Electronic Supplementary Material (ESI) for RSC Advances.
This journal is © The Royal Society of Chemistry 2014

Supporting Information

Synthesis of Isochromene Derivatives using Intramolecular Benzylic C(sp^3)-C(sp^2) Bond Forming Heck Reaction on Vinylogous Carbonates

Santosh J. Gharpure,[a*] Yogesh G. Shelke,[a] S. Raja Bhushan Reddy[b]

[a]*Department of Chemistry, Indian Institute of Technology Bombay,Powai, Mumbai – 400076, India*

[b]*Department of Chemistry, Indian Institute of Technology Madras, Chennai – 600036, Tamil Nadu, India*

sjgharpure@iitb.ac.in

Table of contents

Genreral:

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:

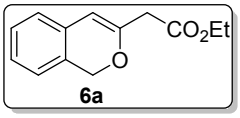
A Schlenk tube with a magnetic stir bar was charged with ethyl propiolate (1.1 mmol), chloroalcohol **15** (1.0 mmol), NMM (1.1 mmol) and Et_3N (15 mmol). The reaction mixture was stirred for 4 hrs at room temperature until completion of starting material (TLC control) and then $Pd(OAc)_2$ (0.05 mmol), 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 ^oC, and the mixture was stirred for 2h. Reaction was quenched with sat. NH_4Cl solution and extracted with EtOAc (3x 10 mL) and dried (anhyd. Na_2SO_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_3N (0.4 mL, 2.94 mmol) and dry DMF (2 mL) were added and it was heated to 100 ^oC 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_4Cl solution (20 mL) and dried (anhyd. Na_2SO_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.



6a

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}.

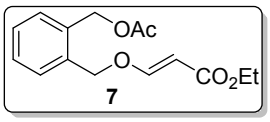
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.20 (t, *J* = 7.4 Hz, 1H), 7.13 (t, *J* = 7.4 Hz, 1H), 6.99 (d, *J* = 7.4 Hz, 1H), 6.95 (d, *J* = 7.4 Hz, 1H), 5.80 (s, 1H), 5.10 (s, 2H), 4.20 (q, *J* = 7.2 Hz, 2H), 3.22 (s, 2H), 1.28 (t, *J* = 7.2 Hz, 3 H).

^{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):



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}.

^{1}H NMR (400 MHz, $CDCl_3$): δ 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).

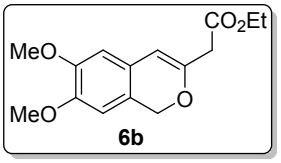
^{13}C NMR (100 MHz, $CDCl_3$, DEPT): δ 170.8 (C), 167.7 (C), 161.8 (CH), 134.5 (C), 133.9 (C), 130.2 (CH), 129.4 (CH), 129.2 (CH), 129.0 (CH), 97.8 (CH), 70.6 (CH_2), 63.8 (CH_2), 60.1 (CH_2), 21.1 (CH_3), 14.5 (CH_3).

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_3N (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.



6b

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}.

^{1}H NMR (400 MHz, $CDCl_3$): δ 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).

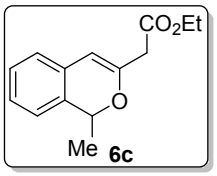
^{13}C NMR (100 MHz, $CDCl_3$, DEPT): δ 170.0 (C), 149.5 (C), 148.9 (C), 147.9 (C), 124.3 (C), 119.7 (C), 108.0 (CH), 107.2 (CH), 104.2 (CH), 68.9 (CH_2), 61.2 (CH_2), 56.3 (CH_3), 56.2 (CH_3), 39.8 (CH_2), 14.3 (CH_3).

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_3N (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, $CDCl_3$): δ 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).

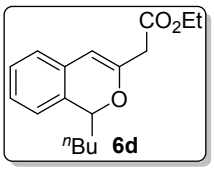
^{13}C NMR (100 MHz, $CDCl_3$, DEPT): δ 169.8 (C), 149.2 (C), 132.0 (C), 130.5 (C), 127.9 (CH), 126.7 (CH), 123.4 (CH), 123.3 (CH), 103.6 (CH), 74.7 (CH), 61.2 (CH_2), 40.2 (CH_2), 20.1 (CH_3), 14.3 (CH_3).

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

HRMS (ESI, M+Na^+): m/z calcd. for $C_{14}H_{16}O_3Na$ 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_3N (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 (dddd, *J* = 14.8, 9.6, 4.8, 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).

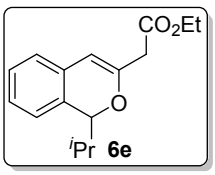
^{13}C NMR (100 MHz, $CDCl_3$, DEPT): δ 169.8 (C), 148.8 (C), 130.9 (C), 130.3 (C), 127.7 (CH), 126.3 (CH), 124.0 (CH), 123.3 (CH), 103.2 (CH), 78.6 (CH), 61.1 (CH_2), 40.2 (CH_2), 33.9 (CH_2), 27.3 (CH_2), 22.6 (CH_2), 14.2 (CH_3), 14.1 (CH_3).

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_3N (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.

R_f: 0.4 (1:9, EtOAc:Hexanes).

IR (neat): 2975, 2962, 2927, 1740, 1656, 1489, 1468, 1455, 1383, 1367, 1149, 1015 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): 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).

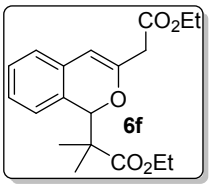
^{13}C NMR (100 MHz, $CDCl_3$, DEPT): δ 169.8 (C), 149.2 (C), 130.8 (C), 129.3 (C), 127.8 (CH), 126.0 (CH), 125.3 (CH), 123.3 (CH), 103.0 (CH), 83.9 (CH), 61.2 (CH_2), 40.2 (CH_2), 31.9 (CH), 19.0 (CH_3), 17.8 (CH_3), 14.3 (CH_3).

LRMS (ESI, M+ H^+): m/z 261.

HRMS (ESI, M+H^+): m/z calcd. for $C_{16}H_{21}O_3$ 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 %), PPh_3 (7 mg, 10 mol%) and Et_3N (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.

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

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

^{1}H NMR (400 MHz, $CDCl_3$): 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).

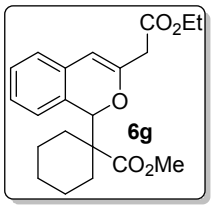
^{13}C NMR (100 MHz, $CDCl_3$, DEPT): δ 175.8 (C), 169.5 (C), 149.6 (C), 131.5 (C), 128.4 (CH), 126.1 (CH), 125.9 (CH), 125.8 (C), 123.5 (CH), 102.5 (CH), 82.6 (CH), 61.2 (CH_2), 60.9 (CH_2), 50.7 (C), 40.2 (CH_2), 21.1 (CH_3), 20.4 (CH_3), 14.2 (CH_3), 14.2 (CH_3).

LRMS (ESI, M+ Na^+): m/z 355.

HRMS (ESI, M+Na^+): m/z calcd. for $C_{19}H_{24}O_5Na$ 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_3N (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}H NMR (400 MHz, $CDCl_3$): δ 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).

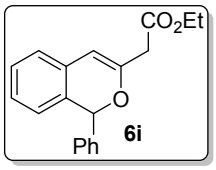
^{13}C NMR (100 MHz, $CDCl_3$, DEPT): δ 174.5 (C), 169.4 (C), 149.9 (C), 131.4 (C), 128.5 (CH), 126.5 (C), 125.9 (CH), 125.4 (CH), 123.3 (CH), 102.6 (CH), 84.3 (CH), 61.2 (CH_2), 55.7 (C), 51.8 (CH_3), 40.2 (CH_2), 30.5 (CH_2), 29.2 (CH_2), 25.6 (CH_2), 23.7 (CH_2), 23.1 (CH_2), 14.3 (CH_3).

LRMS (ESI, M+ H^+): m/z 359.

HRMS (ESI, M+H^+): m/z calcd. for $C_{21}H_{27}O_5$ 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_3N (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, 1598, 1453, 1374, 1189, 1050, 1027, 933, 758, 700 cm^{-1}.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.36 (s, 5H), 7.22 (td, *J* = 6.0, 0.8 Hz, 1H), 7.09 (td, *J* = 6.0, 0.8 Hz, 1H), 7.03 (d, *J* = 6.0 Hz, 1H), 6.69 (d, *J* = 6.0 Hz, 1H), 6.16 (s, 1H), 5.86 (s, 1H), 4.15-4.00 (m, 2H), 3.21 (AB quart., *J* = 12.8 Hz, 2H), 1.20 (t, *J* = 6.6 Hz, 3H).

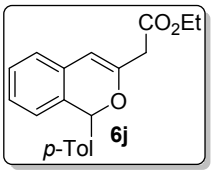
^{13}C NMR (125 MHz, $CDCl_3$, DEPT): δ 169.6 (C), 149.7 (C), 139.9 (C), 131.1 (C), 129.89 (C), 128.5 (3xCH), 128.3 (2xCH), 126.7 (2xCH), 125.5 (CH), 123.3 (CH), 104.2 (CH), 80.6 (CH), 61.2 (CH_2), 40.2 (CH_2), 14.2 (CH_3).

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_3N (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.

R_f: 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, $CDCl_3$): δ 7.25 (bt, *J* = 8.0 Hz, 3H), 7.20 (bt, *J* = 8.0 Hz, 2H), 7.10 (td, *J* = 7.2, 1.2 Hz, 1H), 7.04 (bd, *J* = 7.6 Hz, 1H), 6.72 (bd, *J* = 7.2 Hz, 1H), 6.15 (s, 1H), 5.86 (s, 1H), 4.20-4.00 (m, 2H), 3.22 (s, 2H), 2.38 (s, 3H), 1.21 (t, *J* = 7.2 Hz, 3H).

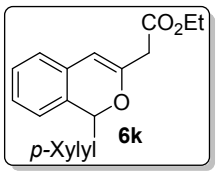
^{13}C NMR (100 MHz, $CDCl_3$, DEPT): δ 169.6 (C), 149.7 (C), 138.2 (C), 136.9 (C), 131.1 (C), 130.0 (C), 129.1 (2xCH), 128.2 (3xCH), 126.6 (CH), 125.5 (CH), 123.2 (CH), 104.1 (CH), 80.4 (CH), 61.1 (CH_2), 40.2 (CH_2), 21.3 (CH_3), 14.2 (CH_3).

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_3N (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.

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

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

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.22 (bt, *J* = 7.5 Hz, 1H), 7.15 (d, *J* = 8.0 Hz, 1H), 7.09 (bd, *J* = 7.0 Hz, 2H), 7.05 (t, *J* = 8.0 Hz, 2H), 6.55 (d, *J* = 7.5 Hz, 1H), 6.33 (s, 1H), 5.90 (s, 1H), 4.20-4.05 (m, 2H), 3.25 (AB, *J* = 16.0 Hz, 2H), 2.34 (s, 3H), 2.31 (s, 3H), 1.24 (t, *J* = 7.0 Hz, 3H).

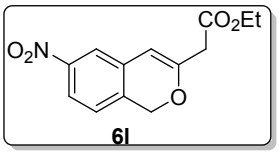
^{13}C NMR (125 MHz, $CDCl_3$, DEPT): δ 169.7 (C), 150.5 (C), 137.0 (C), 135.5 (C), 134.0 (C), 131.7 (C), 130.7 (CH), 129.8 (C), 129.6 (CH), 129.2 (CH), 128.2 (CH), 126.7 (CH), 125.0 (CH), 123.1 (CH), 104.2 (CH), 78.9 (CH), 61.1 (CH_2), 40.0 (CH_2), 21.1 (CH_3), 19.2 (CH_3), 14.2 (CH_3).

LRMS (ESI, M+ Na^+): m/z 345.

HRMS (ESI, M+Na^+): m/z calcd. for $C_{21}H_{22}NaO_3$ 345.1461, found 345.1462.

ethyl 2-(6-nitro-1H-isochromen-3-yl)acetate (6l):

The chloro vinylogous carbonate **5l** (80 mg, 0.27 mmol) was treated with $Pd(OAc)_2$ (4 mg, 5 mol %), PPh_3 (8 mg, 10 mol%) and Et_3N (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.



Physical appearance: viscous liquid.

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

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

^{1}H NMR (500 MHz, $CDCl_3$): δ 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).

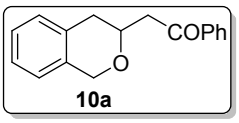
^{13}C NMR (125 MHz, $CDCl_3$, DEPT): δ 169.3 (C), 153.6 (C), 148.4 (C), 133.8 (C), 133.0 (C), 124.8 (CH), 121.6 (CH), 117.5 (CH), 103.4 (CH), 68.7 (CH_2), 61.5 (CH_2), 39.7 (CH_2), 14.3 (CH_3).

LRMS (ESI, M+ H^+): m/z 264.

HRMS (ESI, M+H^+): m/z calcd. for $C_{13}H_{14}NO_5$ 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_3N (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.



10a

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.

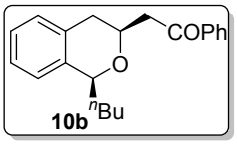
HRMS (ESI, M+Na^+): m/z calcd. for $C_{17}H_{16}NaO_2$ 275.1043, found 275.1040.

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_3N (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.



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

IR (neat): 2931, 1687, 1597, 1450, 1365, 1254, 1109, 752 cm^{-1}.

^{1}H NMR (500 MHz, $CDCl_3$): δ 8.02 (dd, *J* = 8.5, 1.5 Hz, 2H), 7.58 (t, *J* = 7.5 Hz, 1H), 7.48 (t, *J* = 8.0 Hz, 2H), 7.20-7.15 (m, 2H), 7.10-7.05 (m, 2H), 4.77 (bd, *J* = 6.5 Hz, 1H), 4.35-4.30 (m, 1H), 3.50 (ABX, *J* = 16.0, 6.5 Hz, 1H), 3.10 (ABX, *J* = 16.0, 6.0 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 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$, DEPT): δ 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 (CH_2), 35.6 (CH_2), 35.2 (CH_2), 27.2 (CH_2), 22.8 (CH_2), 14.2 (CH_3).

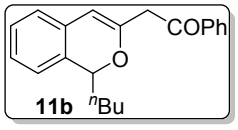
LRMS (ESI, M+ Na^+): m/z 331.

HRMS (ESI, M+Na^+): m/z calcd. for $C_{21}H_{24}NaO_2$ 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 %), PPh_3 (4 mg, 10 mol%) and Et_3N (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.



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

IR (neat): 3048, 2931, 1687, 1597, 1450, 1365, 1254, 1109, 1000, 910, 752 cm^{-1}.

^{1}H NMR (500 MHz, $CDCl_3$): δ 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).

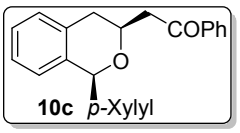
^{13}C NMR (125 MHz, $CDCl_3$, DEPT): δ 196.1 (C), 136.7 (C), 133.4 (C), 130.9 (C), 130.5 (C), 128.8 (2xCH), 128.7 (2xCH), 127.9 (CH), 126.4 (CH), 126.3 (CH), 124.0 (CH), 123.3 (CH), 103.6 (CH), 78.7 (CH), 44.4 (CH_2), 34.0 (CH_2), 27.4 (CH_2), 22.6 (CH_2), 14.1 (CH_3).

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

HRMS (ESI, M+Na^+): m/z calcd. for $C_{21}H_{22}NaO_2$ 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_3N (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, $CDCl_3$): δ 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): δ 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 (2xCH),

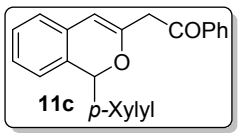
128.6 (CH), 128.3 (2xCH), 126.6 (CH), 126.3 (CH), 123.0 (CH), 78.8 (CH), 71.8 (CH), 45.4 (CH_2), 34.8 (CH_2), 21.1 (CH_3), 19.2 (CH_3).

LRMS (ESI, M+ Na^+): m/z 379.

HRMS (ESI, M+Na^+): m/z calcd. for $C_{25}H_{24}NaO_2$ 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 %), PPh_3 (4 mg, 10 mol%) and Et_3N (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.

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

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

^{1}H NMR (500 MHz, $CDCl_3$): 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).

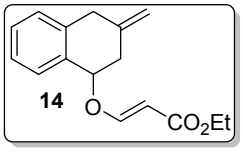
^{13}C NMR (125 MHz, $CDCl_3$, DEPT): δ 195.8 (C), 151.2 (C), 139.6 (C), 137.2 (C), 136.5 (C), 134.0 (CH), 133.9 (C), 131.8 (CH), 130.2 (CH), 129.8 (C), 129.2 (CH), 128.7 (2xCH), 128.6 (2xCH), 128.2 (CH), 126.7 (CH), 126.1 (CH), 125.0 (C), 124.8 (CH), 104.7 (CH) 78.0 (CH), 44.4 (CH_2), 21.2 (CH_3), 19.1 (CH_3).

LRMS (ESI, M+ Na^+): m/z 377.

HRMS (ESI, M+Na^+): m/z calcd. for $C_{25}H_{22}NaO_2$ 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_3N (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, $CDCl_3$): δ 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): δ 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).

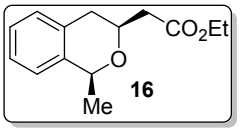
LRMS (ESI, M+ Na^+): m/z 281.

HRMS (ESI, M+Na^+): m/z calcd. for $C_{16}H_{18}O_3Na$ 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 sílica 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, $CDCl_3$): δ 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): δ 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).

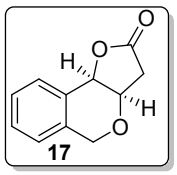
LRMS (ESI, M+ H^+): m/z 235.

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_2Cl_2, *m*-CPBA (123 mg, 0.7151 mmol) was added at 0 ^oC. 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_2Cl_2. Combined organic layer was dried over Na_2SO_4 and concentrated under reduced pressure. This crude reaction mixture was dissolved in freshly distilled dry CH_2Cl_2, cooled to 0 ^oC and triethylsilane (96 µl, 0.605 mmol) and TMSOTf (109 µl, 0.605 mmol) were successively added. Reaction mixture was stirred at 0 ^oC until complete consumption of starting material (TLC control). Reaction was quenched with sat. $NaHCO_3$ solution, extracted with CH_2Cl_2. Combined organic layer was dried (anhyd. Na_2SO_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 ^oC.

IR (neat): 2916, 2873, 1780, 1766, 1599, 1340, 1268, 1192, 1156, 1089, 948, 885, 948 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ 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).

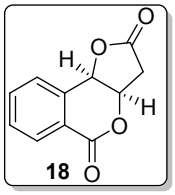
^{13}C NMR (100 MHz, $CDCl_3$, DEPT): δ 174.8 (C), 135.2 (C), 130.8 (CH), 129.5 (CH), 127.7 (CH), 127.7 (C), 124.5 (CH), 75.6 (CH), 73.4 (CH), 66.8 (CH_2), 37.8 (CH_2).

LRMS (ESI, M+ H^+): m/z 191.

HRMS (ESI, M+H^+): m/z calcd. for $C_{11}H_{10}O_3$ 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 CH_2Cl_2, PCC (152 mg, 0.710 mmol) was added portion wise under inert atmosphere. Reaction mixture was heated at 60 ^oC. Once starting material was consumed (TLC control), reaction mixture was passed through celite pad. Resulting solution was concentrated and the residue was purified by sílica gel coloumn 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 ^oC.

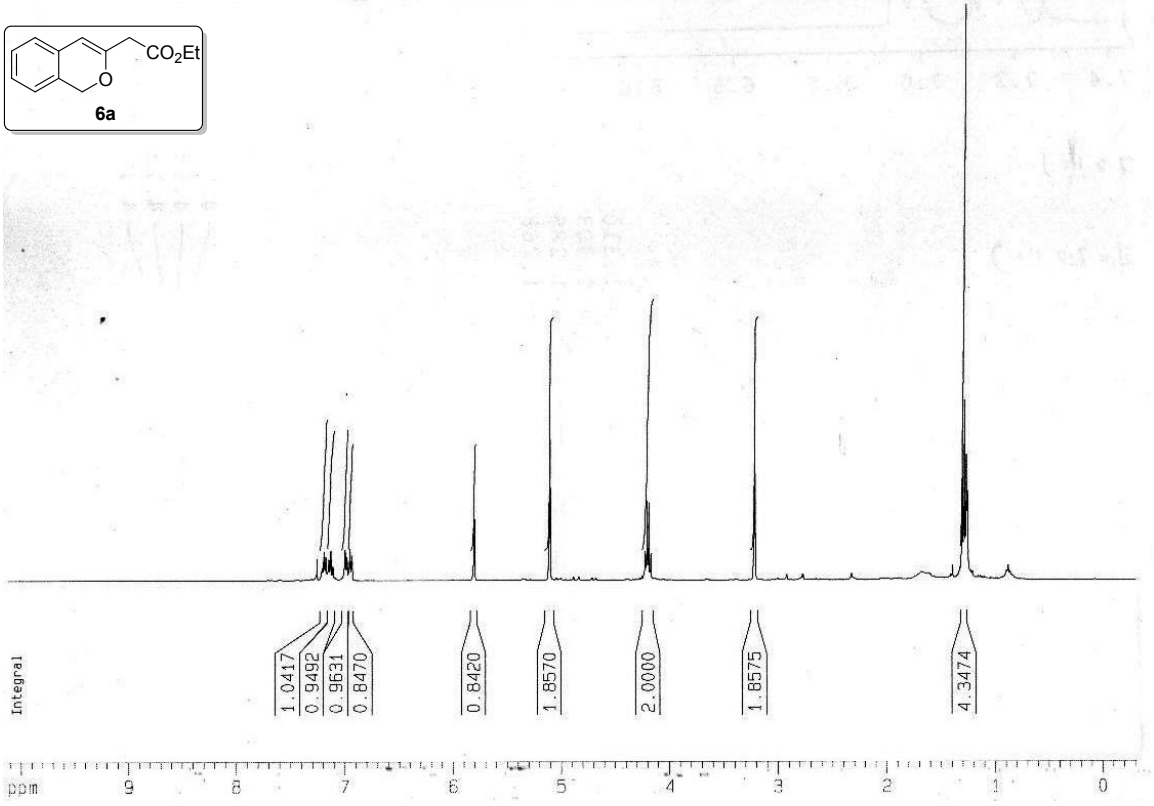
IR (neat): 2917, 2849, 1780, 1721, 1460, 1391, 1354, 1277, 1263, 1116, 1095, 766 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ 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, 1 H), 3.08 (d, *J* = 2.8 Hz, 2H).

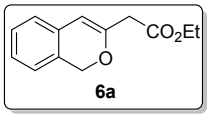
^{13}C NMR (100 MHz, $CDCl_3$, DEPT): δ 173.0 (C), 161.9 (C), 135.0 (CH), 131.7 (CH), 131.5 (C), 130.9 (CH), 130.3 (CH), 123.9 (C), 76.0 (CH), 73.8 (CH), 38.0 (CH_2).

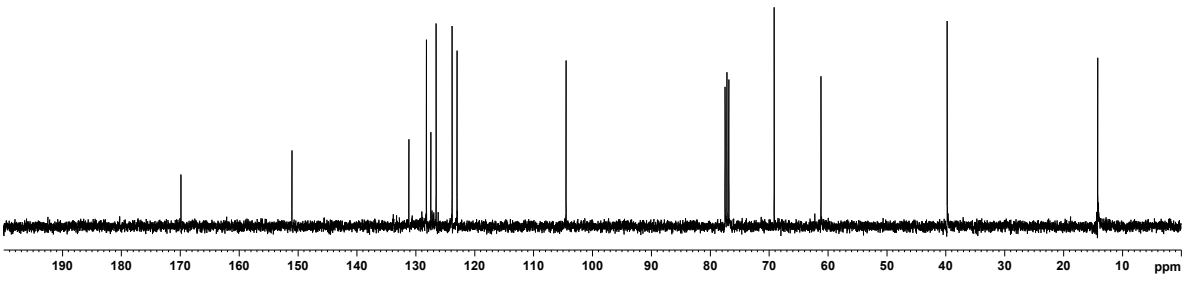
LRMS (ESI, M+ Na^+): m/z 205.

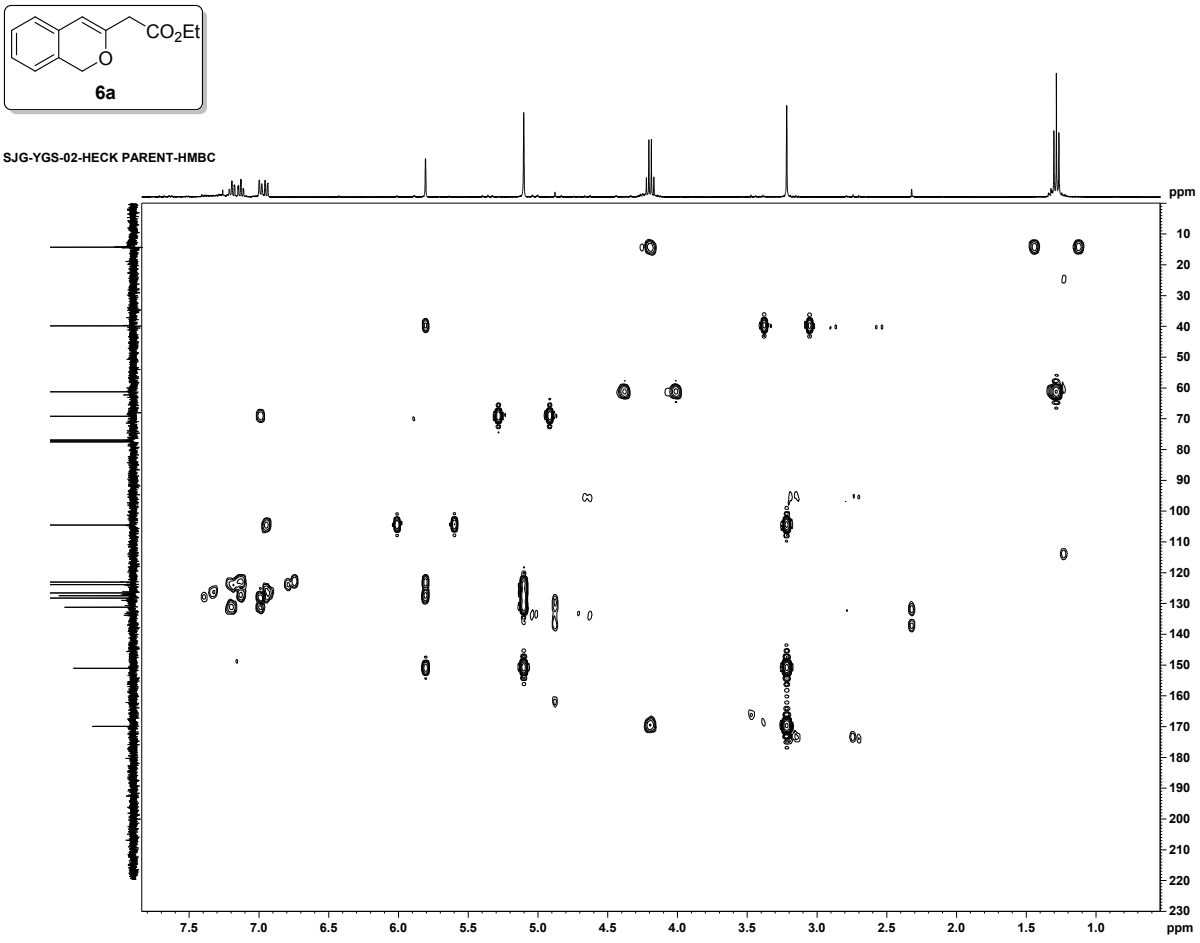
HRMS (ESI, M+H^+): m/z calcd. for $C_{11}H_8O_4$ 205.0501, found 205.0511.



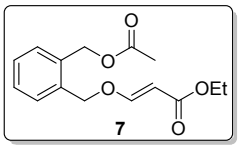
SJG-YGS-02-HECK PARENT-C13

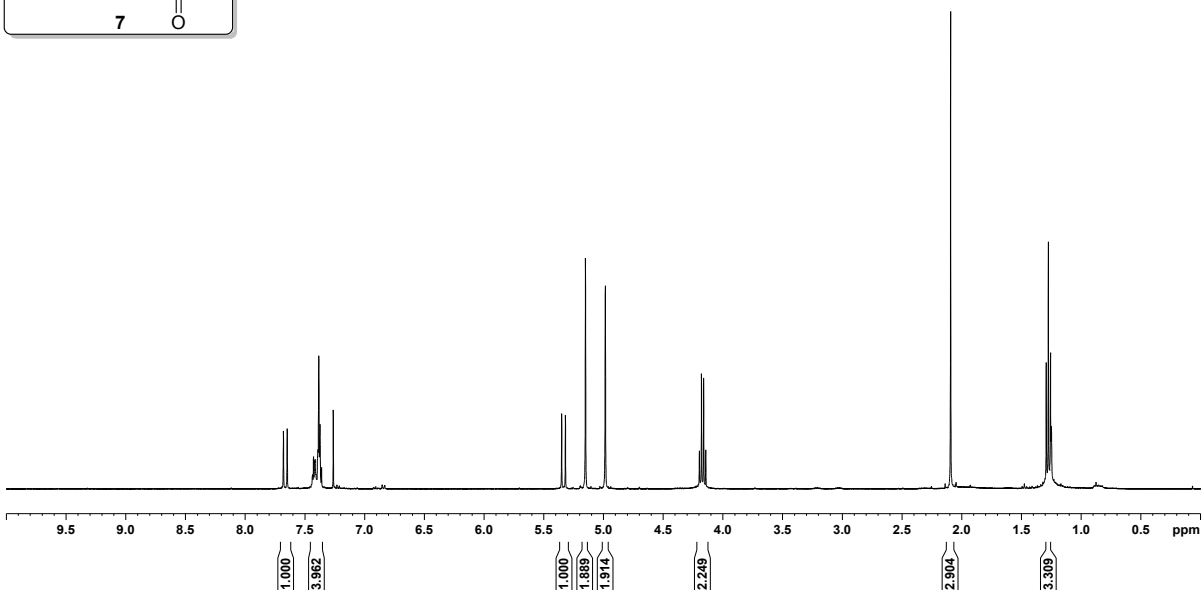




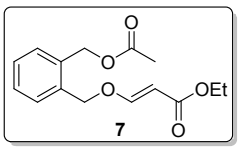

CO2Et
O
6a
SJG-YGS-02-HECK PARENT-HMBC
ppm
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

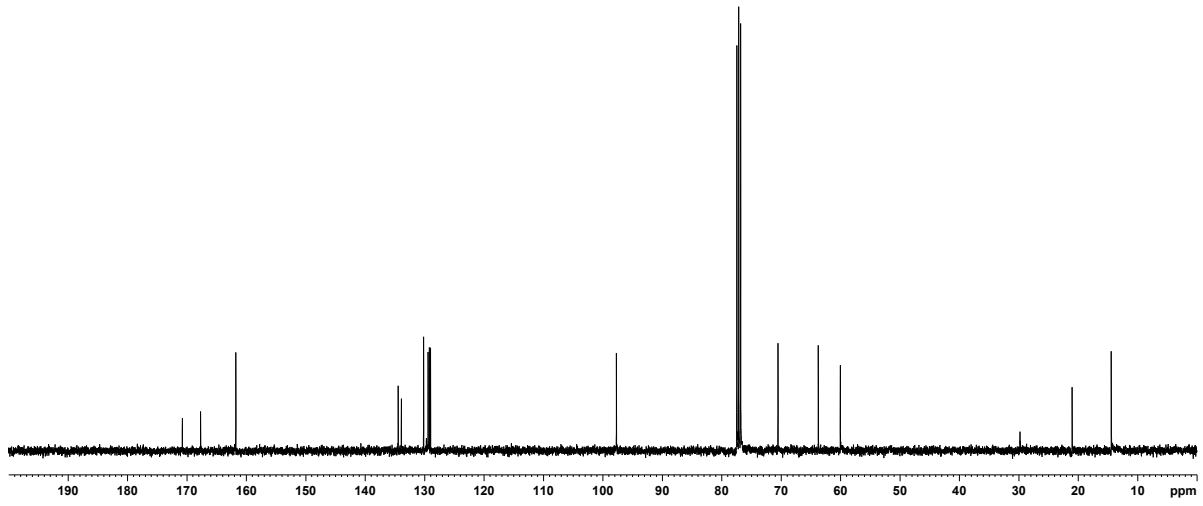
SJG-YGS-02- ACETATE-H1

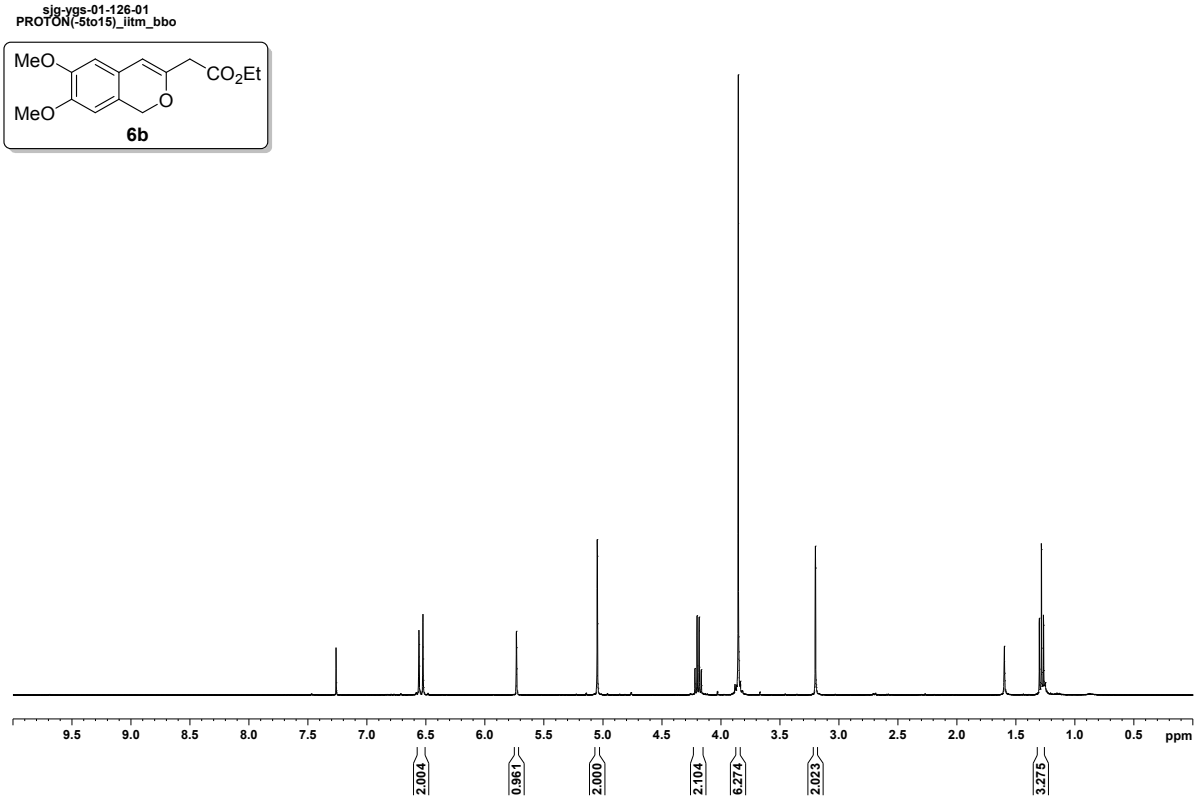


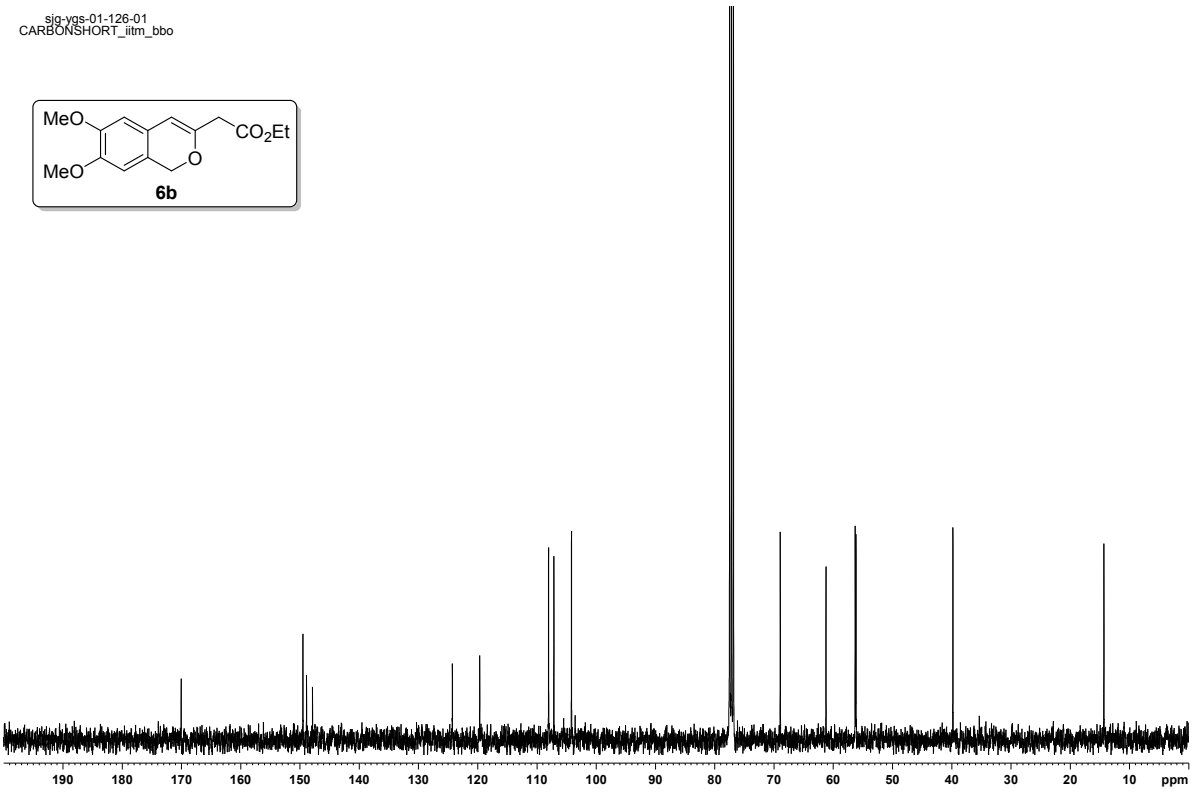


SJG-YGS-02- ACETATE-C13

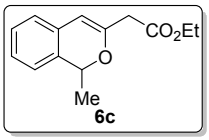


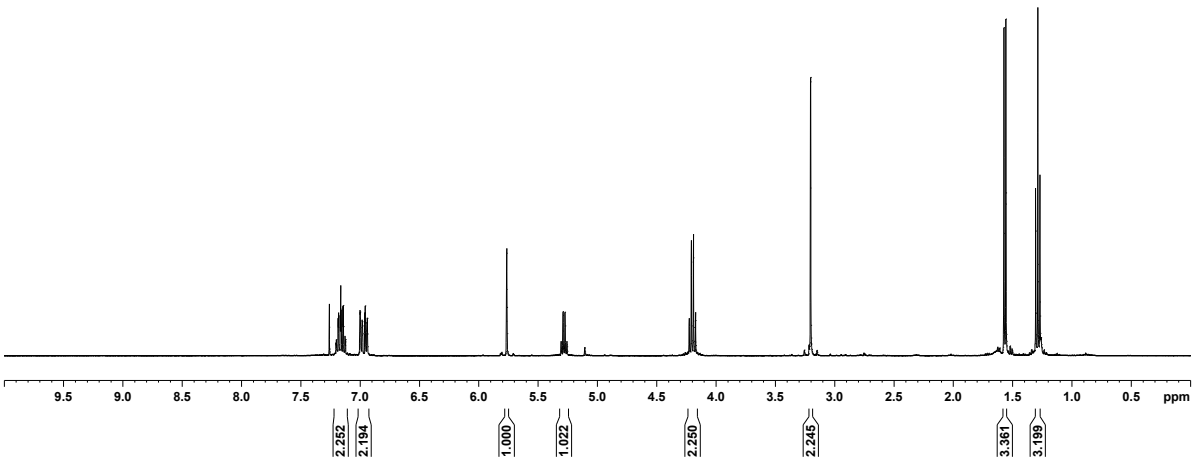



sjg-ygs-01-126-01
PROTON(-5to15)_iitm_bbo
MeO
MeO
CO2Et
O
6b
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.004
0.961
2.000
2.104
6.274
2.023
3.275

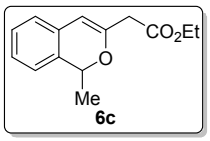

sjg-ygs-01-126-01
CARBONSHORT_iitm_bbo
MeO
MeO
CO2Et
O
6b
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

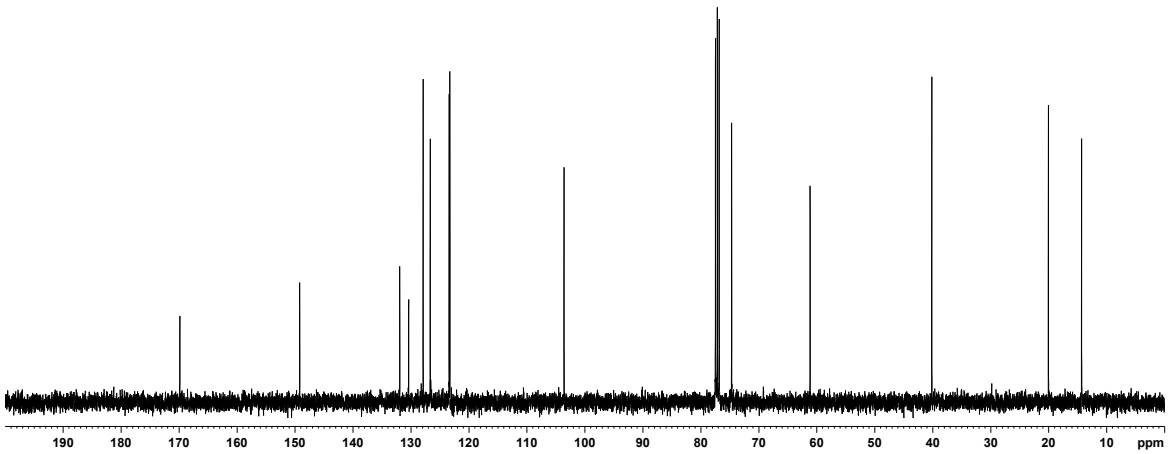
sjg-ygs-01-142-01
PROTON(-5to15)_iitm_bbo

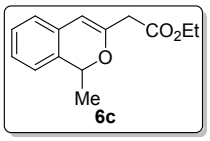




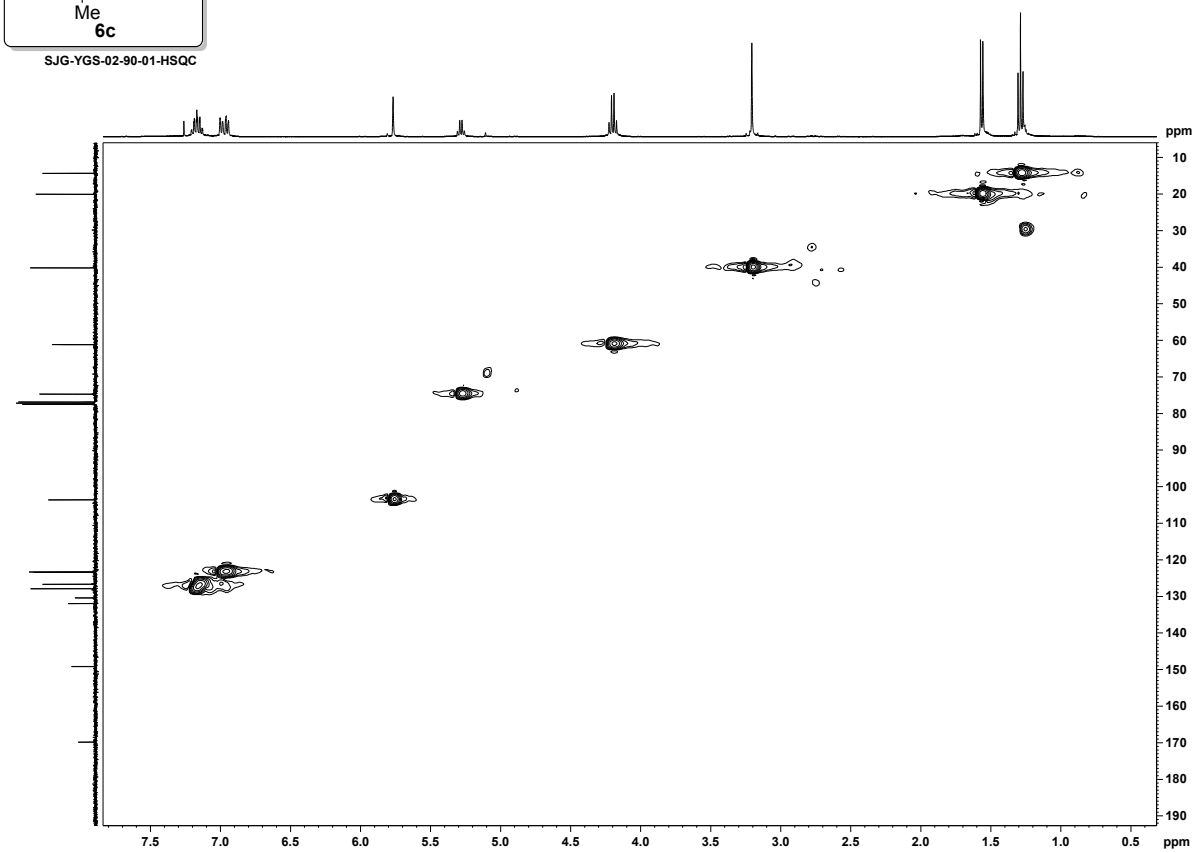
SJG-YGS-02-90-01-C13

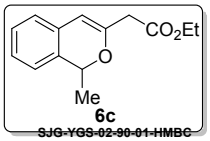




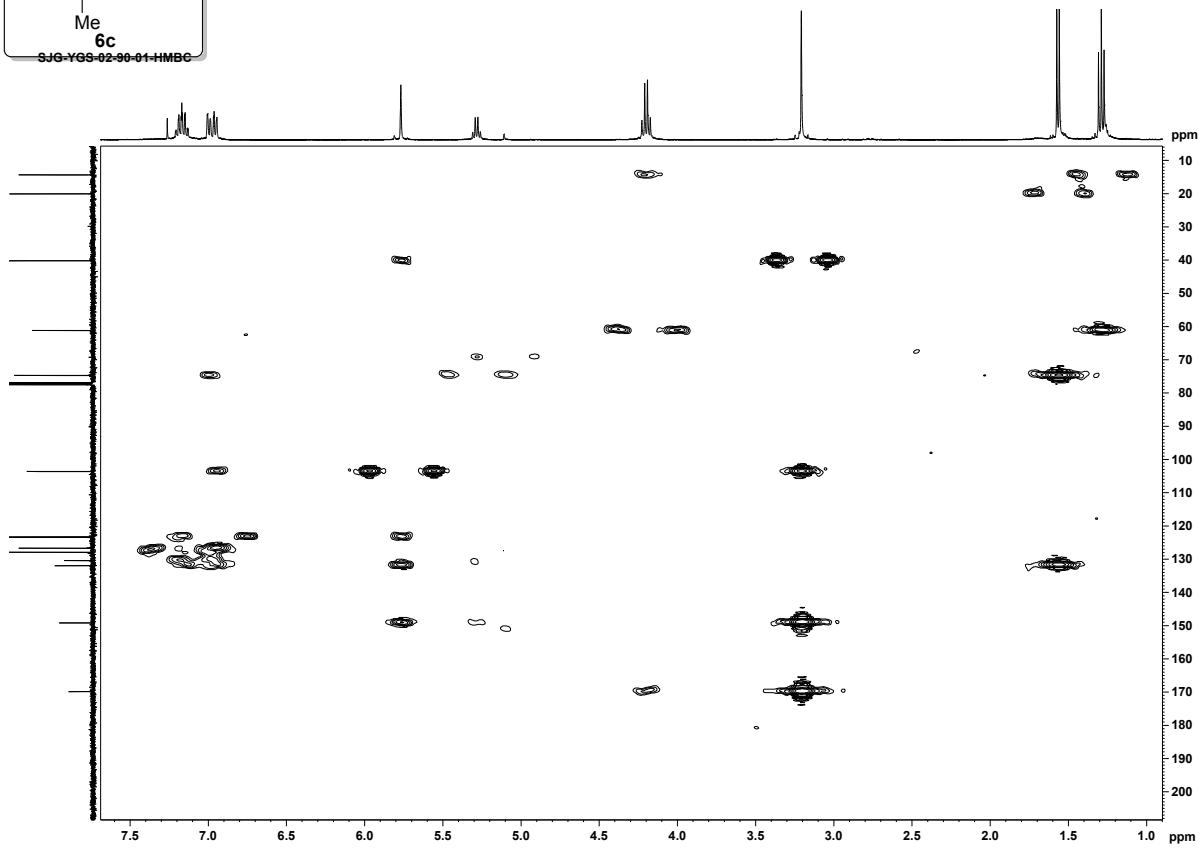


SJG-YGS-02-90-01-HSQC

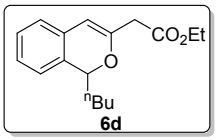




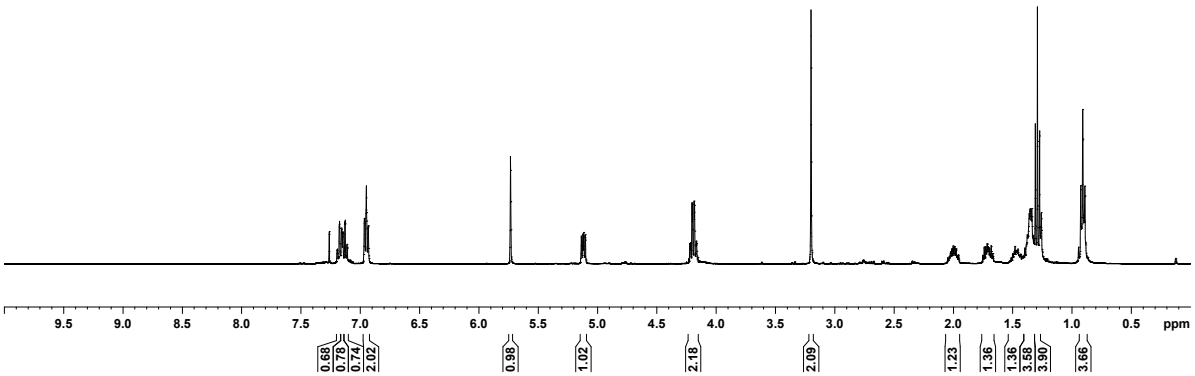
SJG-YGS-02-90-01-HMBC



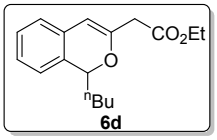
sjg-ygs-01-88-01
PROTON(-5to15)_iitm_bbo



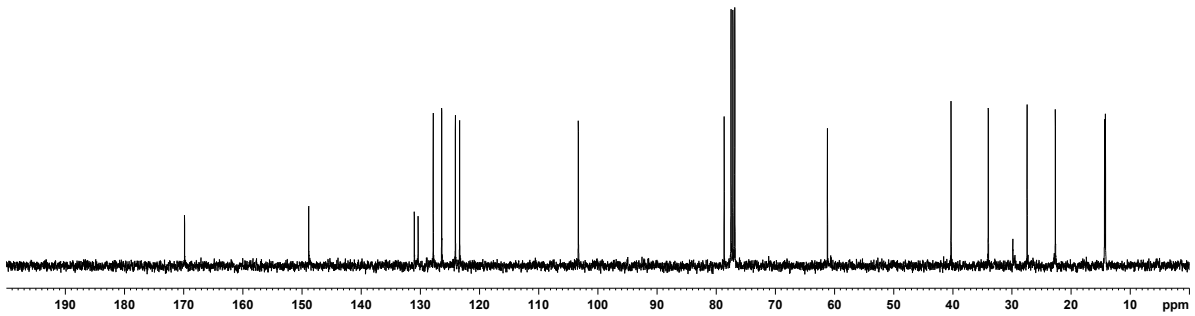
6d



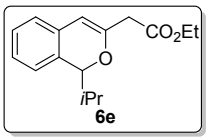
sjg-ygs-01-88-01
CARBONSHORT_iitm_bbo

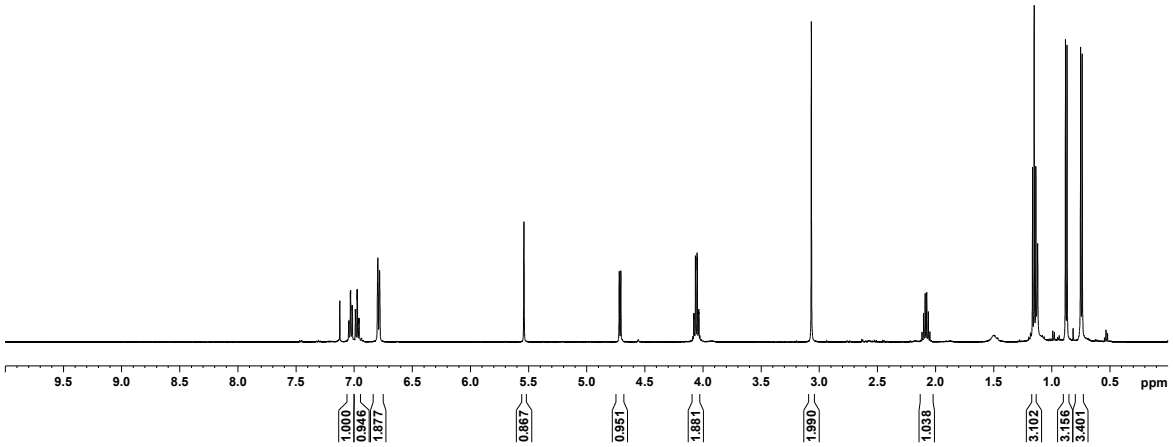


6d

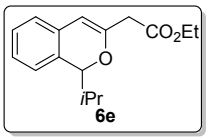


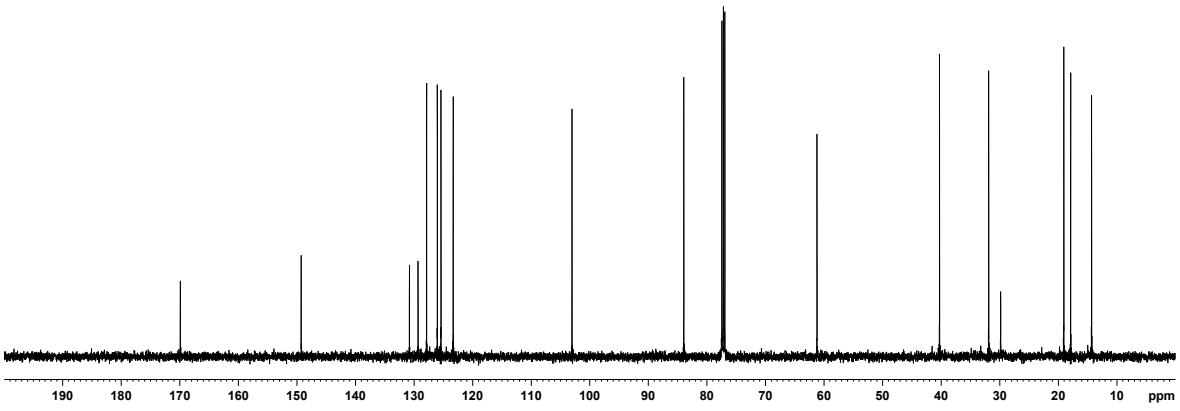
Lab SJG
yogesh
iitm_PROTON CDCl3 /opt/topspin iitm 5



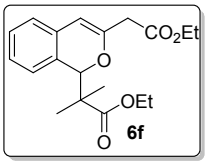


Lab SJG
yogesh
iitm_carbonshort CDCl3 /opt/topspin iitm 5

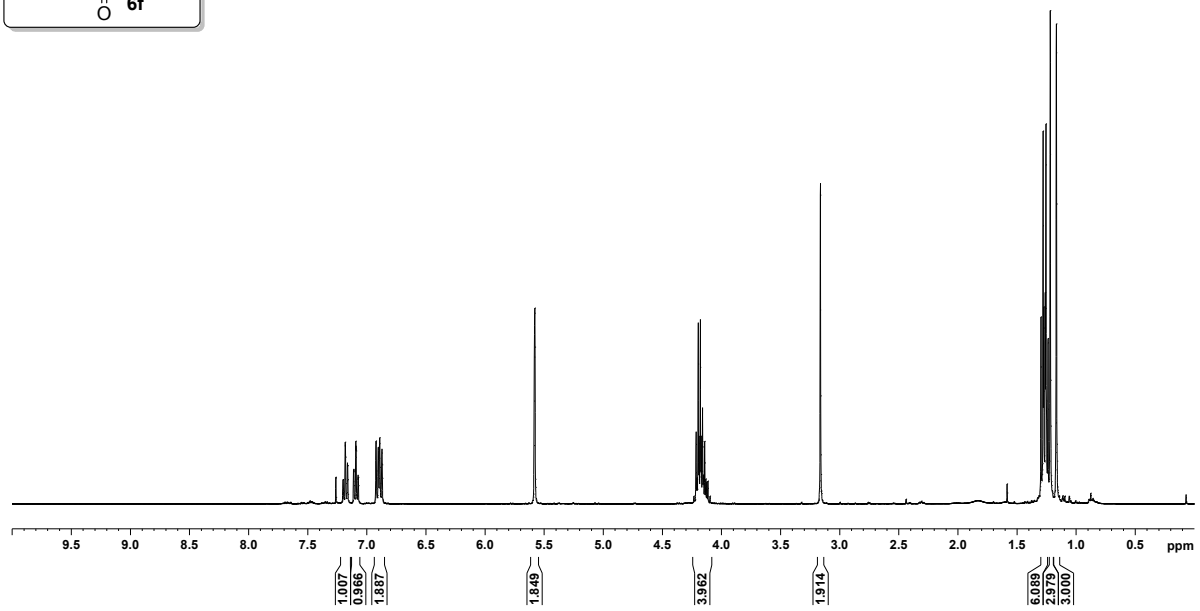




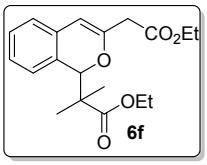
sg-srb-cl-al-isobutyrate-heck
PROTON(-5to15)_iitm_bbo



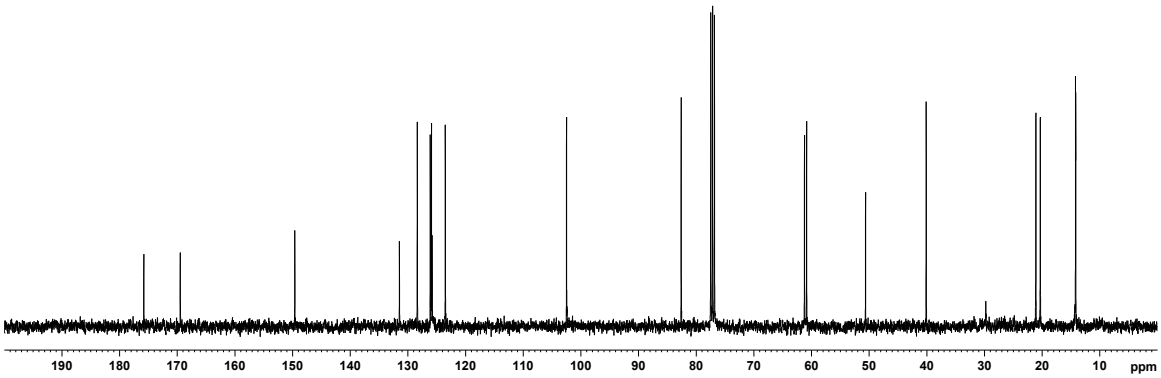
6f

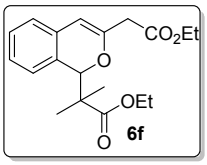


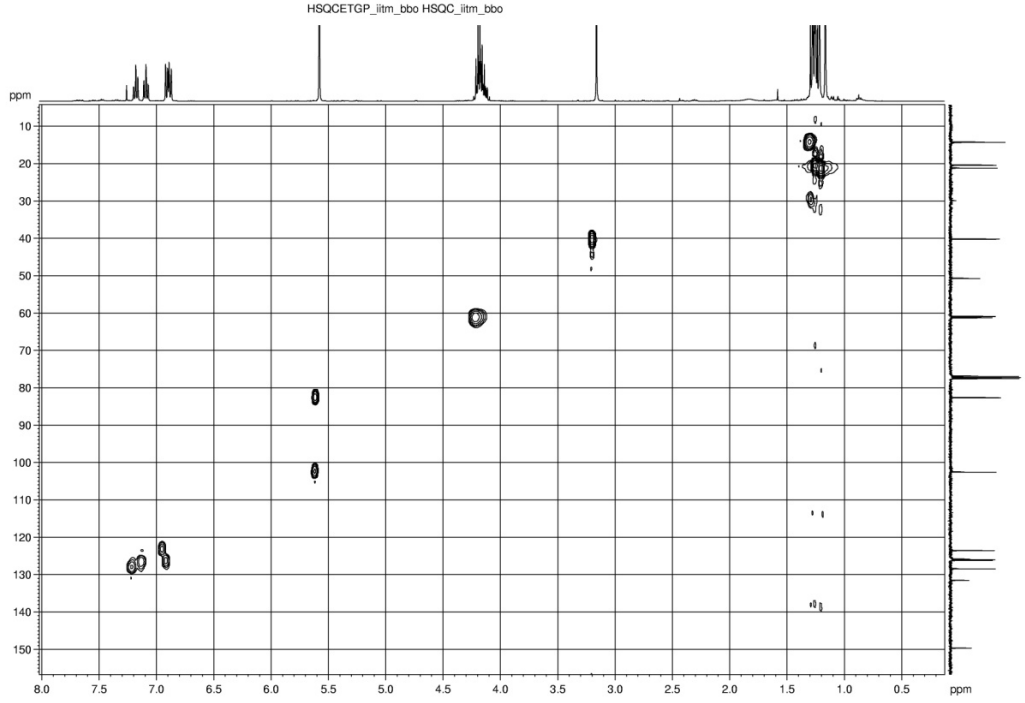
sg-srb-cl-al-isobutyrate-heck
CARBONSHORT_iitm_bbo

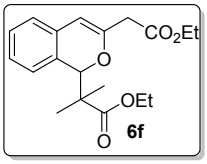


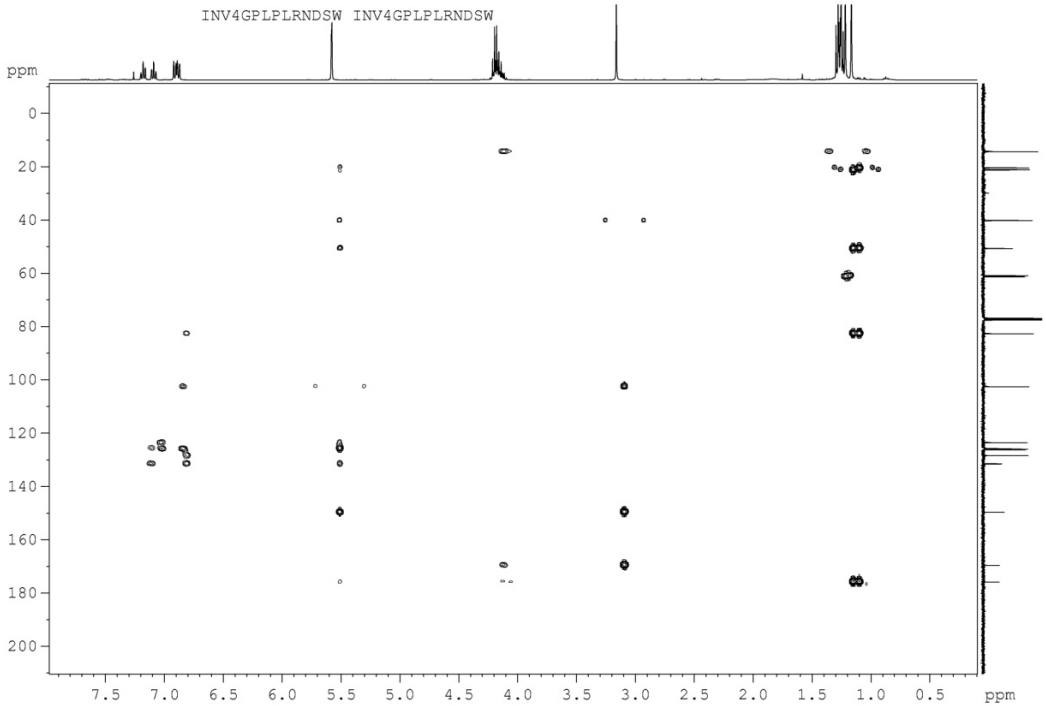
6f

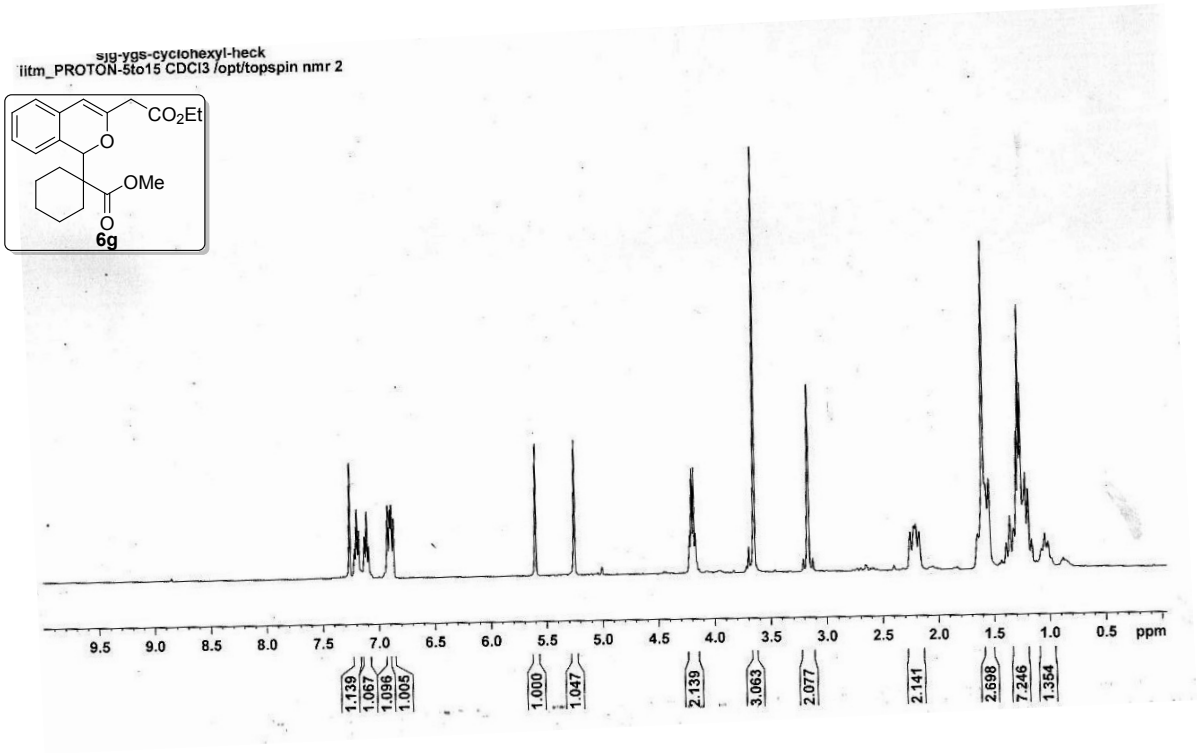


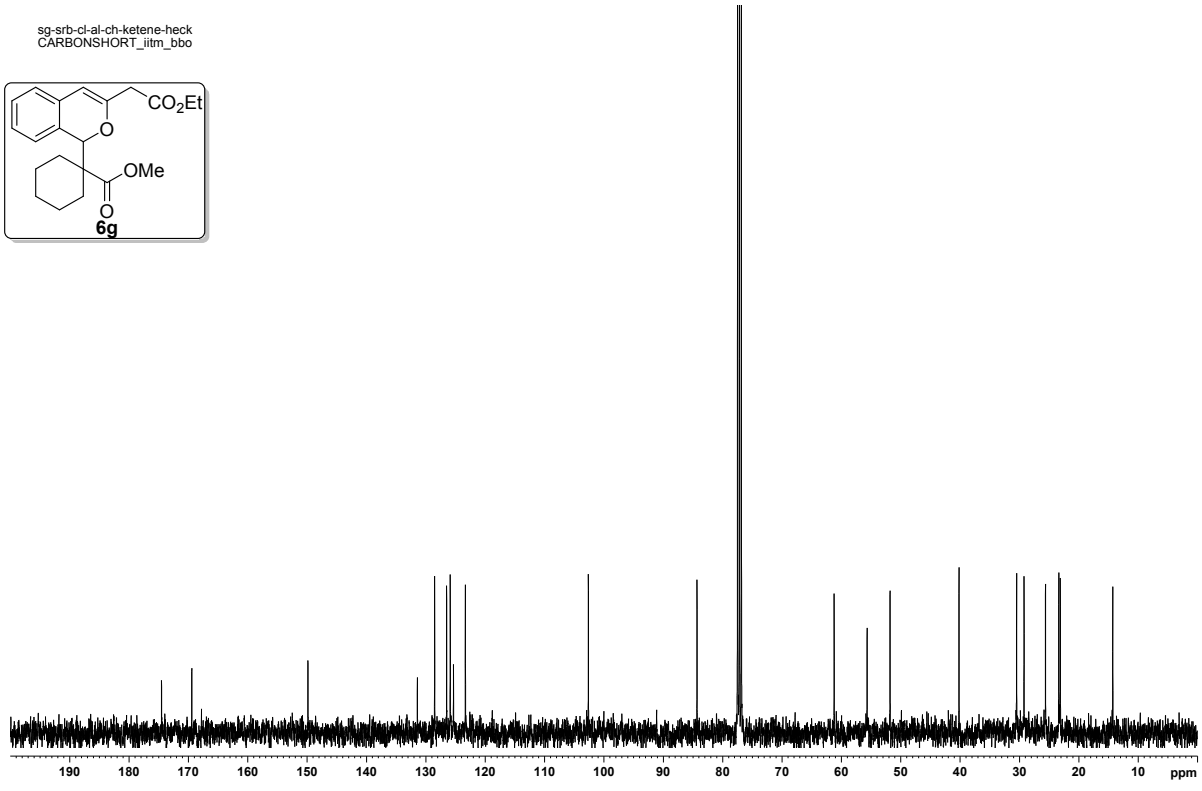

CO2Et
O
OEt
O
6f

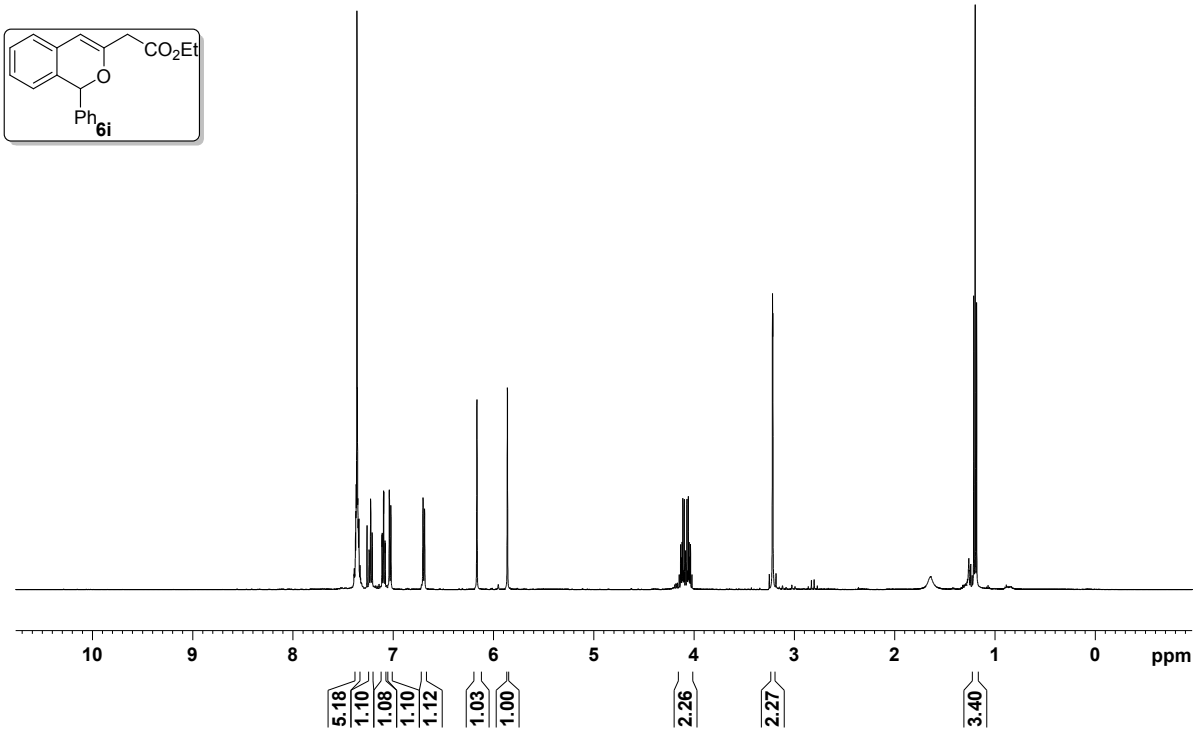

HSQCETGP_iitm_bbo HSQC_iitm_bbo
ppm
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

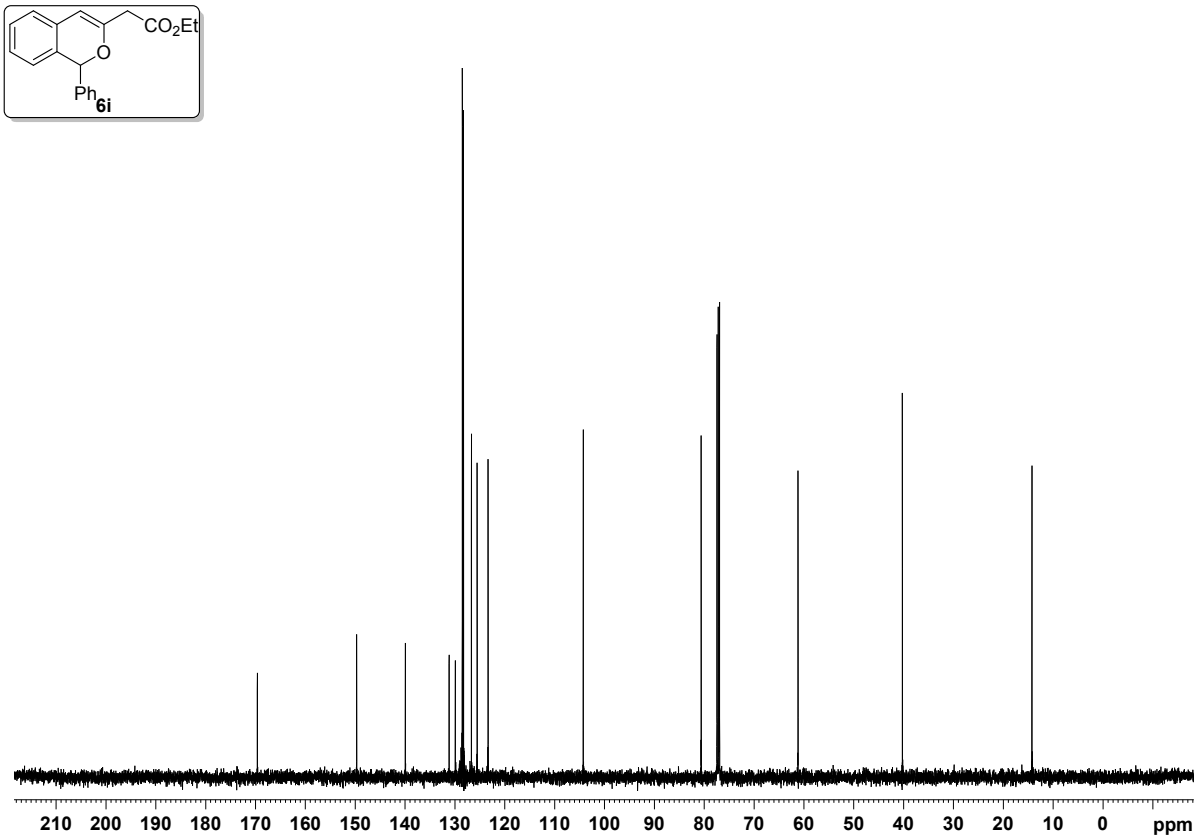

CO2Et
O
OEt
O
6f

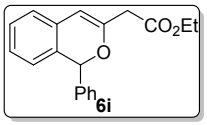

INV4GPLPLRNDSW INV4GPLPLRNDSW
ppm
0
20
40
60
80
100
120
140
160
180
200
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

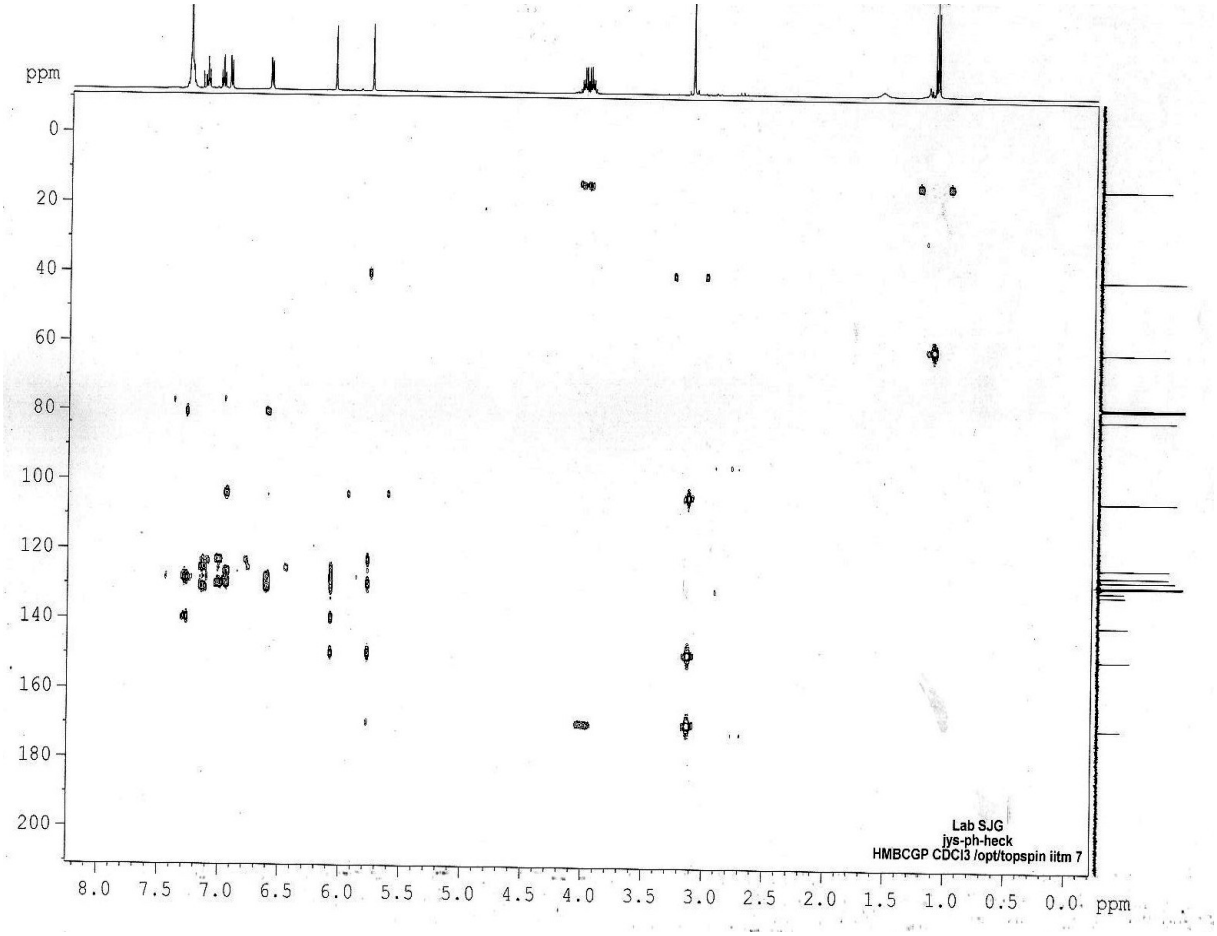

sjg-ygs-cyclohexyl-heck
iitm_PROTON-5to15 CDCl3 /opt/topspin nmr 2
CO2Et
O
OMe
O
6g
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.139
1.067
1.096
1.005
1.000
1.047
2.139
3.063
2.077
2.141
2.698
7.246
1.354


sg-srb-cl-al-ch-ketene-heck
CARBONSHORT_iitm_bbo
CO2Et
O
OMe
O
6g
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

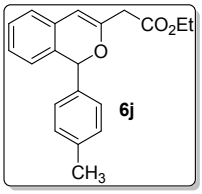

CO2Et
O
Ph
6i
10
9
8
7
6
5
4
3
2
1
0
ppm
5.18
1.10
1.08
1.10
1.12
1.03
1.00
2.26
2.27
3.40

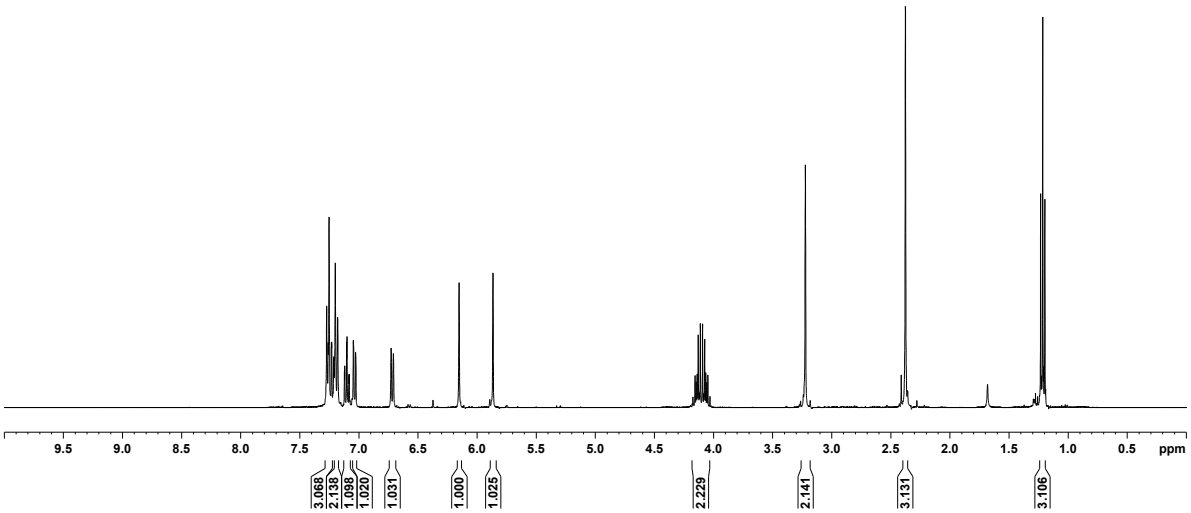

CO2Et
O
Ph
6i
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


CO2Et
O
Ph
6i

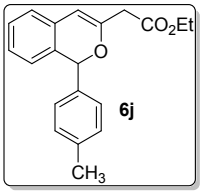

ppm
0
20
40
60
80
100
120
140
160
180
200
Lab SJG
jys-ph-heck
HMBCGP CDCl3 /opt/topspin iitm 7
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm

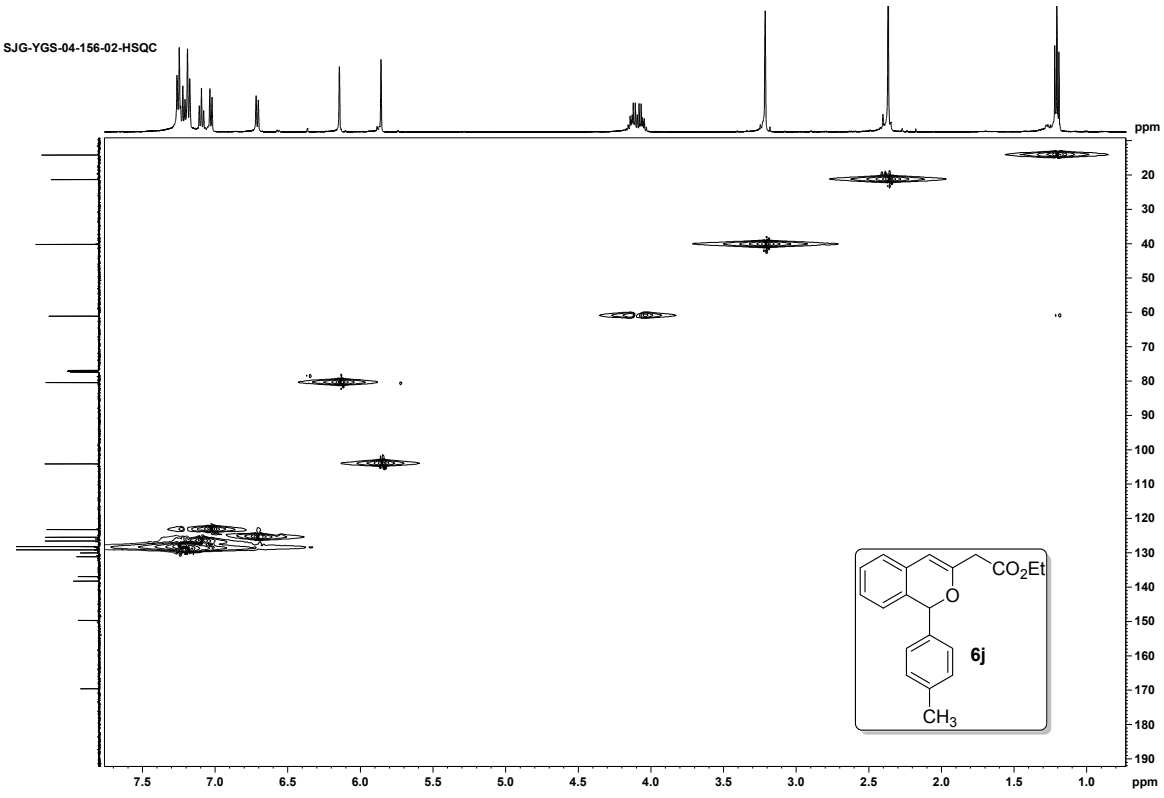
SJG-YGS-04-150-01-1H

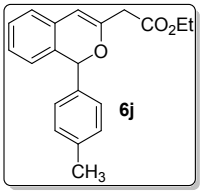


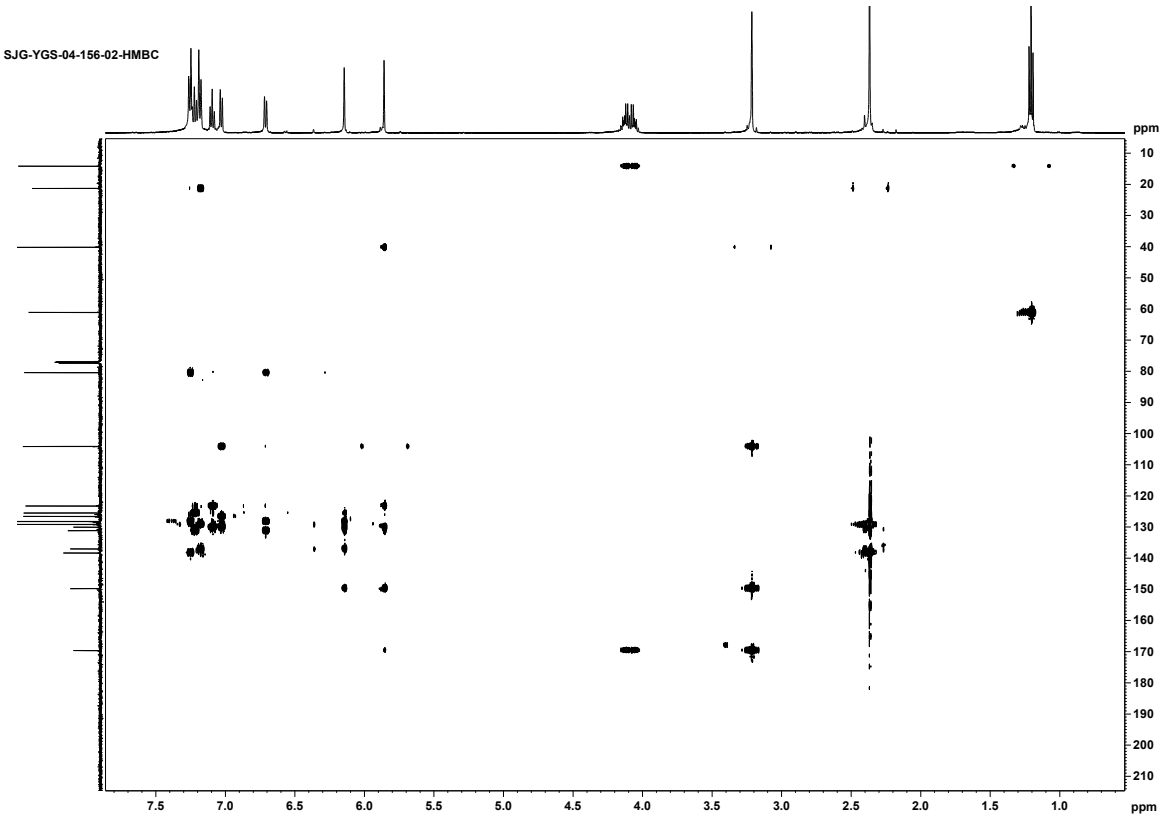


SJG-YGS-04-150-01-13C

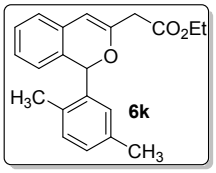



SJG-YGS-04-156-02-HSQC
ppm
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
10 20 30 40 50 60 70 80 90 100 110 120 130 140 150 160 170 180 190
CO2Et
O
6j
CH3

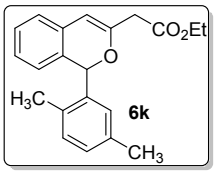

CO2Et
O
6j
CH3

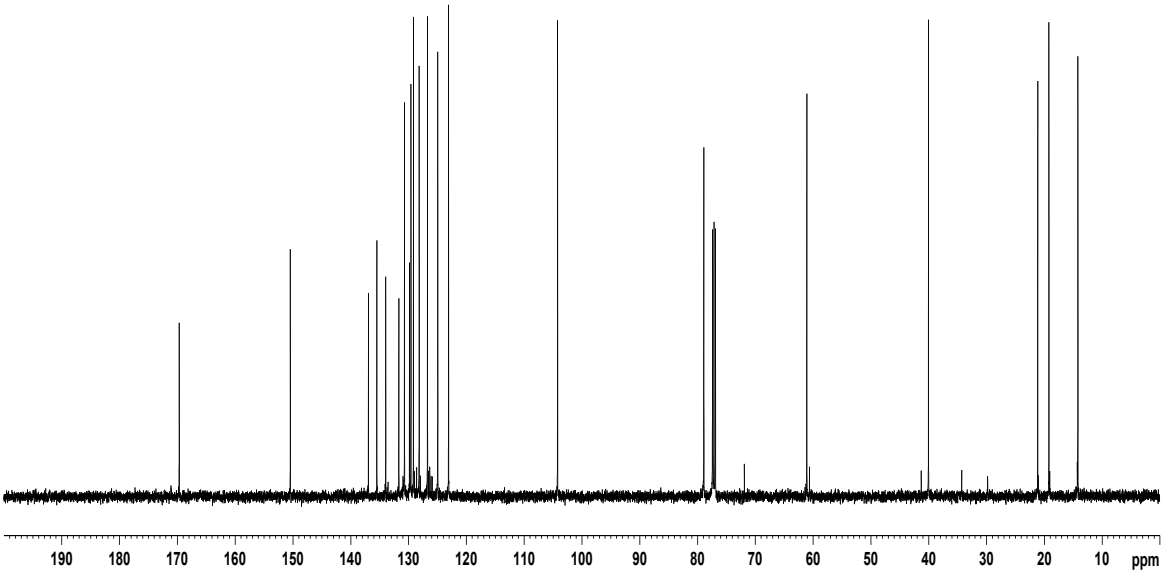

SJG-YGS-04-156-02-HMBC
ppm
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
10 20 30 40 50 60 70 80 90 100 110 120 130 140 150 160 170 180 190 200 210

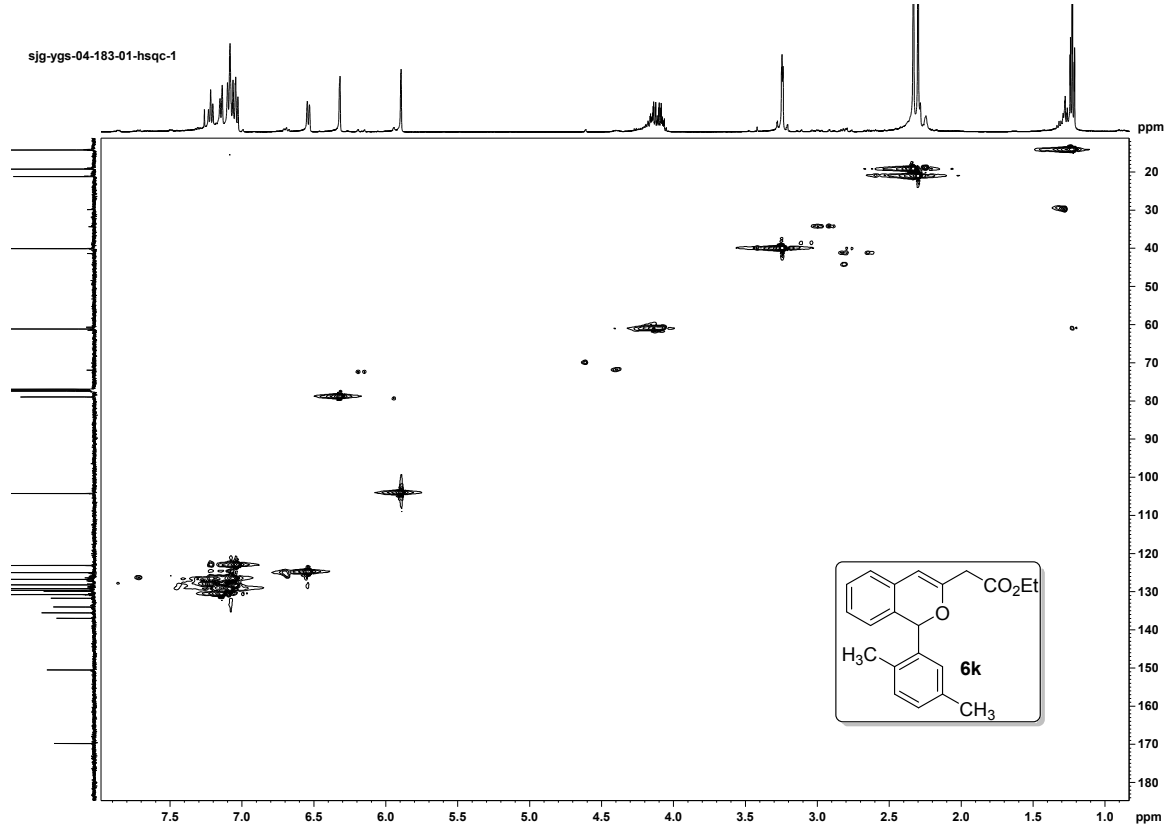
SJG-YGS-04-183-01-1H

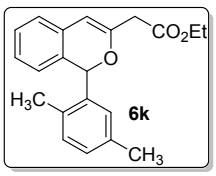


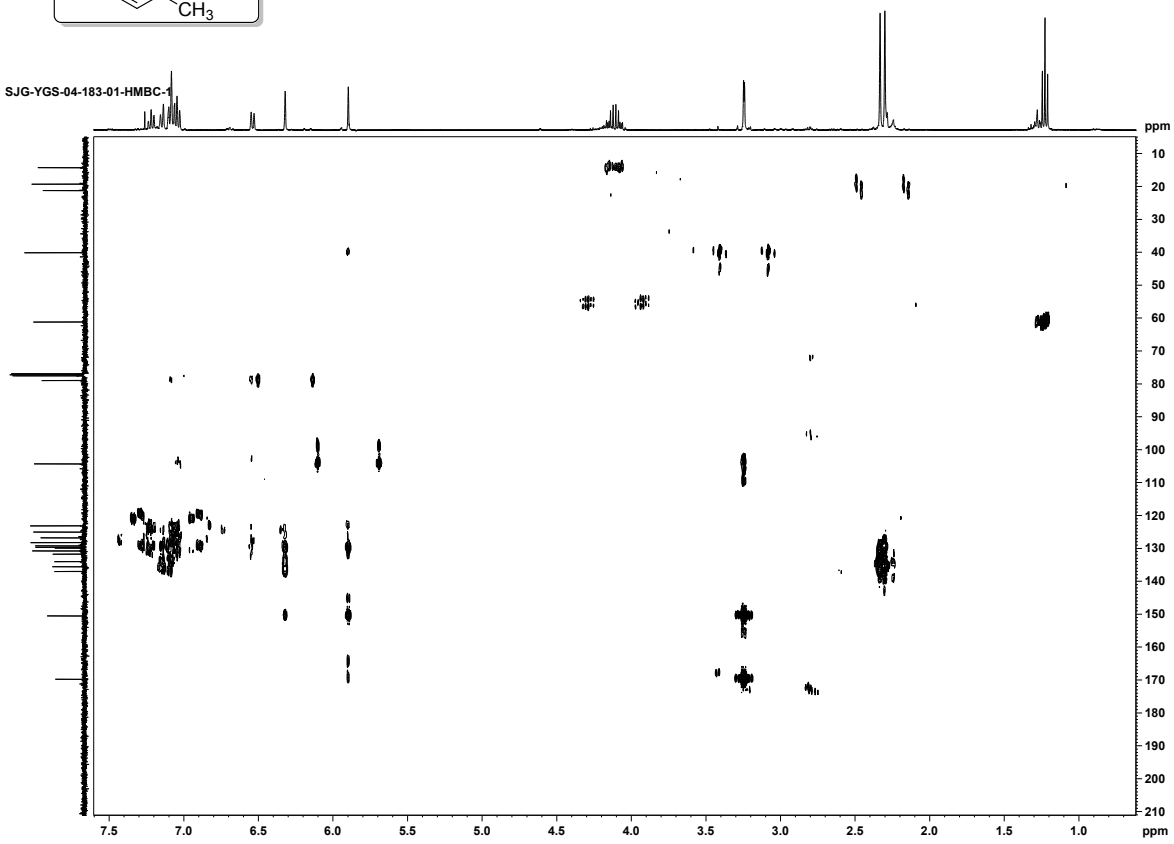
SJG-YGS-04-183-01-13C



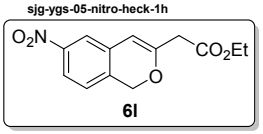



sjg-ygs-04-183-01-hsqc-1
ppm
CO2Et
O
H3C
6k
CH3

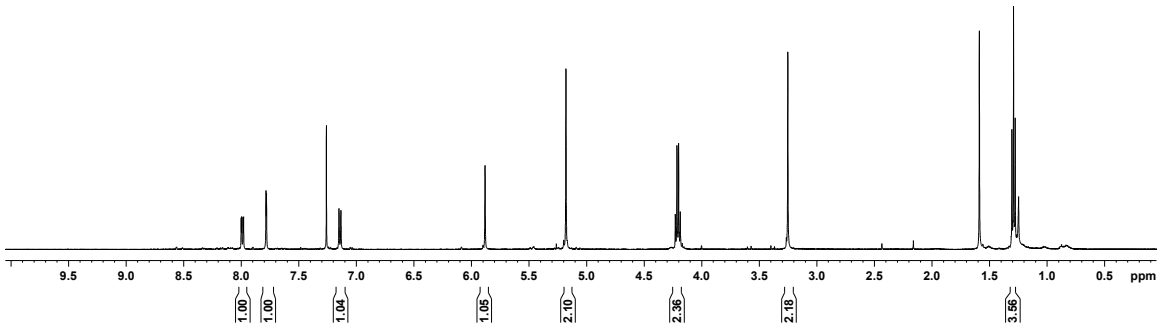

CO2Et
O
H3C
6k
CH3


SJG-YGS-04-183-01-HMBC-1
ppm

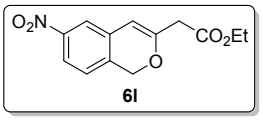
sjg-ygs-05-nitro-heck-1h



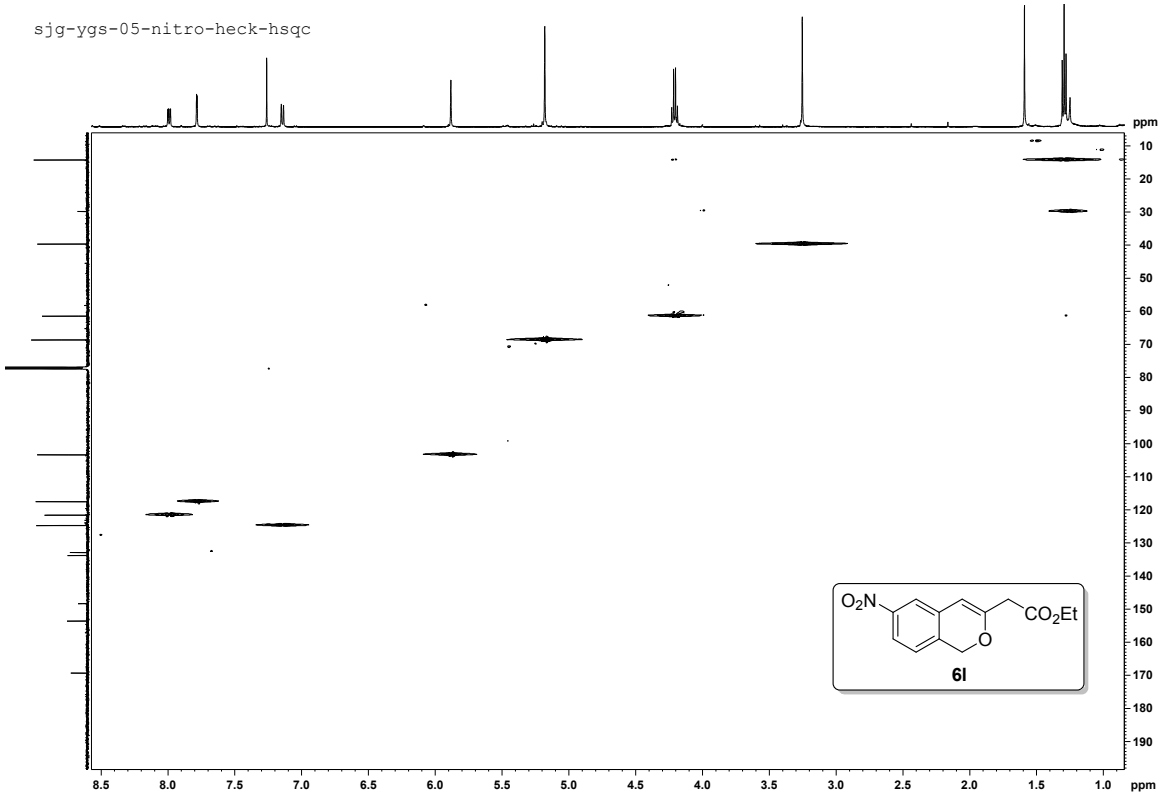
6l

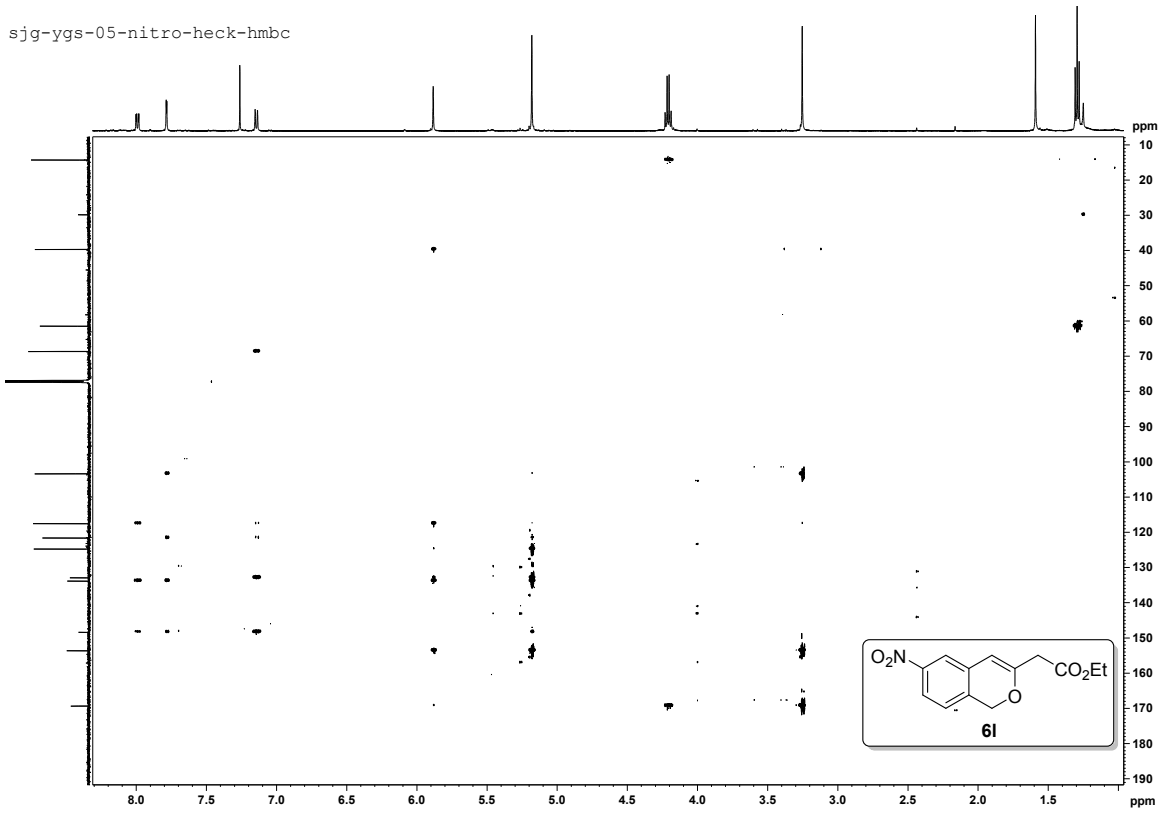


sjg-ygs-05-nitro-heck-13c

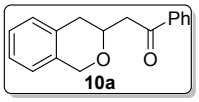


6l

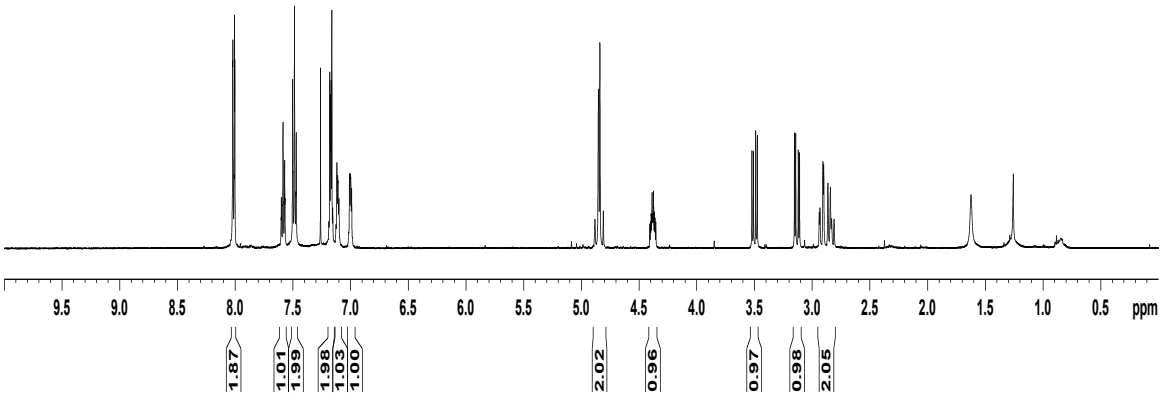

sjg-ygs-05-nitro-heck-hsqc
ppm
O_2N
CO_2Et
O
6l


sjg-ygs-05-nitro-heck-hmbc
ppm
O_2N
CO_2Et
O
6l

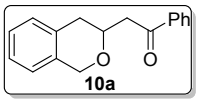
sjg-ygs-04-188-01-1h



10a

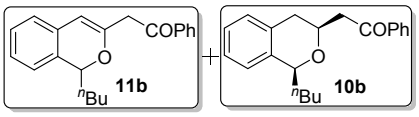


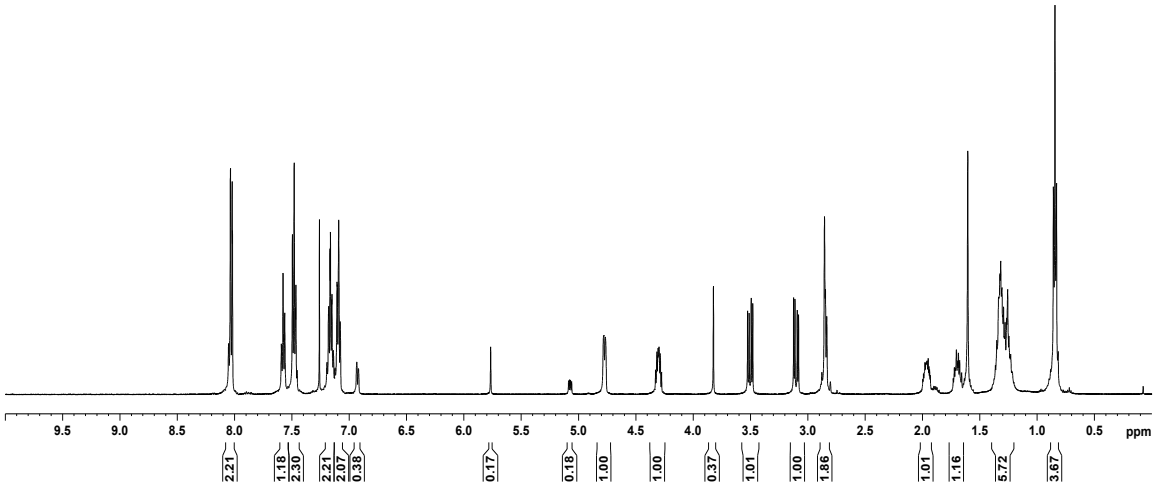
sjg-ygs-04-188-01-13c



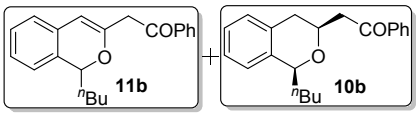
10a

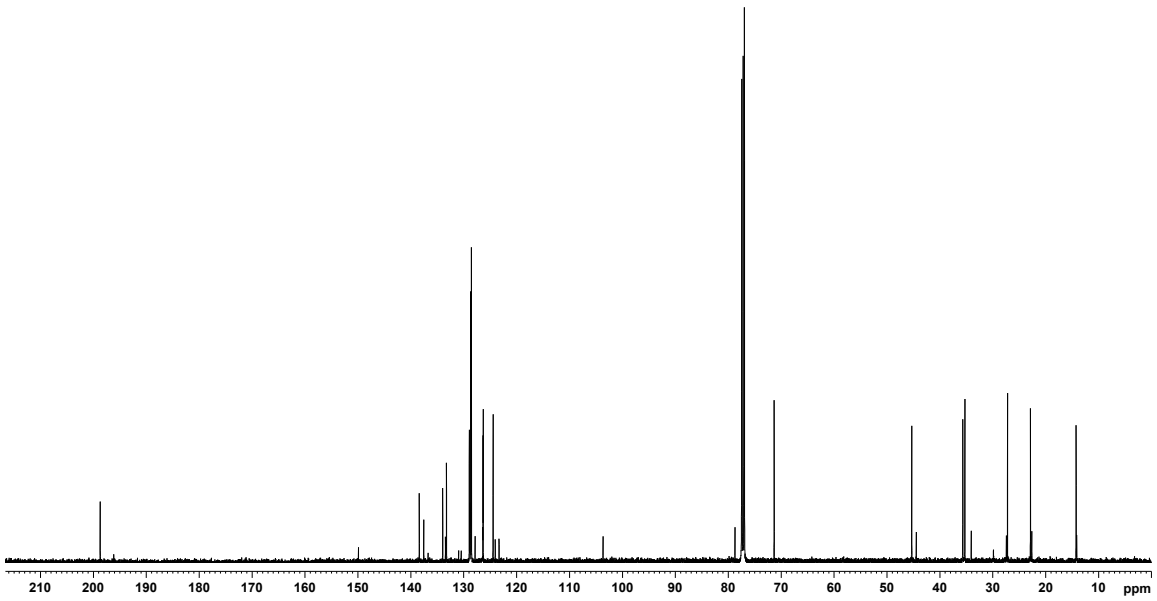
sjg-ygs-05-07-01-1h

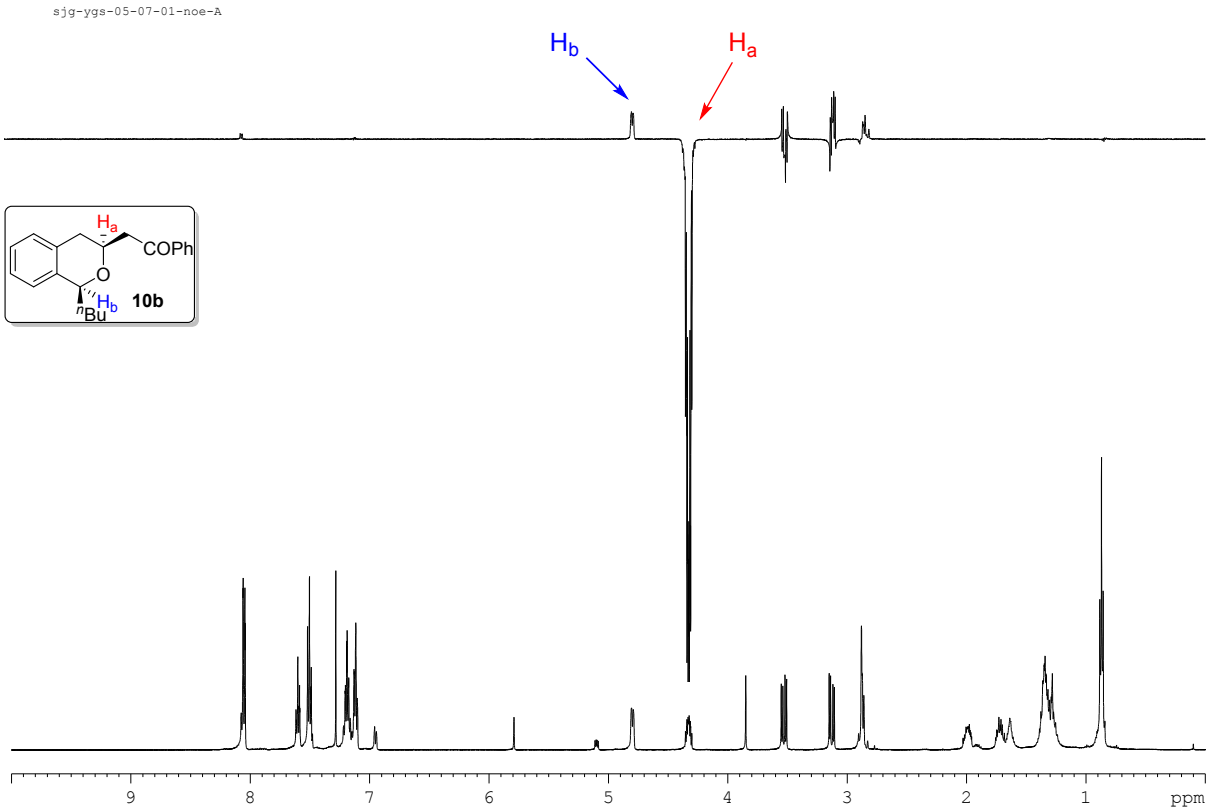




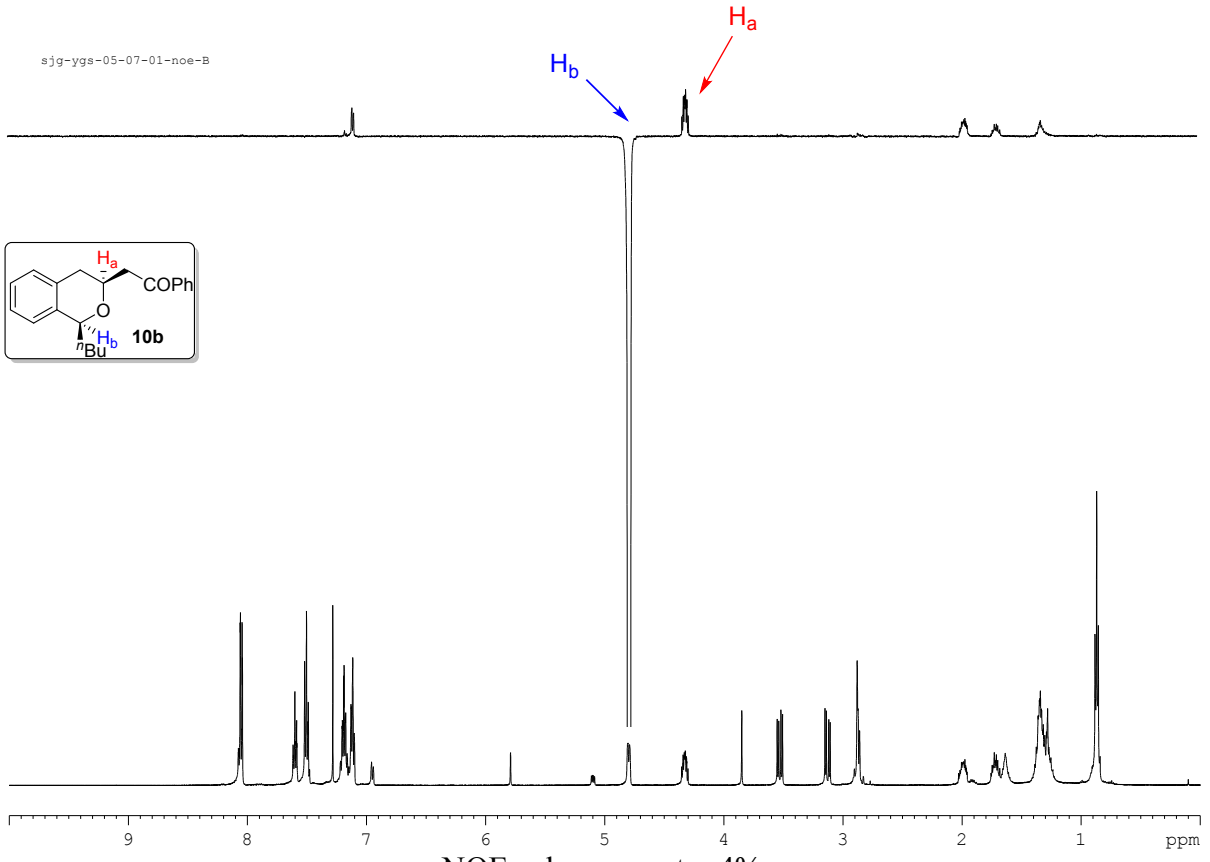
sjg-ygs-05-07-01-13c





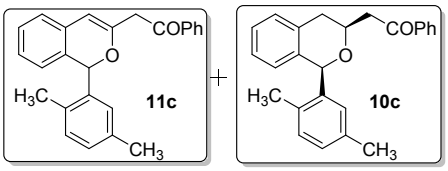


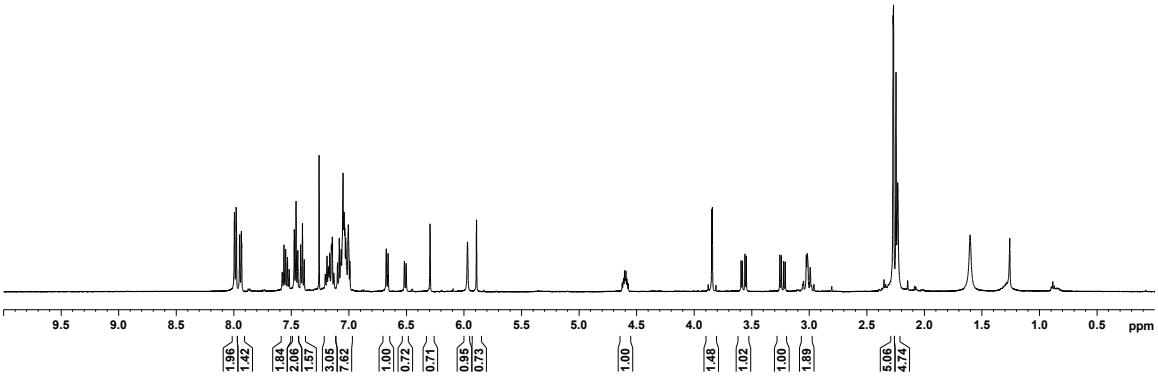
NOE enhancement = 4%



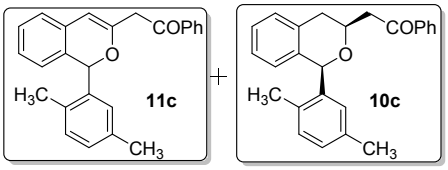
NOE enhancement = 4%

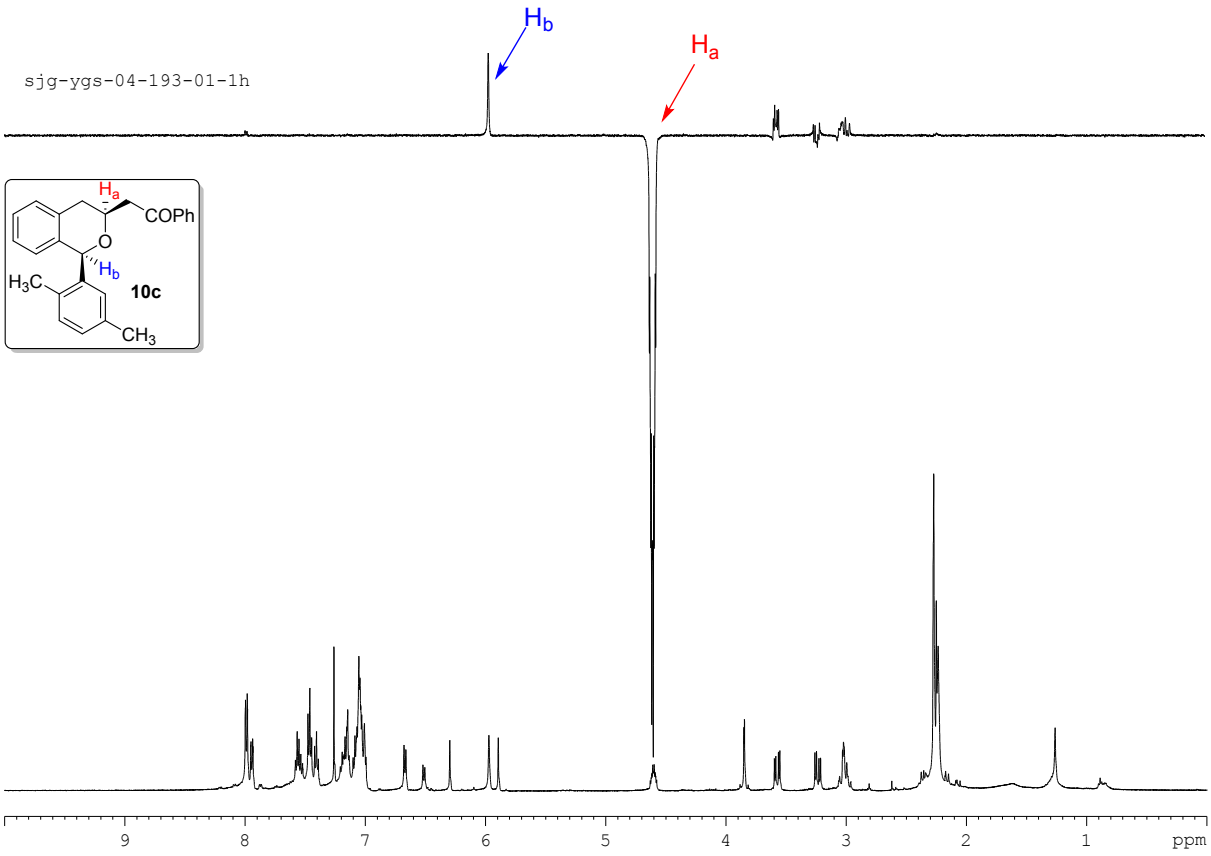
sjg-ygs-04-193-01-1h



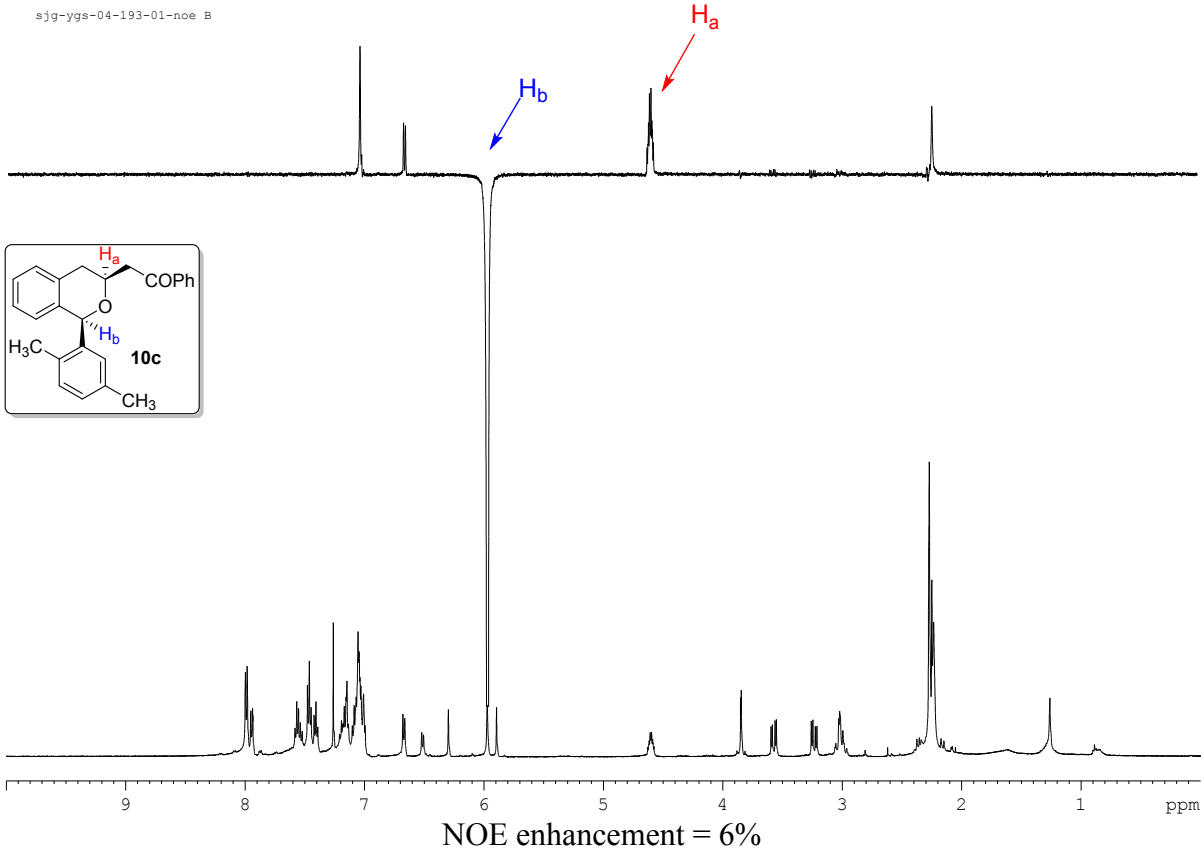


sjg-ygs-04-193-01-13c



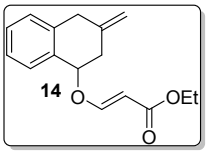


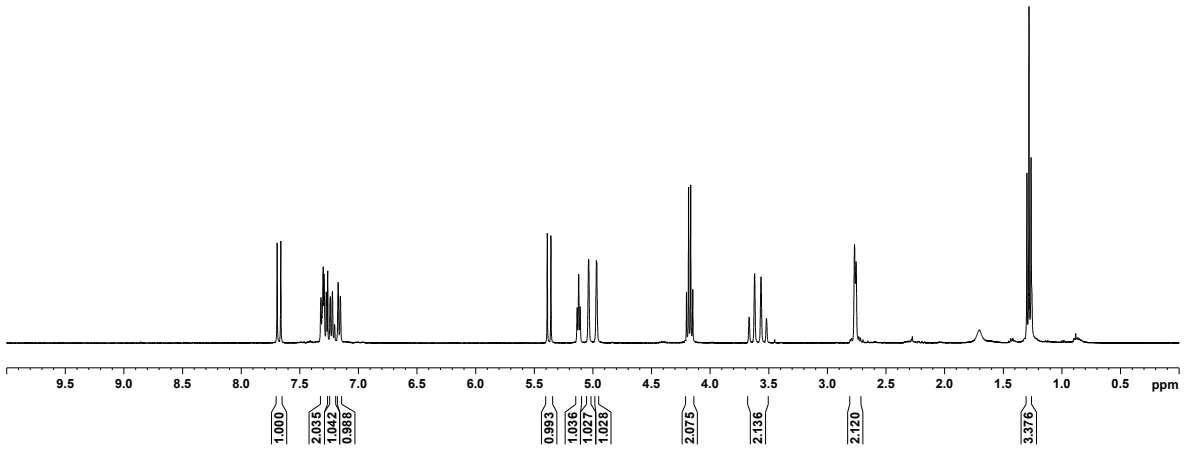
NOE enhancement = 6%

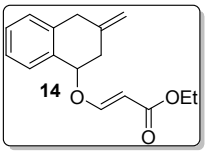


NOE enhancement = 6%

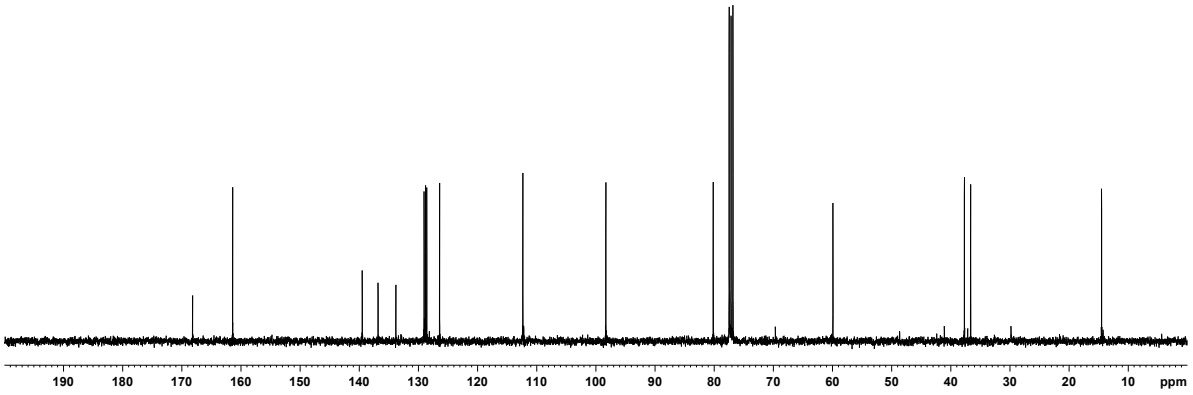
sjg-ygs-02-38-01
iitm_PROTON-5to15



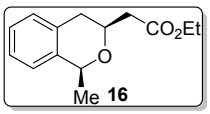




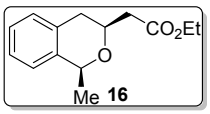
lab sjg
ygs-02-38-01
iitm_carbonshort CDCl3 /opt/topspin nmr 2

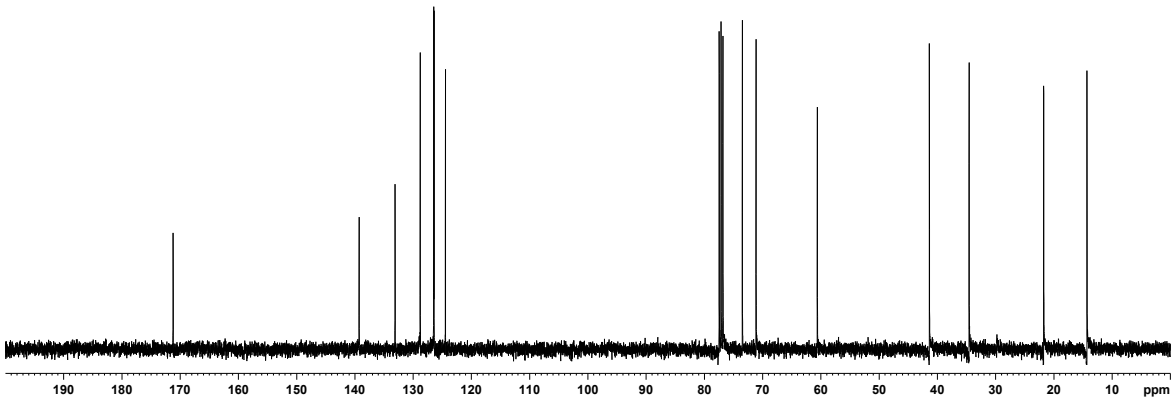


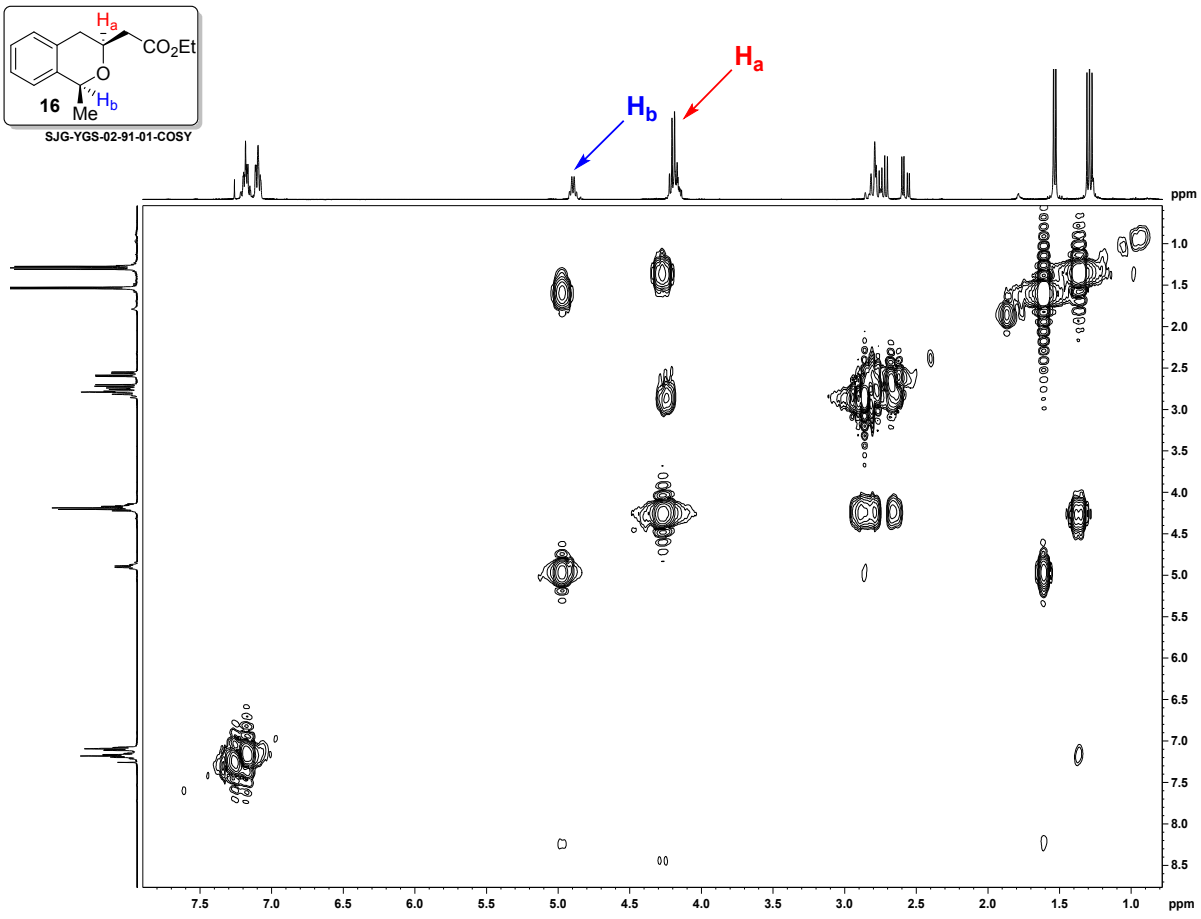
SJG-YGS-02-91-01-H1

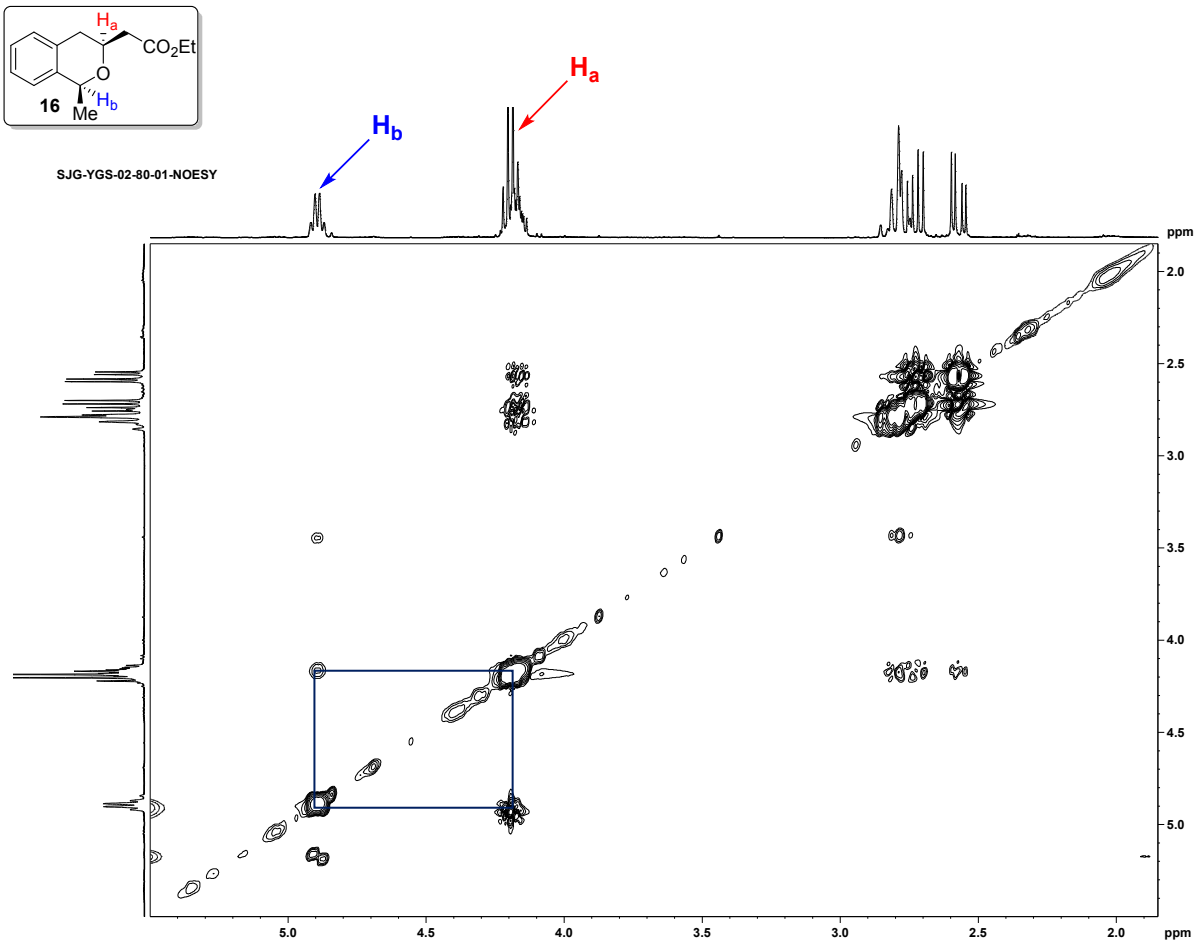


SJG-YGS-02-91-01-C13

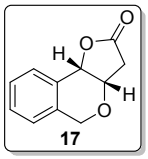


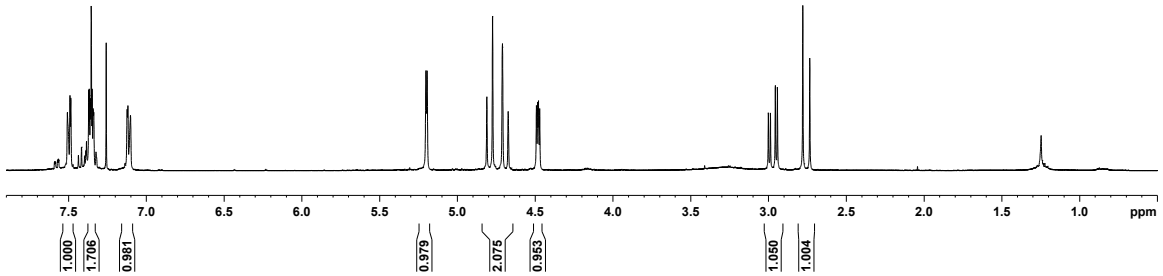



Ha
CO2Et
O
Hb
16
Me
SJG-YGS-02-91-01-COSY
Hb
Ha
ppm
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0

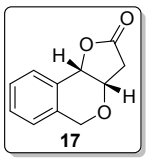

Ha
CO2Et
O
Hb
16
Me
SJG-YGS-02-80-01-NOESY
Hb
Ha
ppm
5.0
4.5
4.0
3.5
3.0
2.5
2.0

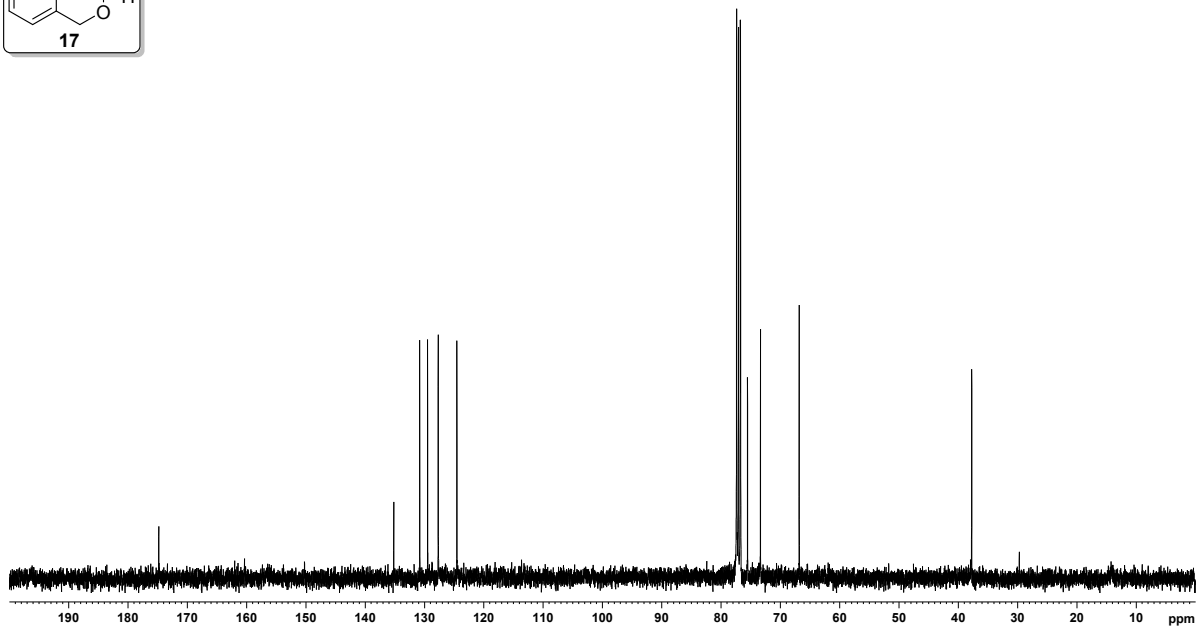
YGS-02-74-03 H1



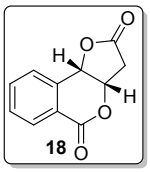


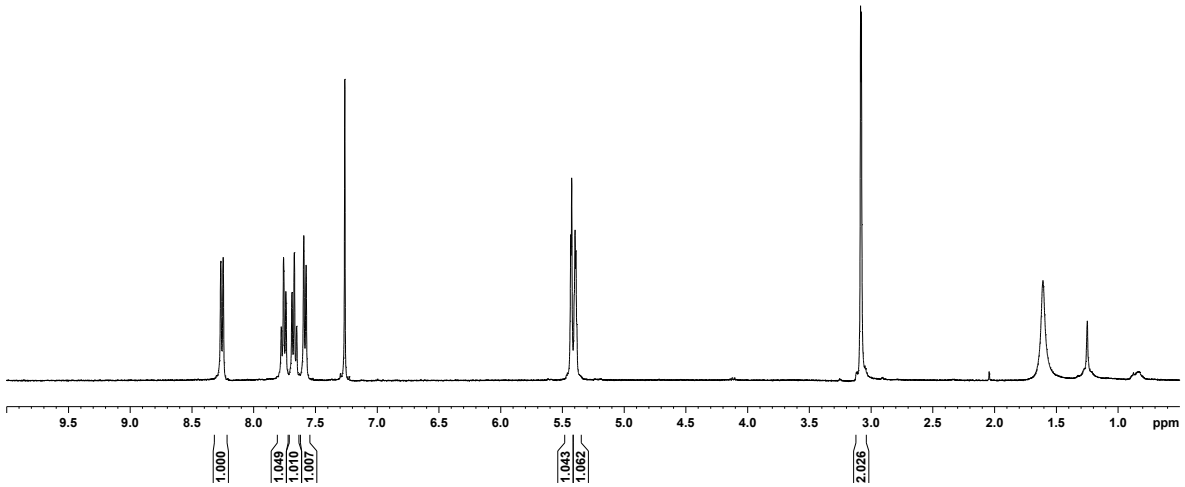
SJG-YGS-02-LACTONE-C13





SJG-YGS-02-86-02-1H





SJG-YGS-02-86-02-C13