Supporting Information

Enantioselective Nozaki-Hiyama-Kishi Allylation-Lactonization for the Syntheses of 3-Substituted Phthalides

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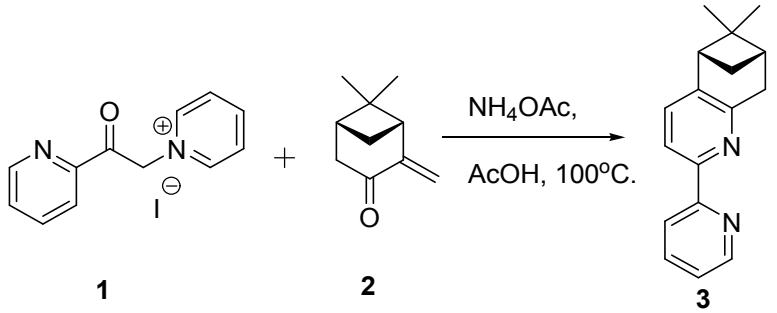
General information:

All reactions were carried out in anhydrous solvents. THF and diethyl ether were distilled from sodium-benzophenone under argon. Toluene, CH_2Cl_2, and hexane were distilled from CaH_2. ^1H NMR spectra were obtained at 300 or 400 MHz (as indicated), and ^{13}C NMR spectra were obtained at 75.5 or 100.6 MHz, using a Bruker NMR spectrometer. Chemical shifts (δ) are reported in ppm relative to $CDCl_3$ (7.26 and 77.0 ppm). Mass spectra (EI-MS) and high resolution mass spectra (HRMS) were determined on a Finnigan/Thermo Quest MAT 95XL mass spectrometer. Infrared spectra were recorded using a JASCO FT/IR S5 410 spectrometer. All asymmetric reactions were conducted in dry glassware under argon. Enantiomeric excesses were determined using Lab Alliance Series III high performance liquid chromatography (HPLC) with a Chiracel OJ column (Daicel Chemical Industries, LTD). Optical rotations were measured using a JASCO P-1010 Polarimeter at the indicated temperature using a sodium lamp (D line, 589 nm). Flash column chromatography was performed using MN silica gel 60 (70–230 mesh) purchased from Macherey-Nagel.

Synthesis and characterization of ligands

1. (1*S*,9*S*)-10,10-dimethyl-5-pyridin-2-yl-6-aza-tricyclo[7.1.1.0^{2,7}]-undeca-2(7)-3,5-triene (3):

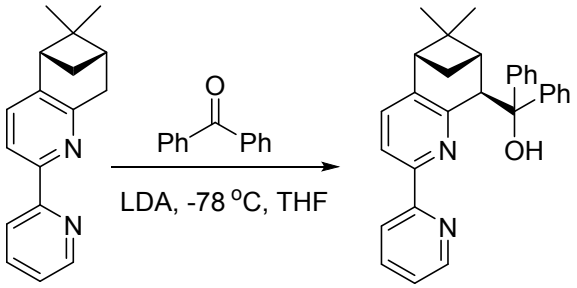


1 **2** **3**

A mixture of compound **1** (6.51 g, 19.97 mmol), compound **2** (3.0 g, 19.97 mmol,), and ammonium acetate (12.31 g, 159.76 mmol) in glacial acetic acid (20.0 mL) under argon atmosphere was heated at 100–110 ºC for 15 h. After cooling to room temperature, the reaction mixture was transferred to a 500-mL conical flask, water (200 mL) was added, and the mixture was basified by sodium hydroxide until it become basic. The aqueous solution was extracted three times with ethyl acetate (3 × 200 mL) and the combined extracts were dried over anhydrous magnesium sulfate. After filtering and concentration, the resulting residue was purified by flash column chromatography using silica gel as the stationary phase and ethyl acetate-hexane (10:90) as the mobile phase to obtain compound **3** (3.80 g, 15.18 mmol) as pale yellow solid. Yield: 76%.

$[\alpha]^{20}{}_D$ –40º (*c* 1.05, $CHCl_3$)., ^{1}H NMR (400 MHz, $CDCl_3$, δ): 8.65 (t, *J* = 3.5 Hz, 1H), 8.37–8.32 (m, 1H), 8.04 (dd, *J* = 3.3, 7.7 Hz, 1H), 7.80–7.73 (m, 1H), 7.32 (dd, *J* = 4.0, 7.7 Hz, 1H), 3.19 (s, 2H), 2.80–2.69 (m, 2H), 2.42–2.30 (m, 1H), 1.42 (s, 3H) 1.32–1.29 (dd, *J* = 4.0, 9.5 Hz, 1H), 0.68 (s, 3H). ^{13}C NMR (100.6 MHz, $CDCl_3$, δ): 156.8, 156.4, 153.5, 149.1, 142.2, 136.7, 133.7, 123.0, 120.8, 117.8, 46.5, 40.3, 39.5, 36.7, 31.9, 26.0, 21.3. IR (KBr): 3049, 2972, 2923, 1920, 1583, 1577, 1558, 1437, 1432, 1382, 1366, 1245, 1144, 1072, 990, 943, 899, 792, 751, 620, 609, 518cm^{-1}. LRMS-EI$^+$ (*m/z*): 251 ([M+H]$^+$, 100), 250 (10) HRMS-TOF-ES$^+$ (*m/z*): [M+H]$^+$ calcd for $C_{17}H_{19}N_2$, 251.1549, found 251.1549.

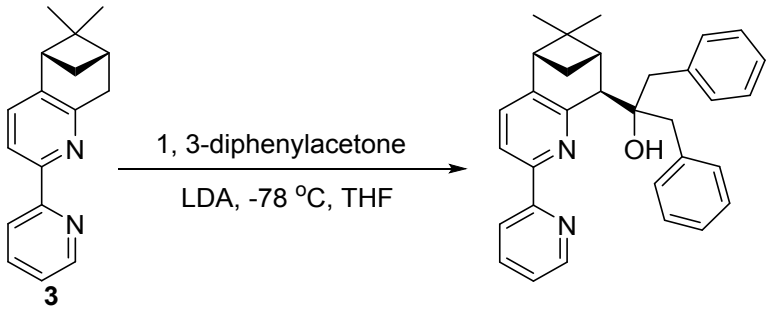
2. (1*S*,8*R*,9*S*)-(10,10-dimethyl-5-pyridin-2-yl-6-aza-tricyclo[7.1.1.0^{2,7}]-undeca-2(7),3,5-trien-8-yl)-diphenyl-methanol (4)



3 **4**

To a solution of Bipy **3** (0.50 g, 2.00 mol) in dry THF (5 mL) the LDA solution (2 M in hexane, 1.2 mL, 2.3966 mmol) was added at –78 °C and stirred for 2 h to generate a dark blue solution. To this dark blue solution was added a solution of benzophenone (0.36 g, 2.00 mmol) in THF (5 mL) and the temperature was slowly raised to room temperature and stirred for 8 h. The reaction was quenched by adding NH_4Cl solution, and extracted with EtOAc. The combined organic phase was dried over anhydrous $MgSO_4$. After concentration to give a residue, the residue was purified by flash column chromatography using silica gel as a stationary phase and using ethyl acetate-hexane (1:9) as the mobile phase, to get product **4** (0.66 g, 1.52 mmol) as off white solid. Yield: 76%. mp: 75–77 °C. $[\alpha]^{20}{}_D$ –452° (*c* 1.77, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$, δ): 9.90 (bs, 1H), 8.69 (d, *J* = 4.4 Hz, 1H), 8.32–8.16 (m, 2H), 7.80–7.74 (m, 1H), 7.56–7.36 (m, 5H), 7.36–7.27 (m, 2H), 7.17–6.96 (m, 5H), 4.48 (s, 1H), 2.75–2.52 (m, 2H), 2.12–2.03 (m, 1H), 1.48 (s, 3H), 0.89 (s, 3H), –0.07– –0.12 (d, *J* = 10.4 Hz, 1H). ^{13}C NMR (100.6 MHz, $CDCl_3$, δ): 156.8, 155.3, 151.4, 149.3, 146.9, 145.8, 144.3, 137.2, 134.9, 128.3, 128.2, 127.8, 127.2, 127.0, 126.4, 123.6, 120.6, 118.7, 81.9, 47.9, 45.8, 43.0, 41.5, 28.7, 26.3, 21.2. IR (KBr): 3445, 3148, 3054, 2972, 2928, 2862, 1577, 1558, 1462, 1432, 1380, 1284, 1152, 987, 938, 897, 858, 787,784, 743, 636, 625, 578, 543, 515 cm^{-1}. LRMS-EI$^+$ (*m/z*): 433 ([M+H]$^+$, 100), 432 (3), 431 (8), 367 (5), 249 (6), 167 (15). HRMS-TOF-ES$^+$ (*m/z*): [M+H]$^+$ calcd for $C_{30}H_{29}N_2O$, 433.2281, found 433.2281.

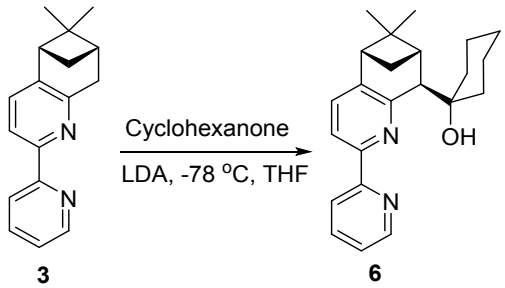
3. (1*S*,8*R*,9*S*)-2-(10,10-dimethyl-5-(pyridin-2-yl)-6-aza-tricyclo[7.1.1.0^{2,7}]-undeca-2(7)-3,5-trien-8-yl)-1,3-diphenyl-propan-2-ol (5):



3 **5**

To a solution of Bipy **3** (0.2 g, 0.80 mmol) in dry THF (3 mL) the LDA solution (0.48 mL, 0.95 mmol, 2 M in hexane) was added at –78 °C and stirred for 2 h to generate a dark blue solution. To this dark blue solution was added a solution of 1,3-diphenyl acetone (0.17 mg, 0.7988 mmol) in THF (2 mL) and the temperature was slowly raised to room temperature and stirred for 3 h. The reaction was quenched by adding NH_4Cl solution, and extracted with EtOAc. The combined organic phase was dried over anhydrous $MgSO_4$. After concentration to give a residue, the residue was purified by flash column chromatography using silica gel as a stationary phase and using ethyl acetate-hexane (1:9) as the mobile phase, to get product **5** (0.27 g, 0.60 mmol) as pale yellow semisolid. Yield: 75%. $[\alpha]^{20}{}_{D}$ –290° (*c* 1.09, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$, δ): 8.70–8.66 (m, 1H), 8.31–8.20 (m, 2H), 8.18 (d, *J* = 7.7 Hz, 1H), 7.88–7.82 (m, 1H), 7.47–7.34 (m, 3H), 7.35–7.11 (m, 8H), 3.21 (s, 1H), 2.94–2.59 (m, 6H), 1.70–1.37 (m, 4H), 1.29–1.25 (m, 2H), 0.40 (s, 3H). ^{13}C NMR (100.6 MHz, $CDCl_3$, δ): 158.4, 155.4, 151.9, 149.2, 144.1, 138.2, 130.0, 137.1, 134.6, 131.1, 130.9, 127.7, 126.0, 125.9, 123.5, 120.5, 118.5, 79.1, 47.7, 46.7, 46.3, 42.1, 41.9, 29.9, 26.3, 20.7. IR (KBr): 3280, 2021, 2923, 1555, 1577, 1492, 1432, 1451, 1382, 1372, 1251, 1152, 1166, 1086, 960, 905, 861, 784, 751, 699, 655, 609, 518 cm^{-1}. LRMS-EI$^+$ (*m*/*z*): 461 ([M+H]$^+$, 100), 443 (3), 180 (1). HRMS-TOF-ES$^+$ (*m*/*z*): [M+H]$^+$ calcd For $C_{32}H_{33}N_2O$, 461.2594, found 461.2596.

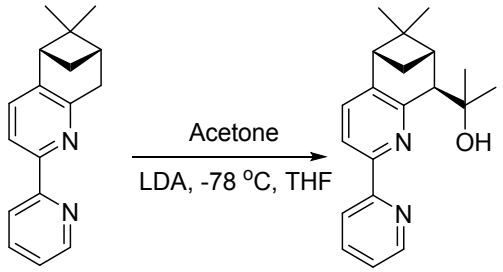
4. (1*S*,8*R*,9*S*)-1-(10,10-dimethyl-5-(pyridin-2-yl)-6-aza-tricyclo[7.1.1.0^{2,7}]-undeca-2(7)-3,5-trien-8-yl)-cyclohexan-1-ol (6)



3 **6**

To a solution of Bipy **3** (0.5 g, 2.00 mmol) in dry THF (5 mL) the LDA solution (1.2 mL, 2.40 mmol, 2 M in hexane) was added at –78° C and stirred for 2 h to generate a dark blue solution. To this dark blue solution was added a solution of cyclohexanone (0.20 mg, 1.9972 mmol) in THF (5 mL) and the temperature was slowly raised to room temperature and stirred for 8 h. The reaction was quenched by adding NH_4Cl solution, and extracted with EtOAc. The combined organic phase was dried over anhydrous $MgSO_4$. After concentration to give a residue, the residue was purified by flash column chromatography using silica gel as a stationary phase and using ethyl acetate-hexane (1:9) as the mobile phase, to get product **6** (0.51 g, 1.46 mmol) as brown oil, Yield: 73%. $[\alpha]^{20}{}_D$ –322° (*c* 1.82, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$, δ): 8.65 (d, *J* = 2.6 Hz, 1H), 8.29–8.11 (m, 2H), 7.90 –7.67 (m, 2H), 7.40 (d, *J* = 7.7 Hz, 1H), 3.21 (d, *J* = 15.4 Hz, 1H), 2.80 (dd, *J* = 5.0, 5.0 Hz, 1H), 2.68–2.60 (m, 1H), 2.47–2.41 (m, 1H), 1.98–1.74 (m, 2H), 1.74–1.50 (m, 4H), 1.50–1.34 (m, 6H), 1.35–1.19 (m, 2H), 1.12 (q, *J* = 13.0 Hz, 1H), 0.69 (s, 3H). ^{13}C NMR (100.6 MHz, $CDCl_3$, δ): 158.0, 155.6, 151.9, 149.1, 143.8, 137.1, 134.5, 123.4, 120.6, 118.5, 75.4, 53.1, 46.5, 42.0, 41.9, 37.2, 35.3, 29.5, 26.0, 21.7, 21.5, 21.1. IR (KBr): 3307, 2928, 2857, 1577, 1555, 1432, 1248, 1218, 1179, 1141, 1091, 1053, 1017, 954, 858, 787, 740, 721, 691, 658, 609, 491 cm^{-1}. LRMS-EI$^+$ (*m/z*): 349 ([M+H]$^+$, 100), 331 (15), 249 (1), 175 (2). HRMS-TOF-ES$^+$ (*m/z*): [M+H]$^+$ calcd for $C_{23}H_{29}N_2O$, 349.2281 found, 349.2278.

5. (1*S*,8*R*,9*S*)-2-(10,10-dimethyl-5-(pyridin-2-yl)-6-aza-tricyclo[7.1.1.0^{2,7}]-undeca-2(7)-3,5-trien-8-yl)-propan-2-ol (7)



3 **7**

To a solution of Bipy **3** (0.2 g, 0.80 mmol) in dry THF (3 mL) the LDA solution (0.48 mL, 0.95 mmol, 2 M in hexane) was added at –78 °C and stirred for 2 h to generate a dark blue solution. To this dark blue solution was added a solution of acetone (0.05 mg, 0.80 mmol) in THF (2 mL) and the temperature was slowly raised to room temperature and stirred for 8 h. The reaction was quenched by adding NH_4Cl solution, and extracted with EtOAc. The combined organic phase was dried over anhydrous $MgSO_4$. After concentration to give a residue, the residue was purified by flash column chromatography using silica gel as a stationary phase and using ethyl acetate-hexane (1:9) as the mobile phase, to get product **7** (0.20 g, 0.65 mmol) as white solid. Yield: 81%. $[\alpha]^{20}{}_D$ –160º (*c* 0.19 $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$, δ): 8.64 (d, *J*

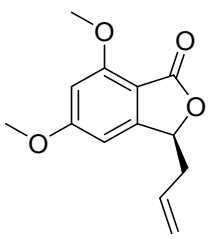
= 4.0 Hz, 1H), 8.33–8.11 (m, 2H), 8.00 (s, 1H), 7.75 (dd, *J* = 7.5, 7.5 Hz, 1H), 7.40 (d, *J* = 7.7 Hz, 1H), 3.29 (s, 1H), 2.80 (dd, *J* = 5.5, 5.5 Hz, 1H), 2.61 (dd, *J* = 5.1, 9.5 Hz, 1H), 2.37 (dd, *J* = 6.8, 6.8 Hz, 1H), 1.54–1.39 (m, 3H), 1.35 (s, 3H), 1.30–1.17 (m, 2H), 1.12 (s, 3H), 0.71 (s, 3H). ^{13}C NMR (100.6 MHz, $CDCl_3$, δ): 158.0, 155.6, 152.1, 149.1, 143.5, 136.9, 134.5, 123.4, 120.5, 118.7, 74.4, 52.8, 46.5, 42.1, 29.5, 27.5, 26.2, 21.0. LRMS-EI$^+$ (*m/z*): 309 ([M+H]$^+$, 100), 291 (13), 249 (3), 148 (1). HRMS-TOF-ES$^+$ (*m/z*): [M+H]$^+$ calcd for $C_{20}H_{25}N_2O$, 309.1968, found 309.1968.

Synthesis and characterization of phthalides

General procedure for preparation of phthalides.

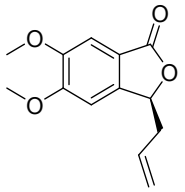
Anhydrous THF (1 mL) was added to a mixture of $CrCl_3$ (8.0 mg, 0.05 mmol, 0.1 eq), Mn (82.4 mg, 0.15 mmol, 3.0 eq, 325 mesh), and dried powder 4Å MS (100 mg) under argon, and the mixture was stirred for 1 h at room temperature. After ligand **6** (65.0 mg, 0.15 mmol, 0.3 eq) and anhydrous NEt_3 (0.042 mL, 0.30 mmol, 0.6 eq) were added, the suspension was stirred for 1 h at room temperature. Subsequently, allyl bromide (0.064 mL, 0.75 mmol, 1.5 eq) was added. After 1 h at room temperature, aldehyde (0.50 mmol, 1 eq) and Me_3SiCl (0.095 mL, 0.75 mmol, 1.5 eq) were added, and the suspension was stirred in ice box at 0 °C for 48 h. Saturated aqueous $NaHCO_3$ was added. Following filtration and evaporation, the aqueous phase was extracted with EtOAc. After the combined organic phases had been evaporated, the residue was dissolved in THF (2 mL). A TBAF solution (1.5 mL, 1.5 mmol, 3 eq, 1M in THF) was added and the mixture was stirred until desilylation was complete (as verified by TLC). Water was added; the solution was extracted using ethyl acetate, and the combined organic phases were dried over $MgSO_4$. Residue was dissolved into THF (2 mL) and *p*-TsOH (cat.) was added and stirred for 2 h at room temperature. Evaporation of the solvent and flash chromatography (EtOAc/hexane 1:19) gave the products. Enantiomeric excess was determined using high-performance liquid chromatography with a chiral column (Chiralcel OJ column, *n*-hexane/2-propanol (90/10), flow rate: 1.0 mL/min^{-1}).

1. 3-allyl-5,7-dimethoxyisobenzofuran-1(3*H*)-one (8) [1]



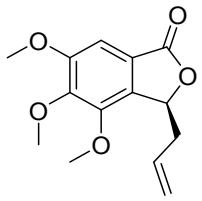
Yield: 87%. Off white solid. $[\alpha]^{25}_{D}$ –66.3 (*c* 0.55, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$, δ): 6.43 (s, 1H), 6.41 (s, 1H), 5.81–5.70 (m, 1H), 5.35–5.32 (t, *J* = 11.7 Hz, 1H), 5.20–5.12 (m, 2H), 3.94 (s, 3H), 3.88 (s, 3H), 2.73–2.67 (m, 1H), 2.60–2.53 (m, 1H). ^{13}C NMR (100.6 MHz, $CDCl_3$, δ): 168.2, 166.6, 159.6, 154.3, 131.4, 119.4, 107.0, 98.7, 97.8, 55.9, 55.9, 39.76. IR (KBr): 3076, 2923, 2846, 1753, 1610, 1459, 1349, 1333, 1218, 1157, 1100, 1034, 836, 757, 691, 554 cm^{-1}. LRMS-EI$^+$ (*m*/*z*): 235 ([M+H]$^+$ 4), 217 (8), 289 (1), 290 (3), 174 (2). HRMS-TOF-ES$^+$ (*m/z*): [M+H]$^+$ calcd for $C_{13}H_{15}O_4$, 235.0971 found 235.0970. 97 % ee by HPLC analysis (Chiralcel OJ column, hexane:2-propanol = 9:1, 1.0 mL/min, 254 nm UV detector).

2. 3-allyl-5,6-dimethoxyisobenzofuran-1(3*H*)-one (9) [2]



Yield: 85%. White solid. $[\alpha]^{25}_{D}$ –52.6° (*c* 0.45, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.32–7.17 (m, 1H), 6.85 (s, 1H), 5.85–5.70 (m, 1H), 5.40 (dd, *J* = 5.9, 5.9 Hz, 1H), 5.30–5.05 (m, 2H), 4.01–3.89 (m, 6H), 2.72–2.60 (m, 2H). ^{13}C NMR (100.6 MHz, $CDCl_3$, δ): 154.7, 150.5, 143.8, 131.4, 119.5, 118.3, 106.2, 103.4, 79.5, 56.3, 56.2, 38.8. IR (KBr): 3076, 2934, 2840, 1753, 1640, 1602, 1500, 1470, 1418, 1336, 1286, 1223, 1133, 1059, 984, 916, 812, 768, 732, 658, 551 cm^{-1}. LRMS-EI$^+$ (*m*/*z*): 235 ([M+H]$^+$, 9), 230 (2), 228 (3), 226 (4), 191 (6). HRMS-TOF-ES$^+$ (*m/z*): [M+H]$^+$ calcd for $C_{13}H_{15}O_4$, 235.0971 found 235.0971. 94 % ee by HPLC analysis (Chiralcel OJ column, hexane:2-propanol = 9:1, 1.0 mL/min, 254 nm UV detector).

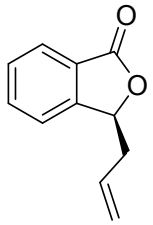
3. 3-allyl-4,5,6-trimethoxyisobenzofuran-1(3*H*)-one (10)



Yield: 90%. Pale yellow oil. $[\alpha]^{25}_{D}$ –29.9 (*c* 0.50, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$, δ): 7.11 (s, 1H), 5.72–5.60 (m, 1H), 5.53–5.48 (m, 1H), 5.10–5.04 (m, 2H), 4.00 (s, 3H), 3.97–3.85 (m, 6H), 3.00–2.91 (m, 1H), 2.58–2.52 (m, 1H); ^{13}C NMR (100.6 MHz, $CDCl_3$, δ): 170.3, 155.7, 147.5, 146.7, 134.4, 131.5, 121.6, 119.1, 102.5, 79.1, 61.1, 60.7, 56.3, 53.4, 37.1, 29.6. IR (KBr): 2918, 2848, 1766, 1615, 1478, 1420, 1344, 1301, 1250, 1198, 1106, 1033, 968, 921, 845, 761, 661 cm^{-1}. LRMS-EI$^+$ (*m*/*z*):265 ([M+H]$^+$, 18), 253 (8), 253 (5) 149 (2). HRMS-TOF-ES$^+$ (*m/z*):

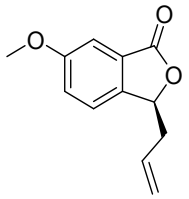
$[M+H]^+$ calcd for $C_{14}H_{16}O_5$, 265.1077, found 265.1080. 99 % ee by HPLC analysis (Chiralcel OJ column, hexane:2-propanol = 9:1, 1.0 mL/min, 254 nm UV detector).

4. 3-allylisobenzofuran-1(3*H*)-one (11) [3]



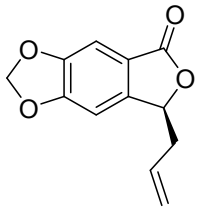
Yield: 86%. Brown oil. $[\alpha]^{25}_D$ –32.6° (*c* 0.35, $CHCl_3$). ^1H NMR (300 MHz, $CDCl_3$, δ): 7.91–7.88 (d, *J* = 7.7 Hz, 1H), 7.69–7.50 (m, 2H), 7.55–7.45 (d, *J* = 7.6 Hz, 1H), 5.82–5.68 (m, 1H), 5.55–5.51 (t, *J* = 12.0 Hz, 1H), 5.21–5.13 (dd, *J* = 1.3, 8.5 Hz, 2H), 2.80–2.60 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$, δ): 149.4, 134.1, 131.1, 129.2, 125.8, 122.0, 119.7, 80.4, 38.6. IR (KBr): 3071, 2917, 2851, 1758, 1643, 1610, 1593, 1462, 1357, 1308, 1286, 1209, 1070, 979, 921, 740, 696, 603, 573 cm^{-1}. LRMS-EI$^+$ (*m/z*): 175 ($[M+H]^+$,1), 174 (4), 169 (10), 168 (40), 157 (70), 14 (50). HRMS-TOF-ES$^+$ (*m/z*): $[M+H]^+$ calcd for $C_{11}H_{11}O_2$, 175.0760, found 175.0760. 96 % ee by HPLC analysis (Chiralcel OJ column, hexane:2-propanol = 9:1, 1.0 mL/min, 254 nm UV detector).

5. 3-allyl-6-methoxyisobenzofuran-1(3*H*)-one (12) [3]



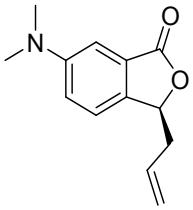
Yield: 90%. White solid. $[\alpha]^{25}_D$ –38.9° (*c* 0.65, $CHCl_3$). ^1H NMR (300 MHz, $CDCl_3$, δ): 7.80–7.77 (d, *J* = 8.4 Hz, 1H), 7.04–7.00 (dd, *J* = 1.7, 2.2 Hz 1H), 6.88–6.87(d, *J* = 2.9 Hz, 1H), 5.84–5.70 (m, 1H), 5.44–5.40 (t, *J* = 11.8 Hz, 1H), 5.29–5.13 (m, 2H), 3.89 (s, 3H), 2.77–2.57 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$, δ): 170.0, 164.6, 152.0, 131.3, 127.2, 119.5, 116.3, 106.1, 79.5, 55.8, 38.7. IR (KBr): 3076, 2923, 2851, 1758, 1604, 1496, 1462, 1349, 1253, 1152, 1102, 1059, 982, 921, 834, 765, 688, 603 cm^{-1}. LRMS-EI$^+$ (*m/z*): 205 ($[M+H]^+$ 0.2), 200 (1), 193 (3), 148 (2). HRMS-TOF-ES$^+$ (*m/z*): $[M+H]^+$ calcd for $C_{12}H_{13}O_3$, 205.0865, found 205.0865. 97 % ee by HPLC analysis (Chiralcel OJ column, hexane:2-propanol = 9:1, 1.0 mL/min, 254 nm UV detector).

6. 7-allyl-[1,3]dioxolo[4,5-f]isobenzofuran-5(7*H*)-one (13) [2]



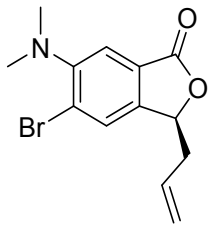
Yield: 76%. Colorless oil. $[\alpha]^{25}_{D}$ –48.9° (*c* 0.45, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.19 (s, 1H), 6.81 (s 1H), 6.11 (s, 2H), 5.78–5.71 (m, 1H), 5.38–5.35 (t, *J* = 12.0 Hz, 1H), 5.20–5.14 (m, 2H), 2.71–2.56 (m, 2H) ^{13}C NMR (75 MHz, $CDCl_3$, δ): 169.8, 153.5, 149.2, 145.9, 131.1, 120.0, 119.7, 104.3, 102.6, 101.7, 79.4, 38.7. IR (KBr): 2923, 1753, 1604, 1500, 1467, 1396, 1341, 1223, 1226, 1097, 1048, 1031, 923, 829, 814, 718,631 cm^{-1}. LRMS-EI$^+$ (*m*/*z*): $[M+H]^+$ 219 ($[M+H]^+$ 14), 208 (4), 193 (4), 166 (6). HRMS-TOF-ES$^+$ (*m/z*): $[M+H]^+$ calcd for $C_{12}H_{11}O_4$, 219.0658, found 219.0658. 96 % ee by HPLC analysis (Chiralcel OJ column, hexane:2-propanol = 9:1, 1.0 mL/min, 254 nm UV detector).

7. 3-allyl-6-(dimethylamino)isobenzofuran-1(3*H*)-one (14) [2]



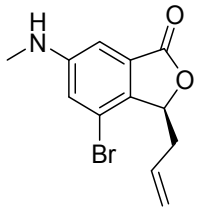
Yield: 70%. Brown oil. $[\alpha]^{25}_{D}$ –48.9° (*c* 0.55, $CHCl_3$).^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.28 (s 1H), 7.11 (d, *J* = 2.2 Hz, 1H), 7.01 (dd, *J* = 2.2, 8.4 Hz, 1H), 5.84–5.72 (m, 1H), 5.43 (t, *J* = 6.1 Hz, 1H), 5.21–5.12 (m, 2H), 3.02 (s, 6H), 2.72–2.56 (m, 2H), 1.27 (s, 2H). ^{13}C NMR (100.6 MHz, $CDCl_3$, δ): 171.3, 151.4, 137.0, 131.8, 127.3, 122.2, 119.2, 118.6, 107.0, 80.1, 40.6, 39.1, 29.7. IR (KBr): 3076, 2923, 1758, 1624, 1551, 1437, 1355, 1316, 1278, 1204, 1061, 982, 916, 817, 773, 598, 556 cm^{-1}. LRMS-EI$^+$ (*m*/*z*): 218 ($[M+H]^+$,52), 180 (4), 174 (5), 158 (4), 152 (2). HRMS-TOF-ES$^+$ (*m/z*): $[M+H]^+$ calcd for $C_{13}H_{16}NO_2$, 218.1182 found 218.1182. 96 % ee by HPLC analysis (Chiralcel OJ column, hexane:2-propanol = 9:1, 1.0 mL/min, 254 nm UV detector).

8. 3-allyl-5-bromo-6-(dimethylamino)isobenzofuran-1(3*H*)-one(15)



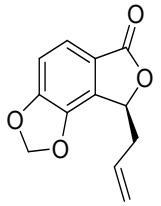
Yield: 90%. Brown solid. $[\alpha]^{25}_{D}$ –56.9° (*c* 0.56, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$, δ): 7.67 (s, 1H), 7.53 (s, 1H), 5.82–5.70 (m, 1H), 5.42 (t, *J* = 5.9 Hz, 1H), 5.24–5.16 (m, 2H), 2.85–2.84 (m, 6H), 2.74–2.58 (m, 2H) ^{13}C NMR (100.6 MHz, $CDCl_3$, δ): 169.7, 153.5, 144.0, 131.0, 127.6, 126.5, 126.4, 119.9, 116.4, 79.4, 44.2, 38.6. IR (KBr): 3082, 2917, 2851, 2780, 1758, 1635, 1610, 1478, 1451, 1396, 1330, 1283, 1256, 1207, 1187, 1157, 1059, 971, 919, 883, 765, 740, 600 cm^{-1}. LRMS-EI$^+$ (*m*/*z*): 296 ([M+H]$^+$ 100), 294, (7), 284 (2), 281 (3), 271 (7), 258 (9), 240 (8). HRMS-TOF-ES$^+$ (*m/z*): [M+H]$^+$ calcd for $C_{13}H_{15}BrNO_2$, 296.0287, found 296.0287. 98 % ee by HPLC analysis (Chiralcel OJ column, hexane:2-propanol = 9:1, 1.0 mL/min, 254 nm UV detector).

9. 3-allyl-4-bromo-6-(methylamino)isobenzofuran-1(3*H*)-one (16)



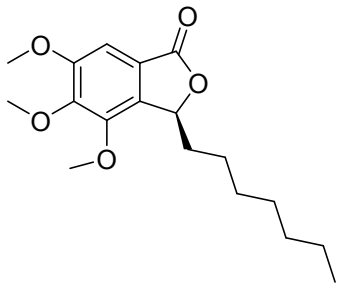
Yield: 90%. Yellow oil. $[\alpha]^{25}_{D}$ –53.7° (*c* 0.48, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$, δ): d 7.53 (s, 1H), 7.01 (s, 1H), 5.81–5.70 (m, 1H), 5.42–5.38 (m, 1H), 5.22–5.14 (m, 2H), 4.65 (s, 1H), 2.97–2.94 (d, *J* = 5.1 Hz, 3H), 2.69–2.56 (m, 2H), ^{13}C NMR (100.6 MHz, $CDCl_3$, δ): 163.0, 146.9, 137.8, 131.2, 126.8, 125.7, 119.6, 116.0, 105.0, 79.4, 38.9, 30.6. IR (KBr): 3395, 2923, 2851, 1753, 1615, 1514, 1456, 1407, 1333, 1270, 119, 1061, 984, 910, 872, 773, 721, 721, 632 cm^{-1}. LRMS-EI$^+$ (*m*/*z*): 296 ([M+H]$^+$,1), 264 (18), 213 (28), 160 (6) HRMS-TOF-ES+ (*m/z*): [M+H]$^+$ calcd for $C_{13}H_{15}BrNO_2$, 296.0287, found 296.0287. 97 % ee by HPLC analysis (Chiralcel OJ column, hexane:2-propanol = 9:1, 1.0 mL/min, 254 nm UV detector).

10. 8-allyl-[1,3]dioxolo[4,5-e]isobenzofuran-6(8*H*)-one (17)



Yield: 77%. Colorless oil. $[\alpha]^{25}_{D}$ –43.9° (*c* 0.50, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$, δ): 7.08–7.06 (d, *J* = 7.9 Hz 1H), 6.85–6.83 (d, *J* = 7.9 Hz 1H), 6.17 (s, 2H), 5.79–5.69 (m, 1H), 5.48–5.45 (t, *J* = 11.6 Hz, 1H), 5.19–5.13 (m, 2H), 2.74–2.57 (m, 2H) ^{13}C NMR (100.6 MHz, $CDCl_3$, δ): 167.2, 149.1, 144.8, 142.2, 131.1, 119.7, 114.3, 109.1, 103.3, 80.7, 39.1. IR (KBr): 2923, 2851, 1761, 1640, 1500, 1473, 1323, 1251, 1240, 1207, 1108, 968, 908, 825, 871, 743, 639 cm^{-1}. LRMS-EI$^+$ (*m*/*z*): 219 ($[M+H]^+$, 3), 217 (3), 210 (1), 166 (4). HRMS-TOF-ES$^+$ (*m*/*z*): $[M+H]^+$ calcd for $C_{12}H_{11}O_4$, 219.0658, found 219.0656. 98 % ee by HPLC analysis (Chiralcel OJ column, hexane:2-propanol = 9:1, 1.0 mL/min, 254 nm UV detector).

11. 3-heptyl-4,5,6-trimethoxyisobenzofuran-1(3*H*)-one (18)



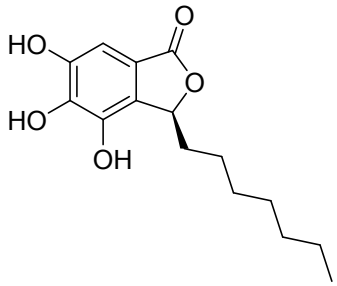
9-BBN (0.18 mL of 0.5 M THF solution, 0.64 mmol, 1.7 eq) was added to olefin **10** (0.10 g, 0.37 mmol, 1 eq) at 25 °C in a flame dried flask under argon. The solution was stirred for 4 h at rt, then Cs_2CO_3 (0.9 mL of a 10 M solution in H_2O, 0.94 mmol, 2.5 eq) was added and the stirring was continued for 10 min. The resulting solution was cannulated into a flask containing 1-bromobutane (41.5 mg, 0.30 mmol, 0.8 eq), $Pd(OAc)_2$ (4.2 mg, 0.0189 mmol) and PCy_3 (10.6 mg, 0.0378 mmol) under Ar(g). The flask and cannula were rinsed once with dioxane (0.50 mL, the solvent was degassed by performing a freeze, pump, thaw cycle 3 times prior to use). The reaction mixture was stirred at 40 °C for 20 h, then diluted with EtOAc (5 mL) and flushed through a plug of celite. The residue was concentrated under reduced pressure and purified by flash column chromatography (10 % EtOAc/Hexanes) to provide (0.11 mg, 0.4312 mmol, Yield 92 %) as pale yellow oil.

^1H NMR (400 MHz, $CDCl_3$, δ): 7.11 (s, 1H), 5.47–5.42 (dd, *J* = 2.8, 7.9 Hz, 1H), 3.96 (s, 3H), 3.93 (s, 3H), 3.91 (s, 3H), 2.20–2.13 (m, 1H), 1.74–1.63 (m, 1H), 1.37–1.22 (m, 10H), 0.91–0.84

(t, *J* = 7.1 Hz, 3H). IR (KBr): 2927, 2856, 1761, 1614, 1479, 1420, 1345, 1253, 1199, 1106, 1036, 967, 848, 765, 725 cm^{-1}. ^{13}C NMR (100.6 MHz, CDCl3, δ): 170.6, 155.5, 147.6, 146.8, 135.4, 121.4, 102.5, 80.2, 61.1, 60.7, 56.4, 33.4, 31.7, 29.2, 29.0, 24.7, 22.6, 14.0. LRMS-EI^+ (*m/z*): $[M+H]^+$, 323 (100), 225 (25), 180 (30), 148 (12). HRMS-TOF-ES^+ (*m/z*): $[M+H]^+$ calcd for $C_{18}H_{26}O_5$, 323.1780, found 323.1860.

[Ref] Keaton, K. A.; Phillips, A. J. *Org. Lett.* **2007**, *9*, 2717–2719.

12. 3 heptyl-4,5,6-trihydroxyisobenzofuran-1(3*H*)-one (19)



To a solution of **19** (0.12 g, 0.372 mmol) in CH_2Cl_2 (2.5 mL) at –30 °C was added BBr_3 (0.37 mL of a 1.0 M solution in CH_2Cl_2, 0.37mmol).[4] After 1h the reaction is warmed to room temperature for 2h after which it was poured into ice-water and the aqueous layer was extracted with CH_2Cl_2 twice. The combined organic layers were washed with brine and dried over Na_2SO_4, and concentrated under reduced pressure. The residue was purified by flash chromatography (5% MeOH/$CHCl_3$) to afford Cytosporone E in 87% yield (0.090 g, 0.3211 mmol). Lit. [5] 98.4%ee, $[\alpha]^{25}_D$ –90.7° (*c* 1.00, acetone). $[\alpha]^{20}_D$ –87.6° (*c* 0.99, acetone). ^1H NMR (400 MHz, DMSO, δ): 9.9 (s, 1H), 9.4 (s, 1H), 9.3 (s 1H), 6.68 (s, 1H), 5.40 (dd, *J* = 2.8, 7.5 Hz, 1H), 2.11 (m, 1H), 1.59 (m, 1H), 1.30–1.16 (m, 10H), 0.84 (t, *J* = 7.1 Hz, 3H). IR (KBr): 3408, 2924, 2853, 2258, 1739, 1628, 1519, 1494, 1346, 1146, 1075, 1024, 999, 869, 765, 637 cm^{-1}. ^{13}C NMR (100.6 MHz, DMSO, δ): 170.8, 147.8, 140.3, 139.9, 129.5, 116.2, 102.0, 79.6, 32.8, 31.6, 29.1, 29.0, 24.6, 22.5, 14.3. . LRMS-EI^+ (*m/z*): $[M-H]^+$ 279 (100), 235 (5). HRMS-TOF-ES^+ (*m/z*): $[M-H]^+$ calcd for $C_{15}H_{19}O_5$, 279.1311, found 279.1233

References

[1] M. A. Brimble and C. J. Bryant, *Org. Biomol. Chem.* 2007, ***5***, 2858.

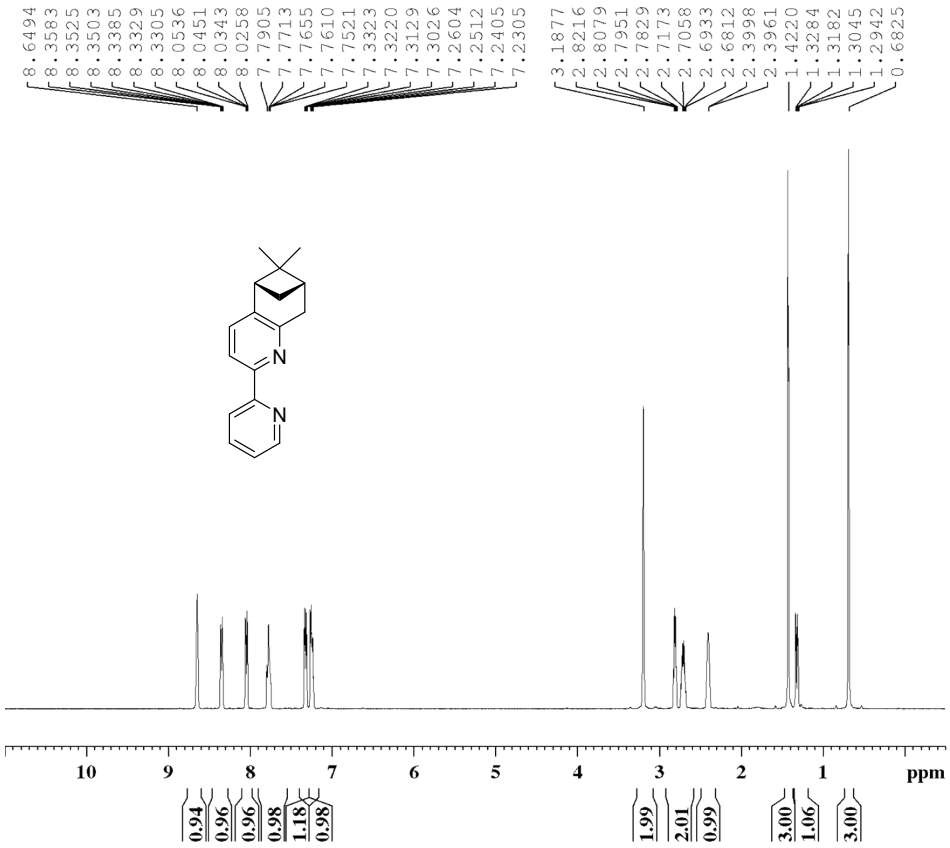
[2] J. M. Cabrera, J. Tauber, and Michael J. Krische, *Angew. Chem. Int. Ed.* 2018, **57**, 1390.

[3] R. Mirabdolbaghi and T. Dudding, *Org. Lett.* 2012, **14**, 3748.

[4] J. D. Hall, N. W. Duncan-Gould, N. A. Siddiqi, J. N. Kelly, L. A. Hoeferlin, S. J. Morrison and J. K. Wyatt, *Bioorg. Med. Chem.* 2005, **13**, 1409.

[5] T. Ohzeki and K. Mori, *Biosci. Biotechnol. Biochem*. 2003, **67**, 2584.

^{1}H NMR and ^{13}C NMR spectra-

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TE                      301.5 K
D1                    2.00000000 sec
TD0                          1

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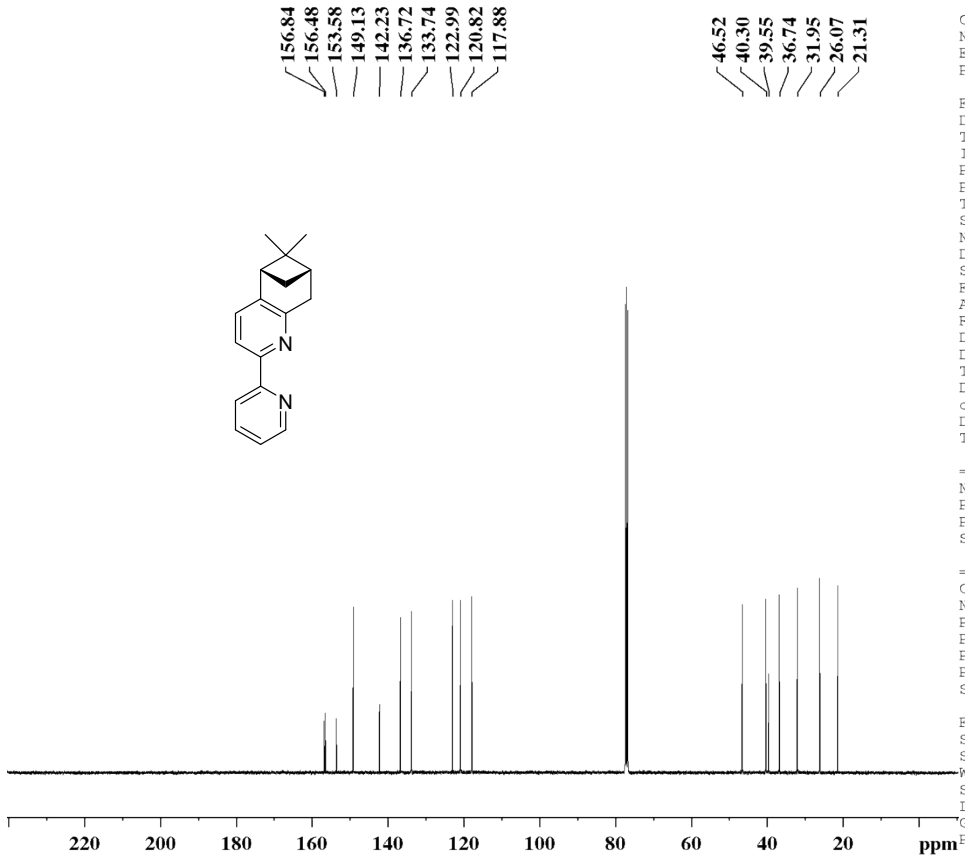


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PL2                -0.40 dB
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SFO2         400.1316005 MHz

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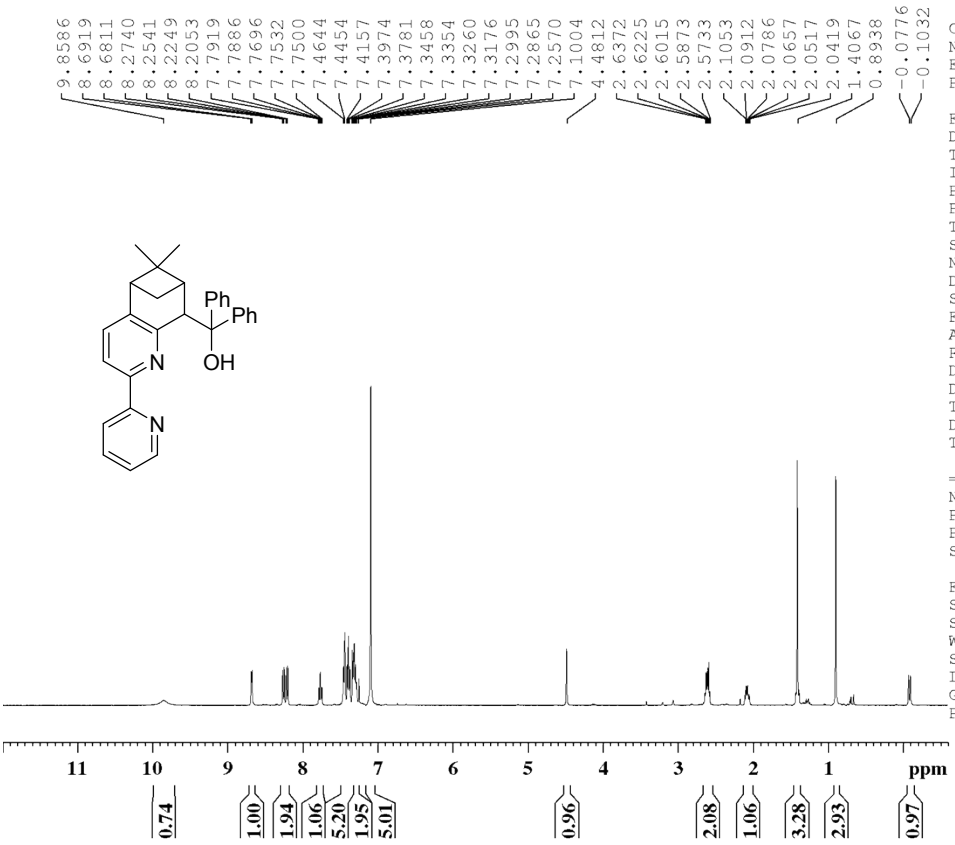


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TD                 32768
SOLVENT            CDCl3
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DS                     0
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FIDRES          0.183399 Hz
AQ             2.7262976 sec
RG                   101
DW                83.200 usec
DE                 6.50 usec
TE                299.5 K
D1           1.00000000 sec
TD0                    1

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P1                15.00 usec
PL1                0.90 dB
SFO1         400.1326008 MHz

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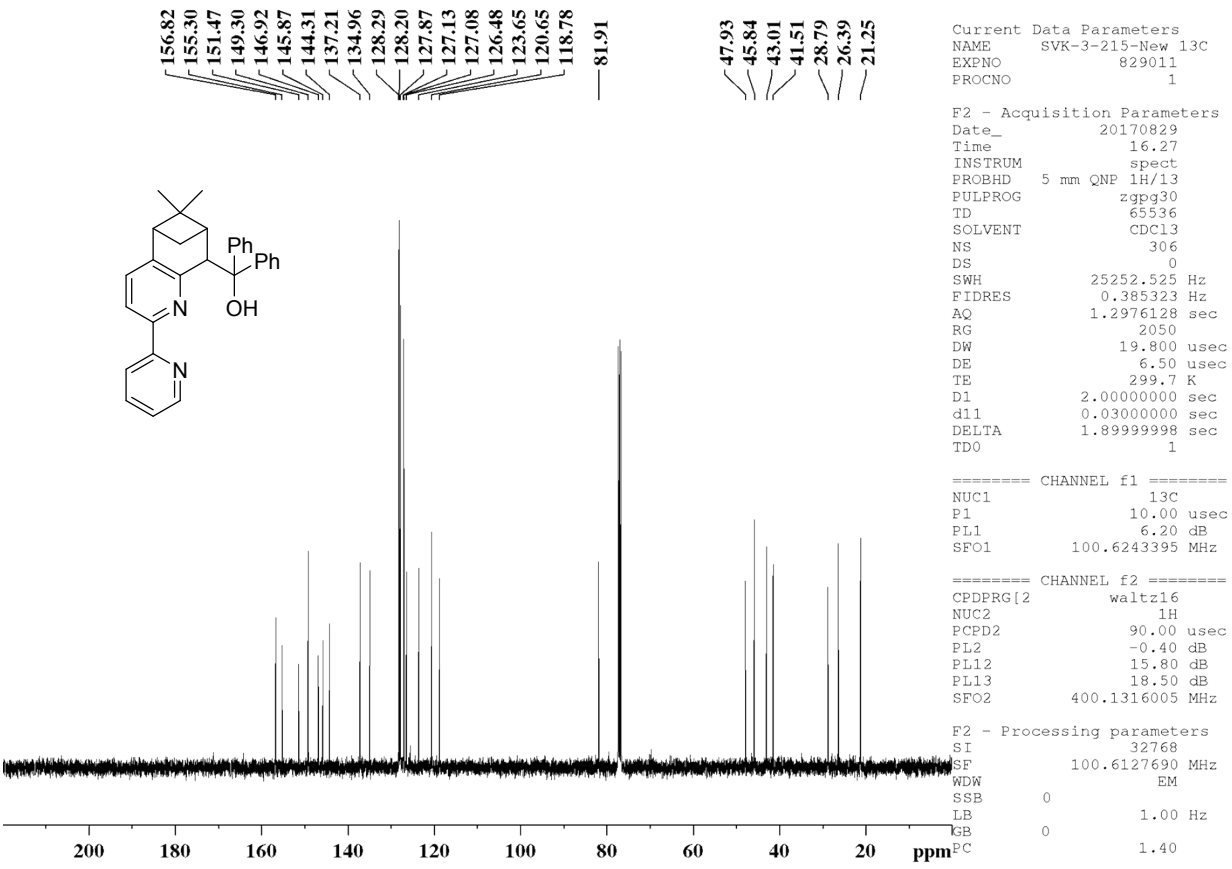


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RG                   2050
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DELTA         1.89999998 sec
TD0                     1

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PL1                  6.20 dB
SFO1         100.6243395 MHz

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SFO2         400.1316005 MHz

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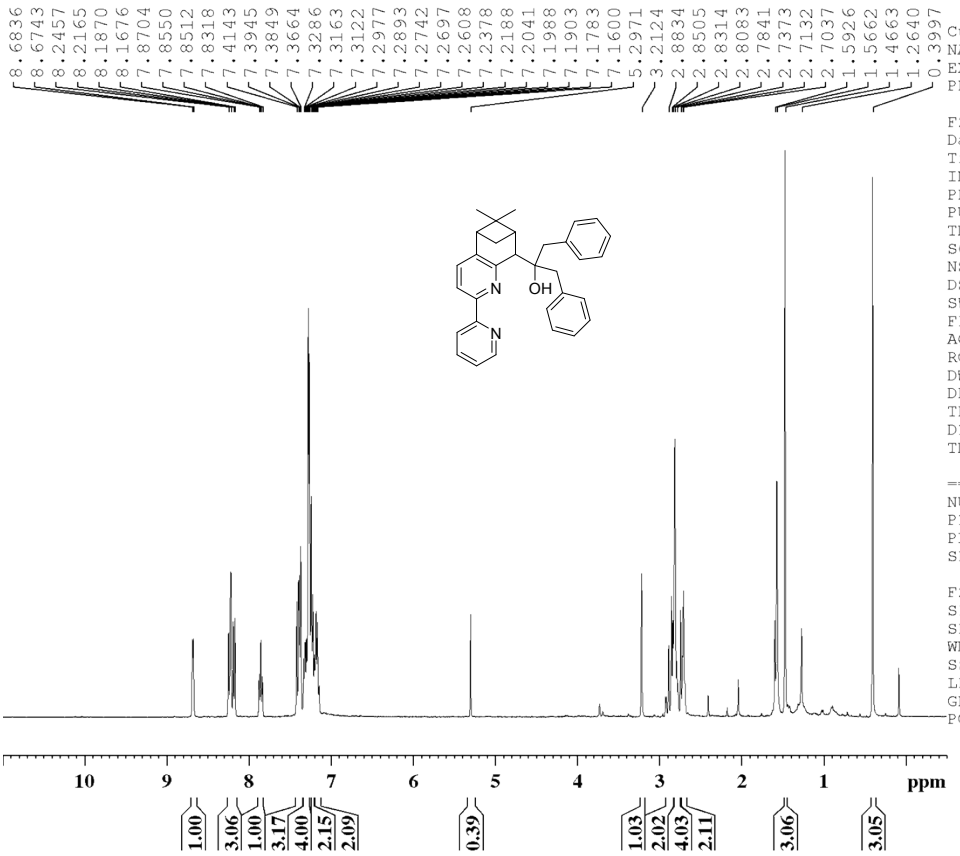


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F2 - Processing parameters
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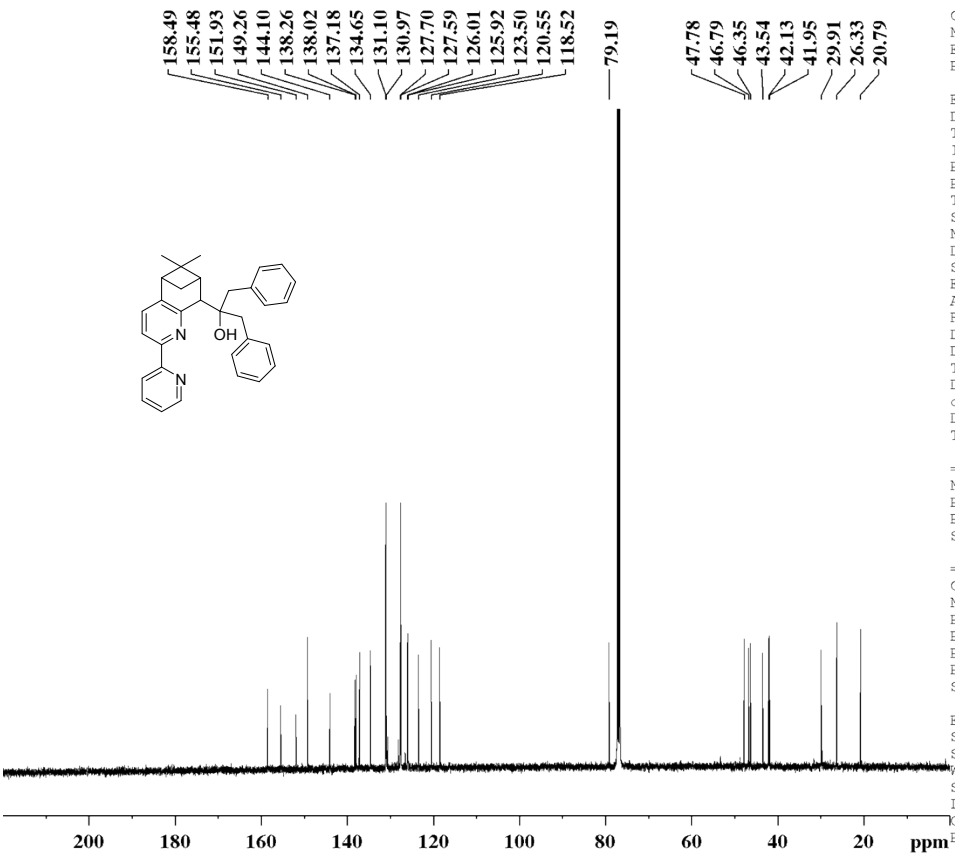


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TD                  65536
SOLVENT             CDCl3
NS                   8028
DS                      0
SWH             25252.525 Hz
FIDRES          0.385323 Hz
AQ             1.2976128 sec
RG                   2050
DW                 19.800 usec
DE                  6.50 usec
TE                 305.3 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                     1

======== CHANNEL f1 ========
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PL1                  6.20 dB
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======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2               90.00 usec
PL2                 -0.40 dB
PL12                15.80 dB
PL13                18.50 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
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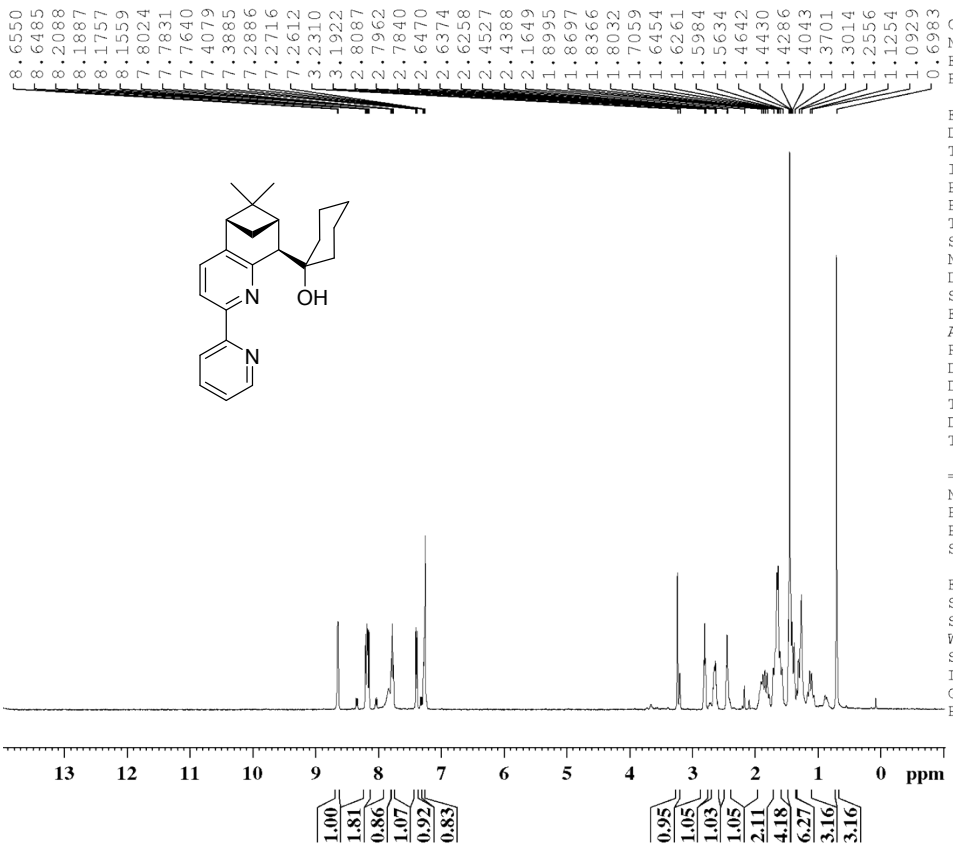
LB                   1.00 Hz
GB        0
PC                   1.40
```

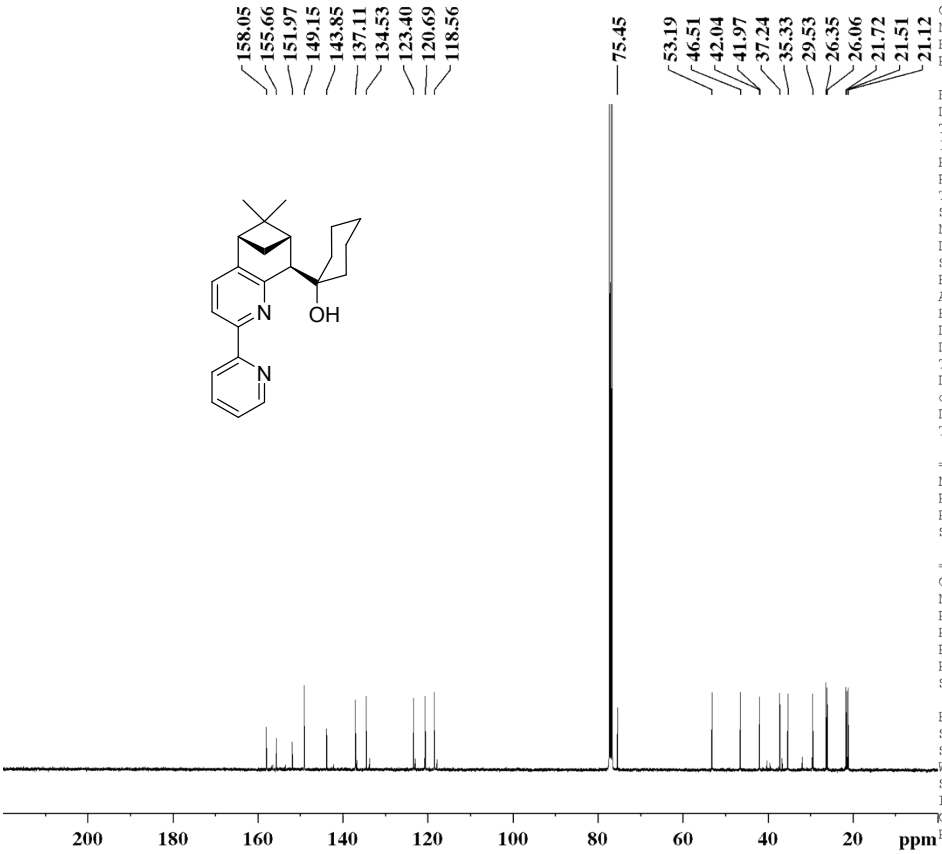



```
Current Data Parameters
NAME                  SVK-3-239
EXPNO                    829021
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20170830
Time                       9.50
INSTRUM                   spect
PROBHD   5 mm QNP 1H/13
PULPROG                    zg30
TD                        32768
SOLVENT                   CDCl3
NS                           10
DS                            0
SWH                    6009.615 Hz
FIDRES                 0.183399 Hz
AQ                    2.7262976 sec
RG                          406
DW                       83.200 usec
DE                        6.50 usec
TE                       303.9 K
D1                  1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                         1H
P1                       15.00 usec
PL1                       0.90 dB
SFO1                400.1326008 MHz

F2 - Processing parameters
SI                        16384
SF                  400.1300097 MHz
WDW                          EM
SSB                   0
LB                        0.30 Hz
GB                    0
PC                        1.00
```



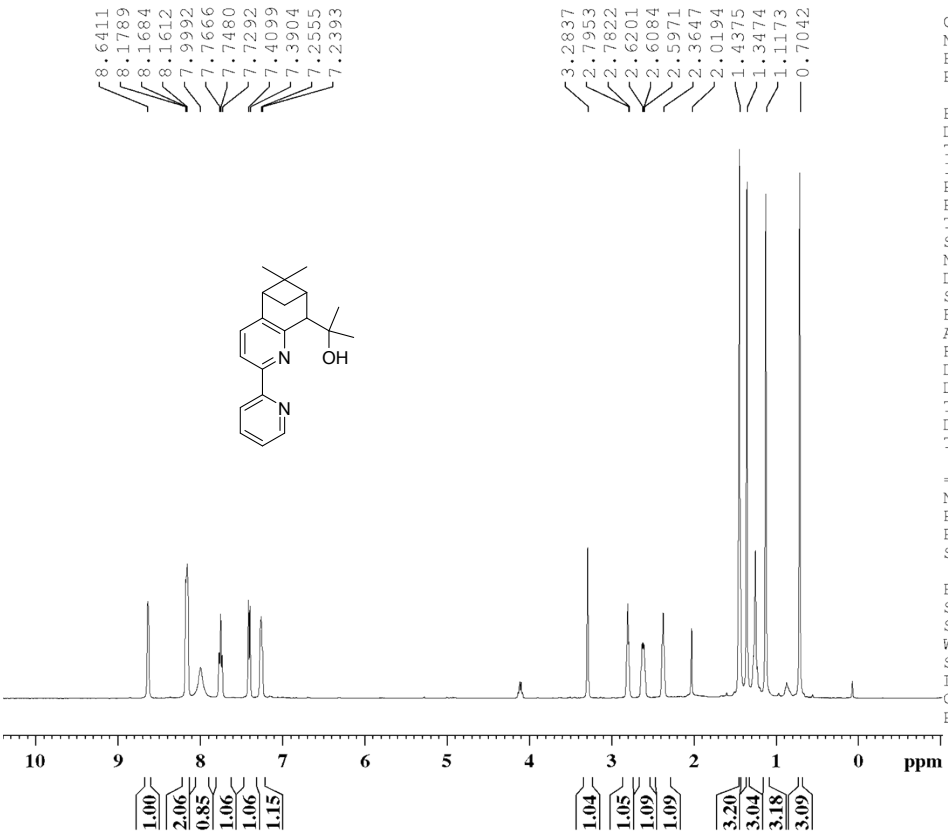

```
Current Data Parameters
NAME      SVK-3-239 new 13C
EXPNO             830041
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170830
Time              23.44
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 9999
DS                    0
SWH           25252.525 Hz
FIDRES         0.385323 Hz
AQ            1.2976128 sec
RG                 2050
DW               19.800 usec
DE                6.50 usec
TE                301.2 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.00 usec
PL1                 6.20 dB
SFO1         100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2              90.00 usec
PL2                -0.40 dB
PL12               15.80 dB
PL13               18.50 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127690 MHz
WDW                  EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

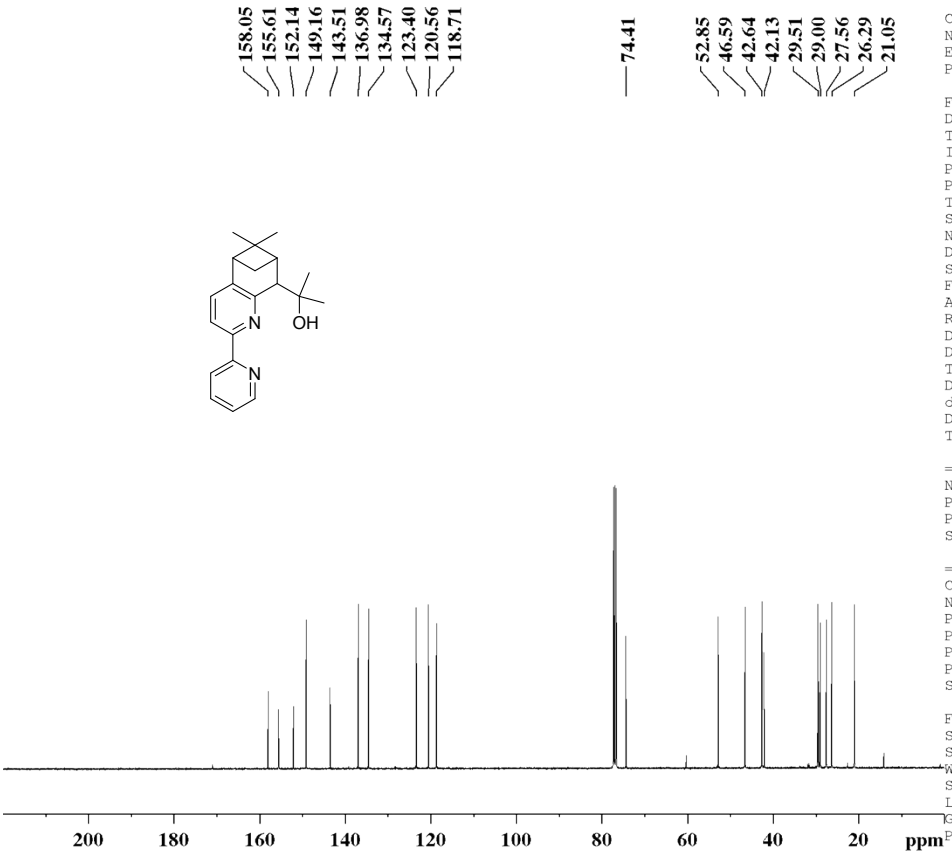



```
Current Data Parameters
NAME      SVk-3-207-acetone
EXPNO              318011
PROCNO                  1

F2 - Acquisition Parameters
Date_            20170318
Time                2.26
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     5
DS                     0
SWH             6009.615 Hz
FIDRES          0.183399 Hz
AQ             2.7262976 sec
RG                   101
DW                83.200 usec
DE                 6.50 usec
TE                307.2 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.00 usec
PL1                0.90 dB
SFO1         400.1326008 MHz

F2 - Processing parameters
SI                 16384
SF           400.1300081 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



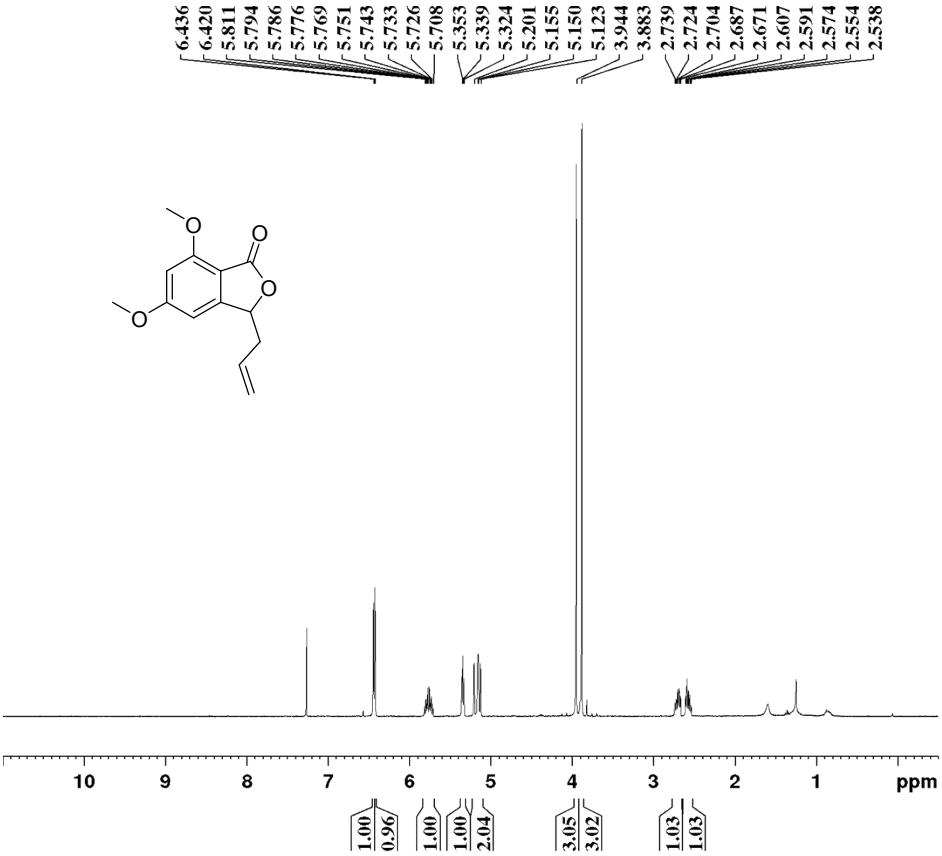

```
Current Data Parameters
NAME      SVk-3-207-acetone 13C
EXPNO              318011
PROCNO                  1

F2 - Acquisition Parameters
Date_            20170318
Time                2.30
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   6600
DS                      0
SWH            25252.525 Hz
FIDRES          0.385323 Hz
AQ             1.2976128 sec
RG                   2050
DW                 19.800 usec
DE                  6.50 usec
TE                 307.3 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  10.00 usec
PL1                  6.20 dB
SFO1         100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2               90.00 usec
PL2                 -0.40 dB
PL12                15.80 dB
PL13                18.50 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                   32768
SF           100.6127690 MHz
WDW                     EM
SSB       0
LB                   1.00 Hz
GB        0
PC                   1.40
```

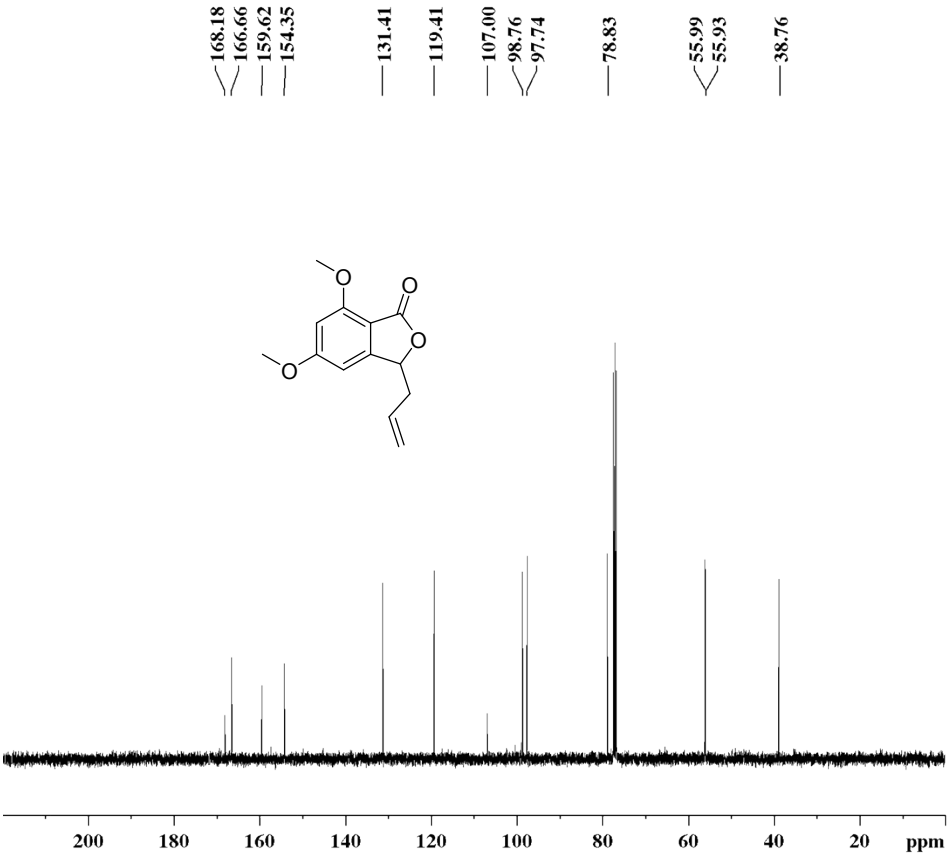



```
Current Data Parameters
NAME          SVK-3-212
EXPNO            323011
PROCNO                1

F2 - Acquisition Parameters
Date_          20170323
Time              15.52
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   10
DS                    0
SWH            6393.862 Hz
FIDRES        0.195125 Hz
AQ            2.5624576 sec
RG                  645
DW               78.200 usec
DE                6.00 usec
TE                294.5 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                11.05 usec
PL1               -3.00 dB
SFO1        400.1324008 MHz

F2 - Processing parameters
SI                16384
SF          400.1300091 MHz
WDW                  EM
SSB       0
LB        0 Hz
GB        0
PC                 1.00
```



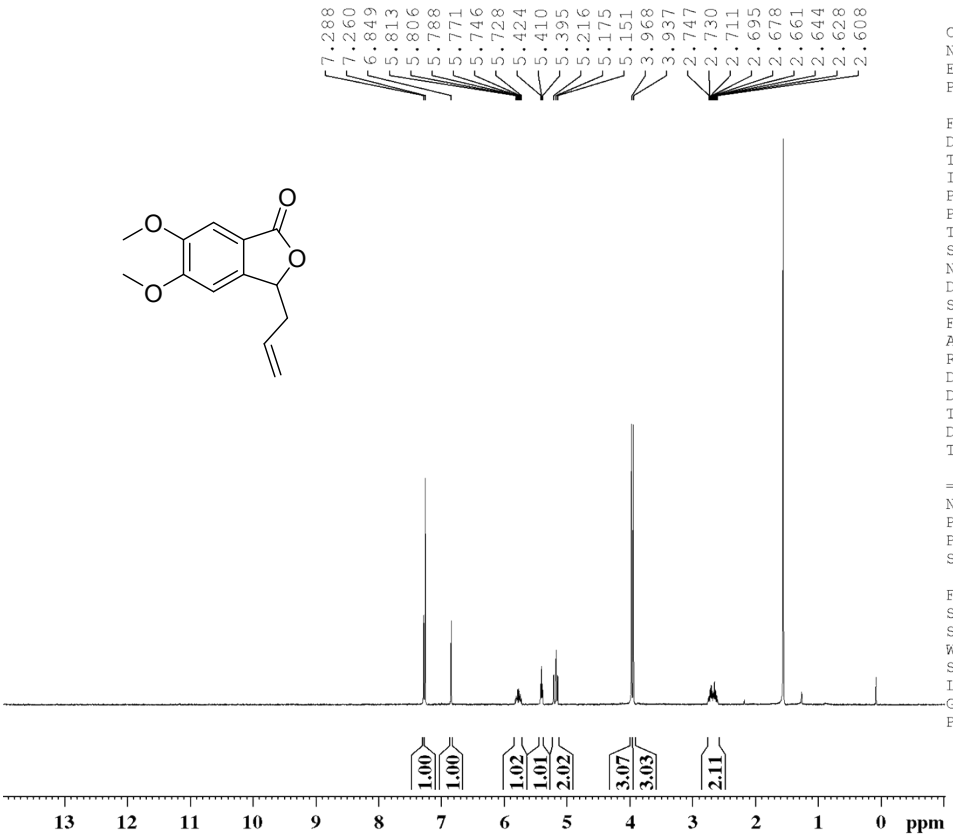

```
Current Data Parameters
NAME      SVK-3-212-new 13C
EXPNO             830031
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170830
Time              16.21
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  165
DS                    0
SWH           25252.525 Hz
FIDRES         0.385323 Hz
AQ            1.2976128 sec
RG                 2050
DW               19.800 usec
DE                6.50 usec
TE                299.8 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                10.00 usec
PL1                6.20 dB
SFO1        100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2             90.00 usec
PL2               -0.40 dB
PL12              15.80 dB
PL13              18.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

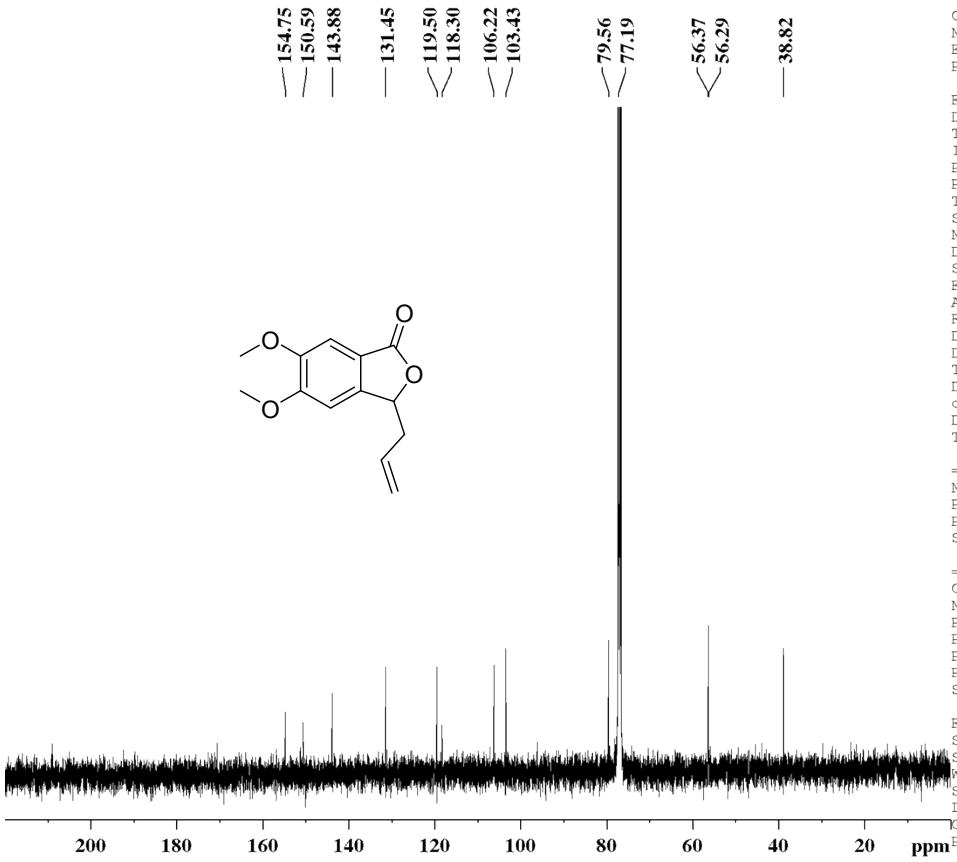



```
Current Data Parameters
NAME          SVK-3-285
EXPNO            727021
PROCNO                1

F2 - Acquisition Parameters
Date_          20170727
Time              23.52
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   10
DS                    0
SWH            6009.615 Hz
FIDRES        0.183399 Hz
AQ           2.7262976 sec
RG                 1030
DW               83.200 usec
DE                 6.50 usec
TE                302.5 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.00 usec
PL1                0.90 dB
SFO1        400.1326008 MHz

F2 - Processing parameters
SI                 16384
SF          400.1300090 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



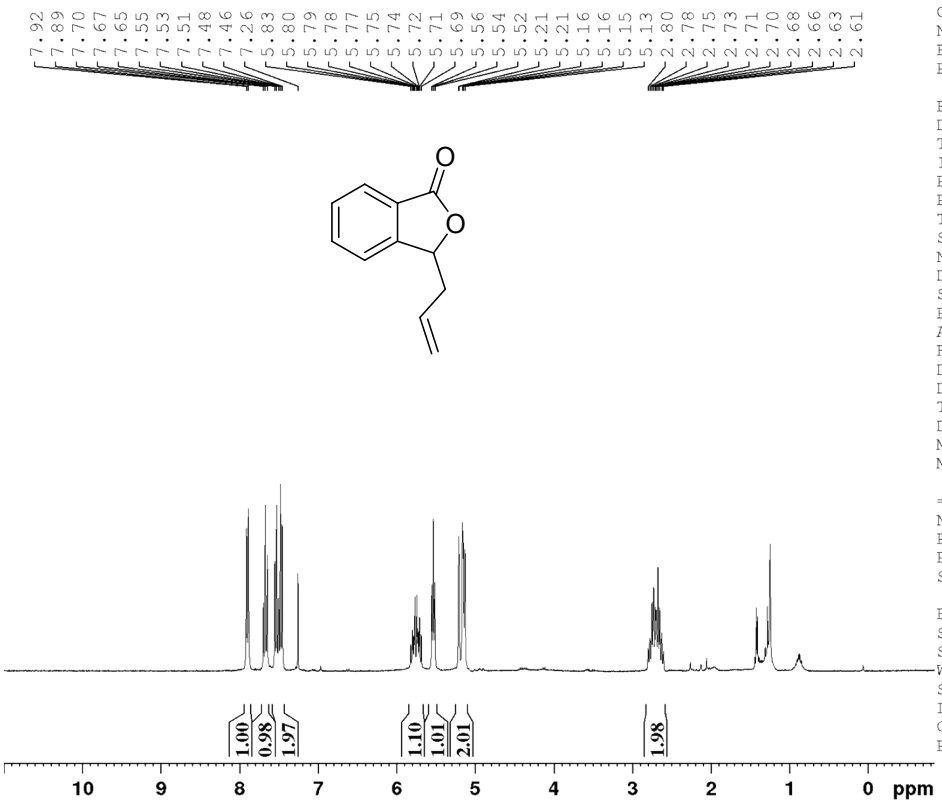

```
Current Data Parameters
NAME       SVK-3-285 13C
EXPNO             727021
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170727
Time              23.57
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 9999
DS                    0
SWH           25252.525 Hz
FIDRES         0.385323 Hz
AQ           1.2976128 sec
RG                 2050
DW               19.800 usec
DE                6.50 usec
TE               302.7 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PL1                6.20 dB
SFO1        100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2               -0.40 dB
PL12              15.80 dB
PL13              18.80 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

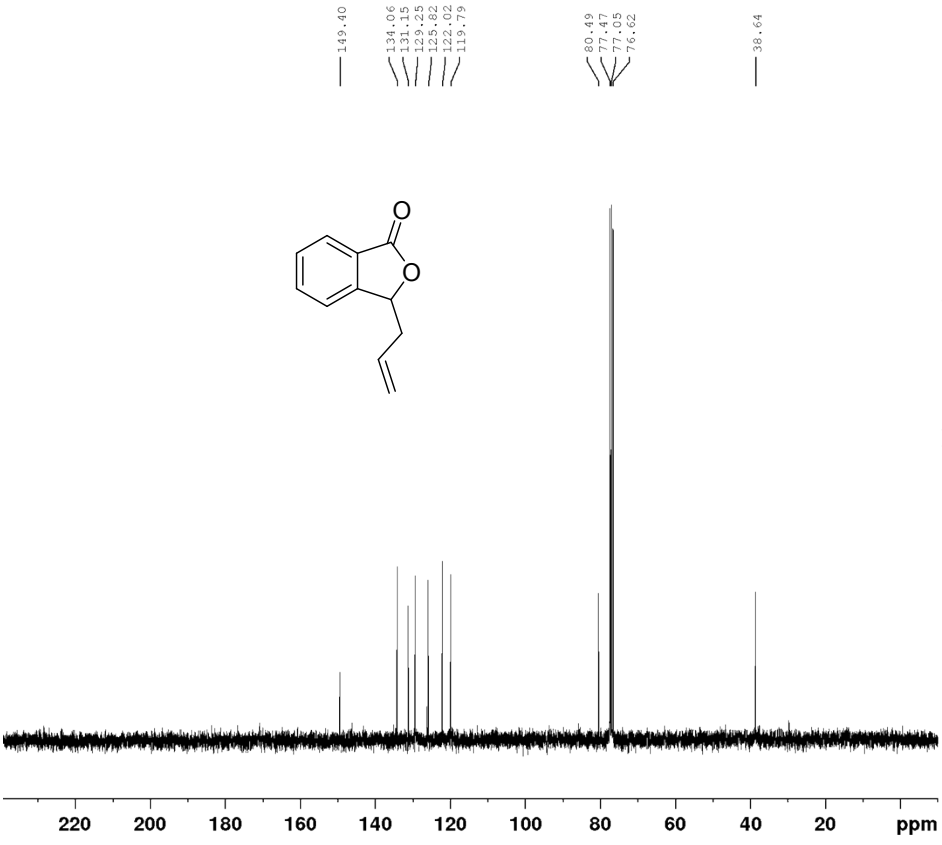



```
Current Data Parameters
NAME           SVK-3-269
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170523
Time               14.44
INSTRUM            spect
PROBHD   5 mm Multinucl
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     8
DS                     0
SWH             4496.403 Hz
FIDRES          0.137219 Hz
AQ              3.6438017 sec
RG                   512
DW                111.200 usec
DE                  6.50 usec
TE                298.2 K
D1             1.50000000 sec
MCREST    0 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                 10.60 usec
PL1        0 dB
SFO1          300.1319508 MHz

F2 - Processing parameters
SI                 16384
SF            300.1300060 MHz
WDW                   EM
SSB        0
LB                  0.10 Hz
GB         0
PC                  1.00
```



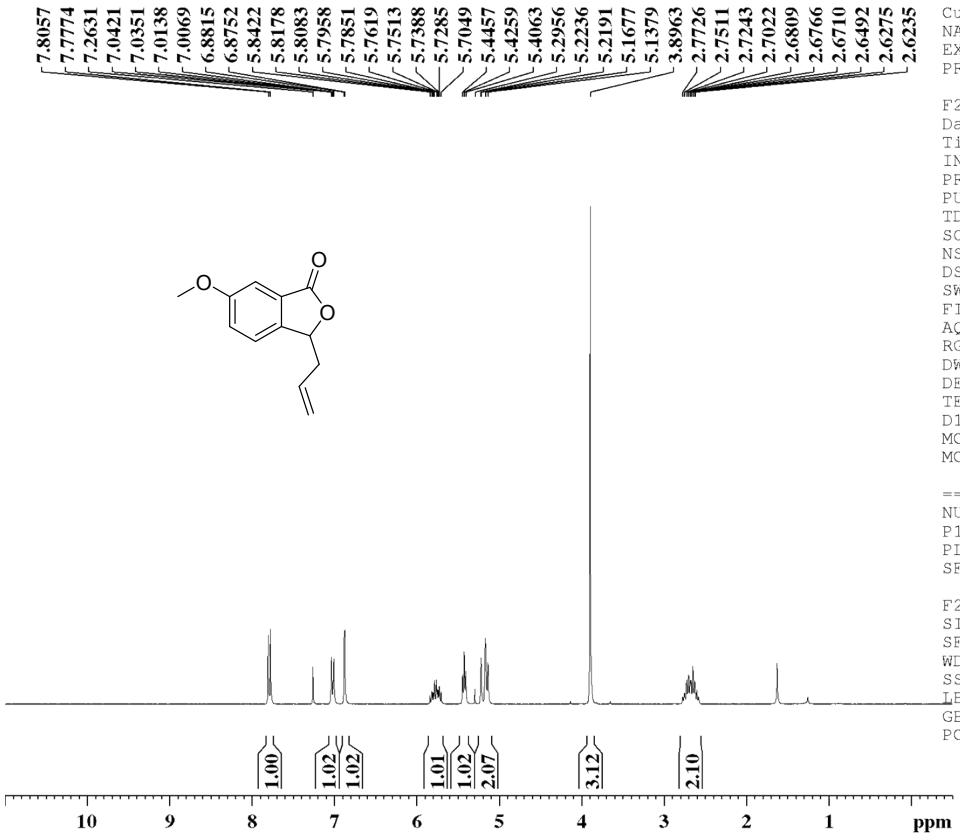

```
Current Data Parameters
NAME       SVK-3-269 13C
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20170523
Time               16.45
INSTRUM            spect
PROBHD   5 mm Multinucl
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   271
DS                     0
SWH            18832.393 Hz
FIDRES          0.287360 Hz
AQ             1.7399808 sec
RG                5792.6
DW                26.550 usec
DE                 6.50 usec
TE                298.2 K
D1            1.50000000 sec
d11           0.03000000 sec
DELTA         1.39999998 sec
MCREST    0 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                11.10 usec
PL1       0 dB
SFO1          75.4763978 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2       0 dB
PL12              18.80 dB
PL13              21.80 dB
SFO2         300.1313506 MHz

F2 - Processing parameters
SI                32768
SF            75.4677477 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.00
```

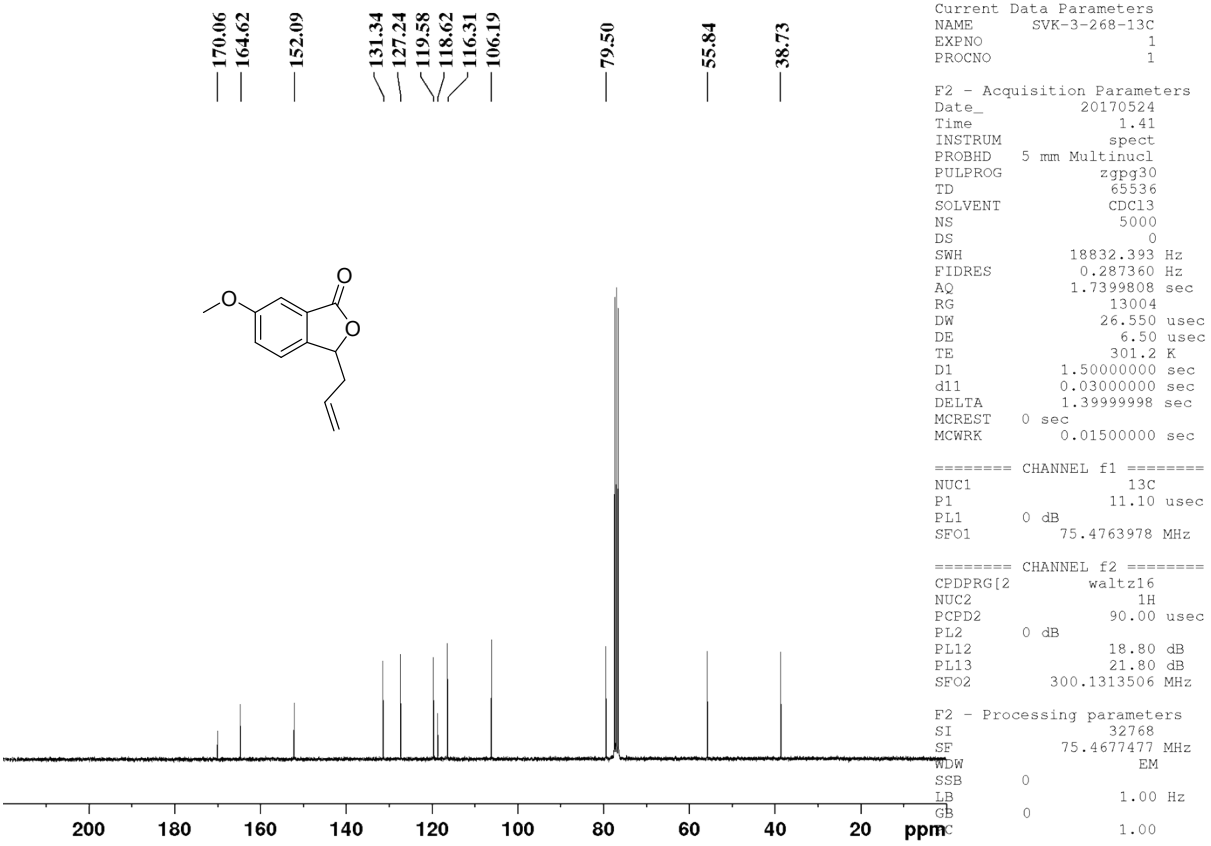



```
Current Data Parameters
NAME          SVK-3-268
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20170524
Time               1.37
INSTRUM           spect
PROBHD   5 mm Multinucl
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                    8
DS                    0
SWH            4496.403 Hz
FIDRES         0.137219 Hz
AQ             3.6438017 sec
RG                645.1
DW               111.200 usec
DE                 6.50 usec
TE                301.2 K
D1             1.50000000 sec
MCREST   0 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                10.60 usec
PL1       0 dB
SFO1         300.1319508 MHz

F2 - Processing parameters
SI                16384
SF          300.1300049 MHz
WDW                  EM
SSB       0
LB                 0.10 Hz
GB        0
PC                 1.00
```



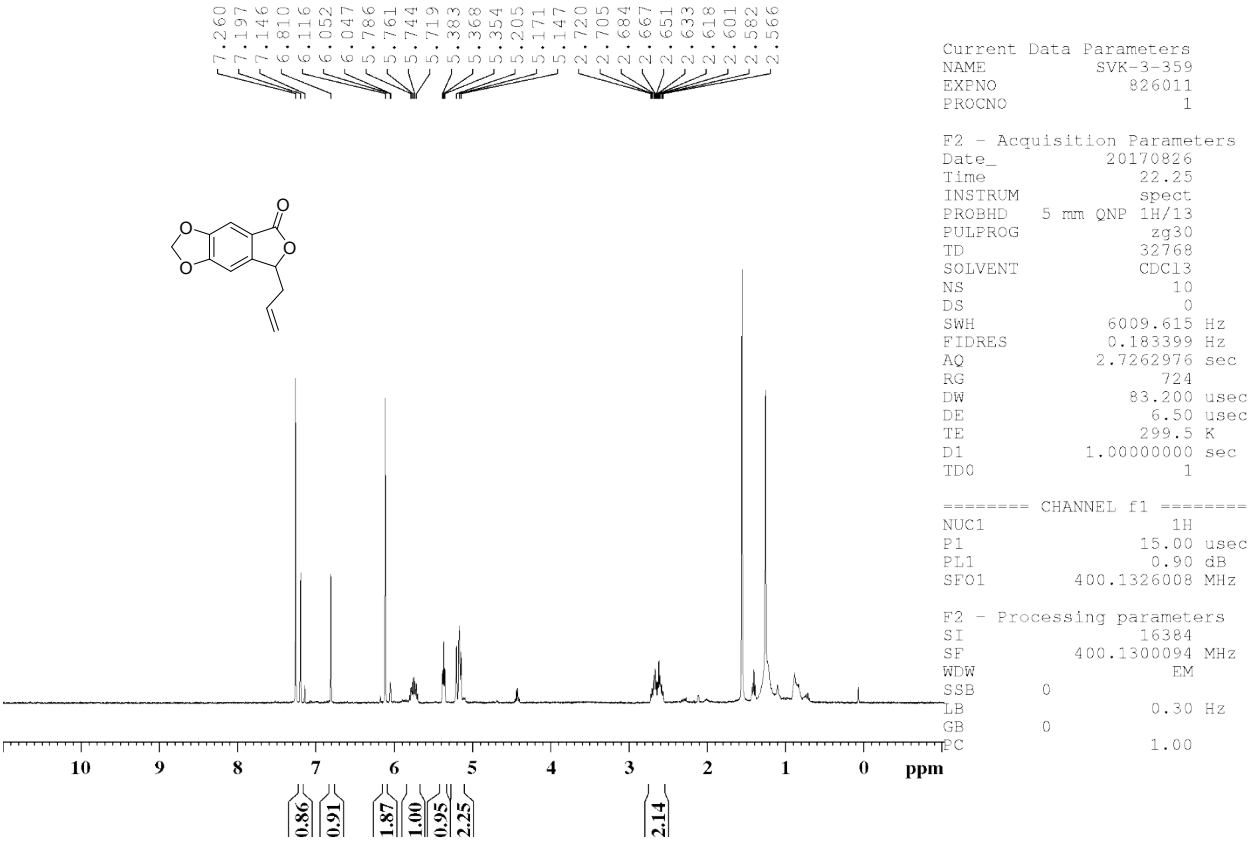

```
Current Data Parameters
NAME       SVK-3-268-13C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170524
Time                1.41
INSTRUM            spect
PROBHD   5 mm Multinucl
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  5000
DS                     0
SWH           18832.393 Hz
FIDRES         0.287360 Hz
AQ            1.7399808 sec
RG                 13004
DW               26.550 usec
DE                6.50 usec
TE                301.2 K
D1           1.50000000 sec
d11          0.03000000 sec
DELTA        1.39999998 sec
MCREST   0 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                11.10 usec
PL1      0 dB
SFO1         75.4763978 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2      0 dB
PL12              18.80 dB
PL13              21.80 dB
SFO2        300.1313506 MHz

F2 - Processing parameters
SI                  32768
SF           75.4677477 MHz
WDW                    EM
SSB      0
LB                  1.00 Hz
GB       0
PC                  1.00
```

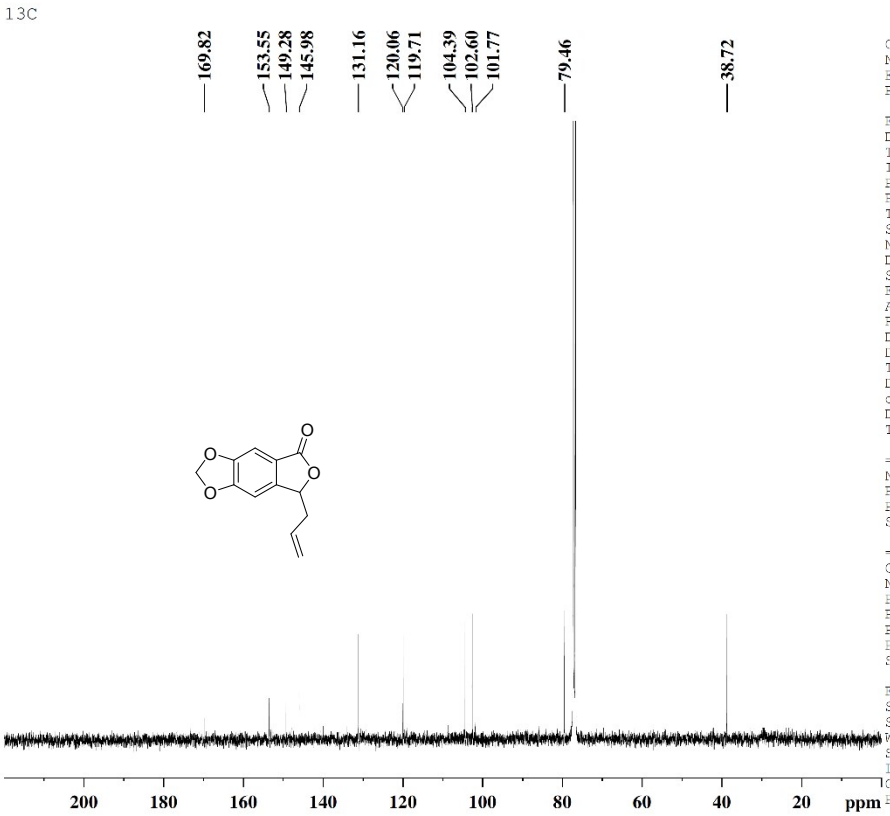


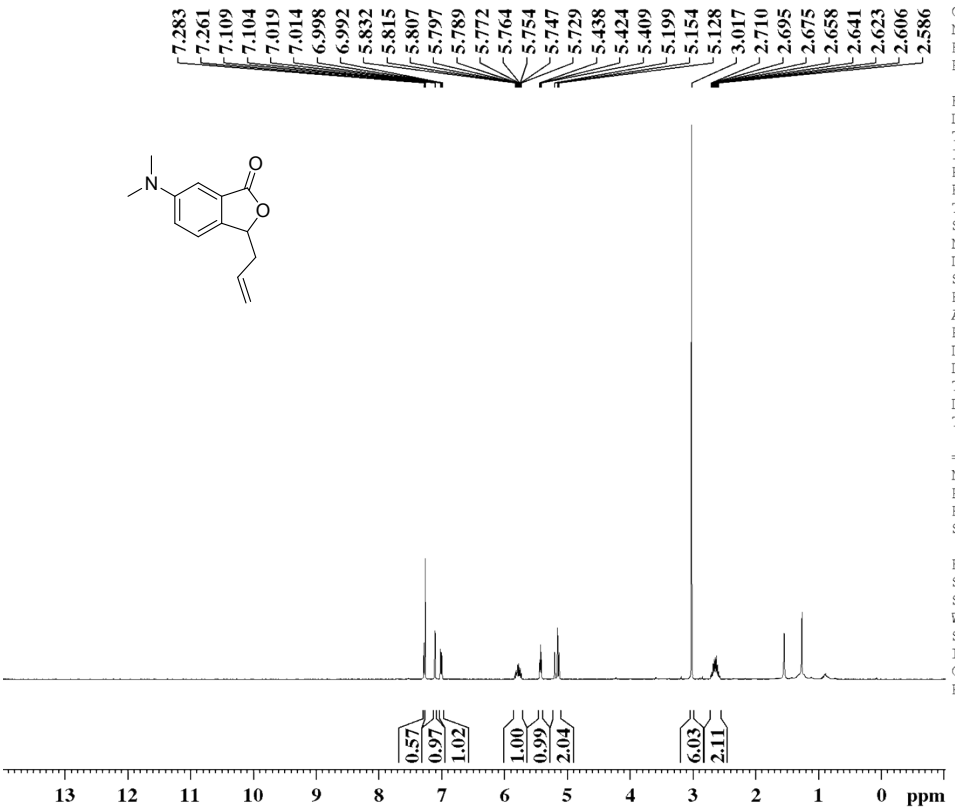

```
Current Data Parameters
NAME                  SVK-3-359
EXPNO                    826011
PROCNO                        1

F2 - Acquisition Parameters
Date_                 20170826
Time                      22.25
INSTRUM                   spect
PROBHD     5 mm QNP 1H/13
PULPROG                   zg30
TD                       32768
SOLVENT                  CDCl3
NS                          10
DS                           0
SWH                  6009.615 Hz
FIDRES               0.183399 Hz
AQ                   2.7262976 sec
RG                         724
DW                      83.200 usec
DE                       6.50 usec
TE                      299.5 K
D1                 1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                         1H
P1                       15.00 usec
PL1                       0.90 dB
SFO1               400.1326008 MHz

F2 - Processing parameters
SI                       16384
SF                 400.1300094 MHz
WDW                         EM
SSB             0
LB                        0.30 Hz
GB              0
PC                        1.00
```


13C
169.82
153.55
149.28
145.98
131.16
120.06
119.71
104.39
102.60
101.77
79.46
38.72
200
180
160
140
120
100
80
60
40
20
ppm
Current Data Parameters
NAME SVK-3-359 13C
EXPNO 826011
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170827
Time 1.18
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 9999
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976128 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 302.4 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 6.20 dB
SFO1 100.6243395 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
PL13 18.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

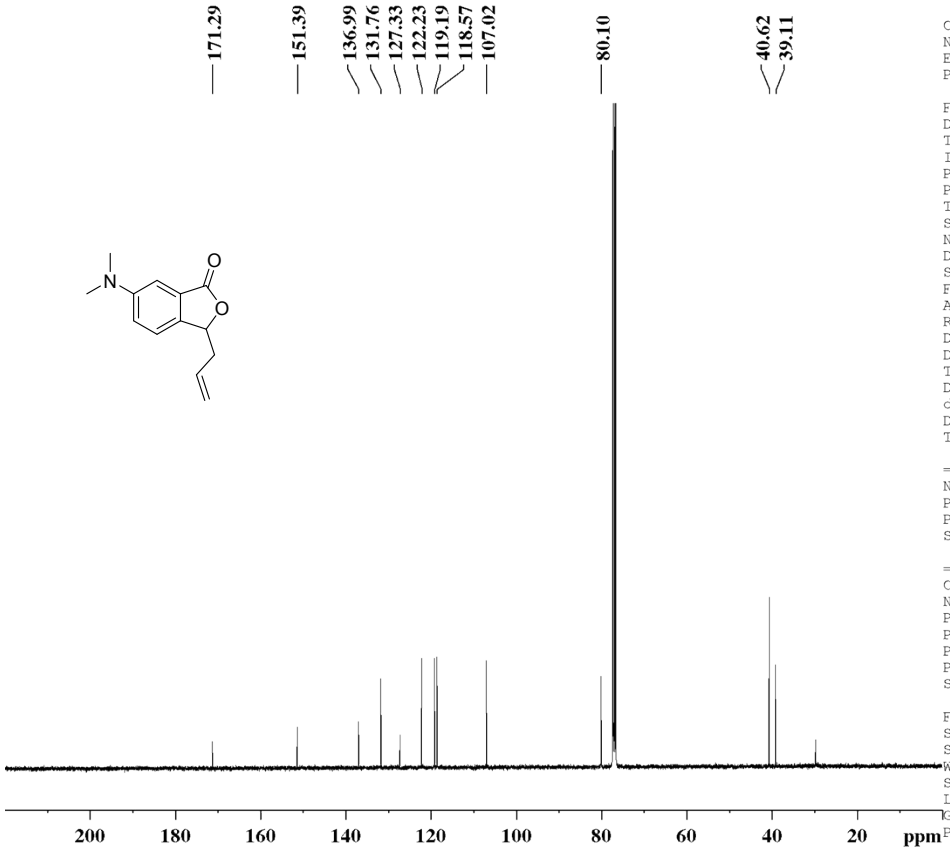



```
Current Data Parameters
NAME                  SVK-4-007
EXPNO                    813011
PROCNO                        1

F2 - Acquisition Parameters
Date_                 20170813
Time                     22.55
INSTRUM                  spect
PROBHD   5 mm QNP 1H/13
PULPROG                  zg30
TD                      32768
SOLVENT                  CDCl3
NS                          10
DS                           0
SWH                   6009.615 Hz
FIDRES                0.183399 Hz
AQ                    2.7262976 sec
RG                         724
DW                      83.200 usec
DE                       6.50 usec
TE                      307.1 K
D1                    1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                         1H
P1                       15.00 usec
PL1                       0.90 dB
SFO1                400.1326008 MHz

F2 - Processing parameters
SI                       16384
SF                  400.1300087 MHz
WDW                         EM
SSB       0
LB                        0.30 Hz
GB        0
PC                        1.00
```



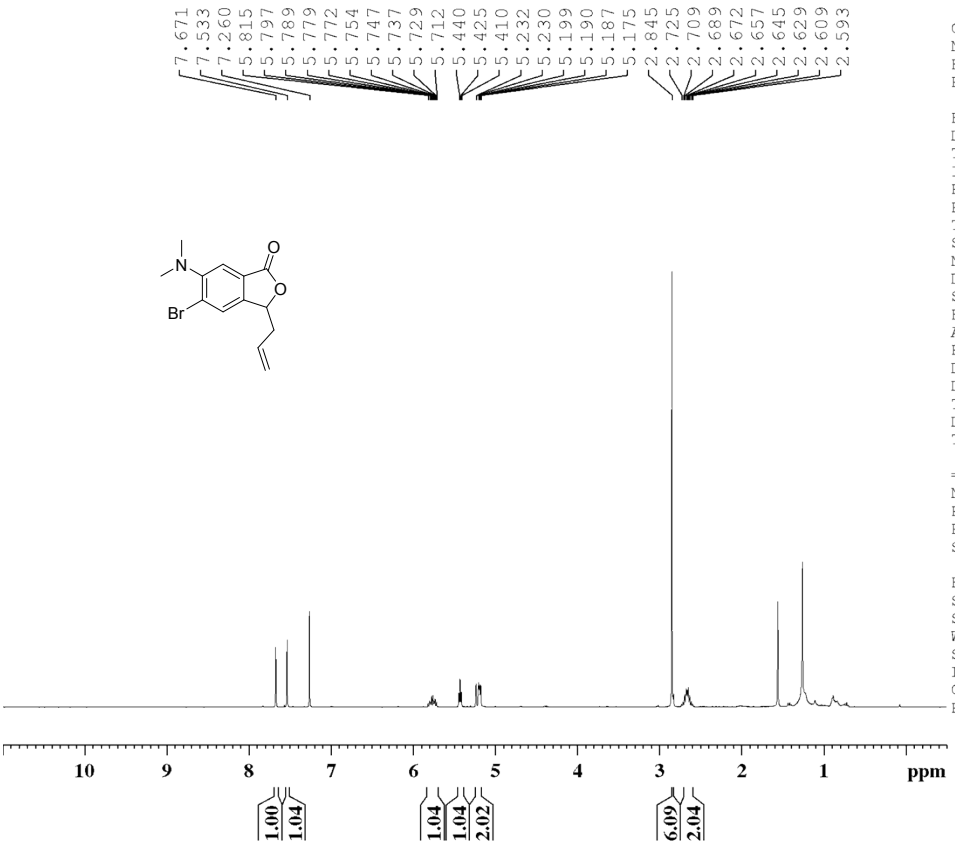

```
Current Data Parameters
NAME       SVK-4-007 13C
EXPNO             813011
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170813
Time              22.59
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                 9999
DS                    0
SWH           25252.525 Hz
FIDRES         0.385323 Hz
AQ            1.2976128 sec
RG                 2050
DW               19.800 usec
DE                6.50 usec
TE               307.2 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PL1                6.20 dB
SFO1        100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2               -0.40 dB
PL12              15.80 dB
PL13              18.80 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

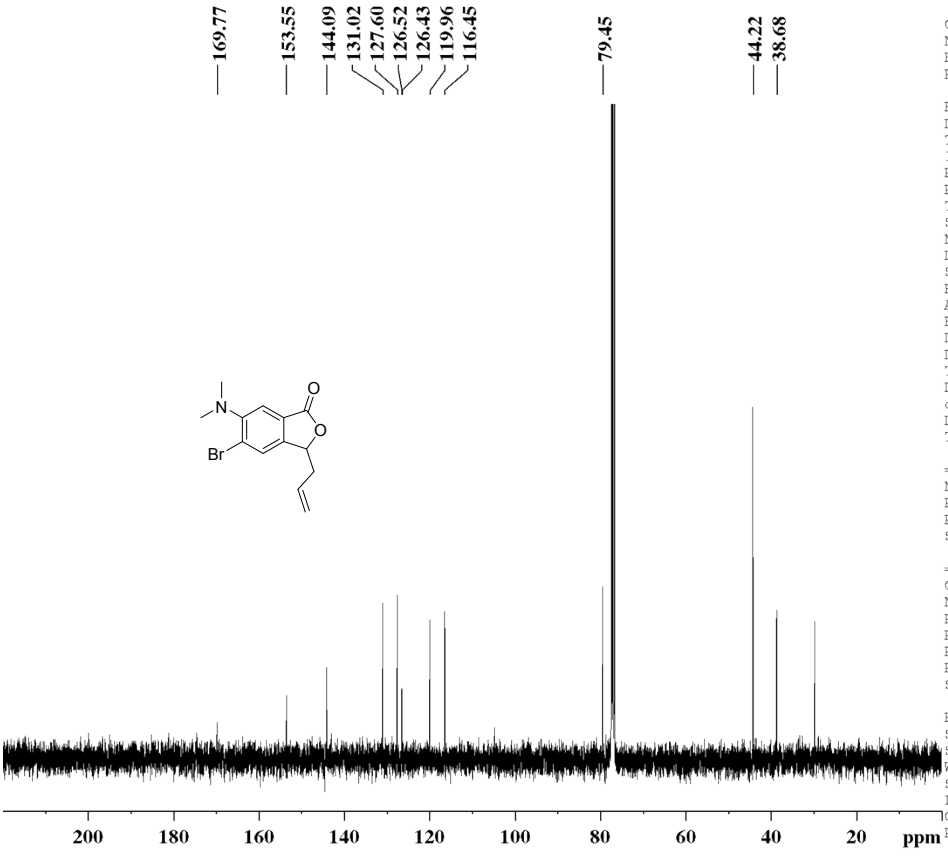



```
Current Data Parameters
NAME          SVK-3-361
EXPNO            828011
PROCNO                1

F2 - Acquisition Parameters
Date_          20170828
Time              22.30
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   10
DS                    0
SWH            6009.615 Hz
FIDRES        0.183399 Hz
AQ            2.7262976 sec
RG                  645
DW               83.200 usec
DE                6.50 usec
TE               300.0 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.00 usec
PL1                0.90 dB
SFO1        400.1326008 MHz

F2 - Processing parameters
SI                16384
SF          400.1300094 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



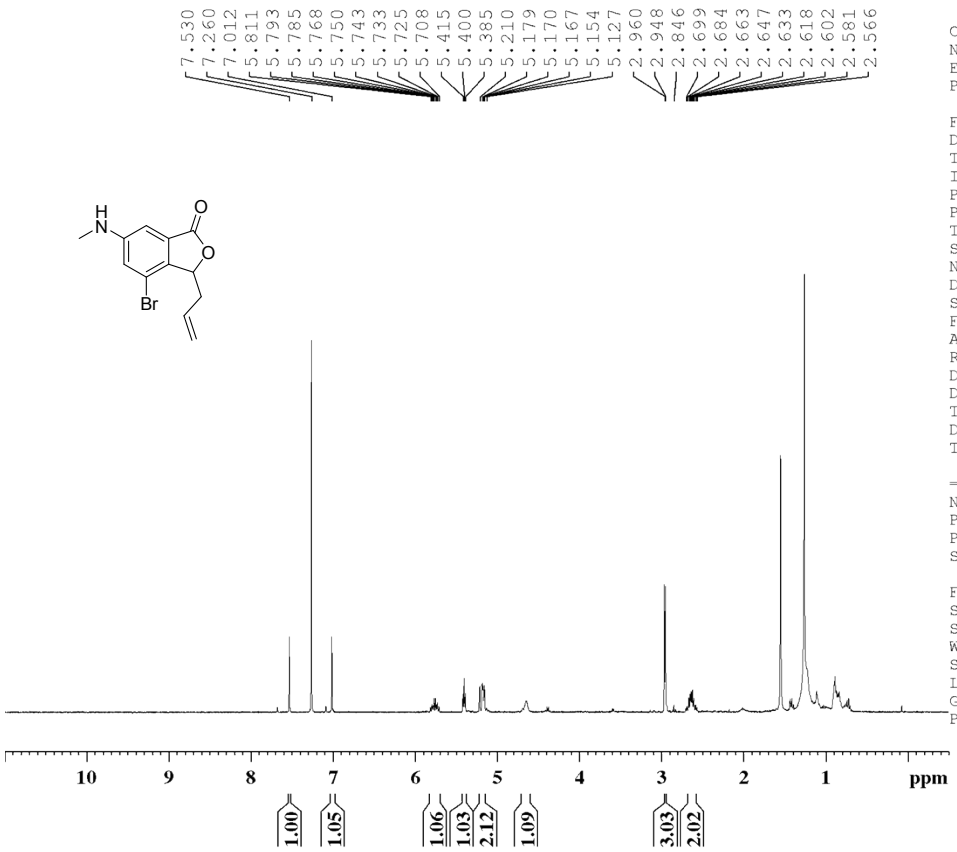

```
Current Data Parameters
NAME      SVK-3-361 13C
EXPNO           828011
PROCNO               1

F2 - Acquisition Parameters
Date_          20170829
Time             12.13
INSTRUM          spect
PROBHD   5 mm QNP 1H/13
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                1023
DS                   0
SWH          25252.525 Hz
FIDRES        0.385323 Hz
AQ           1.2976128 sec
RG                2050
DW              19.800 usec
DE               6.50 usec
TE              300.4 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1               10.00 usec
PL1               6.20 dB
SFO1       100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG[2        waltz16
NUC2               1H
PCPD2            90.00 usec
PL2              -0.40 dB
PL12             15.80 dB
PL13             18.50 dB
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                32768
SF         100.6127690 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

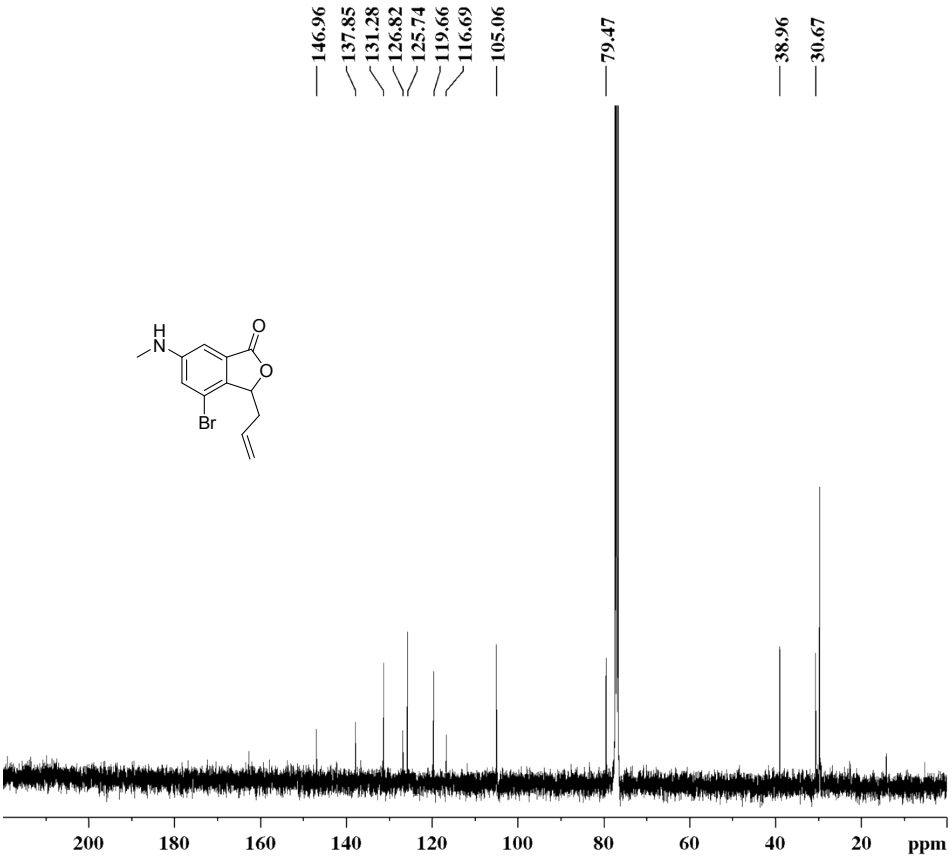



```
Current Data Parameters
NAME            SVK-3-362
EXPNO              828011
PROCNO                  1

F2 - Acquisition Parameters
Date_            20170828
Time                22.36
INSTRUM             spect
PROBHD   5 mm QNP 1H/13
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     10
DS                      0
SWH              6009.615 Hz
FIDRES           0.183399 Hz
AQ             2.7262976 sec
RG                    812
DW                 83.200 usec
DE                   6.50 usec
TE                  300.1 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  15.00 usec
PL1                  0.90 dB
SFO1         400.1326008 MHz

F2 - Processing parameters
SI                  16384
SF           400.1300093 MHz
WDW                    EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
```



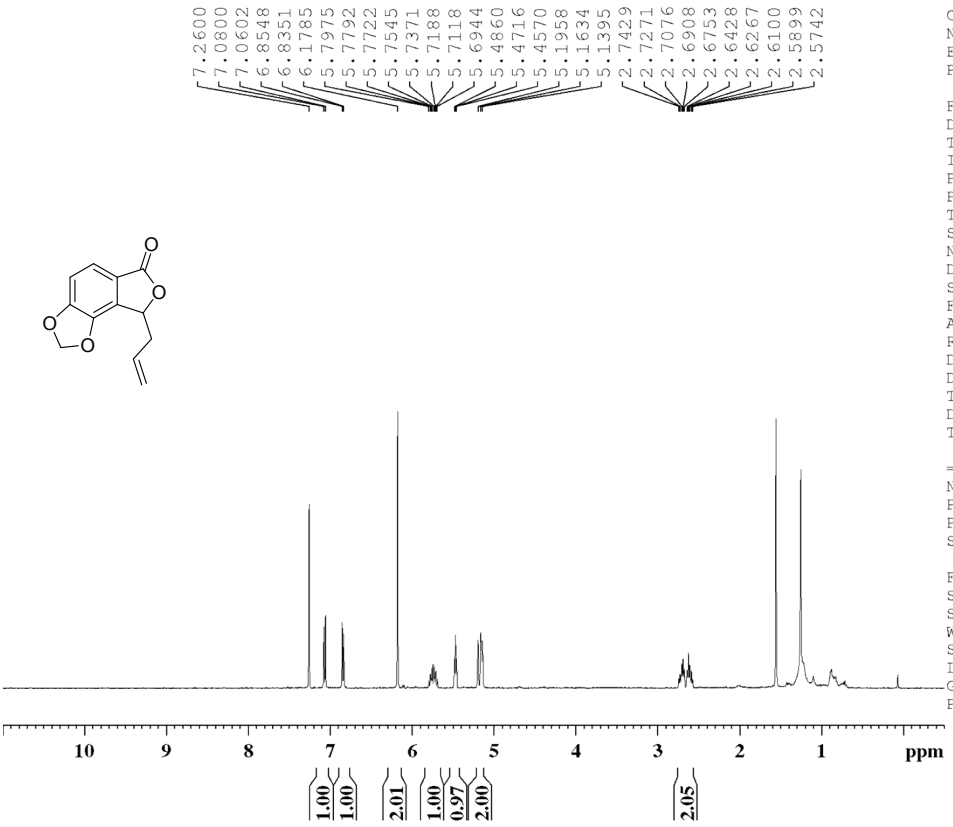

```
Current Data Parameters
NAME      SVK-3-362 2nd time 13C
EXPNO              829021
PROCNO                  1

F2 - Acquisition Parameters
Date_           20170829
Time              23.47
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 9999
DS                    0
SWH           25252.525 Hz
FIDRES         0.385323 Hz
AQ            1.2976128 sec
RG                 2050
DW               19.800 usec
DE                6.50 usec
TE               300.8 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PL1                6.20 dB
SFO1        100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2               -0.40 dB
PL12              15.80 dB
PL13              18.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

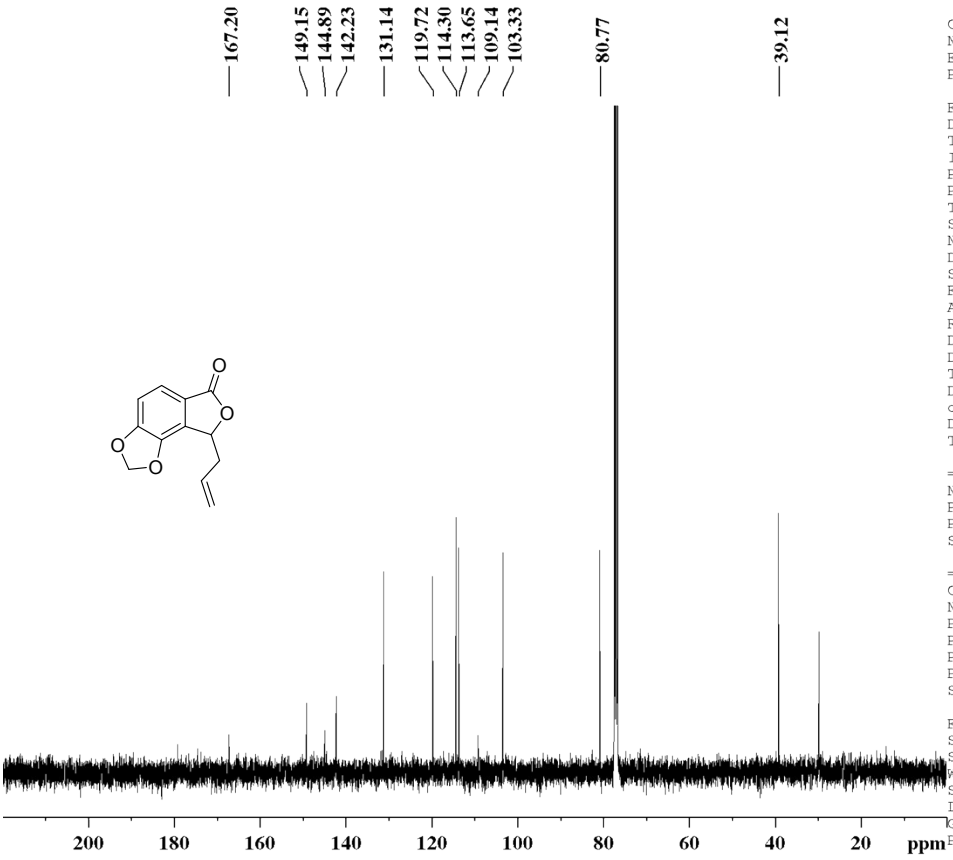



```
Current Data Parameters
NAME          SVK-3-360
EXPNO            826011
PROCNO                1

F2 - Acquisition Parameters
Date_          20170826
Time              22.17
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   10
DS                    0
SWH            6009.615 Hz
FIDRES        0.183399 Hz
AQ           2.7262976 sec
RG                  645
DW               83.200 usec
DE                 6.50 usec
TE                299.3 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.00 usec
PL1                0.90 dB
SFO1        400.1326008 MHz

F2 - Processing parameters
SI                16384
SF          400.1300094 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



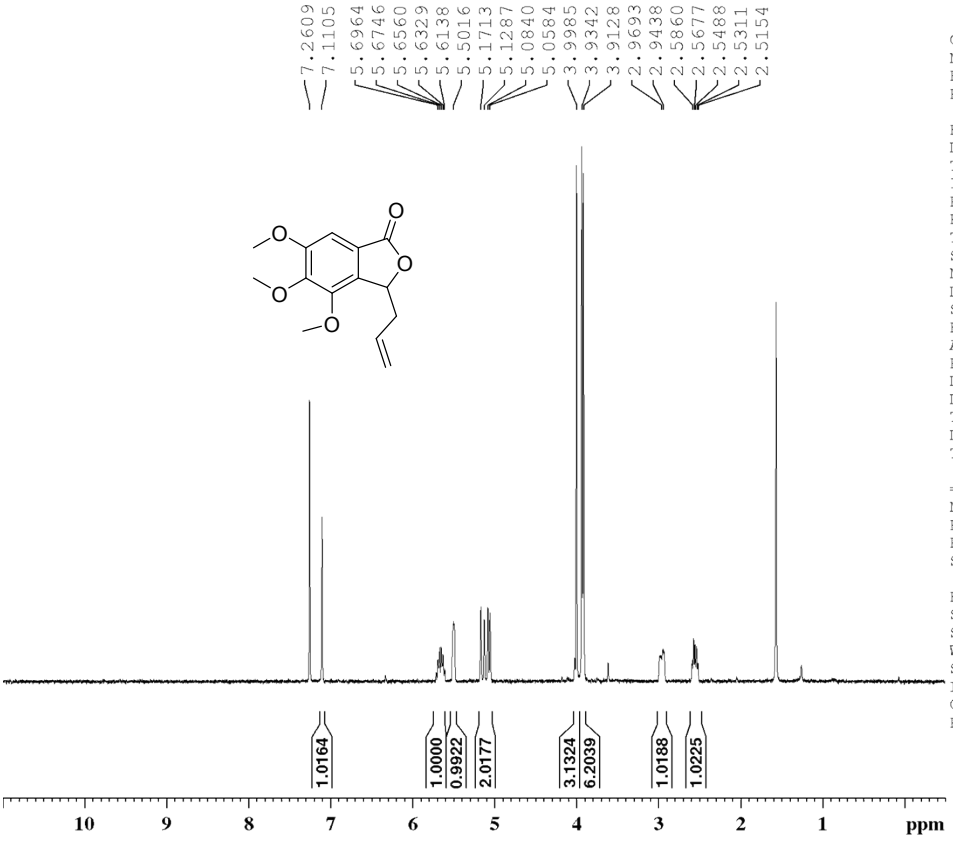

```
Current Data Parameters
NAME      SVK-3-360 13C
EXPNO           826011
PROCNO               1

F2 - Acquisition Parameters
Date_         20170826
Time             22.36
INSTRUM          spect
PROBHD   5 mm QNP 1H/13
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                2889
DS                   0
SWH          25252.525 Hz
FIDRES        0.385323 Hz
AQ           1.2976128 sec
RG                2050
DW              19.800 usec
DE               6.50 usec
TE              300.1 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1               10.00 usec
PL1               6.20 dB
SFO1        100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2            90.00 usec
PL2              -0.40 dB
PL12             15.80 dB
PL13             18.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

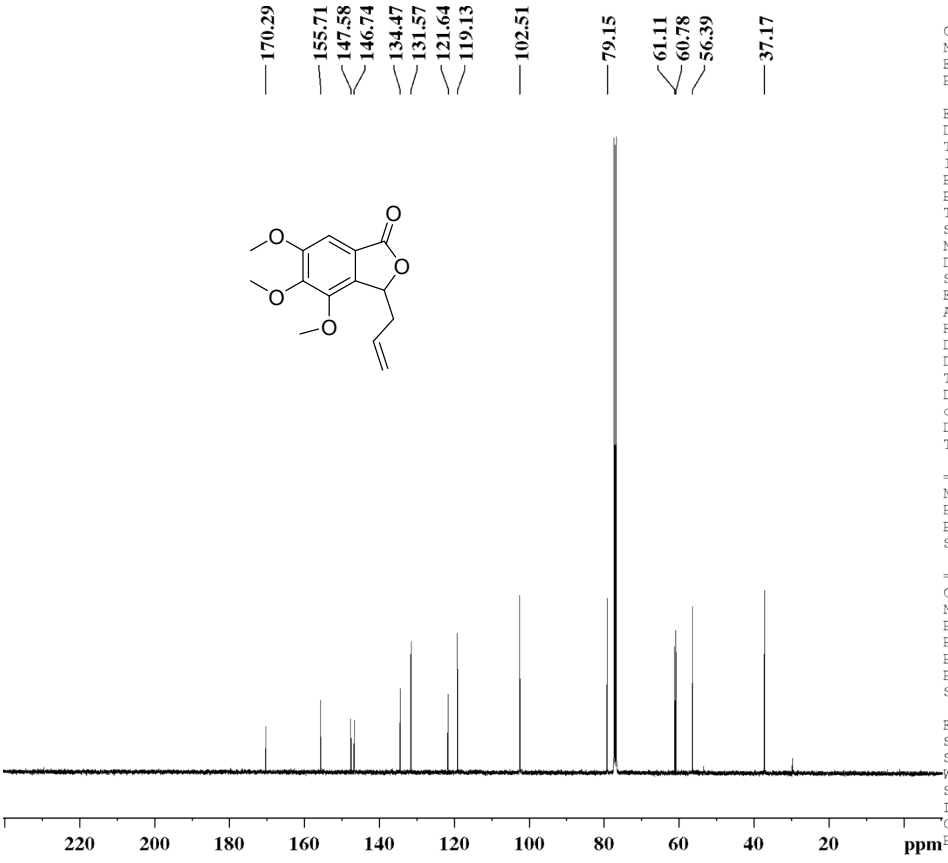



```
Current Data Parameters
NAME         SVK-3-396-I
EXPNO            1213011
PROCNO                 1

F2 - Acquisition Parameters
Date_           20171213
Time                1.38
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    10
DS                     0
SWH             6009.615 Hz
FIDRES          0.183399 Hz
AQ             2.7262976 sec
RG                   101
DW                83.200 usec
DE                 6.50 usec
TE                297.2 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.00 usec
PL1                0.90 dB
SFO1         400.1326008 MHz

F2 - Processing parameters
SI                 16384
SF          400.1300079 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



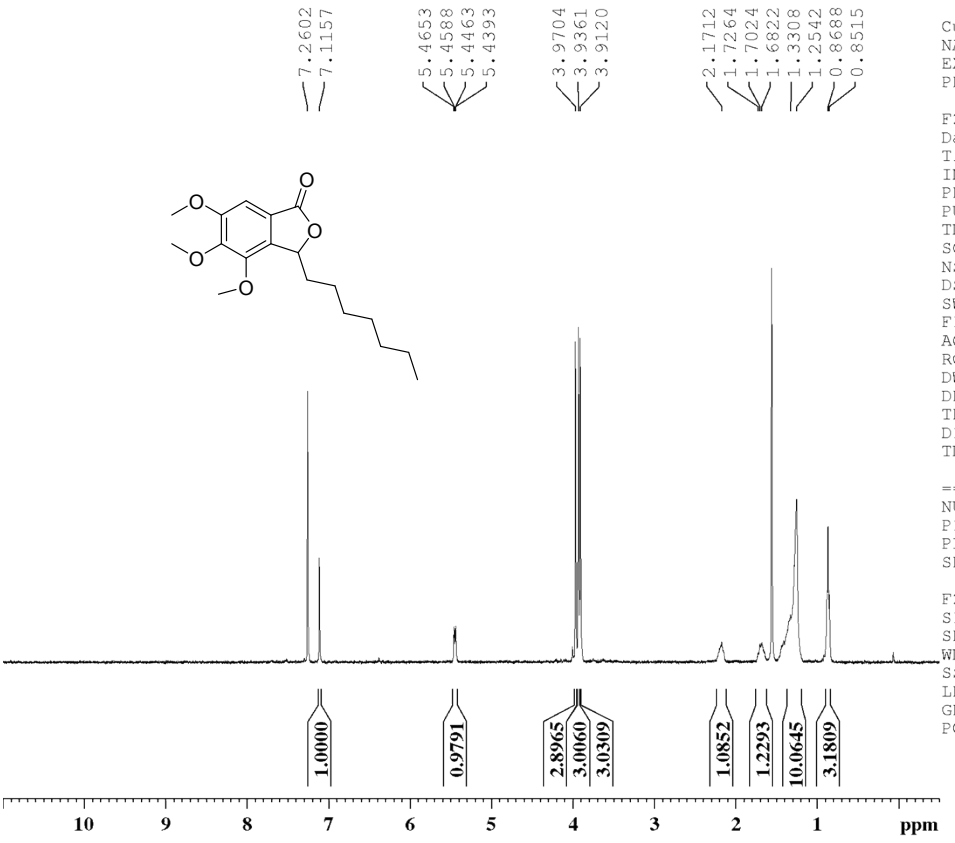

```
Current Data Parameters
NAME      SVK-3-377-I 13C
EXPNO            1025011
PROCNO                 1

F2 - Acquisition Parameters
Date_           20171025
Time               10.42
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 2915
DS                    0
SWH           25252.525 Hz
FIDRES         0.385323 Hz
AQ            1.2976128 sec
RG                 2050
DW               19.800 usec
DE                6.50 usec
TE                299.9 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PL1                6.20 dB
SFO1        100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2               -0.40 dB
PL12              15.80 dB
PL13              18.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

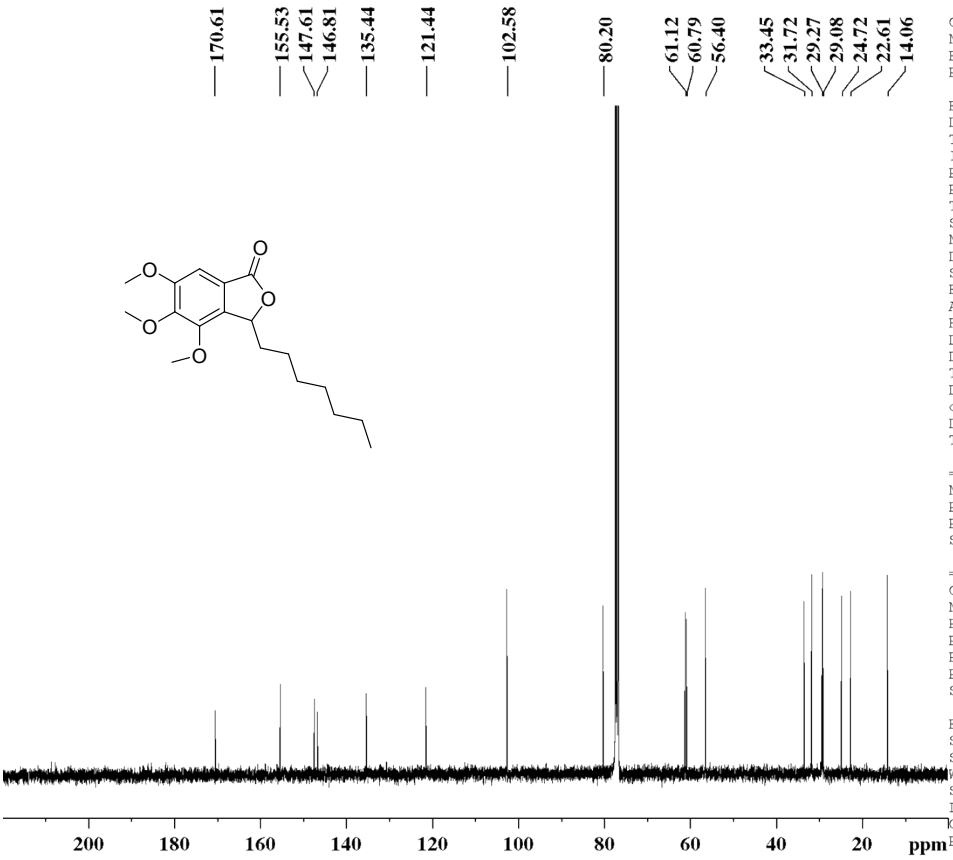



```
Current Data Parameters
NAME          SVK-4-061
EXPNO           1219011
PROCNO                1

F2 - Acquisition Parameters
Date_          20171219
Time              11.26
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                  15
DS                   0
SWH           6009.615 Hz
FIDRES        0.183399 Hz
AQ           2.7262976 sec
RG                 812
DW              83.200 usec
DE               6.50 usec
TE              297.0 K
D1           2.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1              15.00 usec
PL1              0.90 dB
SFO1        400.1326008 MHz

F2 - Processing parameters
SI                16384
SF          400.1300084 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



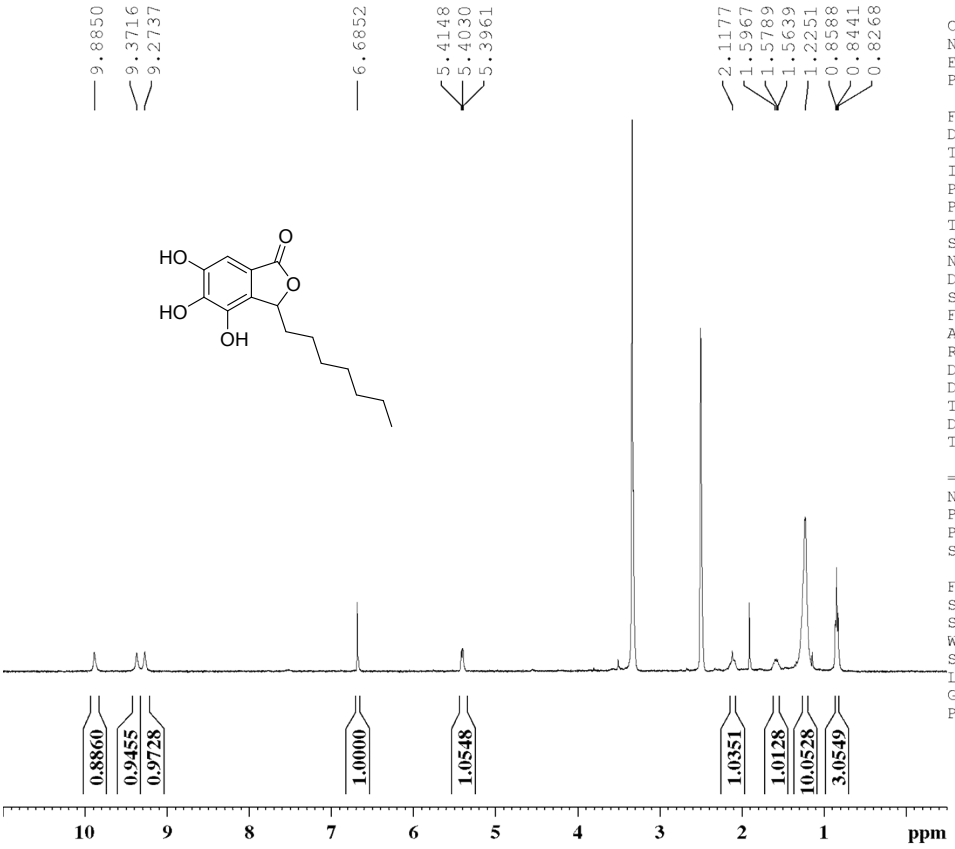

```
Current Data Parameters
NAME           SVK-I 13C
EXPNO            1215011
PROCNO                 1

F2 - Acquisition Parameters
Date_           20171215
Time                3.43
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 5000
DS                    0
SWH           25252.525 Hz
FIDRES         0.385323 Hz
AQ            1.2976128 sec
RG                 2050
DW               19.800 usec
DE                6.50 usec
TE                297.6 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.00 usec
PL1                 6.20 dB
SFO1         100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2              90.00 usec
PL2                -0.40 dB
PL12               15.80 dB
PL13               18.50 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                  32768
SF           100.6127690 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

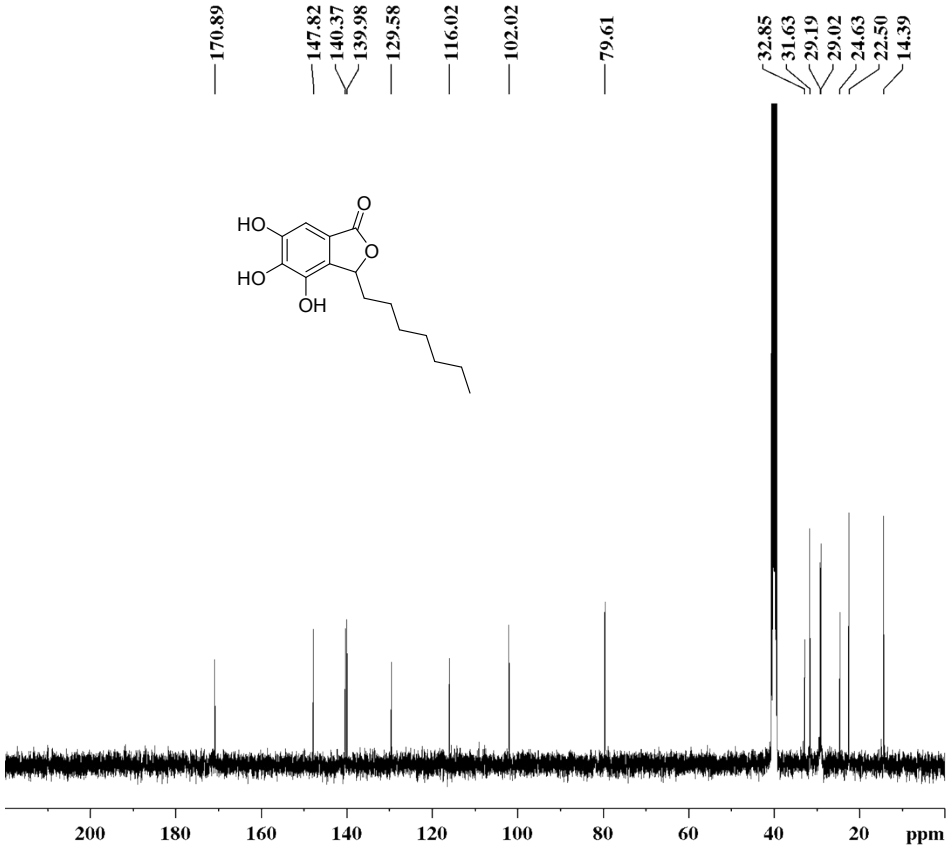



```
Current Data Parameters
NAME          SVK-4-062
EXPNO           1219011
PROCNO                1

F2 - Acquisition Parameters
Date_          20171219
Time              11.38
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   10
DS                    0
SWH            6009.615 Hz
FIDRES        0.183399 Hz
AQ            2.7262976 sec
RG                  645
DW               83.200 usec
DE                6.50 usec
TE                297.1 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1               15.00 usec
PL1               0.90 dB
SFO1        400.1326008 MHz

F2 - Processing parameters
SI                16384
SF          400.1300000 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```




```
Current Data Parameters
NAME      SVK-4-062 DSMO 13C
EXPNO            1216011
PROCNO                 1

F2 - Acquisition Parameters
Date_           20171216
Time               10.19
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1466
DS                    0
SWH           25252.525 Hz
FIDRES         0.385323 Hz
AQ            1.2976128 sec
RG                 2050
DW               19.800 usec
DE                6.50 usec
TE                297.8 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.00 usec
PL1                 6.20 dB
SFO1        100.6243395 MHz

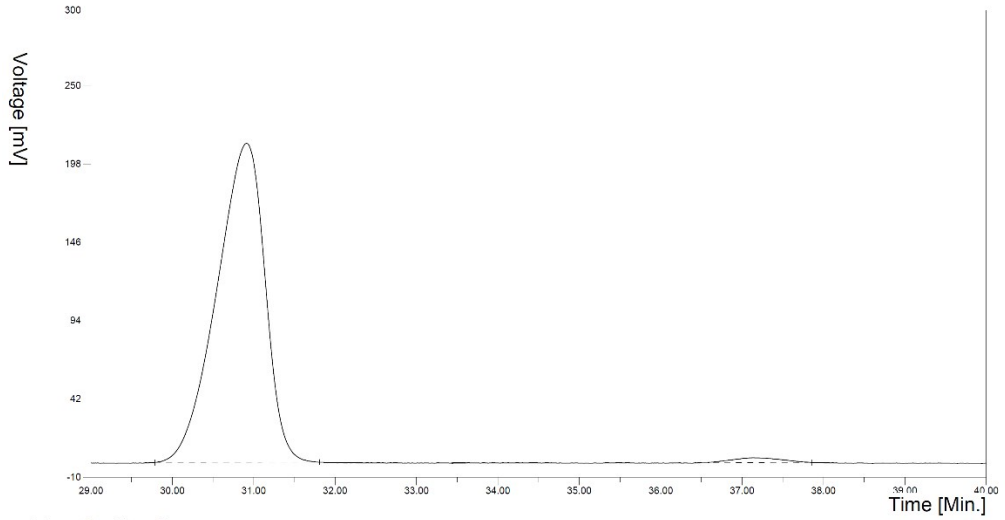
======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2              90.00 usec
PL2                -0.40 dB
PL12               15.80 dB
PL13               18.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

HPLC data:

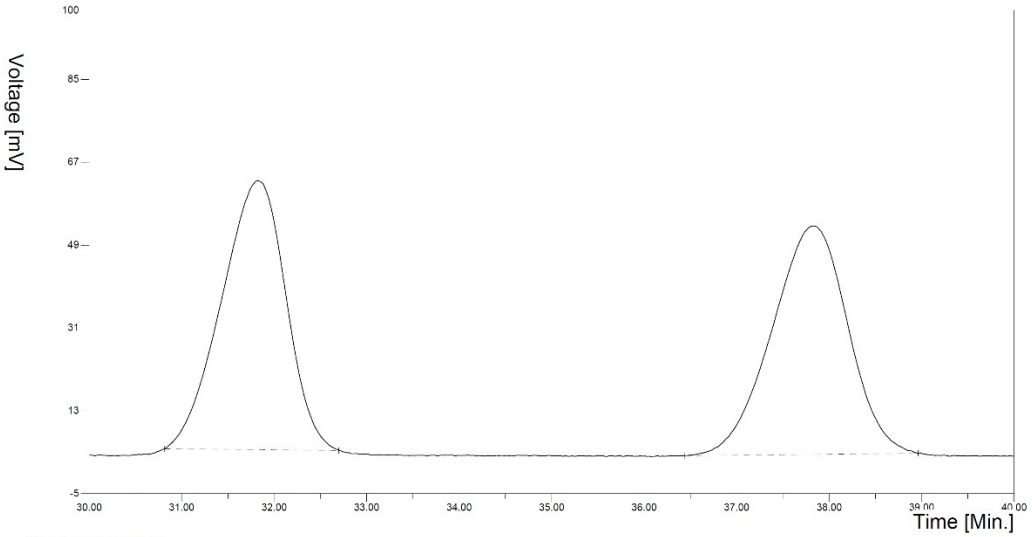
3-allyl-5,7-dimethoxyisobenzofuran-1(3*H*)-one (8)

HPLC conditions: Chiralcel OJ column, *n*-Hexane/2-Propanol (90/10), 1.0 mL/min, (t_{major} = 30.91 min, area% =98.4586), (t_{minor} =37.13 min , area% =1.5414), 254nm UV detector.



Integration Result

Ret. Time(min)	Area Percentage(%)
30.91	98.4586
37.13	1.5414
	100

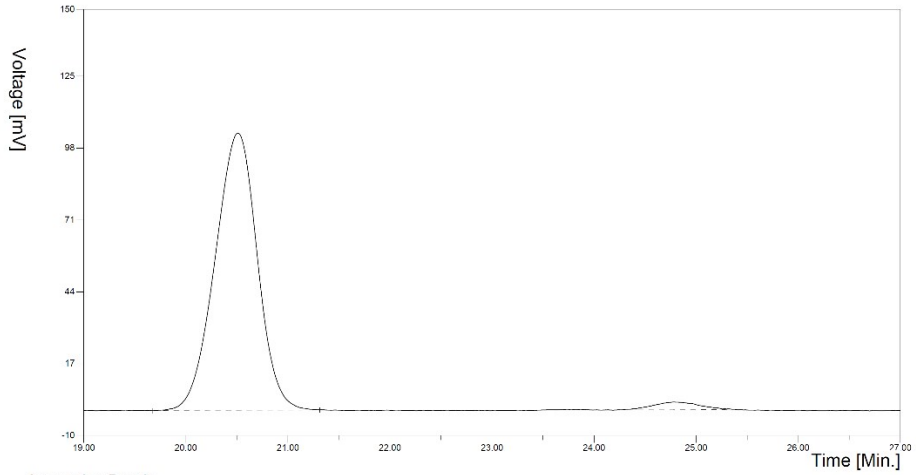


Integration Result

Ret. Time(min)	Area Percentage(%)
31.82	49.9627
37.83	50.0373
	100

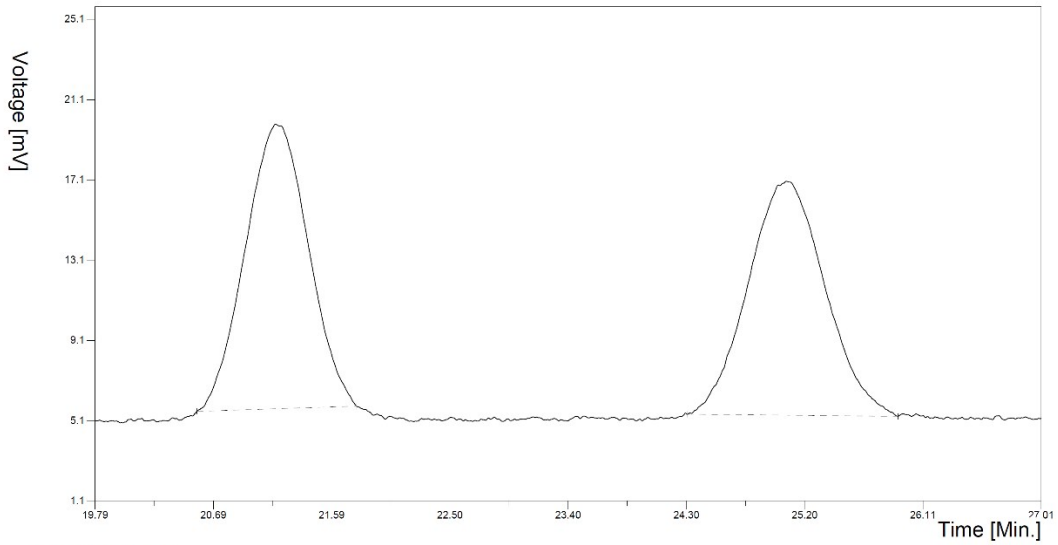
3-allyl-5,6-dimethoxyisobenzofuran-1(3*H*)-one (9)

HPLC conditions: Chiralcel OJ column, *n*-Hexane/2-Propanol (90/10), 1.0 mL/min, (t_{major} =20.51 min, area% =97.0743), (t_{minor} =24.78 min , area% =2.9257), 254nm UV detector.



Integration Result

Ret. Time(min)	Area Percentage(%)
20.51	97.0743
24.78	2.9257
	100

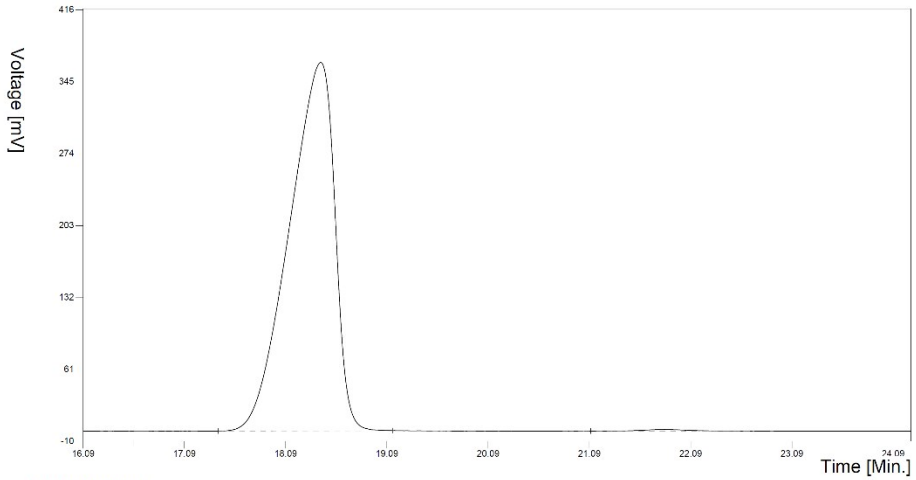


Integration Result

Ret. Time(min)	Area Percentage(%)
21.16	49.9063
25.06	50.0937
	100

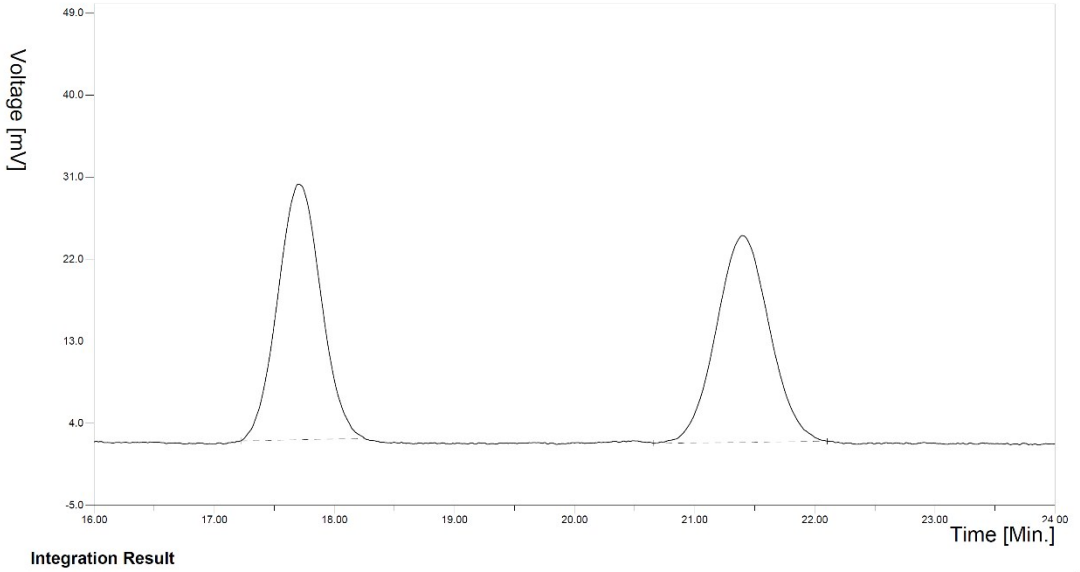
3-allyl-4,5,6-trimethoxyisobenzofuran-1(3*H*)-one (10)

HPLC conditions: Chiralcel OJ column, *n*-Hexane/2-Propanol (90/10), 1 mL/min, (t_{major} =18.43 min, area% =99.5680), (t_{minor} =21.82 min , area% =0.4320), 254 nm UV detector.



Integration Result

Ret. Time(min)	Area Percentage(%)
18.43	99.5680
21.82	0.4320
	100

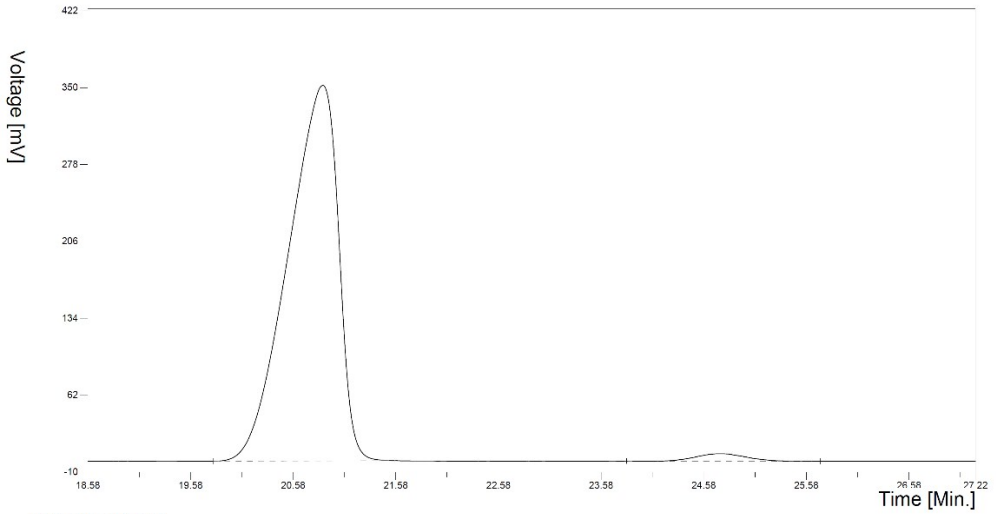


Integration Result

Ret. Time(min)	Area Percentage(%)
17.70	50.2574
21.40	49.7426
	100

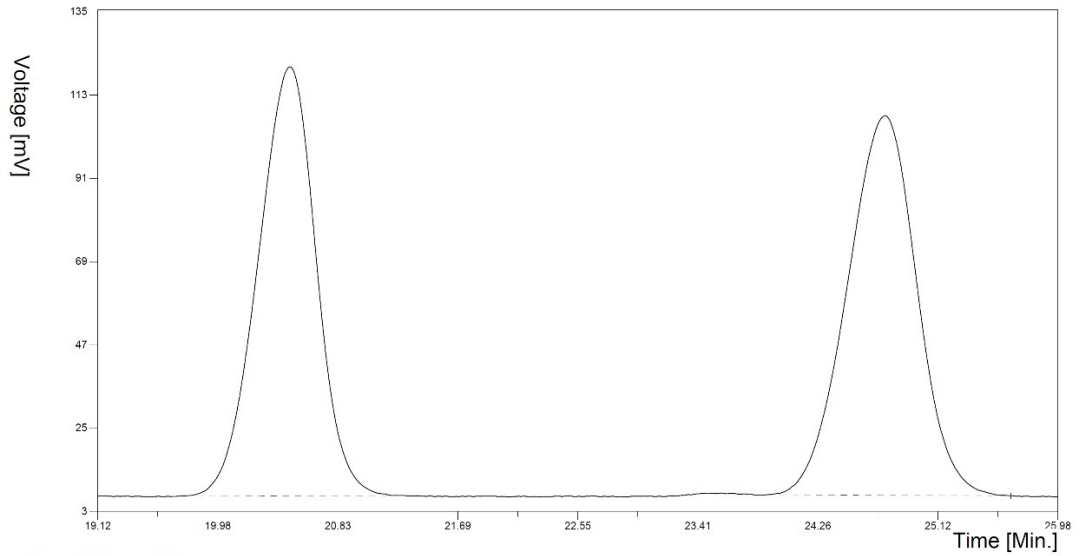
3-allylisobenzofuran-1(3*H*)-one (11)

HPLC conditions: Chiralcel OJ column, *n*-Hexane/2-Propanol (90/10), 1.0 mL/min, (t_{major} = 20.87 min, area% =98.0672), (t_{minor} = 24.72min , area% =1.9328), 254nm UV detector.



Integration Result

Ret. Time(min)	Area Percentage(%)
20.87	98.0672
24.72	1.9328
	100

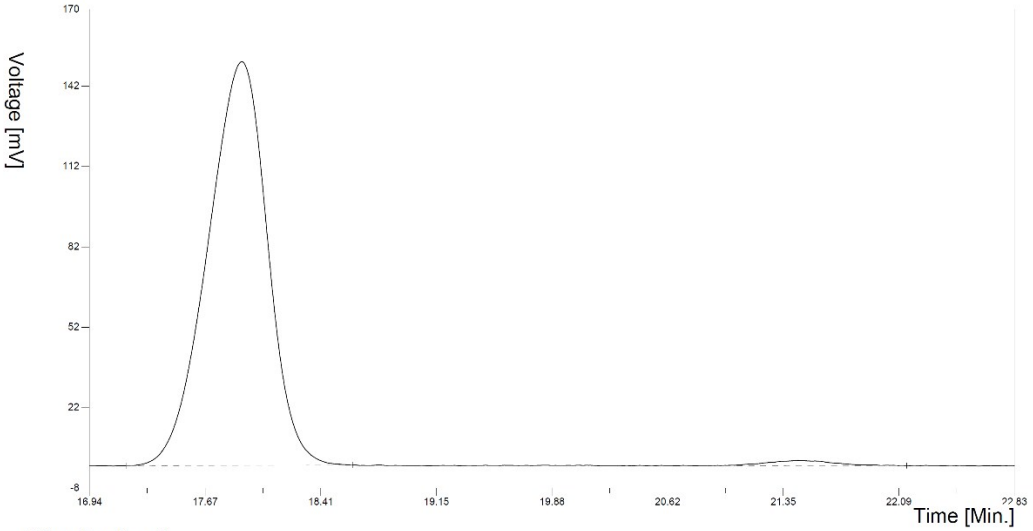


Integration Result

Ret. Time(min)	Area Percentage(%)
20.49	48.6154
24.75	51.3846
	100

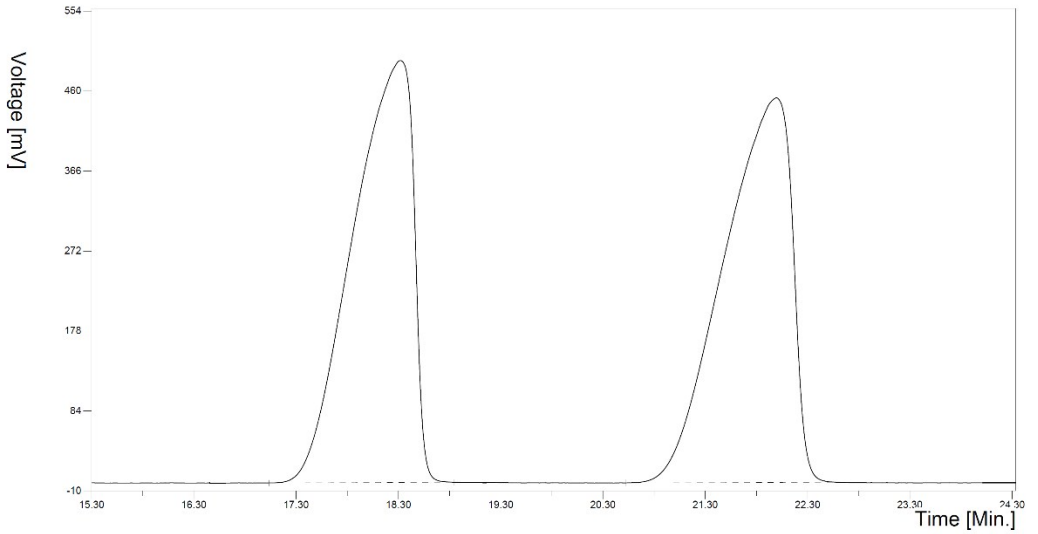
3-allyl-6-methoxyisobenzofuran-1(3*H*)-one (12)

HPLC conditions: Chiralcel OJ column, *n*-Hexane/2-Propanol (90/10), 1.0 mL/min, (t_{major} =17.91 min, area% =98.6356), (t_{minor} =21.45 min, area% =1.3644), 254 nm UV detector.



Integration Result

Ret. Time(min)	Area Percentage(%)
17.91	98.6356
21.45	1.3644
	100

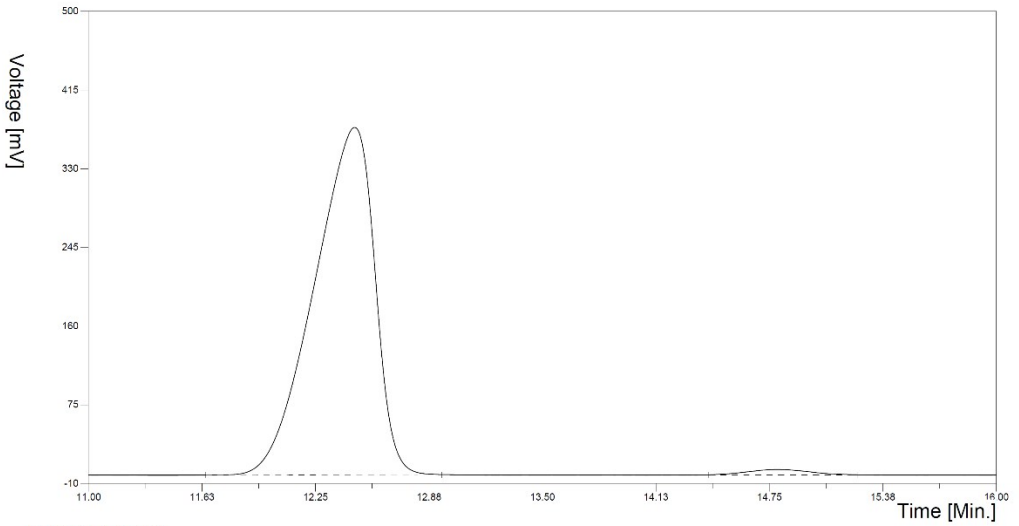


Integration Result

Ret. Time(min)	Area Percentage(%)
18.32	49.2776
22.00	50.7224
	100

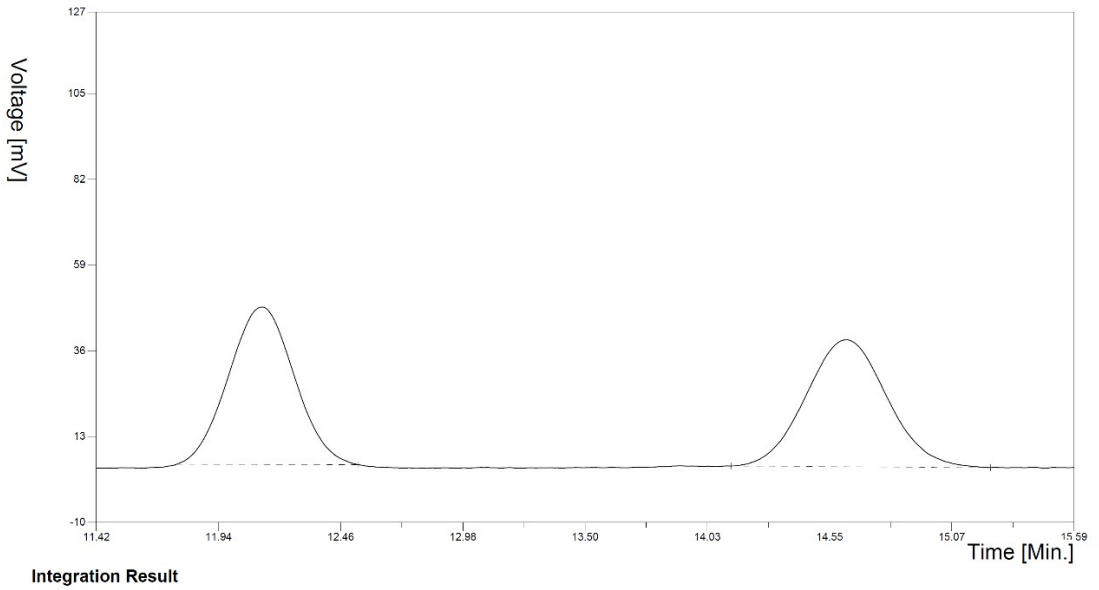
7-allyl-[1,3]dioxolo[4,5-*f*]isobenzofuran-5(7*H*)-one (13)

HPLC conditions: Chiralcel OJ column, *n*-Hexane/2-Propanol (90/10), 1.0 mL/min, (t_{major} =12.47 min, area% =98.5012), (t_{minor} =14.79 min , area% =1.4988), 254nm UV detector.



Integration Result

Ret. Time(min)	Area Percentage(%)
12.47	98.5012
14.79	1.4988
	100

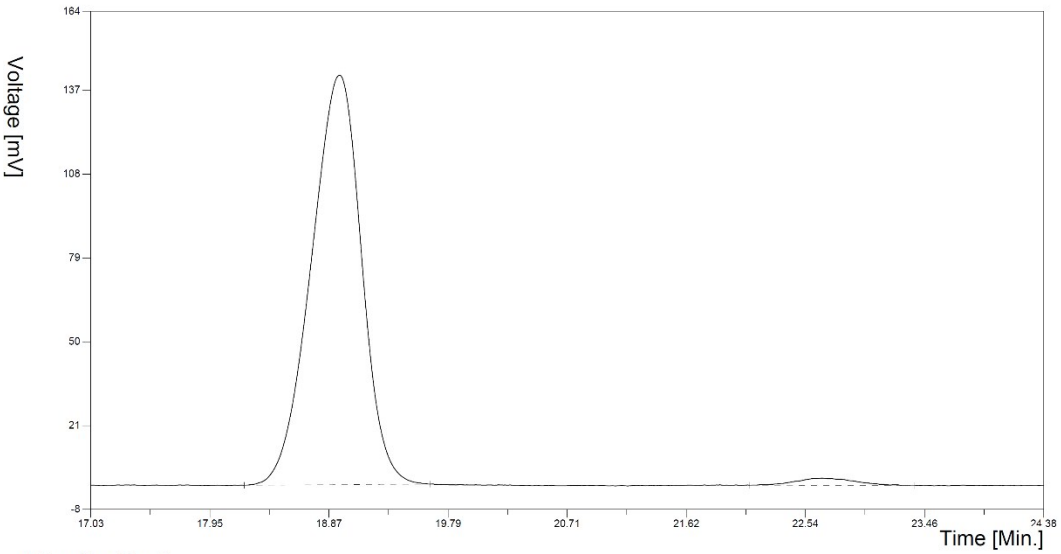


Integration Result

Ret. Time(min)	Area Percentage(%)
12.13	50.1851
14.62	49.8149
	100

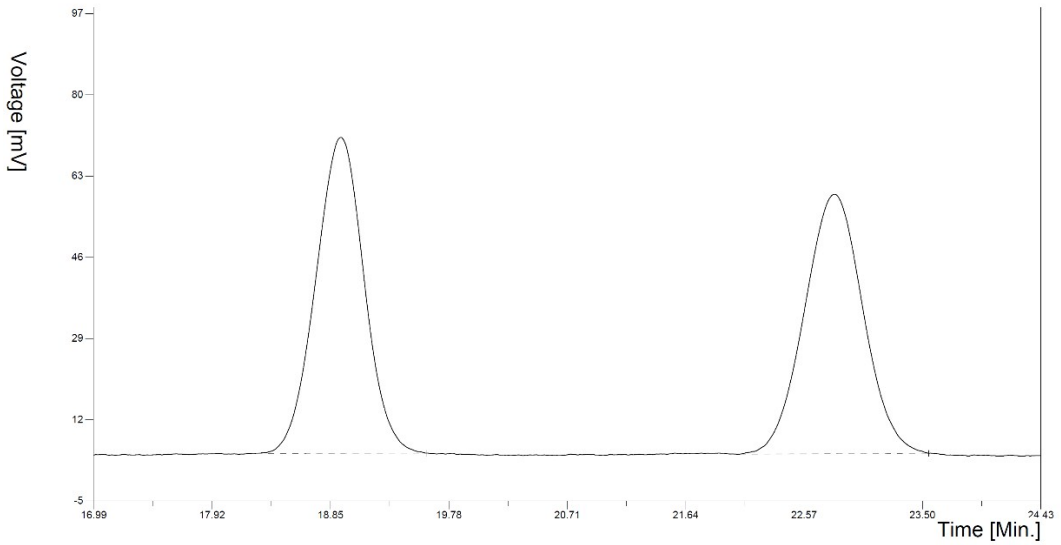
3-allyl-6-(dimethylamino)isobenzofuran-1(3*H*)-one (14)

HPLC conditions: Chiralcel OJ column, *n*-Hexane/2-Propanol (90/10), 1.0 mL/min, (t_{major} =18.95 min, area% =98.0199), (t_{minor} =22.67 min , area% =1.9801), 254nm UV detector.



Integration Result

Ret. Time(min)	Area Percentage(%)
18.95	98.0199
22.67	1.9801
	100

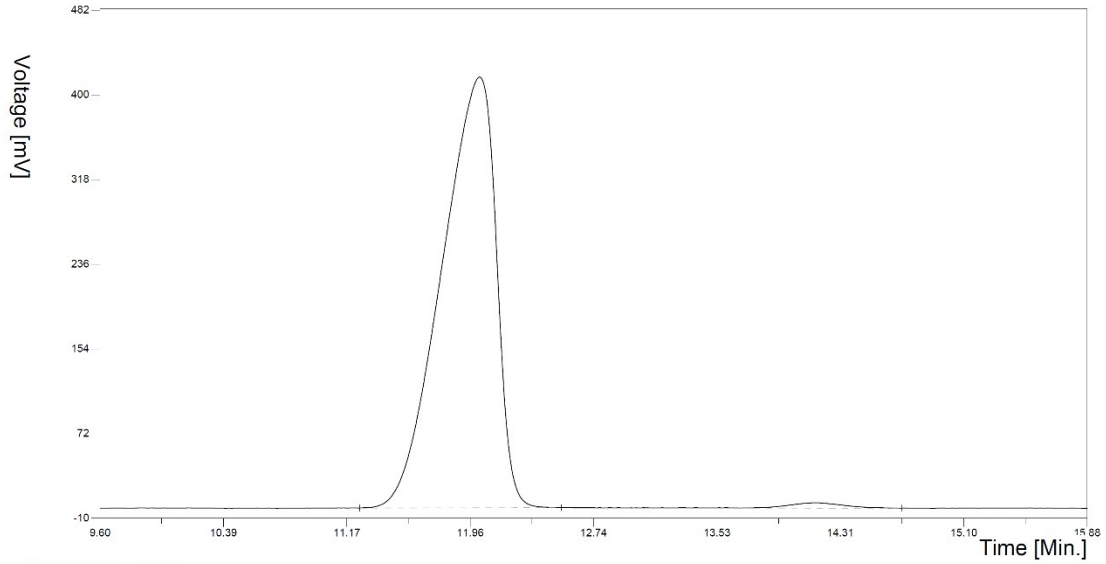


Integration Result

Ret. Time(min)	Area Percentage(%)
18.93	50.1867
22.81	49.8133
	100

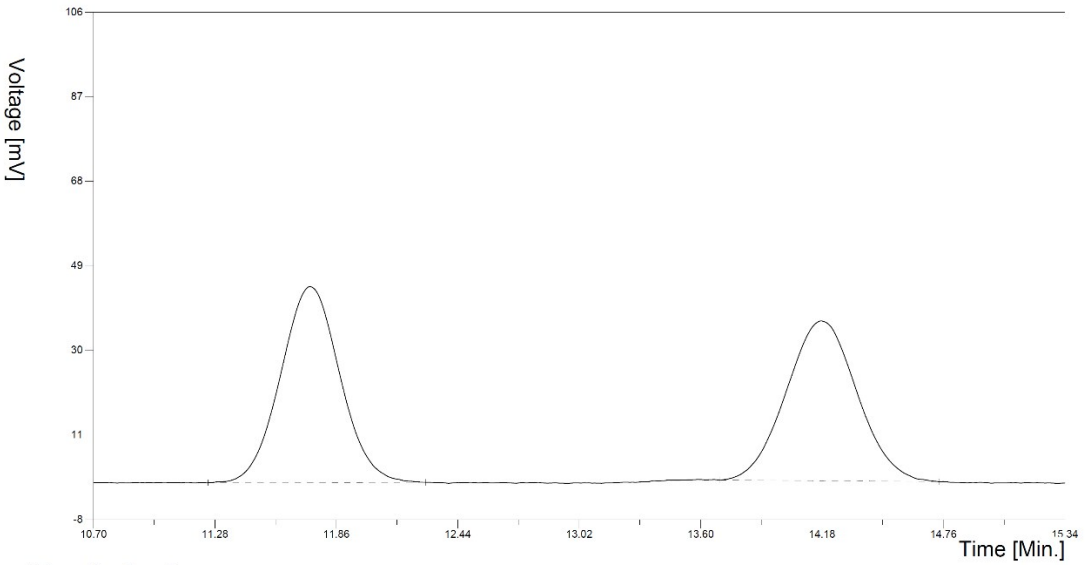
3-allyl-5-bromo-6-(dimethylamino)isobenzofuran-1(3H)-one(15)

HPLC conditions: Chiralcel OJ column, *n*-Hexane/2-Propanol (90/10), 1.0 mL/min, (t_{major} =12.02 min, area% =98.8410), (t_{minor} =14.17min , area% =1.1590), 254nm UV detector.



Integration Result

Ret. Time(min)	Area Percentage(%)
12.02	98.8410
14.17	1.1590
	100

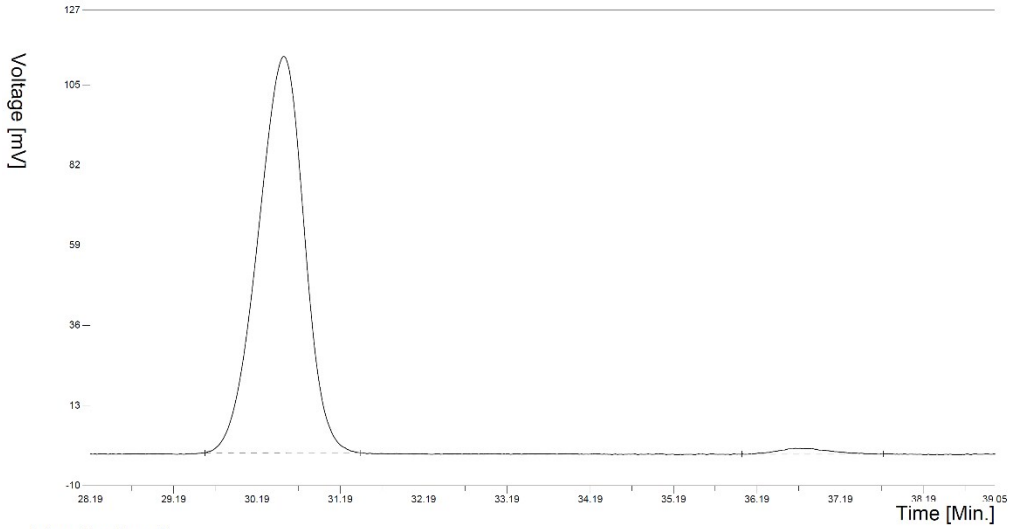


Integration Result

Ret. Time(min)	Area Percentage(%)
11.74	50.7374
14.18	49.2626
	100

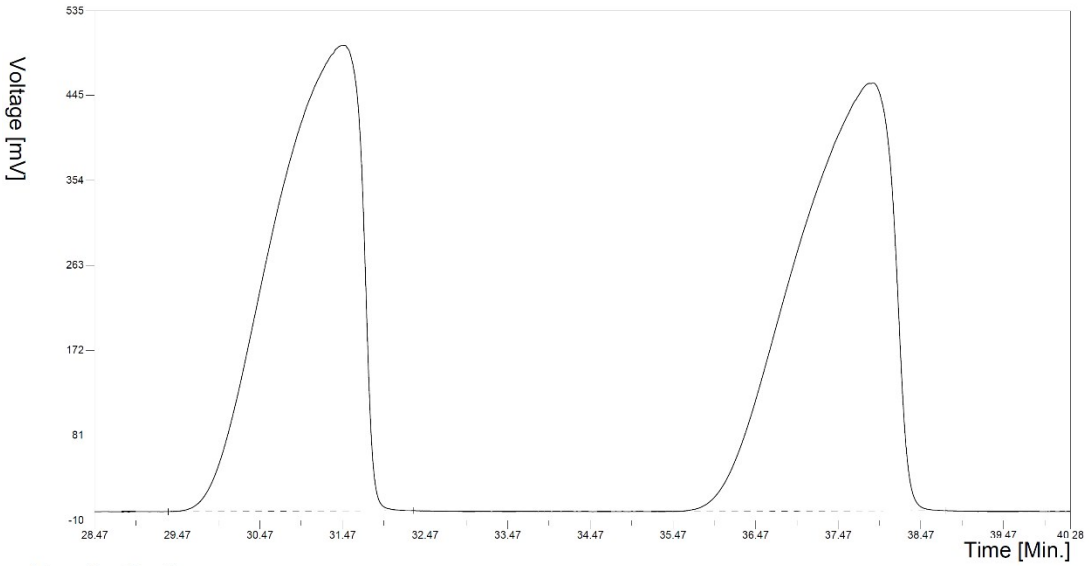
3-allyl-4-bromo-6-(methylamino)isobenzofuran-1(3*H*)-one(16)

HPLC conditions: Chiralcel OJ column, *n*-Hexane/2-Propanol (90/10), 1.0 mL/min, (t_{major} =30.52 min, area% =98.4807), (t_{minor} =36.65 min, area% =1.5193), 254nm UV detector.



Integration Result

Ret. Time(min)	Area Percentage(%)
30.52	98.4807
36.65	1.5193
	100

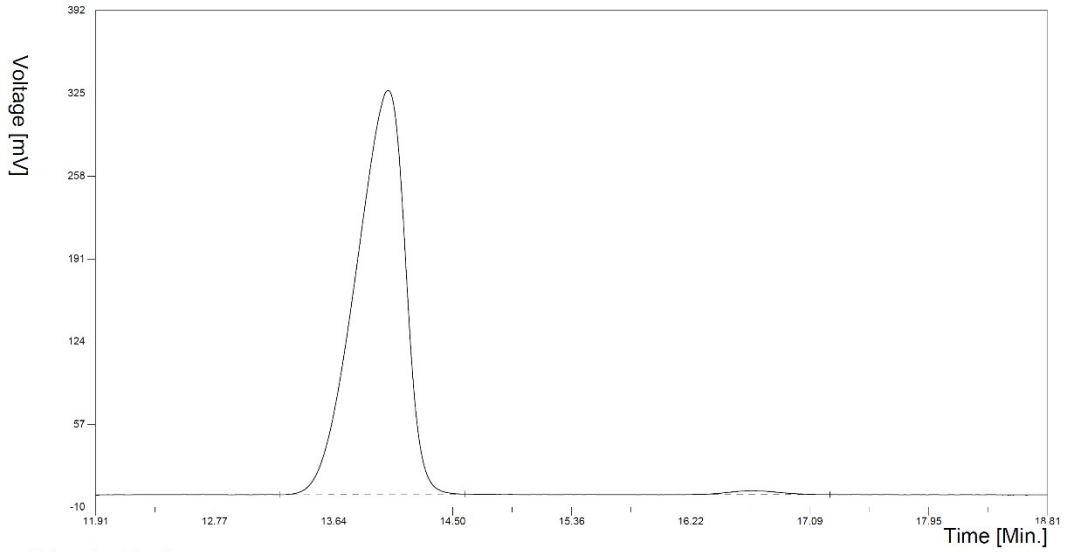


Integration Result

Ret. Time(min)	Area Percentage(%)
31.49	49.5898
37.90	50.4102
	100

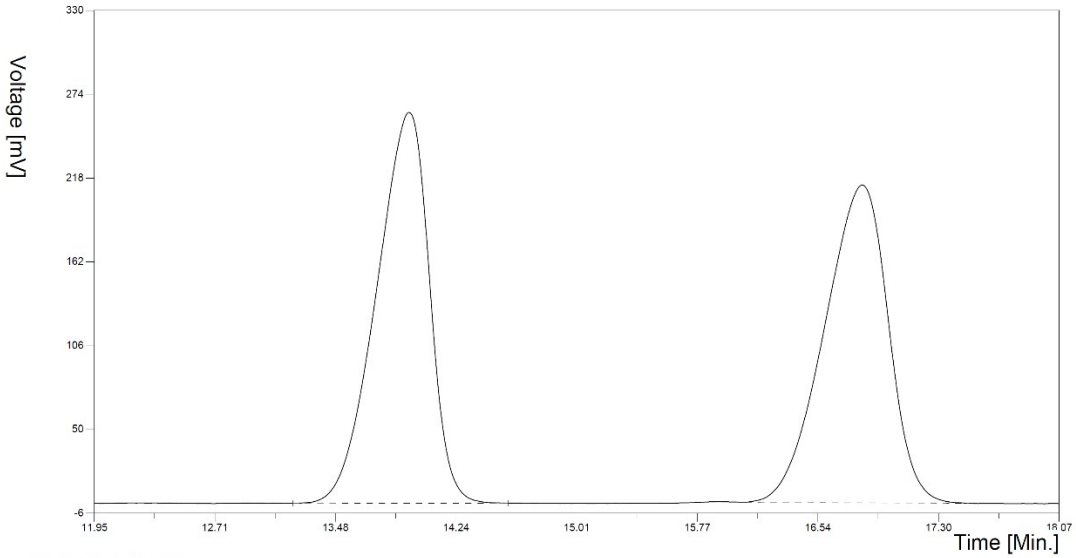
8-allyl-[1,3]dioxolo[4,5-*e*]isobenzofuran-6(8*H*)-one (17)

HPLC conditions: Chiralcel OJ column, *n*-Hexane/2-Propanol (90/10), 1.0 mL/min, (t_{major} =14.03 min, area% =99.0390), (t_{minor} =16.67 min , area% =0.9610), 254nm UV detector.



Integration Result

Ret. Time(min)	Area Percentage(%)
14.03	99.0390
16.67	0.9610
	100

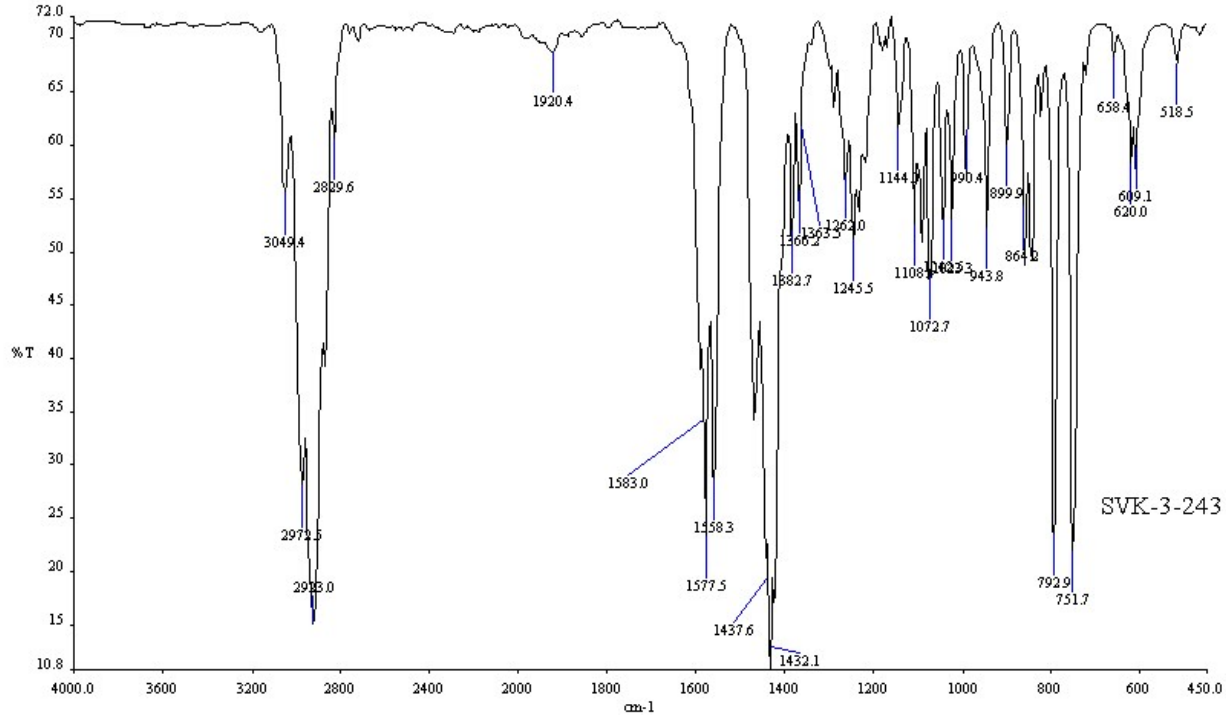


Integration Result

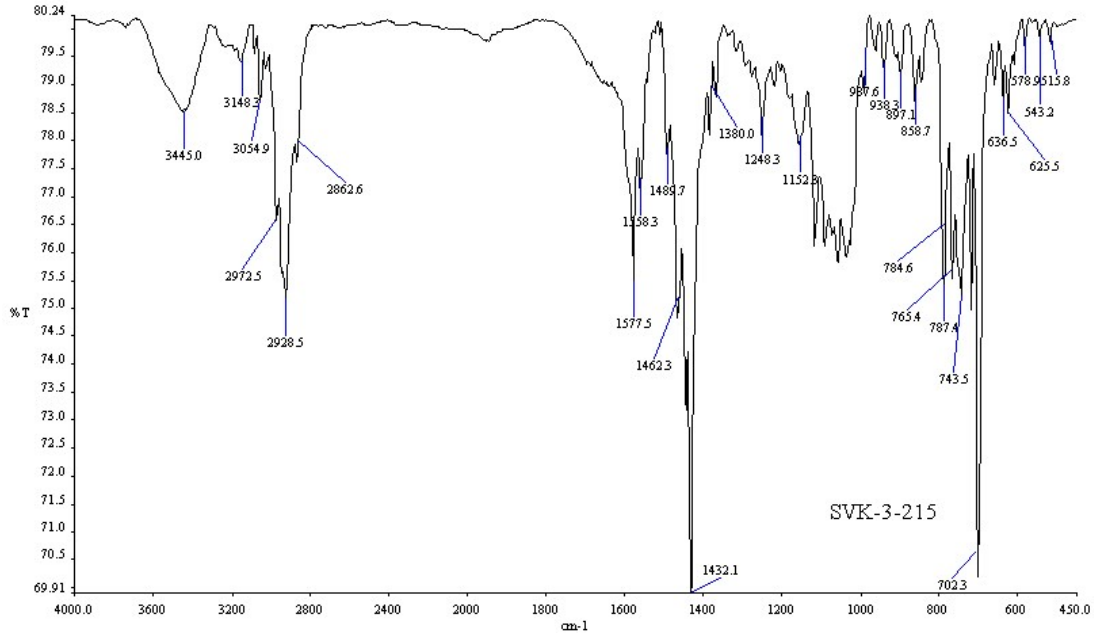
Ret. Time(min)	Area Percentage(%)
13.94	50.4652
16.82	49.5348
	100

IR Spectra

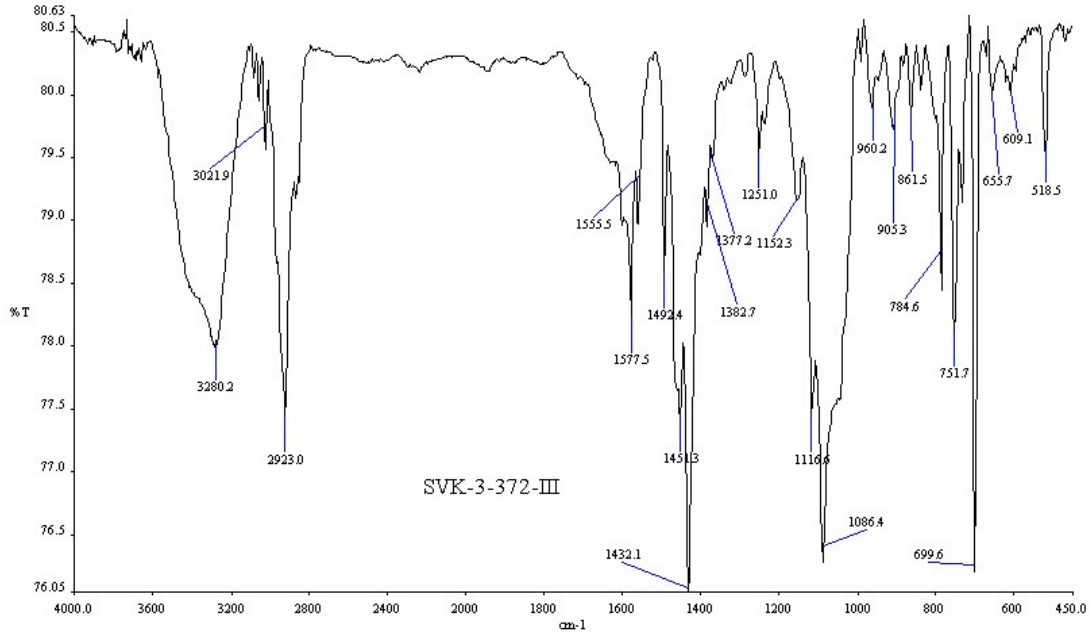
1. (1*S*,9*S*)-10,10-dimethyl-5-pyridin-2-yl-6-aza-tricyclo[7.1.1.0^{2,7}]-undeca-2(7)-3,5-triene (3)



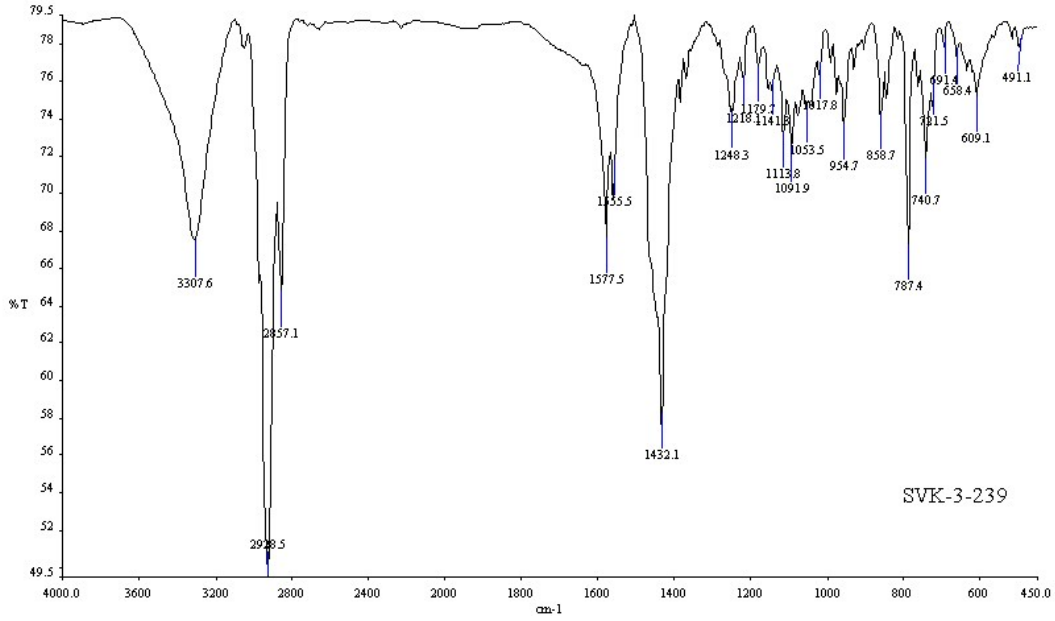
2. (1*S*,8*R*,9*S*)-(10,10-dimethyl-5-pyridin-2-yl-6-aza-tricyclo[7.1.1.0^{2,7}]-undeca-2(7),3,5-trien-8-yl)-diphenyl-methanol (4)



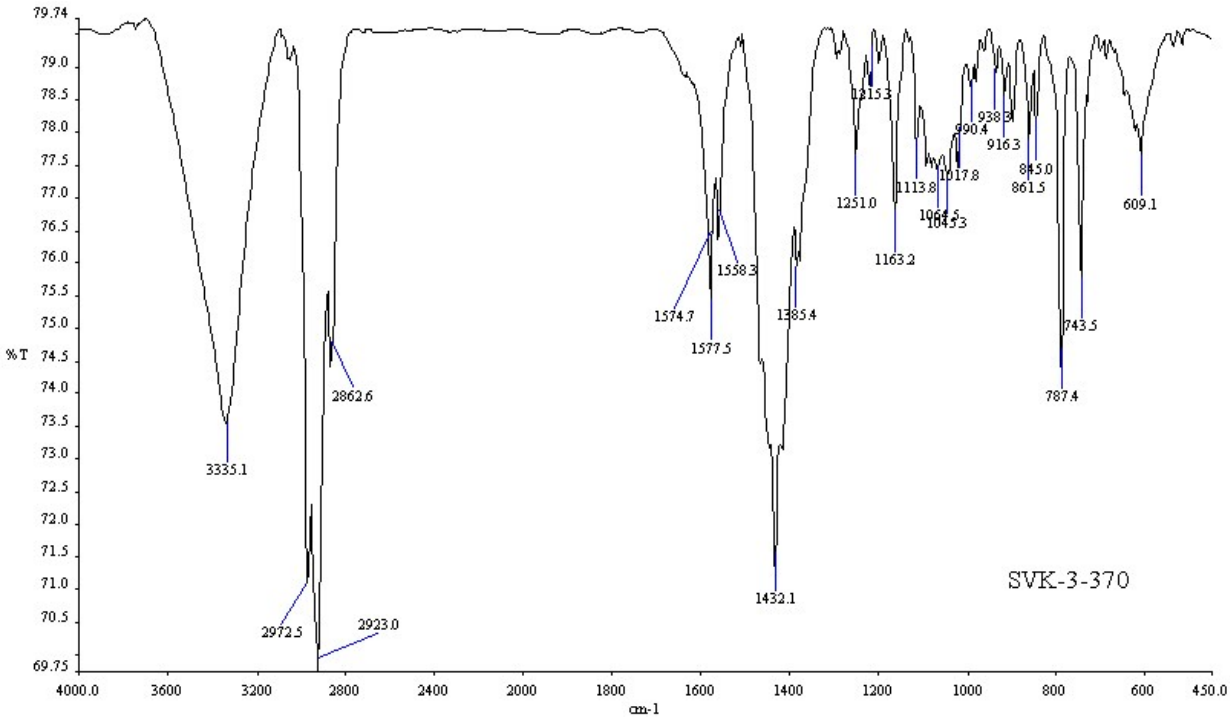
3. (1*S*,8*R*,9*S*)-2-(10,10-dimethyl-5-(pyridin-2-yl)-6-aza-tricyclo[7.1.1.0^{2,7}]-undeca-2(7)-3,5-trien-8-yl)-1,3-diphenyl-propan-2-ol(5)



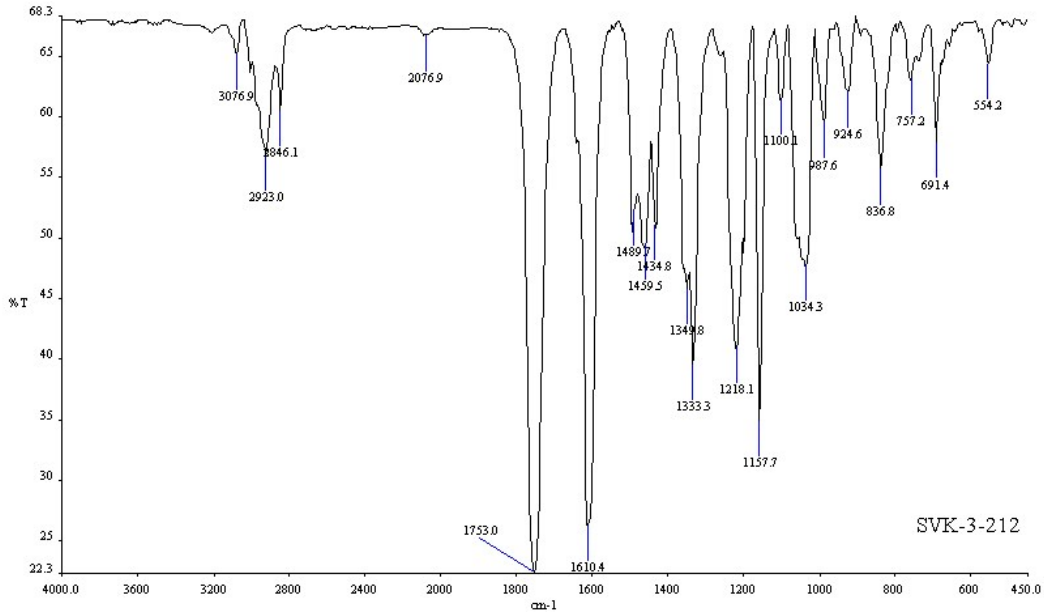
4. (1*S*,8*R*,9*S*)-1-(10,10-dimethyl-5-(pyridin-2-yl)-6-aza-tricyclo[7.1.1.0^{2,7}]-undeca-2(7)-3,5-trien-8-yl)-cyclohexan-1-ol (6)



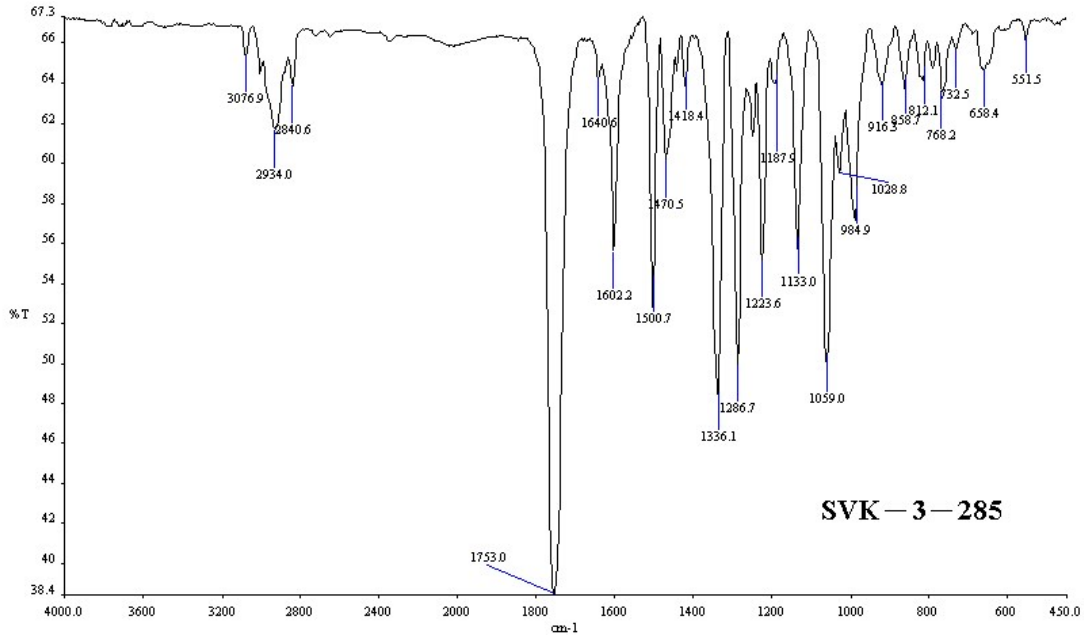
5. (1*S*,8*R*,9*S*)-2-(10,10-dimethyl-5-(pyridin-2-yl)-6-aza-tricyclo[7.1.1.$0^{2,7}$]-undeca-2(7)-3,5-trien-8-yl)-propan-2-ol (7)



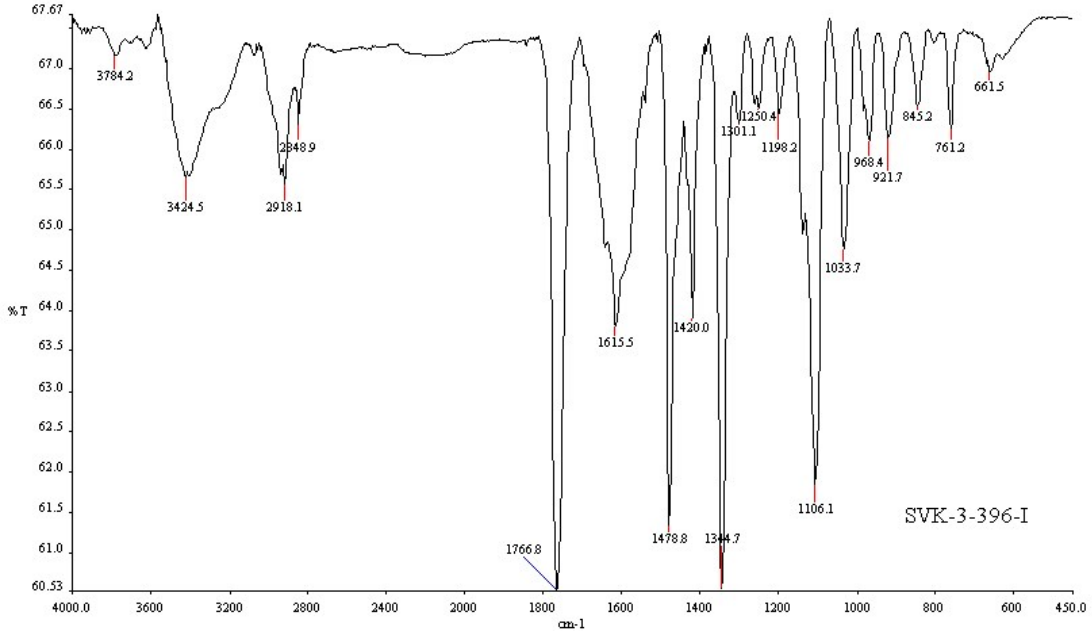
6. 3-allyl-5,7-dimethoxyisobenzofuran-1(3*H*)-one (8)



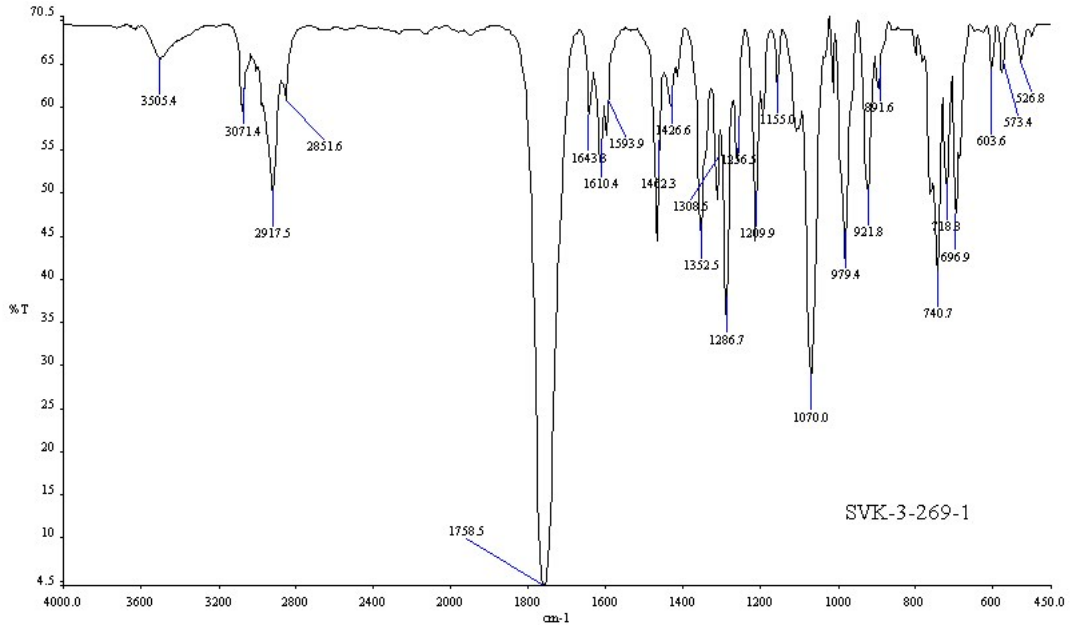
7. 3-allyl-5,6-dimethoxyisobenzofuran-1(3*H*)-one (9)



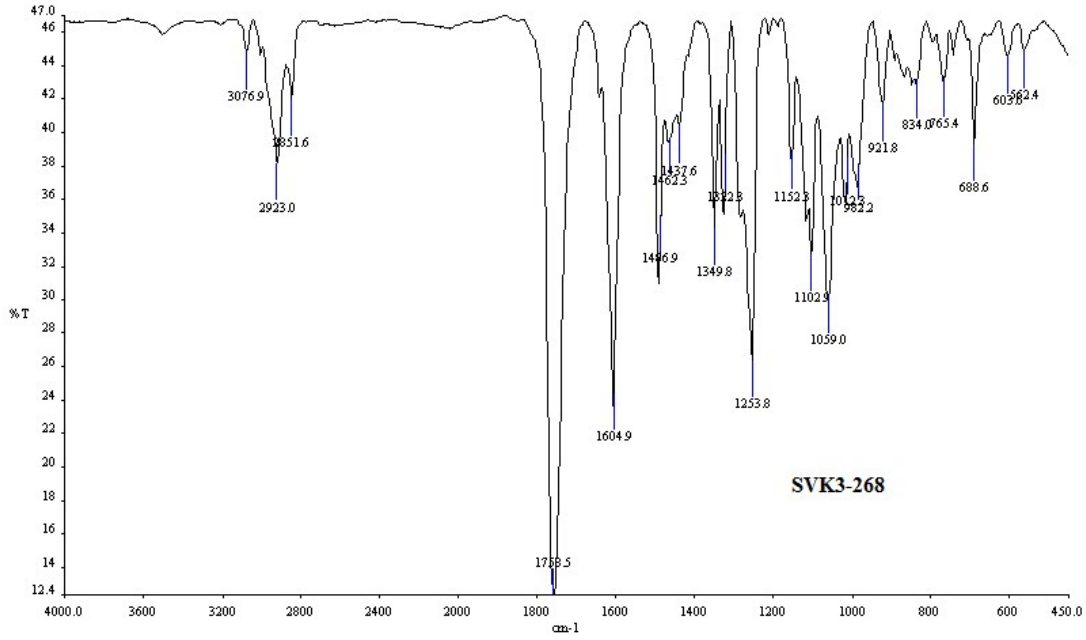
8. 3-allyl-4,5,6-trimethoxyisobenzofuran-1(3*H*)-one (10)



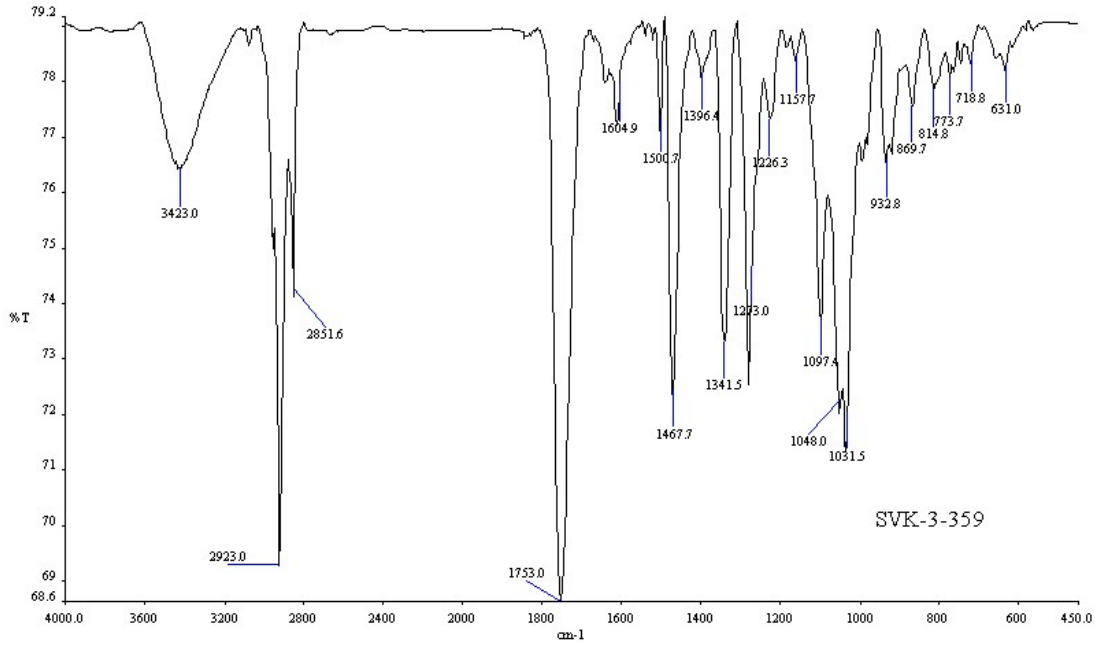
9. 3-allylisobenzofuran-1(3*H*)-one (11)



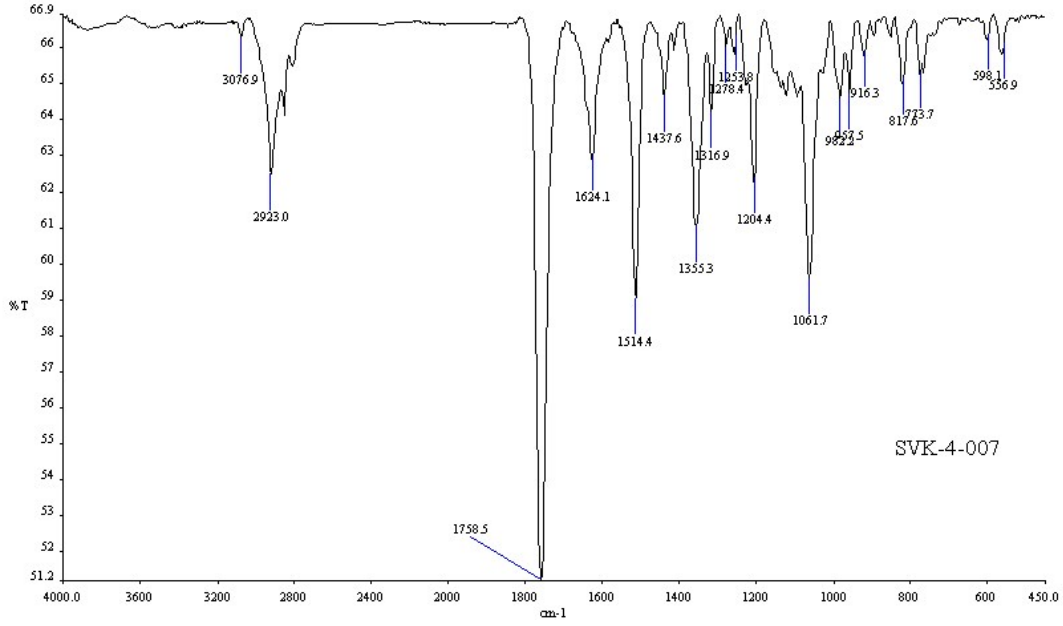
10. 3-allyl-6-methoxyisobenzofuran-1(3*H*)-one (12)



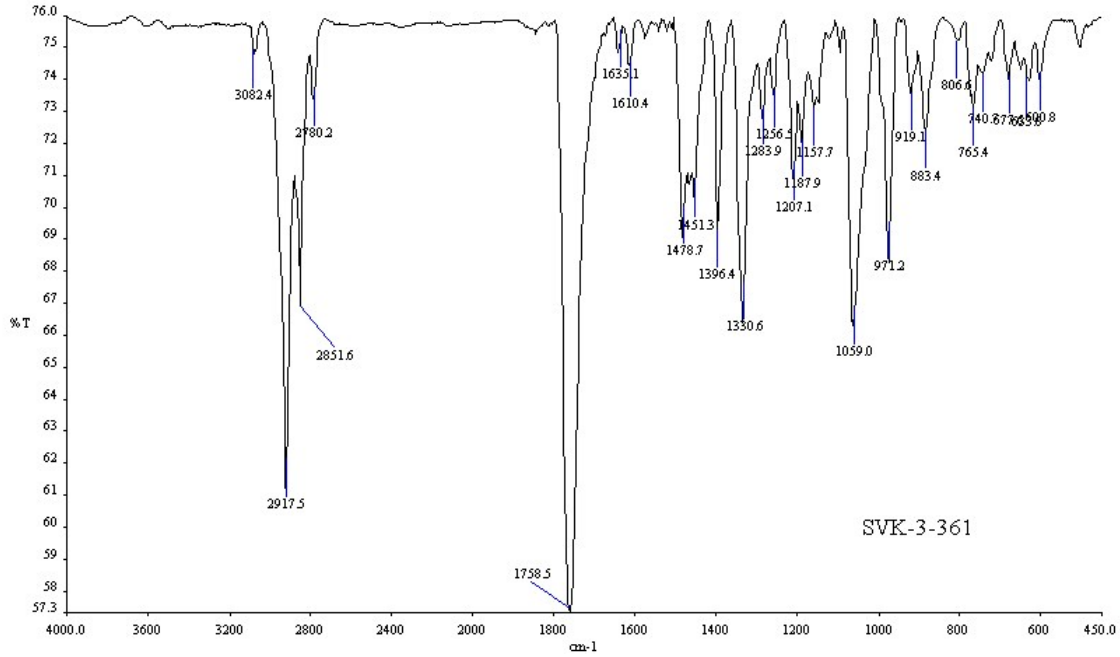
11. 7-allyl-[1,3]dioxolo[4,5-f]isobenzofuran-5(7*H*)-one (13)



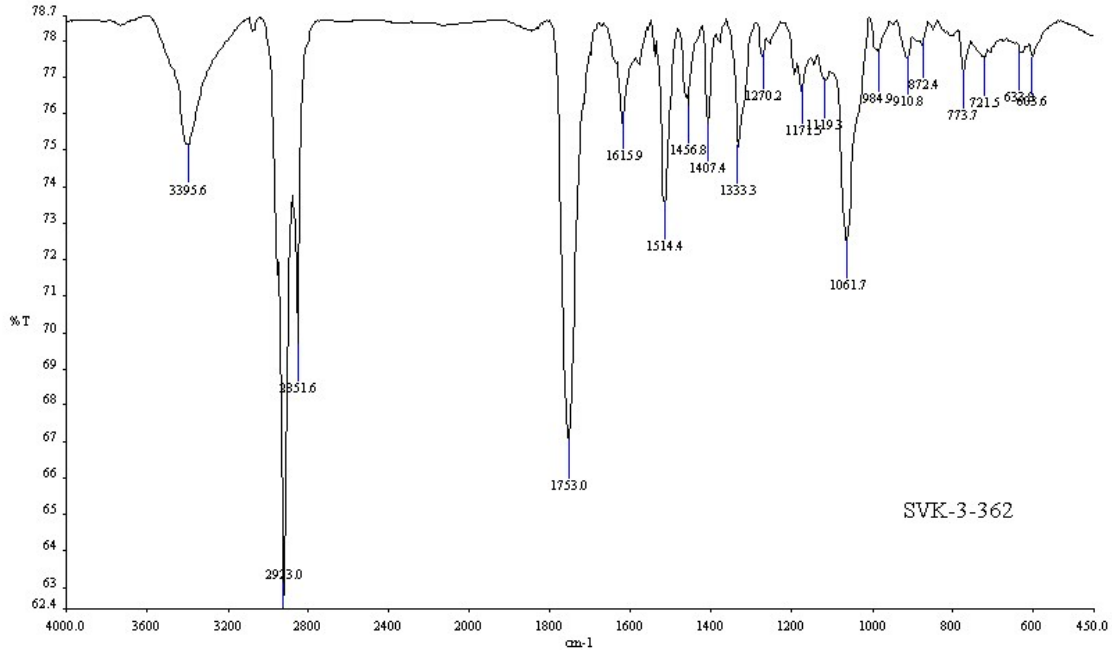
12. 3-allyl-6-(dimethylamino)isobenzofuran-1(3*H*)-one (14)



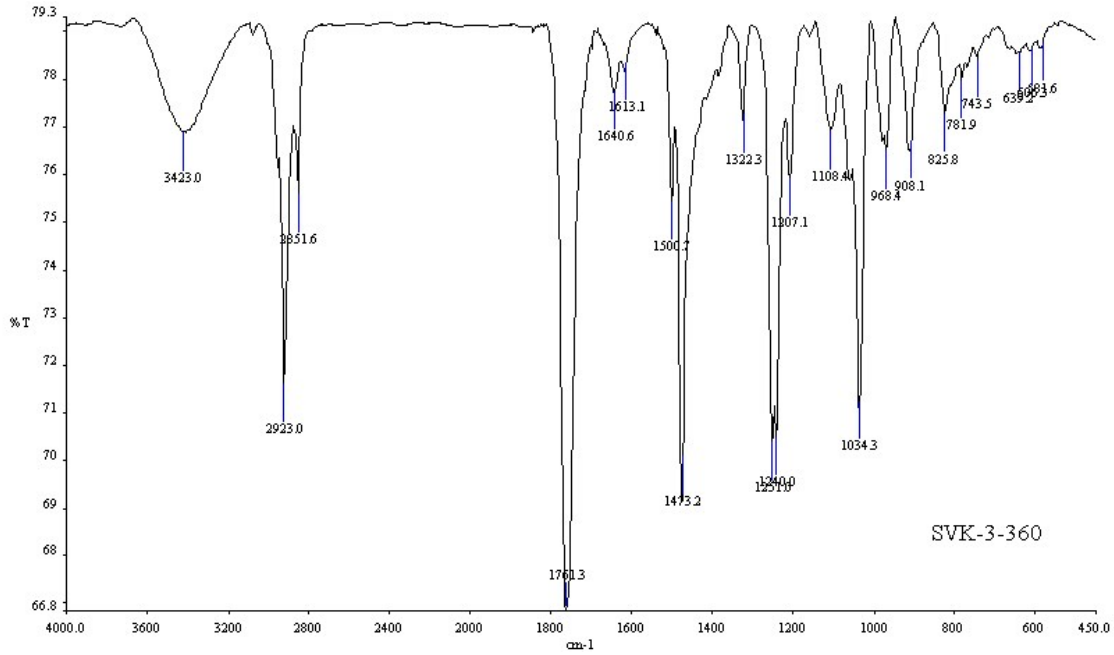
13. 3-allyl-5-bromo-6-(dimethylamino)isobenzofuran-1(3*H*)-one(15)



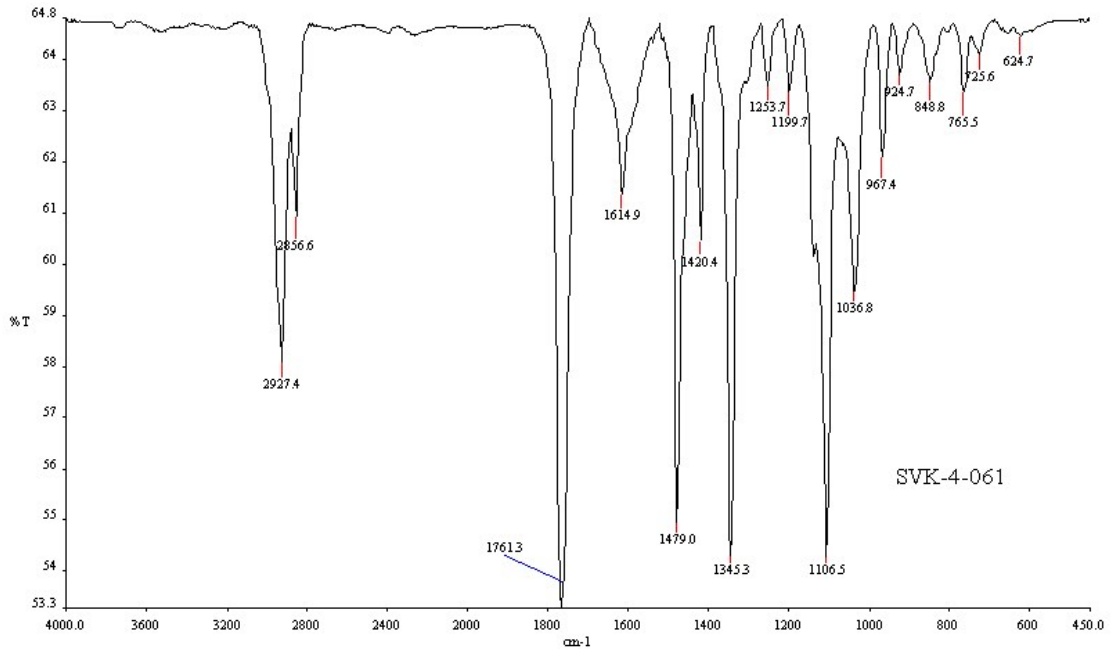
14. 3-allyl-4-bromo-6-(methylamino)isobenzofuran-1(3*H*)-one(16)



15. 8-allyl-[1,3]dioxolo[4,5-*e*]isobenzofuran-6(8*H*)-one (17)



16. 3-heptyl-4,5,6-trimethoxyisobenzofuran-1(3*H*)-one (18)



17. 3-heptyl-4,5,6-trihydroxyisobenzofuran-1(3*H*)-one (19)