

Synthesis and crystal structure of the first metallabicyclic cluster 2a-ruthenacyclopent[*c,d*]indenyl-triruthenium undecacarbonyl $\text{Ru}_4(\text{CO})_{11}\text{C}_{15}\text{H}_{14}$ ¹

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Abstract

The metallabicyclic cluster $\text{Ru}_4(\text{CO})_{11}\text{C}_{15}\text{H}_{14}$ **1** has been obtained from the reaction between $\text{Ru}_3(\text{CO})_{12}$ with 1,3,5-triisopropenylbenzene and represents the first cluster containing a metallabicyclic unit. The organo-ligand is bonded through 3π and 4σ bonds to the Ru_3 metal triangle, donating a total of ten electrons. In all, four C-H bonds have been cleaved in the original ligand.

Keywords: Ruthenium; Cluster; Metallacycle; Bond activation; Cyclometallation; Orthometallation

1. Introduction

The structure and bonding of metallacycle complexes have been under extensive investigation recently because of the diverse array of stoichiometric and catalytic reactions exhibited by this class of compounds [1]. There now exists a wide range of metallacyclopentadienyl complexes but only a few are known to contain a metallabicyclopentadienyl system [2] and no examples of metallabicyclic compounds with more than two metal atoms are known. Earlier, we reported the ruthenacyclopentadienyl cluster $\text{Ru}_3(\text{CO})_8\text{C}_9\text{H}_8$ which is formed on heating the face-capped compound $\text{Ru}_3(\text{CO})_8\text{C}_9\text{H}_{10}$ [3] and which in turn was obtained from the reaction of $\text{Ru}_3(\text{CO})_{12}$ with methylstyrene. This is described in Scheme 1 [4].

In this paper we wish to report the synthesis and full characterisation of the 6-isopropenyl-2a-ruthenacyclopent[*c,d*]indenyl cluster $\text{Ru}_4(\text{CO})_{11}\text{C}_{15}\text{H}_{14}$, **1**, which has been obtained from the reaction of $\text{Ru}_3(\text{CO})_{12}$ with 1,3,5-triisopropenylbenzene in octane followed by separation by thin-layer-chromatography.

2. Results and discussion

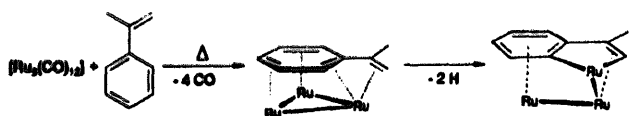
The reaction of $\text{Ru}_3(\text{CO})_{12}$ with 1,3,5-triisopropenylbenzene in octane under reflux for 3 h results in a dark brown solution. Separation of the products from this solution by chromatography on silica, eluting with a solution of 30% dichloromethane hexane gives several compounds including as the top orange band the new cluster $\text{Ru}_4(\text{CO})_{11}\text{C}_{15}\text{H}_{14}$ **1**.

The ¹H NMR spectrum of **1** in CDCl₃ shows eight signals over the range 7.68 to 2.17 ppm. The singlet at the highest frequency may be considered to arise from the hydrogen on the α carbon C(8). The two doublets observed at δ 7.32 and 6.76 ppm are coupled to each other with a coupling constant $J = 1.4$ Hz. These may be assigned to the two protons H3 and H5 on the six-membered ring. The signals at δ 5.48 and 5.22 are considered to arise from the CH₂ protons of the isopropenyl group together with the signal at δ 2.17 for the methyl group. Other singlets at δ 2.73 and 2.31 ppm, each with an integral of 3H, may be assigned to the methyl groups C(9) and C(12). The FAB MS shows the molecular ion at 906 amu and the loss of eight CO ligands; behaviour typical for such clusters.

A crystal of **1** suitable for single-crystal X-ray diffraction analysis was grown from a CH₂Cl₂-hexane solution, and the molecular structures of **1** is shown in

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¹ Dedicated to Professor Jörn Müller on the occasion of his 60th birthday.



Scheme 1. The preparation of $\text{Ru}_3(\text{CO})_8\text{C}_9\text{H}_8$ from $\text{Ru}_3(\text{CO})_{12}$ and methylstyrene.

Fig. 1 and Fig. 2. Relevant structural parameters are listed in Tables 1–3.

Crystal data for $\text{Ru}_4(\text{CO})_{11}\text{C}_{15}\text{H}_{14} \cdot \text{C}_6\text{H}_{14}$, $M = 992.82$, Triclinic, space group $P1$. $a = 9.754(6)$, $b = 13.135(7)$, $c = 14.369(7)$ Å, $\alpha = 69.81(3)$, $\beta = 79.24(4)$, $\gamma = 82.57(4)^\circ$, $V = 1693(2)$ Å³ (from 20 values of 28 reflections ($28 \leq 2\theta \leq 29^\circ$) measured at $\pm\omega$, $\lambda = 0.71073$ Å), $Z = 2$, $D_c = 1.947$ g cm⁻³, $F(000) =$

968. orange needle, $0.64 \times 0.16 \times 0.08$ mm³, $\mu(\text{Mo} - \text{K}\alpha) = 1.808$ mm⁻¹. Stöe stadi 4 diffractometer, equipped with an Oxford Cryosystem low temperature device operating at 150 K. $\omega\theta$ scans, with ω -scan width ($1.2 + 0.35 \tan \theta$)°, graphite monochromated Mo - $\text{K}\alpha$ radiation, 4442 reflections measured ($5 \leq 2\theta \leq 45^\circ$). Following the application of an absorption correction (based on ψ -scans, $T_{\min} = 0.164$, $T_{\max} = 0.716$). The structure was solved by direct methods (SIR 92 [5]) and refined against F^2 (SHELXL 93 [5]); H-atoms were placed partly in calculated or founded positions and refined with an restrained distance to the corresponding C-atom. The refinement converged with anisotropic displacement parameters on all non-H-atoms to a conventional R (based on F 4438 data with $F > 4\sigma(F)$) of 4.69% and $wR_2 = 10.01\%$ (based on all data included in the

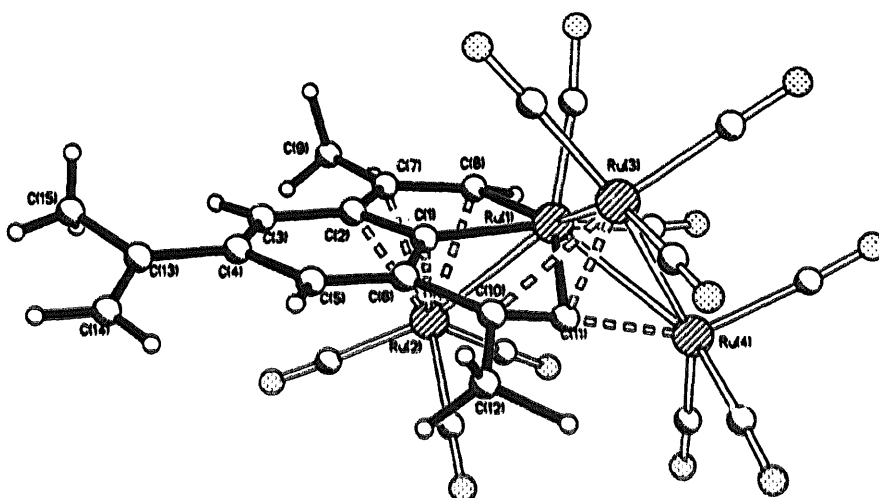


Fig. 1. The molecule structure of 1.

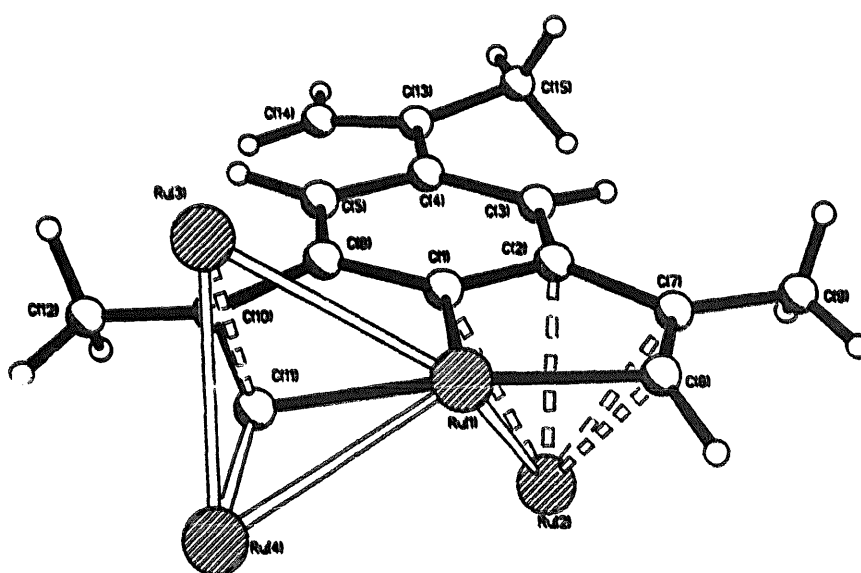


Fig. 2. The molecule structure of 1 without carbonyl ligands.

Table 1
Crystal data and structure refinement for $\text{Ru}_4(\text{CO})_{11}\text{C}_{15}\text{H}_{14}\cdot\text{C}_6\text{H}_{14}$

Empirical formula	$\text{H}_{28}\text{O}_{11}\text{Ru}_4$
Formula weight	992.82
Temperature (K)	150(2)
Wavelength (Å)	0.71073
Crystal system	Triclinic
Space group	$P\bar{1}$
Unit cell dimensions	
a (Å)	9.754(6)
b (Å)	13.135(7)
c (Å)	14.369(7)
α (deg)	69.81(3)
β (deg)	79.24(4)
γ (deg)	82.57(4)
Volume (Å ³)	1693(2)
Z	2
Density (calc) (g cm ⁻³)	1.947
Absorption coefficient (mm ⁻¹)	1.808
$F(000)$	968
Crystal size (mm ³)	$0.64 \times 0.16 \times 0.08$
θ range for data collection (deg)	2.54 to 22.52
Index ranges	$-10 \leq h \leq 10,$ $-12 \leq k \leq 14,$ $0 \leq l \leq 15$
Reflections collected	4442
Independent reflections	4442 ($R_{\text{int}} = 0.0000$)
Max. and min. transmission	0.716 and 0.164
Refinement method	Full-matrix least squares on F^2
Data/restraints/parameters	4438/174/409
Goodness-of-fit on F^2	0.986
Final R indices ($I > 2\sigma(I)$)	$R_1 = 0.0469,$ $wR_2 = 0.0859$
R indices (all data)	$R_1 = 0.0902,$ $wR_2 = 0.1001$
Largest diff. peak and hole (e Å ⁻³)	0.998 and -0.812

refinement) for 409 parameters. The final ΔF map max. and min. were 0.998 and -0.812 e Å^{-3} respectively.

The metal core is unusual in that it consists of a triangular array of three ruthenium atoms attached through one vertex (Ru(1)) to a fourth Ru atom (Ru(2)) which is out of the plane of the Ru_3 -triangle. Atom Ru(1) forms a bridgehead in a nearly flat cyclopent[*c,d*]indenyl ring system and is bound to all other three ruthenium atoms and two carbonyl groups.

Both five-membered metallacycles are π -bonded to other ruthenium atoms. One is η^5 -coordinated to Ru(2) and the other is η^3 -bonded to Ru(3) which lie on opposite sides of the ligand plane. Atom Ru(4) bonds to both Ru(1) and Ru(3) within the metal triangle and is also σ -bonded to carbon atom C(11). This σ -bond must arise from the cleavage the original C–H bond on this carbon. This Ru–C bond distance is 1.947(10) Å, which is short for an Ru–C bond when compared with the other three observed σ -bonds of Ru(1) (2.050(10), 2.090(10) and 2.122(10) Å). The coordination of the

Ru(2) and Ru(3) to the five-membered rings restricts the π system of the organo-moiety essentially to one resonance form and do not allow it to delocalise. This is reflected in the alternation of the C–C bond lengths

Table 2
Atomic coordinates and equivalent isotropic displacement parameters (Å²) for $\text{Ru}_4(\text{CO})_{11}\text{C}_{15}\text{H}_{14}\cdot\text{C}_6\text{H}_{14}$

	x	y	z	U_{eq}^a
Ru(1)	0.28950(8)	0.88859(7)	0.27654(6)	0.0162(2)
Ru(2)	0.54227(8)	0.94596(7)	0.28897(6)	0.0164(2)
Ru(3)	0.18766(8)	0.68860(7)	0.29760(6)	0.0174(2)
Ru(4)	0.17178(8)	0.74701(7)	0.46671(6)	0.0192(2)
C(1)	0.4770(10)	0.8365(8)	0.2100(7)	0.016(2)
C(2)	0.5672(10)	0.9136(8)	0.1366(7)	0.016(2)
C(3)	0.6925(10)	0.8761(9)	0.0877(7)	0.023(3)
C(4)	0.7293(10)	0.7673(8)	0.1095(7)	0.018(2)
C(5)	0.6408(11)	0.6903(9)	0.1849(7)	0.022(3)
C(6)	0.5205(10)	0.7245(8)	0.2355(7)	0.016(2)
C(7)	0.5127(10)	1.0255(8)	0.1275(7)	0.014(2)
C(8)	0.3827(10)	1.0327(8)	0.1888(7)	0.018(2)
C(9)	0.5844(12)	1.1235(9)	0.0552(8)	0.030(3)
C(10)	0.4236(10)	0.6595(8)	0.3218(7)	0.018(2)
C(11)	0.3327(10)	0.7270(8)	0.3702(7)	0.016(2)
C(12)	0.4692(11)	0.5435(8)	0.3747(8)	0.028(3)
C(13)	0.8616(11)	0.7289(9)	0.0584(8)	0.026(3)
C(14)	0.9160(12)	0.6258(10)	0.0942(10)	0.045(4)
C(15)	0.9387(11)	0.8083(9)	$-0.0304(8)$	0.031(3)
C(16)	0.1512(11)	0.9697(9)	0.3450(8)	0.021(3)
C(17)	0.1792(10)	0.8969(8)	0.1790(8)	0.017(2)
C(21)	0.4651(11)	1.0312(9)	0.3717(8)	0.024(3)
C(22)	0.7323(12)	0.9923(9)	0.2622(8)	0.023(3)
C(23)	0.5769(10)	0.8177(9)	0.4037(8)	0.017(2)
C(31)	0.2371(11)	0.6695(9)	0.1677(9)	0.026(3)
C(32)	$-0.0109(13)$	0.7269(9)	0.3002(8)	0.025(3)
C(33)	0.1562(11)	0.5425(10)	0.3746(9)	0.025(3)
C(41)	0.2359(12)	0.8124(9)	0.5509(9)	0.030(3)
C(42)	0.1639(10)	0.6110(10)	0.5639(8)	0.025(3)
C(43)	$-0.0288(13)$	0.7965(9)	0.4898(8)	0.029(3)
O(16)	0.0788(8)	1.0340(6)	0.3720(6)	0.035(2)
O(17)	0.1225(7)	0.9222(6)	0.1099(6)	0.030(2)
O(21)	0.4128(8)	1.0814(7)	0.4205(6)	0.038(2)
O(22)	0.8394(8)	1.0212(7)	0.2449(6)	0.040(2)
O(23)	0.5960(8)	0.7415(6)	0.4674(6)	0.033(2)
O(31)	0.2702(9)	0.6587(7)	0.0914(6)	0.042(2)
O(32)	$-0.1276(8)$	0.7452(6)	0.3031(6)	0.035(2)
O(33)	0.1386(8)	0.4543(7)	0.4229(6)	0.038(2)
O(41)	0.2751(9)	0.8517(8)	0.5980(6)	0.051(2)
O(42)	0.1618(8)	0.5258(7)	0.6253(6)	0.038(2)
O(43)	$-0.1416(8)$	0.8229(7)	0.4985(6)	0.041(2)
C(1S)	0.9133(13)	0.657(2)	0.7861(13)	0.118(7)
C(2S) ^b	0.7761(14)	0.694(2)	0.7459(14)	0.070
C(3S) ^b	0.6636(14)	0.640(2)	0.8276(14)	0.070
C(4S) ^b	0.5245(14)	0.670(2)	0.789(2)	0.070
C(5S) ^b	0.423(2)	0.593(2)	0.866(2)	0.070
C(6S)	0.2776(12)	0.6315(14)	0.8453(13)	0.104(6)
C(2SA) ^b	0.771(2)	0.634(3)	0.8437(14)	0.070
C(3SA) ^b	0.6684(14)	0.649(3)	0.772(2)	0.070
C(4SA) ^b	0.5298(14)	0.617(2)	0.839(2)	0.070
C(5SA) ^b	0.4137(14)	0.673(2)	0.781(2)	0.070

^a U_{eq} is defined as one-third of the trace of the orthogonalized U_{ij} tensor. ^b Disordered part, occupancy 0.548 and 0.452 for the atom A.

within the aromatic ring. Thus, shorter distances are found for C(3)–C(4) 1.367(14) and C(5)–C(6) 1.370(13) Å and longer distances for C(2)–C(3) 1.412(13), C(1)–C(6) 1.415(13) and C(4)–C(5) 1.436(14) Å.

The η^5 -coordinated five-membered ring is almost coplanar with the arene. However, the other five-membered ring is bent with an angle of 31.3° along the Ru(1)–C(10) vector. This deformation in the η^3 -coordinated ring is unusual and may be compared with the structures of $\text{Ru}_3(\text{CO})_8\text{C}_8\text{H}_6$ [7] and $\text{Ru}_3(\text{CO})_8\text{C}_9\text{H}_8$ [3] where the π -coordinated carbon atoms lie in the arene plane. This difference is almost certainly caused by the replacement of a carbonyl ligand by the $\text{Ru}(\text{CO})_2$ unit on Ru(3). The steric interaction between this carbonyl fragment and the organic ligand twists the Ru(3), C(10), C(11) unit out of the plane. In the other clusters the $\text{Ru}(\text{CO})_2$ unit is η^6 -coordinated to the benzene ring and pulls atom Ru(3) in the opposite direction.

The third and only uncoordinated isopropenyl group is not coplanar with the arene and the dihedral angle is 14.3°. Similar angles have been observed for other metallacycle clusters [3,6] containing isopropenyl groups. This loss of coplanarity cannot be attributed to

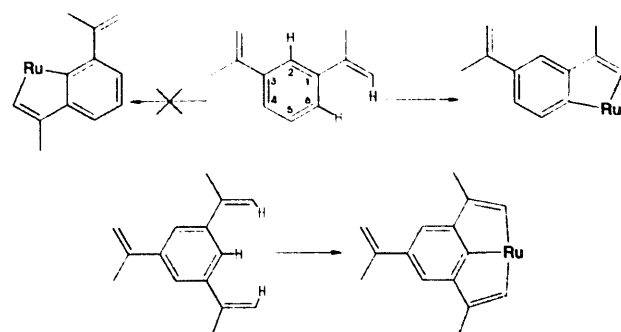


Fig. 3. Possible C–H cleaving in 1,3-diisopropenylbenzene and in 1,3,5-triisopropenylbenzene.

molecular packing in the crystal, and the reason remains unclear.

The molecular structure exhibits some similarities with others reported earlier involving similar ligands [3,6]. Thus, the formation of a metallacycle has been observed in the reaction of $\text{Ru}_3(\text{CO})_{12}$ with both methylstyrene and 1,3-diisopropenylbenzene. The five-membered metallacycle may coordinate in either the η^3 - or η^5 -coordination modes as represented in 1. However, in this case the arene is not involved in coordination to the central cluster unit.

The formation of the metallabicyclic is unexpected, since in the related reaction with 1,3-diisopropenylbenzene no ortho-metallation was observed in the 2-position between the unsaturated side arms. The C–H bond is generally activated and cleaved in the 6-position (see Fig. 3). We suggest that this change in behaviour is brought about by the steric constraints imposed by the triisopropenylbenzene. This would explain the low yield of 1, since there are no other bonding possibilities other than the cleavage of the C–H bond in the C(2) position when the 1,3,5-triisopropenylbenzene ligand is involved.

The cleavage of three C–H bonds is required to form the metallabicyclic. The fourth C–H bond undergoes cleavage by Ru(4) on C(11) to form the remaining σ -bond. Thus, the electron count for 1 is 64 VE; in agreement with the observed four Ru–Ru bonds.

3. Conclusion

In conclusion, it is clear that the availability of additional C=C bonds in the side arm of the substituted arene ligand provides a range of new bonding capabilities. As in many other reactions of ruthenium clusters with organo-species, the chemistry is dominated by the cleavage of C–H bonds with the coincidental formation of Ru–C bonds. In this case, this leads to the formation of the first cluster containing a metallabicyclic unit.

Table 3
Selected bond lengths (Å) and angles (deg) for $\text{Ru}_4(\text{CO})_{11}\text{C}_{15}\text{H}_{14} \cdot \text{C}_6\text{H}_{14}$

Ru(1)–Ru(2)	2.723(2)	C(1)–Ru(1)–C(8)	76.2(4)
Ru(1)–Ru(3)	2.827(2)	C(1)–Ru(1)–C(11)	74.5(4)
Ru(1)–Ru(4)	2.849(2)	C(8)–Ru(1)–C(11)	143.1(4)
Ru(3)–Ru(4)	2.763(2)	C(6)–C(1)–C(2)	118.7(9)
Ru(1)–C(1)	2.050(10)	C(3)–C(2)–C(1)	119.5(9)
Ru(1)–C(8)	2.090(10)	C(3)–C(2)–C(7)	129.3(9)
Ru(1)–C(11)	2.122(10)	C(1)–C(2)–C(7)	111.1(9)
Ru(2)–C(8)	2.244(10)	C(4)–C(3)–C(2)	120.9(10)
Ru(2)–C(7)	2.249(9)	C(3)–C(4)–C(5)	119.4(9)
Ru(2)–C(1)	2.323(9)	C(3)–C(4)–C(13)	120.5(9)
Ru(2)–C(2)	2.334(9)	C(5)–C(4)–C(13)	120.1(9)
Ru(3)–C(11)	2.107(9)	C(6)–C(5)–C(4)	120.9(10)
Ru(3)–C(10)	2.354(10)	C(5)–C(6)–C(1)	120.3(9)
Ru(4)–C(11)	1.947(10)	C(5)–C(6)–C(10)	129.0(9)
C(1)–C(6)	1.415(13)	C(1)–C(6)–C(10)	110.6(8)
C(1)–C(2)	1.435(13)	C(8)–C(7)–C(2)	113.8(8)
C(2)–C(3)	1.412(13)	C(8)–C(7)–C(9)	123.2(9)
C(3)–C(4)	1.367(14)	C(2)–C(7)–C(9)	123.0(9)
C(4)–C(5)	1.436(14)	C(11)–C(10)–C(6)	111.5(9)
C(5)–C(6)	1.370(13)	C(11)–C(10)–C(12)	123.7(9)
C(7)–C(2)	1.467(13)	C(6)–C(10)–C(12)	118.3(9)
C(7)–C(8)	1.415(13)	C(14)–C(13)–C(4)	121.2(10)
C(7)–C(9)	1.505(13)	C(14)–C(13)–C(15)	120.1(10)
C(10)–C(6)	1.483(13)	C(4)–C(13)–C(15)	118.6(9)
C(10)–C(11)	1.431(13)		
C(10)–C(12)	1.500(14)		
C(13)–C(4)	1.480(14)		
C(13)–C(14)	1.35(2)		
C(13)–C(15)	1.486(14)		

4. Experimental

4.1. General procedures and materials

All reactions were carried out in octane purchased from Aldrich Chemicals, and under a nitrogen atmosphere. The separation of products was carried out using standard laboratory-grade solvents. Infrared spectra were recorded on a Perkin–Elmer 1710 Fourier Transform spectrometer. Mass spectra were obtained by positive fast atom bombardment on a Kratos MS50TC, calibrated with CsI. The ^1H NMR spectra were recorded using a Bruker AM360 spectrometer referenced to internal TMS.

The cluster $\text{Ru}_3(\text{CO})_{12}$ was prepared according to literature method, and 1,3,5-triisopropenylbenzene was prepared via bromination and HBr elimination from 1,3,5-triisopropylbenzene [8]. ^1H NMR (CDCl_3): δ 7.50 ppm, s, 3H; δ 5.42 ppm, m, 3H; δ 5.15 ppm, quintet, $J = 1.4$ Hz, 3H; δ 2.20 ppm, dd, $J = 1.4 + 0.7$ Hz, 9H.

4.2. Thermolysis of $\text{Ru}_3(\text{CO})_{12}$ with 1,3,5-triisopropenylbenzene in octane

$\text{Ru}_3(\text{CO})_{12}$ (400 mg) were heated in octane with an excess (0.5 ml) of 1,3,5-triisopropenylbenzene for 3 h. The reaction was monitored by IR spectroscopy and spot TLC plates. The solvent was removed in vacuo and the reaction mixture chromatographed and the products separated using preparative TLC with a solvent mixture 7:3 hexane–dichloromethane.

The top orange band contained 5.0 mg (1.2%) of 1.

Spectroscopic data for 1: ^1H NMR (CDCl_3): H1 (δ 7.32 ppm, d, $J = 1.4$ Hz); H3 (δ 6.76 ppm, d, $J = 1.4$ Hz); H8 (δ 7.68 ppm, s); H9 (δ 2.73 ppm, s, 3H); H12 (δ 2.31 ppm, s, 3H); H14a (δ 5.48 ppm, m); H14b (δ 5.22 ppm, m); H15 (δ 2.17 ppm, t, $J = 0.7$ Hz, 3H). MS (FAB): $m/z = 906$ [M^+]. Calc.: 909. IR (CH_2Cl_2): $\nu(\text{cm}^{-1})$ 2083(w), 2068(s), 2038(vs), 2006(vs), 1965(m), 1947(w), 1928(w).

5. Supplementary material

Tables of anisotropic thermal parameters and fractional atomic coordinates for the non-hydrogen atoms, tables of fractional atomic coordinates for the hydrogen atoms, a complete list of bond lengths and angles, and thermal ellipsoid drawings are available. Ordering information is given in any current masthead page.

Acknowledgements

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References

- [1] N.E. Schore, *Chem. Rev.*, 88 (1988) 1081. J.P. Collman, L.S. Hegeudus, J.R. Norton and R.G. Finke, *Principles and Applications of Organotransition Metal Chemistry*, University Science Books, Mill Valley, CA, 1987, Chapter 9. E. Lindner, *Adv. Heterocycl. Chem.*, 39 (1986) 237. K.P.C. Vollhardt, *Angew. Chem.*, 96 (1984) 525; *Angew. Chem. Int. Ed. Engl.*, 23 (1984) 539. S.D. Chappell and D.J. Cole-Hamilton, *Polyhedron*, 1 (1982) 729.
- [2] G.S.D. King, *Acta Crystallogr.*, 15 (1962) 243; J.M. O'Connor, L. Pu and R. Chadha, *Angew. Chem. Int. Ed. Engl.*, 29 (1990) 543; E. Sappa, L. Milone and G.D. Andretti, *Inorg. Chim. Acta*, 13 (1975) 67.
- [3] P. Dyson, P. Escarpa, B.F.G. Johnson and A. Blake, *Inorg. Chim. Acta*, 241 (1996) 11.
- [4] P. Dyson, P. Escarpa, B.F.G. Johnson, A. Blake, J.J. Byrne, F. Grepioni, E. Parisini and D. Reed, *Organometallics*, 10 (1995) 4892.
- [5] (a) A. Altomare, G. Cascarano, C. Giacomazzo and A. Guagliardi, SIR 92, *J. Appl. Crystallogr.*, 26 (1993) 343. (b) G.M. Sheldrick, SHELX 93, University of Göttingen, Germany, 1993. (c) *International Tables for X-Ray Crystallography*, Vol. IV, Kynoch Press, Birmingham, UK, 1975, p. 99–149.
- [6] P. Dyson, P. Escarpa, B.F.G. Johnson, A. Blake, D. Braga and E. Parisini, *J. Chem. Soc. Dalton Trans.*, (1995) 3431.
- [7] A.J. Arce, Y. De Sanctis, A. Karam and A.J. Deeming, *Angew. Chem. Int. Ed.*, 33 (1994) 1381.
- [8] F. Effenberger and W. Kurtz, *Chem. Ber.*, 106 (1973) 511.