New Building Blocks Based on Truxene Cores: Synthesis of Functionalized *syn*-Tri- and -Hexasubstituted Derivatives

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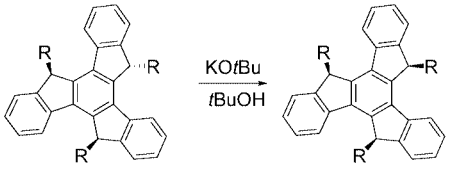
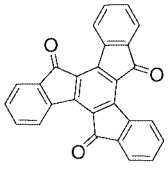
Hexasubstituted truxenes are obtained in one step by alkylation of the potassium anion of truxene. Subsequent derivatization provides truxene derivatives with six carboxy, amino, or hydroxy groups at their peripheries. Alkylation of *syn*-5,10,15-tribenzyl derivatives with a different benzyl bromide derivative affords mixtures of *anti*- and *syn*-hexasubstituted truxenes. Truxenes with phenols or benzoquinone groups have also been synthesized by starting from truxenetrione.

Introduction

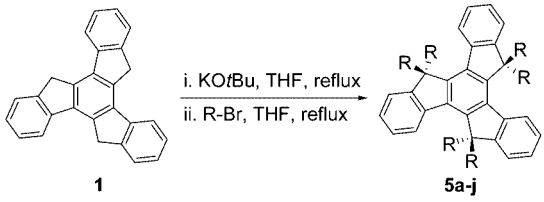
Truxene (10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (**1**, see Scheme 1) has been used for the construction of larger polyarenes[1–5] and for the synthesis of new materials.[6–9] For the synthesis of polyarenes with the topology of the fullerenes,[10–12] we have previously developed a synthesis of derivatives **3** based on treatment of the trianion of **1** with a variety of alkylating agents followed by *anti* to *syn* isomerization with KO*t*Bu in *t*BuOH (Scheme 1).[13,14] We also reported that *syn*-5,10,15-triaryltruxenes **2** (R = aryl)

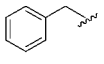
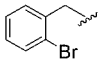
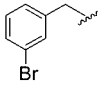
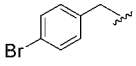
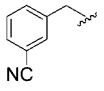
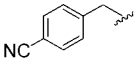
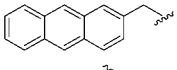
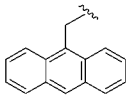
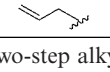




Scheme 1.

Table 1. Synthesis of symmetrical hexasubstituted benzyltruxenes **5a**–**j**.



Entry	R	**5** (Yield, %)
1		**5a** (86; 56[a])
2		**5b** (56; 34[a])
3		**5c** (81)
4		**5d** (92; 60[a])
5		**5e** (75; 35[a])
6		**5f** (94)
7		**5g** (34)
8		**5h** (75)
9		**5i** (83)

[a] This yield refers to the two-step alkylation.

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can be obtained from truxenetrione **4** by addition of aryllithium compounds, reduction of the alcohols with Et_3SiH and BF_3, and *anti* to *syn* equilibration.[15]

Here we report a one-pot synthesis of 5,5,10,10,15,15-hexasubstituted derivatives by simple alkylation of **1** in the presence of excess of base. Several sterically crowded derivatives can be readily prepared by this method. We also report the synthesis of new derivatives substituted at their peripheries with carboxy, amino, or hydroxy groups. A derivative bearing three benzoquinone units as well as the truxene scaffold has also been synthesized. These derivatives could be useful as core structures for the synthesis of dendrimers, as well as in molecular tectonics and for the building of complex supramolecular structures.[16]

Results and Discussion

Synthesis of Hexabenzyltruxenes by Alkylation

Treatment of truxene (**1**) with KO*t*Bu (6 equiv.) gives a green suspension corresponding to a mixture of deprotonated species, which reacts with a variety of alkyl halides to furnish 5,5,10,10,15,15-hexasubstituted truxenes **5a**–**j** in moderate to good yields (Table 1). The synthesis of hexaalkylated truxenes with *n*BuLi (7 equiv.) as the base was also assayed, but mixtures of *syn*- and *anti*-trialkylated truxenes were obtained.[9] Some hexasubstituted derivatives of truxene were synthesized in two steps by initial preparation of *syn*-trialkylated truxenes **3** and subsequent deprotonation with KO*t*Bu (3 equiv.) and addition of RX, but yields were lower by this method. Alkylation with 9-(3-bromopropyl) anthracene[17] failed to provide the corresponding hexasubstituted derivative, and instead gave 9-(prop-1-enyl)anthracene and a mixture of unidentified truxenes.

Derivative **5a** is almost insoluble in common NMR solvents and could be characterized only by FAB mass spectrometry. Similarly, treatment of **1** with 2-(bromomethyl) benzonitrile gave a bluish solid (in ca. 60% yield) that could not be characterized due to its insolubility in all solvents. Interestingly, nitriles **5e** and **5f** are blue in solution and in the solid state. Crystals of nitriles **5e** and **5f** could be grown from DMF/Et_2O solution, and their X-ray diffraction structures show the benzyl groups generally approximately perpendicular to the central truxene plane (Figure 1), although one of the benzyl groups in derivative **5f** adopts a

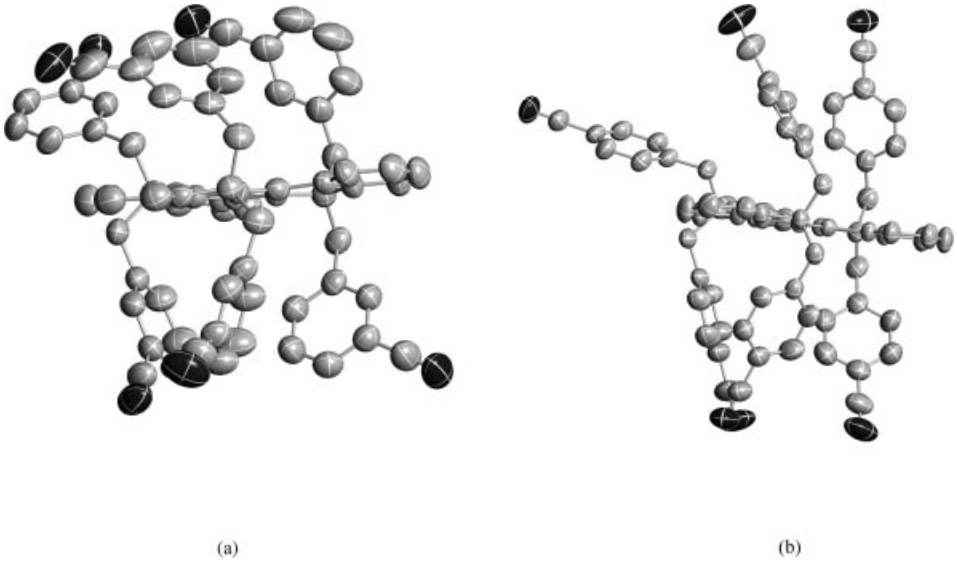


Figure 1. X-ray diffraction structures of nitriles **5e** (a) and **5f** (b).

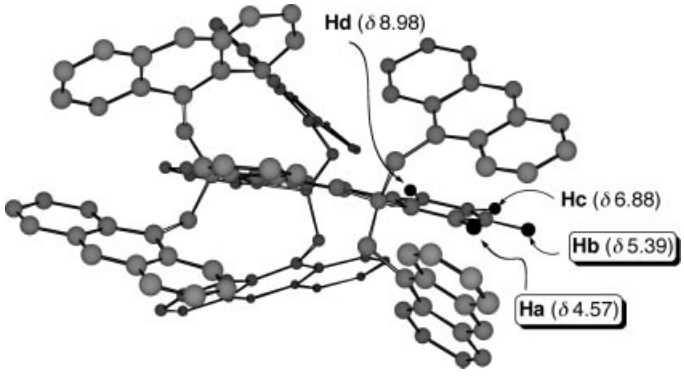


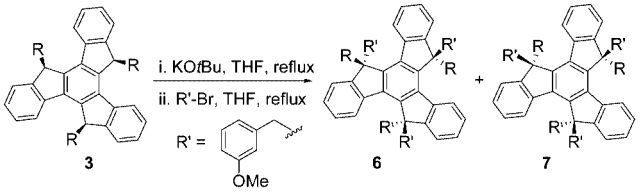
Figure 2. PM3 minimum structure for **5h**.

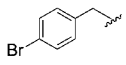
different conformation as the crystal shelters a disordered DMF molecule.

It is interesting to note that the chemical shifts of the truxene hydrogen signals in the hexaalkylated truxenes are concentration-independent in all cases, in contrast with what has been observed with *syn*-trialkylated derivatives **3**,[13] which indicates that hexasubstitution inhibits the self-association. The ^{1}H NMR spectrum of hexakis(9-anthracenyl)methyltruxene **5h** shows signals for Ha (δ = 4.57 ppm) and Hb (δ = 5.39 ppm) shifted upfield relative to those of other truxene derivatives. This effect is the result of the shielding of the hydrogen atoms of the truxene scaffold by the two nearly perpendicular anthracenyl systems, as shown in the PM3-minimized structure of **5h** (Figure 2).

Truxenes with different substituents at their benzylic positions can be obtained by treatment of *syn*-trialkylated truxenes with KO*t*Bu in THF, followed by the addition of alkyl halides to give 1.5:1 to 2:1 mixtures of *anti*- (**6**) and *syn*-hexaalkylated (**7**) truxenes in very good yields (Table 2). The *syn*/*anti* ratios were determined by ^{1}H NMR in the crude reaction mixtures. Hexaalkylated truxenes **6** and **7** could be separated by flash column chromatography.

Table 2. Synthesis of asymmetrical hexabenzyltruxenes.

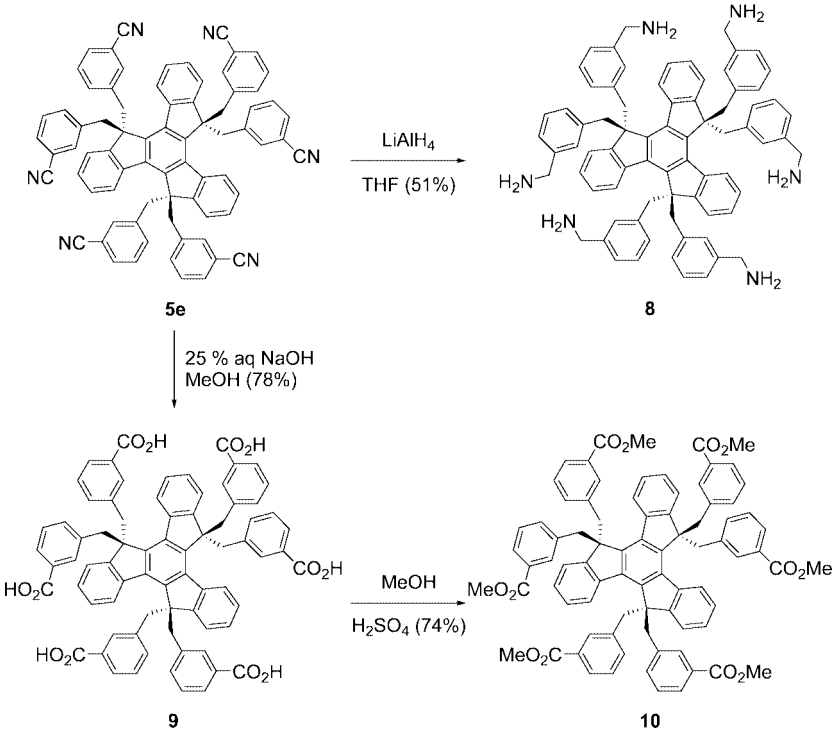


Entry	Truxene **3**	R	**6** (Yield, %)	**7** (Yield, %)
1	**3a**		**6a** (68)	**7a** (29)
2	**3b**		**6b** (66)	**7b** (33)
3	**3c**		**6c** (61)	**7c** (38)

Synthesis of Symmetrical Hexaalkyltruxenes by Functionalization in the Side Chains

Reduction of hexacyano derivative **5e** with $LiAlH_4$ in THF gave the hexaamine **8** in 51% yield (Scheme 2). Compound **5e** also reacted with NaOH in aqueous MeOH under reflux to give derivative **9** in 78% yield. This hexacarboxylic acid shows very broad signals in the ^{1}H NMR spectrum in $CDCl_3$ at room temperature, while the resonances were still not well resolved in 1,1,2,2-tetrachloro[D_2]ethane at 110 °C. The structure of **9** was confirmed by its esterification with MeOH and H_2SO_4 under reflux to give hexaester **10**, showing the expected NMR spectroscopic data, in 74% yield.

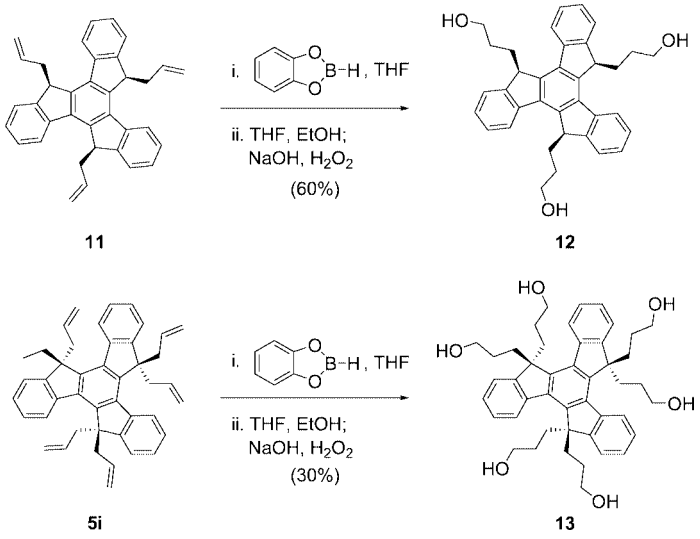
Triptycenes have attracted interest as "spacers" providing crystalline compounds containing channels capable of occluding a variety of other molecules.[18] In consequence, we decided to synthesize a triptycene derivative from hexaanthracenyltruxene **5h** by treatment with anthranilic acid and isoamyl nitrite in 1,2-dichloroethane and benzene under re-



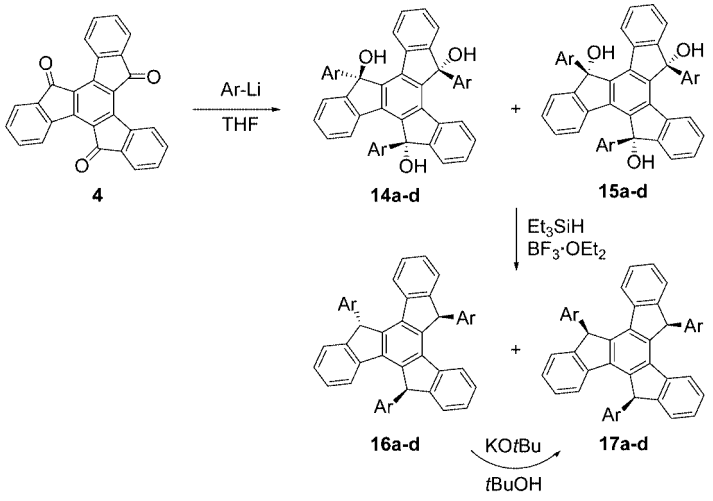
Scheme 2.

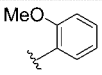
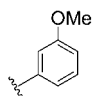
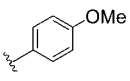
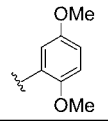
flux.[19,20] However, the resulting yellow solid product could not be characterized, due to its extraordinarily low solubility.

The synthesis of a truxene with six hydroxy substituents was carried out from compound **5i**. Firstly, as a model, *syn*-triallyltruxene **11**[13a] was allowed to react with catecholborane in THF under reflux, followed by oxidative workup, to give trialcohol **12** in 60% yield (Scheme 3). Similarly, **5i** was converted into the hexahydroxy derivative **13**, albeit in lower overall yield. As would be expected from the hydro-



Scheme 3.



Series	Ar	Yield of **4** → **14/15**	**14a–d/15a–d** ratio	Yield of **14/15** → **16/17**	**16a–d/17a–d** ratio	Yield of **16a–d** → **17a–d**
a		96	1:2.8	70	2.6:1	69
b		65	1.1:1	88	10:1	70
c		95	2:1	72	10:1	55
d		90	1:1.1	74	3:1	–

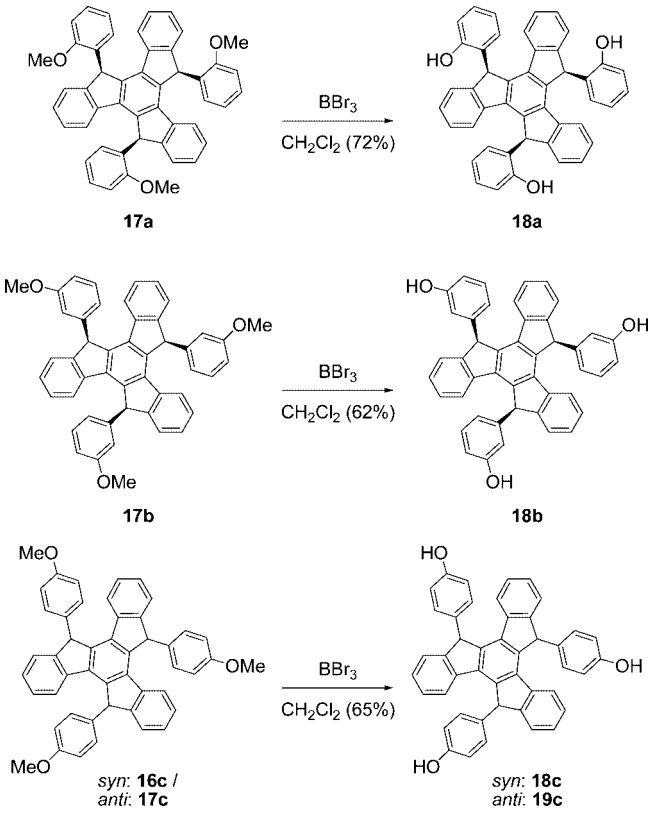
Scheme 4.

philic periphery of **13**, this truxene is more soluble in [D_4] MeOH than in $CDCl_3$.

Synthesis of Functionalized *syn*-Triaryltruxenes

The synthesis of truxenes bearing phenol groups at positions 5, 10, and 15 was carried out by the general approach developed for the preparation of overcrowded *syn*-5,10,15-trisubstituted truxenes.[15b] Thus, truxenetrione **4** was allowed to react with the corresponding aryllithium reagents to give mixtures of *anti* (**14a**–**d**) and *syn* (**15a**–**d**) derivatives (Scheme 4). Reduction of the resulting benzylic alcohols with Et_3SiH and $BF_3{\cdot}OEt_2$ afforded ca. 3–10:1 mixtures of *anti* (**16a**–**d**) and *syn* (**17a**–**d**) derivatives. Finally, base-catalyzed isomerization of *anti* isomers **16a**–**c** furnished pure *syn*-5,10,15-truxenes **17a**–**c**.

Correspondingly, demethylation of compounds **17a** and **17b** with BBr_3 in CH_2Cl_2 at –78 °C provided triphenols **18a** and **18b** in 62–72% yields. No isomerization to the *anti* derivatives was observed under these conditions. Demethylation of a 10:1 mixture of **16c** and **17c** gave a 10:1 mixture of *syn*-**18c** and *anti*-**19c**. Phenols **18a**–**c** are soluble in MeOH and acetone. The ^1H NMR spectra of **18b** and **18c** showed concentration-dependent chemical shifts for the benzylic hydrogen signals, which is characteristic of self-association of *syn*-5,10,15-trisubstituted truxenes.[13] In contrast, the ^1H NMR spectrum of **18a** was not concentration-dependent (Scheme 5).



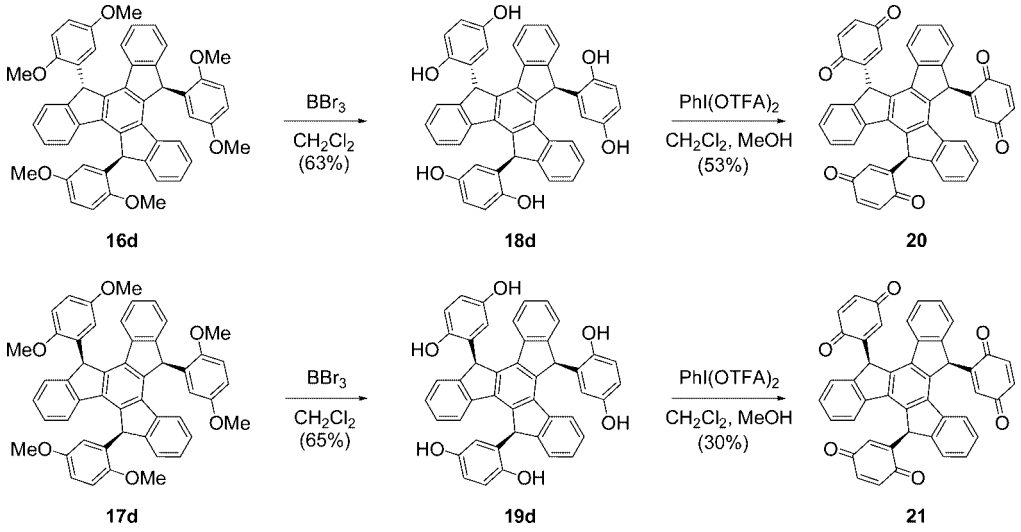
Scheme 5.

Oxidation of a mixture of **16d** and **17d** with ceric ammonium nitrate (CAN) or phenyliodonium bis(trifluoroacetate) (PIFA) failed to give quinones **20** or **21**. However, demethylation of **16d** and **17d** provided **18d** and **19d**, respectively, in satisfactory yields (Scheme 6). These tris(hydroquinones) were oxidized with PIFA in a mixture of MeOH and CH_2Cl_2 to give *anti*-**20** and *syn*-**21** as orange compounds.

Cyclic voltammetry of **20** and **21** (Figure 3) showed two reversible reduction waves corresponding to the formation of the radical anion and the hydroquinone anion. No significant difference in the reduction potentials of stereoisomers **20** and **21** was observed, and they are also very similar to those of 2-methyl- and 2-phenyl-1,4-benzoquinones (Table 3).

Summary

Simple alkylation of the potassium anion of truxene with alkyl halides affords hexasubstituted truxenes in one step.



Scheme 6.

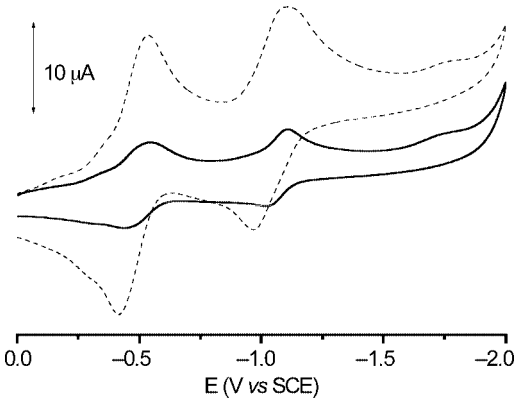


Figure 3. Cyclic voltammogram of tris(quinones) **20** (---) and **21** (−) (room temperature, in CH_2Cl_2 solutions containing 0.1 M $TBAPF_6$ as a supporting electrolyte; scan rate = 100 $mV \cdot s^{-1}$).

Table 3. Reduction potentials of **20**, **21** and reference compounds.

Quinone	Solvent, electrolyte	E^0_I [V]	E^0_{II} [V]
20	CH_2Cl_2, (TBA) PF_6	–0.94	–1.51
21	CH_2Cl_2, (TBA) PF_6	–0.95	–1.53
1,4-Benzoquinone[21]	CH_2Cl_2, (TBA) PF_6	–0.89	–1.80
2-Methyl-1,4-benzoquinone[22]	MeCN, TEAP	–1.04	–1.58
2-Phenyl-1,4-benzoquinone[23]	MeCN, TEAP	–0.95	–1.57

Further derivatization gives rise to truxene derivatives with six carboxy, amino, or hydroxy groups at their peripheries, which could be useful as scaffolds for the construction of larger structures. As expected, alkylation of *syn*-5,10,15-tribenzyl derivatives with a different benzyl bromide affords mixtures of *anti*- and *syn*-hexasubstituted truxenes that can be separated by chromatography. Furthermore, truxenes bearing three pendant phenol, anisole, and benzoquinone units at C5, C10, and C15 have also been synthesized, by starting from truxenetrione. Oxidation of tris(hydroquinone) derivatives affords the corresponding *anti*- and *syn*-tris(benzoquinone) derivatives, which show similar reduction potentials.

Experimental Section

General Remarks: The NMR spectra were determined at 23 °C, unless otherwise stated. The FAB-MS were obtained with *m*-nitrobenzyl alcohol as the matrix. Only the most significant MS fragmentations are given. R_f values were determined on TLC aluminium sheets coated with 0.2 mm GF_{254} silica gel. All reactions were carried out under Ar. Solvents were purified and dried by standard methods. The saturated aqueous NH_4Cl solution was buffered with NH_4OH (pH = 8). Chromatographic purifications were carried out with flash grade silica gel. "Usual workup" means pouring the crude reaction mixture into saturated aqueous NH_4Cl solution, followed by extraction with the stated solvent, drying ($MgSO_4$), and evaporation of the solvent. Truxene (**1**) and truxenetrione **4**,[24] as well as **3a**–**c** and **11**[13] were prepared by the described procedures.

General Procedure for the Synthesis of 5,5′,10,10′,15,15′-Hexaalkyl-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorenes: A mixture of truxene (**1**, 0.58 mmol) and KO*t*Bu (3.48 mmol) in THF (20 mL) was heated under reflux for 30 min. The alkyl bromide (3.48 mmol) in THF (10 mL) was then added to the green suspension. After 16 h, the mixture was cooled and diluted with CH_2Cl_2, washed with saturated aqueous NaCl solution, and dried (Na_2SO_4), and the solvents were evaporated. The residue was usually triturated with CH_2Cl_2 to give pure **5a**–**i**.

5,5,10,10,15,15-Hexakis(phenylmethyl)-10,15-dihydro-5*H*-diindeno-[1,2-*a*;1′,2′-*c*]fluorene (5a): In this case, the solid was filtered off and washed with CH_2Cl_2, acetone, and Et_2O to give **5a** (436 mg, 86%); white solid; m.p. >300 °C. FAB-MS: *m*/*z* (%) = 921 (48) $[M + K]^+$. HR-FAB-MS: *m*/*z* for $C_{69}H_{54}K$: calcd. 921.3863; found 921.3868.

5,5,10,10,15,15-Hexakis[(2-bromophenyl)methyl]-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (5b): Yellow solid (440 mg, 56%): m.p. >300 °C; R_f = 0.18 (10:1 hexane/CH_2Cl_2). ^1H NMR ($CDCl_3$, 300 MHz): δ = 4.17 (d, *J* = 13.7 Hz, 6 H), 4.30 (d, *J* = 13.7 Hz, 6 H), 6.49 (d, *J* = 6.1 Hz, 6 H), 6.59 (t, *J* = 7.3 Hz, 6 H), 6.70 (t, *J* = 8.1 Hz, 6 H), 7.07 (t, *J* = 7.7 Hz, 3 H), 7.21–7.25 (m, 9 H), 7.38 (d, *J* = 8.1 Hz, 3 H), 8.36 (d, *J* = 7.7 Hz, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 40.27 (2 C), 56.74, 124.91, 125.64, 126.00, 126.25, 126.73, 127.62, 129.96, 132.44, 137.52, 146.28, 148.93 (several signals were not observed) ppm. FAB-MS: *m*/*z* (%) = 1395 (34) $[M + K]^+$. HR-FAB-MS: *m*/*z* for $C_{69}H_{48}{}^{79}Br_3{}^{81}Br_3K$: calcd. 1394.8448; found 1394.8432.

5,5,10,10,15,15-Hexakis[(3-bromophenyl)methyl]-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (5c): The residue was triturated with Et_2O to give **5c** (638 mg, 81%); yellow solid; m.p. 140–141 °C; R_f = 0.36 (3:1 hexane/CH_2Cl_2). ^1H NMR ($CDCl_3$, 300 MHz): δ = 3.50 (d, *J* = 13.3 Hz, 6 H), 3.85 (d, *J* = 13.3 Hz, 6 H), 6.04 (d, *J* = 7.9 Hz, 6 H), 6.63 (t, *J* = 7.9 Hz, 6 H), 6.76 (m, 6 H), 7.04 (dd, *J* = 8.1, 1.0 Hz, 6 H), 7.22 (dd, *J* = 7.5, 1.2 Hz, 3 H), 7.40 (t, *J* = 7.5 Hz, 3 H), 7.48 (t, *J* = 7.3 Hz, 3 H), 8.40 (d, *J* = 8.1 Hz, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 125 MHz, DEPT): δ = 40.50 (2 CH_2), 56.85 (C), 121.12 (C), 125.26 (CH), 125.95 (CH), 126.30 (CH), 127.42 (CH), 128.17 (2 CH), 128.94 (CH), 129.08 (CH), 133.31 (CH), 138.52 (C), 139.09 (C), 139.43 (C), 144.35 (C), 145.51 (C) (several signals were not observed, due to overlapping) ppm. MALDI-MS (dithranol + AgTFA): *m*/*z* (%) = 1395 (20) $[M + K]^+$, 1465 (100) $[M + Ag]^+$. HR-MALDI-MS: *m*/*z* for $C_{69}H_{48}{}^{79}Br_3{}^{81}Br_3K$: calcd. 1394.8448; found 1394.8483.

5,5,10,10,15,15-Hexakis[(4-bromophenyl)methyl]-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (5d): Yellow solid (721 mg, 92%): m.p. >300 °C; R_f = 0.35 (2:1 hexane/CH_2Cl_2). ^1H NMR ($CDCl_3$, 300 MHz): δ = 3.41 (d, *J* = 13.7 Hz, 6 H), 3.87 (d, *J* = 13.7 Hz, 6 H), 6.03 (d, *J* = 8.4 Hz, 12 H), 6.92 (d, *J* = 8.1 Hz, 12 H), 7.40–7.48 (m, 9 H), 8.39 (d, *J* = 8.9 Hz, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 40.95 (2 C), 57.17, 120.02, 130.53, 131.39, 135.75 (several signals were not observed) ppm. APCI-MS: *m*/*z* (%) = 1357 (60) $[M + 1]^+$.

5,5,10,10,15,15-Hexakis[(3-cyanophenyl)methyl]-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (5e): The residue was purified by flash chromatography (CH_2Cl_2) and was then triturated with Et_2O to give **5e** (450 mg, 75%); blue solid; m.p. 154–156 °C; R_f = 0.14 (CH_2Cl_2). ^1H NMR ($CDCl_3$, 300 MHz): δ = 3.56 (d, *J* = 13.3 Hz, 6 H), 3.86 (d, *J* = 13.3 Hz, 6 H), 6.33 (dt, *J* = 8.1, 1.2 Hz, 6 H), 6.80 (t, *J* = 1.6 Hz, 6 H), 6.88 (t, *J* = 8.1 Hz, 6 H), 7.23 (dt, *J* = 7.7, 1.2 Hz, 9 H), 7.46 (t, *J* = 7.7 Hz, 3 H), 7.52 (td, *J* = 7.7, 1.2 Hz,

3 H), 8.36 (d, J = 7.7 Hz, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 40.60 (2 C), 56.70, 111.44, 118.50, 125.25, 125.81, 127.31, 128.23, 130.10, 133.45, 138.05, 138.44, 138.86, 143.82, 148.82 (several signals were not observed, due to overlapping) ppm. FAB-MS: m/z (%) 1071 (100) $[M + K]^+$, 1033 (15) $[M + 1]^+$. HR-FAB-MS: m/z for $C_{75}H_{49}N_6$: calcd. 1033.4019; found 1033.4037; for $C_{75}H_{48}N_6K$: calcd. 1071.3602; found 1071.3577.

5,5,10,10,15,15-Hexakis[(4-cyanophenyl)methyl]-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (5f): The solid was filtered off and washed with CH_2Cl_2, acetone, and Et_2O to give **5f** (566 mg, 94%); blue solid; m.p. >300 °C; R_f = 0.08 (CH_2Cl_2). ^{1}H NMR (1,1,2,2-tetrachloro[D_2]ethane, 300 MHz, 80 °C): δ = 3.52 (d, J = 12.8 Hz, 6 H), 3.85 (d, J = 12.8 Hz, 6 H), 6.34 (d, J = 6.5 Hz, 12 H), 7.04 (d, J = 5.7 Hz, 12 H), 7.24 (d, J = 7.7 Hz, 3 H), 7.39 (t, J = 7.3 Hz, 3 H), 7.47 (t, J = 6.9 Hz, 3 H), 8.27 (d, J = 7.3 Hz, 3 H) ppm. ^{13}C NMR (1,1,2,2-tetrachloro[D_2]ethane, 125 MHz, 80 °C): δ = 29.88, 41.91, 110.85, 115.01, 130.65, 131.25, 139.65, 142.40, 172.23 (several signals were not observed) ppm. FAB-MS: m/z (%) = 1071 (100) $[M + K]^+$, 1033 (8) $[M + 1]^+$. MALDI-MS (dithranol): m/z = 1071 $[M + K]^+$. HR-FAB-MS: m/z for $C_{75}H_{49}N_6$: calcd. 1033.4019; found 1033.4029; for $C_{75}H_{48}N_6K$: calcd. 1071.3571; found 1071.3577.

5,5,10,10,15,15-Hexakis(2-methylanthracenyl)-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (5g): The residue was purified by flash chromatography (hexane/CH_2Cl_2, 2:1) and was then triturated with Et_2O to give **5g** (313 mg, 37%); yellow solid; m.p. 200–201 °C; R_f = 0.10 (2:1 hexane/CH_2Cl_2). ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 3.68 (d, J = 13.3 Hz, 6 H), 4.06 (d, J = 13.3 Hz, 6 H), 6.09 (dd, J = 8.9, 1.6 Hz, 6 H), 6.36 (d, J = 8.9 Hz, 6 H), 7.24 (s, 3 H), 7.26–7.45 (m, 27 H), 7.57 (s, 3 H), 7.72–7.81 (m, 12 H), 8.03 (s, 6 H), 8.58 (d, J = 8.1 Hz, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 41.83, 57.53 (2 C), 124.79, 124.99, 125.30, 125.50, 125.74, 125.92, 125.96, 126.11, 126.62, 127.01, 127.73, 127.99, 128.11, 128.47, 129.49, 130.23, 130.26, 130.29, 130.33, 130.49, 130.77, 131.16, 131.38, 131.41, 131.45, 131.48, 131.80, 134.61, 134.66, 134.75, 134.79, 138.85, 139.69, 145.01, 150.50, 150.53 ppm. FAB-MS: m/z (%) = 1483 (18) $[M + 1]^+$. HR-FAB-MS: m/z for $C_{117}H_{79}$: calcd. 1483.6161; found 1483.6181.

5,5,10,10,15,15-Hexakis(9-methylanthracenyl)-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (5h): Yellow solid (647 mg, 75%): m.p. 249–250 °C; R_f = 0.30. ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 4.57 (d, J = 7.3 Hz, 3 H), 4.86 (d, J = 14.6 Hz, 6 H), 5.39 (t, J = 7.3 Hz, 3 H), 5.58 (d, J = 14.6 Hz, 6 H), 6.88 (t, J = 7.3 Hz, 3 H), 7.05–7.18 (m, 24 H), 7.80–7.77 (m, 15 H), 8.17 (s, 6 H), 8.75–7.94 (m, 9 H), 8.98 (d, J = 8.1 Hz, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 34.86, 60.18, 123.77, 125.11, 125.52, 126.03, 127.06, 129.46, 131.85, 132.27, 133.47, 138.91 (the rest of signals were not observed) ppm. MALDI-MS (dithranol): m/z = 1482 $[M]^+$.

5,5,10,10,15,15-Hexakis(3-buten-1-yl)-10,15-dihydro-5*H*-diindeno-[1,2-*a*;1′,2′-*c*]fluorene (5i): The residue was purified by flash chromatography (hexane/CH_2Cl_2, 8.5:1.5) to give **5i** (283 mg, 83%); white solid; m.p. 162–163 °C; R_f = 0.30 (hexane/CH_2Cl_2, 8.5:1.5). ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 2.95 (dd, J = 13.6, 7.2 Hz, 6 H), 3.66 (dd, J = 13.6, 7.6 Hz, 6 H), 4.44 (dd, J = 10.4, 2.0 Hz, 6 H), 4.51 (dd, J = 17.2, 1.6 Hz, 6 H), 4.92–5.02 (m, 6 H), 7.37–7.45 (m, 6 H), 7.56 (dd, J = 7.2, 1.6 Hz, 3 H), 8.32 (d, J = 7.2 Hz, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz, APT): δ = 39.82 (CH_2), 55.15 (C), 116.76 (CH_2), 123.08 (CH), 124.99 (CH), 126.36 (CH), 126.39 (CH), 133.29 (CH), 138.24 (C), 139.76 (C), 143.83 (C), 151.95 (C) ppm. MALDI-MS: m/z = 582 $[M]^+$.

5α,10α,15β-Tris[(2-bromophenyl)methyl]-5β,10β,15α-tris[(3-methoxyphenyl)methyl]-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (6a) and 5α,10α,15α-Tris[(2-bromophenyl)methyl]-5β,10β,15β-tris[(3-methoxyphenyl)methyl]-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (7a): A mixture of 5,10,15-tris(2-bromophenylmethyl)-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (**3a**, 80 mg, 0.09 mmol) and KO*t*Bu (34 mg, 0.28 mmol) in THF (10 mL) was heated under reflux for 30 min. 3-Methoxybenzyl bromide (62 mg, 0.31 mmol) in THF (5 mL) was then added to the green suspension. After 16 h, the mixture was cooled to 23 °C and diluted with CH_2Cl_2, washed with saturated aqueous NaCl solution, and dried (Na_2SO_4). The solvent was evaporated and the residue was triturated with hexane to yield a ca. 2:1 mixture of *anti*-**6a** and *syn*-**7a**. The mixture of isomers could be separated by flash column chromatography (hexane/CH_2Cl_2, 1:1). *anti*-**6a**: 77 mg, 68%; yellow solid; m.p. 200–201 °C; R_f = 0.32 (1:1 hexane/CH_2Cl_2). ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 3.28 (s, 3 H), 3.34 (s, 3 H), 3.48 (s, 3 H), 3.52–4.03 (m, 8 H), 4.08 (d, J = 15.4 Hz, 1 H), 4.18 (d, J = 14.8 Hz, 1 H), 4.32 (d, J = 12.1 Hz, 1 H), 4.46 (d, J = 13.1 Hz, 1 H), 5.42 (d, J = 7.7 Hz, 1 H), 5.62 (dd, J = 7.9, 1.8 Hz, 1 H), 5.90 (d, J = 1.6 Hz, 2 H), 6.06 (d, J = 7.5 Hz, 1 H), 6.18–6.27 (m, 3 H), 6.42 (dd, J = 13.9, 7.7 Hz, 2 H), 6.56–6.68 (m, 2 H), 6.81–7.11 (m, 5 H), 7.51 (dd, J = 7.9, 1.4 Hz, 1 H), 7.17–7.44 (m, 14 H), 7.59 (d, J = 6.5 Hz, 1 H), 8.43 (dd, J = 7.5, 4.6 Hz, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 75 MHz, DEPT): δ = 39.23 (CH_2), 39.31 (CH_2), 39.61 (CH_2), 42.34 (CH_2), 42.45 (CH_2), 42.81 (CH_2), 54.58 (CH_3), 54.70 (CH_3), 54.78 (CH_3), 56.88 (C), 57.13 (C), 57.14 (C), 111.68 (CH), 111.83 (CH), 112.02 (CH), 115.07 (CH), 115.12 (CH), 115.42 (CH), 121.88 (CH), 121.97 (CH), 122.33 (CH), 124.83 (CH), 124.95 (CH), 125.02 (CH), 125.53 (C), 125.61 (CH), 125.70 (CH), 125.80 (CH), 125.91 (C), 126.33 (CH), 126.58 (CH), 126.75 (CH), 127.18 (CH), 127.46 (CH), 127.56 (CH), 127.72 (CH), 128.21 (CH), 129.91 (CH), 130.88 (CH), 131.85 (CH), 132.25 (CH), 132.61 (CH), 137.28 (C), 137.85 (C), 137.93 (C), 138.42 (C), 138.51 (C), 138.57 (C), 138.89 (C), 139.00 (C), 139.25 (C), 145.24 (C), 145.36 (C), 145.57 (C), 149.75 (C), 149.90 (C), 157.85 (C), 158.13 (C), 158.49 (C) (several signals were not observed, due to overlapping) ppm. MALDI-MS (AgTFA): m/z = 1317 $[M + Ag]^+$. HR-MALDI-MS: m/z for $C_{72}H_{57}{}^{107}Ag^{79}Br_3O_3$: calcd. 1313.0908; found 1313.0903. *syn*-**7a**: 33 mg, 29%; yellow solid; m.p. 140–141 °C; R_f = 0.19 (hexane/CH_2Cl_2, 1:1). ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 3.44 (s, 9 H), 3.63 (d, J = 13.3 Hz, 3 H), 3.98 (d, J = 13.1 Hz, 3 H), 4.10 (d, J = 14.3 Hz, 3 H), 4.30 (d, J = 14.6 Hz, 3 H), 5.76 (d, J = 7.7 Hz, 3 H), 6.18 (t, J = 1.6 Hz, 3 H), 6.45 (dd, J = 7.9, 1.8 Hz, 3 H), 6.54 (td, J = 7.3, 1.2 Hz, 6 H), 6.71 (td, J = 8.1, 1.8 Hz, 3 H), 6.74 (t, J = 7.9 Hz, 3 H), 7.21–7.42 (m, 12 H), 8.42 (d, J = 7.7 Hz, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 75 MHz, DEPT): δ = 38.82 (CH_2), 43.12 (CH_2), 54.86 (CH_3), 56.87 (C), 111.97 (CH), 115.65 (CH), 122.33 (CH), 125.03 (CH), 125.55 (CH), 125.77 (C), 125.83 (CH), 126.25 (CH), 126.61 (CH), 127.37 (CH), 128.27 (CH), 129.60 (CH), 132.33 (CH), 137.98 (C), 138.22 (C), 138.74 (C), 138.86 (C), 145.57 (C), 149.71 (C), 158.26 (C) ppm. MALDI-MS (AgTFA): m/z = 1317 $[M + Ag]^+$. HR-MALDI-MS: m/z for $C_{72}H_{57}{}^{107}Ag^{79}Br_3O_3$: calcd. 1313.0908; found 1313.0903.

5α,10α,15β-Tris(3-bromophenylmethyl)-5β,10β,15α-tris[(3-methoxyphenyl)methyl]-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (6a) and 5α,10α,15α-Tris(3-bromophenylmethyl)-5β,10β,15β-tris[(3-methoxyphenyl)methyl]-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (7b): A mixture of 5,10,15-tris(3-bromophenylmethyl)-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (**3b**, 200 mg, 0.24 mmol) and KO*t*Bu (87 mg, 0.71 mmol) in THF (20 mL) was heated under reflux for 30 min. 3-Methoxybenzyl bromide (157 mg, 0.78 mmol) in THF (10 mL) was then added to the green suspension. After 16 h, the mixture was cooled to 23 °C and diluted with CH_2Cl_2, washed with saturated aqueous NaCl solution, and dried (Na_2SO_4). The solvent was evaporated and the residue was tritu-

rated with hexane to yield a ca. 1.5:1 mixture of *anti*-**6b** and *syn*-**7b**. The mixture of isomers could be separated by flash column chromatography (1:1 hexane/CH_2Cl_2). *anti*-**6b**: 190 mg, 66%; yellow solid; m.p. 166–167 °C; R_f = 0.38 (1:1 hexane/CH_2Cl_2). ^1H NMR ($CDCl_3$, 300 MHz): δ = 3.43–3.54 (m, 4 H), 3.56 (s, 3 H), 3.58 (s, 3 H), 3.60 (s, 3 H), 3.63–3.77 (m, 4 H), 3.84 (d, J = 13.4 Hz, 1 H), 3.94 (d, J = 13.4 Hz, 1 H), 4.05 (d, J = 13.4 Hz, 1 H), 4.11 (d, J = 13.4 Hz, 1 H), 5.99 (d, J = 7.7 Hz, 1 H), 5.85 (dd, J = 10.7, 7.7 Hz, 3 H), 6.08 (d, J = 7.5 Hz, 2 H), 6.27 (s, 2 H), 6.35 (s, 1 H), 6.55 (td, J = 6.3, 2.6 Hz, 3 H), 6.62 (t, J = 7.9 Hz, 3 H), 6.75 (d, J = 17.6 Hz, 3 H), 6.82 (d, J = 7.7 Hz, 2 H), 6.89 (t, J = 7.9 Hz, 1 H), 7.02 (dd, J = 16.4, 8.1 Hz, 2 H), 7.30–7.52 (m, 10 H), 8.48 (d, J = 7.7 Hz, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 40.05, 40.44, 40.53, 41.20, 41.39, 41.48, 54.86, 54.91 (2 C), 56.91, 56.99, 57.03, 111.65, 111.81, 115.89, 116.03, 116.20, 120.27, 120.73, 120.87, 122.20, 122.43, 122.50, 122.53, 122.57, 125.21, 125.25, 125.81, 125.89, 125.95, 126.92, 126.95, 127.79, 128.02, 128.07, 128.26, 128.35, 128.39, 128.61, 128.76, 128.81, 128.85, 133.07, 133.17, 138.43, 138.51, 138.54, 138.64, 138.75, 139.38, 139.43, 139.53, 139.61, 139.70, 144.52, 144.68, 144.63, 149.86, 149.90, 158.40, 158.44, 158.52 (several signals were not observed, due to overlapping) ppm. MALDI-MS (AgTFA): m/z = 1317 $[M + Ag]^+$. HR-MALDI-MS: m/z for $C_{72}H_{57}{}^{107}Ag^{79}Br_3O_3$: calcd. 1313.0908; found 1313.0903. *syn*-**7b**: 95 mg, 33%; yellow solid; m.p. 143–144 °C; R_f = 0.16 (1:1 hexane/CH_2Cl_2). ^1H NMR ($CDCl_3$, 300 MHz): δ = 3.53 (br. s, 9 H), 3.54–3.60 (m, 6 H), 3.82–3.95 (m, 6 H), 5.78 (d, J = 7.5 Hz, 3 H), 6.13 (d, J = 7.8 Hz, 3 H), 6.20 (br. s, 3 H), 6.52 (d, J = 8.1 Hz, 3 H), 6.70 (t, J = 7.8 Hz, 3 H), 6.85 (br. s, 3 H), 6.73 (t, J = 7.8 Hz, 3 H), 7.10 (d, J = 8.1 Hz, 3 H), 7.32 (dd, J = 7.3, 2.4 Hz, 3 H), 7.42 (t, J = 7.1 Hz, 3 H), 7.50 (t, J = 7.1 Hz, 3 H), 8.49 (d, J = 7.8 Hz, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 40.77, 40.97, 54.84, 56.93, 111.68, 116.07, 120.99, 122.24, 125.22, 125.79, 125.86, 126.94, 127.84, 128.32, 128.79, 128.89, 133.29, 138.41, 138.53, 139.39, 139.86, 144.61, 149.89, 158.36 ppm. MALDI-MS (AgTFA): m/z = 1317 $[M + Ag]^+$. HR-MALDI-MS: m/z for $C_{72}H_{57}{}^{107}Ag^{79}Br_3O_3$: calcd. 1313.0908; found 1313.0903.

5β,10β,15α-Tris(4-bromophenylmethyl)-5α,10α,15β-tris[(3-methoxyphenyl)methyl]-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (6c) and 5β,10β,15β-Tris(4-bromophenylmethyl)-5α,10α,15α-tris[(3-methoxyphenyl)methyl]-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (7c): A mixture of 5,10,15-tris(4-bromophenylmethyl)-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (**3c**, 100 mg, 0.12 mmol) and KO*t*Bu (43 mg, 0.35 mmol) in THF (10 mL) was heated under reflux for 30 min. 3-Methoxybenzyl bromide (78 mg, 0.39 mmol) in THF (5 mL) was then added to the green suspension. After 16 h, the mixture was cooled to 23 °C and diluted with CH_2Cl_2, washed with saturated aqueous NaCl solution, and dried (Na_2SO_4). The solvent was evaporated and the residue was triturated with hexane to yield a ca. 1.5:1 mixture of *anti*-**6c** and *syn*-**7c**. The mixture of isomers could be separated by flash column chromatography (hexane/CH_2Cl_2 1:1). *anti*-**6c**: 87 mg, 61%; yellow solid; m.p. 130–131 °C; R_f = 0.33 (hexane/CH_2Cl_2, 1:1). ^1H NMR ($CDCl_3$, 300 MHz): δ = 3.50 (s, 3 H), 3.53 (s, 3 H), 3.34–3.59 (m, 8 H), 3.64 (s, 3 H), 3.87–3.95 (m, 2 H), 4.07 (d, J = 13.5 Hz, 1 H), 4.20 (d, J = 13.1 Hz, 1 H), 5.72 (d, J = 7.7 Hz, 1 H), 5.84 (d, J = 7.7 Hz, 1 H), 6.02 (dd, J = 13.3, 8.5 Hz, 5 H), 6.12 (s, 2 H), 6.14 (d, J = 6.2 Hz, 2 H), 6.34 (s, 1 H), 6.50 (dd, J = 7.9, 2.0 Hz, 1 H), 6.62 (ddd, J = 15.8, 7.9, 2.0 Hz, 2 H), 6.75 (dd, J = 15.8, 7.9 Hz, 2 H), 6.83 (dd, J = 10.5, 8.3 Hz, 4 H), 6.92 (t, J = 7.9 Hz, 2 H), 6.99 (d, J = 8.3 Hz, 2 H), 7.33–7.48 (m, 8 H), 8.46 (dd, J = 7.1, 6.2 Hz, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 75 MHz, DEPT): δ = 40.26 (CH_3), 40.93 (CH_3), 41.19 (CH_3), 41.54 (2 CH_3), 41.60 (CH_3), 54.79 (CH_2), 54.81 (CH_2), 54.95 (CH_2), 57.00 (CH_2), 57.22 (CH_2), 57.26 (CH_2), 111.44 (CH), 111.50 (C), 111.55 (C), 111.59 (C), 115.62 (CH), 115.93 (CH), 116.38 (CH), 119.47 (C), 119.63 (C), 119.84 (C), 122.09 (CH), 122.23 (CH), 122.51 (CH), 125.19 (CH), 125.26 (CH), 125.51 (CH), 125.58 (CH), 125.76 (C), 125.82 (CH), 125.86 (CH), 125.90 (CH), 126.92 (CH), 128.19 (CH), 128.56 (CH), 128.62 (CH), 130.08 (C), 130.13 (CH), 130.46 (CH), 130.50 (CH), 131.31 (C), 131.37 (C), 131.41 (CH), 135.94 (C), 136.04 (C), 136.09 (C), 138.59 (C), 138.70 (C), 138.76 (C), 138.80 (C), 139.47 (C), 139.58 (C), 139.61 (C), 144.03 (C), 144.15 (C), 144.47 (C), 149.94 (C), 150.04 (C), 150.08 (C), 158.32 (C), 158.36 (C), 158.57 (C); (several signals were not observed, due to overlapping) ppm. MALDI-MS (AgTFA): m/z = 1317 $[M + Ag]^+$. HR-MALDI-MS: m/z for $C_{72}H_{57}{}^{107}Ag^{79}Br_3O_3$: calcd. 1313.0908; found 1313.0903. *syn*-**7c**: 54 mg, 38%; yellow solid; m.p. 153–154 °C; R_f = 0.43 (hexane/CH_2Cl_2, 1:1). ^1H NMR ($CDCl_3$, 300 MHz): δ = 3.38–3.49 (m, 6 H), 3.58 (s, 9 H), 5.90 (d, J = 7.5 Hz, 3 H), 4.29 (d, J = 13.3 Hz, 6 H), 6.00 (d, J = 8.3 Hz, 6 H), 6.26 (s, 3 H), 6.53 (dd, J = 8.3, 2.4 Hz, 3 H), 6.79 (t, J = 7.5 Hz, 3 H), 6.92 (d, J = 8.3 Hz, 6 H), 7.33–7.48 (m, 9 H), 8.48 (d, J = 7.5 Hz, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 75 MHz, DEPT): δ = 40.38 (CH_2), 41.77 (CH_2), 54.93 (CH_3), 57.21 (C), 111.57 (CH), 116.38 (CH), 119.67 (C), 122.42 (CH), 125.25 (CH), 125.68 (CH), 125.82 (CH), 126.95 (CH), 128.25 (CH), 130.48 (CH), 131.29 (CH), 131.33 (C), 136.07 (C), 138.62 (C), 138.85 (C), 139.54 (C), 144.22 (C), 149.97 (C), 158.47 (C) (several signals were not observed, due to overlapping) ppm. MALDI-MS (AgTFA): m/z = 1317 $[M + Ag]^+$. HR-MALDI-MS: m/z for $C_{72}H_{57}{}^{107}Ag^{79}Br_3O_3$: calcd. 1313.0908; found 1313.0903.

5,5,10,10,15,15-Hexakis{[3-(aminomethyl)phenyl]methyl}-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (8): Truxene **5e** (100 mg, 0.29 mmol) in THF (12 mL) was slowly added at 0 °C to a suspension of $LiAlH_4$ (121 mg, 3.19 mmol) in THF (6 mL), and the mixture was then heated under reflux for 16 h. After the mixture had been cooled to 0 °C, H_2O was added very slowly, together with an aqueous solution of HCl, and the mixture was extracted with CH_2Cl_2. The aqueous layer was basified with an aqueous solution of NaOH and extracted with CH_2Cl_2 and dried ($MgSO_4$), and the solvents were evaporated. The blue oil was triturated with $CHCl_3$/Et_2O (1:100) to give **8** (156 mg, 51%); blue solid; m.p. >300 °C; R_f = 0.14 (CH_2Cl_2). ^1H NMR ($CDCl_3$, 300 MHz): δ = 3.54 (s, 12 H), 3.55 (d, J = 13.4 Hz, 6 H), 3.84 (d, J = 13.4 Hz, 6 H), 6.00 (d, J = 7.3 Hz, 3 H), 6.55 (s, 6 H), 6.78 (t, J = 7.7 Hz, 6 H), 6.86 (d, J = 7.5 Hz, 6 H), 7.34–7.46 (m, 12 H), 8.48 (d, J = 7.7 Hz, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 75 MHz, DEPT): δ = 40.95 (2 CH_2), 46.31 (2 CH_2), 57.16 (C), 124.50 (2 CH), 125.21 (2 CH), 126.21 (CH), 126.48 (CH), 127.50 (2 CH), 128.04 (2 CH), 129.06 (2 CH), 137.47 (2 C), 138.30 (C), 139.69 (C), 142.07 (2 C), 144.85 (C), 150.29 (C) ppm. MALDI-MS (DHB/MeOH): m/z = 1057 $[M + 1]^+$. HR-MALDI-MS: m/z for $C_{75}H_{73}N_6$: calcd. 1057.5891; found 1057.5880.

5,5,10,10,15,15-Hexakis[(3-carboxyphenyl)methyl]-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (9): NaOH (25% aq, 32 mL) was added to a solution of **5e** (400 mg, 0.39 mmol) in MeOH (40 mL) and the mixture was heated under reflux for 16 h. After being cooled, the mixture was poured into HCl solution (10%) and the solid was filtered off and washed with CH_2Cl_2. The aqueous layer was extracted CH_2Cl_2 and dried (Na_2SO_4), the solvent was evaporated, and the residue was triturated with acetone/Et_2O to yield **9** (349 mg, 78%); green solid; m.p. 284–286 °C. ^1H NMR (1,1,2,2-tetrachloro[D_2]ethane, 110 °C, 300 MHz): δ = 3.57–4.40 (m, 12 H), 6.70–7.70 (m, 33 H), 8.00–8.10 (m, 3 H) ppm. ^{13}C NMR (1,1,2,2-tetrachloro[D_2]ethane, 75 MHz, DEPT, 110 °C): δ = 29.69 (CH_2),

30.03 (CH_2), 73.88 (CH), 74.53 (CH) (several signals were not observed) ppm. MALDI-MS (DHB/MeOH): m/z = 1147 $[M + 1]^+$.

5,5,10,10,15,15-Hexakis{[3-(methoxycarbonyl)phenyl]methyl}-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (10): H_2SO_4 (96%, 0.5 mL) was added to a solution of **9** (26 mg, 0.02 mmol) in MeOH (6 mL) and the mixture was heated under reflux for 16 h. After having been cooled, the mixture was diluted with H_2O, extracted with CH_2Cl_2, and dried (Na_2SO_4). The solvent was evaporated and the residue was chromatographed (hexane/EtOAc, 2:1) to give **10** (21 mg, 74%); white solid; m.p. 138–139 °C; R_f = 0.16 (hexane/EtOAc, 2:1). ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 3.47–3.77 (m, 6 H), 3.78 (s, 3 H), 3.79 (s, 3 H), 3.80 (s, 3 H), 3.81 (s, 3 H), 3.82 (s, 3 H), 3.83 (s, 3 H), 3.84–4.06 (m, 6 H), 6.15–6.42 (m, 6 H), 6.71–6.97 (m, 9 H), 7.16–7.52 (m, 15 H), 7.60–7.71 (m, 3 H), 8.38–8.52 (m, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 75 MHz, DEPT): δ = 40.64 (CH_2), 40.74 (CH_2), 51.85 (CH_3), 51.91 (CH_3), 56.60 (C), 56.68 (C), 56.82 (C), 56.92 (C), 110.88 (C), 118.56 (C), 125.22 (CH), 125.34 (CH), 125.63 (CH), 125.81 (CH), 125.92 (CH), 126.05 (CH), 126.60 (CH), 126.78 (CH), 127.13 (CH), 127.30 (CH), 127.95 (CH), 128.03 (CH), 128.91 (CH), 128.99 (CH), 129.11 (CH), 129.51 (CH), 131.47 (CH), 133.60 (CH), 133.70 (CH), 133.89 (CH), 134.02 (CH), 134.10 (CH), 137.07 (C), 137.19 (C), 137.34 (C), 137.43 (C), 138.49 (C), 138.55 (C), 138.69 (C), 138.75 (C), 149.14 (C), 149.47 (C), 149.61 (C), 166.74 (C) (several signals were not observed, due to overlapping) ppm. MALDI-MS (dithranol + NaI): m/z = 1253 $[M + Na]^+$. HR-MALDI-MS: m/z for $C_{81}H_{66}NaO_{12}$: calcd. 1253.4442; found 1253.4446.

5α,10α,15α-Tris(3-hydroxypropyl)-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (12): Catecholborane (0.90 mL, 8.43 mmol) was added very slowly at 0 °C to a mixture of **11** (325 mg, 0.703 mmol) in THF (10 mL), and the mixture was stirred under reflux for 16 h. After the mixture had been cooled to 0 °C, a solution of THF/EtOH (1:1) (12 mL), NaOH (12 mL, 2 M), and H_2O_2 (12 mL) was added. The mixture was extracted with CH_2Cl_2, washed with NaOH solution (1 M), and dried ($MgSO_4$). The solvent was evaporated and the residue was triturated with CH_2Cl_2 to give **11** (218 mg, 60%); yellow solid; m.p. 241–242 °C; R_f = 0.17 (CH_2Cl_2/EtOAc, 1:2). ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 0.74–0.80 (m, 3 H), 0.95–1.05 (m, 3 H), 1.61 (br. s, 3 H), 2.15–2.24 (m, 3 H), 2.33–2.45 (m, 3 H), 2.96–3.16 (m, 6 H), 4.48 (t, J = 4.1 Hz, 3 H), 7.43 (t, J = 7.1 Hz, 3 H), 7.36 (t, J = 7.1 Hz, 3 H), 7.55 (d, J = 7.1 Hz, 3 H), 7.85 (d, J = 7.5 Hz, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 75 MHz, DEPT): δ = 27.11 (CH_2), 27.25 (CH_2), 46.08 (CH), 62.69 (CH_2), 122.63 (CH), 124.84 (CH), 126.55 (CH), 127.07 (CH), 136.80 (C), 140.43 (C), 140.82 (C), 147.99 (C) ppm. MALDI-MS (dithranol + AgTFA): m/z = 539 $[M + Na]^+$. HR-MALDI-MS: m/z for $C_{36}H_{36}NaO_3$: calcd. 539.2562; found 539.2557.

5,5,10,10,15,15-Hexakis(3-hydroxypropyl)-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (13): Catecholborane (1 M in THF, 9.68 mL, 9.68 mmol) was added slowly at 0 °C to a mixture of **5i** (235 mg, 0.403 mmol) in THF (10 mL), and the resulting mixture was stirred under reflux for 16 h. After the mixture had been cooled to 0 °C, a solution of THF/EtOH (1:1, 10 mL), NaOH (10 mL, 2 M), and H_2O_2 (10 mL) was added. The mixture was extracted with CH_2Cl_2 and washed with NaOH solution (1 M). The organic layer was concentrated and the residue was triturated with CH_2Cl_2 (5 mL) and filtered. The filtrate was washed with water and acetone to give **13** (84 mg, 30%); white solid; m.p. 246–248 °C; R_f = 0.40 (CH_2Cl_2/MeOH, 9:1). ^{1}H NMR ([D$_4$]MeOH, 400 MHz): δ = 0.73–0.84 (m, 12 H), 2.28–2.32 (m, 6 H), 3.03–3.16 (m, 18 H), 7.44 (br. s, 6 H), 7.59 (br. s, 3 H), 8.44 (br. s, 3 H) ppm. ^{13}C NMR (CD_3OD, 100 MHz): δ = 28.78, 34.32, 56.30, 63.22, 123.84, 126.09, 127.71, 128.28, 140.32, 141.12, 145.60, 154.22 ppm. ^{13}C NMR ([D$_4$]MeOH, 100 MHz, DEPT): δ = 28.78 (CH_2), 34.32 (CH_2), 56.30 (C), 63.22 (CH_2), 123.84 (CH), 126.09 (CH), 127.71 (CH), 128.28 (CH), 140.32 (C), 141.12 (C), 145.60 (C), 154.22 (C) ppm. HR-EI-MS: m/z for $C_{45}H_{54}NaO_6$: calcd. 713.3818; found 713.3827.

5α,10α,15β-Trihydroxy-5β,10β,15α-tris(2-methoxyphenyl)-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (14a) and 5α,10α,15α-Trihydroxy-5β,10β,15β-tris(2-methoxyphenyl)-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (15a): *n*BuLi (5.6 mL, 14.06 mmol, 2.5 M in hexane) was added at –78 °C to a solution of 2-bromoanisole (1.75 mL, 14.06 mmol) in Et_2O (10 mL). The mixture was warmed up to 10 °C and then a suspension of truxenetrione **4** (600 mg, 1.56 mmol) in THF (50 mL) was added. The mixture was stirred at room temperature for 3 h. After aqueous workup, extraction with EtOAc, and drying with $MgSO_4$, the solvent was evaporated and the residue was triturated with hexane to give a ca. 1:2.8 mixture of **14a** and **15a** (1.06 g, 96%) as a gray solid. The mixture was separated by column chromatography (CH_2Cl_2/EtOAc, 30:1). **14a**: White solid; m.p. 234–236 °C; R_f = 0.39 (CH_2Cl_2/EtOAc, 20:1). ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 4.00 (br. s, 9 H), 6.67–6.75 (m, 3 H), 6.96–7.10 (m, 9 H), 7.14–7.22 (m, 3 H), 7.56–7.75 (m, 9 H) ppm. ^{1}H NMR ([D$_6$]DMSO, 300 MHz): δ = 3.16 (br. s, 9 H), 6.14 (s, 1 H, OH), 6.18 (s, 1 H, OH), 6.22 (s, 1 H, OH), 6.73 (m, 3 H), 6.92–7.03 (m, 12 H), 7.09–7.16 (m, 6 H), 7.84–7.95 (m, 3 H) ppm. ^{13}C NMR ([D$_6$]DMSO, 75 MHz): δ = 55.26, 55.90, 56.04, 81.70 (br), 112.35, 113.38, 113.66, 120.02, 120.44, 123.01, 123.09, 123.20, 125.68, 125.77, 125.93, 126.66, 126.77, 127.08, 127.16, 127.66, 127.72, 128.17, 128.28, 128.36, 132.66, 133.21, 137.68 (br), 138.88 (br), 144.48 (br), 151.15, 151.34, 151.79, 156.61, 156.78, 157.12, 157.17 (some signals were not observed, due to overlapping) ppm. IR: $\tilde{\nu}$ = 3485, 3056, 3928, 1594, 1596, 1483, 1274, 1228, 1040, 1020, 742 cm^{-1}. EI-MS: m/z (%) = 708 (100) $[M]^+$, 691 (18), 674 (9), 601 (19), 583 (8). HRMS: m/z for $C_{48}H_{36}O_6$: calcd. 708.2512; found 708.2508. **15a**: White solid; m.p. >300 °C. R_f = 0.05 (CH_2Cl_2/EtOAc, 20:1). ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 3.69 (br. s, 9 H), 6.90 (br. s, 4 H), 7.01–7.11 (m, 8 H), 7.16–7.22 (m, 4 H), 7.47 (br. s, 4 H), 7.75–7.77 (m, 4 H) ppm. ^{1}H NMR ([D$_6$]DMSO, 200 MHz): δ = 3.12 (br. s, 9 H), 6.19 (s, 1 H, OH), 6.79 (br. s, 3 H), 6.99–7.03 (m, 12 H), 7.12–7.19 (m, 6 H), 7.97 (br. s, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 55.44, 81.58, 112.83, 120.22, 122.93, 125.92, 126.87, 127.18, 127.62, 128.46, 129.10, 132.56, 137.73, 138.53, 151.72, 156.85 ppm. EI-MS: m/z (%) = 708 (43) $[M]^+$, 691 (100), 674 (10). IR: $\tilde{\nu}$ = 3552, 3484, 1730, 1600, 1584, 1490, 1242, 1047, 755, 644 cm^{-1}. HRMS: m/z for $C_{48}H_{36}O_6$: calcd. 708.2512; found 708.2512.

5α,10α,15β-Trihydroxy-5β,10β,15α-tris(3-methoxyphenyl)-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (14b) and 5α,10α,15α-Trihydroxy-5β,10β,15β-tris(3-methoxyphenyl)-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (15b): *n*BuLi (6.16 mL, 15.6 mmol, 2.5 M in hexane) was added at –78 °C to a solution of 3-bromoanisole (1.98 mL, 15.6 mmol) in THF (20 mL). The mixture was warmed up to 10 °C and a suspension of truxenetrione **4** (1.00 g, 2.6 mmol) in THF (80 mL) was then added. The mixture was stirred at room temperature for 17 h. After aqueous workup, extraction with EtOAc, and drying with $MgSO_4$, the solvent was evaporated and the residue was triturated with hexane to give a ca. 1.1:1 mixture of **14b** and **15b** (1.20 g, 65%) as a yellow solid. The mixture was separated by column chromatography (CH_2Cl_2/EtOAc, 40:1). **14b**: Yellow solid; m.p. 176–178 °C; R_f = 0.66 (CH_2Cl_2/EtOAc, 20:1). ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 2.80 (s, 2 H), 2.81 (s, 1 H), 3.69 (s, 6 H), 3.73 (s, 3 H), 6.67–6.72 (m, 3 H), 7.06–7.23 (m, 15 H), 7.31–7.37 (m, 3 H), 7.83–7.87 (m, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 55.05, 55.08, 55.19, 83.50, 83.67,

83.83, 110.86, 111.00, 111.55, 111.83, 112.31, 112.50, 123.52, 123.72, 123.91, 126.23, 126.31, 126.48, 128.68–128.48 (m, 9 C), 129.54, 130.19, 135.93, 136.01, 136.07, 143.20, 143.29, 143.99, 151.07, 151.30, 151.58, 159.64, 159.69 (some signals were not observed, due to overlapping) ppm. IR: $\tilde{\nu}$ = 3478, 3440, 3072, 2932, 2836, 1701, 1602, 1584, 1486, 1460, 1280, 1246, 1147, 1042, 760, 746, 698 cm^{-1}. EI-MS: *m*/*z* (%) = 708 (100) [M]$^+$, 601 (84), 493 (29), 385 (21), 357 (19). HRMS: *m*/*z* for $C_{48}H_{36}O_6$: calcd. 708.2512; found 708.2516. **15b**: White solid; m.p. 264–266 °C; R_f = 0.18 (CH_2Cl_2/EtOAc, 20:1). ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 2.79 (s, 3 H), 3.62 (s, 9 H), 6.60–6.66 (m, 3 H), 6.88 (br. s, 3 H), 6.98–7.06 (m, 6 H), 7.10–7.15 (m, 6 H), 7.197.24 (m, 3 H), 7.75–7.79 (m, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 54.82, 83.32, 110.41, 112.56, 117.16, 123.27, 126.28, 128.46, 128.62, 129.38, 135.57, 139.50, 143.32, 143.68, 151.13, 159.50 ppm. IR: $\tilde{\nu}$ = 3556, 3530, 3470, 3072, 2932, 2838, 1608, 1588, 1480, 1454, 1292, 1250, 762, 702 cm^{-1}. EI-MS: *m*/*z* (%) = 708 (100) [M]$^+$, 601 (73.2), 493 (11), 385 (13), 345 (18). HRMS: *m*/*z* for $C_{48}H_{36}O_6$: calcd. 708.2512; found 708.2518.

5α,10α,15β-Trihydroxy-5β,10β,15α-tris(4-methoxyphenyl)-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (14c) and 5α,10α,15α-Trihydroxy-5β,10β,15β-tris(4-methoxyphenyl)-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (15c): *n*BuLi (11.25 mL, 28.13 mmol, 2.5 M in hexane) was added at –78 °C to a solution of 4-bromoanisole (3.53 mL, 28.13 mmol) in Et_2O (20 mL). The mixture was warmed up to 15 °C and a suspension of truxenetrione **4** (1.20 g, 3.13 mmol) in THF (120 mL) was then added. The mixture was stirred at room temperature for 3 h. After aqueous workup, extraction with EtOAc, and drying with $MgSO_4$, the solvent was evaporated and the residue was triturated with hexane to give a ca. 2:1 mixture of **14c** and **15c** (2.11 g, 95%) as a white solid. The mixture was separated by column chromatography (CH_2Cl_2/EtOAc, 20:1). **14c**: White solid; m.p. 222–224 °C; R_f = 0.77 (CH_2Cl_2/EtOAc, 20:1). ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 2.67 (s, 1 H), 2.68 (s, 1 H), 2.73 (s, 1 H), 3.71 (s, 3 H), 3.72 (s, 6 H), 6.18–6.74 (m, 6 H), 7.05–7.16 (m, 6 H), 7.32–7.35 (m, 3 H), 7.45–7.55 (m, 6 H), 7.77–7.83 (m, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 55.02, 83.30, 83.47, 83.58, 113.76, 113.87, 123.41, 123.66, 126.03, 126.28, 126.36, 126.60, 128.37, 128.43, 134.31, 135.71, 135.82, 139.50, 139.61, 139.78, 143.35, 143.43, 143.44, 151.41, 151.66, 151.80, 158.55, 158.72 (some signals were not observed, due to overlapping) ppm. IR: $\tilde{\nu}$ = 3542, 3442, 1700, 1602, 1576, 1502, 1465, 1242, 1164, 1020, 820, 742, 576 cm^{-1}. EI-MS: *m*/*z* (%) = 708 (100) [M]$^+$, 690 (43), 601 (44), 493 (22), 385 (12), 357 (12). HRMS: *m*/*z* for $C_{48}H_{36}O_6$: calcd. 708.2512; found 708.2513. **15c**: White solid; m.p. >300 °C; R_f = 0.14 (CH_2Cl_2/EtOAc, 20:1). ^{1}H NMR ([D$_6$]acetone, 300 MHz): δ = 2.79 (s, 3 H), 3.65 (s, 9 H), 6.78 (d, *J* = 8.9 Hz, 6 H), 7.00–7.11 (m, 6 H), 7.26 (d, *J* = 8.1 Hz, 3 H), 7.50 (d, *J* = 8.9 Hz, 6 H), 8.11 (d, *J* = 6.9 Hz, 3 H) ppm. ^{13}C NMR ([D$_6$]acetone, 75 MHz): δ = 55.29, 83.91, 114.36, 124.26, 126.97, 127.80, 128.25, 128.47, 136.70, 137.45, 140.16, 145.62, 153.54, 159.51 ppm. IR: $\tilde{\nu}$ = 3556, 1600, 1582, 1514, 1462, 1300, 1249, 1164, 1027, 830, 754 cm^{-1}. EI-MS: *m*/*z* (%) = 708 (100) [M]$^+$, 692 (44), 676 (28),660 (53), 601 (36), 568 (16), 552 (20), 493 (14), 445 (10), 357 (6). HRMS: *m*/*z* for $C_{48}H_{36}O_6$: calcd. 708.2512; found 708.2524.

5α,10α,15β-Trihydroxy-5β,10β,15α-tris(2,5-dimethoxyphenyl)-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (14d) and 5α,10α,15α-Trihydroxy-5β,10β,15β-tris(2,5-dimethoxyphenyl)-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (15d): *n*BuLi (11.25 mL, 28.13 mmol, 2.5 M in hexane) was added at –78 °C to a solution of 1-bromo-2,5-dimethoxybenzene (4.22 mL, 28.13 mmol) in Et_2O (20 mL). The mixture was warmed up to 15 °C and a suspension of truxenetrione **4** (1.20 g, 3.13 mmol) in THF (120 mL) was then added. The mixture was stirred at room temperature for 3 h. After aqueous workup, extraction with EtOAc, and drying with $MgSO_4$, the solvent was evaporated and the residue was triturated with hexane to give a ca. 1:1.1 mixture of **14d** and **15d** (2.25 g, 90%) as a yellow solid. The mixture was separated by column chromatography (CH_2Cl_2/EtOAc, 30:1). **14d**: Pale yellow solid; m.p. 194–196 °C; R_f = 0.68 (CH_2Cl_2/EtOAc, 15:1). ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 3.53 (br. s, 9 H), 3.87 (br. s, 9 H), 6.64–6.70 (m, 3 H), 6.88 (br. s, 3 H), 7.02–7.14 (m, 9 H), 7.58 (br. s, 3 H), 7.75–7.78 (m, 3 H) ppm. ^{1}H NMR ([D$_6$]acetone, 300 MHz): δ = 2.81 (s, 3 H), 3.63 (br. s, 18 H), 5.64 (br. s, 3 H), 6.64–6.71 (m, 3 H), 6.81 (br. s, 3 H), 6.94–7.04 (m, 6 H), 7.34 (br. s, 3 H), 7.91–8.04 (m, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 55.41, 55.58, 55.66, 56.39, 56.54, 56.70, 84.11, 84.52, 85.10, 112.88, 113.05, 113.48, 122.75, 122.83, 127.83, 127.91, 128.15, 128.18, 132.15 (br), 137.10 (br), 138.50, 139.63 (br), 142.39, 151.28, 151.35, 151.53, 153.85, 153.99 (some signals were not observed, due to overlapping) ppm. IR: $\tilde{\nu}$ = 3484, 1606, 1584, 1496, 1222, 1040, 1028, 744 cm^{-1}. EI-MS: *m*/*z* (%) = 798 (100) [M]$^+$, 781 (14), 764 (3), 750 (2), 661 (8), 643 (10). HRMS: *m*/*z* for $C_{51}H_{42}O_9$: calcd. 798.2829; found 798.2824. **15d**: Pale yellow solid; m.p. 184–186 °C; R_f = 0.14 (CH_2Cl_2/EtOAc, 15:1). ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 3.53 (br. s, 9 H), 3.87 (br. s, 9 H), 6.64–6.70 (m, 3 H), 6.88 (br. s, 3 H), 7.02–7.14 (m, 9 H), 7.58 (br. s, 3 H), 7.75–7.78 (m, 3 H) ppm. ^{1}H NMR ([D$_6$]acetone, 300 MHz): δ = 2.79 (s, 3 H), 3.65 (br. s, 18 H), 5.57 (s, 3 H), 6.68 (dd, *J* = 8.9, 2.8 Hz, 3 H), 6.84 (br. s, 3 H), 6.98–7.36 (m, 6 H), 7.36 (br. s, 3 H), 8.01 (br. s, 3 H) ppm. ^{13}C NMR ([D$_6$]acetone, 75 MHz): δ = 55.82, 56.96, 83.70, 113.16, 114.83, 123.68, 127.27, 128.05, 128.19, 134.16, 138.71, 139.60, 144.37, 152.48, 152.90, 154.66 (some signals were not observed, due to overlapping) ppm. IR: $\tilde{\nu}$ = 3484, 1606, 1581, 1496, 1218, 1037, 744, 734 cm^{-1}. EI-MS: *m*/*z* (%) = 798 (100) [M]$^+$, 781 (14), 764 (4), 750 (4), 661 (8), 643 (6). HRMS: *m*/*z* for $C_{51}H_{42}O_9$: calcd. 798.2829; found 798.2830.

5α,10α,15β-Tris(2-methoxyphenyl)-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (16a) and 5α,10α,15α-Tris(2-methoxyphenyl)-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (17a): Et_3SiH (2.67 mL, 16.81 mmol) and BF_3·OEt_2 (1.33 mL, 10.57 mmol) were added at 0 °C to a mixture of **14a** and **15b** (480 mg, 0.68 mmol) in CH_2Cl_2 (15 mL). The mixture was stirred at 0 °C for 45 min and was then partitioned between a saturated aqueous NH_4Cl solution (pH = 7 with NH_4OH) and CH_2Cl_2. After extractive workup, the residue was triturated with EtOAc to give a ca. 2.6:1 mixture of **16a** and **17a** as a yellow solid (309 mg, 70%). The mixture was separated by column chromatography (hexane/CH_2Cl_2, 2:1). **16a**: Yellow solid; m.p. >300 °C; R_f = 0.73 (hexane/CH_2Cl_2, 1:1). ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 4.19 (br. s, 9 H), 6.26–6.38 (m, 4 H), 6.50–6.63 (m, 5 H), 7.06–7.13 (m, 12 H), 7.38–7.46 (m, 6 H) ppm. ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 46.35, 55.76, 55.79, 110.65, 110.76, 121.23 (br), 122.77, 122.85, 124.41, 124.54, 126.45, 126.55, 126.62, 127.60, 127.66, 129.68, 129.75, 129.91, 138.09, 139.10, 139.83, 139.99, 149.62, 149.71, 149.81, 157.14, 157.20 (some signals were not observed, due to overlapping) ppm. EI-MS: *m*/*z* (%) = 660 (100) [M]$^+$, 552 (20), 445 (17). HRMS: *m*/*z* for $C_{48}H_{36}O_3$: calcd. 660.2664; found 660.2677. **17b**: Yellow solid; m.p. >300 °C; R_f = 0.58 (hexane/CH_2Cl_2, 1:1). ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 4.16 (br. s, 9 H), 6.17 (br. s, 3 H), 6.63 (br. s, 6 H), 7.03–7.16 (m, 12 H), 7.39–7.48 (m, 6 H) ppm. ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 48.55, 55.72, 110.77, 121.31, 122.79, 124.46, 126.56, 126.61, 127.67, 129.85, 138.83, 139.86, 157.15 (some signals were not observed, due to overlapping) ppm. EI-MS: *m*/*z* (%) = 660 (100) [M]$^+$, 552 (22), 445 (18). HRMS: *m*/*z* for $C_{48}H_{36}O$: calcd. 660.2664; found 660.2667.

Isomerization of *anti*-16a to *syn*-17a: A suspension of **16a** (71 mg, 0.11 mmol) and KO*t*Bu (15 mg) was heated under reflux in *t*BuOH (10 mL) for 17 h. After having been cooled to room temperature, the mixture was partitioned between water and CH_2Cl_2. Extractive workup and chromatography (hexane/CH_2Cl_2, 2:1) gave **17a** (49 mg, 69%).

5α,10α,15β-Tris(3-methoxyphenyl)-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (16b) and 5α,10α,15α-Tris(3-methoxyphenyl)-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (17b): The procedure described for the synthesis of **16a** and **17a** was applied to **14b/15b** (450 mg, 0.64 mmol), Et_3SiH (2.48 mL, 15.61 mmol), and $BF_3{\cdot}OEt_2$ (1.24 mL, 8.95 mmol). A ca. 10:1 mixture of **16b** and **17b** (370 mg, 88%) was obtained as a yellow solid. The mixture was separated by column chromatography (hexane/EtOAc, 5:1). **16b**: White solid; m.p. 238–240 °C; R_f = 0.41 (3:1 hexane/EtOAc). ^1H NMR ($CDCl_3$, 300 MHz): δ = 3.62 (s, 3 H), 3.65 (s, 3 H), 3.66 (s, 3 H), 5.60 (s, 2 H), 5.62 (s, 1 H), 6.67–6.71 (m, 6 H), 6.85–6.88 (m, 1 H), 6.95–7.01 (m, 2 H), 7.10–7.22 (m, 9 H), 7.42–7.46 (m, 3 H), 7.52–7.61 (m, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 53.93, 54.05, 54.93, 55.08, 11.64, 11.81, 112.14, 112.64, 112.89, 113.40, 120.03, 120.20, 123.46, 123.60, 124.44, 124.61, 124.66, 126.89, 126.95, 129.96, 138.72, 138.80, 139.11, 139.19, 139.25, 139.34, 139.50, 142.90, 142.97, 148.60, 148.73, 160.03 (some signals were not observed, due to overlapping) ppm. EI-MS: *m*/*z* (%) = 660 (100) $[M]^+$, 552 (37), 445 (42), 401 (9). HRMS: *m*/*z* for $C_{48}H_{36}O_3$: calcd. 660.2664; found 660.2658. **17b**: White solid; m.p. >300 °C; R_f = 0.23 (hexane/EtOAc, 3:1). ^1H NMR ($CDCl_3$, 300 MHz): δ = 3.58 (s, 9 H), 4.57 (s, 3 H), 6.42 (s, 3 H), 6.60 (dd, *J* = 8.1, 2.4 Hz, 3 H), 6.66 (d, *J* = 7.3 Hz, 3 H), 7.08 (t, *J* = 7.9 Hz, 3 H), 7.22–7.33 (m, 12 H) ppm. ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 53.07, 54.72, 111.78, 112.42, 119.70, 123.74, 124.69, 126.59, 126.73, 129.77, 138.33, 138.80, 139.03, 142.85, 148.79, 159.72 ppm. EI-MS: *m*/*z* (%) = 660 (100) $[M]^+$, 552 (27), 445 (32), 401 (3). HRMS: *m*/*z* for $C_{48}H_{36}O_3$: calcd. 660.2664; found 660.2669.

Isomerization of *anti*-16b to *syn*-17b: A suspension of **16b** (250 mg, 0.35 mmol) and KO*t*Bu (20 mg) was heated under reflux in *t*BuOH (10 mL) for 17 h. After having been cooled to room temperature, the mixture was partitioned between water and CH_2Cl_2. Extractive workup and trituration with hexane/CH_2Cl_2 gave **17b** (160 mg, 70%).

5α,10α,15β-Tris(4-methoxyphenyl)-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (16c) and 5α,10α,15α-Tris(4-methoxyphenyl)-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (17c): The procedure described for the synthesis of **16a** and **17a** was applied to **14c/15c** (450 mg, 0.64 mmol), Et_3SiH (2.48 mL, 15.61 mmol), and $BF_3{\cdot}OEt_2$ (1.24 mL, 8.95 mmol). A ca. 10:1 mixture of **16c** and **17c** (305 mg, 72%) was obtained as a yellow solid. The mixture was separated by column chromatography (hexane/CH_2Cl_2, 4:1). **16c**: White solid; m.p >300 °C; R_f = 0.5 (hexane/CH_2Cl_2, 2:1). ^1H NMR ($CDCl_3$, 300 MHz): δ = 3.70 (s, 3 H), 3.71 (s, 3 H), 3.72 (s, 3 H), 5.57 (s, 1 H), 5.58 (s, 1 H), 5.60 (s, 1 H), 6.74–6.82 (m, 6 H), 7.08–7.22 (m, 12 H), 7.37–7.42 (m, 3 H), 7.51–7.56 (m, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 300 MHz): δ = 53.21, 53.27, 55.05, 114.34, 114.40, 114.45, 123.54, 123.71, 124.33, 124.58, 126.67, 126.75, 128.26, 133.19, 133.30, 138.55, 138.60, 139.08, 139.10, 139.16, 139.47, 139.64, 149.17, 149.26, 158.29 (some signals were not observed, due to overlapping) ppm. EI-MS: *m*/*z* (%) = 660 (100) $[M]^+$, 552 (48), 445 (25), 401 (8). HRMS: *m*/*z* for $C_{48}H_{36}O_3$: calcd. 660.2664; found 660.2657. **17c**: White solid; m.p. >300 °C; R_f = 0.3 (hexane/CH_2Cl_2, 2:1). ^1H NMR (1,1,2,2-tetrachloro[D_2]ethane, 300 MHz): δ = 3.63 (s, 9 H), 4.99 (s, 3 H), 6.71 (d, *J* = 8.5 Hz, 6 H), 7.01 (d, *J* = 8.4 Hz, 6 H), 7.12–7.19 (m, 6 H), 7.27–7.33 (m, 6 H) ppm. EI-MS: *m*/*z* (%) = 660 (100) $[M]^+$, 568 (14), 552 (41), 445 (21), 401 (7). HRMS: *m*/*z* for $C_{48}H_{36}O_3$: calcd. 660.2664; found 660.2657.

Isomerization of *anti*-16c to *syn*-17c: A suspension of **16c** (250 mg, 0.35 mmol) and KO*t*Bu (20 mg) was heated under reflux in *t*BuOH (10 mL) for 48 h. After having been cooled to room temperature, the mixture was partitioned between water and CH_2Cl_2. Extractive workup and trituration with hexane/CH_2Cl_2 gave **17c** (150 mg, 65%).

5α,10α,15β-Tris(2,5-dimethoxyphenyl)-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (16d) and 5α,10α,15α-Tris(2,5-dimethoxyphenyl)-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (17d): The procedure described for the synthesis of **16a** and **17a** was applied to **14d/15d** (1.80 g, 2.25 mmol), Et_3SiH (6.00 mL, 37.77 mmol), and $BF_3{\cdot}OEt_2$ (3.00 mL, 23.84 mmol). A ca. 3:1 mixture of **16d** and **17d** (1.25 g, 74%) was obtained as a yellow solid. The mixture was separated by column chromatography (hexane/CH_2Cl_2, 1:2). **16d**: White solid; m.p. 278–280 °C; R_f = 0.56 (hexane/CH_2Cl_2, 1:4). ^1H NMR ($CDCl_3$, 300 MHz): δ = 3.34 (s, 6 H), 3.38 (s, 3 H), 4.17 (s, 9 H), 5.94 (br. s, 1 H), 6.00 (br. s, 1 H), 6.08 (br. s, 1 H), 6.23–6.27 (m, 3 H), 6.59–6.65 (m, 3 H), 6.97–7.00 (m, 3 H), 7.10–7.15 (m, 6 H), 7.42–7.52 (m, 6 H) ppm. ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 46.49, 54.97, 55.08, 55.19, 56.25, 11.33, 111.42, 111.53, 111.69, 112.28, 122.74, 122.88, 124.38, 124.55, 126.56, 126.64, 130.88, 130.97, 138.89, 139.00, 139.11, 139.67, 139.78, 149.54, 149.68, 149.76, 151.38, 151.49, 153.61, 153.68 (some signals were not observed, due to overlapping) ppm. EI-MS: *m*/*z* (%) = 750 (100) $[M]^+$, 735 (4), 719 (7), 612 (20), 475 (9). HRMS: *m*/*z* for $C_{51}H_{42}O_6$: calcd. 750.2981; found 750.2988. **17d**: Pale yellow solid; m.p. 292–294 °C; R_f = 0.46 (hexane/CH_2Cl_2, 1:4). ^1H NMR ($CDCl_3$, 300 MHz): δ = 3.33 (s, 9 H), 4.15 (s, 9 H), 6.01 (br. s, 3 H), 6.06 (br. s, 3 H), 6.57 (dd, *J* = 8.9, 3.0 Hz, 3 H), 6.94 (d, *J* = 8.9 Hz, 3 H), 7.01–7.14 (m, 6 H), 7.40–7.42 (m, 3 H), 7.43–7.48 (m, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 46.21, 54.88, 56.19, 111.55, 111.86, 112.22, 122.85, 124.41, 126.59, 126.67, 130.88, 138.69, 139.05, 139.86, 149.68, 151.44, 153.66 ppm. EI-MS: *m*/*z* (%) = 750 (100) $[M]^+$, 735 (3), 719 (6), 612 (18), 475 (8). HRMS: *m*/*z* for $C_{51}H_{42}O_6$: calcd. 750.2981; found 750.2980.

5α,10α,15α-Tris(2-hydroxyphenyl)-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (18a): BBr_3 (0.78 mL, 0.78 mmol, 1 M in CH_2Cl_2) was added at –78 °C to a solution of **17a** (85 mg, 0.13 mmol) in CH_2Cl_2 (10 mL). The mixture was stirred at –78 °C for 1 h and at room temperature for 12 h. After being partitioned between CH_2Cl_2 and water, and extractive workup, the residue was triturated with hexane/CH_2Cl_2, and then with EtOAc to give **18a** (58 mg, 72%): White solid; m.p. >300 °C. ^1H NMR ([D_4]MeOH, 300 MHz): δ = 6.11 (br. s, 3 H), 6.49 (m, 6 H), 6.95–6.97 (m, 6 H), 7.04–7.10 (m, 6 H), 7.54–7.56 (m, 6 H) (3 H corresponding to the OH signals were not observed) ppm. ^{13}C NMR ([D_4]MeOH, 75 MHz): δ = 47.81, 116.41, 120.96, 124.08, 125.67, 127.51, 128.54, 129.43, 140.09, 140.31, 141.26, 151.44, 156.46 (some signals were not observed, due to overlapping) ppm. IR: $\tilde{\nu}$ = 3560, 3470, 3414, 1621, 1592, 1450, 1148, 1082, 743, 614 cm^{-1}. EI-MS: *m*/*z* (%) = 618 (100) $[M]^+$, 524 (40), 431 (23). HRMS: *m*/*z* for $C_{45}H_{30}O_3$: calcd. 618.2194; found 618.2185.

5α,10α,15α-Tris(3-hydroxyphenyl)-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (18b): The same procedure as described for the preparation of **18a** was applied to **17b** (160 mg, 0.24 mmol) in CH_2Cl_2 (10 mL) to give **18b** (92 mg, 62%); white solid; m.p. >300 °C. ^1H NMR ([D_4]MeOH, 300 MHz): δ = 3.89 (s, 3 H), 6.19 (s, 3 H), 6.29 (dd, *J* = 8.1, *J* = 7.1 Hz, 3 H), 6.42 (dd, *J* = 8.1, *J* = 2.4 Hz, 3 H), 6.88 (t, *J* = 7.7 Hz, 3 H), 7.05 (d, *J* = 7.7 Hz, 3 H),

7.18 (t, J = 7.3 Hz, 6 H), 7.32 (t, J = 7.7 Hz, 3 H) ppm. FAB-MS: m/z (%) = 618 $[M]^+$ (4), 525 $[M]^+$ (3). HRMS: m/z for $C_{45}H_{30}O_3$: calcd, 618.2195; found 618.2206.

5α,10α,15β-Tris(4-hydroxyphenyl)-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (18c) and 5α,10α,15α-Tris(4-hydroxyphenyl)-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (19c): The same procedure as described for the preparation of **18a** was applied to a mixture of **16c** and **17c** (1.00 g, 1.51 mmol) in CH_2Cl_2 (45 mL) to give **18c** and **19c** (652 mg, 70%) as a gray solid. The mixture was separated by column chromatography (hexane/CH_2Cl_2, 2:1). **18c**: White solid; m.p. >300 °C; R_f = 0.48 (hexane/EtOAc, 1:1). ^{1}H NMR ([D$_4$]MeOH, 200 MHz): δ = 5.51 (s, 1 H), 5.52 (s, 1 H), 5.58 (s, 1 H), 6.61–6.68 (m, 6 H), 6.98–7.01 (m, 12 H), 7.32–7.34 (m, 3 H), 7.50–7.59 (m, 3 H) ppm. IR: $\tilde{\nu}$ = 3513, 3384 (br), 1694, 1616, 1592, 1514, 1240, 1172, 745 cm^{-1}. FAB-MS: m/z (%) = 618 $[M]^+$ (15), 525 (8), 460 (6), 382 (17). HRMS: m/z for $C_{45}H_{30}O_3$: calcd. 618.2195; found 618.2190. **19c**: White solid; m.p. >300 °C; R_f = 0.18 (hexane/EtOAc, 1:1). ^{1}H NMR ([D$_4$]MeOH, 300 MHz): δ = 3.78 (s, 3 H), 6.42–6.51 (m, 12 H), 6.93 (d, J = 7.7 Hz, 3 H), 7.07–7.13 (m, 6 H), 7.26 (t, J = 7.3 Hz, 3 H) ppm. ^{13}C NMR ([D$_4$]MeOH, 75 MHz): δ = 53.55, 116.52, 125.42, 125.97, 127.31, 127.42, 129.24, 133.62, 139.03, 140.11, 140.81, 151.05, 156.93 ppm. FAB-M: m/z (%) = 618 (1) $[M]^+$.

5α,10α,15β-Tris(2,5-dihydroxyphenyl)-10,15-dihydro-5*H*-diindeno-[1,2-*a*;1′,2′-*c*]fluorene (18d): The same procedure as described for the preparation of **18a** was applied to **16d** (150 mg, 0.20 mmol) in CH_2Cl_2 (15 mL) to give **18d** (84 mg, 63%); gray solid; m.p. >300 °C. ^{1}H NMR ([D$_6$]acetone, 200 MHz): δ = 5.70 (d, J = 3.1 Hz, 1 H), 5.89 (d, J = 3.1 Hz, 1 H), 5.93 (d, J = 3.1 Hz, 1 H), 6.19–6.23 (m, 3 H), 6.38–6.45 (m, 3 H), 6.91 (d, J = 8.7 Hz, 3 H), 7.04–7.15 (m, 6 H), 7.57–7.64 (m, 3 H), 7.67–7.70 (m, 3 H) ppm. ^{13}C NMR ([D$_6$]acetone, 75 MHz): δ = 47.40, 113.41, 113.83, 114.83, 114.92, 116.90, 116.95, 123.84, 125.52, 125.63, 127.44, 127.50, 127.61, 129.20, 129.36, 129.64, 139.74, 139.82, 139.93, 140.74, 140.91, 148.64, 148.77, 151.00, 151.06, 151.15, 151.20 (some signals were not observed, due to overlapping) ppm. IR: $\tilde{\nu}$ = 3504, 3308 (br), 1608, 1506, 1454, 1190, 754 cm^{-1}. FAB-MS: m/z (%) = 666 (100) $[M]^+$, 575 (57), 447 (18). HRMS: m/z for $C_{45}H_{30}O_6$: calcd. 666.2042; found 666.2041.

5α,10α,15α-Tris(2,5-dihydroxyphenyl)-10,15-dihydro-5*H*-diindeno-[1,2-*a*;1′,2′-*c*]fluorene (19d): The same procedure as described for the preparation of **18a** was applied to **17d** (150 mg, 0.20 mmol) in CH_2Cl_2 (15 mL) to give **19d** (87 mg, 65%); gray solid; m.p. >300 °C. ^{1}H NMR ([D$_4$]MeOH, 300 MHz): δ = 6.04 (br. s, 6 H), 6.48 (dd, J = 8.7, 3.0 Hz, 3 H), 6.88 (d, J = 8.5 Hz, 3 H), 7.07–7.15 (m, 6 H), 7.55–57 (m, 3 H), 7.60–7.63 (m, 3 H) ppm. ^{13}C NMR ([D$_4$]MeOH, 75 MHz): δ = 47.83, 114.43, 115.24, 117.11, 124.22, 125.75, 127.59, 130.49, 140.20, 140.31, 141.09, 149.62, 150.93, 151.38 ppm. IR: $\tilde{\nu}$ = 3350 (br), 1694, 1600, 1506, 1446, 1352, 1198, 814, 736 cm^{-1}. FAB-MS: m/z (%) = 666 $[M]^+$ (64), 575 $[M - C_6H_5O_2]^+$ (35), 490 (9). HRMS: m/z for $C_{45}H_{30}O_6$: calcd. 666.2042; found 666.2038.

2α,5α,15β-Tris[(2,5-cyclohexadien-1,4-dione-2-yl)]-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (20): Phenyliodonium bis(trifluoroacetate) (PIFA, 242 mg, 0.56 mmol) was added to a solution of **18d** (25 mg, 0.04 mmol) in CH_2Cl_2 (2 mL) and MeOH (0.2 mL). The mixture was stirred at room temperature for 40 min. After partition between CH_2Cl_2 and water and extractive workup, **20** (13 mg, 53%) was obtained; orange solid; m.p. >300 °C. ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 5.85 (br. s, 1 H), 5.90–6.03 (m, 5 H), 6.64–6.73 (m, 3 H), 7.00 (dd, J = 10.1, 2.4 Hz, 3 H), 7.25–7.45 (m, 12 H) ppm. ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 45.58 (br) 122.60, 122.68, 125.14, 125.23, 125.40, 128.11, 128.23, 129.92 (br), 130.30 (br), 136.35, 136.42, 136.42, 136.46, 136.54, 136.71, 136.75, 137.49, 137.95, 137.99, 138.16, 139.49, 139.51, 145.69, 146.26, 146.35, 147.48, 147.81, 147.96, 187.07, 187.64, 187.69 (some signals were not observed, due to overlapping) ppm. IR: $\tilde{\nu}$ = 2932, 2855, 1650, 1591, 1462, 1292, 916, 736, 420 cm^{-1}. UV/Vis (EtOH): λ_{max} (log ε) = 238 (4.71), 278 (4.57), 299 (4.49) nm. EI-MS: m/z (%) = 660 (100) $[M]^+$, 207 (50), 165 (47). HRMS: m/z for $C_{45}H_{24}O_6$: calcd, 660.1573; found 660.1575.

2α,5α,15α-Tris[(2,5-cyclohexadien-1,4-dione-2-yl)]-10,15-dihydro-5*H*-diindeno[1,2-*a*;1′,2′-*c*]fluorene (21): Phenyliodonium bis(trifluoroacetate) (PIFA, 200 mg, 0.46 mmol) was added to a solution of **19d** (20 mg, 0.03 mmol) in CH_2Cl_2 (2 mL) and MeOH (0.2 mL). The mixture was stirred at room temperature for 40 min. After partitioning between CH_2Cl_2 and water and extractive workup, **21** (6 mg, 30%) was obtained; orange solid; m.p. >300 °C. ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 5.62 (s, 3 H), 6.13 (s, 3 H), 6.69 (dd, J = 10.1, 2.4 Hz, 3 H), 6.96 (d, J = 10.1 Hz, 3 H), 7.24–7.39 (m, 12 H) ppm. ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 46.01, 122.60, 125.28, 128.05, 128.05, 128.17, 131.41 (br), 136.42, 136.74, 138.19, 139.21, 145.73, 147.49, 186.90, 187.55 ppm. IR: $\tilde{\nu}$ = 2924, 2855, 1650, 1600, 1292, 916, 745, 412 cm^{-1}. UV/Vis (EtOH): λ_{max} (log ε) = 206 (4.58), 237 (4.58), 279 (4.44), 300 (4,34). EI-MS: m/z (%) = 660 (4) $[M]^+$, 322 (35), 207 (18), 167 (100) nm.

Table 4. Collecting parameters, crystal and determination data for **5e**.

Diffractometer	Bruker–Siemens Smart CCD
Radiation	Cu-K_α (λ = 1.54178 Å)
T [K]	296(2)
θ [°]	2.37–63.68
Crystal degradation	inappreciable
Index ranges	$-42 \le h \le 43$, $-15 \le k \le 14$, $-21 \le l \le 23$
Empirical formula	$C_{75}H_{48}N_6$
Formula mass	1033.19
Crystal system	monoclinic
Symmetry group	$C2/c$
a [Å]	37.3451(7)
b [Å]	14.3265(3)
c [Å]	22.1848(4)
β [°]	90.8820(10)
Volume [Å^3]	11868.0(4)
Z	8
Calculated density [Mg/m^3]	1.156
F(000)	4320
μ [mm^{-1}]	0.527
Number of reflections observed	9091
Number of independent reflections	5308
Goodness-of-fit	1.002
max/min. $\Delta\rho$ [e Å^{-3}]	1.551/–0.323
R factors	$R1$ = 0.1261, $wR2$ = 0.2883
Final R [$I > 2\sigma(I)$]	$R1$ = 0.0876, $wR2$ = 0.2452

X-ray Crystal Structure Determination of 5e: Blue crystals of **5e** suitable for X-ray diffraction studies were obtained by slow concentration of a DMF/Et_2O (1:100) solution at 23 °C. Crystals were mounted on a Bruker–Siemens Smart CCD diffractometer equipped with a low-temperature device, a normal-focus, 2.4-kW sealed-tube X-ray source (Cu-K_α; λ = 1.54178 Å). The cell parameters were determined by least-squares fit for all reflections collected. Full-matrix least-squares refinements were carried out, min-

imizing $\omega(F_o^2 - F_c^2)^2$. R_w and goodness-of-fit are based on F^2. Most of the calculations were carried out with the SMART software for data collection and reduction and SHELXTL97[25] for structure solution and refinement. The collecting parameters, crystal and determination data are given in Table 4.

X-ray Crystal Structure Determination of 5f: Blue crystals of **5f** suitable for X-ray diffraction studies were obtained by slow concentration of a DMF/Et_2O (1:100) solution at 23 °C. Crystals were mounted on a Bruker–Siemens Smart CCD diffractometer equipped with a low-temperature device, a normal-focus, 2.4-kW sealed-tube X-ray source (Cu-K_α; λ = 1.54178 Å). The cell parameters were determined by least-squares fit for all reflections collected. Full-matrix least-squares refinements were carried out, minimizing $\omega(F_o^2 - F_c^2)^2$. *Rw* and goodness-of-fit are based on F^2. Most of the calculations were carried out with the SMART software for data collection and reduction and SHELXTL97[25] for structure solutions and refinement. The collecting parameters, crystal and determination data are shown in Table 5.

Table 5. Collecting parameters, crystal and determination data for **5f**.

Diffractometer	Bruker–Siemens Smart CCD
Radiation	Cu-K_α (λ = 1.54178 Å)
T [K]	273(2)
θ (°)	2.54–70.54
Crystal degradation	inappreciable
Index ranges	$-21 \le h \le 21$, $0 \le k \le 25$, $0 \le l \le 20$
Empirical formula	$C_{75}H_{48}N_6$
Formula mass	1033.19
Crystal system	monoclinic
Symmetry group	$P2_1/c$
a [Å]	18.0281(3)
b [Å]	21.2291(4)
c [Å]	17.5830(3)
β [°]	105.3230(10)
Volume [Å^3]	6490.2(2)
Z	4
Calculated density [Mg/m^3]	1.207
F(000)	2480
μ [mm^{-1}]	0.574
Number of reflections observed	11833
Number of independent reflections	8066
Goodness-of-fit	1.032
max/min. $\Delta\rho$ [e Å^{-3}]	0.376, 0.286
R factors	$R1$ = 0.0759, $wR2$ = 0.1759
Final R [$I > 2\sigma(I)$]	$R1$ = 0.0759, $wR2$ = 0.1897

CCDC-280573 (for **5e** and **5f**) contains the supplementary crystallographic data. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

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