Electronic Supplementary Material (ESI) for RSC Advances

Electronic Supplementary Information

Cu-catalyzed coupling-cyclization in PEG400 under ultrasound: A highly selective and greener approach towards isocoumarins

R. Gangadhara Chary,[a,b] G. Rajeswar Reddy,[a] Y. S. S. Ganesh,[a] K. Vara Prasad,[a] S. K. Phani Chandra,[a] Soumita Mukherjee,[c] Manojit Pal*[,c]

[a]*Custom Pharmaceutical Services, Dr. Reddy's Laboratories Limited, Bollaram Road Miyapur, Hyderabad 500 049, India*

[b]*Chemistry Division, Institute of Science and Technology, JNT University, Kukatpally, Hyderabad 500072, Andhra Pradesh, India*

[c]*Organic and Medicinal Chemistry Department, Dr. Reddy's Institute of Life Sciences, University of Hyderabad Campus, Gachibowli, Hyderabad 500046, India,*

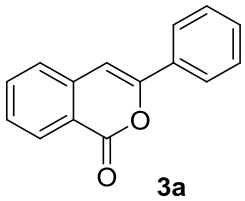
E-mail: manojitpal@rediffmail.com

General methods: Unless stated otherwise, solvents and chemicals were obtained from commercial sources and were used without further purification. Reactions were monitored by thin layer chromatography (TLC) on silica gel plates (60 F254), visualizing with ultraviolet light or iodine spray. Flash chromatography was performed on silica gel (230-400 mesh) using hexane and ethyl acetate. ^{1}H NMR and ^{13}C NMR spectra were determined in DMSO-d_6 solution by using 400 or 100 MHz spectrometers, respectively. Proton chemical shifts (δ) are relative to tetramethylsilane (TMS, δ = 0.00) as internal standard and expressed in ppm. Spin multiplicities are given as s (singlet), d (doublet), t (triplet) and m (multiplet) as well as b (broad). Coupling constants (*J*) are given in hertz. Infrared spectra were recorded on a FT-IR spectrometer. Melting points were determined using melting point B-540 apparatus and are uncorrected. HRMS was determined using waters LCT premier XETOF ARE-047 apparatus.

General procedure for the preparation of 3:

A mixture of 2-iodobenzoic acid (1.0 mmol), terminal alkyne (1.0 mmol), K_2CO_3 (2.0 mmol), and CuI (20 mol%) in polyethylene glycol (5.0 mL) was sonicated at room temperature for 2-3 h under nitrogen. The progress of the reaction was monitored by TLC. After completion of the reaction, the mixture was diluted with EtOAc (10 mL) and filtered through Celite. The filtrate was collected and washed with water (20 mL). The EtOAc layer was collected and concentrated. The residue was purified by column chromatography using EtOAc-petroleum ether.

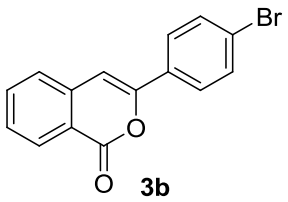
3-Phenyl-1*H*-isochromen-1-one (3a)



3a

Light yellow solid; Yield: 75%; mp: 85.3 - 87.6 °C; IR (KBr): 1776, 1658, 1601 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.97 (d, *J*= 7.3 Hz, 1H), 7.87-7.84 (m, 2H), 7.77-7.79 (m, 2H), 7.57-7.30 (m, 4H), 6.43 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.0, 144.5, 140.5, 134.4, 133.0, 130.0 (2C), 129.7, 128.7 (2C), 128.3, 125.5, 123.3, 119.7, 107.0; HRMS: *m/z* [M + H] calcd for $C_{15}H_{11}O_2$: 223.0759; found: 223.0762.

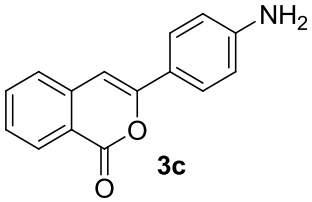
3-(4-Bromophenyl)-1*H*-isochromen-1-one (3b)



3b

White solid; Yield: 80%; mp: 168-172°C; IR (KBr): 1797, 1654 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.95 (d, *J*=7.8 Hz, 1H), 7.79-7.70 (m, 4H), 7.60-7.52 (m, 3H), 6.36 (s, 1H,); $^{13}CNMR$ (100MHz, $CDCl_3$): δ 166.7, 144.9, 140.2, 134.5, 131.9, 131.8 (2C), 131.4 (2C), 129.9, 125.6, 123.3, 122.4, 119.8, 105.6; HRMS: *m/z* [M + H] calcd for $C_{15}H_{10}BrO_2$: 300.9864; found: 300.9862.

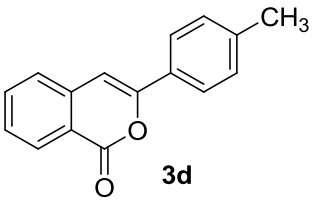
3-(4-Aminophenyl)-1*H*-isochromen-1-one (3c)



3c

Yellow solid; Yield: 65%; mp: 148-151°C; IR (KBr): 3430, 3350, 1739, 1634, cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92 (d, *J*=7.8 Hz, 1H), 7.73-7.66 (m, 4H), 7.51-7.47 (m, 1H), 6.71 (d, *J*=8.8 Hz, 2H), 6.35 (s, 1H), 3.95 (bs, 2H); $^{13}CNMR$ (100MHz, $CDCl_3$): δ 170.6, 151.0, 146.9, 134.2, 131.7, 130.7, 130.1, 128.8, 128.4, 127.8, 125.4, 119.2, 115.0, 113.7, 107.7; HRMS: *m/z* [M + H] calcd for $C_{15}H_{12}NO_2$: 238.0868; found: 238.0874.

3-(*p*-Tolyl)-1*H*-isochromen-1-one (3d)

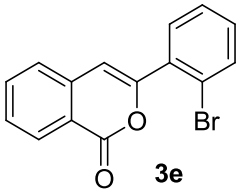


3d

Light yellow solid; Yield: 80%); mp: 150-152.7°C; IR (KBr): 1775, 1659, 1600 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.94 (d, *J*=7.8 Hz, 1H), 7.77-7.69 (m, 4H), 7.56-7.51 (m, 1H), 7.24 (m, 2H), 6.41 (s, 1H), 2.39 (s, 3H). $^{13}CNMR$ (400 MHz, $CDCl_3$): δ 167.1, 143.9, 140.6,

138.6, 134.3, 130.2, 130.0 (2C), 129.4 (3C), 125.4, 123.2, 119.6, 107.1, 21.3; HRMS: *m/z* [M + H] calcd for $C_{16}H_{13}O_2$: 237.0916; found: 237.0918.

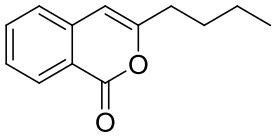
3-(2-Bromophenyl)-1*H*-isochromen-1-one (3e)



3e

White solid; Yield: 80%; mp: 150-154°C; IR (KBr): 1789, 1655, 1604 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 8.30 (dd, *J* = 6.9, 1.5 Hz, 1H), 7.96 (d, *J* =7.6 Hz, 1H), 7.87 (d, *J*=7.6 Hz, 1H), 7.64-7.57 (m, 3H), 7.39 (t, *J* = 7.3 Hz, 1H), 7.19-7.14 (m, 1H), 6.89 (s, 1H); ^{13}CNMR (100 MHz, $CDCl_3$): δ 166.8, 145.7, 140.4, 134.6, 132.9, 132.5, 131.8, 130.2, 129.4, 127.7, 125.5, 124.5, 123.4, 120.2, 105.0; HRMS: *m/z* [M + H] calcd for $C_{15}H_{10}BrO_2$: 300.9864; found: 300.9855.

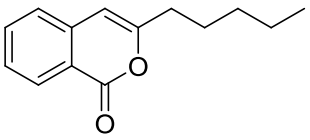
3-Butyl-1*H*-isochromen-1-one (3f)



3f

White solid; Yield: 65%; mp: 40-43.3ºC; IR (KBr): 1728, 1657, 1602 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 8.25 (d, *J*=8.3Hz, 1H), 7.67 (t, *J* = 7.3Hz, 1H), 7.45 (t, *J*=7.3Hz, 1H), 7.35 (d, *J* = 7.8 Hz, 1H), 6.25 (s, 1H), 2.53 (t, *J* = 7.8 Hz, 2H), 1.74-1.66 (m, 2H), 1.44-1.38 (m, 2H), 0.95 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (100MHz, $CDCl_3$): δ 163.0, 158.2, 137.6, 134.6, 129.4, 127.4, 124.9, 120.0, 102.8, 33.1, 28.9, 22.0, 13.7; HRMS: *m/z* [M + H] calcd for $C_{13}H_{15}O_2$: 203.1072; found: 203.1065.

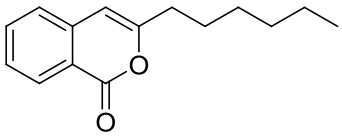
3-Pentyl-1*H*-isochromen-1-one (3g)



3g

Color less liquid; Yield: 60%; IR (Neat): 1723, 1657, 1606 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.25 (d, *J*=7.8 Hz, 1H), 7.69-7.65(m, 1H), 7.47-7.43 (m, 1H), 7.35 (d, *J*=7.8 Hz, 1H), 6.25 (s, 1H), 2.52 (t, *J*=7.5 Hz, 2H) , 1.73-1.69 (m, 2H), 1.38-1.34 (m, 4H), 0.91 (t, *J*=7.3 Hz, 3H); ^{13}CNMR (100MHz, $CDCl_3$): δ 167.2, 145.5, 139.5, 134.1, 129.2, 125.2, 124.4, 119.5, 109.7, 31.4, 28.9, 25.7, 22.4, 13.9; HRMS: *m/z* [M + H] calcd for $C_{14}H_{17}O_2$: 217.1229; found: 217.1220.

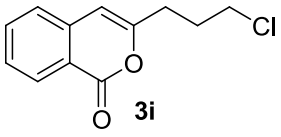
3-Hexyl-1*H*-isochromen-1-one (3h)



3h

Colorless liquid; Yield: 60%; IR (Neat): 1727, 1656, 1606 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.25 (d, *J* = 8.3 Hz, 1H), 7.69-7.65 (m, 1H), 7.47-7.43 (m, 1H), 7.35 (d, *J*=7.8 Hz, 1H), 6.25 (s, 1H), 2.52 (m, *J*=7.8 Hz, 2H), 1.74-1.67 (m, 2H,), 1.45-1.21 (m, 6H), 0.89 (t, *J*=7.3 Hz, 3H); ^{13}C NMR (100MHz, $CDCl_3$): δ 163.0, 158.2, 137.5, 134.6, 129.4, 127.4, 124.9, 120.0, 102.7, 33.4, 31.4, 28.6, 26.8, 22.4, 13.9; HRMS: *m/z* [M + H] calcd for $C_{15}H_{19}O_2$: 231.1385; found: 231.1373.

3-(3-Chloropropyl)-1*H*-isochromen-1-one (3i)

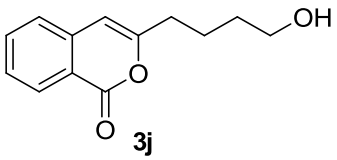


3i

Gummy solid; Yield: 60%; IR (neat): 1725, 1659, 1607 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.26 (d, *J*=7.9, 1H), 7.72-7.67 (m, 1H), 7.50-7.46 (m, 1H), 7.39-7.36 (m, 1H), 6.34 (s, 1H), 3.61 (t, *J*=6.4 Hz, 2H), 2.74 (t, *J*=7.4 Hz, 2H), 2.23-2.16 (m, 2H); ^{13}CNMR (100MHz,

$CDCl_3$): δ 156.0, 137.2, 134.8, 129.5, 127.9, 125.1, 120.2, 103.9, 43.7, 30.9, 30.6, 29.4; HRMS: *m/z* [M + H] calcd for $C_{12}H_{12}ClO_2$: 223.0526; found: 223.0526.

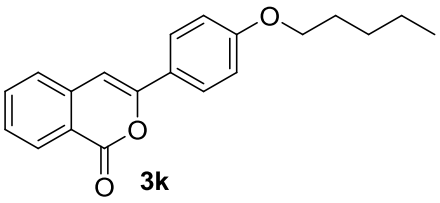
3-(4-Hydroxybutyl)-1*H*-isochromen-1-one (3j)



3j

Gummy solid; Yield: 60%; IR (neat): 3454, 1721, 1656, 1607 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 8.25 (d, *J*=7.8 Hz, 1H), 7.67-7.65 (m, 1H), 7.46 (t, *J* = 7.3 Hz, 1H), 7.35 (d, *J* = 7.8 Hz, 1H), 6.25 (s, 1H), 3.70 (t, *J* = 6.3 Hz, 2H), 2.58 (t, *J* = 7.3 Hz, 2H), 1.86-1.78 (m, 2H), 1.69-1.62 (m, 3H). ^{13}CNMR (100MHz, $CDCl_3$): δ 163.0, 157.7, 137.4, 134.7, 129.4, 127.6, 125.0, 120.0, 103.1, 62.4, 33.1, 31.8, 23.1; HRMS: *m/z* (M + H) calcd for $C_{13}H_{15}O_3$: 219.1021; found 219.1016.

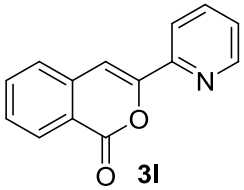
3-(4-(Pentyloxy)phenyl)-1*H*-isochromen-1-one (3k)



3k

Light yellow solid; Yield: 80%; mp: 79-83 °C; IR (KBr): 1784, 1769, 1729, 1599 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.93 (d, *J* = 7.3 Hz, 1H), 7.80 (dd, *J*=6.9, 2.0 Hz, 2H), 7.68-7.76 (m, 2H), 7.54-7.51 (m, 1H), 6.96-6.92 (m, 2H), 6.39 (s, 1H), 4.00 (t, *J* = 7.4 Hz, 2H), 1.81 (t, *J* = 6.8 Hz, 2H), 1.56-1.38 (m, 4H), 0.95 (t, *J*=7.4 Hz, 3H); ^{13}CNMR (100MHz, $CDCl_3$): δ 167.2, 159.3, 142.9, 140.7, 134.3, 131.6, 131.6, 129.1, 125.5, 125.4, 123.0, 119.4, 114.7 (2C), 107.0, 68.0, 28.8, 28.1, 22.4, 13.9; HRMS: *m/z* [M + H] calcd for $C_{20}H_{21}O_3$: 309.1491; found: 309.1488.

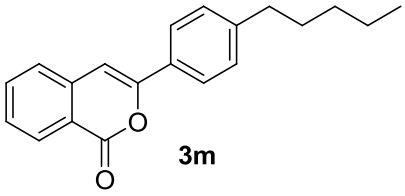
3-(Pyridin-2-yl)-1*H*-isochromen-1-one (3l)



3l

Brown solid; Yield: 75%; mp: 123 -125.6 °C; IR (KBr): 1792, 1787, 1666, 1608 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.63-8.64 (m, 1H), 8.31 (d, J = 7.8 Hz, 1H), 7.96-7.98 (m, 1H), 7.84-7.75 (m, 3H), 7.63-7.59 (m, 1H), 7.26-7.19 (m, 1H), 6.71 (s, 1H); $^{13}CNMR$ (100MHz, $CDCl_3$): δ 166.5, 152.5, 149.5, 146.7, 140.2, 136.5, 134.7, 130.5, 125.6, 125.4, 123.5, 122.3, 120.4, 107.6; HRMS: *m/z* (M + H) calcd for $C_{14}H_{10}NO_2$: 224.0712; found: 224.0718.

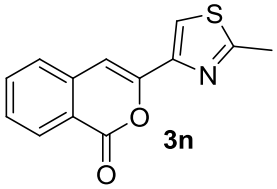
3-(4-pentylphenyl)-1*H*-isochromen-1-one (3m)



3m

White solid; Yield: 80%; mp: 62.1-63.6 °C; IR (KBr): 1780, 1663, 1603 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.94 (d, J=7.8, 1H), 7.78-7.70 (m, 4H), 7.56-7.52 (m, 1H), 7.23 (d, J=8.3 Hz, 2H), 6.42 (s, 1H), 2.61-2.65 (m, 2H), 1.68-1.60 (m, 2H), 1.38-1.30 (m, 4H), 0.89 (m, 3H); $^{13}CNMR$ (100MHz, $CDCl_3$): δ 167.1, 143.9, 143.6, 140.6, 134.3, 130.4, 130.0 (2C), 129.4, 128.8 (2C), 125.4, 123.2, 119.6, 107.2, 35.7, 31.4, 30.9, 22.5, 13.9; HRMS: *m/z* [M + H] calcd for $C_{20}H_{21}O_2$: 293.1542; found: 293.1544.

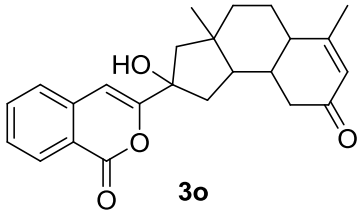
3-(2-Methylthiazol-4-yl)-1*H*-isochromen-1-one (3n)



3n

Brown solid; Yield: 60%; mp: 122-125 °C; IR (KBr): 1793, 1661, 1604 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.96-7.92 (m, 2H), 7.75-7.71 (m, 2H), 7.58-7.50 (m, 1H), 6.75 (s, 1H), 2.75 (s, 3H); $^{13}CNMR$ (100MHz, $CDCl_3$): δ 166.6, 164.9, 148.1, 145.3, 140.0, 134.6, 129.9, 125.6, 123.6, 120.1, 119.8, 100.9, 18.9. HRMS: *m/z* [M + H] calcd for $C_{13}H_{10}NO_2S$: 244.0432; found: 244.0428.

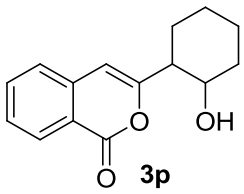
3-(2-Hydroxy-3a,6-dimethyl-8-oxo-2,3,3a,4,5,5a,8,9,9a,9b-decahydro-1*H*-cyclopenta[*a*]naphthalen-2-yl)-1*H*-isochromen-1-one (3o)



3o

White solid; Yield: 80%; mp: 110-115 °C; IR (KBr): 3435, 1730, 1643, 1604 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.27 (d, J = 8.3 Hz, 1H), 7.76-7.71 (m, 1H), 7.50-7.54 (m, 1H), 7.45 (d, J=7.9Hz, 1H), 6.45 (s, 1H), 2.86 (bs, 1H), 2.72 (dd, J = 17.1, 4.4 Hz, 1H), 2.47-1.57 (m, 3H), 2.17-2.35 (m, 3H), 2.03-2.07 (m, 2H), 1.82-1.93 (m, 3H), 1.60-1.74 (m, 3H), 1.56-1.49 (m, 2H), 1.28-1.24 (m, 3H), 1.14-0.97 (m, 1H); ^{13}CNMR (100MHz, $CDCl_3$): δ 198.8, 161.6, 159.2, 157.4, 136.5, 135.0, 130.3, 129.4, 128.4, 126.0, 119.9, 103.7, 84.4, 49.6, 47.4, 39.6, 37.0, 35.0, 32.6, 27.2, 27.0, 23.8, 13.6, 11.0; HRMS: *m/z* [M + H] calcd for $C_{24}H_{27}O_4$: 379.1909; found: 379.1897.

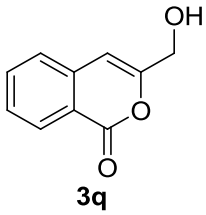
3-(2-Hydroxycyclohexyl)-1*H*-isochromen-1-one (3p)



3p

White solid; Yield: 80%; mp: 94.5-98.5°C; IR (KBr): 3538, 3518, 1788, 1757, 1675, 1608 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.91 (d, J = 7.3 Hz, 1H), 7.64-7.72 (m, 2H), 7.53-7.57 (m, 1H), 5.79 (s, 1H), 2.64 (s, 1H), 1.93-1.70 (m, 8H), 1.68-1.38 (m, 2H); ^{13}CNMR (100 MHz, $CDCl_3$): δ 166.3, 144.2, 139.8, 134.4, 129.9, 125.3, 123.3, 119.8, 115.0, 71.5, 39.5, 38.1, 25.1, 23.0, 22.0; HRMS: *m/z* [M + H] calcd for $C_{15}H_{17}O_3$: 245.1178; found: 245.1173.

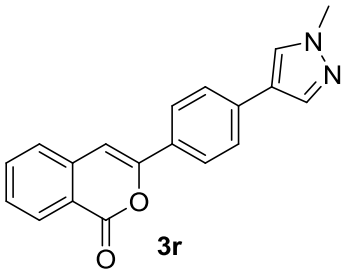
3-(Hydroxymethyl)-1*H*-isochromen-1-one (3q)



3q

White solid; Yield: 85%; mp: 95.6-96.3 °C; IR (KBr): 3279, 3175, 3058, 1781, 1729, 1687, 1609 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92 (d, J=7.3 Hz, 1H), 7.68-7.75 (m, 2H), 7.59-7.55 (m, 1H), 5.83 (t, J=6.8 Hz, 1H), 4.62 (d, J=6.9 Hz, 2H), 1.79 (bs, 1H); ^{13}CNMR (100 MHz, $CDCl_3$): δ 166.5, 146.1, 139.0, 134.5, 130.2, 125.3, 124.4, 120.2, 106.8, 56.9; HRMS: *m/z* [M + H] calcd for $C_{10}H_9O_3$: 177.0552; found: 177.0546.

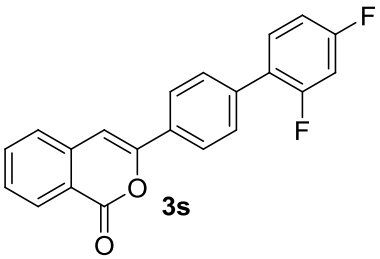
3-(4-(1-Methyl-1*H*-pyrazol-4-yl)phenyl)-1*H*-isochromen-1-one (3r)



3r

Yellow solid; Yield: 80%; mp: 212-218 °C; IR (KBr): 1784, 1652, 1601, 1564 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.96 (d, J=7.8 Hz, 1H), 7.87-7.62 (m, 6H), 7.60-7.49 (m, 3H), 6.43 (s, 1H), 3.96 (s, 3H); ^{13}CNMR (100 MHz, $CDCl_3$): δ 167.1, 144.1, 140.5, 136.8, 134.4, 132.6, 131.0, 130.6 (2C), 129.5, 127.1, 125.5 (3C), 123.2, 122.6, 119.7, 106.8, 39.1; HRMS: *m/z* (M + H) calcd for $C_{19}H_{15}N_2O_2$: 303.1134; found 303.1144.

3-(2',4'-Difluoro-[1,1'-biphenyl]-4-yl)-1*H*-isochromen-1-one (3s)

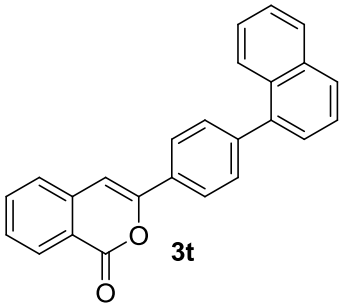


3s

White solid; Yield: 70%; mp: 178-183 °C; IR (KBr): 1785, 1658, 1601 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.97-7.92 (m, 3H), 7.81 (d, J=7.8 Hz, 1H), 7.75 (t, J=7.4 Hz, 1H), 7.58 (t, J=7.8 Hz, 3H), 7.48-7.42 (m, 1H) 7.00-6.91 (m, 2H), 6.47 (s, 1H); ^{13}CNMR (100 MHz, $CDCl_3$): δ 166.9, 144.9, 140.4, 134.8, 134.5, 132.5, 131.9, 131.4, 131.2, 130.2, 130.0, 129.8,

129.1, 125.6, 123.3, 119.8, 111.7, 111.5, 106.4, 105.7, 104.4; HRMS: *m/z* [M + H] calcd for $C_{21}H_{13}F_2O_2$: 335.0884; found: 335.0870.

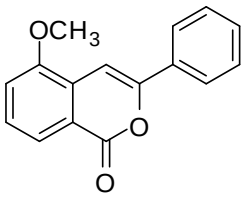
3-(4-(Naphthalen-1-yl) phenyl)-1*H*-isochromen-1-one (3t)



3t

White solid; Yield: 85%; mp: 137-141 °C; IR (KBr): 1761, 1603 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.00-7.82 (m, 7H), 7.80-7.72 (m, 1H), 7.60-7.41 (m, 7H), 6.54 (s, 1H); ^{13}CNMR (100 MHz, $CDCl_3$): δ 167.0, 144.7, 140.8, 139.5, 134.5, 134.5, 133.7, 132.0, 131.9, 131.4, 131.3, 130.4, 130.0, 129.7, 128.3, 127.8, 126.8, 126.1, 125.8, 125.8, 125.6, 125.3, 123.3, 119.8, 106.7; HRMS: *m/z* [M + H] calcd for $C_{25}H_{17}O_2$: 349.1229; found: 349.1223.

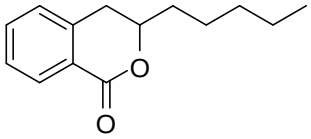
5-Methoxy-3-phenyl-1*H*-isochromen-1-one



A mixture of 2-iodo-3-methoxybenzoic acid (1.0 mmol), phenyl acetylene (1.0 mmol), CuI (0.2mol %) and K_2CO_3 (2.0 mmol) in polyethylene glycol (5.0 mL) was sonicated at room temp for 3 h under nitrogen. The progress of the reaction was monitored by TLC. After completion of the reaction, the mixture was diluted with EtOAc (10 mL) and filtered through Celite. The filtrate was collected and washed with water (20 mL). The EtOAc layer was collected and concentrated. The residue was purified by column chromatography using EtOAc-petroleum ether to give the title compound as off-white solid (yield: 77%); IR ($CHCl_3$): 1724, 1691, 1593 cm^{-1}; ^{1}HNMR (400 MHz, $CDCl_3$): δ7.96-7.98 (m, 1H), 7.61-7.63 (m, 1H), 7.49-7.55 (m, 3H), 7.39 (t, J = 7 Hz, 1H), 6.97 (dd, J_1 =1Hz, J_2 = 7.8 Hz, 1H), 5.59 (s, 1H), 3.92 (s, 3H); ^{13}CNMR(100 MHz, $CDCl_3$): δ 166.6, 158.7, 151.7, 137.5, 134.1, 133.9,

129.3, 128.9 (2C), 127.0(2C), 123.2, 120.0, 113.5, 86.9, 56.8; HRMS: *m/z*[M + NH_4] calcd for $C_{16}H_{16}NO_3$: 270.1130; found: 270.1153.

3-Pentylisochroman-1-one (8)



To a solution of 3-pentyl-1*H*-isochromen-1-one (**3g**) (100 mg, 0.462 mmol) in methanol (10 mL) was added 10%Pd/C (10 mg) and the suspension was stirred for 12 h under a hydrogen atmosphere at ambient pressure. After completion of the reaction, catalyst was filtered off, and the residue was washed with methanol. The combined organic layers were collected, concentrated, and the residue was purified by column chromatography (EtOAc-petroleum ether) to give the desired product (**8**) as pale yellow oil (yield 72%); IR (KBr): 2942, 2853, 1706, 1650, 1454 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 8.08 (d, J = 7.7 Hz, 1H), 7.51 (ddd, J_1 = J_2 = 7.7 Hz, J_3= 1.3 Hz, 1H), 7.37 (dd, J_1 = J_2 = 7.7 Hz, 1H), 7.23 (dd, J_1 = J_2 = 7.7 Hz, 1H), 4.50 (m, 1H), 2.93 (m, 2H), 1.86 (m, 1H), 1.71 (m, 1H), 1.57 (m, 1H), 1.46 (m, 1H), 1.33 (m, 4H), 0.90 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 165.7, 139.3, 133.6, 130.2, 127.6, 127.4, 125.3, 77.4, 34.9, 33.2, 31.6, 24.6, 22.5, 14.0; MS (*m/z*): 218 (M^+, 15), 118 (100).