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Efficient Activation of Aromatic C−H Bonds for Fused Polyaromatic Hydrocarbon Construction

Yimin Hu,* Hao Yao, Yongjie Sun, Jing Wan, Xiangang Lin, and Tao Zhu[a]

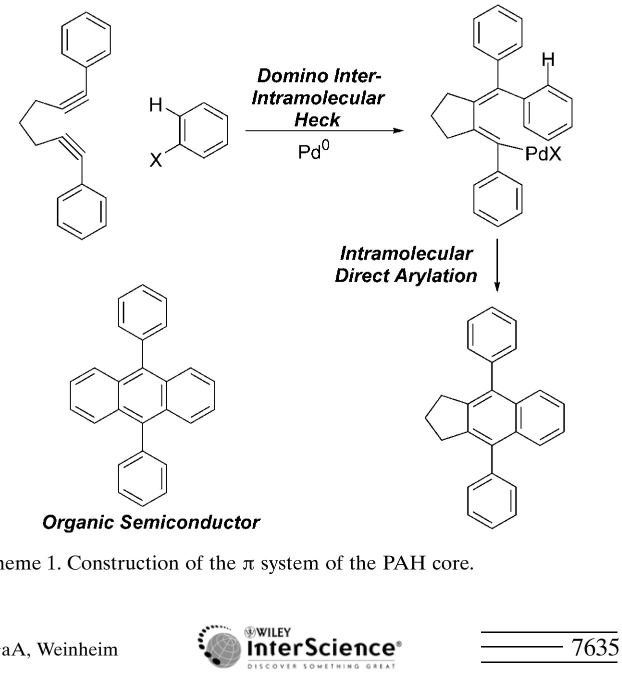
Abstract: An extraordinary domino method for the construction of polycyclic aromatic hydrocarbons has been established. Various 4,9-diphenyl-2,3-dihydro-1*H*-cyclopenta[*b*]naphthalene derivatives are constructed by the palladium(0)-catalyzed reaction of diynes with aryl halides through C–C coupling and C−H bond activation of the incorporated aryl group, which provides an effective π-system synthesis.

Keywords: 1,6-diynes · C−H activation · domino reactions · palladium · polyaromatic hydrocarbons

Introduction

Polyaromatic hydrocarbons (PAHs) possess semiconducting properties that permit their implementation in electronic devices.[1] The chemical flexibility of the organic emitters and their low production cost make them ideal materials for spintronics applications.[2] An important feature of organic electroluminescence is that the charge mobility increases with the number of π-conjugated fused rings.[3] Domino reactions have been established as a powerful tool to accomplish this goal because they offer highly efficient transformations and allow the build up of complex structures over fewer steps with increased overall yields.[4–6] Additionally, Pd-catalyzed olefination of aryl C−H bonds is a potentially powerful platform for a more direct access to carbogenic cores of complex molecules.[7] The first examples of successful intramolecular-Heck direct arylation via alkyl Pd^{II} intermediates have recently been reported.[8,9]

Therefore, we reasoned that the development of new approaches to the synthesis of structurally diverse scaffolds would likely benefit from metal-catalyzed domino inter-intramolecular reactions. The inter-intramolecular nature of these transformations means that the regio- and stereoselectivities are often excellent and a complex structure can be accessed through a sequence of carbon–carbon bond-forming reactions. Given the value of the products and the potential efficiency with which they can be prepared by the use of this method, our observations should enable further development of industrially important PAH[10] molecules. Herein, we report a novel domino cyclization for the preparation of an assortment of 4,9-diphenyl-2,3-dihydro-1*H*-cyclopenta[*b*]naphthalene derivatives from 1,6-diynes and aryl halides. The cascade consists of inter-intramolecular Heck reaction and subsequent regioselective direct arylation by C−H activation of the introduces aryl group (Scheme 1). In a typical procedure, a mixture of diethyl 2,2-bis(3-phenyl-



Scheme 1. Construction of the π system of the PAH core.

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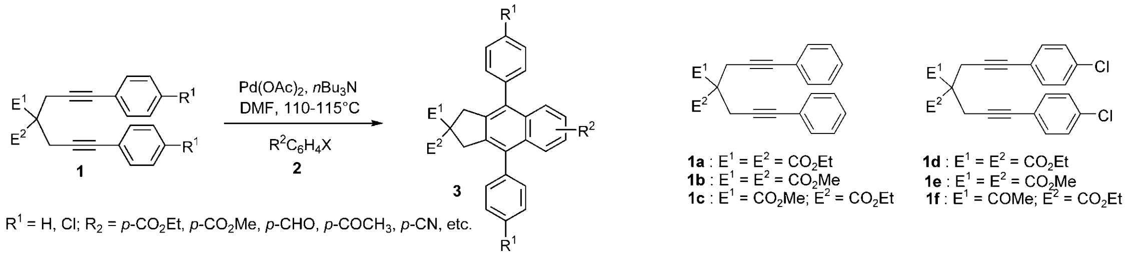
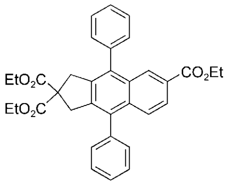
Table 1. The palladium-catalyzed, one-pot reaction for the formation of fused PAHs.[a,b]

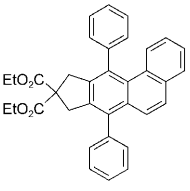


R^1 = H, Cl; R_2 = p-CO$_2$Et, p-CO$_2$Me, p-CHO, p-COCH$_3$, p-CN, etc.

1a : $E^1 = E^2 = CO_2Et$
1b : $E^1 = E^2 = CO_2Me$
1c : $E^1 = CO_2Me$; $E^2 = CO_2Et$
1d : $E^1 = E^2 = CO_2Et$
1e : $E^1 = E^2 = CO_2Me$
1f : $E^1 = COMe$; $E^2 = CO_2Et$



3aa (76%)



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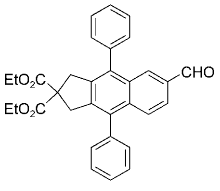

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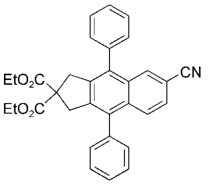

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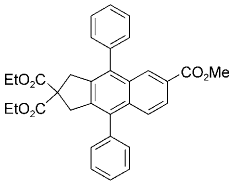

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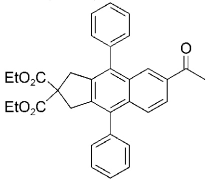

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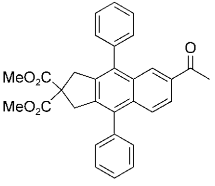

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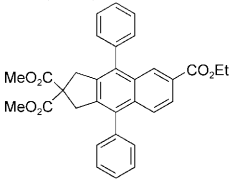

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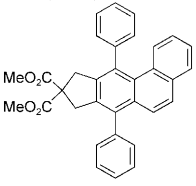
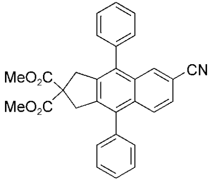


3bc (84%)



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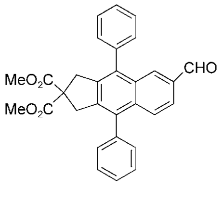
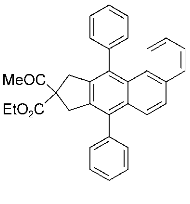


3be (59%)



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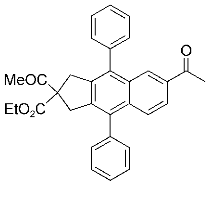

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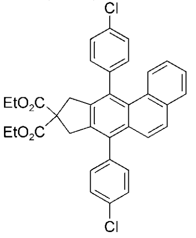

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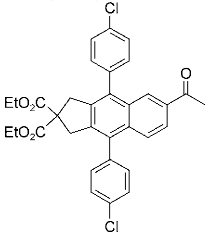
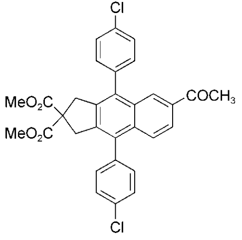


3db (85%)



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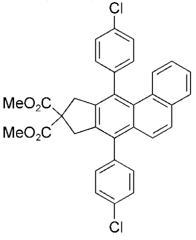

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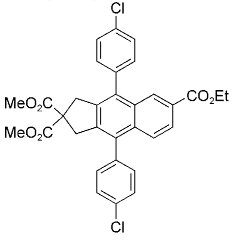
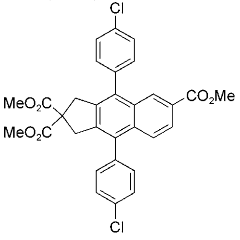
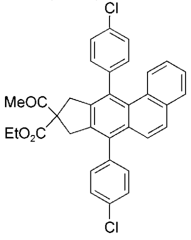


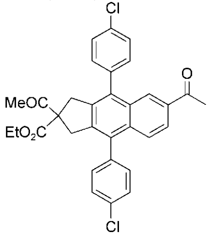
3ec^1 (38%)



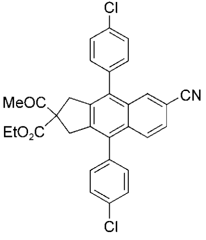
3ed^1 (35%)



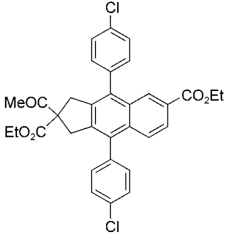
3fa (77%)



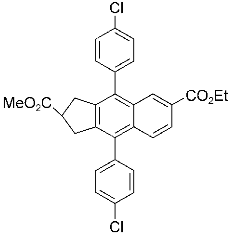
3fb (81%)



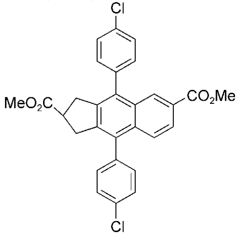
3fc (83%)



3fd (77%)



3ec^2 (49%)



3ed^2 (52%)

[a] Conditions: Substrate **1a–f** (1.0 equiv), R^{2}C$_6$H$_4$X (1.2 equiv), Pd(OAc)$_2$ (2 mol%), PPh$_3$ (4 mol%), nBu$_3$N (2 equiv), DMF (10 mL), 110–115°C, 20 h. [b] Yield given for isolated product.

prop-2-ynyl)malonate (**1a**), 4-bromobenzonitrile, tributylamine, and Pd(OAc)$_2$ was heated in DMF under argon overnight. Standard workup procedures afforded the coupled products **3** with excellent regio- and stereoselectivity for most diynes (Table 1). The temperature is crucial for this reaction; no domino reaction occurred below 90°C. In contrast, higher reaction temperatures (>120°C) led to the decomposition of **3ad**, as indicated by TLC. Reactions that employed other phosphine compounds, such as $P(OMe)_3$, $P(o\text{-tolyl})_3$, and 1,2-bis(diphenylphosphino)ethane (dppe), gave very similar results to those obtained with PPh_3. Among the catalysts tested--$AuCl(PPh_3)/AgSbF_6$, chitosan–Pd, $[Ni(cod)_2]$ (cod = 1,5-cyclooctadiene), $[Pd(PPh_3)_4]$, $[Pd(dba)_2(PPh_3)]$ (dba = dibenzylideneacetone), $[PdCl_2(PPh_3)]$, and $[Pd(OAc)_2(PPh_3)]$—the combination $[Pd(OAc)_2(PPh_3)]$ was the most effective. DMF was found to be a better solvent than *N*,*N*-dimethylacetamide, toluene, dioxane, or CH_3CN for this reaction. $n\mathrm{Bu_3N}$ was more effective than any of the inorganic bases (K_2CO_3, Cs_2CO_3) tested. Thus, the following standard reaction conditions were selected for the following studies: diyne (1 equiv) was treated with aryl halide (1.2 equiv) in the presence of $Pd(OAc)_2$ (2 mol %), PPh_3 (4 mol %), and $n\mathrm{Bu_3N}$ (2 equiv) in DMF at 110 °C.

Results and Discussion

Illustrative examples of the scope of the reaction are shown in Table 1. A variety of diynes and substituted aryl halides are compatible with this palladium-catalyzed domino reaction. A range of 4,9-diphenyl-2,3-dihydro-1*H*-cyclopenta[*b*]-naphthalene derivatives were readily isolated in good to excellent yields, except in the case of **3ec**^{1} and **3ed**^{1}, when aryl halides with a variety of substituted groups were employed. The aryl substituents could be ethoxycarbonyl, keto, naphthalenyl, cyano, or formacyl groups. Reaction of 4-bromobenzonitrile with **1a** or **1f** and of 1-(4-bromophenyl)ethanone with **1d** and **1f** afforded 3,3-disubstituted 2,3-dihydro-1*H*-cyclopenta[*b*]naphthalenes **3ad**, **3fc**, **3db**, and **3fb**, respectively, in yields beyond 83 %. The yield of compound **3ad** was the highest, at 87 %. When 1-bromonaphthalene was used in the reaction with **1a**, **1e**, or **1f**, the desired PAHs **3ab**, **3eb**, and **3fa** were obtained in very good yields, from 76–78 %.

To discover the reactivity with broadly useful substrates for preparing PAHs, in which all the moieties present in the starting material can be used for the subsequent synthetic applications in an atom-economical manner, we chose 1,7-bis(4-chlorophenyl)hepta-1,6-diynes **1d–f** as the coupling partners. Interestingly, it was found that only the C−Br bond reacted with the 1,6-diynes when bromine and chlorine substituents were both present on the benzene ring. The outputs of the reaction of non-chlorinated substrates **1a–c** were close to those of substrates **1d–f**. These results demonstrate that an aryl-C−Cl group can be incorporated into the PAHs as a synthetic handle for further functionalization. However, the cyclic compounds **3ec**^{1} and **3ed**^{1} had low yields (Table 1), 38 and 35 %, respectively. This is probably because of deoxycarbonyl decomposition of the desired products to give **3ec**^{2} and **3ed**^{2}.[11]

All new products were fully characterized by various spectroscopic techniques (^{1}H, ^{13}C NMR, UV, and IR spectroscopies, and HRMS). The molecular structure and relative configuration of **3ab** were confirmed unambiguously by X-ray diffraction (Figure 1).[12] Further details can be found in the Supporting Information (see also the Experimental Section).

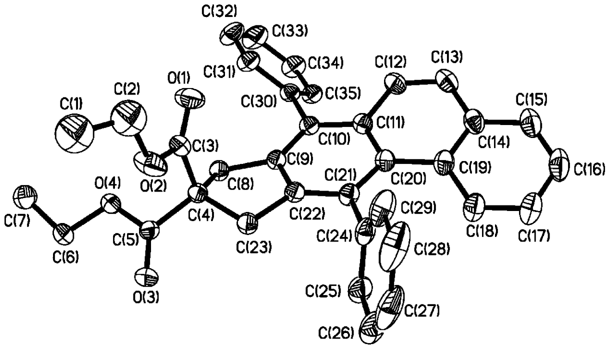
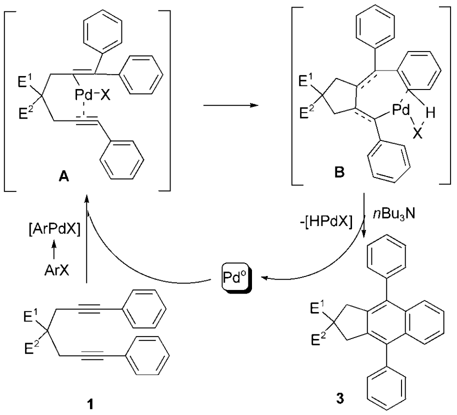


Figure 1. ORTEP plot of **3ab**, shows ellipsoids at the 30 % probability level. Selected bond lengths [Å] and angles [°]: C(9)−C(10) 1.384(2), C(9)−C(22) 1.400(3), C(10)−C(11) 1.427(4), C(11)−C(20) 1.425(4), C(20)−C(21) 1.430(4), C(21)−C(22) 1.380(4); C(9)-C(10)-C(30) 120.9(2), C(11)-C(10)-C(30) 121.0(2), C(20)-C(21)-C(24) 124.6(2), C(22)-C(21)-C(24) 116.6(2).

A plausible mechanism for the cross-coupling reaction is depicted in Scheme 2. Coordination and insertion of arylpalladium(II) halide to the diyne moiety (**1**) produces the intermediate **A**, which then reacts with the second C≡C triple bond through a carbopalladation reaction to afford **B**. A σ-bond metathesis[13] with the aryl group in **B**, followed by a proton abstraction by the base generates **3**.



Scheme 2. Proposed mechanism for the domino reaction.

Conclusion

We have developed a palladium-catalyzed domino reaction for the synthesis of fused polycyclic hydrocarbons through multistep C−C bond formation and C−H activation of the incorporated aryl group by treatment of diynes with a variety of aryl halides. This affords diphenylcyclopentanaphthalene derivatives in moderate to very good yields and with excellent regioselectivities. In addition to synthetic utility, the demonstrated ability to combine direct arylation into this class of tandem process raises confidence in the viability of other domino processes involving alkylpalladium(II) reactive intermediates. This provides a new methodology for the synthesis of fused π-conjugated polycyclic hydrocarbons. Further investigation to understand this catalytic transformation, evaluation of the process with a broader scope of substrates, synthesis of more complex π-system products, and the development of functional organic materials are in progress.

Experimental Section

General: All the catalytic reactions were performed under an argon atmosphere using oven-dried Schlenk flasks. Chemicals were purchased from Alfa Aesar and Acros Chemicals. All solvents and materials were dried, distilled, or recrystallized before use. ^{1}H (300 MHz) and ^{13}C NMR (75 MHz) spectra were recorded on a Bruker Avance 300 spectrometer with $CDCl_3$ as the solvent. Chemical shifts (δ) are reported in ppm relative to TMS (δ = 0.00 ppm) or $CDCl_3$ (δ = 77.0 ppm) for ^{1}H and ^{13}C NMR spectra, respectively. All coupling constants (*J*) are reported in Hz. Column chromatography was performed on silica gel (300–400 mesh). Melting points were determined by using a Gallenkamp melting-point apparatus and are uncorrected. The FTIR spectra were recorded in the 4000–400 cm^{-1} range on a Nicolet 5DX spectrometer as KBr discs or as thin films from $CHCl_3$ on NaCl windows. All HRMS spectra were recorded by EI at 70 eV. X-ray diffraction data of **3ab** was collected at room temperature with a Bruker SMART Apex CCD diffractometer using $Mo_{K\alpha}$ radiation (λ = 0.71073 Å) with a graphite monochromator using the ω-scan mode. Data reductions and absorption corrections were performed with SAINT and SADABS software, respectively.[14] The structure was solved by direct methods and refined on F^2 by full-matrix least squares using SHELXTL.[15] All non-hydrogen atoms were treated anisotropically. The positions of hydrogen atoms were generated geometrically. 1,6-Diynes **1a–1f** were prepared by published procedures.[16]

Typical procedure for the palladium-catalyzed domino reaction of 1,6-diynes with aryl halides: Diyne **1a–f** (1.0 equiv), 4-bromobenzonitrile (1.2 equiv), $Pd(OAc)_2$ (2 mol %), and PPh_3 (4 mol %) were added to a degassed solution of $n\text{Bu}_3N$ (2 equiv) in DMF (5 mL). The mixture was stirred at room temperature for 30 min then heated at 110–115 °C for 20 h. The reaction mixture was cooled, quenched with water, and extracted with EtOAc (3×5 mL). The combined organic layers were washed with hydrochloric acid (5 %), aqueous sodium carbonate (5 %), and saturated aqueous sodium chloride solution. After separation, the organic layer was dried over anhydrous $MgSO_4$ and concentrated under reduced pressure. The residue was purified by flash column chromatography (10:1 petroleum ether/EtOAc) to give the corresponding product **3**.

Compound 3aa: White solid; 408 mg (76 %); R_f = 0.43 (petroleum ether/EtOAc = 10:1); m.p. 176–177 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.40 (s, 1 H; Ar-H), 7.88 (d, *J* = 9.0 Hz, 1 H; Ar-H), 7.66 (d, *J* = 9.0 Hz, 1 H; Ar-H), 7.29–7.42 (m, 5 H; Ar-H), 7.43–7.55 (m, 5 H; Ar-H), 4.32 (q, *J* = 6.6 Hz, 2 H; CH_3CHH-), 4.16 (q, *J* = 6.3 Hz, 4 H; CH_3CHH-), 3.55 (s, 4 H; -CHH-), 1.34 (t, *J* = 6.9 Hz, 3 H; CH_3-), 1.19 ppm (t, *J* = 6.9 Hz, 6 H; CH_3-); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 171.3, 166.9, 139.6, 138.3, 138.0, 137.8, 136.0, 134.6, 134.4, 131.6, 129.9, 129.9, 128.9, 128.7, 127.7, 127.6, 127.0, 126.1, 124.5 , 61.7, 60.9, 60.3, 40.4, 40.3, 14.2, 14.0 ppm; IR (neat): $\tilde{\nu}$ = 1732, 1714, 1275, 1238, 1072, 704, 519 cm^{-1}; UV/Vis (CH_3CN): λ_{max} = 252 nm; HRMS (EI): *m/z*: calcd for $C_{34}H_{32}O_6$: 536.2199; found: 536.2198.

Compound 3ab: White solid; 401 mg (78 %); R_f = 0.42 (petroleum ether/EtOAc = 10:1); m.p. 128–129 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.76 (d, *J* = 7.8 Hz, 1 H; Ar-H), 7.68 (d, *J* = 8.7 Hz, 1 H; Ar-H), 7.03–7.18 (m, 4 H; Ar-H), 7.20–7.38 (m, 5 H; Ar-H), 7.41–7.58 (m, 5 H; Ar-H), 4.15 (q, *J* = 7.2 Hz, 4 H; CH_3CHH-), 3.53 (d, *J* = 18 Hz, 4 H; -CHH-), 1.20 ppm (t, *J* = 7.2 Hz, 6 H; CH_3-); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 171.2, 143.3, 139.4, 139.2, 137.2, 135.4, 132.9, 131.6, 130.1, 130.0, 129.6, 128.9, 128.6, 128.2, 128.1, 127.3, 127.2, 126.8, 125.6, 124.9, 114.9, 61.7, 60.1, 41.3, 40.1, 14.0 ppm; IR (neat): $\tilde{\nu}$ = 1726, 1493, 1442, 1274, 1244, 1193, 1072, 750, 713, 704, 526 cm^{-1}; UV/Vis (CH_3CN): λ_{max} = 260 nm; HRMS (EI): *m/z*: calcd for $C_{35}H_{30}O_4$: 514.2144; found: 514.2141. CCDC-764193 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_ request/cif.

Compound 3ac: White solid; 296 mg (60 %); R_f = 0.42 (petroleum ether/EtOAc = 10:1); m.p. 78–79 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 9.96 (s, 1 H; -CHO), 8.12 (s, 1 H; Ar-H), 7.79 (d, *J* = 8.1 Hz, 1 H; Ar-H), 7.72 (d, *J* = 9.0, 1 H; Ar-H), 7.10–7.37 (m, 5 H; Ar-H), 7.40–7.67 (m, 5 H; Ar-H), 4.16 (q, *J* = 6.6 Hz, 4 H; CH_3CHH-), 3.57 (s, 4 H; -CHH-), 1.20 ppm (t, *J* = 7.2 Hz, 6 H; CH_3-); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 192.5, 171.2, 140.8, 138.4, 138.0, 133.4, 133.0, 129.8, 129.8, 128.8, 128.8, 128.0, 127.7, 127.1, 121.9, 61.8, 60.2, 40.6, 40.2, 14.0 ppm; IR (neat): $\tilde{\nu}$ = 1732, 1694, 1260, 1259, 1186, 752, 702, 518 cm^{-1}; UV/Vis (CH_3CN): λ_{max} = 265 nm; HRMS (EI): *m/z*: calcd for $C_{32}H_{28}O_5$: 492.1937; found: 492.1938.

Compound 3ad: White solid; 426 mg (87 %); R_f = 0.42 (petroleum ether/EtOAc = 10:1); m.p. 142–143 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.02 (s, 1 H; Ar-H), 7.70 (d, *J* = 8.1 Hz, 1 H; Ar-H), 7.57 (d, *J* = 6.0 Hz, 1 H; Ar-H), 7.27–7.39 (m, 5 H; Ar-H), 7.41–7.54 (m, 5 H; Ar-H), 4.15 (q, *J* = 6.6 Hz, 4 H; CH_3CHH-), 3.56 (s, 4 H; CHH), 1.20 ppm (t, *J* = 6.9 Hz, 6 H; CH_3); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 171.1, 140.7, 139.0, 137.6, 137.2, 135.2, 134.9, 133.7, 132.3, 131.5, 129.7, 129.0, 128.8, 128.4, 128.0, 127.9, 127.2, 125.7, 119.6, 108.5, 61.8, 40.5, 40.3, 14.0 ppm; IR (neat): $\tilde{\nu}$ = 1726, 1595, 1274, 1255, 1161, 1070, 829 cm^{-1}; UV/Vis (CH_3CN): λ_{max} = 238 nm; HRMS (EI): *m/z*: calcd for $C_{32}H_{27}NO_4$: 489.1940; found: 489.1943.

Compound 3ae: White solid; 402 mg (77 %); R_f = 0.42 (petroleum ether/EtOAc = 10:1); m.p. 143–144 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.40 (s, 1 H; Ar-H), 7.88 (d, *J* = 8.4 Hz, 1 H; Ar-H), 7.66 (d, *J* = 8.7 Hz, 1 H; Ar-H), (d, *J* = 8.7 Hz, 1 H; Ar-H), 7.29–7.42 (m, 5 H; Ar-H), 7.45–7.56 (m, 5 H; Ar-H), 4.14 (q, *J* = 6.9 Hz, 4 H; CH_3CHH-), 3.86 (s, 3 H; CH_3-O), 3.55 (s, 4 H; -CHH-), 1.19 ppm (t, *J* = 6.9 Hz, 6 H; CH_3-); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 171.3, 167.4, 139.7, 138.2, 137.9, 136.0, 134.5, 131.5, 129.9, 129.9, 128.9, 128.7, 128.7, 127.7, 127.6, 126.7, 126.2, 124.8, 61.7, 60.3, 52.1, 40.5, 40.3, 14.0 ppm; IR (neat): $\tilde{\nu}$ = 1730, 1710, 1441, 1364, 1263, 1236, 1188, 758, 704, 516 cm^{-1}; UV/Vis (CH_3CN): λ_{max} = 248 nm; HRMS (EI): *m/z*: calcd for $C_{33}H_{30}O_6$: 522.2042; found: 522.2043.

Compound 3af: White solid; 355 mg (70 %); R_f = 0.43 (petroleum ether/EtOAc = 10:1); m.p. 141–142 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.27 (s, 1 H; Ar-H), 7.72 (d, *J* = 8.4 Hz, 1 H; Ar-H), 7.68 (d, *J* = 8.7 Hz, 1 H; Ar-H), 7.27–7.38 (m, 5 H; Ar-H), 7.40–7.58 (m, 5 H; Ar-H), 4.14 (q, *J* = 6.9 Hz, 4 H; CH_3CHH-O-), 3.56 (s, 4 H; -CHH-), 2.50 (s, 3 H; -CO-CH_3), 1.20 ppm (t, *J* = 6.6 Hz, 6 H; CH_3-); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 198.3, 171.3, 140.0, 138.1, 138.0, 137.9, 136.2, 134.7, 134.5, 133.8, 131.5, 129.9, 129.8, 128.7, 128.7, 128.2, 127.8, 127.6, 126.4, 123.1, 112.7, 61.8, 60.3, 40.5, 40.3, 26.5, 14.0 ppm; IR (neat): $\tilde{\nu}$ = 1734, 1684, 1602, 1257, 1234, 1190, 1072, 750, 704 cm^{-1}; UV/Vis (CH_3CN): λ_{max} = 259 nm; HRMS (EI): *m/z*: calcd for $C_{33}H_{30}O_5$: 506.2093; found: 506.2092.

Compound 3ba: Yellow oil; 349 mg (73 %); R_f = 0.44 (petroleum ether/EtOAc = 8:1); ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.28 (s, 1 H; Ar-H), 7.87 (d, *J* = 8.7 Hz, 1 H; Ar-H), 7.69 (d, *J* = 8.7 Hz, 1 H; Ar-H), 7.27–7.38 (m, 5 H; Ar-H), 7.40–7.58 (m, 5 H; Ar-H), 3.70 (s, 6 H; CH_3O-), 3.58 (s, 4 H; -CHH-), 2.50 ppm (s, 3 H; -OCCH_3); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 198.2, 171.7, 139.7, 138.0, 137.9, 137.8, 134.7, 134.5, 133.8, 131.6, 129.9, 129.8, 128.7, 128.7, 128.5, 128.2, 127.8, 127.7, 127.1, 126.4, 123.2, 60.1, 53.0, 40.6, 40.4, 26.5 ppm; IR (neat): $\tilde{\nu}$ = 1736, 1681, 1435, 1365, 1259, 1236, 752, 702, 516 cm^{-1}; UV/Vis (CH_3CN): λ_{max} = 261 nm; HRMS (EI): *m/z*: calcd for $C_{31}H_{26}O_5$: 478.1780; found: 478.1781.

Compound 3bb: White solid; 366 mg (72 %); R_f = 0.43 (petroleum ether/EtOAc = 10:1); m.p. 142–143 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.42 (s, 1 H; Ar-H), 8.23 (d, *J* = 7.2 Hz, 1 H; Ar-H), 7.90 (d, *J* = 8.4 Hz, 1 H; Ar-H), 7.27–7.45 (m, 5 H; Ar-H), 7.49–7.68 (m, 5 H; Ar-H), 4.32(q, *J*=7.2 Hz, 2 H; CH_3CHH-O-), 3.69 (s, 6 H; CH_3-), 3.57 (s, 4 H; -CHH-), 1.33 ppm (t, *J* = 6.9 Hz, 3 H; CH_3-); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 171.7, 166.9, 138.2, 137.6, 130.1, 130.0, 129.9, 128.9, 128.7, 128.6, 27.7, 127.6, 127.4, 127.1, 126.1, 126.1, 125.5, 125.5, 125.4, 124.6, 60.9, 60.2, 53.0, 40.4, 40.3, 14.3 ppm; IR (neat): $\tilde{\nu}$ = 1732, 1713, 1618, 1263, 1238, 756, 706, 517 cm^{-1}; UV/Vis (CH_3CN): λ_{max} = 272 nm; HRMS (EI): *m/z*: calcd for $C_{32}H_{28}O_6$: 508.1886; found: 508.1884.

Compound 3bc: White solid; 409 mg (84 %); $R_f=0.44$ (petroleum ether/EtOAc = 10:1); m.p. 174–175 °C; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta=7.77$ (d, $J=7.5$ Hz, 1H; Ar-H), 7.68 (d, $J=7.8$ Hz, 1H; Ar-H), 7.07–7.25 (m, 5H; Ar-H), 7.28–7.39 (m, 5H; Ar-H), 7.42–7.57 (m, 4H; Ar-H), 3.70 (s, 6H; CH_3-O-), 3.56 ppm (d, $J=19.0$ Hz, 4H; -CHH-); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta=172.0$, 143.2, 139.3, 139.0, 137.0, 135.7, 135.4, 133.0, 131.2, 130.1, 130.0, 129.6, 129.0, 128.6, 128.2, 128.1, 127.4, 127.3, 126.8, 125.7, 124.9, 59.9, 52.9, 41.5, 40.7 ppm; IR (neat): $\tilde{\nu}=1732$, 1489, 1435, 1277, 1242, 1159, 1072, 873, 752, 715, 702, 527 cm^{-1}; UV/Vis (CH_3CN): $\lambda_{max}=260$ nm; HRMS (EI): *m/z*: calcd for $C_{33}H_{26}O_4$: 486.1831; found: 486.1834.

Compound 3bd: White solid; 365 mg (79 %); $R_f=0.45$ (petroleum ether/EtOAc = 10:1); m.p. 195–196 °C; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta=8.02$ (s, 1H; Ar-H), 7.72 (d, $J=9.0$ Hz, 1H; Ar-H), 7.57 (d, $J=5.71$ Hz, 1H; Ar-H), 7.27–7.40 (m, 5H; Ar-H), 7.42–7.54 (m, 5H; Ar-H), 3.70 (s, 6H; CH_3-O-), 3.56 ppm (s, 4H; -CHH-); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta=$ 171.5, 140.4, 138.7, 137.4, 137.1, 135.2, 133.7, 132.2, 131.5, 129.7, 129.2, 128.9, 128.8, 128.4, 128.0, 127.9, 127.3, 127.2, 125.7, 119.5, 108.5, 59.9, 53.0, 40.5, 40.3 ppm; IR (neat): $\tilde{\nu}=2226$, 1734, 1433, 1280, 1259, 1205, 1076, 752, 704 cm^{-1}; UV/Vis (CH_3CN): $\lambda_{max}=250$ nm; HRMS (EI): *m/z*: calcd for $C_{30}H_{23}NO_4$: 461.1627; found: 461.1623.

Compound 3be: White solid; 274 mg (59 %); $R_f=0.45$ (petroleum ether/EtOAc = 10:1); m.p. 79–80 °C; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta=9.96$ (s, 1H; -CHO), 8.13 (s, 1H; Ar-H), 7.80 (d, $J=8.7$ Hz, 1H; Ar-H), 7.74 (d, $J=8.7$ Hz, 1H; Ar-H), 7.09–7.37 (m, 5H; Ar-H), 7.39–7.59 (m, 5H; Ar-H), 3.70 (s, 6H; CH_3-O-), 3.59 ppm (s, 4H; -CHH-); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta=192.5$, 171.7, 142.7, 140.5, 138.2, 137.9, 137.7, 135.4, 135.1, 133.4, 132.9, 129.8, 129.8, 129.3, 128.9, 128.8, 128.5, 128.3, 128.0, 127.8, 127.1, 121.9, 60.0, 53.0, 40.7, 40.4 ppm; IR (neat): $\tilde{\nu}=1734$, 1693, 1614, 1602, 1435, 1263, 1203, 1072, 831, 752, 702, 519 cm^{-1}; UV/Vis (CH_3CN): $\lambda_{max}=269$ nm; HRMS (EI): *m/z*: calcd for $C_{30}H_{24}O_5$: 464.1624; found: 464.1621.

Compound 3ca: White solid; 378 mg (78 %); $R_f=0.45$ (petroleum ether/EtOAc = 10:1); m.p. 93–94 °C; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta=7.77$ (d, $J=7.5$ Hz, 1H; Ar-H), 7.66 (d, $J=8.7$ Hz, 1H; Ar-H), 7.04–7.15 (m, 4H; Ar-H), 7.17–7.35 (m, 5H; Ar-H), 7.38–7.55 (m, 5H; Ar-H), 4.16 (q, $J=$ 6.9 Hz, 2H; CH_3CHH-O-), 3.49 (s, 4H; -CHH-), 2.14 (s, 3H; -OCCH_3), 1.21 ppm (t, $J=6.6$ Hz, 3H; CH_3-); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta=$ 202.9, 172.3, 139.4, 137.0, 133.0, 131.7, 130.0, 129.6, 129.6, 128.9, 128.7, 128.6, 128.2, 128.1, 127.4, 127.3, 126.8, 125.7, 124.9, 124.9, 124.9, 108.0, 66.5, 61.8, 40.0, 39.2, 26.3, 14.0 ppm; IR (neat): $\tilde{\nu}=1712$, 1598, 1490, 1440, 1355, 1070, 825, 750, 702, 667, 521 cm^{-1}; UV/Vis (CH_3CN): $\lambda_{max}=275$ nm; HRMS (EI): *m/z*: calcd for $C_{34}H_{28}O_3$: 484.2038; found: 484.2039.

Compound 3cb: Yellow oil; 357 mg (75 %); $R_f=0.41$ (petroleum ether/EtOAc = 8:1); ^{1}H NMR (300 MHz, $CDCl_3$): $\delta=8.26$ (s, 1H; Ar-H), 7.87 (d, $J=8.7$ Hz, 1H; Ar-H), 7.67 (d, $J=8.7$ Hz, 1H; Ar-H), 7.08–7.26 (m, 5H; Ar-H), 7.30–7.59 (m, 5H; Ar-H), 4.17 (q, $J=6.9$ Hz, 2H; CH_3CHH-O-), 3.49 (s, 4H; -CHH-), 2.50 (s, 3H; -OCCH_3), 2.15 (s, 3H; -OCCH_3), 1.21 ppm (t, $J=6.9$ Hz, 3H; CH_3-); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta=$ 202.6, 198.3, 171.1, 139.9, 138.7, 138.1, 137.9, 136.3, 134.8, 134.5, 133.8, 131.6, 130.5, 129.8, 128.8, 128.2, 127.9, 127.7, 126.4, 123.2, 66.7, 62.0, 39.1, 38.9, 26.5, 26.3, 14.0 ppm; IR (neat): $\tilde{\nu}=1714$, 1682, 1601, 1366, 1255, 1232, 754, 702 cm^{-1}; UV/Vis (CH_3CN): $\lambda_{max}=265$ nm; HRMS (EI): *m/z*: calcd for $C_{32}H_{28}O_5$: 476.1988; found: 476.1992.

Compound 3da: White solid; 467 mg (80 %); $R_f=0.45$ (petroleum ether/EtOAc = 10:1); m.p. 94–95 °C; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta=7.79$ (d, $J=7.8$ Hz, 1H; Ar-H), 7.72 (d, $J=8.4$ Hz, 1H; Ar-H), 7.15–7.39 (m, 6H; Ar-H), 7.41–7.61 (m, 6H; Ar-H), 4.17 (q, $J=6.3$ Hz, 4H; CH_3CHH-O-), 3.52 (d, $J=18.9$ Hz, 4H; -CHH-), 1.23 ppm (t, $J=6.3$ Hz, 6H; CH_3-); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta=171.3$, 141.5, 139.3, 137.6, 137.3, 134.6, 134.2, 133.5, 133.2, 132.9, 131.5, 131.3, 130.4, 129.8, 128.9, 128.3, 127.9, 127.1, 125.9, 125.1, 124.4, 61.7, 60.0, 41.2, 40.4, 14.0 ppm; IR (neat): $\tilde{\nu}=$ 1742, 1717, 1489, 1473, 1250, 1178, 1089, 1012, 823, 765, 527 cm^{-1}; UV/Vis (CH_3CN): $\lambda_{max}=276$ nm; HRMS (EI): *m/z*: calcd for $C_{35}H_{28}Cl_2O_4$: 582.1365; found: 582.1361.

Compound 3db: White solid; 489 mg (85 %); $R_f=0.45$ (petroleum ether/EtOAc = 8:1); m.p. 144–145 °C; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta=8.23$ (s, 1H; Ar-H), 7.88 (d, $J=8.7$ Hz, 1H; Ar-H), 7.65 (d, $J=9.0$ Hz, 1H; Ar-H), 7.55 (t, $J=6.9$, 4H; Ar-H); 7.34 (t, $J=8.4$ Hz, 4H; Ar-H), 4.16 (q, $J=$ 6.9 Hz, 4H; CH_3CHH-O-), 3.53 (s, 4H; -CHH-), 2.53 (s, 3H; -CO-CH_3), 1.20 ppm (t, $J=6.9$ Hz, 6H; CH_3-); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta=$ 198.1, 171.1, 140.1, 138.2, 136.4, 136.2, 135.2, 134.3, 134.0, 133.2, 133.7, 131.4, 131.2, 131.2, 129.1, 129.1, 127.7, 126.3, 123.6, 119.4, 61.9, 60.3, 40.4, 40.2, 26.7, 14.0 ppm; IR (neat): $\tilde{\nu}=1732$, 1682, 1491, 1367, 1258, 1236, 1188, 1091, 819, 524 cm^{-1}; UV/Vis (CH_3CN): $\lambda_{max}=277$ nm; HRMS (EI): *m/z*: calcd for $C_{33}H_{28}Cl_2O_5$: 574.1314; found: 574.1311.

Compound 3ea: White solid; 339 mg (62 %); $R_f=0.42$ (petroleum ether/EtOAc = 8:1); m.p. 135–136 °C; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta=8.24$ (s, 1H; Ar-H), 7.89 (d, $J=8.7$ Hz, 1H; Ar-H), 7.66 (d, $J=8.7$ Hz, 1H; Ar-H), 7.55 (s, 4H; Ar-H), 7.35 (d, $J=8.1$ Hz, 4H; Ar-H), 3.71 (s, 6H; CH_3-O-), 3.55 (s, 4H; -CHH-), 2.53 ppm (s, 3H; -CO-CH_3); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta=198.2$, 171.5, 139.9, 138.0, 136.3, 136.1, 135.2, 134.3, 134.1, 134.0, 133.9, 133.7, 131.4, 131.2, 131.2, 129.1, 129.1, 127.7, 126.3, 123.7, 60.0, 53.1, 40.5, 40.3, 26.6 ppm; IR (neat): $\tilde{\nu}=1739$, 1680, 1610, 1490, 1256, 1091, 1012, 841, 822, 609, 525 cm^{-1}; UV/Vis (CH_3CN): $\lambda_{max}=$ 256 nm; HRMS (EI): *m/z*: calcd for $C_{31}H_{24}Cl_2O_5$: 546.1001; found: 546.0999.

Compound 3eb: White solid; 422 mg (76 %); $R_f=0.44$ (petroleum ether/EtOAc = 10:1); m.p. 242–243 °C; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta=7.78$ (d, $J=7.8$ Hz, 1H; Ar-H), 7.69 (d, $J=8.4$ Hz, 1H; Ar-H), 7.13–7.55 (m, 12H; Ar-H), 3.71 (s, 6H; CH_3-O-), 3.51 ppm (d, $J=19.8$ Hz, 4H; -CHH-); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta=171.8$, 142.2, 139.4, 139.1, 137.0, 135.7, 135.4, 133.0, 131.1, 130.4, 130.1, 129.6, 129.0, 128.6, 128.2, 128.1, 127.4, 127.3, 126.8, 123.7, 124.9, 59.9, 52.8, 41.5, 40.6 ppm; IR (neat): $\tilde{\nu}=1736$, 1492, 1429, 1281, 1250, 1152, 1091, 1072, 808, 750, 529 cm^{-1}; UV/Vis (CH_3CN): $\lambda_{max}=265$ nm; HRMS (EI): *m/z*: calcd for $C_{33}H_{24}Cl_2O_4$: 554.1052; found: 554.1056.

Compound 3ec^1: White solid; 219 mg (38 %); $R_f=0.44$ (petroleum ether/EtOAc = 10:1); m.p. 164–165 °C; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta=8.37$ (s, 1H; Ar-H), 7.92 (d, $J=8.4$ Hz, 1H; Ar-H), 7.64 (d, $J=8.7$ Hz, 1H; Ar-H), 7.27–7.55 (m, 8H; Ar-H), 4.34 (q, $J=6.6$ Hz, 2H; CH_3-CHH-O-), 3.71 (s, 6H; CH_3-O-), 3.54 (s, 4H; -CHH-), 1.35 ppm (t, $J=6.6$ Hz, 3H; -CH_3); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta=171.5$, 166.6, 139.4, 137.7, 136.3, 136.1, 135.0, 134.2, 133.8, 133.7, 133.6, 131.3, 131.2, 131.1, 129.0, 128.5, 127.3, 125.9, 124.9, 61.0, 60.0, 53.0, 40.4, 40.3, 14.2 ppm; IR (neat): $\tilde{\nu}=$ 1738, 1709, 1492, 1269, 1259, 1096, 1014, 824, 761, 524 cm^{-1}; UV/Vis (CH_3CN): $\lambda_{max}=251$ nm; HRMS (EI): *m/z*: calcd for $C_{32}H_{26}Cl_2O_6$: 576.1106; found: 576.1102.

Compound 3ed^1: White solid; 197 mg (35 %); m.p. 141–142 °C; $R_f=0.44$ (petroleum ether/EtOAc = 10:1); ^{1}H NMR (300 MHz, $CDCl_3$): $\delta=8.35$ (s, 1H; Ar-H), 7.92 (d, $J=8.7$ Hz, 1H; Ar-H), 7.64 (d, $J=8.7$ Hz, 1H; Ar-H), 7.27–7.54 (m, 8H; Ar-H), 3.89 (s, 3H; CH_3-O-), 3.70 (s, 6H; CH_3-O-), 3.54 ppm (s, 4H; -CHH-); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta=171.5$, 167.1, 139.5, 137.8, 136.3, 136.1, 135.0, 134.3, 133.8,133.8, 133.6, 131.3, 131.2, 131.1, 130.6, 129.0, 129.0, 128.5, 127.0, 125.9, 124.9, 59.9, 53.0, 52.2, 40.4, 40.3 ppm; IR (neat): $\tilde{\nu}=1738$, 1717, 1489, 1435, 1300, 1271, 1161, 1093, 1012, 844, 823, 763, 524 cm^{-1}; UV/Vis (CH_3CN): $\lambda_{max}=252$ nm; HRMS (EI): *m/z*: calcd for $C_{31}H_{24}Cl_2O_6$: 562.0950; found: 562.0928.

Compound 3fa: White solid; 426 mg (77 %); $R_f=0.44$ (petroleum ether/EtOAc = 10:1); m.p. 142–143 °C; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta=7.78$ (d, $J=7.5$ Hz, 1H; Ar-H), 7.78 (d, $J=8.7$ Hz, 1H; Ar-H), 7.13–7.40 (m, 6H; Ar-H), 7.43–7.60 (m, 6H; Ar-H), 4.18 (q, $J=6.9$ Hz, 2H; CH_3CHH-O-), 3.43 (d, $J=17.4$ Hz, 4H; -CHH-), 2.15 (s, 3H; -OCCH_3), 1.22 ppm (t, $J=6.9$ Hz, 3H; CH_3-); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta=202.4$, 172.0, 141.5, 139.1, 137.6, 137.1, 133.5, 133.2, 132.9, 131.5, 131.2, 130.3, 129.9, 129.0, 129.8, 129.0, 128.3, 127.9, 127.2, 125.9, 125.1, 124.4, 66.4, 61.9, 39.8, 39.0, 26.1, 13.9 ppm; IR (neat): $\tilde{\nu}=1741$, 1714, 1489, 1232, 1092, 1072, 1015, 827, 766, 524 cm^{-1}; UV/Vis (CH_3CN): $\lambda_{max}=207$ nm; HRMS (EI): *m/z*: calcd for $C_{34}H_{26}Cl_2O_3$: 552.1259; found:552.1251.

Compound 3fb: White solid; 442 mg (81 %); $R_f=0.44$ (petroleum ether/EtOAc = 8:1); m.p. 173–174 °C; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta=8.21$ (s, 1H; Ar-H), 7.88 (d, $J=8.7$ Hz, 1H; Ar-H), 7.63 (d, $J=9.0$ Hz, 1H; Ar-H), 7.26–7.58 (m, 8H; Ar-H), 4.18 (q, $J=6.6$ Hz, 2H; CH_3CHH-O-), 3.46 (s, 4H; -CHH-), 2.53 (s, 3H; -OCCH_3), 2.16 (s, 3H; -OCCH_3), 1.22 ppm (t, $J=6.6$ Hz, 3H; CH_3-); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta=202.2$, 198.0, 171.8, 139.9, 138.0, 136.4, 136.2, 135.2, 134.3, 134.1, 133.9, 133.8, 131.4, 131.2, 129.2, 129.1, 129.1, 127.6, 126.2, 123.7, 66.7, 62.0, 39.0, 38.8, 26.6, 26.2, 14.0 ppm; IR (neat): $\tilde{\nu}$ = 1712, 1681, 1491, 1364, 1253, 1092, 1012, 829, 523 cm^{-1}; UV/Vis (CH_3CN): λ_{max} = 262 nm; HRMS (EI): m/z: calcd for $C_{32}H_{26}C_{12}O_4$: 544.1208; found: 544.1197.

Compound 3fc: White solid; 439 mg (83 %); R_f = 0.43 (petroleum ether/EtOAc = 10:1); m.p. 174–175 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.95 (s, 1H; Ar-H), 7.67 (d, J = 8.7 Hz, 1H; Ar-H), 7.58 (d, J = 6.6 Hz, 1H; Ar-H), 7.27–7.54 (m, 8H; Ar-H), 4.17 (q, J = 6.6 Hz, 2H; CH_3CHH-O-), 3.45 (s, 4H; -CHH-), 2.15 (s, 3H; -$OCCH_3$), 1.22 ppm (t, J = 6.9 Hz, 3H; CH_3-); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 201.9, 171.6, 140.6, 138.9, 135.7, 135.3, 134.3, 134.1, 133.5, 131.9, 131.3, 131.0, 130.7, 129.3, 129.3, 129.2, 129.2, 128.6, 126.9, 126.0, 119.2, 111.4 ,108.9, 66.5, 62.1, 38.9, 38.7, 26.1, 14.0 ppm; IR (neat): $\tilde{\nu}$ = 2226, 1712, 1491, 1364, 1230, 1090, 1015, 820, 518 cm^{-1}; UV/Vis (CH_3CN): λ_{max} = 253 nm; HRMS (EI): m/z: calcd for $C_{31}H_{23}Cl_2NO_3$: 527.1055; found: 524.1042.

Compound 3fd: White solid; 443 mg (77 %); R_f = 0.46 (petroleum ether/EtOAc = 10:1); m.p. 175–176 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.35 (s, 1H; Ar-H), 7.92 (d, J = 8.7 Hz, 1H; Ar-H), 7.62 (d, J = 9.0 Hz, 1H; Ar-H), 7.27–7.56 (m, 8H; Ar-H), 4.35 (q, J = 6.6 Hz, 2H; CH_3CHH-O-), 4.18 (q, J = 6.9 Hz, 2H; CH_3CHH-O-), 3.45 (s, 4H; -CHH-), 2.15 (s, 3H; -$OCCH_3$), 1.35 (t, J = 6.9 Hz, 3H; CH_3-), 1.22 ppm (t, J = 6.9 Hz, 3H; CH_3-); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 202.2, 171.8, 166.6, 139.5, 137.7, 136.4, 136.2, 135.0, 134.2, 133.8, 133.7, 133.6, 131.3, 131.1, 131.1, 129.0, 129.0, 128.5, 127.3, 125.8, 124.9, 66.6, 61.9, 61.0, 38.9, 38.8, 26.1, 14.2, 13.9 ppm; IR (neat): $\tilde{\nu}$ = 1714, 1645, 1361, 1263, 1234, 1091, 1014, 819, 762, 518 cm^{-1}; UV/Vis (CH_3CN): λ_{max} = 251 nm; HRMS (EI): m/z: calcd for $C_{33}H_{28}Cl_2O_5$: 574.1314; found: 574.1309.

Compound 3ec^2: White solid; 254 mg (49 %); R_f = 0.46 (petroleum ether/EtOAc = 10:1); m.p. 171–172 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.37 (s, 1H; Ar-H), 7.92 (d, J = 8.4 Hz, 1H; Ar-H), 7.63 (d, J = 8.7 Hz, 1H; Ar-H), 7.27–7.54 (m, 8H; Ar-H), 4.36 (q, J = 6.9 Hz, 2H; CH_3CHH-O-), 3.68 (s, 3H; CH_3-O-), 3.28 (d, J = 11.8 Hz, 4H; -CHH-), 3.15 (m, 1H; -CH-), 3.12–3.16 ppm (m, 1H; -CH-); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 175.0, 166.7, 141.2, 139.5, 136.7, 136.4, 134.8, 134.1, 133.7, 133.6, 133.4, 131.3, 131.2, 131.1, 131.1, 129.0, 128.8, 128.5, 127.2, 125.8, 124.7, 61.0, 52.0, 43.5, 36.1, 35.9, 14.2 ppm; IR (neat): $\tilde{\nu}$ = 1735, 1362, 1262, 1254, 1090, 1013, 819, 722, 522 cm^{-1}; UV/Vis (CH_3CN): λ_{max} = 256 nm; HRMS (EI): m/z: calcd for $C_{30}H_{24}Cl_2O_4$: 518.1052; found: 518.1047.

Compound 3ed^2: White solid; 263 mg (52 %); R_f = 0.46 (petroleum ether/EtOAc = 10:1); m.p. 173–174 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.35 (s, 1H; Ar-H), 7.92 (d, J = 8.7 Hz, 1H; Ar-H), 7.64 (d, J = 8.7 Hz, 1H; Ar-H), 7.27–7.54 (m, 8H; Ar-H), 3.89 (s, 3H; CH_3-O-), 3.68 (s, 3H; CH_3-O-), 3.27 (d, J = 12.6 Hz, 4H; -CHH-), 3.12–3.17 ppm (m, 1H; -CH-); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 175.0, 167.2, 141.3, 139.5, 136.6, 136.4, 134.8, 134.1, 133.7, 133.6, 131.2, 131.1, 129.0, 129.0, 128.8, 128.8, 128.5, 126.9, 125.9, 124.8, 52.1, 52.0, 43.4, 36.3, 36.1 ppm; IR (neat): $\tilde{\nu}$ = 1738, 1713, 1645, 1634, 1493, 1435, 1261, 1089, 1012, 819, 763, 717, 524 cm^{-1}; UV/Vis (CH_3CN): λ_{max} = 254 nm; HRMS (EI): m/z: calcd for $C_{29}H_{22}Cl_2O_4$: 504.0895; found: 504.0883.

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