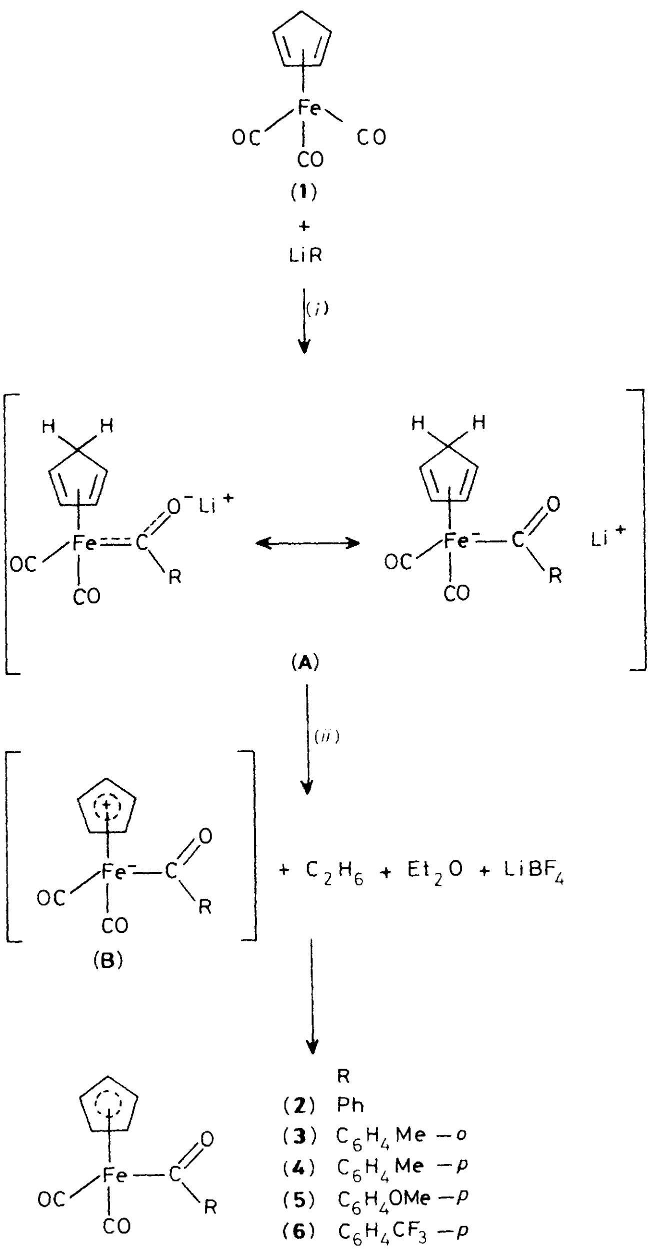
Complex	I.r. (ν_{CO}/cm^{-1})[a]	^{1}H N.m.r. (δ)[b]	Mass spectrum[c]
(**2**)	2 006vs, 1 929vs, 1 585vs (2 015vs, 1 960vs)	5.12 (s, 5 H, C_5H_5) 7.40—7.49 (m, 5 H, C_6H_5)	282 M^+ (1) 254 $[M - CO]^+$ (40) 226 $[M - 2CO]^+$ (72)
(**3**)	2 005vs, 1 928vs, 1 580s (2 010vs, 1 960vs)	2.22 (s, 3 H, CH_3) 5.11 (s, 5 H, C_5H_5) 7.04—7.30 (m, 4H, C_6H_4)	296 M^+ (1) 268 $[M - CO]^+$ (33) 240 $[M - 2CO]^+$ (24)
(**4**)	2000vs, 1 927vs, 1 575s (2 012vs, 1 955vs)	2.34 (s, 3 H, CH_3) 5.10 (s, 5 H, C_5H_5) 7.20 (d, 2 H, C_6H_4) 7.46 (d, 2 H, C_6H_4)	296 M^+ (3) 268 $[M - CO]^+$ (42) 240 $[M - 2CO]^+$ (43)
(**5**)	1 990vs, 1 927vs, 1 576s (2 010vs, 1 955vs)	3.86 (s, 3 H, OCH_3) 5.10 (s, 5 H, C_5H_5) 6.94 (d, 2 H, C_6H_4) 7.62 (d, 2 H, C_6H_4)	312 M^+ (2) 284 $[M - CO]^+$ (49) 256 $[M - 2CO]^+$ (79)
(**6**)	2 013vs, 1 956vs, 1 587s (2 020vs, 1 965vs)	5.18 (s, 5 H, C_5H_5) 7.62 (d, 2 H, C_6H_4) 7.76 (d, 2 H, C_6H_4)	350 M^+ (6) 322 $[M - CO]^+$ (59) 294 $[M - 2CO]^+$ (53)

[a] Potassium chloride pellet (in n-hexane solution). [b] In $(CD_3)_2CO$. [c] Relative intensity (%) in parentheses.

were used for the reaction with (**A**) in CH_2Cl_2 or in aqueous solution the expected product was not obtained.

Crystal and Molecular Structure of Complex (**6**).—The configuration of complex (**6**) is shown in the Figure. The results of the crystal structure analysis demonstrated that in the reaction one of the hydrogen atoms at the saturated carbon of the η^4-cyclopentadiene ligand was eliminated with the aid of the Fe atom so as to convert the η^4 ligand into an η^5 ligand.

The average C–C bond length of the cyclopentadienyl ring is 1.412 Å, and the average distance from the Fe atom to the five carbon atoms of this ring is 2.117 Å, consistent with that expected. The distance from the Fe atom to the least-squares plane of the cyclopentadienyl ring is 1.743 Å. The Fe–C(7) bond length of 1.972(4) Å is shorter than that of a normal M–C bond, which can probably be attributed to the formation of a conjugated system of Fe, C(7), and O(1). However, this system does not involve the benzene ring, since the results reveal



Scheme. (*i*) Et_2O, −60 °C; (*ii*) Et_3OBF_4, CH_2Cl_2 at −60 °C or water at 0 °C different distances between Fe, C(7), O(11), and the plane of the benzene ring, which might be ascribed to the intramolecular steric hindrance.

Experimental

I.r., ^{1}H n.m.r., and mass spectra were recorded on Zeiss Specord-75, Varian XL-200, and Finnigan 4021 GC/MS/DS spectrometers, respectively. Melting points were taken in sealed capillaries and uncorrected.

All manipulations were performed under a purified nitrogen atmosphere with standard Schlenk techniques. Light petroleum (b.p. 30—60 °C), diethyl ether, and CH_2Cl_2 were saturated with N_2 and distilled from CaH_2, sodium diphenylketyl, and P_2O_5 respectively prior to use. The alumina (neutral) used for chromatography was deoxygenated in a high vacuum for 16 h, deactivated with 5% (w/w) water, and stored under N_2. Tricarbonyl(η-cyclopenta-1,3-diene)iron (**1**),[4] Et_3OBF_4,[10] and aryl-lithium reagents [11–15] were prepared by literature methods.

Typical Procedures for the Preparation of Complexes (**2**)—(**6**).—To a solution of complex (**1**) (0.5 g, 2.4 mmol) in diethyl ether (50 cm^3) was added dropwise the aryl-lithium reagent (2.4 mmol) in ether (20 cm^3) at −60 °C within 15 min, with efficient stirring. The solution turned gradually from light yellow to red. It was slowly allowed to warm to −40 °C and stirred at this temperature for 3—5 h. After evaporation of solvent in a high vacuum, the red residue was dissolved in CH_2Cl_2 (30 cm^3) and cooled to −60 °C. To this solution was immediately added Et_3OBF_4 (0.5 g, 2.6 mmol) in CH_2Cl_2 (15 cm^3) with vigorous stirring. The reaction mixture was stirred at −60 to −50 °C for 0.5 h and became orange-yellow. The

Table 3. Positional parameters and their estimated standard deviations for complex (**6**)

Atom	*x*	*y*	*z*
Fe	0.743 02(6)	0.461 34(5)	0.265 57(4)
F(1)	0.727 2(5)	−0.222 5(3)	−0.331 2(3)
F(2)	0.761 0(6)	−0.050 2(4)	−0.428 5(3)
F(3)	0.969 5(4)	−0.068 5(5)	−0.298 5(4)
O(1)	0.417 4(3)	0.360 2(3)	0.084 9(3)
O(2)	0.457 0(3)	0.579 5(3)	0.388 7(3)
O(3)	0.703 0(4)	0.190 9(3)	0.372 4(3)
C(1)	0.634 5(4)	0.231 2(4)	−0.000 1(3)
C(2)	0.607 6(5)	0.249 6(4)	−0.125 6(4)
C(3)	0.660 7(5)	0.152 0(4)	−0.228 9(4)
C(4)	0.740 0(5)	0.030 6(4)	−0.204 9(4)
C(5)	0.767 0(5)	0.010 6(4)	−0.081 1(4)
C(6)	0.716 5(5)	0.110 6(4)	0.022 3(4)
C(7)	0.570 6(4)	0.342 7(4)	0.107 2(3)
C(8)	0.567 0(4)	0.530 4(4)	0.340 0(3)
C(9)	0.718 5(4)	0.295 4(4)	0.328 6(3)
C(10)	0.798 2(5)	−0.076 1(4)	−0.315 0(5)
C(11)	0.985 8(5)	0.470 7(5)	0.190 6(4)
C(12)	1.025 8(5)	0.531 1(5)	0.329 5(4)
C(13)	0.948 0(5)	0.661 8(5)	0.362 4(4)
C(14)	0.859 7(5)	0.682 3(4)	0.245 5(5)
C(15)	0.885 1(5)	0.565 3(5)	0.139 5(4)

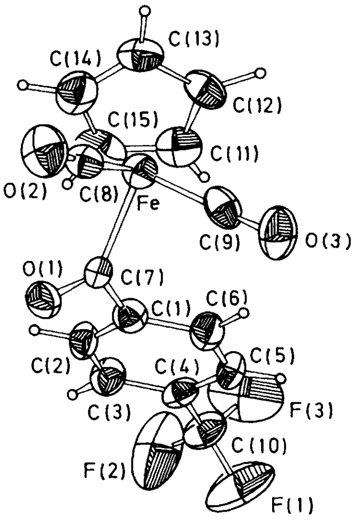


Figure. Molecular structure of complex (**6**)

Table 4. Bond lengths (Å) and angles (°) for complex (**6**)

Fe–C(7)	1.972(4)	Fe–C(15)	2.107(6)	C(1)–C(2)	1.386(7)	C(5)–C(6)	1.387(7)
Fe–C(8)	1.772(5)	F(1)–C(10)	1.303(6)	C(1)–C(6)	1.398(8)	C(11)–C(12)	1.427(7)
Fe–C(9)	1.768(6)	F(2)–C(10)	1.302(8)	C(1)–C(7)	1.520(6)	C(11)–C(15)	1.406(9)
Fe–C(11)	2.104(5)	F(3)–C(10)	1.300(7)	C(2)–C(3)	1.388(7)	C(12)–C(13)	1.399(8)
Fe–C(12)	2.132(4)	O(1)–C(7)	1.217(6)	C(3)–C(4)	1.401(8)	C(13)–C(14)	1.417(9)
Fe–C(13)	2.136(5)	O(2)–C(8)	1.139(6)	C(4)–C(5)	1.370(8)	C(14)–C(15)	1.410(7)
Fe–C(14)	2.106(6)	O(3)–C(9)	1.140(7)	C(4)–C(10)	1.497(8)		
C(7)–Fe–C(8)	90.3(2)	C(9)–Fe–C(14)	158.1(2)	C(2)–C(3)–C(4)	118.6(5)	F(3)–C(10)–C(4)	112.7(4)
C(7)–Fe–C(9)	90.5(2)	C(9)–Fe–C(15)	141.8(2)	C(3)–C(4)–C(5)	120.6(5)	Fe–C(11)–C(12)	71.4(3)
C(7)–Fe–C(11)	100.7(2)	C(11)–Fe–C(12)	39.4(2)	C(3)–C(4)–C(10)	119.4(5)	Fe–C(11)–C(15)	70.6(3)
C(7)–Fe–C(12)	139.6(2)	C(11)–Fe–C(13)	65.1(2)	C(5)–C(4)–C(10)	120.0(5)	C(12)–C(11)–C(15)	108.3(5)
C(7)–Fe–C(13)	148.9(2)	C(11)–Fe–C(14)	65.3(2)	C(4)–C(5)–C(6)	120.5(5)	Fe–C(12)–C(11)	69.2(3)
C(7)–Fe–C(14)	110.4(2)	C(11)–Fe–C(15)	39.0(2)	C(1)–C(6)–C(5)	119.9(5)	Fe–C(12)–C(13)	71.0(3)
C(7)–Fe–C(15)	86.0(2)	C(12)–Fe–C(13)	38.3(2)	Fe–C(7)–O(1)	124.2(3)	C(11)–C(12)–C(13)	107.7(5)
C(8)–Fe–C(9)	93.1(2)	C(12)–Fe–C(14)	65.0(2)	Fe–C(7)–C(1)	118.9(3)	Fe–C(13)–C(12)	70.7(3)
C(8)–Fe–C(11)	158.4(2)	C(12)–Fe–C(15)	65.6(2)	O(1)–C(7)–C(1)	116.8(4)	Fe–C(13)–C(14)	69.4(3)
C(8)–Fe–C(12)	129.2(2)	C(13)–Fe–C(14)	39.0(2)	Fe–C(8)–O(2)	177.7(4)	C(12)–C(13)–C(14)	108.0(5)
C(8)–Fe–C(13)	96.1(2)	C(13)–Fe–C(15)	65.4(2)	Fe–C(9)–O(3)	178.2(4)	Fe–C(14)–C(13)	71.6(3)
C(8)–Fe–C(14)	93.5(2)	C(14)–Fe–C(15)	39.1(2)	F(1)–C(10)–F(2)	104.3(4)	Fe–C(14)–C(15)	70.5(3)
C(8)–Fe–C(15)	124.9(2)	C(2)–C(1)–C(6)	119.1(4)	F(1)–C(10)–F(3)	104.9(5)	C(13)–C(14)–C(15)	108.5(5)
C(9)–Fe–C(11)	105.2(2)	C(2)–C(1)–C(7)	117.8(4)	F(1)–C(10)–C(4)	113.1(5)	Fe–C(15)–C(11)	70.4(3)
C(9)–Fe–C(12)	94.8(2)	C(6)–C(1)–C(7)	123.2(5)	F(2)–C(10)–F(3)	106.1(6)	Fe–C(15)–C(14)	70.4(3)
C(9)–Fe–C(13)	119.4(2)	C(1)–C(2)–C(3)	121.3(5)	F(2)–C(10)–C(4)	114.8(5)	C(11)–C(15)–C(14)	107.5(5)

solvent was removed under vacuum, and the solid residue was subjected to column chromatography on alumina at −20 °C with light petroleum followed by light petroleum–ether (5:1) as eluant. After removal of solvent under vacuum, the crude product was recrystallized from light petroleum at −80 °C to give light yellow needles.

Complexes (**2**)—(**6**) can also be formed when the acylmetalate intermediates are allowed to react with Et_3OBF_4 in aqueous solution at 0 °C, but the yield is often slightly lower than in CH_2Cl_2 at −60 °C. The detailed procedures are as follows. After the reaction mixture of complex (**1**) with aryl-lithium had been evaporated to dryness in a high vacuum, the residue was dissolved in nitrogen-saturated water (50 cm^3) at 0 °C and covered with light petroleum. Immediately Et_3OBF_4 was added portionwise, with vigorous stirring, until the solution became acidic. It was extracted with light petroleum, and the combined extracts dried over anhydrous Na_2SO_4. After removal of solvent in vacuum, further treatment of the residue obtained as described above gave acyl complexes (**2**)—(**6**).

Structure Determination of Complex (**6**).—Crystals of complex (**6**) suitable for *X*-ray diffraction were obtained by recrystallization from light petroleum at −80 °C. A single crystal was sealed in a capillary under N_2.

Crystal data. $C_{15}H_9F_3FeO_3$, $M = 350.08$, triclinic, space group $P\bar{1}$, $a = 7.670(7)$, $b = 9.008(4)$, $c = 10.662(1)$ Å, $\alpha = 103.70(2)$, $\beta = 96.496(8)$, $\gamma = 100.03(2)°$, $U = 695.47$ Å^3, $Z = 2$, $D_c = 1.67$ g cm^{-3}, λ(Mo-K_α) = 0.710 69 Å, $\mu = 11.2$ cm^{-1}.

The measurement of cell parameters and the collection of diffraction data were carried out with Mo-K_α radiation on a CAD4 diffractometer using the ω–2θ scan technique. The diffraction range was $2 \leqslant 2\theta \leqslant 54°$. 3 265 Unique reflections, of which 2 834 with $I > 3\sigma(I)$ were considered observed, were obtained. The intensity data were corrected for Lorentz and polarization factors, but not for absorption.

The structure was solved by direct methods using the Enraf-Nonius structure determination program. The co-ordinates of 22 non-hydrogen atoms were derived by Fourier syntheses, and the positions of nine hydrogen atoms were obtained by calculation using the HYDRO program. All the non-hydrogen atoms with anisotropic thermal parameters and the hydrogen atoms with isotropic thermal parameters were refined by block-diagonal matrix least squares. The agreement factor converged to $R = \Sigma||F_o| - |F_c||/\Sigma|F_o| = 0.039$, $R' = [\Sigma w(|F_o| - |F_c|)^2/\Sigma w|F_o|^2]^{\frac{1}{2}} = 0.045$. The final atomic co-ordinates are given in Table 3, bond lengths and angles in Table 4.

Additional material available from the Cambridge Crystallographic Data Centre comprises H-atom co-ordinates and thermal parameters.

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