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Intramolecular versus intermolecular oxidative couplings of ester tethered di-aryl ethers

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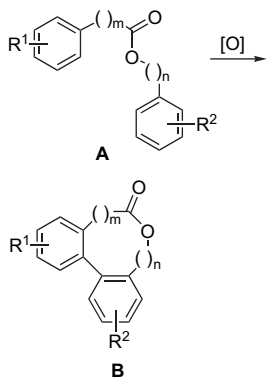
Abstract—The oxidative cyclization of 3,4-dimethoxyphenyl 3,4-dimethoxyphenylacetate, through intramolecular biphenyl bond formation, was successful and gave the target seven-membered lactone in good yield (85–86%). All other ester substrates gave biphenyl products or their further oxidized products via intermolecular coupling of their radical cation intermediate with the neutral substrate. It appears that matching of the oxidation potentials and nucleophilicity of the two phenyl rings, the positioning of the ring substituents and the ease of *E* to *Z* isomerization about the ester C–O bond are important factors contributing to these product outcomes.

1. Introduction

As a part of a medicinal chemistry project we have explored the oxidative coupling reactions of the ester tethered di-aryl esters **A** aimed at the synthesis of lactones of type **B** as shown in Scheme 1. While both the intramolecular and the intermolecular oxidative couplings of phenyl ethers to give biphenyls using one electron oxidants have been reported, these have generally been restricted to electron rich substrates.[1–27] The intramolecular versions of these reactions have been used to prepare six-[28–31] and seven-membered[23] carbocyclic ring products using an all-carbon tether between the two participating aryl ethers,[32] while six-,[31] seven-[23,33,34] and eight-membered[33,34] heterocyclic ring products have been obtained when the tether contains a heteroatom (N, O, S and Si). The use of an ester tether in these types of reactions has not been reported.[35] It was thus of interest to explore the oxidative coupling reactions of **A** with single electron oxidants and to determine the effects of ring size and the electronic properties of the two coupling partners on the efficiencies and product distributions of such reactions.



Scheme 1.

2. Results and discussion

2.1. Oxidative coupling reactions of substituted phenyl phenylacetates

Initial experiments focussed on the study of the oxidative coupling reactions of the tetramethoxy substituted phenyl phenylacetate derivative **1a** with the one electron oxidants, $FeCl_3/SiO_2$,[1,2] $MoCl_5$,[3–7] VOF_3,[8–14] thallium(III) trifluoroacetate (TTFA),[15–20] $Ce(OH)_4$[21,22] and phenyl iodine(III) bis(trifluoroacetate) (PIFA),[23–27] using literature procedures (Scheme 2). Reactions with the latter three oxidizing reagents required the addition of $BF_3 \cdot Et_2O$. The results of these reactions are summarized in Table 1.

Clearly the use of PIFA and $BF_3 \cdot Et_2O$ in MeCN solution at rt for 10 min (Table 1, entry 7) gave the best overall performance in terms of the yield (85%) of **2** and reaction time. This procedure was far more convenient than the one using

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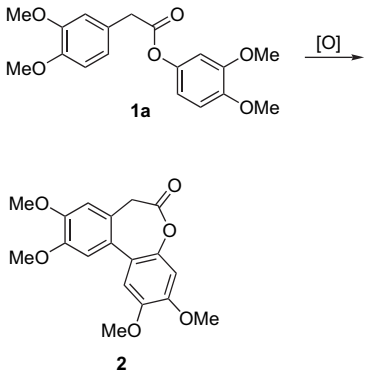


Scheme 2.

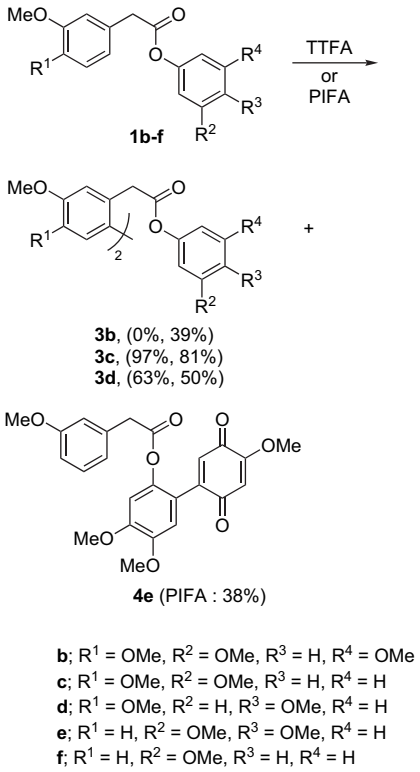
Table 1

Entry	Oxidant	Conditions	Time	Yield of **2** (%)
1	$FeCl_3/SiO_2$	CH_2Cl_2, rt	6 h	0
2	$MoCl_5$	CH_2Cl_2, 0 °C to rt	2 h	26
3	VOF_3	TFA, TFAA, CH_2Cl_2, EtOAc, 0 °C	1 h	60
4	TTFA[a]	TFAA, 0 °C	1 h	60
5	TTFA	MeCN, $BF_3{\cdot}Et_2O$, 0 °C	1 h	74
6	$Ce(OH)_4$	CH_2Cl_2, TFA, $BF_3{\cdot}Et_2O$, 0 °C	6 h	86
7	PIFA[b]	MeCN, $BF_3{\cdot}Et_2O$, rt	10 min	85

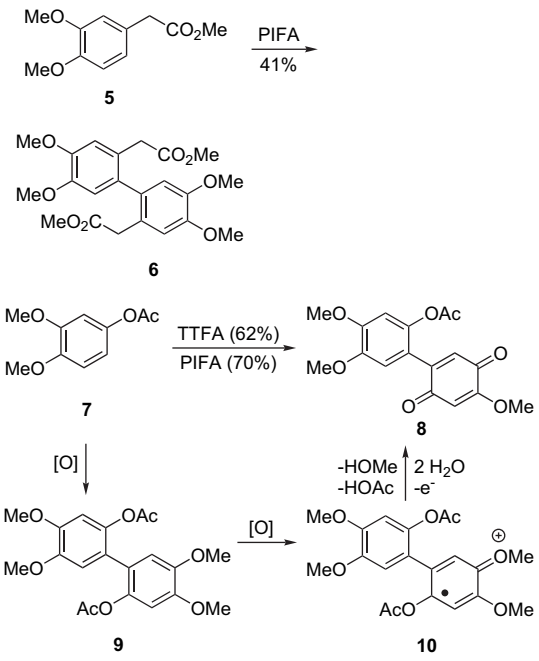
[a] TTFA is thallium(III) trifluoroacetate.
[b] PIFA is phenyl iodine(III) bis(trifluoroacetate).

hygroscopic $Ce(OH)_4$, even though this reagent gave a slightly higher yield (86%) of **2** (Table 1, entry 6). Thus the PIFA method and that using TTFA (Table 1, entry 5) were employed in subsequent oxidative coupling reactions.

The results of the oxidative couplings of the substituted phenyl phenylacetates **1b–f** with TTFA or PIFA, under the conditions shown in Table 1, are summarized in Scheme 3 (the yields in brackets refer to the reactions with TTFA and PIFA, respectively). Unlike the reactions of **1a**, none of these substrates gave the desired intramolecular cyclization product (**B**, m=1, n=0). Esters **1b–d** (R^1=OMe) gave the biphenyl products **3b–d**, through intermolecular coupling of the phenylacetate rings of **1b–d**, while the ester **1e** (R^1=H) gave the biphenyl **4e**, formed via the intermolecular coupling of the more electron rich dimethoxy-substituted phenoxy ring, followed by further oxidation of the biphenyl ring system and then hydrolysis to give a *para*-quinone (see Scheme 4 for more mechanistic details).[35] The ester **1f**, having only one methoxy group on each aromatic ring, was unreactive to the PIFA oxidative conditions. The structure of **3c** was confirmed by a single crystal X-ray study (Fig. 1; CCDC 647893). The simpler esters **5** and **7**, representing the phenylacetate and phenoxy ring moieties of **1a–d** and **1e**, respectively, gave the related biphenyl product **6** and the *para*-quinone **8**, respectively, upon treatment with TTFA or PIFA (Scheme 4). The structure of **8** was confirmed by a single crystal X-ray study (Fig. 2; CCDC 647894). These results indicated that both aromatic rings of **1a** are readily oxidized. This was further supported by the measurements of the oxidation potentials (E^0) of compounds **5** and **7** (see Supplementary data for details), which were 1.40 V and 1.41 V, respectively.



Scheme 3. The yields in brackets refer to the reactions with TTFA and PIFA, respectively.



Scheme 4.

Notably, **1b**, the isomeric 3,5-dimethoxyphenyl ester of **1a** failed to provide the corresponding cyclization product analogous to **2**, even though the phenoxy ring of **1b** had the same number of activating methoxy groups as **1a**. Two possible mechanisms for the formation of **2** from **1a** are shown in Scheme 5. In the first mechanism, the radical cation intermediate **Ca** undergoes intramolecular electrophilic attack by

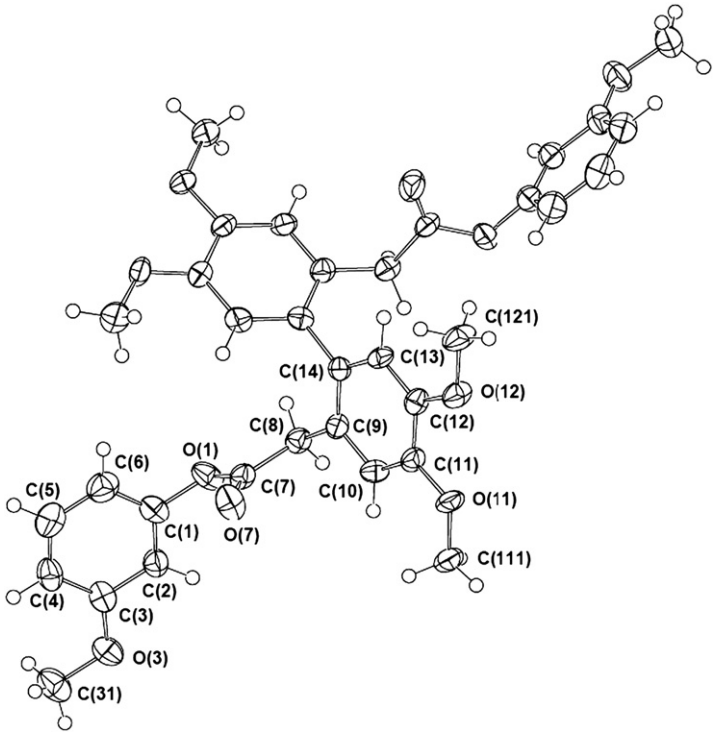


Figure 1. Molecular projection of **3c** (50% probability displacement amplitude ellipsoids for non-hydrogen atoms, hydrogen atoms having arbitrary radii of 0.1 Å here and in Figs. 2 and 3).

the 3,4-dimethoxyphenyloxyacyl ring, *para* to the activating methoxy group, which also stabilizes intermediate **Da**. While such stabilization is also possible in intermediate **Db**, derived from cyclization of **1b**, this intermediate would be destabilized relative to **Da** due to an unfavourable steric interaction between X (X=OMe) and the CH of the adjoining six-membered ring (Scheme 5). An alternative mechanism involving the intermediate **E** followed by a dienone-phenol-like rearrangement is possible for **Ea** but not for **Eb** in which Y=H because of the relatively poorer stabilization of the intermediate cation.[33]

An alternative path, involving oxidation of the phenoxy ring of **1a** first, is also possible. Cyclization would lead to an intermediate related to **Da** in which the two six-membered rings had the reverse electronic nature.

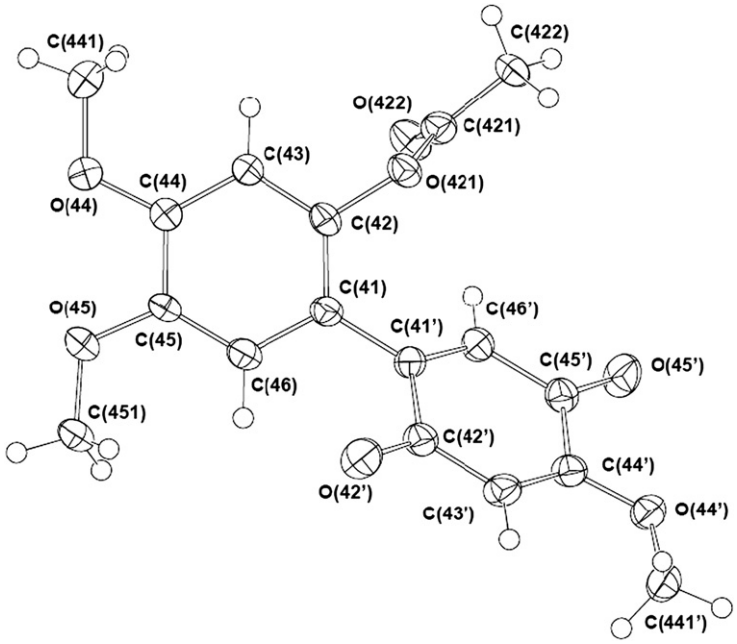
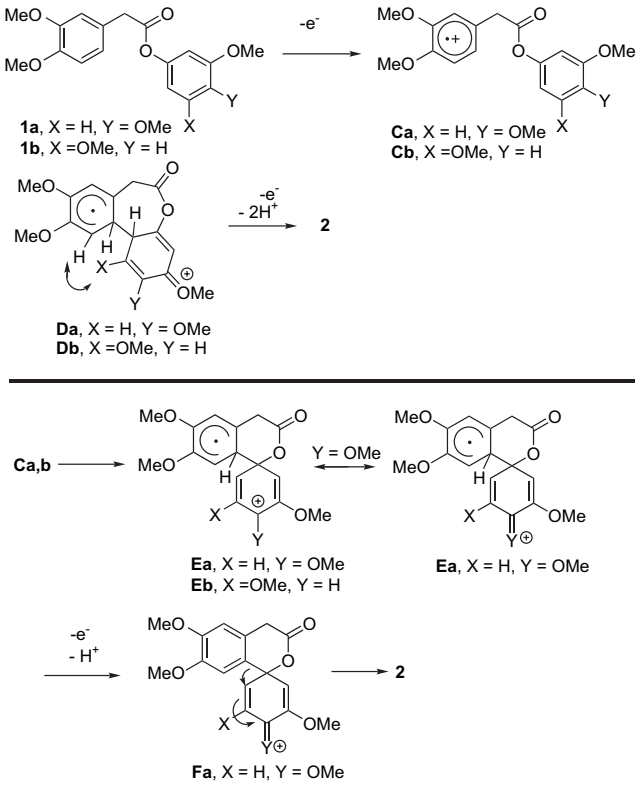


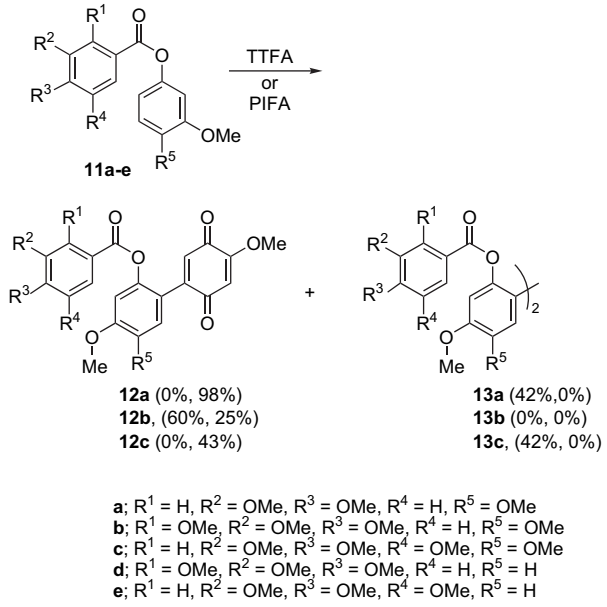
Figure 2. Molecular projection of **8** (molecule 1: there are four molecules in the asymmetric unit, molecules 3 and 4 differing from molecules 1 and 2 by rotation of ca. 180° about the pendant acetate bond).



Scheme 5.

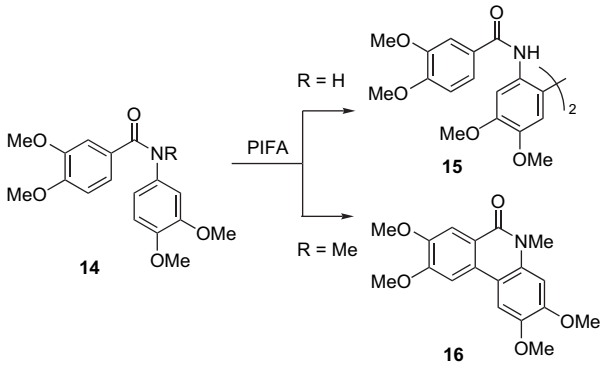
2.2. Oxidative coupling reactions of substituted phenyl benzoates

Treatment of the substituted phenyl benzoates **11a–c** (R^5=OMe) with PIFA under similar reaction conditions as applied to **1a–f** provided the *para*-quinones **12a–c** in variable yields (Scheme 6). Treatment of **11b** with TTFA also provided the quinone **12b**, whereas the esters **11a** and **11c** gave the corresponding biphenyls **13a** and **13c**, respectively (Scheme 6). These products were a result of the initial oxidation and dimerization of the more electron rich 3,4-dimethoxyphenoxy ring. In contrast the esters **11d** and **11e** (R^5=H) having only one methoxy group of the phenoxy ring, but in the case of **11d** three methoxy groups on the benzoate ring, gave no isolable oxidation products and in each case the starting ester was recovered (24–57%). These latter results indicated that both the trimethoxybenzoate and the 3-methoxyphenoxy rings in **11d** and **11e** were too deactivated (the former by the carboxylate group) to undergo smooth oxidation.



Scheme 6. The yields in brackets refer to the reactions with TTFA and PIFA, respectively.

The analogous tetramethoxy substituted benzamide **14** (R=H) to **11a** was reported to give the corresponding biphenyl **15** (R=H) through dimerization of the more electron rich aniline ring, while its *N*-methyl derivative **14** (R=Me) gave the cyclization product **16** (Scheme 7).[31] The unsuccessful cyclization of **14** (R=H) has been attributed to the inaccessibility of the *s*-cis (*E*) amide isomer that is required for cyclization. While oxidative cyclization of the esters **1** and **11** would also require them to adopt the energetically less favourable *s*-cis (*E*) isomer, this isomer is more readily accessible in the case of esters (*E–Z* energy difference 18–22 kcal mol^{-1} for amides and 5–6 kcal mol^{-1} for esters)[36] but may also be a contributing factor in the lack of cyclization products being produced from oxidation of these ester substrates. This effect would be more pronounced in the phenyl benzoate ester **11a** when compared to **1a**, since the *s*-cis (*E*) isomer would be of higher energy due to the closer proximity of the two phenyl groups.

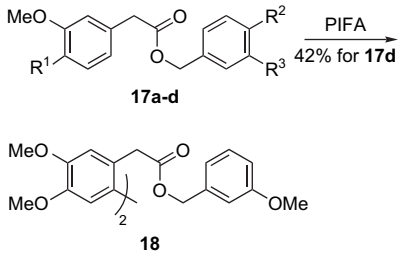


Scheme 7.

2.3. Oxidative coupling reactions of substituted benzyl phenylacetates

Not surprisingly, exposure of the methoxy substituted benzyl phenylacetates **17a–c** to the above oxidative conditions resulted in oxidative cleavage of the *O*-benzyl group and formation of 3,4-dimethoxy- or 4-methoxybenzaldehyde (Scheme 8). None of the desired cyclization products or the corresponding biphenyls could be detected from analysis of the crude reaction mixtures. In contrast, the 3-methoxybenzyl ester **17d** was less prone to oxidative cleavage and gave the biphenyl **18** in 45% yield using PIFA. Oxidative coupling of **17d** had occurred through the more electron rich 3,4-dimethoxyphenylacetate aromatic ring. The structure

of **18** was confirmed by a single crystal X-ray analysis (Fig. 3; CCDC 647895).



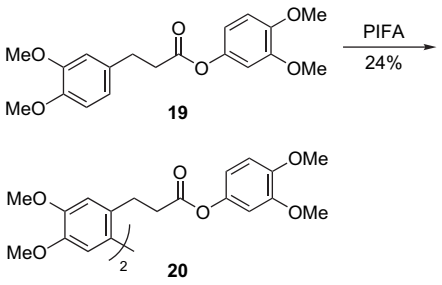
a; R^1 = OMe, R^2 = OMe, R^3 = OMe
b; R^1 = OMe, R^2 = OMe, R^3 = H
c; R^1 = H, R^2 = H, R^3 = OMe
d; R^1 = OMe, R^2 = H, R^3 = OMe

Scheme 8.

The dihydrocinnamate ester **19**, the homologue of **1a**, underwent intermolecular oxidative coupling through the cinnamate aryl ring to give the biphenyl **20**. None of the desired cyclized product was formed, although desired cyclized product would require cyclization through an unfavourable, eight-membered ring transition state or a less likely seven-membered spiro-intermediate, a homologue of intermediate **E** (Scheme 5). Notably, intramolecular biaryl couplings to form eight-membered heterocyclic rings have been reported using $–CH_2N(TFA)CH_2CH_2–$ as a tether on substrates that have the same or less number of methoxy groups as **19** (Scheme 9).[33]

3. Conclusion

In conclusion, the oxidative cyclization of the ester **1a**, through intramolecular biphenyl bond formation, was successful and the target seven-membered lactone **2** was obtained in good yield (85–86%). All other ester substrates gave biphenyl products or their further oxidized products via intermolecular coupling of their radical cation intermediate with the neutral substrate. It appears that matching of the oxidation potentials and nucleophilicity of the two phenyl rings, the positioning of the ring substituents and the ease of *E* to *Z* isomerization about the ester C–O bond are important factors contributing to these product outcomes.



Scheme 9.

4. Experimental

4.1. General

PS refers to the fraction of petroleum spirit with a boiling point of 40–60 °C. DCM refers to dichloromethane. All ^{1}H NMR spectra were measured at 300 MHz and all ^{13}C NMR (DEPT) spectra at 75 MHz in $CDCl_3$ solution, unless otherwise noted. All spectra were referenced to $CDCl_3$ (^{1}H NMR δ 7.26 ppm and ^{13}C NMR δ 77.00 ppm). ^{1}H NMR assignments were achieved with the aid of gCOSY, and in some cases NOESY and TOCSY experiments. ^{13}C NMR assignments were based upon DEPT, gHSQC and gHMBC experiments. In the NMR assignments Q refers to quinone NMR signals.

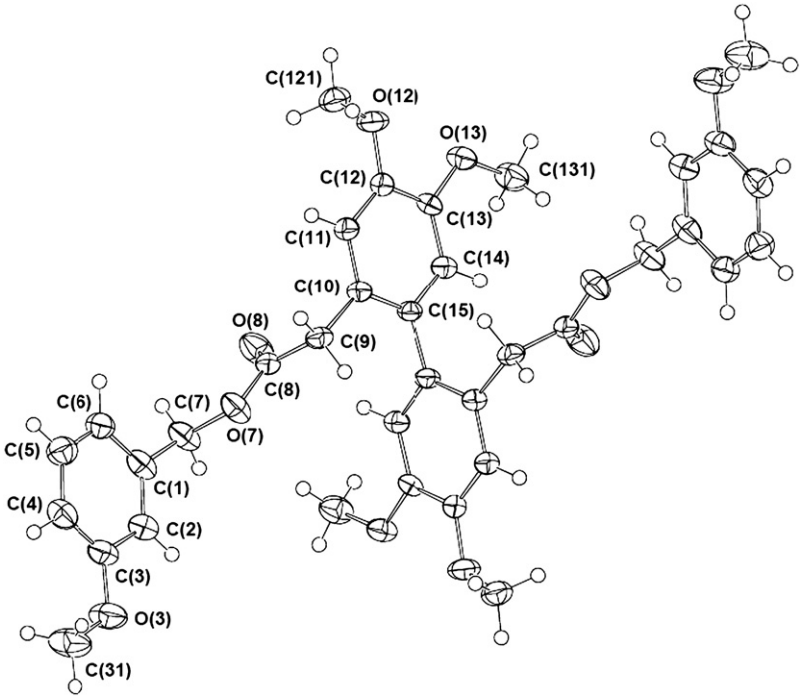


Figure 3. Molecular projection of **18**.

4.2. General methods for ester formation (see Supplementary data for data and procedures for other esters)

4.2.1. 3,4-Dimethoxyphenyl 3,4-dimethoxyphenylacetate 1a. To a stirred solution of 3,4-dimethoxyphenyl acetic acid (500 mg, 2.54 mmol), DCC (578 mg, 2.80 mmol) and DMAP (77 mg, 0.637 mmol) in dry DCM (10 mL) was added a solution of 3,4-dimethoxyphenol (373 mg, 2.42 mmol) in dry DCM (2 mL). The reaction was stirred at rt for 18 h under N_2, diluted with DCM (20 mL), filtered and the filtrate was washed with water (20 mL) and satd $NaHCO_3$ solution (20 mL). The organic phase was dried over $MgSO_4$, filtered, evaporated and the residue was chromatographed, using EtOAc/PS (1:1) as the mobile phase, to yield the title compound as a white solid (727 mg, 90%). Spectral data were consistent with that reported in the literature.[37] Mp 110–112 °C (lit.[37] mp 109–110 °C).

4.3. General methods for oxidative couplings

4.3.1. Method A—hypervalent iodine (PIFA).

4.3.1.1. 2,3,9,10-Tetramethoxydibenzo[*b*,*d*]oxepin-6-(7*H*)-one 2. To a solution of **1a** (61 mg, 0.18 mmol) and PIFA (82 mg, 0.19 mmol) in dry MeCN (2 mL) at 0 °C under N_2 was added $BF_3 \cdot Et_2O$ (100 μL). After 10 min the mixture was diluted with water (15 mL) and extracted with DCM (2×20 mL). The extracts were combined, washed with satd aqueous $NaHCO_3$ (20 mL), dried over $MgSO_4$, filtered and evaporated. Purification by flash silica gel chromatography using EtOAc/PS (1:1) as the eluent yielded the title compound as a white solid (52 mg, 85%). This compound was also prepared by oxidative coupling methods B, C, D and E.

4.3.2. Method B—thallium(III) trifluoroacetate (TTFA). To a solution of **1a** (100 mg, 0.30 mmol) and TTFA (163 mg, 0.30 mmol) in dry MeCN (4 mL) at 0 °C under N_2 was added $BF_3 \cdot Et_2O$ (200 μL). After 1 h the mixture was diluted with water (15 mL) and extracted with DCM (2×20 mL). The extracts were combined, washed with satd aqueous $NaHCO_3$ (20 mL), dried over $MgSO_4$, filtered and evaporated. Purification by flash silica gel chromatography using EtOAc/PS (1:1) as the eluent yielded the title compound as a white solid (74 mg, 74%).

4.3.3. Method C—$MoCl_5$. Compound **1a** (50 mg, 0.15 mmol) was dissolved in dry DCM (2 mL) and was stirred with powdered molecular sieves (4 Å, 100 mg) for 30 min, then the mixture was cooled to 0 °C. $MoCl_5$ (90 mg, 0.33 mmol) was added to the reaction mixture and stirring was continued at 0 °C for 2 h after which the mixture was diluted with water (15 mL) and extracted with DCM (2×20 mL). The extracts were combined, washed with satd aqueous $NaHCO_3$ (20 mL), dried over $MgSO_4$, filtered and evaporated. Purification by flash silica gel chromatography using EtOAc/PS (1:1) as the eluent yielded the title compound as a white solid (13 mg, 26%).

4.3.4. Method D—$Ce(OH)_4$. To a solution of **1a** (50 mg, 0.15 mmol), $Ce(OH)_4$ (156 mg, 0.75 mmol), TFA (2 mL) and trifluoroacetic anhydride (0.4 mL) in dry DCM (7 mL) at 0 °C under N_2 was added $BF_3 \cdot Et_2O$ (38 μL). The ice bath was removed and the reaction mixture was warmed to rt over 6 h. The reaction was quenched with water (15 mL) and extracted with DCM (2×20 mL). The combined extracts were washed with satd aqueous $NaHCO_3$ (20 mL), dried over $MgSO_4$, filtered and evaporated. Purification by flash silica gel chromatography using EtOAc/PS (1:1) as the eluent yielded the title compound as a white solid (43 mg, 86%).

4.3.5. Method E—VOF_3. To a solution of TFA/trifluoroacetic anhydride (20:1, 1.5 mL) in dry EtOAc (1.5 mL) at 0 °C was added VOF_3 (47 mg, 0.37 mmol), followed by a solution of **1a** (50 mg, 0.15 mmol) in dry DCM (3 mL). The ice bath was removed and the reaction mixture was warmed to rt over 3 h. The reaction was quenched with water (15 mL) and extracted with DCM (2×20 mL). The extracts were combined, washed with satd aqueous $NaHCO_3$ (20 mL), dried over $MgSO_4$, filtered and evaporated. Purification by flash silica gel chromatography using EtOAc/PS (1:1) as the eluent yielded the title compound as a white solid (30 mg, 60%). Starting material (14 mg, 28%) was also obtained from the column. Mp 176–178 °C. ^{1}H NMR: δ 7.02 (s, 1H, Ar–*H*-11), 6.94 (s, 1H, Ar–*H*-1), 6.82 (s, 1H, Ar–*H*-8), 6.76 (s, 1H, Ar–*H*-4), 3.92 (s, 6H, OCH_3-2, 3), 3.88 (s, 3H, OCH_3-10), 3.87 (s, 3H, OCH_3-9), 3.51 (ABq, 2H, J=12.6 Hz, Ar–CH_2). ^{13}C NMR: δ 169.4 (*C*=O), 149.6 (Ar–*C*–OCH$_3$-9), 149.3 (Ar–*C*–OCH$_3$-2), 149.2 (Ar–*C*–OCH$_3$-3), 146.5 (Ar–*C*–OCH$_3$-10), 143.5 (Ar–*C*-4a), 127.4 (Ar–*C*-11a), 123.1 (Ar–*C*-8a), 121.2 (Ar–*C*-1a), 111.1 (Ar–*C*–H-8), 110.9 (Ar–*C*–H-1), 110.2 (Ar–*C*–H-11), 104.3 (Ar–*C*–H-4), 56.4 (Ar–O*C*H$_3$-2), 56.2 (Ar–O*C*H$_3$-3), 56.1 (Ar–O*C*H$_3$-10), 56.0 (Ar–O*C*H$_3$-9), 39.6 (Ar–*C*H$_2$). MS (CI$^+$): *m*/*z* 331 (M+1, 100%). HRMS (EI$^+$) calcd for $C_{18}H_{18}O_6$: 330.1103 (M$^+$), found: 330.1102.

4.3.6. (Di-3,5-dimethoxyphenyl)-2,2′-(4,4′,5,5′-tetramethoxybiphenyl-2,2′-diyl)diacetate 3b. The title compound was prepared in 39% yield (white solid, 37 mg) from **1b** (100 mg, 0.30 mmol) in the presence of PIFA (129 mg, 0.30 mmol), $BF_3 \cdot Et_2O$ (100 μL) and MeCN (3 mL) according to oxidative coupling method A. Mp 114–116 °C. ^{1}H NMR: δ 6.97 (s, 2H, Ar–*H*-6), 6.79 (s, 2H, Ar–*H*-3), 6.29 (t, 2H, J=2.2 Hz, Ar–*H*-4′), 6.13 (d, 4H, J=2.1 Hz, Ar–*H*-2′, 6′), 3.94 (s, 6H, OCH_3), 3.81 (s, 6H, OCH_3), 3.71 (s, 12H, 4×OCH_3), 3.61 (ABq, 4H, J=15.0 Hz, Ar–CH_2). ^{13}C NMR: δ 170.2 (*C*=O), 161.0 (2×Ar–*C*–OCH$_3$-3′, 5′), 152.1 (Ar–*C*-1′), 148.4 (Ar–*C*–OCH$_3$-4), 147.8 (Ar–*C*–OCH$_3$-5), 133.0 (Ar–*C*-1), 124.3 (Ar–*C*-2), 113.4 (Ar–*C*–H-3), 112.7 (Ar–*C*–H-6), 99.9 (2×Ar–*C*–H-2′, 6′), 98.1 (Ar–*C*–H-4′), 55.98 (Ar–O*C*H$_3$), 55.92 (Ar–O*C*H$_3$), 55.37 (2×Ar–O*C*H$_3$), 38.3 (Ar–*C*H$_2$). MS (ES$^+$): *m*/*z* 663 (M+H, 100%). HRMS (ES$^+$) calcd for $C_{36}H_{39}O_{12}$: 663.2442 (M+H$^+$), found: 663.2438.

4.3.7. Di-(3-methoxyphenyl)-2,2′-(4,4′,5,5′-tetramethoxybiphenyl-2,2′-diyl)diacetate 3c. The title compound was prepared in 81% yield (pale yellow solid, 107 mg) from **1c** (150 mg, 0.49 mmol) in the presence of PIFA (227 mg, 0.53 mmol), $BF_3 \cdot Et_2O$ (130 μL) and MeCN (5 mL) according to oxidative coupling method A. The title compound was prepared in 97% yield (yellow film, 116 mg) from **1c** (117 mg, 0.38 mmol) in the presence of TTFA (210 mg, 0.38 mmol), $BF_3 \cdot Et_2O$ (2000 μL) and MeCN (4 mL)

according to oxidative coupling method B. Mp 122–124 °C. ^{1}H NMR: δ 7.20 (t, 2H, J=8.1 Hz, Ar–*H*-5′), 6.97 (s, 2H, Ar–*H*-6), 6.79 (s, 2H, Ar–*H*-3), 6.74 (dd, 2H, J=8.1, 2.1 Hz, Ar–*H*-6′), 6.54 (dd, 2H, J=8.1, 2.1 Hz, Ar–*H*-4′), 6.50 (t, 2H, J=2.1 Hz, Ar–*H*-2′), 3.94 (s, 6H, OCH_3-4), 3.79 (s, 6H, OCH_3-3), 3.73 (s, 6H, OCH_3-3′), 3.62 (ABq, 4H, J=16.2 Hz, Ar–CH_2). ^{13}C NMR: δ 170.3 (*C*=O), 160.3 (Ar–*C*–OCH$_3$-3′), 151.5 (Ar–*C*-1′), 148.4 (Ar–*C*–OCH$_3$-3), 147.7 (Ar–*C*–OCH$_3$-4), 132.9 (Ar–*C*-1), 129.6 (Ar–*C*–H-5′), 124.3 (Ar–*C*-2), 113.5 (Ar–*C*–H-5), 113.4 (Ar–*C*–H-4′), 112.7 (Ar–*C*–H-2), 111.5 (Ar–*C*–H-6′), 107.4 (Ar–*C*–H-2′), 55.9 (Ar–O*C*H$_3$-4), 55.8 (Ar–O*C*H$_3$-3), 55.2 (Ar–O*C*H$_3$-3′), 38.3 (Ar–*C*H$_2$–CO). MS (EI$^+$): *m*/*z* 602 (M$^+$, 10%), 299 (100%). HRMS (EI$^+$) calcd for $C_{34}H_{34}O_{10}$: 602.2151 (M$^+$), found: 602.2160.

4.3.8. Di-(4-methoxyphenyl)-2,2′-(4,4′,5,5′-tetramethoxybiphenyl-2,2′-diyl)diacetate 3d. The title compound was prepared in 50% yield (white solid, 49.5 mg) from **1d** (100 mg, 0.28 mmol) in the presence of PIFA (149 mg, 0.34 mmol), $BF_3\cdot Et_2O$ (83 μL) and MeCN (5 mL) according to oxidative coupling method A. The title compound was also prepared in 63% yield (clear film, 49.5 mg) from **1d** (50 mg, 0.14 mmol) in the presence of TTFA (44 mg, 0.082 mmol), $BF_3\cdot Et_2O$ (100 μL) and MeCN (1 mL) according to oxidative coupling method B. Mp 102–104 °C. ^{1}H NMR: δ 6.97 (s, 2H, Ar–*H*-6), 6.86 (d, 4H, J=9.0 Hz, Ar–*H*-2′, 6′), 6.82 (d, 4H, J=9.0 Hz, Ar–*H*-3′, 5′), 6.79 (s, 2H, Ar–*H*-3), 3.95 (s, 6H, OCH_3), 3.79 (s, 6H, OCH_3), 3.77 (s, 6H, OCH_3), 3.59 (ABq, 4H, J=16.2 Hz, Ar–CH_2). ^{13}C NMR: δ 170.8 (*C*=O), 157.1 (Ar–*C*–OCH$_3$-4′), 148.3 (Ar–*C*–OCH$_3$-5), 147.6 (Ar–*C*–OCH$_3$-4), 144.0 (Ar–*C*–1′), 132.9 (Ar–*C*-1), 124.3 (Ar–*C*-2), 122.1 (2×Ar–*C*–H-2′, 6′), 114.2 (2×Ar–*C*–H-3′, 5′), 113.3 (Ar–*C*–H-3), 112.6 (Ar–*C*–H-6), 55.9 (Ar–O*C*H$_3$), 55.8 (Ar–O*C*H$_3$), 55.4 (Ar–O*C*H$_3$), 38.2 (Ar–*C*H$_2$–CO). MS (EI$^+$): *m*/*z* 602 (M$^+$, 2%). HRMS (EI$^+$) calcd for $C_{34}H_{34}O_{10}$: 602.2151 (M$^+$), found: 602.2182.

4.3.9. 4,5-Dimethoxy-2-(4-methoxy-3,6-dioxocyclohexa-1,4-dien-1-yl)phenyl (3-methoxyphenyl)acetate 4e. The title compound was prepared in 38% yield (pale film, 34 mg) from 3,4-dimethoxyphenyl (3-methoxyphenyl)acetate **1e** (123 mg, 0.41 mmol) in the presence of PIFA (184 mg, 0.43 mmol), $BF_3\cdot Et_2O$ (104 μL) and MeCN (5 mL) according to oxidative coupling method A. ^{1}H NMR: δ 7.16 (t, 1H, J=7.8 Hz, Ar–*H*-5), 6.82 (dd, 1H, J=7.8, 2.7 Hz, Ar–*H*-6), 6.79 (d, 1H, J=2.1 Hz, Ar–*H*-2), 6.76 (dd, 1H, J=7.8, 2.7 Hz, Ar–*H*-4), 6.71 (s, 1H, Ar–*H*-6′), 6.65 (s, 1H, Ar–*H*-3′), 6.52 (s, 1H, Q–*H*-b), 5.78 (s, 1H, Q–*H*-e), 3.87 (s, 3H, OCH_3-5′), 3.83 (s, 3H, OCH_3-4′), 3.81 (s, 3H, OCH_3-3), 3.77 (s, 3H, Q–OCH_3), 3.66 (s, 2H, Ar–CH_2). ^{13}C NMR: δ 185.5 (Q–*C*=O-c), 181.4 (Q–*C*=O-f), 169.3 (*C*=O), 159.6 (Ar–*C*–OCH$_3$-3), 158.0 (Q–*C*–OCH$_3$-d), 150.5 (Ar–*C*–OCH$_3$-4′), 146.7 (Ar–*C*–OCH$_3$-5′), 144.0 (Ar–*C*-1′), 141.8 (Q–*C*-a), 134.2 (Ar–*C*-1), 132.4 (Q–*C*–H-b), 129.7 (Ar–*C*–H-5), 121.5 (Ar–*C*–H-6), 117.4 (Ar–*C*-2′), 114.5 (Ar–*C*–H-2), 113.1 (Ar–*C*–H-4), 112.5 (Ar–*C*–H-3′), 107.5 (Q–*C*–H-e), 106.4 (Ar–*C*–H-6′), 56.2 (Ar–O*C*H$_3$-4′), 56.1 (Ar–O*C*H$_3$-5′), 56.0 (Ar–O*C*H$_3$-3), 55.1 (Q–O*C*H$_3$), 41.3 (Ar–*C*H$_2$). MS (EI$^+$): *m*/*z* 438 (M$^+$, 16%), 292 (100%). HRMS (EI$^+$) calcd for $C_{24}H_{22}O_8$: 438.1314 (M$^+$), found: 438.1331.

4.3.10. Dimethyl 2,2′-(4,4′,5,5′-tetramethoxybiphenyl-2,2′-diyl)diacetate 6.[38] The title compound was prepared in 41% yield (clear crystals, 53 mg) from **5** (129 mg, 0.62 mmol) in the presence of PIFA (250 mg, 0.58 mmol), $BF_3\cdot Et_2O$ (150 μL) and MeCN (10 mL) according to oxidative coupling method A. Mp 142–144 °C (lit.[38] mp 145 °C). ^{1}H NMR: δ 6.84 (s, 2H, Ar–*H*-6), 6.72 (s, 2H, Ar–*H*-3), 3.92 (s, 6H, OCH_3-5), 3.83 (s, 6H, OCH_3-4), 3.60 (s, 6H, CO_2CH_3), 3.35 (ABq, 4H, J=16.5 Hz, Ar–CH_2). ^{13}C NMR: δ 172.4 (*C*=O), 148.1 (Ar–*C*–OCH$_3$-4), 147.4 (Ar–*C*–OCH$_3$-5), 132.8 (Ar–*C*-1), 124.6 (Ar–*C*-2), 113.2 (Ar–*C*–H-3), 112.5 (Ar–*C*–H-6), 55.8 (Ar–O*C*H$_3$), 55.7 (Ar–O*C*H$_3$), 51.8 (CO_2CH_3), 37.9 (Ar–*C*H$_2$). MS (CI$^+$): *m*/*z* 419 (M+H, 100%). HRMS (EI$^+$) calcd for $C_{22}H_{26}O_8$: 418.1627 (M$^+$), found: 418.1615.

4.3.11. 4,5-Dimethoxy-2-(4-methoxy-3,6-dioxocyclohexa-1,4-dien-1-yl)phenyl acetate 8. The title compound was prepared in 70% yield (cloudy film, 49 mg) from **7** (100 mg, 0.51 mmol) in the presence of PIFA (227 mg, 0.53 mmol), $BF_3\cdot Et_2O$ (130 μL) and MeCN (5 mL) according to oxidative coupling method A. The title compound was also prepared in 62% yield (clear film, 44 mg) from 3,4-dimethoxyphenyl-acetate (100 mg, 0.51 mmol) in the presence of TTFA (44 mg, 0.082 mmol), $BF_3\cdot Et_2O$ (100 μL) and MeCN (1 mL) according to oxidative coupling method B. ^{1}H NMR: δ 6.75 (s, 1H, Ar–*H*-3), 6.73 (s, 1H, Ar–*H*-6), 6.72 (s, 1H, Q–*H*-b), 6.03 (s, 1H, Q–*H*-e), 3.89 (s, 3H, –OCH_3-5), 3.87 (s, 3H, OCH_3-4), 3.86 (s, 3H, Q–OCH_3), 2.16 (s, 3H, COCH_3). ^{13}C NMR: δ 185.4 (Q–*C*=O-c), 181.8 (Q–*C*=O-f), 168.9 (*C*=O), 158.4 (Q–*C*–OCH$_3$), 150.6 (Ar–*C*–OCH$_3$-5), 146.5 (Ar–*C*–OCH$_3$-4), 144.2 (Ar–*C*-1), 142.1 (Q–*C*-a), 132.2 (Q–*C*–H-b), 117.1 (Ar–*C*-2), 112.8 (Ar–*C*–H-3), 107.7 (Q–*C*–H-e), 106.6 (Ar–*C*–H-6), 56.2 (Ar–O*C*H$_3$-5), 56.1 (Ar–O*C*H$_3$-4), 56.0 (Q–O*C*H$_3$), 20.8 (CO*C*H$_3$). MS (EI$^+$): *m*/*z* 332 (M$^+$, 16%), 292 (100%). HRMS (EI$^+$) calcd for $C_{17}H_{16}O_7$: 332.0896 (M$^+$), found: 332.0896.

4.3.12. 4,5-Dimethoxy-2-(4-methoxy-3,6-dioxocyclohexa-1,4-dien-1-yl)phenyl 3,4-dimethoxybenzoate 12a. The title compound was prepared in 98% yield (red crystals, 55 mg) from **11a** (79 mg, 0.25 mmol) in the presence of PIFA (112 mg, 0.26 mmol), $BF_3\cdot Et_2O$ (63 μL) and DCM (5 mL) according to oxidative coupling method A. Mp 196–198 °C. ^{1}H NMR: δ 7.71 (dd, 1H, J=8.7, 2.1 Hz, Ar–*H*-6), 7.54 (d, 1H, J=2.1 Hz, Ar–*H*-2), 6.91 (d, 1H, J=8.7 Hz, Ar–*H*-5), 6.84 (s, 1H, Ar–*H*-6′), 6.81 (s, 1H, Ar–*H*-3′), 6.79 (s, 1H, Q–*H*-b), 5.94 (s, 1H, Q–*H*-e), 3.95 (s, 3H, OCH_3), 3.93 (s, 3H, OCH_3), 3.91 (s, 3H, OCH_3), 3.90 (s, 3H, OCH_3), 3.79 (s, 3H, Q–OCH_3). ^{13}C NMR: δ 185.5 (Q–*C*=O-c), 181.7 (Q–*C*=O-f), 164.4 (*C*=O), 158.3 (Q–*C*–OCH$_3$), 153.6 (Ar–*C*–OCH$_3$-3), 150.7 (Ar–*C*–OCH$_3$-4′), 148.7 (Ar–*C*–OCH$_3$-4), 146.6 (Ar–*C*–OCH$_3$-5′), 144.3 (Ar–*C*-1′), 142.5 (Q–*C*-a), 132.5 (Q–*C*–H-b), 124.3 (Ar–*C*–H-6), 121.1 (Ar–*C*-1), 117.4 (Ar–*C*-2′), 112.8 (Ar–*C*–H-3′), 112.2 (Ar–*C*–H-2), 110.4 (Ar–*C*–H-5), 107.7 (Q–*C*–H-e), 106.7 (Ar–*C*–H-6′), 56.3 (Ar–O*C*H$_3$), 56.2 (Ar–O*C*H$_3$), 56.1 (Ar–O*C*H$_3$), 56.0 (Ar–O*C*H$_3$), 55.9 (Q–O*C*H$_3$). MS (EI$^+$): *m*/*z* 454 (M$^+$, 2%). HRMS (EI$^+$) calcd for $C_{24}H_{22}O_9$: 454.1263 (M$^+$), found: 454.1256.

4.3.13. 4,5-Dimethoxy-2-(4-methoxy-3,6-dioxocyclohexa-1,4-dien-1-yl)phenyl 2,3,4-trimethoxybenzoate 12b. The

title compound was prepared in 25% yield (white film, 17 mg) from **11b** (100 mg, 0.28 mmol) in the presence of PIFA (132 mg, 0.35 mmol), $BF_3 \cdot Et_2O$ (73 μL) and MeCN (4 mL) according to method A. The title compound was also prepared in 60% yield (42 mg, 97% br sm) from **11b** (100 mg, 0.28 mmol) in the presence of TTFA (93 mg, 0.17 mmol), $BF_3 \cdot Et_2O$ (73 μL) and MeCN (4 mL) according to oxidative coupling method B. ^{1}H NMR: δ 7.63 (d, 1H, J=8.7 Hz, Ar–*H*-6), 6.83 (s, 1H, Ar–*H*-6′), 6.79 (s, 1H, Ar–*H*-3′), 6.78 (s, 1H, Q–*H*-b), 6.70 (d, 1H, J=8.7 Hz, Ar–*H*-5), 5.95 (s, 1H, Q–*H*-e), 3.91 (s, 3H, OCH_3-3), 3.90 (s, 3H, OCH_3-5′), 3.89 (s, 3H, OCH_3-2), 3.88 (s, 3H, OCH_3-4), 3.85 (s, 3H, OCH_3-4′), 3.79 (s, 3H, Q–OCH_3). ^{13}C NMR: δ 185.5 (Q–*C*=O-c), 181.8 (Q–*C*=O-f), 163.1 (*C*=O), 158.2 (Q–*C*–OCH$_3$-d), 157.8 (Ar–*C*–OCH$_3$-4), 155.3 (Ar–*C*–OCH$_3$-2), 150.6 (Ar–*C*–OCH$_3$-4′), 146.5 (Ar–*C*–OCH$_3$-5′), 144.2 (Ar–*C*-1′), 142.9 (Q–*C*-a), 142.5 (Ar–*C*–OCH$_3$-3), 132.4 (Q–*C*–H-b), 127.3 (Ar–*C*–H-6), 117.5 (Ar–*C*-2′), 116.1 (Ar–*C*-1), 112.9 (Ar–*C*–H-3′), 107.7 (Q–*C*–H-e), 106.9 (Ar–*C*–H-5), 106.8 (Ar–*C*–H-6′), 61.6 (Ar–O*C*H$_3$-2), 60.8 (Ar–O*C*H$_3$-4), 56.3 (Ar–O*C*H$_3$-4′), 56.2 (Ar–O*C*H$_3$-5′), 56.1 (Ar–O*C*H$_3$-3), 56.0 (Q–O*C*H$_3$). MS (EI$^+$): *m*/*z* 484 (M$^+$, 4%), 195 (100%). HRMS (CI$^+$) calcd for $C_{25}H_{25}O_{10}$: 484.1447 (MH$^+$), found: 484.1459.

4.3.14. 4,5-Dimethoxy-2-(4-methoxy-3,6-dioxocyclohexa-1,4-dien-1-yl)phenyl 3,4,5-trimethoxybenzoate 12c. The title compound was prepared in 43% yield (white solid, 70 mg) from **11c** (100 mg, 0.28 mmol) in the presence of PIFA (132 mg, 0.35 mmol), $BF_3 \cdot Et_2O$ (73 μL) and MeCN (4 mL) according to oxidative coupling method A. ^{1}H NMR: δ 7.29 (s, 2H, Ar–*H*-2, 6), 6.82 (s, 1H, Ar–*H*-6′), 6.80 (s, 1H, Ar–*H*-3′), 6.79 (s, 1H, Q–*H*-b), 5.93 (s, 1H, Q–*H*-e), 3.91 (s, 3H, OCH_3-4), 3.90 (s, 3H, OCH_3-5′), 3.89 (s, 9H, OCH_3-3, 5, 4′), 3.78 (s, 3H, Q–OCH_3). ^{13}C NMR: δ 185.5 (Q–*C*=O-c), 181.7 (Q–*C*=O-f), 164.3 (*C*=O), 158.4 (Q–*C*–OCH$_3$), 153.0 (2×Ar–*C*–OCH$_3$-3, 5), 150.8 (Ar–*C*–OCH$_3$-4′), 146.7 (Ar–*C*–OCH$_3$-5′), 144.3 (Ar–*C*-1′), 142.9 (Ar–*C*–OCH$_3$-4), 142.4 (Q–*C*-a), 132.6 (Q–*C*–H-b), 123.6 (Ar–*C*-1), 117.4 (Ar–*C*-2′), 112.8 (Ar–*C*–H-3′), 107.7 (Q–*C*–H-e), 107.2 (2×Ar–*C*–H-2, 6), 106.7 (Ar–*C*–H-6′), 60.9 (Ar–O*C*H$_3$-4), 56.3 (Ar–O*C*H$_3$-5′), 56.2 (3×Ar–O*C*H$_3$-3, 5, 4′), 56.1 (Q–O*C*H$_3$). MS (EI$^+$): *m*/*z* 484 (M$^+$, 24%). HRMS (EI$^+$) calcd for $C_{25}H_{24}O_{10}$: 484.1369 (M$^+$), found: 484.1367.

4.3.15. 4,4′,5,5′-Tetramethoxybiphenyl-2,2′-diyl di-(3,4-dimethoxydibenzoate) 13a. The title compound was prepared in 42% yield (clear film, 41 mg) from **11a** (100 mg, 0.29 mmol) in the presence of TTFA (86 mg, 0.16 mmol), $BF_3 \cdot Et_2O$ (73 μL) and MeCN (5 mL) according to oxidative coupling method B. ^{1}H NMR: δ 7.65 (dd, 2H, J=8.4, 2.1 Hz, Ar–*H*-6), 7.46 (d, 2H, J=2.1 Hz, Ar–*H*-2), 6.85 (s, 2H, Ar–*H*-3′), 6.84 (d, 2H, J=8.4 Hz, Ar–*H*-5), 6.81 (s, 2H, Ar–*H*-6′), 3.91 (s, 6H, OCH_3-3), 3.85 (s, 6H, OCH_3-5′), 3.84 (s, 6H, OCH_3-4′), 3.73 (s, 6H, OCH_3-4). ^{13}C NMR: δ 165.0 (*C*=O), 153.3 (Ar–*C*–OCH$_3$-3), 148.6 (Ar–*C*–OCH$_3$-4′), 148.5 (Ar–*C*–OCH$_3$-5′), 146.3 (Ar–*C*–OCH$_3$-4), 141.7 (Ar–*C*-2′), 124.1 (Ar–*C*–H-6), 121.6 (Ar–*C*-1, Ar–*C*-1′), 113.0 (Ar–*C*–H-5), 112.1 (Ar–*C*–H-2), 110.2 (Ar–*C*–H-6′), 106.3 (Ar–*C*–H-3′), 55.9 (3×Ar–O*C*H$_3$-3, 4′, 5′), 55.8 (Ar–O*C*H$_3$-4). MS (EI$^+$): *m*/*z* 634 (M$^+$, 9%), 167 (100%). HRMS (EI$^+$) calcd for $C_{34}H_{34}O_{12}$: 634.2050 (M$^+$), found: 634.2047.

4.3.16. 4,4′,5,5′-Tetramethoxy-2,2′-diyl di-(3,4,5-trimethoxydibenzoate) 13c. The title compound was prepared in 42% yield (clear film, 41 mg) from **11c** (100 mg, 0.29 mmol) in the presence of TTFA (86 mg, 0.16 mmol), $BF_3 \cdot Et_2O$ (73 μL) and MeCN (5 mL) according to oxidative coupling method B. ^{1}H NMR: δ 7.22 (s, 4H, Ar–*H*-2, 6), 6.86 (s, 4H, Ar–*H*-3′, 6′), 3.89 (s, 6H, OCH_3-4), 3.86 (s, 6H, OCH_3-5′), 3.81 (s, 12H, OCH_3-3, 5), 3.78 (s, 6H, OCH_3-4′). ^{13}C NMR: δ 164.8 (*C*=O), 152.9 (2×Ar–*C*–OCH$_3$-3, 5), 148.8 (Ar–*C*–OCH$_3$-5′), 146.5 (Ar–*C*–OCH$_3$-4′), 142.6 (Ar–*C*–OCH$_3$-4), 141.8 (Ar–*C*-2′), 124.2 (Ar–*C*-1), 121.6 (Ar–*C*-1′), 113.1 (Ar–*C*–H-6′), 107.2 (2×Ar–*C*–H-2, 6), 106.1 (Ar–*C*–H-3′), 60.9 (Ar–O*C*H$_3$-5′), 56.2 (Ar–O*C*H$_3$-4′), 56.1 (2×Ar–O*C*H$_3$-3, 5), 56.0 (Ar–O*C*H$_3$-4). MS (EI$^+$): *m*/*z* 694 (M$^+$, 18%), 195 (100%). HRMS (EI$^+$) calcd for $C_{36}H_{38}O_{14}$: 694.2262 (M$^+$), found: 694.2282.

4.3.17. [Di-(3-methoxybenzyl)]-(4,4′,5,5′-tetramethoxybiphenyl-2,2′-diyl)diacetate 18. The title compound was prepared in 42% yield (white solid, 41 mg) from **17d** (100 mg, 0.28 mmol) in the presence of PIFA (142 mg, 0.33 mmol), $BF_3 \cdot Et_2O$ (73 μL) and MeCN (4 mL) according to oxidative coupling method A. Mp 102–104 °C. ^{1}H NMR: δ 7.22 (t, 2H, J=7.8 Hz, Ar–*H*-5′), 6.84–6.80 (m, 4H, Ar–*H*-4′, 6′), 6.82 (s, 2H, Ar–*H*-6), 6.76 (t, 2H, J=2.1 Hz, Ar–*H*-2′), 6.70 (s, 2H, Ar–*H*-3), 5.00 (ABq, 4H, J=12.3 Hz, Ar–CH_2–O), 3.86 (s, 6H, OCH_3-4), 3.75 (s, 6H, OCH_3-3′), 3.72 (s, 6H, OCH_3-3), 3.36 (s, 4H, Ar–CH_2–CO). ^{13}C NMR: δ 171.7 (*C*=O), 159.6 (Ar–*C*–OCH$_3$-3′), 148.2 (Ar–*C*–OCH$_3$-4), 147.4 (Ar–*C*–OCH$_3$-3), 137.2 (Ar–*C*-1′), 132.8 (Ar–*C*-1), 129.5 (Ar–*C*–H-5′), 124.6 (Ar–*C*-2), 120.1 (Ar–*C*–H-6′), 113.6 (Ar–*C*–H-4′), 113.4 (Ar–*C*–H-3), 113.2 (Ar–*C*–H-6), 112.5 (Ar–*C*–H-2′), 66.2 (Ar–*C*H$_2$–O), 55.8 (Ar–O*C*H$_3$-4), 55.7 (Ar–O*C*H$_3$-3), 55.1 (Ar–O*C*H$_3$-3′), 38.1 (Ar–*C*H$_2$–CO). MS (EI$^+$): *m*/*z* 630 (M$^+$, 10%), 121 (100%). HRMS (EI$^+$) calcd for $C_{36}H_{38}O_{10}$: 630.2465 (M$^+$), found: 630.2439.

4.3.18. Di-(3,4-dimethoxyphenyl) 3,3′-(4,4′,5′,5′-tetramethoxybiphenyl-2,2′-diyl)dipropanoate 20. The title compound was prepared in 24% yield (yellow film, 24 mg) from **19** (100 mg, 0.28 mmol) in the presence of PIFA (130 mg, 0.30 mmol), $BF_3 \cdot Et_2O$ (100 μL) and MeCN (3 mL) according to oxidative coupling method A. ^{1}H NMR: δ 6.88 (s, 2H, Ar–*H*-6), 6.78 (d, 2H, J=8.7 Hz, Ar–*H*-5′), 6.72 (s, 2H, Ar–*H*-3), 6.49 (dd, 2H, J=8.7, 2.4 Hz, Ar–*H*-6′), 6.45 (d, 2H, J=2.4 Hz, Ar–*H*-2′), 3.91 (s, 6H, OCH_3), 3.85 (s, 6H, OCH_3), 3.84 (s, 6H, OCH_3), 3.80 (s, 6H, OCH_3), 2.93–2.72 (m, 4H, Ar–CH_2), 2.64 (t, 4H, J=7.2 Hz, Ar–CH$_2$–CH_2). ^{13}C NMR: δ 171.6 (*C*=O), 149.2 (Ar–*C*–OCH$_3$-3′), 148.2 (Ar–*C*–OCH$_3$-3), 147.0 (Ar–*C*–OCH$_3$-4), 146.7 (Ar–*C*–OCH$_3$-4′), 144.1 (Ar–*C*-1′), 132.5 (Ar–*C*-1), 130.2 (Ar–*C*-2), 113.4 (Ar–*C*–H-3), 112.6 (Ar–*C*–H-6′), 112.1 (Ar–*C*–H-6), 111.0 (Ar–*C*–H-5′), 105.5 (Ar–*C*–H-2′), 56.0 (Ar–O*C*H$_3$), 55.9 (2×Ar–O*C*H$_3$), 55.8 (Ar–O*C*H$_3$), 35.3 (Ar–CH$_2$–*C*H$_2$), 28.2 (Ar–*C*H$_2$). MS (EI$^+$): *m*/*z* 690 (M$^+$, 14%). HRMS (ESI$^+$) calcd for $C_{38}H_{43}O_{12}$: 691.2755 (MH$^+$), found: 691.2726.

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Supplementary data

Supplementary data associated with this article can be found in the online version, at doi:10.1016/j.tet.2007.08.082.

References and notes

1. Jempty, T. C.; Gogins, K. A. Z.; Mazur, Y.; Miller, L. L. *J. Org. Chem.* **1981**, *46*, 4545–4551.
2. Jempty, T. C.; Miller, L. L.; Mazur, Y. *J. Org. Chem.* **1980**, *45*, 749–751.
3. Waldvogel, S. R. *Synlett* **2002**, 622–624.
4. Kramer, B.; Frohlich, R.; Bergander, K.; Waldvogel, S. R. *Synthesis* **2003**, *1*, 91–96.
5. Kumar, S.; Manickam, M. *Chem. Commun.* **1997**, 1615–1616.
6. Mirk, D.; Wibbeling, B.; Frohlich, R.; Waldvogel, S. R. *Synlett* **2004**, 1970–1974.
7. Waldvogel, S. R.; Aits, E.; Holst, C.; Frohlich, R. *Chem. Commun.* **2002**, 1278–1279.
8. Schwartz, M. A.; Rose, B. F.; Holton, R. A.; Scott, S. W.; Vishnuvajjala, B. *J. Am. Chem. Soc.* **1977**, *99*, 2571–2578.
9. Elliot, I. W. *J. Org. Chem.* **1977**, *42*, 1090–1093.
10. Kupchan, S. M.; Liepa, A. J.; Kameswaran, V.; Bryan, R. F. *J. Am. Chem. Soc.* **1973**, *95*, 6861–6863.
11. Damon, R. E.; Schlessinger, R. H.; Blount, J. F. *J. Org. Chem.* **1976**, *41*, 3772–3773.
12. Kupchan, S. M.; Dhingra, O. P.; Kim, C.-K.; Kameswaran, V. *J. Org. Chem.* **1976**, *41*, 4047–4049.
13. Kupchan, S. M.; Dhingra, O. P.; Kim, C.-K.; Kameswaran, V. *J. Org. Chem.* **1978**, *43*, 2521–2529.
14. Kupchan, S. M.; Kameswaran, V.; Lynn, J. T.; Williams, D. K.; Liepa, A. J. *J. Am. Chem. Soc.* **1975**, *97*, 5622–5623.
15. Morimoto, T.; Chiba, M.; Achiwa, K. *Tetrahedron* **1993**, *49*, 1793–1806.
16. Kende, A. S.; Liebeskind, L. S.; Mills, J. E.; Rutledge, P. S.; Curran, D. P. *J. Am. Chem. Soc.* **1977**, *99*, 7082–7083.
17. Evans, D. A.; Cain, P. A.; Wong, R. Y. *J. Am. Chem. Soc.* **1977**, *99*, 7083–7085.
18. McKillop, A.; Turrell, A. G.; Young, D. W.; Taylor, E. C. *J. Am. Chem. Soc.* **1980**, *102*, 6504–6512.
19. Taylor, E. C.; Andrade, J. G.; Rall, G. J. H.; McKillop, A. *J. Am. Chem. Soc.* **1980**, *102*, 6513–6519.
20. Lau, W.; Kochi, J. K. *J. Am. Chem. Soc.* **1984**, *106*, 7100–7112.
21. Norman, R. O. C.; Thomas, C. B.; Ward, P. J. *J. Chem. Soc., Perkin Trans. 1* **1973**, 2914–2917.
22. Planchenault, D.; Dhal, R.; Robin, J.-P. *Tetrahedron* **1993**, *49*, 5823–5830.
23. Tohma, H.; Morioka, H.; Takizawa, S.; Arisawa, M.; Kita, Y. *Tetrahedron* **2001**, *57*, 345–352.
24. Arisawa, M.; Utsumi, S.; Nakajima, M.; Ramesh, N. G.; Tohma, H.; Kita, Y. *Chem. Commun.* **1999**, 469–470.
25. Kita, Y.; Takada, T.; Gyoten, M.; Tohma, H.; Zenk, M. H.; Eichhorn, J. *J. Org. Chem.* **1996**, *61*, 5857–5864.
26. Hamamoto, H.; Anilkumar, G.; Tohma, H.; Kita, Y. *Chem. Commun.* **2002**, 450–451.
27. Tohma, H.; Kita, Y. *Top. Curr. Chem.* **2003**, *224*, 209–248.
28. Olivera, R.; San Martin, R.; Dominguez, E. *J. Org. Chem.* **2000**, *65*, 7010–7019.
29. Churruca, F.; San Martin, R.; Tellitu, I.; Dominguez, E. *Eur. J. Org. Chem.* **2005**, 2481–2490.
30. Moreno, I.; Tellitu, I.; Dominguez, E.; San Martin, R. *Eur. J. Org. Chem.* **2002**, 2126–2135.
31. Moreno, I.; Tellitu, I.; Etayo, J.; San Martin, R.; Dominguez, E. *Tetrahedron* **2001**, *57*, 5403–5411.
32. Carbocycles have also been generated from bis(3,4-dimethoxyphenyl) alkanes phenyl ethers by electrochemical oxidation, see: Ronlan, A.; Parker, V. D. *J. Am. Chem. Soc.* **1974**, *39*, 1014–1016.
33. Hamamoto, H.; Anilkumar, G.; Tohma, H.; Kita, Y. *Chem.—Eur. J.* **2002**, *8*, 5377–5383.
34. Takada, T.; Arisawa, M.; Gyoten, M.; Hamada, R.; Tohma, H.; Kita, Y. *J. Org. Chem.* **1998**, *63*, 7698–7706.
35. The electrochemical oxidation of di-aryl esters has been shown to give in one case a diphenyl derivative from intermolecular coupling. Further oxidation of this compound gave a *para*-quinone product. Other esters gave oligomeric products, see: Sainsbury, M.; Wyatt, J. *J. Chem. Soc., Perkin Trans. 1* **1979**, 108–114.
36. *Stereochemistry of Organic Compounds*; Eliel, E. L., Wilen, S. H., Eds.; Wiley: New York, NY, 1994; pp 618–620.
37. Elliott, I. W.; Sloan, M. J.; Tate, E. *Tetrahedron* **1996**, *52*, 8063–8072.
38. Cromartie, R. I. T.; Harley-Mason, J.; Wannigama, D. G. P. *J. Chem. Soc.* **1958**, 1982–1985.