Supporting information

**T3P® Mediated Domino C(sp2)-H Sulfenylation/Annulation of Enaminones and Methylsulfinyls for the synthesis of Chromone Thioethers Derivatives**

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General Methods

Dry solvents were purchased from chemical suppliers and used without further purification. Analytical thin-layer chromatography (TLC) was performed on commercially available Merck TLC Silica gel 60 F254. Silica gel column chromatography was performed on silica gel (spherical 100-200 µm). IR spectra were recorded on Perkin-Elmer FT/IR-4000 using ATR. ¹H-NMR spectra were recorded on Varian-400 (400 MHz) spectrometer. Chemical shifts of ¹H-NMR spectra were reported relative to tetra methyl silane (¹³C NMR spectra were recorded on Varian-400 (100 MHz) spectrometer. Chemical shifts of ¹³C NMR spectra were reported to relative to CDCl₃ (77.16) and DMSO-d₆ (39.5). Splitting patterns were reported as s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; br, broad. Melting points (m.p.) were measured by Büchi 510 melting point apparatus and uncorrected.

General procedure for the synthesis of Enaminone:

A mixture of 2-hydroxy acetophenone 1 (200 mg, 1.47 mmol) and DMF-DMA (0.96 ml, 1.47 mmol) were introduced into a 2–5 mL initiator reaction vial. The mixture was irradiated for 10 minutes at 100 °C under MW. The reaction mixture was cooled to room temperature then wash with diethyl ether and filtered to get yellow solid.

General procedure for the synthesis 3-(methylthio)-4H-chromen-4-one (3a):

Enaminone 1a (100 mg, 0.523 mmol) was added to a dried sealed tube containing DMSO (0.297 mL, 4.18 mmol) in THF (1 mL) followed by the addition of T3P® (343 mg, 1.04 mmol) at 10 °C. After 15 min, the reaction mixture was heated to 100 °C for 12 h. The reaction progress was monitored by TLC. Then the reaction mixture was cooled to room temperature, diluted with ethyl acetate, washed with water and brine, dried with anhydrous Na₂SO₄, and concentrated under vacuum. The residue was purified by flash chromatography (silica gel, hexane/EtOAc) to give desired product 3a (91%) as a colourless solid. The same procedure was used to prepare compounds 3b–3n.

General procedure for the synthesis 3-(phenylthio)-4H-chromen-4-one (4a):

Enaminone 1a (100 mg, 0.523 mmol) was added to a dried sealed tube containing DMAC (0.3 mL), followed by the addition of T3P® (343 mg, 1.04 mmol.) and methyl phenyl sulfoxide (145 mg, 1.04 mmol) at 0 °C. After 15 min, the reaction mixture was heated to 90 °C for 10 h. The reaction progress was monitored by TLC. Then the reaction mixture was cooled to room temperature, diluted with ethyl acetate, washed with water and brine,
dried with anhydrous NaSO₄, and concentrated under vacuum. The crude residue was purified by flash chromatography (silica gel, hexane /EtOAc) to give desired product 4a (81 %) as a colourless solid. The same procedure was used to prepare compounds 4b–4p

3-(methylthio)-4H-chromen-4-one:

![3a](image)

Off white solid; Yield (91%); m.p. 104-108 °C; IR (KBr, cm⁻¹): 3066, 1625, 1559, 1463, 1359, 1106, 1078, 891, 755; ¹H-NMR (400 MHz, CDCl₃): 8.27-8.25 (d, J= 7.6 Hz, 1H), 8.06 (s, 1H), 7.68 (s, 1H), 7.45-7.43 (m, 2H), 2.41 (s, 3H); ¹³C NMR (400 MHz,CDCl₃): 175.58, 156.23, 153.86, 133.74, 126.06, 125.42, 123.12, 121.80, 118.03, 16.24; MS (EI): m/z 193 (M+1, 100); HRMS: (ESI): Calcd for C₁₀H₈O₇S[M+H⁺]:193.0323, Found: 193.0329.

6-bromo-3-(methylthio)-4H-chromen-4-one:

![3b](image)

Yellow solid; Yield (82%); m.p. 124-127 °C; IR (KBr, cm⁻¹): 1627, 1463, 1432, 1311, 1120, 1080, 815, 754; ¹H-NMR (500 MHz, CDCl₃): 8.37 (d, J = 2.4 Hz, 1H), 8.0 (s, 1H), 7.76-7.74 (m, 1H), 7.37 (d, J = 8.8 Hz, 1H), 2.40 (s, 3H); ¹³C NMR (400 MHz,CDCl₃): 174.32, 155.00, 153.67, 136.75, 128.63, 124.31, 122.28, 120.04, 118.80, 16.11; MS (EI): m/z 270 (M+1, 100); HRMS: (ESI): Calcd for C₁₀H₉BrO₂S[M+H⁺]:270.9430, Found: 270.9428

6-fluoro-3-(methylthio)-4H-chromen-4-one:

![3c](image)

Colourless liquid; Yield (80%); IR (KBr, cm⁻¹): 3079, 1628, 1558, 1479, 1370, 1102, 884, 814; ¹H-NMR (400 MHz, CDCl₃): 8.06 (s, 1H), 7.90-7.85 (m, 1H), 7.49-7.37 (m, 2H), 2.40 (s, 3H); ¹³C NMR (125 MHz, CDCl₃): 174.89, 160.55, 158.59, 153.91, 152.49, 124.23, 124.17, 122.18, 121.98, 121.36, 120.27, 120.20, 110.89, 110.70, 16.15; MS (EI): m/z 211 (M+1, 100); HRMS: (ESI): Calcd for C₁₀H₇F₂O₇S[M+H⁺]:211.0999, Found: 211.0231.

6-chloro-3-(methylthio)-4H-chromen-4-one:

![3d](image)

White Solid; Yield (74%); m.p.133-137 °C. IR (KBr, cm⁻¹): 3086, 1628, 1549, 1465, 1359, 1117, 1080, 814, 643; ¹H-NMR (400 MHz, CDCl₃): 8.21 (d, J = 2.8 Hz, 1H), 8.03 (s, 1H), 7.63-7.60 (m, 1H), 7.43 (d, 1H), 2.40 (s, 3H); ¹³C NMR (400 MHz,CDCl₃): 174.47, 154.56, 153.70, 134.00, 131.36, 125.41, 123.95, 122.17, 119.82, 16.11; MS (EI): m/z 227 (M+1, 100); HRMS: (ESI): Calcd for C₁₀H₇ClO₂S[M+H⁺]:226.9927, Found: 226.9934.

6-methyl-3-(methylthio)-4H-chromen-4-one:
Found: 122.92, 122.84, 119.92, 15.67; (s, 1220, 1080, 1058, 885, 734); 1H-NMR (400 MHz, CDCl3): 8.05 (s, 1H), 7.60 (d, J=3Hz, 1H), 7.40-7.25 (m, 2H), 3.90 (s, 3H), 2.40 (s, 3H); 13C NMR (400 MHz, CDCl3): 175.47, 157.07, 153.89, 151.12, 123.93, 123.75, 120.80, 119.47, 104.87, 55.90, 16.34; MS (EI): m/z 223 (M+1, 100); HRMS: (ESI): Calcd for C13H10O5S[M+H]:222.9985; Found: 222.9987.

6-methoxy-3-(methylthio)-4H-chromen-4-one:

Off white Solid; Yield (83%); m.p. 97-101 °C; IR (KBr, cm⁻¹): 2918, 1639, 1482, 1367, 867, 824, 779, 720; 1H-NMR (400 MHz, CDCl3): 8.05-8.03 (m, 2H), 7.49-7.47 (m, 1H), 7.36 (d, J=8.8Hz, 1H), 2.46 (s, 3H), 2.40 (s, 3H); 13C NMR (400 MHz, CDCl3): 175.65, 154.52, 153.96, 135.46, 135.01, 125.28, 122.81, 121.43, 117.78, 20.90, 16.34; MS (EI): m/z 207 (M+1, 100); HRMS: (ESI): Calcd for C13H10O5S[M+H]:207.0488; Found: 207.0488.

8-fluoro-3-(methylthio)-4H-chromen-4-one:

Off white Solid; Yield (81%); m.p. 237-241 °C; IR (KBr, cm⁻¹): 3079, 1630, 1557, 1484, 1340, 1256, 1099, 885; 1H-NMR (500 MHz, CDCl3): 8.05 (s, 1H), 8.02-8.00 (m, 1H),7.46-7.43 (m, 1H), 7.38-7.35 (m, 1H), 2.41 (s, 3H); 13C NMR (400 MHz, CDCl3): 174.61, 174.66, 174.59, 152.76, 152.00, 149.99, 145.12, 145.03, 125.07, 125.02, 124.82, 122.81, 121.09, 121.06, 119.52, 119.39, 15.98; MS (EI): m/z 211 (M+1, 100); HRMS: (ESI): Calcd for C13H10F2O5S[M+H]:211.0229; Found: 211.0222.

3-(methylthio)-6-nitro-4H-chromen-4-one:

Yellow Solid; Yield (72%); m.p. 150-152 °C; IR (KBr, cm⁻¹): 3095, 1642, 1517, 1448, 1337, 1220, 1080, 1058, 885, 734; 1H-NMR (400 MHz, CDCl3): 9.12 (d, J=2.8Hz, 1H), 8.52-8.49 (dd, J=9.2Hz, 1H), 8.02 (s, 1H), 7.64 (d, 1H), 2.43 (s, 3H); 13C NMR (400 MHz, CDCl3): 174.14, 158.93, 152.74, 144.87, 128.02, 123.74, 122.92, 122.84, 119.92, 15.67; MS (EI): m/z 238 (M+1, 100); HRMS: (ESI): Calcd for C13H10NO5S[M+H]:236.0018; Found: 236.0021.

7-methoxy-3-(methylthio)-4H-chromen-4-one:

Off white Solid; Yield (76%); m.p. 153-157 °C; IR (KBr, cm⁻¹): 3073, 1622, 1615, 1606, 1435, 1233, 1090, 935, 828; 1H-NMR (500 MHz, CDCl3): 8.16 (d, J= 9.2Hz, 1H), 7.97 (s, 1H), 7.00-6.97 (m, 1H),
6.82 (d, J=2.4Hz, 1H), 3.90 (s, 3H), 2.39 (s, 3H), 13C NMR (400 MHz, CDCl3): 174.93, 164.10, 158.04, 153.30, 127.46, 121.71, 117.05, 114.83, 100.07, 55.81, 16.27; MS (EI): m/z 223 (M+1,100); HRMS: (ESI): Calcd for C15H15O5S[M+H]:223.0428, Found: 223.0429.

3-(methylthio)-4H-benzo[h]chromen-4-one:

![Diagram of 3-(methylthio)-4H-benzo[h]chromen-4-one]

Light yellow Solid; Yield (87%); m.p. 134-138 °C; IR (KBr, cm−1): 3063, 1559, 1387, 1207, 1105, 884, 759, 576; 1H-NMR (400 MHz, CDCl3): 8.49 (d, J=8, 1H), 8.20-8.18 (m, 2H), 7.95-7.93 (m, 1H), 7.80 (d, J=10 Hz, 1H) 7.74-7.67 (m, 2H), 2.46 (s, 3H); 13C NMR (400 MHz, CDCl3): 175.35, 153.75, 152.05, 135.74, 129.43, 128.09, 127.22, 125.58, 123.85, 123.83, 122.18, 120.86, 119.18, 15.84; MS (EI): m/z 243 (M+1,100); HRMS: (ESI): Calcd for C16H17O5S[M+H]:243.0480, Found: 243.0479.

3-(methylthio)-6-phenyl-4H-chromen-4-one:

![Diagram of 3-(methylthio)-6-phenyl-4H-chromen-4-one]

Off white solid; Yield (84%); m.p. 146-150 °C; IR (KBr, cm−1): 2918, 1643, 1559, 1460, 1316, 1072, 762, 700; 1H-NMR (500 MHz, CDCl3): 8.4 (d, J= 2.4 Hz, 1H), 8.08 (s, 1H), 7.93-7.90 (m, 1H), 7.67-7.65 (m, 2H), 7.54-7.39 (m, 4H), 2.42 (s, 3H); 13C NMR (400 MHz, CDCl3): 175.63, 155.62, 153.80, 139.13, 138.64, 132.65, 128.96, 127.90, 127.15, 123.83, 123.24, 121.88, 118.56, 16.27; MS (EI): m/z 268 (M+1,100); HRMS: (ESI): Calcd for C16H15O5S[M+H]:269.0663, Found: 269.0637

6-(4-fluorophenyl)-3-(methylthio)-4H-chromen-4-one:

![Diagram of 6-(4-fluorophenyl)-3-(methylthio)-4H-chromen-4-one]

Light Yellow solid; Yield (86%); m.p. 120-125 °C; IR (KBr, cm−1): 2919, 1633, 1472, 1324, 1220, 1079, 863, 814; 1H-NMR (500 MHz, CDCl3): 8.41 (d, J= 2.4Hz, 1H), 8.07 (s, 1H), 7.87-7.85 (m, 1H), 7.63-7.59 (m, 2H), 7.54 (d, J= 8.8 Hz , 1H), 7.18-7.14 (m, 2H), 2.42 (s, 3H); 13C NMR (400 MHz, CDCl3): 175.56, 163.98, 161.52, 155.55, 153.77, 137.64, 135.29, 135.26, 132.46, 128.83, 128.75, 123.75, 122.32, 121.94, 118.66, 116.01, 115.79, 16.23; MS (EI): m/z 287 (M+1,100); HRMS: (ESI): Calcd for C16H11FO5S[M+H]:287.0542, Found: 287.0546.

6-(4-methoxyphenyl)-3-(methylthio)-4H-chromen-4-one:

![Diagram of 6-(4-methoxyphenyl)-3-(methylthio)-4H-chromen-4-one]

Off white solid; Yield (84%); m.p. 114-117 °C; IR (KBr, cm−1): 3445, 3423, 2922, 1635, 1558, 1465, 1246, 811, 532; 1H-NMR (500 MHz, CDCl3): 8.41 (d, J= 2.4Hz, 1H), 8.0 (s, 1H), 7.89-7.86 (m, 1H), 7.60-7.58 (m, 2H), 7.51 (d, J= 8.8 Hz , 1H), 7.01 (m, 2H), 3.86 (s, 3H), 2.42 (s, 3H); 13C NMR (400 MHz, CDCl3): 175.64, 159.53, 155.19, 153.74, 138.20, 132.23, 131.52, 128.16, 123.16, 122.95, 121.67, 118.44, 114.34, 55.30, 16.23; MS (EI): m/z 299 (M+1, 100); HRMS: (ESI): Calcd for C17H16O5S[M+H]:299.0742, Found: 299.0744.

3-(methylthio)-6-(5-phenyl-1H-pyrazol-1-yl)-4H-chromen-4-one:
Off white solid; Yield (87%); m.p. 167-171 °C; IR (KBr, cm⁻¹): 1624, 1497, 1456, 1118, 1090, 967, 934, 908; ¹H-NMR (500 MHz, CDCl₃): 8.77 (d, J= 2.4Hz, 1H), 8.49 (s, 1H), 8.35-8.32 (m, 2H), 7.97-7.95 (m, 2H), 7.89-7.87 (m, 1H), 7.45-7.38 (m, 3H), 7.39 (s, 3H); ¹³C NMR (400 MHz, CDCl₃): 175.21, 154.20, 153.87, 153.0, 152.6, 132.69, 128.69, 128.39, 128.21, 125.85, 125.49, 121.81, 119.71, 113.54, 105.87, 16.21; MS (EI): m/z 335 (M+1, 100); HRMS: (ESI): Calcd for C₁₉H₁₄N₂O₂S: [M+H]:335.0854, Found: 335.0862

3-(phenylthio)-4H-chromen-4-one

Color less liquid; Yield (81%); IR (KBr, cm⁻¹): 3061, 1652, 1467, 1350, 1313, 1109, 753, 692; ¹H-NMR (400 MHz, CDCl₃): 8.27 (d, J=8.4Hz, 1H), 8.16 (s, 1H), 7.72 (m, 1H), 7.49-7.38 (m, 4H), 7.30-7.20 (m, 3H); ¹³C NMR (125 MHz, CDCl₃): 175.16, 157.47, 156.43, 134.10, 129.91, 129.29, 127.21, 126.53, 125.84, 123.76, 120.00, 118.25; MS (EI): m/z 255 (M+1,100); HRMS: (ESI): Calcd for C₁₅H₁₀O₂S: [M+H]:255.0480, Found: 255.0483.

3-(p-tolylthio)-4H-chromen-4-one:

Yellow solid; Yield (73%); m.p. 102-107 °C; IR (KBr, cm⁻¹): 3418, 2921, 1643, 1547, 1455, 1346, 1098, 750; ¹H-NMR (400 MHz, CDCl₃): 8.25-8.23 (m, 1H), 8.04 (s, 1H), 7.70-7.65 (m, 1H), 7.46-7.40 (m, 2H), 7.36-7.33 (m, 2H), 7.12-7.10 (m, 2H), 2.31 (s, 3H); ¹³C NMR (125 MHz, CDCl₃): 175.04, 156.25, 156.21, 137.55, 133.83, 130.95, 130.00, 129.77, 126.34, 125.53, 125.31, 121.06, 118.06, 21.04; MS (EI): m/z 269 (M+1,100); HRMS: (ESI): Calcd for C₁₆H₁₂O₂S: [M+H]:269.0636, Found: 269.0642.

8-fluoro-3-(p-tolylthio)-4H-chromen-4-one:

Yellow solid; Yield (70%); m.p. 139-143 °C; IR (KBr, cm⁻¹): 3055, 1653, 1544, 1484, 1306, 1255, 1101, 751; ¹H-NMR (400 MHz, CDCl₃): 7.99-7.97 (m, 2H), 7.44-7.42 (m, 1H), 7.38-7.33 (m, 3H), 7.14-7.12 (m, 2H), 2.32 (s, 3H); ¹³C NMR (125 MHz, CDCl₃): 174.05, 174.03, 154.80, 152.00, 149.98, 145.11, 145.02, 138.09, 131.65, 130.16, 128.83, 125.18, 125.14, 122.44, 121.31, 121.27, 119.60, 119.47, 21.08; MS (EI): m/z 287 (M+1,100); HRMS: (ESI): Calcd for C₁₆H₁₁FOS: [M+H]:287.0542, Found: 287.0545.
8-fluoro-3-(phenylthio)-4H-chromen-4-one:

White solid; Yield (79%); m.p. 114-118 °C; IR (KBr, cm⁻¹): 3057, 1655, 1559, 1474, 1350, 1257, 1106, 750; ¹H-NMR (400 MHz, CDCl₃): 8.10 (s, 1H), 8.01 (d, J = 8.0 Hz, 1H), 7.49-7.43 (m, 3H), 7.41-7.24 (m, 4H); ¹³C NMR (125 MHz, CDCl₃): 174.17, 174.14, 156.11, 152.39, 149.86, 145.16, 133.20, 130.63, 129.42, 127.66, 125.78, 125.74, 125.50, 125.43, 122.12, 121.95, 121.50, 121.46, 121.35, 119.90, 119.73, 118.24, MS (EI): m/z 273 (M+1,100); HRMS: (ESI): Calcd for C₁₅H₉FO₂S [M+H]: 273.0386, Found: 237.0390.

6-chloro-3-(p-tolylthio)-4H-chromen-4-one:

Yellow solid; Yield (71%); m.p. 123-127 °C; IR (KBr, cm⁻¹): 3066, 1651, 1461, 1298, 1085, 910, 817, 654; ¹H-NMR (400 MHz, CDCl₃): 8.19 (d, J = 2.4 Hz, 1H), 7.97, (s, 1H), 7.62-7.59 (m, 1H), 7.41-7.39 (d, J = 7.2 Hz, 1H), 7.36-7.34 (m, 2H), 7.13-7.11 (d, J = 8.0 Hz, 2H), 2.32 (s, 3H). ¹³C NMR (125 MHz, CDCl₃): 173.94, 155.78, 154.58, 137.95, 134.07, 131.52, 131.40, 130.13, 129.14, 125.67, 124.33, 121.70, 119.83, 21.08; MS (EI): m/z 303 (M+1,100); HRMS: (ESI): Calcd for C₁₆H₁₁ClO₂S [M+H]: 303.0247, Found: 303.0255.

6-methoxy-3-(phenylthio)-4H-chromen-4-one:

Light yellow solid; Yield (74%); m.p. 94-98 °C; IR (KBr, cm⁻¹): 3064, 1640, 1479, 1431, 1363, 1311, 1016, 729; ¹H-NMR (400 MHz, CDCl₃): 8.17 (s, 1H), 7.60 (d, J = 3.2 Hz, 1H), 7.43-7.37 (m, 3H), 7.30-7.21 (m, 4H), 3.95 (s, 3H); ¹³C NMR (125 MHz, CDCl₃): 174.96, 157.30, 151.17, 134.24, 133.75, 130.20, 129.86, 129.62, 129.20, 129.11, 126.96, 124.33, 124.08, 119.55, 118.82, 105.32, 55.92; MS (EI): m/z 285 (M+1,100); HRMS: (ESI): Calcd for C₁₆H₁₂O₃S [M+H]: 285.0585, Found: 285.0587.

6-bromo-3-(p-tolylthio)-4H-chromen-4-one:

Yellow solid; Yield (75%); m.p. 234-238 °C; IR (KBr, cm⁻¹): 3061, 1647, 1458, 1296, 1115, 1084, 906, 816; ¹H-NMR (400 MHz, CDCl₃): 8.36 (d, J = 2 Hz, 1H), 7.97, (s, 1H), 7.76-7.73 (m, 1H), 7.36-7.33 (m, 3H), 7.13 (d, J = 8 Hz, 2H), 2.32 (s, 3H). ¹³C NMR (125 MHz, CDCl₃): 173.80, 155.76, 155.02, 137.98, 136.83, 131.42, 130.14, 129.11, 128.89, 124.69, 121.83, 120.05, 118.97, 21.09; MS (EI): m/z 347 (M+1,100); HRMS: (ESI): Calcd for C₁₆H₁₁BrO₂S [M+H]: 346.9741, Found: 346.9746.

3-[(4-chlorophenyl)thio]-4H-chromen-4-one:
White solid; Yield (72%); m.p. 154-158 °C; IR (KBr, cm⁻¹): 3418, 3047, 1640, 1547, 1462, 1379, 1099, 750; ¹H-NMR (400 MHz, CDCl₃): 8.24-8.22 (m, 2H), 7.73-7.69 (m, 1H), 7.49-7.42 (m, 2H), 7.33-7.31 (m, 2H), 7.26-7.24 (m, 2H); ¹³C NMR (125 MHz, CDCl₃): 175.07, 158.02, 156.48, 134.29, 133.22, 132.92, 131.00, 129.39, 126.57, 126.02, 123.84, 119.35, 118.33; MS (EI): m/z 289 (M⁺,100); HRMS: (ESI): Calcd for C₁₅H₁₉ClO₂S: 289.0090, Found: 289.0079.

6-methyl-3-[(p-tolylthio)-4H-chromen-4-one:

Yellow solid; Yield (76%); m.p. 122-126 °C; IR (KBr, cm⁻¹): 3061, 1645, 1557, 1484, 1304, 1115, 814, 786, 511; ¹H-NMR (400 MHz, CDCl₃): 8.04-8.02 (m, 2H), 7.48-7.46 (m, 1H), 7.35-7.32 (m, 3H), 7.10-7.09 (m, 2H), 2.45 (s, 3H), 2.31 (s, 3H); ¹³C NMR (125 MHz, CDCl₃): 175.31, 156.56, 154.70, 137.53, 135.80, 135.26, 130.85, 130.21, 130.12, 125.76, 120.70, 117.97, 21.20, 21.08; MS (EI): m/z 283 (M⁺,100); HRMS: (ESI): Calcd for C₁₇H₁₄O₂S: 283.0793, Found: 283.0801.

3-[(4-chlorophenyl)thio]-6-methoxy-4H-chromen-4-one:

Off white solid; Yield (70%); m.p. 161-165 °C; IR (KBr, cm⁻¹): 3072, 1649, 1562, 1478, 1432, 1303, 1084, 818; ¹H-NMR (400 MHz, CDCl₃): 8.23 (s, 1H), 7.58-7.57 (d, J= 3.2Hz, 1H), 7.43 (d, J=8.8Hz, 1H), 7.32-7.22 (m, 5H), 3.88 (s, 3H); ¹³C NMR (125 MHz, CDCl₃): 175.01, 157.91, 157.56, 151.35, 133.16, 133.12, 130.86, 129.36, 124.55, 124.40, 119.76, 118.33, 105.46, 56.17; MS (EI): m/z 319 (M⁺,100); HRMS: (ESI): Calcd for C₁₆H₁₁ClO₃S: 319.0196, Found: 319.0200.

6-fluoro-3-[(p-tolylthio)-4H-chromen-4-one:

Yellow solid; Yield (73%); m.p. 151-155 °C; IR (KBr, cm⁻¹): 3073, 1650, 1557, 1478, 1346, 1302, 1103, 816; ¹H-NMR (400 MHz, CDCl₃): 8.01 (s, 1H), 7.87-7.84 (m, 1H), 7.48-7.40 (m, 2H), 7.39-7.34 (m, 2H), 7.13-7.11 (m, 2H), 2.32 (s, 3H); ¹³C NMR (125 MHz, CDCl₃): 174.51, 161.03, 158.56, 156.21, 152.65, 152.67, 138.00, 131.38, 130.25, 129.46, 124.81, 124.74, 122.44, 122.18, 120.95, 120.46, 120.38, 111.39, 111.16, 21.23; MS (EI): m/z 287 (M⁺,100); HRMS: (ESI): Calcd for C₁₅H₁₁FO₂S: 319.0196, Found: 319.0200.

6-methoxy-3-[(p-tolylthio)-4H-chromen-4-one:

Pale Yellow solid; Yield (77%); m.p. 121-126 °C; IR (KBr, cm⁻¹): 3425, 2920, 1639, 1434, 1303, 1270, 1083, 810; ¹H-NMR (400 MHz, CDCl₃): 8.06 (s, 1H), 7.59 (d, J= 2.0Hz, 1H), 7.40 (m, 1H), 7.38-7.09 (m, 5H), 3.88 (s, 3H), 2.30 (s, 3H); ¹³C NMR (125 MHz, CDCl₃): 174.54, 157.37, 156.44, 151.34, 137.61, 130.95,
130.25, 130.15, 124.40, 124.19, 119.69, 105.42, 56.10, 21.23; MS (EI): m/z 299 (M+, 100); HRMS (ESI): Calcd for C_{17}H_{14}O_{3}S[M+H]^+: 299.0749; Found: 299.0742.

3-(p-tolylthio)-4H-benzo[h]chromen-4-one:

Yellow solid; Yield (80%); m.p. 131-134 °C; IR (KBr, cm⁻¹): 3425, 2920, 1639, 1434, 1303, 1270, 1083, 810; ¹H-NMR (400 MHz, CDCl₃): 8.45 (d, J= 8 Hz, 1H), 8.18-8.16 (d, J= 8.8Hz, 1H), 7.16 (d, J= 8 Hz, 2H), 7.16 (d, J= 8 Hz, 1H), 7.10 (s, 1H), 7.94 (d, J= 7.6 Hz, 1H), 7.79-7.70 (m, 3H), 7.69-7.40 (m, 2H), 7.16 (d, J= 8 Hz, 2H), 2.33 (s, 3H); ¹³C NMR (125 MHz, CDCl₃): 174.94, 154.38, 153.87, 138.09, 135.91, 131.80, 130.32, 129.61, 129.22, 128.25, 127.41, 125.83, 123.93, 123.57, 122.28, 121.17, 119.73, 21.26; MS (EI): m/z 319 (M+, 100); HRMS (ESI): Calcd for C_{20}H_{14}O_{2}S[M+H]^+: 319.0798; Found: 319.0793.

6-phenyl-3-(p-tolylthio)-4H-chromen-4-one:

Off white solid; Yield (76%); m.p. 154-159 °C; IR (KBr, cm⁻¹): 3438, 3063, 2920, 1645, 1551, 1464, 1083, 697; ¹H-NMR (400 MHz, CDCl₃): 8.45 (d, J= 2.4 Hz, 1H), 8.06 (s, 1H), 7.34-7.25 (m, 6H), 7.27-7.18 (m, 2H), 7.13 (d, J= 8.0 Hz, 2H), 2.33 (s, 3H); ¹³C NMR (125 MHz, CDCl₃): 175.28, 156.32, 155.80, 139.23, 138.93, 137.76, 132.89, 131.15, 130.21, 129.90, 129.14, 128.10, 127.31, 124.29, 123.80, 121.28, 118.76, 21.24; MS (EI): m/z 345 (M+, 100); HRMS (ESI): Calcd for C_{22}H_{16}O_{2}S[M+H]^+: 345.0798; Found: 345.0793.

6-(4-chlorophenyl)thio)-8-fluoro-4H-chromen-4-one:

Off white solid; Yield (72%); m.p. 177-181 °C; IR (KBr, cm⁻¹): 3063, 1645, 1556, 1479, 1259, 1112, 807, 759; ¹H-NMR (400 MHz, CDCl₃): 8.20 (s, 1H), 8.00-7.97 (m, 1H), 7.50-7.45 (m, 1H), 7.40-7.34 (m, 3H), 7.27-7.25 (m, 2H); ¹³C NMR (125 MHz, CDCl₃): 173.95, 156.68, 152.03, 150.01, 145.18, 145.09, 133.57, 131.94, 131.50, 129.38, 125.57, 125.52, 125.40, 121.39, 121.36, 120.44, 119.95, 119.82; MS (EI): m/z 307 (M+, 100); HRMS (ESI): Calcd for C_{16}H_{8}ClFO_{2}S[M+H]^+: 306.9996; Found: 307.0003.

7-methoxy-3-(p-tolylthio)-4H-chromen-4-one:

Pale yellow solid; Yield (74%); m.p. 121-125 °C; IR (KBr, cm⁻¹): 3425, 2920, 1639, 1434, 1303, 1270, 1083, 810; ¹H-NMR (400 MHz, CDCl₃): 8.14 (d, J= 7.2 Hz, 1H), 7.96 (s, 1H), 7.34-7.32 (m, 2H), 7.11-7.09 (m, 2H), 6.96-6.82 (m, 1H), 6.81 (s, 1H), 3.89 (s, 3H), 2.30 (s, 3H); ¹³C NMR (125 MHz, CDCl₃): 174.55, 164.33, 158.22, 156.00, 137.58, 130.97, 130.14, 127.93, 121.02, 117.61, 115.03, 100.36, 56.01, 21.22; MS (EI): m/z 299 (M+, 100); HRMS (ESI): Calcd for C_{17}H_{14}O_{2}S[M+H]^+: 299.0742; Found: 299.0753.
RND-FA-3.5 MIN.M
Column: ACQUITY UPLC BEH C18 (50mmx2.1mm, 1.7um)
Mobile Phase: B1: 0.1 % FA IN WATER A1: 0.1%FA IN ACN
Gradient: Time (min) /%A1: 0/2, 0.4/2, 2.8/98, 3.4/98, 3.41/2, 3.5/2
Column Flow Rate: 0.6 ml/min
Column Temperature: 60°C
Single Mass Analysis
Tolerance = 1000.0 PPM / DBE: min = -1.5, max = 50.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
5 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)

Elements Used:
C: 0-10  H: 0-9  O: 0-2  S: 0-1

3a

Date of analysis: 04-Oct-2017 15:10:46
Instrument ID: ANL-MC13 LC/MS-851
1: TOF MS ES+

Minimum: 193.0329
Maximum: 193.7302

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Date of Analysis : 8/21/2017 9:39:31 PM  Injection Vol : 0.300µL  
Acq. Method : RND-FA-3.5mins  Instrument ID : ANL-MOL5-LCMS-   
Sample Name : GVK-PHD-VK-KLU-MB-142

RND-FA-3.5 MIN.M  
Column: ACQUITY UPLC BEH C18 (50mmx2.1mm, 1.7µm)  
Mobile Phase: B1: 0.1 % FA IN WATER A1: 0.1%FA IN ACN  
Gradient: Time (min) /%A1: 0/2, 0.2/2, 2.3/98, 3.4/98, 3.41/2, 3.5/2  
Column Flow Rate: 0.8 ml/min  
Column Temperature: 56°C

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Single Mass Analysis
Tolerance = 1000.0 PPM / DEB: min = -1.5, max = 50.0
Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions
11 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)
Elements Used:
C: 0-10 H: 0-8 O: 0-2 S: 0-1 Br: 0-1

Minimum: 5.0 1000.0 50.0

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Date of Analysis: 8/10/2017 8:24:01 PM  
Injection Vol: 0.300μL
Acq. Method: RND-FA-3.5mins  
Instrument ID: ANL-MCL5-ICMS-001
Sample Name: GVK-CBK-3-Phd-40

RND-FA-3.5 MIN.M
Column: ACQUITY UPLC BEH C18 (50mmx2.1mm, 1.7μm)
Mobile Phase: B1: 0.1% FA IN WATER A1: 0.1% FA IN ACN
Gradient: Time (min) /%A1: 0/2, 0.2/2, 2.3/95.3/0, 3.4/95.3.41/2, 3.5/2
Column Flow Rate: 0.8 ml/min
Column Temperature: 50°C


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**Single Mass Analysis**

Tolerance = 1000.0 PPM / DBE: min = -1.5, max = 50.0
Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions
10 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)

**Elements Used:**
C: 0-10  H: 0-8  O: 0-2  F: 0-1  S: 0-1

GVK Bio sciences (pvt) Ltd
Analytical Research and Development
511802393935 7 (0.262) AM (Cen:2, 50.00, Ar:5.0, 196.11, 1.00, 1.55); Si (Mg, 5c:2.00); Eb (1.49, 99); Cm (S:15)

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![Graphical representation of the mass spectrum](image1)

![Graphical representation of the mass spectrum](image2)
**Sample Name**: GVK-CBK-1-47

**Acq. Method**: RND-FA-3.5mins

**Column**: ACQUITY UPLC BEH C18 (50mmx2.1mm, 1.7um)

**Gradient**: Time (min) /%A: 0/2, 0.2/2, 2.3/98, 3.4/98, 3.41/2, 3.5/2

**Column Flow Rate**: 0.8 ml/min

**Column Temperature**: 50°C

---

### DAD1 A, Sig=215,4 Ref-off

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Single Mass Analysis
Tolerance = 1000.0 PPM / DBE: min = -1.5, max = 50.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
11 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)
Elements Used:
C: 0-10  H: 0-8  O: 0-2  S: 0-1  Cl: 0-1

GVK Bio sciences (pvt) Ltd
Analytical Research and Development
51170084416 13 (0.335) AM (Cen,4, 20.00, Ar,5.0,195,11,1.00,L S S); Sm (Mn,5x2.00); Sb (1.40.00)

Minimum:                  Maximum:                  DBE  1-FIT  Formula
226.9927  226.9934  -0.7  -3.1  6.5  382.7  C10 H8 O2 S Cl

Comment: IN Kbr
GVK-CBK-1-47

Date: 04-Oct-2017 15:55:31
Instrum ID: ANL-MCL-0560-001

1.44e4
**GVK BIOSCIENCES PVT. LTD.**

**MEDICINAL CHEMISTRY LABORATORY - ANALYTICAL RESEARCH**

**LCMS REPORT**

---

**Date of Analysis:** 8/17/2017 6:42:56 PM  
**Injection Vol:** 0.300 uL  
**Acq. Method:** RND-FA-3.5mnS  
**Instrument ID:** ANL-MLG5-LCMS-  
**Sample Name:** GVR-CBK-1-42

---

**RND-FA-3.5 MIN.M**

- **Column:** ACQUITY UPLC BEH C18 (50mmx2.1mm, 1.7um)
- **Mobile Phase:** B: 0.1% FA IN WATER  A: 0.1% FA IN ACN
- **Gradient Time:** 0.2, 0.2/2, 2.3/98, 3.4/98, 3.41/2, 3.5/2
- **Column Flow Rate:** 0.8 ml/min  
**Column Temperature:** 50°C

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MS Spectrum

MSD1 SPC, time=2.039 of C:\CHEM320\DATA\2017\AUG\PRO\0511\068997.D  MN-ES+APCI, Pos, Scan, Frag. 70, *+VE*

Max: 562432

3e

H₃C

O

S

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207.0

200.0

140  160  180  200  220  240  260
m/z

28
Single Mass Analysis
Tolerance = 1000.0 PPM / DBE: min = -1.5, max = 50.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
5 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)
Elements Used:
C: 0.11  H: 0.11  O: 0.2  S: 0.1

Instrument ID: ANL-MCLS-1CMS-001
1-SCF MS ES+ 1.79e4

Minimum: 5.0  1000.0  50.0

Mass  Calc. Mass  mDa  PPM  DBE  i-PII  Formula
207.0488 207.0480  0.8  3.9  6.5  5044.3  C11 H11 O2 S

Comment: EN Xcr
No. of Scans:
Date: 11/30/2018 4:14:59 PM

29
**LCMS Report**

**Vial position:** P2-A-01

**Date of Analysis:** 8/18/2017 11:07:04 PM  
**Injection Vol:** 0.300mL

**Acq. Method:** RND-FA-3.5mins  
**Instrument ID:** ANL-MCL5-LCMS-0C

**Sample Name:** E119051-46

---

**RND-FA-3.5 MIN.M**

**Column:** ACQUITY UPLC BEH C18 (50mmx2.1mm, 1.7um)

**Mobile Phase:** B1: 0.1% FA IN WATER  
A1: 0.1%FA IN ACN

**Gradient:** Time (min)  
/4A1: 0/2, 0.2/2, 2.3/98, 3.4/98, 3.41/2, 3.5/2

**Column Flow Rate:** 0.8 ml/min

**Column Temperature:** 50°C

---

![Graph](image.png)

**DAD1 A, Sig=215,4 Ref=off**

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*Graph and data as per report.*
Elemental Composition Report

Single Mass Analysis (displaying only valid results)
Tolerance = 1000.0 PPM / DBE: min = -1.5, max = 50.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
7 formula(s) evaluated with 1 results within limits (up to 1 closest results for each mass)

Elements Used:
C: 0.11  H: 0.11  O: 0.3  S: 0.1

Date of analysis: 07-Mar-2018 12:42:04
Instrument ID: ANL.MC13.LCMS.201
1: TOF MS ES+

Minimum: 
Maximum: 

Mass  Calc. Mass  m/z  PPM  DBE  i-FIT  Formula
223.0429  223.0429  -4.9  -22.0  6.5  15.4  Cl1  H11  O3  S

Comment: IN Ker
No. of Scans: 
Resolution: 
 Acquisition: 

Date: 13/2017 12:16:18 PM
User: Admin
Single Mass Analysis
Tolerance = 1000.0 PPM / DBE: min = -1.5, max = 50.0
Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions
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Elements Used:
C: 0.10  H: 0.8  O: 0.2  F: 0.1  S: 0.1

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Date of analysis: 04-Oct-2017
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1: TOF MS ESI+ 1.394
Vial position : F1-E-03
Date of Analysis : 9/30/2017 9:43:50 PM Injection Vol : 0.300μL
Acq. Method : RND-FA-3.5mms Instrument ID : ANL-MCLS-LCMS-00:
Sample Name : GVR-VK-PHD-32

RND-FA-3.5 MIN.M
Column: ACQUITY UPLC BEH C18 (50mmx2.1mm, 1.7μm)
Mobile Phase: B1: 0.1% FA IN WATER A1: 0.1% FA IN ACN
Gradient: Time (min) /%B1: 0/2, 0.2/2, 2.3/98,3.4/98,3.41/2,3.5/2
Column Flow Rate: 0.8 mL/min
Column Temperature: 50°C

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Single Mass Analysis (displaying only valid results)
Tolerance = 1000.0 PPM / DBE: min = -1.5, max = 50.0
Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions
18 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)
Elements Used:
C: 0-10  H: 0-8  N: 0-1  O: 0-4  S: 0-1

GVK Bio sciences (pvt) Ltd
Analytical Research and Development
511712A0385 8 (0.326) AM (Cn,2,50.00, Ar,5.56,194.98,1.00,LS 5); Sm (Mn, Sn2.00); Sb (1.40); Cn (2.22)

Minimum: 236.0022
Maximum: 236.0018
Mass  Calc. Mass  m/z  PPM  DBE  i-FIT  Formula
236.0022  236.0018  0.3  1.3  8.5  31.0  C10 H6 N O4 S

Date of analysis 08-Dec-2017 16:53:57
Instrument ID: ANL-231, LCMS-201

1. TOF MS ES+

57.0
LC/MS REPORT

Date of Analysis: 9/9/2017  TIME: 2:21:41 PM  Vial position: P1-B-08
Sample Name: GVK-RS-009-70  Injection Vol. 0.200 mL
Acq. Method: C:\Chem32\METHODS\RND-FA-3.2-MIN.M  Instrument Name:ANL-MCL5-LCMS-002

Acq Method Conditions: RND-FA-3.2-MIN
Column: Aquity UPLC BEH C18 (50mmx2.1 mm, 1.7um)
Mobile phase: A: 0.1% of Formic Acid in Water, B: 0.1% of Formic acid in Acetonitrile
Gradient: Time(min)/%A 0/0.2/2/1.5/98/2.6/98/2.61/2.3/2/2
Column temperature: 45 C, Flow rate: 0.8 ml/min

<table>
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<td>1.781</td>
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DAD1 A, Sig=215,4 Ref-off

ES-API, Pos, Scan, Freq: 100, *vea*
**Elemental Composition Report**

**Single Mass Analysis**

Tolerance = 1000.0 PPM / DBE: min = -1.5, max = 50.0

Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions
7 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)

Elements Used:
C: 0-11  H: 0-11  O: 0-3  S: 0-1

GVK-RK-009-70

GVK Bio sciences (pvt) Ltd
Analytical Research and Development

511706847288 9 (0.278) AM (Carb, 20.00, Ar, 5.0, 105.15, 10.00, LS 5); Sm (Mn, 562.00); Sb (1.46.00)

223.0428

Minimum: 5.0  1000.0  50.0

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<th>PPM</th>
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**Comment:** IN Rile

GVK-RK-1-Pub-70

No. of Scans: 1

Resolution: 1

Averages: 1

Date 11/02/18 11:56:16 AM

User: Admin

44
GOV BIOTECHNOLOGIES PVT. LTD.
MEDICINAL CHEMISTRY LABORATORY - ANALYTICAL RESEARCH
LCMS REPORT

Date of Analysis: 8/14/2017 2:17:57 PM
Injection Vol: 0.300uL
Acq. Method: RND-FA-3.5min
Instrument ID: AHL-MCL5-LOMS-0
Sample Name: GVP-CSK-3-Phd-43

RND-FA-3.5 MIN.M
Column: ACQUITY UPLC BEH C18 (50mmx2.1mm, 1.7um)
Mobile Phase: B1: 0.1 % FA IN WATER A1: 0.1%FA IN ACN
Gradient: Time (min) /%A1: 0/2, 0.2/2, 2.3/98, 3.4/98, 3.41/2, 3.5/2
Column Flow Rate: 0.8 ml/min
Column Temperature: 50°C


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<td>12.27</td>
<td>555.519</td>
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46
### Elemental Composition Report

**Single Mass Analysis**

- **Tolerance:** 1000.0 PPM / DBE: min = -1.5, max = 50.0
- **Selected filters:** None

**Monoisotopic Mass, Odd and Even Electron Ions**

5 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)

**Elements Used:**
- C: 0.14
- H: 0.11
- O: 0.2
- S: 0.1

**Data of analysis:**
- Date: 04-Oct-2017 13:06
- Instrument ID: ANL-MCL-1-LCMS-003
- 1: TOF MS ESI+

**Minimum:**
- 1.5

**Maximum:**
- 5.0

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<th>DBE</th>
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<td>Cl4 H11 O2 S</td>
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</table>

**Comment:**
- EN Khr

**User:** Admin

**Date:** 04-Oct-2017 13:06
RND-FA-3.5 MIN.M
Column: ACQUITY UPLC BEH C18 (50mmx2.1mm, 1.7um)
Mobile Phase: B1: 0.1 % FA IN WATER A1: 0.1%FA IN ACN
Gradient: Time (min) /%A1: 0/2, 0.2/2, 2.3/98, 3.4/98, 3.41/2, 3.5/2
Column Flow Rate: 0.8 ml/min
Column Temperature: 50°C

<table>
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<td>[1]</td>
<td>1.71</td>
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**Single Mass Analysis**
Tolerance = 1000.0 PPM  /  DBE: min = -1.5, max = 50.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
5 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)
Elements Used:
C: 0.16  H: 0.13  O: 0.2  S: 0.1

[Graph showing mass spectrum]

Minimum:  5.0  1000.0  -1.5
Maximum:  50.0

Mass  Calc. Mass  mDa  PPM  DBE  1-FIT  Formula
269.0637  269.0636  0.1  0.4  10.5  17759.7  C16 H13 O2 S
**Single Mass Analysis**

Tolerance = 1000.0 PPM / DBE: min = -1.5, max = 50.0

Selected filters: None

**Monoisotopic Mass, Odd and Even Electron Ions**

10 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)

Elements Used:

C: 0-16  H: 0-12  O: 0-2  F: 0-1  S: 0-1

---

**Mass**

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Single Mass Analysis
Tolerance = 1000.0 PPM / DBE: min = -1.5, max = 50.0
Selected filters: None

Monoisotopic Mass, Even Electron Ion
7 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)

Elements Used:
C: 0-17  H: 0-15  O: 0-3  S: 0-1

GVCK-RK-009-62

GVK Bio sciences (pvt) Ltd
Analytical Research and Development

31170984730 10 (0.346) AM (Cen,4, 20.00, Ar,5.0, 195.13,1,00,10,5); Sm (Mn, 3x2.00); Sb (1,40.00)

299.0744  299.0742  0.2  0.7  10.5  8182.5  C17 H15 O3 S

Date of analysis: 04-Oct-201715:40:38
Instruments ID: ANL-MCL-3-LCMS-021
1 TOF MS ES+
1.6904
Date of Analysis: 9/4/2017 1:48:16 PM
Injection Vol: 0.300μL
Acq. Method: RND-FA-3.5mns
Sample Name: GVK-PHD-VK-BALA-33-1

Column: ACQUITY UPLC BEH C18 (50mmx2.1mm, 1.7μm)
Mobile Phase: H2: 0.1% FA in WATER A1: 0.1% FA in ACN
Gradient: Time (min) /%/A1: 0/2, 0.2/2, 2.3/98, 3.4/98, 3.41/2, 3.5/2
Column Flow Rate: 0.8 ml/min
Column Temperature: 50°C

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Elemental Composition Report

Single Mass Analysis
Tolerance = 1000.0 PPM / DBE: min = -1.5, max = 50.0
Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions
12 formulas(s) evaluated with 1 results within limits (up to 1 closest results for each mass)
Elements Used:
C: 0-19  H: 0-15  N: 0-2  O: 0-1
GVK Bio sciences (pvt)Ltd
Analytical Research and Development
511802B35851. 9 (0.369) AM (Con,2, 50.00, Ar,5.0, 195.69, 1,00,LS S); Sm (Me, 5x2.00); Sb (1,40.00); Cln (4:18)

Minimum:
Maximum:
Mass  Calc. Mass  mDa  PPM  DBE  1-FIT  Formula
335.0062  335.0054  0.8  2.4  13.5  31.6  C19 H15 N2 O2 S

Date of analysis: 19-Feb-2018 11:50:51
Instrument ID: ANL-MCL-LEMS-001
1: TOF MS ES+ 3.4064

Comment: IN-Kne
GVK-Bio (pvt) Ltd
No of Spec: Resolution
Resolution
User: Adhira
Elemental Composition Report

Single Mass Analysis (displaying only valid results)
Tolerance = 1000.0 PPM / DBE: min = -1.5, max = 50.0
Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions
5 formula(e) evaluated with 1 results within limits (up to 1 best isotopic matches for each mass)
Elements Used:
C: 0-15  H: 0-11  O: 0-2  S: 0-1

511846B8827 (0.252) AM (Cen,2, 50.00, Ar,5,0,195.10,1.00,LS 5); Sm (Mn,5x2.00); Sb (1,40.00); Cm (4:14)
255.0493

Minimum:  1000.0
Maximum:  50.0

Mass  Calc. Mass  mDa  PPM  DBE  1-FIT  Formula
255.0483  255.0480  0.3  1.2  10.5  264.0  C15 H11 O2 S
Single Mass Analysis (displaying only valid results)
Tolerance = 1000.0 PPM / DBE: min = -1.5, max = 50.0
Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions
5 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)
Elements Used:
C: 0.16  H: 0.13  O: 0.2  S: 0.1

51171276056 8 (0.324) AM (Con,2, 50.0, Ar,5,0,105,13,1,00,LS S); Sim (Mn, 8d2.00); Sb (1,40.00)
269.0642

Minimum:                  Maximum:
Calc. Mass               1000.0  50.0
mDa                     5.0
P AM 5.0
DBE                   1-PIT 845.6  16  H13 02 S
Sample Code: GVK-CBK-1-51

Analytical Research and Development
GVK BIOSCIENCES PVT LTD

Date of Analysis: 28-Aug-2017 09:44:20
Instrument ID: ANL-MCL3-LCMS-010

Column: ACQUITY UPLC BEH C18 1.7μm, 2.1x150mm
Mobile Phases: A: 0.1% FA in Water, B: 0.1% FA in Acetonitrile
Flow Rate: 0.35ml/min
Temperature: 40°C

Scan: 133.0-1619.0 m/z

Figure 1: LC/MSchromatogram of compound 4c.

Figure 2: MS/MS spectra of compound 4c.

Figure 3: Mass spectrum of compound 4c.

Figure 4: High-resolution mass spectrum of compound 4c.
Elemental Composition Report

Single Mass Analysis (displaying only valid results)
Tolerance = 1000.0 PPM / DBE: min = -1.5, max = 50.0
Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions
10 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)

Elements Used:
C: 0-16  H: 0-12  O: 0-2  F: 0-1  S: 0-1

GVK Bio sciences (pvt) Ltd
Analytical Research and Development

51171248035 8 (0.330) AM (Cen,2, 80.00, Ar,5,0,196,09,1.00,LS 5); Sm (Mn,9x2.00); Sb (1,40.00) 287.0545

Minimum: 5.0  Maximum: 1000.0  Tolerance: 50.0

Mass  Calc. Mass  mDa  DBE  1-FIT  Formula
287.0545  287.0542  0.3  1.0  10.5  748.4  C16 H12 O2 F S
Elemental Composition Report

Single Mass Analysis (displaying only valid results)
Tolerance = 1000.0 PPM / DBE: min = -1.5, max = 50.0
Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions
10 formula(e) evaluated with 1 results within limits (up to 1 best isotopic matches for each mass)
Elements Used:
C: 0-15  H: 0-10  O: 0-2  F: 0-1  S: 0-1

51818829837 (0.285) AM (Cen,2, 50.00, Ar,6.0,0,195.11,1.00,LS 5); Sm (Mn, 5x2.00; Sb 1,40.00); Cm (5:13)

GWK Bio sciences (pvt)Ltd
Analytical Research and Development

Date of analysis: 15 Apr 2018 05:28
Instrument ID: ANL-MCL-4A; EMS-001
1 TOF MS 2.5e-4

Minimum: -1.5
Maximum: 1000.0

Mass Calc. Mass mDa FPPM DBE 1-FIT Formula
273.0390 273.0386 0.4 1.5 10.5 414.9 C15 H10 O2 F S
Elemental Composition Report

Single Mass Analysis
Tolerance = 1000.0 PPM / DBE: min = -1.5, max = 50.0
Selected filters: None

Monosotopic Mass, Odd and Even Electron Ions
11 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)
Elements Used:
C: 0-16  H: 0-12  O: 0-2  S: 0-1  Cl: 0-1

GWK-CIKK-I-Ph-52

Date of analysis: 16-Feb-2018 15:20:15
Instrument ID: ANL MCL-5 LCM-016

TOF MS ES+
7.47e4

Mass  Calc. Mass  mDa  PPM  DBE  1-FIT  Formula
303.0255  303.0247  0.8  2.6  10.5  917.1  C16 H12 O2 S Cl
Elemental Composition Report

Single Mass Analysis (displaying only valid results)
Tolerance = 1000.0 PPM / DBE: min = -1.5, max = 50.0
Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions
7 formula(s) evaluated with 1 results within limits (up to 1 best isotopic matches for each mass)
Elements Used:
C: 0-16 H: 0-13 O: 0-3 S: 0-1
GVK Bio sciences (pvt)Ltd
Analytical Research and Development
511643B6886 7 (0.281) AM (Cen,2.50.00, Ar,5,0,195,11,1.00,LS,5); Sm (Mn, 5x2.00); Sb (1.40.00); Cm (4.13)

Minimum: 59.0
Maximum: -1.5
Mass Calc. Mass m/z PPM DBE 1-PIT Formula
285.0587 285.0585 0.2 0.7 10.5 319.5 C16 H13 O3 S
**Elemental Composition Report**

**Single Mass Analysis (displaying only valid results)**
Tolerance = 1000.0 PPM / DBE: min = -1.5, max = 50.0
Selected filters: None

C: 0.16  H: 0.12  O: 0-2  S: 0-1  Br: 0-1

11 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)

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<td>10.5</td>
<td>C16 H12 O2 S Br</td>
</tr>
</tbody>
</table>

**Graphical Representation**

- Diagram 1: Molecular structure of 4g with Br, S, and other atoms.
- Diagram 2: Mass spectrum with peaks at 346.9746 and 346.9741.

**Additional Details**
- Date of analysis: 08-Dec-2015 13:58:27
- Instrument ID: ANL/MCL-3/SCID-501
- TOF MS ESI- 1.9463

**Comment:** BS Kbr

**Date:** 12/23/2017 12:31:22 PM

**User:** Admin

**Page 1**
**Acq. Method Conditions:** RND X-Bridge 5.0 Min
COLUMN: X-BRIDGE C18 (4.6mm x 75mm) 3.5 um
MOBILE PHASE A: 10mM Ammonium Acetate in water, B 100% ACN
Gradient: % of B 0.0/10.0,2/10,2.5/75,3.0/100,4.6/100,5.0/10
Column Temp: 35°C
Flow rate: 2.0 ml/min

---

**PEAK No | RT min | Area | Area %**
---|---|---|---|
| 1 | 2.176 | 338.080 | 6.548 |
| 2 | 3.071 | 4565.847 | 88.432 |
| 3 | 3.604 | 212.711 | 4.120 |
| 4 | 3.851 | 46.480 | 0.900 |

---
**Elemental Composition Report**

**Single Mass Analysis (displaying only valid results)**

**Tolerance = 1000.0 PPM / DBE: min = -1.5, max = 50.0**

**Selected filters: None**

**Monoisotopic Mass, Odd and Even Electron Ions**

11 formula(e) evaluated with 1 results within limits (up to 1 best isotopic matches for each mass)

**Elements Used:**

- C: 0-15
- H: 0-10
- O: 0-2
- S: 0-1
- Cl: 0-1

**GVK Bio sciences (pvt)Ltd**

**Analytical Research and Development**

**51184563677 7 (0.263) AM (Cen,2, 50.00, Ar,5.0,165.09,1.00,LS S); Sm (Mn, 5x2.00); Sb (1.40,00 ); Cm (5:10)**

**Date of analysis: 11 Apr-201816:08:55**

**Instrum ent ID: ANL-MCL2-LCMS-001**

**TOF MS EQ+ 7.29x3**

**Minimum:**

- 50.0
- 1000.0
- -1.5

**Maximum:**

- 50.0

**Mass Calc. Mass mDa PPM DBE 1-FIT Formula**

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<td>34.5</td>
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Comment: IN Kbr

 GVK-CBRX-1-15

**No. of Scales:**

- Resolution

**Apodization:**

**Date:** 4/2/2018 1:07:44 PM

**User:** Admin
Single Mass Analysis (displaying only valid results)
Tolerance = 1000.0 PPM / DBE: min = -1.5, max = 50.0
Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions
5 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)
Elements Used:
C: 0-17  H: 0-15  O: 0-2  S: 0-1

Minimum: 283.0801
Maximum: 494.6085
Mass  Calc. Mass  mDa  PPM  DBE  1-FIT  Formula
283.0801  283.0793  0.8  2.8  10.5  746.4  C17 H15 O2 S
**LC/MS REPORT**

Data file : C:\EZKDATA\03-28-18\GVK-CBK-1--. Vial position: Vial 57
Instrument Name: ANL-MCL2-LCMS-002
Injection Date: 28-Mar-2018 03:40:11 PM Injection Vol: 4.000
Sample Name: GVK-CBK-1-76

Acq. Method Conditions: RHN X-Bridge 5.0 Min
COLUMN: X-BRIDGE C18 (4.6mm x 75mm) 3.5 μm
MOBILE PHASE A: 10mM Ammonium Acetate in water, B 100% ACN
Gradient: % of B 0.0/10,0.2/10,2.5/75,3.0/100,4.0/100,5.0/10
Column Temp: 35°C
Flow rate: 2.0 ml/min

<table>
<thead>
<tr>
<th>PEAK No</th>
<th>RT min</th>
<th>Area</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2.321</td>
<td>85,025</td>
<td>5.777</td>
</tr>
<tr>
<td>2</td>
<td>3.137</td>
<td>1295.772</td>
<td>88.041</td>
</tr>
<tr>
<td>3</td>
<td>3.790</td>
<td>90,990</td>
<td>6.182</td>
</tr>
</tbody>
</table>

**Graphs**

1. DAD1 B, Sig=215,4 Ref-off (03-28-18;GVK-CBK-1-76 2D472_D - 03 28-18 BLANK 02435.D)
2. PSD TIC, MS File (C:\EZKDATA\03-28-18;GVK-CBK-1-76 2D472.D) MM-ESI+APCI, Pos, Scan, Frag 70, **+VE**
Elemental Composition Report

Single Mass Analysis (displaying only valid results)
Tolerance = 1000.0 PPM / DBE: min = -1.5, max = 50.0
Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions
15 formula(e) evaluated with 1 results within limits (up to 1 best isotopic matches for each mass)

Elements Used:
C: 0-16 H: 0-12 O: 0-3 S: 0-1 Cl: 0-1

Date of analysis: 11-Apr-2018 16:36:29
Instruments: Q-TOF Q-TOF ES+

C16H12O3S Cl

Comment: Error
No of Rows: Resolution:
Resolution:

Date: 8/2/2018 11:03:32 PM
User: Admin

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Elemental Composition Report

Single Mass Analysis (displaying only valid results)
Tolerance = 1000.0 PPM / DBE: min = -1.5, max = 50.0
Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions
10 formula(s) evaluated with 1 results within limits (up to 1 closest results for each mass)
Elements Used:
C: 0.16  H: 0.12  O: 0.2  F: 0.1  S: 0.1

511712A8071 8 (0.326) AM (Con,H, 50.00, Ar,E,0.196,12,1.60,LS 8); Sn (Sb, 0.00); Sb (1.40,00)
287.0553

Minimum: 5.0  1000.0  50.0
Maximum: 1.1  3.8  10.5  904.9  C16 H12 O2 P S

Date of analysis: 08-Dec-2016:13:36
Instrument ID: ANL-1MC2,LCMS-001
1: TCF MS ES+ 6.00e2

Comment: IN Khr
RoK-C61-1-PhD-50
Resolution: User: Admin
Acquisition: Due:12/22/2017 12:26:31 PM
Analytical Research And Development

Date of Analysis: 29-Aug-2017 02:14:19

Time | Height | Area | Area%  
--- | --- | --- | ---  
0.74 | 5970 | 159.72 | 0.43  
1.26 | 6718 | 557.25 | 0.55  
1.66 | 27188 | 555.92 | 1.46  
1.84 | 7464 | 193.15 | 0.57  
2.05 | 5409 | 120.88 | 0.32  
2.20 | 20218 | 443.08 | 1.18  
2.25 | 1459589 | 35168.39 | 93.91  
2.31 | 5857 | 249.02 | 0.66  
2.63 | 22415 | 458.06 | 1.09  

Column - ACQUITY UPLC BEH C18 1.7μm, 2.1x50mm  
Mobile phase-B: 0.1% FA IN H2O, A: 0.1% FA IN ACN  
1% of B: 0.97 0.97 2.72 2.52 2.3 2.19 0.97  
Flow: 0.6 mL/min,  
Temp: 35°C
Single Mass Analysis (displaying only valid results)
Tolerance = 1000.0 PPM / DBE: min = -1.5, max = 50.0
Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions
7 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)

Elements Used:
C: 0.17  H: 0.15  O: 0.3  S: 0.1

511712A6573 9 (0.370) AM (Cen,2, 50.00, Ar,5.0, 195.13, 1.00, LE 50; Sm (5x2.00); Sb (1,45.00 )

299.0740

Minimum:
MAXIMUM:

Mass    Calc. Mass  mDa  PPM  DBE  1-FIT  Formula
299.0749 299.0742  0.7  2.3  10.5  939.5  C17 H15 O3 S
Elemental Composition Report

Single Mass Analysis
Tolerance = 1000.0 PPM / DBE: min = -1.5, max = 50.0
Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions
3 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)
Elements Used:
C: 0-20  H: 0-15  O: 0-2  S: 0-1

1102383638 7 (0.265) AM (Cen,2, 50,00, Ar,5,0,195,10,0,LS S); Sm (Mn, 5x2,00,); Sb (1,4,0,0,); Cm (5,1,0)
519.0796  319.0793  0.5  1.6  13.5  549.2  C20 H15 O2 S

Date of analysis: 16-Feb-2018 10:26:05
Instrument: ANL-MC13 LCMS001
1-TOF MS ES+
7.1264

Min:  5.0  1000.0  50.0
Max: -1.5

Calc. Mass  mDa  PPM  DBE  1-FIT  Formula
319.0796  319.0793  0.5  1.6  13.5  549.2  C20 H15 O2 S

Page 1

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Elemental Composition Report

Single Mass Analysis (displaying only valid results)
Tolerance = 1000.0 PPM  /  DBE: min = -1.5, max = 50.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
3 formula(s) evaluated with 1 results within limits (up to 1 closest results for each mass)

Elements Used:
C: 0.22  H: 0.17  O: 0.2  S: 0.1

GVK Bio sciences (p) Ltd
Analytical Research and Development

Date of analysis: 06-Dec-2017 16:13
Instrum used I.D: AML-MCS-001
2: TOF MS ES+
345.0941  S11

Minimum:  5.0  1000.0  50.0
Maximum:
Mass  Calc. Mass  mDa  PPM  DBE  i-FIT  Formula
345.0941  345.0949  -0.8  -2.3  10.5  106.0  C22 H17 O2 S

Comments: IN-258
GVK Bio - I Proj 39

No. of Scans:  
Resolution:  
Apcrization:  

User  Admin

Date 17/11/2013 11:59:31 AM

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**LC/MS REPORT**

Data file: \C:\EZXDATA\03-27-18\GVK-CBK-1-77-02410.D
Injection Date: 27-Mar-2018 08:25:15 PM  Vial position: Vial 84
Sample Name: BIAT-005554C(BI01700605)  Instrument Name:ANL-MIL2-LCMS-6
Injection Vol:: 4.000

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GVK_LCMS_19

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**DAD1 B, Sig=215,4 Ref=off (03-27-16/GVK-CBK-1-77-02410/D - 03-23-16/BLANK-1-02195.D)**

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**MSD1 TIC, MS File (C:\EZXDATA/03-27-19/GVK-CBK-1-77-02410.D) MM+ES+APCI, Pos, Scan, Frag: 70 "+VE"**

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**MSD2 TIC, MS File (C:\EZXDATA/03-27-19/GVK-CBK-1-77-02410.D) MM+ES+APCI, Neg, Scan, Frag: 70 "-VE"**

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**DAD1 B, Sig=215,4 Ref=off**

<table>
<thead>
<tr>
<th>PEAK No</th>
<th>RT min</th>
<th>Area</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2.320</td>
<td>219.153</td>
<td>8.040</td>
</tr>
<tr>
<td>2</td>
<td>3.131</td>
<td>2479.407</td>
<td>90.966</td>
</tr>
<tr>
<td>3</td>
<td>3.560</td>
<td>27.080</td>
<td>0.994</td>
</tr>
</tbody>
</table>
# Elemental Composition Report

**Single Mass Analysis (displaying only valid results)**

**Tolerance** = 1000.0 PPM  /  **DBE** min = -1.5, max = 50.0  
**Selected filters:** None

**Monoisotopic Mass, Odd and Even Electron Ions**

22 formula(e) evaluated with 1 results within limits (up to 1 best isotopic matches for each mass)

<table>
<thead>
<tr>
<th>Elements Used:</th>
<th>C: 0.15</th>
<th>H: 0.9</th>
<th>O: 0.2</th>
<th>F: 0.1</th>
<th>S: 0.1</th>
<th>Cl: 0.1</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>GVK-CBK-177</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SI184793898.8 (0.325) AM (Cen, 2, 50.00, Ar, 5.0, 105.09, 1.00, LS 5); Sm (Sn, 5c2.00); Sn (1.40.00); Cm (4:16)</td>
<td>307.0003</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Date of analysis:** 11-Apr-2018 16:13:30  
**Instrument ID:** MCL3-MSMS-001  
**1: TOF MS ES+**  
**1.45e+4**

<table>
<thead>
<tr>
<th>Mass</th>
<th>Calc. Mass</th>
<th>mDa</th>
<th>PPM</th>
<th>DBE</th>
<th>i-FIT</th>
<th>Formula</th>
</tr>
</thead>
<tbody>
<tr>
<td>307.0003</td>
<td>306.9996</td>
<td>0.7</td>
<td>2.3</td>
<td>10.5</td>
<td>54.1</td>
<td>C15 H9 O2 F S Cl</td>
</tr>
</tbody>
</table>

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**Graphical Representation:**

- **4o**

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**Notes:**

- **Resolution:**
- **Apodization:**
- **Date:** 4/2/2018 16:13:31 PM
- **User:** Admin

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Elemental Composition Report

Single Mass Analysis (displaying only valid results)
Tolerance = 1000.0 PPM / DBE: min = -1.5, max = 50.0
Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions
7 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)
Elements Used:
C: 0-17 H: 0-15 O: 0-3 S: 0-1

Date of analysis: 08-Dec-201716:00:26
Instrument ID:ANL-MCL3-LCMS-001
1. TOF MS ES+

Min: 5.0 1000.0 50.0
Max: 5.0 1000.0 50.0

Mass  Calc. Mass  mDa  PPM  DBE  i-FIT  Formula
299.0753 299.0742  1.1  3.7  10.5  999.6  C17 H13 O3 S

Comment: IN Khr
Rev.: PKH
A2-10477