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# 2,3,4,7-Tetrahydro-1*H*-inden-2-ol: synthesis, molecular structure and coordination chemistry

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**Abstract**

2,3,4,7-Tetrahydro-1*H*-inden-2-ol (**1**) has been synthesised by Birch reduction of indan-2-ol. A single-crystal X-ray structure analysis of **1** shows the compound to exist as hydrogen-bonded polymers along the *c*-axis due to the presence of a series of hydrogen-bonds between hydroxo functions. The ferrocene ester derivatives, ferrocene carboxylic acid 2,3,4,7-tetrahydro-1*H*-inden-2-yl ester (**2**) and ferrocene carboxylic acid indan-2-yl ester (**3**) have been prepared by peptidic coupling of ferrocene carboxylic acid with **1** and with indan-2-ol, respectively. The single-crystal X-ray structure analyses of **2** and **3** reveal in both cases the cyclopentadienyl rings to adopt an eclipsed conformation with the indenyl substituent being rotated out of the Cp ester plane by almost 90°, allowing no efficient interaction between the π-system of the Cp ring and the indene moiety. The dienyl derivative **2** reacts with $RuCl_3 \cdot nH_2O$ in refluxing ethanol to afford $[Ru(arene)Cl_2]_2$ (**4**) (arene = ferrocene carboxylic acid indan-2-yl ester) as a mixture of isomers.

*Keywords:* Arene ligand; Ferrocene carboxylic acid; Ferrocene derivative; Ruthenium

## 1. Introduction

A wide variety of methods for the synthesis of ferrocenoyl ester derivatives are found in the literature, among them reaction of $FeCl_2$ with cyclopentadienyl ester derivatives [1], condensation of chlorocarbonylferrocene with hydroxo derivatives [2], oxazoline ring-opening reaction of ferrocenoyloxazole derivatives [3], reaction of ferrocene carboxylic acid with diazomethane [4], reaction of ferrocenoyl imidazolide with hydroxo derivatives [5], haloferrocene with acids [6], reaction of ferrocene carboxylic acid with hydroxo derivatives [7], and reaction of 2-(*N*,*N*-dimethylaminoethylferrocene)carboxylic acid with sodium bicarbonate and methyl iodide [8].

Recently, we reported the synthesis of ferrocene ester derivatives by standard peptidic coupling conditions [9]. The method has been proven to be an efficient synthetic route for the synthesis of ferrocene ester derivatives [10]. Herein, we report the synthesis of two new ferrocenoyl ester derivatives by coupling of ferrocene carboxylic acid with 2,3,4,7-tetrahydro-1*H*-inden-2-ol and indan-2-ol. The dienyl-ester derivative reacts with $RuCl_3 \cdot nH_2O$ to form a dinuclear arene–ruthenium complex incorporating ferrocenoyl groups. The single-crystal structure analyses of ferrocene carboxylic acid 2,3,4,7-tetrahydro-1*H*-inden-2-yl ester, ferrocene carboxylic acid indan-2-yl ester and the new 2,3,4,7-tetrahydro-1*H*-inden-2-ol are presented.

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## 2. Experimental

### 2.1. General

All manipulations were carried out using routine procedures, under a nitrogen atmosphere. Organic solvents were distilled under nitrogen prior to use. NMR spectra were recorded using a Varian Gemini 200 MHz spectrometer and a Bruker 400 MHz spectrometer. IR spectra were recorded on a Perkin–Elmer Spectrum One

FT-IR spectrometer (4000–400 $cm^{-1}$). Microanalyses were performed by the Laboratory of Pharmaceutical Chemistry, University of Geneva (Switzerland). Electrospray mass spectra were obtained in positive-ion mode with an LCQ Finnigan mass spectrometer. All reagents were purchased (Fluka or Aldrich) and used as received.

### 2.2. Syntheses

#### 2.2.1. 2,3,4,7-Tetrahydro-1H-inden-2-ol (1)

**1** was synthesised by sodium reduction of indan-2-ol in liquid ammonia by analogy to published methods [11]. Crystals suitable for X-ray analysis were obtained by slow evaporation of a dichloromethane solution of **1**.

*Spectroscopic data for* **1**: $^1$H NMR (400 MHz, $CDCl_3$): $\delta = 5.75$ (m, 2H, ethylenic *H*), 4.50 (m, 1H, –C*H*OH), 2.66 (m, 4H, –C=C–C$H_2$–), 2.62 (m, 2H, –C$H_2$CHOH), 2.34 (s, 1H, –CHO*H*), 2.22 (dd, 2H, $^2J = 17.02$ Hz, $^3J = 1.02$ Hz, –C$H_2$CHOH). $^{13}$C{$^1$H} NMR (100 MHz, $CDCl_3$): $\delta = 129.91$, 124.89, 70.90, 46.08, 27.70. MS (EI, acetone): *m*/*z*: 136 $[M]^+$. *Anal.* Calc. for $C_9H_{12}O$: C, 79.37; H, 8.88. Found: C, 79.41; H, 8.82%.

#### 2.2.2. Ferrocene carboxylic acid 2,3,4,7-tetrahydro-1H-inden-2-yl ester (2)

A solution of ferrocene carboxylic acid (3.30 g, 14.34 mmol), *N*,*N*-dicyclohexylcarbodiimide (4.54 g, 22.06 mmol), 4-(dimethylamino)pyridine (1.35 g, 11.03 mmol), 4-pyrrolidinopyridine (1.63 g, 11.03 mmol), and 2,3,4,7-tetrahydro-1*H*-inden-2-ol (1.50 g, 11.03 mmol) in $CH_2Cl_2$ (150 mL) was stirred under nitrogen at room temperature for 3 days. The resulting solution was filtered through celite to remove *N*,*N*-dicyclohexylurea, and the filtrate concentrated under reduce pressure. The residue was separated by column chromatography on silica gel eluting with hexane/acetone (20:1). The pure product was isolated from the first fraction, giving **2** as red-orange solid. Yield: 3.50 g (91%). Crystals suitable for X-ray analysis were grown by slow evaporation of an acetone/hexane solution of **2**.

*Spectroscopic data for* **2**: IR (KBr, $cm^{-1}$): $\nu = 1709$ vs (C=O ester), 1460m, 1277s, 1134s. $^1$H NMR (400 MHz, $CDCl_3$): $\delta = 5.80$ (m, 2H, ethylenic *H*), 5.53 (m, 1H, –C*H*O–), 4.84 (m, 2H, Cp*H*), 4.42 (m, 2H, Cp*H*), 4.24 (s, 5H, Cp*H*), 2.81 (dd, 2H, $^2J = 16.38$ Hz, $^3J = 6.14$ Hz, –C$H_2$CHO–), 2.71 (m, 4H, –C=C–C$H_2$–), 2.42 (dd, 2H, $^2J = 16.38$ Hz, $^3J = 2.36$ Hz, –C$H_2$CHO). $^{13}$C{$^1$H} NMR (50 MHz, $CDCl_3$): $\delta = 171.84$, 129.84, 129.77, 71.96, 71.46, 71.68, 70.09, 70.00, 43.01, 27.47. MS (EI, acetone): *m*/*z*: 348 $[M]^+$. *Anal.* Calc. for $C_{20}H_{20}Fe_1O_2$: C, 68.98; H, 5.79. Found: C, 68.93; H, 5.82%.

#### 2.2.3. Ferrocene carboxylic acid indan-2-yl ester (3)

A solution of ferrocene carboxylic acid (2.00 g, 8.70 mmol), *N*,*N*-dicyclohexylcarbodiimide (2.75 g, 13.38 mmol), 4-(dimethylamino)pyridine (0.82 g, 6.69 mmol), 4-pyrrolidinopyridine (0.99 g, 6.69 mmol), and indan-2-ol (0.90 g, 6.72 mmol) in $CH_2Cl_2$ (80 mL) was stirred under nitrogen at room temperature for 3 days. The resulting solution was filtered through celite to remove *N*,*N*-dicyclohexylurea, and the filtrate concentrated under reduce pressure. The residue was separated by column chromatography on silica gel eluting with hexane/acetone (20:1). The pure product was isolated from the first fraction, giving **3** as red-orange solid. Yield: 1.72 g (81%). Crystals suitable for X-ray analysis were grown by slow evaporation of an acetone/hexane solution of **3**.

*Spectroscopic data for* **3**: IR (KBr, $cm^{-1}$): $\nu = 1692$vs (C=O ester), 1459m, 1277s, 1141s. $^1$H NMR (200 MHz, $CDCl_3$): $\delta = 7.26$ (m, 4H, $H_{arom}$), 5.70 (m, 1H, –C*H*O–), 4.81 (m, 2H, Cp*H*), 4.41 (m, 2H, Cp*H*), 4.20 (s, 5H, Cp*H*), 3.44 (dd, 2H, $^2J = 16.66$ Hz, $^3J = 6.22$ Hz, –C$H_2$CHO–), 3.15 (dd, 2H, $^2J = 16.66$ Hz, $^3J = 2.19$ Hz, –C$H_2$CHO–). $^{13}$C{$^1$H} NMR (50 MHz, $CDCl_3$): $\delta = 171.93$, 140.87, 128.14, 126.05, 75.23, 73.45, 71.68, 70.92, 70.09, 40.05. MS (EI, acetone): *m*/*z*: 346 $[M]^+$. *Anal.* Calc. for $C_{20}H_{18}Fe_1O_2$: C, 69.39; H, 5.24. Found: C, 69.37; H, 5.23%.

#### 2.2.4. [Ru(arene)Cl$_2$]$_2$ (4) (arene = ferrocene carboxylic acid indan-2-yl ester)

To a solution of ruthenium trichloride hydrate (470 mg, 1.80 mmol) in ethanol (50 mL) was added **2** (2.50 g, 7.18 mmol), and the mixture was refluxed for 4 h. After cooling to room temperature, half of the volume was evaporated in vacuo, the orange-brown precipitate was filtered, washed with ether and acetone, dissolved in $CHCl_3$ and filtered through celite to eliminate insoluble particles. After evaporation to dryness, **4** was obtained as orange powder. Yield: 495 mg (53%).

*Spectroscopic data for* **4**: IR (KBr, $cm^{-1}$): $\nu = 1709$ vs (C=O ester), 1458m, 1276s, 1137s. $^1$H NMR for **4a** (400 MHz, dmso-$d_6$): $\delta = 5.88$ (m, 8H, Ru–$H_{arom}$), 5.55 (m, 2H, –C*H*O–), 4.79 (m, 4H, Cp*H*), 4.53 (m, 4H, Cp*H*), 4.26 (s, 10H, Cp*H*), 3.36 (dd, 4H, $^2J = 16.18$ Hz, $^3J = 5.56$ Hz, –C$H_2$CHO–), 2.87 (dd, 4H, $^2J = 16.18$ Hz, $^3J = 2.03$ Hz, –C$H_2$CHO–). $^1$H NMR for **4b** (400 MHz, dmso-$d_6$): $\delta = 6.03$ (m, 8H, Ru–$H_{arom}$), 5.63 (m, 2H, –C*H*O–), 4.90 (m, 4H, Cp*H*), 4.46 (m, 4H, Cp*H*), 4.29 (s, 10H, Cp*H*), 3.27 (dd, 4H, $^2J = 16.22$ Hz, $^3J = 5.60$ Hz, –C$H_2$CHO–), 3.15 (dd, 4H, $^2J = 16.22$ Hz, $^3J = 2.07$ Hz, –C$H_2$CHO–). MS (EI, acetone): *m*/*z*: 1000.5 $[M - Cl]^+$. *Anal.* Calc. for $C_{20}H_{18}Fe_1O_2$: C, 46.36; H, 3.50. Found: C, 46.33; H, 3.52%.

### 2.3. X-ray crystallography

Crystals of **1** and **2** were mounted on a Stoe Image Plate Diffraction system equipped with a $\phi$ circle goniometer, using Mo K$\alpha$ graphite monochromated radiation ($\lambda = 0.71073$ Å) with $\phi$ range 0–200°, increment of

Table 1
Crystallographic and selected experimental data of **1**–**3**

| | **1** | **2** | **3** |
|---|---|---|---|
| Chemical formula | $C_9H_{12}O$ | $C_{20}H_{20}FeO_2$ | $C_{20}H_{18}FeO_2$ |
| Formula weight | 136.19 | 348.21 | 346.19 |
| Crystal system | tetragonal | monoclinic | triclinic |
| Space group | $I4_1/a$ | $P2_1/a$ | $P\bar{1}$ |
| Crystal colour and shape | colourless block | orange block | orange block |
| Crystal size | $0.50 \times 0.50 \times 0.50$ | $0.40 \times 0.40 \times 0.25$ | $0.38 \times 0.23 \times 0.19$ |
| $a$ (Å) | 21.9204(14) | 17.315(1) | 8.078(2) |
| $b$ (Å) | 21.9204(14) | 10.7738(8) | 9.886(2) |
| $c$ (Å) | 6.2552(4) | 19.478(1) | 11.171(2) |
| $\alpha$ (°) | 90 | 90 | 64.66(3) |
| $\beta$ (°) | 90 | 116.360(6) | 77.44(3) |
| $\gamma$ (°) | 90 | 90 | 78.53(3) |
| $V$ (Å$^3$) | 3005.7(3) | 3255.8(4) | 781.3(4) |
| $Z$ | 16 | 8 | 2 |
| $T$ (K) | 153(2) | 153(2) | 293(2) |
| $D_c$ (g cm$^{-3}$) | 1.204 | 1.421 | 1.472 |
| $\mu$ (mm$^{-1}$) | 0.076 | 0.933 | 0.972 |
| Scan range (°) | $5.26 < 2\theta < 51.92$ | $4.20 < 2\theta < 51.92$ | $4.08 < 2\theta < 50.24$ |
| Unique reflections | 1432 | 6328 | 2712 |
| Reflections used [$I > 2\sigma(I)$] | 1150 | 4580 | 2410 |
| $R_{int}$ | 0.0274 | 0.0435 | 0.0719 |
| Final $R$ indices [$I > 2\sigma(I)$] | 0.0353, $wR_2$ 0.0914 | 0.1102, $wR_2$ 0.2591 | 0.0628, $wR_2$ 0.1646 |
| $R$ indices (all data) | 0.0449, $wR_2$ 0.0948 | 0.1295, $wR_2$ 0.2645 | 0.0698, $wR_2$ 0.1695 |
| Goodness-of-fit | 1.054 | 1.184 | 1.212 |
| Maximum and minimum $\Delta\rho$ (e Å$^{-3}$) | 0.164 and −0.117 | 1.522 and −1.126 | 0.737 and −0.843 |

1.1° and 1.2°, $D_{max} - D_{min} = 12.45 - 0.81$ Å. Crystal of **3** was mounted on a Stoe AED2 4-circle diffractometer using Mo K$\alpha$ graphite monochromated radiation with $\omega/2\theta$ scans in the $2\theta$ range 4–135°. The structures were solved by direct methods using the program SHELXS-97 [12]. The refinement and all further calculations were carried out using SHELXL-97 [13]. In **1**–**3**, the hydrogen atoms have been included in calculated positions and treated as riding atoms using the SHELXL default parameters, except for the hydroxo H-atom in **1** which was located from difference Fourier maps and refined isotropically. All non-H atoms were refined anisotropically, using weighted full-matrix least-square on $F^2$. Crystallographic details are summarised in Table 1. Figs. 1 and 3 were drawn with ORTEP [14] and Fig. 2 with the program POV-RAY [15].

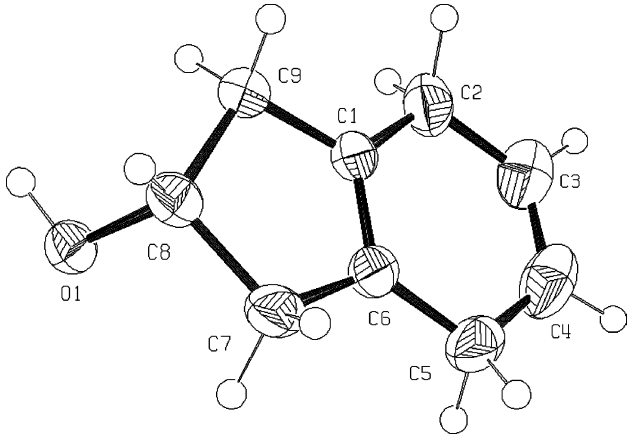



Fig. 1. ORTEP view of **1**, displacement ellipsoids are drawn at the 50% probability level. Selected bond lengths (Å) and angles (°): O(1)–C(8) 1.4321(14), C(1)–C(6) 1.3521(18); O(1)–C(8)–C(7) 107.67(10), O(1)–C(8)–C(9) 111.64(10).

## 3. Results and discussion

Indan-2-ol as well as 2,3,4,7-tetrahydro-1*H*-inden-2-ol (**1**), accessible by standard Birch reduction of indan-2-ol, react with ferrocenyl carboxylic acid in dichloromethane in the presence of condensation agents (*N*,*N*-dicyclohexylcarbodiimine, 4-(dimethylamino)pyridine, 4-pyrrolidinopyridine) to give the corresponding ferrocenoyl ester derivatives, ferrocene carboxylic acid 2,3,4,7-tetrahydro-1*H*-inden-2-yl ester (**2**) and ferrocene carboxylic acid indan-2-yl ester (**3**), see Scheme 1. Compound **1** can be recrystallised from a slow evaporation of a dichloromethane solution to give colourless crystals, while **2** and **3** can be recrystallised from an acetone–hexane solution to give orange, air-stable crystals.

The molecular structure of **1** is shown in Fig. 1. The indenyl moiety [C(1)–C(9)] is almost planar with a maximum deviation from the mean plane of −0.114(1) Å for C(8), with the oxygen lying out of the plane by 1.446(1) Å.

The hydrogen atom of the hydroxo function, which was located from difference Fourier maps, is involved in a hydrogen bond with the oxygen of a neighbouring

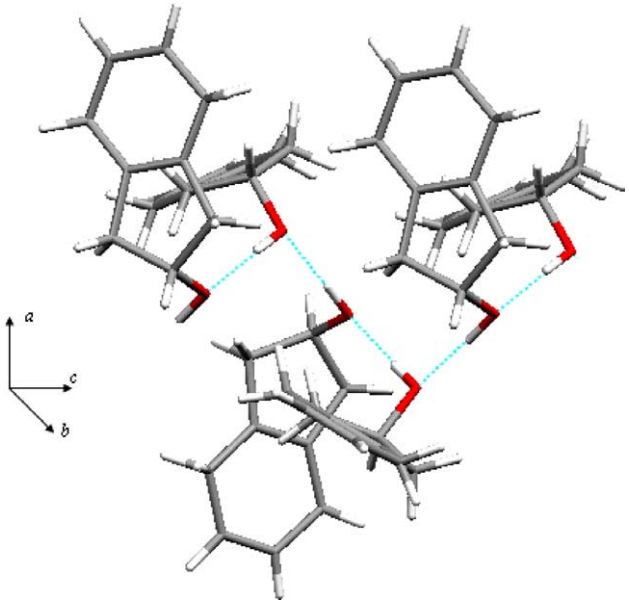
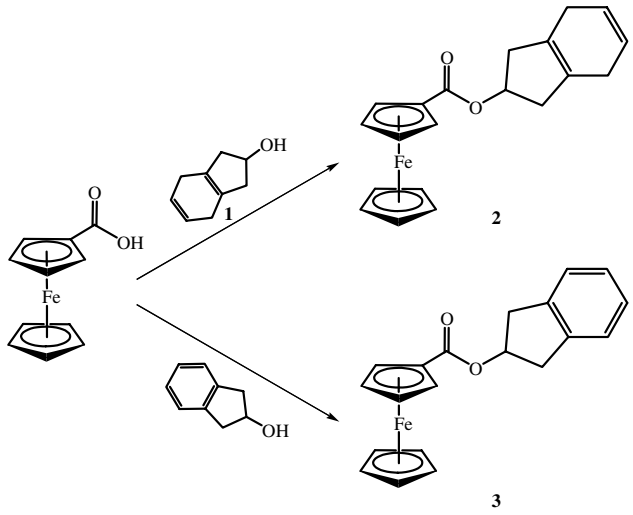



Fig. 2. Hydrogen bonded arrangement of **1** along the *c*-axis.




Scheme 1.

molecule, distance O···O being 2.7376(9) Å with a O–H···O angle of 175.5(15)°. They form one-dimensional hydrogen-bonded chains along the *c*-axis of the crystal, see Fig. 2.

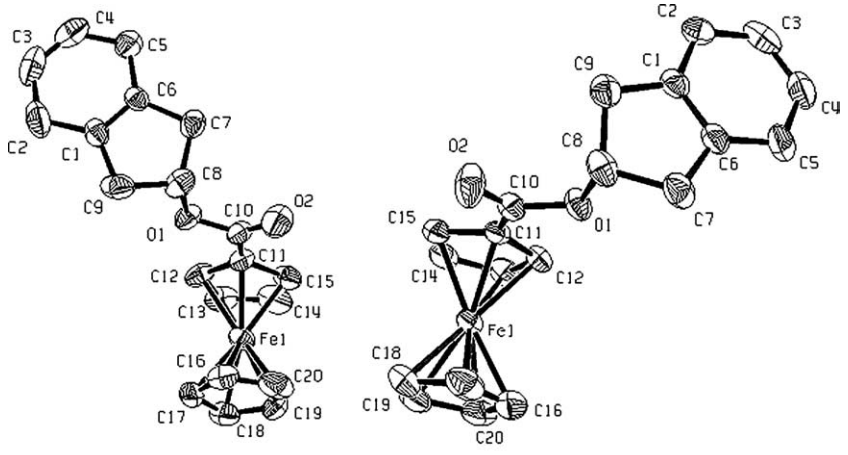
The infrared spectra of **2** and **3** exhibit the characteristic $\nu_{CO}$ absorption at 1709 $cm^{-1}$ for **2** and 1692 $cm^{-1}$ for **3** of the ester function and the bands at 1460, 1277 and 1134 $cm^{-1}$ for **2** and 1459, 1277 and 1141 $cm^{-1}$ for **3** due to the presence of a ferrocene moiety. Single-crystal structure analyses of **2** and **3** have been performed, see Fig. 3. The molecular structure of **2** comprises two independent molecules per asymmetric unit. Fig. 3 shows only one independent molecule of **2**. Selected bond lengths (Å) and angles (°) are given in Table 2.

In both cases, the ferrocene moiety is in the eclipsed conformation. The Cp rings are almost parallel to each other, the angle between the two Cp planes are 2.1(1)°, 3.4(1)° and 2.1(3)°, respectively. The indenyl substituents are rotated out of the Cp-ester plane by 79.7(2)°, 80.6(3)° and 84.5(1)°, respectively, allowing no efficient interaction between the π-system of the Cp ring and the indene moiety. A similar behavior has been observed for

Table 2
Selected bond lengths (Å) and angles (°) for compounds **2** and **3**

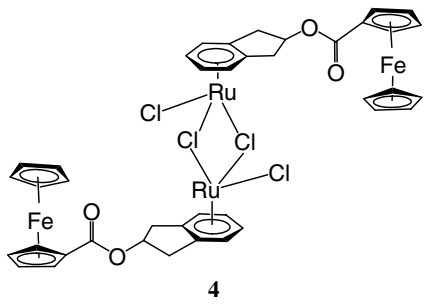
| | **2** | **2**[a] | **3** |
|---|---|---|---|
| *Interatomic distances* | | | |
| O(1)–C(10) | 1.370(12) | 1.342(11) | 1.355(5) |
| O(2)–C(10) | 1.194(12) | 1.212(12) | 1.204(6) |
| C(10)–C(11) | 1.454(13) | 1.459(13) | 1.466(6) |
| O(1)–C(8) | 1.459(11) | 1.466(11) | 1.466(5) |
| C(1)–C(2) | 1.483(16) | 1.480(13) | 1.399(7) |
| *Bond angles* | | | |
| O(1)–C(10)–C(11) | 111.0(8) | 112.0(8) | 111.8(4) |
| O(2)–C(10)–C(11) | 125.9(10) | 124.8(9) | 125.3(4) |
| O(2)–C(10)–O(1) | 123.0(9) | 123.2(9) | 122.8(4) |
| C(8)–O(1)–C(10) | 116.0(8) | 116.2(8) | 116.3(3) |

[a] Values for the second independent molecule of **2**.




**2** **3**

Fig. 3. ORTEP view of **2** and **3**, displacement ellipsoids are drawn at the 50% probability level, hydrogen atoms are omitted for clarity.




**4**

Scheme 2.

benzoylferrocene [16] and for methylenebenzotriazolylferrocene derivatives [10a]. The C(10)–O(1) distance [1.355(5) Å] is shorter than that of ferrocene carboxylic acid benzotriazole ester [1.427(2) Å], which is known to have a weak and reactive ester bond [7a].

The dienyl function of **2** reacts with $RuCl_3 \cdot nH_2O$, in refluxing ethanol to afford in moderate yield $[Ru(arene)Cl_2]_2$ (**4**) (arene = ferrocene carboxylic acid indan-2-yl ester), see Scheme 2.

In **4**, as observed for related complexes $[Ru(trindane)Cl_2]_2$ [17] and $[Ru(indane)Cl_2]_2$ [18], the complexation of the indane to the ruthenium atoms generates diastereotopic methylene protons, the two faces of the arene being non-equivalent. The *endo* and *exo* benzylic protons integrate for two whereas the central methylene proton for one. In principle, the central methylene proton can be folded toward (*endo*) or away (*exo*) from the metal, giving rise to three possible isomers in a 2:1:1 ratio (*exo–endo*:*exo–exo*:*endo–endo*). In fact, only two isomers have been observed by $^1H$ NMR in a 2:1 ratio (**4a**:**4b**). We believed that the *endo–endo* isomer, where the central methylene protons are folded away from the metals, is sterically disfavoured.

## 4. Supplementary material

Full tables of atomic parameters, bond lengths and angles are deposited at the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44-1223-336033; E-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk). Deposition numbers: (**1**) 218893, (**2**) 220073, (**3**) 218892.

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