Synthesis of Trinuclear Complexes with Mixed Bridging Ligands. X-Ray Structure of $[Pd\{Rh(\mu\text{-}pz)(\mu\text{-}SBu^t)(CO)_2\}_2]$ (pz = pyrazolate)†

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The formation and properties of the trinuclear heterobridged complexes of general formula $[Pd\{Rh(\mu\text{-}pz)(\mu\text{-}SBu^t)L_2\}_2]$ [L_2 = cycloocta-1,5-diene **1**; L_2 = $(CO)_2$ **2**; L_2 = CO, PPh_3 **3** and L_2 = CO, $P(OMe)_3$ **4**; pz = pyrazolate] are reported. Complexes **2** and **4** react with molecular iodine to give the compounds $[Pd\{Rh(\mu\text{-}pz)(\mu\text{-}SBu^t)I_2L_2\}_2]$ [L_2 = $(CO)_2$ **5** and L_2 = CO, $P(OMe)_3$ **6**]. The structure of $[Pd\{Rh(\mu\text{-}pz)(\mu\text{-}SBu^t)(CO)_2\}_2]$ **2** has been determined by X-ray diffraction methods. The trinuclear complex is centrosymmetric with the Pd atom lying on the centre of symmetry. The Rh and Pd atoms, separated by 3.686(1) Å, are connected by a double bridge, the pyrazolate ligand bridging through the two N atoms and the SBu^t ligand bridging through the S atom.

Binuclear rhodium or iridium pyrazolate complexes have attracted considerable interest in recent years.[1–8] In these complexes the individual metal centres, in oxidation states I, II or III, are generally joined to one another by a pair of bridging exo-bidentate pyrazolate (pz) ligands in such a way that the cyclic-bridged framework deviates from planarity. The boat conformation adopted results in a wide range of intermetallic separations [Rh–Rh 2.581–3.568, Ir–Ir 2.623–3.290 Å].[2a,9,10]

As suggested by Trofimenko,[1b] controlled extension of the $M(\mu\text{-}pz)_2M$ core to form well defined tri- or tetra-nuclear compounds may constitute an interesting area of research. Very recently we have reported the synthesis of homo- and hetero-trinuclear complexes containing the metallocycles $M(\mu\text{-}pz)_2M'(\mu\text{-}pz)_2M$.[11] Our interest in this area is focused on proposing an easy method for building a multinuclear framework with the possibility of varying the metal centres and their environment.

In this context, the present study deals with the preparation of a family of trinuclear heterobridged complexes having the $\{Rh(\mu\text{-}pz)(\mu\text{-}SBu^t)Pd(\mu\text{-}pz)(\mu\text{-}SBu^t)Rh\}$ core, as well as the X-ray structure of the complex $[Pd\{Rh(\mu\text{-}pz)(\mu\text{-}SBu^t)(CO)_2\}_2]$.

Results and Discussion

The reaction of the complex $[Pd\{Rh(\mu\text{-}pz)(\mu\text{-}Cl)(cod)\}_2]$ (cod = cycloocta-1,5-diene), prepared *in situ* by linkage of the fragments [Rh(acac)(cod)] (acac = acetylacetonate) and $[PdCl_2(Hpz)_2]$, with $KSBu^t$ affords the mixed-bridged trinuclear complex $[Pd\{Rh(\mu\text{-}pz)(\mu\text{-}SBu^t)(cod)\}_2]$ (equation 1).

$$2[Rh(acac)(cod)] + [PdCl_2(Hpz)_2] \xrightarrow{-2Hacac} [Pd\{Rh(\mu\text{-}pz)(\mu\text{-}Cl)(cod)\}_2] \xrightarrow[-2KCl]{2KSBu^t} \underset{\mathbf{1}}{[Pd\{Rh(\mu\text{-}pz)(\mu\text{-}SBu^t)(cod)\}_2]} \quad (1)$$

Compound **1** can exist as three geometrical isomers, but, according to the ^1H NMR spectrum of the isolated complex, only one of the possible isomers is present in solution. Taking into account the *trans* geometry present in the palladium complex $[PdCl_2(Hpz)_2]$[11] and the steric influence of the *tert*-butyl groups, we suggest that a *trans* configuration is also present in trinuclear compound **1**.

It is interesting to note, that we have recently synthesized some homo-binuclear complexes of general formula $[M_2(\mu\text{-}pz)(\mu\text{-}SBu^t)(L_2)_2]$ (M = Rh[12] or Ir[13]), but all the attempts to prepare mixed-bridged hetero-binuclear complexes were unsuccessful due to the operation of redistribution reactions.

Bubbling of carbon monoxide through a dichloromethane solution of complex **1** leads to the displacement of cod and to the formation of the carbonyl derivative $[Pd\{Rh(\mu\text{-}pz)(\mu\text{-}SBu^t)(CO)_2\}_2]$ **2**. The IR spectrum of complex **2** in dichloromethane shows two strong ν(CO) bands as predicted for *cis*-dicarbonyl structures.[14,15]

Addition of triphenyl phosphine or trimethyl phosphite to diethyl ether suspensions of complex **2** leads, as expected, to evolution of carbon monoxide and the formation of derivatives of the type $[Pd\{Rh(\mu\text{-}pz)(\mu\text{-}SBu^t)(CO)L\}_2]$ [L = PPh_3 **3**, $P(OMe)_3$ **4**]. Complexes **3** and **4** are characterized by a doublet in their $^{31}P\text{-}\{^1H\}$ NMR spectra at δ 35.9 (J_{RhP} 153) and 138.5 (J_{RhP} 242 Hz) respectively, indicating that they are symmetrical species in which the phosphorus nuclei are chemically equivalent.

Complexes **2** and **4** react readily with molecular iodine (1:2 molar ratio) to give complexes **5** and **6** respectively, which on the basis of elemental analysis, IR and NMR spectral data are formulated as the rhodium(III)–palladium(II)–rhodium(III) complexes $[Pd\{Rh(\mu\text{-}pz)(\mu\text{-}SBu^t)I_2(CO)(L)\}_2]$ [L = CO **5**, $P(OMe)_3$ **6**]. The ν(CO) bands of the oxidation compounds are shifted towards higher frequencies relative to those for the parent compounds (**5**: 75 and 105; **6**: 85 cm^{-1}), the observed shifts supporting the formulation of the compounds as rhodium(III) derivatives. The ^1H and $^{31}P\text{-}\{^1H\}$ NMR spectra of complex **6** show the presence of a small quantity of an isomer which has escaped isolation upon fractional crystallization. Indeed, the $^{31}P\text{-}\{^1H\}$ NMR spectrum of complex **6** contains one

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† Di-μ-*tert*-butylthio-1:2κ^2*S*; 2:3κ^2*S*-tetracarbonyl-1κ^2*C*,3κ^2*C*-di-μ-pyrazolato-1:2κ*N*^{1}:*N*^{2}; 2:3κ*N*^{1}:*N*^{2}-palladiumdirhodium.

Supplementary data available: see Instructions for Authors, *J. Chem. Soc., Dalton Trans.*, 1991, Issue 1, pp. xviii–xxii.

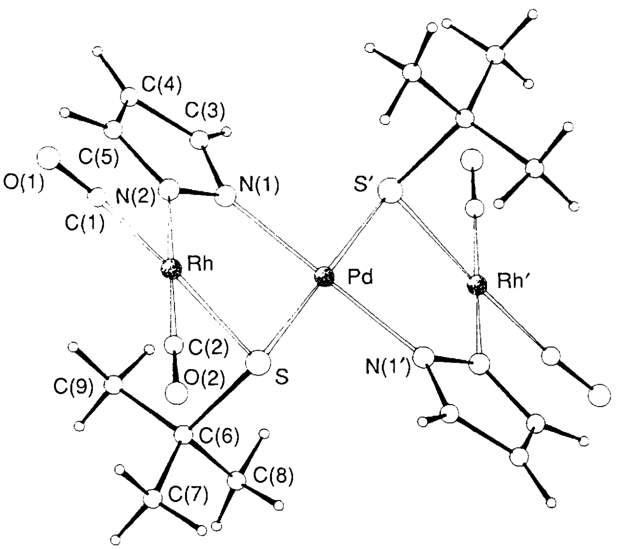

Fig. 1 Perspective view of the molecular structure of the complex [Pd{Rh(μ-pz)(μ-SBu^t)(CO)$_2$}$_2$], showing the atomic numbering scheme

Table 1 Bond distances (Å) and angles (°) in complex **2***

Rh–S	2.349(2)	C(3)–C(4)	1.377(12)
Rh–N(2)	2.039(5)	C(4)–C(5)	1.362(10)
Rh–C(1)	1.879(8)	C(1)–O(1)	1.136(10)
Rh–C(2)	1.827(6)	C(2)–O(2)	1.167(8)
Pd–N(1)	2.007(6)	S–C(6)	1.860(6)
Pd–S	2.317(2)	C(6)–C(7)	1.506(9)
N(1)–N(2)	1.406(8)	C(6)–C(8)	1.545(12)
N(1)–C(3)	1.347(7)	C(6)–C(9)	1.515(10)
N(2)–C(5)	1.337(9)		

N(2)–Rh–C(1)	91.5(3)	Rh–N(2)–N(1)	124.1(3)
S–Rh–N(2)	88.7(2)	Rh–N(2)–C(5)	129.0(5)
C(2)–Rh–S	88.3(2)	N(1)–N(2)–C(5)	106.6(5)
C(1)–Rh–C(2)	91.4(3)	N(1)–C(3)–C(4)	109.6(6)
S–Pd–N(1)	89.3(1)	C(3)–C(4)–C(5)	105.7(7)
Rh–S–Pd	104.4(1)	N(2)–C(5)–C(4)	111.0(7)
Rh–S–C(6)	105.8(2)	S–C(6)–C(7)	106.6(5)
Pd–S–C(6)	108.1(2)	S–C(6)–C(8)	106.3(5)
Rh–C(1)–O(1)	175.8(7)	S–C(6)–C(9)	110.9(5)
Rh–C(2)–O(2)	178.9(6)	C(7)–C(6)–C(8)	109.1(6)
Pd–N(1)–N(2)	124.4(4)	C(7)–C(6)–C(9)	115.1(6)
Pd–N(1)–C(3)	128.5(5)	C(8)–C(6)–C(9)	108.6(6)
N(2)–N(1)–C(3)	107.1(5)		

* The primed atoms are related tc the unprimed ones by the transformation $1 - x, -y, -z$.

strong and one weak signal; each signal is coupled with ^{103}Rh and the two $^1J(^{103}Rh^{31}P)$ coupling constants are nearly the same. The spectroscopic data indicate the existence of two isomers, probably due to the different relative positions of the SBu^t and pyrazolate groups. The above mentioned results demonstrate the high stability of the five-membered cyclic core {Rh(μ-pz)(μ-SBu^t)Pd(μ-pz)(μ-SBu^t)Rh}. Various exchange reactions can be carried out on the terminal ligands leaving the central ring intact; oxidative additions to two metal atoms can also take place.

Description of the Crystal Structure of the Complex [Pd{Rh(μ-pz)(μ-SBu^t)(CO)$_2$}$_2$].—The structure of complex **2** is shown in Fig. 1 together with the atomic numbering scheme; bond distances and angles are given in Table 1. The trinuclear complex has a crystallographically imposed C_i symmetry with the Pd atom lying on the centre of symmetry. The Rh, Pd and Rh′ metals form a linear chain in which each Rh and Pd couple, at a distance of 3.686(1) Å which excludes any metal–metal interaction, is connected by a double bridge, with a pyrazolate ligand bridging through the two N atoms, and a SBu^t ligand through the S atom. Each metal is in a square-planar arrangement. The two Rh atoms are bound to the S atom, to the N(2) atom, and to the C(1) and C(2) atoms from terminal carbonyl groups, whereas the central Pd atom is bound to two N(1) and to two S atoms, each couple being centrosymmetrically related. The Pd atom and the co-ordinated atoms are perfectly coplanar for imposed symmetry conditions, whereas the S, N(2), C(1) and C(2) atoms deviate by −0.001(1), 0.008(5), −0.021(7) and 0.020(7) Å from the mean plane through them, with the Rh atom out by 0.053(1) Å from this mean plane. The complex exhibits a step conformation, the dihedral angle between the mean co-ordination planes around Rh and Pd atoms being 29.8(1)°.

The Rh–N [2.039(5) Å] and Pd–N [2.007(6) Å] bond distances involving the pyrazolate ligand are comparable to those [2.063(7), 2.062(6) and 1.988(6), 1.992(7) Å respectively], found in the very similar complex [Pd{Rh(μ-pz)$_2$(CO)$_2$}$_2$] in which each Rh–Pd couple is doubly bridged by pyrazolate ligands [Rh ••• Pd separation 3.579(1) Å].[11] The value of the Rh–S bond distance, 2.349(2) Å, is comparable to those found, 2.352(2) and 2.376(3) Å, in the binuclear complex [Rh$_2$(μ-pz)(μ-SBu^t)(CO)$_2${P(OMe)$_3$}$_2$] with the same SBu^t bridging ligand,[12] and the Pd–S bond distances, 2.317(2) Å, are very close to the mean value of this distance, 2.328(7) Å, in four-co-ordinate Pd complexes with bridging alkanethiolate ligands.[16]

The relatively short intermolecular Rh ••• Rh($-x$, $-y$, $-z$) separation, 3.447(1) Å, for complex **2** is noteworthy. The deviation of the Rh atom by 0.053(1) Å from the mean plane through the co-ordinated atoms pointing towards the other Rh in a direction roughly normal to the Rh co-ordination plane could be indicative of a weak metal–metal interaction. Taking into account these weak interactions, in the crystal the complexes are joined in chains running along the centres of symmetry. Weak metal–metal interactions have been postulated especially for dimeric complexes, in which short intramolecular Rh ••• Rh separations have been found, whereas examples of short intermolecular Rh ••• Rh separations are much rarer.[10,17]

Experimental

Elemental analyses were carried out with a Perkin-Elmer 240B microanalyser. IR spectra were recorded on a Perkin-Elmer 783 spectrophotometer. ^{1}H and ^{31}P-{^{1}H} NMR spectra were carried out in CDCl$_3$ solution at room temperature on a Varian XL 200 spectrometer. All preparations were carried out under nitrogen on a vacuum line using Schlenk techniques. Solvents were purified by standard methods prior to use. The reactants were of commercial origin and used without further purification. The complexes [PdCl$_2$(Hpz)$_2$][11] and [Rh(acac)(cod)][18] were prepared according to literature methods.

Preparation of [Pd{Rh(μ-pz)(μ-SBu^t)(cod)}$_2$] **1**.—To a solution of the species [Pd{Rh(μ-pz)(μ-Cl)(cod)}$_2$] {prepared by reaction of [PdCl$_2$(Hpz)$_2$] (84.6 mg, 0.27 mmol) with [Rh(acac)(cod)] (167.4 mg, 0.54 mmol)} in acetone (20 cm^3) was added a mixture of Bu^{t}SH (33.0 μl, 0.27 mmol) and potassium hydroxide (0.27 mmol) in methanol (10 cm^3) to give a yellow solution. The reaction was continued for 48 h at room temperature to afford a pale yellow suspension of complex **1**. Then the suspension was evaporated to dryness and the residue extracted with dichloromethane (20 cm^3). Concentration of the filtrate to 2 cm^3 and addition of methanol (10 cm^3) gave complex **1** which was filtered off, washed with cold methanol and vacuum dried (yield 75%) (Found: C, 42.2; H, 5.7; N, 6.8. Calc. for $C_{30}H_{48}N_4PdRh_2S_2$: C, 42.8; H, 5.7; N, 6.6%). ^{1}H NMR (CDCl$_3$): δ 1.34 (s, SBu^t), 2.26 (br, cod), 2.33 (br, cod), 4.24 (br, cod), 4.63 (br, cod), 6.07 (t, pz, $^3J_{HH}$ 2.0), 7.08 (d, pz, $^3J_{HH}$ 2.0) and 7.77 (d, pz, $^3J_{HH}$ 2.0 Hz).

Table 2 Fractional atomic coordinates ($\times 10^4$) with estimated standard deviations in parentheses for the non-hydrogen atoms of complex **2**

Atom	*X/a*	*Y/b*	*Z/c*
Pd	5000	0	0
Rh	1333(1)	1076(0)	575(1)
S	3156(2)	1465(1)	−426(1)
O(1)	−925(6)	773(5)	1959(4)
O(2)	−1185(6)	2507(5)	−1090(5)
N(1)	4592(6)	−199(4)	1485(4)
N(2)	3160(6)	161(4)	1694(4)
C(1)	−81(8)	846(6)	1427(6)
C(2)	−199(8)	1958(6)	−433(6)
C(3)	5584(9)	−671(6)	2453(5)
C(4)	4823(9)	−644(6)	3267(7)
C(5)	3354(9)	−115(6)	2771(6)
C(6)	4305(8)	2728(5)	245(6)
C(7)	3155(9)	3687(5)	−208(8)
C(8)	5796(9)	2822(6)	−184(8)
C(9)	4955(11)	2622(7)	1532(6)

Preparation of [Pd{Rh(μ-pz)(μ-SBu^t)(CO)$_2$}$_2$] **2**.—Carbon monoxide was bubbled through a solution of complex **1** (100.0 mg, 0.14 mmol) in dichloromethane (25 cm^3) for 30 min, and the red compound [Pd{Rh(μ-pz)(μ-SBu^t)(CO)$_2$}$_2$] **2** precipitated spontaneously. The solvent was evaporated under vacuum to *ca.* 1 cm^3 and hexane (10 cm^3) was added. The solid was filtered off, washed with cold hexane and vacuum dried (yield 70%) (Found: C, 29.3; H, 3.4; N, 7.7. Calc. for $C_{18}H_{24}N_4O_4PdRh_2S_2$: C, 29.3; H, 3.3; N, 7.6%). IR (CH_2Cl_2): ν(CO) 2060 and 2000 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 1.25 (s, SBu^t), 6.19 (t, pz, $^3J_{HH}$ 2.2), 7.63 (d, pz, $^3J_{HH}$ 2.2) and 8.06 (d, pz, $^3J_{HH}$ 2.2 Hz).

Preparation of [Pd{Rh(μ-pz)(μ-SBu^t)(CO)(PPh$_3$)}$_2$] **3**.—A stoichiometric amount of solid triphenylphosphine (124.7 mg, 0.47 mmol) was added to a suspension of [Pd{Rh(μ-pz)(μ-SBu^t)(CO)$_2$}$_2$] **2** (176.9 mg, 0.23 mmol) in diethyl ether and the resulting yellow solution was stirred for 1 h. Addition of hexane (10 cm^3) to the concentrated solution (2 cm^3) gave a yellow solid which was filtered off, washed with hexane and vacuum dried (yield 80%) (Found: C, 51.0; H, 4.3; N, 4.8. Calc. for $C_{52}H_{54}N_4O_2P_2PdRh_2S_2$: C, 51.8; H, 4.5; N, 4.6%). IR (CH_2Cl_2): ν(CO) 1970 cm^{-1}. NMR ($CDCl_3$): ^{1}H, δ 1.24 (s, SBu^t), 6.15 (t, pz, $^3J_{HH}$ 2.2 Hz) and 7.62 (m, PPh$_3$); ^{31}P-{^{1}H}, δ 35.9 (d, J_{RhP} 153 Hz).

Preparation of [Pd{Rh(μ-pz)(μ-SBu^t)(CO)P(OMe)$_3$}$_2$] **4**.—Addition of trimethyl phosphite (113.0 μl, 0.47 mmol) to a suspension of [Pd{Rh(μ-pz)(μ-SBu^t)(CO)$_2$}$_2$] **2** (176.9 mg, 0.23 mmol) in diethyl ether caused evolution of carbon monoxide. The resulting yellow solution was evaporated to *ca.* 2 cm^3 and hexane (10 cm^3) was added to give a pale yellow solid which was filtered off, washed with hexane and vacuum dried (yield 70%) (Found: C, 29.0; H, 4.9; N, 6.0. Calc. for $C_{22}H_{42}N_4O_8P_2PdRh_2S_2$: C, 28.4; H, 4.5; N, 6.0%). IR (CH_2Cl_2): ν(CO) 1995 cm^{-1}. NMR ($CDCl_3$): ^{1}H, δ 1.19 (s, SBu^t), 3.69 [d, P(OMe)$_3$, $^3J_{PH}$ 12.6], 6.10 (t, pz, $^3J_{HH}$ 2.2), 7.58 (d, pz, $^3J_{HH}$ 2.2) and 8.05 (d, pz, $^2J_{HH}$ 2.2 Hz); ^{31}P-{^{1}H}, δ 138.5 (d, J_{RhP} 242 Hz).

Preparation of [Pd{Rh(μ-pz)(μ-SBu^t)I$_2$(CO)$_2$}$_2$] **5**.—To a solution of [Pd{Rh(μ-pz)(μ-SBu^t)(CO)$_2$}$_2$] **2** (100.0 mg, 0.13 mmol) in dichloromethane (40 cm^3) was added a stoichiometric amount of iodine (68.9 mg, 0.27 mmol). The colour of the solution changed from orange to brown and the progress of the reaction was followed by IR spectroscopy. After stirring for 30 min the solvent was evaporated off to 2 cm^3 and hexane (10 cm^3) was added to give a dark-brown solid, which was filtered off, washed with hexane and dried under vacuum (yield 85%) (Found: C, 16.2; H, 1.5; N, 4.0. Calc. for $C_{18}H_{24}I_4N_4O_4PdRh_2S_2$: C, 16.0; H, 1.7; N, 4.1%). IR (CH_2Cl_2): ν(CO) 2135 and 2105 cm^{-1}. ^{1}H NMR: 1.34 (s, SBu^t), 6.22 (br, pz), 7.82 (br, pz) and 8.56 (br, pz).

Preparation of [Pd{Rh(μ-pz)(μ-SBu^t)I$_2$(CO)[P(OMe)$_3$]}$_2$] **6**.—Iodine (54.6 mg, 0.22 mmol) was added to a solution of [Pd{Rh(μ-pz)(μ-SBu^t)(CO)[P(OMe)$_3$]}$_2$]**4**(100mg,0.11mmol) in dichloromethane (20 cm^3); the resulting brown solution was stirred at room temperature. After 30 min, the IR spectrum revealed the absence of any starting material. Addition of hexane (15 cm^3) to the concentrated solution (1 cm^3) gave a brown solid, which was filtered off, washed with hexane and vacuum dried (yield 80%) (Found: C, 18.6; H, 2.9; N, 3.8. Calc. for $C_{22}H_{42}I_4N_4O_8P_2PdRh_2S_2$: C, 18.4; H, 2.9; N, 3.9%). IR (CH_2Cl_2): ν(CO) 2080 (br) cm^{-1}. NMR ($CDCl_3$): major species, ^{1}H, δ 1.28 (s, SBu^t), 3.77 [d, P(OMe)$_3$, $^3J_{PH}$ 11.2], 6.12 (t, pz, $^3J_{HH}$ 2.0), 8.02 (d, pz, $^3J_{HH}$ 2.0) and 8.33 (d, pz, $^3J_{HH}$ 2.0 Hz); ^{31}P-{^{1}H}, δ 98.5 (d, J_{RhP} 125 Hz); minor species, ^{1}H, δ 1.41 (s, SBu^t), 4.07 [d, P(OMe)$_3$, $^3J_{PH}$ 10.9], 6.21 (t, pz, $^3J_{HH}$ 2.2), 8.13 (d, pz, $^3J_{HH}$ 2.2) and 8.59 (d, pz, $^3J_{HH}$ 2.2 Hz); ^{31}P-{^{1}H}, δ 96.7 (d, J_{RhP} 124 Hz).

Crystal-structure Determination of the Complex [Pd{Rh(μ-pz)(μ-SBu^t)(CO)$_2$}$_2$] **2**.—A crystal of approximate dimensions 0.24 × 0.27 × 0.35 mm was used for the X-ray analysis.

Crystal data. $C_{18}H_{24}N_4O_4PdRh_2S_2$, M = 736.74, monoclinic, space group $P2_1/n$, a = 8.567(3), b = 12.123(5), c = 12.446(6) Å, β = 109.58(2)°, U = 1217.9(9) Å^3 (by least-squares refinement from the θ values of 30 accurately measured reflections with θ in the range 11.1–19.0°, λ = 0.7107 Å), Z = 2, D_c = 2.009 g cm^{-3}, F(000) = 720, μ(Mo-Kα) = 22.36 cm^{-1}.

Data were collected at room temperature on Siemens AED single-crystal diffractometers using niobium-filtered Mo-Kα radiation and the θ–2θ scan mode. All reflections with θ in the range 3–30° were measured; of 3691 independent reflections, 2765, having $I > 3\sigma(I)$, were considered observed and used in the analysis. The individual profiles were analysed according to Lehmann and Larsen.[19] The intensity of one standard reflection was measured after 50 reflections as a general check on crystal and instrument stability. No significant change in the measured intensities was observed during the data collection. A correction for the absorption effects was applied (maximum and minimum transmission factors values were 1.30 and 0.77).[20]

The structure was solved by Patterson and Fourier methods, and refined by full-matrix least squares first with isotropic and then with anisotropic thermal parameters in the last cycles for all the non-hydrogen atoms. The hydrogen atoms were placed at their calculated positions (C–H 1.00 Å) and refined 'riding' on the corresponding carbon atoms. A weighting scheme $w = [\sigma^2(F_o)]^{-1}$ was used in the last cycles of refinement. Final R and R' values were 0.0363 and 0.0359. The SHELX 76 and SHELXS 86 systems of computer programs were used.[21] Atomic scattering factors, corrected for anomalous dispersion, were taken from ref. 22. Final atomic coordinates for the non-hydrogen atoms are given in Table 2. All calculations were carried out on the CRAY X-MP/48 computer of the Centro di Calcolo Elettronico Interuniversitario dell'Italia Nord-Orientale, Bologna and on the GOULD POWERNODE 6040 of the Centro di Studio per la Strutturistica Diffrattometrica del C.N.R., Parma.

Additional data available from the Cambridge Crystallographic Data Centre comprises H-atom coordinates, thermal parameters and remaining bond distances and angles.

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