Synthesis and bioactivity investigation of benzophenone and its derivatives

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1 General procedures and NMR data of compounds

1.1 The synthesis of compound **1**

Compound **s3** (100 mg, 0.37 mmol), 2-chloroacetyl chloride (56 mg, 0.49 mmol), triethylamine (0.08 mL), a small amount of 4-dimethylaminopyridine, and anhydrous 1,4-dioxane (5 mL) were added to a test tube in sequence. The reaction liquid was heated to 100 °C, stirred for 30 min, and cooled; then, few drops of water were added to quench the reaction, and the reaction liquid was poured into an appropriate amount of water. The organic portion was extracted with ethyl acetate and dried with anhydrous Na_2SO_4. The solvent was removed by evaporation under reduced pressure to obtain the crude product, which was purified by medium-pressure preparative chromatography with ethyl acetate/petroleum ether to afford compound **1** (40 mg, 31%).

5-(2-Hydroxy-3,5-dimethylbenzoyl)-2-methoxyphenyl 2-chloroacetate (**1**): grass green solid, mp: 103-105 °C; IR (KBr) ν_{max} 639, 789, 816, 831, 936, 1016, 1128, 1262, 1276, 1345, 1511, 1606, 1627, 1782, 2958, 2999, 3436 cm^{-1}; ^1H NMR (500 MHz, acetone-d_6): δ 2.23 (s, 6H, 3'-CH_3, 5'-CH_3), 3.97 (s, 3H, CH_3O-), 4.61 (s, 2H, -CH_2-), 7.29 (s, 1H, H-4'), 7.31 (s, 1H, H-6'), 7.31 (d, 1H, J = 8.5 Hz, H-3), 7.56 (d, 1H, J = 2.2 Hz, H-2), 7.70 (dd, 1H, J = 2.2, 8.5 Hz, H-4), 11.89 (s, 1H, 2'-OH); ^{13}C NMR (126 MHz, acetone-d_6): δ 15.5, 20.4, 41.4, 56.8, 113.0, 119.0, 124.9, 127.6, 128.0, 130.5, 131.3 (C-5, C-5'), 138.9, 140.0, 155.4, 159.9, 166.2, 200.0; HRMS(ESI) m/z calcd for $C_{18}H_{17}ClNaO_5$ $[M + Na]^+$ 371.0657, found 371.0663.

1.2 The synthesis of compound **2**

Compound **s3** (100 mg, 0.37 mmol), ethyl 2-chloroacetate (79 μL, 0.74 mmol), anhydrous K_2CO_3 (51 mg, 0.37 mmol), and acetone (10 mL) were added to a test tube in turn, heated to 50 °C, stirred for 24 h, and cooled; then, the mixture was poured into an appropriate amount of water. The organic portion was extracted with ethyl acetate and dried with anhydrous Na_2SO_4. The solvent was removed by evaporation under reduced pressure to obtain the crude product. Finally, the crude product was purified by medium-pressure preparative chromatography with ethyl acetate/petroleum ether to afford compound **2** (76 mg, 57%).

Ethyl 2-(5-(2-hydroxy-3,5-dimethylbenzoyl)-2-methoxyphenoxy)acetate (**2**): grass green solid, mp: 90-93 °C; IR (KBr) ν_{max} 459, 781, 871, 1009, 1136, 1186, 1252, 1332, 1437, 1581, 1627, 1756,

2916, 3614 cm^{-1}; ^1H NMR (500 MHz, acetone-d_6): δ 1.22 (t, 3H, J = 7.2 Hz, -CH_3), 2.04 (m, acetone-d_6), 2.22 (s, 3H, 3''-CH_3), 2.24 (s, 3H, 5''-CH_3), 2.81 (H_2O), 3.96 (s, 3H, -OCH_3), 4.80 (s, 2H, -CH_2-), 7.16 (d, 1H, J = 8.4 Hz, H-3'), 7.27 (s, 1H, H-4''), 7.30 (d, 1H, J = 2.0 Hz, H-6'), 7.31 (s, 1H, H-6''), 7.40 (dd, 1H, J = 2.0, 8.4 Hz, H-4'), 12.0 (s, 1H, 2''-OH); ^{13}C NMR (126 MHz, acetone-d_6): δ 14.4, 15.5, 20.4, 56.3, 61.4, 66.6, 112.2, 112.6, 116.5, 119.1, 120.3, 125.7, 126.8, 127.9, 131.5, 138.6, 148.1, 154.2, 169.4, 191.2; HRMS(ESI) m/z calcd for $C_{20}H_{23}O_6$ $[M + H]^+$ 359.1489, found 359.1494.

1.3 The synthesis of compound **3**

Compound **2** (65 mg, 0.2 mmol), hydrochloric acid (0.48 mmol), and tetrahydrofuran (10 mL) were successively added to a test tube, heated to reflux, and stirred for 6 to 24 h. The reaction was monitored by thin layer chromatography (TLC). After the disappearance of the raw material, the reaction was stopped and the mixture was cooled; then, the solvent was removed by evaporation under reduced pressure to obtain the crude product. The latter was purified by medium-pressure preparative chromatography with ethyl acetate/petroleum ether to afford compound **3** (61 mg, 85%).

2-(5-(2-Hydroxy-3,5-dimethylbenzoyl)-2-methoxyphenoxy)acetic acid (**3**): yellow solid, mp: 140-142 °C; IR (KBr) ν_{max} 624, 759, 786, 1131, 1262, 1349, 1512, 1607, 1624, 1707, 1738, 2849, 2919, 3446 cm^{-1}; ^1H NMR (500 MHz, acetone-d_6): δ 2.04 (m, acetone-d_6), 2.22 (s, 6H, -CH_3 × 2), 3.92 (s, 3H, CH_3O-), 4.82 (s, 2H, -CH_2-), 7.16 (d, 1H, J = 8.2 Hz, H-3'), 7.26 (s, 1H, H-4''), 7.31 (s, 1H, H-6''), 7.32 (d, 1H, J = 2.0 Hz, H-6'), 7.40 (dd, 1H, J = 2.0, 8.2 Hz, H-4'), 12.0 (s, 1H, 2''-OH); ^{13}C NMR (126 MHz, acetone-d_6): δ 15.5, 20.4, 56.3, 66.1, 112.2, 116.2, 119.1, 125.6, 127.4, 127.9, 131.1, 131.5, 138.6, 148.0, 154.2, 159.8, 170.1, 200.8; HRMS(ESI) m/z calcd for $C_{18}H_{19}O_6$ $[M + H]^+$ 331.1176, found 331.1167.

1.4 The synthesis of compound **4**

Compound **s3** (150 mg, 0.37 mmol), 4-chloro-*N*-methylpicolinamide (**s4**, 116 mg, 0.66 mmol), anhydrous K_2CO_3 (92 mg, 0.66 mmol), and dimethyl sulfoxide (5 mL) were added to a test tube in turn, heated to 130 °C, stirred for 1.5 h, and cooled; then, the mixture was poured into an

appropriate amount of water. The organic portion was extracted by ethyl acetate, combined with the organic layers, washed with saturated salt water (10 mL × 3), and dried with anhydrous Na_2SO_4. The solvent was removed by evaporation under reduced pressure to obtain the crude product. The latter was purified by medium-pressure preparative chromatography with ethyl acetate/petroleum ether to afford compound **4** (32 mg, 21%).

4-(5-(2-Hydroxy-3,5-dimethylbenzoyl)-2-methoxyphenoxy)-*N*-methylpicolinamide (**4**): light yellow solid, mp: 137-139 °C; IR (KBr) ν_{max} 566, 624, 787, 811, 936, 1018, 1118, 1223, 1294, 1423, 1510, 1532, 1568, 1599, 1679, 1736, 2929, 3392 cm^{-1}; ^1H NMR (500 MHz, acetone-d_6): δ 2.04 (m, acetone-d_6), 2.21 (s, 3H, 4”-CH_3), 2.22 (s, 3H, 6”-CH_3), 2.83 (H_2O), 2.92 (d, 3H, J = 5.0 Hz, -NCH_3), 3.91 (s, 3H, CH_3O-), 7.08 (dd, 1H, J = 2.6, 5.6 Hz, H-5), 7.28 (s, 1H, H-4’’), 7.34 (s, 1H, H-6’’), 7.41 (d, 1H, J = 8.6 Hz, H-3’), 7.53 (d, 1H, J = 2.6 Hz, H-3), 7.61 (d, 1H, J = 2.1 Hz, H-6’), 7.77 (dd, 1H, J = 2.1, 8.6 Hz, H-4’), 8.31 (s, 1H, -NH-), 8.45 (d, 1H, J = 5.6 Hz, H-6), 11.89 (s, 1H, 2’’-OH); ^{13}C NMR (126 MHz, acetone-d_6): δ 15.5, 20.4, 26.2, 56.7, 109.3, 113.8, 114.1, 119.1, 125.0, 127.5, 128.1, 130.2, 131.2, 132.1, 138.9, 142.2, 150.9, 153.7, 155.9, 159.9, 164.7, 166.8, 200.0; HRMS(ESI) m/z calcd for $C_{23}H_{23}N_2O_5$ $[M + H]^+$ 407.1601, found 407.1607.

1.5 General procedure for the synthesis of compounds **5**-**7**

Phosphorus pentoxide (0.29 g, 2.0 mmol) and methanesulfonic acid (10 mL) were added to a test tube in turn, heated to 90 °C and stirred to dissolve them; then, phenol (2.0 mmol) and cinnamic acid compound (2.0 mmol) were added in turn and stirred at 90 °C for 1 h. After the reaction was completed, the reaction liquid was cooled and poured into an appropriate amount of water to precipitate a viscous fluid. Then, the viscous fluid was extracted with ethyl acetate (10 mL×3) and dried with anhydrous Na_2SO_4. The solvent was removed by evaporation under reduced pressure to obtain the crude product, which was purified by medium-pressure preparative chromatography with ethyl acetate/petroleum ether to afford compounds **5**–**7**.

4-(6-Isopropyl-2-oxochroman-4-yl)-2-methoxyphenyl methanesulfonate (**5**): orange-red solid (228 mg, 35%), mp: 94-96 °C; IR (KBr) ν_{max} 516, 550, 673, 855, 972, 1028, 1115, 1179, 1366, 1505, 1602, 1764, 2871, 2963, 3017 cm^{-1}; ^1H NMR (500 MHz, acetone-d_6): δ 1.16 (d, 3H, J = 3.0 Hz, CH_3-), 1.18 (d, 3H, J = 3.0 Hz, CH_3-), 2.04-2.05 (m, acetone-d_6), 2.83-2.89 (m, 1H, tertiary H in

isopropyl), 2.87 (H_2O), 3.11 (dd, 1H, J = 6.2, 14.0 Hz, Ha in -CH_2-), 3.17 (dd, 1H, J = 6.2, 14.0 Hz, Hb in -CH_2-), 3.24 (s, 3H, CH_3SO_3-), 3.88 (s, 3H, CH_3O-2), 4.54 (t, 1H, J = 6.2 Hz, H-4'), 6.76-6.78 (dd, 1H, J = 2.2, 8.4 Hz, H-5), 7.04 (d, 1H, J = 8.4 Hz, H-6), 7.05 (d, 1H, J = 2.2 Hz, H-3), 7.14 (d, 1H, J = 2.2 Hz, H-5'), 7.23-7.26 (dd, 1H, J = 2.2, 8.4 Hz, H-7'), 7.25 (d, 1H, J = 8.4 Hz, H-8'); ^{13}C NMR (125 MHz, acetone-d_6): δ 24.3 (2 CH_3-), 34.2, 37.3, 38.5 (CH_3SO_3-), 41.1 (chiral C-4'), 56.5 (CH_3O-2), 113.5, 117.5, 120.3, 125.3, 126.2, 127.2, 127.5, 138.4, 142.9, 146.0, 150.9, 152.8, 167.8; HRMS(ESI) m/z calcd for $C_{20}H_{23}O_6S$ $[M + H]^+$ 391.1210, found 391.1201.

6-Ethyl-4-(4-hydroxy-3,5-dimethoxyphenyl)chroman-2-one (**6**): orange-red solid (203 mg, 31%), mp: 94-96 °C; IR (KBr) ν_{max} 529, 828, 1012, 1093, 1147, 1201, 1259, 1316, 1464, 1482, 1591, 1698, 1765, 2845, 2937, 2964, 3005 cm^{-1}; ^{1}H NMR (500 MHz, acetone-d_6): δ 1.12 (t, 3H, J = 7.6 Hz, CH_3-), 2.52-2.57 (q, 2H, J = 7.6 Hz, -CH_2-), 3.05 (d, 1H, J = 6.1 Hz, Ha in -CH_2-3), 3.06 (d, 1H, J = 7.5 Hz, Hb in -CH_2-3), 3.75 (s, 6H, CH_3O-3', CH_3O-5'), 4.36 (t, 1H, J = 7.5 Hz, H-4), 6.53 (s, 2H, H-2', H-6'), 6.93 (d, 1H, J = 1.9 Hz, H-5), 6.99 (d, 1H, J = 8.3 Hz, H-8), 7.15-7.17 (dd, 1H, J = 1.9, 8.3 Hz, H-7), 7.28 (s, 1H, HO-4'); ^{13}C NMR (125 MHz, acetone-d_6): δ 16.2, 28.7, 37.6, 41.2, 56.6 (CH_3O-3', CH_3O-5'), 106.0 (C-2', C-6'), 117.2, 127.4, 128.5, 128.6, 132.4, 136.1, 141.2, 148.9, 150.8 (C-3', C-5'), 168.2; HRMS(ESI) m/z calcd for $C_{19}H_{21}O_5$ $[M + H]^+$ 329.1384, found 329.1389. Simultaneously, the rotating frame overhauser effect spectroscopy (ROESY) experiment of compound **6** was performed in order to clarify its structural elucidation. In the ROESY spectrum, H-5 coupled with H-2', H-6', and -CH_2- in ethyl group, while H-4 was coupled with H-2' and H-6', and -CH_2-3 was coupled with H-2', H-6', and H-4. In addition, in this spectrum, H-2' was coupled with CH_3O-3', and H-6' was coupled with CH_3O-5'. Therefore, the analysis of these spectra was coordinated.

4-(6-Ethyl-2-oxochroman-4-yl)-2,6-dimethoxyphenyl methanesulfonate (**7**): white solid (103 mg, 15%), mp: 168-171 °C; IR (KBr) ν_{max} 511, 865, 975, 1130, 1152, 1202, 1240, 1354, 1426, 1465, 1493, 1605, 1764, 2855, 2938, 2962, 3019, 3031 cm^{-1}; ^{1}H NMR (500 MHz, acetone-d_6): δ 1.14 (t, 3H, J = 7.6 Hz, CH_3-), 2.03-2.05 (m, acetone-d_6), 2.55-2.59 (q, 2H, J = 7.6 Hz, -CH_2-), 2.87 (H_2O), 3.13-3.14 (dd, 2H, J = 0.8, 6.7 Hz, -CH_2-3'), 3.30 (s, 3H, CH_3SO_3-), 3.80 (s, 6H, CH_3O-2, CH_3O-6), 4.49 (t, 1H, J = 6.8 Hz, H-4'), 6.67 (s, 2H, H-3, H-5), 6.98 (d, 1H, J = 1.5 Hz, H-5'), 7.02 (d, 1H, J = 8.3 Hz, H-8'), 7.19-7.21 (dd, 1H, J = 1.5, 8.3 Hz, H-7'); ^{13}C NMR (125 MHz, acetone-d_6): δ 16.2, 28.7, 37.2, 40.2 (CH_3SO_3-), 41.5 (C-4'), 56.5 (2 CH_3O-), 105.4 (C-3, C-5), 117.4, 126.4,

128.5, 129.0, 141.4 (C-4, C-4'a), 142.1, 150.9, 154.4 (C-2, C-6), 167.9; HRMS(ESI) m/z calcd for $C_{20}H_{23}O_7S$ $[M + H]^+$ 407.1159, found 407.1166.

1.6 The synthesis of compound **8**

Compound **s3** (136 mg, 0.50 mmol), sodium acetate (58 mg, 0.70 mmol), and acetic anhydride (6 mL) were added to a test tube in turn, heated to 130 °C, stirred for 2 h, and cooled; then, ethyl acetate (35 mL) was added to the test tube. The combined organic layers were then washed with brine (3 × 10 mL) and finally dried over anhydrous Na_2SO_4. The solvent was removed by evaporation under reduced pressure to obtain the crude product. The latter was purified by medium-pressure preparative chromatography with ethyl acetate/petroleum ether to afford compound **8** (99 mg, 59%).

5-(6,8-Dimethyl-2-oxo-2*H*-chromen-4-yl)-2-methoxyphenyl acetate (**8**): grass green solid, mp: 140-142 °C; IR (KBr) ν_{max} 474, 520, 635, 728, 936, 1017, 1122, 1196, 1262, 1280, 1347, 1472, 1514, 1624, 1767, 2850, 2919, 3022, 3421 cm^{-1}; ^1H NMR (500 MHz, acetone-d_6) : δ 2.03-2.05 (m, acetone-d_6), 2.23 (s, 6H, CH_3-6', CH_3-8'), 2.28 (s, 3H, CH_3C=O), 2.81 (H_2O), 3.95 (s, 3H, CH_3O-2), 7.27 (d, 1H, J = 8.6 Hz, H-3), 7.28 (s, 1H, H-7'), 7.39 (s, 1H, H-5'), 7.48 (d, 1H, J = 2.2 Hz, H-6), 7.66 (dd, 1H, J = 2.2, 8.6 Hz, H-4), 11.9 (s, 1H, H-3'); ^{13}C NMR (126 MHz, acetone-d_6): δ 15.5, 20.4 (C-6' and CH_3- in acetyl), 56.6, 112.7 (C-3, C-3'), 119.0, 125.2 (C-4, C-5'), 127.5, 128.0, 129.9, 131.2, 131.3, 138.8, 140.4, 155.7, 159.9, 169.0, 200.2; HRMS(ESI) m/z calcd for $C_{20}H_{19}O_5$ $[M + H]^+$ 339.1227, found 339.1224.

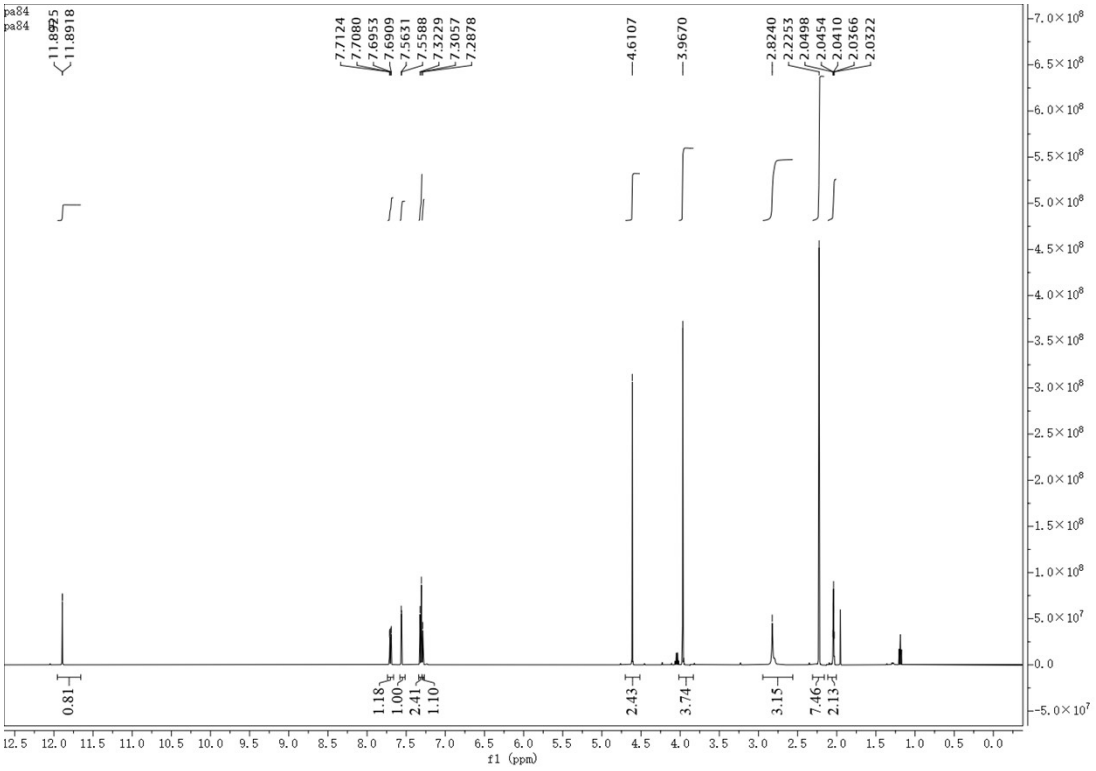
1.7 The synthesis of compound **9**

Compound **8** (60 mg, 0.18 mmol), hydrochloric acid (1.62 mmol), and ethanol (5 mL) were successively added to a test tube, heated to reflux and stirred for 5 h. After cooling, the reaction liquid was poured into a round-bottom flask, and the solvent was removed by evaporation under reduced pressure to obtain the crude product, which was purified by medium-pressure preparative chromatography with ethyl acetate/petroleum ether to afford compound **9** (52 mg, 98%).

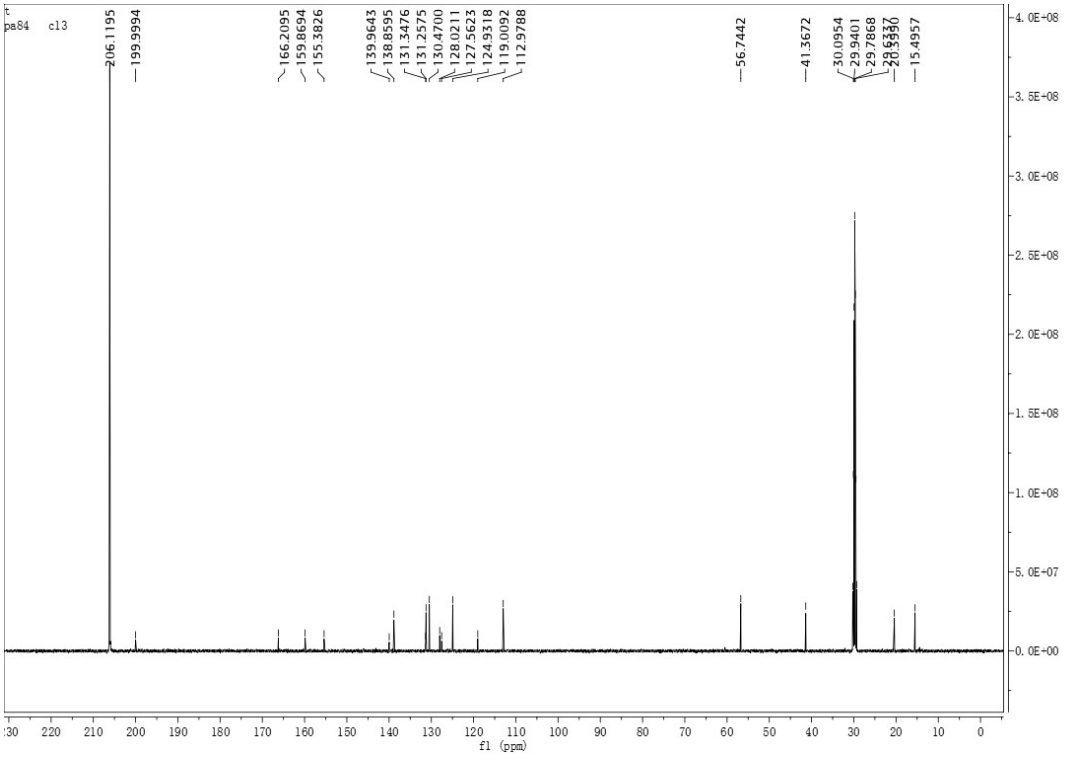
4-(3-Hydroxy-4-methoxyphenyl)-6,8-dimethyl-2*H*-chromen-2-one (**9**): green solid, 125-128 °C; IR (KBr) ν_{max} 547, 627, 789, 1020, 1134, 1214, 1272, 1361, 1508, 1560, 1618, 1726, 2850, 2916,

2975, 3420 cm^{-1}; ^{1}H NMR (500 MHz, acetone-d_6) : δ 2.03-2.05 (m, acetone-d_6), 2.23 (s, 6H, CH_3-6, CH_3-8), 2.82 (s, H_2O), 3.93 (s, 3H, CH_3O-4'), 7.10 (d, 1H, J = 8.0 Hz, H-5'), 7.23 (d, 1H, J = 2.1 Hz, H-2'), 7.24 (dd, 1H, J = 2.1, 8.0 Hz, H-6'), 7.27 (s, 1H, H-7), 7.33 (s, 1H, H-5), 8.08 (s, 1H, HO-3'), 12.0 (s, 1H, H-3); ^{13}C NMR (126 MHz, acetone-d_6): δ 15.5, 20.4, 56.3, 111.4, 116.8, 119.2, 122.8, 123.4, 127.4, 127.7, 128.1, 131.4, 131.8, 132.2, 138.5, 152.0, 159.9, 201.2; HRMS(ESI) m/z calcd for $C_{18}H_{17}O_4$ $[M + H]^+$ 297.1121, found 297.1126.

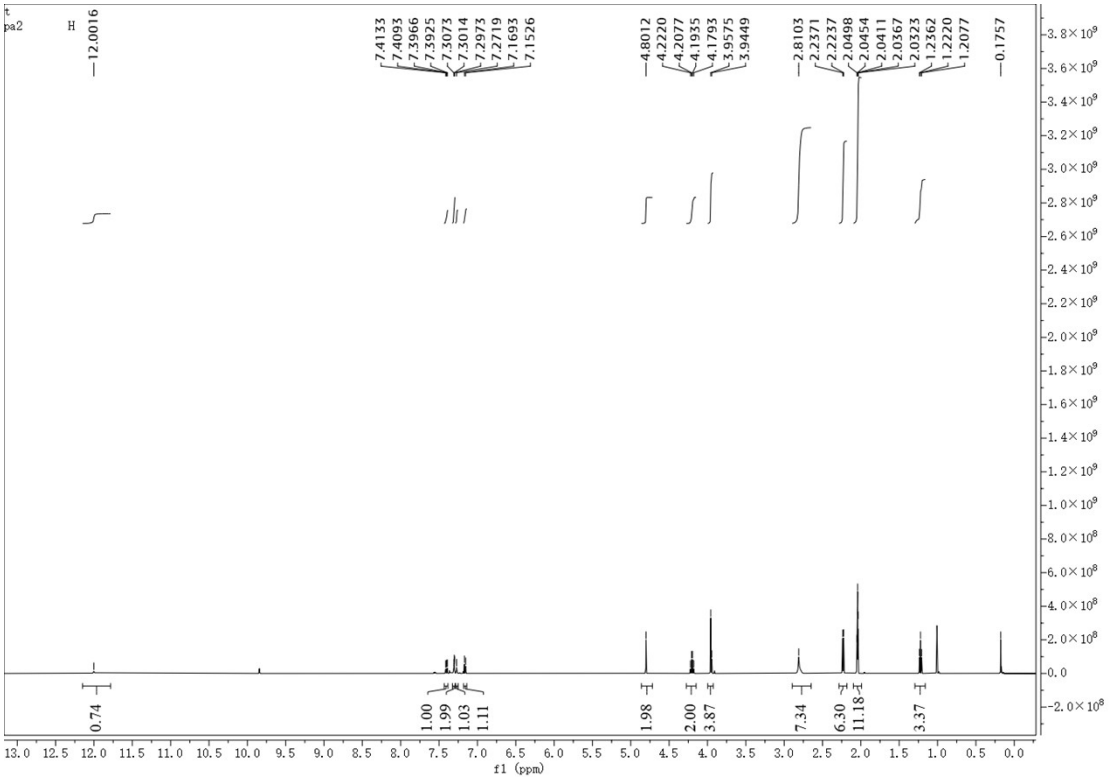
1. The ^{1}H NMR spectrum of 5-(2-hydroxy-3,5-dimethylbenzoyl)-2-methoxyphenyl 2-chloroacetate (acetone-d_6, 500 MHz) (**1**)



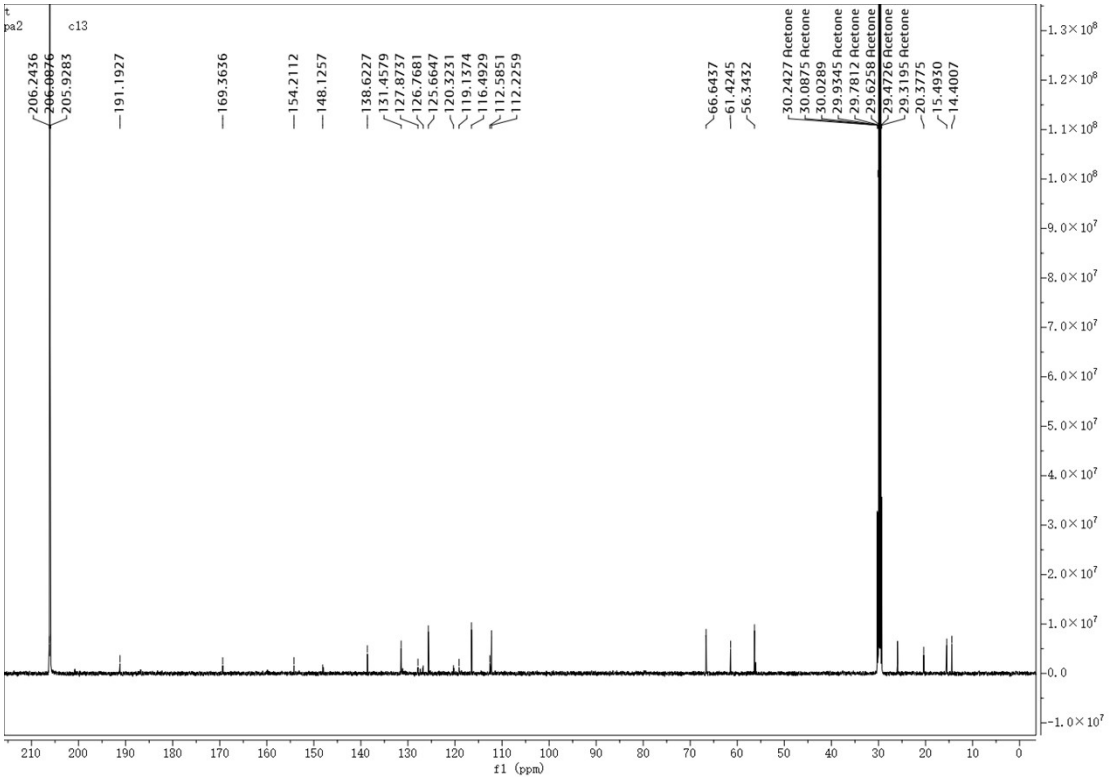
The ^{13}C NMR spectrum of 5-(2-hydroxy-3,5-dimethylbenzoyl)-2-methoxyphenyl 2-chloroacetate (acetone-d_6, 125 MHz) (**1**)



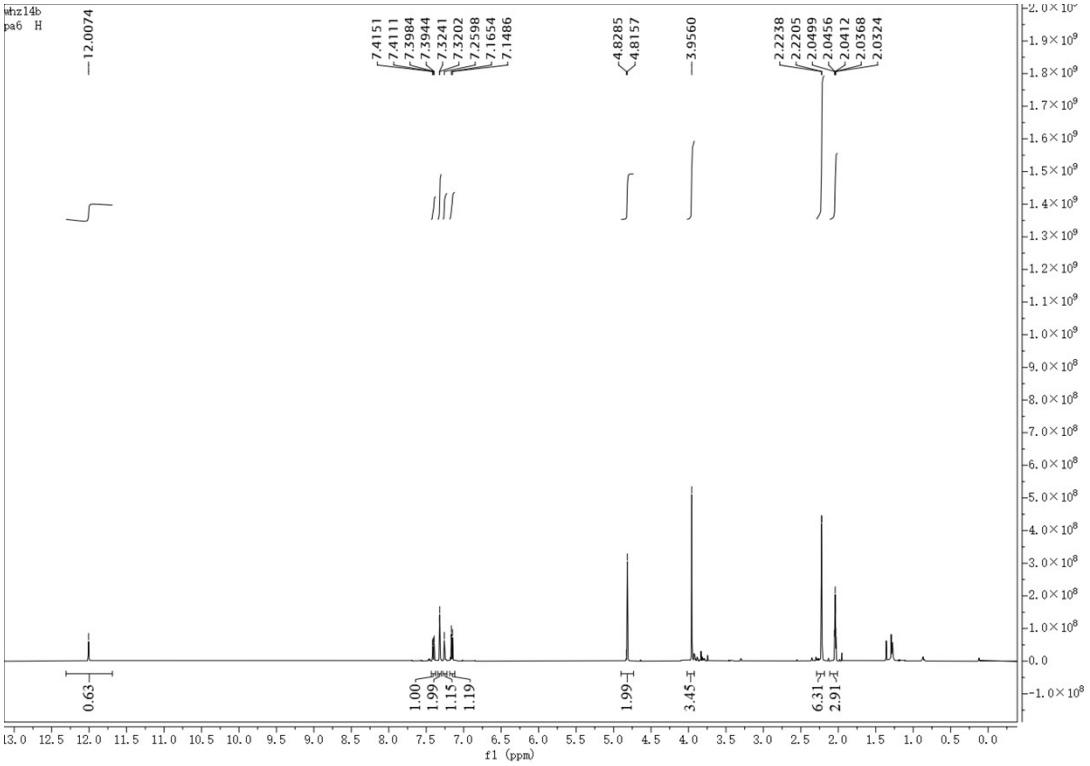
2. The ^{1}H NMR spectrum of ethyl 2-(5-(2-hydroxy-3,5-dimethylbenzoyl)-2-methoxyphenoxy)acetate (acetone-d_6, 500 MHz) (**2**)



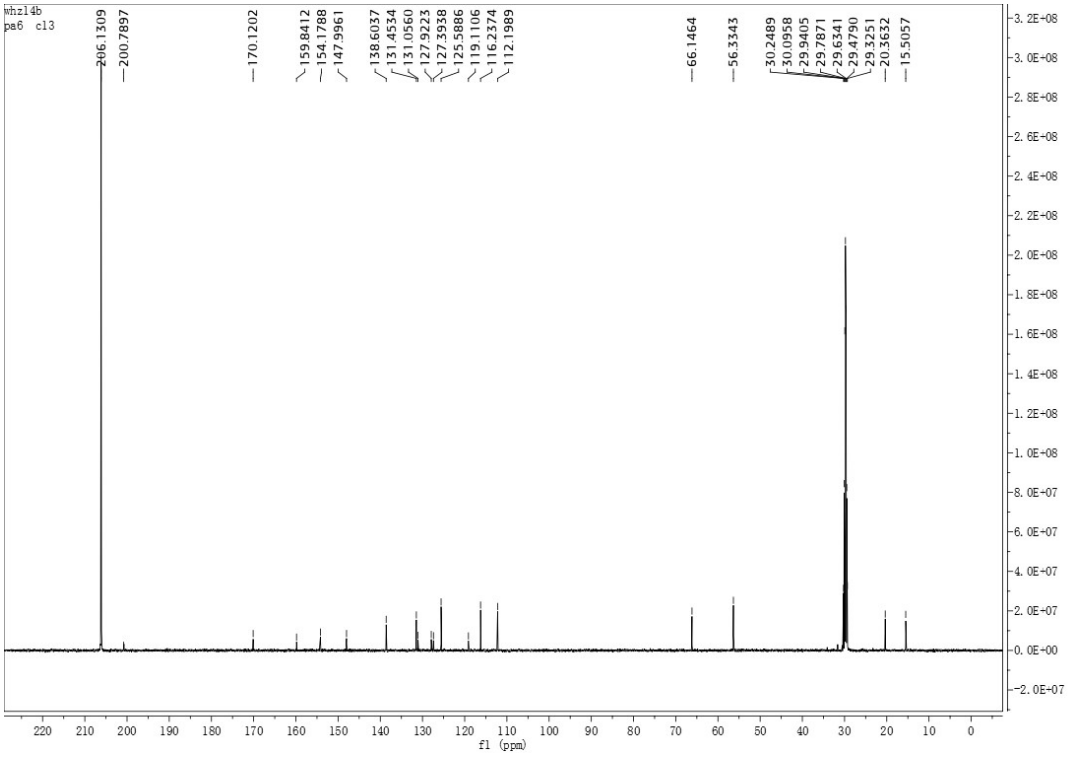
The ^{13}C NMR spectrum of ethyl 2-(5-(2-hydroxy-3,5-dimethylbenzoyl)-2-methoxyphenoxy)acetate (acetone-d_6, 125 MHz) (**2**)



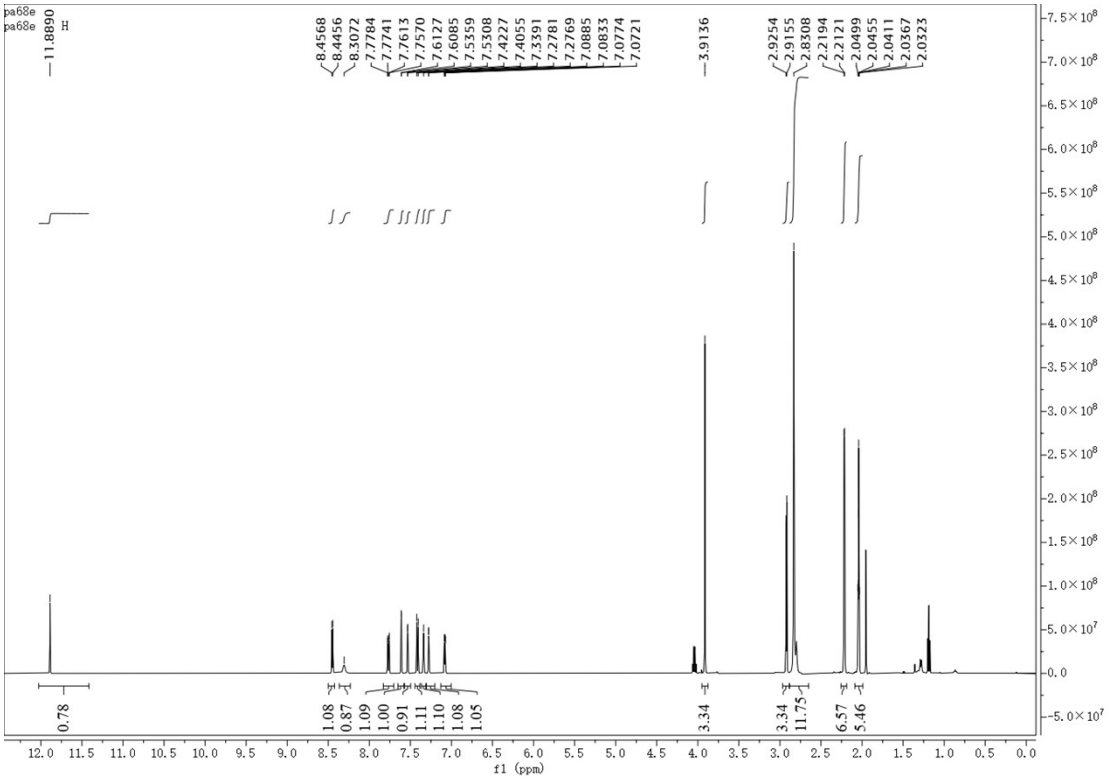
3. The ^{1}H NMR spectrum of 2-(5-(2-hydroxy-3,5-dimethylbenzoyl)-2-methoxyphenoxy)acetic acid (acetone-d_6, 500 MHz) (**3**)



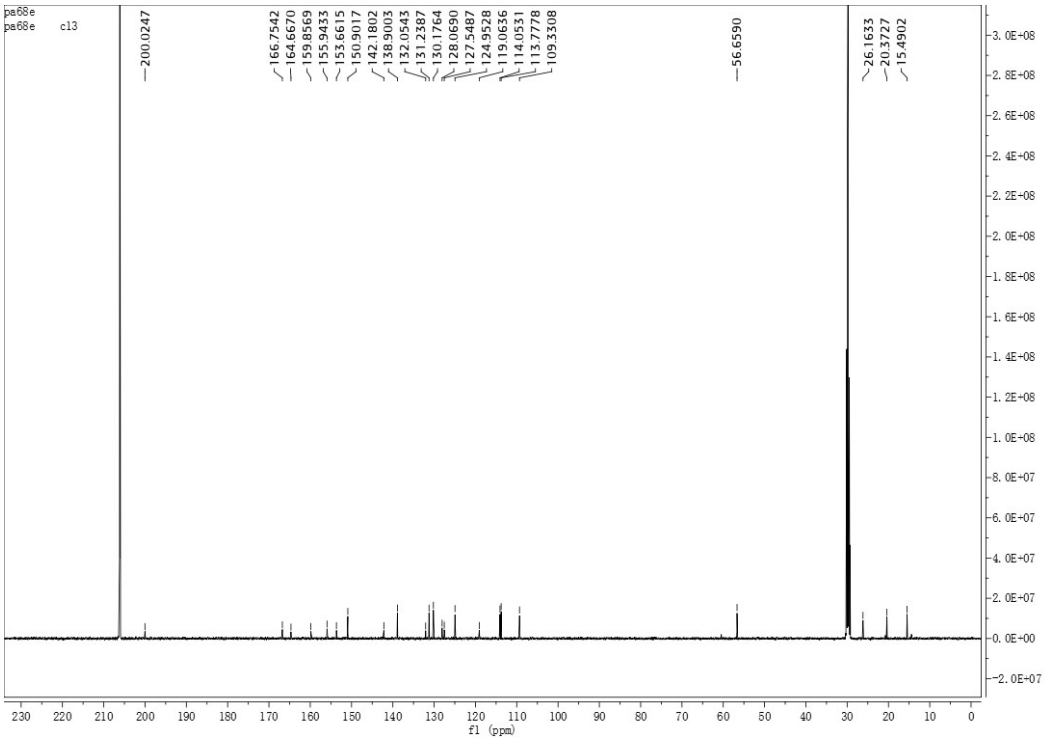
The ^{13}C NMR spectrum of 2-(5-(2-hydroxy-3,5-dimethylbenzoyl)-2-methoxyphenoxy)acetic acid (acetone-d_6, 125 MHz) (**3**)



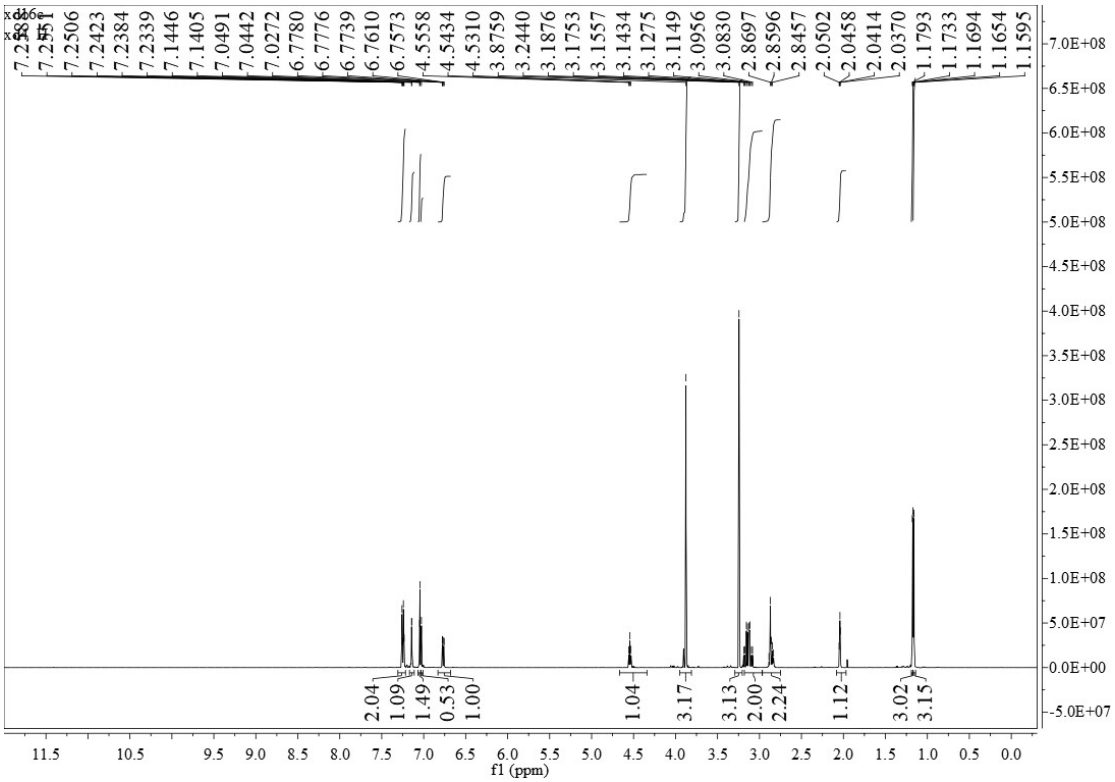
4. The ^{1}H NMR spectrum of 4-(5-(2-hydroxy-3,5-dimethylbenzoyl)-2-methoxyphenoxy)-*N*-methylpicolinamide (acetone-d_6, 500 MHz) (**4**)



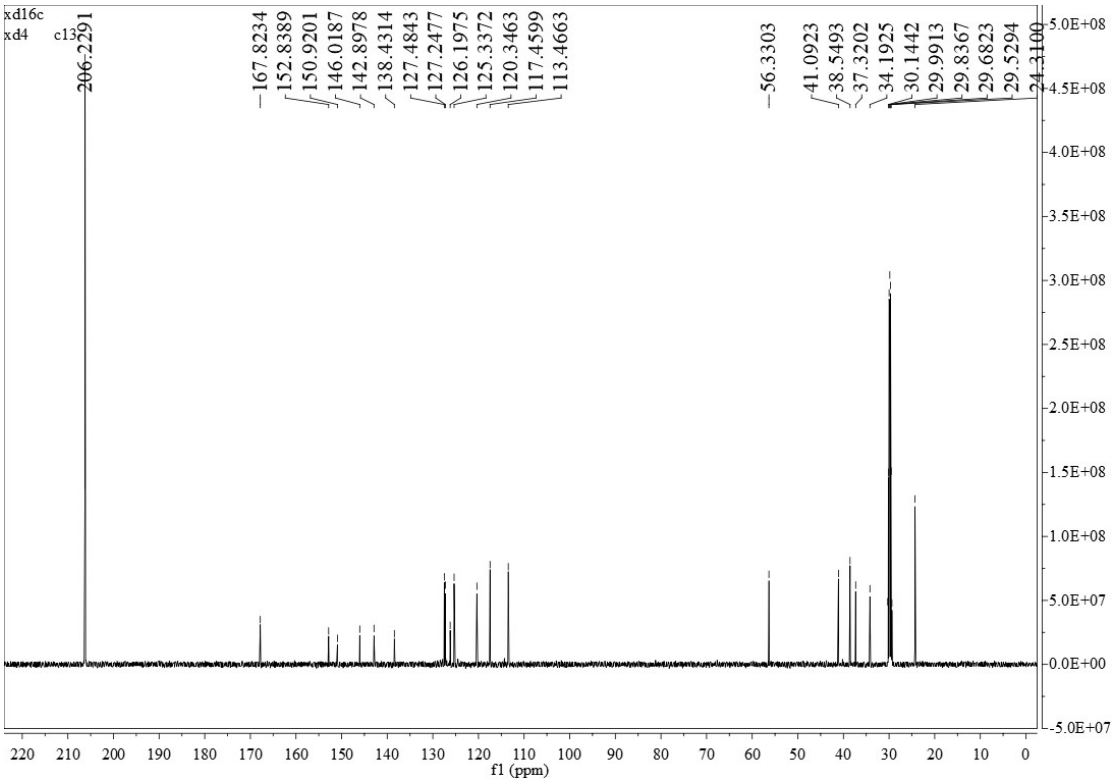
The ^{13}C NMR spectrum of 4-(5-(2-hydroxy-3,5-dimethylbenzoyl)-2-methoxyphenoxy)-*N*-methylpicolinamide (acetone-d_6, 125 MHz) (**4**)



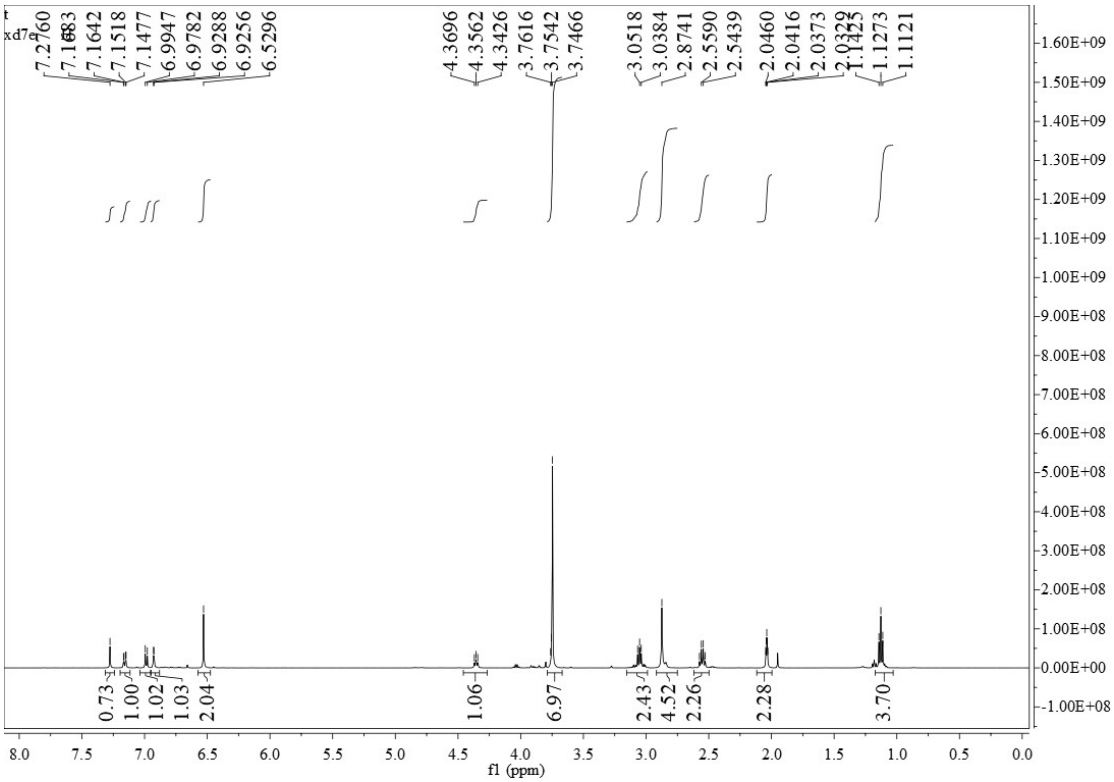
5. The ^{1}H NMR spectrum of 4-(6-isopropyl-2-oxochroman-4-yl)-2-methoxyphenyl methanesulfonate (acetone-d_6, 500 MHz) (**5**)



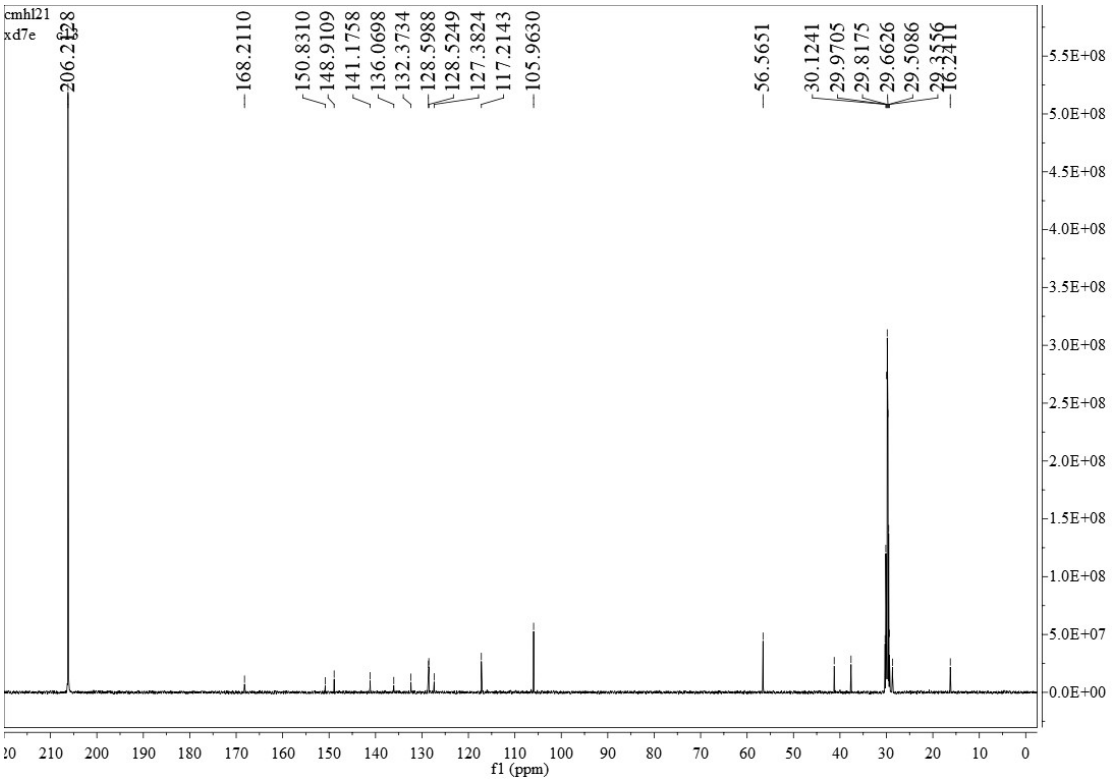
The ^{13}C NMR spectrum of 4-(6-isopropyl-2-oxochroman-4-yl)-2-methoxyphenyl methanesulfonate (acetone-d_6, 125 MHz) (**5**)



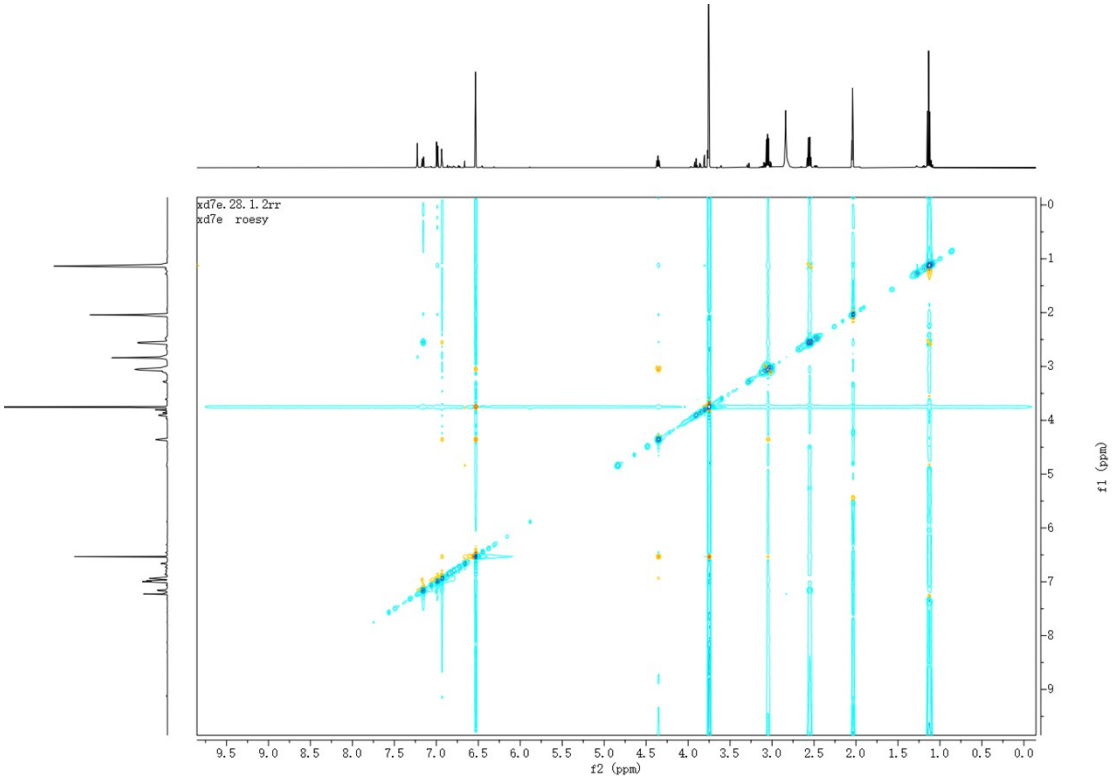
6. The ^{1}H NMR spectrum of 6-ethyl-4-(4-hydroxy-3,5-dimethoxyphenyl)chroman-2-one (acetone-d_6, 500 MHz) (**6**)



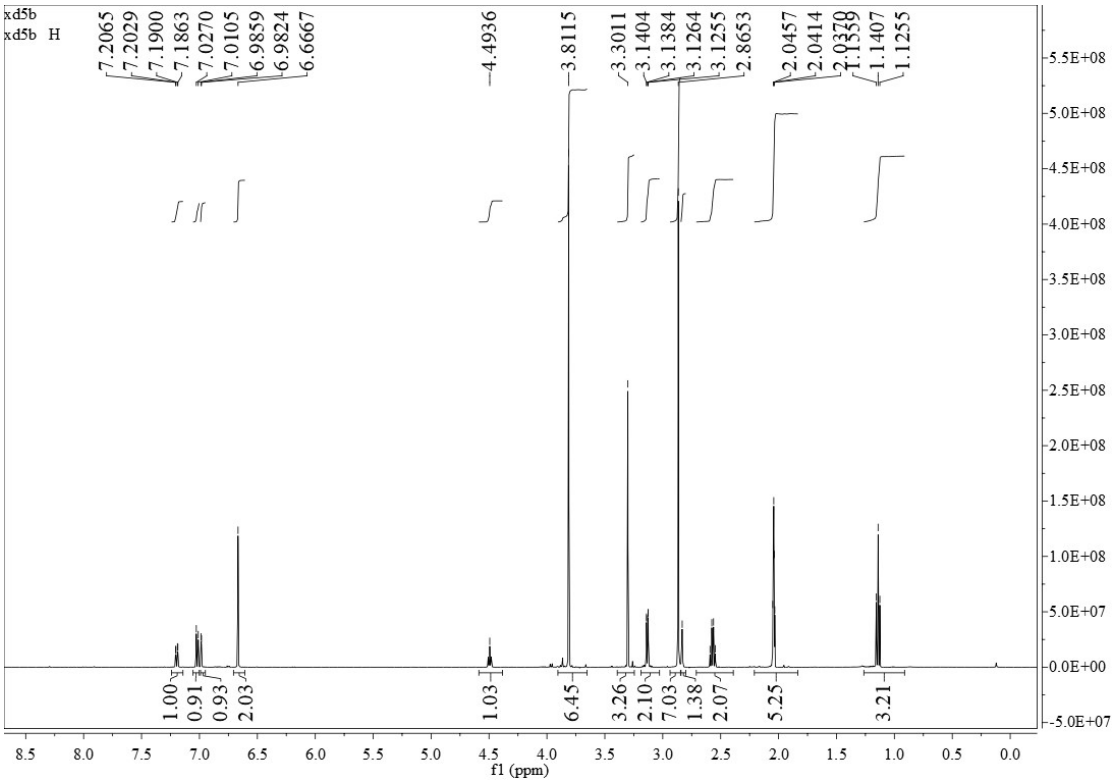
The ^{13}C NMR spectrum of 6-ethyl-4-(4-hydroxy-3,5-dimethoxyphenyl)chroman-2-one (acetone-d_6, 125 MHz) (**6**)



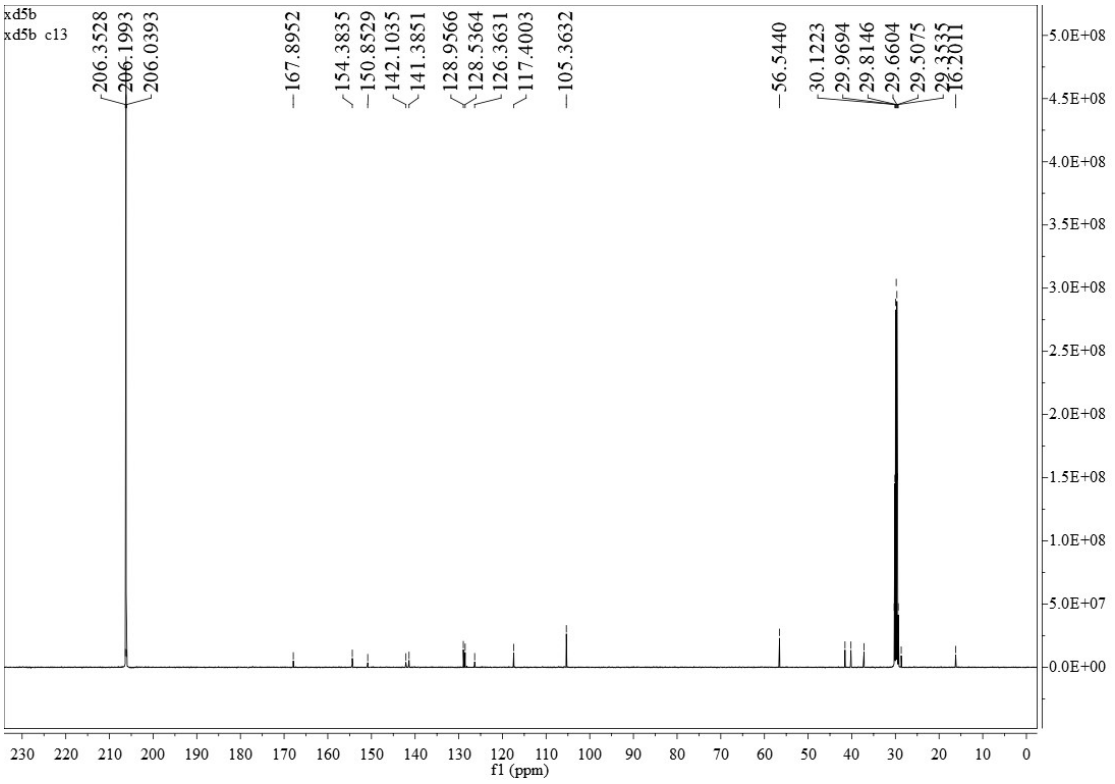
The ROESY spectrum of 6-ethyl-4-(4-hydroxy-3,5-dimethoxyphenyl)chroman-2-one (acetone-d_6, 500 MHz) (**6**)



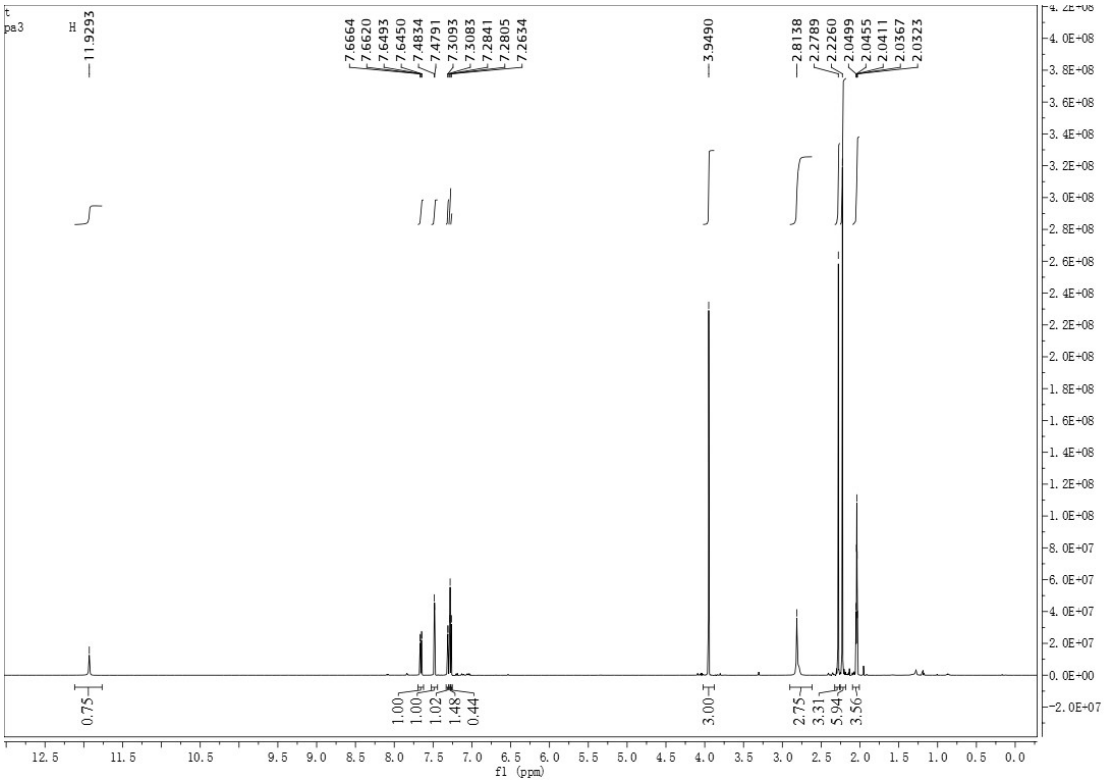
7. The ^{1}H NMR spectrum of 4-(6-ethyl-2-oxochroman-4-yl)-2,6-dimethoxyphenyl methanesulfonate (acetone-d_6, 500 MHz) (**7**)



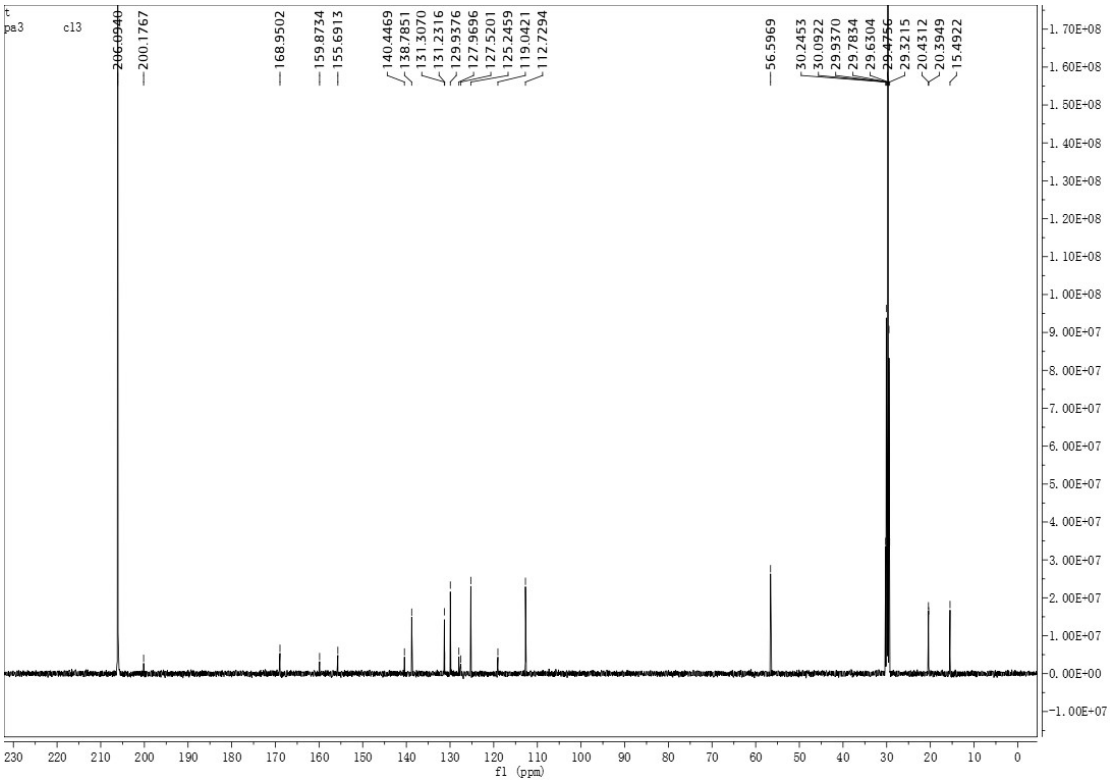
The ^{13}C NMR spectrum of 4-(6-ethyl-2-oxochroman-4-yl)-2,6-dimethoxyphenyl methanesulfonate (acetone-d_6, 125 MHz) (**7**)



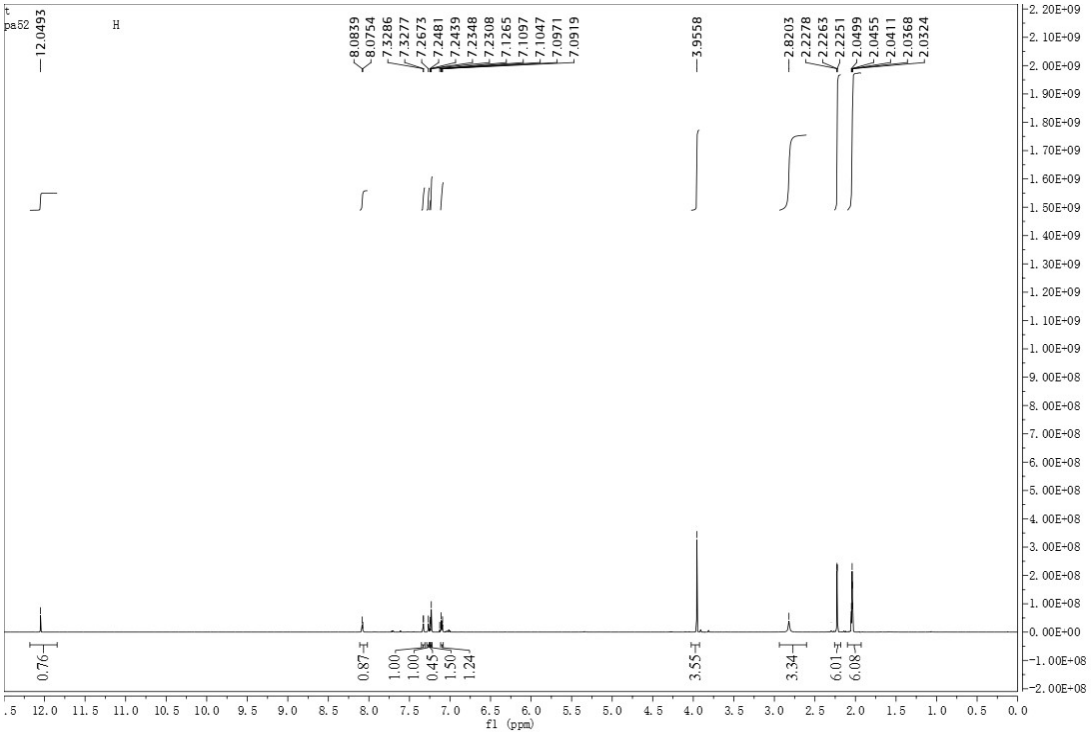
8. The ^{1}H NMR spectrum of 5-(6,8-dimethyl-2-oxo-2*H*-chromen-4-yl)-2-methoxyphenyl acetate (acetone-d_6, 500 MHz) (**8**)



The ^{13}C NMR spectrum of 5-(6,8-dimethyl-2-oxo-2*H*-chromen-4-yl)-2-methoxyphenyl acetate (acetone-d_6, 125 MHz) (**8**)



9. The ^{1}H NMR spectrum of 4-(3-hydroxy-4-methoxyphenyl)-6,8-dimethyl-2*H*-chromen-2-one (acetone-d_6, 500 MHz) (**9**)



The ^{13}C NMR spectrum of 4-(3-hydroxy-4-methoxyphenyl)-6,8-dimethyl-2*H*-chromen-2-one (acetone-d_6, 125 MHz) (**9**)