New Soluble Bis[nona-, octa-, and pentamethylferrocenes] as "Molecular Wires" with a Metal-to-Metal Distance of up to 40 Å[#]

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Methylated ferrocenes are useful building blocks for novel materials in molecular electronics with advantageous properties in comparison to normal ferrocene derivatives. The presence of nine, eight, or five methyl substituents leads to (i) a decrease in oxidation potential, (ii) amplified donor capacity with correspondingly increased stability of the ferrocenium salts, and, most significantly, (iii) increased solubility. A modular synthetic approach based on standard Wittig chemistry affords π -conjugated soluble nona-, octa-, and pentamethylated biferrocenes, bridged by up to five vinylene-phenylene subunits. These biferrocenes with spacers are "molecular wires" with a metal-to-metal distance of up to 40 Å and an effective conjugation pathway of up to 50 Å.

Introduction

In comparison to the well-developed chemistry of ferrocene with its numerous applications¹ in organic synthesis, homogeneous catalysis, and materials science, the analogous chemistry of methylated ferrocene derivatives is limited.² Ferrocenes made up of one pentamethylcyclopentadienide (Cp*) and one substituted cyclopentadienide [(H₃C)_nCp-R; n = 0-4; R = functional group or bridging ligand] should display favorable properties in terms of increased solubility,³ lowered oxidation potential, amplified donor capacity, and hence increased stability³ of the corresponding

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Scheme 1. Principal Components of Molecular Wires



ferrocenium cations, which could be exploited for improved materials in molecular electronics.⁴ Molecular wires⁵ are mixed-valence bimetallic compounds with a conjugating bridging ligand that allows electronic communication between the two redox termini (Scheme 1). For example, such systems are known with oligoene bridging ligands between ruthenium pentamine complexes⁶ or between simple ferrocenes,⁷ where the electronic coupling can be experimentally verified for up to 18 Å separated electrophores.

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In this contribution we report on the synthesis of soluble methylated biferrocenes with increased (up to 40Å) metal-to metal distances, using standard Wittig and/or Wittig-Horner olefination procedures.⁸ The bridging ligands were selected to be phenylene-vinylene oligomers, which combine chemical stability with good charge conducting properties in comparison to oligoene,⁷ oligoyne,⁹ and oligophenyleneyne compounds.¹⁰ The number of solubilizing and cation-stabilizing methyl substituents on the metallocenvl termini were systematically varied from 9 to 5 with the anticipation of (i) increasing the solubility with the number of methyl groups attached to the ferrocenyl electrophores and (ii) progressively stabilizing the oxidized ferrocenium species according to the number of electron-donating methyl substituents.

Results and Discussion

(a) General Synthetic Approach. Biferrocenes with olefinic conjugating bridging ligands are most easily prepared by Wittig and/or Wittig-Horner reactions,⁸ similar to published results¹¹ on related oligo-(phenylene-vinylenes). The necessary synthons for the stepwise construction of such biferrocenes are thus methylated ferrocene aldehydes, terephthaldialdehyde, and the hydroxymethyl derivatives as the phosphonium progenitors, respectively. Fortunately, in contrast to the normal product distribution of Wittig protocols,⁸ the desired (E)-vinylferrocenes are favored for the first homologation step, although in general always E/Zmixtures are formed.¹² The isomer distribution of the Wittig products of ferrocene aldehydes¹³ is strongly influenced by the base used in the preparation of the ylide, and potassium tert-butoxide14 seems to be the base of choice for maximized yields of (E)-olefins. The resulting isomer mixtures are usually difficult to separate by chromatography, but sometimes the (E)-olefins can be obtained by selective crystallization (see Experimental Section). In addition, according to our own experience, acidic isomerizations¹² to the thermodynamically favored (E)-olefins are not applicable for highly methylated ferrocenes. To circumvent these restrictions, Wittig-Horner olefinations utilizing pphenylene bis(phosphonate)¹⁵ as a convenient "bis-(ylide)" progenitor are preferable, especially for the construction of the central phenylene-vinylene bridging units in the higher oligomers. (Ferrocenylmethyl)-

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Figure 1. Molecular structure of 4, showing the atomnumbering scheme. Hydrogen atoms are omitted for clarity. The cyclopentadienyl carbons of ferrocene 1 are C(1)-C(10), and the methyl carbons of ferrocene 1 are C(01)-C(09). Torsion angles: $C(12)-C(11)-C(10)-C(9) = -6.0(3.2)^{\circ}$; $C(11)-C(12)-C(13)-C(18) = 10.8(3.4)^{\circ}$. Angle of Cp plane [C(6)-C(10)] to phenyl plane $[C(13)-C(18)] = 10.5(1.1)^{\circ}$.

triphenylphosphonium and 1,1'-ferrocenediylbis(methyltriphenylphosphonium) salts¹⁶ are usually synthesized by multistep procedures via the methyl iodides of the corresponding ((dimethylamino)methyl)metallocenes. Since the availability of numerous metallocenyl aldehydes and the alcohols derived thereof has been significantly improved during the recent years,¹⁷ it seemed encouraging to apply a one-pot elimination-substitution sequence according to Hamanaka and Kosuge,¹⁸ which proved to be the most simple and efficient route to the desired ylide progenitors.

(b) Nona- and Octamethylated Ferrocenes (Scheme 2). In this manner, ((octa- and nonamethylferrocenyl)methyl)triphenylphosphonium salts 1a,b and 2 can be obtained in high yield. In addition to their characterization by the usual spectroscopic methods (see Experimental Section), X-ray structure analyses of 1b and 2 confirm their identity. These two solid-state structures are very similar to the published structure of (ferrocenylmethyl)triphenylphosphonium iodide¹⁶ and present no unexpected features; therefore the details of these crystal structures are included in the Supporting Information only.

Besides these normal (monocationic) phosphonium salts, the stabilizing effect of the methyl substituents allows also the isolation of the green air-stable ((octamethylferroceniumyl)methyl)triphenylphosphonium dication (1c), whose single-crystal structure is included in the Supporting Information. Starting from phosphonium salts 1a,b and 2 the corresponding ylides afford the "spacered" aldehydes 3 and 4 by reaction with excess terephthaldialdehyde. In accord with NMR analysis the solid-state structures of 3 (Supporting Information) and 4 (Figure 1, Table 1) clearly show an *E*-configuration. Only twinned crystals could be obtained in the case of 4; however the structure could be solved with correspondingly rather high residuals in the final structure refinement, but the connectivity of the molecule is nevertheless unequivocally determined.

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As will also be seen in other following structures, the vinylene–phenylene subunit(s) is (are) usually out of plane to the ferrocenyl group and with respect to each other, resulting in a twisted conformation in the solid state with the consequence of crystallographically imposed chirality for these molecules. Therefore the contents of the unit cell correspond to a racemic mixture in these cases. For compound **4** the respective torsion angles are -6.0(3.2) and $10.8(3.4)^{\circ}$ (Figure 1). Similar values for successive twisting of vinylene and phenylene segments have recently been published for a purely organic all-*E* oligo(phenylene–vinylene).^{11d}

Reaction of the ylide derived from phosphonium salt **1a** with octamethylferrocenyl aldehyde yields an E/Z-mixture of the symmetrical olefin **5a,b**, whereas the *E*-isomer **5a** can be also obtained more conveniently by a McMurry¹⁹ reaction from the aldehyde, in analogy to other carbonyl ferrocenes.²⁰

Figure 2 (see also Table 1) shows the result of an X-ray structure analysis of **5a** which can be compared to the structure of (*E*)-diferrocenylethylene.²¹ The overall structural parameters are of course very similar;

only the torsion angle between the cyclopentadienyl plane and the double bond is higher in value $(23.28^{\circ}$ for **5a** and 15.1° for (*E*)-diferrocenylethylene), most likely due to the steric requirements of the methyl substituents.

Bis-olefination of terephthaldialdehyde with 2 equiv of octamethylferrocenyl ylide yields the vinylene-phenylene-vinylene bridged biferrocene **6** in the all-*E* conformation as is evidenced by the observation of the typical *E*-coupling constants of 16 Hz in the ¹H NMR spectrum. The solid-state structure (Figure 3, Table 1) confirms this stereochemistry with regard to the double bonds and shows that the vinylene phenylene cyclopentadienyl subunits are twisted in relation to each other with torsion angles of 20.68 and 12.60°, respectively.

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Figure 2. Molecular structure of **5a**, showing the atomnumbering scheme. Hydrogen atoms are omitted for clarity. Numbering of the ferrocenyl carbons is analogous to **4**. Distances: Fe(1) to Cp plane [C(6)-C(10)] = 1.656(1) Å; Fe(1) to Cp plane [C(1)-C(5)] = 1.662(1) Å. Angles: Cp plane [C(6)-C(10)] to Cp plane $[C(1)-C(5)] = 0.51(0.17)^\circ$; $C(6)-C(10)-C(11)-C(11a) = -24.90(0.48)^\circ$.



Figure 3. Molecular structure of **6**, showing the atomnumbering scheme. Hydrogen atoms are omitted for clarity. Numbering of the ferrocenyl carbons is analogous to **4**. Distances: Fe(1)–Fe(1a) = 13.332(3) Å; Fe(1) to Cp plane [C(6)-C(10)] = 1.653(1) Å; Fe(1) to Cp plane [C(1)-C(5)]= 1.659(1) Å. Angles: Cp plane [C(6)-C(10)] to Cp plane $[C(1)-C(5)] = 1.94(23)^\circ$; C(12)–C(11)–C(10)–C(6) = 20.68-(49)°; C(11)–C(12)–C(13)–C(14) = 12.6(47)°; Cp plane [C(6)-C(10)] to phenyl plane $[C(13)-C(13a)] = 32.18(35)^\circ$.

The metal-to-metal distance (through space) equals 13.332(3) Å, and the sum of bond lengths (corresponding to the conjugation pathway from Fe(1) to Fe(1a)) is 16.845 Å. As can be seen from a packing plot (Figure 4), the relatively large angle (32.18°) of the plane of the phenylene ring [C(13)–C(15) and C(13a)–C(15a)] to the plane of the cyclopentadienyl ring [C(6)–C(10)] might be due to steric repulsion by neighboring molecules.

Further elongation of **3** (see Scheme 2) with the bis-(ylide) prepared from *p*-xylylene-bis(triphenylphosphonium bromide) gives access to the (vinylene)₄-(phenylene)₃-bridged biferrocene **7** which precipitates during its formation from the reaction mixture and is insoluble in all common solvents (with the exception of hot toluene). Due to this poor solubility, characterization of **7** is limited to IR and FAB-mass spectroscopy and no NMR-spectroscopic determination of the E/Z stereochemistry is possible, but the low solubility indicates the existence of **7** as a rigid rod with an all-*E* configuration. Thus within this series of octa- or nonamethylated biferrocenes, despite the anticipated solubilizing properties of 16 or 18 methyl groups, the disappointing low solubility of 7 precludes the synthesis of longer systems.

(c) Pentamethylferrocenes (Scheme 3). By essentially analogous chemistry as described above the corresponding pentamethylated compounds are accessible: Reduction of pentamethylferrocenyl aldehyde^{2c} with lithium triethylborohydride affords (hydroxymethyl)pentamethylferrocene (8), which is a progenitor of (pentamethylferrocenyl)methylphosphonium salt and the ylide derived thereof, prepared by the method of Hamanaka and Kosuge.¹⁸ Biferrocenes **9a,b** are obtained as an E/Z-mixture, from which the *E*-isomer (**9a**) can be separated by selective crystallization.

The X-ray structure of compound 9a is interesting from a crystallographer's point of view: Only merohedrically twinned crystals were available, but the structure could be solved without problems with good final *R* values (Table 1); there are two independent halfmolecules (with an inversion center located in the center of the olefinic bond) in the asymmetric unit. One of the molecules (Figure 5) is regularly ordered, whereas the second molecule (Figure 6) is disordered in a 50:50% ratio with regard to the orientations of the *trans*-olefinic moiety. Interestingly, the very same type of disordering has been published recently for the homologous (E)-1,2bis(pentamethylruthenocenyl)ethylene.²² The conformation of 9a in the solid state [angle C(11a)-C(11)- $C(10)-C(9) = -7.51^{\circ}$ can be compared to the analogous 5a, which shows increased torsion due to steric hindrance by the methyl groups of the substituted cyclopentadienyl rings (see above).

Reaction of 2 equiv of pentamethylferrocenyl aldehyde with the bis(ylide) derived from *p*-xylylenebis(triphenylphosphonium bromide) yields the (vinylene)₂(phenylene)₁ "spacered" biferrocene **10**, which is the system analogous to compound **6**. The gross features of the crystal structures of **10** (Figure 7, Table 1) and **6** (see above) are quite similar, but in the case of **10** smaller torsion angles corresponding to less twisting of the vinylene phenylene cyclopentadienyl subunits [angle $C(11)-C(12)-C(13)-C(14) = -1.79^{\circ}$; angle C(12)- $C(11)-C(10)-C(9) = -11.37^{\circ}$] are observed, due to less interaction in the solid state by neighboring molecules. In accord with these smaller distortions, the metal-tometal distance of **10** is slightly shortened to 13.291(2) Å in comparison to 13.332(3) Å in **6**.

Further elongation by similar methods as described for the octa- and nonamethylated compounds gives access to the (vinylene)₂(phenylene)₂-spacered aldehydes **11a,b**, which are a chiral molecules due to successive twisting of the cyclopentadienyl, vinylene, and phenylene subunits with respect to each other. In the crystalline state (Figure 8, Table 1) a racemic mixture of both enantiomers make up the contents of the unit cell, analogous to the structure of the one vinylene– phenylene unit shorter nonamethylated aldehyde **4**. The helical twisting in **11a** is reflected in torsion angles of -5.75, -2.67, 13.53, and 12.07° , respectively, similar in value to that for compound **4** and to that for a recently published all-*E* oligo(phenylene–vinylene).^{11d}

Coupling of the mono(ylide) derived from *p*-xylylenebis(diethylphosphonate) with pentamethylferrocenyl al-

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Figure 4. Packing of molecules of 6.



Figure 5. Molecular structure of **9a** (nondisordered molecule A), showing the atom-numbering scheme (hydrogen atoms omitted for clarity). Distances: Fe(1) to Cp plane [C(6)-C(10)] = 1.660(4) Å; Fe(1) to Cp* plane [C(1)-C(5)] = 1.644(4) Å. Angles: Cp plane [C(6)-C(10)] to Cp* plane $[C(1)-C(5)] = 0.00(65)^\circ$; C(11a)-C(11)-C(10)-C(9) = -7.51-(206)^\circ.

dehyde in situ and subsequent coupling with 11a yields (vinylene)₄(phenylene)₃-spacered biferrocene 12, which shows good solubility in contrast to the essentially insoluble nonamethylated analogue 7. The exact stereochemistry with regard to E or Z configurations of the newly formed double bonds cannot be determined due to the observation of only broad signals with unresolved coupling in the ¹H NMR spectrum. Although X-rayquality crystals have not yet been obtained, 12 represents a soluble biferrocene with a conjugation pathway of 33.6 Å and an estimated metal-to-metal distance of 26.5 Å, assuming (i) an all *E*-configuration with respect to the double bonds, (ii) a trans conformation with respect to the terminal metallocenyl units, and (iii) twist angles of similar values as in the structurally characterized shorter compounds described above.

Reaction of 2 equiv of spacered aldehyde **11a** with the bis(ylide) of *p*-xylylenebis(triphenylphosphonium bromide) [Wittig reaction] or *p*-xylylenebis(diethylphosphonate) [Wittig–Horner reaction] affords α, ω -bis(pentamethylferrocenyl)(vinylene)₆(phenylene)₅ **(13)** in 59% or 42% yield, respectively, as a red powder with a melting

point of 275-80 and 180-185 °C, respectively. Also, in the UV-vis spectra different λ_{max} absorptions (354) and 388 nm) are observed, indicating different stereochemistry of the newly formed double bonds. The Wittig-Horner methodology is known to favor E-configurated products,⁸ suggesting an all-E configuration to the latter product. Similarly to 12, NMR analysis did not give any conclusive result due to unresolved couplings and X-ray crystallography was not possible until now because the obtained crystals were to small for a single-crystal analysis. Nevertheless, compound 13 is still soluble with good solubility in dichloromethane and THF and constitutes to our knowledge the longest soluble π -conjugated biferrocene prepared so far. Under the same assumptions as stated for 12 (see above), the metal-to-metal distance in 13 can be estimated to approximately 40 Å with a conjugation pathway of 51 Å (Figure 9).

Conclusions and Perspectives. Nona-, octa-, and pentamethylated ferrocenyl aldehydes or methylphosphonium salts are the key synthons for the preparation of soluble, conjugated biferrocenes by standard Wittig and/or Wittig-Horner olefinations with vinylene-phenylene oligomers as bridging ligands between the two redox-active metallocenyl termini. The methyl substituents of the metallocenyl groups are necesssary to ensure increased solubility and enhanced stability of the oxidized species in comparison to nonmethylated spacered biferrocenes. Interestingly, the pentamethylated systems show superior solubility in direct comparison to their higher methylated analogues. These compounds serve as model systems for molecular wires, allowing the examination of the intervalence transfer (up to 40 Å) between the terminal electrophores of the mixed-valence biferrocenes. Such investigations are in progress and will be published elsewhere.

Experimental Section

General Comments. Standard techniques and instrumentation for spectroscopic and physical measurements have been described previously.²³ Tetramethylcyclopentadiene as a starting compound for the synthesis of octamethylferrocene and octamethylferrocene aldehyde was obtained from Raylo Chemicals, Edmonton, Alberta, Canada.

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Scheme 3. Synthesis of Compounds 8-13



^{*a*} Legend: (i) LiB(C₂H₅)₃H; (ii) P(C₆H₅)₃·HBr/-H₂O; (iii) (H₅C₆)₃P=CHC₆H₄CH=P(C₆H₅)₃; (iv) (H₅C₆)₃P=CHC₆H₄CH=O/*t*-BuOK/ O=CHC₆H₄CH=O; (v) (EtO)₂P(O)=CHC₆H₄CH=Q(O)(OEt)₂/*t*-BuOK; (vi) (EtO)₂P(O)=CHC₆H₄CH=P(O)(OEt)₂.

X-ray Structure Determinations of 1b,c, 2–4, 5a, 6, 9a, 10a, and 11a. A Siemens P4 diffractometer with graphitemonochromatized Mo K α radiation (λ = 71.073 pm) was used for data collection. Crystal data, data collection, and refinement parameters of **4, 5a, 6, 9a, 10**, and **11a** are summarized in Table 1 (for **1b,c, 2**, and **3**, see Supporting Information). The unit cells were determined by the automatic indexing of 25 centered reflections and confirmed by examination of the axial photographs. Data were measured via ω -scan and corrected for Lorentz and polarization effects. Scattering factors for neutral atoms and anomalous dispersion corrections were taken from the ref 24, and an empirical absorption correction²⁵ was made. The structures were solved by direct methods, SHELXS-86,²⁶ and refined by a full-matrix leastsquares procedure using SHELXL-93.²⁷ All non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atoms were placed in calculated positions. For tables of complete crystallographic data, bond lengths, bond angles, anisotropic thermal parameters, calculated hydrogen atomic coordinates, and final atomic coordinates, see the Supporting Information.

(1',2,2',3,3',4,4',5-Octamethylferrocenyl)methyl)triphenylphosphonium Bromide (1a) (Protocol According to Ref 18). A mixture of 1-(hydroxymethyl)-1',2,2',3,3',4,4',5octamethylferrocene²ⁱ (1.300 g, 3.96 mmol) [CAS Reg. No. 128925-13-3], triphenylphosphine hydrobromide (1.360 g, 3.96 mmol, 1 mequiv), and 120 mL of toluene was refluxed for 30 min until separation from the eutectic condensate was com-

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Figure 6. Molecular structure of **9a** (disordered molecule B), showing the two orientations (1:1) of the *E*-olefinic moiety C(30)-C(31a)-C(31c)-C(30a) and C(30)-C(31b)-C(31d)-C(30a), respectively.



Figure 7. Molecular structure of **10**, showing the atomnumbering scheme (hydrogen atoms are omitted for clarity). Distances: Fe(1)-Fe(1a) = 13.291(2) Å; Fe(1) to Cp plane [C(6)-C(10)] = 1.660(2) Å; Fe(1) to Cp* plane [C(1)-C(5)] = 1.645(2) Å. Angles: Cp plane [C(6)-C(10)] to Cp* plane [C(1)-C(5)] = 2.71(0.33)°; C(12)-C(11)-C(10)-C(9) = -11.37(0.66)°; C(11)-C(12)-C(13)-C(14) = -1.79(0.66)°; Cp plane [C(6)-C(10)] to phenyl plane [C(13)-C(13a)] = 13.73°.



Figure 8. Molecular structure of **11a**, showing the atomnumbering scheme (hydrogen atoms are omitted for clarity). Distances: Fe(1) to Cp plane [C(6)-C(10)] = 1.649(5)Å; Fe(1) to Cp* plane [C(1)-C(5)] = 1.655(5) Å. Angles: Cp plane [C(6)-C(10)] to Cp* plane $[C(1)-C(5)] = 1.70(0.96)^{\circ}$; Cp plane [C(6)-C(10)] to phenyl plane [C(13)-C(16)] =7.28(0.78)°; phenyl plane [C(13)-C(16)] to phenyl plane $[C(21)-C(26)] = 26.92(0.50)^{\circ}$; C(12)-C(11)-C(10)-C(6) = $-5.75(1.9)^{\circ}$; C(11)-C(12)-C(13)-C(18) = $-2.67(1.99)^{\circ}$; C(17)-C(16)-C(19)-C(20) = $13.53(2.11)^{\circ}$; C(19)-C(20)-C(21)-C(22) = $12.07(1.62)^{\circ}$.

plete in a Dean–Stark trap. By cooling, a yellow solid precipitated, which was filtered off and washed with 20 mL of ether and dried. The organic yellow filtrate, which contains some additional product, was reduced in volume (to about 20 mL) and extracted with 70 mL of water. The aqueous layer was washed with 30 mL of ether and re-extracted with CH_{2} -



Figure 9. Estimated distances for 13.

 $Cl_2.$ The CH_2Cl_2 layers were combined, and the solvent was evaporated. Yield: 2.264 g (88%) of yellow air-stable micro-crystalline solid.

Data for 1a. Mp: 212–214 °C. Anal. Calcd for $C_{37}H_{42}$ -BrFeP: C, 68.01; H, 6.48. Found: C, 67.81; H, 6.45. MS (FAB): m/z 573.4 (M⁺ of cation, 100%). UV–vis (CH₂Cl₂ [λ_{max} (nm)/log ϵ]): 266/4.03; 273/3.98; 397.0/2.87. IR data (KBr): 3054 w, 2961 m, 2940 m, 2900 m, 2853 m, 1586 m, 1481 m, 1437 s, 1381 m, 1107 s, 1032 m, 997 m, 864 m, 754 vs, 740 vs, 719 m, 713 m, 692 vs, 511 s, 499 s, 472 m, 449 m cm⁻¹. ¹H NMR (CD₃CN): δ 0.61, 1.29 , 2.25 (26H, 3s, CH₃ and CH₂); 3.71 (1H, bs, C–H of Cp); 7.64, 7.68, 7.84, 7.88 (15H, m, C₆H₅). ¹³C NMR (CD₃CN): δ 8.70, 9.48, 10.65 (CH₃ and CH₂); 130.37, 135.15, 135.57 (C₆H₅).

((1',2,2',3,3',4,4',5-Octamethylferrocenyl)methyl)triphenylphosphonium Tetrafluoroborate (1b). To a solution of tetrafluoroboric acid (54% in ether) (0.050 g, 0.027 g, 0.30 mmol) in 40 mL of toluene was added a solution of triphenylphosphine (0.080 g, 0.30 mmol) in 40 mL of toluene and a solution of 1-(hydroxymethyl)-1',2,2',3,3',4,4',5-octamethylferrocene²⁸ (0.100 g, 0.30 mmol) [128925-13-3] in 30 mL of toluene. The mixture was refluxed for 10 min, until the separation from a eutectic condensate was complete in a Dean-Stark trap. TLC (silica/ether) showed no remaining starting material. By cooling, a yellow solid precipitated, which was filtered off and dried. Yield: 0.184 g (91.5%) of yellow air-stable microcrystalline solid.

Data for 1b. Mp: 248-249 °C. Anal. Calcd for $C_{37}H_{42}$ -BF₄FeP: C, 67.30; H, 6.41. Found: C, 67.23; H, 6.39. MS (FAB): m/z 573.2 (M⁺ of cation, 100%). HR-MS (FAB): m/z 573.23266 (M⁺ of cation; exact mass calcd for $C_{37}H_{42}$ FeP, 573.2327). IR data (KBr): 3064 w, 2965 m, 2944 m, 2902 m, 1588 m, 1485 s, 1441 vs, 1381 s, 1111 vs (b), 997 s, 752 s, 739 s, 719 s, 692 vs, 519 s, 509 s, 497 s, 470 m, 451 m cm⁻¹. Single crystals were obtained from dichloromethane/acetone (v:v = 1:1) (Supporting Information, Figure 10).

((1',2,2',3,3',4,4',5-Octamethylferroceniumyl)methyl)triphenylphosphonium Bis(tetrafluoroborate) (1c). A 5 mg (0.0075 mmol) amount of 1b was dissolved in 5 mL of a mixture (v:v = 1:1) of dichloromethane and acetone; under stirring, 3 drops of a 54% solution of tetrafluoroboric acid in ether and 5 drops of a 30% solution of H_2O_2 in water were added, and the mixture changed color to green. Upon standing the product crystallized and was isolated by filtration (2 mg, 35.4% yield) as dark green air-stable crystals.

Data for 1c. Mp: 285–290 °C. Anal. Calcd for $C_{37}H_{42}$ -B₂F₈FeP: C, 59.48; H, 5.67. Found: due to only 2 mg of material available, no analysis was possible. MS (FAB): not observed, 286.6 (M⁺/2 of bis(cation)). UV–vis (CH₂Cl₂ [λ_{max} (nm)/log ϵ]): 276.0/4.24; 317.0/4.05. IR data (KBr): 3424 m (b), 2963 w, 2923 w, 2853 w, 1655 w, 1638 w, 1470 w, 1459 w, 1437 m, 1391 m, 1109 s, 1090 s, 1038 s, 758 m, 744 m, 721 m, 694 m, 515 w, 493 w cm⁻¹. Single crystals were obtained from dichloromethane/acetone (v:v = 1:1) (Supporting Information, Figure 11).

((Nonamethylferrocenyl)methyl)triphenylphosphonium Bromide (2) (Procedure According to the Method of Refs 18 and 28). To a cold solution $(-50 \ ^{\circ}C)$ of formylnonamethylferrocene^{2h,i} (0.634 g, 1.86 mmol) [90310-18-2] in 25 mL of anhydrous THF was added by syringe 2.05 mL of a 1 M solution of lithium triethylborohydride in THF (0.217 g, 2.05 mmol). The color of the solution immediately changed

Soluble "Molecular Wires"

from deep red to dark yellow. The mixture was allowed to warm to ambient temperature and was stirred further for 90 min at room temperature. TLC (silica/*n*-hexane/ether = 1:1) showed no remaining starting material. After the THF was removed in vacuo the residue was extracted with 30 mL of water and 50 mL of ether, the combined etheral layers were washed with 80 mL of water, the ether was evaporated, the remaining crude solid product was dissolved in 100 mL of toluene, and triphenylphosphine hydrobromide (0.639 g, 1.86 mmol, 1 mequiv) was added in one portion. The mixture was refluxed for 15 min until the separation from eutectic condensate was complete in a Dean–Stark trap. By cooling, a yellow solid precipitated, which was filtered off, washed with 20 mL ether, and dried, affording 0.829 g (66.7%) of **2** as a yellow air-stable microcrystalline solid.

Data for 2. Mp: 228–233 °C, dec. Anal. Calcd for C₃₈-H₄₄BrFeP: C, 68.38; H, 6.64. Found: C, 68.15; H, 6.62. MS (FAB): m/z 588 (M⁺ of cation, 11%), 325.3 (M⁺ – P(C₆H₅)₃, 100%). UV–vis (CH₂Cl₂ [λ_{max} (nm)/log ϵ]): 266.5/4.07; 273.5/4.04. IR data (KBr): 3052 w, 2967 m, 2940 m, 2900 m, 2861 m, 1481 m, 1437 vs, 1379 m, 1107 vs, 1032 m, 995 m, 754 s, 735 vs, 719 m, 690 s, 511 m, 501 s, 493 m cm⁻¹. Single crystals were obtained from CH₃CN (Supporting Information, Figure 12).

4-[2-(1',2,2',3,3',4,4',5-Octamethylferrocenyl)ethenyl]benzaldehyde (3). A Schlenk vessel was charged with ((1',2,2',3,3',4,4',5-octamethylferrocenyl)methyl)triphenylphosphonium bromide (1a) (1.00 g, 1.53 mmol) and 15 mL of anhydrous THF, the resulting suspension was cooled to -40 °C, potassium tert-butoxide (0.189 g, 1.68 mmol, 1.1 mequiv) was added in one portion, and the red suspension was stirred for 10 min in an ice/water bath. Subsequently, terephthalaldehyde (0.821 g, 6.12 mmol, 4 mequiv) was added, and the deep purple solution was allowed to warm to room temperature during 1 h. At this point TLC (silica/CH₂Cl₂/n-hexane = 2:1) showed no further reaction. The solvent was removed in vacuo, the residue was washed with 35 mL of saturated ammonium chloride solution and extracted with 50 mL ether, the combined organic layers were dried with Na₂SO₄, solvents were evaporated, and the crude product was purified by column chromatography (silica/CH₂Cl₂/n-hexane = 2:1), yielding 0.336 g (51.3%) of 3 as a purple air-stable solid.

Data for 3. Mp: 93-95 °C. Anal. Calcd for C₂₇H₃₃FeO: C, 75.52; H, 7.75. Found: C, 75.55; H, 7.75. MS (EI, 70 eV): m/z 428.5 (M⁺, 100%). HR-MS (FAB): m/z 429.18804 (M⁺; exact mass calcd for C₂₇H₃₃FeO, 429.18808). UV-vis (CH₂- $Cl_2 [\lambda_{max} (nm)/log \epsilon]$: 360.5/4.36; 530.0/3.64. IR data (KBr): 3040 w, 2961 w, 2900 w, 1697 vs, 1593 m, 1564 m, 1308 m, 1213 m, 1165 s, 1030 m, 962 s, 860 s, 843 m, 821 vs, 798 m, 787 s, 501 s, 468 s, 459 s, 451 s cm $^{-1}$. ¹H NMR (CD₃CN): δ 1.62, 1.66, 1.81, 1.97 (12H, each signal: 3H, s, CH₃ of Cp); 2.21 (12H, s, 4 × CH₃ of Cp); 3.31 (1H, s, CH of Cp); 6.77 (1H, d, ${}^{3}J_{\text{trans}}({}^{1}\text{H}-{}^{1}\text{H}) = 16$ Hz, CH=CH); 7.14 (1H, d, ${}^{3}J_{\text{trans}}({}^{1}\text{H}-{}^{1}\text{H})$ ¹H) = 16 Hz, CH=CH); 7.66, 7.82 (4H, m, C_6H_4); 9.92 (1H, s, CH=O). ¹³C NMR (CD₃CN): δ 9.52, 10.09, 11.39 (8 × CH₃); 72.39 (CH of Cp); 77.72, 80.75, 81.66, 83.57 (Cp); 125.1, 126.5, 131.1, 133.3 (C₆H₄ and CH=CH); 192.8 (CH=O). Single crystals were obtained from CH₂Cl₂ (Supporting Information, Figure 13).

4-[2-(Nonamethylferrocenyl)ethenyl]benzaldehyde (4). In analogy to the synthesis of **3**, compound **4** was prepared by starting from ((nonamethylferrocenyl)methyl)triphenylphosphonium bromide **(2)** (0.300 g, 0.45 mmol), resulting in 0.118 g (59.3% yield) of 4-[2-(nonamethylferrocenyl)ethenyl]benzaldehyde **(4)** as air-stable purple flakes.

Data for 4. Mp: 174–175 °C. Anal. Calcd for $C_{28}H_{35}$ -FeO: C, 75.84; H, 7.96. Found: C, 75.87; H, 7.98. MS (EI, 70 eV): m/z 442 (M⁺, 100%). HR-MS (FAB): m/z 443.203749 (M⁺; exact mass calcd for $C_{28}H_{35}$ FeO, 443.203727). UV–vis (CH₂Cl₂ [λ_{max} (nm)/log ϵ]): 365.5/4.35; 539.0/3.64. IR data (KBr): 2965 w, 2948 w, 2903 w, 2857 w, 1694 m, 1626 w, 1598 vs, 1562 w, 1377 w, 1215 w, 1165 m, 1028 w, 820 w cm⁻¹. ¹H NMR (CDCl₃): δ 1.67 (15H, s, 5 × CH₃ of unsubst. Cp); 1.78 (6H, s, 2 × CH₃ of subst. Cp); 1.95 (6H, s, 2 × CH₃ of subst. Cp); 6.70 (1H, d, ${}^{3}J_{trans}({}^{1}H^{-1}H) = 16$ Hz, CH=CH); 7.03 (1H, d, ${}^{3}J_{trans}({}^{1}H^{-1}H) = 16$ Hz, CH=CH); 7.56, 7.82 (4H, m, C₆H₄); 9.96 (1H, s, CH=O). ${}^{13}C$ NMR (CDCl₃): δ 9.30, 10.60 (9 × CH₃); 79.46, 79.73, 82.37 (Cp); 124.29, 125.34, 130.39, 132.15, 134.05, 145.09 (C₆H₄ and CH=CH); 191.46 (CH=O). Single crystals were obtained from CH₂Cl₂ (Table 1, Figure 1, Supporting Information).

1,2-Bis(1',2,2',3,3',4,4',5-octamethylferrocenyl)ethene (5a,b). Method A (Wittig Reaction). A suspension of ((1',2,2',3,3',4,4',5-octamethylferrocenyl)methyl)triphenylphosphonium bromide (1a) (0.350 g, 0.54 mmol) in 15 mL of anhydrous THF in a Schlenk vessel was cooled to -40 °C and converted to the corresponding ylide in the usual manner (see above) with 0.066 g (0.59 mmol, 1.1 mequiv) of potassium tertbutoxide. The red mixture was stirred for 5 min with the cooling bath removed, and 0.192 g (0.59 mmol, 1.1 mequiv) of formyl-1',2,2',3,3',4, 4',5-octamethylferrocene [CAS No. 128925-12-2] was added. The mixture was stirred at room temperature for 30 min and refluxed for a further 150 min. The THF was removed in vacuo, and the crude product was purified by column chromatography (silica, CH_2Cl_2/n -hexane = 2:1), yielding 0.067 g of **5a,b** (20.2% or 37.5% based on recovered starting material) as a red air-stable solid, consisting of a 1:3 mixture of trans (5a) and cis (5b) according to NMR analysis.

Method B (McMurry Reaction). A suspension of 5.254 g (14.2 mmol) of TiCl₃·3THF in 40 mL of DME (dimethoxyethane) was reduced with 325 mg (46.9 mmol) of lithium powder by immersing the reaction vessel in an ultrasonic cleaning bath at room temperature for 30 min. To the resulting black suspension of low-valent titanium was added 300 mg (0.920 mmol) of formyl-1',2,2',3,3',4, 4',5-octamethylferrocene [CAS No. 128925-12-2], and sonication was continued for an additional 1/2 h. Workup: DME was removed in vacuo, the mixture was poured into water, the organic material was extracted with three portions of ether, and the combined organic layers were washed with water and dried with Na₂-SO₄. After removal of solvents, the crude product mixture was purified by chromatography as described above, yielding 0.164 g (28.7%) of a mixture of *trans* (5a) and *cis* (5b) according to NMR analysis. Fractional crystallization from n-hexane afforded 69 mg (12.1% yield) of large single crystals of trans-(5a).

Data for 5a,b. MS (EI, 70 eV): m/z 620.5 (M⁺, 100%); 312 (CH₃)₈FcCH₂, 97%). IR data (KBr): 3056 w, 2905 w, 2716 w, 1628 w, 1449 m, 1427 m, 1375 m, 1261 m, 1084 m, 1028 m, 955 m, 858 m, 820 s, 802 s, 694 m, 507 m, 464 s cm⁻¹. ¹H NMR (CDCl₃): δ 1.43, 1.56, 1.60, 1.63, 1.67, 1.69, 1.72, 1.74, 1.81, 1.86, 1.98 (CH₃); 3.30 (s, CH of Cp); 6.68, 6.85, 7.33, 7.42 (CH=CH). ¹³C NMR (CDCl₃): δ 9.06, 9.28, 9.66, 9.75, 9.83, 10.93, 11.02, 11.08, 11.15 (8 × CH₃); 71.28, 79.20, 79.48, 80.60, 80.75, 81.74 (Cp); 125.63, 126.38, 126.47, 128.47, 128.62, 133.48, 133.87 (CH of the *E* and *Z* double bonds).

Data for 5a. Mp: 219–220 °C. Anal. Calcd for $C_{38}H_{52}$ -Fe₂: C, 73.55; H, 8.45. Found: C, 73.54; H, 8.43. MS (EI, 70 eV): m/z 620.5 (M⁺, 100%). UV–vis (CH₂Cl₂ [λ_{max} (nm)/log ϵ]): 318.0/4.23; 461.5/3.26. IR data (KBr): 3060 w, 2965 s, 2944 m, 2859 s, 2714 m, 1630 w, 1476 w, 1458 m, 1439 m, 1374 s, 1261 w, 1111 m, 1080 w, 1028 s, 966 m, 818 m, 515 w, 482 w, 449 s cm⁻¹. ¹H NMR (CD₂Cl₂): δ 1.60, 1.63, 1.69, 1.79 (CH₃); 3.30 (s, CH of Cp); 6.24 (2H, s(br), CH=CH). ¹³C NMR (CD₂Cl₂): δ 9.36, 9.91, 11.25 (CH₃); 71.76, 78.48, 80.85, 81.02, 81.51, 81.72 (Cp); 125.42 (CH=CH). Single crystals were obtained from *n*-hexane (Table 1, Figure 2; Supporting Information).

1,4-Bis[2-(1',2,2',3,3',4,4',5-octamethylferrocenyl)ethenyl]benzene (6). Similarly to the synthesis of compounds **5a,b**, $((1',2,2',3,3',4,4',5\text{-octamethylferrocenyl)methyl)triphenylphos$ phonium bromide**(1a)**(0.250 g, 0.383 mmol) was converted

Table 1. Crystal and Structure Refinement for Data for 4, 5a, 6, 9a, 10, and 11a

		-	•
	4	5a	6
mol formula	CarHarFeO	CueHraFea	CuaHraEea
f	449 40	C4011501 C2 C40 50	799 69
IW .	442.40	042.30	122.02
cryst system	monoclinic	monoclinic	monoclinic
space group	$P2_1/c$ (No. 14)	$P2_1/c$ (No. 14)	$P2_1/c$ (No. 14)
<i>a</i> (pm)	1425.0(5)	956.5(3)	995.4(2)
b (pm)	1114.9(5)	1241.5(3)	1176.2(2)
c (pm)	1497.4(12)	1414.3(3)	1659.7(4)
a (deg)	90	90	90
β (deg)	104 58(4)	106 11(2)	102 90(2)
μ (deg)	00	00	00
y (ueg)	90 9.000/0)	90 1 0195 (7)	
V (nm ³)	2.302(2)	1.6135(7)	1.8941(7)
Z	4	2	2
temp (K)	213(2)	213(2)	223(2)
d(calcd) (Mg/m ³)	1.276	1.322	1.267
abs coeff (mm ⁻¹)	0.672	0.926	0.797
<i>F</i> (000)	944	684	772
color habit	red platelet	red column	red prism
cryst size (mm)	$0.38 \times 0.32 \times 0.10$	$0.85 \times 0.3 \times 0.2$	$0.6 \times 0.3 \times 0.11$
(1 yst size (iiiii)	$0.36 \times 0.32 \times 0.19$	$0.03 \times 0.3 \times 0.2$	$0.0 \times 0.3 \times 0.11$
<i>θ</i> range for data collen (deg)	3.35-20.50	3.20-24.99	3.06-24.00
index ranges	$0 \le h \le 14$	$0 \le h \le 11$	$-1 \le h \le 10$
	$-1 \leq k \leq 11$	$-1 \leq k \leq 14$	$-1 \leq k \leq 12$
	$-14 \leq l \leq 15$	$-16 \leq l \leq 16$	$-18 \leq l \leq 18$
no. of rflns collcd	2286	3295	3803
no, of indep rflps	$2015 (R_{int} = 0.0426)$	$2824 (R_{int} = 0.0217)$	$2880 (R_{int} = 0.0251)$
no of rflps with $I > 2\sigma(I)$	1587	2/12	2315
abc con	1507	2112 N 6000	2010
	none		ψ -scall
max and min transm		0.643 and 0.589	0.925 and 0.808
refinement method	full-matrix least-squares on F^{z}	full-matrix least-squares on F^{z}	full-matrix least-squares on F^{z}
data/restraints/parms	1962/0/270	2818/0/189	2880/0/333
goodness-of-fit on F ²	1.115	1.042	1.038
final R indices $[I > 2\sigma(I)]$	$R_1 = 0.1106$	$R_1 = 0.0334$	$R_1 = 0.0354$
	$wR_2 = 0.2940$	$wR_2 = 0.0782$	$wR_2 = 0.0815$
R indices (all data)	$R_1 = 0.1445$	$R_1 = 0.0444$	$R_1 = 0.0519$
n mates (an data)	$n_1 = 0.1445$ $m_2 = 0.4002$	$m_1 = 0.0444$	$m_1 = 0.0010$ $m_2 = 0.0007$
1 = 1 = 1 = 1	$WR_2 = 0.4003$	$WR_2 = 0.0883$	$WR_2 = 0.0097$
largest diff peak and hole (e-nm 3)	1590 and -657	294 and -403	244 and -234
	9a	10	11a
	9a	10	11a
mol formula	9a C ₃₂ H ₄₀ Fe ₂	10 C ₄₀ H ₄₆ Fe ₂	11a C ₃₂ H ₃₂ FeO
mol formula fw	9a C ₃₂ H ₄₀ Fe ₂ 536.34	10 C ₄₀ H ₄₆ Fe ₂ 638.47	11a C ₃₂ H ₃₂ FeO 488.43
mol formula fw cryst system	9a C ₃₂ H ₄₀ Fe ₂ 536.34 monoclinic	10 C ₄₀ H ₄₆ Fe ₂ 638.47 triclinic	11a C ₃₂ H ₃₂ FeO 488.43 monoclinic
mol formula fw cryst system space group	9a C ₃₂ H ₄₀ Fe ₂ 536.34 monoclinic <i>P</i> 2 ₁ / <i>n</i> (No. 14)	10 C ₄₀ H ₄₆ Fe ₂ 638.47 triclinic <i>P</i> 1 (No. 2)	11a C ₃₂ H ₃₂ FeO 488.43 monoclinic <i>P</i> 2 ₁ (No. 4)
mol formula fw cryst system space group a (pm)	9a C ₃₂ H ₄₀ Fe ₂ 536.34 monoclinic <i>P</i> 2 ₁ / <i>n</i> (No. 14) 1759.0(6)	$\begin{array}{c} 10\\ \hline C_{40}H_{46}Fe_2\\ 638.47\\ triclinic\\ P\bar{1}\ (No.\ 2)\\ 831.6(2) \end{array}$	11a C ₃₂ H ₃₂ FeO 488.43 monoclinic <i>P</i> 2 ₁ (No. 4) 838.9(4)
mol formula fw cryst system space group <i>a</i> (pm) <i>b</i> (pm)	9a C ₃₂ H ₄₀ Fe ₂ 536.34 monoclinic <i>P</i> 2 ₁ / <i>n</i> (No. 14) 1759.0(6) 1085.6(3)	$\begin{array}{c} \textbf{10} \\ \hline C_{40}H_{46}Fe_2 \\ 638.47 \\ triclinic \\ P\bar{1} \ (No. \ 2) \\ 831.6(2) \\ 937 \ 7(2) \end{array}$	11a C ₃₂ H ₃₂ FeO 488.43 monoclinic <i>P</i> 2 ₁ (No. 4) 838.9(4) 883 3(6)
mol formula fw cryst system space group <i>a</i> (pm) <i>b</i> (pm) <i>c</i> (pm)	9a C ₃₂ H ₄₀ Fe ₂ 536.34 monoclinic <i>P</i> 2 ₁ / <i>n</i> (No. 14) 1759.0(6) 1085.6(3) 1672.0(2)	10 C ₄₀ H ₄₆ Fe ₂ 638.47 triclinic <i>P</i> I (No. 2) 831.6(2) 937.7(2) 1230.9(2)	11a C ₃₂ H ₃₂ FeO 488.43 monoclinic <i>P</i> 2 ₁ (No. 4) 838.9(4) 883.3(6) 1705 9(9)
mol formula fw cryst system space group <i>a</i> (pm) <i>b</i> (pm) <i>c</i> (pm) c (pm)	9a C ₃₂ H ₄₀ Fe ₂ 536.34 monoclinic <i>P</i> 2 ₁ / <i>n</i> (No. 14) 1759.0(6) 1085.6(3) 1673.0(3) 90	$\begin{array}{c} \textbf{10} \\ \hline C_{40}H_{46}Fe_2 \\ 638.47 \\ triclinic \\ P\bar{1} \ (No. \ 2) \\ 831.6(2) \\ 937.7(2) \\ 1239.9(2) \\ 09.82(1) \\ \end{array}$	11a C ₃₂ H ₃₂ FeO 488.43 monoclinic <i>P</i> 2 ₁ (No. 4) 838.9(4) 883.3(6) 1795.9(9) 00
mol formula fw cryst system space group a (pm) b (pm) c (pm) α (deg)	9a C ₃₂ H ₄₀ Fe ₂ 536.34 monoclinic <i>P</i> 2 ₁ / <i>n</i> (No. 14) 1759.0(6) 1085.6(3) 1673.0(3) 90 4142.02(2)	$\begin{array}{c} \textbf{10} \\ \hline C_{40}H_{46}Fe_2 \\ 638.47 \\ triclinic \\ P\bar{1} \ (No. \ 2) \\ 831.6(2) \\ 937.7(2) \\ 1239.9(2) \\ 98.86(1) \\ 102.07(2) \end{array}$	11a C ₃₂ H ₃₂ FeO 488.43 monoclinic <i>P</i> 2 ₁ (No. 4) 838.9(4) 883.3(6) 1795.9(9) 90 404 04(0)
mol formulafwcryst systemspace group a (pm) b (pm) c (pm) α (deg) β (deg)	$\begin{array}{c} \textbf{9a} \\ \hline C_{32}H_{40}Fe_2 \\ 536.34 \\ monoclinic \\ P2_1/n \ (No. \ 14) \\ 1759.0(6) \\ 1085.6(3) \\ 1673.0(3) \\ 90 \\ 118.32(2) \end{array}$	$\begin{array}{c} \textbf{10} \\ \hline C_{40}H_{46}Fe_2 \\ 638.47 \\ triclinic \\ P\bar{1} \ (No. \ 2) \\ 831.6(2) \\ 937.7(2) \\ 1239.9(2) \\ 98.86(1) \\ 108.86(1) \end{array}$	11a C ₃₂ H ₃₂ FeO 488.43 monoclinic P21 (No. 4) 838.9(4) 883.3(6) 1795.9(9) 90 101.24(3)
mol formula fw cryst system space group a (pm) b (pm) c (pm) α (deg) β (deg) γ (deg)	$\begin{array}{c} \textbf{9a} \\ \hline C_{32}H_{40}Fe_2 \\ 536.34 \\ monoclinic \\ P2_1/n \ (No. \ 14) \\ 1759.0(6) \\ 1085.6(3) \\ 1673.0(3) \\ 90 \\ 118.32(2) \\ 90 \\ \end{array}$	$\begin{array}{c} \textbf{10} \\ \hline C_{40}H_{46}Fe_2 \\ 638.47 \\ triclinic \\ P\bar{1} \ (No. \ 2) \\ 831.6(2) \\ 937.7(2) \\ 1239.9(2) \\ 98.86(1) \\ 108.86(1) \\ 107.41(1) \end{array}$	$\begin{array}{c} 11a \\ \hline C_{32}H_{32}FeO \\ 488.43 \\ monoclinic \\ P2_1 (No. 4) \\ 838.9(4) \\ 883.3(6) \\ 1795.9(9) \\ 90 \\ 101.24(3) \\ 90 \\ \end{array}$
mol formula fw cryst system space group a (pm) b (pm) c (pm) α (deg) β (deg) γ (deg) V (nm ³)	$\begin{array}{c} \textbf{9a} \\ \hline C_{32}H_{40}Fe_2 \\ 536.34 \\ monoclinic \\ P2_1/n \ (No. \ 14) \\ 1759.0(6) \\ 1085.6(3) \\ 1673.0(3) \\ 90 \\ 118.32(2) \\ 90 \\ 2.8123(13) \end{array}$	$\begin{array}{c} \textbf{10} \\ \hline C_{40}H_{46}Fe_2 \\ 638.47 \\ triclinic \\ P\bar{1} \ (No. \ 2) \\ 831.6(2) \\ 937.7(2) \\ 1239.9(2) \\ 98.86(1) \\ 108.86(1) \\ 107.41(1) \\ 0.8387(3) \end{array}$	11a C ₃₂ H ₃₂ FeO 488.43 monoclinic P2 ₁ (No. 4) 838.9(4) 883.3(6) 1795.9(9) 90 101.24(3) 90 1.3052(13)
mol formula fw cryst system space group a (pm) b (pm) c (pm) α (deg) β (deg) γ (deg) γ (deg) V (nm ³) Z	$\begin{array}{c} \textbf{9a} \\ \hline C_{32}H_{40}Fe_2 \\ 536.34 \\ monoclinic \\ P2_1/n \ (No. \ 14) \\ 1759.0(6) \\ 1085.6(3) \\ 1673.0(3) \\ 90 \\ 118.32(2) \\ 90 \\ 2.8123(13) \\ 4 \end{array}$	$\begin{array}{c} \textbf{10} \\ \hline C_{40}H_{46}Fe_2 \\ 638.47 \\ triclinic \\ P\bar{1} \ (No. \ 2) \\ 831.6(2) \\ 937.7(2) \\ 1239.9(2) \\ 98.86(1) \\ 108.86(1) \\ 107.41 \ (1) \\ 0.8387(3) \\ 1 \end{array}$	$\begin{array}{c} 11a\\ \hline C_{32}H_{32}FeO\\ 488.43\\ monoclinic\\ P2_1 (No. 4)\\ 838.9(4)\\ 883.3(6)\\ 1795.9(9)\\ 90\\ 101.24(3)\\ 90\\ 1.3052(13)\\ 2\end{array}$
mol formula fw cryst system space group a (pm) b (pm) c (pm) α (deg) β (deg) γ (deg) V (nm ³) Z temp (K)	$\begin{array}{c} \textbf{9a} \\ \hline C_{32}H_{40}Fe_2 \\ 536.34 \\ monoclinic \\ P2_1/n (No. 14) \\ 1759.0(6) \\ 1085.6(3) \\ 1673.0(3) \\ 90 \\ 118.32(2) \\ 90 \\ 2.8123(13) \\ 4 \\ 208(2) \end{array}$	$\begin{array}{c} \textbf{10} \\ \hline C_{40}H_{46}Fe_2 \\ 638.47 \\ triclinic \\ P\bar{1} \ (No. \ 2) \\ 831.6(2) \\ 937.7(2) \\ 1239.9(2) \\ 98.86(1) \\ 108.86(1) \\ 107.41(1) \\ 0.8387(3) \\ 1 \\ 213(2) \end{array}$	11a C ₃₂ H ₃₂ FeO 488.43 monoclinic P21 (No. 4) 838.9(4) 883.3(6) 1795.9(9) 90 101.24(3) 90 1.3052(13) 2 213(2)
mol formula fw cryst system space group a (pm) b (pm) c (pm) $\alpha (deg)$ $\beta (deg)$ $\gamma (deg)$ $V (nm^3)$ Z temp (K) $d(cad) (Mg/m^3)$	$\begin{array}{c} \textbf{9a} \\ \hline C_{32}H_{40}Fe_2 \\ 536.34 \\ monoclinic \\ P2_1/n (No. 14) \\ 1759.0(6) \\ 1085.6(3) \\ 1673.0(3) \\ 90 \\ 118.32(2) \\ 90 \\ 2.8123(13) \\ 4 \\ 208(2) \\ 1 267 \end{array}$	$\begin{array}{c} 10\\ \hline C_{40}H_{46}Fe_2\\ 638.47\\ triclinic\\ P\bar{1}\ (No.\ 2)\\ 831.6(2)\\ 937.7(2)\\ 1239.9(2)\\ 98.86(1)\\ 108.86(1)\\ 107.41(1)\\ 0.8387(3)\\ 1\\ 213(2)\\ 1\\ 213(2)\\ 1\\ 264 \end{array}$	$\begin{array}{c} 11a\\ \hline C_{32}H_{32}FeO\\ 488.43\\ monoclinic\\ P2_1 (No. 4)\\ 838.9(4)\\ 883.3(6)\\ 1795.9(9)\\ 90\\ 101.24(3)\\ 90\\ 1.3052(13)\\ 2\\ 213(2)\\ 1.243\\ \end{array}$
mol formula fw cryst system space group a (pm) b (pm) c (pm) α (deg) β (deg) γ (deg) V (nm ³) Z temp (K) d(calcd) (Mg/m ³) abe coeff (mm ⁻¹)	$\begin{array}{r} \textbf{9a} \\ \hline C_{32}H_{40}Fe_2 \\ 536.34 \\ monoclinic \\ P2_1/n \ (No. \ 14) \\ 1759.0(6) \\ 1085.6(3) \\ 1673.0(3) \\ 90 \\ 118.32(2) \\ 90 \\ 2.8123(13) \\ 4 \\ 208(2) \\ 1.267 \\ 1.049 \\ \end{array}$	$\begin{array}{c} \textbf{10} \\ \hline C_{40}H_{46}Fe_2 \\ 638.47 \\ triclinic \\ P\bar{1} (No. 2) \\ 831.6(2) \\ 937.7(2) \\ 1239.9(2) \\ 98.86(1) \\ 108.86(1) \\ 107.41(1) \\ 0.8387(3) \\ 1 \\ 213(2) \\ 1.264 \\ 0.891 \\ \end{array}$	$\begin{array}{c} 11a\\ \hline C_{32}H_{32}FeO\\ 488.43\\ monoclinic\\ P2_1 (No. 4)\\ 838.9(4)\\ 883.3(6)\\ 1795.9(9)\\ 90\\ 101.24(3)\\ 90\\ 1.3052(13)\\ 2\\ 213(2)\\ 1.243\\ 0.590\\ \end{array}$
mol formula fw cryst system space group a (pm) b (pm) c (pm) α (deg) β (deg) γ (deg) V (nm ³) Z temp (K) d(calcd) (Mg/m ³) $abs coeff (mm^{-1})$	$\begin{array}{r} \textbf{9a} \\ \hline C_{32}H_{40}Fe_2 \\ 536.34 \\ monoclinic \\ P2_1/n \ (No. \ 14) \\ 1759.0(6) \\ 1085.6(3) \\ 1673.0(3) \\ 90 \\ 118.32(2) \\ 90 \\ 2.8123(13) \\ 4 \\ 208(2) \\ 1.267 \\ 1.049 \\ 1120 \end{array}$	$\begin{array}{c} \textbf{10} \\ \hline C_{40}H_{46}Fe_2 \\ 638.47 \\ triclinic \\ P\bar{1} \ (No. \ 2) \\ 831.6(2) \\ 937.7(2) \\ 1239.9(2) \\ 98.86(1) \\ 108.86(1) \\ 107.41(1) \\ 0.8387(3) \\ 1 \\ 213(2) \\ 1.264 \\ 0.891 \\ 239 \end{array}$	$\begin{array}{c} 11a\\ \hline C_{32}H_{32}FeO\\ 488.43\\ monoclinic\\ P2_1 (No. 4)\\ 838.9(4)\\ 883.3(6)\\ 1795.9(9)\\ 90\\ 101.24(3)\\ 90\\ 1.3052(13)\\ 2\\ 213(2)\\ 1.243\\ 0.599\\ 510\\ \end{array}$
mol formula fw cryst system space group a (pm) b (pm) c (pm) α (deg) β (deg) γ (deg) V (nm ³) Z temp (K) d(calcd) (Mg/m ³) abs coeff (mm ⁻¹) F(000)	$\begin{array}{r} \textbf{9a} \\ \hline C_{32}H_{40}Fe_2 \\ 536.34 \\ monoclinic \\ P2_1/n (No. 14) \\ 1759.0(6) \\ 1085.6(3) \\ 1673.0(3) \\ 90 \\ 118.32(2) \\ 90 \\ 2.8123(13) \\ 4 \\ 208(2) \\ 1.267 \\ 1.049 \\ 1136 \\ 0 \\ 1.049 \\ 1136 \\ 0 \\ 1.041 \\ 0 \\ 1.041 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ $	$\begin{array}{c} \textbf{10} \\ \hline C_{40}H_{46}Fe_2 \\ 638.47 \\ triclinic \\ P\bar{1} (No. 2) \\ 831.6(2) \\ 937.7(2) \\ 1239.9(2) \\ 98.86(1) \\ 108.86(1) \\ 107.41(1) \\ 0.8387(3) \\ 1 \\ 213(2) \\ 1.264 \\ 0.891 \\ 338 \\ \end{array}$	$\begin{array}{c} 11a\\ \hline C_{32}H_{32}FeO\\ 488.43\\ monoclinic\\ P2_1 (No. 4)\\ 838.9(4)\\ 883.3(6)\\ 1795.9(9)\\ 90\\ 101.24(3)\\ 90\\ 1.3052(13)\\ 2\\ 213(2)\\ 1.243\\ 0.599\\ 516\\ \hline \end{array}$
mol formula fw cryst system space group a (pm) b (pm) c (pm) α (deg) β (deg) γ (deg) γ (deg) V (nm ³) Z temp (K) d(calcd) (Mg/m ³) abs coeff (mm ⁻¹) F(000) color, habit	$\begin{array}{c} \textbf{9a} \\ \hline C_{32}H_{40}Fe_2 \\ 536.34 \\ monoclinic \\ P2_1/n (No. 14) \\ 1759.0(6) \\ 1085.6(3) \\ 1673.0(3) \\ 90 \\ 118.32(2) \\ 90 \\ 2.8123(13) \\ 4 \\ 208(2) \\ 1.267 \\ 1.049 \\ 1136 \\ red platelet \end{array}$	$\begin{array}{c} \textbf{10} \\ \hline C_{40}H_{46}Fe_2 \\ 638.47 \\ triclinic \\ P\bar{1} (No. 2) \\ 831.6(2) \\ 937.7(2) \\ 1239.9(2) \\ 98.86(1) \\ 108.86(1) \\ 107.41(1) \\ 0.8387(3) \\ 1 \\ 213(2) \\ 1.264 \\ 0.891 \\ 338 \\ red platelet \end{array}$	$\begin{array}{c} 11a \\ \hline C_{32}H_{32}FeO \\ 488.43 \\ monoclinic \\ P2_1 (No. 4) \\ 838.9(4) \\ 883.3(6) \\ 1795.9(9) \\ 90 \\ 101.24(3) \\ 90 \\ 1.3052(13) \\ 2 \\ 213(2) \\ 1.243 \\ 0.599 \\ 516 \\ red platelet \\ \end{array}$
mol formula fw cryst system space group a (pm) b (pm) c (pm) α (deg) β (deg) γ (deg) V (nm ³) Z temp (K) d(calcd) (Mg/m ³) $abs coeff (mm^{-1})$ F(000) color, habit cryst size (nm)	$\begin{array}{c} \textbf{9a} \\ \hline C_{32}H_{40}Fe_2 \\ 536.34 \\ monoclinic \\ P2_1/n \ (No. \ 14) \\ 1759.0(6) \\ 1085.6(3) \\ 1673.0(3) \\ 90 \\ 118.32(2) \\ 90 \\ 2.8123(13) \\ 4 \\ 208(2) \\ 1.267 \\ 1.049 \\ 1136 \\ red platelet \\ 0.55 \times 0.55 \times 0.12 \\ \end{array}$	$\begin{array}{c} \textbf{10} \\ \hline C_{40}H_{46}Fe_2 \\ 638.47 \\ triclinic \\ P\bar{1} (No. 2) \\ 831.6(2) \\ 937.7(2) \\ 1239.9(2) \\ 98.86(1) \\ 108.86(1) \\ 108.86(1) \\ 107.41(1) \\ 0.8387(3) \\ 1 \\ 213(2) \\ 1.264 \\ 0.891 \\ 338 \\ red platelet \\ 0.5 \times 0.25 \times 0.09 \\ \end{array}$	$\begin{array}{c} 11a\\ \hline C_{32}H_{32}FeO\\ 488.43\\ monoclinic\\ P2_1 (No. 4)\\ 838.9(4)\\ 883.3(6)\\ 1795.9(9)\\ 90\\ 101.24(3)\\ 90\\ 1.3052(13)\\ 2\\ 213(2)\\ 1.243\\ 0.599\\ 516\\ red platelet\\ 0.3 \times 0.3 \times 0.05\\ \end{array}$
mol formula fw cryst system space group a (pm) b (pm) c (pm) α (deg) β (deg) γ (deg) V (nm ³) Z temp (K) d(calcd) (Mg/m ³) $abs coeff (mm^{-1})$ F(000) color, habit cryst size (nm) θ range for data collcn (deg)	$\begin{array}{r} \textbf{9a} \\ \hline C_{32}H_{40}Fe_2 \\ 536.34 \\ monoclinic \\ P2_1/n \ (No. \ 14) \\ 1759.0(6) \\ 1085.6(3) \\ 1673.0(3) \\ 90 \\ 118.32(2) \\ 90 \\ 2.8123(13) \\ 4 \\ 208(2) \\ 1.267 \\ 1.049 \\ 1136 \\ red platelet \\ 0.55 \times 0.55 \times 0.12 \\ 3.07-21.00 \\ \end{array}$	$\begin{array}{c} 10\\ \hline C_{40}H_{46}Fe_2\\ 638.47\\ triclinic\\ P\bar{1}\ (No.\ 2)\\ 831.6(2)\\ 937.7(2)\\ 1239.9(2)\\ 98.86(1)\\ 108.86(1)\\ 108.86(1)\\ 107.41(1)\\ 0.8387(3)\\ 1\\ 213(2)\\ 1.264\\ 0.891\\ 338\\ red platelet\\ 0.5\times 0.25\times 0.09\\ 3.19-23.00\\ \end{array}$	$\begin{array}{c} 11a\\ \hline C_{32}H_{32}FeO\\ 488.43\\ monoclinic\\ P2_1 (No. 4)\\ 838.9(4)\\ 883.3(6)\\ 1795.9(9)\\ 90\\ 101.24(3)\\ 90\\ 1.3052(13)\\ 2\\ 213(2)\\ 1.243\\ 0.599\\ 516\\ red platelet\\ 0.3 \times 0.3 \times 0.05\\ 2.31-18.50\\ \end{array}$
mol formula fw cryst system space group <i>a</i> (pm) <i>b</i> (pm) <i>c</i> (pm) α (deg) β (deg) γ (deg) V (nm ³) Z temp (K) d(calcd) (Mg/m ³) abs coeff (mm ⁻¹) <i>F</i> (000) color, habit cryst size (nm) θ range for data collcn (deg) index ranges	$\begin{array}{c} \textbf{9a} \\ \hline C_{32}H_{40}Fe_2 \\ 536.34 \\ monoclinic \\ P2_1/n \ (No. \ 14) \\ 1759.0(6) \\ 1085.6(3) \\ 1673.0(3) \\ 90 \\ 118.32(2) \\ 90 \\ 2.8123(13) \\ 4 \\ 208(2) \\ 1.267 \\ 1.049 \\ 1136 \\ red \ platelet \\ 0.55 \times 0.55 \times 0.12 \\ 3.07-21.00 \\ -17 \leq h \leq 6 \end{array}$	$\begin{array}{c} 10\\ \hline C_{40}H_{46}Fe_2\\ 638.47\\ triclinic\\ P\bar{1}\ (No.\ 2)\\ 831.6(2)\\ 937.7(2)\\ 1239.9(2)\\ 98.86(1)\\ 108.86(1)\\ 107.41(1)\\ 0.8387(3)\\ 1\\ 213(2)\\ 1.264\\ 0.891\\ 338\\ red\ platelet\\ 0.5\times 0.25\times 0.09\\ 3.19-23.00\\ -1\leq h\leq 8 \end{array}$	$\begin{array}{c} 11a\\ \hline C_{32}H_{32}FeO\\ 488.43\\ monoclinic\\ P2_1 (No. 4)\\ 838.9(4)\\ 883.3(6)\\ 1795.9(9)\\ 90\\ 101.24(3)\\ 90\\ 1.3052(13)\\ 2\\ 213(2)\\ 1.243\\ 0.599\\ 516\\ red platelet\\ 0.3 \times 0.3 \times 0.05\\ 2.31-18.50\\ -1 \leq h \leq 7 \end{array}$
mol formula fw cryst system space group a (pm) b (pm) c (pm) α (deg) β (deg) γ (deg) V (nm ³) Z temp (K) d(calcd) (Mg/m ³) abs coeff (mm ⁻¹) F(000) color, habit cryst size (nm) θ range for data collcn (deg) index ranges	$\begin{array}{c} \textbf{9a} \\ \hline C_{32}H_{40}Fe_2 \\ 536.34 \\ monoclinic \\ P2_1/n (No. 14) \\ 1759.0(6) \\ 1085.6(3) \\ 1673.0(3) \\ 90 \\ 118.32(2) \\ 90 \\ 2.8123(13) \\ 4 \\ 208(2) \\ 1.267 \\ 1.049 \\ 1136 \\ red platelet \\ 0.55 \times 0.55 \times 0.12 \\ 3.07-21.00 \\ -17 \leq h \leq 6 \\ -10 \leq k \leq 1 \\ \end{array}$	$\begin{array}{c} 10\\ \hline C_{40}H_{46}Fe_2\\ 638.47\\ triclinic\\ P\bar{1}\ (No.\ 2)\\ 831.6(2)\\ 937.7(2)\\ 1239.9(2)\\ 98.86(1)\\ 107.41(1)\\ 0.8387(3)\\ 1\\ 213(2)\\ 1.264\\ 0.891\\ 338\\ red\ platelet\\ 0.5\times 0.25\times 0.09\\ 3.19-23.00\\ -1\leq h\leq 8\\ -9\leq k\leq 9 \end{array}$	11a $C_{32}H_{32}FeO$ 488.43 monoclinic $P2_1$ (No. 4) 838.9(4) 883.3(6) 1795.9(9) 90 101.24(3) 90 1.3052(13) 2 213(2) 1.243 0.599 516 red platelet 0.3 × 0.3 × 0.05 2.31-18.50 $-1 \le h \le 7$ $-16 \le l \le 15$
mol formula fw cryst system space group a (pm) b (pm) c (pm) α (deg) β (deg) γ (deg) γ (deg) V (nm ³) Z temp (K) $d(calcd) (Mg/m^3)$ $abs coeff (mm^{-1})$ F(000) color, habit cryst size (nm) θ range for data collcn (deg) index ranges	$\begin{array}{c} \textbf{9a} \\ \hline \\ C_{32}H_{40}Fe_2 \\ 536.34 \\ monoclinic \\ P2_1/n (No. 14) \\ 1759.0(6) \\ 1085.6(3) \\ 1673.0(3) \\ 90 \\ 118.32(2) \\ 90 \\ 2.8123(13) \\ 4 \\ 208(2) \\ 1.267 \\ 1.049 \\ 1136 \\ red platelet \\ 0.55 \times 0.55 \times 0.12 \\ 3.07-21.00 \\ -17 \leq h \leq 6 \\ -10 \leq k \leq 1 \\ -12 \leq k \leq 14 \\ \end{array}$	$\begin{array}{c} \textbf{10} \\ \hline C_{40}H_{46}Fe_2 \\ 638.47 \\ triclinic \\ P\overline{1} (No. 2) \\ 831.6(2) \\ 937.7(2) \\ 1239.9(2) \\ 98.86(1) \\ 108.86(1) \\ 107.41(1) \\ 0.8387(3) \\ 1 \\ 213(2) \\ 1.264 \\ 0.891 \\ 338 \\ red platelet \\ 0.5 \times 0.25 \times 0.09 \\ 3.19-23.00 \\ -1 \leq h \leq 8 \\ -9 \leq k \leq 9 \\ -13 \leq l \leq 13 \end{array}$	$\begin{array}{c} 11a\\ \hline C_{32}H_{32}FeO\\ 488.43\\ monoclinic\\ P2_1 (No. 4)\\ 838.9(4)\\ 883.3(6)\\ 1795.9(9)\\ 90\\ 101.24(3)\\ 90\\ 1.3052(13)\\ 2\\ 213(2)\\ 1.243\\ 0.599\\ 516\\ red platelet\\ 0.3\times0.3\times0.05\\ 2.31-18.50\\ -1\leq h\leq7\\ -16\leq l\leq15\\ -16\leq l\leq15\\ \end{array}$
mol formula fw cryst system space group a (pm) b (pm) c (pm) α (deg) β (deg) γ (deg) V (nm ³) Z temp (K) d(calcd) (Mg/m ³) $abs coeff (mm^{-1})$ F(000) c color, habit cryst size (nm) θ range for data collcn (deg) index ranges	$\begin{array}{c} \textbf{9a} \\ \hline C_{32}H_{40}Fe_2 \\ 536.34 \\ monoclinic \\ P2_1/n (No. 14) \\ 1759.0(6) \\ 1085.6(3) \\ 1673.0(3) \\ 90 \\ 118.32(2) \\ 90 \\ 2.8123(13) \\ 4 \\ 208(2) \\ 1.267 \\ 1.049 \\ 1136 \\ red platelet \\ 0.55 \times 0.55 \times 0.12 \\ 3.07-21.00 \\ -17 \leq h \leq 6 \\ -10 \leq k \leq 1 \\ -12 \leq k \leq 14 \\ 3113 \end{array}$	$\begin{array}{c} 10\\ \hline C_{40}H_{46}Fe_2\\ 638.47\\ triclinic\\ P\bar{1}\ (No.\ 2)\\ 831.6(2)\\ 937.7(2)\\ 1239.9(2)\\ 98.86(1)\\ 108.86(1)\\ 107.41(1)\\ 0.8387(3)\\ 1\\ 213(2)\\ 1.264\\ 0.891\\ 338\\ red\ platelet\\ 0.5\times 0.25\times 0.09\\ 3.19-23.00\\ -1\leq h\leq 8\\ -9\leq k\leq 9\\ -13\leq l\leq 13\\ 2843\\ \end{array}$	$\begin{array}{c} 11a\\ \hline C_{32}H_{32}FeO\\ 488.43\\ monoclinic\\ P2_1 (No. 4)\\ 838.9(4)\\ 883.3(6)\\ 1795.9(9)\\ 90\\ 101.24(3)\\ 90\\ 1.3052(13)\\ 2\\ 213(2)\\ 1.243\\ 0.599\\ 516\\ red platelet\\ 0.3\times0.3\times0.05\\ 2.31-18.50\\ -1\leq h\leq7\\ -16\leq l\leq15\\ -16\leq l\leq15\\ -16\leq l\leq15\\ 2100\\ \end{array}$
mol formula fw cryst system space group a (pm) b (pm) c (pm) α (deg) β (deg) γ (deg) V (nm ³) Z temp (K) d(calcd) (Mg/m ³) $abs coeff (mm^{-1})$ F(000) color, habit cryst size (nm) θ range for data collcn (deg) index ranges no. of rflns collcd no. of rflns collcd	$\begin{array}{c} 9a \\ \hline C_{32}H_{40}Fe_2 \\ 536.34 \\ monoclinic \\ P2_1/n (No. 14) \\ 1759.0(6) \\ 1085.6(3) \\ 1673.0(3) \\ 90 \\ 118.32(2) \\ 90 \\ 2.8123(13) \\ 4 \\ 208(2) \\ 1.267 \\ 1.049 \\ 1136 \\ red platelet \\ 0.55 \times 0.55 \times 0.12 \\ 3.07-21.00 \\ -17 \leq h \leq 6 \\ -10 \leq k \leq 1 \\ -12 \leq k \leq 14 \\ 3113 \\ 2ee5 (R_{-} = 0.0104) \\ \end{array}$	$\begin{array}{c} 10\\ \hline C_{40}H_{46}Fe_2\\ 638.47\\ triclinic\\ P\bar{1}\ (No.\ 2)\\ 831.6(2)\\ 937.7(2)\\ 1239.9(2)\\ 98.86(1)\\ 108.86(1)\\ 108.86(1)\\ 107.41(1)\\ 0.8387(3)\\ 1\\ 213(2)\\ 1.264\\ 0.891\\ 338\\ red platelet\\ 0.5 \times 0.25 \times 0.09\\ 3.19-23.00\\ -1 \le h \le 8\\ -9 \le k \le 9\\ -13 \le l \le 13\\ 2843\\ 2844\ (B_{-}=0.0242)\\ \end{array}$	$\begin{array}{c} 11a\\ \hline C_{32}H_{32}FeO\\ 488.43\\ monoclinic\\ P2_1 (No. 4)\\ 838.9(4)\\ 883.3(6)\\ 1795.9(9)\\ 90\\ 101.24(3)\\ 90\\ 101.24(3)\\ 90\\ 1.3052(13)\\ 2\\ 213(2)\\ 1.243\\ 0.599\\ 516\\ red platelet\\ 0.3 \times 0.3 \times 0.05\\ 2.31-18.50\\ -1 \leq h \leq 7\\ -16 \leq l \leq 15\\ -16 \leq l \leq 15\\ -16 \leq l \leq 15\\ 2100\\ 1767 (B_{-}=0.0287)\\ \end{array}$
mol formula fw cryst system space group <i>a</i> (pm) <i>b</i> (pm) <i>c</i> (pm) α (deg) β (deg) γ (deg) V (nm ³) Z temp (K) d(calcd) (Mg/m ³) abs coeff (mm ⁻¹) <i>F</i> (000) color, habit cryst size (nm) θ range for data collcn (deg) index ranges no. of rflns collcd no. of indep rflns	$\begin{array}{c} \textbf{9a} \\ \hline C_{32}H_{40}Fe_2 \\ 536.34 \\ monoclinic \\ P2_1/n (No. 14) \\ 1759.0(6) \\ 1085.6(3) \\ 1673.0(3) \\ 90 \\ 118.32(2) \\ 90 \\ 2.8123(13) \\ 4 \\ 208(2) \\ 1.267 \\ 1.049 \\ 1136 \\ red platelet \\ 0.55 \times 0.55 \times 0.12 \\ 3.07-21.00 \\ -17 \leq h \leq 6 \\ -10 \leq k \leq 1 \\ -12 \leq k \leq 14 \\ 3113 \\ 2635 (R_{int} = 0.0194) \\ 2020 \\ \end{array}$	$\begin{array}{c} 10\\ \hline C_{40}H_{46}Fe_2\\ 638.47\\ triclinic\\ P\bar{1}\ (No.\ 2)\\ 831.6(2)\\ 937.7(2)\\ 1239.9(2)\\ 98.86(1)\\ 108.86(1)\\ 107.41(1)\\ 0.8387(3)\\ 1\\ 213(2)\\ 1.264\\ 0.891\\ 338\\ red platelet\\ 0.5\times 0.25\times 0.09\\ 3.19-23.00\\ -1\le h\le 8\\ -9\le k\le 9\\ -13\le l\le 13\\ 2843\\ 2284\ (R_{int}=0.0243)\\ 1000\\ $	$\begin{array}{c} 11a\\ \hline C_{32}H_{32}FeO\\ 488.43\\ monoclinic\\ P2_1 (No. 4)\\ 838.9(4)\\ 883.3(6)\\ 1795.9(9)\\ 90\\ 101.24(3)\\ 90\\ 1.3052(13)\\ 2\\ 213(2)\\ 1.243\\ 0.599\\ 516\\ red platelet\\ 0.3\times 0.3\times 0.05\\ 2.31-18.50\\ -1\leq h\leq 7\\ -16\leq l\leq 15\\ -16\leq l\leq 15\\ -16\leq l\leq 15\\ 2100\\ 1767 (R_{int}=0.0287)\\ 1400\\ \end{array}$
mol formula fw cryst system space group <i>a</i> (pm) <i>b</i> (pm) <i>c</i> (pm) <i>a</i> (deg) β (deg) γ (deg) γ (deg) V (nm ³) Z temp (K) d(calcd) (Mg/m ³) abs coeff (mm ⁻¹) <i>F</i> (000) color, habit cryst size (nm) θ range for data collcn (deg) index ranges no. of rflns collcd no. of indep rflns no. of rflns with $I > 2\sigma(I)$	$\begin{array}{c} \textbf{9a} \\ \hline \\ C_{32}H_{40}Fe_2 \\ 536.34 \\ monoclinic \\ P2_1/n (No. 14) \\ 1759.0(6) \\ 1085.6(3) \\ 1085.6(3) \\ 1673.0(3) \\ 90 \\ 118.32(2) \\ 90 \\ 2.8123(13) \\ 4 \\ 208(2) \\ 1.267 \\ 1.049 \\ 1136 \\ red platelet \\ 0.55 \times 0.55 \times 0.12 \\ 3.07-21.00 \\ -17 \leq h \leq 6 \\ -10 \leq k \leq 1 \\ -12 \leq k \leq 14 \\ 3113 \\ 2635 (R_{int} = 0.0194) \\ 2198 \\ \end{array}$	$\begin{array}{c} 10\\ \hline C_{40}H_{46}Fe_2\\ 638.47\\ triclinic\\ P\bar{1}\ (No.\ 2)\\ 831.6(2)\\ 937.7(2)\\ 1239.9(2)\\ 98.86(1)\\ 107.41(1)\\ 0.8387(3)\\ 1\\ 107.41(1)\\ 0.8387(3)\\ 1\\ 213(2)\\ 1.264\\ 0.891\\ 338\\ red platelet\\ 0.5\times 0.25\times 0.09\\ 3.19-23.00\\ -1\leq h\leq 8\\ -9\leq k\leq 9\\ -13\leq l\leq 13\\ 2843\\ 2284\ (R_{int}=0.0243)\\ 1838\\ \end{array}$	$\begin{array}{c} 11a\\ \hline C_{32}H_{32}FeO\\ 488.43\\ monoclinic\\ P2_1 (No. 4)\\ 838.9(4)\\ 883.3(6)\\ 1795.9(9)\\ 90\\ 101.24(3)\\ 90\\ 1.3052(13)\\ 2\\ 213(2)\\ 1.243\\ 0.599\\ 516\\ red platelet\\ 0.3\times0.3\times0.05\\ 2.31-18.50\\ -1\leq h\leq7\\ -16\leq l\leq15\\ -16\leq l\leq15\\ 2100\\ 1767 (R_{int}=0.0287)\\ 1430\\ \end{array}$
mol formula fw cryst system space group <i>a</i> (pm) <i>b</i> (pm) <i>c</i> (pm) α (deg) β (deg) γ (deg) V (nm ³) Z temp (K) d(calcd) (Mg/m ³) abs coeff (mm ⁻¹) <i>F</i> (000) color, habit cryst size (nm) θ range for data collcn (deg) index ranges no. of rflns collcd no. of indep rflns no. of rflns with $I > 2\sigma(I)$ abs cor	$\begin{array}{r} \textbf{9a} \\ \hline C_{32}H_{40}Fe_2 \\ 536.34 \\ monoclinic \\ P2_1/n (No. 14) \\ 1759.0(6) \\ 1085.6(3) \\ 1085.6(3) \\ 1673.0(3) \\ 90 \\ 118.32(2) \\ 90 \\ 2.8123(13) \\ 4 \\ 208(2) \\ 1.267 \\ 1.049 \\ 1136 \\ red platelet \\ 0.55 \times 0.55 \times 0.12 \\ 3.07-21.00 \\ -17 \leq h \leq 6 \\ -10 \leq k \leq 1 \\ -12 \leq k \leq 14 \\ 3113 \\ 2635 (R_{int} = 0.0194) \\ 2198 \\ none \\ \end{array}$	$\begin{array}{c} 10\\ \hline C_{40}H_{46}Fe_2\\ 638.47\\ triclinic\\ P\bar{1}\ (No.\ 2)\\ 831.6(2)\\ 937.7(2)\\ 1239.9(2)\\ 98.86(1)\\ 108.86(1)\\ 108.86(1)\\ 107.41(1)\\ 0.8387(3)\\ 1\\ 213(2)\\ 1.264\\ 0.891\\ 338\\ red platelet\\ 0.5\times 0.25\times 0.09\\ 3.19-23.00\\ -1\leq h\leq 8\\ -9\leq k\leq 9\\ -13\leq l\leq 13\\ 2843\\ 2284\ (R_{int}=0.0243)\\ 1838\\ \psi\text{-scan} \end{array}$	$\begin{array}{c} 11a\\ \hline C_{32}H_{32}FeO\\ 488.43\\ monoclinic\\ P2_1 (No. 4)\\ 838.9(4)\\ 883.3(6)\\ 1795.9(9)\\ 90\\ 101.24(3)\\ 90\\ 1.3052(13)\\ 2\\ 213(2)\\ 1.243\\ 0.599\\ 516\\ red platelet\\ 0.3\times0.3\times0.05\\ 2.31-18.50\\ -1\leq h\leq7\\ -16\leq l\leq15\\ -16\leq l\leq15\\ -16\leq l\leq15\\ 2100\\ 1767 (R_{int}=0.0287)\\ 1430\\ \psi\text{-scan} \end{array}$
mol formula fw cryst system space group <i>a</i> (pm) <i>b</i> (pm) <i>c</i> (pm) α (deg) β (deg) γ (deg) V (nm ³) Z temp (K) d(calcd) (Mg/m ³) abs coeff (mm ⁻¹) <i>F</i> (000) color, habit cryst size (nm) θ range for data collcn (deg) index ranges no. of rflns collcd no. of rflns with $I > 2\sigma(I)$ abs cor max and min transm	$\begin{array}{r} \textbf{9a} \\ \hline C_{32}H_{40}Fe_2 \\ 536.34 \\ monoclinic \\ P2_1/n (No. 14) \\ 1759.0(6) \\ 1085.6(3) \\ 1673.0(3) \\ 90 \\ 118.32(2) \\ 90 \\ 2.8123(13) \\ 4 \\ 208(2) \\ 1.267 \\ 1.049 \\ 1136 \\ red platelet \\ 0.55 \times 0.55 \times 0.12 \\ 3.07-21.00 \\ -17 \leq h \leq 6 \\ -10 \leq k \leq 1 \\ -12 \leq k \leq 14 \\ 3113 \\ 2635 (R_{int} = 0.0194) \\ 2198 \\ none \\ \end{array}$	$\begin{array}{c} 10\\ \hline C_{40}H_{46}Fe_2\\ 638.47\\ triclinic\\ P\bar{1}\ (No.\ 2)\\ 831.6(2)\\ 937.7(2)\\ 1239.9(2)\\ 98.86(1)\\ 108.86(1)\\ 108.86(1)\\ 107.41(1)\\ 0.8387(3)\\ 1\\ 213(2)\\ 1.264\\ 0.891\\ 338\\ red platelet\\ 0.5\times 0.25\times 0.09\\ 3.19-23.00\\ -1\leq h\leq 8\\ -9\leq k\leq 9\\ -13\leq l\leq 13\\ 2843\\ 2284\ (R_{int}=0.0243)\\ 1838\\ \psi\text{-scan}\\ 0.861\ and\ 0.750\\ \end{array}$	$\begin{array}{c} 11a\\ \hline C_{32}H_{32}FeO\\ 488.43\\ monoclinic\\ P2_1 (No. 4)\\ 838.9(4)\\ 883.3(6)\\ 1795.9(9)\\ 90\\ 101.24(3)\\ 90\\ 1.3052(13)\\ 2\\ 213(2)\\ 1.243\\ 0.599\\ 516\\ red platelet\\ 0.3\times0.3\times0.05\\ 2.31-18.50\\ -1\leq h\leq 7\\ -16\leq l\leq 15\\ -16\leq l\leq 15\\ -16\leq l\leq 15\\ 2100\\ 1767 (R_{int}=0.0287)\\ 1430\\ \psi\text{-scan}\\ 0.926 and 0.862\\ \end{array}$
mol formula fw cryst system space group <i>a</i> (pm) <i>b</i> (pm) <i>c</i> (pm) α (deg) β (deg) γ (deg) V (nm ³) Z temp (K) d(calcd) (Mg/m ³) abs coeff (mm ⁻¹) <i>F</i> (000) color, habit cryst size (nm) θ range for data collcn (deg) index ranges no. of rflns collcd no. of rflns with $I > 2\sigma(I)$ abs cor max and min transm refinement method	$\begin{array}{c} \textbf{9a} \\ \hline C_{32}H_{40}Fe_2 \\ 536.34 \\ monoclinic \\ P2_1/n (No. 14) \\ 1759.0(6) \\ 1085.6(3) \\ 1673.0(3) \\ 90 \\ 118.32(2) \\ 90 \\ 2.8123(13) \\ 4 \\ 208(2) \\ 1.267 \\ 1.049 \\ 1136 \\ red platelet \\ 0.55 \times 0.55 \times 0.12 \\ 3.07-21.00 \\ -17 \leq h \leq 6 \\ -10 \leq k \leq 1 \\ -12 \leq k \leq 14 \\ 3113 \\ 2635 (R_{int} = 0.0194) \\ 2198 \\ none \\ \hline full-matrix least-squares on F^2$	$\begin{array}{c} 10\\ \hline C_{40}H_{46}Fe_2\\ 638.47\\ triclinic\\ P\bar{1}\ (No.\ 2)\\ 831.6(2)\\ 937.7(2)\\ 1239.9(2)\\ 98.86(1)\\ 108.86(1)\\ 107.41(1)\\ 0.8387(3)\\ 1\\ 213(2)\\ 1.264\\ 0.891\\ 338\\ red platelet\\ 0.5\times 0.25\times 0.09\\ 3.19-23.00\\ -1\le h\le 8\\ -9\le k\le 9\\ -13\le l\le 13\\ 2843\\ 2284\ (R_{int}=0.0243)\\ 1838\\ \psi\ scan\\ 0.861\ and\ 0.750\\ full-matrix\ least-squares\ on\ F^2\\ \end{array}$	$\begin{array}{c} 11a\\ \hline C_{32}H_{32}FeO\\ 488.43\\ monoclinic\\ P2_1 (No. 4)\\ 838.9(4)\\ 883.3(6)\\ 1795.9(9)\\ 90\\ 101.24(3)\\ 90\\ 101.24(3)\\ 90\\ 1.3052(13)\\ 2\\ 213(2)\\ 1.243\\ 0.599\\ 516\\ red platelet\\ 0.3 \times 0.3 \times 0.05\\ 2.31-18.50\\ -1 \leq h \leq 7\\ -16 \leq l \leq 15\\ -16 \leq l \leq 15\\ -16 \leq l \leq 15\\ 2100\\ 1767 (R_{int} = 0.0287)\\ 1430\\ \psi \ scan\\ 0.926 \ and 0.862\\ full-matrix \ least-squares on \ F^2 \end{array}$
mol formula fw cryst system space group <i>a</i> (pm) <i>b</i> (pm) <i>c</i> (pm) <i>a</i> (deg) β (deg) γ (deg) γ (deg) V (nm ³) Z temp (K) d(calcd) (Mg/m ³) abs coeff (mm ⁻¹) <i>F</i> (000) color, habit cryst size (nm) θ range for data collcn (deg) index ranges no. of rflns collcd no. of rflns with $I \ge 2\sigma(I)$ abs cor max and min transm refinement method data/restraints/narms	$\begin{array}{c} \textbf{9a} \\ \hline C_{32}H_{40}Fe_2 \\ 536.34 \\ monoclinic \\ P2_1/n (No. 14) \\ 1759.0(6) \\ 1085.6(3) \\ 1673.0(3) \\ 90 \\ 118.32(2) \\ 90 \\ 2.8123(13) \\ 4 \\ 208(2) \\ 1.267 \\ 1.049 \\ 1136 \\ red platelet \\ 0.55 \times 0.55 \times 0.12 \\ 3.07-21.00 \\ -17 \leq h \leq 6 \\ -10 \leq k \leq 1 \\ -12 \leq k \leq 14 \\ 3113 \\ 2635 (R_{int} = 0.0194) \\ 2198 \\ none \\ \hline full-matrix least-squares on F^2 \\ 2635/0/321 \\ \end{array}$	$\begin{array}{c} 10\\ \hline C_{40}H_{46}Fe_2\\ 638.47\\ triclinic\\ P\bar{1}\ (No.\ 2)\\ 831.6(2)\\ 937.7(2)\\ 1239.9(2)\\ 98.86(1)\\ 108.86(1)\\ 107.41(1)\\ 0.8387(3)\\ 1\\ 213(2)\\ 1.264\\ 0.891\\ 338\\ red\ platelet\\ 0.5\times 0.25\times 0.09\\ 3.19-23.00\\ -1\le h\le 8\\ -9\le k\le 9\\ -13\le l\le 13\\ 2843\\ 2284\ (R_{int}=0.0243)\\ 1838\\ \psi\ -scan\\ 0.861\ and\ 0.750\\ full-matrix\ least-squares\ on\ F^2\\ 2284/0/195\end{array}$	$\begin{array}{c} 11a \\ \hline C_{32}H_{32}FeO \\ 488.43 \\ monoclinic \\ P2_1 (No. 4) \\ 838.9(4) \\ 883.3(6) \\ 1795.9(9) \\ 90 \\ 101.24(3) \\ 90 \\ 101.24(3) \\ 90 \\ 1.3052(13) \\ 2 \\ 213(2) \\ 1.243 \\ 0.599 \\ 516 \\ red platelet \\ 0.3 \times 0.3 \times 0.05 \\ 2.31-18.50 \\ -1 \leq h \leq 7 \\ -16 \leq l \leq 15 \\ -16 \leq l \leq 15 \\ -16 \leq l \leq 15 \\ 2100 \\ 1767 (R_{int} = 0.0287) \\ 1430 \\ \psi$ -scan \\ 0.926 and 0.862 \\ full-matrix least-squares on F^2
mol formula fw cryst system space group <i>a</i> (pm) <i>b</i> (pm) <i>c</i> (pm) α (deg) β (deg) γ (deg) V (nm ³) Z temp (K) d(calcd) (Mg/m ³) abs coeff (mm ⁻¹) <i>F</i> (000) color, habit cryst size (nm) θ range for data collcn (deg) index ranges no. of rflns collcd no. of rflns with $I > 2\sigma(I)$ abs cor max and min transm refinement method data/restraints/parms goodness-of.fit on F^2	$\begin{array}{r} \textbf{9a} \\ \hline C_{32}H_{40}Fe_2 \\ 536.34 \\ monoclinic \\ P2_1/n (No. 14) \\ 1759.0(6) \\ 1085.6(3) \\ 1673.0(3) \\ 90 \\ 118.32(2) \\ 90 \\ 2.8123(13) \\ 4 \\ 208(2) \\ 1.267 \\ 1.049 \\ 1136 \\ red platelet \\ 0.55 \times 0.55 \times 0.12 \\ 3.07-21.00 \\ -17 \leq h \leq 6 \\ -10 \leq k \leq 1 \\ -12 \leq k \leq 14 \\ 3113 \\ 2635 (R_{int} = 0.0194) \\ 2198 \\ none \\ \hline full-matrix least-squares on F^2 \\ 2635/0/321 \\ 1.069 \\ \end{array}$	$\begin{array}{c} 10\\ \hline C_{40}H_{46}Fe_2\\ 638.47\\ triclinic\\ P\bar{1}\ (No.\ 2)\\ 831.6(2)\\ 937.7(2)\\ 1239.9(2)\\ 98.86(1)\\ 108.86(1)\\ 108.86(1)\\ 107.41(1)\\ 0.8387(3)\\ 1\\ 213(2)\\ 1.264\\ 0.891\\ 338\\ red platelet\\ 0.5\times 0.25\times 0.09\\ 3.19-23.00\\ -1\leq h\leq 8\\ -9\leq k\leq 9\\ -13\leq l\leq 13\\ 2843\\ 2284\ (R_{int}=0.0243)\\ 1838\\ \psi\text{-scan}\\ 0.861\ and\ 0.750\\ full-matrix\ least-squares\ on\ F^2\\ 2284/0/195\\ 1\ 0.44\\ \end{array}$	$\begin{array}{r} 11a \\ \hline C_{32}H_{32}FeO \\ 488.43 \\ monoclinic \\ P2_1 (No. 4) \\ 838.9(4) \\ 883.3(6) \\ 1795.9(9) \\ 90 \\ 101.24(3) \\ 90 \\ 1.3052(13) \\ 2 \\ 213(2) \\ 1.243 \\ 0.599 \\ 516 \\ red platelet \\ 0.3 \times 0.3 \times 0.05 \\ 2.31-18.50 \\ -1 \leq h \leq 7 \\ -16 \leq l \leq 15 \\ -16 \leq l \leq 15 \\ 2100 \\ 1767 (R_{int} = 0.0287) \\ 1430 \\ \psi$ -scan \\ 0.926 and 0.862 \\ full-matrix least-squares on F^2 1767/1/312 \\ 1090 \\ \end{array}
mol formula fw cryst system space group <i>a</i> (pm) <i>b</i> (pm) <i>c</i> (pm) α (deg) β (deg) γ (deg) V (nm ³) Z temp (K) d(calcd) (Mg/m ³) abs coeff (mm ⁻¹) <i>F</i> (000) color, habit cryst size (nm) θ range for data collcn (deg) index ranges no. of rflns collcd no. of rflns with $I > 2\sigma(I)$ abs cor max and min transm refinement method data/restraints/parms goodness-of-fit on F^2 final B indices $I > 2\sigma(D)$	$\begin{array}{r} \textbf{9a} \\ \hline C_{32}H_{40}Fe_2 \\ 536.34 \\ monoclinic \\ P2_1/n (No. 14) \\ 1759.0(6) \\ 1085.6(3) \\ 1085.6(3) \\ 1073.0(3) \\ 90 \\ 118.32(2) \\ 90 \\ 2.8123(13) \\ 4 \\ 208(2) \\ 1.267 \\ 1.049 \\ 1136 \\ red platelet \\ 0.55 \times 0.55 \times 0.12 \\ 3.07-21.00 \\ -17 \leq h \leq 6 \\ -10 \leq k \leq 1 \\ -12 \leq k \leq 14 \\ 3113 \\ 2635 (R_{int} = 0.0194) \\ 2198 \\ none \\ \hline full-matrix least-squares on F^2$	10 $C_{40}H_{46}Fe_2$ 638.47 triclinic $P\bar{1}$ (No. 2) 831.6(2) 937.7(2) 1239.9(2) 98.86(1) 108.86(1) 107.41(1) 0.8387(3) 1 213(2) 1.264 0.891 338 red platelet 0.5 × 0.25 × 0.09 3.19-23.00 -1 ≤ h ≤ 8 -9 ≤ k ≤ 9 -13 ≤ l ≤ 13 2843 2284 ($R_{int} = 0.0243$) 1838 ψ -scan 0.861 and 0.750 full-matrix least-squares on F^2 2284/0/195 1.044	11a $C_{32}H_{32}FeO$ 488.43monoclinic $P2_1$ (No. 4)838.9(4)883.3(6)1795.9(9)90101.24(3)901.3052(13)2213(2)1.2430.599516red platelet0.3 × 0.3 × 0.052.31-18.50 $-1 \le h \le 7$ $-16 \le l \le 15$ 2100 1767 ($R_{int} = 0.0287$)1430 ψ -scan0.926 and 0.862full-matrix least-squares on F^2 1767/1/3121.090 $R_{-0} 0.0466$
mol formula fw cryst system space group <i>a</i> (pm) <i>b</i> (pm) <i>c</i> (pm) <i>a</i> (deg) β (deg) γ (deg) <i>V</i> (nm ³) Z temp (K) d(calcd) (Mg/m ³) abs coeff (mm ⁻¹) <i>F</i> (000) color, habit cryst size (nm) θ range for data collcn (deg) index ranges no. of rflns collcd no. of rflns with $I > 2\sigma(I)$ abs cor max and min transm refinement method data/restraints/parms goodness-of-fit on F^2 final R indices [$I > 2\sigma(I)$]	9a $C_{32}H_{40}Fe_2$ 536.34 monoclinic $P2_1/n$ (No. 14) 1759.0(6) 1085.6(3) 1673.0(3) 90 118.32(2) 90 2.8123(13) 4 208(2) 1.267 1.049 1136 red platelet 0.55 × 0.55 × 0.12 3.07-21.00 -17 ≤ h ≤ 6 -10 ≤ k ≤ 1 -12 ≤ k ≤ 14 3113 2635 ($R_{int} = 0.0194$) 2198 none full-matrix least-squares on F^2 2635/0/321 1.069 $R_1 = 0.0511$	10 $C_{40}H_{46}Fe_2$ 638.47 triclinic $P\bar{1}$ (No. 2) 831.6(2) 937.7(2) 1239.9(2) 98.86(1) 108.86(1) 107.41(1) 0.8387(3) 1 213(2) 1.264 0.891 338 red platelet 0.5 × 0.25 × 0.09 3.19-23.00 $-1 \le h \le 8$ $-9 \le k \le 9$ $-13 \le l \le 13$ 2843 2284 ($R_{int} = 0.0243$) 1838 ψ -scan 0.861 and 0.750 full-matrix least-squares on F^2 2284/0/195 1.044 $R_1 = 0.0434$	11a $C_{32}H_{32}FeO$ 488.43monoclinic $P2_1$ (No. 4)838.9(4)883.3(6)1795.9(9)90101.24(3)901.3052(13)2213(2)1.2430.599516red platelet $0.3 \times 0.3 \times 0.05$ 2.31-18.50 $-1 \le h \le 7$ $-16 \le l \le 15$ $-16 \le l \le 15$ 21001767 ($R_{int} = 0.0287$)1430 ψ -scan0.926 and 0.862full-matrix least-squares on F^2 1767/1/3121.090 $R_1 = 0.0466$ $w_P = c = 0.022$
mol formula fw cryst system space group <i>a</i> (pm) <i>b</i> (pm) <i>c</i> (pm) <i>a</i> (deg) β (deg) γ (deg) γ (deg) V (nm ³) Z temp (K) d(calcd) (Mg/m ³) abs coeff (mm ⁻¹) <i>F</i> (000) color, habit cryst size (nm) θ range for data collcn (deg) index ranges no. of rflns collcd no. of rflns with $I > 2\sigma(I)$ abs cor max and min transm refinement method data/restraints/parms goodness-of-fit on F^2 final R indices [$I > 2\sigma(I)$]	9a $C_{32}H_{40}Fe_2$ 536.34 monoclinic $P2_1/n$ (No. 14) $1759.0(6)$ $1085.6(3)$ $1673.0(3)$ 90 $118.32(2)$ 90 $2.8123(13)$ 4 $208(2)$ 1.267 1.049 1136 red platelet $0.55 \times 0.55 \times 0.12$ $3.07-21.00$ $-17 \le h \le 6$ $-10 \le k \le 1$ $-12 \le k \le 14$ 3113 $2635(R_{int} = 0.0194)$ 2198 none full-matrix least-squares on F^2 $2635/0/321$ 1.069 $R_1 = 0.0511$ $wR_2 = 0.1131$	$\begin{array}{c} 10\\ \hline C_{40}H_{46}Fe_2\\ 638.47\\ triclinic\\ P\overline{1}\ (No.\ 2)\\ 831.6(2)\\ 937.7(2)\\ 1239.9(2)\\ 98.86(1)\\ 107.41(1)\\ 0.8387(3)\\ 1\\ 213(2)\\ 1.264\\ 0.891\\ 338\\ red platelet\\ 0.5\times 0.25\times 0.09\\ 3.19-23.00\\ -1\le h\le 8\\ -9\le k\le 9\\ -13\le 1\le 13\\ 2843\\ 2284\ (R_{int}=0.0243)\\ 1838\\ \psi\mbox{-scan}\\ 0.861\ and\ 0.750\\ full-matrix least-squares on\ F^2\\ 2284/0/195\\ 1.044\\ R_1=0.0434\\ wR_2=0.968\\ D\le 0.016$	11a $C_{32}H_{32}FeO$ 488.43monoclinic $P2_1$ (No. 4)838.9(4)838.9(4)838.3(6)1795.9(9)90101.24(3)901.3052(13)2213(2)1.2430.599516red platelet $0.3 \times 0.3 \times 0.05$ $2.31-18.50$ $-1 \le h \le 7$ $-16 \le l \le 15$ 2100 1767 ($R_{int} = 0.0287$)1430 ψ -scan 0.926 and 0.862 full-matrix least-squares on F^2 1767/1/3121.090 $R_1 = 0.0466$ $wR_2 = 0.0933$
mol formula fw cryst system space group <i>a</i> (pm) <i>b</i> (pm) <i>c</i> (pm) α (deg) β (deg) γ (deg) V (nm ³) Z temp (K) d(calcd) (Mg/m ³) abs coeff (mm ⁻¹) <i>F</i> (000) color, habit cryst size (nm) θ range for data collcn (deg) index ranges no. of rflns collcd no. of indep rflns no. of rflns with $I > 2\sigma(I)$ abs cor max and min transm refinement method data/restraints/parms goodness-of-fit on <i>F</i> ² final R indices [$I > 2\sigma(I)$] <i>R</i> indices (all data)	$\begin{array}{r} \textbf{9a} \\ \hline C_{32}H_{40}Fe_2 \\ 536.34 \\ monoclinic \\ P2_1/n (No. 14) \\ 1759.0(6) \\ 1085.6(3) \\ 1073.0(3) \\ 90 \\ 118.32(2) \\ 90 \\ 2.8123(13) \\ 4 \\ 208(2) \\ 1.267 \\ 1.049 \\ 1136 \\ red platelet \\ 0.55 \times 0.55 \times 0.12 \\ 3.07-21.00 \\ -17 \leq h \leq 6 \\ -10 \leq k \leq 1 \\ -12 \leq k \leq 14 \\ 3113 \\ 2635 (R_{int} = 0.0194) \\ 2198 \\ none \\ \hline full-matrix least-squares on F^2 \\ 2635/0/321 \\ 1.069 \\ R_1 = 0.0511 \\ wR_2 = 0.1131 \\ R_1 = 0.0679 \\ \end{array}$	$\begin{array}{c} 10\\ \hline C_{40}H_{46}Fe_2\\ 638.47\\ triclinic\\ P\bar{1}\ (No.\ 2)\\ 831.6(2)\\ 937.7(2)\\ 1239.9(2)\\ 98.86(1)\\ 108.86(1)\\ 108.86(1)\\ 107.41(1)\\ 0.8387(3)\\ 1\\ 213(2)\\ 1.264\\ 0.891\\ 338\\ red platelet\\ 0.5\times 0.25\times 0.09\\ 3.19-23.00\\ -1\leq h\leq 8\\ -9\leq k\leq 9\\ -13\leq l\leq 13\\ 2843\\ 2284\ (R_{int}=0.0243)\\ 1838\\ \psi\text{-scan}\\ 0.861\ and\ 0.750\\ full-matrix\ least-squares\ on\ F^2\\ 2284/0/195\\ 1.044\\ R_1=0.0434\\ wR_2=0.968\\ R_1=0.0613\\ \end{array}$	$\begin{array}{r} 11a\\ \hline C_{32}H_{32}FeO\\ 488.43\\ monoclinic\\ P2_1 (No. 4)\\ 838.9(4)\\ 883.3(6)\\ 1795.9(9)\\ 90\\ 101.24(3)\\ 90\\ 1.3052(13)\\ 2\\ 213(2)\\ 1.243\\ 0.599\\ 516\\ red platelet\\ 0.3\times0.3\times0.05\\ 2.31-18.50\\ -1\leq h\leq 7\\ -16\leq l\leq 15\\ -16\leq l\leq 15\\ -16\leq l\leq 15\\ 2100\\ 1767 (R_{int}=0.0287)\\ 1430\\ \psi\text{-scan}\\ 0.926 \text{ and } 0.862\\ full-matrix least-squares on F^2\\ 1767/1/312\\ 1.090\\ R_1=0.0466\\ wR_2=0.0933\\ R_1=0.0702\\ \end{array}$
mol formula fw cryst system space group <i>a</i> (pm) <i>b</i> (pm) <i>c</i> (pm) <i>a</i> (deg) β (deg) γ (deg) V (nm ³) Z temp (K) d(calcd) (Mg/m ³) abs coeff (mm ⁻¹) <i>F</i> (000) color, habit cryst size (nm) θ range for data collcn (deg) index ranges no. of rflns collcd no. of rflns with $I > 2\sigma(I)$ abs cor max and min transm refinement method data/restraints/parms goodness-of-fit on F^2 final R indices [$I > 2\sigma(I)$] <i>R</i> indices (all data)	$\begin{array}{c} 9a \\ \hline C_{32}H_{40}Fe_2 \\ 536.34 \\ monoclinic \\ P2_1/n (No. 14) \\ 1759.0(6) \\ 1085.6(3) \\ 1085.6(3) \\ 1085.6(3) \\ 1085.6(3) \\ 90 \\ 118.32(2) \\ 90 \\ 2.8123(13) \\ 4 \\ 208(2) \\ 1.267 \\ 1.049 \\ 1136 \\ red platelet \\ 0.55 \times 0.55 \times 0.12 \\ 3.07-21.00 \\ -17 \leq h \leq 6 \\ -10 \leq k \leq 1 \\ -12 \leq k \leq 14 \\ 3113 \\ 2635 (R_{int} = 0.0194) \\ 2198 \\ none \\ \hline full-matrix least-squares on F^2 \\ 2635/0/321 \\ 1.069 \\ R_1 = 0.0511 \\ wR_2 = 0.1131 \\ R_1 = 0.0679 \\ wR_2 = 0.1222 \\ \end{array}$	$\begin{array}{c} 10\\ \hline C_{40}H_{46}Fe_2\\ 638.47\\ triclinic\\ P\bar{1}\ (No.\ 2)\\ 831.6(2)\\ 937.7(2)\\ 1239.9(2)\\ 98.86(1)\\ 108.86(1)\\ 108.86(1)\\ 107.41(1)\\ 0.8387(3)\\ 1\\ 213(2)\\ 1.264\\ 0.891\\ 338\\ red platelet\\ 0.5\times 0.25\times 0.09\\ 3.19-23.00\\ -1\leq h\leq 8\\ -9\leq k\leq 9\\ -13\leq l\leq 13\\ 2843\\ 2284\ (R_{int}=0.0243)\\ 1838\\ \psi\text{-scan}\\ 0.861\ and\ 0.750\\ full-matrix\ least-squares\ on\ F^2\\ 2284/0/195\\ 1.044\\ R_1=0.0434\\ wR_2=0.968\\ R_1=0.0613\\ wR_2=0.1061\\ \end{array}$	$\begin{array}{c} 11a \\ \hline C_{32}H_{32}FeO \\ 488.43 \\ monoclinic \\ P2_1 (No. 4) \\ 838.9(4) \\ 883.3(6) \\ 1795.9(9) \\ 90 \\ 101.24(3) \\ 90 \\ 1.3052(13) \\ 2 \\ 213(2) \\ 1.243 \\ 0.599 \\ 516 \\ red platelet \\ 0.3 \times 0.3 \times 0.05 \\ 2.31-18.50 \\ -1 \leq h \leq 7 \\ -16 \leq l \leq 15 \\ -16 \leq l \leq 15 \\ -16 \leq l \leq 15 \\ 2100 \\ 1767 (R_{int} = 0.0287) \\ 1430 \\ \psi$ -scan \\ 0.926 and 0.862 \\ full-matrix least-squares on F^2 $1767/1/312 \\ 1.090 \\ R_1 = 0.0466 \\ wR_2 = 0.0933 \\ R_1 = 0.0702 \\ wR_2 = 0.1035 \\ \end{array}$
mol formula fw cryst system space group <i>a</i> (pm) <i>b</i> (pm) <i>c</i> (pm) α (deg) β (deg) γ (deg) V (nm ³) Z temp (K) d(calcd) (Mg/m ³) abs coeff (mm ⁻¹) <i>F</i> (000) color, habit cryst size (nm) θ range for data collcn (deg) index ranges no. of rflns collcd no. of rflns with $I > 2\sigma(I)$ abs cor max and min transm refinement method data/restraints/parms goodness-of-fit on F^2 final R indices $[I > 2\sigma(I)]$ <i>R</i> indices (all data) largest diff peak and hole (e-nm ⁻³)	$\begin{array}{c} \textbf{9a} \\ \hline C_{32}H_{40}Fe_2 \\ 536.34 \\ monoclinic \\ P2_1/n (No. 14) \\ 1759.0(6) \\ 1085.6(3) \\ 1073.0(3) \\ 90 \\ 118.32(2) \\ 90 \\ 2.8123(13) \\ 4 \\ 208(2) \\ 1.267 \\ 1.049 \\ 1136 \\ red platelet \\ 0.55 \times 0.55 \times 0.12 \\ 3.07-21.00 \\ -17 \leq h \leq 6 \\ -10 \leq k \leq 1 \\ -12 \leq k \leq 14 \\ 3113 \\ 2635 (R_{int} = 0.0194) \\ 2198 \\ none \\ \hline full-matrix least-squares on F^2 \\ 2635/0/321 \\ 1.069 \\ R_1 = 0.0511 \\ wR_2 = 0.1131 \\ R_1 = 0.0679 \\ wR_2 = 0.1222 \\ 373 and -428 \\ \end{array}$	$\begin{array}{c} 10\\ \hline C_{40}H_{46}Fe_2\\ 638.47\\ triclinic\\ P\bar{1}\ (No.\ 2)\\ 831.6(2)\\ 937.7(2)\\ 1239.9(2)\\ 98.86(1)\\ 108.86(1)\\ 108.86(1)\\ 107.41(1)\\ 0.8387(3)\\ 1\\ 213(2)\\ 1.264\\ 0.891\\ 338\\ red platelet\\ 0.5 \times 0.25 \times 0.09\\ 3.19-23.00\\ -1 \leq h \leq 8\\ -9 \leq k \leq 9\\ -13 \leq l \leq 13\\ 2843\\ 2284\ (R_{int}=0.0243)\\ 1838\\ \psi\text{-scan}\\ 0.861\ and\ 0.750\\ full-matrix\ least-squares\ on\ F^2\\ 2284/0/195\\ 1.044\\ R_1=0.0434\\ wR_2=0.968\\ R_1=0.0613\\ wR_2=0.1061\\ 352\ and\ -261\\ \end{array}$	$\begin{array}{c} 11a\\ \hline C_{32}H_{32}FeO\\ 488.43\\ monoclinic\\ P2_1 (No. 4)\\ 838.9(4)\\ 883.3(6)\\ 1795.9(9)\\ 90\\ 101.24(3)\\ 90\\ 101.24(3)\\ 90\\ 1.3052(13)\\ 2\\ 213(2)\\ 1.243\\ 0.599\\ 516\\ red platelet\\ 0.3 \times 0.3 \times 0.05\\ 2.31-18.50\\ -1 \leq h \leq 7\\ -16 \leq l \leq 15\\ -16 \leq l \leq 15\\ -16 \leq l \leq 15\\ 2100\\ 1767 (R_{int} = 0.0287)\\ 1430\\ \psi\text{-scan}\\ 0.926 \text{ and } 0.862\\ full-matrix least-squares on F^2\\ 1767/1/312\\ 1.090\\ R_1 = 0.0466\\ wR_2 = 0.0933\\ R_1 = 0.0702\\ wR_2 = 0.1035\\ 246 \text{ and } -203\\ \end{array}$

with 0.047 g (0.420 mmol, 1.1 mequiv) of potassium *tert*butoxide to the corresponding ylide and olefinated with 0.026 g (0.191 mmol, 0.5 mequiv) of terephthalaldehyde. After the usual workup (see above) and chromatography (silica, CH_2 - $Cl_2/n\mbox{-hexane}=2\mbox{:}1)$ 0.075 g (54.4%) of ${\bf 6}$ was obtained as an air-stable, orange-red microcrystalline solid.

Data for (6). Mp: 158–161 °C. Anal. Calcd for C₄₆H₅₈-Fe₂: C, 76.45; H, 8.09. Found: C, 76.53; H, 8.06. MS (EI, 70 eV): m/z 722.5 (M⁺, 100%). HR-MS (FAB): m/z 722.32348 (M⁺; exact mass calcd for C₄₆H₅₈Fe₂, 722.32372). UV-vis (CH₂-Cl₂ [λ_{max} (nm)/log ϵ]): 362/4.29; 490/3.60. IR data (KBr): 3056 w, 2963 w, 2902 w, 1626 w, 1468 w, 1447 w, 1427 w, 1373 m, 1261 m, 1095 m, 1028 s, 955 m, 858 m, 820 vs, 800 vs, 505 m, 460 vs, 445 s cm⁻¹. ¹H NMR (CDCl₃): δ 1.52, 1.65, 1.70, 1.80, 1.96 (48H, 5 × s, 16 × CH₃); 3.29 (2H, s, CH of Cp); 6.65 (2H, d, ³J_{trans}(¹H-¹H) = 16 Hz, CH=CH); 6.85 (2H, d, ³J_{trans}(¹H-¹H) = 16 Hz, CH=CH); 6.85 (2H, d, ³J_{trans}(¹H-¹H) = 16 Hz, CH=CH); 7.40 (4H, s, C₆H₄). ¹³C NMR (CDCl₃): δ 8.44, 8.83, 9.31, 9.44, 9.88, 10.65, 11.13, 11.18 (16 × CH₃); 71.47, 79.37, 80.82, 80.96, 81.94 (Cp); 125.68, 126.47, 126.51 (C₆H₄ and CH=CH). Single crystals were obtained from acetone (Table 1, Figures 3 and 4; Supporting Information).

1,4-Bis{2-[4-(2-(1',2,2',3,3',4,4',5-octamethylferrocenyl)ethenyl)phenyl]ethenyl}benzene (7). *p*-Xylylenebis(triphenylphosphonium bromide) (0.167 g, 0.21 mmol) [CAS Reg. No. 40817-03-6] was converted to the corresponding bis(ylide) with potassium *tert*-butoxide (0.050 g, 0.45 mmol, 2.1 mequiv) in THF and was allowed to react with 2 equiv of 4-[2-(1',2,2',-3,3',4,4',5-octamethylferrocenyl)ethenyl]benzaldehyde **(3)** (0.200 g, 0.47 mmol, 2.2 mequiv) for 2 h at room temperature and for additional 15 min at reflux temperature. During the course of the reaction the product precipitated as a red solid. After cooling to ambient temperature 30 mL of ether was added and the precipitate was filtered off, washed with one portion of ether, water, and acetone, and dried in vacuo, yielding 0.040 g (20.4%) of **7** as an air-stable red solid.

Data for 7. Mp: 200–205 °C, dec. Anal. Calcd for $C_{62}H_{70}$ -Fe₂: C, 80.34; H, 7.61. Found: C, 80.40; H, 7.58. MS (FAB): m/z 926.5 (M⁺, 3%); 307 (CH₃)₈Fc, 100%). UV–vis (CH₂Cl₂ [λ_{max} (nm)/log ϵ]): 400.5/4.87; 503.0/4.12. IR data (KBr): 3023 m, 2965 m, 2944 m, 2902 m, 2859 m, 1626 s, 1595 s, 1516 s, 1425 m, 1377 m, 1109 m, 1080 m, 1030 s, 960 vs, 854 m, 825 s, 547 s, 466 s cm⁻¹. Due to insolubility in all common solvents, no NMR analysis was possible.

1,2,3,4,5-Pentamethyl-1'-(hydroxymethyl)ferrocene (8). A solution of 820 mg (2.9 mmol) of 1,2,3,4,5-pentamethyl-1'-formylferrocene in 30 mL of THF was cooled to 0 °C, and 4.4 mL of a 1.0 M THF solution of lithium triethylborohydride (4.4 mmol) was added. After the solution was stirred for 5 min the cooling bath was removed, and stirring was continued for a further 60 min. Aqueous workup and chromatography (aluminum oxide, *n*-hexane/ether (v/v = 1/1) as eluent) afforded 700 mg (2.5 mmol, 86.2% yield) of 1,2,3,4,5-pentamethyl-1'-(hydroxymethyl)ferrocene **(8)** as yellow crystals.

Data for 8. Mp: 90–92 °C. Anal. Calcd for $C_{16}H_{22}FeO$: C, 67.15; H, 7.75. Found: C, 66.88; H, 7.72. MS (EI, 70 eV): m/z 286 (M⁺, 98%); 270 (M⁺ – OH, 23%); 256 (M⁺ – CH₂OH, 38%). IR data (KBr): 3664–3269 m, br; 2900 m, 1478 m, 1382 s, 1038 s, 814 s cm⁻¹. ¹H NMR (CD₂Cl₂): δ 1.65 (15H, s, CH₃); 3.72 (5H, br m, subst. Cp and OH); 4.20 (2H, s, CH₂). ¹³C NMR (CD₂Cl₂): δ 10.4 (CH₃); 59.9 (CH₂); 71.0, 73.0 (Cp); 79.9 (C₁' of subst. Cp); 80.4 (Cp*).

1,2-Bis(1,2,3,4,5-pentamethylferrocen-1'-yl)ethylene ("(α,ω-bis(pentamethylferrocenyl)[vinylene]") (9a,b). In a Schlenk vessel 200 mg (0.7 mmol) of 1,2,3,4,5-pentamethyl-1'-(hydroxymethyl)ferrocene (8) was dissolved in 20 mL of dry dichloromethane, and the contents were cooled to -80 °C. To the stirred solution was added 0.22 mL (1.6 mmol) of a 54% etheral solution of tetrafluoroboric acid, causing immediate change in color from orange to intense red, indicative of formation of the corresponding (pentamethylferrocenyl)methylium tetrafluoroborate. The mixture was stirred for a further 10 min at a temperature of -80 °C, and 184 mg (0.7 mmol) of triphenylphosphine was added; the cooling bath was removed, and stirring was continued for 1 h, during which time the color changed from red to brownish yellow. Solvents and volatile materials were removed in vacuo, the residue was triturated four times with 20 mL portions of diethyl ether, and the resulting green ((1,2,3,4,5-pentamethylferrocen-1'-yl)methyl)triphenylphosphonium tetrafluoroborate was dried in vacuo, yielding 340 mg (0.56 mmol, 78.6%) of green, solid material. Mp: 85–90 °C. MS (FAB): m/z 531.29 (M⁺ of cation, 100%). IR (KBr): 3058 m, 2964 m, 2906 m, 1629 w, 1588 w, 1482 m, 1385 m, 1111 s, 1061 s, 1040 s, 998 s, 920 m, 745 s cm⁻¹. No NMR spectra could be obtained, probably due to paramagnetic impurities, but the phosphonium salt is sufficiently pure for the following step:

((Pentamethylferrocenyl)methyl)phosphonium tetrafluoroborate (108 mg, 0.17 mmol) was converted to the correspondig ylide with 19 mg (0.17 mmol) of potassium *tert*-butoxide in THF. Addition of 48 mg (0.17 mmol) of 1,2,3,4,5-pentamethyl-1'-formylferrocene and aqueous workup and chromatography (silica, *n*-hexane) yielded a red oil, consisting of *trans*-1,2-bis-(1,2,3,4,5-pentamethylferrocen-1'-yl)ethylene **(9a)** and *cis*-1,2bis-(1,2,3,4,5-pentamethylferrocen-1'-yl)ethylene **(9b)**, respectively. From this mixture, crystallization afforded red crystals (35 mg, 0.065 mmol, 38.4% yield) of the *trans*-isomer **(9a)**: Mp 210 °C.

Data for the Mixture of 9a and 9b. Anal. Calcd for C₃₂-H₄₀Fe₂: C, 71.66; H, 7.52. Found: C, 71.59; H, 7.54. MS (EI, 70 eV): m/z 536.5 (M⁺, 59%); 404.5 (M⁺ − Cp^{*}, 44%). HR-MS (FAB): m/z 536.18280 (M⁺; exact mass calcd for C₃₂H₄₀Fe₂, 536.18288). UV−vis (THF [λ_{max} (nm)/log ϵ]): 453.0/2.99; 460.0/ 2.99. IR data (KBr): 3081 m, 2964 s, 2946 s, 2904 s, 2858 s, 1623 w, 1476 s, 1453 s, 1428 m, 1380 s, 1262 m, 1071 s, 1032 s, 951 m, 851 m cm⁻¹. ¹H NMR (CD₂Cl₂): δ 1.82−1.87 (30H, 3s, CH₃); 3.46−3.80 (8H, 6m, subst. Cp); 6.02, 6.12 (2H, 2s, CH=CH). ¹³C NMR (CDCl₃): δ 11.0−11.1 (CH₃); 69.1−80.1 (Cp and Cp^{*}); 123.3, 124.3 (CH=CH). Single crystals of **9a** were obtained from *n*-hexane (Table 1, Figures 5 and 6; Supporting Information).

1,4-Bis(1,2,3,4,5-pentamethylferrocen-1'-yl-ethenyl)benzene ("(α,ω -Bis(pentamethylferrocenyl)[(vinylene)₂-(phenylene)]") (10). A 126 mg (0.33 mmol) amount of *p*-xylylenebis(diethylphosphonate) [CAS Reg. No. 4546-04-7],¹⁵ dissolved in 30 mL THF, was converted to the corresponding bis(ylide) by addition of 79 mg (0.7 mmol) of potassium *tert*butoxide at a temperature of -80 °C and subsequent warming to room temperature under efficient stirring. The resulting orange solution was cooled to -80 °C, and 200 mg (0.7 mmol) of 1,2,3,4,5-pentamethy-1'-formylferrocene was added. The cooling bath was removed, and the mixture was stirred for 6 h. Aqueous workup and chromatography (silica, ether/*n*hexane (v/v = 1/1) as eluent) afforded 155 mg (0.24 mmol, 72.7% yield) of *trans,trans*-1,4-bis((1,2,3,4,5-pentamethylferrocen-1'-yl)ethenyl)benzene (10) as red crystals.

Data for 10. Mp: 199–203 °C. Anal. Calcd for $C_{40}H_{46}$ -Fe₂: C, 75.25; H, 7.26. Found: C, 75.18; H, 7.29. MS (EI, 70 eV): m/z 638.5 (M⁺, 100%); 448.5 (M⁺ – FeCp*, 19%). HR-MS (FAB): m/z 638.23864 (M⁺; exact mass calcd for $C_{40}H_{46}$ -Fe₂, 638.22982). UV–vis (THF [λ_{max} (nm)/log ϵ]): 358/4.57; 481/3.97. IR data (KBr): 2964 s, 2906 s, 2856 s, 1630 m, 1513 m, 1474 m, 1459 m, 1380 m, 1262 m, 1096 m, 1071 m, 1032 s, 953 s, 861 m cm⁻¹. ¹H NMR (CD₂Cl₂): δ 1.83 (30H, s, CH₃); 3.88 (4H, m, subst. Cp); 3.94 (4H, subst. Cp); 6.56, 6.72 (each signal: 2H, d, ³J_{trans}(¹H–¹H) = 15.9 Hz, CH=CH); 7.42 (4H, s, C₆H₄). ¹³C NMR (CD₂Cl₂): δ 11.1 (CH₃); 69.8, 73.8 (Cp); 81.3 (Cp*); 83.8 (C₁ of subst. Cp); 125.2, 126.1, 126.7, 137.0 (CH=CH and (C₆H₄). Single crystals were obtained from CH₂-Cl₂/EtOH (Table 1, Figure 7; Supporting Information).

4-(2-(1,2,3,4,5-Pentamethylferrocen-1'-yl)ethenyl)-4'formylstilbene ("Pentamethylferrocenyl[(vinylene)₂-(phenylene)₂] Aldehyde") (11a,b). To a suspension of 3 g (3.8 mmol) of *p*-xylylenebis(triphenylphosphonium bromide) [CAS Reg. No. 40817-03-6] in 50 mL of THF at a temperature of -30 °C was added 430 mg (3.8 mmol) of potassium *tert*butoxide, and the mixture was stirred for 0.5 h at -30 °C. The cooling bath was removed, and stirring was continued for 45 min, during which time the color of the solution gradually changed to orange, indicative of formation of the mono(ylide). The mixture was coooled to -30 °C, 700 mg (2.5 mmol) of 1,2,3,4,5-pentamethyl-1'-formylferrocene was added, and further stirring for 2 h afforded the *mono*-olefination product in solution. The mixture was once again cooled to -30 °C, and with 430 mg (3.8 mmol) of potassium *tert*-butoxide the second phosphonium group was converted to the ylide, which was olefinated with 510 mg (3.8 mmol) of terephthalaldehyde in a similar manner as described above. After the mixture was stirred overnight, aqueous workup and chromatography (silica, ether/hexane (v/v = 1/2) as eluent) produced three products. The first orange fraction yielded 250 mg of a mixture of **10** and 4-(2-(1,2,3,4,5-pentamethylferrocen-1'-yl)ethenyl)-4'-meth-ylstilbene methylferrocene, and the second and third red fractions were different isomers (700 mg, 1.43 mmol, 57.3% yield of *trans*-isomer **(11a)** and 240 mg, 0.49 mmol, 19.6% yield of *cis/trans*-isomer **(11b)**) of the desired aldehyde:

Data for 11a. Red crystals formed. Mp: 220 °C, dec. Anal. Calcd for $C_{32}H_{32}FeO$: C, 78.69; H, 6.60. Found: C, 78.65; H, 6.56. MS (EI, 70 eV): m/z 488.5 (M⁺, 100%); 353.5 (M⁺ - Cp^{*}, 63%). HR-MS (FAB): m/z 488.17828 (M⁺; exact mass calcd for $C_{32}H_{32}Fe_2$, 488.18026). IR data (KBr): 2966 w, 2906 m, 2856 w, 2746 w, 1698 s, 1690 s, 1659 s, 1598 s, 1383 m, 1034 m, 824 s cm⁻¹. ¹H NMR (acetone- d_6): δ 1.82 (15H, s, CH₃); 3.87-4.28 (4H, m, subst. Cp); 6.64-7.93 (12H, m, olefinic and aromatic H); 9.71, 10.01 (1H, 2s, CHO). ¹³C NMR (acetone- d_6): δ 11.0 (CH₃); 70.3-77.9 (Cp and Cp^{*}); 125.1-132.7 (olefinic and aromatic C); 192.1 (CHO). Single crystals were obtained from *n*-hexane (Table 1, Figure 8; Supporting Information).

Data for 11b. A red oil formed. Mp: not available. MS (FAB): m/z 489 (M⁺, 23%); 461.6 (M⁺ – CHO, 15%); 281.2 (M⁺ – CH=CHC₆H₄CHO, 97%). IR data (KBr): 2962 w, 2927 w, 2860 w, 1729 s, 1702 s, 1602 s, 1382 m, 1119 s, 1040 s, 807 s cm⁻¹. ¹H NMR (CDCl₃): δ 1.81 (15H, s, CH₃); 3.84–4.32 (4H, m, subst. Cp); 6.54–7.72 (12H, m, olefinic and aromatic H); 9.93, 9.95 (1H, 2s, CHO). ¹³C NMR (CDCl₃): δ 11.0 (CH₃); 68.2–76.2 (Cp and Cp*); 126.1–131.9 (olefinic and aromatic C); not observed, CHO.

 α, ω -Bis(pentamethylferrocenyl)[(vinylene)₄(phenylene)₃] (12). A 57 mg (0.15 mmol) amount of p-xylylenebis-(diethylphosphonate) [CAS Reg. No. 4546-04-7],¹⁵ dissolved in 20 mL of THF, was converted to the corresponding mono(ylide) by addition of 17 mg (0.15 mmol) of potassium tert-butoxide at a temperature of -80 °C and subsequent warming to room temperature under efficient stirring. The resulting orange solution was cooled to -80 °C, 56 mg (0.15 mmol) of 1,2,3,4,5pentamethyl-1'-formylferrocene was added, and the mixture was refluxed for 3 h to complete the mono-olefination. After cooling of the mixture to -80 °C a second portion of 17 mg (0.15 mmol) of potassium tert-butoxide was added and the mixture was allowed to warm to room temperature. The so formed dark solution of the ylide was olefinated by addition of 73 mg (0.15 mmol) of 4-(2-(1,2,3,4,5-pentamethylferrocen-1'-yl)ethenyl)-4'-formylstilbene (11a) at a temperature of -80 °C and subsequent refluxing for 12 h. Aqueous workup and chromatography (silica, ether/*n*-hexane (v/v = 1/1)) afforded 50 mg (0.06 mmol, 40% yield) of 12 as red crystals.

Data for 12. Mp: 185–190 °C. Anal. Calcd for $C_{56}H_{58}$ -Fe₂: C, 79.81; H, 6.94. Found: C, 80.06; H, 6.97. MS (FAB): m/z 843.1 (M⁺, 16%); 652.3 (M⁺ – FeCp^{*}, 10%). HR-MS (FAB): m/z 842.32238 (M⁺; exact mass calcd for $C_{56}H_{58}$ Fe₂, 842.32373). UV–vis (THF [λ_{max} (nm)/log ϵ]): 359/4.38; 481/ 3.73. IR data (KBr): 2964 m, 2925 m, 1630 w, 1262 s, 1100 s, 1028 s, 861 m, 803 s cm⁻¹. ¹H NMR (CD₂Cl₂): δ 1.84 (30H, s, CH₃); 3.86 (4H, m, subst. Cp); 3.92 (4H, subst. Cp); 6.53– 6.76 (8H, m, CH=CH); 7.41 (12H, s, C₆H₄). ¹³C NMR (CD₂-Cl₂): δ 11.1 (CH₃); 69.8, 73.7 (Cp); 81.3 (Cp^{*}); 83.8 (C₁ of subst. Cp); 125.2–126.7 (CH=CH and C₆H₄).

 α,ω -Bis(pentamethylferrocenyl)[(vinylene)₆(phenylene)₅] (13). Method A (Wittig Reaction). A 100 mg (0.125

mmol) amount of *p*-xylylenebis(triphenylphosphonium bromide) was converted to the corresponding bis(ylide) by deprotonation with 31 mg (0.25 mmol) of potassium *tert*-butoxide in a similar manner as described above. Addition of 120 mg (0.25 mmol) of 4-(2-(1,2,3,4,5-pentamethylferrocen-1'-yl)ethenyl)-4'-formylstilbene ("pentamethylferrocenyl][(ene)₂(phenylene)₂]aldehyde") **(11a)** at a temperature of -80 °C and stirring overnight gave a red solution. Aqueous workup and chromatography (silica, dichloromethane/hexane (v/v = 1/2)) yielded 46 mg (0.05 mmol, 59%) α, ω -bis(pentamethylferrocenyl)[(ene)₆-(phenylene)₅] **(13)** as the first fraction, and 40 mg of unreacted pentamethylferrocenyl[(ene)₂(phenylene)₂] aldehyde **(11a)** as the second fraction.

Method B (Wittig–Horner Reaction). A 57 mg (0.15 mmol) amount of *p*-xylylenebis(diethylphosphonate)¹⁵ was converted to the corresponding bis(ylide) by deprotonation with 35 mg (0.31 mmol) of potassium *tert*-butoxide in a similar manner as described above. Addition of 150 mg (0.31 mmol) of 4-(2-(1,2,3,4,5-pentamethylferrocen-1'-yl)ethenyl)-4'-formyl-stilbene ("pentamethylferrocenyl[(ene)₂(phenylene)₂] aldehyde") **(11a)** at a temperature of -80 °C and subsequent refluxing for 5 days gave a red solution. Aqueous workup and chromatography (silica, dichloromethane/hexane (v/v = 1/2)) yielded 50 mg (0.05 mmol, 42%) of α, ω -bis(pentamethylferrocenyl]((ene)₆(phenylene)₅] **(13)** as the first fraction and 35 mg of unreacted pentamethylferrocenyl](ene)₂(phenylene)₂] aldehyde **(11a)** as the second fraction.

Data for 13. A red powder formed. Mp: 275–280 °C (product of method A); mp 180–185 °C (product of method B). Anal. Calcd for $C_{72}H_{70}Fe_2$: C, 82.59; H, 6.74. Found: C, 82.67; H, 6.81. MS (FAB): m/z 1046.7 (M⁺, 12%); 842.5 (M⁺ – FeCp^{*} – Me, 77%); 780.5 (M⁺ – CHCpFeCp^{*}, 15%); 576.2 (M⁺ – C₆H₄-CH=CHCpFeCp^{*}, 100%). HR-MS (FAB): m/z 1046.43908 (M⁺; exact mass calcd for $C_{72}H_{70}Fe_2$, 1046.41815). UV–vis for product of method B (THF [λ_{max} (nm)/log ϵ]): 387.5/4.82; 500/ shoulder. IR data (KBr): 3020 w, 2964 s, 2923 s, 2854 s, 1630 m, 1515 m, 1459 m, 1380 m, 1262 s, 1098 s, 1030 s, 807 s cm⁻¹. ¹H NMR (CD₂Cl₂): δ 1.84 (30H, s, CH₃); 3.86 (4H, m, subst. Cp); 3.92 (4H, subst. Cp); 6.53–6.76 (8H, m, CH=CH); 7.41 (12H, s, C₆H₄). ¹³C NMR (CD₂Cl₂): δ 11.1 (CH₃); 69.8, 73.7 (Cp); 81.3 (Cp^{*}); 83.8 (C1 of subst. Cp); 125.2–126.7 (CH=CH and C₆H₄).

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Supporting Information Available: Tables of crystal data and structure refinement details, anisotropic thermal parameters, fractional atomic coordinates and isotropic thermal parameters for the non-hydrogen atoms, all bond lengths and angles, and fractional atomic coordinates for the hydrogen atoms for **1a,b**, **2–4**, **5a**, **6**, **9a**, **10**, and **11a** and ORTEP diagrams for **1b,c**, **2**, and **3** (Figures 10–13) (76 pages). Ordering information is given on any current masthead page. The authors have deposited atomic coordinates for structures **1b,c**, **2–4**, **5a**, **6**, **9a**, **10**, and **11a** with the Cambridge Crystallographic Data Centre. The coordinates can be obtained, on request, from the Director, Cambridge Crystallographic Data Centre, Lensfield Road, Cambridge, CB2 1EW, U.K.

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