A palladium-catalyzed cascade approach for the synthesis of 3,3a,4,6,7,8,9,9a-octahydro-1H-benzo[f]isoindole-1,5(2H)-diones

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Abstract
A synthetic method for the preparation of 3,3a,4,6,7,8,9,9a-octahydro-1H-benzo[f]isoindole-1,5(2H)-diones from 1,6-dienes and vinyl iodides is developed using PdCl₂(PPh₃)₂ as the catalyst. The presented approach exhibits a good functional group tolerance and affords moderate yields of the products. A mechanism is also proposed.

Keywords
1,6-dienes, cascade reactions, enones and their derivatives, Pd-catalyzed, polycyclic compounds

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Introduction
Due to the benefits of atom economy, saving time, and less waste generation, cascade reactions that allow the generation of molecular complexity in a single operation have attracted significant attention.1–5 Although cascade reactions have been successfully employed for the synthesis of the core skeleton of many important natural products, the design and performance of cascade reactions remains a challenging aspect of organic chemistry.6–10 Recently, the α-arylation of enones and their derivatives has been developed.11–14 For example, Liu reported an efficient synthesis of N-fused polycyclic indoles via a palladium-catalyzed annulation/acyl migration cascade reaction (Scheme 1a).11 McGlacken reported that 1,3-diketone-derived enol ethers could undergo Heck cyclization, offering tricyclic oxoisochromene derivatives in good yields (Scheme 1b).12 In these cases, the introduction of a halogen atom into certain molecules was necessary.15,16 Meanwhile, reports on alkylations at the α-position of enones and their derivatives are comparatively scarce.17,18 Starting from electron-deficient vinyl iodides and propargyl ethers, Huang provided an efficient synthesis of structurally complex polycycles with 2,3-dihydrofuran units (Scheme 1c).18 Given this background, we envisioned that Pd-catalyzed reactions of 1,6-dienes and vinyl iodides would proceed via a cascade reaction to form polycyclic compounds through alkylation of the α-position of the enone (Scheme 1d).

Results and discussion
Our initial study was carried out with the reaction between N-allyl-N-benzylacrylamide (1a) and 3-iodocyclohex-2-en-1-one (2a) in order to examine the reaction parameters (Table 1). When the reaction was performed in the presence of Pd(OAc)₂ in toluene at 130 °C under a nitrogen atmosphere using K₂CO₃ as the base and TBAB as an additive, 2-benzyl-3,3a,4,6,7,8,9,9a-octahydro-1H-benzo[f]isoindole-1,5(2H)-dione (3a) was obtained in 36% yield (Table 1, Entry 1). Subsequently, various palladium salts were tested, among which PdCl₂(PPh₃)₂ provided the best result (Table 1, Entries 1–3). A series of bases, including DABCO, n-Bu₃N, LiOH, and KOAc, also gave compound (3a), albeit in lower yields (Table 1, Entries 4–7). Furthermore, as revealed in Table 1, other solvents such as DMSO, DMF, and DMAc were investigated (Table 1, Entries 8–10), with DMSO found to be optimal, giving (3a) in 60% yield. Moreover, when TCAB19 was used as the additive, the product (3a) could be obtained in 61% yield (Table 1, Entry 11).
Finally, the reaction temperature was investigated and after a brief screening of different temperatures, we found that the highest yield was achieved at 130 °C (Table 1, Entries 11–13).

With optimized reaction conditions in hand, we next explored the scope and generality of the method. Pleasingly, N-allyl-N-benzylacrylamides substituted with an electron-donating group (Me) or an electron-withdrawing group (Cl) on the aromatic ring were tolerated, leading to the formation of target products 3b, 3c in moderate yields (Table 2, Entries 2 and 3). To further study the scope of this method, the reactions of different N-allyl-N-phenylacrylamides with 3-iodo-cyclohex-2-en-1-one (2a) were investigated (Table 2, Entries 4–8). Gratifyingly, all the reactions proceeded smoothly and gave the expected products in moderate yields. A substrate with an electron-withdrawing substituent on the phenyl ring gave a slightly higher yield than that with an electron-donating substituent (Table 2, Entries 7 and 8).

The generality of the reaction was also examined by the employment of 3-iodo-5,5-dimethylcyclohex-2-en-1-one, and the results are given in Table 2 (Entries 9–15). All the reactions proceeded smoothly under the standard conditions, affording the desired products 3i-o in moderate yields (35%–63%). The crystal structure of 3K is shown in Figure 1 with the relevant crystal data given in Table 3. We can see from the crystal structure that the five and six member rings are transfused.

A plausible reaction mechanism has been proposed (Scheme 2). Initial oxidative addition and carbo-palladation gives Intermediate I, which undergoes an intramolecular tandem reaction to provide Intermediate III. Considering a standard Heck mechanism, the Intermediate III does not allow for syn-β-hydride elimination from C-1-H. Alternatively, C-2-H in the appropriate configuration can be eliminated to give Intermediate IV. Subsequent isomerization would lead to the observed product.

Scheme 1. Previous work and our synthetic strategy: (a) Liu’s work, (b) McGlacken’s work, (c) Huang’s work, and (d) this work.
Table 1. Optimization of the reaction conditions.\textsuperscript{a}

| Entry | Base       | Solvent | Pd source         | Additive | Temp | Reaction time (h) | Yield (%)\textsuperscript{b} |
|-------|------------|---------|-------------------|----------|------|-------------------|-----------------------------|
| 1     | K\textsubscript{2}CO\textsubscript{3} | Toluene | Pd(OAc)\textsubscript{2} | TBAB     | 130  | 12                | 36                          |
| 2     | K\textsubscript{2}CO\textsubscript{3} | Toluene | PdCl\textsubscript{2}  | TBAB     | 130  | 12                | 30                          |
| 3     | K\textsubscript{2}CO\textsubscript{3} | Toluene | PdCl\textsubscript{2}(PPh\textsubscript{3})\textsubscript{2} | TBAB     | 130  | 12                | 42                          |
| 4     | DABCO      | Toluene | PdCl\textsubscript{2}(PPh\textsubscript{3})\textsubscript{2} | 12       | 130  | 12                | 40                          |
| 5     | n-Bu\textsubscript{3}N | Toluene | PdCl\textsubscript{2}(PPh\textsubscript{3})\textsubscript{2} | 12       | 130  | 12                | 35                          |
| 6     | LiOH       | Toluene | PdCl\textsubscript{2}(PPh\textsubscript{3})\textsubscript{2} | TBAB     | 130  | 12                | 12                          |
| 7     | K\textsubscript{2}OAc | Toluene | PdCl\textsubscript{2}(PPh\textsubscript{3})\textsubscript{2} | TBAB     | 130  | 12                | 39                          |
| 8     | K\textsubscript{2}CO\textsubscript{3} | DMSO    | PdCl\textsubscript{2}(PPh\textsubscript{3})\textsubscript{2} | TBAB     | 130  | 12                | 60                          |
| 9     | K\textsubscript{2}CO\textsubscript{3} | DMF     | PdCl\textsubscript{2}(PPh\textsubscript{3})\textsubscript{2} | TBAB     | 130  | 12                | 56                          |
| 10    | K\textsubscript{2}CO\textsubscript{3} | DMAc    | PdCl\textsubscript{2}(PPh\textsubscript{3})\textsubscript{2} | TCAB     | 130  | 12                | 34                          |
| 11    | K\textsubscript{2}CO\textsubscript{3} | DMSO    | PdCl\textsubscript{2}(PPh\textsubscript{3})\textsubscript{2} | TCAB     | 130  | 12                | 61                          |
| 12    | K\textsubscript{2}CO\textsubscript{3} | DMSO    | PdCl\textsubscript{2}(PPh\textsubscript{3})\textsubscript{2} | TCAB     | 140  | 12                | 50                          |

\textsuperscript{a}Reaction conditions: 1a (0.30 mmol), 2a (0.36 mmol), Pd (5.0 mol%), additive (10.0 mol%), base (0.36 mmol), solvent (5.0 mL).

\textsuperscript{b}Yield of isolated product.

Table 2. Substrate scope of the reaction.\textsuperscript{a}

| Entry | R          | R\textsuperscript{1} | Product | Reaction time (h) | Yield (%)\textsuperscript{b} |
|-------|------------|----------------------|---------|-------------------|-----------------------------|
| 1     | Bn         | H                    | 3a      | 12                | 67                          |
| 2     | p-MeC\textsubscript{6}H\textsubscript{4}CH\textsubscript{2} | H       | 3b      | 12                | 62                          |
| 3     | p-ClC\textsubscript{6}H\textsubscript{4}CH\textsubscript{2} | H       | 3c      | 12                | 64                          |
| 4     | C\textsubscript{6}H\textsubscript{5}  | H        | 3d      | 12                | 47                          |
| 5     | p-MeC\textsubscript{6}H\textsubscript{5} | H        | 3e      | 12                | 46                          |
| 6     | m-MeC\textsubscript{6}H\textsubscript{5} | H        | 3f      | 12                | 42                          |
| 7     | p-MeOC\textsubscript{6}H\textsubscript{5} | H        | 3g      | 12                | 32                          |
| 8     | p-ClC\textsubscript{6}H\textsubscript{5} | H        | 3h      | 12                | 53                          |
| 9     | C\textsubscript{6}H\textsubscript{5}  | Me       | 3i      | 12                | 46                          |
| 10    | p-MeC\textsubscript{6}H\textsubscript{5} | Me       | 3j      | 12                | 48                          |
| 11    | p-MeOC\textsubscript{6}H\textsubscript{5} | Me       | 3k      | 12                | 35                          |
| 12    | p-ClC\textsubscript{6}H\textsubscript{5} | Me       | 3l      | 12                | 55                          |
| 13    | Bn         | Me       | 3m      | 12                | 58                          |
| 14    | p-MeC\textsubscript{6}H\textsubscript{4}CH\textsubscript{2} | Me       | 3n      | 12                | 52                          |
| 15    | p-ClC\textsubscript{6}H\textsubscript{4}CH\textsubscript{2} | Me       | 3o      | 12                | 63                          |

\textsuperscript{a}Reaction conditions: 1 (0.30 mmol), 2 (0.36 mmol), PdCl\textsubscript{2}(PPh\textsubscript{3})\textsubscript{2} (5.0 mol%), TCAB (10.0 mol%), and K\textsubscript{2}CO\textsubscript{3} (0.36 mmol), DMSO (5.0 mL).

\textsuperscript{b}Yield of isolated product.

Conclusion

In summary, we have developed an efficient method for the synthesis of polycyclic oxindole moieties in the presence of a PdCl\textsubscript{2}(PPh\textsubscript{3})\textsubscript{2} catalytic system under mild conditions. The ready accessibility of the starting materials, the wide range of compatibility of the substrates, and the generality of this make the reported process advantageous.
All starting materials and reagents are commercially available and were used without further purification. The organic solvents used in this study were dried over appropriate drying agents and distilled prior to use. Melting points were determined with an X-4 apparatus and are uncorrected. IR spectra were recorded with a Shimadzu FTIR-8300 spectrophotometer. High-resolution mass spectra (HRMS) were obtained on a waters G2-Xs with an ESI source (Waters, Manchester, UK). The 1H NMR spectra were determined in CDCl3 using TMS as an internal reference with a Bruker 500 FT NMR spectrometer operating at 500 MHz. The 13C NMR spectra were determined in CDCl3 using TMS as an internal reference with a Bruker 500 FT NMR spectrometer operating at 125 MHz.

Polycyclic Oxindoles 3 general procedure

The 1,6-diene 1 (0.30 mmol), vinyl iodide 2 (0.36 mmol), 

\[ \text{PdCl}_2(PPh_3)_2 \] (5 mol%, 0.015 mmol, 10.5 mg), TCAB (10 mol%, 0.03 mmol, 8.33 mg), and K₂CO₃ (0.36 mmol, 49.68 mg) were stirred in DMSO (5.0 mL) at 130 °C in a 20 mL tube under a N₂ atmosphere. When the reaction was complete (monitoring by TLC), the mixture was cooled to room temperature. The reaction was quenched with HCl (5%, 10 mL) and extracted with Et₂O (3 × 10 mL). The combined organic layers were dried over anhydrous MgSO₄ and then evaporated in vacuum. The residue was purified by column chromatography on silica gel (hexanes/ethyl acetate) to afford the corresponding polycyclic oxindoles 3.

2-Benzyl-3,3a,4,6,7,8,9,9a-octahydro-1H-benzo[\( f \)]isoindole-1,5(2H)-dione (3a): Yield: 59.3 mg (67%); white solid; m.p. 218 °C–224 °C (PE/EtOAc); IR (film) 2984, 2938, 2902, 1700, 1672, 1510, 1087, 732 cm⁻¹; 1H NMR (500 MHz, CDCl₃): δ 7.35–7.26 (m, 3H), 7.23–7.22 (m, 2H), 4.51 (d, \( J = 15 \) Hz, 1H), 4.44 (d, \( J = 15 \) Hz, 1H), 3.34–3.31 (m, 1H), 3.03 (t, \( J = 9.5 \) Hz, 1H), 2.71–2.69 (m, 1H), 2.64–2.59 (m, 1H), 2.47–2.35 (m, 6H), 2.19–2.13 (m, 1H), 2.02–1.91 (m, 4H); 13C NMR (125 MHz, CDCl₃): δ 201.0, 174.6, 136.6, 132.1, 128.8, 128.1, 127.7, 50.9, 64.6, 43.8, 37.8, 37.4, 32.2, 31.6, 26.5, 22.3; HRMS (ESI): m/z [M+H]⁺ calcd for C₁₉H₂₂NO₂: 296.1651; found: 296.1647.

2-(4-Methylbenzyl)-3,3a,4,6,7,8,9,9a-octahydro-1H-benzo[\( f \)]isoindole-1,5(2H)-dione (3b): Yield: 57.5 mg (62%); white solid; m.p. 218 °C–224 °C (PE/EtOAc); IR (film) 2984, 2938, 2902, 1700, 1672, 1510, 1087, 732 cm⁻¹; 1H NMR (500 MHz, CDCl₃): δ 7.35–7.26 (m, 3H), 7.23–7.22 (m, 2H), 4.51 (d, \( J = 15 \) Hz, 1H), 4.44 (d, \( J = 15 \) Hz, 1H), 3.34–3.31 (m, 1H), 3.03 (t, \( J = 9.5 \) Hz, 1H), 2.71–2.69 (m, 1H), 2.64–2.59 (m, 1H), 2.47–2.35 (m, 6H), 2.19–2.13 (m, 1H), 2.02–1.91 (m, 4H); 13C NMR (125 MHz, CDCl₃): δ 201.0, 174.6, 136.6, 132.1, 129.4, 128.1, 127.7, 50.9, 46.3, 43.8, 37.8, 37.4, 32.2, 31.6, 26.5, 22.3; HRMS (ESI): m/z [M+H]⁺ calcd for C₂₀H₂₄NO₂: 310.1807; found: 310.1809.
2-(4-Chlorobenzyl)-3,3a,4,6,7,8,9,9a-octahydro-1H-benzo[f]isoindole-1,5(2H)-dione (3c): Yield: 63.2 mg (62%); white solid; m.p. 182 °C–186 °C (PE/EtOAc); IR (film) 2943, 2928, 2895, 1702, 1656, 1490, 1135, 772 cm$^{-1}$; $^1$H NMR (500 MHz, CDCl$_3$): $\delta$ 7.30 (d, $J = 8.5$ Hz, 2H), 7.17 (d, $J = 8.5$ Hz, 2H), 4.48 (d, $J = 15$ Hz, 1H), 4.40 (d, $J = 15$ Hz, 1H), 3.34–3.32 (m, 1H), 3.03 (t, $J = 10$ Hz, 1H), 2.73–2.70 (m, 1H), 2.64–2.59 (m, 1H), 2.48–2.36 (m, 5H), 2.18–2.12 (m, 1H), 2.04–1.89 (m, 4H); $^{13}$C NMR (125 MHz, CDCl$_3$): $\delta$ 198.6, 174.6, 156.3, 135.2, 133.5, 132.0, 129.4, 128.9, 50.9, 45.9, 43.7, 37.8, 37.4, 32.1, 31.6, 26.5, 22.3; HRMS (ESI): $m/z$ [M+H]$^+$ calcd for C$_{19}$H$_{21}$ClNO$_2$: 330.1261; found: 330.1257.

2-Phenyl-3,3a,4,6,7,8,9,9a-octahydro-1H-benzo[f]isoindole-1,5(2H)-dione (3d): Yield: 39.6 mg (47%); white solid; m.p. 221 °C–227 °C (PE/EtOAc); IR (film) 2943, 2895, 1702, 1656, 1490, 1135, 772 cm$^{-1}$; $^1$H NMR (500 MHz, CDCl$_3$): $\delta$ 7.63–7.61 (m, 2H), 7.39–7.36 (m, 2H), 7.16–7.13 (m, 1H), 3.94–3.91 (m, 1H), 3.64 (t, $J = 9.5$ Hz, 1H), 2.88–2.85 (m, 1H), 2.68–2.63 (m, 1H), 2.51–2.32 (m, 6H), 2.11–1.96 (m, 1H); $^{13}$C NMR (125 MHz, CDCl$_3$): $\delta$ 198.6, 174.6, 156.3, 135.2, 133.5, 132.0, 129.4, 128.9, 50.9, 45.9, 43.7, 37.8, 37.4, 32.1, 31.6, 26.5, 22.3; HRMS (ESI): $m/z$ [M+H]$^+$ calcd for C$_{18}$H$_{20}$NO$_2$: 282.1494; found: 282.1496.

2-(p-Tolyl)-3,3a,4,6,7,8,9,9a-octahydro-1H-benzo[f]isoindole-1,5(2H)-dione (3e): Yield: 40.7 mg (46%); white solid; m.p. 193 °C–196 °C (PE/EtOAc); IR (film) 2943, 2928, 2895, 1702, 1656, 1490, 1135, 772 cm$^{-1}$; $^1$H NMR (500 MHz, CDCl$_3$): $\delta$ 7.50 (d, $J = 8.5$ Hz, 2H), 7.19 (d, $J = 8.5$ Hz, 2H), 3.92–3.89 (m, 1H), 3.63 (t, $J = 9.5$ Hz, 1H), 2.88–2.86 (m, 1H), 2.68–2.64 (m, 1H), 2.50–2.31 (m, 9H), 2.12–1.98 (m, 4H); $^{13}$C NMR (125 MHz, CDCl$_3$): $\delta$ 198.7, 174.6, 156.3, 135.2, 133.9, 131.9, 129.4, 119.6, 53.0, 44.9, 37.8, 36.5, 32.1, 31.6, 26.5, 22.3, 20.9; HRMS (ESI): $m/z$ [M+H]$^+$ calcd for C$_{19}$H$_{22}$NO$_2$: 296.1651; found: 296.1655.

2-(m-Tolyl)-3,3a,4,6,7,8,9,9a-octahydro-1H-benzo[f]isoindole-1,5(2H)-dione (3f): Yield: 37.2 mg (42%); white solid; m.p. 200 °C–206 °C (PE/EtOAc); IR (film) 2952, 2852, 1687, 1654, 1322, 1206, 738 cm$^{-1}$; $^1$H NMR (500 MHz, CDCl$_3$): $\delta$ 7.45–7.39 (m, 2H), 7.25–7.24 (m, 1H), 6.97–6.95 (m, 1H), 3.92–3.89 (m, 1H), 3.65–3.61 (m, 1H), 2.87–2.84 (m, 1H), 2.66–2.62 (m, 1H), 2.51–2.32 (m, 9H), 2.10–1.96 (m, 4H); $^{13}$C NMR (125 MHz, CDCl$_3$): $\delta$ 198.8, 173.6, 165.4, 137.1, 133.9, 131.9, 129.6, 119.6, 53.0, 44.9, 37.8, 36.5, 32.1, 31.6, 26.5, 22.3, 20.9; HRMS (ESI): $m/z$ [M+H]$^+$ calcd for C$_{19}$H$_{22}$NO$_2$: 296.1651; found: 296.1655.

2-(4-Methoxyphenyl)-3,3a,4,6,7,8,9,9a-octahydro-1H-benzo[f]isoindole-1,5(2H)-dione (3g): Yield: 29.9 mg (32%); white solid; m.p. 205 °C–213 °C (PE/EtOAc); IR (film) 2961, 2928, 2904, 1688, 1680, 1659, 1619, 835 cm$^{-1}$; $^1$H NMR (500 MHz, CDCl$_3$): $\delta$ 7.52 (d, $J = 9.0$ Hz, 2H),

Scheme 2. A plausible reaction mechanism.
2-(4-Chlorophenyl)-3,3a,4,6,7,8,9a-octahydro-1H-benzo[f]isoindole-1,5(2H)-dione (3h): Yield: 50.1 mg (53%); white solid; m.p. 195°C–198°C (PE/EtOAc); IR (film) 2962, 2920, 2885, 1687, 1662, 1619, 1169, 757 cm⁻¹; ¹H NMR (500 MHz, CDCl₃): δ 7.48 (d, J = 11 Hz, 2H), 7.17 (d, J = 10.5 Hz, 2H), 3.90–3.87 (m, 1H), 3.64–3.59 (m, 1H), 2.89–2.87 (m, 1H), 2.63–2.58 (m, 1H), 2.41–2.25 (m, 9H), 2.10–2.06 (m, 2H), 1.07 (s, 3H), 1.00 (s, 3H); ¹³C NMR (125 MHz, CDCl₃): δ 198.8, 173.5, 153.8, 130.8, 129.4, 119.7, 53.1, 54.0, 45.7, 45.1, 36.7, 33.2, 29.4, 27.1, 26.3, 20.9; HRMS (ESI): m/z [M+H]⁺ calculated for C₂₁H₂₅ClNO₂: 358.1512; found: 358.1512.

7,7-Dimethyl-2-phenyl-3,3a,4,6,7,8,9a-octahydro-1H-benzo[f]isoindole-1,5(2H)-dione (3i): Yield: 42.8 mg (46%); white solid; m.p. 242°C–246°C (PE/EtOAc); IR (film) 2962, 2920, 2885, 1687, 1662, 1619, 1169, 757 cm⁻¹; ¹H NMR (500 MHz, CDCl₃): δ 7.64–7.62 (m, 2H), 7.41–7.37 (m, 2H), 7.18–7.15 (m, 1H), 3.96–3.93 (m, 1H), 3.68–3.64 (m, 1H), 2.92–2.90 (m, 1H), 2.64–2.61 (m, 1H), 2.43–2.37 (m, 6H), 2.12–2.09 (m, 2H), 1.10 (s, 3H), 1.03 (s, 3H); ¹³C NMR (125 MHz, CDCl₃): δ 198.8, 173.7, 153.8, 139.6, 130.7, 129.4, 124.4, 119.6, 53.0, 51.4, 45.7, 45.1, 36.6, 33.1, 32.3, 29.4, 27.1, 26.3, 20.9; HRMS (ESI): m/z [M+H]⁺ calculated for C₂₁H₂₅NO₂: 310.1807; found: 310.1811.

7,7-Dimethyl-2-(p-toly)-3,3a,4,6,7,8,9a-octahydro-1H-benzo[f]isoindole-1,5(2H)-dione (3j): Yield: 46.5 mg (48%); white solid; m.p. 195°C–200°C (PE/EtOAc); IR (film) 2952, 2922, 1678, 1653, 1615, 1426, 1367, 799 cm⁻¹; ¹H NMR (500 MHz, CDCl₃): δ 7.48 (d, J = 11 Hz, 2H), 7.17 (d, J = 10.5 Hz, 2H), 3.90–3.87 (m, 1H), 3.64–3.59 (m, 1H), 2.89–2.87 (m, 1H), 2.63–2.58 (m, 1H), 2.41–2.25 (m, 9H), 2.10–2.06 (m, 2H), 1.07 (s, 3H), 1.00 (s, 3H); ¹³C NMR (125 MHz, CDCl₃): δ 198.8, 173.5, 153.8, 130.8, 129.4, 119.7, 53.1, 51.4, 45.7, 45.1, 36.7, 33.2, 32.3, 29.4, 27.1, 26.3, 20.9; HRMS (ESI): m/z [M+H]⁺ calculated for C₂₁H₂₅NO₂: 324.1964; found: 324.1961.

2-(4-Methoxyphenyl)-2-(4-Methoxyphenyl)-7,7-dimethyl-3,3a,4,6,7,8,9a-octahydro-1H-benzo[f]isoindole-1,5(2H)-dione (3k): Yield: 50.7 mg (52%); white solid; m.p. 178°C–180°C (PE/EtOAc); IR (film) 2969, 2934, 2875, 1687, 1654, 1507, 1224, 773 cm⁻¹; ¹H NMR (500 MHz, CDCl₃): δ 6.74–6.72 (m, 2H), 6.41–6.37 (m, 2H), 3.96–3.93 (m, 1H), 3.68–3.64 (m, 1H), 2.92–2.90 (m, 1H), 2.64–2.61 (m, 1H), 2.43–2.37 (m, 6H), 2.12–2.09 (m, 2H), 1.10 (s, 3H), 1.03 (s, 3H); ¹³C NMR (125 MHz, CDCl₃): δ 198.8, 173.7, 153.8, 139.6, 130.7, 129.4, 124.4, 119.6, 53.0, 51.4, 45.7, 45.1, 36.6, 33.1, 32.3, 29.4, 27.1, 26.3, 20.9; HRMS (ESI): m/z [M+H]⁺ calculated for C₂₁H₂₅NO₂: 310.1807; found: 310.1811.

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21. CCDC 2113876 (3k) contain the supplementary crystallographic data for this paper. These data can be obtained free charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/datarequest/cif