Electronic supplementary information

Co-catalyzed decarbonylative alkylation esterification of styrenes with aliphatic aldehydes and hypervalent iodine(III) reagents

Yong Peng, Yuan-Yuan Jiang, Xue-Jiao Du, Da-You Ma, Luo Yang*

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I. General information

Unless otherwise noted, all commercially available compounds were used as purchased without further purification. Dry solvents (toluene, ethyl acetate, dichloromethane, acetonitrile, chlorobenzene, fluorobenzene, trifluoromethyl benzene) were used as commercially available. Thin-layer chromatography (TLC) was performed using E. Merck silica gel 60 F254 precoated plates (0.25 mm) or Sorbent Silica Gel 60 F254 plates. The developed chromatography was analyzed by UV lamp (254 nm). High-resolution mass spectra (HRMS) were obtained from a JEOL JMS-700 instrument (ESI) or Thermo Scientific LTQ Orbitrap XL (ESI). Melting points are uncorrected. Nuclear magnetic resonance (NMR) spectra were recorded on a Bruker Avance 400 spectrometer at ambient temperature. Chemical shifts for $^1$H NMR spectra are reported in parts per million (ppm) from tetramethylsilane with the solvent resonance as the internal standard (chloroform: $\delta$ 7.26 ppm). Chemical shifts for $^{13}$C NMR spectra are reported in parts per million (ppm) from tetramethylsilane with the solvent as the internal standard (CDCl$_3$: $\delta$ 77.16 ppm). Data are reported as following: chemical shift, multiplicity (s = singlet, d = doublet, dd = doublet of doublets, t = triplet, q = quartet, m = multiplet, br = broad signal), coupling constant (Hz), and integration.

II. General experimental procedure

A general experimental procedure for the synthesis of acetates (3a-3m, 4b-4m) is described as following:

An oven-dried reaction vessel was successively charged with Co(OAc)$_2$·4H$_2$O (0.01 mmol, 5 mol%), iodo benzene diacetate (0.4 mmol, 2.0 equiv), styrene (1a, 0.2 mmol, 1.0 equiv), isobutyraldehyde (2a, 0.6 mmol, 3 equiv), trifluoromethyl benzene (1.0 mL) and di-tert-butyl
peroxide (DTBP, 0.24 mmol, 1.2 equiv). The vessel was sealed and stirred at 120 °C (oil bath temperature) for 12 h. Afterwards the resulting mixture was cooled to room temperature, the solvent was removed in vacuum. The residue was purified by column chromatography on silica gel with a mixture of dichloromethane/petroleum ether as eluent to give products 3a.

A general experimental procedure for the synthesis of benzoates (6a-6f) is described as following:

An oven-dried reaction vessel was successively charged with Co(OAc)2·4H2O (0.01 mmol, 5 mol%), iodobenzene diacetate (0.3 mmol, 1.5 equiv), PhCOOH (0.64 mmol, 3.2 equiv), Na2CO3 (0.32 mmol, 1.6 equiv), styrene (1a, 0.2 mmol, 1.0 equiv), isobutyraldehyde (2a, 0.6 mmol, 3 equiv), trifluoromethyl benzene (1.2 mL) and di-tert-butyl peroxide (DTBP, 0.4 mmol, 2 equiv). The vessel was sealed and stirred at 120 °C (oil bath temperature) for 12 h. Afterwards the resulting mixture was cooled to room temperature, the solvent was removed in vacuum. The residue was purified by column chromatography on silica gel with a mixture of dichloromethane/petroleum ether as eluent to give products 6a.

### III. Condition optimization

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Reaction conditions: 1a (0.2 mmol, 1.0 equiv), 2a (0.6 mmol, 3.0 equiv), PhI(OAc)<sub>2</sub> (0.4 mmol, 2.0 equiv), DTBP (0.6 mmol, 3.0 equiv), Cat. (0.01 mmol, x mol%), solvent (1.0 mL), stirred at y °C for 12 h under air.

**IV Spectra data of products 3a-3m, 4b-4m, 6a-6f**

**3a** 3-methyl-1-phenylbutyl acetate

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\[\text{OC} \quad \text{CH}_3 \quad \text{CH} \quad \text{CH}_2 \quad \text{CH}_2 \quad \text{CO} \]
```

The title compound was prepared according to the general procedure described above by the reaction between styrene (1a) with iodobenzene diacetate and isobutyraldehyde (2a), and purified by flash column chromatography as colorless oil (37.1 mg, 90%).

**1**H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.33 – 7.26 (m, 5H), 5.83 – 5.80 (m, 1H), 2.05 (d, J = 1.6 Hz, 3H), 1.85 (dd, J = 9.2, 16.4 Hz, 1H), 1.62 – 1.57 (m, 2H), 0.94 (t, J = 13.2 Hz, 6H). **1**C NMR (100 MHz, CDCl<sub>3</sub>) δ 170.48, 141.23, 128.54, 127.93, 126.63, 74.66, 45.54, 24.83, 22.88, 21.40. IR (cm<sup>-1</sup>): 2958, 2871, 2837, 1736, 1073, 699.

**3b** 1-(4-methoxyphenyl)-3-methylbutyl acetate

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\[\text{OC} \quad \text{CH}_3 \quad \text{CH} \quad \text{CH}_2 \quad \text{CH}_2 \quad \text{CO} \]
```

The title compound was prepared according to the general procedure described above by the reaction between 4-methoxystyrene (1b) with iodobenzene diacetate and isobutyraldehyde (2a), and purified by flash column chromatography as colorless oil (27.4 mg, 58%).

**1**H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.28 (d, J = 8.8 Hz, 2H), 6.87 (d, J = 8.4 Hz, 2H), 5.79 (dd, J = 8.4, 6.0 Hz, 1H), 3.79 (s, 3H), 2.03 (s, 3H), 1.88 – 1.81 (m, 1H), 1.62 – 1.50 (m, 2H), 0.92 (dd, J = 10.8,
The title compound was prepared according to the general procedure described above by the reaction between 4-tert-butylstyrene (1c) with iodosobenzene diacetate and isobutyraldehyde (2a), and purified by flash column chromatography as colorless oil (47.3 mg, 83%).

**1H NMR (400 MHz, CDCl₃)** δ 7.36 – 7.34 (m, 2H), 7.27 – 7.25 (m, 2H), 5.83 – 5.80 (m, 1H), 2.04 (s, 3H), 1.88 – 1.83 (m, 1H), 1.61 – 1.56 (m, 2H), 1.31 (s, 9H), 0.96 – 0.92 (m, 6H).

**13C NMR (100 MHz, CDCl₃)** δ 170.58, 150.84, 138.15, 126.42, 125.45, 74.51, 45.49, 34.66, 31.57, 31.47, 24.88, 22.92, 22.52, 21.48. IR (cm⁻¹): 2959, 2870, 1736, 1240, 812.


### (3d) 3-methyl-1-(p-tolyl)butyl acetate

The title compound was prepared according to the general procedure described above by the reaction between 4-methylstyrene (1d) with iodosobenzene diacetate and isobutyraldehyde (2a), and purified by flash column chromatography as colorless oil (34.3 mg, 78%).

**1H NMR (400 MHz, CDCl₃)** δ 7.23 (t, J = 4.4 Hz, 2H), 7.14 (d, J = 7.6 Hz, 2H), 5.80 – 5.77 (m, 1H), 2.33 (s, 3H), 2.03 (s, 3H), 1.88 – 1.82 (m, 1H), 1.61 – 1.51 (m, 2H), 0.93 (dd, J = 10.4, 6.4 Hz, 6H).


### (3e) 1-(4-(chloromethyl)phenyl)-3-methylbutyl acetate

The title compound was prepared according to the general procedure described above by the reaction between 4-chloromethylstyrene (1e) with iodosobenzene diacetate and isobutyraldehyde (2a), and purified by flash column chromatography as colorless oil (47.6 mg, 86%).
**1H NMR (400 MHz, CDCl₃) δ 7.37 – 7.32 (m, 4H), 5.82 – 5.79 (m, 1H), 4.57 (s, 2H), 2.05 (s, 3H), 1.89 – 1.80 (m, 1H), 1.59 – 1.53 (m, 2H), 0.95 – 0.92 (dd, J = 9.2, 6.4 Hz, 6H). 13C NMR (100 MHz, CDCl₃) δ 170.47, 141.59, 137.12, 128.84, 127.05, 74.30, 46.04, 45.49, 24.83, 22.88, 22.47, 21.39. IR (cm⁻¹): 2958, 2870, 1736, 1236, 826. HRMS: calcd. for C₁₄H₁₉ClO₂Na⁺ [M+Na⁺]: 277.0966; Found: 277.0951.**

(3f) 3-methyl-1-(4-(trifluoromethyl)phenyl)butyl acetate

![Chemical structure](image)

The title compound was prepared according to the general procedure described above by the reaction between 4-trifluoromethylstyrene (1f) with iodobenzene diacetate and isobutyraldehyde (2a), and purified by flash column chromatography as colorless oil (33.8 mg, 57%).

**1H NMR (400 MHz, CDCl₃) δ 7.60 (d, J = 8.4 Hz, 2H), 7.44 (d, J = 8 Hz, 2H), 5.85 – 5.81 (m, 1H), 2.07 (s, 3H), 1.89 – 1.83 (m, 1H), 1.59 – 1.52 (m, 2H), 0.97 – 0.93 (t, J = 7 Hz, 6H). 13C NMR (100 MHz, CDCl₃) δ 170.45, 145.36, 130.12 (q, J = 32.1 Hz), 127.06, 125.61 (q, J = 2.7 Hz), 124.18 (d, J = 270.6 Hz), 74.03, 45.55, 24.82, 22.91, 22.38, 21.30. IR (cm⁻¹): 2959, 2870, 1741, 1125, 825. HRMS: calcd. for C₁₄H₁₇F₃O₂Na⁺ [M+Na⁺]: 297.1073; Found: 297.1074.**

(3g) 1-(4-chlorophenyl)-3-methylbutyl acetate

![Chemical structure](image)

The title compound was prepared according to the general procedure described above by the reaction between 4-chlorostyrene (1g) with iodobenzene diacetate and isobutyraldehyde (2a), and purified by flash column chromatography as colorless oil (45.2 mg, 86%).

**1H NMR (400 MHz, CDCl₃) δ 7.32 – 7.30 (m, 2H), 7.28 (d, J = 6.4 Hz, 2H), 5.78 – 5.75 (m, 1H), 2.05 (s, 3H), 1.87 – 1.80 (m, 1H), 1.57 – 1.51 (m, 2H), 0.93 (d, J = 9.2, 6.4 Hz, 6H). 13C NMR (400 MHz, CDCl₃) δ 170.43, 139.79, 133.71, 128.77, 128.10, 73.99, 45.40, 24.82, 22.85, 22.47, 21.37. IR (cm⁻¹): 2959, 2870, 2097, 1736, 699. HRMS: calcd. For C₁₃H₁₇ClO₂Na⁺ [M+Na⁺]: 263.0809; Found: 263.0795.**

(3h) 1-(3-chlorophenyl)-3-methylbutyl acetate

![Chemical structure](image)
The title compound was prepared according to the general procedure described above by the reaction between 3-chlorostyrene (1h) with iodobenzene diacetate and isobutyraldehyde (2a), and purified by flash column chromatography as colorless oil (42.1 mg, 80%).

1H NMR (400 MHz, CDCl₃) δ 7.32 (s, 1H), 7.26 – 7.25 (m, 2H), 7.21 – 7.19 (m, 1H), 5.78 – 5.74 (m, 1H), 2.07 (s, 3H), 1.86 – 1.80 (m, 2H), 1.59 – 1.52 (m, 2H), 0.96 – 0.92 (m, 6H). 13C NMR (100 MHz, CDCl₃) δ 170.38, 143.42, 134.48, 129.86, 128.10, 126.69, 124.85, 73.93, 45.53, 24.81, 22.92, 22.39, 21.33. IR (cm⁻¹): 2959, 2871, 1736, 1158, 867. HRMS: calcd. For C₁₃H₁₇ClO₂Na⁺ [M+Na⁺]: 263.0809; Found: 263.0809.

(3i) 1-(2-chlorophenyl)-3-methylbutyl acetate

The title compound was prepared according to the general procedure described above by the reaction between 2-chlorostyrene (1i) with iodobenzene diacetate and isobutyraldehyde (2a), and purified by flash column chromatography as colorless oil (38.9 mg, 74%).

1H NMR (400 MHz, CDCl₃) δ 7.37 (dd, J = 8, 2 Hz, 1H), 7.33 (dd, J = 8, 1.2 Hz, 1H), 7.25 – 7.19 (m, 2H), 6.21 (dd, J = 9.2, 3.6 Hz, 1H), 2.10 (s, 3H), 1.80 – 1.69 (m, 2H), 1.58 – 1.54 (m, 1H), 0.97 (dd, J = 17.2, 6.4 Hz, 6H). 13C NMR (100 MHz, CDCl₃) δ 170.20, 139.65, 132.14, 129.71, 128.73, 127.17, 126.81, 71.37, 44.86, 25.11, 23.43, 21.89, 21.25. IR (cm⁻¹): 2958, 2870, 1736, 1123, 785. HRMS: calcd. For C₁₃H₁₇ClO₂Na⁺ [M+Na⁺]: 263.0809; Found: 263.0795.

(3j) 1-(4-fluorophenyl)-3-methylbutyl acetate

The title compound was prepared according to the general procedure described above by the reaction between 4-fluorostyrene (1j) with iodobenzene diacetate and isobutyraldehyde (2a), and purified by flash column chromatography as colorless oil (39.0 mg, 79%).

1H NMR (400 MHz, CDCl₃) δ 7.33 – 7.29 (m, 2H), 7.04 – 7.00 (m, 2H), 5.80 – 5.77 (m, 1H), 2.04 (s, 3H), 1.86 – 1.80 (m, 1H), 1.57 – 1.51 (m, 2H), 0.95 – 0.91 (m, 6H). 13C NMR (100 MHz, CDCl₃) δ 170.49, 162.42 (d, J = 244.6 Hz), 137.04 (d, J = 3.2 Hz), 128.47 (d, J = 8.1 Hz), 115.45 (d, J = 21.3 Hz), 74.02, 45.44, 24.83, 22.83, 22.50, 21.41. IR (cm⁻¹): 2959, 2870, 1740, 1158, 837. HRMS: calcd. For C₁₃H₁₇FO₂Na⁺ [M+Na⁺]: 247.1105; Found: 247.1091.

(3k) 1-(4-bromophenyl)-3-methylbutyl acetate
The title compound was prepared according to the general procedure described above by the reaction between 4-boromostyrene (1k) with iodobenzene diacetate and isobutyraldehyde (2a), and purified by flash column chromatography as colorless oil (45.4 mg, 74%).

\( ^1H \text{NMR} \) (400 MHz, CDCl\(_3\)) \( \delta \) 7.46 (d, \( J = 8.4 \) Hz, 2H), 7.21 (d, \( J = 8.4 \) Hz, 2H), 5.77 - 5.73 (m, 1H), 2.05 (s, 3H), 1.88 - 1.78 (m, 1H), 1.59 - 1.50 (m, 2H), 0.93 (dd, \( J = 2 \), 6.4 Hz, 6H). \(^{13}C \text{NMR} \) (100 MHz, CDCl\(_3\)) \( \delta \) 170.40, 140.30, 131.71, 128.41, 121.82, 74.01, 45.35, 24.79, 22.84, 22.45, 21.34. IR (cm\(^{-1}\)): 2958, 2870, 1736, 1299, 817.

HRMS: calcd. For \( C_{13}H_{17}BrO_2Na^+ \) [M+Na]\(^{+}\): 307.0304; Found: 307.0296.

(3l) 1-(3-bromophenyl)-3-methylbutyl acetate

The title compound was prepared according to the general procedure described above by the reaction between 3-boromostyrene (1l) with iodobenzene diacetate and isobutyraldehyde (2a), and purified by flash column chromatography as colorless oil (47.9 mg, 78%).

\( ^1H \text{NMR} \) (400 MHz, CDCl\(_3\)) \( \delta \) 7.47 (t, \( J = 1.8 \) Hz, 1H), 7.42 - 7.39 (m, 1H), 7.25 - 7.18 (m, 2H), 5.76 - 5.73 (m, 1H), 2.07 (s, 3H), 1.87 - 1.80 (m, 1H), 1.58 - 1.50 (m, 2H), 0.94 (dd, \( J = 4.8 \), 6.8 Hz, 6H). \(^{13}C \text{NMR} \) (100 MHz, CDCl\(_3\)) \( \delta \) 170.37, 143.67, 131.04, 130.16, 129.59, 125.33, 122.68, 73.86, 45.54, 24.81, 22.92, 22.38, 21.34. IR (cm\(^{-1}\)): 2958, 2870, 1739, 782. HRMS: calcd. For \( C_{13}H_{17}BrO_2Na^+ \) [M+Na]\(^{+}\): 307.0304; Found: 307.0296.

(3m) 3-methyl-1,2-diphenylbutyl acetate

The title compound was prepared according to the general procedure described above by the reaction between trans-1,2-diphenylethene (1m) with iodobenzene diacetate and isobutyraldehyde (2a), and purified by flash column chromatography as colorless oil (47.9 mg, 78%, d.r. = 1.7 : 1).

\( ^1H \text{NMR} \) (400 MHz, CDCl\(_3\)) \( \delta \) 7.24 - 7.07 (m, 9H), 6.93 - 6.91 (m, 1H), 6.25 (dd, \( J = 17.6 \), 7.6 Hz, 1H), 3.10 - 2.78 (m, 1H), 2.80 (t, \( J = 7.2 \) Hz, 0.37×1H), 2.28 - 2.20 (m, 0.6×1H), 2.08 (s, 1.88×1H), 1.90 (s, 1.12×1H), 1.88 - 1.83 (m, 0.37×1H), 0.87 (m, 6H). \(^{13}C \text{NMR} \) (100 MHz, CDCl\(_3\)) \( \delta \) 170.33, 170.24, 140.08, 139.42, 138.85, 137.93, 130.31, 129.84, 128.24, 127.86, 127.78, 127.66, 127.64, 127.56, 126.93, 126.44, 76.53, 76.18, 58.36, 57.01, 28.74, 28.44, 21.88, 21.86, 21.44, 21.10, 19.29, 18.44. IR (cm\(^{-1}\)): 2960, 2930, 2873, 1739, 1234, 761. HRMS: calcd. For \( C_{19}H_{22}O_2Na^+ \) [M+Na]\(^{+}\): 305.1512; Found: 305.1503.

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The title compound was prepared according to the general procedure described above by the reaction between styrene (1a) with iodobenzene diacetate and pivaldehyde (2b), and purified by flash column chromatography as colorless oil (37.8 mg, 86%).

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.33 – 7.24 (m, 5H), 5.88 – 5.85 (m, 1H), 2.03 (s, 3H), 1.99 – 1.93 (m, 1H), 1.63 – 1.61 (m, 1H), 0.95 (s, 9H). $^13$C NMR (100 MHz, CDCl$_3$) $\delta$ 170.42, 142.56, 128.58, 127.80, 126.45, 74.11, 50.05, 30.60, 30.02, 21.60.

IR (cm$^{-1}$): 3030, 2955, 2870, 1739.


(4c) 3-methyl-1-phenylpentyl acetate

The title compound was prepared according to the general procedure described above by the reaction between styrene (1a) with iodobenzene diacetate and 2-methylbutanal (2c), and purified by flash column chromatography as colorless oil (36.5 mg, 75%, d.r. = 1.2 : 1).

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.34 – 7.25 (m, 5H), 5.86 – 5.80 (m, 1H), 2.05 (d, $J$ = 6.4 Hz, 3H), 1.76 (t, $J$ = 7.0 Hz, 1H), 1.50 – 1.12 (m, 4H), 0.93 – 0.81 (m, 6H). $^13$C NMR (100 MHz, CDCl$_3$) $\delta$ 170.56, 170.50, 141.59, 141.03, 128.56, 128.01, 127.89, 126.84, 126.52, 126.17, 74.89, 74.36, 43.72, 43.08, 31.05, 29.78, 29.33, 21.48, 21.42, 19.36, 19.05, 11.26, 11.10. IR (cm$^{-1}$): 2964, 2930, 2875, 1739, 1236, 698.

HRMS: calcd. For C$_{14}$H$_{20}$O$_2$Na$^+$ [M+Na]$^+$: 257.1512; Found: 257.1509.

(4d) 3-ethyl-1-phenylpentyl acetate

The title compound was prepared according to the general procedure described above by the reaction between styrene (1a) with iodobenzene diacetate and 2-ethylbutanal (2d), and purified by flash column chromatography as colorless oil (41.6 mg, 81%).

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.34 – 7.25 (m, 5H), 5.81 (dd, $J$ = 8.8, 6.0 Hz, 1H), 2.05 (s, 3H), 1.91 – 1.84 (m, 1H), 1.65 (dd, $J$ = 13.2, 6.0 Hz, 1H), 1.40 – 1.28 (m, 4H), 1.22 (dd, $J$ = 12.4, 6.0 Hz, 1H), 0.83 (dt, $J$ = 19.2, 7.2 Hz, 6H). $^13$C NMR (100 MHz, CDCl$_3$) $\delta$ 170.53, 141.36, 128.54, 127.93, 126.66, 74.70, 39.98, 36.79, 25.48, 25.15, 21.42, 10.67, 10.51. IR (cm$^{-1}$): 2963, 2932, 2875, 1736, 1236, 699.

HRMS: calcd. For C$_{15}$H$_{22}$O$_2$Na$^+$ [M+Na]$^+$: 257.1512; Found: 257.1509.

(4e) 3-ethyl-1-phenylheptyl acetate
The title compound was prepared according to the general procedure described above by the reaction between styrene (1a) with iodobenzene diacetate and 2-ethylhexanal (2e), and purified by flash column chromatography as colorless oil (47.9 mg, 84%, d.r. = 1.2 : 1).

\[ ^1H\text{ NMR} \ (400\text{ MHz}, \text{CDCl}_3) \delta \ 7.33 - 7.25 \ (m, 5H), \ 5.81 \ (t, \ J = 7.0\text{ Hz}, 1H), \ 2.05 \ (s, 3H), \ 1.89 - 1.85 \ (m, 1H), \ 1.65 \ (d, \ J = 8.8\text{ Hz}, 1H), \ 1.35 - 1.21 \ (m, 9H), \ 0.89 - 0.79 \ (m, 6H). \]

\[ ^{13}C\text{ NMR} \ (100\text{ MHz}, \text{CDCl}_3) \delta \ 170.52, \ 141.40, \ 141.34, \ 128.53, \ 127.92, \ 126.68, \ 126.64, \ 74.77, \ 74.72, \ 40.45, \ 40.40, \ 35.38, \ 32.87, \ 32.59, \ 28.72, \ 28.58, \ 26.03, \ 25.66, \ 23.16, \ 23.10, \ 14.22, \ 10.62, \ 10.47. \]

IR (cm\(^{-1}\)): 2963, 2932, 2875, 1739, 1235, 699.

HRMS: calcd. For C\(_{17}\)H\(_{26}\)O\(_2\)Na\(^+\) [M+Na\(^+\)]: 285.1825; Found: 285.1822.

(4f) 2-cyclohexyl-1-phenylethyl acetate

The title compound was prepared according to the general procedure described above by the reaction between styrene (1a) with iodobenzene diacetate and cyclohexanecarboxaldehyde (2f), and purified by flash column chromatography as colorless oil (43.0 mg, 80%).

\[ ^1H\text{ NMR} \ (400\text{ MHz}, \text{CDCl}_3) \delta \ 7.33 - 7.26 \ (m, 5H), \ 5.84 \ (dd, \ J = 8.8, 6.0\text{ Hz}, 1H), \ 2.05 \ (s, 3H), \ 1.88 - 1.81 \ (m, 1H), \ 1.76 - 1.61 \ (m, 6H), \ 1.29 - 1.14 \ (m, 4H), \ 1.01 - 0.90 \ (m, 2H). \]

\[ ^{13}C\text{ NMR} \ (100\text{ MHz}, \text{CDCl}_3) \delta \ 170.52, \ 141.37, \ 128.54, \ 127.90, \ 126.63, \ 74.11, \ 44.20, \ 34.19, \ 33.60, \ 33.09, \ 26.58, \ 26.27, \ 26.18, \ 21.45. \]

IR (cm\(^{-1}\)): 3064, 2923, 2851, 1736, 1237, 699. HRMS: calcd. For C\(_{16}\)H\(_{22}\)O\(_2\)Na\(^+\) [M+Na\(^+\)]: 269.1512; Found: 269.1503.

(4g) 2-cyclopentyl-1-phenylethyl acetate

The title compound was prepared according to the general procedure described above by the reaction between styrene (1a) with iodobenzene diacetate and cyclopentanecarbaldehyde (2g), and purified by flash column chromatography as colorless oil (37.6 mg, 81%).

\[ ^1H\text{ NMR} \ (400\text{ MHz}, \text{CDCl}_3) \delta \ 7.34 - 7.28 \ (m, 5H), \ 5.78 - 5.74 \ (m, 1H), \ 2.06 \ (s, 3H), \ 1.99 - 1.92 \ (m, 1H), \ 1.81 - 1.67 \ (m, 4H), \ 1.59 - 1.41 \ (m, 4H), \ 1.20 - 1.08 \ (m, 2H). \]

\[ ^{13}C\text{ NMR} \ (100\text{ MHz}, \text{CDCl}_3) \delta \ 170.52, \ 141.37, \ 128.54, \ 127.90, \ 126.63, \ 74.11, \ 44.20, \ 34.19, \ 33.60, \ 33.09, \ 26.58, \ 26.27, \ 26.18, \ 21.45. \]

IR (cm\(^{-1}\)): 3064, 2950, 2867, 1736, 1237, 699. HRMS: calcd. For C\(_{15}\)H\(_{20}\)O\(_2\)Na\(^+\) [M+Na\(^+\)]: 255.1356; Found: 255.1370.
(4h) 4,6,6-trimethyl-1-phenylheptyl acetate

The title compound was prepared according to the general procedure described above by the reaction between styrene (1a) with iodobenzene diacetate and 3,5,5-trimethylhexanal (2h), and purified by flash column chromatography as colorless oil (40.9 mg, 78%, d.r. = 1.2 : 1).

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.34 –7.28 (m, 5H), 5.72 – 5.64 (m, 1H), 2.06 (s, 3H), 1.92 – 1.73 (m, 2H), 1.49 – 1.42 (m, 1H), 1.23 – 1.14 (m, 2H), 1.10 – 1.01 (m, 2H), 0.90 – 0.85 (m, 12H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 170.56, 141.06, 140.94, 128.53, 127.96, 126.73, 126.67, 126.65, 76.62, 76.48, 51.15, 50.69, 38.33, 35.12 (d, $J = 5.4$ Hz), 34.07, 31.31, 31.16, 30.13, 29.19, 29.11, 27.73, 25.62, 24.22, 22.64, 22.61, 21.43. IR (cm$^{-1}$): 3064, 2950, 2867, 1736, 1236, 699. HRMS: calcd. For C$_{18}$H$_{28}$O$_2$Na$^+ [M+Na]$^+$: 299.1982; Found: 299.1987.

(4i) 5-methyl-1-phenylhexyl acetate

The title compound was prepared according to the general procedure described above by the reaction between styrene (1a) with iodobenzene diacetate and 4-methylpentanal (2i), and purified by flash column chromatography as colorless oil (33.7 mg, 72%).

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.34 – 7.28 (m, 5H), 5.70 (dd, $J = 7.2$, 6.4 Hz, 1H), 2.07 (s, 3H), 1.95 – 1.85 (m, 1H), 1.81 – 1.72 (m, 1H), 1.57 – 1.49 (m, 1H), 1.26 – 1.19 (m, 1H), 1.13 – 1.06 (m, 1H), 0.86 (dd, $J = 6.4$, 2.4 Hz, 6H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 170.56, 141.01, 128.54, 127.95, 126.68, 76.56, 34.66, 34.35, 27.98, 22.63, 22.60, 21.44. IR (cm$^{-1}$): 3064, 2950, 2870, 1736, 1237, 700. HRMS: calcd. For C$_{14}$H$_{20}$O$_2$Na$^+ [M+Na]$^+$: 243.1356; Found: 243.1366.

(4j) 4,4-dimethyl-1-phenylpentyl acetate

The title compound was prepared according to the general procedure described above by the reaction between styrene (1a) with iodobenzene diacetate and 3,3-dimethylbutanal (2j), and purified by flash column chromatography as colorless oil (29.6 mg, 63%).

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.35 – 7.27 (m, 5H), 5.69 – 5.65 (m, 1H), 2.07 (s, 3H), 1.92 – 1.83 (m, 1H), 1.79 – 1.71 (m, 1H), 1.26 – 1.21 (m, 1H), 1.10 – 0.94 (m, 1H), 0.85 (s, 9H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 170.57, 141.00, 128.55, 127.97, 126.72, 76.99, 39.63, 31.68, 30.19, 29.39, 21.45. IR (cm$^{-1}$): 3064, 2956, 2870, 1736, 1236, 699. HRMS: calcd. For C$_{15}$H$_{22}$O$_2$Na$^+ [M+Na]$^+$: 257.1512; Found: 257.1499.
(4k) 1-phenylbutyl acetate

The title compound was prepared according to the general procedure described above by the reaction between styrene (1a) with iodobenzene diacetate and propionaldehyde (2k), and purified by flash column chromatography as colorless oil (20.7 mg, 54%).

1H NMR (400 MHz, CDCl3) δ 7.34 – 7.27 (m, 5H), 5.76 – 5.72 (m, 1H), 2.06 (s, 3H), 1.93 – 1.86 (m, 1H), 1.77 – 1.71 (m, 1H), 1.40 – 1.32 (m, 1H), 1.29 – 1.23 (m, 1H), 0.91 (t, J = 7.2 Hz, 3H). 13C NMR (100 MHz, CDCl3) δ 170.5, 141.00, 128.52, 127.93, 126.65, 76.05, 38.56, 21.42, 18.92, 13.92. IR (cm⁻¹): 3064, 2956, 2870, 1736, 1236, 699. HRMS: calcd. For C12H16O2Na⁺ [M+Na]⁺: 215.1043; Found: 215.1050.

(4l) 3-acetoxy-3-phenylpropyl benzoate

The title compound was prepared according to the general procedure described above by the reaction between styrene (1a) with iodobenzene diacetate and 2-oxopropyl benzoate (2l), and purified by flash column chromatography as colorless oil (32.2 mg, 54%).

1H NMR (400 MHz, CDCl3) δ 8.02 – 7.98 (m, 2H), 7.56 (t, J = 7.4 Hz, 1H), 7.44 (t, J = 7.6 Hz, 2H), 7.37 – 7.28 (m, 5H), 5.97 (dd, J = 8.0, 6.0 Hz, 1H), 4.42 – 4.28 (m, 2H), 2.45 – 2.36 (m, 1H), 2.30 – 2.22 (m, 1H), 2.07 (s, 3H). 13C NMR (100 MHz, CDCl3) δ 170.31, 166.57, 139.99, 133.30, 133.16, 130.14, 129.84, 129.72, 128.78, 128.51, 128.32, 126.55, 73.23, 61.36, 35.49, 21.33. IR (cm⁻¹): 3063, 2962, 2933, 1720, 700. HRMS: calcd. For C18H18O4Na⁺ [M+Na]⁺: 321.1097; Found: 321.1083.

(4m) 2-(2,2-dimethyl-1,3-dioxolan-4-yl)-1-phenylethyl acetate

The title compound was prepared according to the general procedure described above by the reaction between styrene (1a) with iodobenzene diacetate and (R)-2,2-dimethyl-1,3-dioxolane-4-carbaldehyde (2m), and purified by flash column chromatography as colorless oil (33.8 mg, 64%, d.r. = 1.5 : 1).

1H NMR (400 MHz, CDCl3) δ 7.35 – 7.29 (m, 5H), 5.89 – 5.85 (m, 1H), 4.18 – 4.12 (m, 1H), 4.05 – 4.01 (m, 1H), 3.59 – 3.52 (m, 1H), 2.08 (s, 3H), 1.42 – 1.34 (m, 6H), 1.30 (s, 1H), 1.26 (s, 1H). 13C NMR (100 MHz, CDCl3) δ 170.18, 140.64, 128.72, 128.20, 126.69, 126.31, 108.93, 73.39,
73.06, 69.60, 41.08, 40.32, 27.13, 25.86, 21.34. IR (cm⁻¹): 2959, 2870, 2097, 1245, 699. HRMS: calcd. For C₁₅H₂₀O₄Na⁺ [M+Na⁺]: 301.1410; Found: 301.1420.

(6a) 3-methyl-1-phenylbutyl benzoate

![Chemical Structure](image)

The title compound was prepared according to the general procedure described above by the reaction between styrene (1a) with benzoic acid (5a) and isobutyraldehyde (2a), and purified by flash column chromatography as colorless oil (43.4 mg, 81%).

¹H NMR (400 MHz, CDCl₃) δ 8.07 (d, J = 8.0 Hz, 2H), 7.56 – 7.52 (m, 1H), 7.45 – 7.41 (m, 4H), 7.34 (t, J = 7.6 Hz, 2H), 7.27 (t, J = 7.4 Hz, 1H), 6.07 (dd, J = 8.8, 4.8 Hz, 1H), 2.05 – 1.99 (m, 1H), 1.74 – 1.66 (m, 2H), 0.97 (dd, J = 8.4, 6.4 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 165.93, 141.32, 132.96, 130.62, 129.71, 128.57, 128.42, 127.94, 126.52, 75.29, 45.82, 24.95, 23.01, 22.47. IR (cm⁻¹): 3089, 3064, 2957, 2870, 1716, 1271. HRMS: calcd. For C₁₅H₂₀O₄Na⁺ [M+Na⁺]: 291.1356; Found: 291.1360.

(6b) 3-methyl-1-phenylbutyl 4-fluorobenzoate

![Chemical Structure](image)

The title compound was prepared according to the general procedure described above by the reaction between styrene (1a) with 4-fluorobenzoic acid (5b) and isobutyraldehyde (2a), and purified by flash column chromatography as colorless oil (42.3 mg, 74%).

¹H NMR (400 MHz, CDCl₃) δ 8.08 (dd, J = 8.8, 5.6 Hz, 2H), 7.41 (d, J = 7.6 Hz, 2H), 7.34 (t, J = 7.4 Hz, 2H), 7.30 – 7.26 (m, 1H), 7.10 (t, J = 8.6 Hz, 2H), 6.05 (dd, J = 8.4, 4.8 Hz, 1H), 2.06 – 2.00 (m, 1H), 1.75 – 1.63 (m, 2H), 0.98 (dd, J = 9.2, 6.4 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 167.13, 165.04, 164.61, 141.18, 132.33, 132.23, 128.65, 128.07, 126.57, 115.71, 115.49, 45.76, 24.98, 23.00, 22.51. IR (cm⁻¹): 2958, 2871, 2837, 1736, 1073, 699. HRMS: calcd. For C₁₅H₁₉FO₂Na⁺ [M+Na⁺]: 309.1261; Found: 309.1257.

(6c) 4-methoxybenzoic acid

![Chemical Structure](image)

The title compound was prepared according to the general procedure described above by the reaction between styrene (1a) with 4-methoxybenzoic acid (5c) and isobutyraldehyde (2a), and purified by flash column chromatography as colorless oil (50.1 mg, 84%).

¹H NMR (400 MHz, CDCl₃) δ 8.03 (d, J = 8.8 Hz, 2H), 7.41 (d, J = 7.2 Hz, 2H), 7.34 (t, J = 7.4 Hz, 2H), 7.29 – 7.25 (m, 1H), 6.92 (d, J = 9.2 Hz, 2H), 6.03 (dd, J = 8.8, 5.2 Hz, 1H), 3.85 (s, 3H), 2.06 – 1.97 (m, 1H), 1.74 – 1.64 (m, 2H), 0.97 (dd, J = 8.0, 6.4 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ
165.76, 163.44, 141.58, 131.77, 128.57, 127.87, 126.51, 123.05, 113.70, 77.48, 77.16, 76.84, 74.95, 55.54, 45.91, 24.97, 23.06, 22.51. IR (cm\(^{-1}\)): 3018, 2871, 2834, 1746, 1073, 699. HRMS: calcd. For C\(_{19}\)H\(_{22}\)O\(_3\)Na\(^{+}\) [M+Na\(^{+}\)]: 321.1461; Found: 321.1476.

(6d) 3-methyl-1-phenylbutyl 2-naphthoate

![Structure of 3-methyl-1-phenylbutyl 2-naphthoate](image)

The title compound was prepared according to the general procedure described above by the reaction between styrene (1a) with 2-naphthoic acid (5d) and isobutyraldehyde (2a), and purified by flash column chromatography as colorless oil (50 mg, 79%).

\(^{1}\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.63 (s, 1H), 8.09 (dd, \(J = 8.4, 1.2\) Hz, 1H), 7.96 (d, \(J = 8.0\) Hz, 1H), 7.87 (d, \(J = 8.4\) Hz, 2H), 7.60 – 7.52 (m, 2H), 7.47 (d, \(J = 7.2\) Hz, 2H), 7.36 (t, \(J = 7.4\) Hz, 2H), 7.29 (d, \(J = 7.2\) Hz, 1H), 7.13 (dd, \(J = 8.8, 4.8\) Hz, 1H), 2.13 – 2.06 (m, 1H), 1.80 – 1.71 (m, 2H), 1.01 (dd, \(J = 9.6, 6.4\) Hz, 6H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 166.18, 141.40, 135.67, 132.65, 131.17, 129.49, 128.65, 128.33, 128.25, 128.01, 127.89, 126.74, 126.64, 125.46, 124.97, 124.89, 25.05, 23.07, 22.57. IR (cm\(^{-1}\)): 2958, 2871, 2837, 1736, 1073, 699.

HRMS: calcd. For C\(_{22}\)H\(_{22}\)O\(_2\)Na\(^{+}\) [M+Na\(^{+}\)]: 341.1512; Found: 341.1517.

(6e) 3-methyl-1-phenylbutyl furan-2-carboxylate

![Structure of 3-methyl-1-phenylbutyl furan-2-carboxylate](image)

The title compound was prepared according to the general procedure described above by the reaction between styrene (1a) with furan-2-carboxylic acid (5e) and isobutyraldehyde (2a), and purified by flash column chromatography as colorless oil (30 mg, 58%).

\(^{1}\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.57 (s, 1H), 7.41 (d, \(J = 7.2\) Hz, 2H), 7.34 (t, \(J = 7.0\) Hz, 2H), 7.29 (d, \(J = 6.4\) Hz, 1H), 7.20 (s, 1H), 6.50 (s, 1H), 6.06 – 6.03 (m, 1H), 2.01 (t, \(J = 8.0\) Hz, 1H), 1.73 – 1.64 (m, 2H), 0.97 (t, \(J = 5.6\) Hz, 6H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 158.26, 146.39, 145.10, 140.92, 128.63, 128.11, 126.71, 117.99, 111.89, 75.32, 45.62, 24.93, 23.00, 22.49. IR (cm\(^{-1}\)): 2958, 2871, 2837, 1736, 1073, 699. HRMS: calcd. For C\(_{16}\)H\(_{18}\)O\(_3\)Na\(^{+}\) [M+Na\(^{+}\)]: 281.1148; Found: 281.1151.

(6f) 3-methyl-1-phenylbutyl thiophene-2-carboxylate

![Structure of 3-methyl-1-phenylbutyl thiophene-2-carboxylate](image)

The title compound was prepared according to the general procedure described above by the reaction between styrene (1a) with thiophene-2-carboxylic acid (5f) and isobutyraldehyde (2a), and purified by flash column chromatography as colorless oil (35.1 mg, 64%).
$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.81 (d, $J = 3.2$ Hz, 1H), 7.55 (d, $J = 4.8$ Hz, 1H), 7.41 (d, $J = 7.6$ Hz, 2H), 7.34 (t, $J = 7.4$ Hz, 2H), 7.29 (d, $J = 7.2$ Hz, 1H), 7.09 (t, $J = 4.4$ Hz, 1H), 6.01 (dd, $J = 8.8$, 4.8 Hz, 1H), 2.03 – 1.99 (m, 1H), 1.70 – 1.67 (m, 2H), 0.98 (t, $J = 6.2$ Hz, 6H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 161.71, 141.15, 134.32, 133.51, 132.42, 128.63, 128.04, 127.86, 126.58, 75.64, 45.80, 24.99, 23.05, 22.52. IR (cm$^{-1}$): 2958, 2871, 2837, 1736, 1073, 699. HRMS: calcd. For C$_{16}$H$_{18}$O$_2$SNa$^+$ [M+Na$^+$]: 297.0920; Found: 297.0936.

V. References


VI. Copies of $^1$H and $^{13}$C NMR spectra of products 3a-3m, 4b-4m, 6a-6f
S25