Supporting Information

Synthesis of Isochromene Derivatives using Intramolecular Benzylic C(sp³)-C(sp²) Bond Forming Heck Reaction on Vinylogous Carbonates

Santosh J. Gharpure,a* Yogesh G. Shelke,a S. Raja Bhushan Reddyb

aDepartment of Chemistry, Indian Institute of Technology Bombay, Powai, Mumbai – 400076, India

bDepartment of Chemistry, Indian Institute of Technology Madras, Chennai – 600036, Tamil Nadu, India

sjgharpure@iitb.ac.in

Table of contents

I. General ........................................................................................................S2

II. Experimental procedures and experimental data...............................S2-S19

III. Copies of 1H and 13C NMR spectra for all compounds .................S20-S48
General:

Melting points are recorded using Tempo melting point apparatus in capillary tubes and are uncorrected. IR spectra were recorded on Nicolet 6700 spectrophotometer and JASCO FT IR-4100 spectrophotometer. 1H (400 MHz, 500 MHz) and 13C (100 MHz, 125 MHz) NMR spectrums were recorded on Bruker Avance 400 spectrophotometer and Bruker Avance 500 spectrophotometer, respectively. The chemical shifts (ppm) and coupling constants (Hz) are reported in the standard fashion with reference to chloroform. In the 13C NMR spectra, the nature of the carbons (C, CH, CH$_2$ or CH$_3$) was determined by recording the DEPT-135 experiment, and is given in parentheses. High resolution mass measurements were carried out using Micromass Q-ToF instrument using direct inlet mode. Analytical thin-layer chromatographies (TLC) were performed on glass plates (7.5 × 2.5 and 9 × 5.0 cm) coated with Merck or Acme's silica gel G containing 13% calcium sulfate as binder or on pre-coated 0.2 mm thick Merck 60 F$_{245}$ silica plates and various combinations of ethyl acetate and hexanes were used as eluent. Visualization of spots was accomplished by either exposure to iodine vapour or KMnO$_4$ stain. Acme's silica gel (100-200 mesh) was used for column chromatography (approximately 15-20 g per 1 g of the crude product). All small-scale dry reactions were carried out using standard syringe septum technique. Low temperature reactions were conducted in jullabo. Dry THF and dry ether were obtained by distillation over sodium-benzophenone ketyl. Dry dichloromethane and dry DMF were prepared by distilling over calcium hydride. All the commercial reagents were used as such without further purification.

Typical Procedure for the Synthesis of isochromenes in One Pot:

A Schlenk tube with a magnetic stir bar was charged with ethyl propiolate (1.1 mmol), chloroalcohol 15 (1.0 mmol), NMM (1.1 mmol) and Et$_3$N (15 mmol). The reaction mixture was stirred for 4 hrs at room temperature until completion of starting material (TLC control) and then Pd(OAc)$_2$ (0.05 mmol), PPh$_3$ (0.10 mmol), was added to the above Schlenk tube. The reaction vessel was placed in an oil bath and heated at 100 ℃, and the mixture was stirred for 2h. Reaction was quenched with sat. NH$_4$Cl solution and extracted with EtOAc (3x 10 mL) and dried (anhyd. Na$_2$SO$_4$). Combined organic layer was concentrated under reduced
pressure and purification of residue on a silica gel column using EtOAc/Hexanes (1:19) as eluent furnished the isochromene derivative 6a (62%) as a colourless oil.

**Representative experimental procedures:**

*Ethyl 2-(1H-isochromen-3-yl)acetate (6a):*

To chloro vinylogous carbonate 5a (50 mg, 0.196 mmol) in a sealed reaction tube were added Pd(OAc)$_2$ (2 mg, 5 mol%) and PPh$_3$ (6 mg, 10 mol%) under nitrogen atmosphere. This reaction tube was evacuated for some time. To this reaction tube, Et$_3$N (0.4 mL, 2.94 mmol) and dry DMF (2 mL) were added and it was heated to 100 °C with vigorous stirring. Progress of the reaction was monitored by TLC until disappearance of starting material. Reaction mixture was diluted with water and extracted with ethyl acetate (3 x 10 mL). The combined organic layers were washed with sat. NH$_4$Cl solution (20 mL) and dried (anhyd. Na$_2$SO$_4$). Evaporation of the solvent and purification of residue on a silica gel column using EtOAc/Hexanes (1:19) as eluent furnished the isochromene derivative 6a (37 mg, 86%) as a colourless oil.

**Physical appearance:** colourless liquid.

R$_f$: 0.4 (1:9, EtOAc:Hexanes).

**IR (neat):** 3542, 2982, 1730, 1647, 1475, 1375, 1200, 1045, 764, 672 cm$^{-1}$.

**$^1$H NMR (400 MHz, CDCl$_3$):** δ 7.20 (t, $J = 7.4$ Hz, 1H), 7.13 (t, $J = 7.4$ Hz, 1H), 6.99 (d, $J = 7.4$ Hz, 1H), 6.95 (d, $J = 7.4$ Hz, 1H), 5.80 (s, 1H), 5.10 (s, 2H), 4.20 (q, $J = 7.2$ Hz, 2H), 3.22 (s, 2H), 1.28 (t, $J = 7.2$ Hz, 3 H).

**$^{13}$C NMR (100 MHz, CDCl$_3$, DEPT):** δ 169.9 (C), 151.1 (C), 131.3 (C), 128.3 (CH), 127.5 (C), 126.6 (CH), 123.9 (CH), 123.1 (CH), 104.5 (CH), 69.2 (CH$_2$), 61.2 (CH$_2$), 39.9 (CH$_2$), 14.3 (CH$_3$).

**LRMS (ESI, M+ Na$^+$):** m/z 241.

**HRMS (ESI, M+Na$^+$):** m/z calcd. for C$_{13}$H$_{14}$NaO$_3$ 241.0841, found 241.0832.
(E)-ethyl 3-(2-(acetoxymethyl)benzyloxy)acrylate (7):

**Physical appearance:** viscous liquid.

R_f: 0.8 (1:9, EtOAc:Hexanes).

**IR (neat):** 2928, 2852, 1742, 1711, 1643, 1626, 1458, 1382, 1324, 1234, 1134, 1045 cm^{-1}.

\[ ^1H \text{ NMR (400 MHz, CDCl}_3 \text{):}\] \( \delta \) 7.66 (d, \( J = 12.8 \) Hz, 1H), 7.40-7.35 (m, 4H), 5.33 (d, \( J = 12.8 \) Hz, 1H), 5.15 (s, 2H), 4.98 (s, 2H), 4.17 (q, \( J = 7.2 \) Hz, 2H), 2.09 (s, 3H), 1.27 (t, 3H).

\[ ^{13}C \text{ NMR (100 MHz, CDCl}_3 \text{, DEPT):}\] \( \delta \) 170.8 (C), 167.7 (C), 161.8 (CH), 134.5 (C), 133.9 (C), 130.2 (CH), 129.4 (CH), 129.2 (CH), 129.0 (CH), 97.8 (CH), 70.6 (CH\(_2\)), 63.8 (CH\(_2\)), 60.1 (CH\(_2\)), 21.1 (CH\(_3\)), 14.5 (CH\(_3\)).

**LRMS (ESI, M+ Na\(^+\):** m/z 301.

**HRMS (ESI, M+Na\(^+\):** m/z calcd. for C\(_{15}\)H\(_{18}\)NaO\(_5\) 301.1052, found 301.1045.

**Ethyl 2-(6,7-dimethoxy-1H-isochromen-3-yl)acetate (6b):**

The chloro vinylogous carbonate 5b (45 mg, 0.15 mmol) was treated with Pd(OAc)_2 (2 mg, 5 mol %), PPh\(_3\) (4 mg, 10 mol%) and Et\(_3\)N (0.3 mL, 2.24 mmol) in dry DMF (3 mL) as described for compound 6a followed by purification on silica gel column chromatography using EtOAc/Hexanes (1:19) as eluent furnished the isochromene derivative 6b (31 mg, 74 %) as a viscous liquid.

**Physical appearance:** viscous liquid.

R_f: 0.6 (1:9, EtOAc:Hexanes).

**IR (neat):** 2978, 2835, 1737, 1653, 1609, 1511, 1465, 1456, 1391, 1284, 1249, 1232, 1121, 1039, 999, 861, 837, 753 cm^{-1}.

\[ ^1H \text{ NMR (400 MHz, CDCl}_3 \text{):}\] \( \delta \) 6.56 (s, 1H), 6.52 (s, 1H), 5.73 (s, 1H), 5.04 (s, 2H), 4.19 (q, \( J = 7.2 \) Hz, 2H), 3.85 (s, 6H), 3.20 (s, 2H), 1.28 (t, \( J = 7.2 \) Hz, 3H).
$^{13}$C NMR (100 MHz, CDCl$_3$, DEPT): $\delta$ 170.0 (C), 149.5 (C), 148.9 (C), 147.9 (C), 124.3 (C), 119.7 (C), 108.0 (CH), 107.2 (CH), 104.2 (CH), 68.9 (CH$_2$), 61.2 (CH$_2$), 56.3 (CH$_3$), 56.2 (CH$_3$), 39.8 (CH$_2$), 14.3 (CH$_3$).

LRMS (ESI, M+ H$^+$): m/z 279.

HRMS (ESI, M+H$^+$): m/z calcd. for C$_{15}$H$_{19}$O$_5$ 279.1232, found 279.1241.

Ethyl 2-(1-methyl-1H-isochromen-3-yl)acetate (6c):

The chloro vinylogous carbonate 5c (51 mg, 0.19 mmol) was treated with Pd(OAc)$_2$ (2 mg, 5 mol %), PPh$_3$ (5 mg, 10 mol%) and Et$_3$N (0.4 mL, 2.86 mmol) in dry DMF (3 mL) as described for compound 6a followed by purification on silica gel column chromatography using EtOAc/Hexanes (1:19) as eluent furnished the isochromene derivative 6c (32 mg, 72 %) as a viscous liquid.

Physical appearance: viscous liquid.

R$_f$: 0.4 (1:9, EtOAc:Hexanes).

IR (neat): 3442, 2983, 1736, 1657, 1375, 1338, 1282, 1190, 1132, 1030, 762 cm$^{-1}$.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.15 (m, 2H), 6.99 (dd, $J = 7.6$, 0.8 Hz, 1H), 6.95 (dd, $J = 7.6$, 1.6 Hz, 1H), 5.76 (s, 1H), 5.28 (q, $J = 7.2$ Hz, 1H), 4.20 (q, $J = 7.2$ Hz, 2H), 3.20 (s, 2H), 1.56 (d, $J = 7.2$ Hz, 3H), 1.29 (t, $J = 7.2$ Hz, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$, DEPT): $\delta$ 169.8 (C), 149.2 (C), 132.0 (C), 130.5 (C), 127.9 (CH), 126.7 (CH), 123.4 (CH), 123.3 (CH), 103.6 (CH), 74.7 (CH), 61.2 (CH$_2$), 40.2 (CH$_2$), 20.1 (CH$_3$), 14.3 (CH$_3$).

LRMS (ESI, M+ Na$^+$): m/z 255.

HRMS (ESI, M+Na$^+$): m/z calcd. for C$_{14}$H$_{16}$O$_3$Na 255.0997, found 255.1003.

Ethyl 2-(1-butyl-1H-isochromen-3-yl)acetate (6d):
The chloro vinylogous carbonate 5d (88 mg, 0.28 mmol) was treated with Pd(OAc)$_2$ (3 mg, 5 mol %), PPh$_3$ (8 mg, 10 mol%) and Et$_3$N (0.6 mL, 4.25 mmol) in dry DMF (4 mL) as described for compound 6a followed by purification on silica gel column chromatography using EtOAc/Hexanes (1:19) as eluent furnished the isochromene derivative 6d (52 mg, 67 %) as a viscous liquid.

**Physical appearance:** viscous liquid.

**R$_f$:** 0.4 (1:9, EtOAc:Hexanes).

**IR (neat):** 2956, 2932, 2859, 1740, 1656, 1488, 1457, 1385, 1251, 1149, 1034, 751 cm$^{-1}$.

**$^1$H NMR (400 MHz, CDCl$_3$):** δ 7.15 (m, 2H), 6.94 (t, $J = 6.8$ Hz, 2H), 5.73 (s, 1H), 5.11 (dd, $J = 8.4$, 5.2 Hz, 1H), 4.19 (qd, $J = 7.2$, 0.8 Hz, 2H), 3.20 (s, 2H), 2.00 (m, 1H), 1.71 (ddddd, $J = 14.8$, 9.6, 4.8 Hz, 1H), 1.40-1.50 (m, 1H), 1.30-1.35 (m, 3H), 1.29 (t, $J = 7.2$ Hz, 3H), 0.90 (t, $J = 7.2$ Hz, 3H).

**$^{13}$C NMR (100 MHz, CDCl$_3$, DEPT):** δ 169.8 (C), 148.8 (C), 130.9 (C), 130.3 (C), 127.7 (CH), 126.3 (CH), 124.0 (CH), 123.3 (CH), 103.2 (CH), 78.6 (CH), 61.1 (CH$_2$), 40.2 (CH$_2$), 33.9 (CH$_2$), 27.3 (CH$_2$), 22.6 (CH$_2$), 14.2 (CH$_3$), 14.1 (CH$_3$).

**LRMS (ESI, M+H$^+$):** m/z 275.

**HRMS (ESI, M+H$^+$):** m/z calcd. for C$_{17}$H$_{23}$O$_3$ 275.1647, found 275.1635.

*Ethyl 2-(1-isopropyl-1H-isochromen-3-yl)acetate (6e):*

The chloro vinylogous carbonate 5e (62 mg, 0.21 mmol) was treated with Pd(OAc)$_2$ (2 mg, 5 mol %), PPh$_3$ (6 mg, 10 mol%), and Et$_3$N (0.4 mL, 3.12 mmol) in dry DMF (4 mL) as described for compound 6a followed by purification on silica gel column chromatography using EtOAc/Hexanes (1:19) as eluent furnished the isochromene derivative 6e (38 mg, 70 %) as a viscous liquid.

**Physical appearance:** viscous liquid.
Rf: 0.4 (1:9, EtOAc:Hexanes).

IR (neat): 2975, 2962, 2927, 1740, 1656, 1489, 1468, 1383, 1367, 1149, 1015 cm⁻¹.

¹H NMR (400 MHz, CDCl₃): δ 7.17 (t, J = 7.2 Hz, 1H), 7.11 (t, J = 7.2 Hz, 1H), 6.92 (d, J = 7.2 Hz, 2H), 5.68 (s, 1H), 4.85 (d, J = 6.4 Hz, 1H), 4.19 (q, J = 7.2 Hz, 2H), 3.20 (s, 2H), 2.25-2.15 (m, 1H), 1.29 (t, J = 7.2 Hz, 3H), 1.01 (d, J = 6.4 Hz, 3H), 0.88 (d, J = 6.8 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃, DEPT): δ 169.8 (C), 149.2 (C), 130.8 (C), 129.3 (C), 127.8 (CH), 126.0 (CH), 125.3 (CH), 123.3 (CH), 103.0 (CH), 83.9 (CH), 61.2 (CH₂), 40.2 (CH₂), 31.9 (CH), 19.0 (CH₃), 17.8 (CH₃), 14.3 (CH₃).

LRMS (ESI, M+ H⁺): m/z 261.

HRMS (ESI, M+H⁺): m/z calcd. for C₁₆H₂₁O₃ 261.1491, found 261.1488.

Ethyl 2-(3-(2-ethoxy-2-oxoethyl)-1H-isochromen-1-yl)-2-methylpropanoate (6f):
The chloro vinylogous carbonate 5f (100 mg, 0.27 mmol) was treated with Pd(OAc)₂ (3 mg, 5 mol %), PPh₃ (7 mg, 10 mol%) and Et₃N (0.6 mL, 4.07 mmol) in dry DMF (4 mL) as described for compound 6a followed by purification on silica gel column chromatography using EtOAc/Hexanes (1:19) as eluent furnished the isochromene derivative 6f (60 mg, 67 %) as a viscous liquid.

Physical appearance: viscous liquid.

Rf: 0.6 (1:9, EtOAc:Hexanes).

IR (neat): 2980, 1738, 1662, 1625, 1578, 1491, 1388, 1367, 1254, 1174, 1031, 946, 894, 754 cm⁻¹.

¹H NMR (400 MHz, CDCl₃): δ 7.18 (td, J = 12.6, 0.8 Hz, 1H), 7.09 (td, J = 12.6, 0.8 Hz, 1H), 6.91 (d, J = 12.6 Hz, 1H), 6.88 (d, J = 12.6 Hz, 1H), 5.58 (s, 2H), 4.18 (q, J = 7.2 Hz, 2H), 4.18 (q, J = 7.2 Hz, 2H), 3.16 (s, 2H), 1.28 (t, J = 7.2 Hz, 3H), 1.25 (t, J = 7.2 Hz, 3H), 1.22 (s, 3H), 1.16 (s, 3H).
\( ^{13} \text{C NMR (100 MHz, CDCl}_3, \text{ DEPT)}: \delta \) 175.8 (C), 169.5 (C), 149.6 (C), 131.5 (C), 128.4 (CH), 126.1 (CH), 125.9 (CH), 125.8 (C), 123.5 (CH), 102.5 (CH), 82.6 (CH), 61.2 (CH\(_2\)), 60.9 (CH\(_2\)), 50.7 (C), 40.2 (CH\(_2\)), 21.1 (CH\(_3\)), 20.4 (CH\(_3\)), 14.2 (CH\(_3\)), 14.2 (CH\(_3\)),

**LRMS (ESI, M+ Na\(^{+}\)):** m/z 355.

**HRMS (ESI, M+Na\(^{+}\)):** m/z calcd. for \( \text{C}_{19}\text{H}_{24}\text{O}_5\text{Na} \) 355.1521, found 355.1505.

**Methyl-1-(3-(2-ethoxy-2-oxoethyl)-1H-isochromen-1-yl)cyclohexanecarboxylate (6g):**

The chloro vinylogous carbonate 5g (90 mg, 0.23 mmol) was treated with Pd(OAc)\(_2\) (3 mg, 5 mol %), PPh\(_3\) (6 mg, 10 mol%) and Et\(_3\)N (0.5 mL, 4.03 mmol) in dry DMF (4 mL) as described for compound 6a followed by purification on silica gel column chromatography using EtOAc/Hexanes (1:19) as eluent furnished the isochromene derivative 6g (59 mg, 72 %) as a viscous liquid.

**Physical appearance:** viscous liquid.

**R\(_f\):** 0.7 (1:9, EtOAc:Hexanes).

**IR (neat):** 2998, 1742, 1662, 1487, 1448, 1385, 1371, 1305, 1213, 1013, 943, 750 cm\(^{-1}\).

\( ^1\text{H NMR (400 MHz, CDCl}_3): \delta \) 7.20 (t, \( J = 7.6 \) Hz, 1H), 7.11 (t, \( J = 7.6 \) Hz, 1H), 6.91 (d, \( J = 7.6 \) Hz, 1H), 6.88 (d, \( J = 7.2 \) Hz, 1H), 5.60 (s, 1H), 5.25 (s, 1H), 4.19 (q, \( J = 7.2 \) Hz, 2H), 3.65 (s, 3H), 3.17 (AB quart., \( J = 16.0 \) Hz, 2H), 2.21 (dd, \( J = 20, 13.2 \) Hz, 2H), 1.65-1.55 (m, 5H), 1.40-1.15 (m, 5H), 1.05-1.00 (m, 1H).

\( ^{13} \text{C NMR (100 MHz, CDCl}_3, \text{ DEPT)}: \delta \) 174.5 (C), 169.4 (C), 149.9 (C), 131.4 (C), 128.5 (CH), 126.5 (C), 125.9 (CH), 125.4 (CH), 123.3 (CH), 102.6 (CH), 84.3 (CH), 61.2 (CH\(_2\)), 55.7 (C), 51.8 (CH\(_3\)), 40.2 (CH\(_2\)), 30.5 (CH\(_2\)), 29.2 (CH\(_2\)), 25.6 (CH\(_2\)), 23.7 (CH\(_2\)), 23.1 (CH\(_2\)), 14.3 (CH\(_3\)).

**LRMS (ESI, M+ H\(^{+}\)):** m/z 359.

**HRMS (ESI, M+H\(^{+}\)):** m/z calcd. for \( \text{C}_{21}\text{H}_{27}\text{O}_5 \) 359.1858, found 359.1843.
**Ethyl 2-(1-phenyl-1H-isochromen-3-yl)acetate (6i):**

The chloro vinylogous carbonate 5i (87 mg, 0.246 mmol) was treated with Pd(OAc)$_2$ (3 mg, 5 mol %), PPh$_3$ (7 mg, 10 mol%) and Et$_3$N (0.5 mL, 3.70 mmol) in dry DMF (4 mL) as described for compound 6a followed by purification on silica gel column chromatography using EtOAc/Hexanes (1:19) as eluent furnished the isochromene derivative 6i (65 mg, 88 %) as a viscous liquid.

**Physical appearance:** viscous liquid.

**R$_f$:** 0.5 (1:9, EtOAc:Hexanes).

**IR (neat):** 2921, 2850, 2358, 1733, 1374, 1189, 1050, 1027, 933, 758, 700 cm$^{-1}$.

**$^1$H NMR (500 MHz, CDCl$_3$):** δ 7.36 (s, 5H), 7.22 (td, $\text{J} = 6.0$, 0.8 Hz, 1H), 7.09 (td, $\text{J} = 6.0$, 0.8 Hz, 1H), 7.03 (d, $\text{J} = 6.0$ Hz, 1H), 6.69 (d, $\text{J} = 6.0$ Hz, 1H), 6.16 (s, 1H), 5.86 (s, 1H), 4.15-4.00 (m, 2H), 3.21 (AB quart., $\text{J} = 12.8$ Hz, 2H), 1.20 (t, $\text{J} = 6.6$ Hz, 3H).

**$^{13}$C NMR (125 MHz, CDCl$_3$, DEPT):** δ 169.6 (C), 149.7 (C), 139.9 (C), 131.1 (C), 129.89 (C), 128.5 (3xCH), 128.3 (2xCH), 126.7 (2xCH), 125.5 (CH), 123.3 (CH), 104.2 (CH), 80.6 (CH), 61.2 (CH$_2$), 40.2 (CH$_2$), 14.2 (CH$_3$).

**LRMS (ESI, M+ H$^+$):** m/z 295.

**HRMS (ESI, M+H$^+$):** m/z calcd. for C$_{19}$H$_{19}$O$_3$ 295.1334, found 295.1322.

---

**Ethyl 2-(1-(p-tolyl)-1H-isochromen-3-yl)acetate (6j):**

The chloro vinylogous carbonate 5j (95 mg, 0.276 mmol) was treated with Pd(OAc)$_2$ (3 mg, 5 mol %), PPh$_3$ (7 mg, 10 mol%) and Et$_3$N (0.6 mL, 4.140 mmol) in dry DMF (4 mL) as described for compound 6a followed by purification on silica gel column chromatography using EtOAc/Pet ether (1:19) as eluent furnished the isochromene derivative 6j (70 mg, 82 %) as a colourless liquid.
Physical appearance: colourless liquid.

Rf: 0.5 (1:9, EtOAc:Pet ether).

IR (neat): 2982, 1738, 1654, 1483, 1371, 1254, 1149, 1032, 912, 802, 753 cm\(^{-1}\).

\(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta 7.25\) (bt, \(J = 8.0 \text{ Hz}, 3\text{H}\)), \(7.20\) (bt, \(J = 8.0 \text{ Hz}, 2\text{H}\)), \(7.10\) (td, \(J = 7.2, 1.2 \text{ Hz}, 1\text{H}\)), \(7.04\) (bd, \(J = 7.6 \text{ Hz}, 1\text{H}\)), \(6.72\) (bd, \(J = 7.2 \text{ Hz}, 1\text{H}\)), \(6.15\) (s, \(1\text{H}\)), \(5.86\) (s, \(1\text{H}\)), \(4.20-4.00\) (m, \(2\text{H}\)), \(3.22\) (s, \(2\text{H}\)), \(2.38\) (s, \(3\text{H}\)), \(1.21\) (t, \(J = 7.2 \text{ Hz}, 3\text{H}\)).

\(^13\)C NMR (100 MHz, CDCl\(_3\), DEPT): \(\delta 169.6\) (C), \(149.7\) (C), \(138.2\) (C), \(136.9\) (C), \(131.1\) (C), \(130.0\) (C), \(129.1\) (2xCH), \(128.2\) (3xCH), \(126.6\) (CH), \(125.5\) (CH), \(123.2\) (CH), \(104.1\) (CH), \(80.4\) (CH), \(61.1\) (CH\(_2\)), \(40.2\) (CH\(_2\)), \(21.3\) (CH\(_3\)), \(14.2\) (CH\(_3\)).

LRMS (ESI, M+ Na\(^+\)): m/z 331.

HRMS (ESI, M+Na\(^+\)): m/z calcd. for C\(_{20}\)H\(_{20}\)NaO\(_3\) 331.1301, found 331.1307.

*Ethyl 2-(1-(2,5-dimethylphenyl)-1H-isochromen-3-yl)acetate (6k)*:

The chloro vinylogous carbonate 5k (105 mg, 0.293 mmol) was treated with Pd(OAc)$_2$ (4 mg, 5 mol %), PPh$_3$ (8 mg, 10 mol%) and Et$_3$N (0.6 mL, 4.396 mmol) in dry DMF (4 mL) as described for compound 6a followed by purification on silica gel column chromatography using EtOAc/Pet ether (1:19) as eluent furnished the isochromene derivative 6k (68 mg, 72 %) as a colourless liquid.

Physical appearance: colourless liquid.

Rf: 0.5 (1:9, EtOAc:Pet ether).

IR (neat): 3012, 1783, 1738, 1655, 1602, 1483, 1455, 1339, 1251, 1151, 1032, 917, 809, 734 cm\(^{-1}\).

\(^1\)H NMR (500 MHz, CDCl\(_3\)): \(\delta 7.22\) (bt, \(J = 7.5 \text{ Hz}, 1\text{H}\)), \(7.15\) (d, \(J = 8.0 \text{ Hz}, 1\text{H}\)), \(7.09\) (bd, \(J = 7.0 \text{ Hz}, 2\text{H}\)), \(7.05\) (t, \(J = 8.0 \text{ Hz}, 2\text{H}\)), \(6.55\) (d, \(J = 7.5 \text{ Hz}, 1\text{H}\)), \(6.33\) (s, \(1\text{H}\)), \(5.90\) (s, \(1\text{H}\)), \(4.20-4.05\) (m, \(2\text{H}\)), \(3.25\) (AB, \(J = 16.0 \text{ Hz}, 2\text{H}\)), \(2.34\) (s, \(3\text{H}\)), \(2.31\) (s, \(3\text{H}\)), \(1.24\) (t, \(J = 7.0 \text{ Hz}, 3\text{H}\)).
\[ ^{13}\text{C} \text{NMR (125 MHz, CDCl}_3, \ DEPT) : \delta 169.7 (C), 150.5 (C), 137.0 (C), 135.5 (C), 134.0 (C), 131.7 (C), 130.7 (CH), 129.8 (C), 129.6 (CH), 129.2 (CH), 128.2 (CH), 126.7 (CH), 125.0 (CH), 123.1 (CH), 104.2 (CH), 78.9 (CH), 61.1 (CH), 40.0 (CH), 21.1 (CH), 19.2 (CH) , 14.2 (CH).
\]

\text{LRMS (ESI, M+ Na\textsuperscript{+}) : m/z 345.}

\text{HRMS (ESI, M+Na\textsuperscript{+}) : m/z calcd. for C\textsubscript{21}H\textsubscript{22}NaO\textsubscript{3} 345.1461, found 345.1462.}

\text{ethyl 2-(6-nitro-1H-isochromen-3-yl)acetate (6l):}

The chloro vinylogous carbonate 5l (80 mg, 0.27 mmol) was treated with Pd(OAc)\textsubscript{2} (4 mg, 5 mol %), PPh\textsubscript{3} (8 mg, 10 mol%) and Et\textsubscript{3}N (0.56 mL, 4 mmol) in dry DMF (3 mL) as described for compound 6a followed by purification on silica gel column chromatography using EtOAc/Hexanes (1:19) as eluent furnished the isochromene derivative 6l (46 mg, 65 %) as a viscous liquid.

\text{Physical appearance: viscous liquid.}

\text{Rf : 0.8 (1:9, EtOAc:Hexanes).}

\text{IR (neat): 2950, 1736, 1526, 1349, 1258, 1155, 1038, 911, 739 cm\textsuperscript{-1}.}

\[ ^{1}\text{H} \text{NMR (500 MHz, CDCl}_3) : \delta 7.99 (dd, J = 8.0, 2.5 Hz, 1H), 7.78 (d, J = 2.0 Hz, 1H), 7.14 (d, J = 8.0 Hz, 1H), 5.88 (s, 1H), 5.18 (s, 2H), 4.21 (q, J = 7.5 Hz, 2H), 3.25 (s, 2H), 1.29 (t, J = 7.5 Hz, 3H).
\]

\[ ^{13}\text{C} \text{NMR (125 MHz, CDCl}_3, \ DEPT) : \delta 169.3 (C), 153.6 (C), 148.4 (C), 133.8 (C), 133.0 (C), 124.8 (CH), 121.6 (CH), 117.5 (CH), 103.4 (CH), 68.7 (CH), 61.5 (CH), 39.7 (CH), 14.3 (CH).
\]

\text{LRMS (ESI, M+ H\textsuperscript{+}) : m/z 264.}

\text{HRMS (ESI, M+H\textsuperscript{+}) : m/z calcd. for C\textsubscript{13}H\textsubscript{14}NO\textsubscript{5} 264.0866, found 264.0861.}
2-(isochroman-3-yl)-1-phenylethan-1-one (10a):

The chloro vinylogous ester 9a (65 mg, 0.227 mmol) was treated with Pd(OAc)$_2$ (3 mg, 5 mol %), PPh$_3$ (6 mg, 10 mol%) and Et$_3$N (0.5 mL, 3.40 mmol) in dry DMF (3 mL) as described for compound 6a followed by purification on silica gel column chromatography using EtOAc/Pet ether (1:19) as eluent furnished the isochroman derivative 10a (30 mg, 52 %) as a colourless liquid.

**Physical appearance:** colourless liquid.

R$_f$: 0.4 (1:9, EtOAc:Pet ether).

**IR (neat):** 3061, 2925, 1686, 1597, 1495, 1448, 1372, 1297, 1204, 1096, 1036, 1001, 910, 825, 747, 690, 650 cm$^{-1}$.

**$^1$H NMR (500 MHz, CDCl$_3$):** δ 8.01 (dd, $J = 8.7, 1.2$ Hz, 2H), 7.58 (tt, $J = 7.5, 1.2$ Hz, 1H), 7.49 (td, $J = 7.5, 1.7$ Hz, 2H), 7.20-7.15 (m, 2H), 7.10 (t, $J = 7.5$ Hz, 1H), 7.00 (t, $J = 8.0$ Hz, 1H), 4.85 (AB, $J = 15.0$ Hz, 2H), 4.40-4.35 (m, 1H), 3.50 (ABX, $J = 16.5, 7.0$ Hz, 1H), 3.13 (ABX, $J = 16.5, 6.0$ Hz, 1H), 2.95-2.80 (m, 2H).

**$^{13}$C NMR (125 MHz, CDCl$_3$, DEPT):** δ 198.0 (C), 137.2 (C), 134.6 (C), 133.4 (CH), 133.0 (C), 129.0 (CH), 128.8 (2xCH), 128.4 (2xCH), 126.6 (CH), 126.2 (CH), 124.4 (CH), 71.5 (CH$_2$), 68.4 (CH$_2$), 45.0 (CH$_2$), 34.1 (CH$_2$).

**LRMS (ESI, M+ Na$^+$):** m/z 275.

**HRMS (ESI, M+Na$^+$):** m/z calcd. for C$_{17}$H$_{16}$NaO$_2$ 275.1043, found 275.1040.

2-((1S,3S)-1-butylisochroman-3-yl)-1-phenylethan-1-one (10b):

The chloro vinylogous ester 9b (55 mg, 0.161 mmol) was treated with Pd(OAc)$_2$ (2 mg, 5 mol %), PPh$_3$ (4 mg, 10 mol%) and Et$_3$N (0.4 mL, 2.411 mmol) in dry DMF (2 mL) as described for compound 6a followed by purification on silica gel column chromatography
using EtOAc/Pet ether (1:19) as eluent furnished the isochroman derivative **10b** (35 mg, 71 %, combined with **11b**) as a colourless liquid.

**Physical appearance:** colourless liquid.

**Rf:** 0.4 (1:9, EtOAc:Pet ether).

**IR (neat):** 2931, 1687, 1597, 1365, 1254, 1109, 752 cm⁻¹.

**1H NMR (500 MHz, CDCl₃):** \(\delta 8.02 (dd, J = 8.5, 1.5 \text{ Hz}, 2H), 7.58 (t, J = 7.5 \text{ Hz}, 1H), 7.48 (t, J = 8.0 \text{ Hz}, 2H), 7.20-7.15 (m, 2H), 7.10-7.05 (m, 2H), 4.77 (bd, J = 6.5 \text{ Hz}, 1H), 4.35-4.30 (m, 1H), 3.50 (ABX, \(J = 16.0, 6.5 \text{ Hz}, 1H\)), 3.10 (ABX, \(J = 16.0, 6.0 \text{ Hz}, 1H\)), 2.90-2.85 (m, 2H), 2.00-1.95 (m, 1H), 1.75-1.65 (m, 1H), 1.35-1.25 (m, 4H), 0.85 (t, \(J = 7.0 \text{ Hz}, 3H\)).

**13C NMR (125 MHz, CDCl₃, DEPT):** \(\delta 198.7 (C), 138.4 (C), 137.5 (C), 134.0 (C), 133.2 (CH), 128.9 (CH), 128.7 (2xCH), 128.5 (2xCH), 126.3 (CH), 126.3 (CH), 124.4 (CH), 76.8 (CH), 71.3 (CH), 45.3 (CH₂), 35.6 (CH₂), 35.2 (CH₂), 27.2 (CH₂), 22.8 (CH₂), 14.2 (CH₃).

**LRMS (ESI, M+ Na⁺):** \(m/z 331\).

**HRMS (ESI, M+Na⁺):** \(m/z\) calcd. for C₂₁H₂₄NaO₂ 331.1669, found 331.1668.

---

2-(1-butyl-1H-isochromen-3-yl)-1-phenylethan-1-one (11b):

The chloro vinylogous ester **9b** (55 mg, 0.161 mmol) was treated with Pd(OAc)₂ (2 mg, 5 mol %), PPh₃ (4 mg, 10 mol%) and Et₃N (0.4 mL, 2.411 mmol) in dry DMF (2 mL) as described for compound **6a** followed by purification on silica gel column chromatography using EtOAc/Pet ether (1:19) as eluent furnished the isochromene derivative **11b** (35 mg, 71 %, combined with **10b**) as a colourless liquid.

**Physical appearance:** colourless liquid.

**Rf:** 0.4 (1:9, EtOAc:Pet ether).

**IR (neat):** 3048, 2931, 1687, 1597, 1450, 1365, 1254, 1109, 1000, 910, 752 cm⁻¹.

---

S13
$^1$H NMR (500 MHz, CDCl$_3$): $\delta$ 8.05-8.00 (m, 2H), 7.60-7.55 (m, 1H), 7.50-7.45 (m, 2H), 7.20-7.15 (m, 2H), 6.93 (bd, $J$ = 7.5 Hz, 2H), 5.77 (s, 1H), 5.08 (dd, $J$ = 8.5, 4.5 Hz, 1H), 3.82 (s, 2H), 2.00-1.95 (m, 1H), 1.75-1.65 (m, 1H), 1.35-1.25 (m, 4H), 0.85 (t, $J$ = 7.0 Hz, 3H).

$^{13}$C NMR (125 MHz, CDCl$_3$, DEPT): $\delta$ 196.1 (C), 136.7 (C), 133.4 (C), 130.9 (C), 130.5 (C), 128.8 (2xC), 128.7 (2xC), 127.9 (CH), 126.4 (CH), 126.3 (CH), 124.0 (CH), 123.3 (CH), 103.6 (CH), 78.7 (CH), 44.4 (CH$_2$), 34.0 (CH$_2$), 27.4 (CH$_2$), 22.6 (CH$_2$), 14.1 (CH$_3$).

LRMS (ESI, M+ Na$^+$): m/z 329.

HRMS (ESI, M+Na$^+$): m/z calcd. for C$_{21}$H$_{22}$NaO$_3$ 329.1512, found 329.1510.

2-((1S,3S)-1-(2,5-dimethylphenyl)isochroman-3-yl)-1-phenylethan-1-one (10c):

The chloro vinylogous ester 9c (50 mg, 0.128 mmol) was treated with Pd(OAc)$_2$ (2 mg, 5 mol %), PPh$_3$ (4 mg, 10 mol%) and Et$_3$N (0.3 mL, 1.922 mmol) in dry DMF (2 mL) as described for compound 6a followed by purification on silica gel column chromatography using EtOAc/Pet ether (1:19) as eluent furnished the isochroman derivative 10c (25 mg, 78 % combined with 11c) as a Yellowish liquid.

Physical appearance: Yellowish liquid.

$R_f$: 0.6 (1:9, EtOAc:Pet ether).

IR (neat): 2923, 1687, 1450, 1334, 1268, 1210, 1152, 988, 907, 811 cm$^{-1}$.

$^1$H NMR (500 MHz, CDCl$_3$): $\delta$ 7.99 (dd, $J$ = 8.5, 1.5 Hz, 2H), 7.56 (t, $J$ = 7.5 Hz, 1H), 7.46 (t, $J$ = 8.0 Hz, 2H), 7.10-7.00 (m, 6H), 6.67 (d, $J$ = 7.5 Hz, 1H), 5.95 (s, 1H), 4.65-4.60 (m, 1H), 3.57 (ABX, $J$ = 17.0, 6.0 Hz, 1H), 3.23 (ABX, $J$ = 17.0, 7.0 Hz, 1H), 3.05-2.95 (m, 2H), 2.27 (s, 3H), 2.27 (s, 3H).

$^{13}$C NMR (125 MHz, CDCl$_3$, DEPT): $\delta$ 197.8 (C), 137.8 (C), 136.9 (C), 135.5 (C), 135.5 (C), 133.8 (C), 133.3 (CH), 131.0 (C), 130.7 (CH), 129.5 (CH), 129.0 (CH), 128.7 (2xC)},
128.6 (CH), 128.3 (2xCH), 126.6 (CH), 126.3 (CH), 123.0 (CH), 78.8 (CH), 71.8 (CH), 45.4 (CH₂), 34.8 (CH₂), 21.1 (CH₃), 19.2 (CH₃).

LRMS (ESI, M+ Na⁺): m/z 379.

HRMS (ESI, M+Na⁺): m/z calcd. for C₂₅H₂₄NaO₂ 379.1669, found 379.1662.

2-(1-(2,5-dimethylphenyl)-1H-isochromen-3-yl)-1-phenylethan-1-one (11c):

The chloro vinylogous ester 9c (50 mg, 0.128 mmol) was treated with Pd(OAc)₂ (2 mg, 5 mol %), PPh₃ (4 mg, 10 mol%) and Et₃N (0.3 mL, 1.922 mmol) in dry DMF (2 mL) as described for compound 6a followed by purification on silica gel column chromatography using EtOAc/Pet ether (1:19) as eluent furnished the isochromene derivative 11c (25 mg, 78 % combined with 10c) as a Yellowish liquid.

Physical appearance: Yellowish liquid.

Rf: 0.6 (1:9, EtOAc:Pet ether).

IR (neat): 3044, 2923, 1687, 1600, 1450, 1334, 1210, 1153, 1106, 988, 907, 811, 751 cm⁻¹.

¹H NMR (500 MHz, CDCl₃): δ 7.94 (dd, J = 8.5, 1.0 Hz, 2H), 7.54 (t, J = 7.5 Hz, 1H), 7.40 (t, J = 8.0 Hz, 2H), 7.20-7.10 (m, 6H), 6.51 (d, J = 7.5 Hz, 1H), 6.29 (s, 1H), 5.96 (s, 1H), 3.85 (AB quart., J = 15.7 Hz, 2H), 2.25 (s, 3H), 2.23 (s, 3H).

¹³C NMR (125 MHz, CDCl₃, DEPT): δ 195.8 (C), 151.2 (C), 139.6 (C), 137.2 (C), 136.5 (C), 134.0 (CH), 133.9 (C), 131.8 (CH), 130.2 (CH), 129.8 (C), 129.2 (CH), 128.7 (2xCH), 128.6 (2xCH), 128.2 (CH), 126.7 (CH), 126.1 (CH), 125.0 (C), 124.8 (CH), 104.7 (CH) 78.0 (CH), 44.4 (CH₂), 21.2 (CH₃), 19.1 (CH₃).

LRMS (ESI, M+ Na⁺): m/z 377.

HRMS (ESI, M+Na⁺): m/z calcd. for C₂₅H₂₂NaO₂ 377.1512, found 377.1517.

(E)-ethyl 3-(3-methylene-1,2,3,4-tetrahydronaphthalen-1-yloxy)acrylate (14):
The chloro vinylogous carbonate 5m (79 mg, 0.27 mmol) was treated with Pd(OAc)$_2$ (3.0 mg, 5 mol %), PPh$_3$ (7.05 mg, 10 mol%), and Et$_3$N (0.561 mL, 4.03 mmol) in dry DMF (4 mL) as described for compound 6a followed by purification on silica gel column chromatography using EtOAc/Hexanes (1:19) as eluent furnished the tetrahydronaphthalene derivative 14 (50 mg, 72 %) as a viscous liquid.

**Physical appearance:** viscous liquid.

**R$_f$:** 0.5 (1:9, EtOAc:Hexanes).

**IR (neat):** 2982, 2930, 1705, 1640, 1456, 1371, 1199, 1128, 1044, 955, 754 cm$^{-1}$.

**$^1$H NMR (400 MHz, CDCl$_3$):** $\delta$ 7.68 (d, $J = 12.4$ Hz, 1H), 7.30-7.25 (m, 2H), 7.22 (d, $J = 7.6$ Hz, 1H), 7.16 (d, $J = 7.6$ Hz, 1H), 5.37 (d, $J = 12.4$ Hz, 1H), 5.21 (t, $J = 5.2$ Hz, 1H), 5.04 (s, 1H), 4.97 (d, $J = 1.2$ Hz, 1H), 4.17 (q, $J = 7.2$ Hz, 2H), 3.59 (AB quart., $J = 18.8$ Hz, 2H), 2.76 (d, $J = 5.2$ Hz, 2H), 1.28 (t, $J = 7.2$ Hz, 3H).

**$^{13}$C NMR (100 MHz, CDCl$_3$, DEPT):** $\delta$ 168.2 (C), 161.4 (CH), 139.5 (C), 136.8 (C), 133.8 (C), 129.1 (CH), 128.8 (CH), 128.6 (CH), 126.4 (CH), 122.4 (CH$_2$), 98.3 (CH), 80.2 (CH), 59.9 (CH$_2$), 37.7 (CH$_2$), 36.6 (CH$_2$), 14.5 (CH$_3$).

**LRMS (ESI, M+ Na$^+$):** m/z 281.

**HRMS (ESI, M+Na$^+$):** m/z calcd. for C$_{16}$H$_{18}$O$_3$Na 281.1148, found 281.1147.

**ethyl 2-((1S,3S)-1-methylisochroman-3-yl)acetate (16):**

Isochromene derivative 6c (54 mg, 0.232mmol) was dissolved in ethyl acetate (8 ml) in a flame dried RB flask. Then 10% Pd/C (10.8 mg, 20% W/W) was added and stirred under hydrogen atmosphere (H$_2$ balloon). Once starting material was consumed (TLC control), reaction mixture was filtered through celite pad. Resulting solution was concentrated and
purified by silica gel column chromatography using ethyl acetate-Pet ether (1:9) to furnish
ischroman derivative 16 (43 mg, 80%) as colourless liquid.

**Physical appearance:** colourless liquid.

$R_f$: 0.4 (1:9, EtOAc:Pet ether).

**IR (neat):** 3065, 2981, 2933, 1733, 1493, 1450, 1399, 1338, 1219, 1150, 1104, 1042, 1027,
948, 857, 764 cm$^{-1}$.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.20-7.15 (m, 2H), 7.10 (td, $J = 7.6$, 1.8 Hz, 2H), 4.90 (q, $J =
6.4$ Hz, 1H), 4.20-4.15 (m, 3H), 2.85-2.75 (m, 2H), 2.74 (ABX, $J = 15.6$, 7.6 Hz, 1H), 2.58
(ABX, $J = 15.6$, 5.6 Hz, 1H), 1.54 (d, $J = 6.4$ Hz, 3H), 1.29 (t, $J = 7.2$ Hz, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$, DEPT): $\delta$ 171.2 (C), 139.3 (C), 133.1 (C), 128.8 (CH), 126.5
(CH), 126.4 (CH), 124.5 (CH), 73.5 (CH), 71.2 (CH), 60.6 (CH$_2$), 41.4 (CH$_2$), 34.6 (CH$_2$),
21.8 (CH$_3$), 14.3 (CH$_3$).

**LRMS (ESI, M+H$^+$):** m/z 235.

**HRMS (ESI, M+H$^+$):** m/z calcd. for C$_{14}$H$_{18}$O$_3$ 235.1334, found 235.1345.

**3,3a,5,9b-tetrahydro-2H-furo[3,2-c]isochromen-2-one (17):**

To a stirred solution of isochromene 6a (120 mg, 0.550 mmol) in dry CH$_2$Cl$_2$, $m$-CPBA (123
mg, 0.7151 mmol) was added at 0 °C. Resulting mixture was stirred at room temperature until
disappearance of starting material. Reaction mixture was quenched with sat. NaHCO$_3$
solution and extracted with CH$_2$Cl$_2$. Combined organic layer was dried over Na$_2$SO$_4$ and
concentrated under reduced pressure. This crude reaction mixture was dissolved in freshly
distilled dry CH$_2$Cl$_2$, cooled to 0 °C and triethylsilane (96 µl, 0.605 mmol) and TMSOTf (109
µl, 0.605 mmol) were successively added. Reaction mixture was stirred at 0 °C until complete
consumption of starting material (TLC control). Reaction was quenched with sat. NaHCO$_3$
solution, extracted with CH$_2$Cl$_2$. Combined organic layer was dried (anhyd. Na$_2$SO$_4$) and
concentrated. The residue was purified on a silica gel column using ethyl acetate-Pet ether (4:6) to furnish lactone 17 (65 mg, 62%) as a white solid.

**Physical appearance:** White solid.

**R_f:** 0.3 (4:6, EtOAc:Pet ether).

**M.P.:** 76-78 °C.

**IR (neat):** 2916, 2873, 1780, 1766, 1599, 1340, 1268, 1192, 1156, 1089, 948, 885, 948 cm⁻¹.

**1H NMR (400 MHz, CDCl₃):** \( \delta \) 7.50 (dd, \( J = 6.8, 2.0 \) Hz, 1H), 7.40-7.35 (m, 2H), 7.11 (dd, \( J = 6.4, 2.0 \) Hz, 1H), 5.20 (d, \( J = 2.8 \) Hz, 1H), 4.79 (d, \( J = 15.2 \) Hz, 1H), 4.69 (d, \( J = 15.2 \) Hz, 1H), 4.48 (dd, \( J = 5.2, 3.2 \) Hz, 1H), 2.97 (dd, \( J = 17.6, 5.2 \) Hz, 1H), 2.76 (d, \( J = 18.0 \) Hz, 1H).

**13C NMR (100 MHz, CDCl₃, DEPT):** \( \delta \) 174.8 (C), 135.2 (C), 130.8 (CH), 129.5 (CH), 127.7 (CH), 127.7 (C), 124.5 (CH), 75.6 (CH), 73.4 (CH), 66.8 (CH₂), 37.8 (CH₂).

**LRMS (ESI, M+ H⁺):** m/z 191.

**HRMS (ESI, M+H⁺):** m/z calcd. for C₁₁H₁₀O₃ 191.0708, found 191.0714.

### 3,3a-dihydro-2H-furo[3,2-c]isochromene-2,5(9bH)-dione (18):

To the solution of lactone derivative 17 (45 mg, 0.236 mmol) in CH₂Cl₂, PCC (152 mg, 0.710 mmol) was added portion wise under inert atmosphere. Reaction mixture was heated at 60 °C. Once starting material was consumed (TLC control), reaction mixture was passed through celite pad. Resulting solution was concentrated and the residue was purified by silica gel column chromatography using ethyl acetate-Pet ether (5:5) to give bis-lactone derivative 18 (28 mg, 58%) as a white solid.

**Physical appearance:** White solid.

**R_f:** 0.2 (5:5, EtOAc:Pet ether).

**M.P.:** 148-150 °C.

**IR (neat):** 2917, 2849, 1780, 1721, 1460, 1391, 1354, 1277, 1263, 1116, 1095, 766 cm⁻¹.
$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 8.25 (d, $J = 7.6$ Hz, 1H), 7.76 (t, $J = 7.4$ Hz, 1H), 7.67 (t, $J = 7.4$ Hz, 1H), 7.58 (d, $J = 7.6$ Hz, 1H), 5.43 (d, $J = 2.8$ Hz, 1H), 5.39 (q, $J = 2.8$ Hz, 1H), 3.08 (d, $J = 2.8$ Hz, 2H).

$^{13}$C NMR (100 MHz, CDCl$_3$, DEPT): $\delta$ 173.0 (C), 161.9 (C), 135.0 (CH), 131.7 (CH), 131.5 (C), 130.9 (CH), 130.3 (CH), 123.9 (C), 76.0 (CH), 73.8 (CH), 38.0 (CH$_2$).

LRMS (ESI, M+ Na$^+$): m/z 205.

HRMS (ESI, M+H$^+$): m/z calcd. for C$_{11}$H$_8$O$_4$ 205.0501, found 205.0511.
NOE enhancement = 6%