

A facile approach for the synthesis of novel 1-oxa- and 1-aza-flavonyl-4-methyl-1H-benzo[d][1,3]oxazin-2(4H)-ones by microwave enhanced Suzuki-Miyaura coupling using bidentate chromen-4-one-based Pd(II)-diimine complex as catalyst

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SUPPORTING INFORMATION

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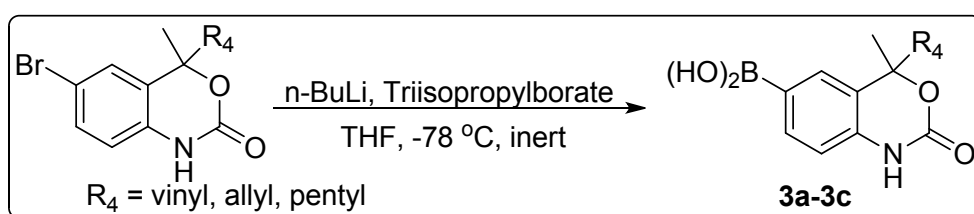
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General Information

Unless otherwise noted, chemicals were purchased from commercial suppliers at the highest purity grade available and were used without further purification. Solvents were distilled by standard methods. Thin layer chromatography was performed on Merck precoated 0.25 mm silica gel plates (60F-254) using UV light as visualizing agent and/or iodine as developing agent. Silica gel (100-200 mesh) was used for column chromatography. Melting points were performed with Ambassador[®] and Digital Melting point apparatus (Nutronics), Popular India. IR spectra were recorded on FT-IR spectrometer and expressed as wave numbers (cm⁻¹). ¹H and ¹³C NMR spectra were recorded on a Brüker Avance 500 & Jeol Resonance ECX 400 spectrometer. Spectra were referenced internally to the residual proton resonance in CDCl₃ (δ 7.26 ppm) or with tetramethylsilane (TMS, δ 0.00 ppm) as the internal standard. Spectra were processed using Bruker Topspin[®] 3.0.b.8. Chemical shifts (δ) were reported as part per million (ppm) in δ scale downfield from TMS. ¹³C NMR spectra were referenced to CDCl₃ (δ 77.23 ppm, the middle peak). Coupling constants are expressed in Hz. The following abbreviations are used to explain the multiplicities: s = singlet, d = doublet, t = triplet, dd = doublet of doublets, m = multiplet, br = broad. High-resolution mass spectra (HRMS) were obtained on a Brüker micrOTOF[™]-Q II mass spectrometer (ESIMS).

General procedure for synthesis of Heteroarylboronic acids (3a-3c).

The synthesis followed the literature procedure.

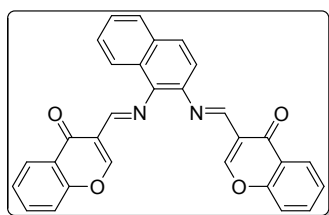


General procedure for synthesis of compounds (4a-r, 5a-c, 6a-c): To a G-4 process vial capped with Teflon septum was added 3-bromo flavone (1 mmol), boronic acid (1.1 mmol),

Pd-complex **C** (0.3 mol%, 1.6 mg), TBAB (0.25 mmol), K₂CO₃ (2.5 mmol) and 6 ml of ethanol. After a pre-stirring for one minute, the vial was subjected to microwave irradiation time of 18-20 minute at 60 °C. It was then cooled to room temperature, diluted with water, and extracted with EtOAc for three times. The organic phase was dried with Na₂SO₄ and concentrated to yield the product. The crude material was chromatographed on a silica gel column eluting with a mixture of ethyl acetate and hexane. (The purified products were identified by FTIR, NMR and HRMS spectra).

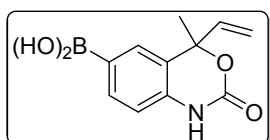
Characterization Data

3,3'-((1E,1'E)-(naphthalene-1,2-diylbis(azanylylidene))bis(methanylylidene))bis(4H-chromen-4-one) (L)



Yield: 85% as yellow solid. ¹H NMR (500 MHz, CDCl₃, ppm): δ 8.44-8.35 (m, 1H), 8.28-8.20 (m, 2H), 7.98-7.96 (m, 1H), 7.83-7.73 (m, 2H), 7.56-7.55 (m, 1H), 7.52-7.47 (m, 3H), 7.43-7.40 (m, 2H), 7.33-7.26 (m, 2H), 7.25-7.21 (m, 2H), 7.15-7.13 (m, 1H), 6.82 (m, 1H). ¹³C NMR (100 MHz, CDCl₃, ppm): δ 178.3, 161.8, 154.7, 151.8, 143.1, 137.4, 136.4, 134.8, 127.6, 126.3, 126.0, 125.1, 124.6, 121.9, 120.3, 118.2, 113.2, 108.8. FTIR (KBr, ν = cm⁻¹): 1715, 1645, 1624. Anal. Calcd (in %) for C₃₀H₁₈N₂O₄: C, 76.59; H, 3.86; N, 5.95. Found: C, 76.43; H, 3.72; N, 5.79. HRMS (ESI⁺): m/z calcd for C₃₀H₁₈N₂NaO₄ [M+Na]⁺: 493.1158, found: 493.1158.

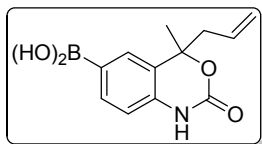
(4-methyl-2-oxo-4-vinyl-2,4-dihydro-1H-benzo[d][1,3]oxazin-6-yl)boronic acid (3a)



Yield: 81% as white solid; ¹H NMR (500 MHz, DMSO-d₆, ppm): δ 10.29 (s, 1H, D₂O exchangeable), 7.97 (s, 2H, D₂O exchangeable), 7.68 (d, *J* = 10Hz, 2H), 6.83 (d, *J* = 8Hz, 1H), 6.01 (dd, *J* = 10.5, 18Hz, 1H), 5.16 (d, *J* = 10.5Hz, 1H), 4.98 (d, *J* = 17Hz, 1H), 1.73 (s, 3H). ¹³C NMR (100 MHz, DMSO-d₆, ppm): δ 153.4, 140.6, 137.4, 136.5, 131.4, 128.9, 123.9, 116.2, 115.0, 85.1,

26.3. FTIR (KBr, $\nu = \text{cm}^{-1}$): 3408, 3279, 2978, 2930, 1718, 1645, 1513, 1355, 1248, 1088, 925. HRMS (ESI+): m/z calcd for $\text{C}_{11}\text{H}_{13}\text{BNO}_4$ $[\text{M}+\text{H}]^+$: 234.0932 found: 234.0930.

(4-allyl-4-methyl-2-oxo-2,4-dihydro-1H-benzo[d][1,3]oxazin-6-yl)boronic acid (3b)



Yield: 80% as white solid; ^1H NMR (500 MHz, DMSO-d_6 , ppm): δ

10.16 (s, 1H, D_2O exchangeable), 7.92 (s, 2H, D_2O , exchangeable),

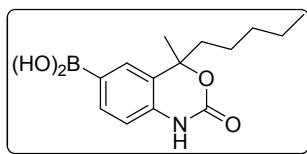
7.61 (t, $J = 3.5\text{Hz}$, 2H), 6.77 (d, $J = 8.5\text{Hz}$, 1H), 5.62-5.56 (m, 1H),

5.07-5.02 (m, 2H), 2.66-2.62 (m, 1H), 2.55-2.51 (m, 1H), 1.55 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6 , ppm): δ 153.2, 137.3, 136.1, 132.8, 131.2, 128.7, 124.6, 121.2, 114.9, 85.9, 45.1,

27.7. FTIR (KBr, $\nu = \text{cm}^{-1}$): 3430, 3158, 2978, 2934, 1720, 1656, 1383, 1342, 1257, 1109,

1042. HRMS (ESI+): m/z calcd for $\text{C}_{12}\text{H}_{16}\text{BNO}_4$ $[\text{M}+\text{H}]^+$: 248.1088 found: 248.1087.

(4-methyl-2-oxo-4-pentyl-2,4-dihydro-1H-benzo[d][1,3]oxazin-6-yl)boronic acid (3c)



Yield: 95% as yellow solid; ^1H NMR (500 MHz, CDCl_3 , ppm): δ

9.76 (s, 1H, D_2O exchangeable), 7.41 (d, $J = 8.5\text{ Hz}$, 1H), 7.18 (s,

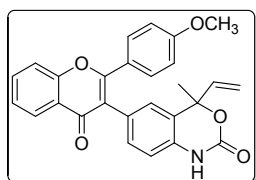
1H), 6.86 (d, $J = 8.5\text{ Hz}$, 1H), 2.03-1.98 (m, 2H), 1.75 (s, 3H),

1.49-1.35 (m, 6H), 0.94 (t, $J = 7\text{ Hz}$, 3H). ^{13}C NMR (125 MHz, CDCl_3 , ppm): δ 153.0, 133.6,

131.8, 127.0, 126.9, 116.5, 115.8, 85.5, 41.1, 31.9, 27.2, 23.3, 22.6, 14.8. HRMS (ESI+): m/z

calcd. for $\text{C}_{14}\text{H}_{21}\text{BNO}_4$ $[\text{M}+\text{H}]^+$: 278.1558.

6-(2-(4-methoxyphenyl)-4-oxo-4H-chromen-3-yl)-4-methyl-4-vinyl-1H-benzo[d][1,3]oxazin-2(4H)-one (4a)



Yield: 95% as brown solid, mp 160-168 $^\circ\text{C}$; ^1H NMR (500 MHz,

CDCl_3 , ppm): δ 9.08 (s, 1H, D_2O exchangeable), 8.27 (d, $J = 8\text{Hz}$, 1H),

7.80 (d, $J = 9\text{Hz}$, 1H), 7.7 (t, $J = 8.5\text{ Hz}$, 1H), 7.48 (d, $J = 8\text{ Hz}$, 1H),

7.44 (t, $J = 7.5\text{ Hz}$, 1H), 7.37 (dd, $J = 1.5, 8.5\text{ Hz}$, 1H), 7.13 (d, $J = 8\text{Hz}$, 1H), 7.08-7.02 (m,

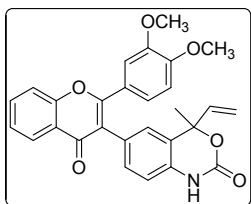
3H), 6.87 (d, $J = 8\text{Hz}$, 1H), 6.06-5.97 (m, 1H), 5.26-5.06 (m, 2H), 3.90 (s, 3H), 1.82 (s, 3H).

^{13}C NMR (125 MHz, CDCl_3 , ppm): δ 174.8, 164.5, 161.8, 155.9, 153.0, 139.4, 134.2, 132.3,

131.5, 129.4, 127.5, 127.3, 126.8, 125.9, 124.5, 123.6, 120.0, 117.7, 115.7, 114.8, 113.8,

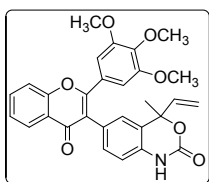
113.7, 87.7, 55.6, 25.7. FTIR (KBr, $\nu = \text{cm}^{-1}$): 3256, 2955, 1709, 1655, 1615, 1587, 1505, 1463, 1345, 1333, 1306. HRMS (ESI+): m/z calcd. for $\text{C}_{27}\text{H}_{22}\text{NO}_5$ $[\text{M}+\text{H}]^+$: 440.1492 found : 440.1495.

6-(2-(3,4-dimethoxyphenyl)-4-oxo-4H-chromen-3-yl)-4-methyl-4-vinyl-1H-benzo[d][1,3]oxazin-2(4H)-one (4b)



Yield: 91% as brown solid, mp 139-143 °C; ^1H NMR (500 MHz, CDCl_3 , ppm): δ 8.93 (s, 1H, D_2O exchangeable), 8.28 (d, $J = 8$ Hz, 1H), 7.70 (t, $J = 8$ Hz, 1H), 7.51 (t, $J = 8$ Hz, 2H), 7.46 (d, $J = 7.5$ Hz, 1H), 7.35 (dd, $J = 1.5, 8$ Hz, 1H), 7.13 (d, $J = 7.5$ Hz, 1H), 7.06 (t, $J = 7.5$ Hz, 1H), 7.00 (d, $J = 8.5$ Hz, 1H), 6.87 (d, $J = 7.5$ Hz, 1H), 6.05-5.96 (m, 1H), 5.25-5.05 (m, 2H), 3.95 (s, 6H), 1.81 (s, 3H). ^{13}C NMR (125 MHz, CDCl_3 , ppm): δ 173.4, 161.9, 155.7, 151.6, 148.6, 139.4, 134.6, 134.2, 132.3, 129.4, 127.5, 126.7, 125.8, 125.2, 124.5, 123.8, 123.5, 123.3, 121.9, 117.9, 114.8, 112.6, 110.7, 108.7, 83.8, 56.2, 25.7. FTIR (KBr, $\nu = \text{cm}^{-1}$): 3267, 2975, 1713, 1652, 1611, 1557, 1505, 1463, 1350, 1328, 1306. HRMS (ESI+): m/z calcd. for $\text{C}_{28}\text{H}_{23}\text{NNaO}_6$ $[\text{M}+\text{Na}]^+$: 492.1417 found : 492.1431.

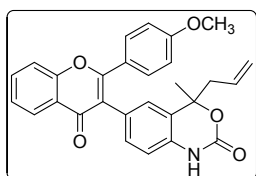
4-methyl-6-(4-oxo-2-(3,4,5-trimethoxyphenyl)-4H-chromen-3-yl)-4-vinyl-1H-benzo[d][1,3]oxazin-2(4H)-one (4c)



Yield: 89% as brown solid, mp 140-145 °C; ^1H NMR (500 MHz, CDCl_3 , ppm): δ 8.95 (s, 1H, D_2O exchangeable), 8.28 (d, $J = 8$ Hz, 1H), 7.72-7.68 (m, 1H), 7.51 (s, 1H), 7.36 (dd, $J = 2, 8.5$ Hz, 1H), 7.12 (d, $J = 7.5$ Hz, 1H), 7.06 (t, $J = 7.5$ Hz, 1H), 7.00 (d, $J = 8.5$ Hz, 1H), 6.87 (d, $J = 8$ Hz, 1H), 6.04-5.96 (m, 1H), 5.25-5.05 (m, 2H), 3.96 (s, 3H), 3.95 (s, 6H), 1.81 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 173.3, 161.9, 155.6, 153.1, 139.4, 134.4, 132.3, 129.4, 127.9, 127.5, 126.7, 125.9, 124.5, 123.5, 118.0, 115.7, 114.7, 107.1, 82.4, 61.2, 56.5, 25.7. FTIR (KBr, $\nu = \text{cm}^{-1}$): 3269,

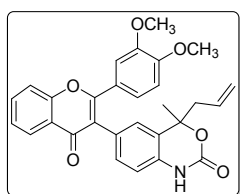
2959, 1713, 1690, 1652, 1621, 1555, 1513, 1473, 1359, 1336, 1321. HRMS (ESI+): m/z calcd. for $C_{29}H_{26}NO_7$ $[M+H]^+$: 522.1523 found : 522.1501.

4-allyl-6-(2-(4-methoxyphenyl)-4-oxo-4H-chromen-3-yl)-4-methyl-1H-benzo[d][1,3]oxazin-2(4H)-one (4d)



Yield: 94% as brown solid, mp 124-125 °C; 1H NMR (500 MHz, $CDCl_3$, ppm): δ 9.74 (s, 1H, D_2O exchangeable), 8.27 (d, $J = 8$ Hz, 1H), 7.72 (s, 1H), 7.53-7.50 (m, 1H), 7.46-7.42 (m, 1H), 7.31 (d, $J = 8.5$ Hz, 2H), 7.17 (s, 1H), 7.01 (s, 2H), 7.00 (d, $J = 8.5$ Hz, 1H), 6.78 (d, $J = 8.5$ Hz, 1H), 5.75-5.66 (m, 1H), 5.13-5.01 (m, 2H), 3.9 (s, 3H), 2.69-2.60 (m, 2H), 1.67 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$, ppm): δ 173.5, 162.0, 155.7, 152.5, 151.6, 148.6, 135.2, 134.3, 133.7, 132.0, 131.1, 127.1, 125.9, 123.3, 121.8, 120.5, 117.9, 116.4, 115.7, 112.5, 110.6, 108.6, 84.3, 56.3, 45.5, 26.3. FTIR (KBr, $\nu = cm^{-1}$): 3267, 2975, 1713, 1692, 1652, 1611, 1557, 1505, 1463, 1350, 1328, 1306. HRMS (ESI+): m/z calcd. for $C_{28}H_{24}NO_5$ $[M+H]^+$: 454.1648 found : 454.1636.

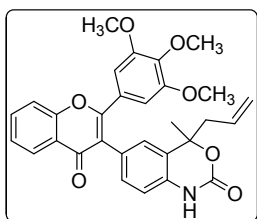
4-allyl-6-(2-(3,4-dimethoxyphenyl)-4-oxo-4H-chromen-3-yl)-4-methyl-1H-benzo[d][1,3]oxazin-2(4H)-one (4e)



Yield: 92% as brown solid, mp 117-119 °C; 1H NMR (500 MHz, $CDCl_3$, ppm): δ 9.40 (s, 1H, D_2O exchangeable), 8.29 (d, $J = 8$ Hz, 1H), 7.91 (s, 1H), 7.27 (t, $J = 8$ Hz, 1H), 7.53-7.45 (m, 2H), 7.32 (d, $J = 8$ Hz, 1H), 7.18 (s, 1H), 7.10 (d, $J = 18$ Hz, 2H), 6.79 (d, $J = 8.5$ Hz, 1H), 5.73-5.65 (m, 1H), 5.14-5.09 (m, 2H), 3.93 (d, 6H), 2.67-2.63 (m, 2H), 1.68 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$, ppm): δ 173.5, 162.0, 155.7, 152.6, 151.6, 148.6, 135.2, 134.3, 133.7, 132.0, 131.1, 127.1, 126.6, 125.9, 125.1, 123.4, 121.8, 120.5, 118.0, 116.4, 115.7, 112.5, 110.7, 108.7, 84.6, 56.3, 56.2, 45.5, 26.3. FTIR (KBr, $\nu = cm^{-1}$): 3265, 2975, 1713, 1652, 1641, 1555, 1513, 1473, 1349,

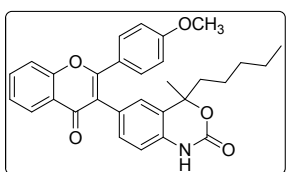
1331, 1311. HRMS (ESI+): m/z calcd. for $C_{29}H_{25}NNaO_6$ $[M+Na]^+$:506.1574 found : 506.1575.

4-allyl-4-methyl-6-(4-oxo-2-(3,4,5-trimethoxyphenyl)-4H-chromen-3-yl)-1H-benzo[d][1,3]oxazin-2(4H)-one (4f)



Yield: 88% as brown solid, mp 118-120 °C; 1H NMR (500 MHz, $CDCl_3$, ppm): δ 10.08 (s, 1H, D_2O exchangeable), 8.19 (dd, $J = 8$ Hz, 1H), 7.75 (s, 1H), 7.69-7.64 (m, 1H), 7.55-7.47 (m, 1H), 7.43-7.35 (m, 1H), 7.12-7.10 (m, 2H), 7.04 (s, 1H), 6.80-6.78 (m, 1H), 5.68-5.60 (m, 1H), 5.08-5.04 (m, 2H), 3.90 (s, 6H), 3.8 (s, 3H), 2.64-2.54 (m, 2H), 1.62 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$, ppm): δ 173.4, 162.0, 153.1, 152.4, 140.6, 135.0, 134.4, 133.7, 132.0, 131.1, 127.9, 127.2, 126.7, 126.6, 126.0, 121.8, 121.4, 120.5, 118.0, 116.4, 115.7, 107.2, 84.4, 61.2, 56.6, 45.5, 26.3. FTIR (KBr, $\nu = cm^{-1}$): 3235, 2934, 1716, 1686, 1645, 1592, 1463, 1406. HRMS (ESI+): m/z calcd. for $C_{30}H_{27}NNaO_7$ $[M+Na]^+$: 536.1679 found : 536.1678.

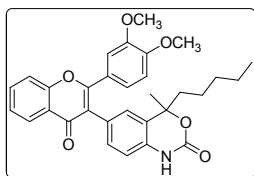
6-(2-(4-methoxyphenyl)-4-oxo-4H-chromen-3-yl)-4-methyl-4-pentyl-1H-benzo[d][1,3]oxazin-2(4H)-one (4g)



Yield: 90% as white solid, mp 113-114 °C; 1H NMR (500 MHz, $CDCl_3$, ppm): δ 9.90 (s, 1H, D_2O exchangeable), 8.14 (dd, $J = 8$ Hz, 1H), 7.78 (d, $J = 8.5$ Hz, 1H), 7.67 (t, $J = 8$ Hz, 1H), 7.42 (m, 2H), 7.28 (d, $J = 8.5$ Hz, 1H), 7.15 (s, 2H), 6.99 (d, $J = 8.5$ Hz, 2H), 6.79 (d, $J = 8.5$ Hz, 1H), 3.86 (s, 3H), 1.91-1.87 (m, 2H), 1.64 (s, 3H), 1.37-1.24 (m, 6H), 0.82 (t, $J = 8$ Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$, ppm): δ 178.0, 155.9, 155.4, 152.8, 146.4, 146.0, 133.9, 133.7, 131.7, 127.0, 126.9, 125.8, 125.3, 124.2, 118.0, 116.5, 115.7, 113.3, 112.6, 105.5, 85.4, 41.0, 31.8, 27.1, 23.2, 22.5, 14.1. FTIR (KBr, $\nu = cm^{-1}$): 3228, 2934, 2869, 1705, 1687, 1615,

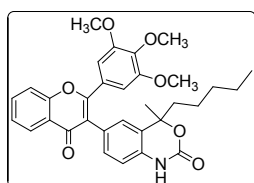
1463, 1406, 1352. HRMS (ESI+): m/z calcd. for $C_{30}H_{29}NNaO_5$ $[M+Na]^+$: 506.1937 found : 506.1921.

6-(2-(3,4-dimethoxyphenyl)-4-oxo-4H-chromen-3-yl)-4-methyl-4-pentyl-1H-benzo[d][1,3]oxazin-2(4H)-one (4h)



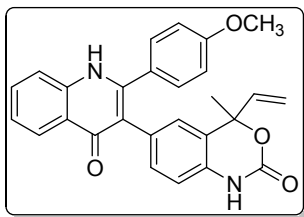
Yield: 89% as white solid, mp 109-110 °C; 1H NMR (500 MHz, $CDCl_3$, ppm): δ 9.61 (s, 1H, D_2O exchangeable), 7.80-7.69 (m, 1H), 7.51 (t, J = 7.5 Hz, 1H), 7.46-7.41 (m, 1H), 7.31 (d, J = 8.5 Hz, 1H), 7.15 (s, 1H), 6.78 (d, J = 8 Hz, 1H), 3.95-3.96 (d, 6H), 1.92-1.88 (m, 2H), 1.65 (s, 3H), 1.37-1.28 (m, 8H), 0.84 (t, J = 8 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$, ppm): δ 173.4, 161.9, 152.7, 152.9, 151.6, 148.6, 134.2, 133.6, 131.8, 127.0, 126.9, 126.7, 125.8, 125.2, 123.3, 121.8, 117.9, 116.5, 115.8, 112.6, 110.7, 108.7, 85.5, 56.3, 56.2, 41.0, 31.9, 27.2, 23.3, 22.5, 14.1. HRMS (ESI+): m/z calcd. for $C_{31}H_{31}NNaO_6$ $[M+Na]^+$: 536.2043 found: 536.2034.

4-methyl-6-(4-oxo-2-(3,4,5-trimethoxyphenyl)-4H-chromen-3-yl)-4-pentyl-1H-benzo[d][1,3]oxazin-2(4H)-one (4i)



Yield: 87% as white solid, mp 155-158 °C; 1H NMR (500 MHz, $CDCl_3$, ppm): δ 9.63 (s, 1H, D_2O exchangeable), 8.28 (d, J = 8 Hz, 1H), 7.72 (t, J = 8 Hz, 1H), 7.52-7.44 (m, 2H), 7.31 (d, J = 8.5 Hz, 1H), 7.16 (s, 1H), 7.09 (s, 2H), 6.79 (d, J = 8 Hz, 1H), 3.93 (d, 9H), 1.92-1.89 (m, 2H), 1.65 (s, 3H), 1.31-1.23 (m, 6H), 0.83 (t, J = 7 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$, ppm): δ 173.3, 161.9, 155.6, 153.1, 152.9, 140.6, 134.4, 133.6, 131.8, 127.9, 127.0, 126.9, 126.7, 126.0, 121.8, 118.0, 116.5, 115.8, 109.1, 107.2, 85.5, 61.1, 56.5, 41.0, 31.8, 27.2, 23.2, 22.5, 14.1. HRMS (ESI+): m/z calcd. for $C_{32}H_{33}NNaO_7$ $[M+Na]^+$: 566.2149 found: 566.2151.

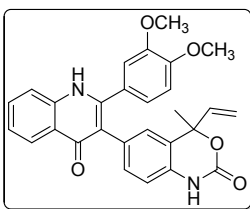
6-(2-(4-methoxyphenyl)-4-oxo-1,4-dihydroquinolin-3-yl)-4-methyl-4-vinyl-1H-benzo[d][1,3]oxazin-2(4H)-one (4j)



Yield: 86% as brown solid, mp 159-161 °C; ¹H NMR (500 MHz, CDCl₃, ppm): δ9.49 (s, 1H, D₂O exchangeable), 9.08 (s, 1H, D₂O exchangeable), 8.28 (d, *J* = 8 Hz, 1H), 7.79 (d, *J* = 9 Hz, 1H), 7.70 (t, *J* = 8.5 Hz, 1H), 7.37 (dd, *J* = 1.5, 8.5 Hz, 1H), 7.13 (d, *J* = 8

Hz, 1H), 7.08-7.02 (m, 2H), 6.87 (d, *J* = 8 Hz, 1H), 6.84 (d, *J* = 8 Hz, 1H), 6.06-6.00 (m, 1H), 5.26-5.06 (m, 2H), 3.90 (s, 3H), 1.82 (t, *J* = 8 Hz, 3H). ¹³C NMR (125 MHz, CDCl₃, ppm): δ174.8, 164.5, 161.8, 155.9, 139.4, 138.7, 134.6, 134.2, 132.3, 131.5, 129.4, 127.5, 123.6, 120.0, 117.7, 115.7, 114.8, 113.7, 87.7, 55.6, 25.7. FTIR (KBr, ν = cm⁻¹): 3305, 3267, 2975, 1714, 1692, 1641, 1557, 1505, 1463. HRMS (ESI⁺): *m/z* calcd. for C₂₇H₂₂N₂NaO₄ [M+Na]⁺ : 461.1471 found : 461.1467.

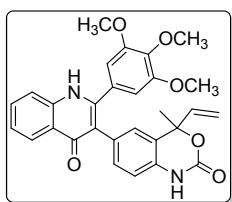
6-(2-(3,4-dimethoxyphenyl)-4-methyl-4-vinyl-1H-benzo[d][1,3]oxazin-2(4H)-one (4k)



Yield: 85% as Brown solid, mp 147-150 °C; ¹H NMR (500 MHz, CDCl₃, ppm): δ9.31 (s, 1H, D₂O exchangeable), 8.93 (s, 1H, D₂O exchangeable), 8.28 (d, *J* = 8 Hz, 1H), 7.70 (t, *J* = 8 Hz, 1H), 7.51 (t, *J* = 8.5 Hz, 2H), 7.46 (d, *J* = 8 Hz, 1H), 7.36 (dd, *J* = 1.5, 8.5 Hz, 1H), 7.13 (d, *J* = 7.5 Hz, 1H),

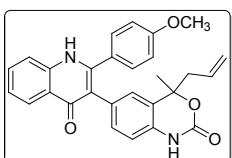
7.06 (t, *J* = 7.5 Hz, 1H), 6.99 (d, *J* = 8.5 Hz, 1H), 6.87 (d, *J* = 7.5 Hz, 1H), 6.05-5.98 (m, 1H), 5.20-5.05 (m, 2H), 3.95 (s, 6H), 1.81 (t, *J* = 8 Hz, 3H). ¹³C NMR (125 MHz, CDCl₃, ppm): δ174.8, 164.5, 161.8, 156.0, 153.2, 139.4, 138.7, 134.6, 134.2, 133.8, 132.3, 131.5, 129.4, 127.5, 126.9, 125.9, 124.5, 123.8, 123.6, 120.1, 117.7, 115.8, 114.8, 113.7, 84.3, 55.8, 25.8. HRMS (ESI⁺): *m/z* calcd. for C₂₈H₂₄N₂NaO₅ [M+Na]⁺: 491.1577 found : 491.1572.

4-methyl-6-(4-oxo-2-(3,4,5-trimethoxyphenyl)-1,4-dihydroquinolin-3-yl)-4-vinyl-1H-benzo[d][1,3]oxazin-2(4H)-one (4l)



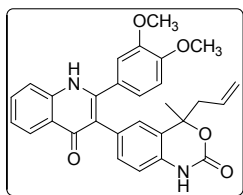
Yield: 85% as Brown solid, mp 112-115 °C; ¹H NMR (500 MHz, CDCl₃, ppm): δ9.31 (s, 1H, D₂O exchangeable), 8.93 (s, 1H, D₂O exchangeable), 8.26 (d, *J* = 8Hz, 1H), 7.70-7.68 (m, 1H), 7.50 (s, 1H), 7.34 (dd, *J* = 2, 8.5 Hz, 1H), 7.11 (d, *J* = 7.5 Hz, 1H), 7.04 (t, *J* = 7.5 Hz, 1H), 6.98 (d, *J* = 8.5 Hz, 1H), 6.86(d, *J* = 8 Hz, 1H), 6.03-5.94 (m, 1H), 5.23-5.03 (m, 2H), 3.95 (s, 3H), 3.94 (s, 6H), 1.79 (s, 3H). ¹³C NMR (125 MHz, CDCl₃, ppm): δ174.7, 164.1, 161.5, 156.5, 153.6, 139.1, 138.5, 134.1, 134.0, 133.6, 132.2, 131.4, 129.3, 127.4, 126.8, 125.4, 124.2, 123.7, 123.5, 120.1, 117.5, 115.7, 84.3, 60.5, 55.8, 25.7. HRMS (ESI+): *m/z* calcd. for C₂₉H₂₆N₂NaO₆ [M+Na]⁺ : 521.1683 found : 521.1682.

4-allyl-6-(2-(4-methoxyphenyl)-4-oxo-1,4-dihydroquinolin-3-yl)-4-methyl-1H-benzo[d][1,3]oxazin-2(4H)-one (4m)



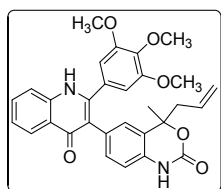
Yield: 86% as Brown solid, mp 138-141 °C; ¹H NMR (500 MHz, CDCl₃, ppm): δ9.61 (s, 1H, D₂O exchangeable), 8.26 (dd, *J* = 1, 8 Hz, 1H), 7.80 (d, *J* = 8.5 Hz, 2H), 7.68 (t, *J* = 7.5 Hz, 1H), 7.48 (d, *J* = 8.5 Hz, 1H), 7.43 (t, *J* = 7 Hz, 1H), 7.32 (dd, *J* = 1.5, 8.5 Hz, 1H), 7.18 (s, 1H), 7.02 (d, *J* = 8.5 Hz, 2H), 6.77 (d, *J* = 8.5 Hz, 1H), 6.23 (s, 1H, D₂O exchangeable), 5.72-5.65 (m, 1H), 5.14-5.09 (m, 2H), 3.89 (s, 3H, 2.70-2.62 (m, 2H), 1.68 (s, 3H). ¹³C NMR (125 MHz, CDCl₃, ppm): δ175.0, 164.6, 161.8, 155.9, 152.5, 134.3, 133.7, 132.0, 131.9, 131.5, 131.1, 127.2, 127.1, 126.8, 126.6, 125.9, 120.5, 120.0, 117.7, 116.6, 115.7, 113.7, 84.3, 55.6, 45.5, 26.3. FTIR (KBr, ν = cm⁻¹): 3394, 3293, 2957, 2930, 1712, 1686, 1646, 1610, 1572, 1355. HRMS (ESI+): *m/z* calcd. for C₂₈H₂₄N₂NaO₄ [M+Na]⁺ : 475.1628 found : 475.1631.

4-allyl-6-(2-(3,4-dimethoxyphenyl)-4-oxo-1,4-dihydroquinolin-3-yl)-4-methyl-1H-benzo[d][1,3]oxazin-2(4H)-one (4n)



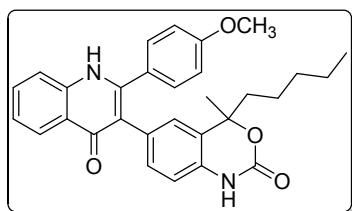
Yield: 85% as Brown solid, mp 160-165 °C; ¹H NMR (500 MHz, CDCl₃, ppm): δ 9.66 (s, 1H, D₂O exchangeable), 8.28 (dd, *J* = 1, 8 Hz, 1H), 7.74-7.71 (m, 2H), 7.52 (d, *J* = 8 Hz, 1H), 7.46 (t, *J* = 7 Hz, 1H), 7.31 (dd, *J* = 1.5, 8 Hz, 1H), 7.17 (d, *J* = 1.5 Hz, 1H), 7.09 (s, 2H), 6.78 (d, *J* = 8.5 Hz, 1H), 5.71-5.67 (m, 1H), 5.52 (s, 1H, D₂O exchangeable), 5.13-5.09 (m, 2H), 3.94 (s, 3H), 2.69-2.60 (m, 2H), 1.67 (s, 3H). ¹³C NMR (125 MHz, CDCl₃, ppm): δ 174.1, 152.4, 150.5, 150.3, 148.5, 139.4, 135.2, 133.7, 132.2, 132.0, 131.1, 127.2, 126.6, 124.4, 123.5, 122.3, 121.9, 120.5, 116.4, 115.7, 112.6, 110.6, 84.3, 56.1, 56.0, 45.5, 26.3. FTIR (KBr, ν = cm⁻¹): 3375, 3290, 2987, 2927, 1713, 1677, 1648, 1601, 1407, 1352. HRMS (ESI⁺): *m/z* calcd. for C₂₉H₂₆N₂NaO₅ [M+Na]⁺: 505.1733 found: 505.1733.

4-allyl-4-methyl-6-(4-oxo-2-(3,4,5-trimethoxyphenyl)-1,4-dihydroquinolin-3-yl)-1H-benzo[d][1,3]oxazin-2(4H)-one (4o)



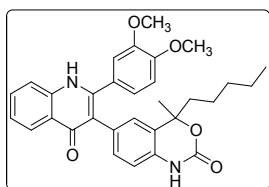
Yield: 81% as Brown solid, mp 178-180 °C; ¹H NMR (500 MHz, CDCl₃, ppm): δ 9.71 (s, 1H, D₂O exchangeable), 9.20 (s, 1H, D₂O exchangeable), 8.24 (d, *J* = 8 Hz, 1H), 7.83 (d, *J* = 8 Hz, 1H), 7.63-7.58 (m, 2H), 7.34-7.28 (m, 2H), 7.15 (s, 1H), 7.03 (s, 1H), 6.78 (d, *J* = 8.5 Hz, 1H), 5.71-5.63 (m, 1H), 5.12-5.07 (m, 2H), 3.77 (s, 9H), 2.67-2.58 (m, 2H), 1.65 (s, 3H). ¹³C NMR (125 MHz, CDCl₃, ppm): δ 173.9, 152.8, 152.3, 150.5, 139.4, 138.9, 135.1, 133.7, 131.9, 131.1, 130.3, 127.1, 126.5, 126.0, 124.5, 123.4, 121.7, 120.4, 118.7, 116.4, 115.6, 106.8, 84.2, 60.9, 56.3, 45.5, 26.3. HRMS (ESI⁺): *m/z* calcd. for C₃₀H₂₈N₂NaO₆ [M+Na]⁺: 535.1839 found: 535.1841.

6-(2-(4-methoxyphenyl)-4-oxo-1,4-dihydroquinolin-3-yl)-4-methyl-4-pentyl-1H-benzo[d][1,3]oxazin-2(4H)-one (4p)



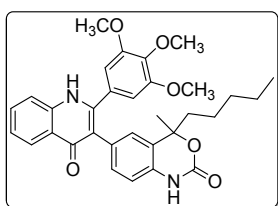
Yield: 83% as white solid, mp 145-148 °C; ¹H NMR (500 MHz, CDCl₃, ppm): δ 9.90 (s, 1H, D₂O exchangeable), 8.24 (d, *J* = 8 Hz, 1H), 7.78 (d, *J* = 8.5 Hz, 1H), 7.65 (t, *J* = 7.5 Hz, 1H), 7.46-7.39 (m, 2H), 7.29 (d, *J* = 8.5 Hz, 1H), 7.15 (s, 2H), 7.00 (d, *J* = 8.5 Hz, 2H), 6.80 (d, *J* = 8.5 Hz, 1H), 5.82 (s, 1H, D₂O exchangeable), 3.86 (s, 3H), 1.92-1.87 (m, 2H), 1.64 (s, 3H), 1.37-1.29 (m, 6H), 0.82 (s, 3H). ¹³C NMR (125 MHz, CDCl₃, ppm): δ 174.9, 164.5, 161.8, 155.9, 153.0, 145.1, 134.2, 131.7, 131.5, 127.2, 127.0, 126.9, 126.8, 125.9, 120.0, 117.6, 116.5, 115.7, 113.7, 85.4, 55.6, 41.0, 31.8, 27.1, 23.2, 22.5, 14.0. FTIR (KBr, ν = cm⁻¹): 3375, 3290, 2987, 2927, 1710, 1677, 1643, 1407, 1352. HRMS (ESI⁺): *m/z* calcd. for C₃₀H₃₀N₂NaO₄ [M+Na]⁺ : 505.2097 found : 505.2096.

6-(2-(3,4-dimethoxyphenyl)-4-oxo-1,4-dihydroquinolin-3-yl)-4-methyl-4-pentyl-1H-benzo[d][1,3]oxazin-2(4H)-one (4q)



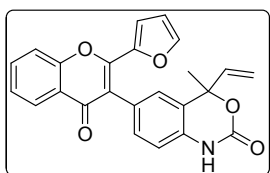
Yield: 85% as white solid, mp 117-118 °C; ¹H NMR (500 MHz, DMSO, ppm): δ 12.18, (s, 1H, D₂O exchangeable), 10.28 (s, 1H, D₂O exchangeable), 8.16 (d, *J* = 8 Hz, 1H), 7.68 (d, *J* = 3.5 Hz, 2H), 7.41-7.38 (m, 3H), 7.23-7.13 (m, 2H), 7.02 (s, 1H), 6.82 (d, *J* = 8.5 Hz, 1H), 3.84 (s, 3H), 3.82 (s, 3H), 1.91 (t, *J* = 13 Hz, 2H), 1.56 (s, 3H), 1.28-0.80 (m, 6H), 0.79 (t, *J* = 7 Hz, 3H). ¹³C NMR (125 MHz, CDCl₃, ppm): δ 173.4, 161.9, 155.6, 152.9, 151.6, 148.6, 134.2, 133.6, 131.8, 126.9, 126.6, 125.8, 125.2, 123.3, 121.8, 117.9, 116.5, 115.8, 112.5, 110.7, 108.7, 85.5, 56.3, 56.2, 41.0, 31.8, 27.2, 23.2, 22.5, 14.1. HRMS (ESI⁺): *m/z* calcd. for C₃₁H₃₂N₂NaO₅ [M+Na]⁺ : 535.2203 found: 535.2170.

4-methyl-6-(4-oxo-2-(3,4,5-trimethoxyphenyl)-1,4-dihydroquinolin-3-yl)-4-pentyl-1H-benzo[d][1,3]oxazin-2(4H)-one (4r)



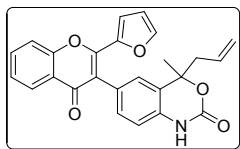
Yield: 84% as white solid, mp 110-113 °C; ¹H NMR (500 MHz, DMSO, ppm): δ 12.25 (s, 1H, D₂O exchangeable), 10.27 (s, 1H, D₂O exchangeable), 8.29-8.22 (m, 1H), 8.15 (t, *J* = 8 Hz, 1H), 7.41-7.38 (m, 4H), 7.28 (s, 1H), 6.93 (d, *J* = 21.5 Hz, 1H), 6.82 (d, *J* = 8.5 Hz, 1H), 3.83 (s, 6H), 3.74 (s, 3H), 1.93-1.74 (m, 2H), 1.56 (s, 3H), 1.27-1.16 (m, 6H), 0.79 (t, *J* = 10 Hz, 3H). ¹³C NMR (125 MHz, CDCl₃, ppm): δ 173.3, 161.9, 155.6, 153.1, 152.9, 140.6, 134.4, 133.6, 131.8, 127.9, 127.0, 126.9, 126.7, 126.0, 121.8, 118.0, 116.5, 115.8, 109.1, 107.2, 85.5, 61.1, 56.5, 41.0, 31.8, 27.2, 23.2, 22.5, 14.1. HRMS (ESI⁺): *m/z* calcd. for C₃₂H₃₄N₂NaO₆ [M+Na]⁺ : 565.2309 found : 565.2304.

6-(2-(furan-2-yl)-4-oxo-4H-chromen-3-yl)-4-methyl-4-vinyl-1H-benzo[d][1,3]oxazin-2(4H)-one (5a)



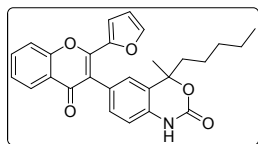
Yield: 90% as brown solid, mp 97-99 °C; ¹H NMR (500 MHz, CDCl₃, ppm): δ 8.75 (s, 1H, D₂O exchangeable), 8.19 (d, *J* = 7.5 Hz, 1H), 7.65 (t, *J* = 8 Hz, 1H), 7.61 (m, 2H), 7.48 (d, *J* = 8.5 Hz, 2H), 7.38 (t, *J* = 7.5 Hz, 1H), 7.12 (d, *J* = 2.5 Hz, 1H), 6.78 (s, 1H), 6.59 (s, 1H), 6.04-5.96 (m, 1H), 5.24-5.05 (m, 2H), 1.81 (s, 3H). ¹³C NMR (100 MHz, CDCl₃, ppm): δ 178.0, 155.9, 155.3, 145.9, 139.4, 138.7, 133.9, 132.2, 129.3, 127.4, 125.8, 125.3, 124.5, 123.5, 118.0, 116.5, 115.7, 114.7, 113.3, 112.7, 105.57, 84.2. FTIR (KBr, ν = cm⁻¹): 3290, 2987, 1712, 1677, 1652, 1635, 1586, 1422, 1352. HRMS (ESI⁺): *m/z* calcd. for C₂₄H₁₇NNaO₅ [M+Na]⁺ : 422.0998 found : 422.0995.

4-allyl-6-(2-(furan-2-yl)-4-oxo-4H-chromen-3-yl)-4-methyl-1H-benzo[d][1,3]oxazin-2(4H)-one (5b)



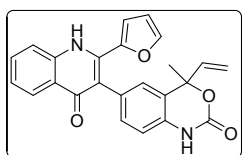
Yield: 91% as brown solid, mp 95-97 °C; ¹H NMR (500 MHz, CDCl₃, ppm): δ 9.82 (s, 1H, D₂O exchangeable), 8.17 (d, *J* = 8 Hz, 1H), 7.76 (s, 1H), 7.65 (t, *J* = 7.5 Hz, 1H), 7.59 (s, 1H), 7.47 (d, *J* = 8.5 Hz, 1H), 7.37 (t, *J* = 7 Hz, 1H), 7.30 (d, *J* = 8.5 Hz, 1H), 7.16 (s, 1H), 7.12 (d, *J* = 8.5 Hz, 1H), 6.80 (d, *J* = 8 Hz, 1H), 5.73-5.65 (m, 1H), 5.13-5.08 (m, 2H), 2.66-2.62 (m, 2H), 1.66 (s, 3H). ¹³C NMR (125 MHz, CDCl₃, ppm): δ 178.2, 156.0, 155.5, 152.5, 146.5, 146.1, 134.0, 132.0, 131.2, 127.1, 126.6, 125.9, 125.4, 124.3, 121.8, 120.5, 118.1, 116.5, 115.7, 113.4, 112.8, 105.5, 84.3, 45.6, 26.3. FTIR (KBr, ν = cm⁻¹): 3284, 1713, 1697, 1609, 1556, 1372, 1303, 1236. HRMS (ESI⁺): *m/z* calcd. for C₂₅H₁₉NNaO₅ [M+Na]⁺ : 436.1155 found : 436.1161.

6-(2-(furan-2-yl)-4-oxo-4H-chromen-3-yl)-4-methyl-4-pentyl-1H-benzo[d][1,3]oxazin-2(4H)-one (5c)



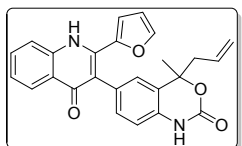
Yield: 92% as brown solid, mp 96-99 °C; ¹H NMR (500 MHz, CDCl₃, ppm): δ 9.69 (s, 1H, D₂O exchangeable), 8.17 (d, *J* = 6.5 Hz, 1H), 7.64-7.59 (m, 2H), 7.46 (d, *J* = 7.5 Hz, 1H), 7.36 (t, *J* = 8 Hz, 1H), 7.29 (d, *J* = 7 Hz, 1H), 7.12 (d, *J* = 20 Hz, 2H), 6.79 (d, *J* = 8 Hz, 1H), 6.58 (s, 1H), 1.91-1.86 (m, 2H), 1.64 (s, 3H), 1.37-1.23 (m, 6H), 0.82 (t, *J* = 8 Hz, 3H). ¹³C NMR (125 MHz, CDCl₃, ppm): δ 178.0, 155.9, 155.4, 152.8, 146.4, 146.0, 133.9, 133.7, 131.7, 127.0, 126.9, 125.8, 125.3, 124.2, 118.0, 116.5, 115.7, 113.3, 112.6, 105.5, 85.4, 41.0, 31.8, 27.1, 23.2, 22.5, 14.1. HRMS (ESI⁺): *m/z* calcd. for C₂₇H₂₅NNaO₅ [M+Na]⁺ : 466.1624 found : 466.1636.

6-(2-(furan-2-yl)-4-oxo-1,4-dihydroquinolin-3-yl)-4-methyl-4-vinyl-1H-benzo[d][1,3]oxazin-2(4H)-one (6a)



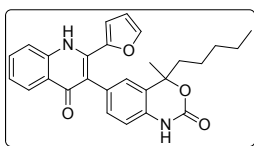
Yield: 89% as brown solid, mp 127-130 °C; ¹H NMR (500 MHz, CDCl₃, ppm): δ 9.17 (s, 1H, D₂O exchangeable), 8.75 (s, 1H, D₂O exchangeable), 8.19 (d, *J* = 9 Hz, 1H), 7.66 (t, *J* = 7.5 Hz, 1H), 7.61 (m, 2H), 7.48 (d, *J* = 8.5 Hz, 2H), 7.38 (t, *J* = 7.5 Hz, 1H), 7.12 (d, *J* = 2.5 Hz, 1H), 6.78 (s, 1H), 6.59 (s, 1H), 6.04-5.96 (m, 1H), 5.24-5.05 (m, 2H), 1.81 (s, 3H). ¹³C NMR (100 MHz, CDCl₃, ppm): δ 178.1, 155.8, 155.3, 145.9, 139.4, 138.7, 133.9, 132.2, 129.3, 127.4, 125.8, 125.3, 124.5, 123.5, 118.0, 116.5, 115.7, 114.7, 113.3, 112.7, 105.57, 84.2. FTIR (KBr, ν = cm⁻¹): 3486, 3229, 2869, 1717, 1693, 1645, 1546, 1462, 1341, 1259. HRMS (ESI⁺): *m/z* calcd. for C₂₄H₁₈N₂NaO₄ [M+Na]⁺ : 421.1158 found : 421.1152.

4-allyl-6-(2-(furan-2-yl)-4-oxo-1,4-dihydroquinolin-3-yl)-4-methyl-1H-benzo[d][1,3]oxazin-2(4H)-one (6b)



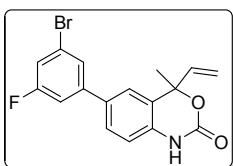
Yield: 87% as brown solid, mp 146-148 °C; ¹H NMR (400 MHz, CDCl₃, ppm): δ 9.90 (s, 1H, D₂O exchangeable), 8.17 (dd, *J* = 2, 10 Hz, 1H), 7.67-7.63 (m, 1H), 7.59 (d, *J* = 1 Hz, 1H), 7.47 (d, *J* = 10 Hz, 1H), 7.39-7.35 (m, 1H), 7.30-7.27 (m, 1H), 7.15 (d, *J* = 2.5 Hz, 1H), 7.11 (d, *J* = 4.5 Hz, 1H), 6.80 (d, *J* = 10.5 Hz, 1H), 6.59-6.57 (m, 1H), 6.24 (s, 1H, D₂O exchangeable), 5.73-5.63 (m, 1H), 5.12-5.07 (m, 2H), 2.69-2.58 (m, 2H), 1.66 (s, 3H). ¹³C NMR (125 MHz, CDCl₃, ppm): δ 178.2, 156.1, 155.4, 152.5, 146.5, 146.1, 134.0, 132.0, 131.2, 127.1, 126.6, 125.9, 125.4, 124.3, 121.8, 120.5, 118.1, 116.5, 115.7, 113.4, 112.8, 105.5, 84.3, 45.6, 26.3. FTIR (KBr, ν = cm⁻¹): 3492, 3278, 2849, 1715, 1689, 1665, 1609, 1565, 1412, 1343, 1259. HRMS (ESI⁺): *m/z* calcd. for C₂₅H₂₀N₂NaO₄ [M+Na]⁺ : 435.1315 found : 435.1314.

6-(2-(furan-2-yl)-4-oxo-1,4-dihydroquinolin-3-yl)-4-methyl-4-pentyl-1H-benzo[d][1,3]oxazin-2(4H)-one (6c)



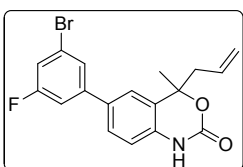
Yield: 91% as brown solid, mp 153-157 °C; ¹H NMR (500 MHz, CDCl₃, ppm): δ 9.69 (s, 1H, D₂O exchangeable), 8.17 (d, *J* = 6.5 Hz, 1H), 7.64-7.59 (m, 2H), 7.46 (d, *J* = 7.5 Hz, 1H), 7.36 (t, *J* = 8 Hz, 1H), 7.29 (d, *J* = 7 Hz, 1H), 7.12 (d, *J* = 20 Hz, 2H), 6.79 (d, *J* = 8 Hz, 1H), 6.58 (s, 1H), 4.23 (s, 1H, D₂O exchangeable), 1.91-1.86 (m, 2H), 1.64 (s, 3H), 1.37-1.23 (m, 6H), 0.82 (t, *J* = 7 Hz, 3H). ¹³C NMR (125 MHz, CDCl₃, ppm): δ 178.0, 155.9, 155.4, 152.8, 146.4, 146.0, 133.9, 133.7, 131.8, 127.1, 126.9, 125.8, 125.3, 124.3, 118.0, 116.5, 115.7, 113.3, 112.7, 105.5, 85.4, 41.0, 31.8, 27.2, 23.2, 22.5, 14.1. HRMS (ESI⁺): *m/z* calcd. for C₂₇H₂₆N₂NaO₄ [M+Na]⁺ : 465.1784 found : 465.1781.

6-(3-bromo-5-fluorophenyl)-4-methyl-4-vinyl-1H-benzo[d][1,3]oxazin-2(4H)-one (6aa)



Yield: 94% as white solid; ¹H NMR (500 MHz, CDCl₃, ppm): δ 8.53 (s, 1H, D₂O exchangeable), 7.46 (s, 2H), 7.28 (s, 1H), 7.22 (d, *J* = 8 Hz, 1H), 7.16 (d, *J* = 9.5 Hz, 1H), 6.93 (d, *J* = 8.5 Hz, 1H), 6.08 (dd, *J* = 10.5, 17 Hz, 1H), 5.28 (d, *J* = 11 Hz, 1H), 5.16 (d, *J* = 17 Hz, 1H), 1.25 (s, 3H). ¹³C NMR (100 MHz, CDCl₃, ppm): δ 164.4, 161.9, 153.0, 144.0, 134.7, 134.1, 131.4, 128.0, 125.9, 125.3, 122.9, 120.5, 118.0, 115.5, 113.1, 85.1, 26.6. FTIR (KBr, ν = cm⁻¹): 3142, 2912, 1701, 1656. HRMS (ESI⁺): *m/z* calcd for C₁₇H₁₄BrFNO₂ [M+H]⁺: 362.0192 found: 362.0189.

4-allyl-6-(3-bromo-5-fluorophenyl)-4-methyl-1H-benzo[d][1,3]oxazin-2(4H)-one (7aa)



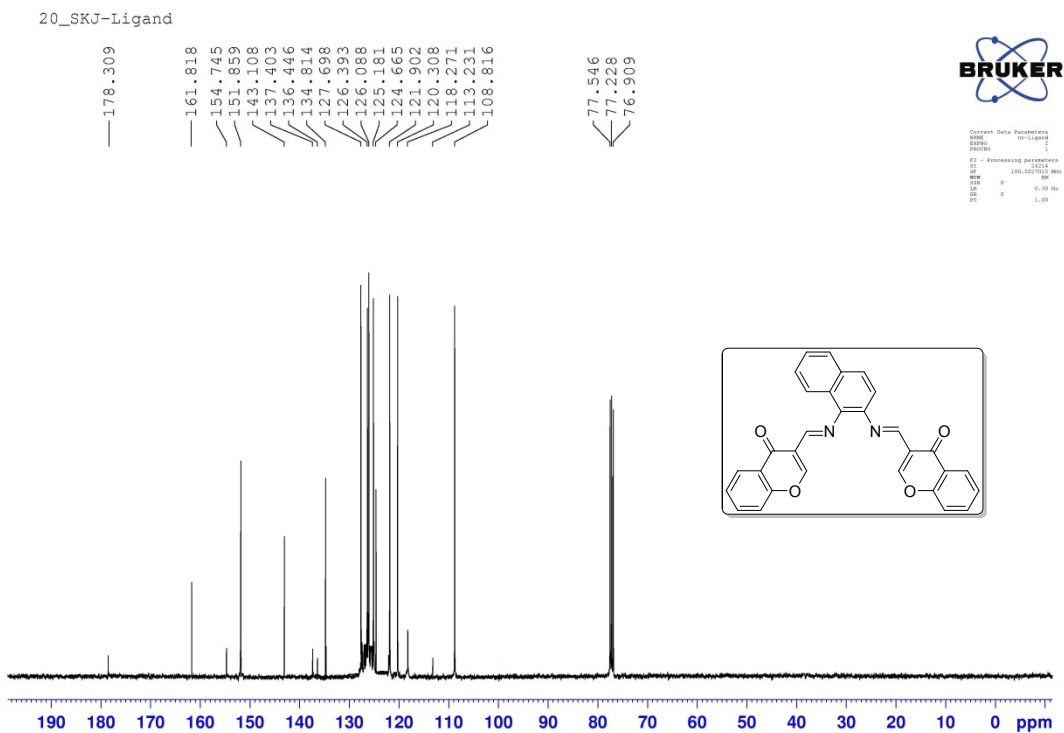
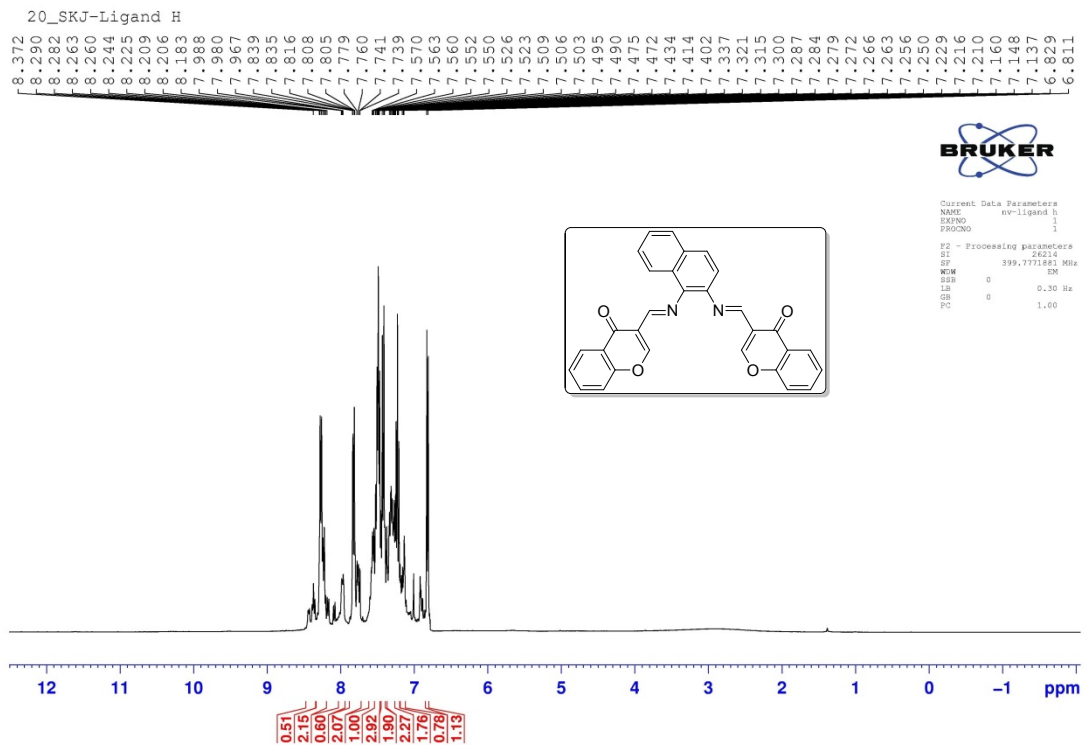
Yield: 94% as white solid; ¹H NMR (500 MHz, CDCl₃, ppm): δ 9.52 (s, 1H, D₂O exchangeable), 7.42 (t, *J* = 6.5 Hz, 2H), 7.21 (s, 2H), 7.15 (d, *J* = 9 Hz, 1H), 6.97 (d, *J* = 8.5 Hz, 1H), 5.80-5.72 (m, 1H), 5.17-5.14 (m, 2H), 2.81-2.71 (m, 2H), 1.78 (s, 3H). ¹³C NMR (100 MHz, CDCl₃, ppm): δ 164.5, 161.9, 153.2, 144.2, 134.9, 134.2, 131.6, 128.1, 126.0, 125.4, 123.3, 120.6, 118.1, 115.7, 113.2,

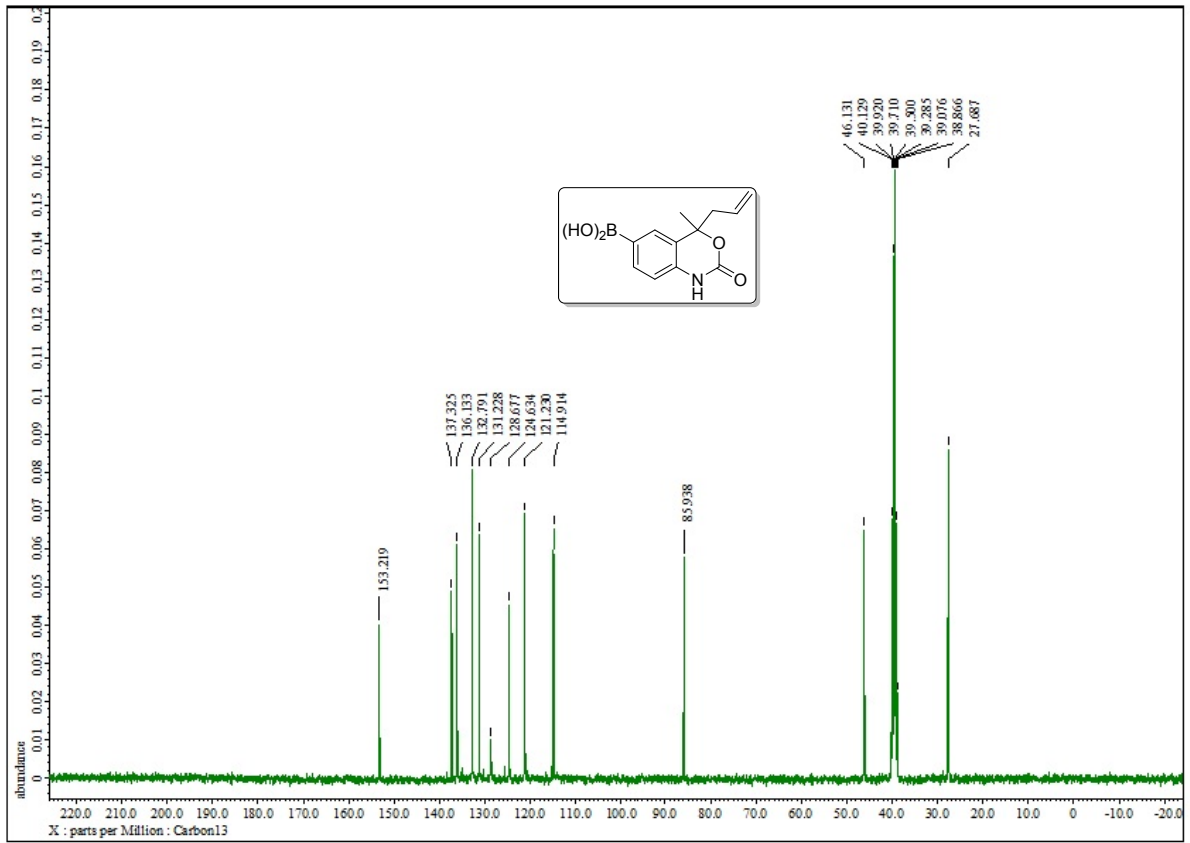
85.2, 45.9, 26.7. FTIR (KBr, $\nu = \text{cm}^{-1}$): 3140, 2913, 1704, 1655, 1573, 1516, 1431, 1356, 1304, 1168, 823, 768. HRMS (ESI+): m/z calcd for $\text{C}_{18}\text{H}_{15}\text{BrFNNaO}_2$ $[\text{M}+\text{Na}]^+$: 398.0168 found: 398.0184.

Reference:

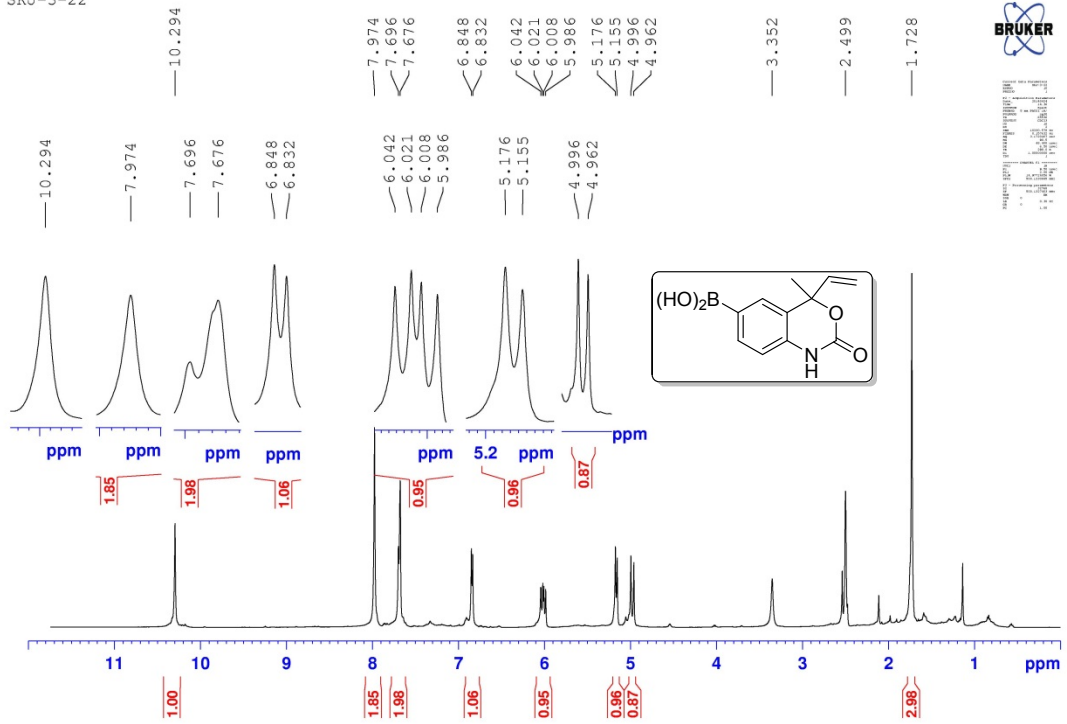
1. P. Zhang, P. Zhang, E. A. Terefenko, A. Fensome, J. Wrobel, R. Winneker, S. Lundeen, K. B. Marschke, Z. Zhang, *J. Med. Chem.* 2002, **45**, 4379.

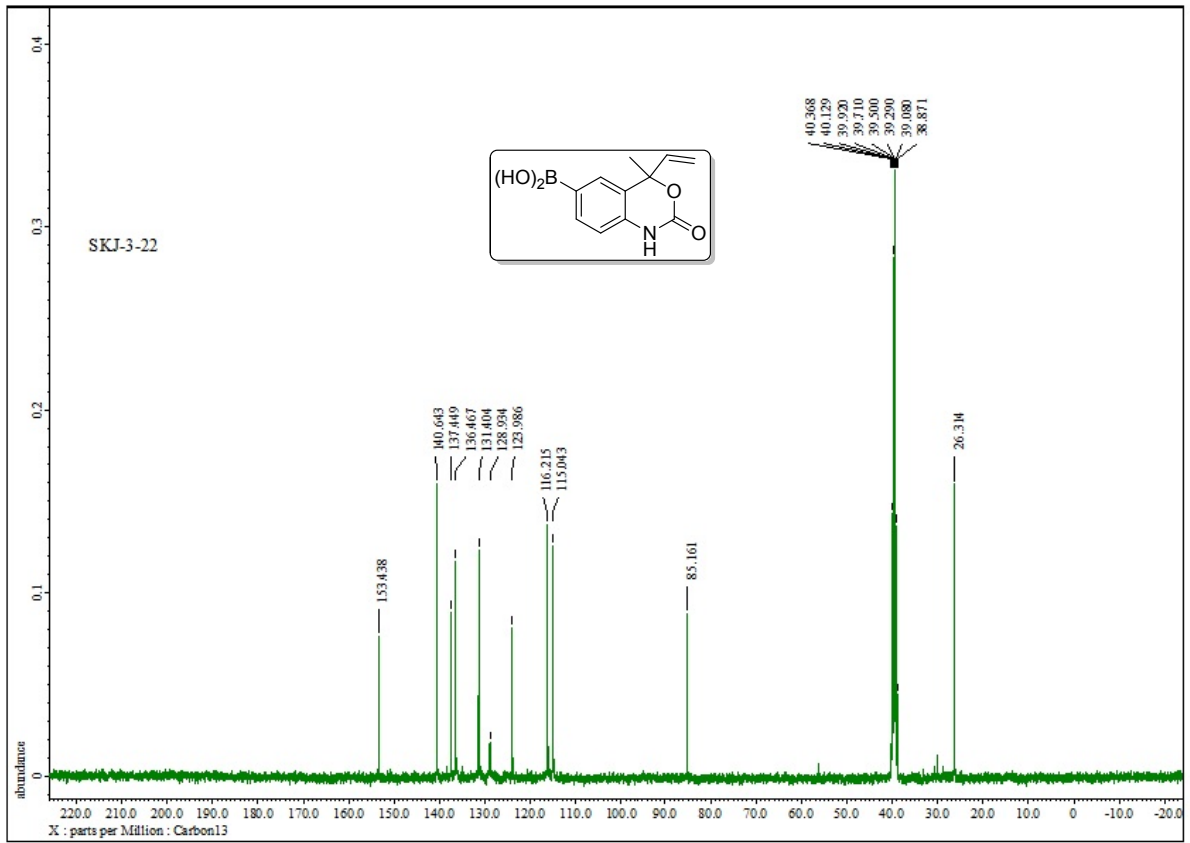
¹H and ¹³C Spectra



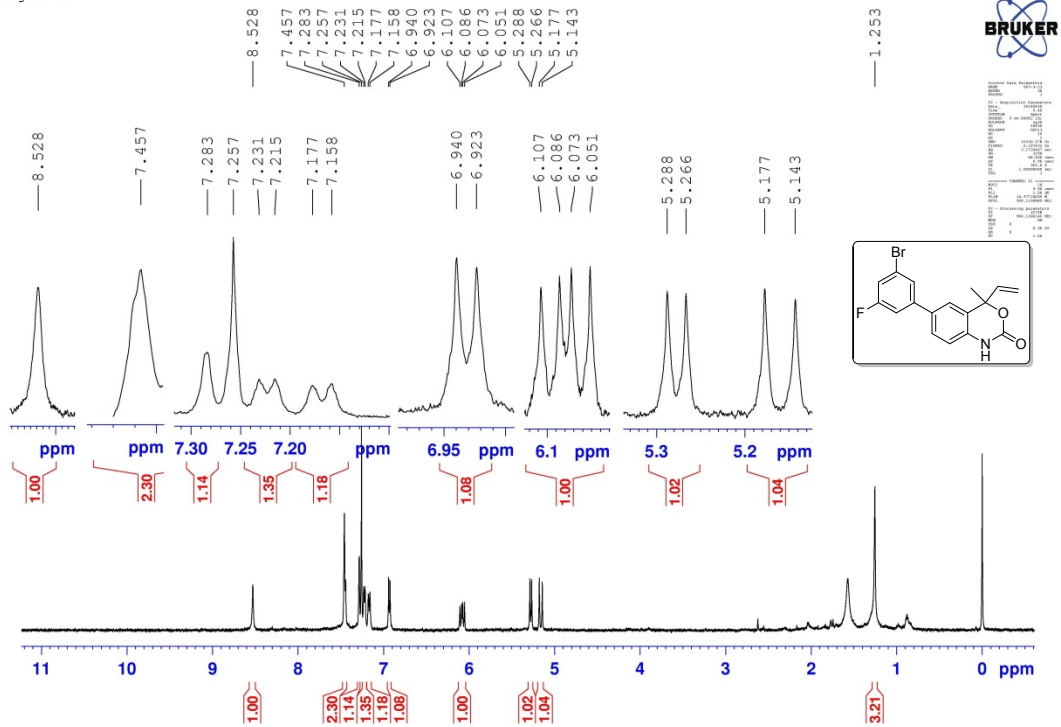


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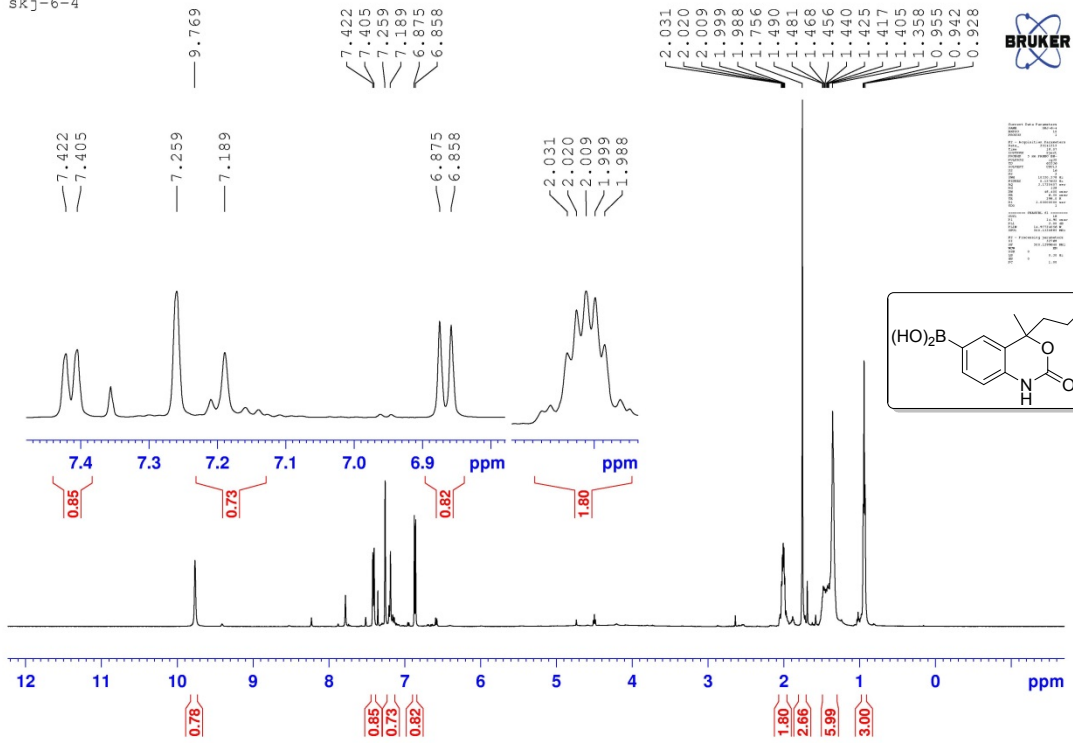




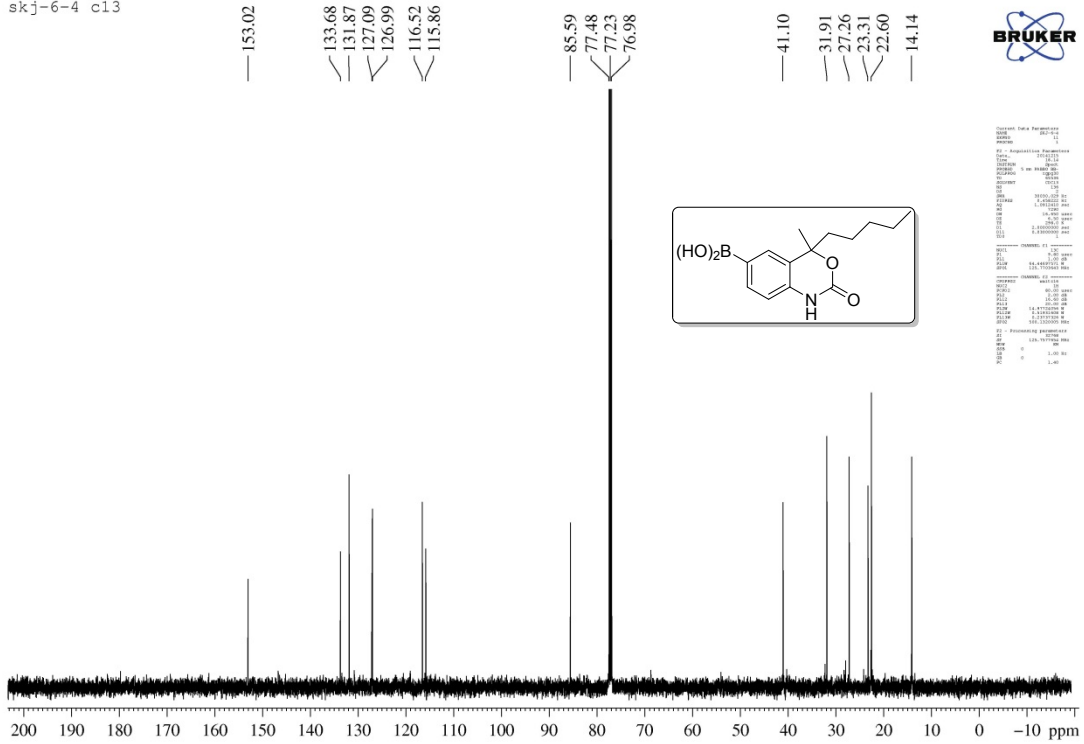
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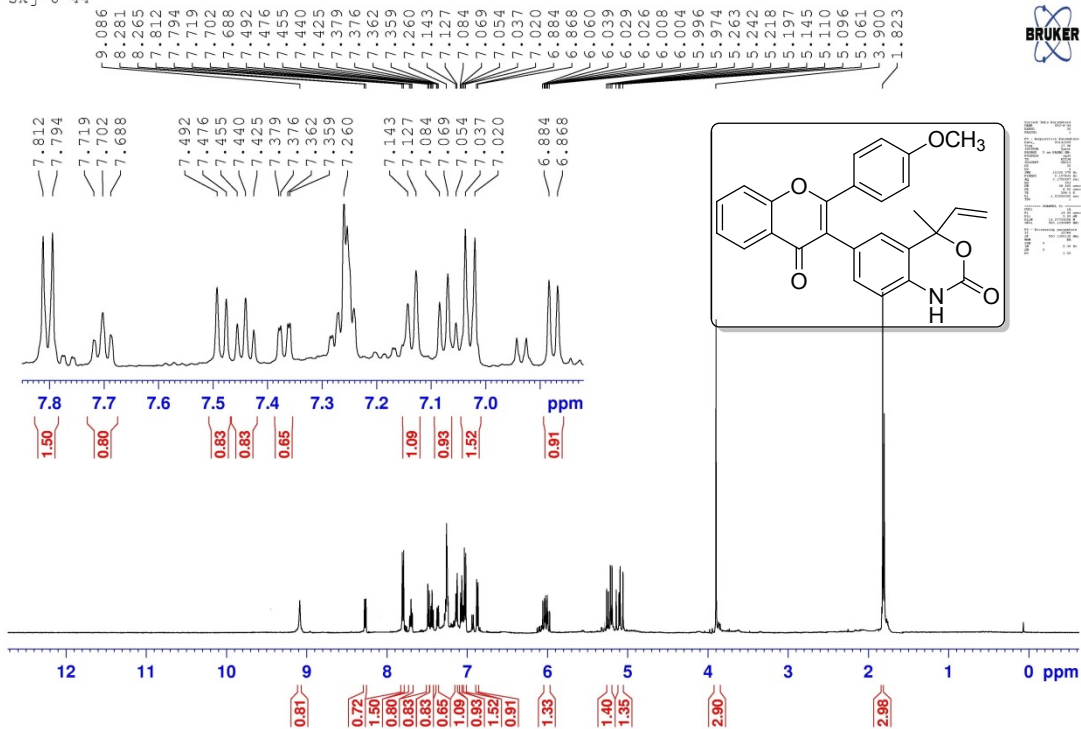
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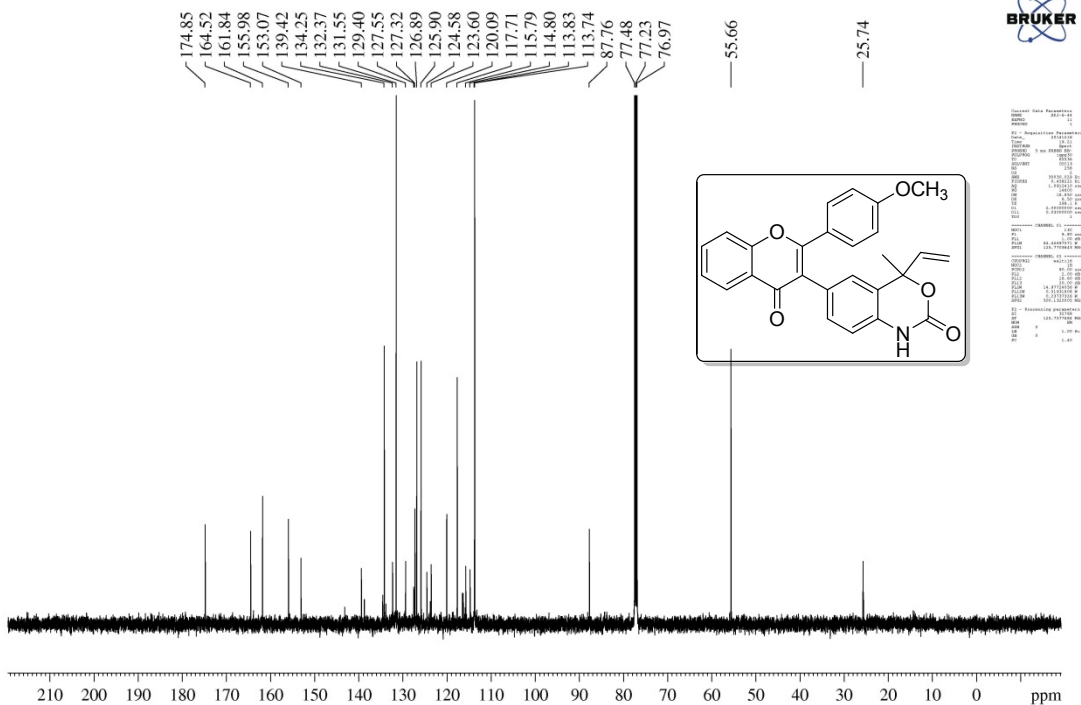
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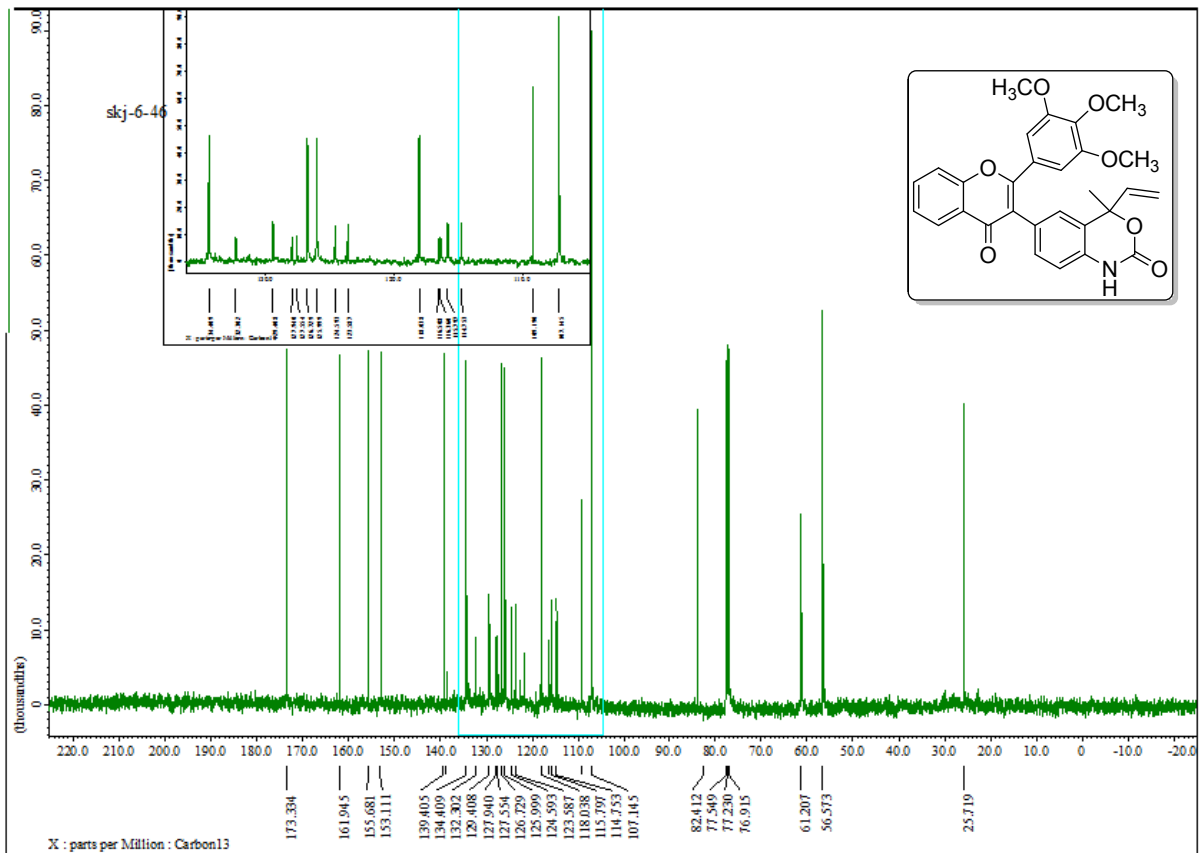
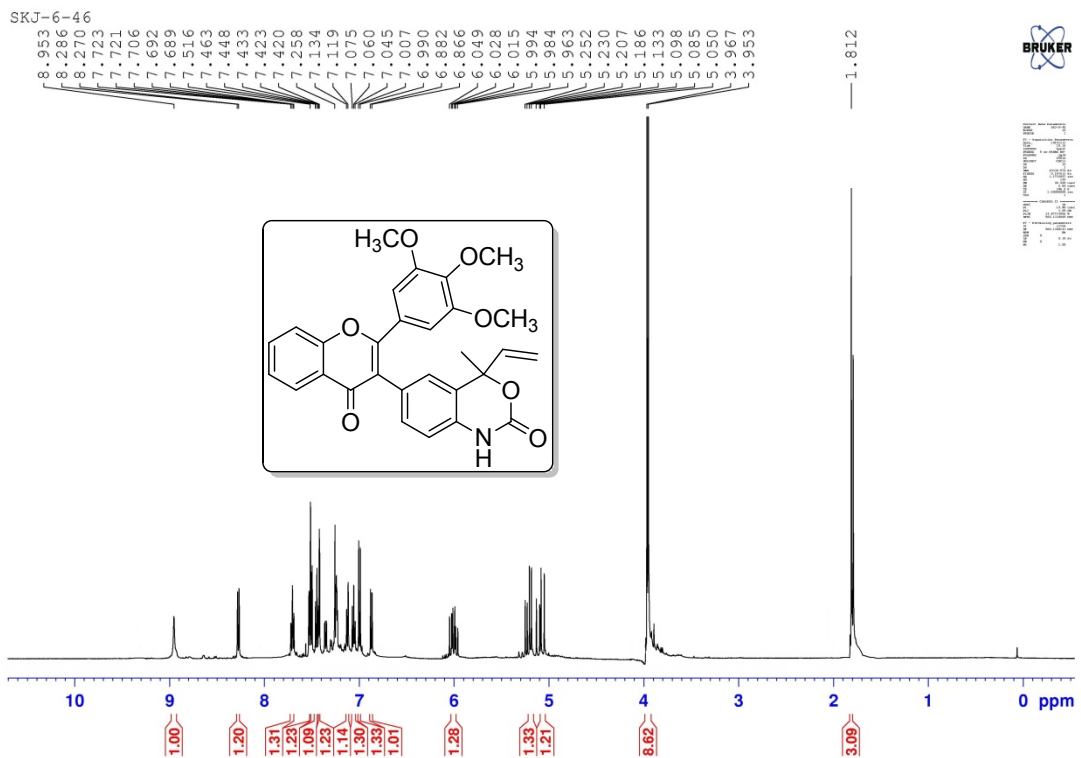


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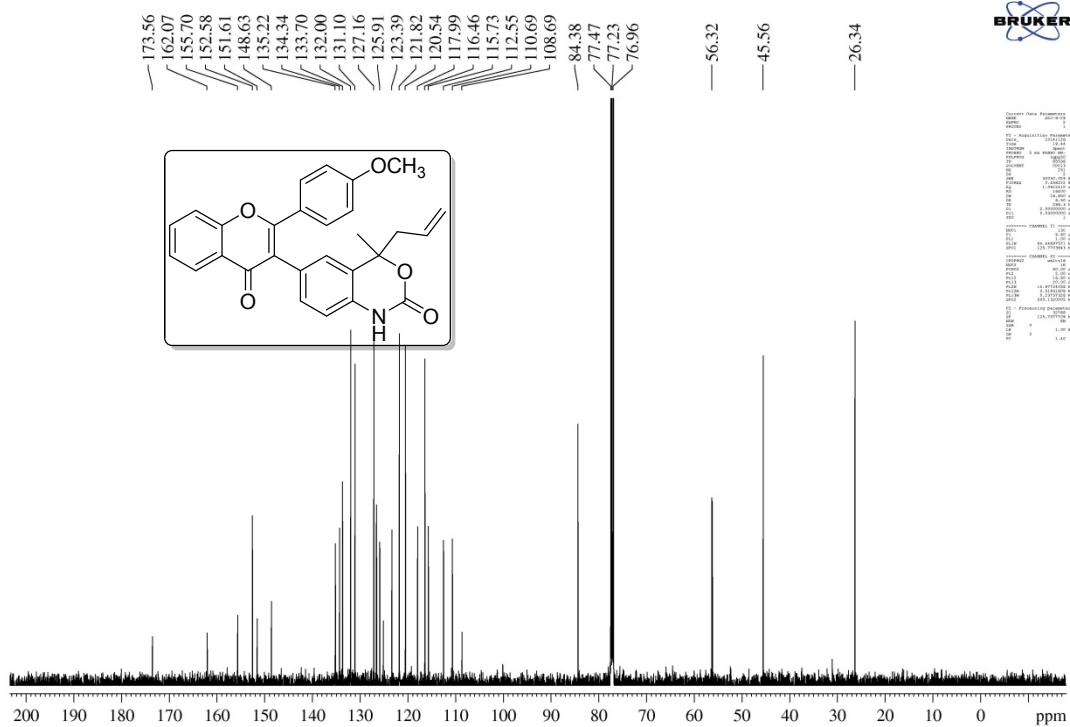
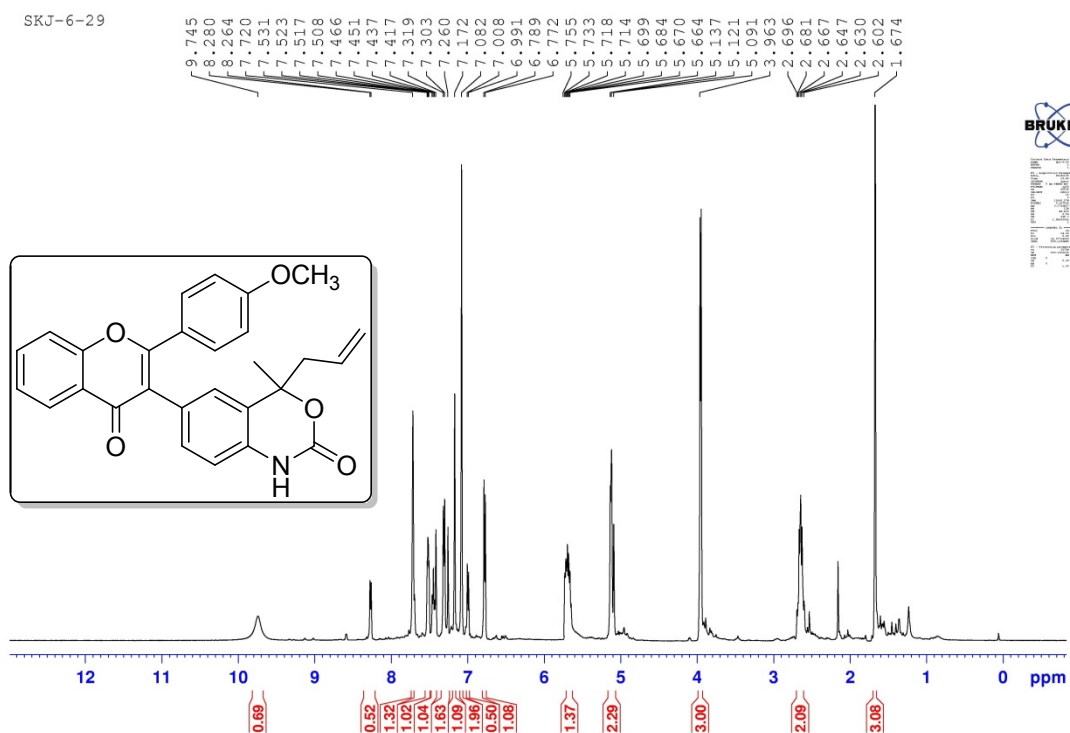


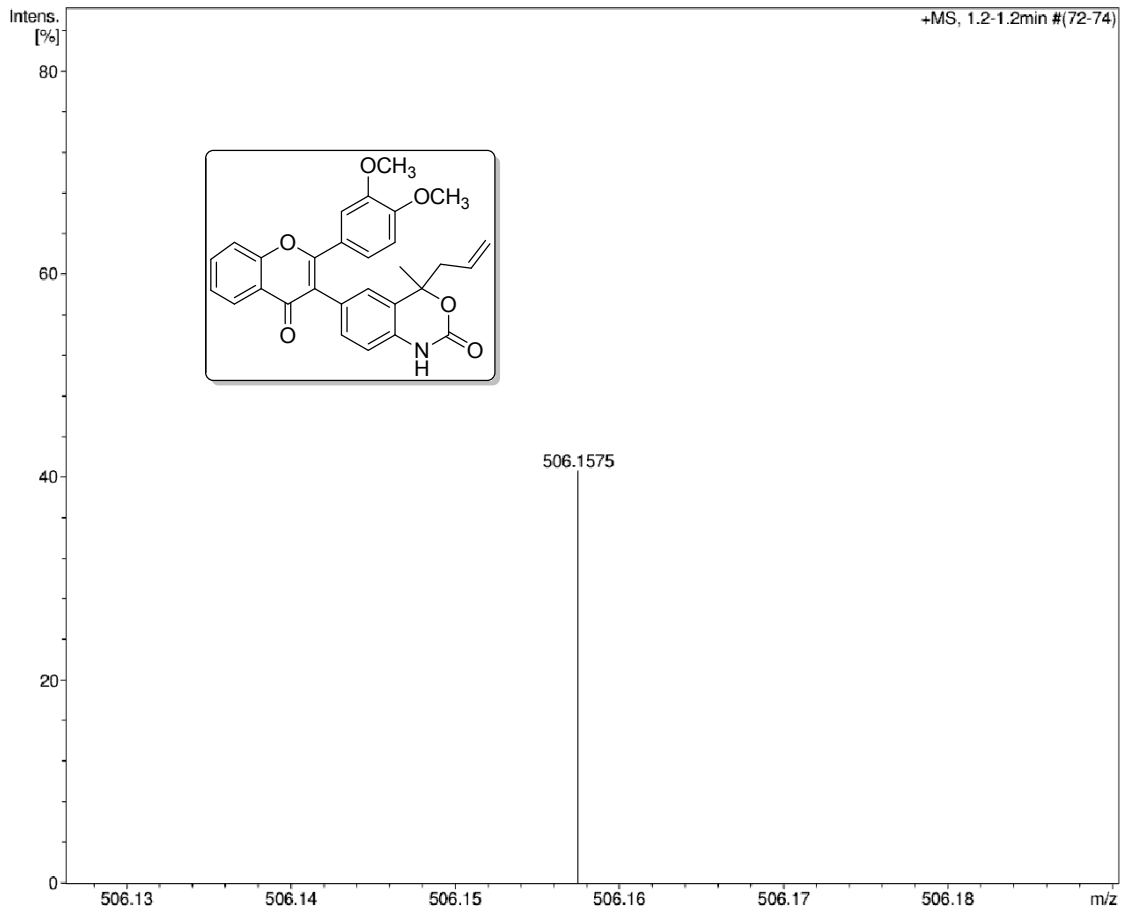
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SKJ-6-29



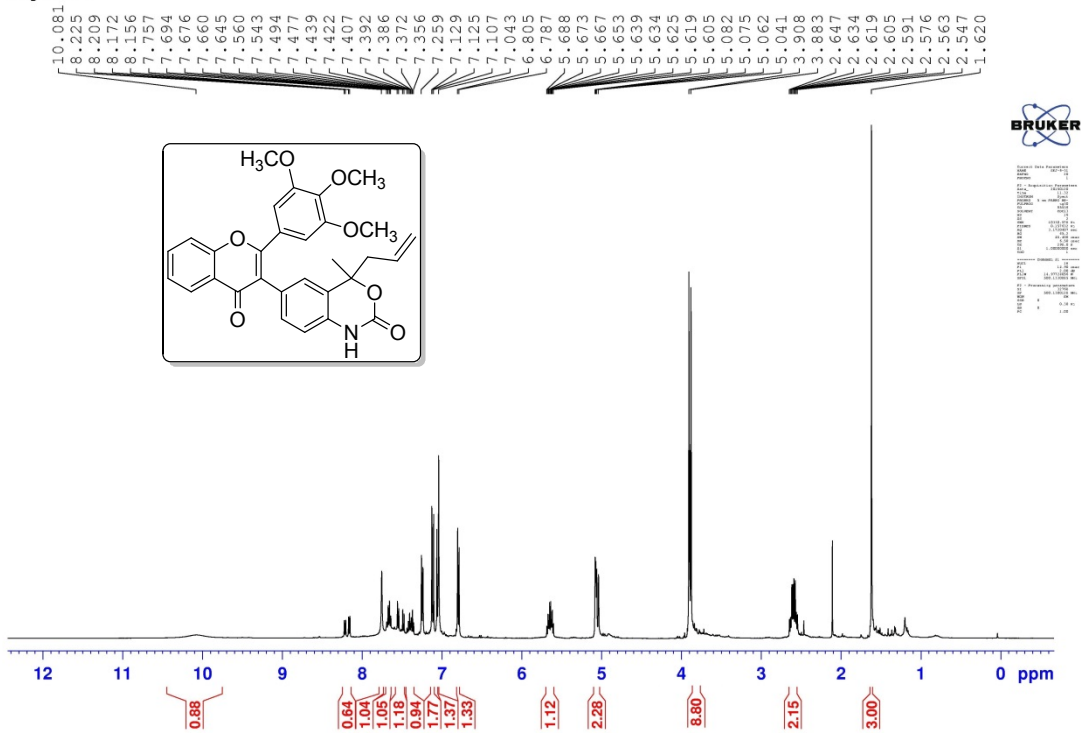


Bruker Compass DataAnalysis 4.0

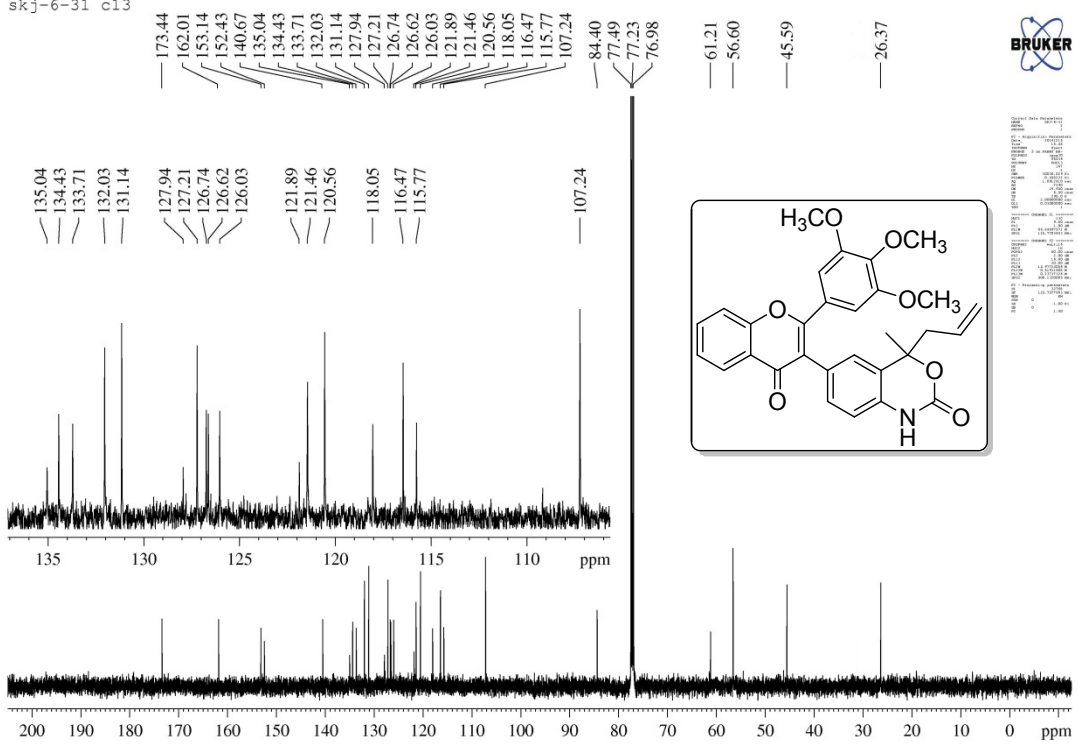
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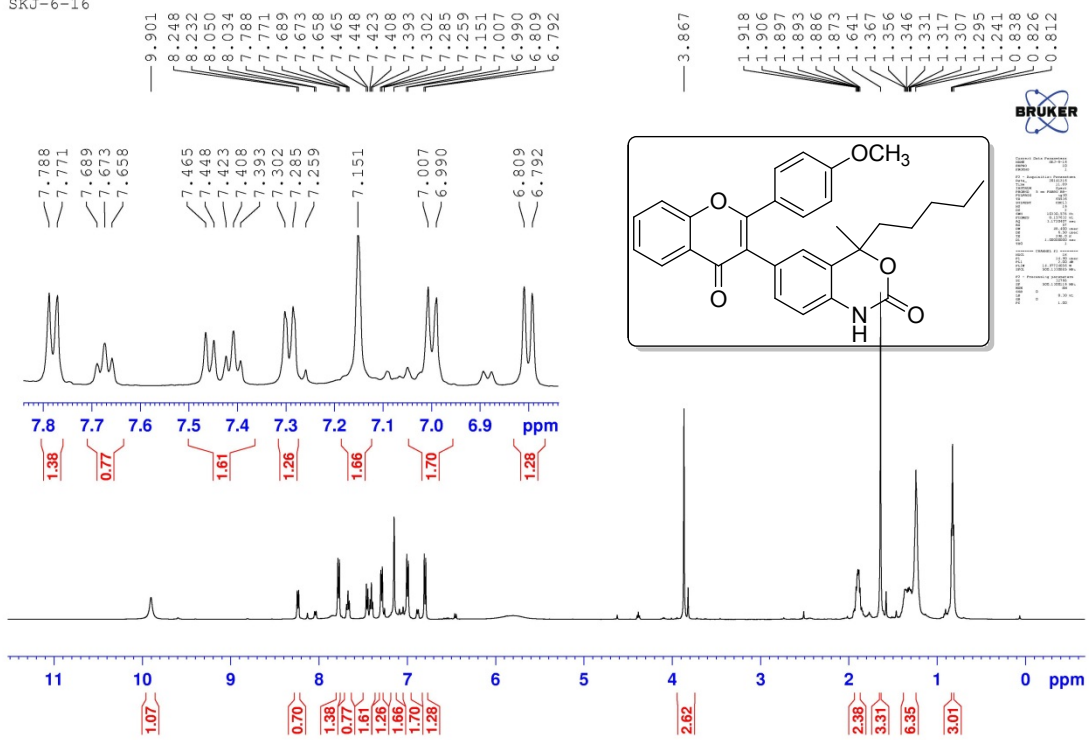
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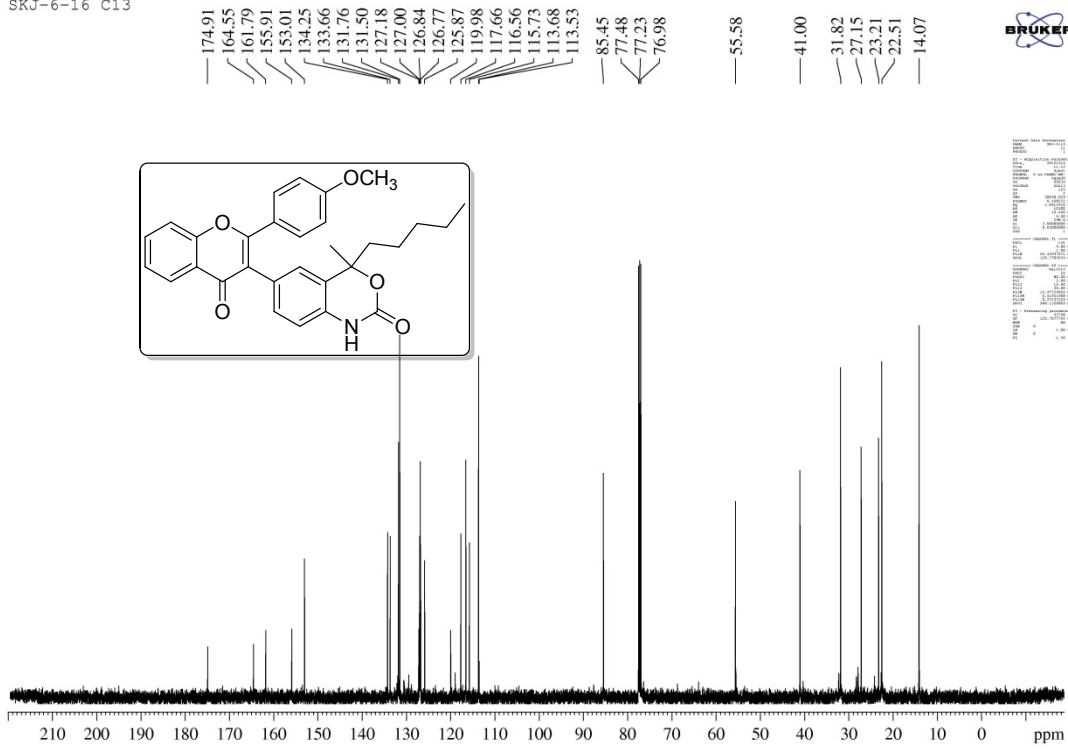
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SKJ-6-16

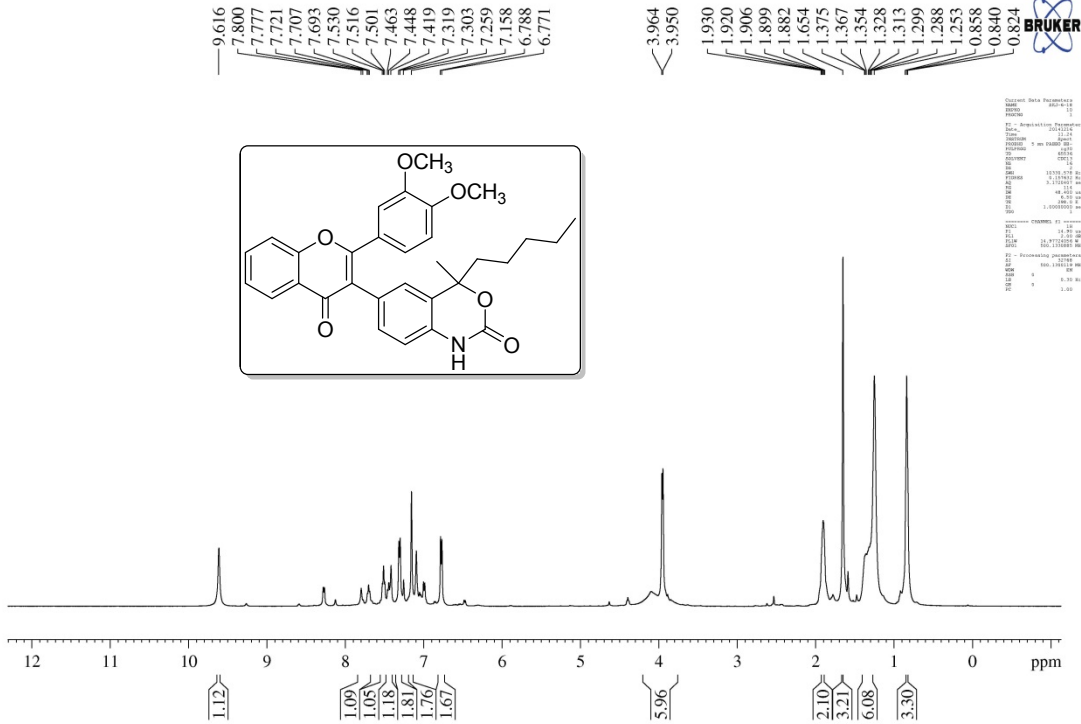


SKJ-6-16 C13



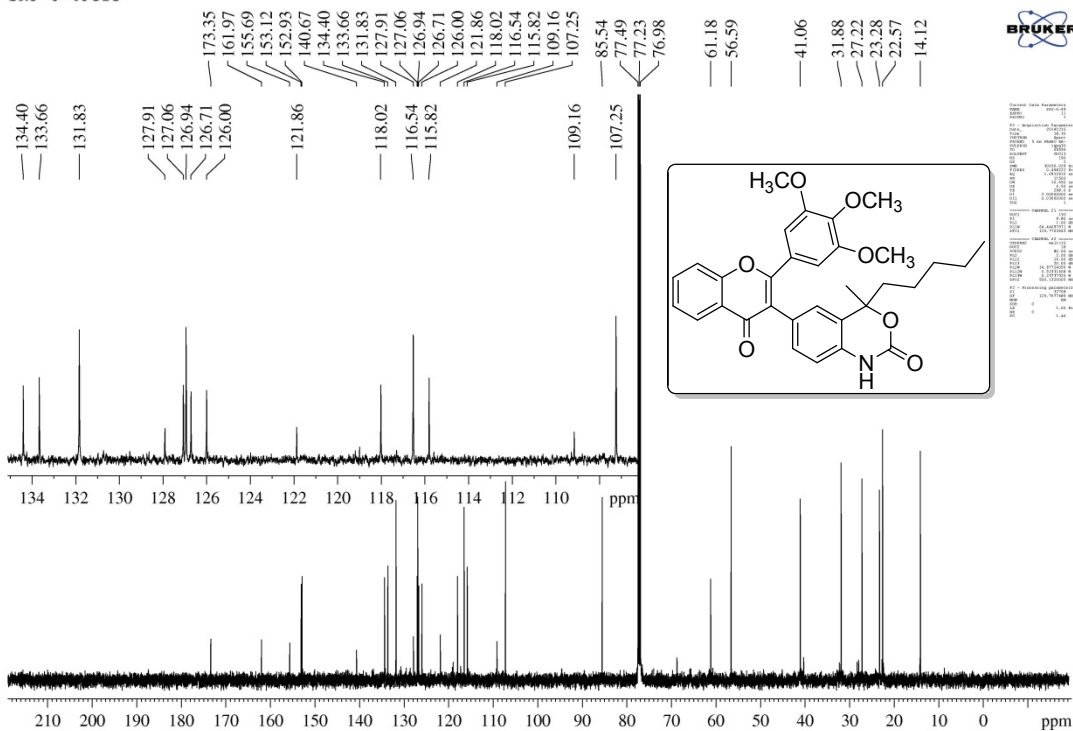
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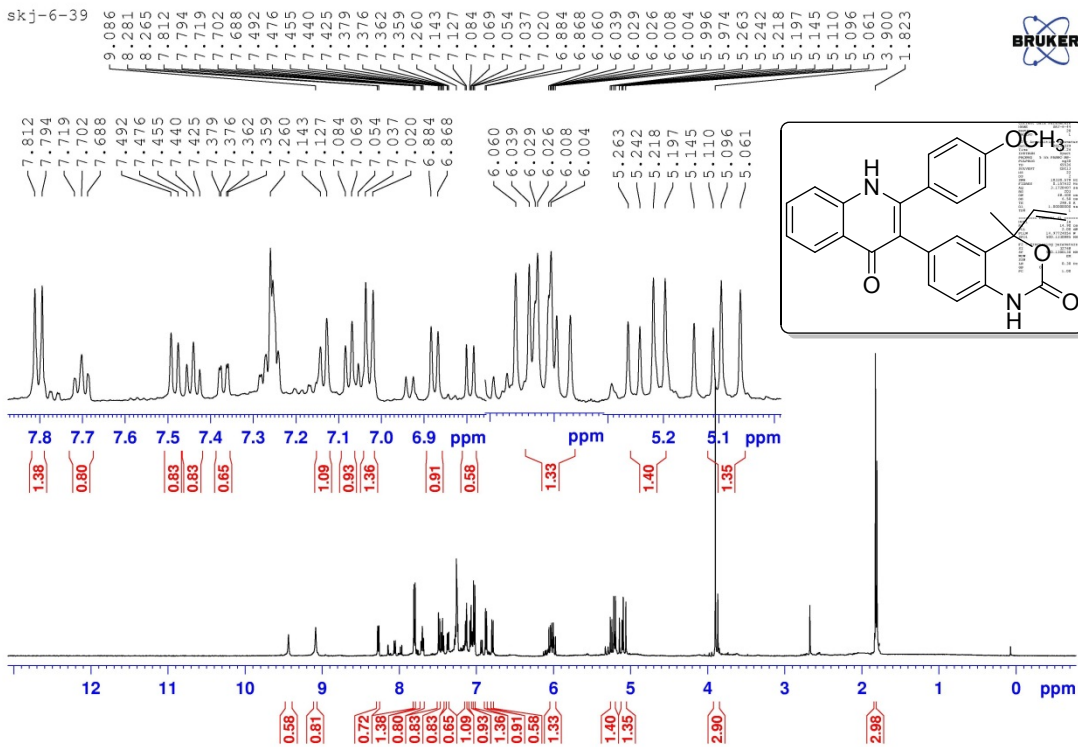


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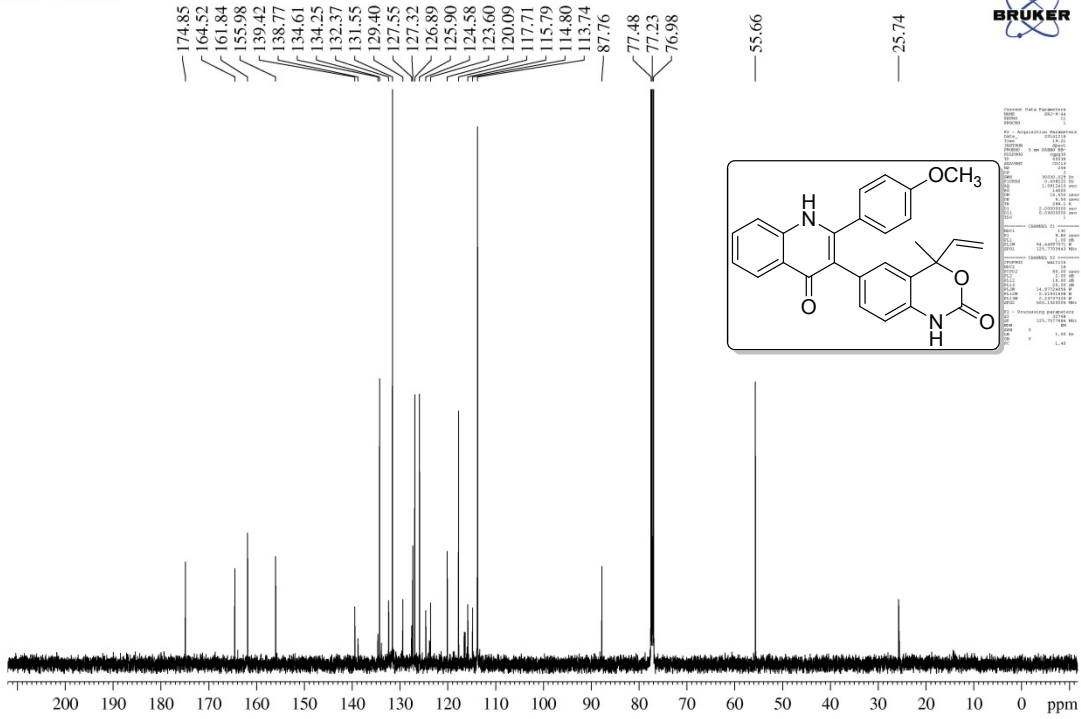
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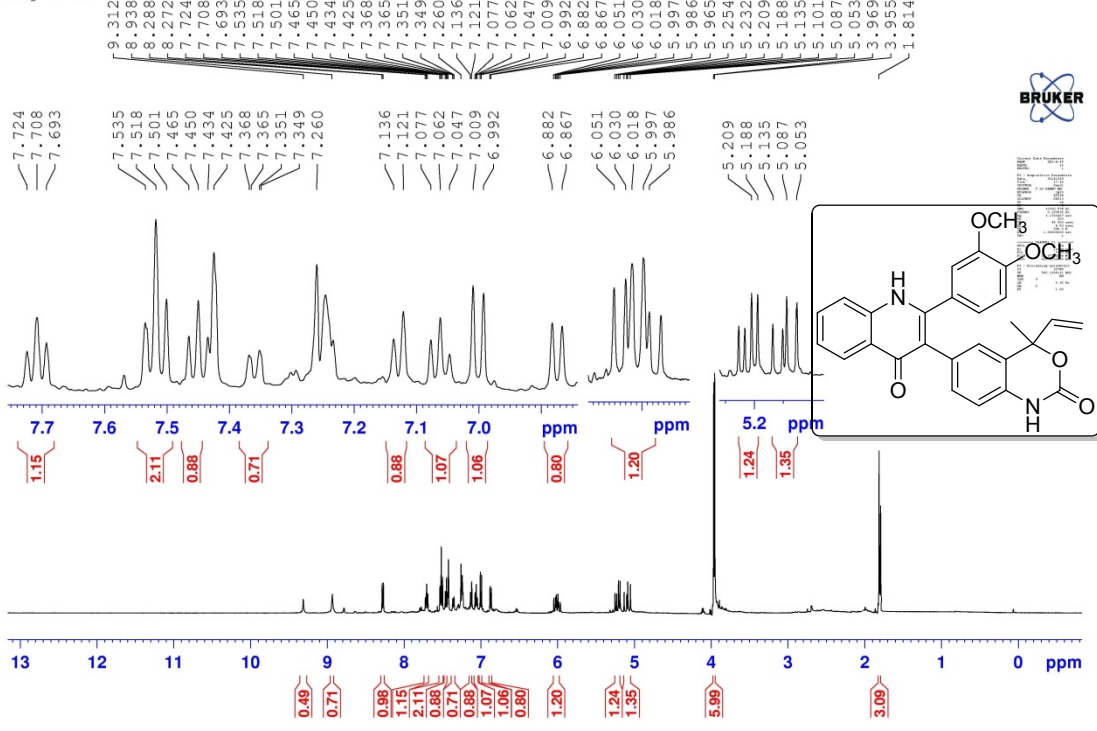
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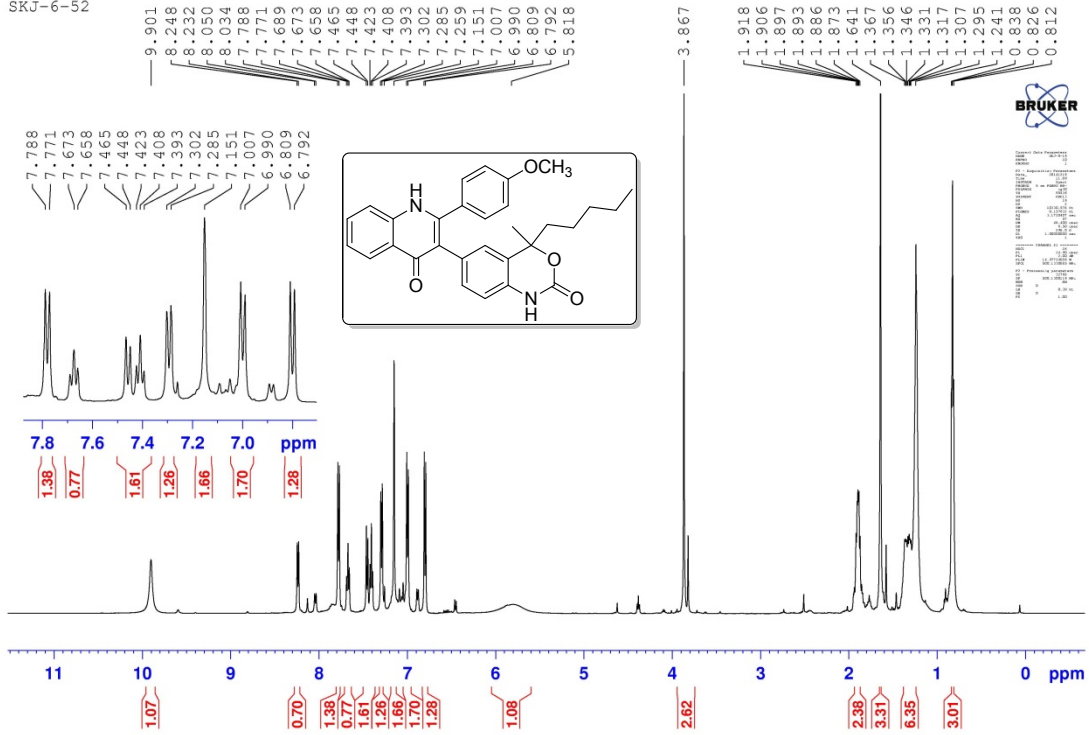
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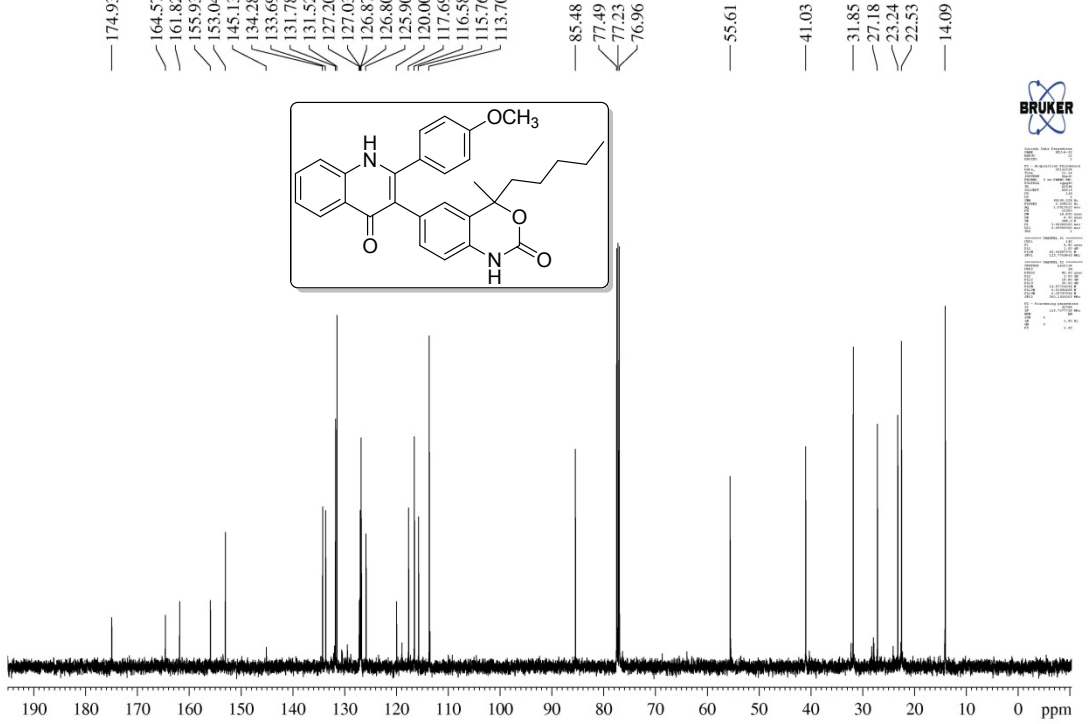
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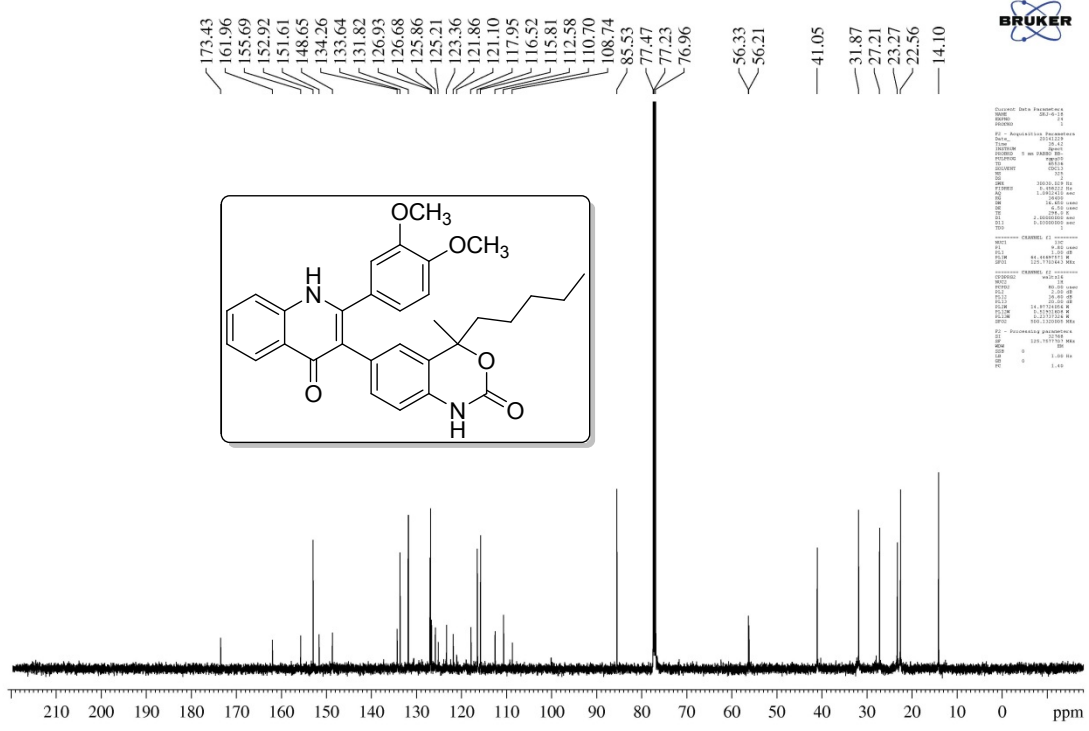
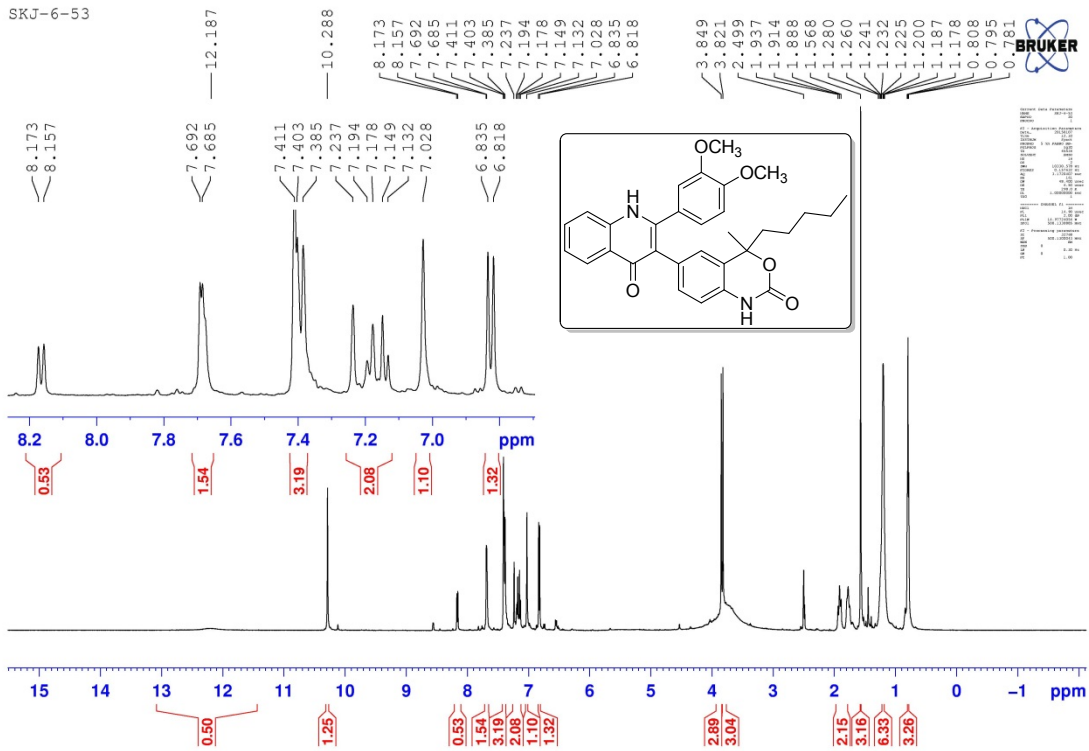
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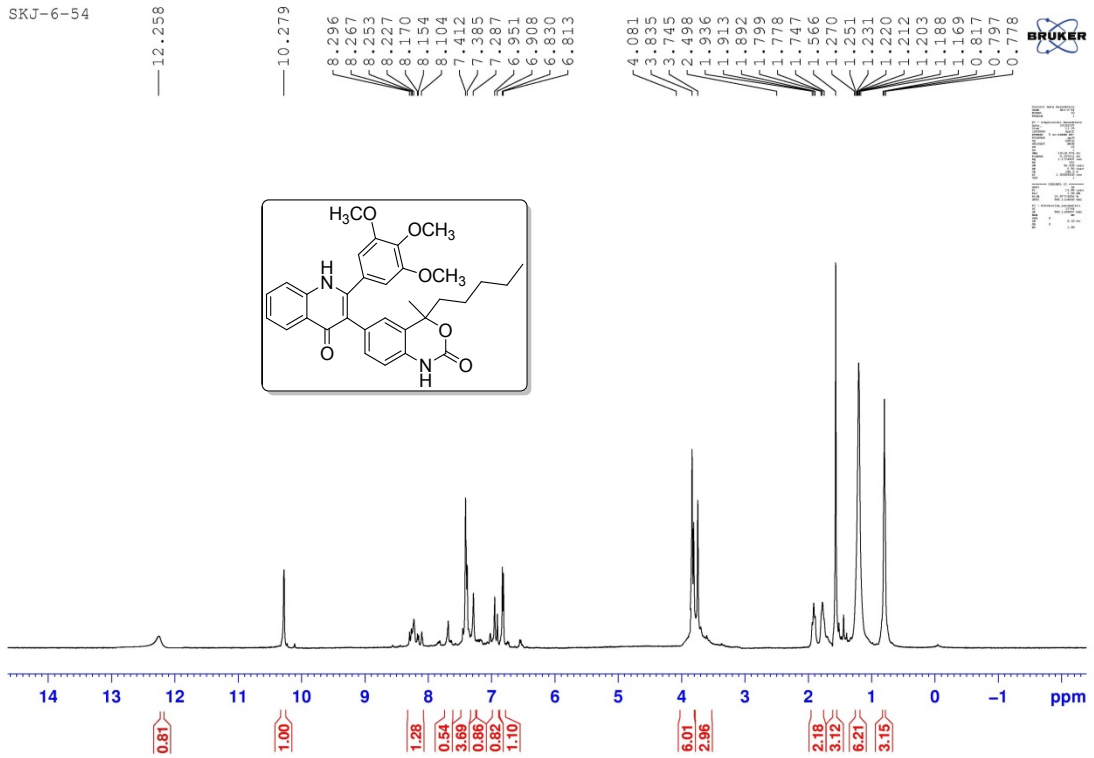
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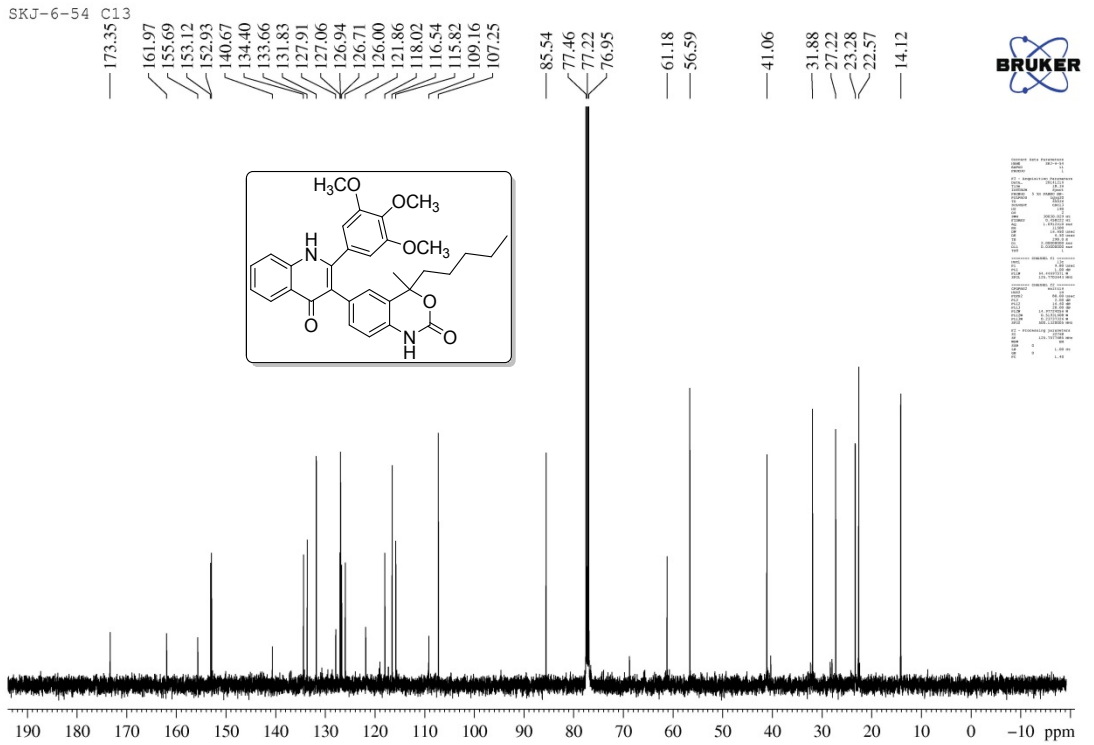
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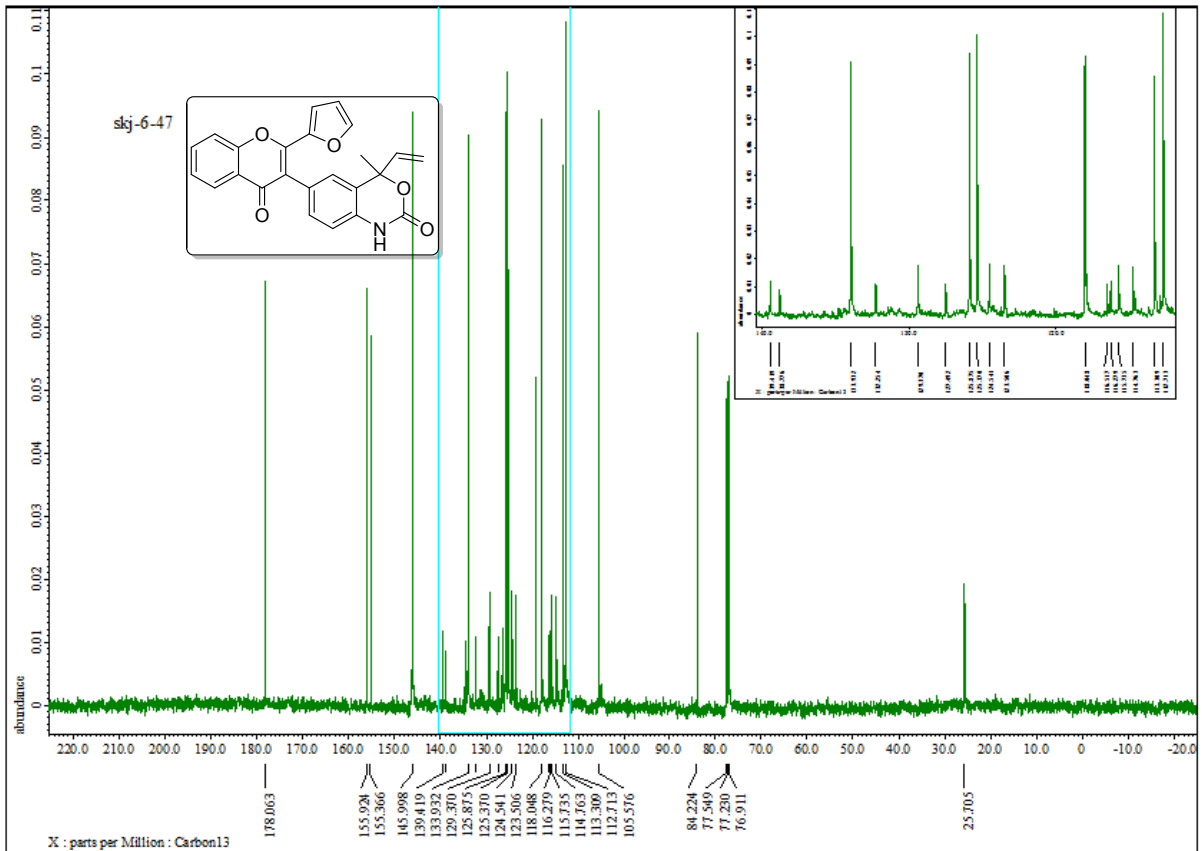
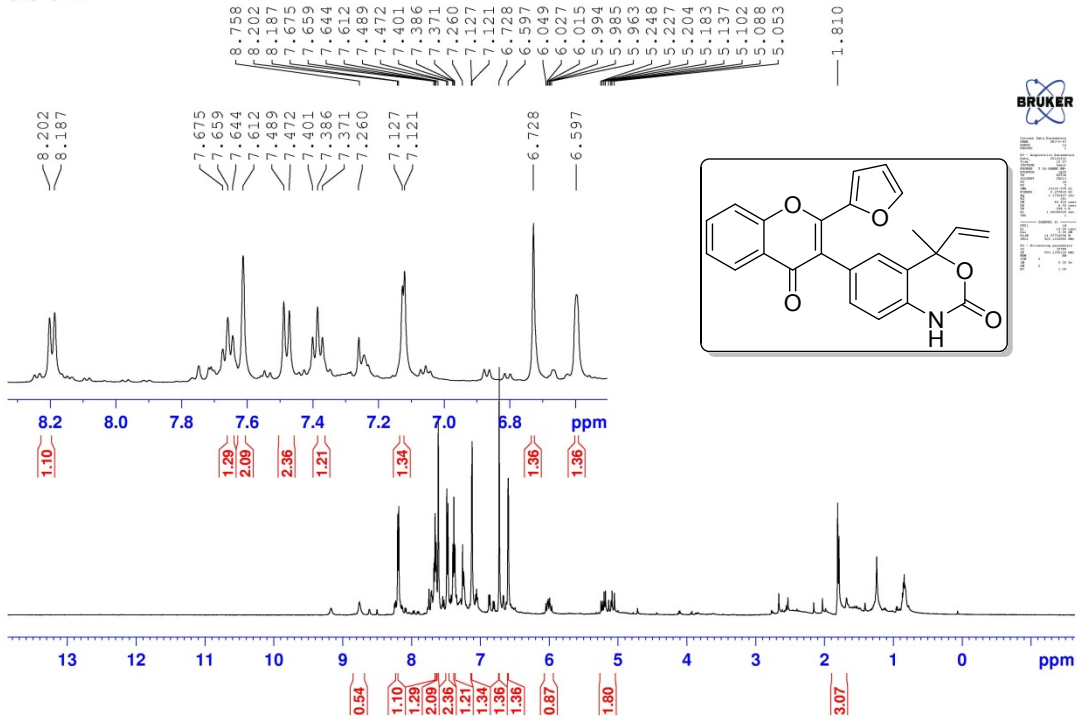
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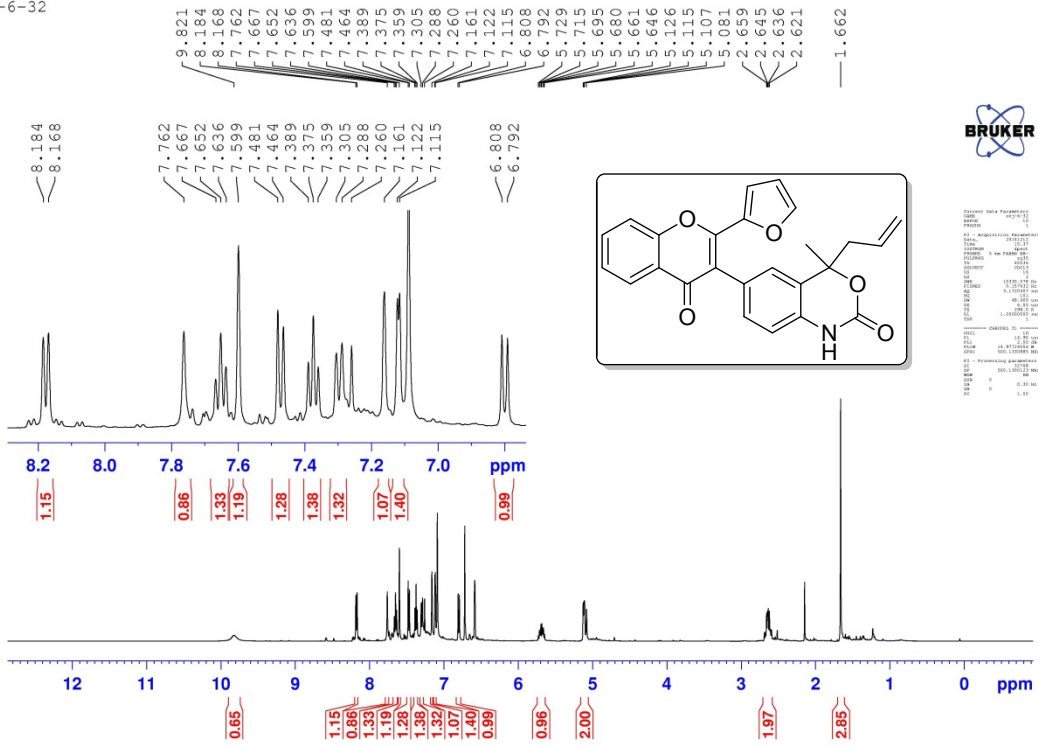
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SKJ-6-47

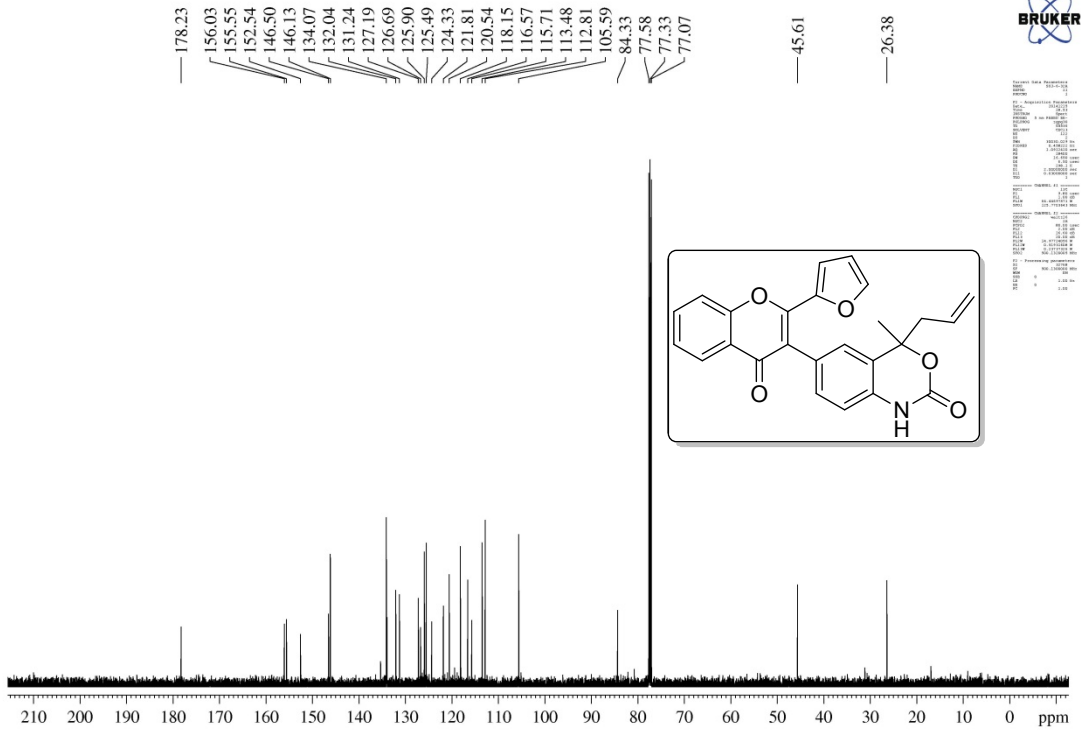


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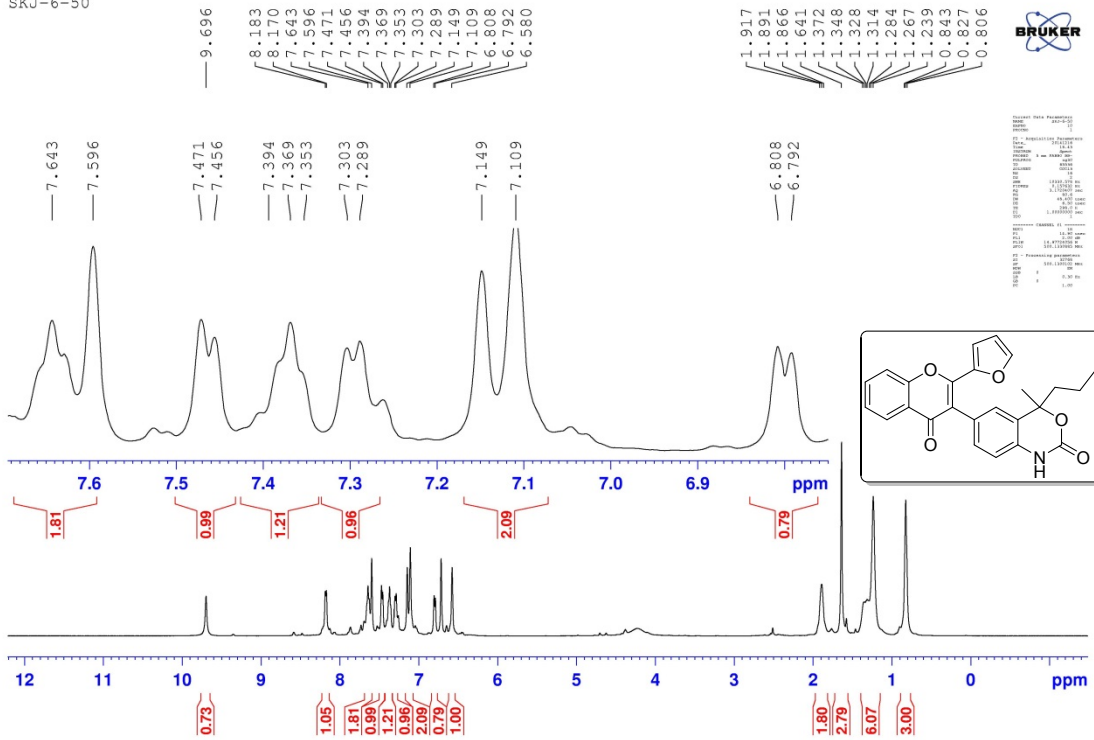
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SKJ-6-32A

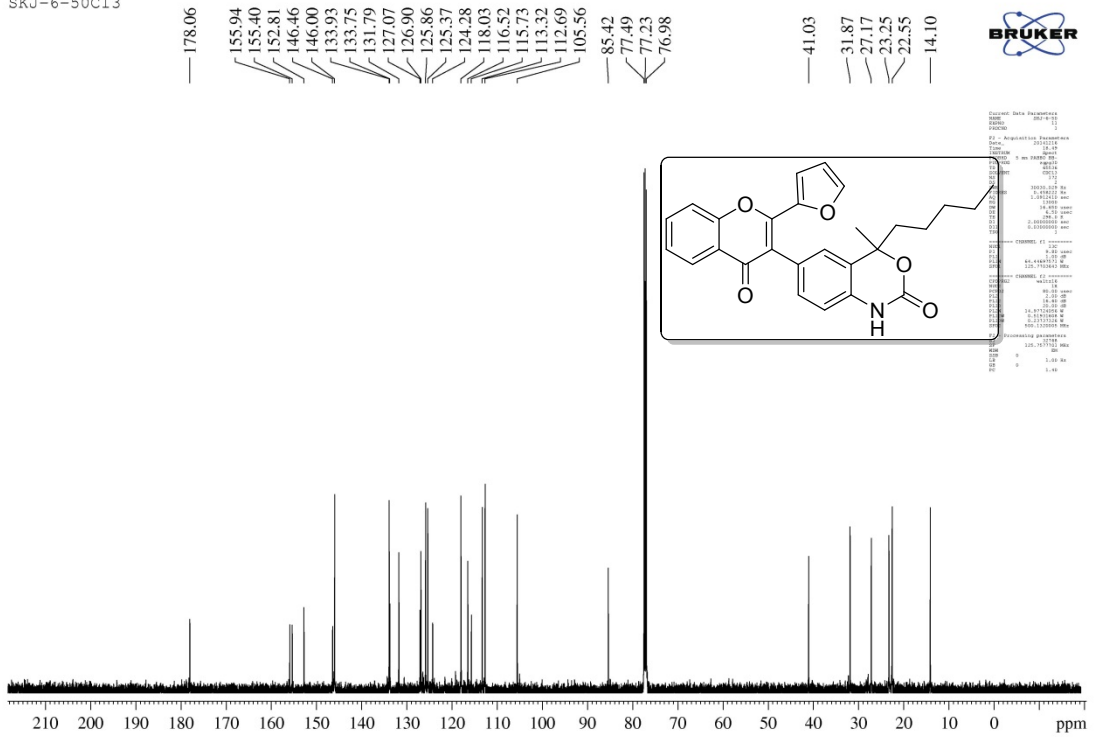


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SKJ-6-50



SKJ-6-50C13



SKJ-6-42

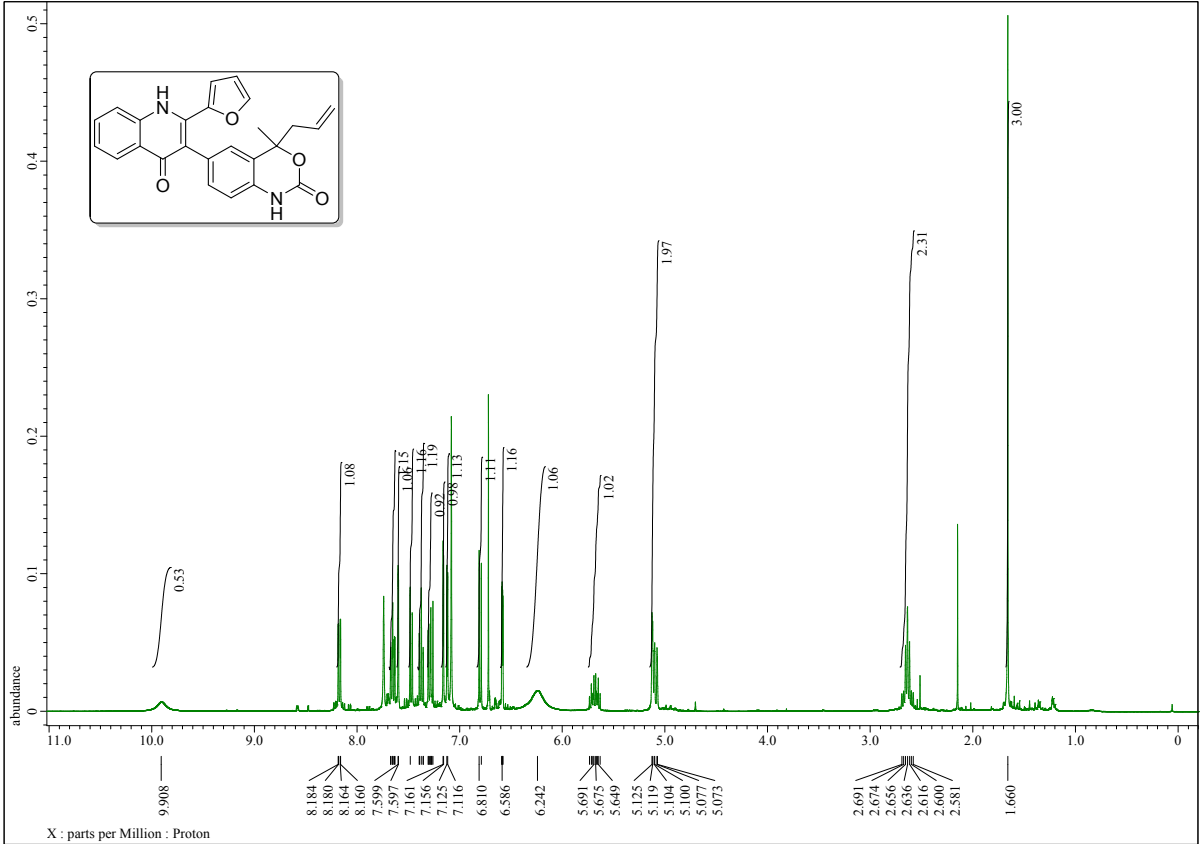
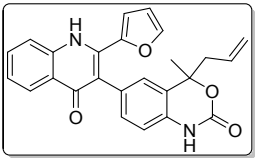
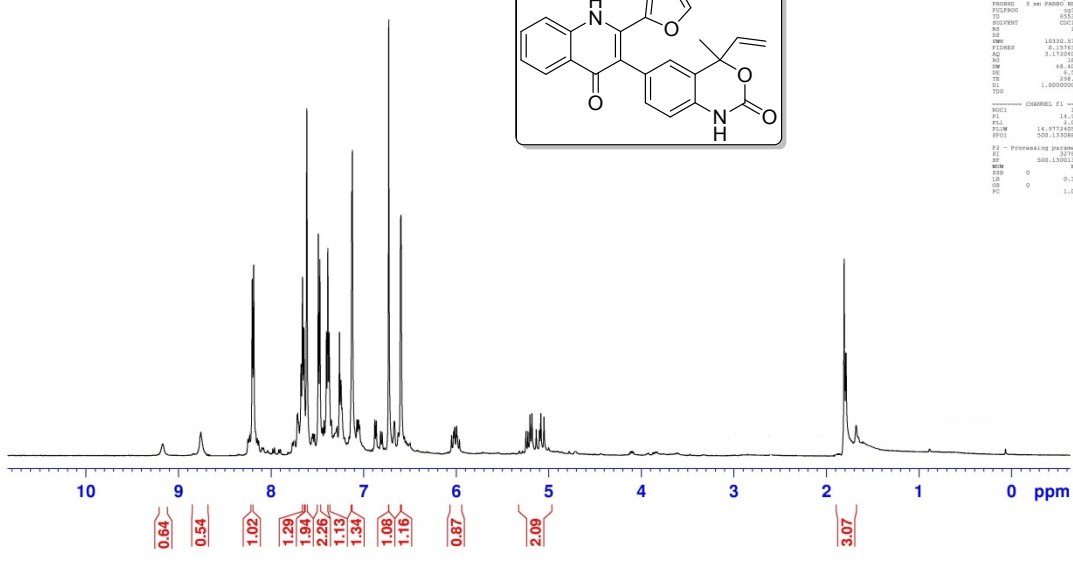
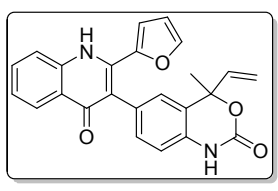
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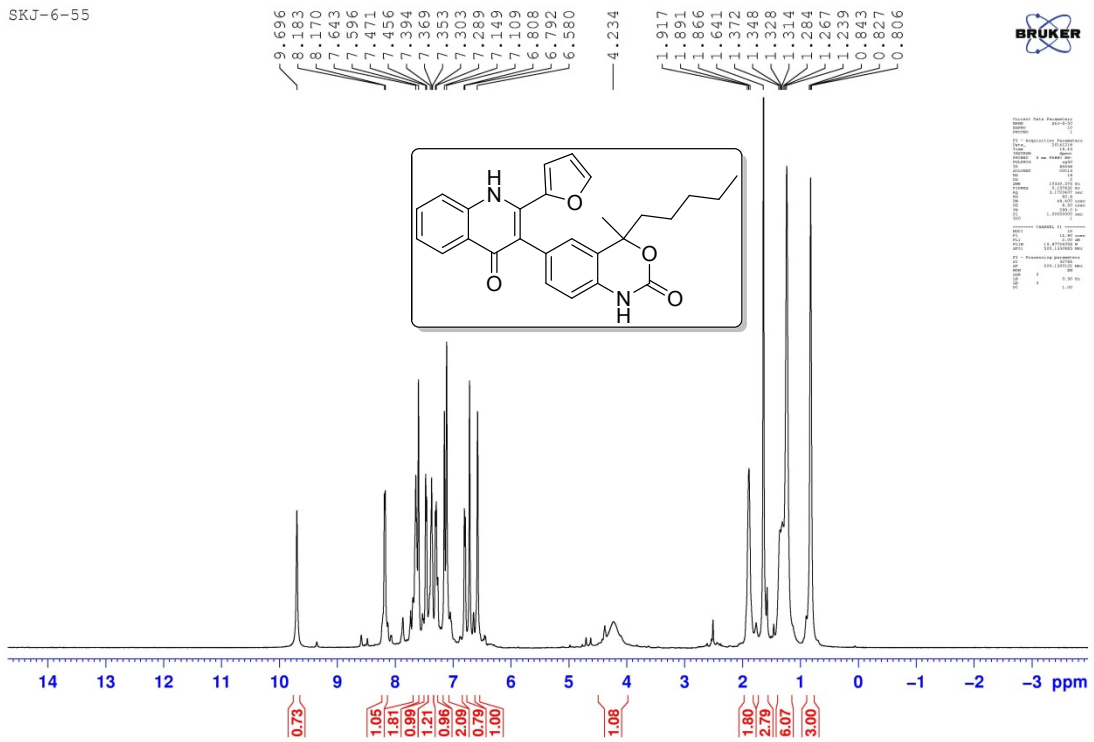


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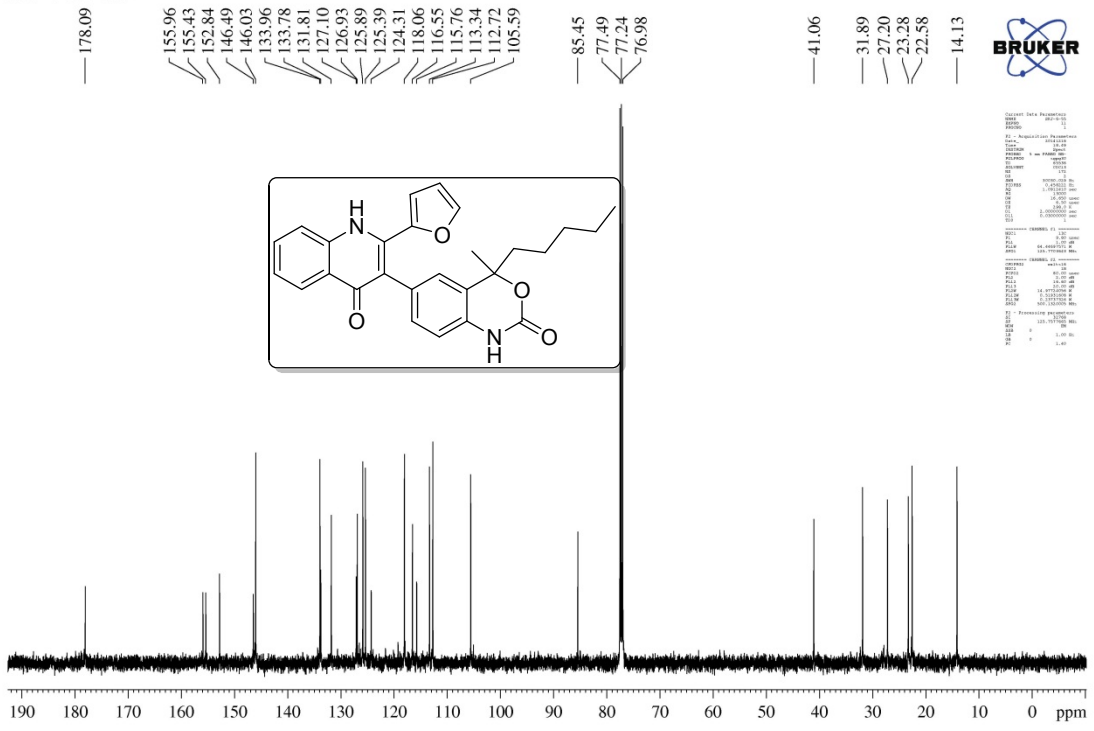
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SFO2:
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FIDRES: 0.131410 Hz
AQ: 3.117047 Hz
RG: 48.433 Hz
SF: 225.000 MHz
WDW: EM
SSB: 0
LB: 2.00 Hz
GB: 0
PC: 1.0000000 sec
TD: 65536
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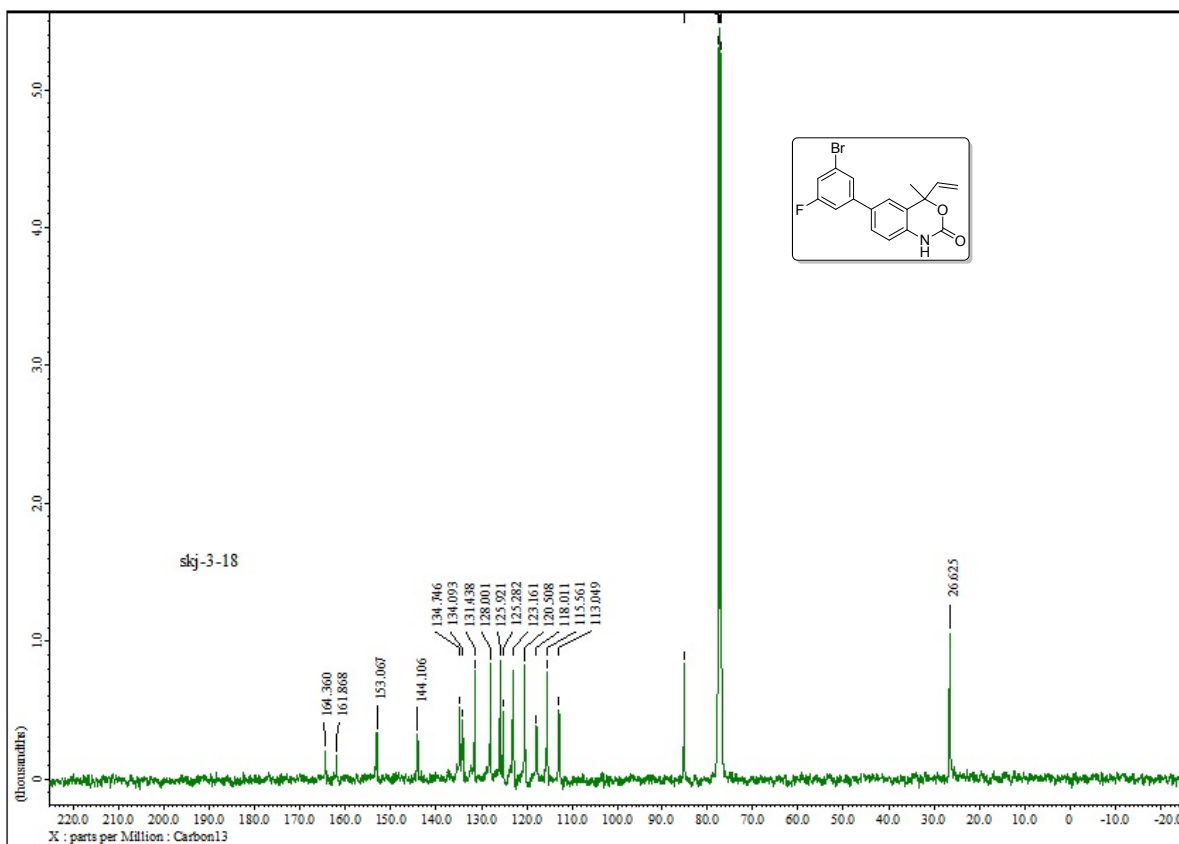
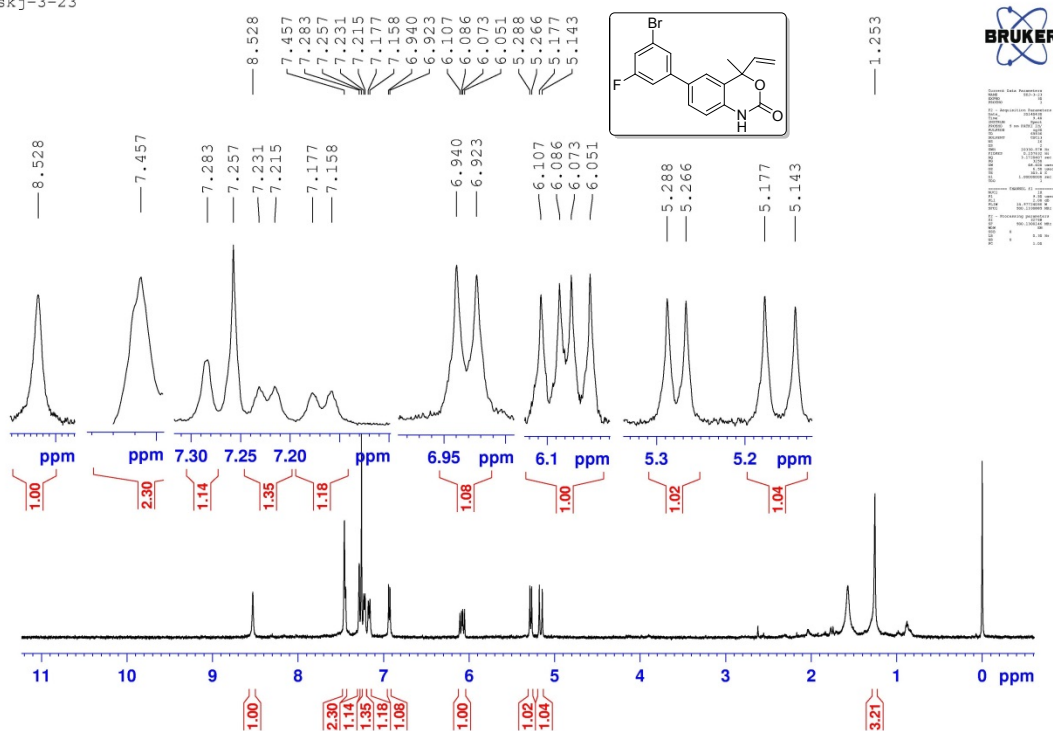
SKJ-6-55



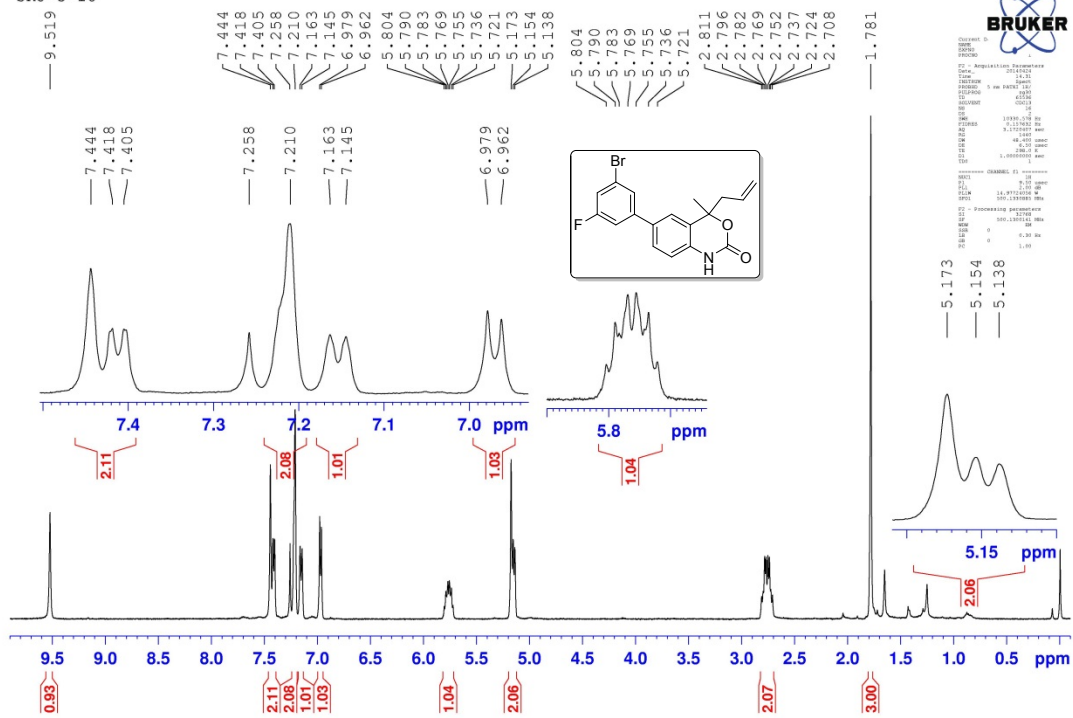
SKJ-6-55 C13



skj-3-23



SKJ-3-18



BRUKER

CHANNEL F1
 Name: SKJ-3-18
 Date: 11/11/2011
 Time: 14:25
 Program: zgpg30
 Processor: zgpg30
 Acquisition Parameters
 Date_ : 11/11/2011
 Time_ : 14:25
 Name_ : SKJ-3-18
 ExpNO : 1
 F2 - Acquisition Parameters
 Date_ : 11/11/2011
 Time_ : 14:25
 Name_ : SKJ-3-18
 ExpNO : 1
 F2 - Processing parameters
 Date_ : 11/11/2011
 Time_ : 14:25
 Name_ : SKJ-3-18
 ExpNO : 1
 F2 - Reference parameters
 Date_ : 11/11/2011
 Time_ : 14:25
 Name_ : SKJ-3-18
 ExpNO : 1

