Supplementary Information

An efficient CuO/rGO/TiO_2 photocatalyst for the synthesis of benzopyranopyrimidine compounds under visible light irradiation

Maryam Mirza-Aghayan,[1]* Mandana Saeedi,[1] Rabah Boukherroub[2]

[1] Chemistry and Chemical Engineering Research Center of Iran (CCERCI), P. O. BOX 14335-186, Tehran, Iran

[2] Univ. Lille, CNRS, Centrale Lille, Univ. Polytechnique Hauts-de-France, UMR 8520-IEMN, F-59000 Lille, France

Experimental details

Elmasonic P ultrasonic cleaning unit with a frequency of 37 kHz and ultrasonic homogenizer Bandelin Sonoplus HD 3100 with probe model MS 73 at 100% power were used for the synthesis of rGO/TiO_2 and CuO/rGO/TiO_2 nanocomposites. Scanning electron microscopy (SEM) and energy dispersive X-ray (EDS) data were recorded using a VEGA3 LMU TESCAN SEM and MIRA III TESCAN FESEM. X-ray diffraction (XRD) patterns were acquired using a PW1730 (PHILIPS Company). FT-IR spectra were obtained using KBr disks with a Bruker Vector 22 FT-IR spectrometer. Copper content in CuO/rGO/TiO_2 catalyst was determined using ICP-OES Varian 735 ES configuration torch redial. Melting points were determined in evacuated capillaries with a Buchi B-545 apparatus. ^{1}H and ^{13}C NMR spectra were performed on Bruker 300, 125 and 75 MHz spectrometers using tetramethylsilane as internal standard. X-ray photoelectron spectroscopy (XPS) measurements were carried out using a Bes Tec (Germany) instrument at a pressure of 10×10^{-10} mbar. High resolution transmission electron microscopy (HRTEM) analysis was acquired using FEI Tecnai G2 F20 SuperTwin TEM (accelerating voltage: 200 kV). Cyclic voltammetry (CV) analysis was performed by Ivium electrochemical

* Corresponding author: m.mirzaaghayan@ccerci.ac.ir
Tel.: +98 21 44787720; Fax: +98 2144787762

workstation. Photoluminescence (PL) measurements were recorded using a Varian Cary Eclipse Spectrometer (light source: Xenon Flash Lamp, Wavelength range: 200-900 nm).

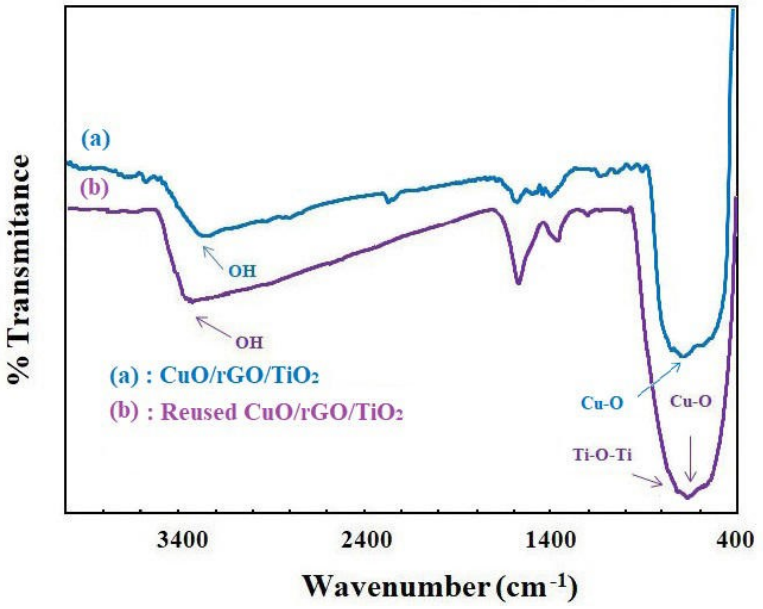


Fig. S1. The FT-IR spectra of fresh and reused $CuO/rGO/TiO_2$ catalyst.

2-(4-Morpholino-5*H*-benzopyrano[2,3-*d*]pyrimidin-2-yl)phenol (1a): White powder; Mp = 192-194°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm) = 3.49 (t, 4H, $^3J_{HH}$ = 4.3 Hz, $2CH_2$), 3.78 (t, 4H, $^3J_{HH}$ = 4.7 Hz, $2CH_2$), 3.99 (s, 2H, CH_2), 6.88-6.93 (m, 2H, CH_{Ar}), 7.14-7.35 (m, 5H, CH_{Ar}), 8.24 (dd, 1H, $^3J_{HH}$ = 9 and 1.5Hz, CH_{Ar}), 13.06 (s, 1H, OH).

4-Bromo-2-(7-bromo-4-morpholino-5*H*-benzopyrano[2,3-d]pyrimidin-2-yl)phenol (1b): Yellow solid; Mp = 194-196°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm) = 3.48 (t, 4H, $^3J_{HH}$ = 4.1 Hz, $2CH_2$), 3.78 (t, 4H, $^3J_{HH}$ = 4.6 Hz, $2CH_2$), 3.99 (s, 2H, CH_2), 6.87 (d, 1H, $^3J_{HH}$ = 8.7 Hz, CH_{Ar}), 7.14 (d, 1H, $^3J_{HH}$ = 8.7 Hz, CH_{Ar}), 7.41-7.57 (m, 3H, CH_{Ar}), 8.26 (d, 1H, $^3J_{HH}$ = 2.5 Hz, CH_{Ar}), 13.09 (s, 1H, OH).

2-(4-(Piperidin-1-yl)-5*H*-benzopyrano[2,3-d]pyrimidin-2-yl)phenol (1c): Yellow solid; Mp = 168-170°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm) = 1.68 (s, 6H, 3CH_2), 3.47 (s, 4H, N(CH_2)$_2$), 3.98 (s, 2H, CH_2), 6.91 (t, 2H, $^3J_{HH}$ = 9 Hz, CH_{Ar}), 7.14-7.39 (m, 5H, CH_{Ar}), 8.25 (dd, 1H, $^3J_{HH}$ = 8.3 and 1.3 Hz, CH_{Ar}), 13.28 (s, 1H, OH).

4-Bromo-2-(7-bromo-4-(piperidin-1-yl)-5*H*- benzopyrano[2,3-d]pyrimidin-2-yl)phenol (1d): Yellow solid; Mp = 222-224°C; ^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm) = 1.70 (m, 6H, 3CH_2), 3.48-3.50 (m, 4H, N(CH_2)$_2$), 4.05 (s, 2H, CH_2), 6.93 (d, 1H, $^3J_{HH}$ = 5.25 Hz, CH_{Ar}), 7.2 (d, 1H, $^3J_{HH}$ = 5.22 Hz, CH_{Ar}), 7.47 (dd, 1H, $^3J_{HH}$ = 5.4 and 1.5 Hz, CH_{Ar}), 7.54 (dd, 1H, $^3J_{HH}$ = 3 and 1.5 Hz, CH_{Ar}), 7.65 (d, 1H, $^3J_{HH}$ = 1.4 Hz, CH_{Ar}), 8.34 (d, 1H, $^3J_{HH}$ = 1.4 Hz, CH_{Ar}), 13.36 (s, 1H, OH).

2-(4-(4-Methylpiperidin-1-yl)-5*H*-benzopyrano[2,3-d]pyrimidin-2-yl)phenol (1e): Yellow solid; Mp = 158-159°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm) = 0.97 (d, 3H, $^3J_{HH}$ = 6.2 Hz, CH_3), 1.25-1.37 (m, 2H, CH_2), 1.66-1.78 (m, 3H, CH and CH_2), 3.01 (t, 2H, $^3J_{HH}$ = 12.6 Hz, N(CH_2)), 3.93-3.98 (broad, m, 4H, CH_2, N(CH_2)), 6.89-6.94 (m, 2H, CH_{Ar}), 7.12-7.39 (m, 5H, CH_{Ar}), 8.25 (dd, 1H, $^3J_{HH}$ = 8.12 Hz, 1.3 Hz, CH_{Ar}), 13.26 (d, 1H, $^4J_{HH}$ = 3.4 Hz, OH).

4-Bromo-2-(7-bromo-4-(4-methylpiperidin-1-yl)-5*H*-benzopyrano[2,3-d]pyrimidin-2-yl)phenol (1f): Yellow solid; Mp = 233-235°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm) = 0.96 (d, 3H, $^3J_{HH}$= 6.2 Hz, CH_3), 1.25-1.36 (m, 2H, CH_2), 1.66-1.78 (broad, m, 3H, CH and CH_2), 3.02 (t, 2H, $^3J_{HH}$ = 11.9 Hz, N(CH_2)), 3.90-3.99 (broad , m, 4H, CH_2, N(CH_2)), 6.88 (d, 1H, $^3J_{HH}$

= 7.8 Hz, CH_{Ar}), 7.15 (d, 1H, $^{3}J_{HH}$ = 8.6 Hz, CH_{Ar}), 7.42-7.52 (m, 2H, CH_{Ar}), 7.60 (s, 1H, CH_{Ar}), 8.28 (d, 1H, $^{3}J_{HH}$ = 2.1 Hz, CH_{Ar}), 13.32 (s, 1H, OH).

2-(4-(Dimethylamino)-5*H*-benzopyrano[2,3-d]pyrimidin-2-yl)phenol (1g): Yellow solid; Mp = 178-180°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm) = 3.31 (s, 6H, $2CH_3$), 4.18 (s, 2H, CH_2), 6.88-7.35 (m, 7H, CH_{Ar}), 8.24-8.27 (m, 1H, CH_{Ar}), 13.37 (s, 1H, OH).

4-Bromo-2-(7-bromo-4-(dimethylamino)-5*H*-benzopyrano[2,3-d]pyrimidin-2-yl)phenol (1h): Yellow solid; Mp = 195-197°C; ^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm) = 3.19 (s, 3H, CH_3), 3.28 (s, 3H, CH_3), 4.17 (s, 2H, CH_2), 6.86 (d, 1H, $^{3}J_{HH}$ =8.8, CH_{Ar}), 7.10 (d, 1H, $^{3}J_{HH}$ = 8.8, CH_{Ar}), 7.41 (d, 1H, $^{3}J_{HH}$ = 8.65, CH_{Ar}), 7.47 (d, 1H, $^{3}J_{HH}$ = 8.8, CH_{Ar}), 7.51 (s, 1H, CH_{Ar}), 8.27 (s, 1H, CH_{Ar}), 13.36 (s, H, OH).

2-(4-(Butyl(methyl)amino)-5*H*-benzopyrano [2,3-d]pyrimidin-2-yl)phenol (1i): Yellow solid; Mp = 150-152°C; IR (KBr): 3432, 2959, 2929, 1624, 1602, 1580, 1489, 1439, 1389, 1256, 1068, 761 cm^{-1}; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm) = 0.92 (t, 3H, $^{3}J_{HH}$ = 7.3 Hz, CH_3), 1.28-1.41 (m, 2H, CH_2), 1.60-1.71 (m, 2H, CH_2), 3.22 (s, 3H, CH_3), 3.55 (t, 2H, $^{3}J_{HH}$ = 7.6 Hz, CH_2), 4.15 (s, 2H, CH_2), 6.88-6.93 (m, 2H, CH_{Ar}), 7.10-7.38 (m, 5H, CH_{Ar}), 8.23 (dd, 1H, $^{3}J_{HH}$ = 7.6 and 1.6 Hz, CH_{Ar}), 13.31 (s, 1H, OH); ^{13}C NMR (75 MHz, $CDCl_3$) (ppm) = 13.9, 20.0, 26.0, 29.9, 39.0, 52.5, 94.3, 116.9, 117.4, 118.5, 118.7, 119.2, 124.2, 128.1, 128.5, 129.1, 132.6, 150.3, 160.4, 161.4, 163.8; MS (EI) (70 ev), m/z (%): 363 (20) $[M+2]^+$, 361 (77) $[M]^+$, 346 (24), 332 (8), 318 (32), 304 (100), 290 (14), 275 (8), 171 (9), 151 (20), 128 (9), 102 (8).

4-Bromo-2-(7-bromo-4-(butyl(methyl)amino)-5*H*-benzopyrano[2,3-d]pyrimidin-2-yl)phenol (1j): Yellow solid; Mp = 226-228°C; IR (KBr): 3431, 2954, 2857, 1622, 1597, 1539, 1487, 1369, 1260, 1186, 1045, 817, 682 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6) δ (ppm) = 0.94 (t, 3H, $^3J_{HH}$ = 7.4 Hz, CH_3), 1.32-1.45 (m, 2H, CH_2), 1.60-1.71 (m, 2H, CH_2), 3.23 (s, 3H, CH_3), 3.54 (t, 2H, $^3J_{HH}$ = 7.7 Hz, CH_2), 4.18 (s, 2H, CH_2), 6.92 (d, 1H, $^3J_{HH}$ = 8.7 Hz, CH_{Ar}), 7.14 (d, 1H, $^3J_{HH}$ = 8.7 Hz, CH_{Ar}), 7.42-7.61 (m, 3H, CH_{Ar}), 8.30 (d, 1H, $^3J_{HH}$ = 8.7 Hz, CH_{Ar}), 13.33 (s, 1H, OH); ^{13}C NMR (75 MHz, $CDCl_3$) (ppm) = 13.9, 20.0, 25.8, 29.8, 39.1, 52.7, 93.7, 110.8, 116.7, 119.5, 119.9, 121.2, 131.3, 135.4, 136.9, 146.8, 149.2, 159.4, 163.6, 165.3; MS (EI) (70 ev), m/z (%): 519 (100) $[M+2]^+$, 517 (48.8) $[M]^+$, 504 (17.6) $[M+2\text{-}CH_3]^+$, 476 (28) $[M+2\text{-}CH_3\text{-}(CH_2)_2]^+$, 462 (91) $[M+2\text{-}CH_3\text{-}(CH_2)_3]^+$, 281 (30), 207 (67), 191 (30), 127 (22).

2-(4-(Benzyl(methyl)amino)-5*H*-benzopyrano[2,3-d]pyrimidin-2-yl)phenol (1k): Yellow solid; Mp = 168-170°C; IR (KBr): 3430, 3061, 2899, 1627, 1605, 1583, 1493, 1488, 1262, 1059, 947, 754, 692 cm^{-1}; ^1H NMR (300 MHz, DMS-d_6) δ (ppm) = 3.20 (s, 3H, CH_3), 4.19 (s, 2H, CH_2), 4.86 (s, 2H, CH_2), 6.85-6.88 (m, 2H, CH_{Ar}), 7.08-7.39 (m, 10H, CH_{Ar}), 8.19 (dd, 1H, $^3J_{HH}$ = 8.1 Hz and 1.5 Hz, CH_{Ar}), 13.12 (s, 1H, OH); ^{13}C NMR (125 MHz, DMSO-d6) δ (ppm) = 25.7, 39.1, 56.1, 94.5, 116.8, 117.6, 118.1, 119, 119.2, 124, 125.9,126.7, 127.1, 127.7, 128.3, 128.6, 128.9, 129.2, 133.0, 137.0, 150.0, 160.3, 164.2; MS (EI) (70 ev), m/z (%): 397 (8) $[M+2]^+$, 395 (25) $[M]^+$, 304 (100), 207 (8), 185 (9), 171 (5), 120 (5), 91 (16).

2-(4-(Benzyl(methyl)amino)-7-bromo-5*H*-benzopyrano[2,3-d]pyrimidin-2-yl)-4-bromophenol (1l): Yellow solid; Mp = 198-200°C; IR (KBr): 3433, 2896, 1615, 1561, 1545, 1434, 1180, 1056, 817, 802, 696 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6) δ (ppm) = 3.26 (s, 3H, CH_3), 4.24 (s, 2H, CH_2), 4.85 (s, 2H, CH_2), 6.85 (d, 1H, $^3J_{HH}$ = 9 Hz, CH_{Ar}), 7.14 (d, 1H, $^3J_{HH}$ =

9 Hz, CH_{Ar}), 7.25-7.49 (m, 8H, CH_{Ar}), 8.21 (d, 1H, $^3J_{HH}$ = 9 Hz, CH_{Ar}), 13.10 (s, 1H, OH); ^{13}C NMR (75 MHz, $CDCl_3$) (ppm) = 25.7, 38.9, 56.0, 94.5, 116.8, 117.5, 118.4, 118.7, 119.2, 124.3, 127, 128.1, 128.5, 128.9, 129.2, 132.8, 137.2, 150.1, 160.4, 161.3, 163.7, 164.2.

2-Imino-2*H*-benzopyran-3-carbonitrile (2a) Yellow solid; Mp = 160-162°C; IR (KBr): 3340, 3294, 2226, 2196, 1647, 1600, 1453, 1417, 1220, 1191, 1060, 760 cm^{-1}. ^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm) = 7.20 (d, 1H, $^3J_{HH}$ = 8.8 Hz, CH_{Ar}), 7.27 (t, 2H, $^3J_{HH}$ = 7.4 Hz, CH_{Ar}), 7.60 (d, 1H, $^3J_{HH}$ = 5.6 Hz, CH_{Ar}), 8.38 (s, 1H, CH_{Ar}), 8.85 (s, 1H, NH).

4-Morpholino-2-(naphthalen-2-yl)-5*H*-benzopyrano[2,3-d]pyrimidine (3a): Yellow solid; Mp = 182-183°C; IR (KBr): 2930, 2852, 1598, 1572, 1536, 1418, 1383, 1235, 1109, 929, 790, 755 cm^{-1}; 1H NMR (500 MHz, DMSO-d_6) δ (ppm) = 3.53 (t, 4H, $3J_{HH}$ = 5 Hz, $2CH_2$), 3.80 (t, 4H, $3J_{HH}$ = 4.9 Hz, $2CH_2$), 4.10 (s, 2H, CH_2), 7.16-7.20 (m, 2H, CH_{Ar}), 7.30 (t, 1H, $3J_{HH}$ = 8.2 Hz, CH_{Ar}) 7.40 (d, 1H, $3J_{HH}$ = 7.5 Hz, CH_{Ar}), 7.59-7.67 (m, 3H, CH_{Ar}), 8.02 (d, 1H, $3J_{HH}$ = 7.5 Hz, CH_{Ar}), 8.06 (d, 2H, $3J_{HH}$ = 7.5 Hz, CH_{Ar}), 8.78 (d, 1H, $3J_{HH}$ = 7Hz, CH_{Ar}); ^{13}C NMR (125 MHz, DMSO-d_6) δ (ppm) = 25.2, 48.7, 66.7, 98.5, 116.7, 120.5, 124.8, 125.6, 126.3, 126.4, 127.1, 128.5, 128.8, 129.2, 129.5, 130.6, 130.9, 134.0, 135.6, 150.8, 163.2, 164.9, 165.8; MS (EI) (70 ev), m/z (%): 397 (4) [M+2]+, 395 (28) [M]+, 393 (100), 364 (16), 350 (22), 324 (16), 309 (28), 197 (8), 153 (13), 127 (12), 102 (9), 84 (24).

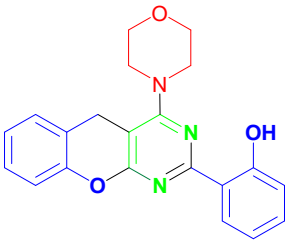
2-(Naphthalen-2-yl)-4-(piperidin-1-yl)-5*H*-benzopyrano[2,3-d]pyrimidine (3b): Yellow solid; Mp = 163-165°C; IR (KBr): 2843, 1598, 1575, 1423, 1386, 1231, 1119, 1011, 933, 759 cm^{-1}; ^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm) = 1.70 (s, 6H, $3CH_2$), 3.47 (s, 4H, $N(CH_2)_2$), 4.06

(s, 2H, CH_2), 7.15-7.22 (m, 2H, CH_{Ar}), 7.29 (t, 1H, $^3J_{HH}$ = 8 Hz, CH_{Ar}), 7.40 (d, 1H, $^3J_{HH}$ = 7.7 Hz, CH_{Ar}), 7.56-7.58 (m, 2H, CH_{Ar}), 7.62 (t, 1H, $^3J_{HH}$ = 7.8 Hz, CH_{Ar}), 8.01 (d, 1H, $^3J_{HH}$ = 6.8 Hz, CH_{Ar}), 8.05 (d, 2H, $^3J_{HH}$ = 7.7 Hz, CH_{Ar}), 8.78 (d, 1H, $^3J_{HH}$ = 7 Hz, CH_{Ar}); ^{13}C NMR (125 MHz, DMSO-d_6) δ (ppm) = 24.5, 25.5, 26.3, 49.5, 97.9, 116.7, 120.9, 121.5, 124.7, 125.8, 126.4, 126.9, 128.5, 128.7, 129.0, 129.5, 130.8, 130.9, 134.0, 135.8, 163.0, 165.0, 166.5; MS (EI) (70 ev), m/z (%): 395 (12.8) $[M+2]^+$, 393 (45.6) $[M]^+$, 309 (13), 281 (52), 207 (100), 191 (12), 153 (8), 96 (80), 84 (13), 73 (11).

4-Morpholino-2-(thiophen-2-yl)-5*H*-benzopyrano[2,3-d]pyrimidine (3c): Yellow solid; Mp = 210-212°C; IR (KBr): 2854, 1600, 1575, 1531, 1427, 1246, 1013, 920, 780 cm^{-1}; ^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm) = 3.51 (t, 4H, $^3J_{HH}$ = 4.4 Hz, $2CH_2$), 3.78 (t, 4H, $^3J_{HH}$ = 5 Hz, $2CH_2$), 4.00 (s, 2H, CH_2), 7.13-7.19 (m, 3H, CH_{Ar}), 7.28 (t, 1H, $^3J_{HH}$ = 7.25Hz, CH_{Ar}) 7.35 (1H, d, $^3J_{HH}$ = 7.3 Hz, CH_{Ar}), 7.71 (d, 1H, $^3J_{HH}$ = 6.1 Hz, CH_{Ar}), 7.86 (d, 1H, $^3J_{HH}$ = 6.1 Hz,, CH_{Ar}); ^{13}C NMR (125 MHz, DMSO-d_6) δ (ppm) = 25.2, 48.6, 54.9, 66.5, 97.8, 116.7, 120.5, 124.8, 128.5, 128.7, 129.5, 130.7, 150.5, 157.5, 165.5, 184.2, 186.3; MS (EI) (70 ev), m/z (%): 353 (9) $[M+2]^+$, 351 (100) $[M]^+$, 319 (11), 306 (48), 293 (61), 281 (28), 265 (25), 207 (42), 191 (6), 155 (15), 130 (16), 119 (22), 102 (16), 77 (8).

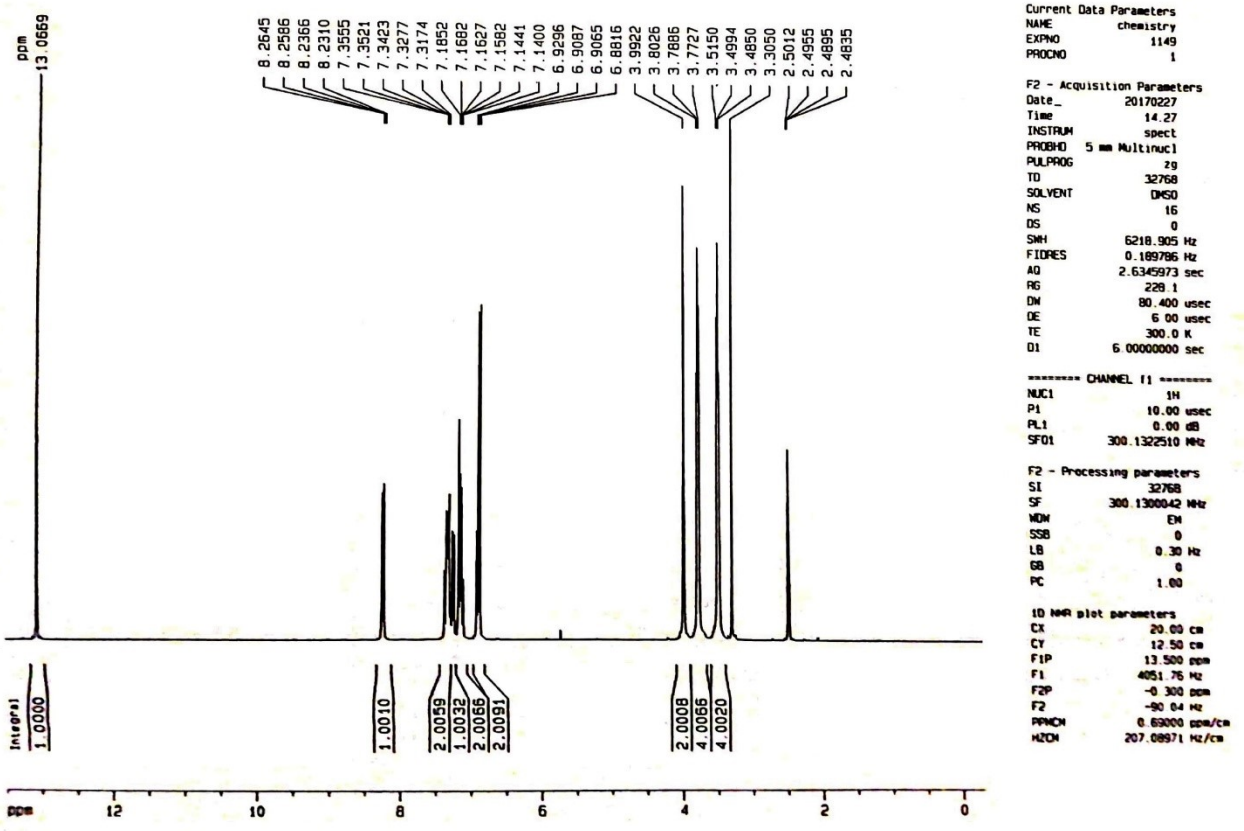
N-Benzyl-N-methyl-2-(naphthalen-2-yl)-5*H*-benzopyrano[2,3-d]pyrimidin-4-amine (3d): Yellow solid; Mp = 225-227°C; IR (KBr): 3087, 3044, 2694, 1622, 1598, 1536, 1383, 1235, 1109, 929, 790, 735 cm^{-1}; ^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm) = 3.23 (s, 3H, CH_3), 4.26 (d, 2H, $^4J_{HH}$ = 30 Hz, CH_2), 4.88 (d, 2H, $^4J_{HH}$ = 9.5 Hz, CH_2), 6.90 (t, 1H, $^3J_{HH}$ = 7.8 Hz, CH_{Ar}), 7.12-7.18 (m, 3H, CH_{Ar}), 7.28-7.40 (m, 6H, CH_{Ar}), 7.52 (t, 1H, $^3J_{HH}$ = 8.6 Hz, CH_{Ar}), 7.58 (t,

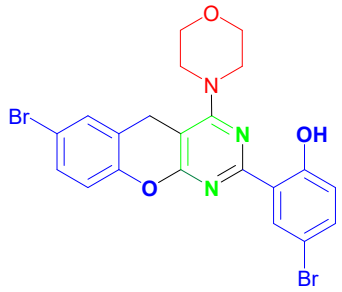
1H, $^{3}J_{HH}$ = 8.6 Hz, CH_{Ar}), 7.96-8.03 (m, 2H, CH_{Ar}), 8.22 (d, 1H, $^{3}J_{HH}$ = 8.4 Hz, CH_{Ar}), 8.69 (d, 1H, $^{3}J_{HH}$ = 8.5 Hz, CH_{Ar}); ^{13}C NMR (125 MHz, DMSO-d_6) δ (ppm) = 25.5, 31.1, 55.7, 94.9, 116.5, 117.8, 119.2, 124.5, 125.5, 126.2, 126.4, 126.8, 127.5, 127.6, 129.0, 129.1, 129.5, 133.9, 138.2, 138.7, 150.6, 160.2, 175.4, 181.6; MS (EI) (70 ev), m/z (%): 431 (2) $[M+2]^{+}$, 429 (16) $[M]^{+}$, 338 (100), 281 (8), 207 (16), 185 (8), 91 (16).



2-(4-Morpholino-5*H*-benzopyrano[2,3-*d*]pyrimidin-2-yl)phenol (1a)

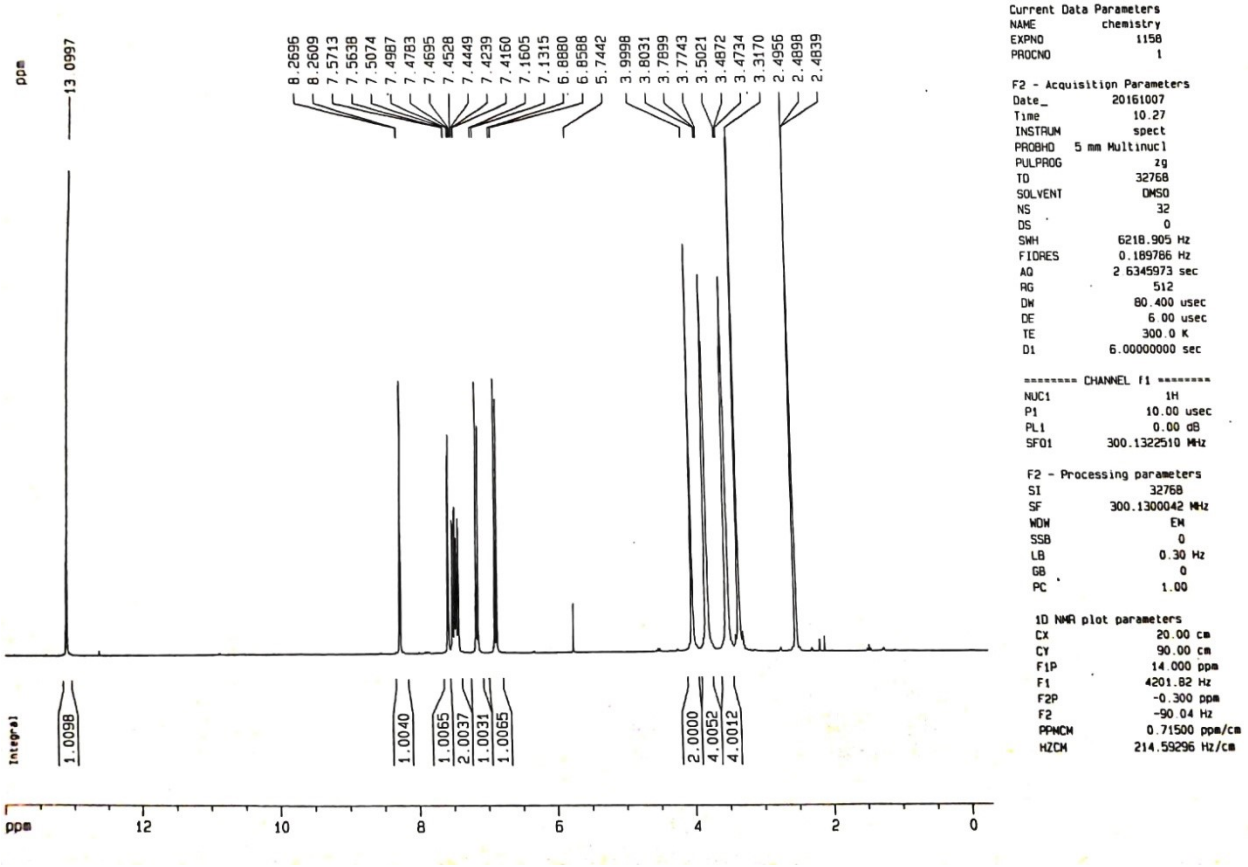
^{1}H NMR spectrum



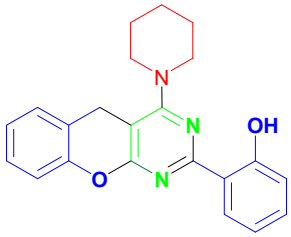


4-Bromo-2-(7-bromo-4-morpholino-5*H*-benzopyrano[2,3-d]pyrimidin-2-yl)phenol (1b)

^{1}H NMR spectrum

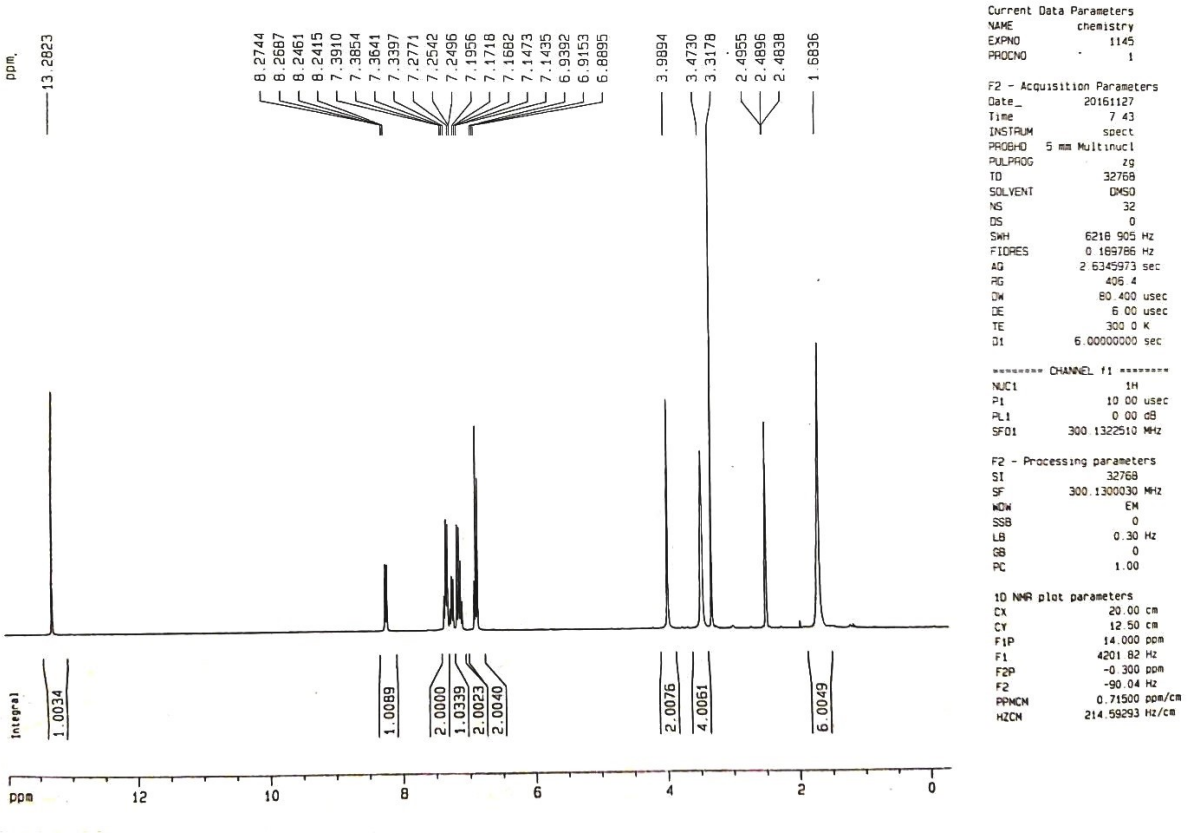



```
Current Data Parameters
NAME                  chemistry
EXPNO                      1158
PROCNO                        1

F2 - Acquisition Parameters
Date_                 20161007
Time                     10.27
INSTRUM                  spect
PROBHD    5 mm Multinucl
PULPROG                     zg
TD                       32768
SOLVENT                  DMSO
NS                         32
DS                          0
SWH                   6218.905 Hz
FIDRES                0.189786 Hz
AQ                    2.6345973 sec
RG                        512
DW                     80.400 usec
DE                      6.00 usec
TE                      300.0 K
D1                    6.00000000 sec

======== CHANNEL f1 ========
NUC1                        1H
P1                      10.00 usec
PL1                      0.00 dB
SFO1                300.1322510 MHz

F2 - Processing parameters
SI                       32768
SF                  300.1300042 MHz
WDW                         EM
SSB                          0
LB                      0.30 Hz
GB                           0
PC                        1.00

1D NMR plot parameters
CX                      20.00 cm
CY                      90.00 cm
F1P                    14.000 ppm
F1                   4201.82 Hz
F2P                    -0.300 ppm
F2                    -90.04 Hz
PPMCM                 0.71500 ppm/cm
HZCM                214.59296 Hz/cm
```

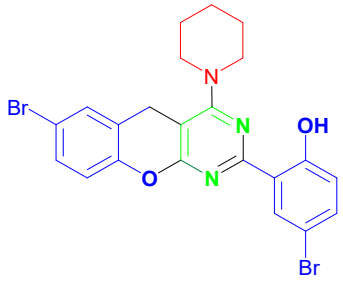


2-(4-(Piperidin-1-yl)-5*H*-benzopyrano[2,3-d]pyrimidin-2-yl)phenol (1c)

^{1}H NMR spectrum

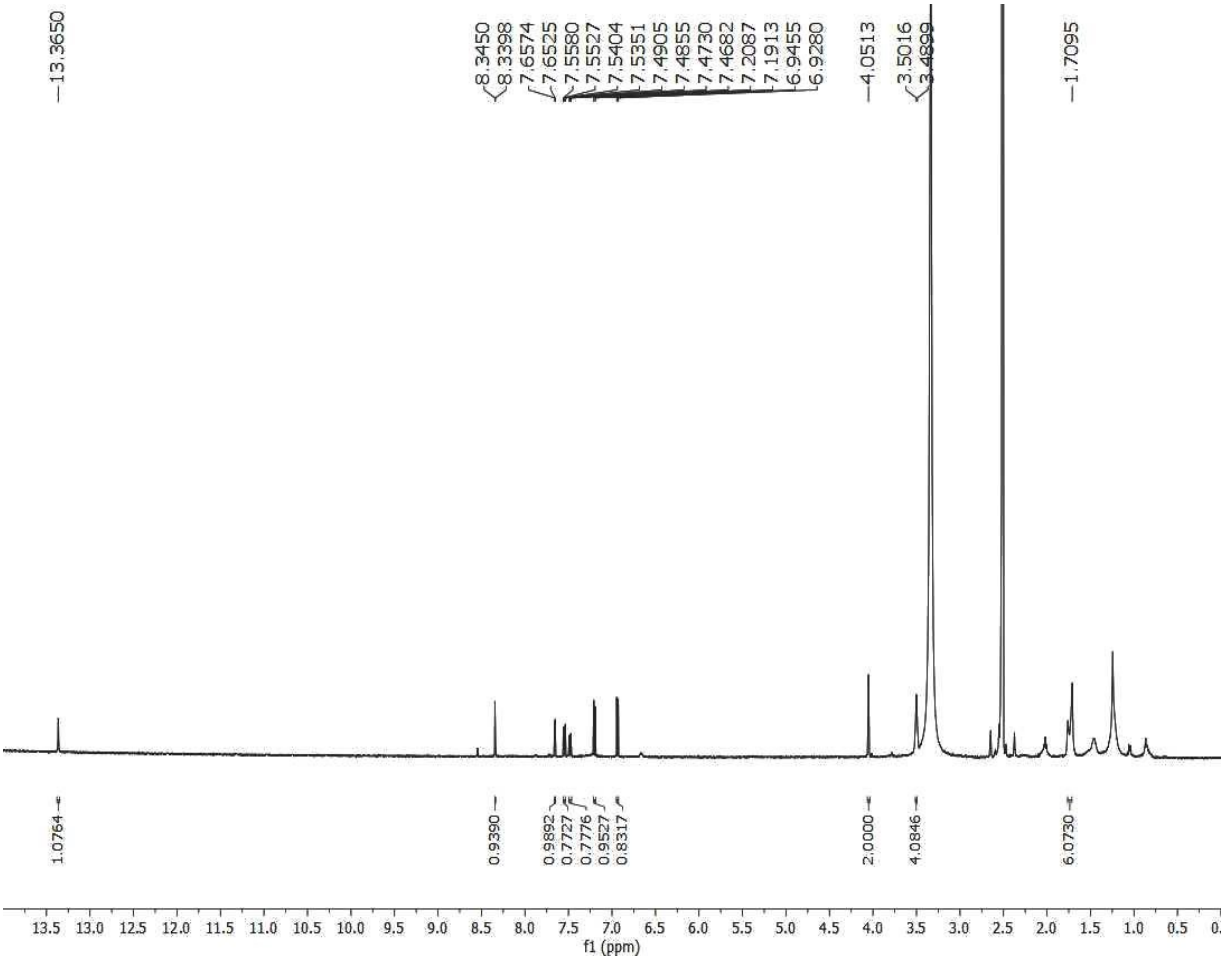



```
Current Data Parameters
NAME            chemistry
EXPNO                1145
PROCNO                  1

F2 - Acquisition Parameters
Date_           20161127
Time                7.43
INSTRUM             spect
PROBHD   5 mm Multinucl
PULPROG               zg
TD                 32768
SOLVENT             DMSO
NS                    32
DS                     0
SWH              6218.905 Hz
FIDRES           0.189786 Hz
AQ               2.6345973 sec
RG                   406.4
DW                  80.400 usec
DE                   6.00 usec
TE                  300.0 K
D1               6.00000000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                  10.00 usec
PL1                  0.00 dB
SFO1           300.1322510 MHz

F2 - Processing parameters
SI                 32768
SF            300.1300030 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 20.00 cm
CY                 12.50 cm
F1P               14.000 ppm
F1              4201.82 Hz
F2P               -0.300 ppm
F2               -90.04 Hz
PPMCM          0.71500 ppm/cm
HZCM          214.59293 Hz/cm
```

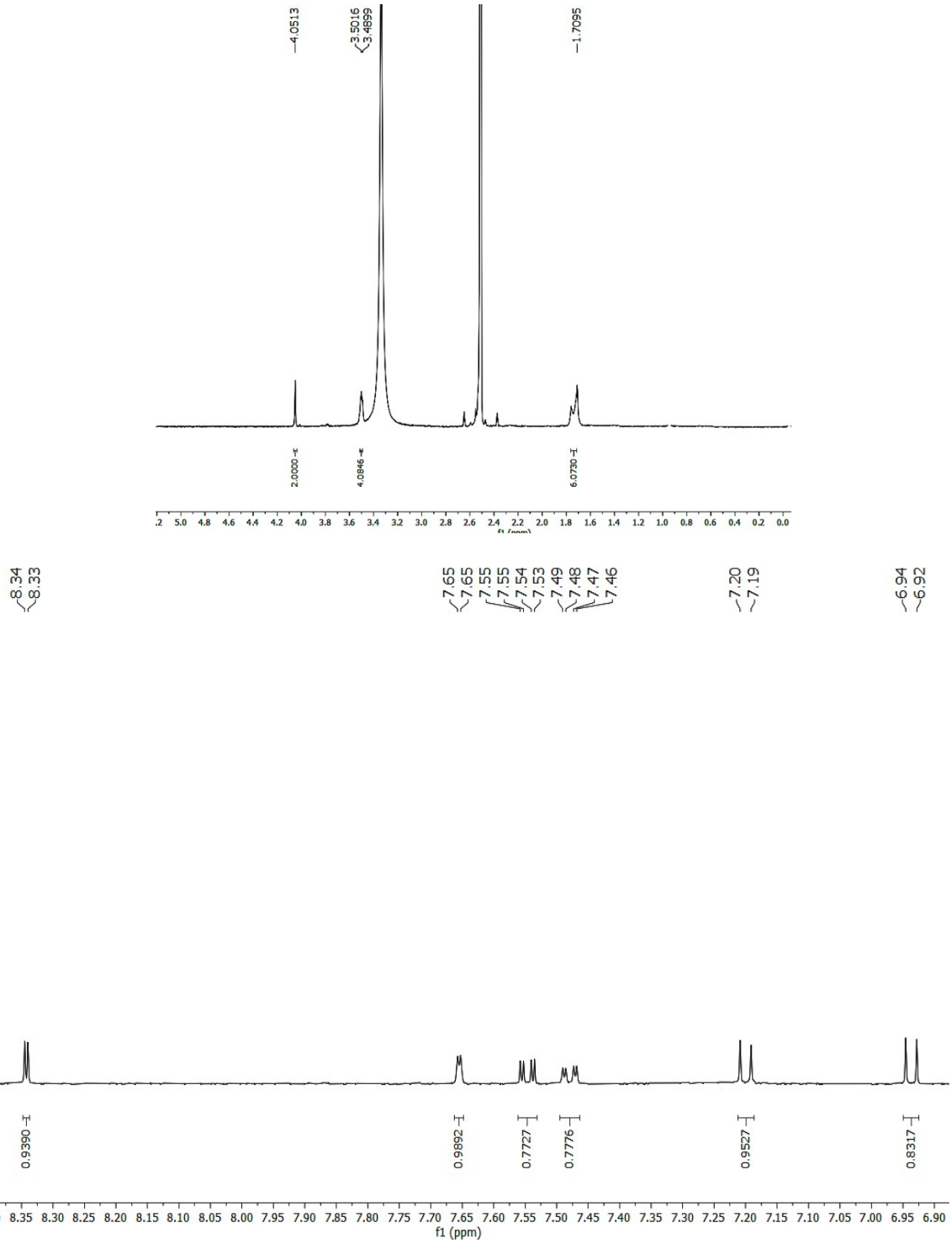


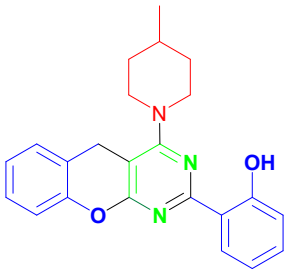
4-Bromo-2-(7-bromo-4-(piperidin-1-yl)-5*H*-benzopyrano[2,3-d]pyrimidin-2-yl)phenol (1d)

^{1}H NMR spectrum



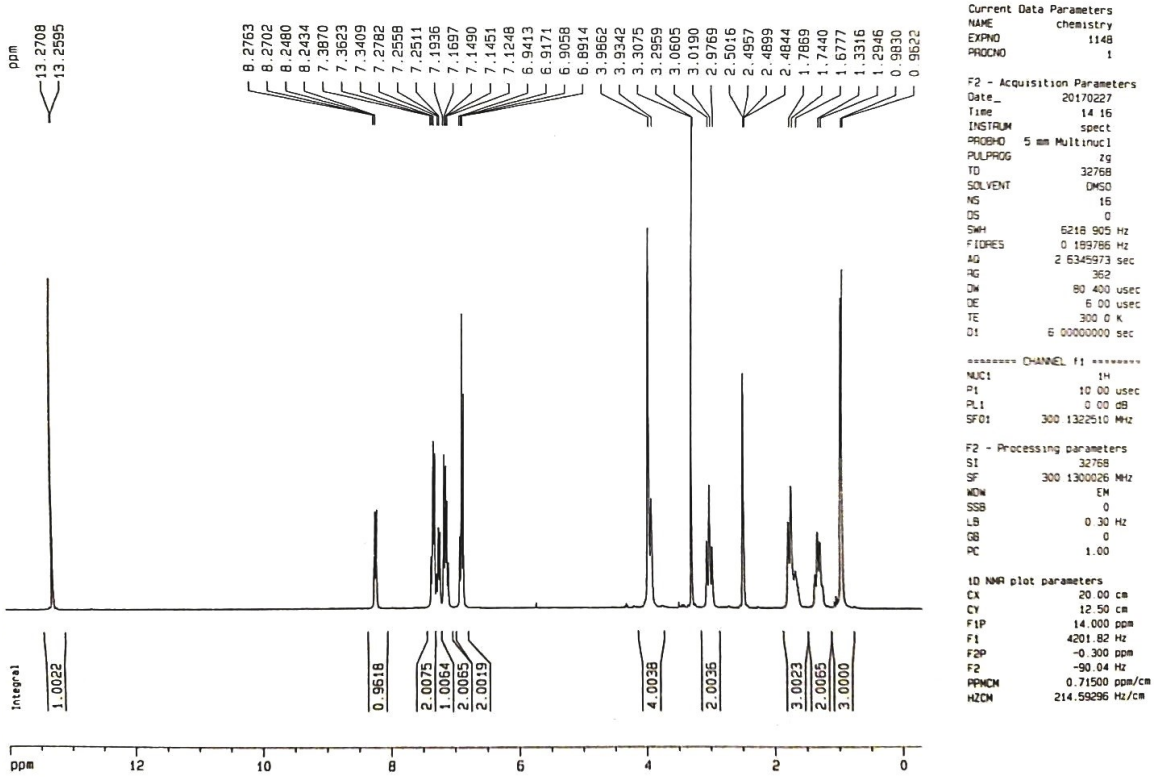
Expanded ^{1}H NMR spectrum

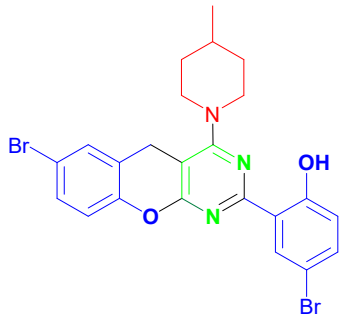




2-(4-(4-Methylpiperidin-1-yl)-5*H*-benzopyrano[2,3-d]pyrimidin-2-yl)phenol (1e)

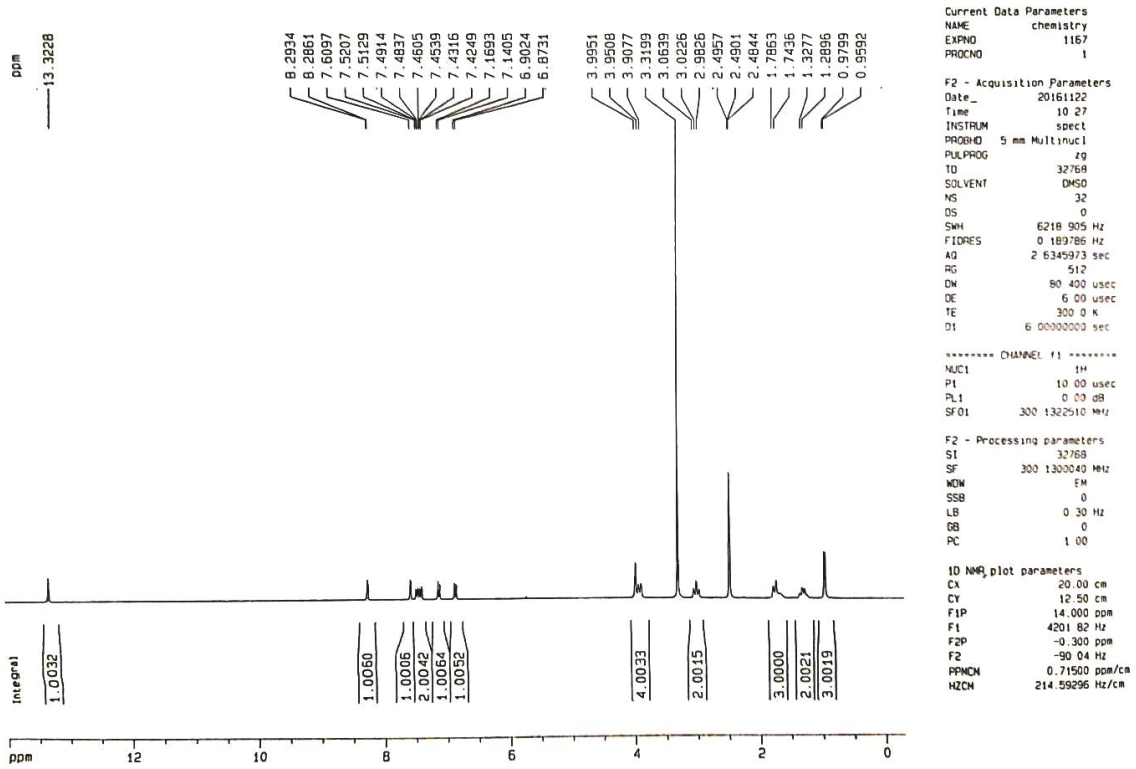
^{1}H NMR spectrum



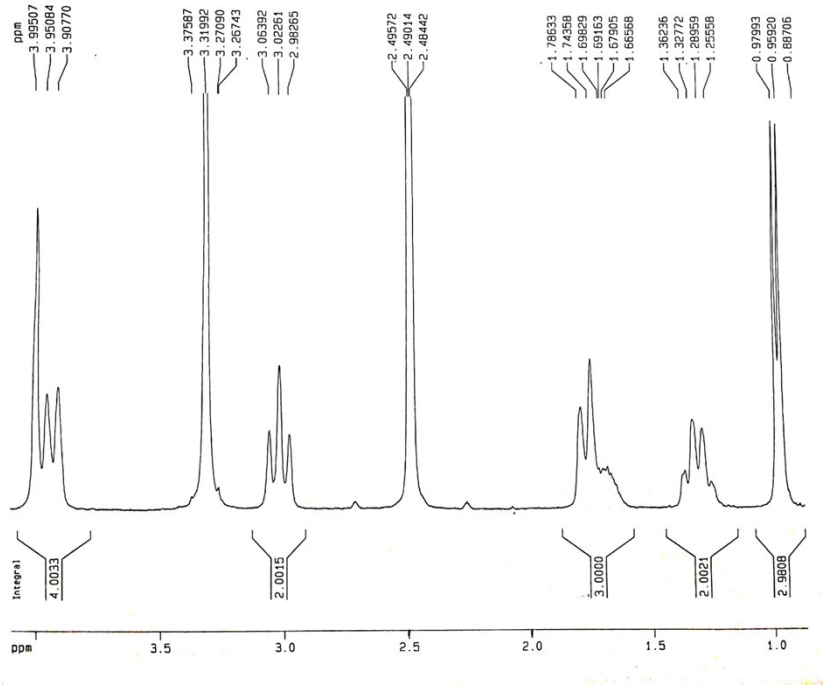


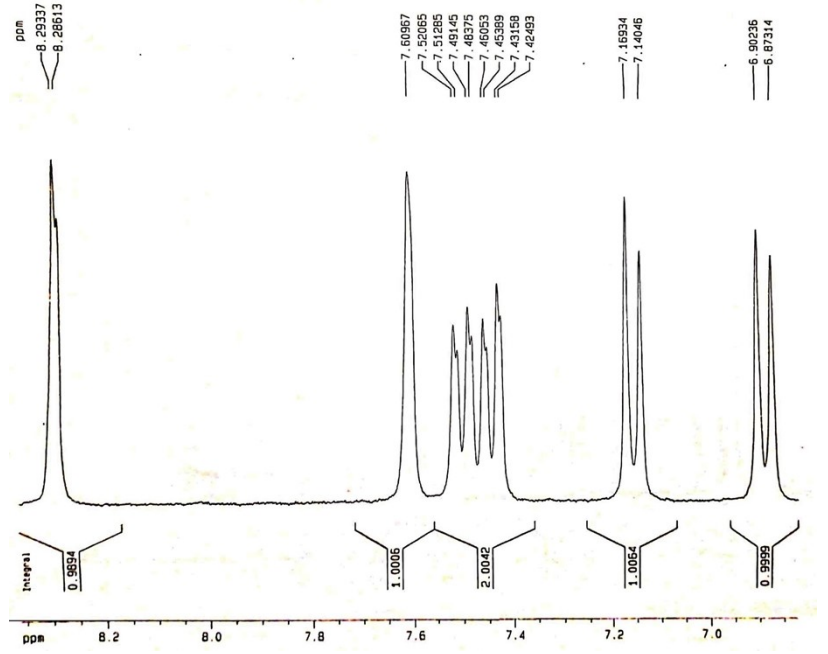
4-Bromo-2-(7-bromo-4-(4-methylpiperidin-1-yl)-5*H*-benzopyrano[2,3-d]pyrimidin-2-yl)phenol (1f)

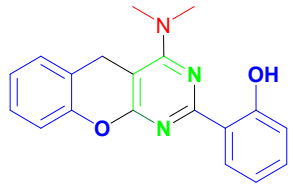
^{1}H NMR spectrum



Expanded ^{1}H NMR spectrum

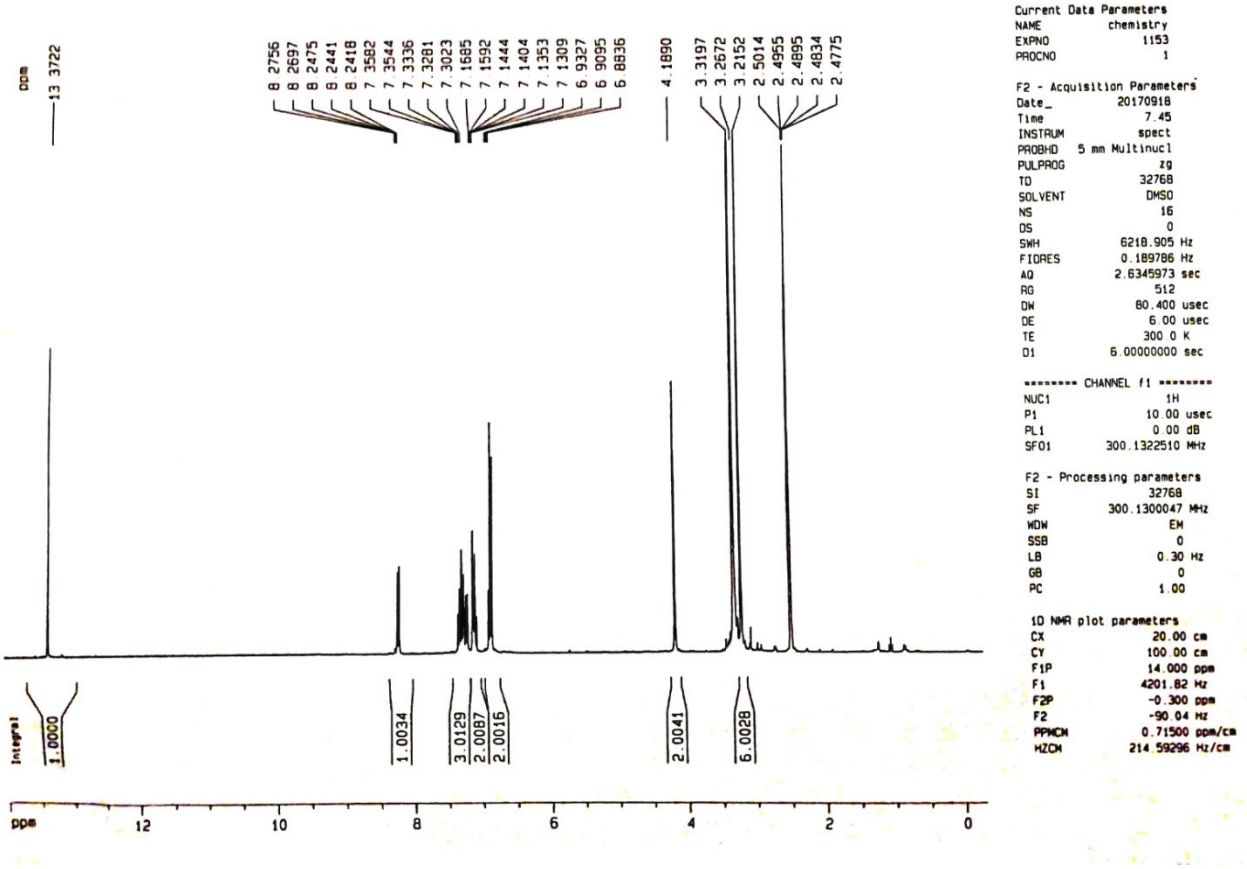




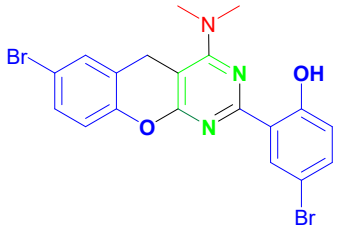


2-(4-(Dimethylamino)-5*H*-benzopyrano[2,3-d]pyrimidin-2-yl)phenol (1g)

^{1}H NMR spectrum

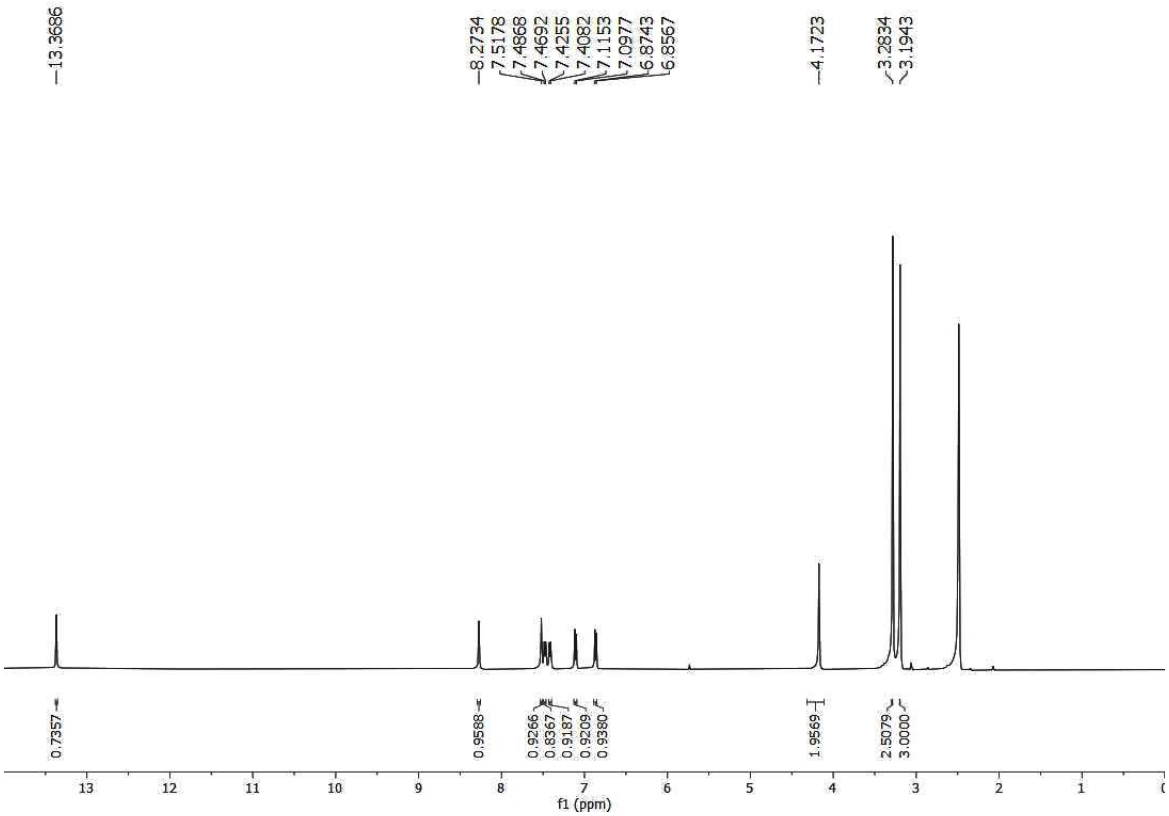



```
Current Data Parameters
NAME                  chemistry
EXPNO                      1153
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20170918
Time                       7.45
INSTRUM                   spect
PROBHD    5 mm Multinucl
PULPROG                     zg
TD                         32768
SOLVENT                    DMSO
NS                           16
DS                            0
SWH                    6218.905 Hz
FIDRES                 0.189786 Hz
AQ                     2.6345973 sec
RG                           512
DW                      80.400 usec
DE                       6.00 usec
TE                       300.0 K
D1                     6.00000000 sec

======== CHANNEL f1 ========
NUC1                         1H
P1                      10.00 usec
PL1                      0.00 dB
SFO1                300.1322510 MHz

F2 - Processing parameters
SI                         32768
SF                  300.1300047 MHz
WDW                           EM
SSB                           0
LB                       0.30 Hz
GB                            0
PC                         1.00

1D NMR plot parameters
CX                       20.00 cm
CY                      100.00 cm
F1P                     14.000 ppm
F1                     4201.82 Hz
F2P                     -0.300 ppm
F2                      -90.04 Hz
PPMCM                 0.71500 ppm/cm
HZCM                214.59296 Hz/cm
```

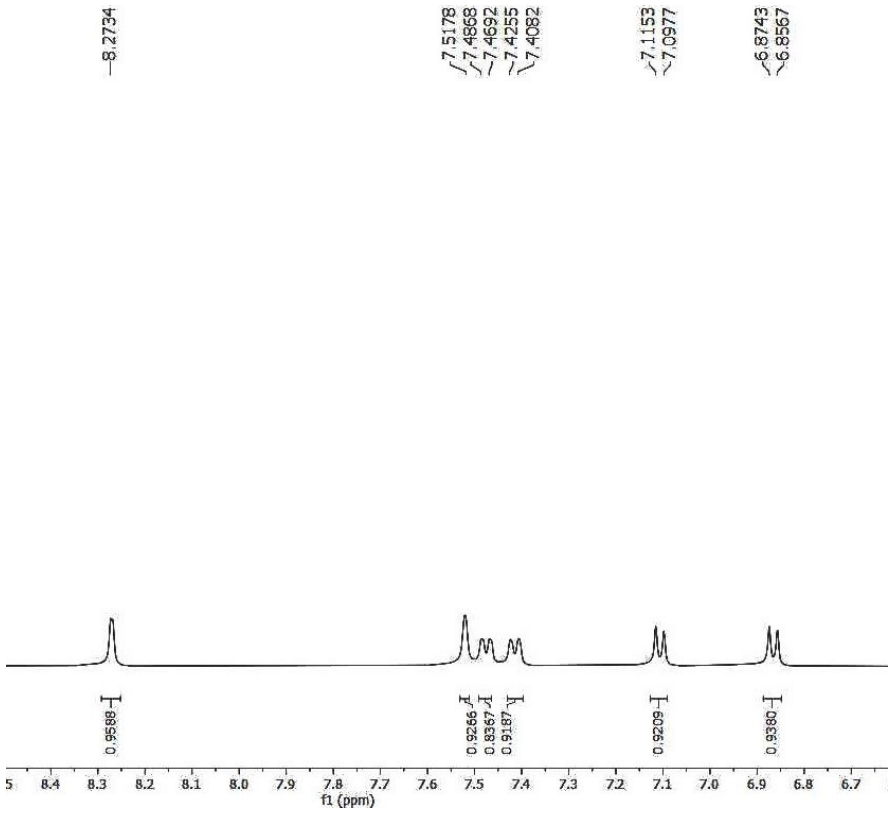


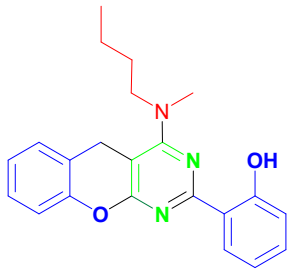
4-Bromo-2-(7-bromo-4-(dimethylamino)-5*H*-benzopyrano[2,3-d]pyrimidin-2-yl)phenol (1h)

^{1}H NMR spectrum



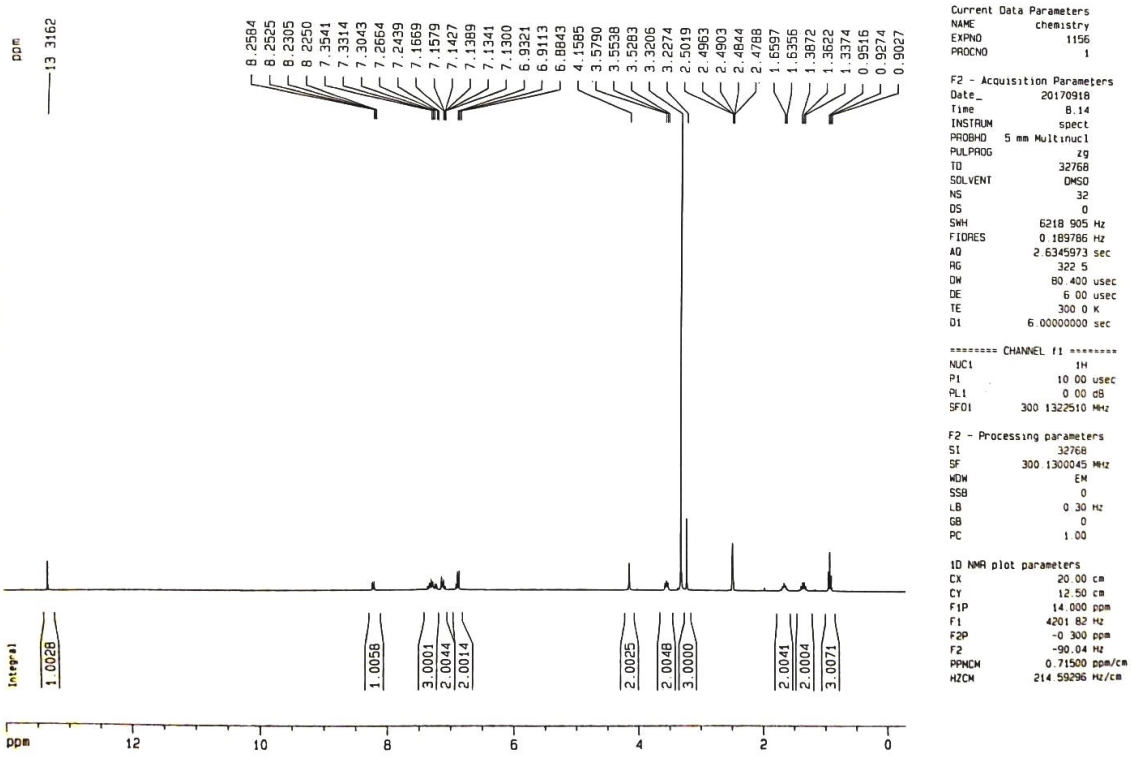
Expanded ^{1}H NMR spectrum





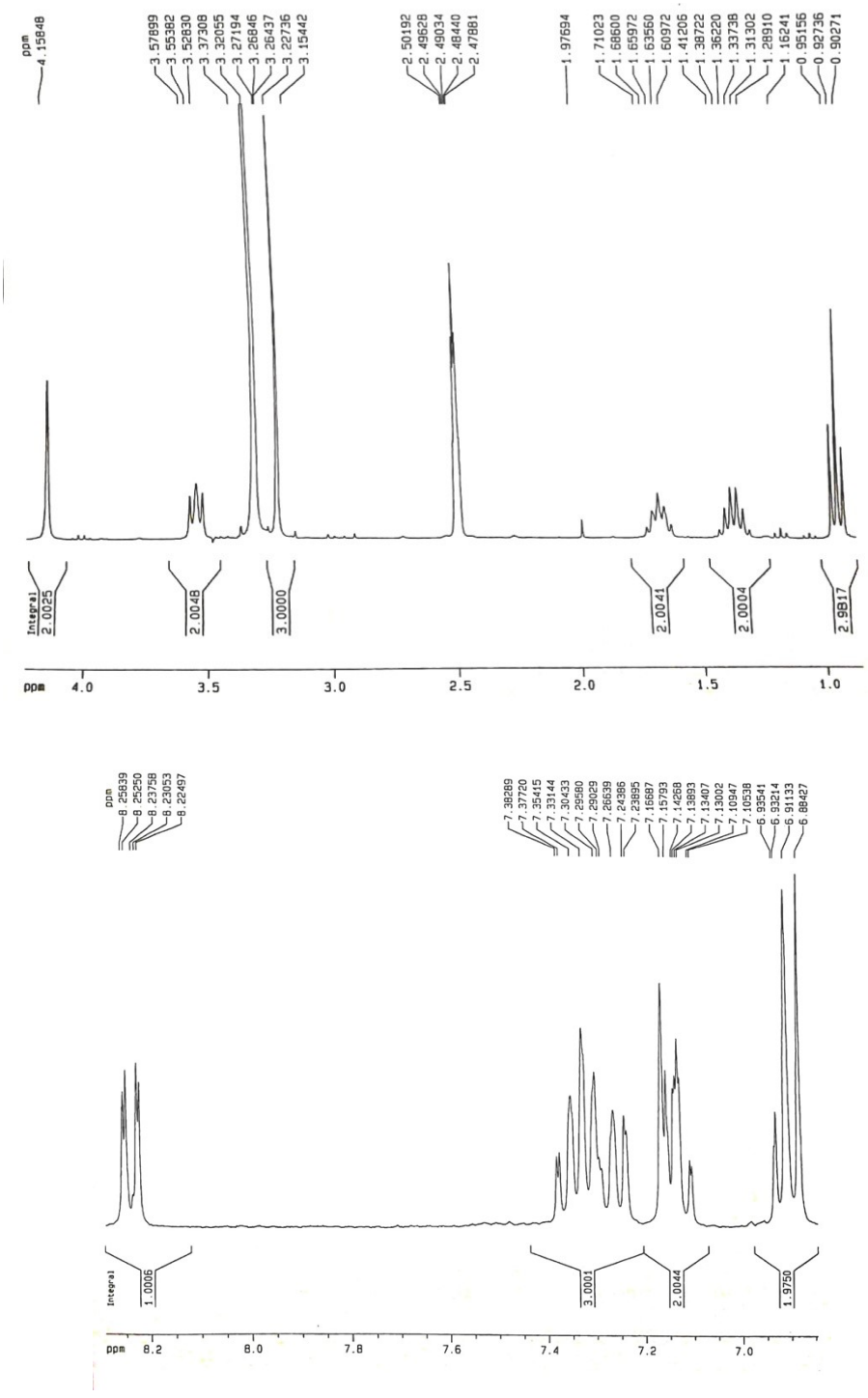
2-(4-(Butyl(methyl)amino)-5*H*-benzopyrano [2,3-d]pyrimidin-2-yl)phenol (1i)

^{1}H NMR spectrum

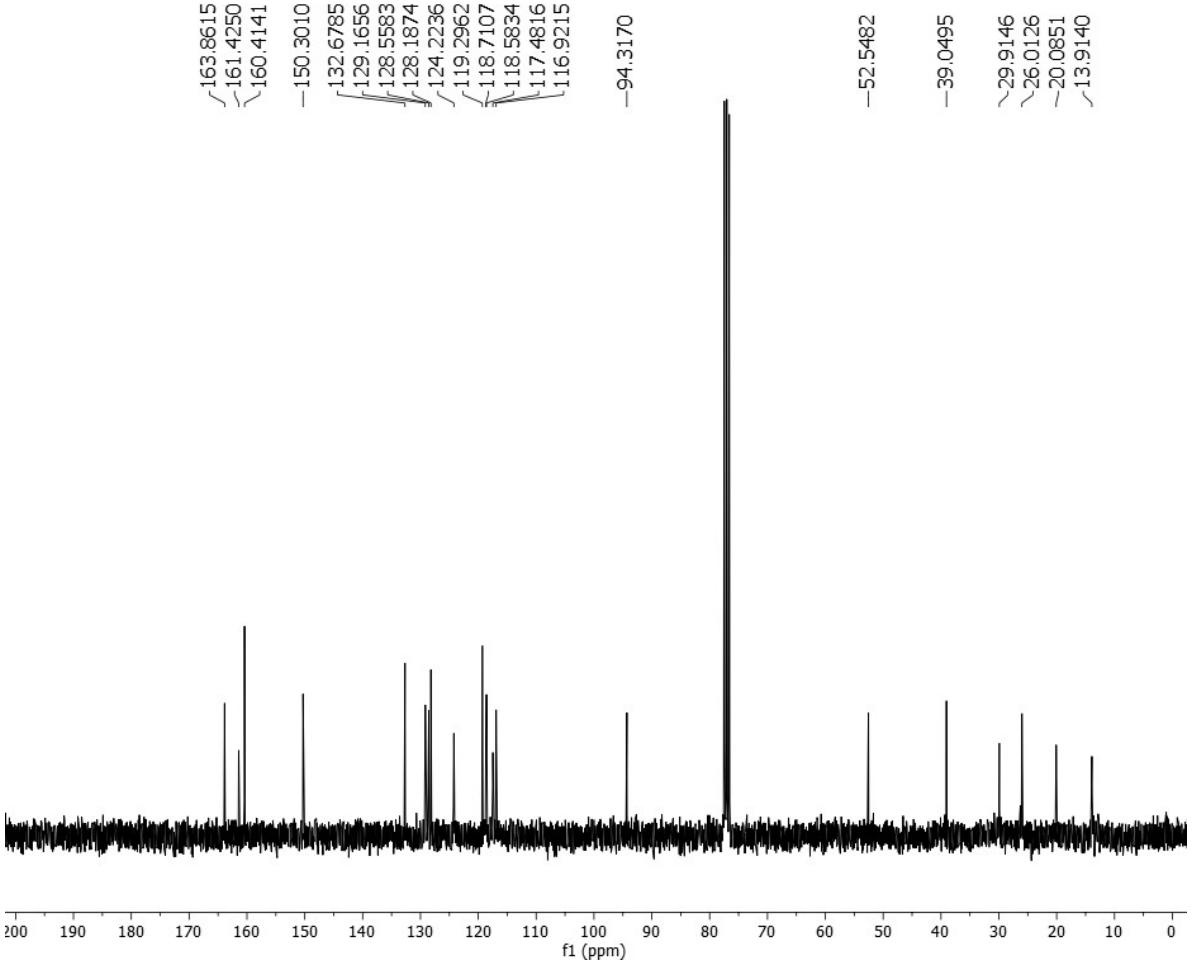



```
Current Data Parameters
NAME            chemistry
EXPNO              1156
PROCNO                1

F2 - Acquisition Parameters
Date_           20170918
Time                8.14
INSTRUM            spect
PROBHD   5 mm Multinucl
PULPROG              zg
TD                32768
SOLVENT            DMSO
NS                   32
DS                    0
SWH           6218.905 Hz
FIDRES        0.189786 Hz
AQ            2.6345973 sec
RG                322.5
DW               80.400 usec
DE                6.00 usec
TE               300.0 K
D1            6.00000000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                10.00 usec
PL1                0.00 dB
SFO1        300.1322510 MHz

F2 - Processing parameters
SI                32768
SF          300.1300045 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
CY                12.50 cm
F1P               14.000 ppm
F1              4201.82 Hz
F2P               -0.300 ppm
F2              -90.04 Hz
PPMCM          0.71500 ppm/cm
HZCM          214.59296 Hz/cm
```

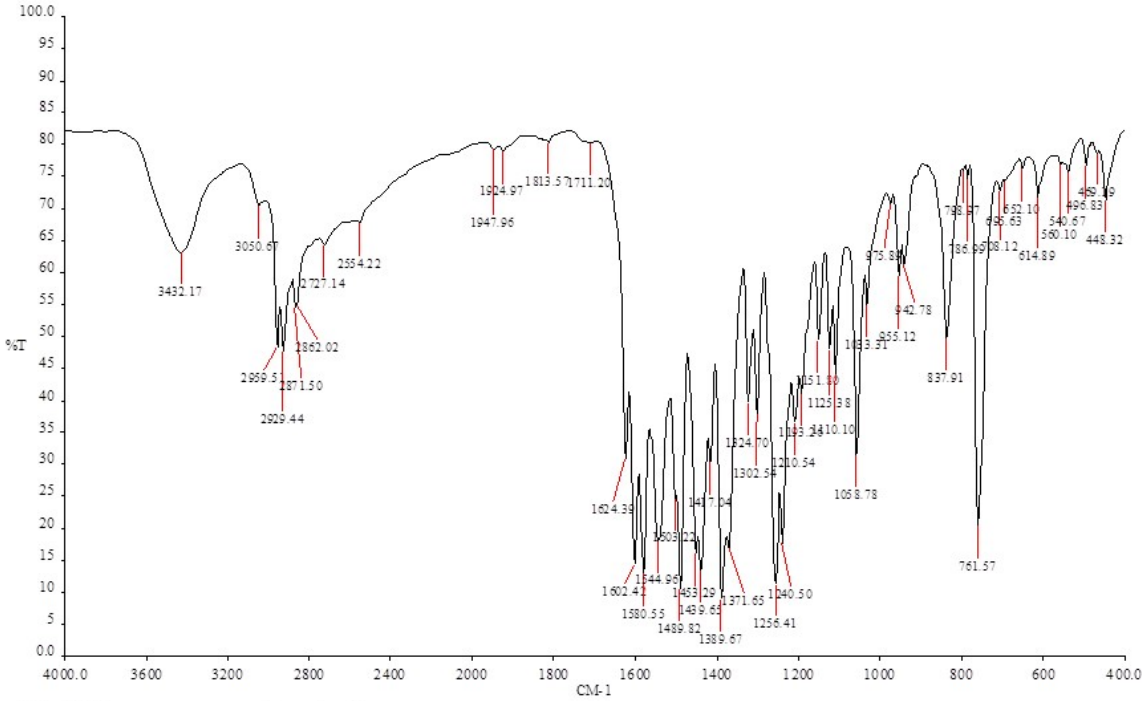
Expanded ^{1}H NMR spectrum



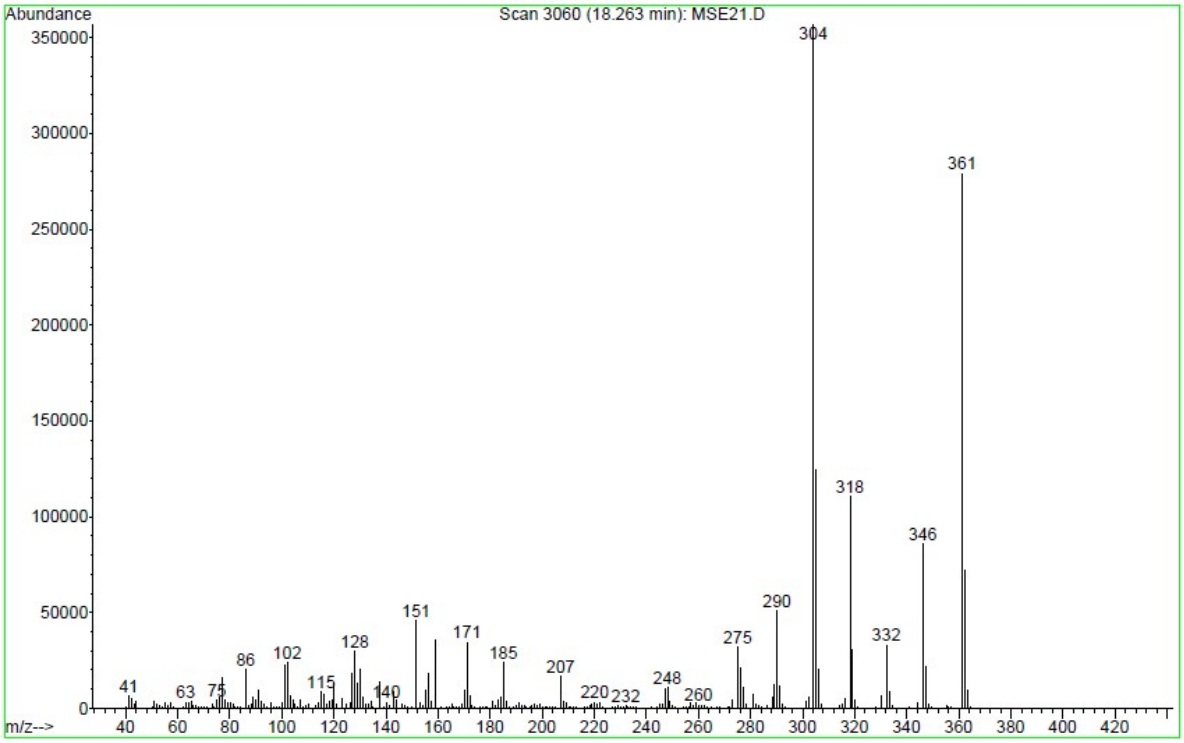
^{13}C NMR spectrum

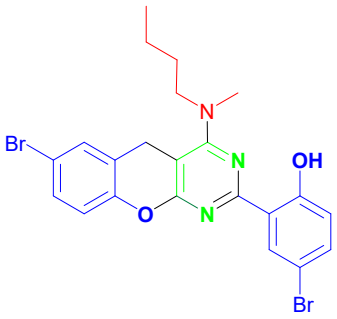


FTIR spectrum



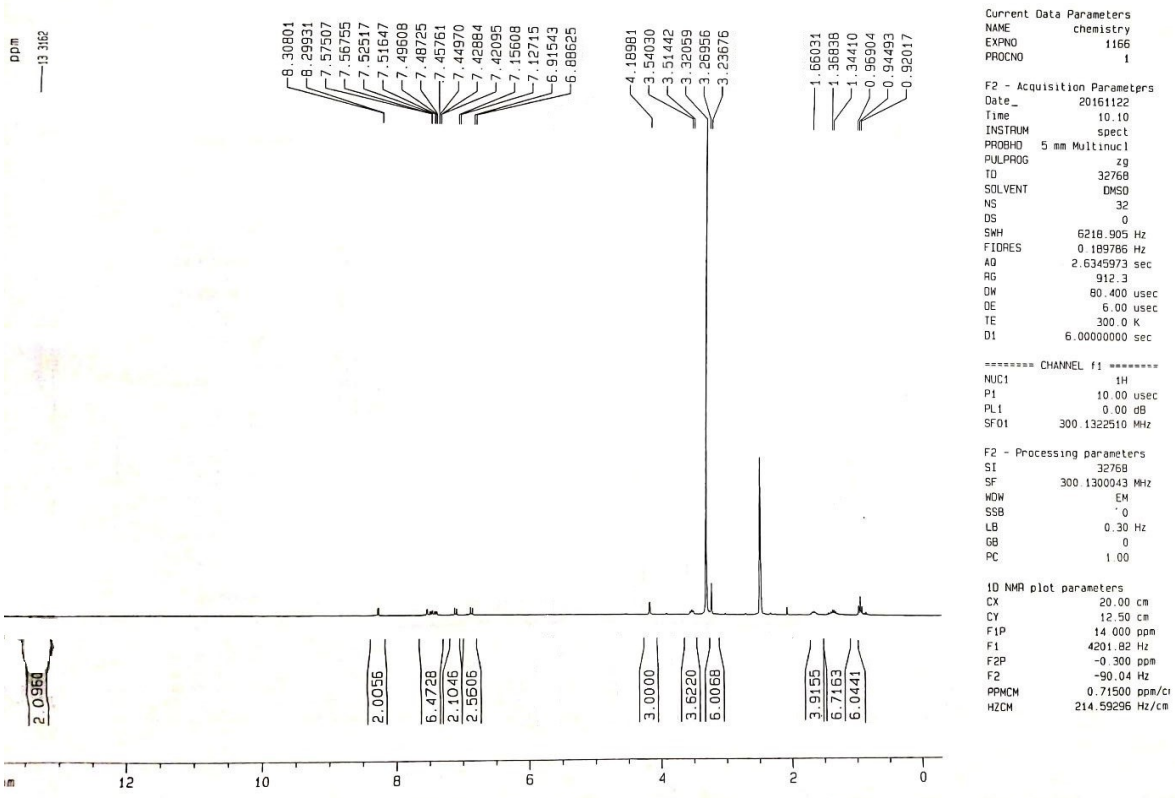
Mass spectrum



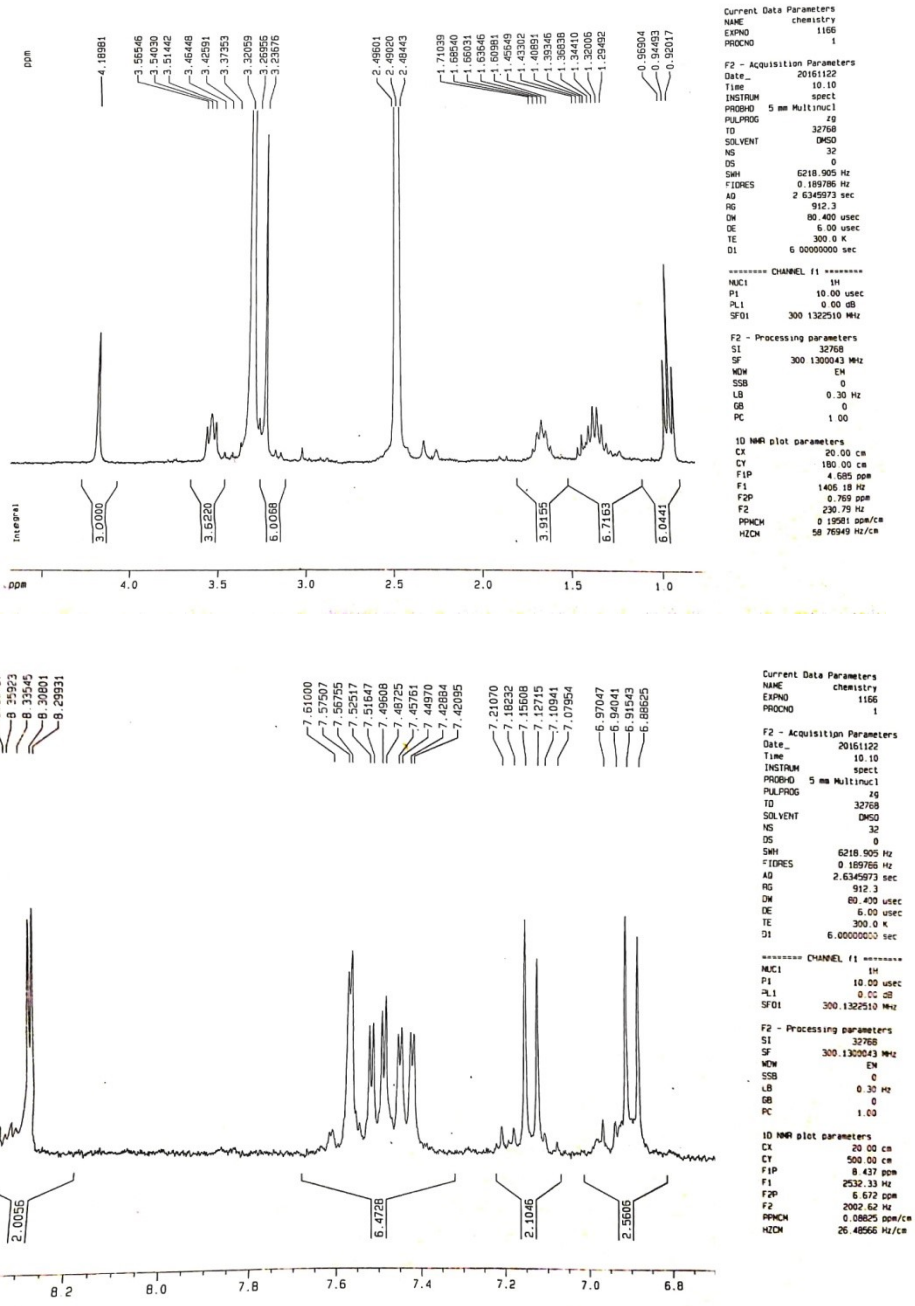


4-Bromo-2-(7-bromo-4-(butyl(methyl)amino)-5*H*-benzopyrano[2,3-d]pyrimidin-2-yl)phenol (1j)

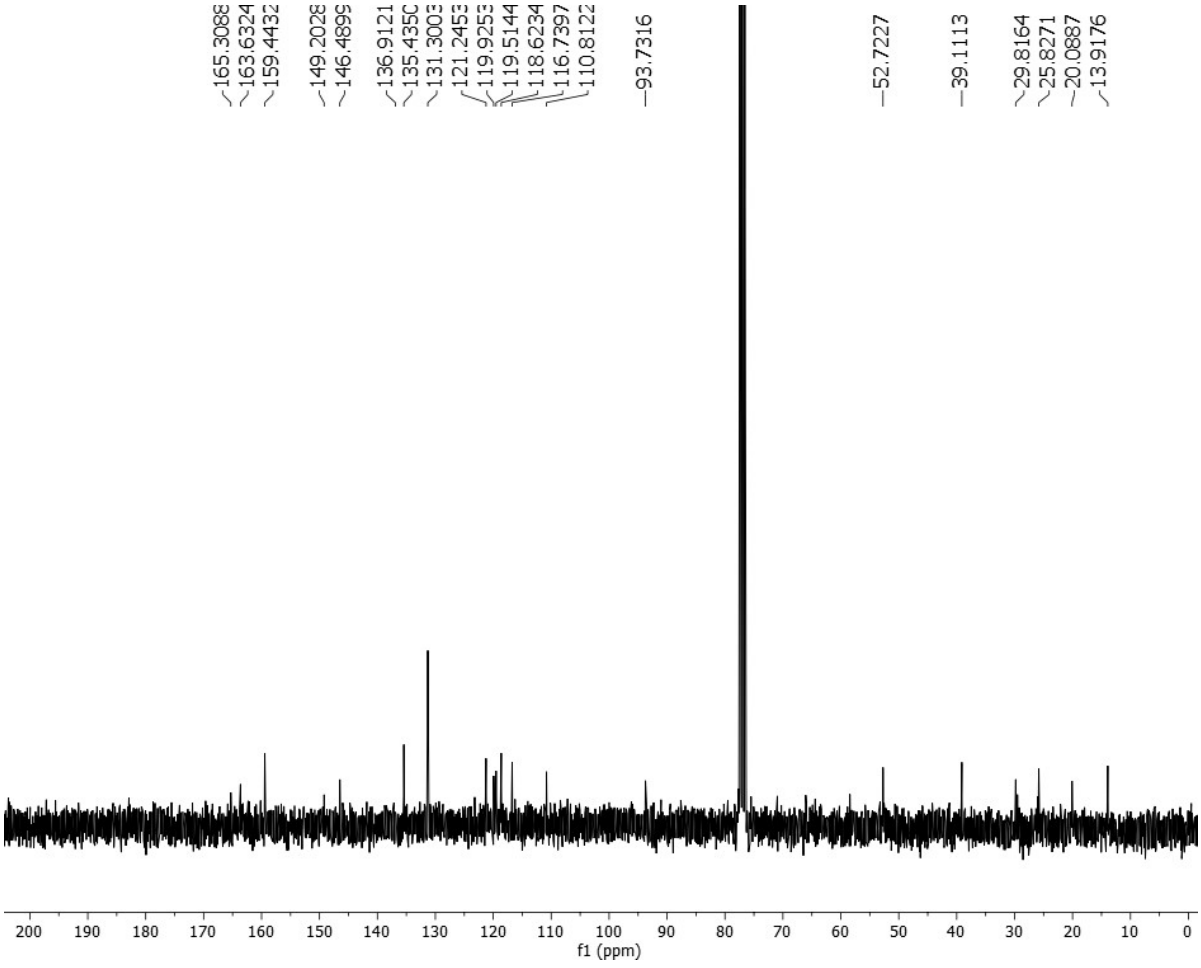
^{1}H NMR spectrum



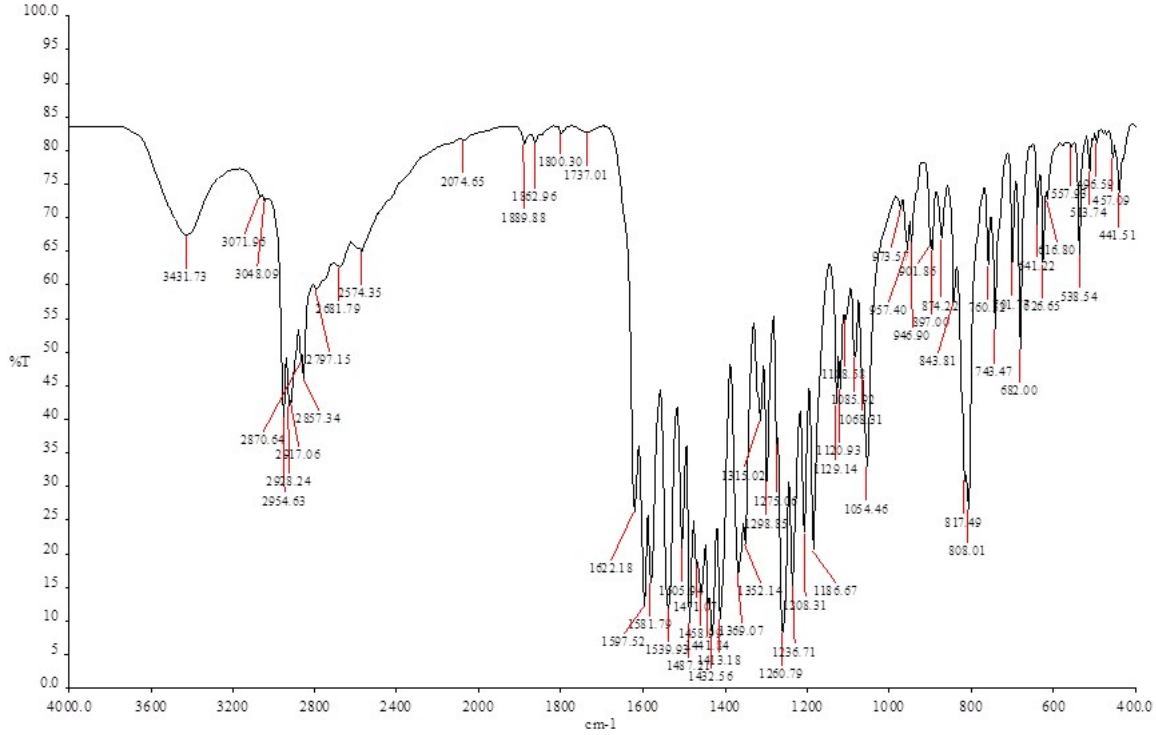
Expanded ^{1}H NMR spectrum



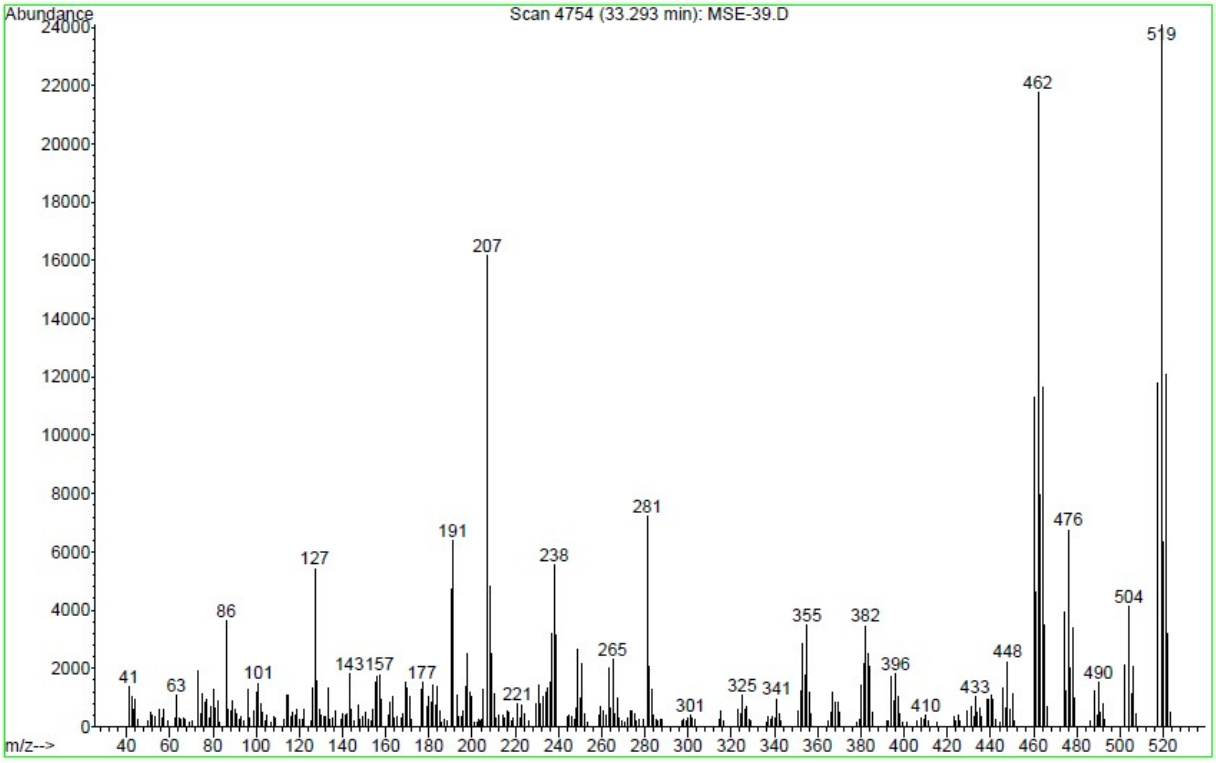
^{13}C NMR spectrum

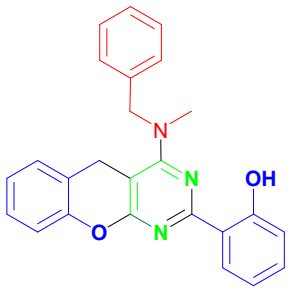


FTIR spectrum



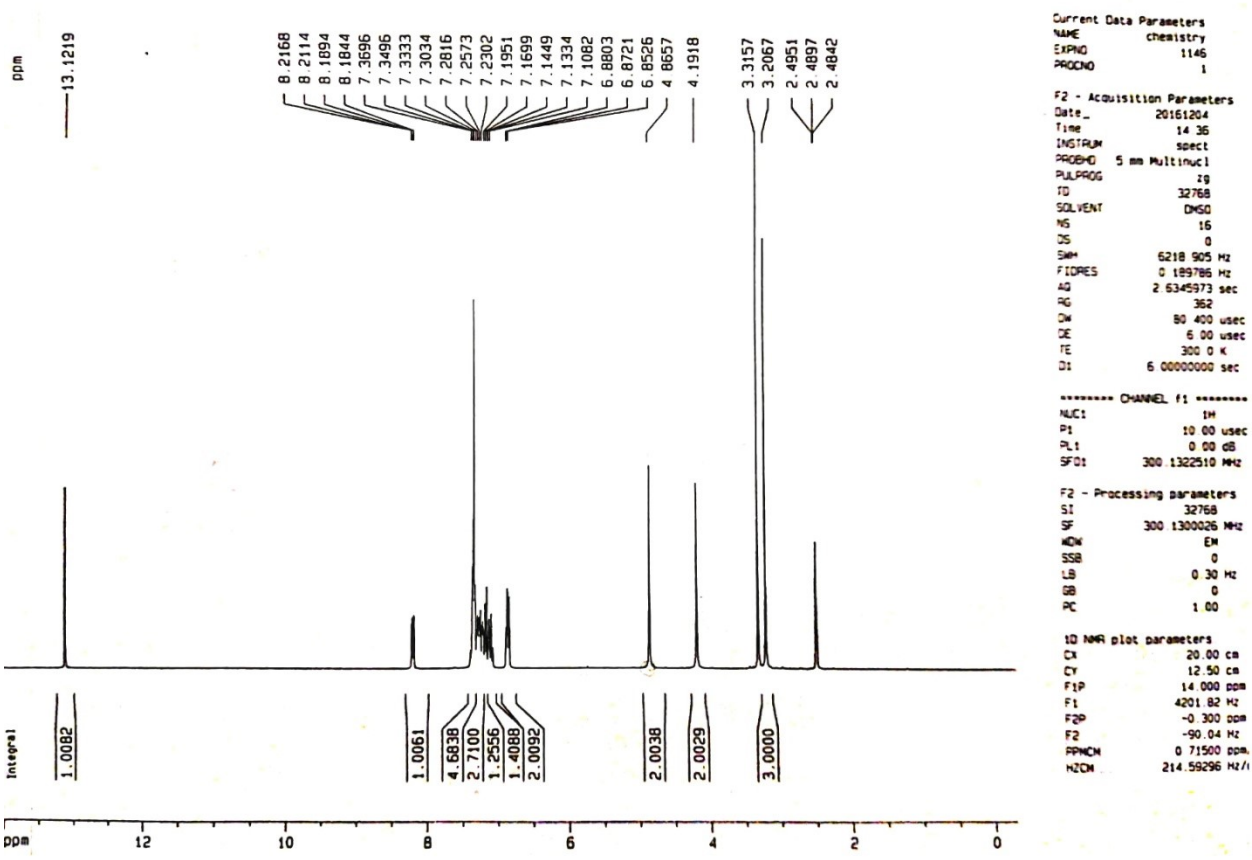
Mass spectrum



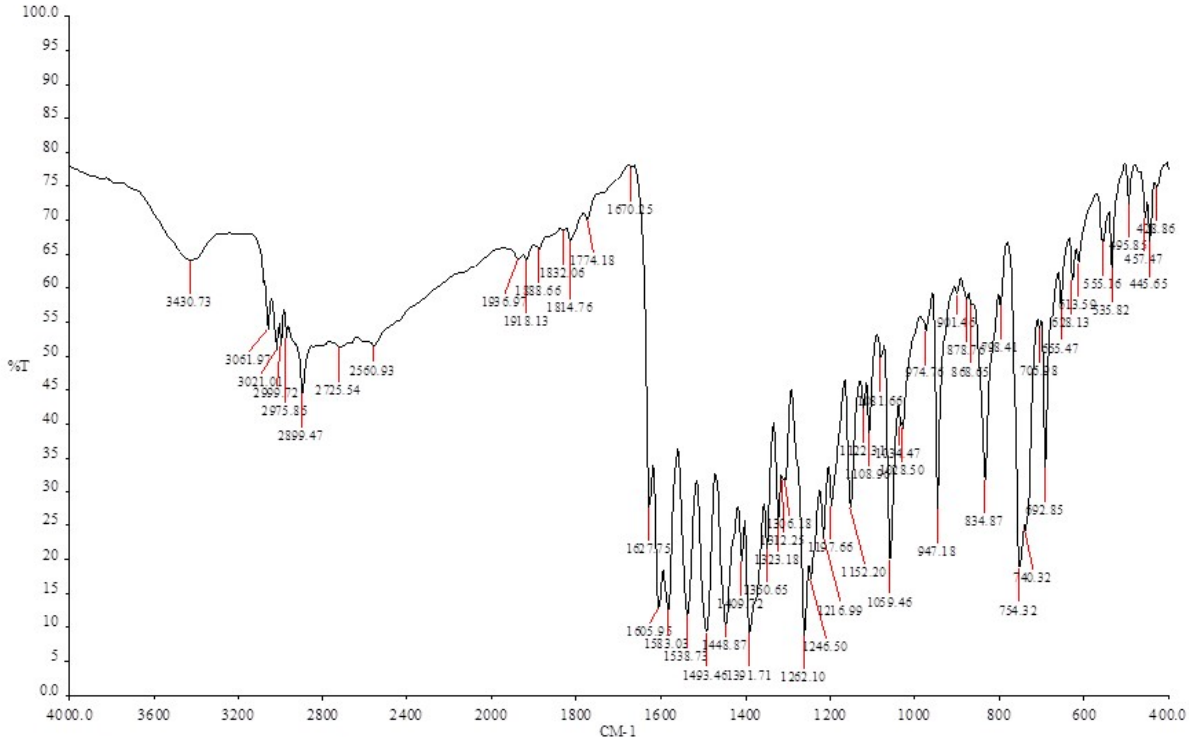


2-(4-(Benzyl(methyl)amino)-5*H*- benzopyrano[2,3-d]pyrimidin-2-yl)phenol (1k)

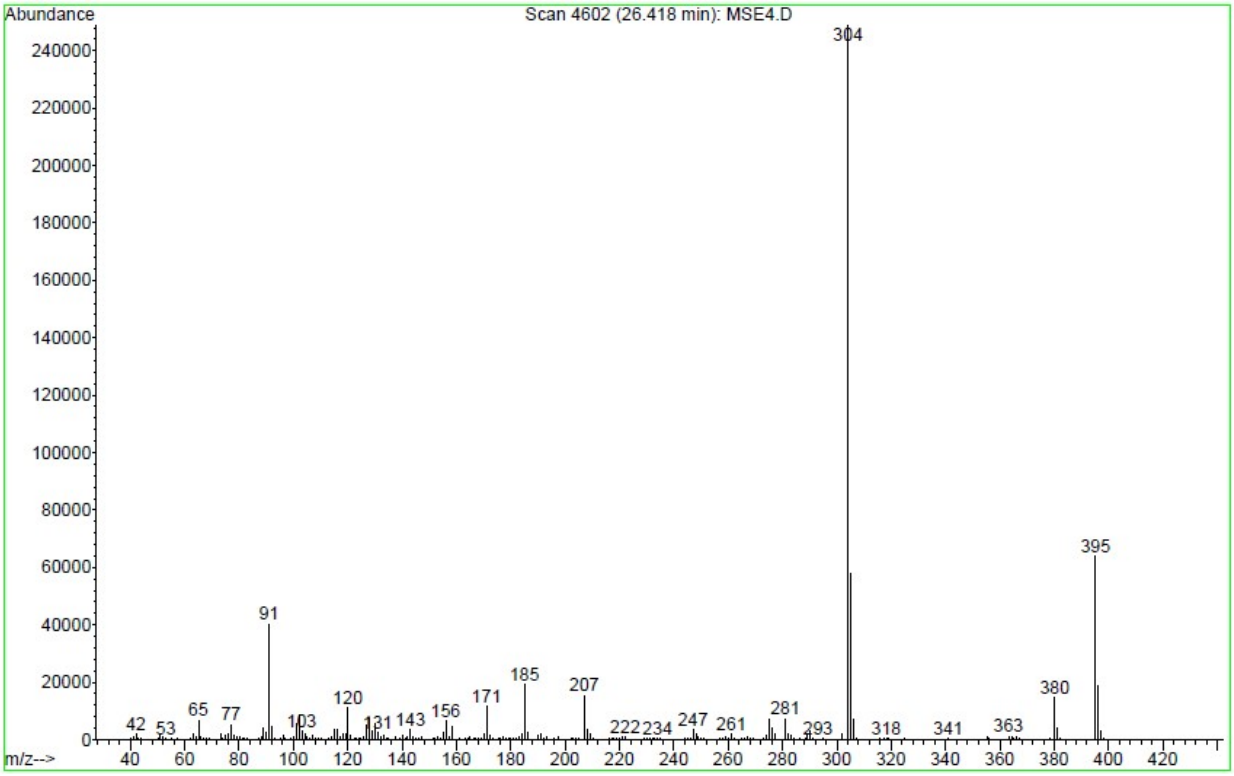
^{1}H NMR spectrum



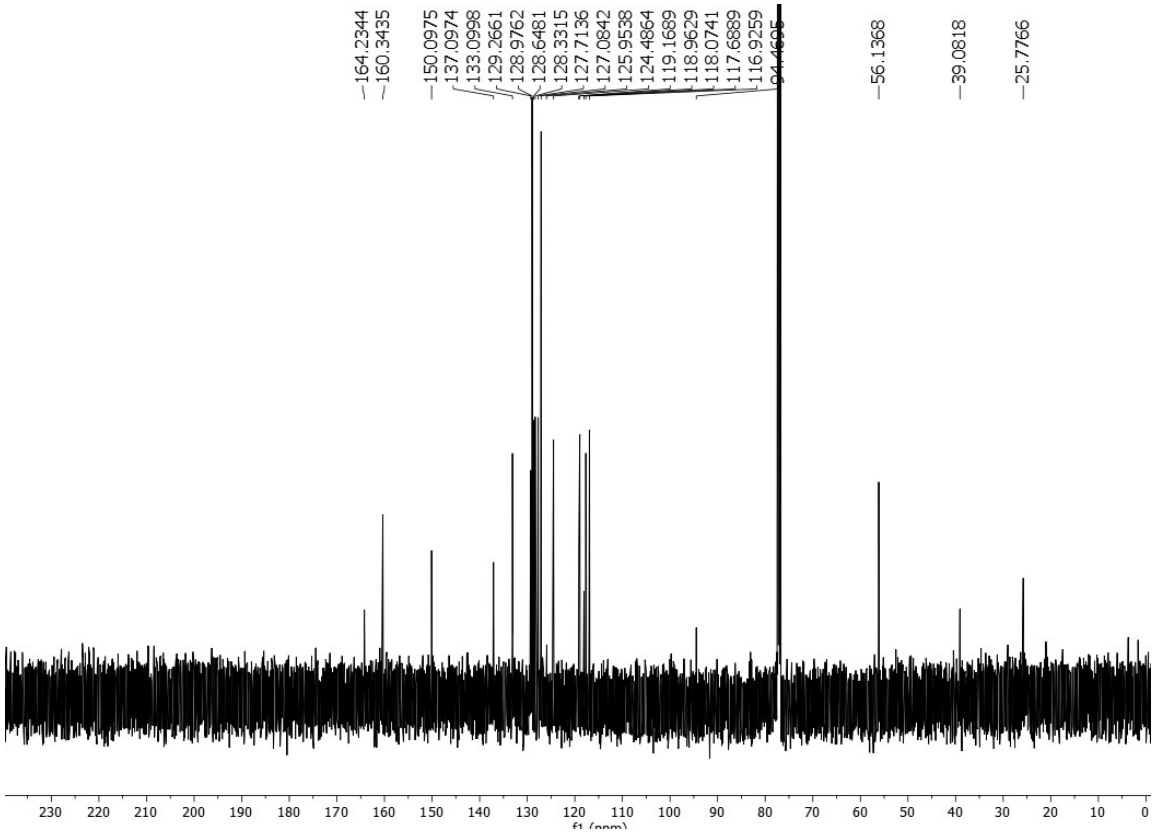
FTIR spectrum

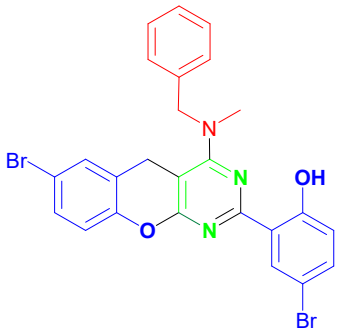


Mass spectrum



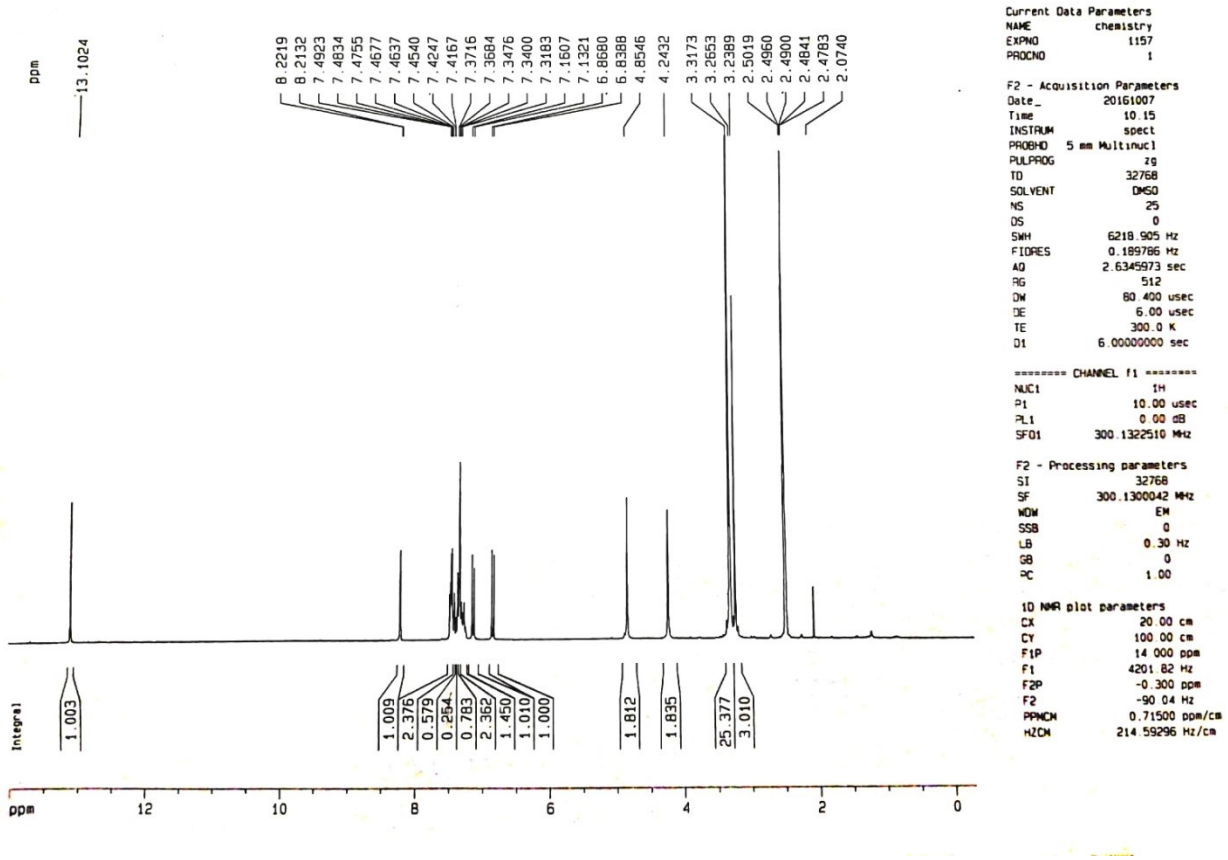
^{13}C NMR spectrum



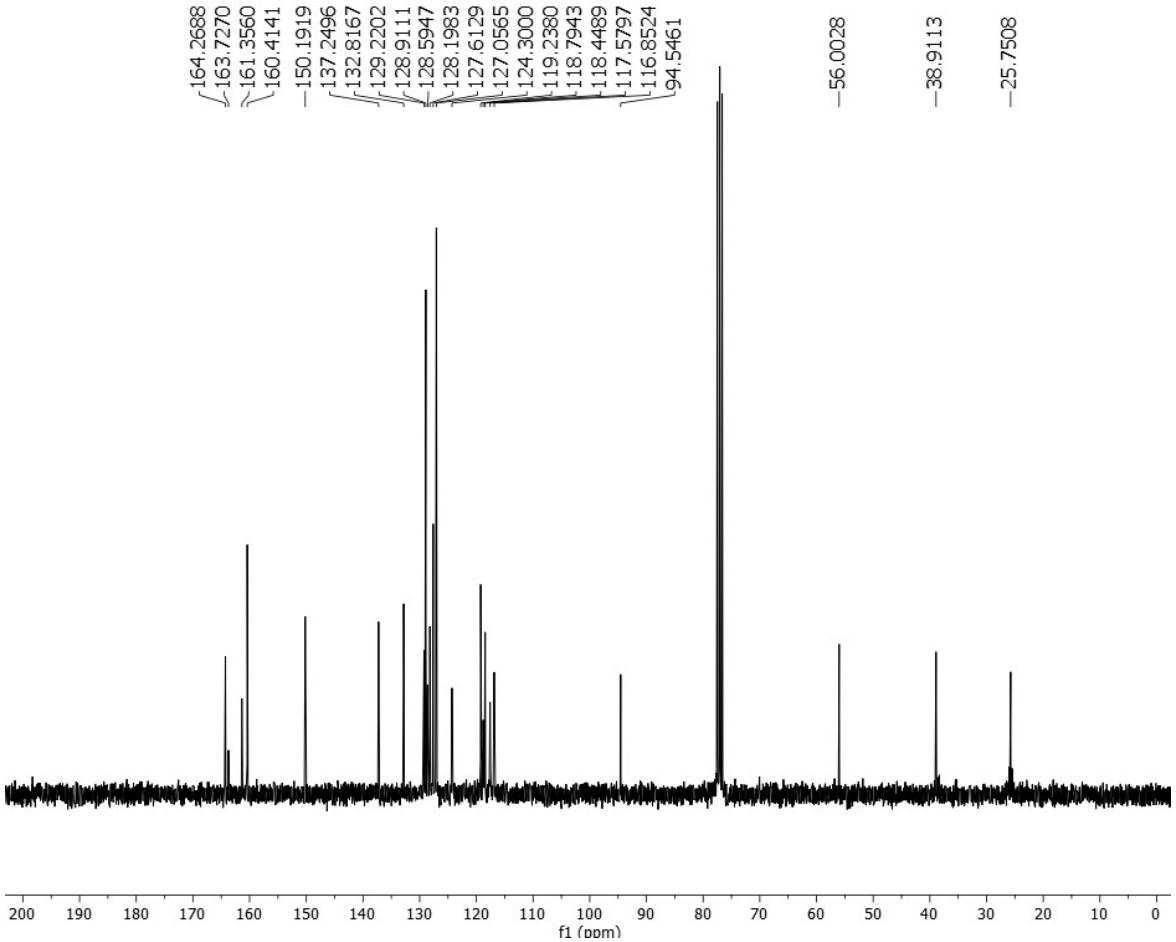


2-(4-(Benzyl(methyl)amino)-7-bromo-5*H*-benzopyrano[2,3-d]pyrimidin-2-yl)-4-bromophenol (1l)

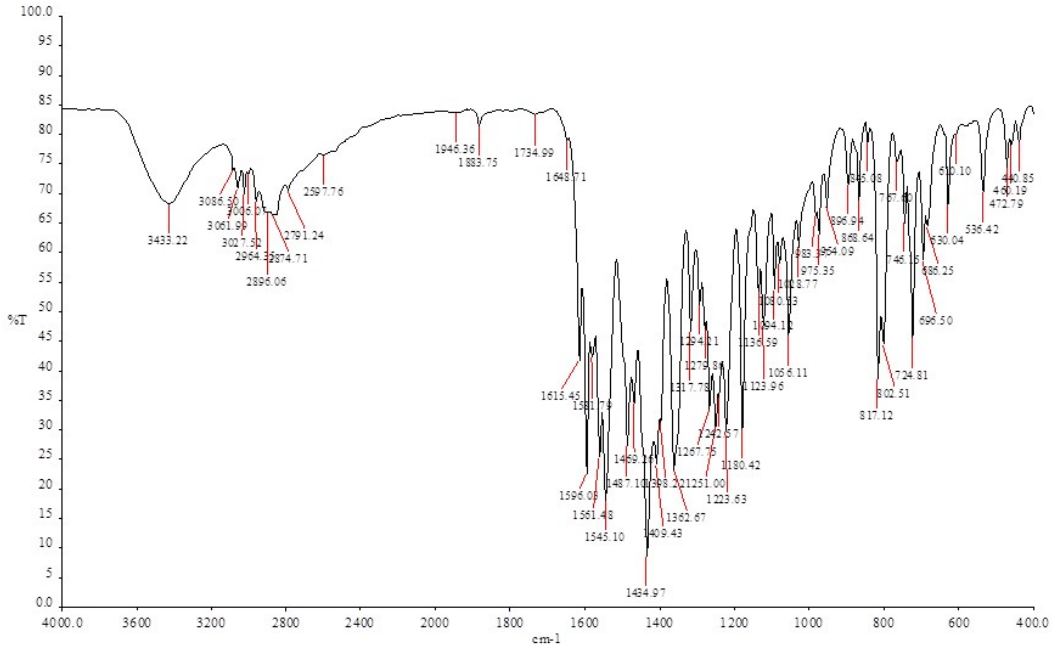
^{1}H NMR spectrum

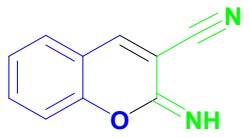


^{13}C NMR spectrum



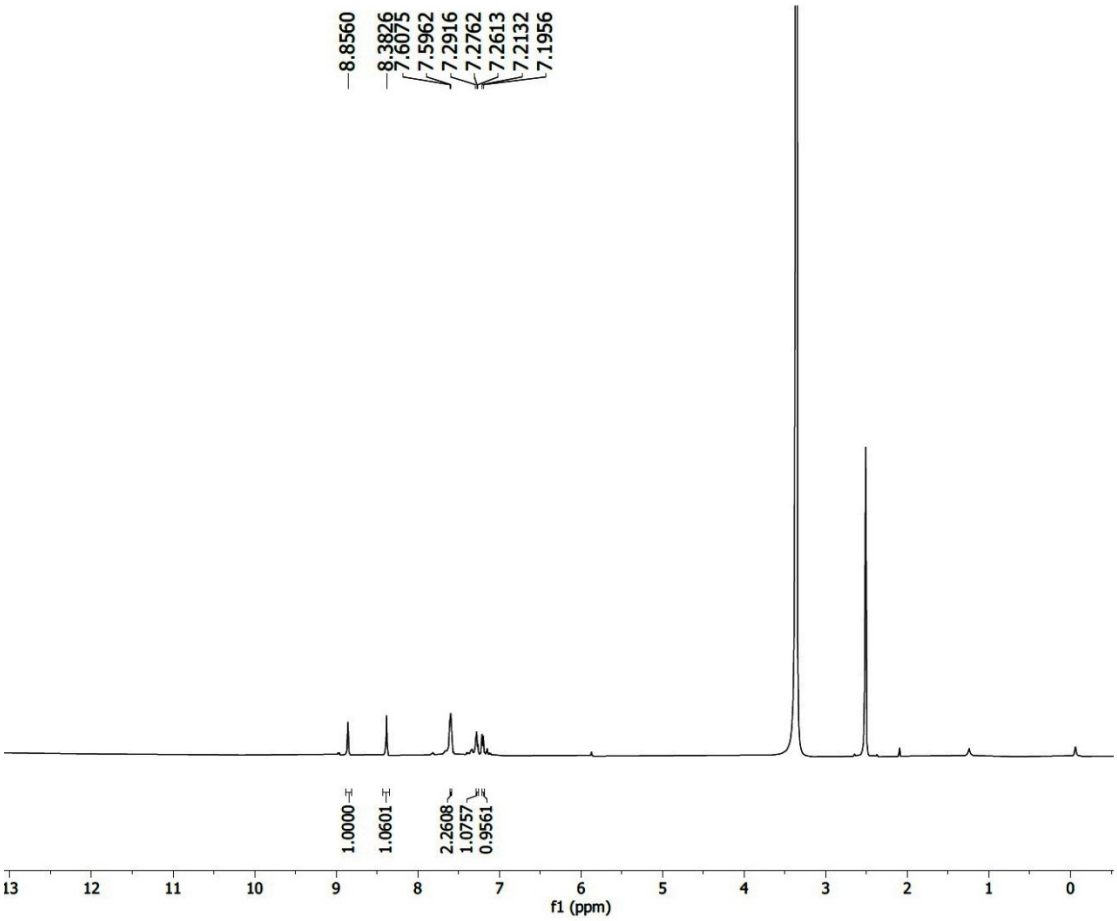
FTIR spectrum



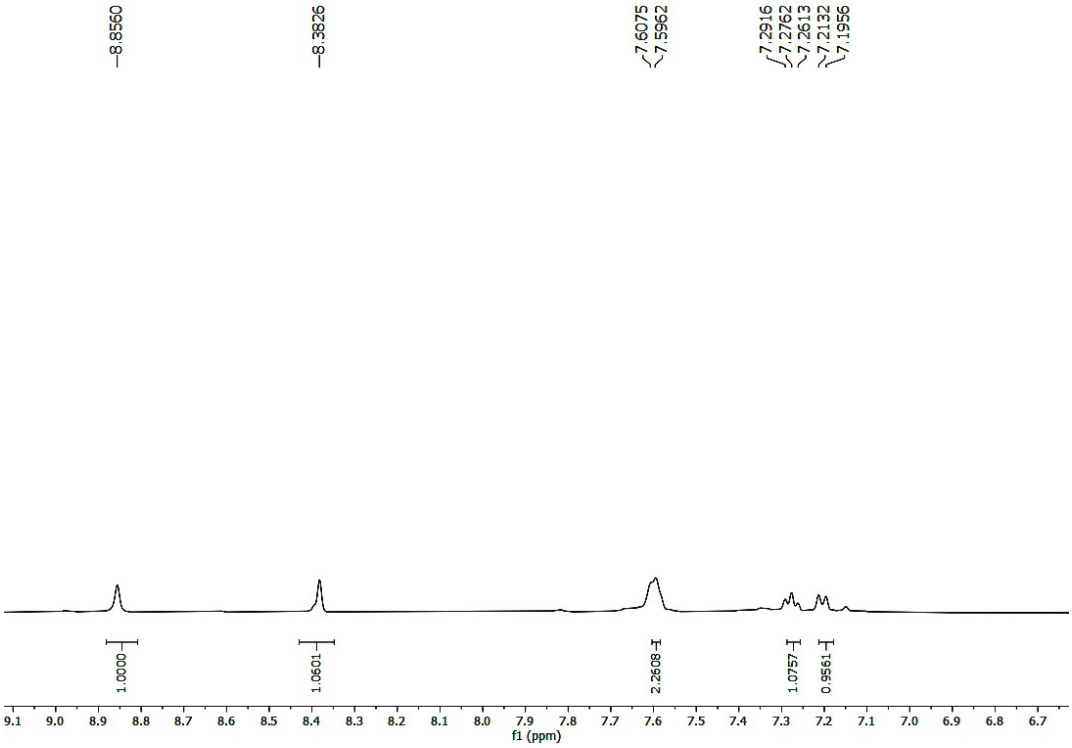


2-Imino-2*H*-benzopyran-3-carbonitrile (2a)

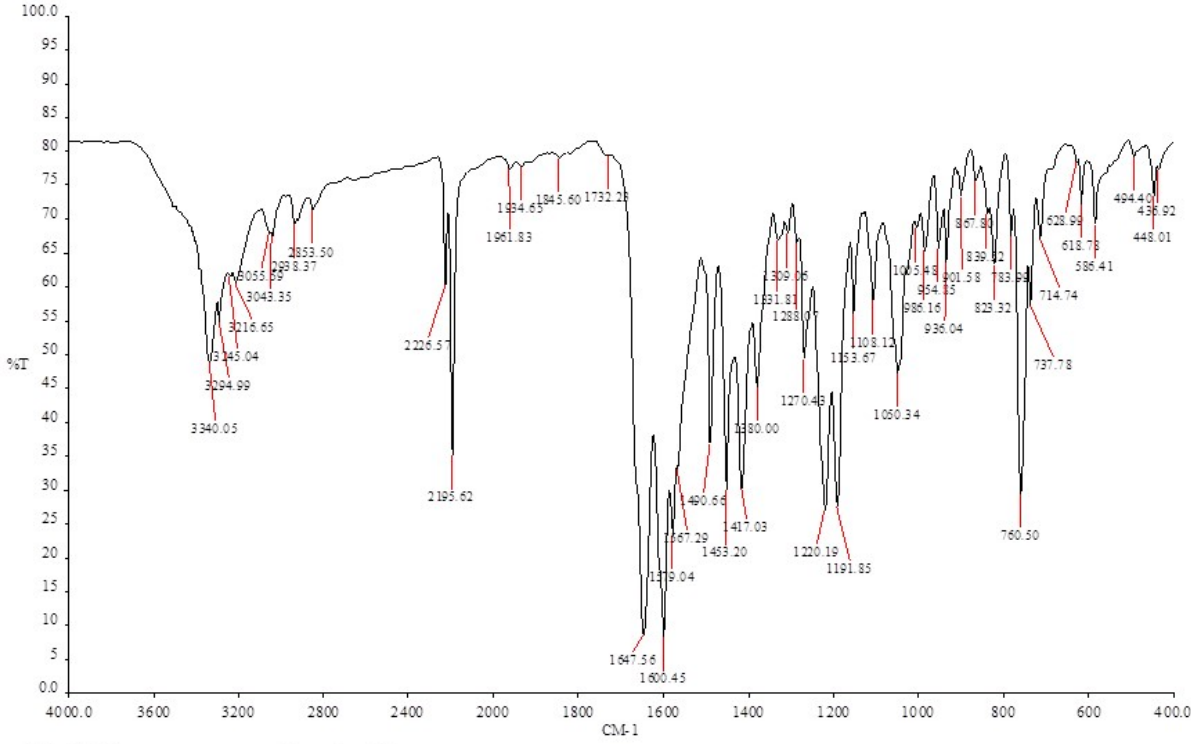
1H NMR spectrum



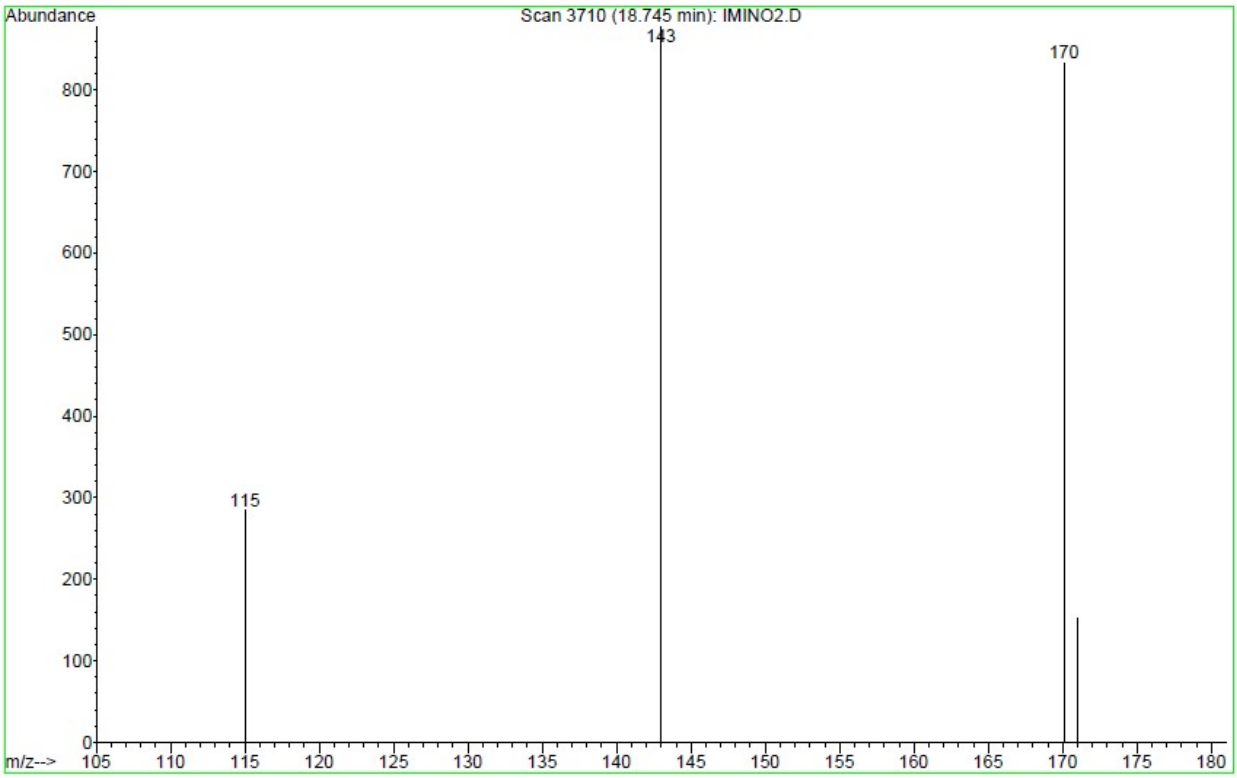
Expanded ^{1}H NMR spectrum

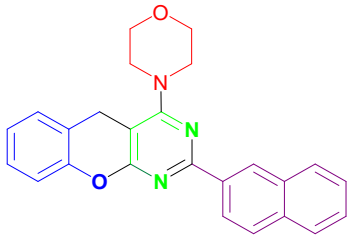


FTIR spectrum



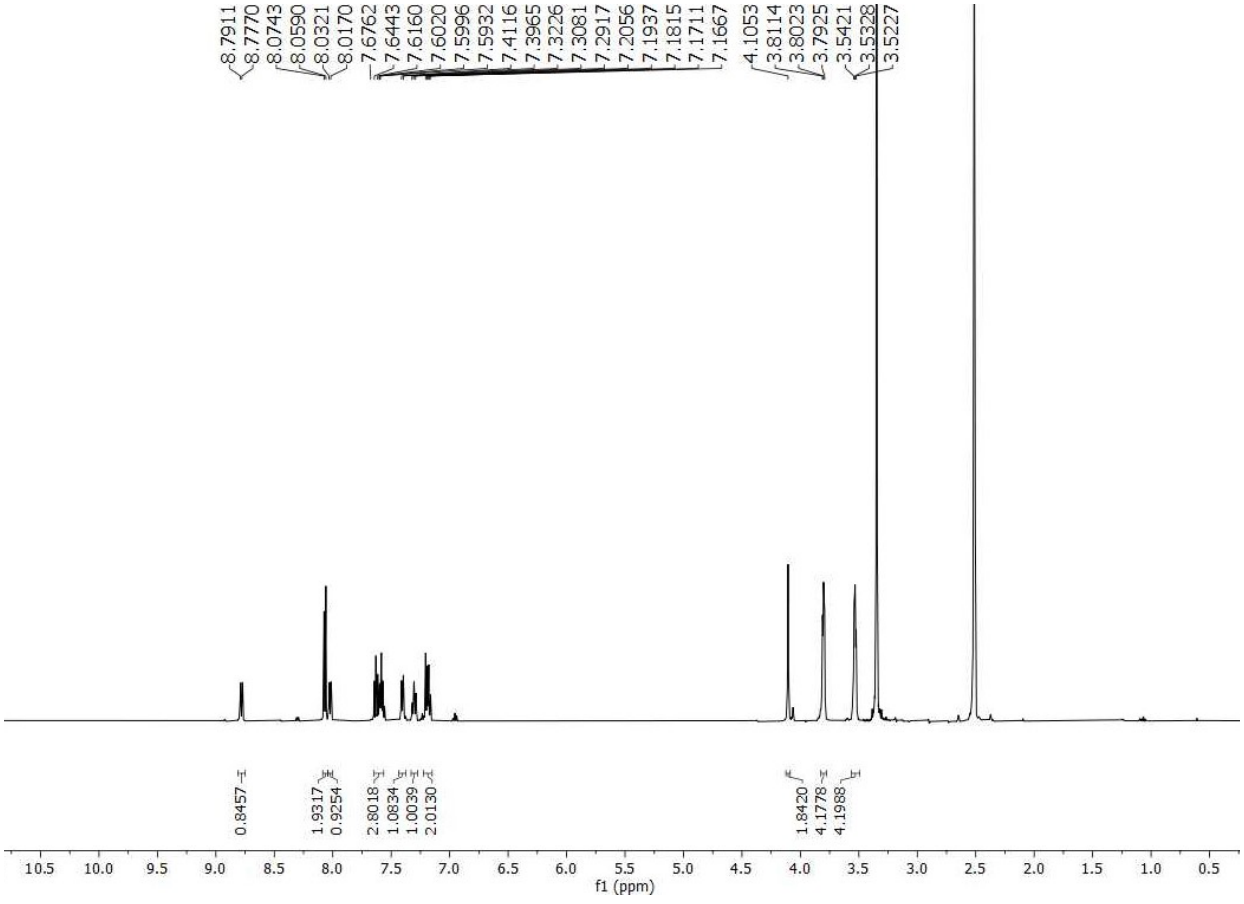
Mass spectrum



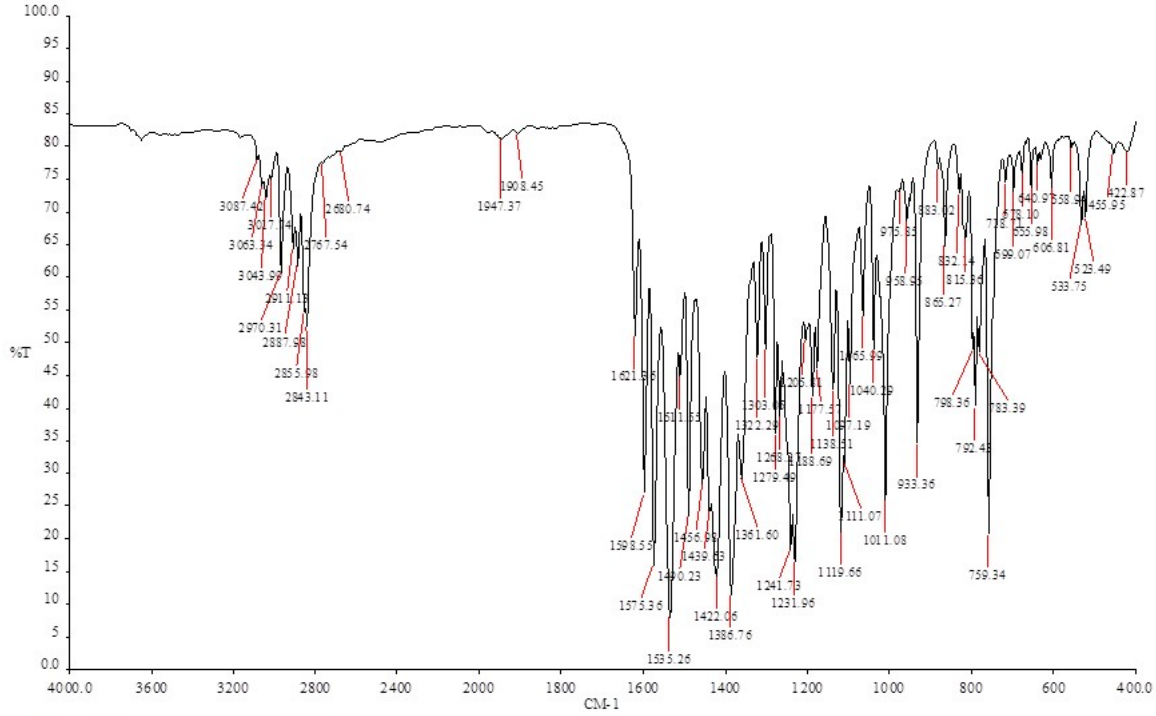


4-Morpholino-2-(naphthalen-2-yl)-5*H*-benzopyrano[2,3-d]pyrimidine (3a)

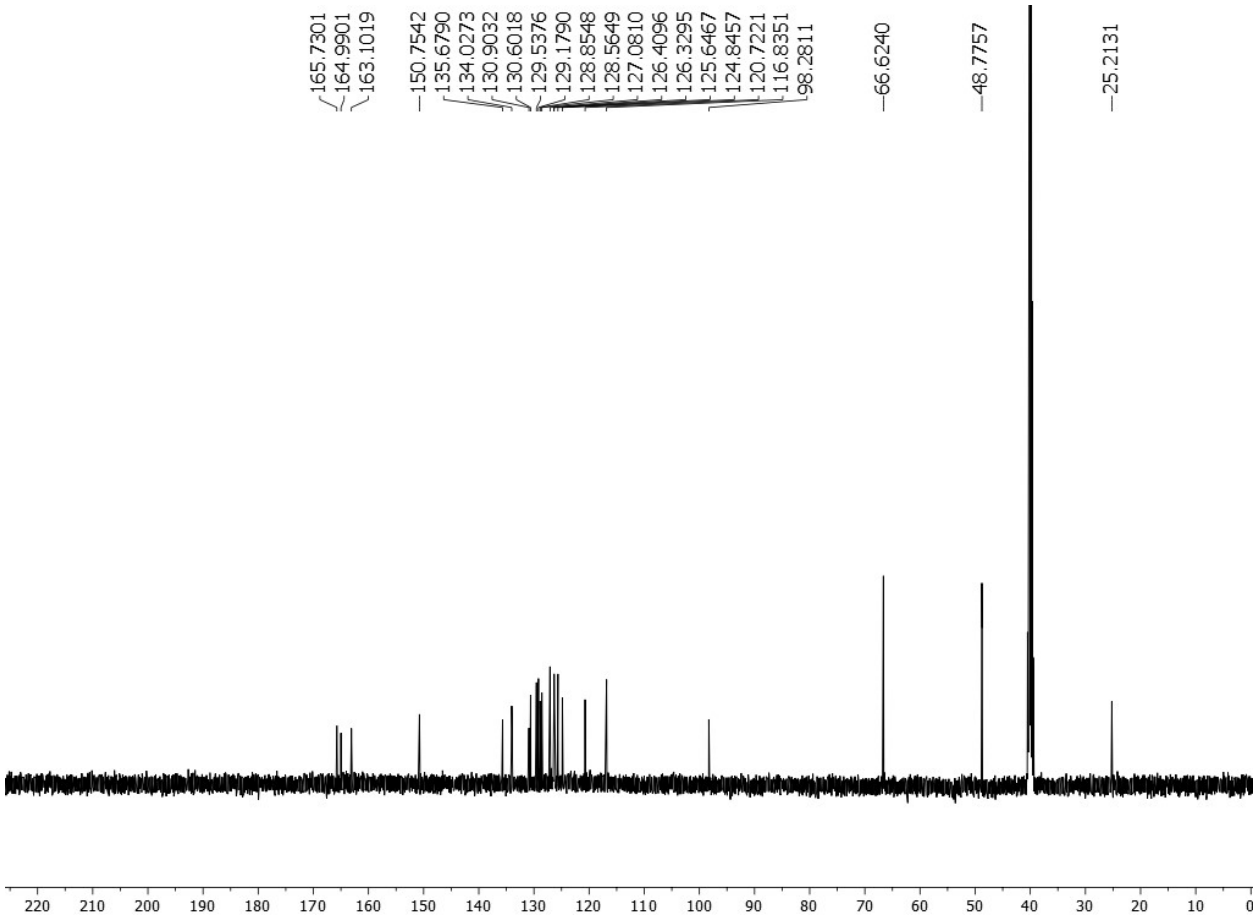
^{1}H NMR spectrum



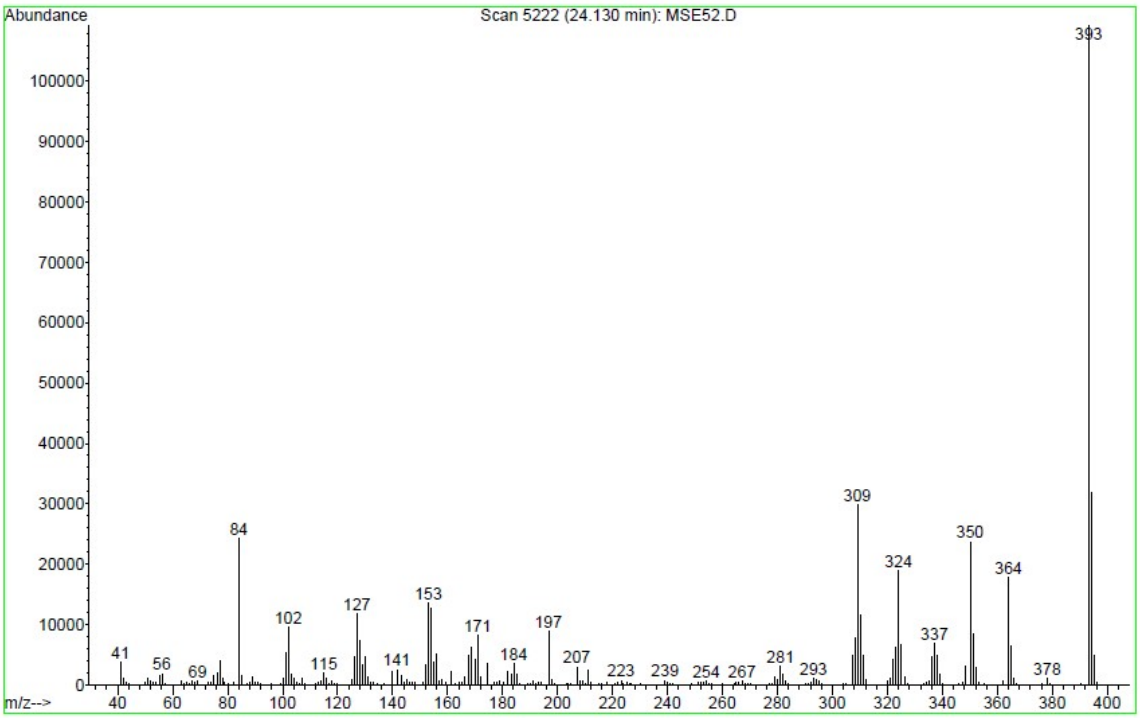
FTIR spectrum

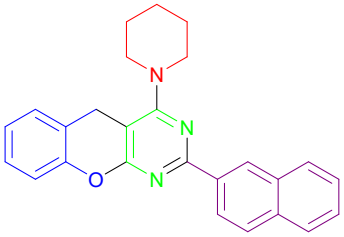


^{13}C NMR spectrum



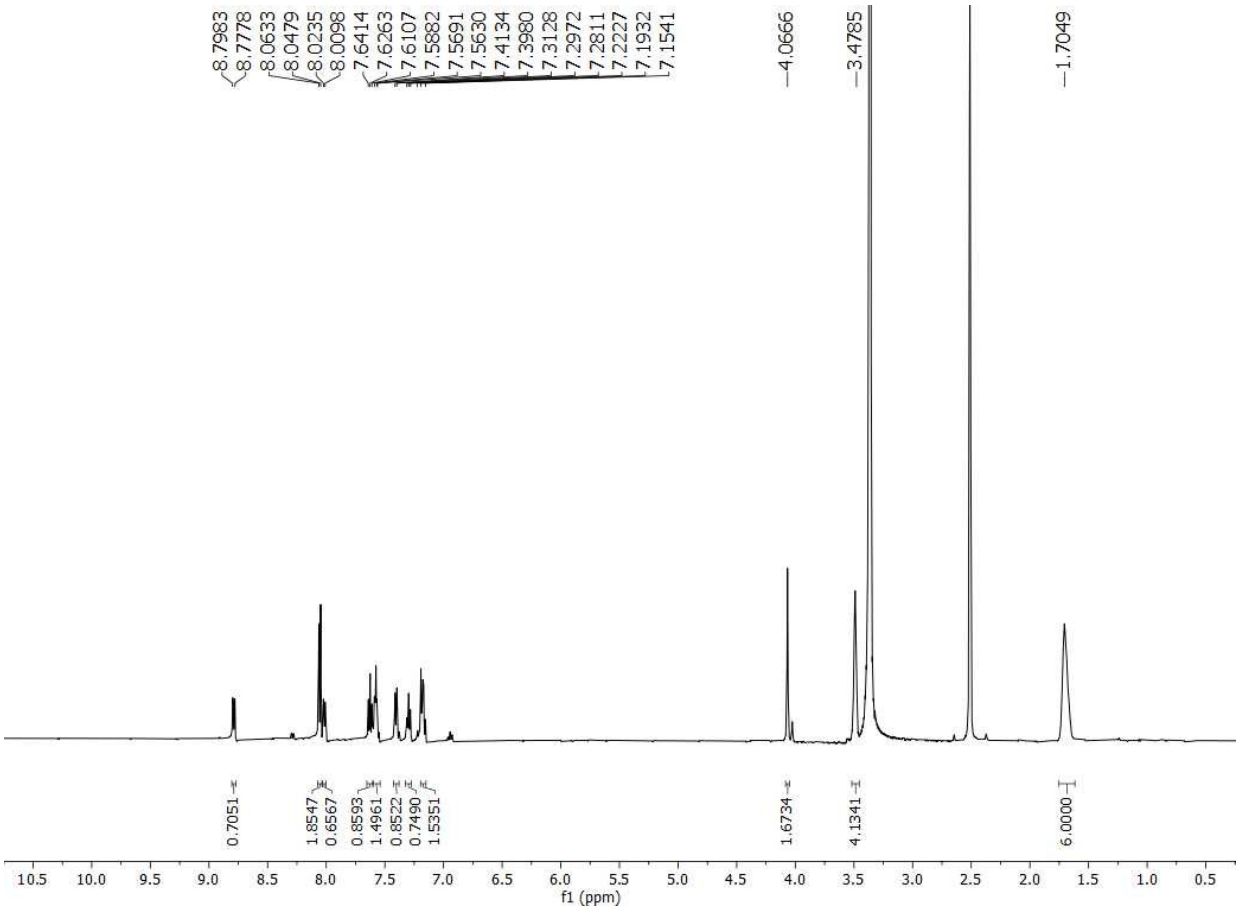
Mass spectrum



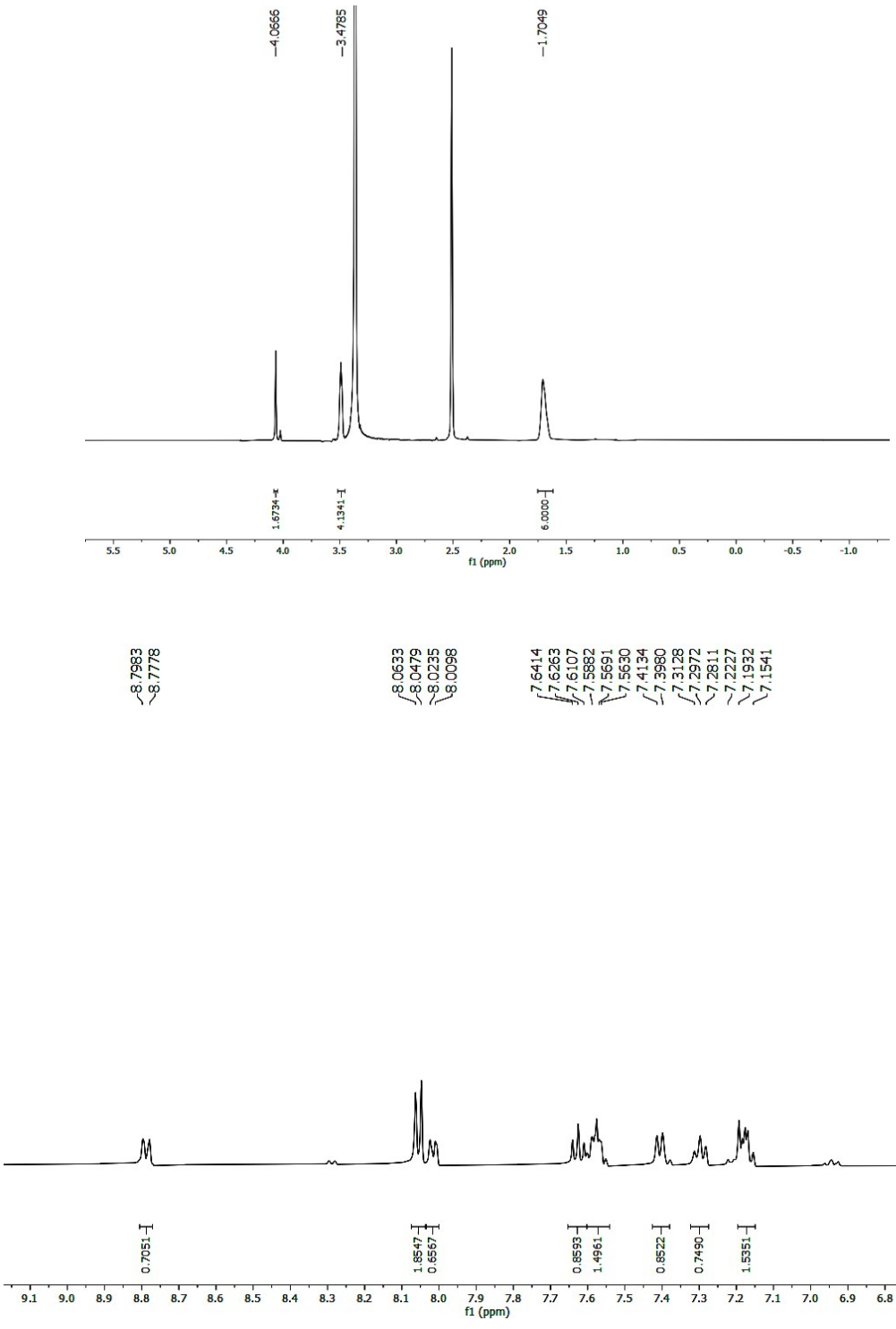


2-(Naphthalen-2-yl)-4-(piperidin-1-yl)-5*H*-benzopyrano[2,3-d]pyrimidine (3b)

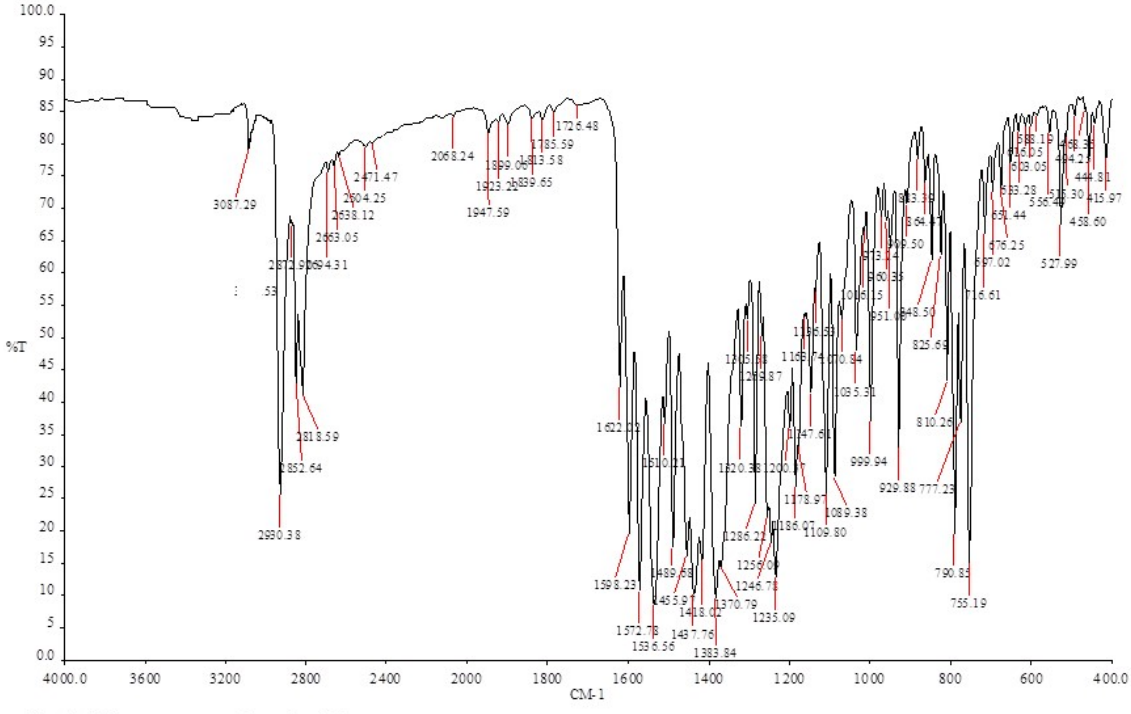
^{1}H NMR spectrum



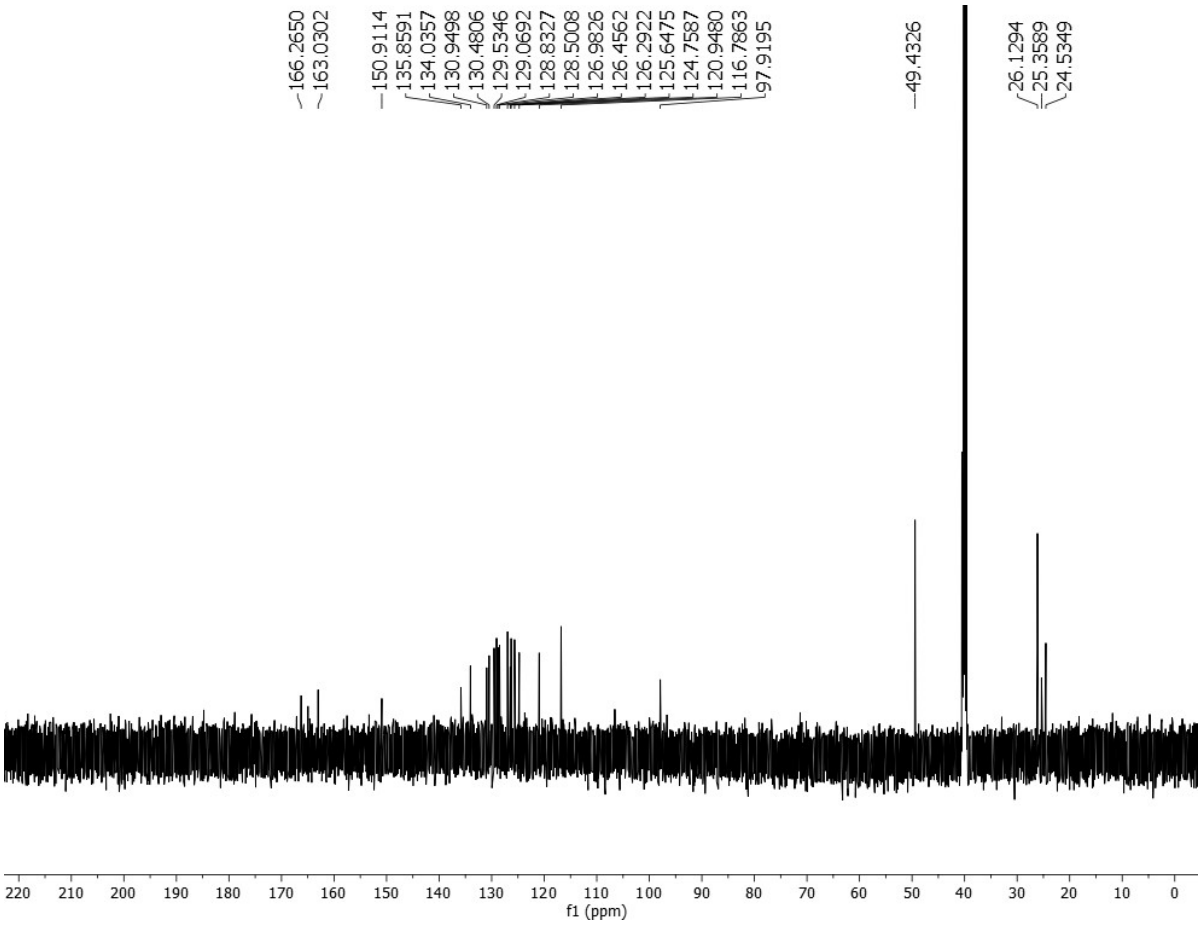
Expanded ^{1}H NMR spectrum



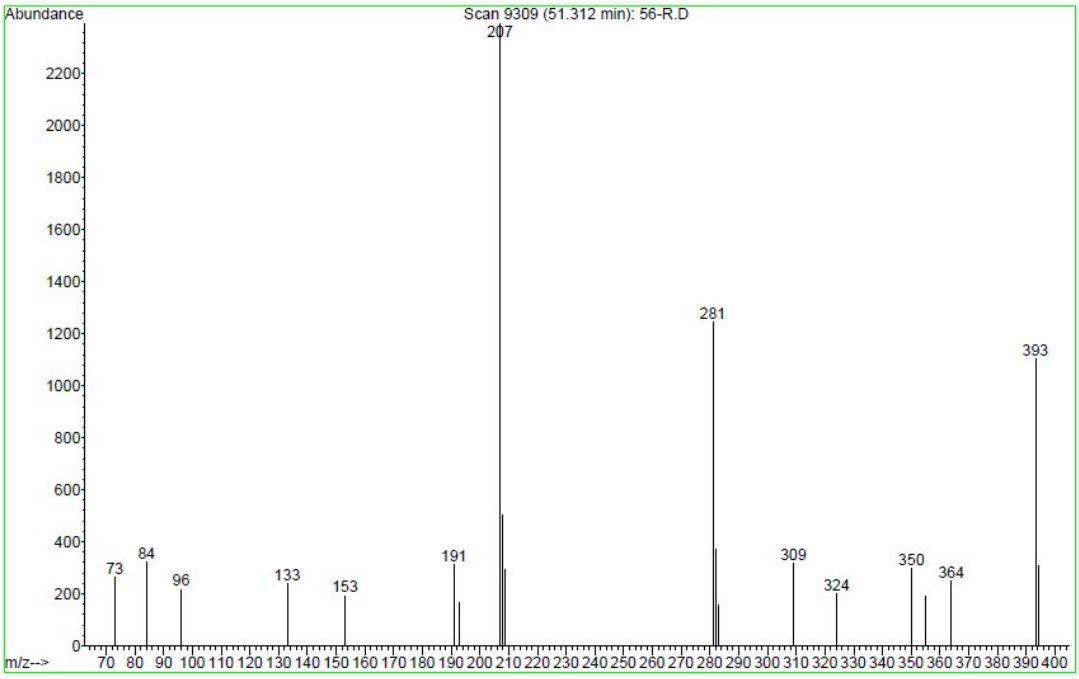
FTIR spectrum

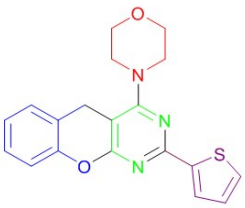


^{13}C NMR spectrum



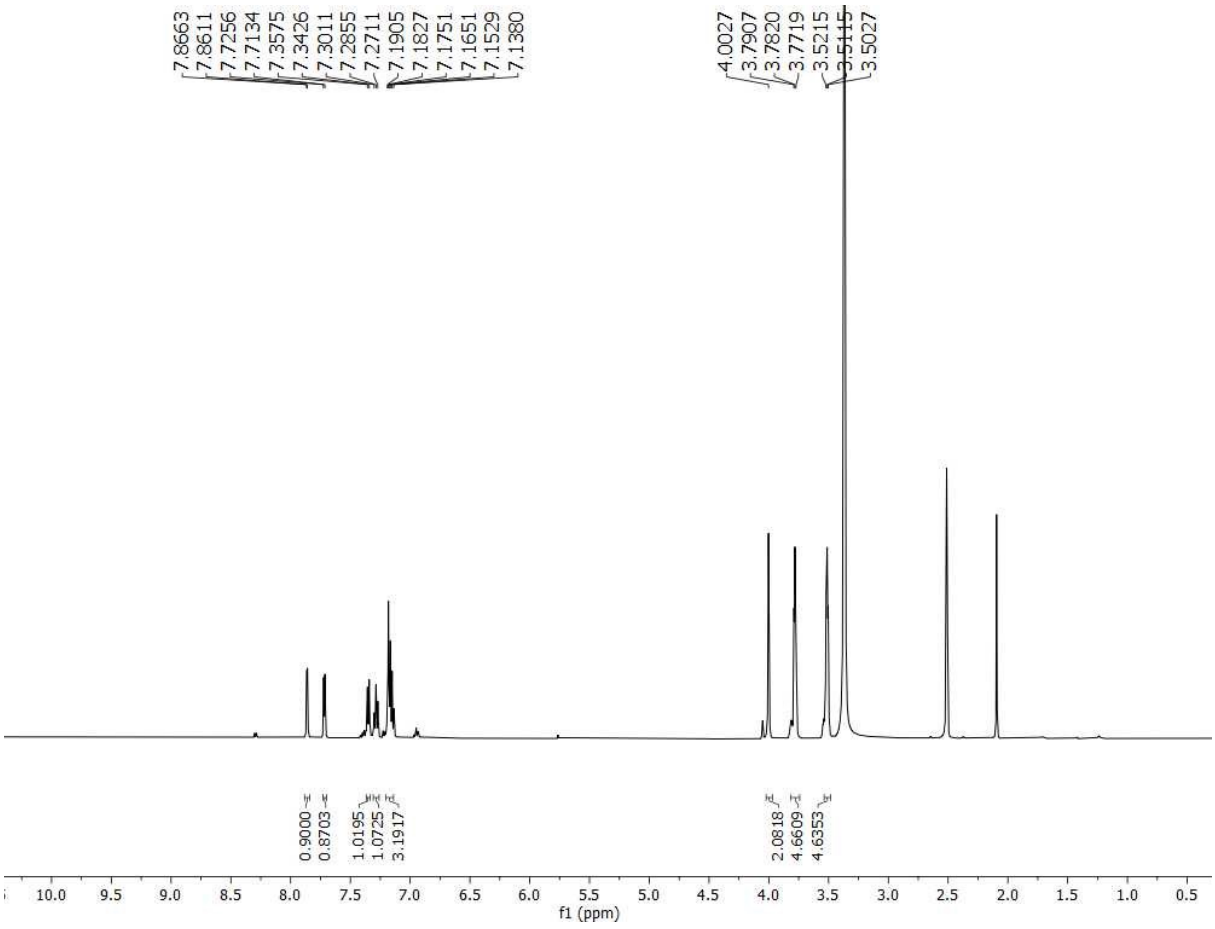
Mass spectrum



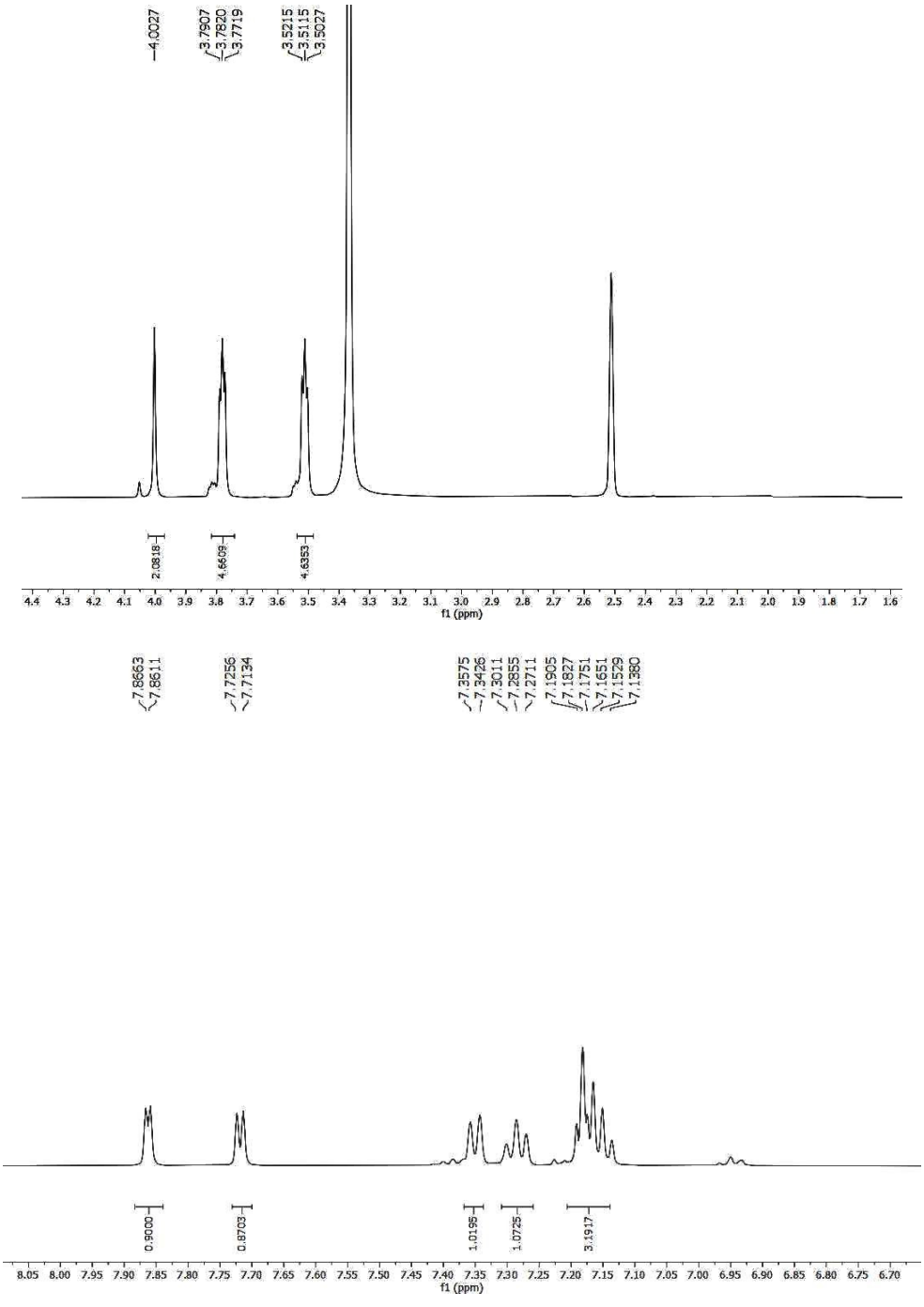


4-Morpholino-2-(thiophen-2-yl)-5*H*- benzopyrano[2,3-d]pyrimidine (3c)

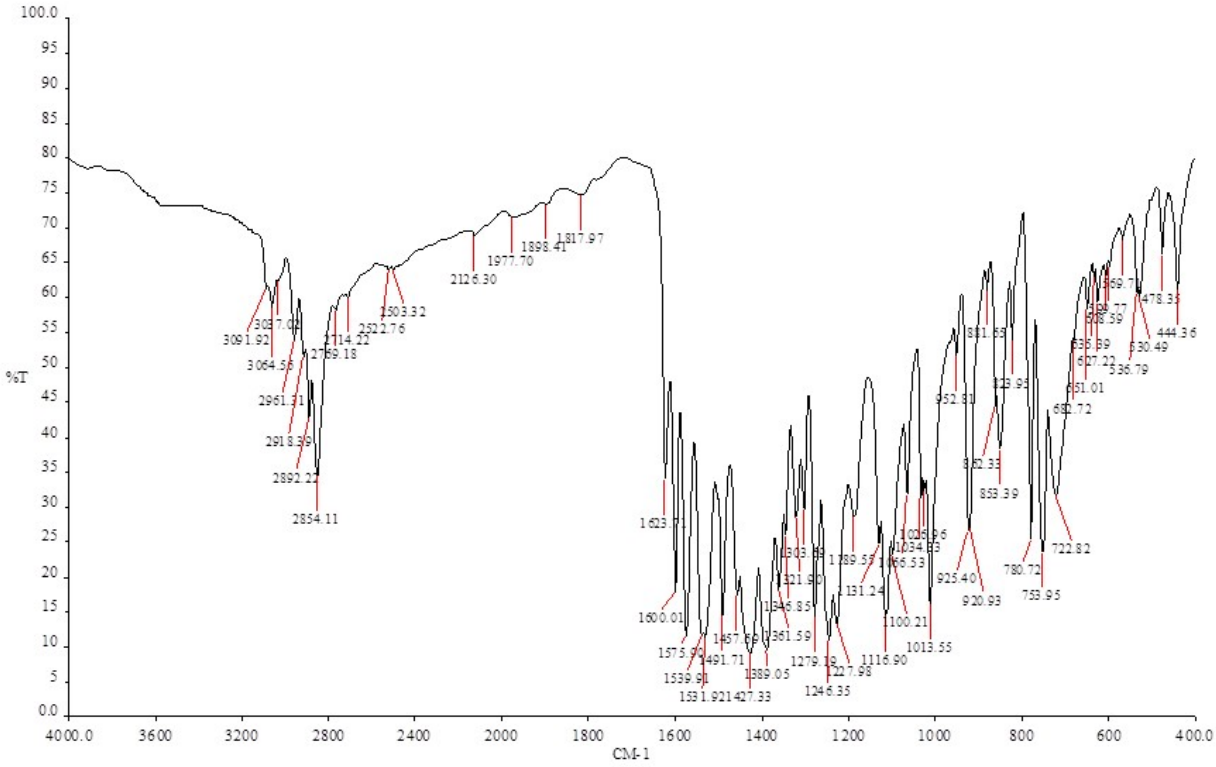
^{1}H NMR spectrum



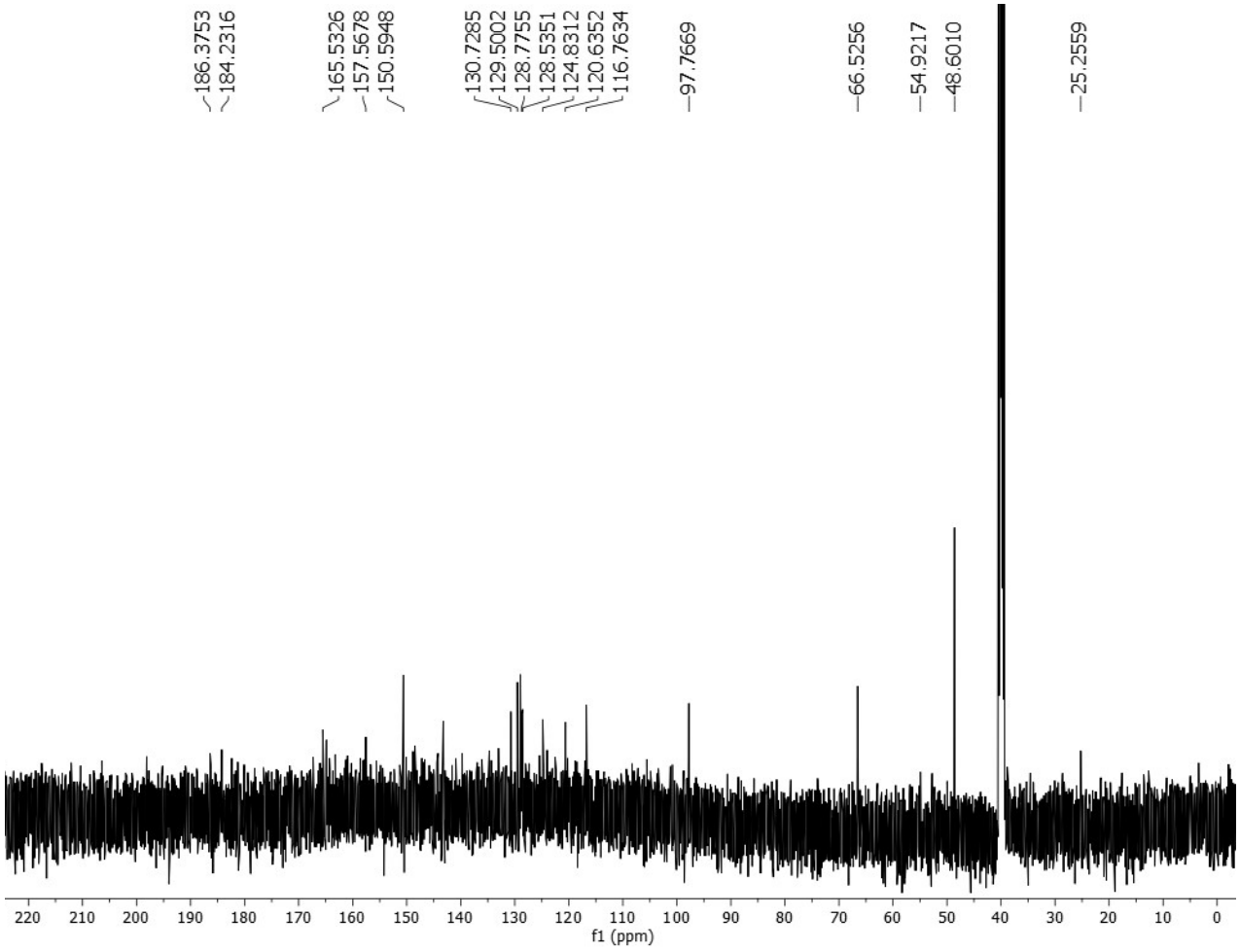
Expanded ^{1}H NMR spectrum



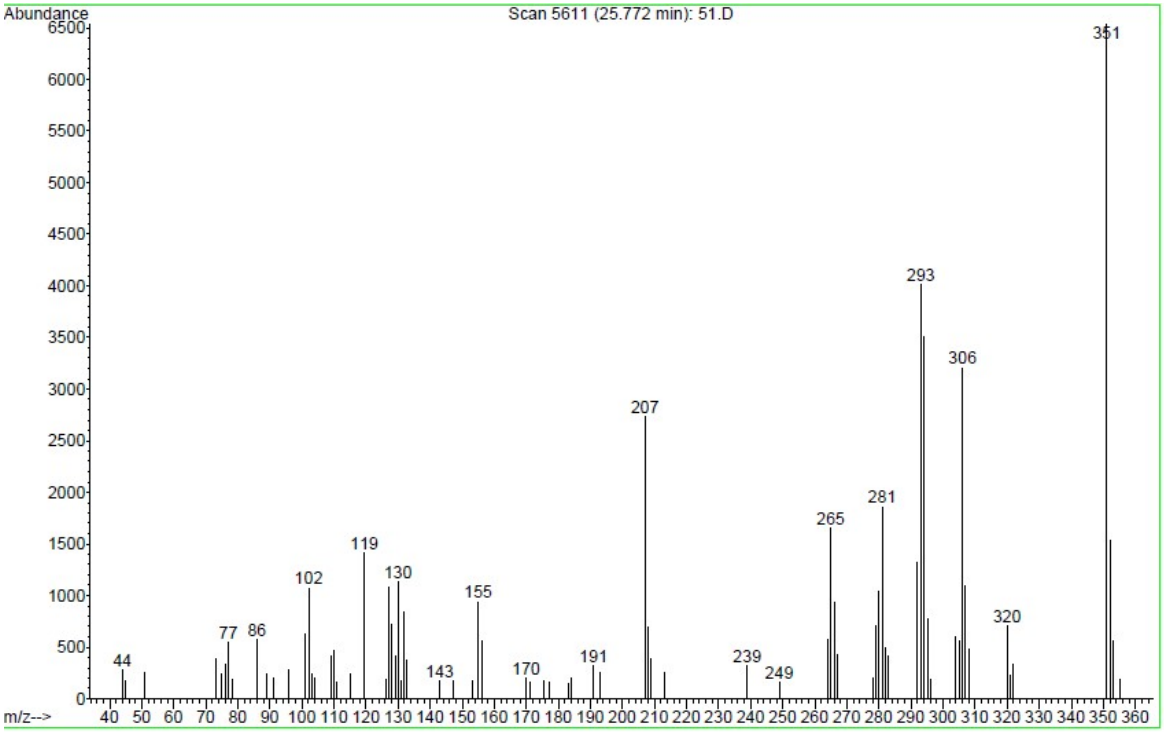
FTIR spectrum

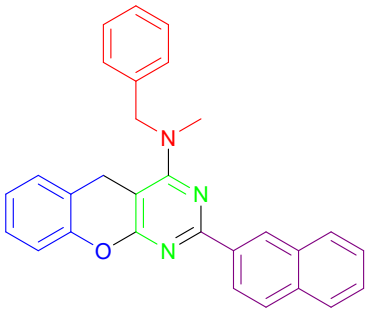


^{13}C NMR spectrum



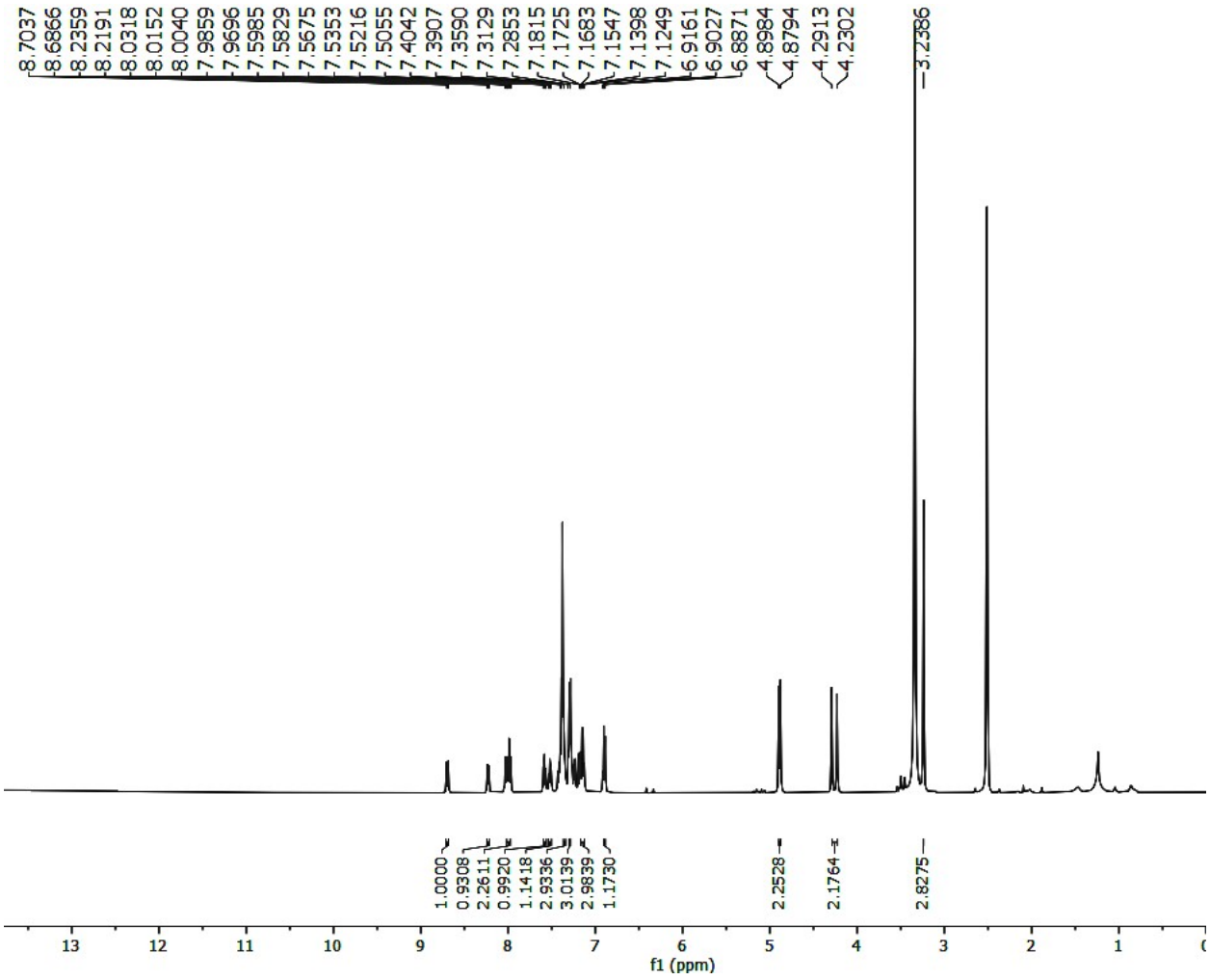
Mass spectrum



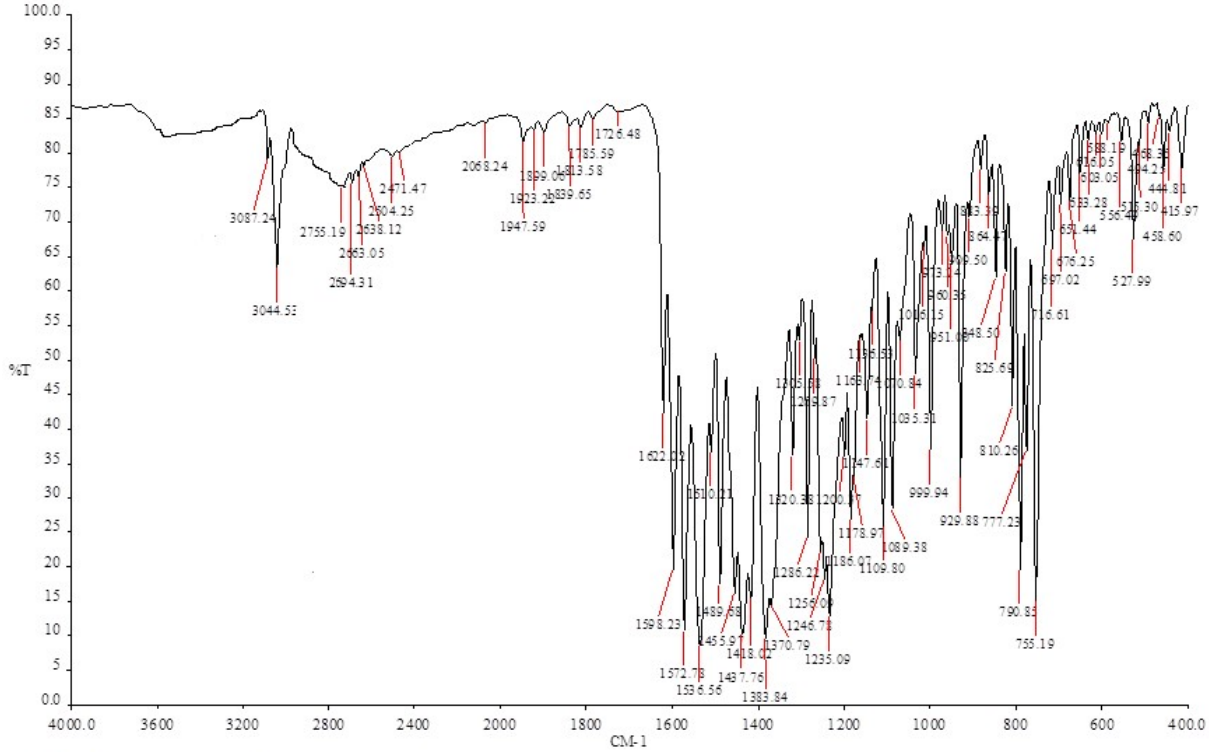


N-Benzyl-N-methyl-2-(naphthalen-2-yl)-5*H*-benzopyrano[2,3-d]pyrimidin-4-amine (3d)

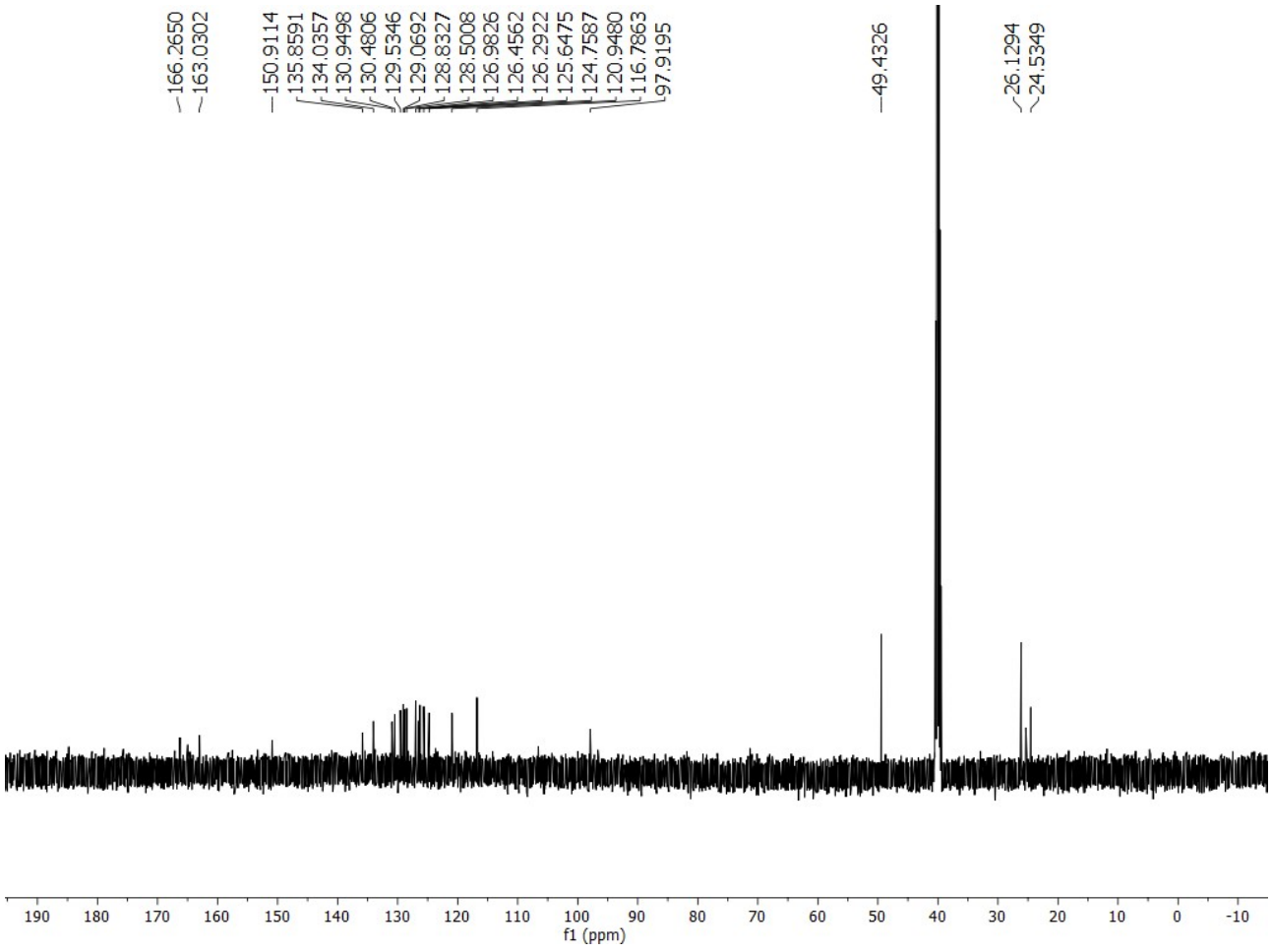
^{1}H NMR spectrum



FTIR spectrum



^{13}C NMR spectrum



Mass spectrum