Synthesis and Chemistry of 1,1-Bis(3-indenyl)ethane; Crystal Structure of $[\{Ru(CO)_2\}_2\{\eta^5:\eta^5\text{-}CH_3CH(C_9H_6)_2\}]$†

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The compound 1,1-bis(3-indenyl)ethane has been synthesised. Its reaction with $[Ru_3(CO)_{12}]$ yielded $[\{Ru(CO)_2\}_2\{\eta^5:\eta^5\text{-}CH_3CH(C_9H_6)_2\}]$ as a mixture of diastereoisomers which have different faces of each five-membered ring of the indenyl units complexed to a $Ru(CO)_2$ fragment. The crystal structure of **1** shows that the major diastereoisomers present are bound to inequivalent faces of the ligand [trigonal, space group $P3_121$ (D_3^4, no. 152), $Z = 3$, $a = 10.226(6)$, $c = 16.896(8)$ Å and $\gamma = 120°$; R 0.0384 for 1666 unique reflections]. The reaction of 1,1-bis(3-indenyl)ethane with $[Mo(CO)_3(NCMe)_3]$ or $[Cr(CO)_3(NH_3)_3]$ gave $[M\{\eta^6\text{-}CH_3CH(C_9H_7)_2\}(CO)_3]$ (M = Mo or Cr respectively) as a mixture of diastereoisomers. In all cases ^1H and ^{13}C NMR spectroscopy proved to be useful tools for detecting the diastereoisomers.

We describe herein the synthesis of the new ligand 1,1-bis(3-indenyl)ethane. This ligand has several features of interest to organometallic chemists; for example, it could bond to metals in a variety of ways either *via* the five-membered or six-membered rings, or a combination of both. In addition, the asymmetry of the ligand gives rise to a number of interesting types of chirality; thus, not only are the two faces of each indenyl substituent prochiral with respect to complexation, but bonding to only one indenyl fragment generates a chiral centre at the bridging carbon atom. These are features which are illustrated in this paper; however, given that the ligand is readily synthesised in high yield from readily available starting materials, there are other features of this ligand that are noteworthy. In particular given the interest in heterobinuclear compounds, the ligand clearly has potential in this area.[1] Further, the enhanced reactivity of indenyl complexes is well established[2] and this should be particularly evident when both indenyl fragments are complexed either in a mono- or bi-nuclear compound.

Results and Discussion

1,1-Bis(3-indenyl)ethane was originally synthesised by serendipity in the course of synthesising 1-neomenthylindene.[3] Thus, reaction of LiBu with indene in tetrahydrofuran and subsequent treatment with menthyl tosylate under reflux for 6 h gave the compound in approximately 10% yield (based on indene). No impurity at this level was present in the starting materials and it is therefore presumed that it originated from the reported cleavage of tetrahydrofuran by LiBu.[4] A more rational synthesis, the reaction of lithium indenide with 1,1-dichloroethane, gave a 68% yield.

Reaction of 1,1-bis(3-indenyl)ethane with $[Ru_3(CO)_{12}]$ gave $[\{Ru(CO)_2\}_2\{\eta^5:\eta^5\text{-}CH_3CH(C_9H_6)_2\}]$ **1** as confirmed by X-ray crystallography. In this molecule the two faces of the asymmetric indenyl units are inequivalent giving rise to four possible arrangements **I**–**IV**; however, **III** and **IV** are enantiomeric. The infrared spectrum of **1** (Experimental section) shows no evidence of such a complex mixture and is similar to that assigned to the *cis* form of the unsubstituted indenyl complex $[\{Ru(\eta^5\text{-}C_9H_7)(CO)_2\}_2]$ which displays ν(CO) at 2003, 1961, 1815vw and 1779 cm^{-1} (in $CHCl_3$).[5] In contrast, the

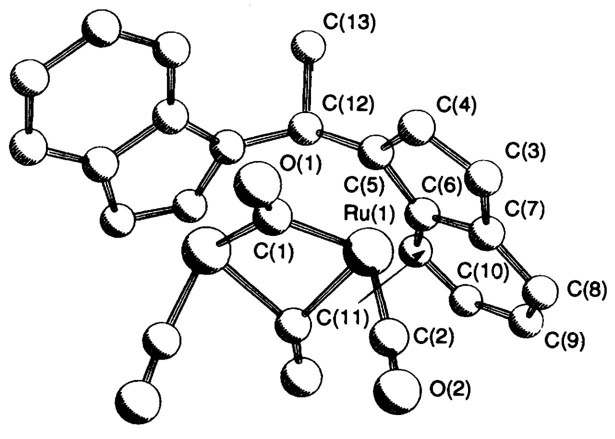
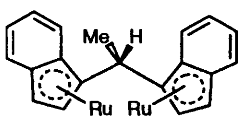
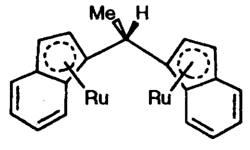


Fig. 1 Molecular structure of $[\{Ru(CO)_2\}_2\{\eta^5:\eta^5\text{-}CH_3CH(C_9H_6)_2\}]$ **1**



I



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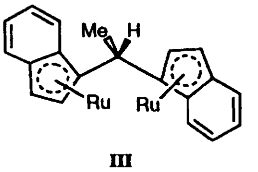
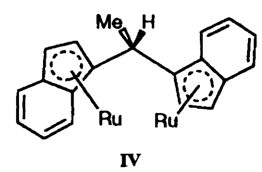


III



IV

^1H and ^{13}C NMR spectra of **1** show the presence of two species in the ratio 4:1, the major species having inequivalent indenyl groups and is assigned to a mixture of the asymmetric isomers **III** and **IV**. The minor species has equivalent indenyl groups and is assigned to a mixture of the isomers **I** and **II**, both of which have a plane of symmetry through the bridging carbon atom.

Although attempts to separate the isomeric species were unsuccessful the crystal selected for X-ray analysis is that of one of the major species, **III**, and the molecular structure is illustrated in Fig. 1. It comprises a binuclear complex, which

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† *Supplementary data available:* see Instructions for Authors, *J. Chem. Soc., Dalton Trans.*, 1994, Issue 1, pp. xxiii–xxviii.

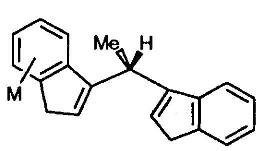
possesses crystallographically imposed C_2 symmetry, in which the two symmetry related ruthenium atoms each carry a terminal carbonyl ligand, and are bridged by two symmetry related carbonyl groups and by a 1,1-bis(3-indenyl)ethane ligand. Each ruthenium has a 'piano stool' geometry, with the three carbonyl groups in the basal sites. The bond lengths from the ruthenium atoms to the bridging carbonyls at 2.009(10) Å are intermediate to those found in [{Ru(η^5-C_5R_4R')(CO)}$_2$(μ-CO)$_2$] [*i.e.* 1.99 Å when R = R′ = H, **2a**;[6] 2.042(4) Å when R = Me, R′ = Et, **2b**[7]] whereas those to the terminal carbonyl ligands are approximately 0.14 Å shorter and virtually identical to those found in **2a** and **2b**. The Ru–Ru distance in **1** at 2.656(2) Å is significantly shorter than in the cyclopentadienyl analogues [*i.e.* 2.735 Å in **2a** and 2.7584(5) Å in **2b**]; also, the bridge bond angle C(5)–C(12)–C(5a) is opened up to 112.9(13)°. Thus, it appears that the 1,1-bis(3-indenyl)ethane ligand is slightly strained in bridging the two metal centres. The indenyl fragment is attached asymmetrically to the five-membered ring, with the shortest distances to the two carbons which carry hydrogens; however, at 2.282(9) Å the mean Ru–C(indenyl) distance is similar to that found in other ruthenium–indenyl compounds.[8] The two rings of this ligand are each planar (root mean square deviations 0.020 and 0.014 Å) but mutually inclined by 4.0° about their common bond so that the six-membered ring is bent away from the ruthenium. The two disorder components of the linking carbon atom of the ethane fragment lie 0.056 and 0.008 Å from the mean plane of the five-membered ring; the methyl group is displaced more than 1 Å out of the plane away from the metal.

Reaction of 1,1-bis(3-indenyl)ethane with [Mo(CO)$_3$(NCMe)$_3$] yielded a bright yellow compound which, on the basis of microanalytical and spectroscopic data is formulated as [Mo{η^6-$CH_3CH(C_9H_7)_2$}(CO)$_3$] **3** in which a Mo(CO)$_3$ unit is complexed to one of the six-membered rings. This bonding mode is clearly evident from the ^{1}H and ^{13}C NMR spectra of **3** which show the characteristic upfield shift for the signals of the complexed arene ring.[9] Once again the NMR spectrum shows the presence of two isomers, this time in the ratio 1.7 : 1 and these are assigned to the diastereoisomers **V** and **VI**, together with their mirror images. The diastereoisomerism arises from the inequivalent faces of the six-membered ring and the chirality of the bridging carbon atom. Again we were unsuccessful in separating the diastereoisomers. As expected the infrared spectrum of **3** (Experimental section) is similar to that reported for the analogous indene complex [Mo(η^6-C_9H_8)(CO)$_3$], *i.e.* ν(CO) at 1976 and 1895 cm^{-1} (in tetrahydrofuran).[10]

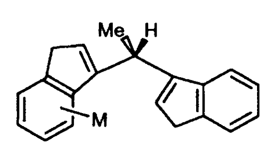
A similar reaction with [Cr(CO)$_3$(NH$_3$)$_3$] gave the analogous [Cr{η^6-$CH_3CH(C_9H_7)_2$}(CO)$_3$] **4** as a mixture of diastereoisomers. Interestingly, the ratio of the diastereoisomers was 1.7:1, the same as that observed for the corresponding molybdenum complex. The spectroscopic properties of **4** were virtually identical to those of the corresponding molybdenum complex **3** and the infrared spectrum closely resembles that reported for the indene complex [Cr(η^6-C_9H_8)(CO)$_3$], *i.e.* ν(CO) at 1950 and 1860 cm^{-1} (KBr).[11] We also note that the related compound [Cr(η^6-$C_9H_7CH_2CH_2C_9H_6Me$)(CO)$_3$] has recently been synthesised but, in marked contrast to **4**, one diastereoisomer is formed almost exclusively.[12]

Experimental

Microanalytical data were obtained by the University of Sheffield Microanalytical Service. The ^{1}H and ^{13}C NMR spectra were recorded on a Bruker AM 250 spectrometer using SiMe$_4$ as an internal reference. Infrared spectra were measured on a PE-157G spectrometer. All reactions were carried out under an atmosphere of nitrogen and all solvents were dried and degassed before use.



V



VI

Synthesis of 1,1-*Bis*(3-*indenyl*)*ethane*.—A thoroughly degassed solution of redistilled indene (46.83 g, 0.403 mol) in tetrahydrofuran (150 cm^3) was treated with butyllithium (2.6 mol dm^{-3} in hexane, 155 cm^3, 0.403 mol) at 0 °C under nitrogen. The mixture was stirred and allowed to warm to room temperature over a period of 30 min. 1,1′-Dichloroethane (19.98 g, 0.202 mol) in tetrahydrofuran (50 cm^3) was added dropwise maintaining the temperature of the reaction mixture at 15–20 °C. Stirring was continued for 30 min after the addition was complete and then the mixture was heated under reflux for a further 6 h. The solution was cooled in an ice-bath and water (100 cm^3) was carefully added to destroy excess indenyllithium. Tetrahydrofuran was removed on the rotary evaporator and the residue was extracted with diethyl ether (3 × 90 cm^3). The combined organic phase was dried over anhydrous MgSO$_4$, filtered and then concentrated on a rotary evaporator to yield a brown oil. Vacuum distillation of this oil first removed traces of tetrahydrofuran, then an unidentified royal blue distillate [*ca.* 1 cm^3; b.p. 72–82 °C (0.6 mmHg)] and left the crude product as a light brown viscous oil (35.4 g, 68%). This product was sufficiently pure to be used in synthesis but an analytically pure sample was obtained by chromatography on alumina using hexane as the eluent; this product was a colourless viscous oil which slowly solidified over a period of days (Found: C, 92.1; H, 6.9. $C_{20}H_{18}$ requires C, 93.0; H, 7.0%); *m*/*z* 258 (34, M^+), 143 (100, $M - C_9H_7$), 128 (81, $M - C_{10}H_{10}$), 115 (74%, C_9H_7). NMR: δ_H(CDCl$_3$) 7.42 (2 H, d, *J* 6, aryl), 7.34 (2 H, m, aryl), 7.25–7.10 (4 H, m, aryl), 6.25 (2 H, m, olefinic CH), 4.11 (1 H, q, *J* 6 Hz, C*H*Me), 3.35 (4 H, br s, 2 CH$_2$), 1.66 (3 H, d, CH*Me*); δ_C(CDCl$_3$) 147.0 [2 C, s, *C*CH(Me)*C*], 144.7 (2 C, s, aryl C), 144.6 (2 C, s, aryl C), 128.2, 125.8, 124.4, 123.7 (8 C, all s, aryl CH), 119.6 (2 C, s, olefinic CH), 37.5 (2 C, s, CH$_2$), 31.6 (1 C, s, *C*HMe), 19.0 (1 C, s, Me).

Synthesis of [{Ru(CO)$_2$}$_2${η^5:η^5-$CH_3CH(C_9H_6)_2$}] **1**.—A mixture of [Ru$_3$(CO)$_{12}$] (0.250 g, 0.391 mmol) and 1,1-bis(3-indenyl)ethane (0.152 g, 0.588 mmol) in light petroleum (b.p. > 120 °C, 40 cm^3) was heated under reflux (128 °C) for 18 h under nitrogen but *without any initial degassing of air*. After cooling, the reaction mixture was filtered and the solvent was removed on a rotary evaporator. The brown residue was chromatographed on alumina; eluting with dichloromethane first yielded traces of unreacted [Ru$_3$(CO)$_{12}$] and then a yellow band. Removal of the solvent from the latter yielded the product **1** as a yellow-orange solid (0.160 g, 48%) (Found: C, 50.5; H, 3.2. $C_{24}H_{16}O_4Ru_2$ requires C, 50.5; H, 2.8%); $\tilde{\nu}_{max}$/cm^{-1} (CO) 1997vs, 1960m, 1816w, 1784s (CH$_2$Cl$_2$). NMR major isomer: δ_H(CDCl$_3$) 7.65–7.10 (8 H, m, aryl), 6.18 (1 H, d, *J* 3, CH), 6.12 (1 H, d, *J* 3, CH), 5.53 (1 H, d, *J* 3, CH), 5.42 (1 H, d, *J* 3, CH), 4.06 (1 H, q, *J* 7.5 Hz, C*H*Me), 1.72 (3 H, d, Me); δ_C(CDCl$_3$) 126.8 (2 C), 126.0, 125.6, 124.5, 123.0, 119.2, 119.0 (uncomplexed aromatic CH), 114.8, 112.1, 106.9, 106.7 (aromatic C), 97.1, 95.6, 76.3, 74.7 (complexed aromatic CH), 93.8, 93.2 (*C*CHMe), 29.6 (*C*HMe), 20.7 (Me). NMR minor isomer: δ_H(CDCl$_3$) 7.65–7.10 (8 H, m, aryl), 6.15 (1 H, d, *J* 3, CH), 5.46 (1 H, d, *J* 3, CH), 4.44 (1 H, q, *J* 7.5 Hz, C*H*Me), 1.57 (3 H, d, Me); δ_C(CDCl$_3$) 126.5, 126.2, 123.1, 118.4 (uncomplexed aromatic CH), 113.5, 107.2 (aromatic C), 95.9, 75.3 (complexed aromatic CH), 93.1 (*C*CHMe), 29.3 (*C*HMe), 21.4 (Me).

Synthesis of [Mo{η^6-$CH_3CH(C_9H_7)_2$}(CO)$_3$] **3**.—[Mo(CO)$_3$(NCMe)$_3$] was synthesised from [Mo(CO)$_6$] (0.85 g, 3.22 mmol) by the published procedure.[13] A solution of 1,1-

bis(3-indenyl)ethane (0.78 g, 3.02 mmol) in tetrahydrofuran (70 cm^3) was added to the solid [$Mo(CO)_3(NCMe)_3$] under nitrogen and the mixture heated under reflux for 20 h. After cooling, the solvent was removed *in vacuo* and the dark yellow residue chromatographed on alumina under nitrogen. A yellow band eluted with dichloromethane–light petroleum (b.p. 40–60 °C), 1 : 1, and removal of the solvent *in vacuo* yielded a bright yellow solid (0.686 g, 52%) (Found: C, 62.9; H, 4.2. $C_{23}H_{18}MoO_3$ requires C, 63.0; H, 4.1%); $\tilde{\nu}_{max}$/cm^{-1} (CO) 1960vs, 1881vs(br)(CH_2Cl_2). NMR major isomer: δ_H($CDCl_3$) 7.65–7.00 (4 H, m, uncomplexed aromatics), 6.30 (1 H, br s, =CH), 6.20 (1 H, br s, =CH), 5.81 (1 H, d, *J* 7), 5.58 (1 H, d, *J* 7), 5.24 (1 H, m), 5.13 (1 H, t, *J* 7) (complexed aromatics), 3.91 (1 H, q, *J* 7.5 Hz, C*H*Me), 3.45 (4 H, br s, 2CH$_2$), 1.65 (3 H, d, Me); δ_C($CDCl_3$) 233.8 (CO), 146.2, 145.1, 144.7, 144.0 (uncomplexed aromatic C and =*C*CHMe), 130.7, 128.9, 126.1, 124.8, 123.9, 119.4 (uncomplexed aromatic CH and =CH), 116.0, 114.5 (complexed aromatic C), 90.9, 90.3, 90.2, 87.8 (complexed aromatic CH), 37.7, 37.6 (CH$_2$), 31.4 (*C*HMe), 18.8 (Me). NMR minor isomer: δ_H($CDCl_3$) 7.65–7.00 (4 H, m, uncomplexed aromatics), 6.42 (1 H, br s, =CH), 6.30 (1 H, br s, =CH), 5.73 (1 H, d, *J* 7), 5.63 (1 H, d, *J* 7), 5.34 (1 H, m), 5.03 (1 H, t, *J* 7) (complexed aromatics), 3.91 (1 H, q, *J* 7.5 Hz, C*H*Me), 3.36 (4 H, br s, 2CH$_2$), 1.62 (3 H, d, Me); δ_C($CDCl_3$) 233.7 (CO), 146.2, 145.0, 144.7, 144.2 (uncomplexed aromatic C and =*C*CHMe), 131.0, 129.6, 126.0, 124.7, 123.8, 119.4 (uncomplexed aromatic CH and =CH), 115.4, 114.5 (complexed aromatic C), 92.8, 89.8, 88.4, 88.0 (complexed aromatic CH), 37.8, 37.7 (CH$_2$), 32.0 (*C*HMe), 18.9 (Me).

Synthesis of [$Cr\{\eta^6\text{-}CH_3CH(C_9H_7)_2\}(CO)_3$] **4**.—The compound [$Cr(CO)_3(NH_3)_3$] was synthesised from [$Cr(CO)_6$] by the published procedure.[14] A mixture of 1,1-bis(3-indenyl)ethane (1.06 g, 4.10 mmol) and [$Cr(CO)_3(NH_3)_3$] (1 g, 5.34 mmol) in dioxane (50 cm^3) was heated under reflux (10 h) with stirring under nitrogen and in the dark. The solvent was removed *in vacuo* without any heat to leave a green solid which was chromatographed on alumina. A yellow band eluted with dichloromethane–light petroleum (3:2) and removal of the solvent gave the desired product **4** as a yellow solid (1.30 g, 72%) (Found: C, 69.5; H, 4.7. $C_{23}H_{18}CrO_3$ requires C, 70.0; H, 4.6%); *m/z* 394 (M^+, 9), 310 (M − 3 CO, 53), 308 ([M − C_2H_4, CH_3]$^+$, 57), 258 [M − $Cr(CO)_3$, 33], 143 [M − $Cr(CO)_3C_9H_7$, 100%]; $\tilde{\nu}_{max}$/cm^{-1} (CO) 1960vs, 1880vs (br) (CH_2Cl_2). NMR major isomer: δ_H($CDCl_3$) 7.65–7.00 (4 H, m, uncomplexed aromatics), 6.30 (1 H, br s, =CH), 6.21 (1 H, br s, =CH), 5.81 (1 H, d, *J* 6), 5.61 (1 H, d, *J* 6), 5.26 (1 H, m), 5.13 (1 H, t, *J* 6) (complexed aromatics), 3.91 (1 H, br m, C*H*Me), 3.46 (4 H, br s, 2CH$_2$), 1.64 (3 H, d, *J* 7.5 Hz, Me); δ_C($CDCl_3$) 233.7 (CO), 145.1, 144.7, 144.6, 143.9 (uncomplexed aromatic C and =*C*CHMe), 130.6, 129.4, 126.1, 124.8, 123.9, 119.4 (uncomplexed aromatic CH and =CH), 115.3, 114.4 (complexed aromatic C), 90.8, 90.3, 90.1, 87.8 (complexed aromatic CH), 37.8, 37.7 (CH$_2$), 31.4 (*C*HMe), 18.8 (Me). NMR minor isomer: δ_H($CDCl_3$) 7.65–7.00 (4 H, m, uncomplexed aromatics), 6.44 (1 H, br s, =CH), 6.28 (1 H, br s, =CH), 5.73 (1 H, d, *J* 6.5), 5.66 (1 H, d, *J* 6.5), 5.36 (1 H, m), 5.04 (1 H, t, *J* 6.5) (complexed aromatics), 3.91 (1 H, br m, C*H*Me), 3.36 (4 H, br s, 2CH$_2$), 1.62 (3 H, d, *J* 7 Hz, Me); δ_C($CDCl_3$) 233.6 (CO), 146.2, 144.8, 144.5, 144.2 (uncomplexed aromatic C and =*C*CHMe), 130.9, 128.8, 126.0, 124.7, 123.8, 119.5 (uncomplexed aromatic CH and =CH), 116.0, 114.4 (complexed aromatic C), 92.7, 89.7, 88.4, 88.0 (complexed aromatic CH), 37.7, 37.6 (CH$_2$), 32.0 (*C*HMe), 18.9 (Me).

Crystal-structure Determination of [$\{Ru(CO)_2\}_2\{\eta^5:\eta^5\text{-}CH_3CH(C_9H_6)_2\}$] **1**.—Suitable crystals of **1** were grown by slow evaporation under nitrogen of a solution made up from acetone–dichloromethane (1 : 1). The crystals formed as orange bricks; crystal dimensions 0.15 × 0.17 × 0.25 mm.

Crystal data. $C_{24}H_{16}O_4Ru_2$, M = 570.53, trigonal, space group $P3_121$ ($D_3{}^4$, no. 152), a = 10.226(6), c = 16.896(8) Å, γ = 120°, U = 1530.3(14) Å^3, Z = 3, D_c = 1.857 g cm^{-3}, λ(Mo-Kα) = 0.710 69 Å, μ(Mo-Kα) = 14.80 cm^{-1}, F(000) = 839.90.

Table 1 Atom coordinates (× 10^4) for compound **1**

Atom	*x*	*y*	*z*
Ru(1)	6 402(1)	5 336(1)	553(1)
O(1)	4 097(7)	6 144(7)	1 077(3)
O(2)	3 739(7)	2 301(7)	939(4)
C(1)	4 848(8)	5 985(8)	622(4)
C(2)	4 792(11)	3 423(11)	794(5)
C(3)	8 005(9)	5 937(12)	1 549(5)
C(4)	8 097(10)	7 249(12)	1 285(6)
C(5)	8 636(9)	7 561(10)	500(6)
C(6)	9 019(8)	6 416(9)	299(4)
C(7)	8 602(9)	5 385(10)	933(5)
C(8)	8 843(11)	4 142(11)	882(6)
C(9)	9 556(11)	4 057(12)	230(6)
C(10)	9 984(10)	5 065(12)	−389(6)
C(11)	9 704(10)	6 211(12)	−366(5)
C(12)	8 971(24)	8 764(18)	−146(14)
C(13)	9 928(24)	10 343(18)	194(23)

Atoms C(12) and C(13), and their associated hydrogen atoms, have occupancies of 0.5, since they constitute one of two symmetry related sites of a disordered CH$_3$CH fragment which bridges two symmetry related indenyl ligands across the crystallographic C_2 axis at (x,x,0).

Table 2 Bond lengths (Å) and angles (°) for compound **1**

Ru(1)–C(1)	2.009(10)	Ru(1)–C(2)	1.866(8)
Ru(1)–C(3)	2.212(9)	Ru(1)–C(4)	2.230(9)
Ru(1)–C(5)	2.282(7)	Ru(1)–C(6)	2.369(8)
Ru(1)–C(7)	2.316(10)	Ru(1)–Ru(1a)	2.656(2)
Ru(1)–C(1a)	2.039(7)	O(1)–C(1)	1.154(11)
O(2)–C(2)	1.140(9)	C(3)–C(4)	1.372(18)
C(3)–C(7)	1.456(15)	C(4)–C(5)	1.410(14)
C(5)–C(6)	1.448(16)	C(5)–C(12)	1.549(24)
C(5)–C(12a)	1.506(28)	C(6)–C(7)	1.411(12)
C(6)–C(11)	1.394(14)	C(7)–C(8)	1.414(17)
C(8)–C(9)	1.347(16)	C(9)–C(10)	1.376(15)
C(10)–C(11)	1.338(20)	C(12)–C(13)	1.521(26)
C(1)–Ru(1)–C(2)	84.5(5)	C(1)–Ru(1)–C(1a)	92.0(4)
C(2)–Ru(1)–C(1a)	89.4(3)	Ru(1)–C(1)–O(1)	141.1(7)
Ru(1)–C(1)–Ru(1a)	82.0(3)	O(1)–C(1)–Ru(1a)	136.8(8)
Ru(1)–C(2)–O(2)	174.8(12)	C(4)–C(3)–C(7)	108.0(8)
C(3)–C(4)–C(5)	110.8(11)	C(4)–C(5)–C(6)	105.8(9)
C(4)–C(5)–C(12)	138.4(14)	C(6)–C(5)–C(12)	115.8(12)
C(4)–C(5)–C(12a)	115.3(13)	C(6)–C(5)–C(12a)	138.7(12)
C(5)–C(6)–C(7)	108.8(8)	C(5)–C(6)–C(11)	132.1(9)
C(7)–C(6)–C(11)	119.1(10)	C(3)–C(7)–C(6)	106.4(9)
C(3)–C(7)–C(8)	133.5(9)	C(6)–C(7)–C(8)	120.1(9)
C(7)–C(8)–C(9)	116.8(10)	C(8)–C(9)–C(10)	123.5(13)
C(9)–C(10)–C(11)	120.4(11)	C(6)–C(11)–C(10)	119.9(9)
C(5)–C(12)–C(13)	110.3(20)	C(5)–C(12)–C(5a)	112.9(13)
C(13)–C(12)–C(5a)	108.8(19)		

Structure analysis and refinement. Three-dimensional, room temperature X-ray data were collected in the range 3.5 < 2θ < 50° on a Nicolet R3 diffractometer by the ω-scan method. The 1666 reflections (of 3019 measured assuming only monoclinic symmetry) for which $|F|/\sigma(|F|) > 3.0$ were corrected for Lorentz and polarisation effects, and for absorption by analysis of three azimuthal scans (minimum and maximum transmission coefficients 0.762 and 0.813). The structure was solved by conventional Patterson and Fourier techniques and refined by blocked-cascade least-squares methods. The CH$_3$CH fragment which bridges the two indenyl groups, is disordered between two sites across the crystallographic C_2 axis, and constraints were applied to the bond lengths and angles of this fragment. Hydrogen atoms were included in calculated positions with isotropic thermal parameters related to those of the supporting atom. Refinement converged at a final R 0.0384 (145 parameters, final mean and maximum δ/σ 0.012 and 0.170 respectively), with allowance for the anisotropic thermal vibrations of all non-hydrogen atoms. The space group was distinguished from the enantiomeric $P3_221$ on the basis of the more complete refinement in the chosen space group. Complex scattering factors were taken from ref. 15 and from the program package SHELXTL[16] as implemented on the Data General Nova 3 computer. Unit weights were used throughout; the final minimum and maximum difference electron density was -0.70 and $+1.12$ e Å^{-3}. Table 1 lists atomic positional parameters with estimated standard deviations and bond distances and angles are listed in Table 2.

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

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