Nano-kaolin/Ti^{4+}/Fe_3O_4: a magnetic reusable nano-catalyst for the synthesis of pyrimido[2,1-*b*]benzothiazoles

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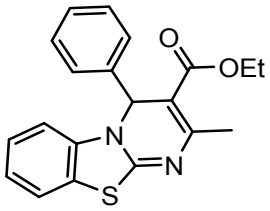
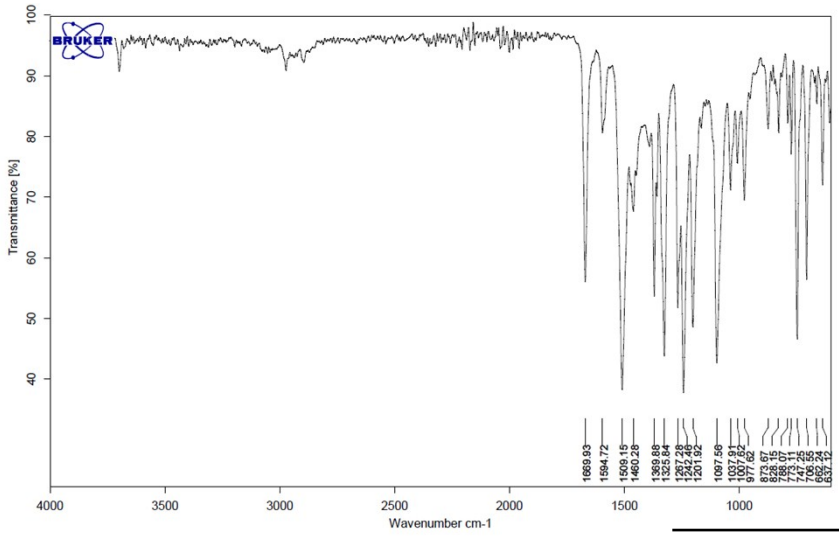
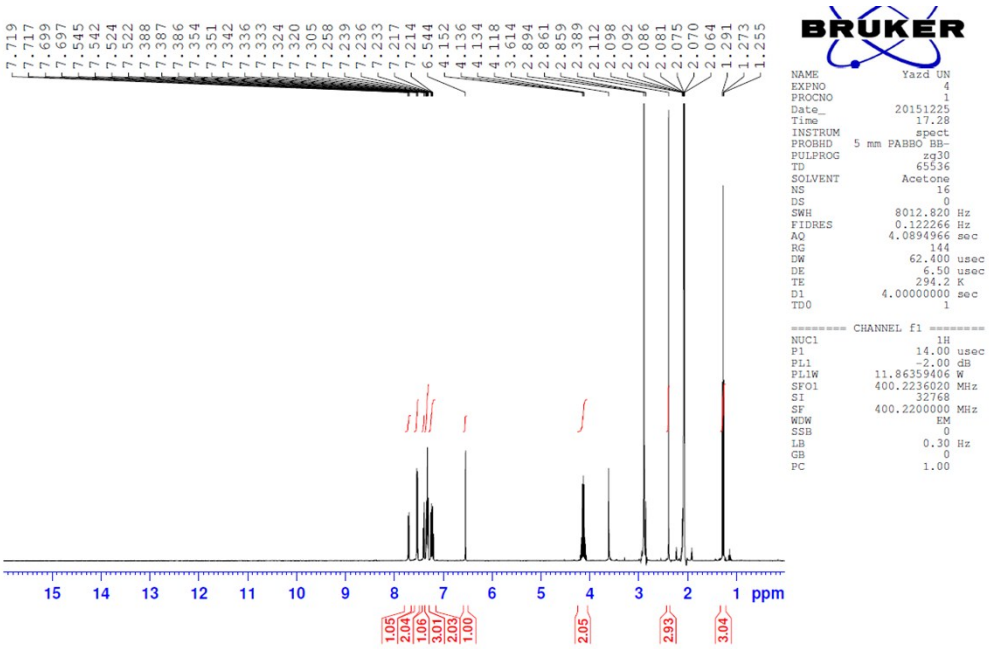
Ethyl-2-methyl-4-(phenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate (table 2, IV_a).



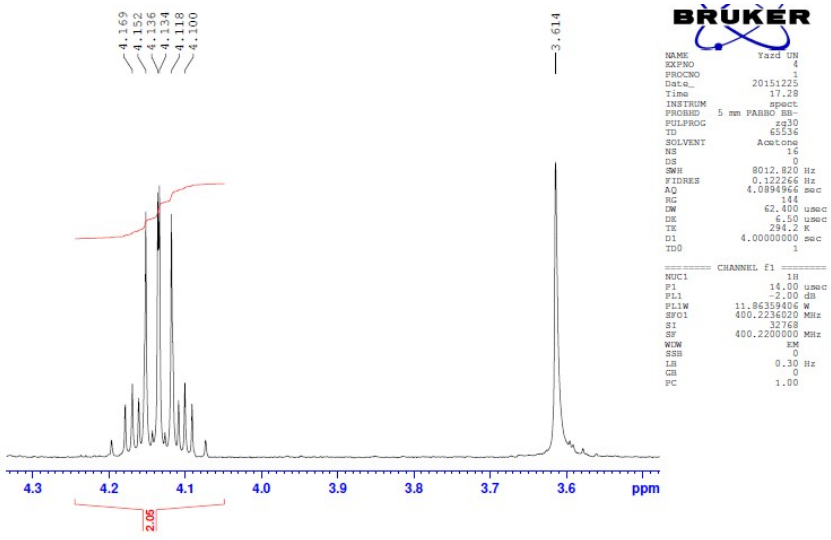
Pale yellow solid. ^{1}H NMR (Acetone-d_6, 400 MHz): δ 7.70 (dd, *J*=8, 0.8 Hz, 1H), 7.52-7.54 (m, 2H), 7.40 (dd, *J*=8, 1.2 Hz, 1H), 7.30-7.35 (m, 3H), 7.19-7.26 (m, 2H), 6.54 (s, 1H), 4.07-4.20 (m, 2H), 2.38 (s, 3H), 1.27 (t, 3H, J=7.2 Hz). ^{13}C NMR (DMSO-d_6, 400 MHz): 165.9, 163.1, 154.5, 142.0, 138.0, 129.0, 128.7, 127.4, 127.2, 124.5, 123.3, 112.7, 103.2, 59.9, 57.1, 23.6, 14.5, δ IR (KBr): 2974, 1669, 1594, 1460, 1242, 747 cm^{-1}. mp: 178-180 ˚C.



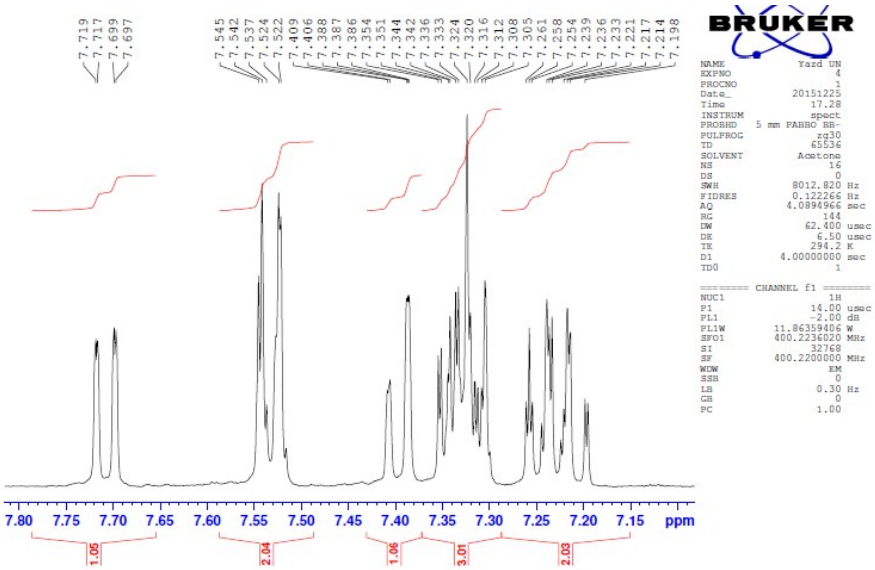
The FT-IR spectrum of product (IV_a)



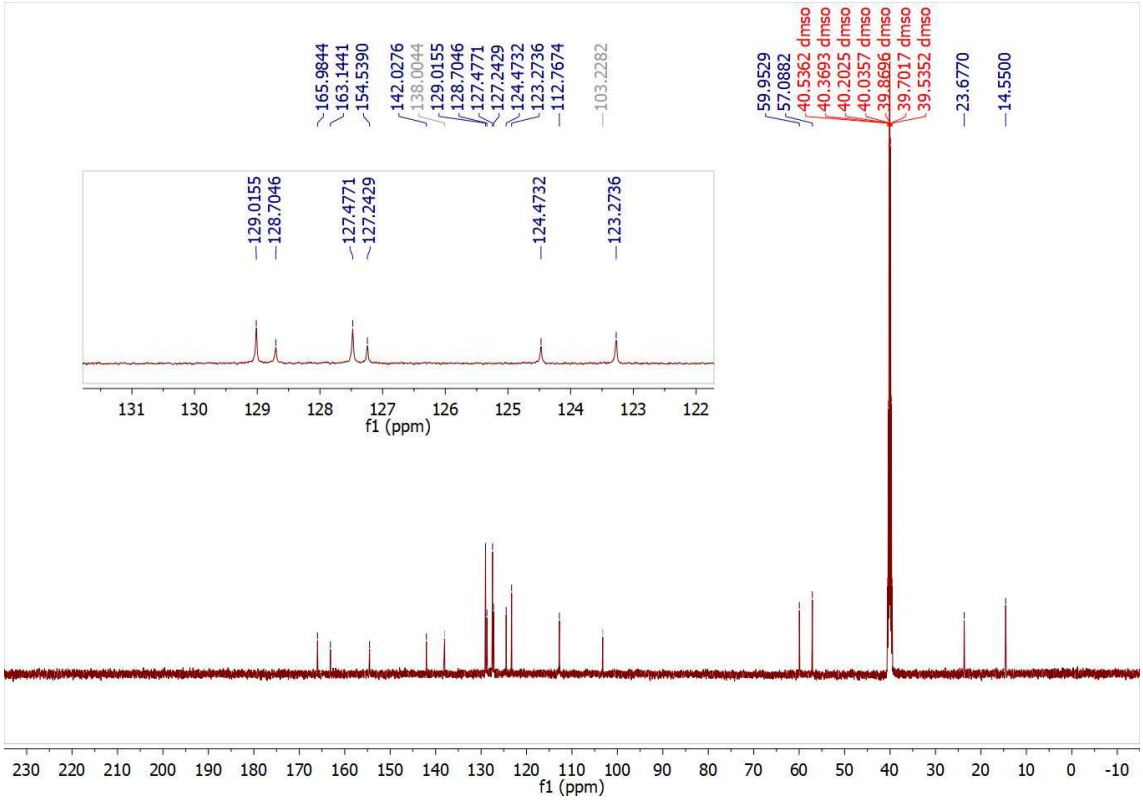
The ^{1}H NMR (400MHz) spectrum of product (IV_a)



The ^{1}H NMR (400MHz) spectrum of product (IV_a)



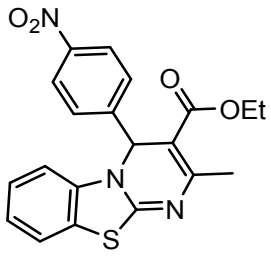
The ^{1}H NMR (400MHz) spectrum of product (IV_a)



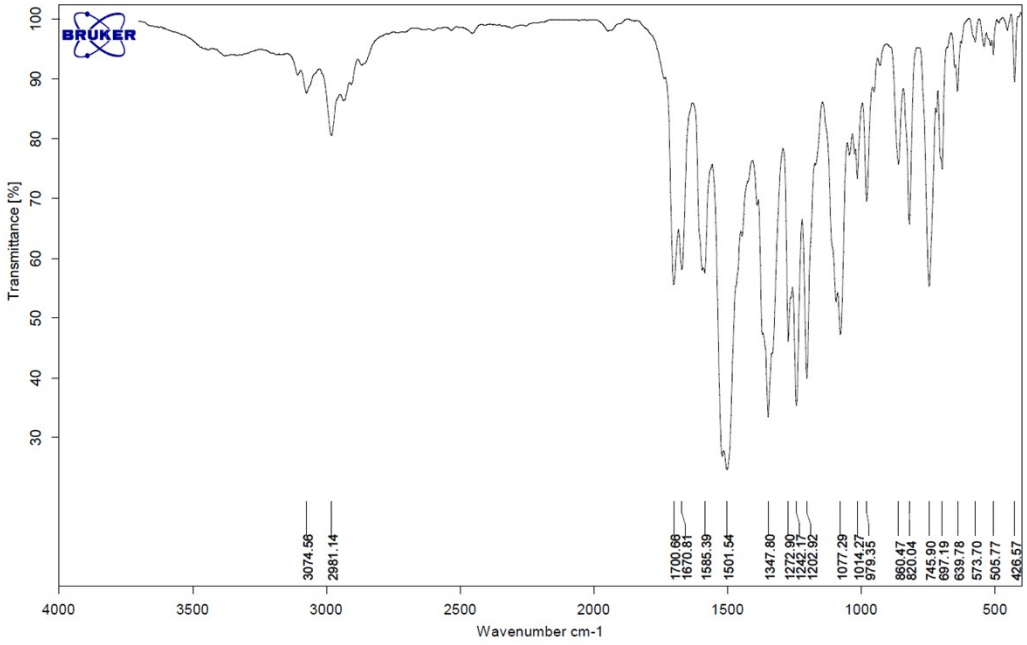
The ^{13}C NMR (400MHz) spectrum of product (IV_a)

Ethyl-2-methyl-4-(4-nitrophenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate

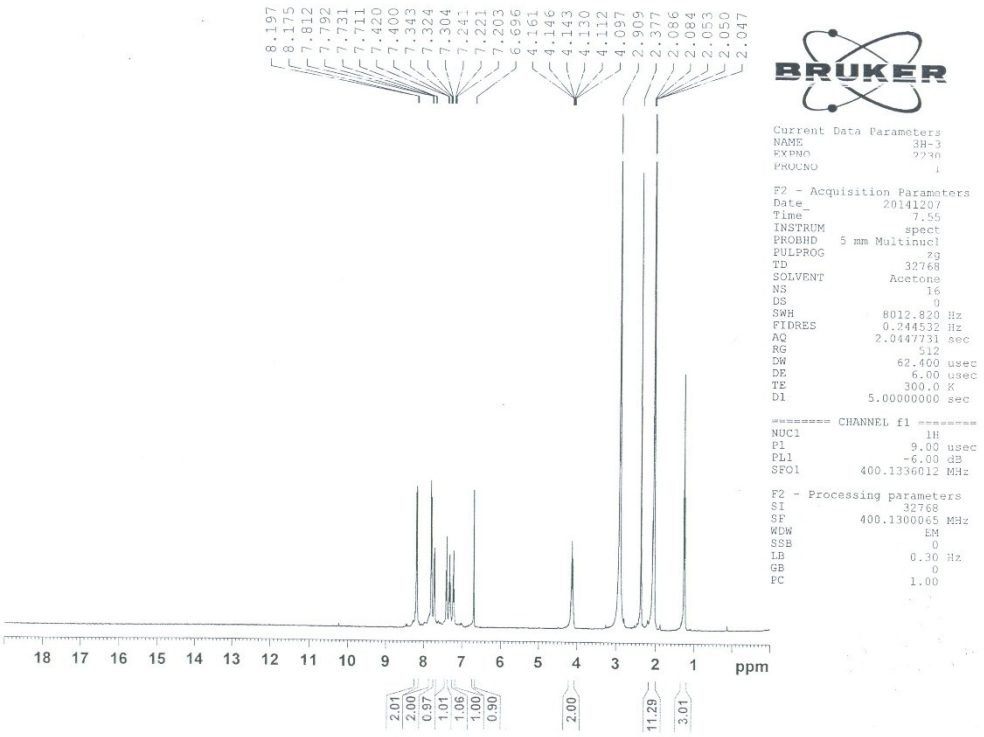
(table 2, IV_b).



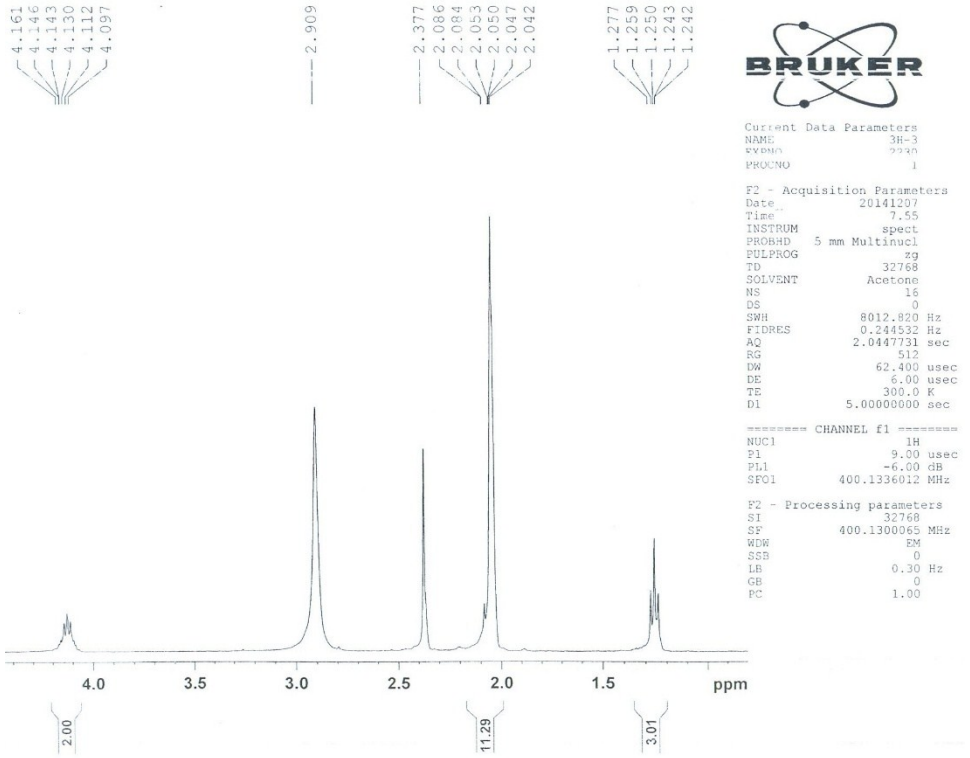
Yellow solid. ^{1}H NMR (Acetone-d_6, 400 MHz): δ 8.18 (d, *J*=8.8 Hz, 2H), 7.80 (d, *J*=8 Hz, 2H), 7.72 (d, *J*=8 Hz, 1H), 7.41 (d, *J*=8 Hz, 1H), 7.32 (t, *J*=8 Hz, 1H), 7.22 (t, *J*=7.2 Hz, 1H), 6.69 (s, 1H), 4.09-4.16 (m, 2H), 2.37 (s, 3H), 1.25 (t, *J*=7.2 Hz, 3H). ^{13}C NMR (DMSO-d_6, 400 MHz): δ 165.7, 163.4, 155.5, 151.7, 148.6, 137.7, 128.9, 127.4, 124.7, 124.3, 123.4, 123.2, 112.7, 102.3, 60.1, 56.4, 23.8, 14.5, IR (KBr): 3074, 2981, 1700, 1670, 1585, 1501, 1347, 1272, 1242, 1202, 745 cm^{-1}. mp: 171-173 ˚C.



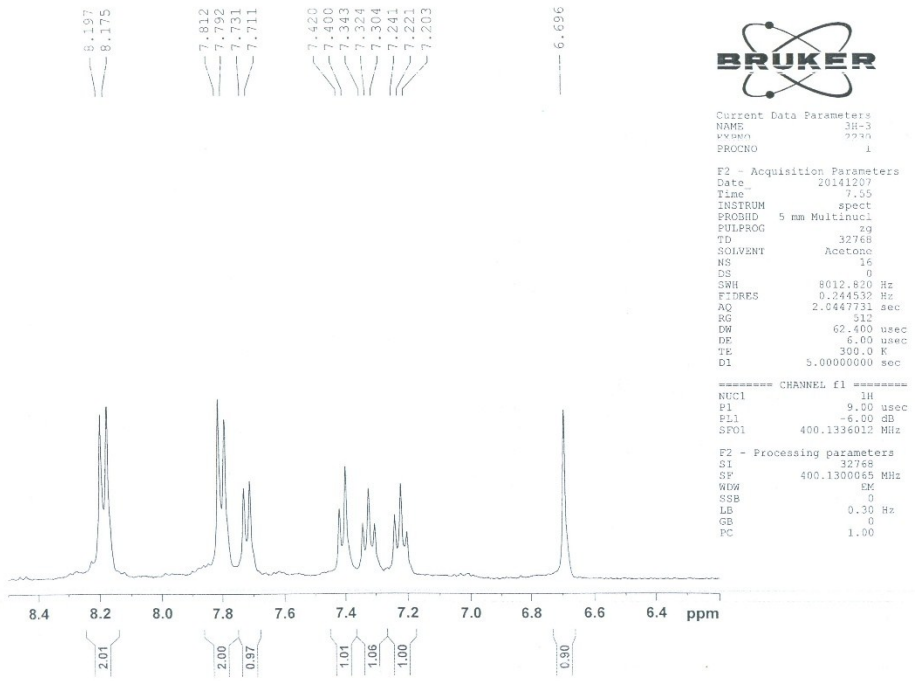
The FT-IR spectrum of product (IV_b)



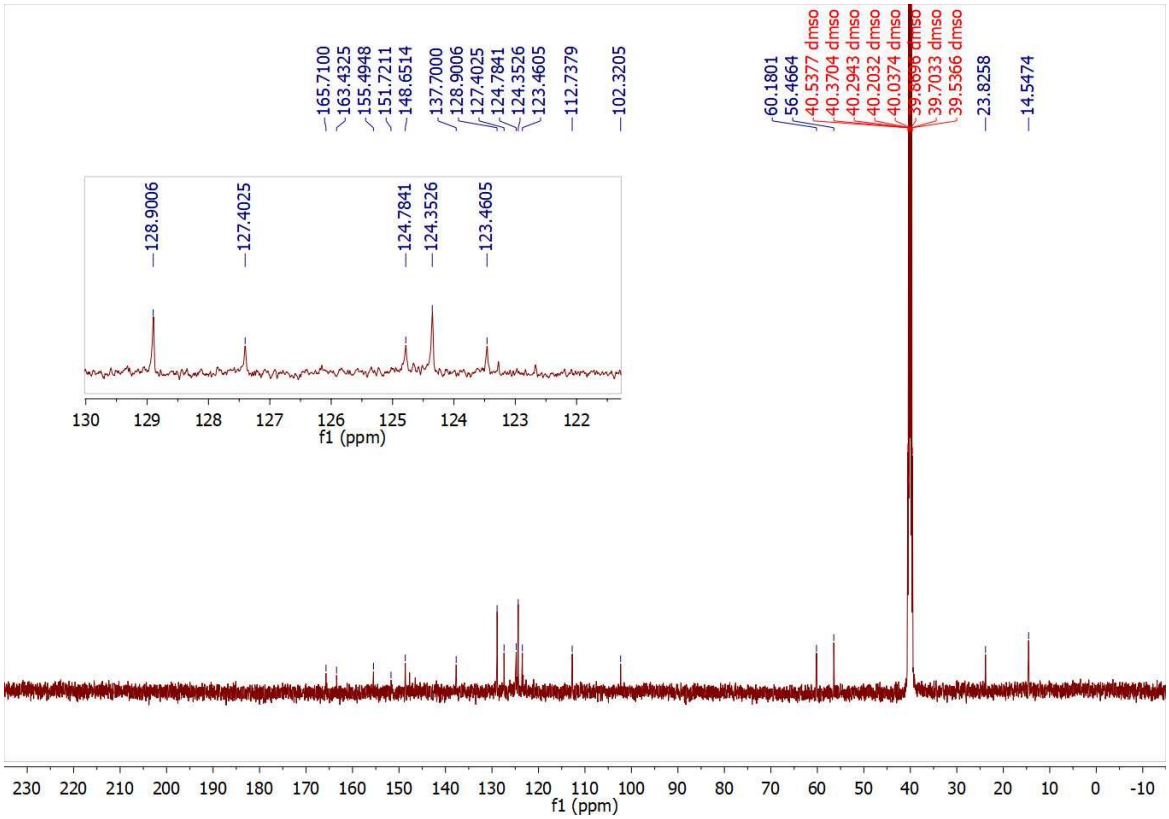
The ^{1}H NMR (400MHz) spectrum of product (IV_b)



The ^{1}H NMR (400MHz) spectrum of product (IV_b)

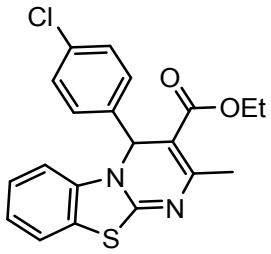


The ^{1}H NMR (400MHz) spectrum of product (IV_b)

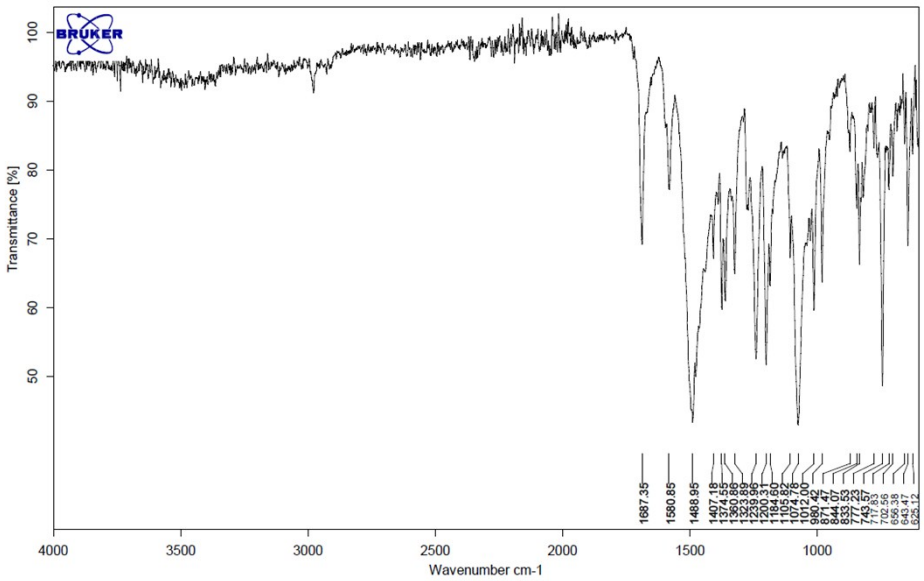


The ^{13}C NMR (400MHz) spectrum of product (IV_b)

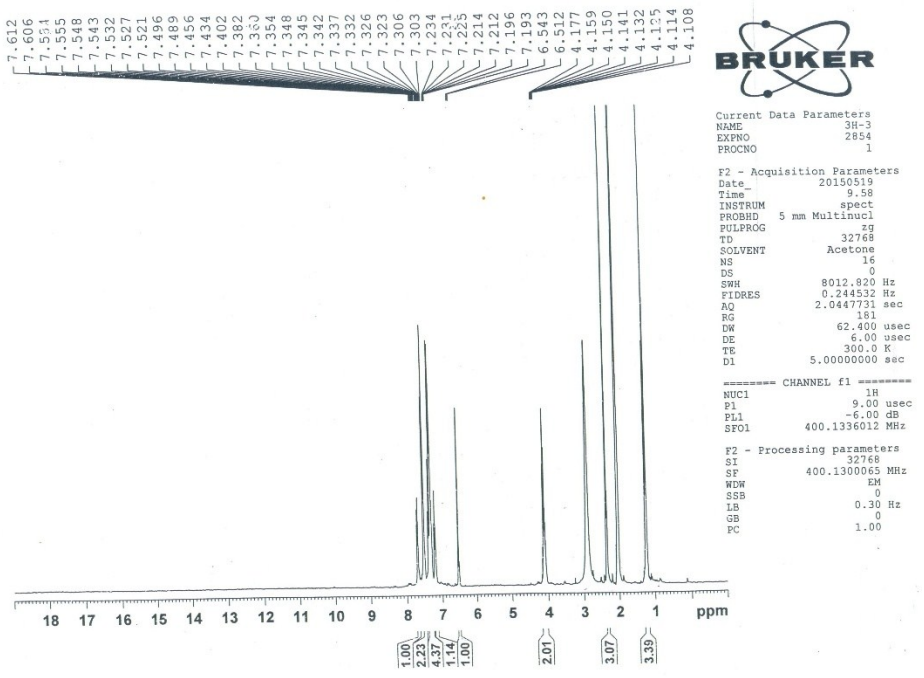
Ethyl-2-methyl-4-(4-chlorophenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate (table 2, IV_c).



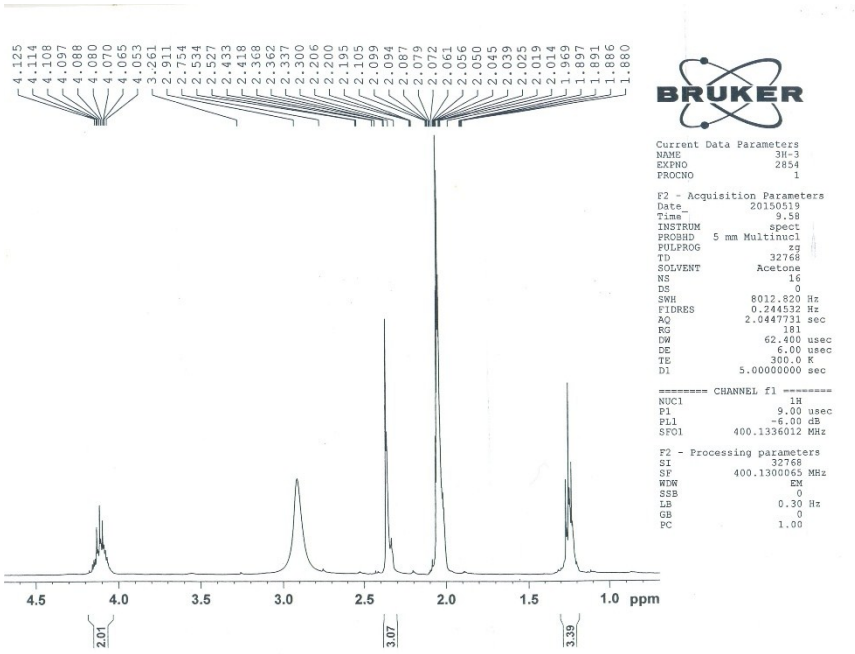
Yellow solid. ^{1}H NMR (Acetone-d_6, 400 MHz): δ 7.70 (d, *J*=8 Hz, 1H), 7.50-7.55 (m, 2H), 7.30-7.41 (m, 4H), 7.21 (td, *J*=7.2, 1.2 Hz, 1H), 6.54 (s, 1H), 4.05-4.12 (m, 2H), 2.36 (s, 3H), 1.20-1.30 (m, 3H). ^{13}C NMR (DMSO-d_6, 400 MHz): 165.8, 163.1, 154.9, 140.9, 137.8, 133.3, 129.4, 129.1, 127.2, 124.6, 123.2, 112.7, 102.8, 60.1, 56.4, 23.7, 14.5, IR (KBr): 2978, 1687, 1580, 1488, 1239, 1200, 1074, 833, 743 cm^{-1}. mp: 87-89 ˚C.



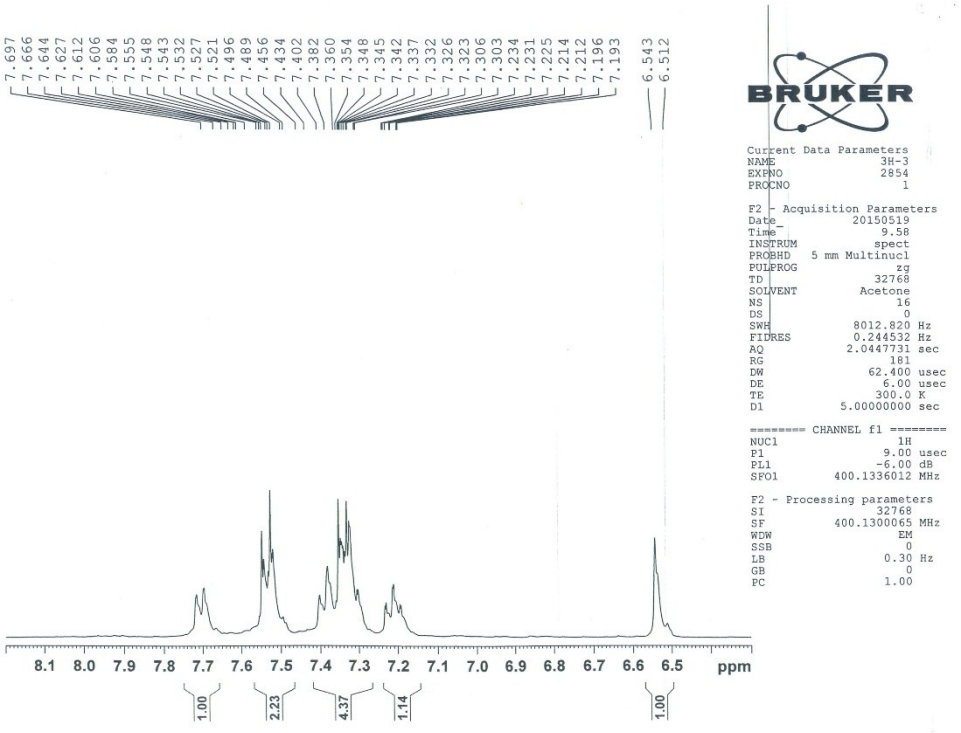
The FT-IR spectrum of product (IV_c)



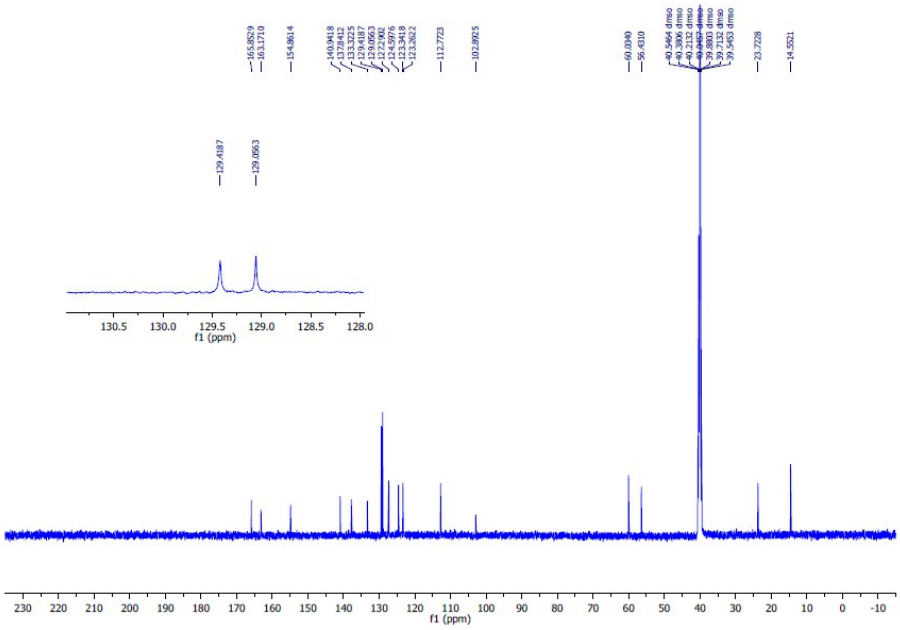
The ^1H NMR (400MHz) spectrum of product (IV_c)



The ^{1}H NMR (400MHz) spectrum of product (IV_c)

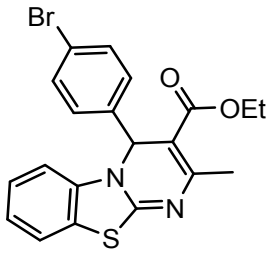


The ^{1}H NMR (400MHz) spectrum of product (IV_c)

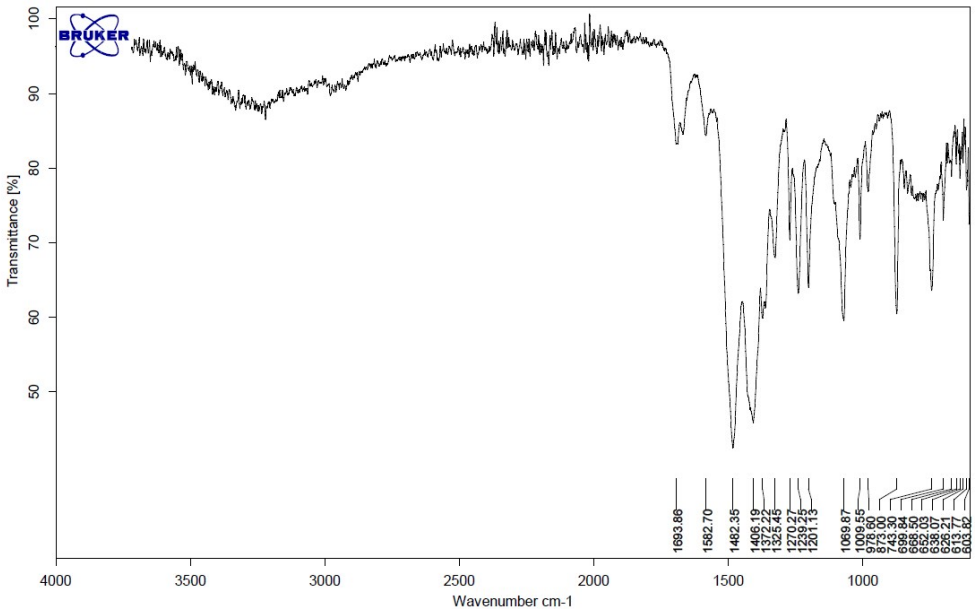


The ^{13}C NMR (400MHz) spectrum of product (IV_c)

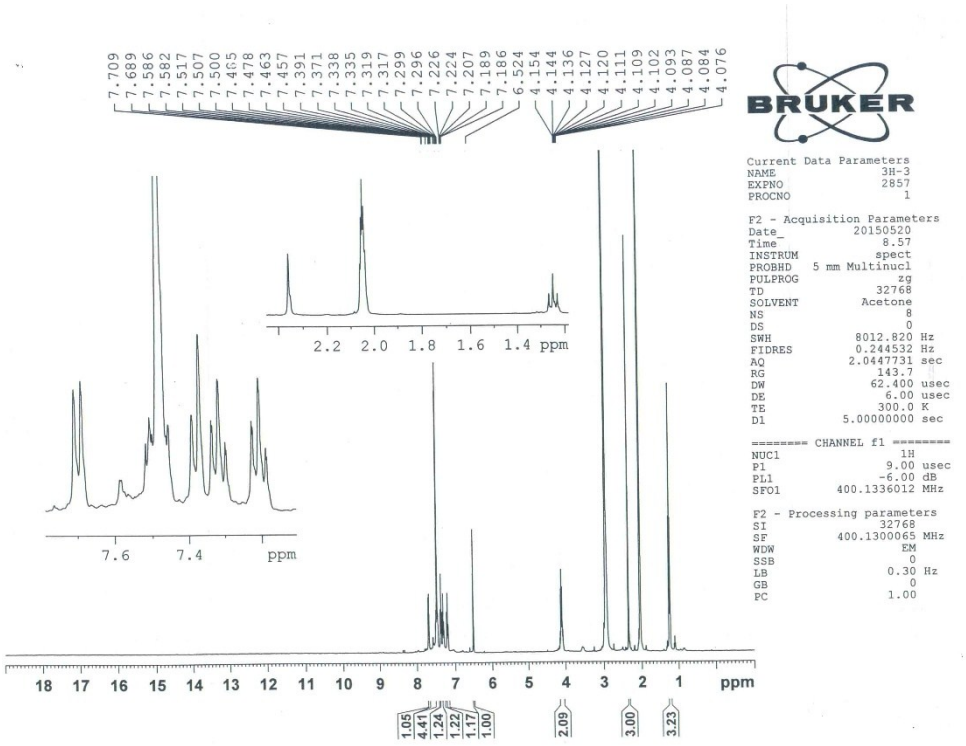
Ethyl-2-methyl-4-(4-bromo phenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate (table 2, IV_d).



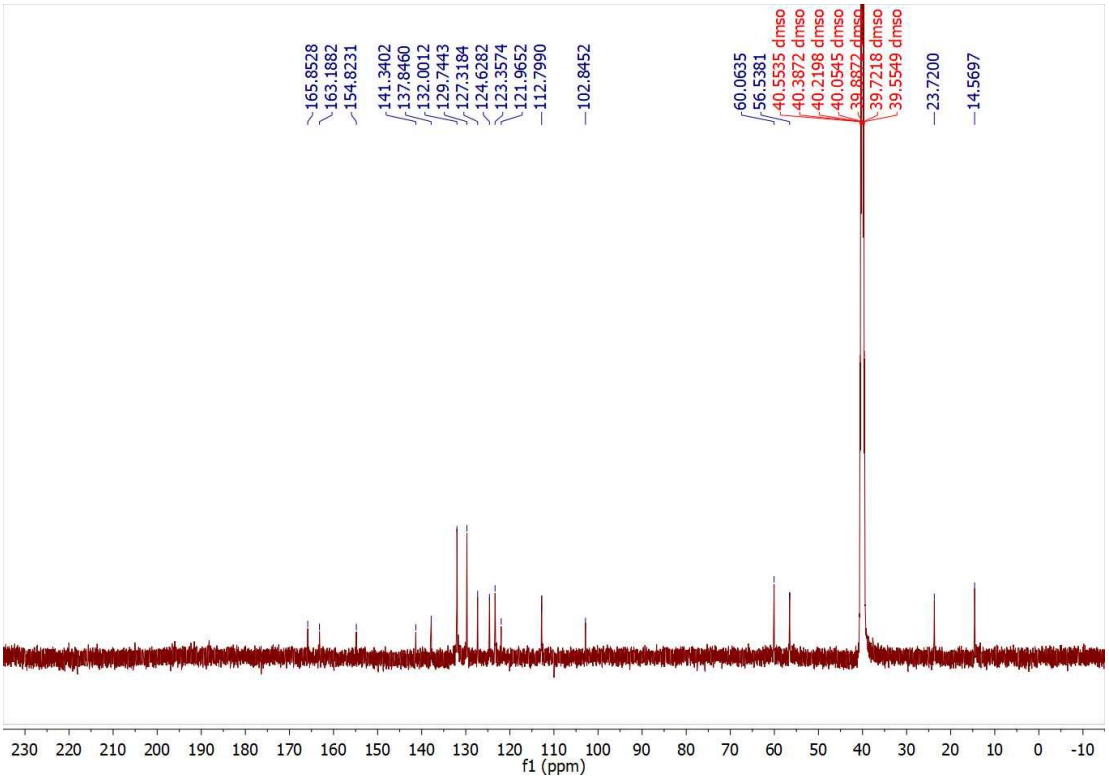
Orange solid. ^1H NMR (Acetone-d_6, 400 MHz): δ 7.70 (d, *J=8* Hz, 1H), 7.43-7.51 (m, 4H), 7.38 (d, *J*=8 Hz, *1H*), 7.32 (td, *J*=8, *1.2, Hz,* 1H), 7.20 (td, *J*=8, *1.2, Hz, 1H*), 6.52 (s, 1H), 4.07-4.15 (m, 2H), 2.37 (s, 3H), 1.20 (t, 3H). ^{13}C NMR (DMSO-d_6, 400 MHz): δ 165.8, 163.2, 154.8, 141.3, 137.8, 132, 129.7, 127.3, 124.6, 123.3, 121.9, 112.8, 102.8, 60.1, 56.5, 23.7, 14.5, IR (KBr): 1693, 1582, 1482, 1406, 1270, 1239, 1201, 1069, 873, 743 cm^{-1}. mp: 110-114 ˚C.



The FT-IR spectrum of product (IV_d)

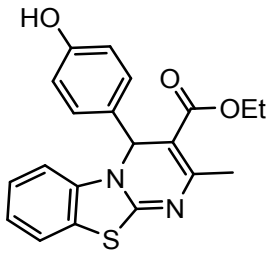


The ^{1}H NMR (400MHz) spectrum of product (IV_d)

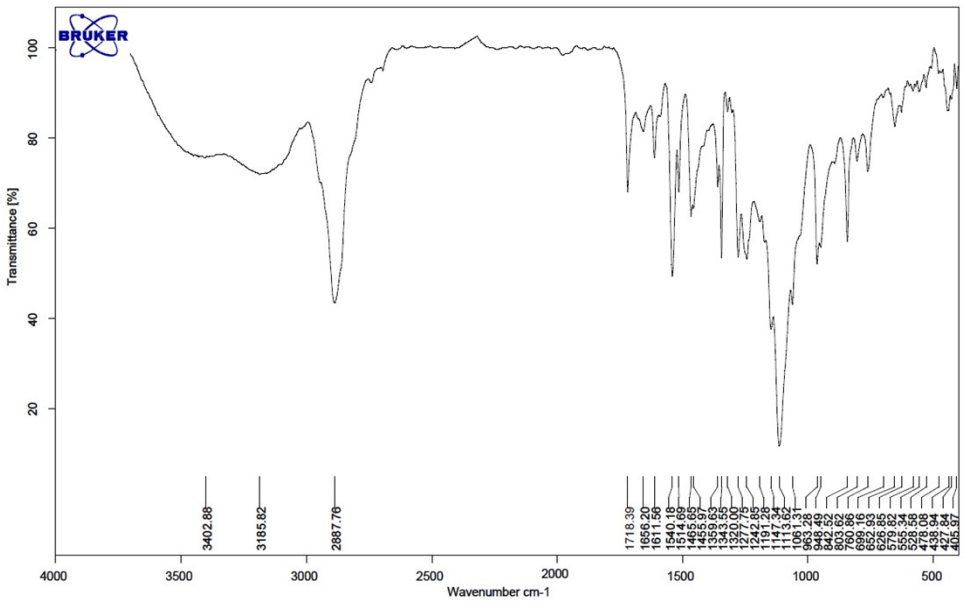


The ^{13}C NMR (400MHz) spectrum of product (IV_d)

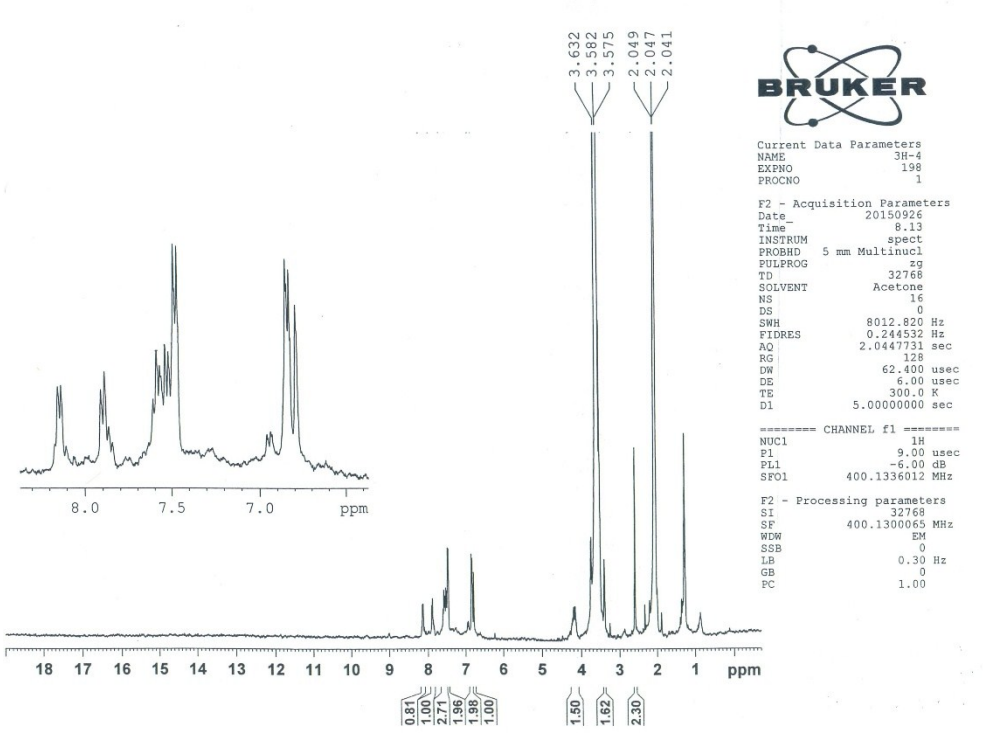
Ethyl-2-methyl-4-(4-hydroxy phenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate (table 2, IV_e).



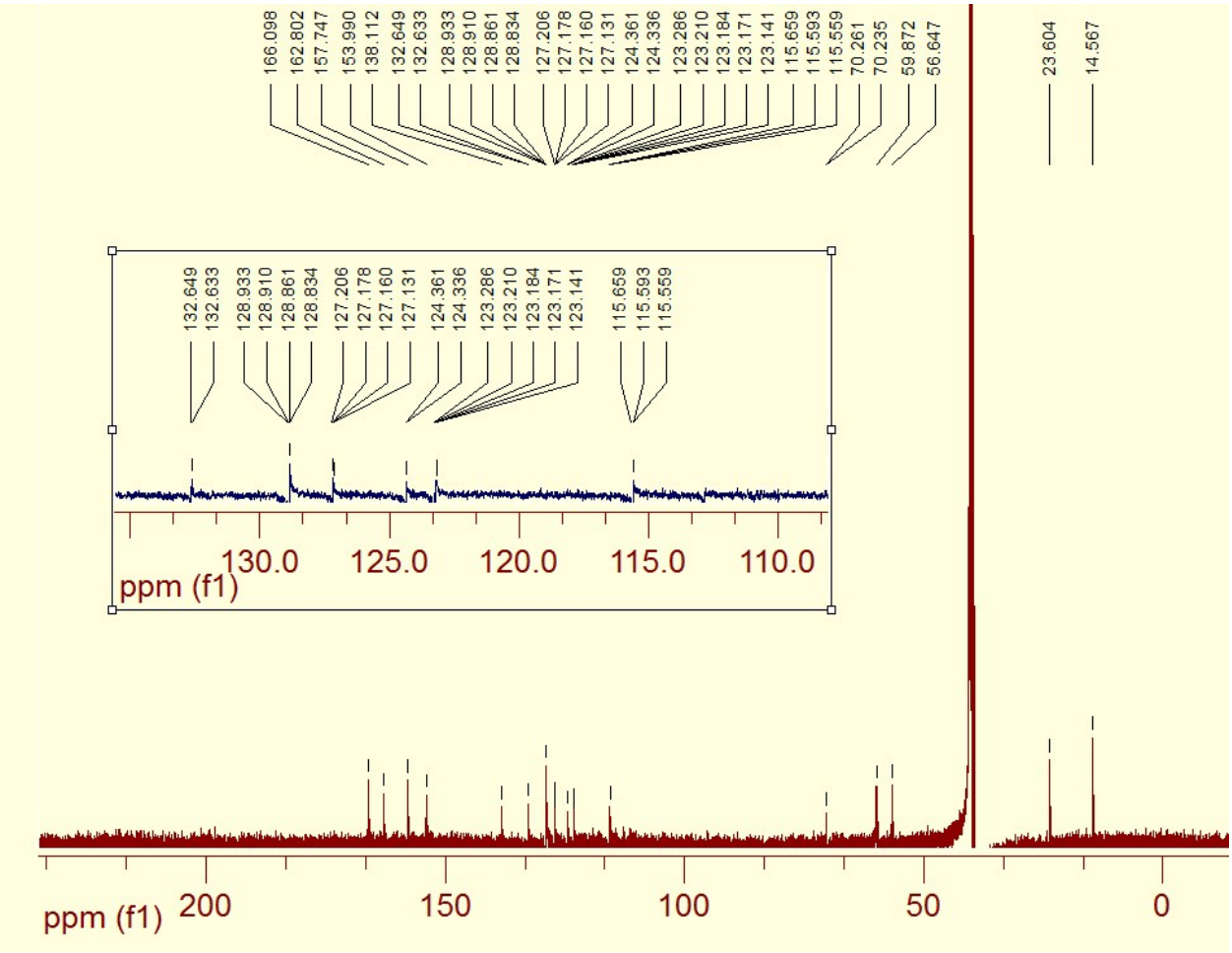
Pale yellow solid. ^1H NMR (Acetone-d_6, 400 MHz): δ 8.10-8.20 (m, 1H), 7.80-7.91 (m, 1H), 7.40-7.60 (m, 5H), 6.80-690 (m, 2H), 6.78 (s, 1H), 4.10-4.40 (m, 2H), 2.60 (s, 3H), 1.30 (m, 3H). ^{13}C NMR (DMSO-d_6, 400 MHz): δ 166.1, 162.8, 157.7, 153.9, 138.1, 132.6, 128.1, 127.1, 124.3, 123.2, 115.6, 70.2, 59.8, 56.6, 23.6, 14.5. IR (KBr): 3402, 2887, 1718, 1611, 1540, 1514, 1465, 1343, 1277, 1242, 1113, 963, 843 cm^{-1}. mp: 210-212 ˚C.



The FT-IR spectrum of product (IV_e)

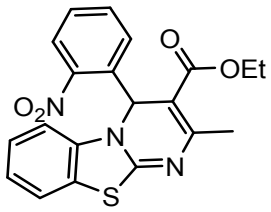


The ^{1}H NMR (400MHz) spectrum of product (IV_e)

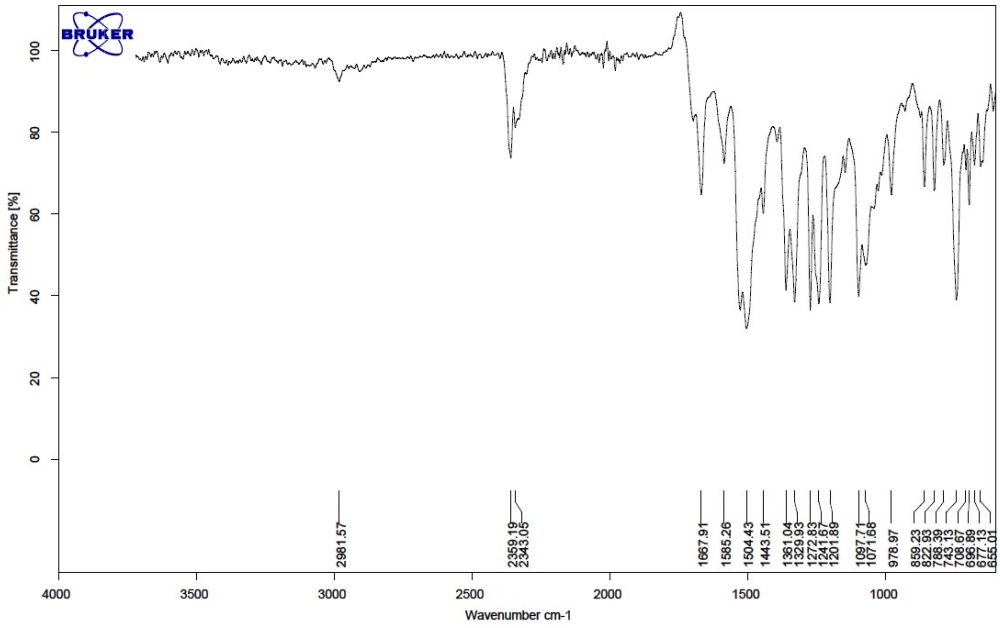


The ^{13}C NMR (400MHz) spectrum of product (IV_e)

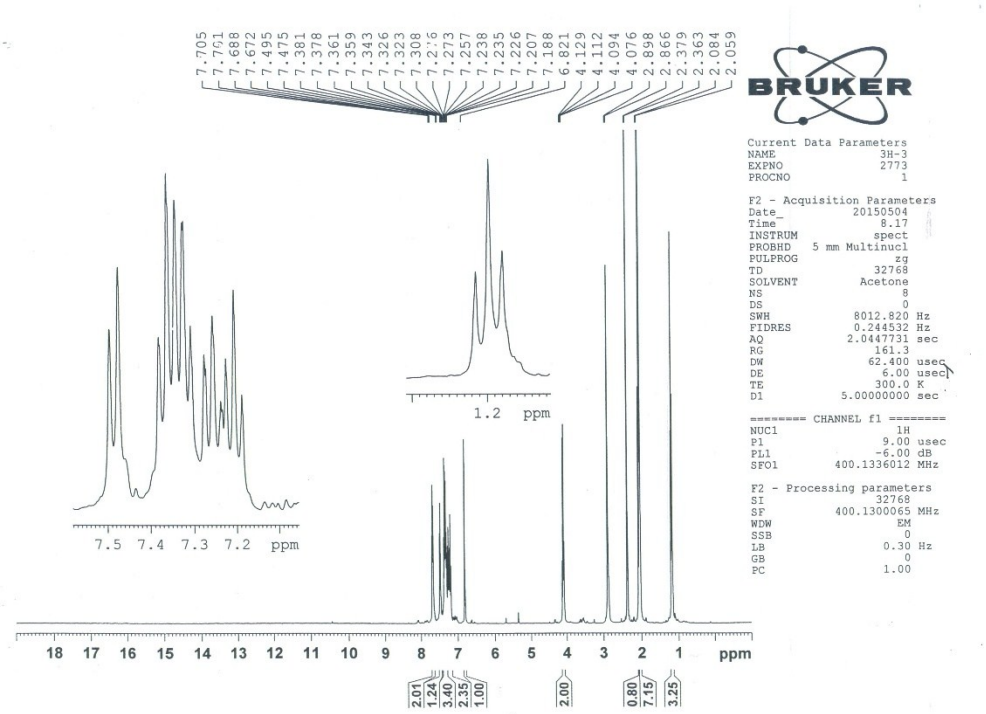
Ethyl-2-methyl-4-(2-nitrophenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate (table 2, IV_f).



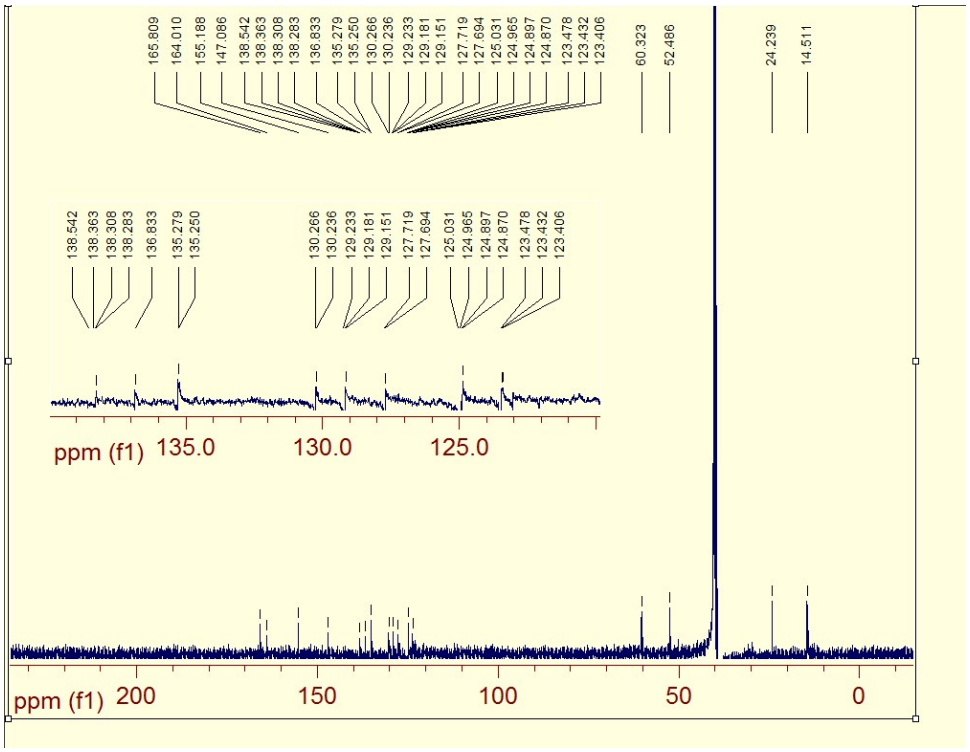
Red orange solid. ^{1}H NMR (Acetone-d_6, 400 MHz): δ 7.70 (m, 2H), 7.49 (d, *J*=8 Hz, 1H), 7.30-7.38 (m, 3H), 7.27 (td, *J*=6.4, 1.2 Hz, 1H), 7.22 (t, *J*=7.6 Hz, 1H), 6.82 (s, 1H), 4.11 (q, *J*=7.2 Hz, 2H), 2.37 (s, 3H), 1.20 (t, J=7.2 Hz, 3H). ^{13}C NMR (DMSO-d_6, 400 MHz): 165.8, 164.1, 155.2, 146.1, 138.2, 136.8, 135.2,130.3, 129.2, 127.7, 127.6, 124.8, 124.7, 123.3, 123.05, 122.2, 60.3, 52.5, 24.2, 14.5, IR (KBr): 2981, 1667, 1585, 1504, 1443, 1361, 1329, 1241, 1201, 1097, 743 cm^{-1}. mp: 122-125 ˚C.



The FT-IR spectrum of product (IV_f)

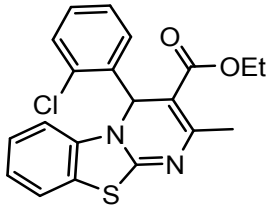


The ^{1}H NMR (400 MHz) spectrum of product (IV_f)

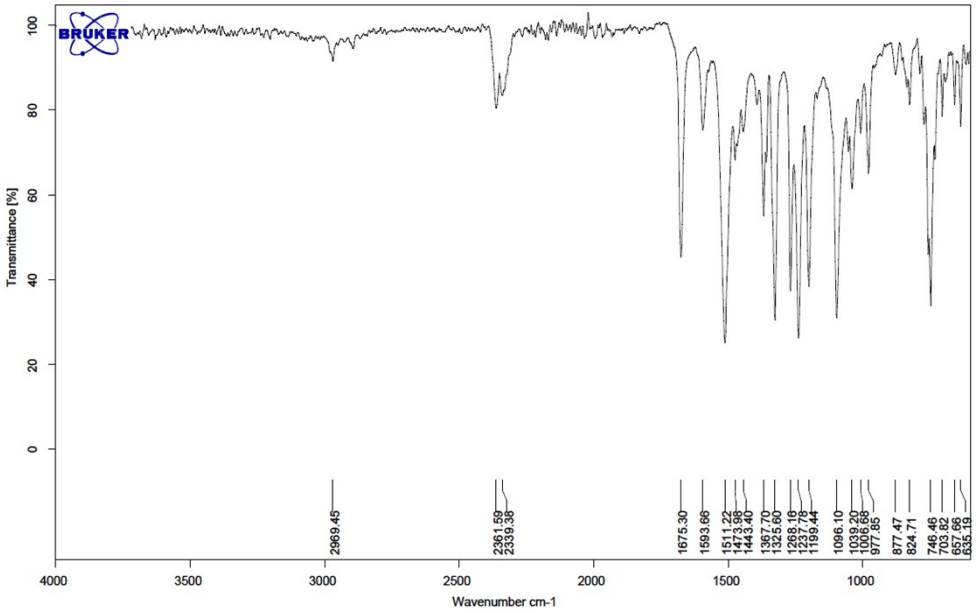


The ^{13}C NMR (400MHz) spectrum of product (IV_f)

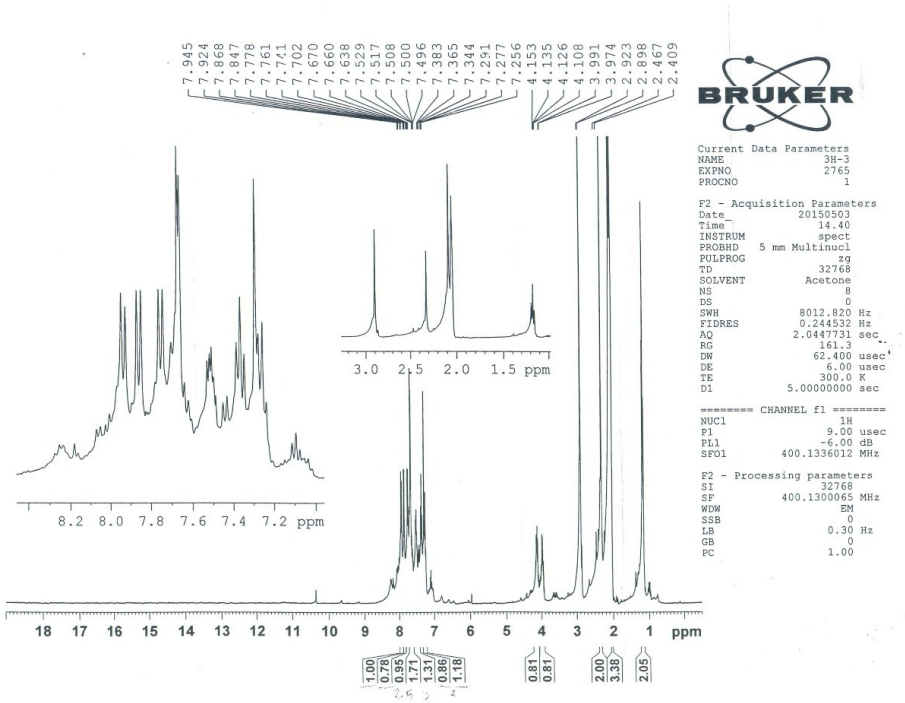
Ethyl-2-methyl-4-(2-chlorophenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate (table 2, IV_g).



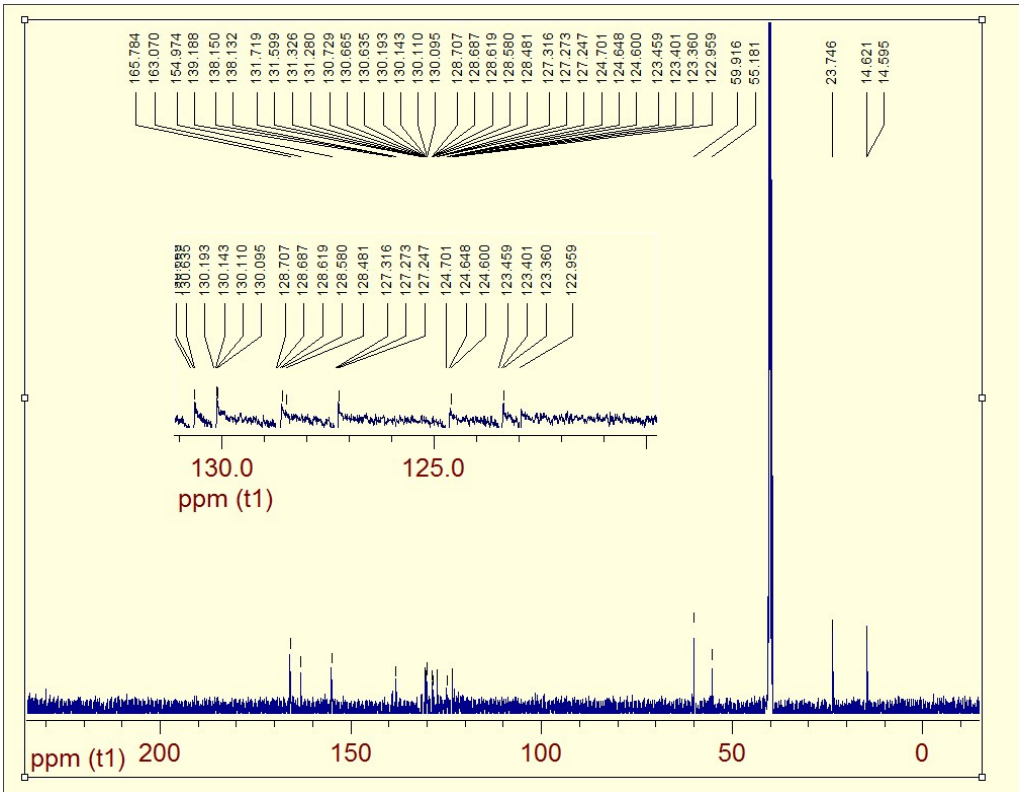
Yellow solid. ^{1}H NMR (Acetone-d_6, 400 MHz): δ 7.94 (d, J=8 Hz, 1H), 7.84 (d, J=8 Hz, 1H), 7.74 (d, J=8 Hz, 1H), 7.67 (m, 1H), 7.49-7.54 (m, 2H), 7.38 (t, J=7.2 Hz, 1H), 7.25-7.31 (m, 2H), 3.97-3.99 (m, 1H), 4.01-4.15 (m, 1H), 2.30 (s, 1H), 1.2 (t, 3H). ^{13}C NMR (DMSO-d_6, 400 MHz): 165.7, 163.1, 154.9, 139.5, 138.1, 131.3, 130.6, 130.1, 128.4, 127.2, 124.7, 123.4, 59.9, 55.2, 23.7, 14.5, IR (KBr): 2969, 1675, 1593, 1473, 1367, 1325, 1268, 1237, 1096, 746 cm^{-1}. mp: 124-126 ˚C.



The FT-IR spectrum of product (IV_g)

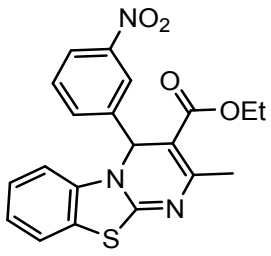


The ^{1}H NMR (400MHz) spectrum of product (IV_g)

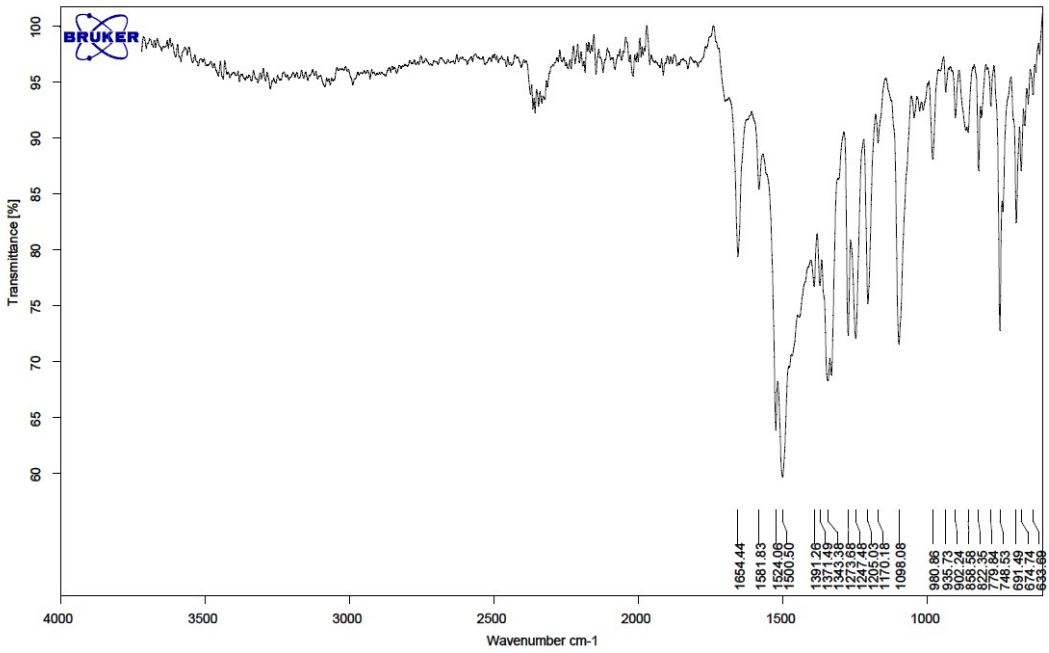


The ^{13}C NMR (400MHz) spectrum of product (IV_g)

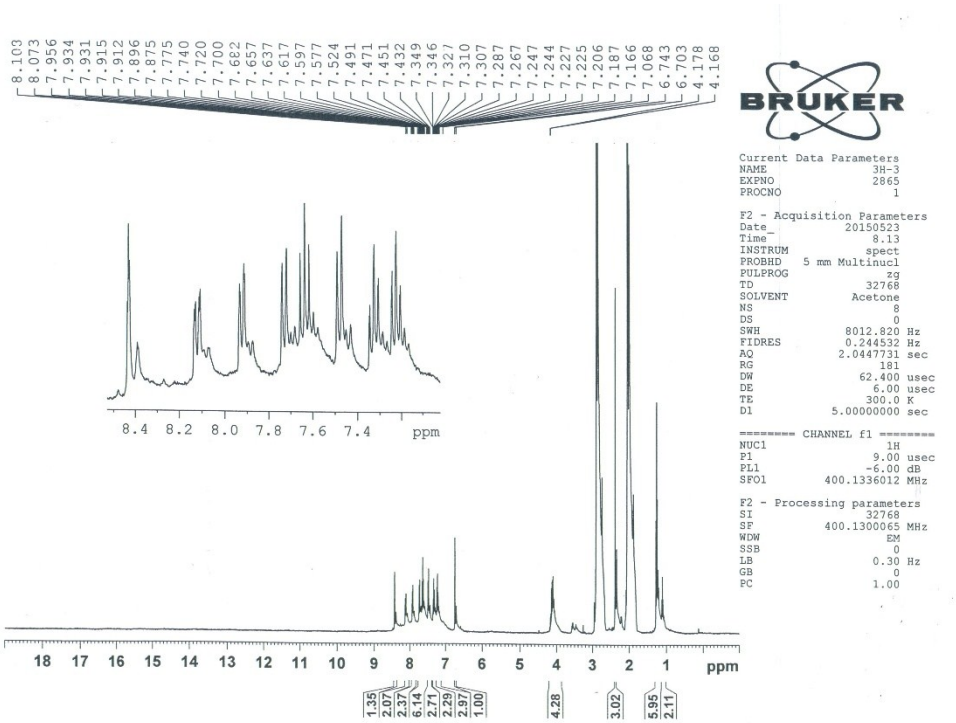
Ethyl-2-methyl-4-(3-nitrophenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate (table 2, IV_h).



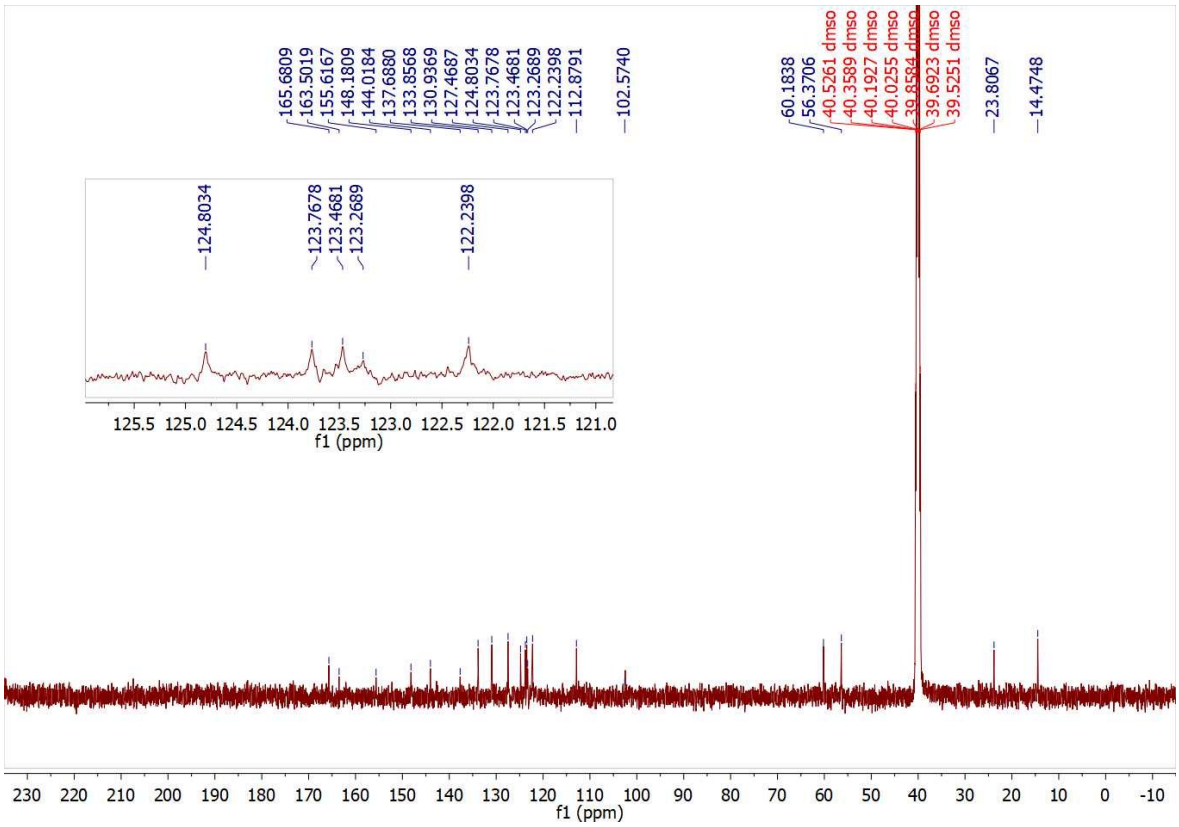
Light yellow solid. ^{1}H NMR (Acetone-d_6, 400 MHz): δ 8.45 (s, 1H), 8.13 (d, *J*=8 Hz, 1H), 7.91 (d, *J*=8 Hz, 1H), 7.71 (d, J=8 Hz, 1H), 7.65 (t, *J*=8 Hz, 1H), *7*.47 (d, *J*=8 Hz, *1H*), 7.33 (t, *J*=8 Hz, *1H*), 7.25 (t, *J*=8 Hz, 1H), 6.74 (s, 1H), 3.90-4.20 (m, 2H), 2.30 (s, 3H), 1.30 (m, 3H). ^{13}C NMR (DMSO-d_6, 400 MHz): 165.7, 163.5, 155.6, 148.2, 144.0, 137.7, 133.8, 130.9, 127.4, 124.8, 123.7, 123.4, 123.2, 122.2, 112.8, 102.5, 60.1, 56.3, 23.8, 14.4, δ IR (KBr): 1654, 1581, 1500, 1343, 1273, 1247, 1205, 1098, 748 cm^{-1}. M.P: 222-224 ˚C.



The FT-IR spectrum of product (IV_h)

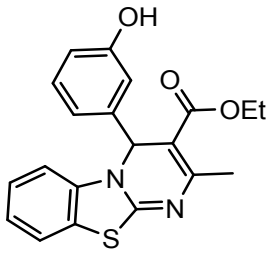


The ^{1}H NMR (400MHz) spectrum of product (IV_h)

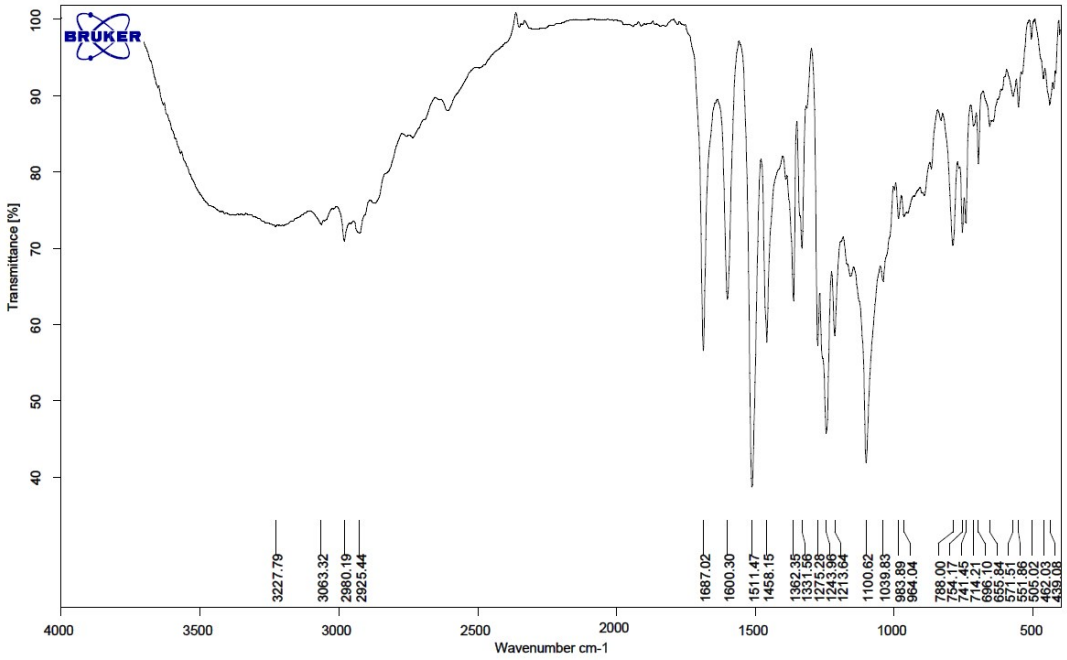


The ^{13}C NMR (400MHz) spectrum of product (IV_h)

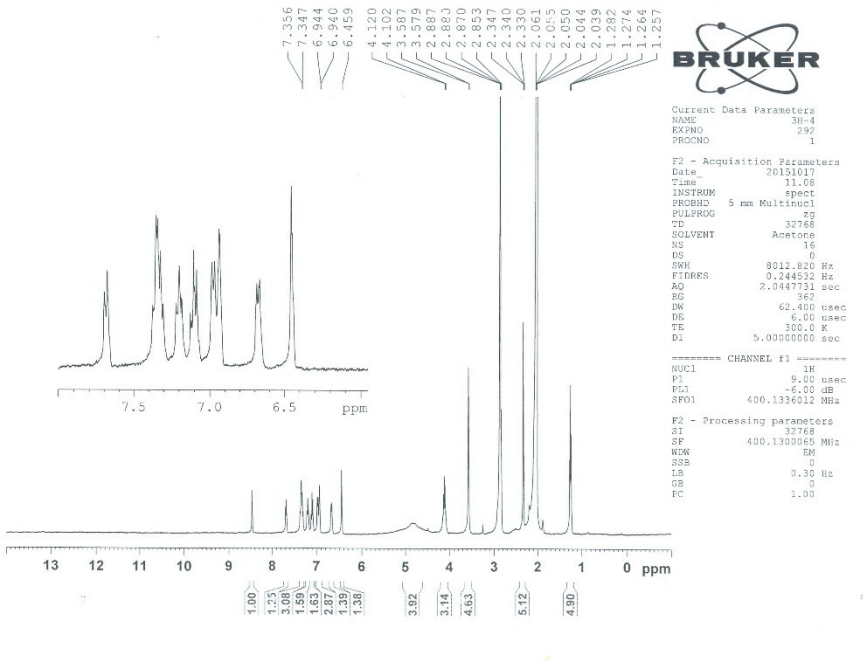
Ethyl-2-methyl-4-(3-hydroxy phenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate (table 2, IV_i).



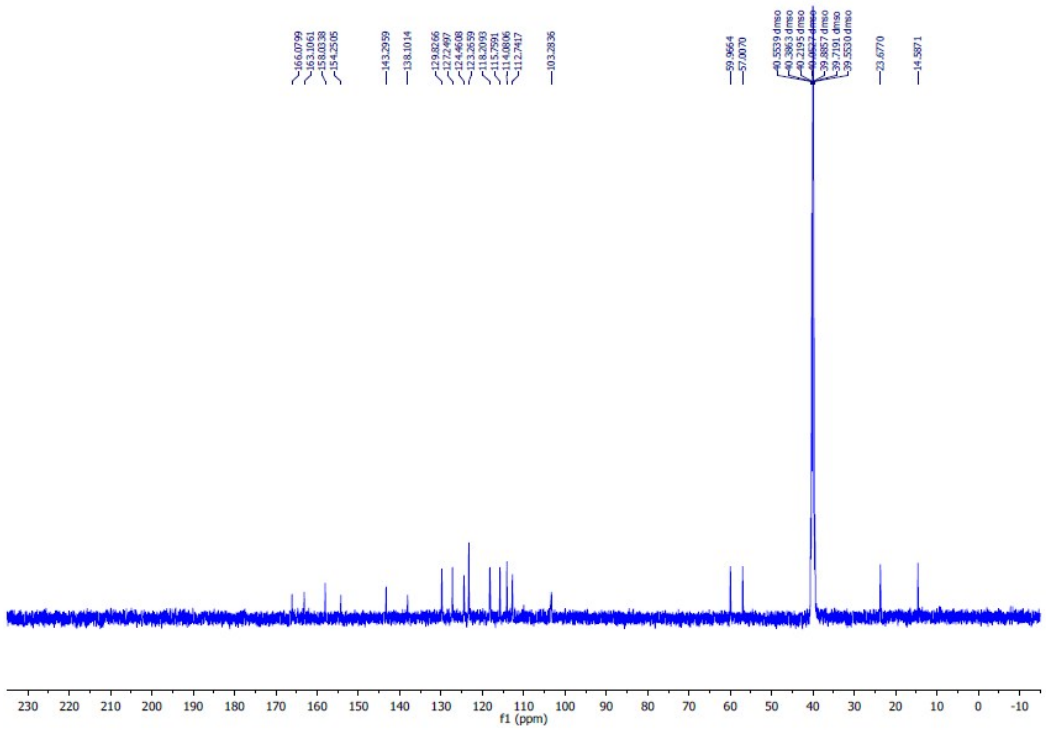
Yellow solid. ^1H NMR (Acetone-d_6, 400 MHz): δ 8.50 (s, 1H), 7.70 (m, 1H), 7.35 (m, 2H), 7.20 (m, 1H), 7.10 (m, 1H), 6.94 (m, 2H), 6.7 (m, 1H), 6.45 (s, 1H), 4.10 (q, *J*=7 Hz, 2H), 2.33 (s, 3H), 1.27 (t, *J*=7 Hz, 3H). ^{13}C NMR (DMSO-d_6, 400 MHz): 166.1, 163.1, 158.0, 154.3, 143.3, 138.1, 129.8, 127/2, 124.5, 123.3, 118.2, 115.8, 114.1, 112.8, 103.3, 59.9, 57, 23.6, 14.6, IR (KBr): 3227, 3063, 2980, 2925, 1687, 1600, 1511, 1458, 1275, 1243, 1213, 1100, 788 cm^{-1}. mp: 260-263 ˚C.



The FT-IR spectrum of product (IV_i)

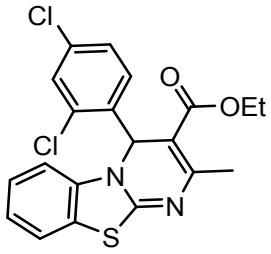


The ^{1}H NMR (400MHz) spectrum of product (IV_i)

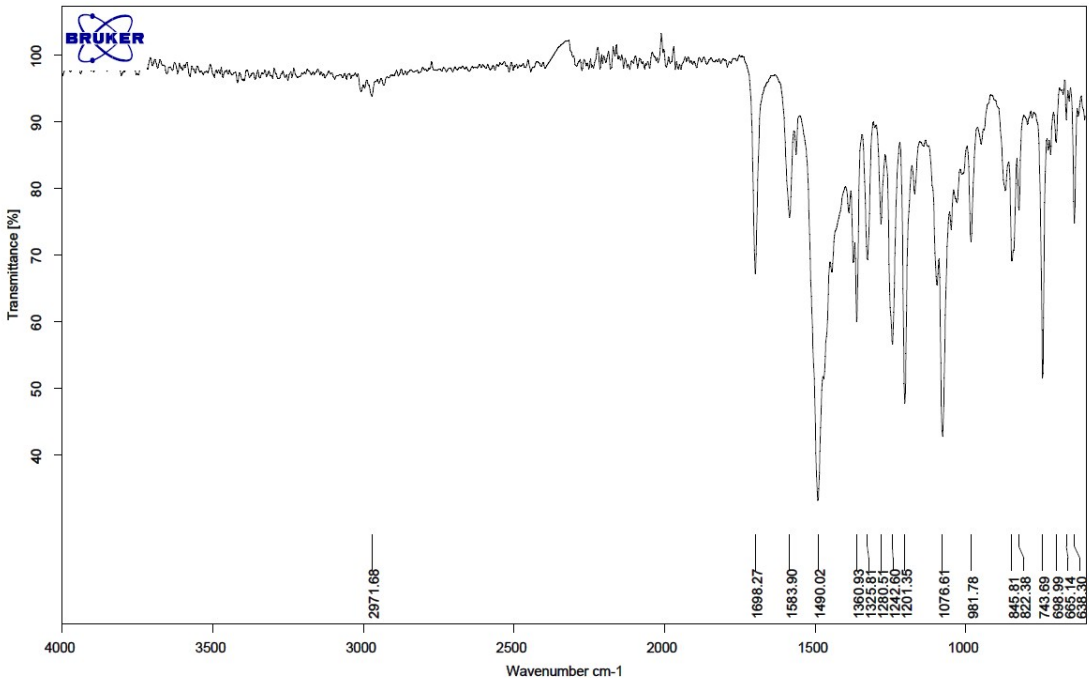


The ^{13}C NMR (400MHz) spectrum of product (IV_i)

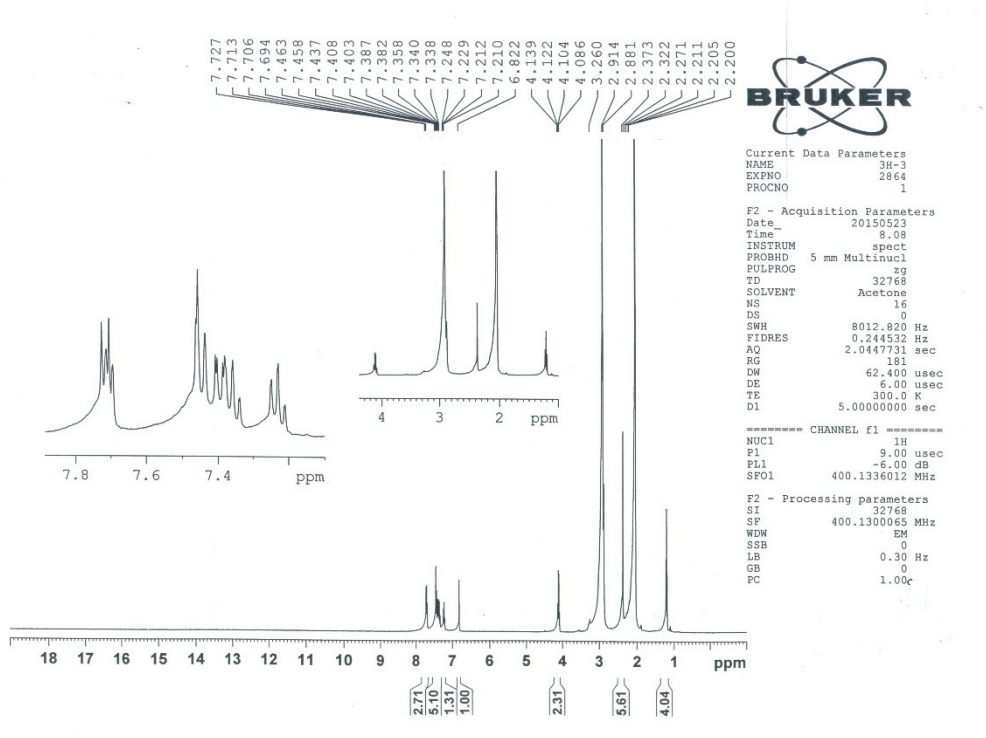
Ethyl-2-methyl-4-(2,4-dichlorophenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate (table 2, IV_j).



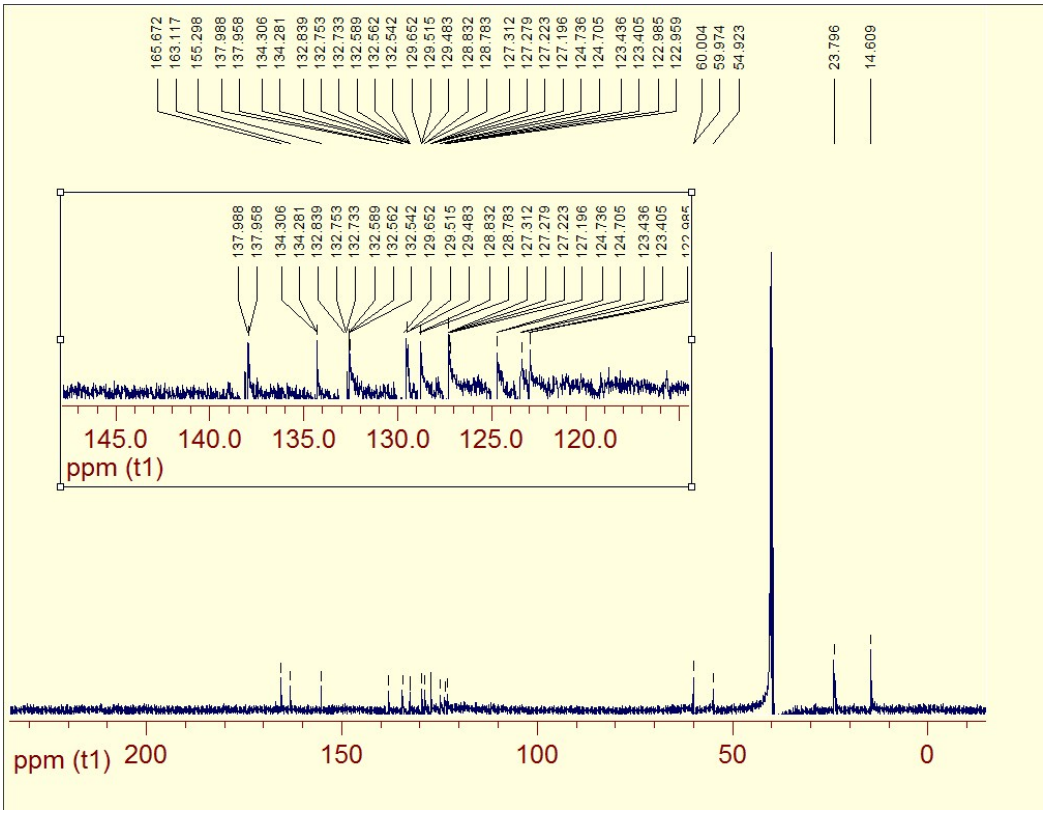
Yellow solid. ^1H NMR (Acetone-d_6, 400 MHz): δ 7.69-7.72 (m, 2H), 7.33-7.46 (m, 4H), 7.22 (t, *J*=7.6 Hz, 1H), 6.82 (s, 1H), 4.11 (q, *J*=6.8 Hz, 2H), 2.37 (s, 3H), 1.20 (t, *J*=6.8 Hz, 3H). ^{13}C NMR (DMSO-d_6, 400 MHz): 165.6, 163.1, 155.3, 137.9, 134.3, 132.5, 129.5, 128.8, 127.3, 127.2, 124.7, 123.4, 122.9, 59.9, 54.9, 23.8, 14.6, IR (KBr): 3007, 2971, 1698, 1583, 1490, 1360, 1242, 1201, 1076, 845, 743 cm^{-1}. M.P: 133-135 ˚C.



The FT-IR spectrum of product (IV_j)

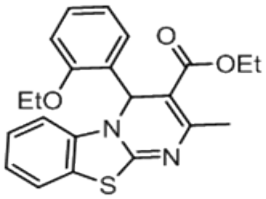


The ^{1}H NMR (400MHz) spectrum of product (IV_j)

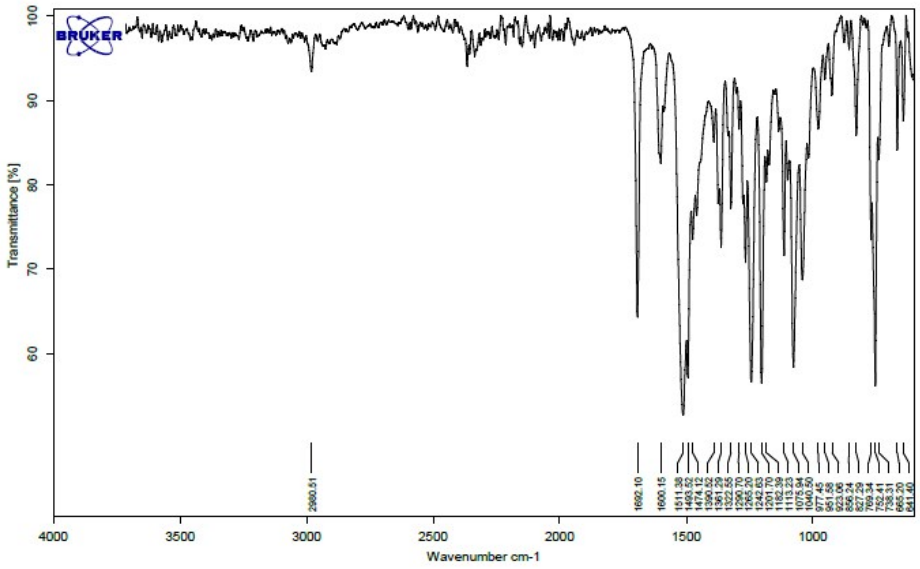


The ^{13}C NMR (400MHz) spectrum of product (IV_j)

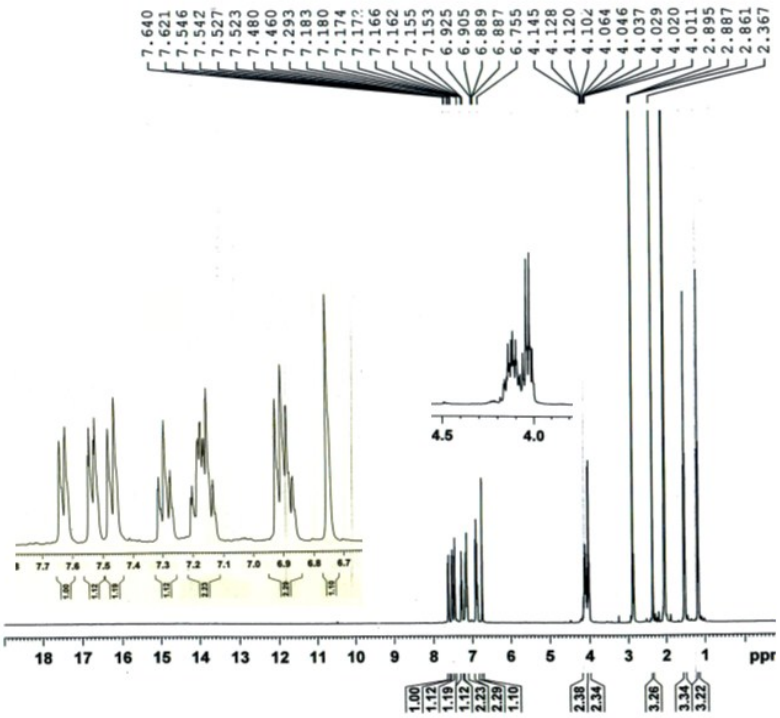
Ethyl-2-methyl-4-(2, ethoxyphenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate (table 2, IV_k).



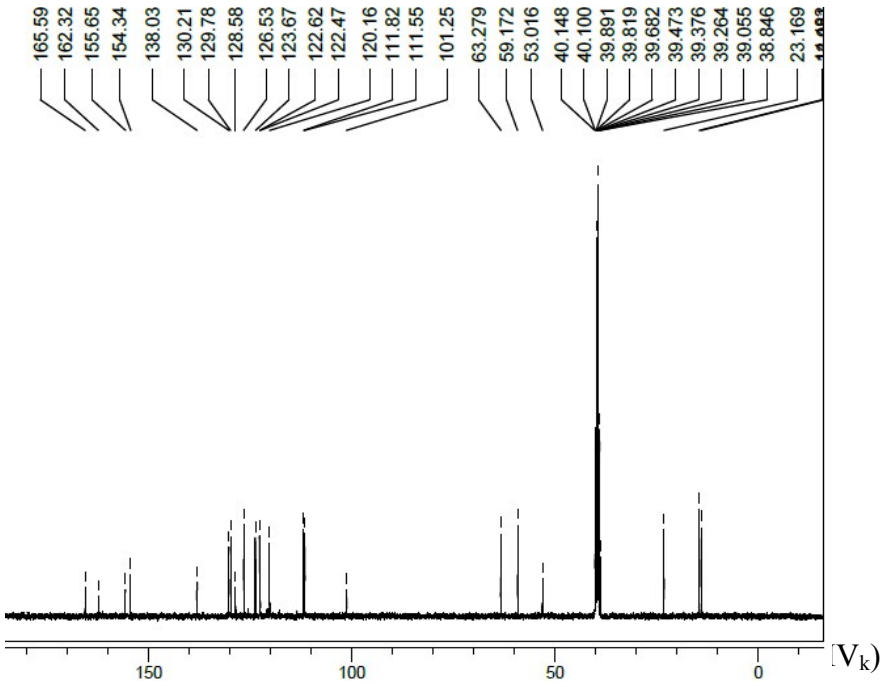
Yellow solid, ^1H NMR (Acetone-d_6, 400 MHz) : δ ppm: 7.63 (d, 1H, *J*=8 Hz), 7.53 (dd, 1H, *J*=7.6 Hz, *J*=1.6 Hz), 7.47 (d, 1H, *J*=8 Hz), 7.29 (m, 1H), 7.15-7.18 (m, 2H), 6.88-6.92 (m, 2H), 6.75 (s, 1H, CH), 4.10-4.14 (m, 2H), 4.03 (q, 2H, ^3J=7.2 Hz), 2.37 (s, 3H), 1.50-1.54 (m, 3H), 1.16-1.20 (m, 3H). ^{13}C NMR (DMSO-d_6, 100 MHz) :δ ppm: 13.98, 14.48, 23.16, 53.01, 59.17, 63.27, 101.25, 111.55, 111.82, 120.16, 122.47, 122.62, 123.67, 126.53, 128.58, 129.78, 130.21, 138.03, 154.34, 155.65, 162.32, 165.59. FT-IR (ATR) $\bar{\upsilon}$ (cm^{-1}): 2980, 1692, 1600, 1511, 1493, 1242, 1201, 1075, 1040, 752 cm^{-1}. m.p: 171-175 ˚C.



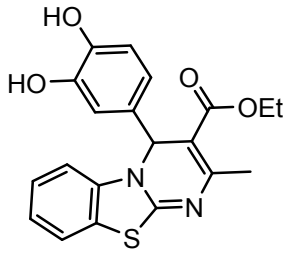
The FT-IR spectrum of product (IV_k



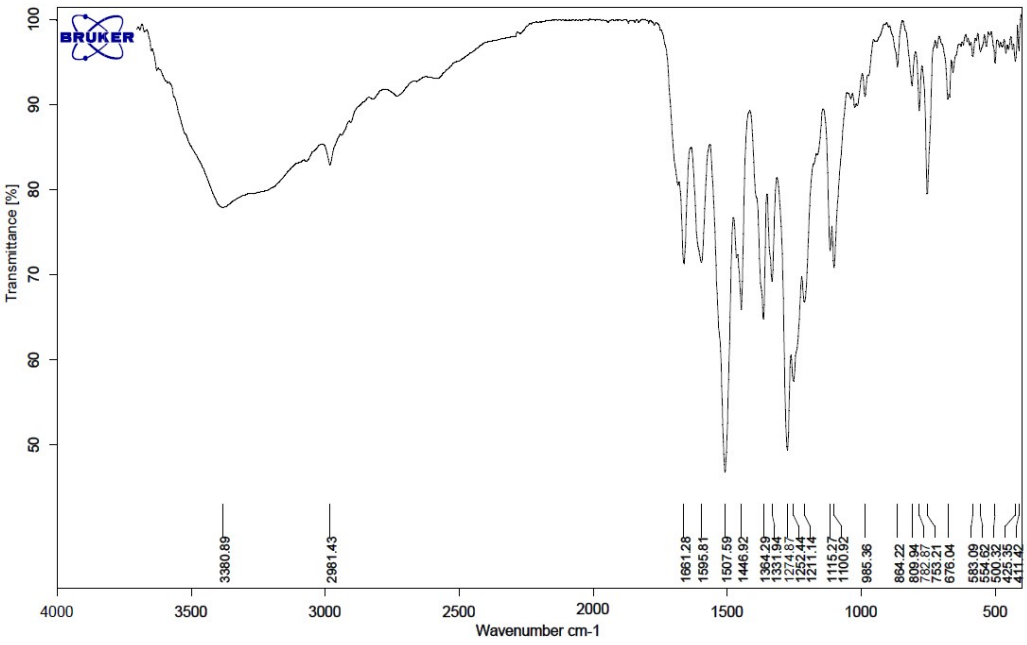
The ^1H NMR (400MHz) spectrum of product (IV_k)



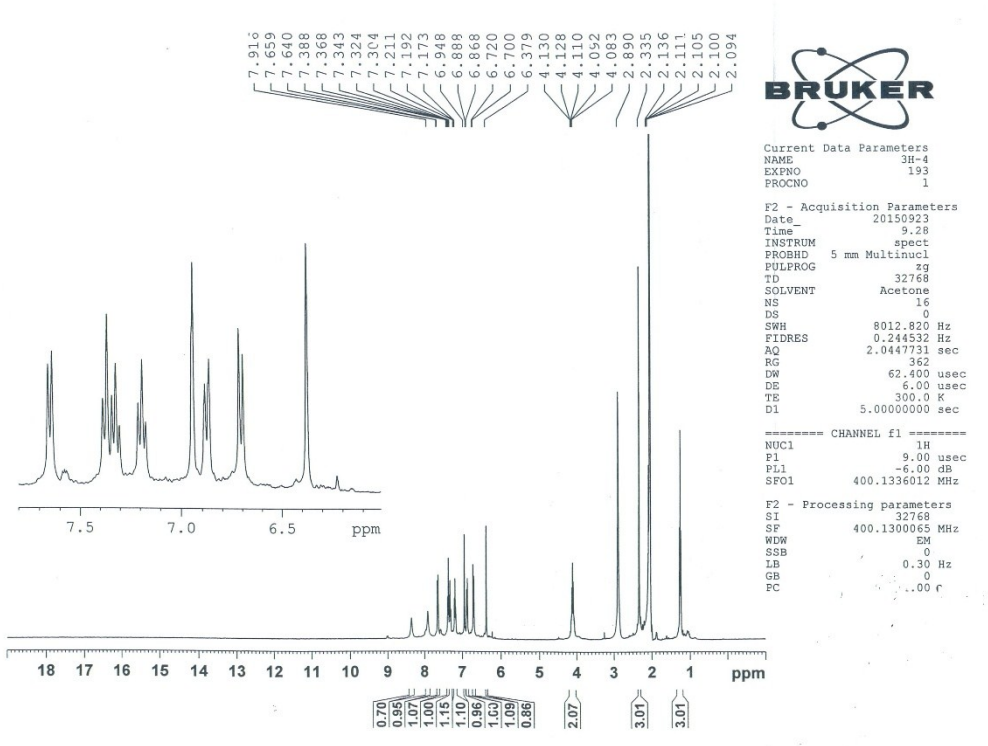
Ethyl-2-methyl-4-(3,4-dihydroxyphenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate (table 2, IV_l).



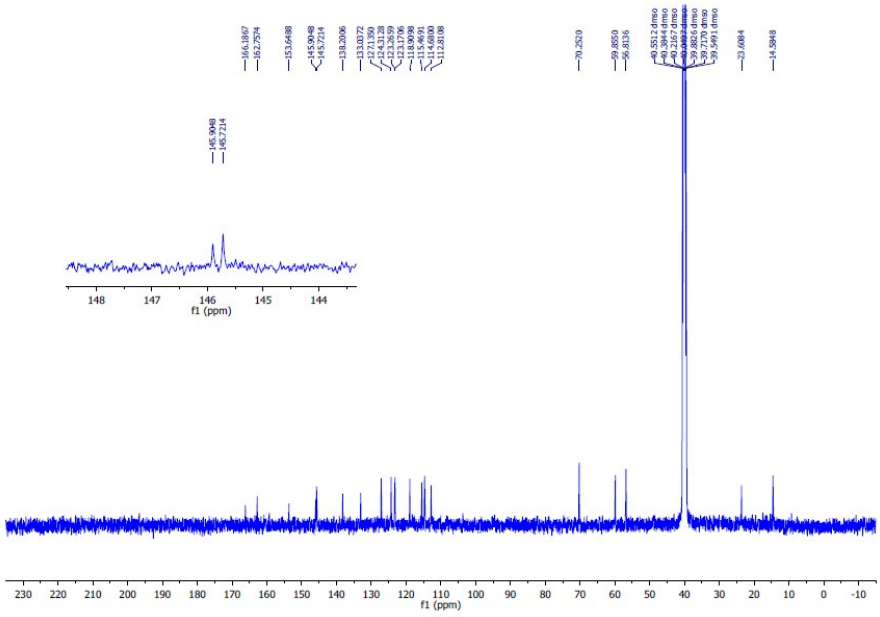
Gray solid. ^{1}H NMR (Acetone-d_6, 400 MHz): δ 8.30 (s, 1H), 7.91 (s, 1H), 7.64 (d, *J*=7.6 Hz, 1H), 7.37 (d, *J*=8 Hz, 1H), 7.32 (t, *J*=7.6 Hz, 1H), 7.19 (t, *J*=7.6 Hz, 1H), 6.94 (s, 1H), 6.87 (d, *J*=8 Hz, 1H), 6.71 (d, *J*=8 Hz, 1H), 6.37 (s, 1H), 4.12 (q, *J*=7.2 Hz, 2H), 2.33 (s, 3H), 1.20 (t, *J*=7.2 Hz, 3H). ^{13}C NMR (DMSO-d_6 and $CDCl_3$, 400 MHz): 166.2, 162.7, 153.6, 145.9, 145.7, 138.2, 133.0, 127/1, 124.3, 123.3, 123.2, 118.9, 115.4, 114.6, 112.8, 59.8, 56.8, 23.6, 12.4, IR (KBr): 3380, 2981, 1661, 1595, 1507, 1446, 1274, 1252, 1211, 1100, 753 cm^{-1}. mp: 225-227 ˚C.



The FT-IR spectrum of product (IV_l)



The ^{1}H NMR (400MHz) spectrum of product (IV_l)



The ^{13}C NMR (400MHz) spectrum of product (IV_l)