Preparation and characterization of Nano guar gum/BF_3/Fe_3O_4 as a novel And a natural-based lewis acid catalysts for the one-pot green synthesis of pyrimidobenzothiazoles under solvent-free conditions

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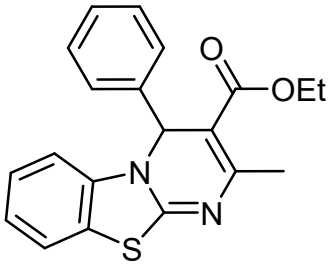
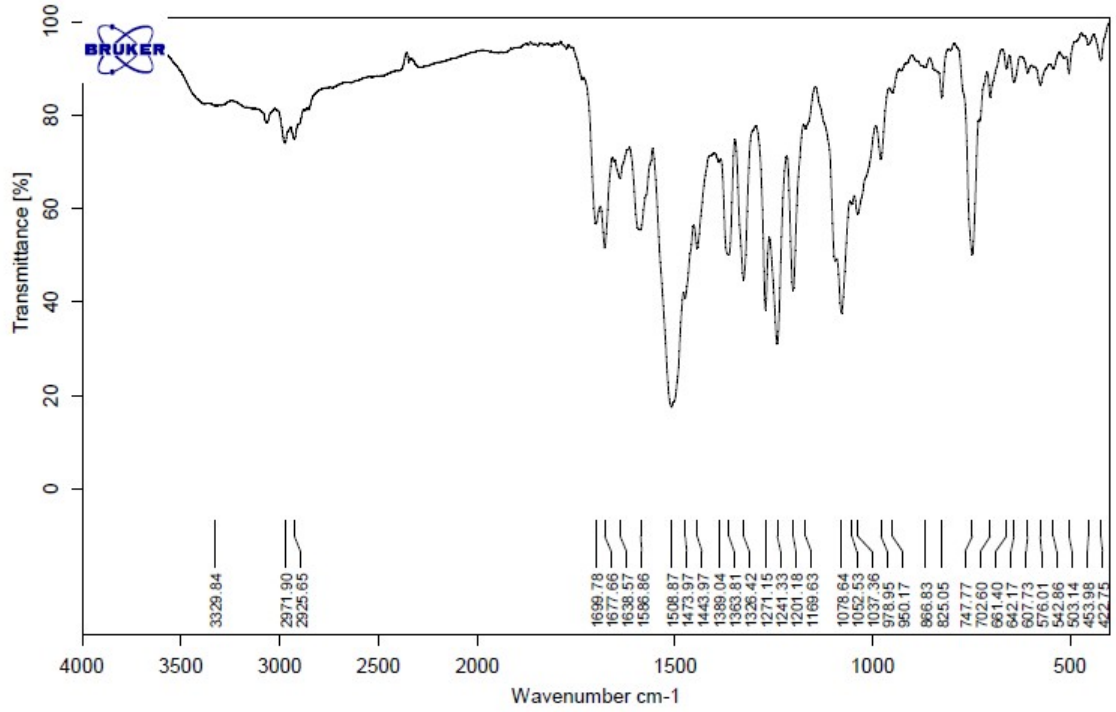
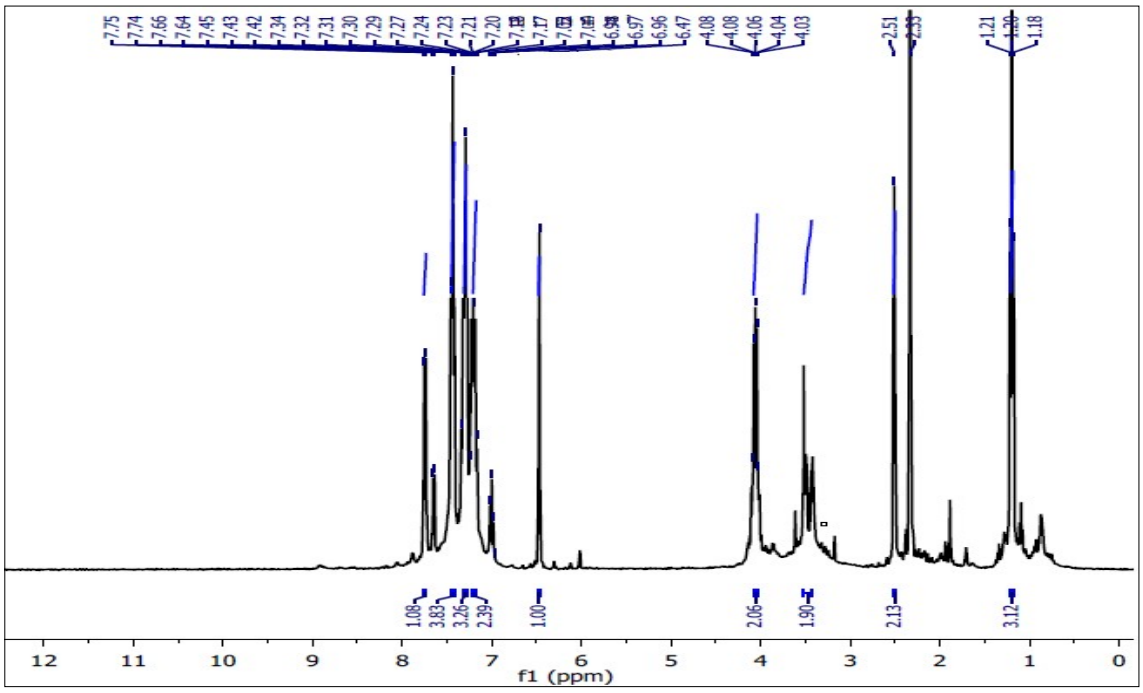
Ethyl-2-methyl-4-(phenyl)-4H-pyrimido[2,1-b][1,3]benzothiazole-3-carboxylate



Pale yellow solid, Melting point: 177-180 °C. FT-IR (KBr) $\bar{\upsilon}$ (cm^{-1}): 2971, 1699, 1677, 1508, 1241, 747. ^1H NMR (DMSO-d_6, 400 MHz): δ 7.73 (d, *J*=7.7 Hz, 1H), 7.41 (t, *J*=7 Hz, 2H), 7.27-7.34 (m, 4H), 7.16-7.24 (m, 2H), 6.46 (s, 1H), 4.02-4.08 (m, 2H), 2.33 (s, 3H), 1.17 (t, *J* = 7 Hz, 3H).

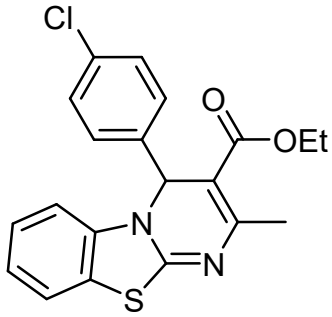


The FT-IR of Ethyl-2-methyl-4-(phenyl)-4H-pyrimido[2,1-b][1,3]benzothiazole-3-carboxylate

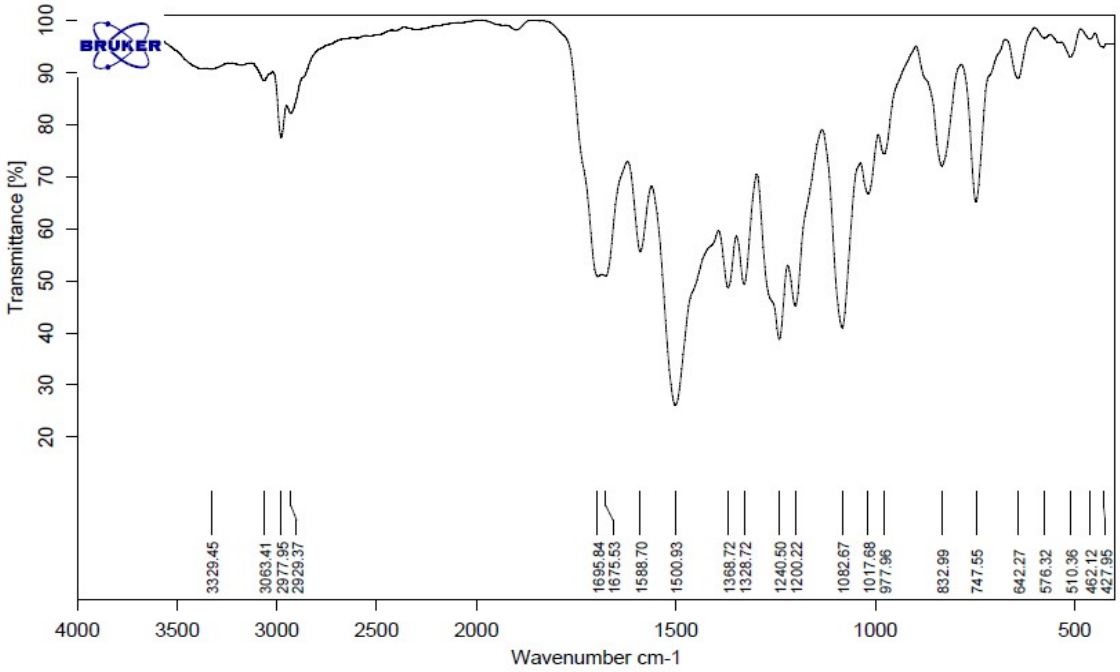


The ^{1}H NMR spectrum of Ethyl-2-methyl-4-(phenyl)-4H-pyrimido[2,1-b][1,3]benzothiazole-3-carboxylate

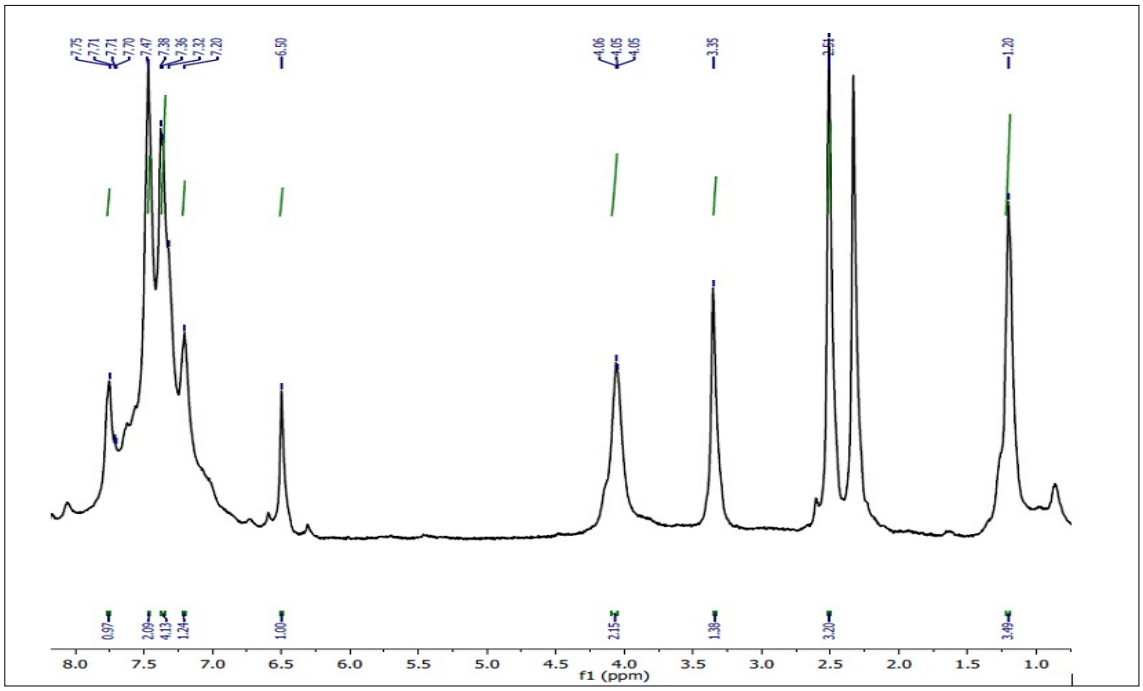
Ethyl-2-methyl-4-(4-chlorophenyl)-4H-pyrimido[2,1-b][1,3]benzothiazole-3-carboxylate



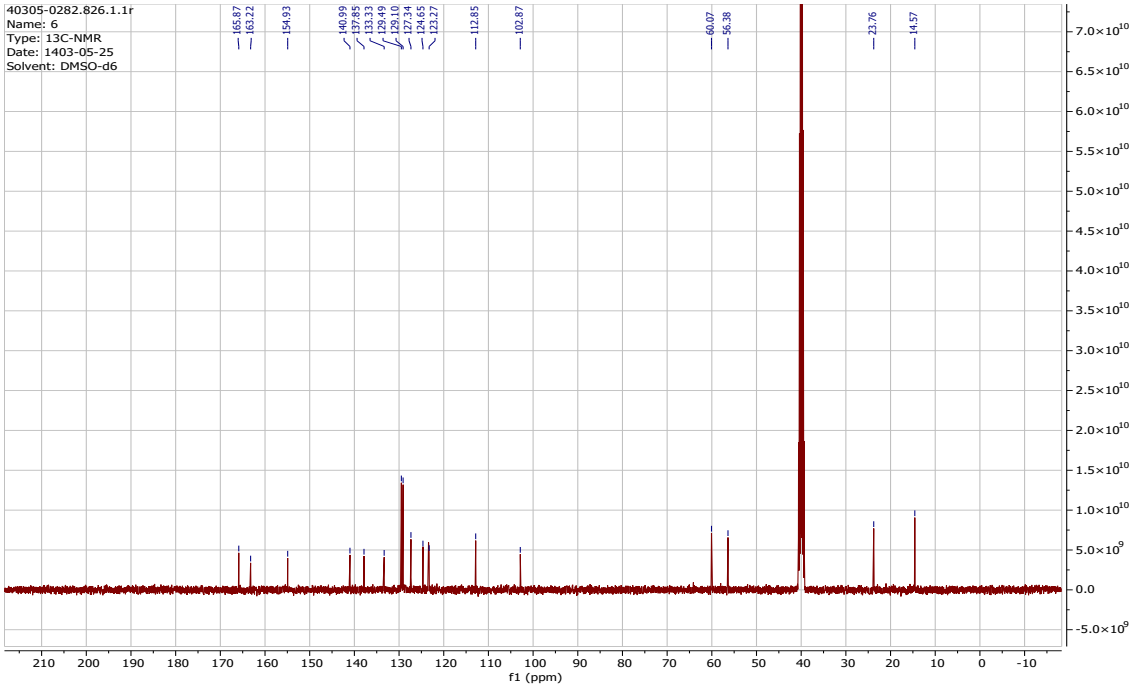
Yellow solid, Melting point: 89-91 °C. FT-IR (KBr) ῡ (cm^{-1}): 2977, 1695, 1675, 1588, 1500, 1240, 1200, 1082, 832, 747. ^{1}H NMR (DMSO-d6, 400 MHz): δ 7.75 (br. s, 1H), 7.46-7.50 (m, 2H), 7.32-7.37 (m, 4H), 7.20 (br. s, 1H), 6.49 (s, 1H), 4.04-4.06 (t, J = 4.5 Hz, 2H), 2.5 (s, 3H), 1.10-1.30 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6): 14.57, 23.76, 56.38, 60.07, 102.87, 112.85, 123.27, 124.65, 127.34, 129.10, 129.49, 133.33, 137.85, 140.99,154.93, 163.22, 165.87.



The FT-IR of Ethyl-2-methyl-4-(4-chlorophenyl)-4H-pyrimido[2,1-b][1,3]benzothiazole-3-carboxylate

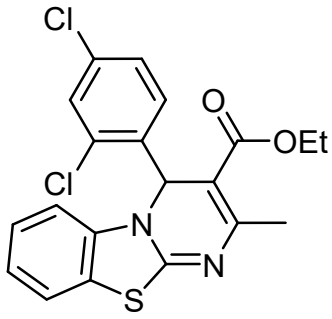


The ^{1}H NMR spectrum of Ethyl-2-methyl-4-(4-chlorophenyl)-4H-pyrimido[2,1-b][1,3]benzothiazole-3-carboxylate

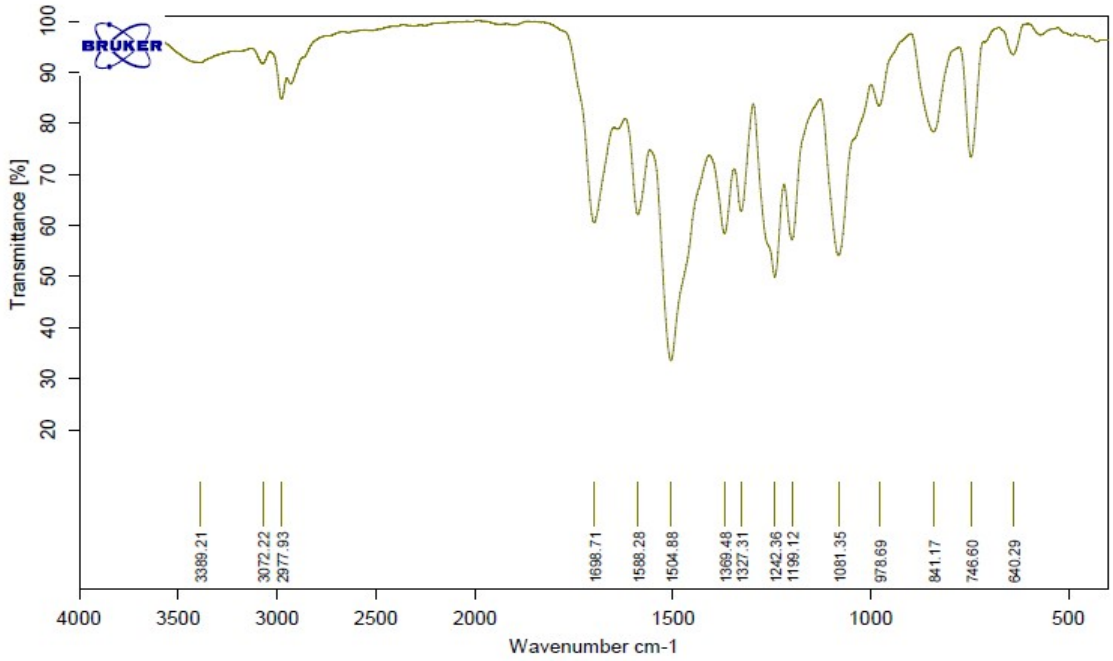


The ^{13}C NMR spectrum of Ethyl-2-methyl-4-(4-chlorophenyl)-4H-pyrimido[2,1-b][1,3]benzothiazole-3-carboxylate

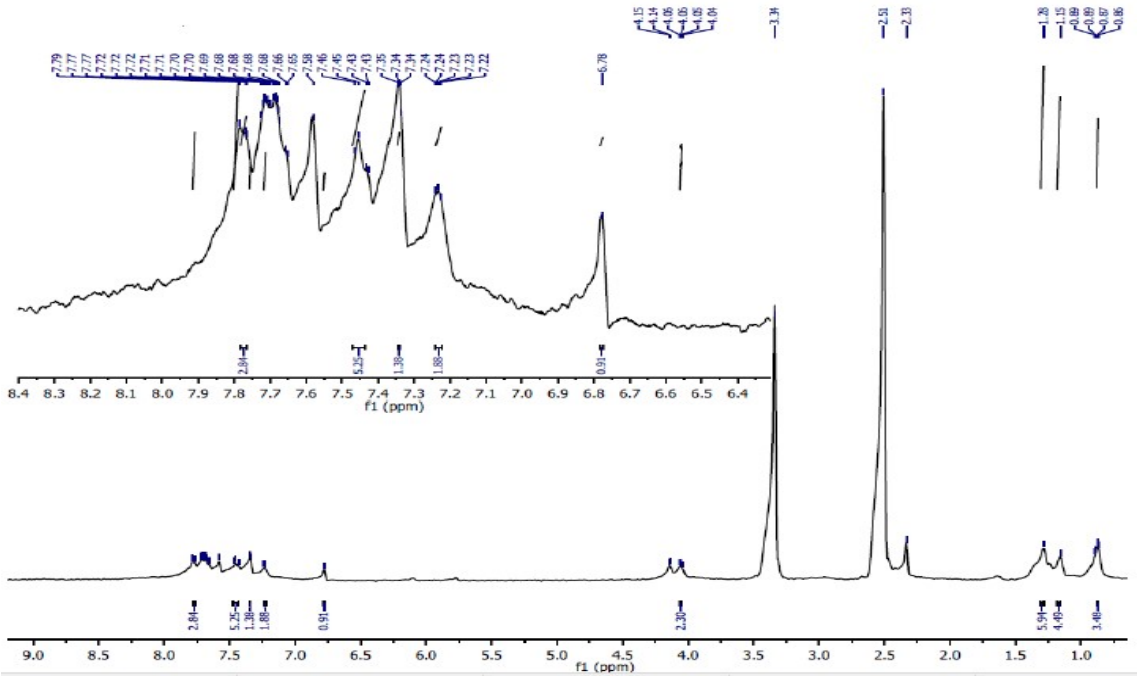
Ethyl-2-methyl-4-(2,4-dichlorophenyl)-4H-pyrimido[2,1-b][1,3]benzothiazole-3-Carboxylate



Yellow solid, Melting point: 132-134 °C. FT-IR (KBr) $\bar{\upsilon}$ (cm^{-1}): 2977, 1698, 1588, 1504, 1369, 1327, 1242, 1199, 1081, 841, 746. ^{1}H NMR (DMSO-d$_6$, 400 MHz): δ 7.69-7.72 (m, 2H), 7.33-7.46 (m, 4H), 7.22 (m, 1H), 6.77 (s, 1H), 4.04-4.06 (q, J= 6.8 Hz, 2H), 2.37 (s, 3H), 1.15 (t, J= 6.8 Hz, 3H).

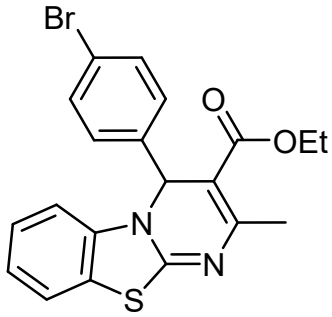


The FT-IR of Ethyl-2-methyl-4-(2,4-dichlorophenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-Carboxylate

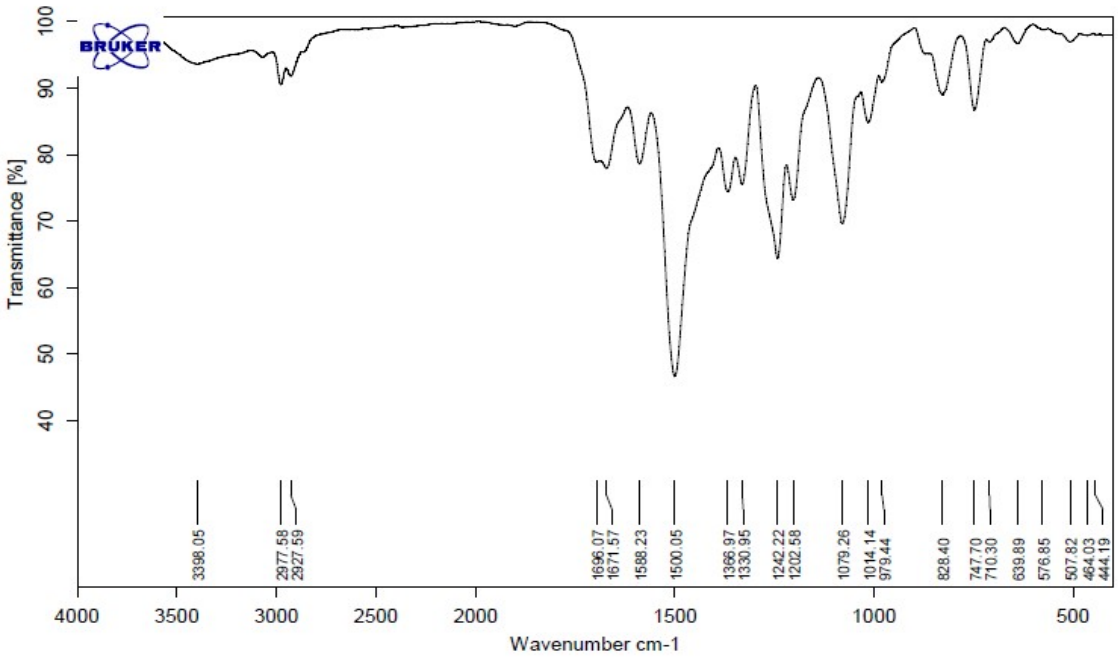


The ^{1}H NMR spectrum of Ethyl-2-methyl-4-(2,4-dichlorophenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-Carboxylate

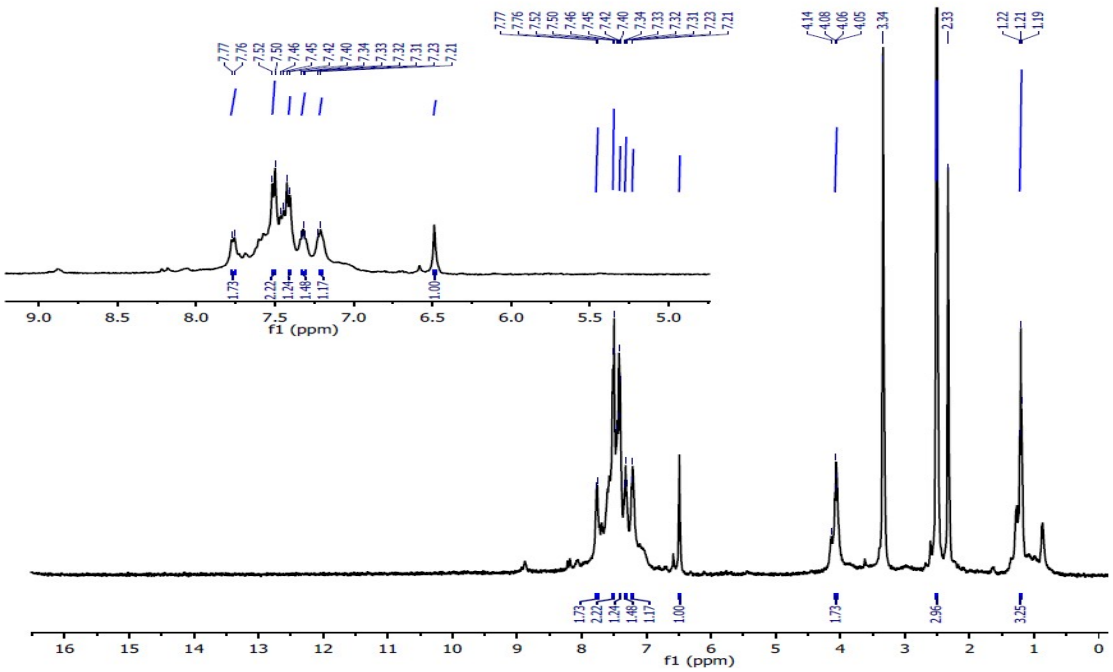
Ethyl-2-methyl-4-(4-bromo phenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-Carboxylate



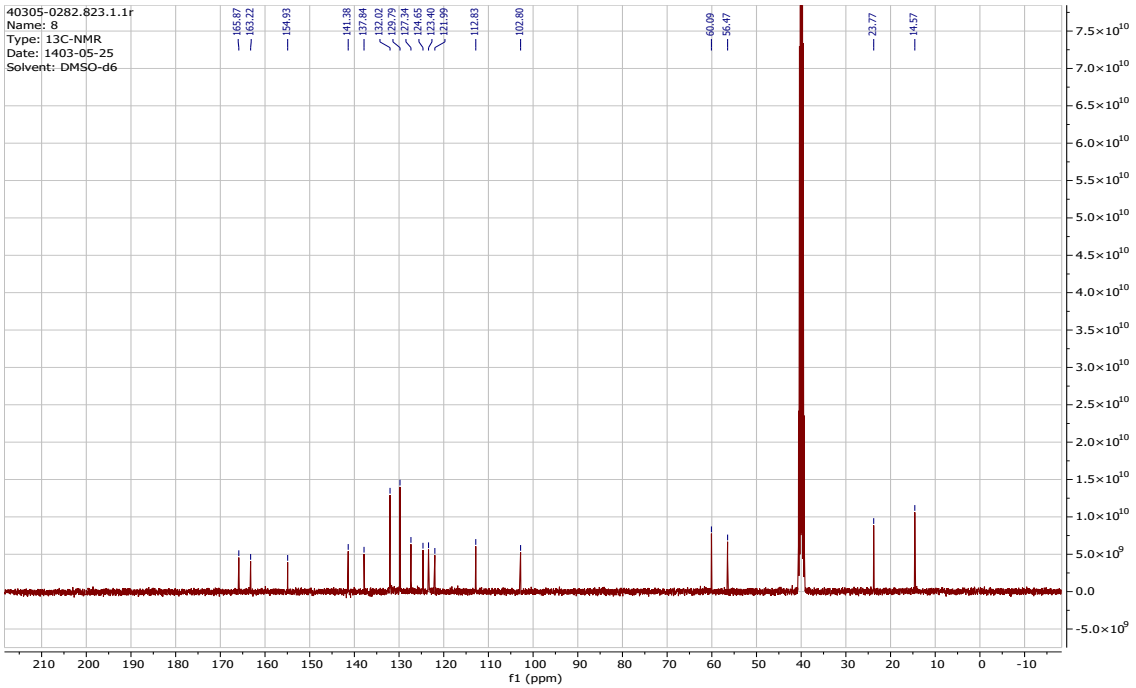
Orange solid, Melting point: 112-115 °C. FT-IR (KBr) ῡ (cm-1): 2977, 1696, 1671, 1588, 1500, 1366, 1270, 1242, 1202, 1079, 828, 747. ^{1}H NMR (DMSO-d_6, 400 MHz): δ 7.75 (d, *J*=6.2 Hz, 1H), 7.40-7.51 (m, 5H), 7.21 (t, *J*= 6 Hz 1H), 6.52 (s, 1H), 4.04-4.08 (t, *J*=7 Hz, 2H), 2.50 (s, 3H), 1.10 (t, *J*= 6.8 Hz, 3H). ^{13}C NMR (100 MHz, DMSO-d_6): 14.57, 23.77, 56.47, 60.09, 102.80, 112.83, 121.99, 123.40, 124.65, 127.34, 129.79, 132.03, 137.84, 141.39,154.95, 163.21, 165.89.



The FT-IR of Ethyl-2-methyl-4-(4-bromo phenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-Carboxylate

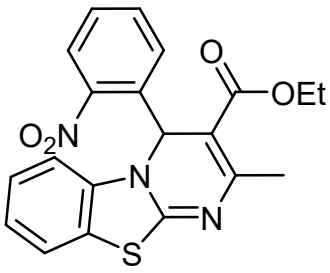


The ^{1}H NMR spectrum of Ethyl-2-methyl-4-(4-bromo phenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-Carboxylate

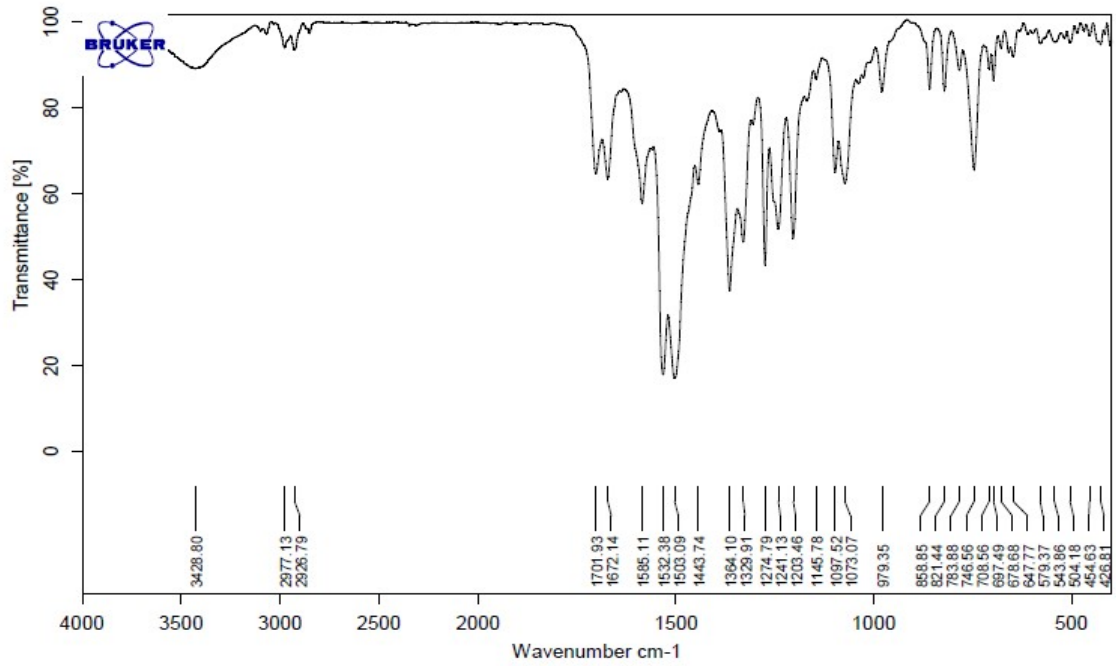


The ^{13}C NMR spectrum of Ethyl-2-methyl-4-(4-bromo phenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-Carboxylate

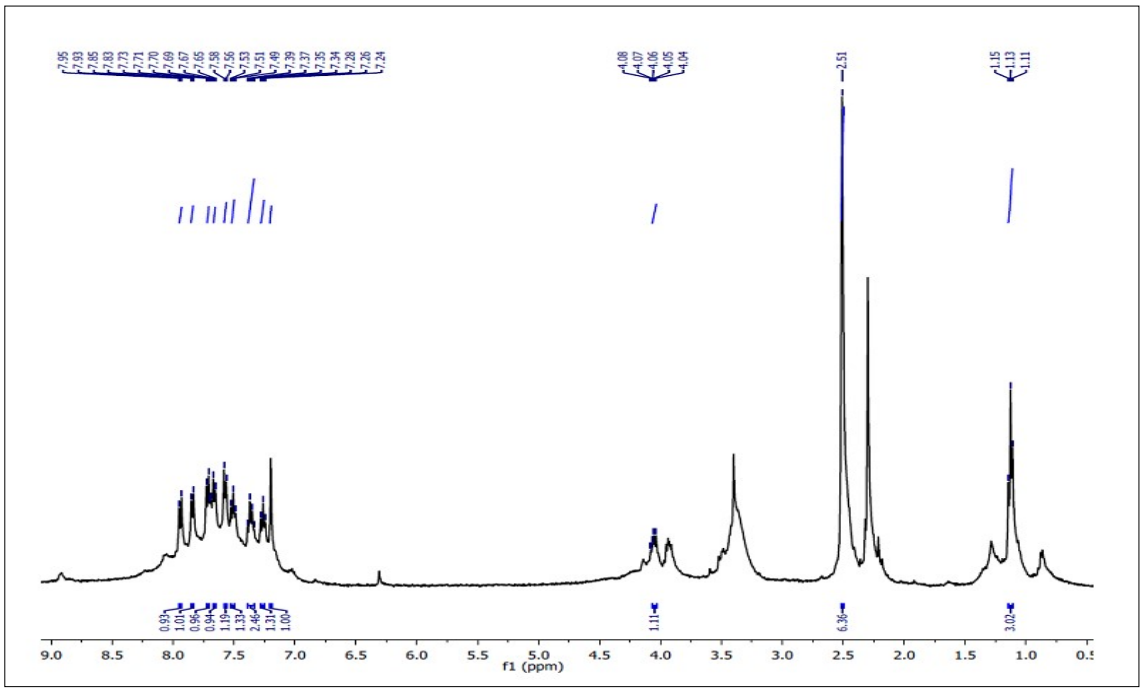
Ethyl-2-methyl-4-(2-nitrophenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-Carboxylate



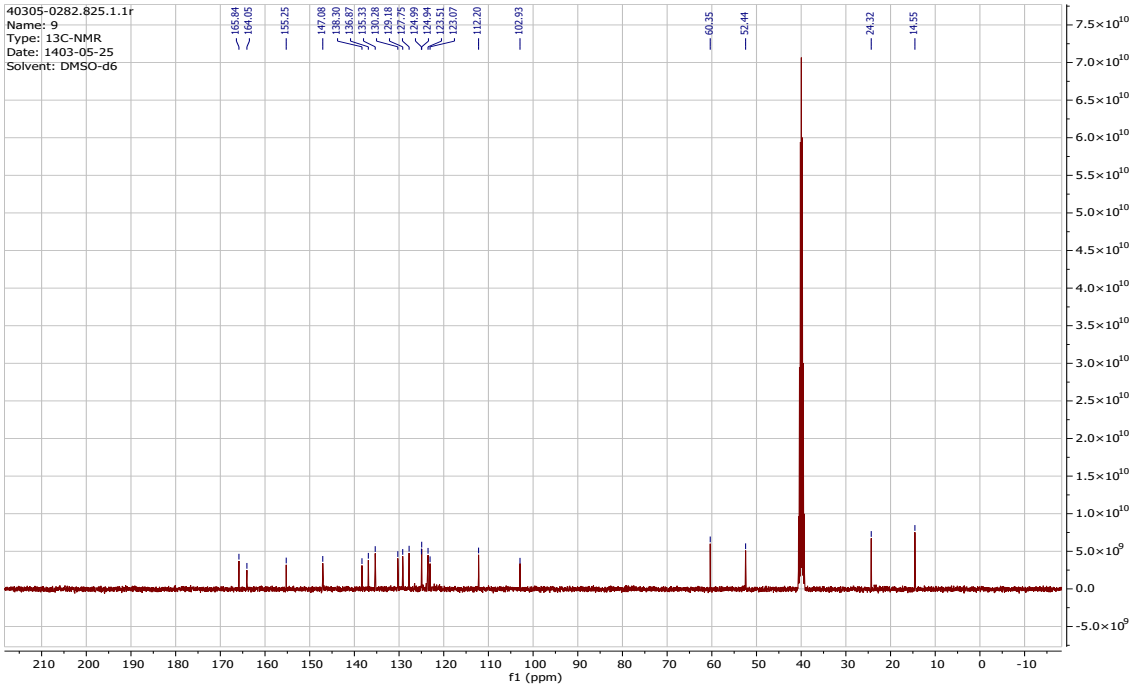
Red orange solid, Melting point: 122-125 °C. FT-IR (KBr) ῡ (cm-1): 2977, 1701, 1672, 1585, 1532, 1503, 1443, 1364, 1329, 1241, 1203, 1097, 746. ^{1}H NMR (DMSO-d_6, 400 MHz): δ 7.9 (d, J = 8 Hz, 1H) ,7.80 (d, J=7.7 Hz, 1H), 7.65-7.72 (m, 2H), 7.56 (d, J=7.5 Hz, 1H),7.48 (t, J=7.6 Hz, 1H) 7.33 (q, 1H), 7.24 (t, J= 8 Hz, 1H), 7.19 (s, 1H), 4.03-4.08 (m, 2H), 2.51 (s, 3H), 1.11 (t, J=6.7 Hz, 3H).^{13}C NMR (100 MHz, DMSO-d_6): 14.57, 24.35, 52.47, 60.39, 102.93, 112.20, 123.07, 123.53, 124.99, 127.75, 129.18, 130.28, 135.33, 136.87, 138.30, 147.09 ,155.25, 164.05, 165.84.



The FT-IR of Ethyl-2-methyl-4-(2-nitrophenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-Carboxylate

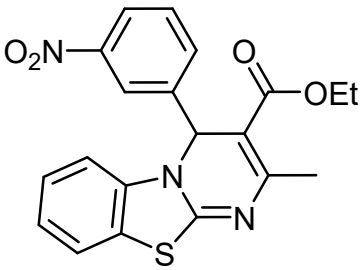


The ^{1}H NMR of Ethyl-2-methyl-4-(2-nitrophenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-Carboxylate

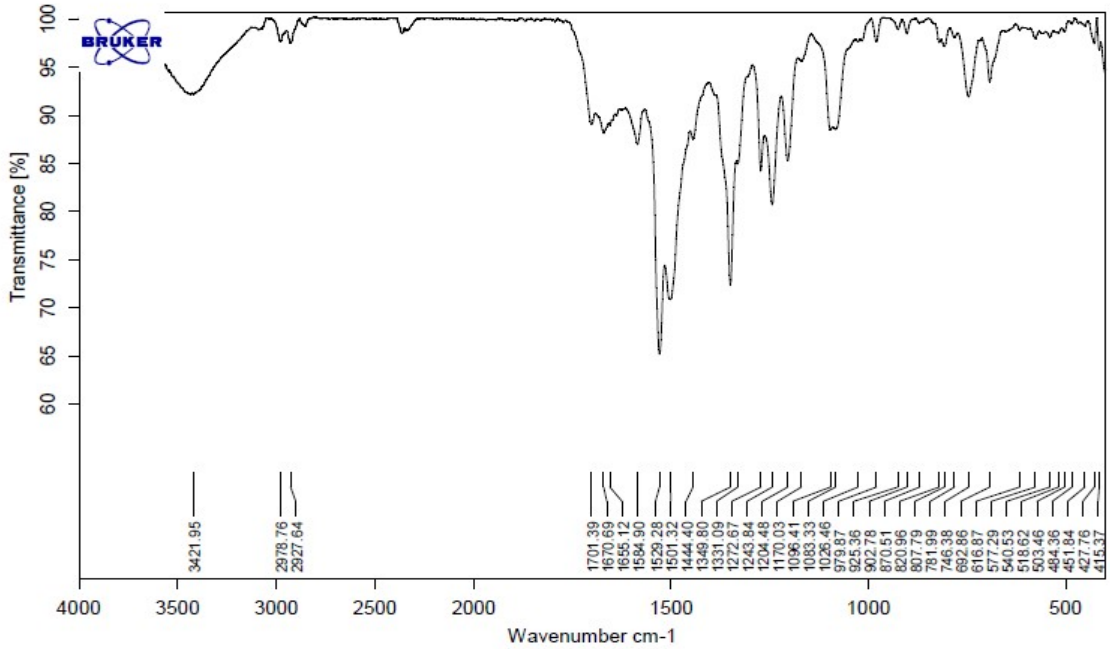


The ^{13}C NMR of Ethyl-2-methyl-4-(2-nitrophenyl)-4H-pyrimido[2,1-b][1,3]benzothiazole-3-Carboxylate

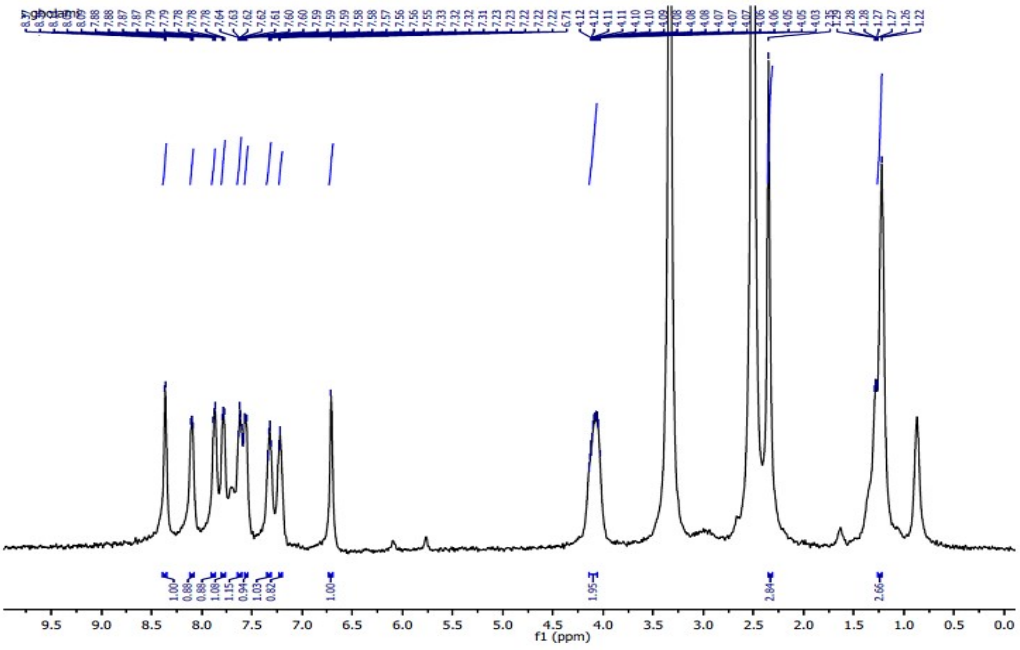
Ethyl-2-methyl-4-(3-nitrophenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate



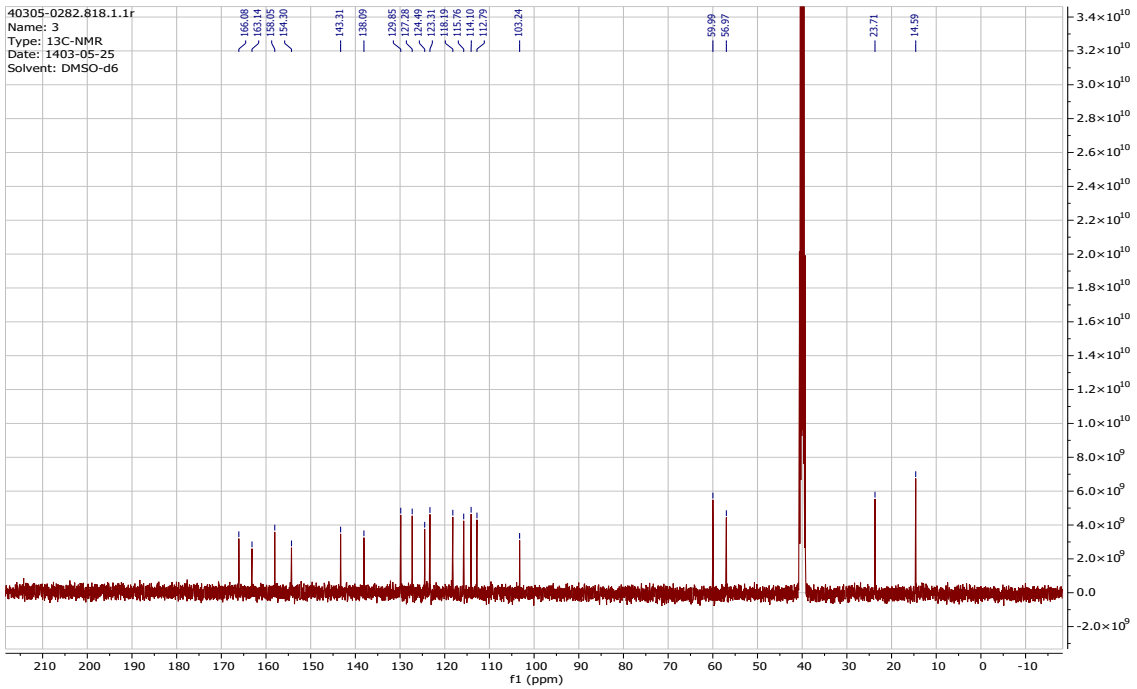
Light yellow solid. Melting point: 223-225 °C. FT-IR (KBr) ῡ (cm-1): 2978, 1701, 1670, 1655, 1529, 1501, 1349, 1272, 1243, 1204, 1096, 746. 1H NMR (DMSO-d6, 400 MHz): δ 8.36 (s, 1H), 8.09 (m, 1H), 7.91 (m, 1H), 7.86 (m, 1H), 7.77 (m, 1H), 7.55 (m, 1H), 7.31 (m, 1H), 7.21 (m, 1H), 6.71 (s, 1H), 4.03-4.14 (m, 2H), 2.35 (s, 3H), 1.22 (m, 3H). ^{13}C NMR (100 MHz, DMSO-d_6): 14.59, 23.75, 56.97, 59.99, 103.23, 112.80,114.10, 115.76, 118.19, 123.31, 124.49, 127.28, 129.85, 138.09, 143.31, 154.30, 158.05, 163.14, 166.08.



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

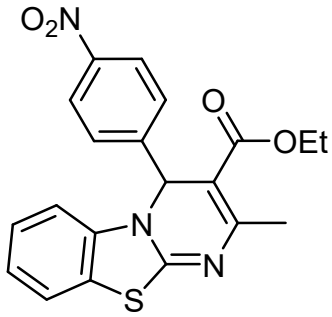


The ^{1}H NMR spectrum of Ethyl-2-methyl-4-(3-nitrophenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate

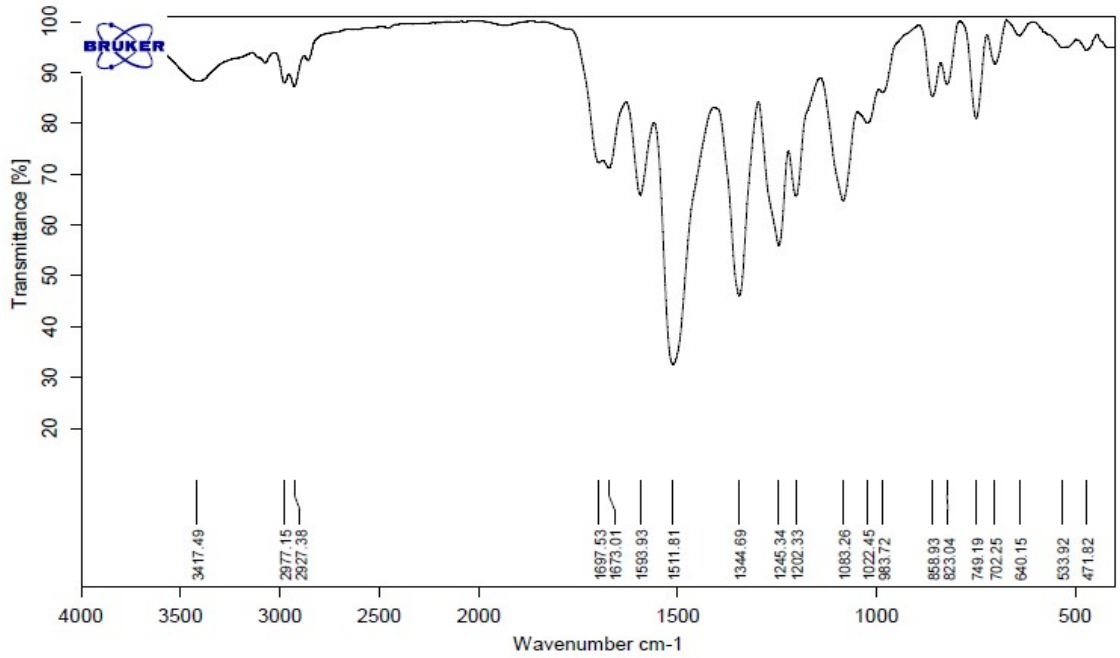


The ^{13}C NMR spectrum of Ethyl-2-methyl-4-(3-nitrophenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate

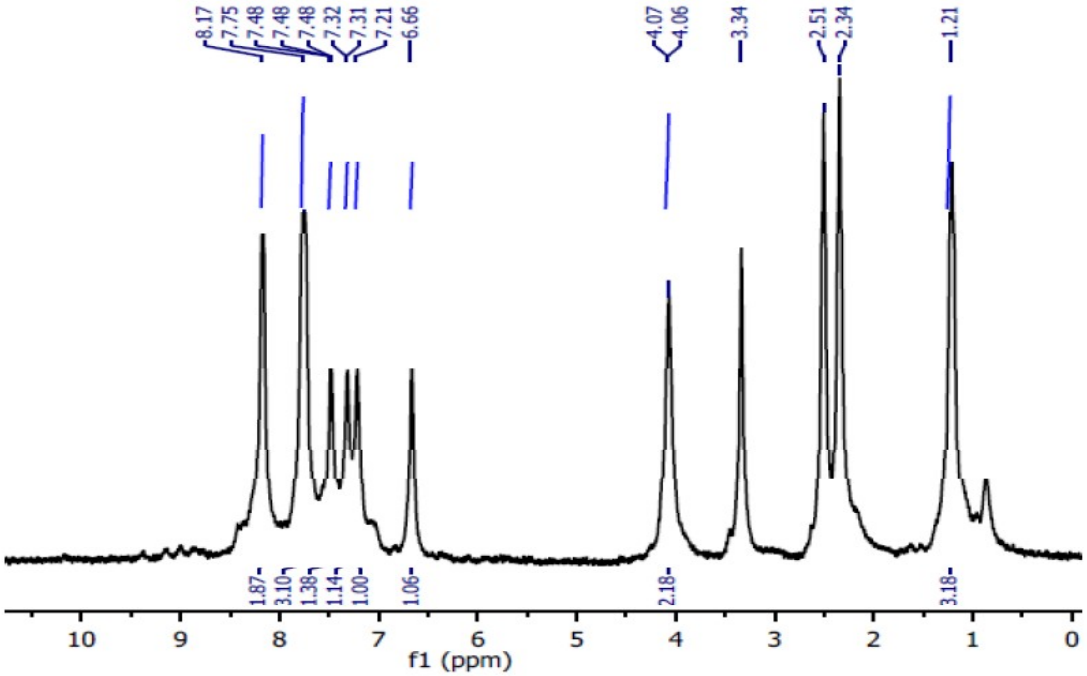
Ethyl-2-methyl-4-(4-nitrophenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate



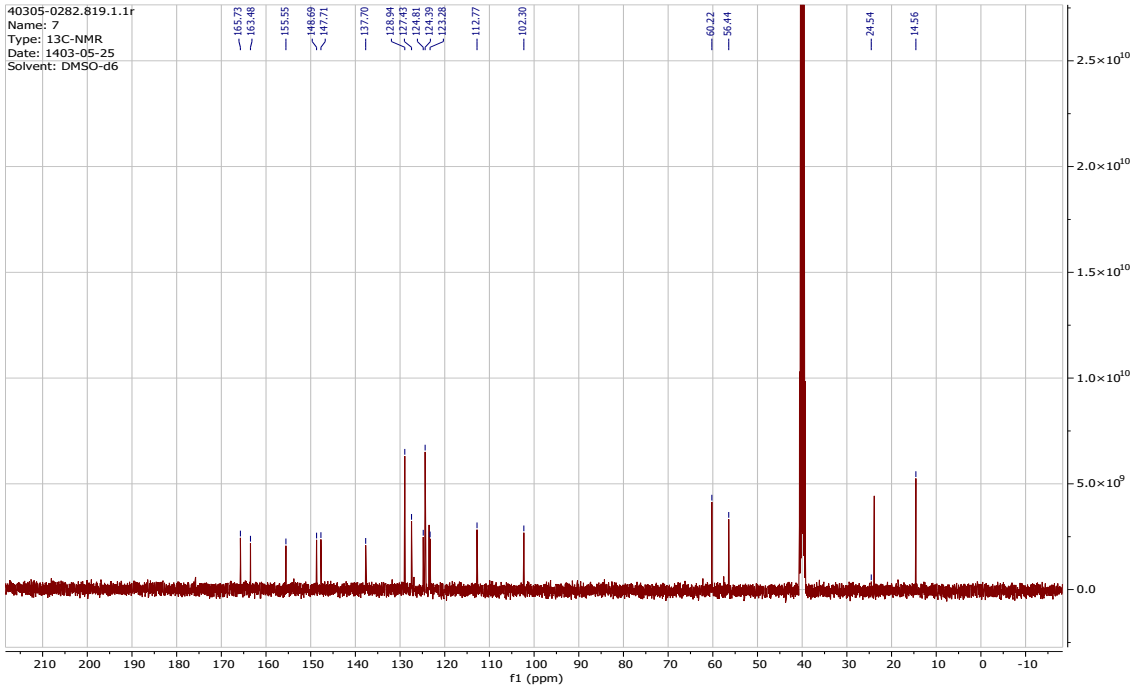
Yellow solid, Melting point: 172-174 °C. FT-IR (KBr) $\bar{\upsilon}$ (cm^{-1}): 2977, 1697, 1673, 1593, 1511, 1344, 1245, 1202, 749^{1}H NMR (DMSO-d6, 400 MHz): δ 8.17 (br. s, 2H), 7.75 (br. s, 3H), 7.47 (br. s, 1H), 7.31 (br. s, 1H), 7.21 (br. s, 1H), 6.66 (s, 1H), 4.06 (br. s, 2H), 2.37 (s, 3H), 1.21 (br. s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6): 14.56, 24.51, 56.47, 60.22, 102.30, 112.77, 123.28, 124.39, 124.81, 127.43, 128.94, 137.70, 147.71, 148.69, 155.55, 163.48, 165.77.



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

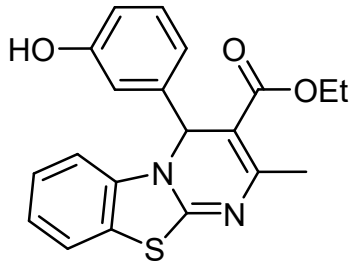


The ^{1}H NMR spectrum of Ethyl-2-methyl-4-(4-nitrophenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate

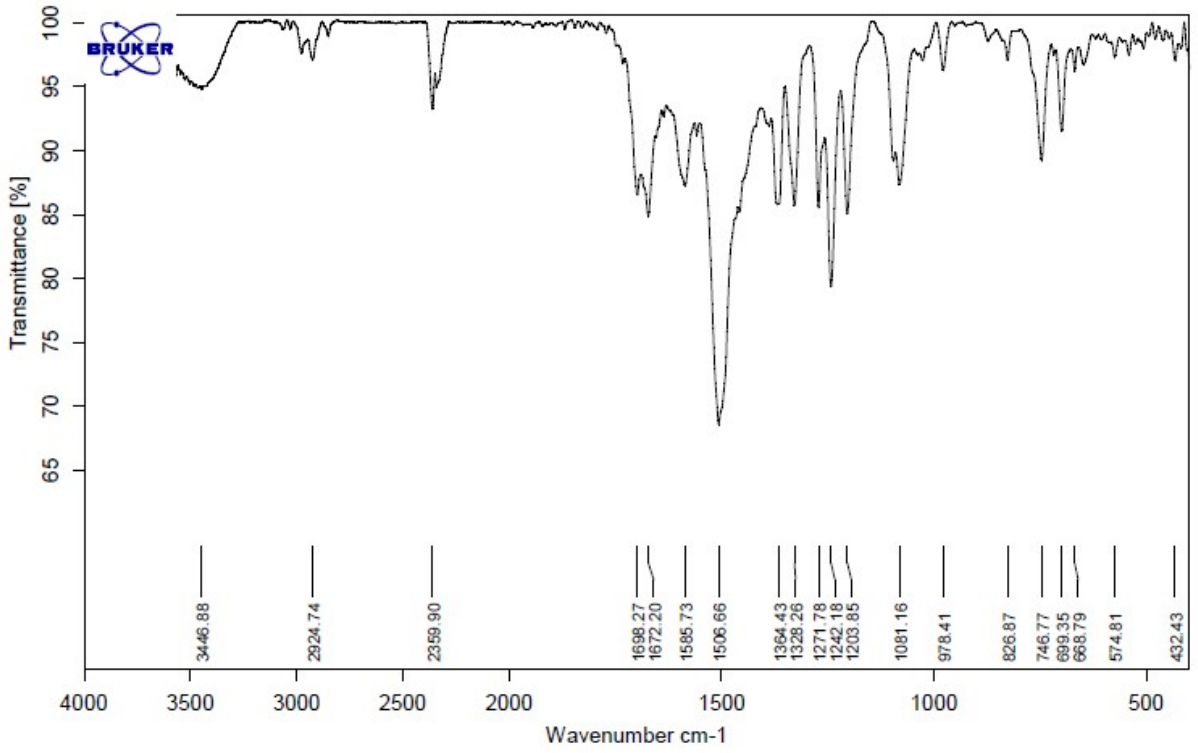


The ^{13}C NMR spectrum of Ethyl-2-methyl-4-(4-nitrophenyl)-4H-pyrimido[2,1-b][1,3]benzothiazole-3-carboxylate

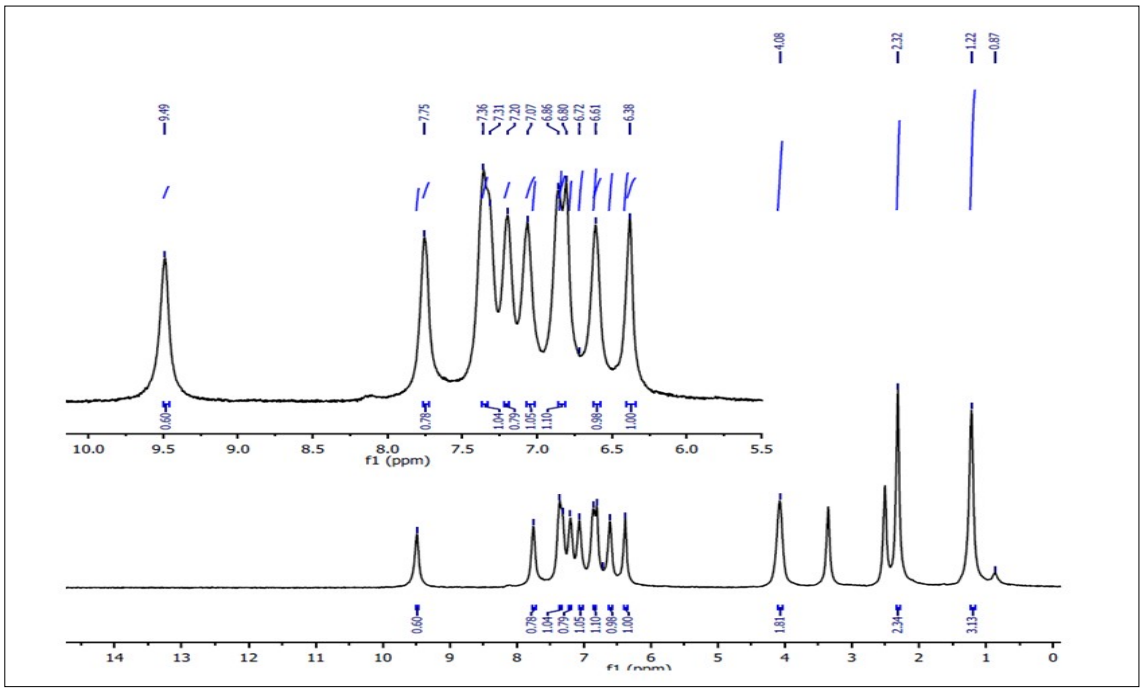
Ethyl-2-methyl-4-(3-hydroxy phenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate



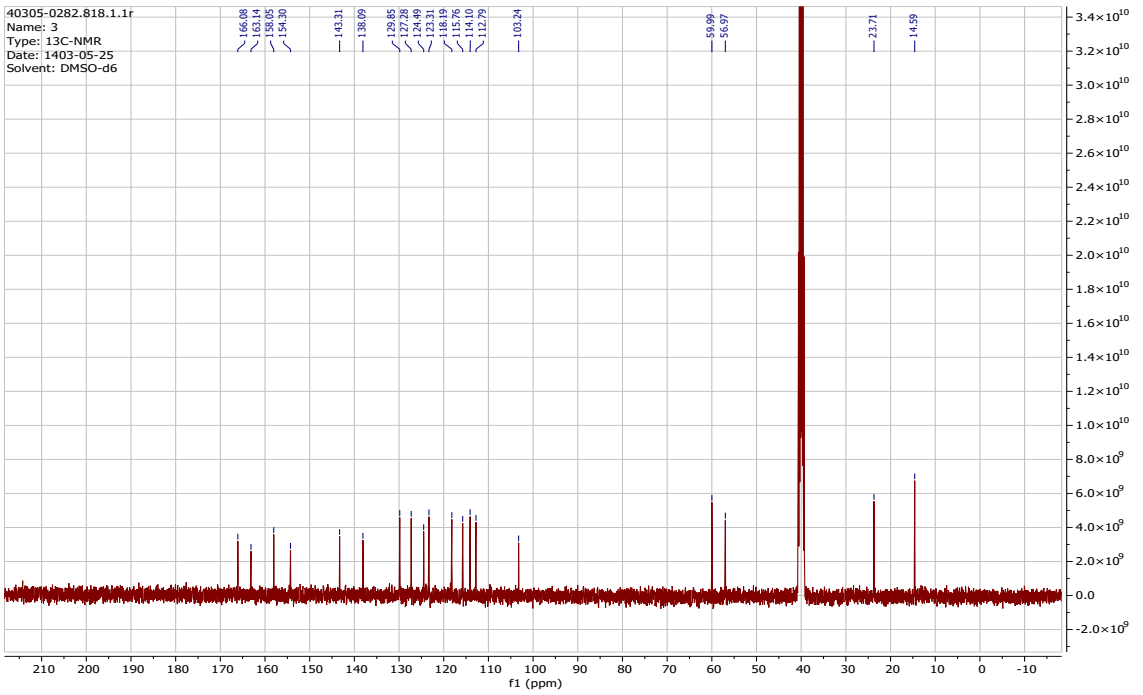
Yellow solid. Melting point: 260-262 °C. FT-IR (KBr) ῡ (cm^{-1}): 3446, 2970, 2924, 1698, 1672, 1506, 1364, 1271, 1242, 1203, 1081, 746. ^{1}H NMR (DMSO-d6, 400 MHz): δ 9.4 (s, 1H), 7.75 (s, 1H), 7.35 (m, 2H), 7.32 (m, 1H), 7.19 (s, 1H), 7.0 (s, 2H),6.80 (m, 1H), 6.61 (s, 1H), 6.38 (s, 1H), 4.07 (m, 2H), 2.31 (s, 3H), 1.21 (br. s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6): 14.59, 23.71, 56.97, 59.99, 103.24, 112.79, 114.10, 115.76, 118.19, 123.31, 124.49, 127.28, 129.85, 138.09, 143.31, 154.30, 158.02, 163.14, 166.08.



The FT-IR of Ethyl-2-methyl-4-(3-hydroxy phenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate

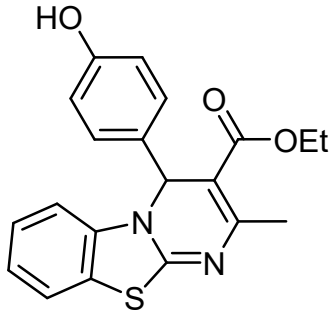


The ^{1}H NMR spectrum of Ethyl-2-methyl-4-(3-hydroxy phenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate

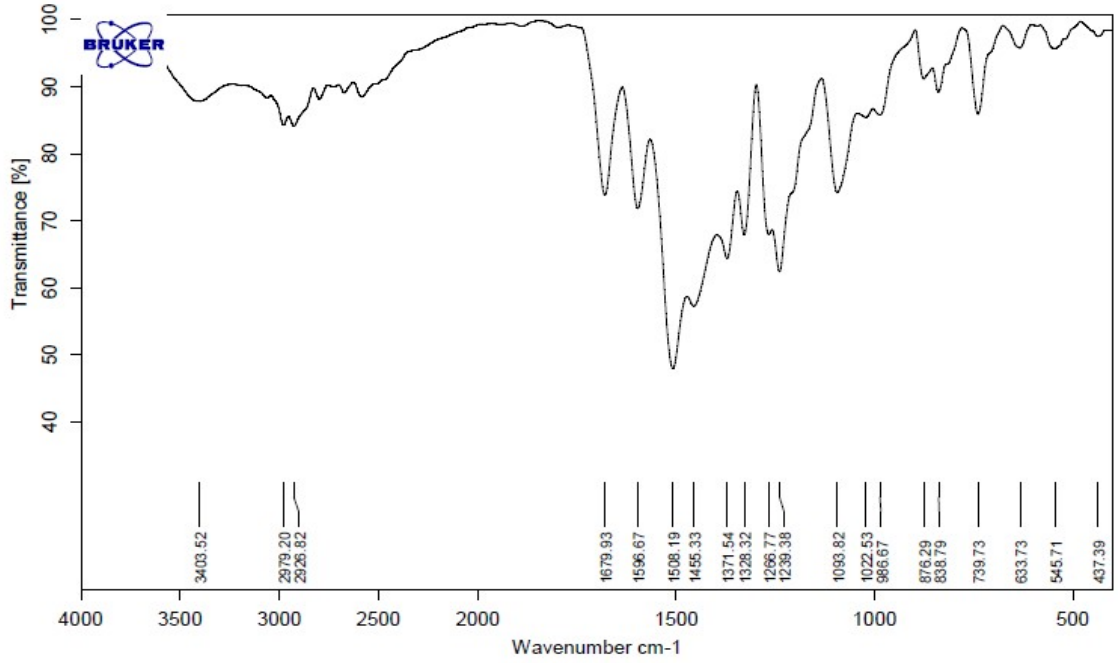


The ^{13}C NMR spectrum of Ethyl-2-methyl-4-(3-hydroxy phenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate

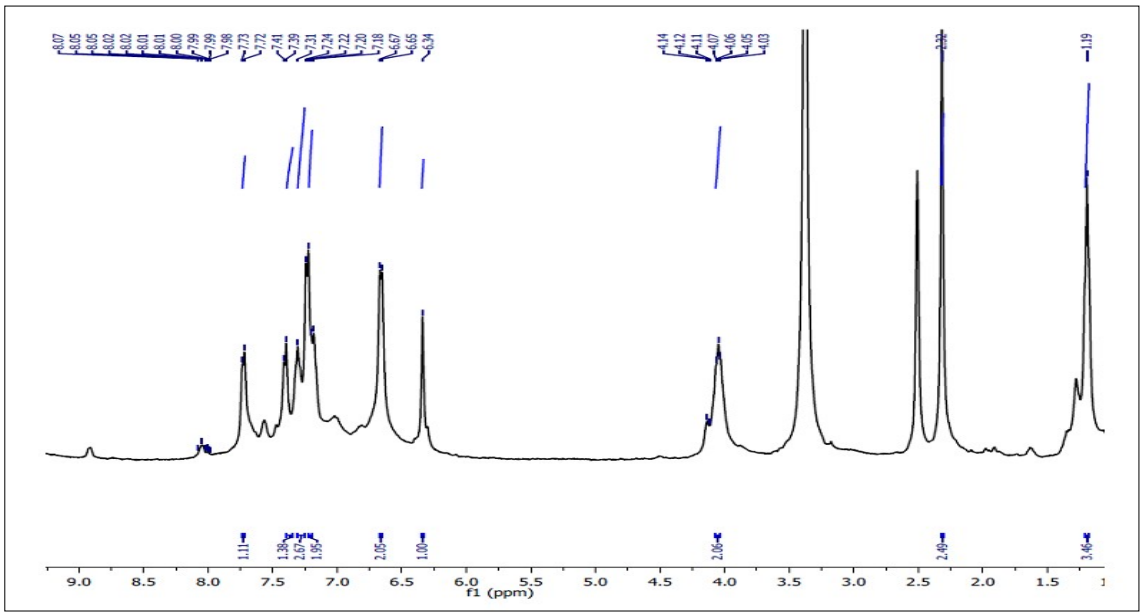
Ethyl-2-methyl-4-(4-hydroxy phenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate



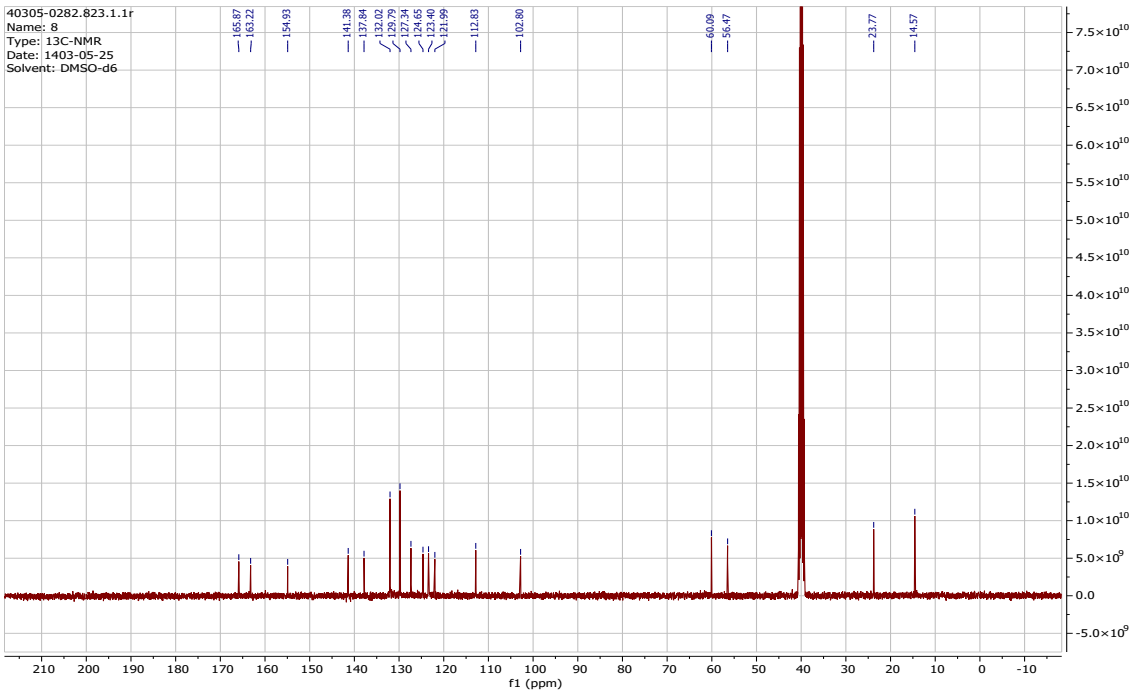
Pale yellow solid, Melting point: 210-212 ◦C. FT-IR (KBr) ῡ (cm^{-1}): 3403, 2926, 1679, 1596, 1508, 1455, 1371, 1266, 1239, 1093, 986, 838. ^{1}H NMR (DMSO-d6, 400 MHz): δ 9.50 (s, 1H), 7.71 (d, *J*=6.2 Hz, 1H), 7.18-7.41 (m, 5H), 6.65 (d, *J*=7.7 Hz, 2H), 6.33 (s, 1H), 4.03-4.14 (m, 2H), 2.31 (s, 3H), 1.19 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6): 14.57, 23.77, 56.47, 60.09, 102.80, 112.83, 121.99, 123.40, 124.65, 127.34, 129.79, 132.09, 137.84, 141.38, 154.93, 163.22, 165.87.



The FT-IR of Ethyl-2-methyl-4-(4-hydroxy phenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate

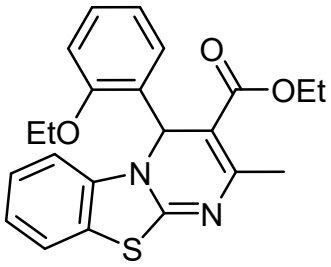


The ^{1}H NMR spectrum of Ethyl-2-methyl-4-(4-hydroxy phenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate

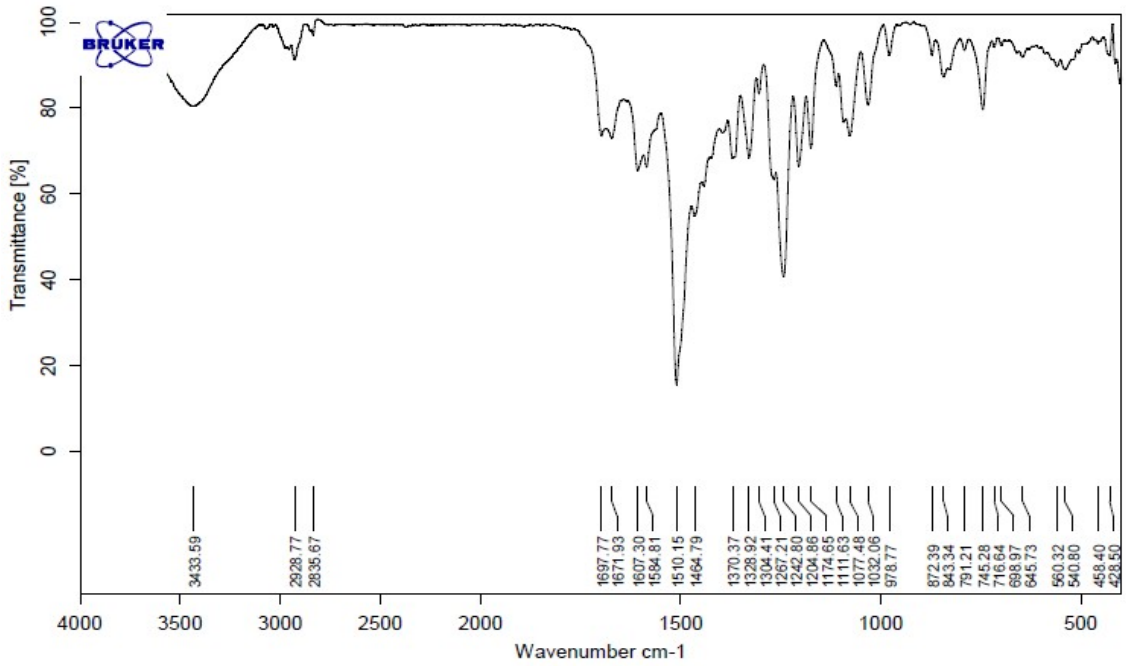


The ^{13}C NMR spectrum of Ethyl-2-methyl-4-(4-hydroxy phenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate

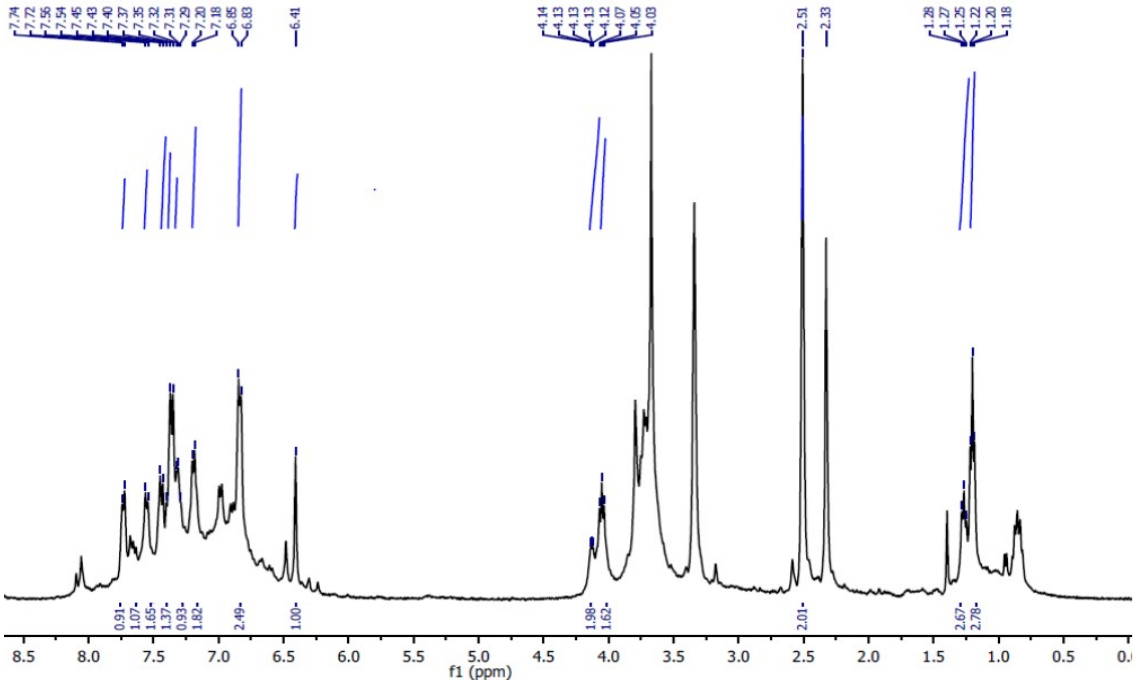
Ethyl-2-methyl-4-(2-ethoxy phenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate



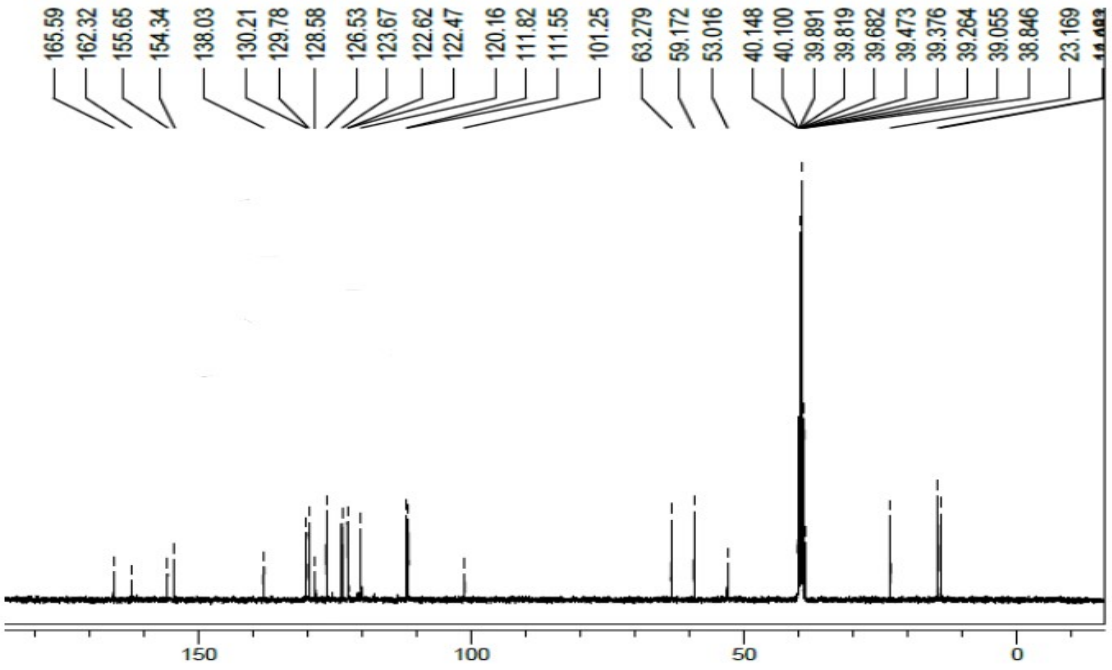
Yellow solid, Melting point: 173-176 °C. FT-IR (KBr) ῡ (cm^{-1}): 2928, 1697, 1607, 1510, 1464, 1370, 1242, 1204, 1077, 1032, 745. ^{1}H NMR (DMSO-d6, 400 MHz): δ 7.72 (d, *J*=7.4 Hz, 1H), 7.54 (d, *J*=8 Hz, 1H), 7.22-744 (m, 4H), 6.82 (d, *J*=8 Hz, 2H), 6.40 (s, 1H), 4.11-4.13 (m, 2H), 4.03 (t, *J*=7 Hz, 2H), 2.32 (s, 3H), 1.24 (t, *J*=6.8 Hz, 3H), 1.18 (t, *J*=7 Hz 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.



The FT-IR of Ethyl-2-methyl-4-(2-ethoxy phenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate

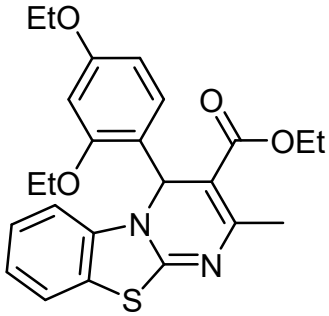


The ^{1}H NMR spectrum of Ethyl-2-methyl-4-(2-ethoxy phenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate

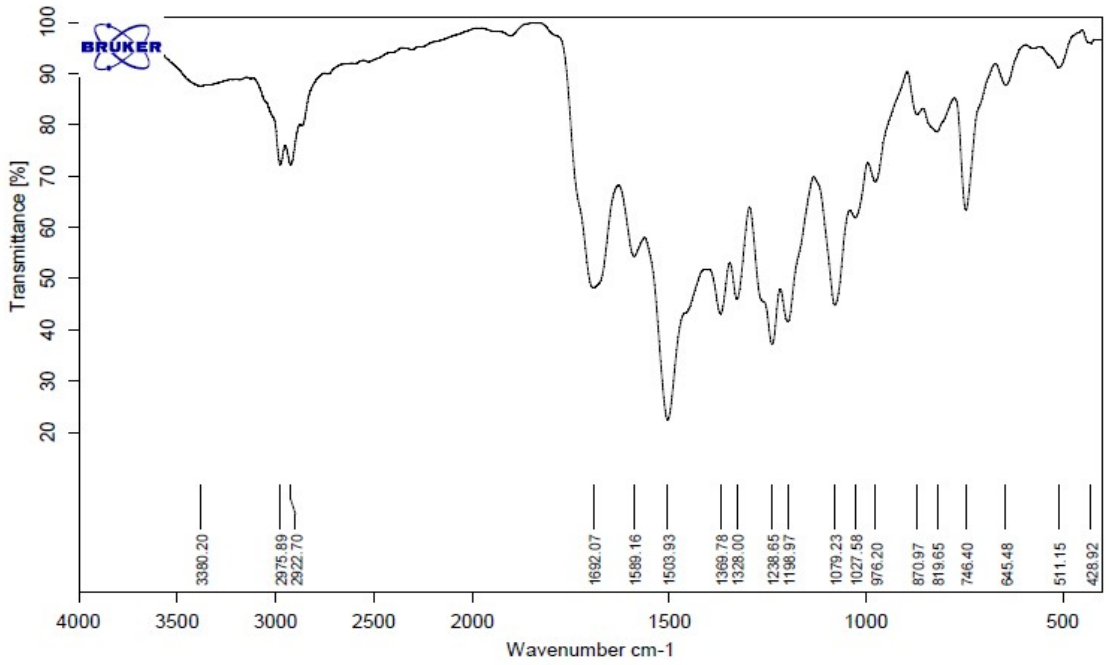


The ^{13}C NMR spectrum of Ethyl-2-methyl-4-(2-ethoxy phenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate

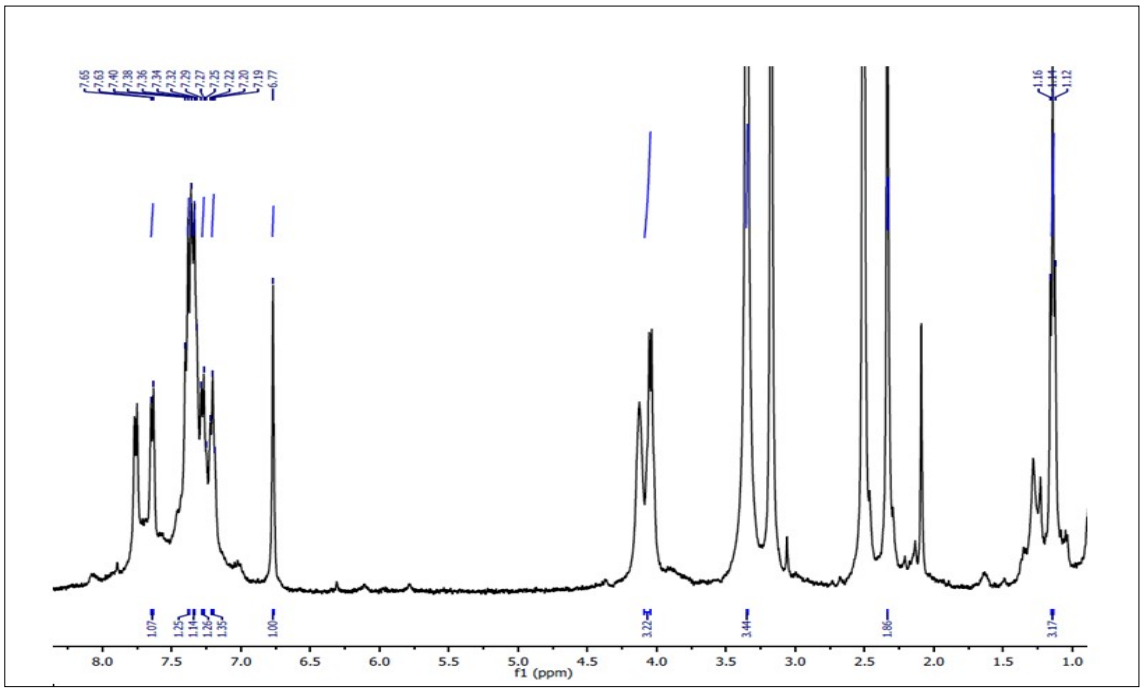
Ethyl-2-methyl-4-(2,4- ethoxyphenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate



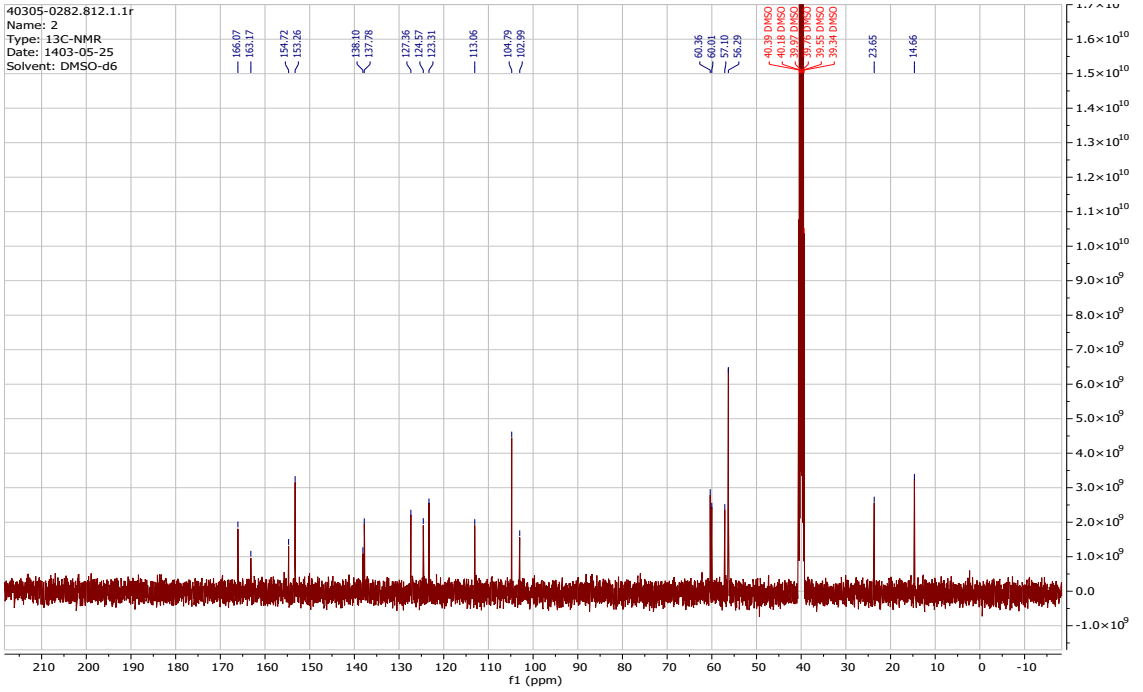
Yellow solid, Melting point: 165-167 °C. FT-IR (KBr) ῡ (cm^{-1}): 2975, 2922, 1692, 1589, 1503, 1328, 1238, 1198, 1079, 819, 746. . ^{1}H NMR (DMSO-d6, 400 MHz): δ 7.62 (d, J=7.6 Hz, 1H), 7.45 (d, J= 8 Hz, 1H), 7.37 (d, J = 8.4 Hz, 1H), 7.30 (td, J = 8 Hz, 1.2 Hz, 1H), 7.15 (td, J = 7.6 Hz, 1.2 Hz, 1H), 6.68(s, 1H), 6.44-6.48 (m, 2H), 4.05 (q, J = 6.8 Hz, 2H), 3.93 (s, 3H), 3.71 (s, 3H), 2.36 s, 3H), 1.19 (t, J = 6.8 Hz, 3H). ^{13}C NMR (DMSO-d_6, 100 MHz) /δ ppm: 14.66, 23.66, 56.29, 57.10, 60.01, 60.35, 102.99, 104.79, 113.06, 123.31, 124.57, 127.36, 137.78, 138.10, 153.26, 154.72, 163.17, 166.07.



The FT-IR of Ethyl-2-methyl-4-(2,4- ethoxyphenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate

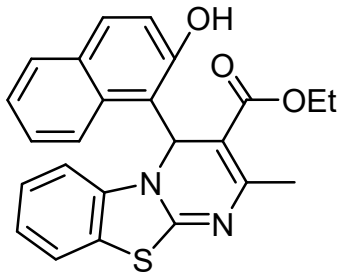


The ^{1}H NMR spectrum of Ethyl-2-methyl-4-(2,4- ethoxyphenyl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate

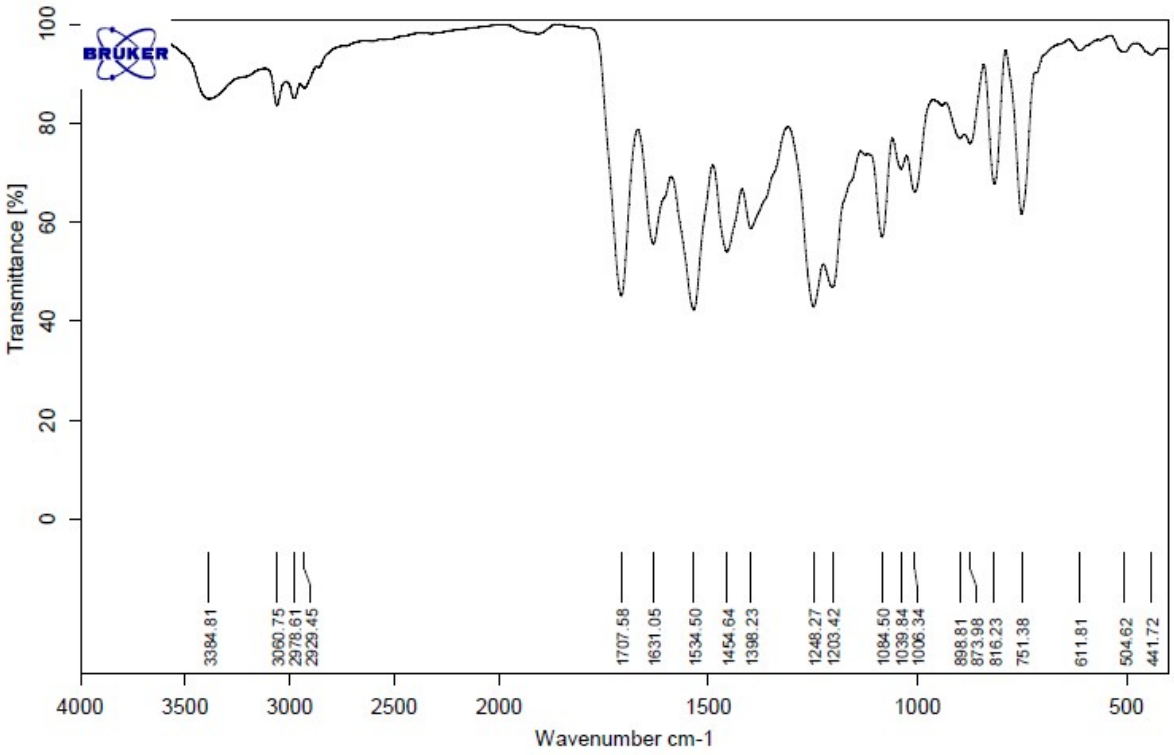


The ^{13}C NMR spectrum of Ethyl-2-methyl-4-(2,4- ethoxyphenyl)-4H-pyrimido[2,1-b][1,3]benzothiazole-3-carboxylate

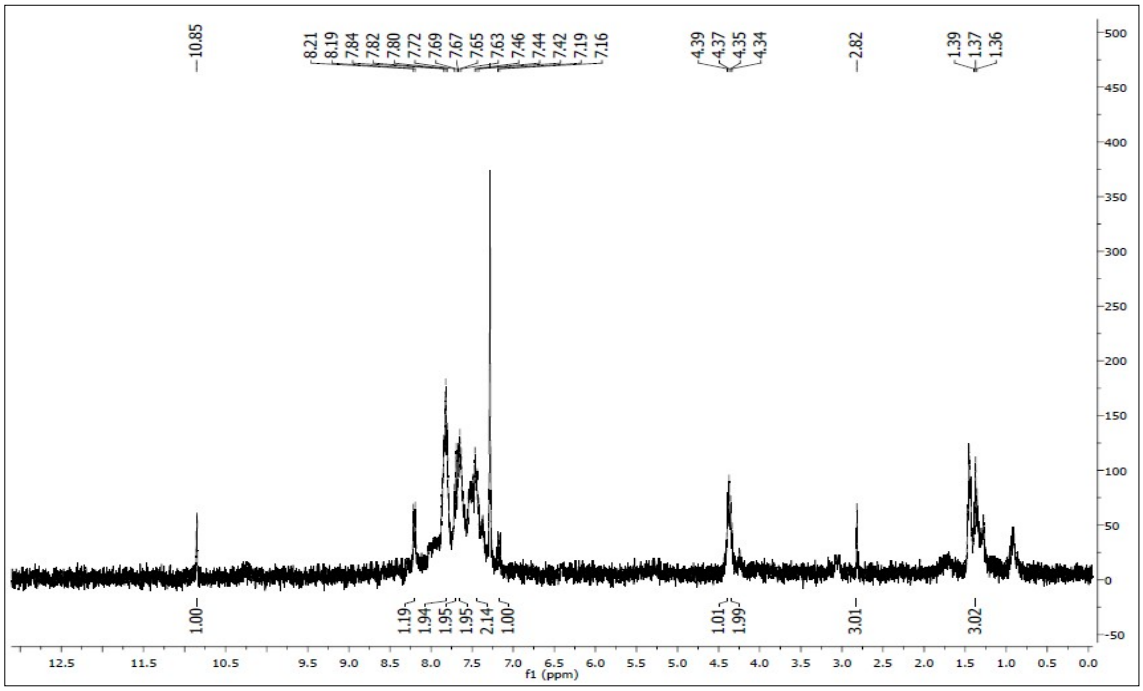
Ethyl-2-methyl-4-(2-hydroxynaphthalen-1-yl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate



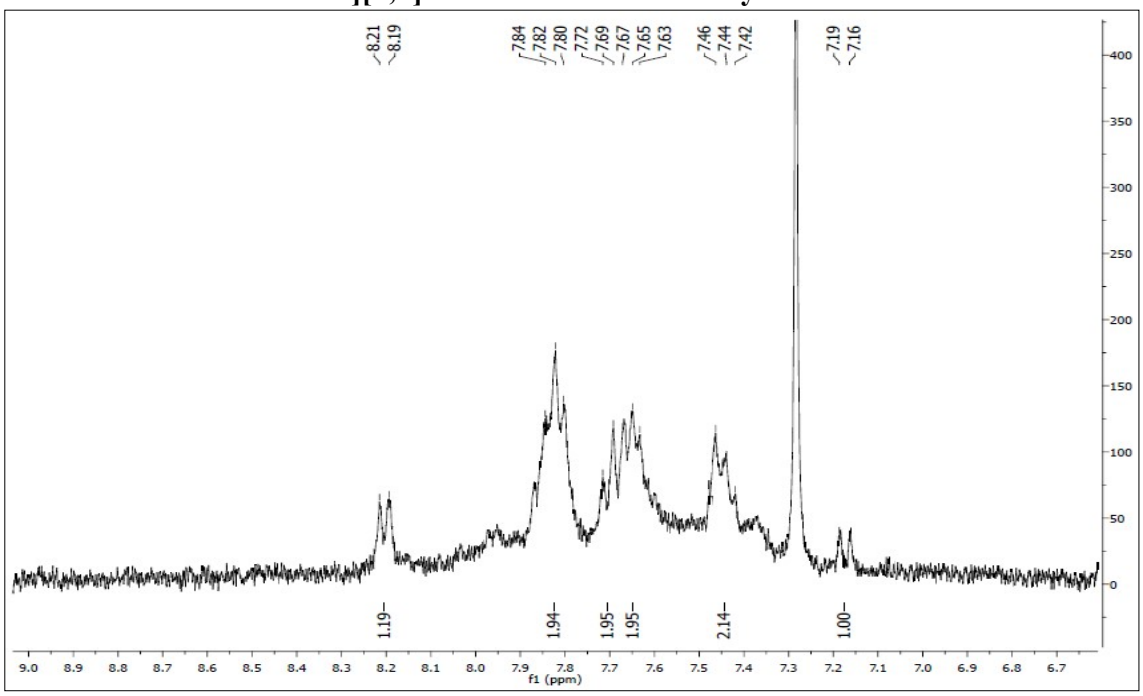
Pale Brown solid, Melting point: 220-222 °C. FT-IR (KBr) $\bar{\upsilon}$ (cm^{-1}): 3094, 3060, 2978, 1707, 1631, 1534, 1454, 1398, 1248, 1203, 1084, 1039, 816, 751. ^{1}H NMR ($CDCl_3$-d_6, 400 MHz): δ 10.85 (s, 1H), 8.19 (m, 1H), 7.82 (m, 2H), 7.69 (m, 2H), 7.65 (m, 2H), 7.4 (m, 2H), 7.1 (m, 1H), 4.38 (s, 1H), 4.34 (s, 2H), 2.82 (s, 3H), 1.37 (s, 3H). ^{13}C NMR ($CDCl_3$-d_6, 100 MHz) /δ ppm: 14.40, 30.37, 61.22, 62.06, 116.57, 117.37, 119.00, 119.32, 121.26, 121.77, 121.86, 124.32, 124.88, 126.04, 128.90, 129.27, 132.22, 133.38, 136.36, 143.38, 152.85, 156.26, 164.39, 165.25.



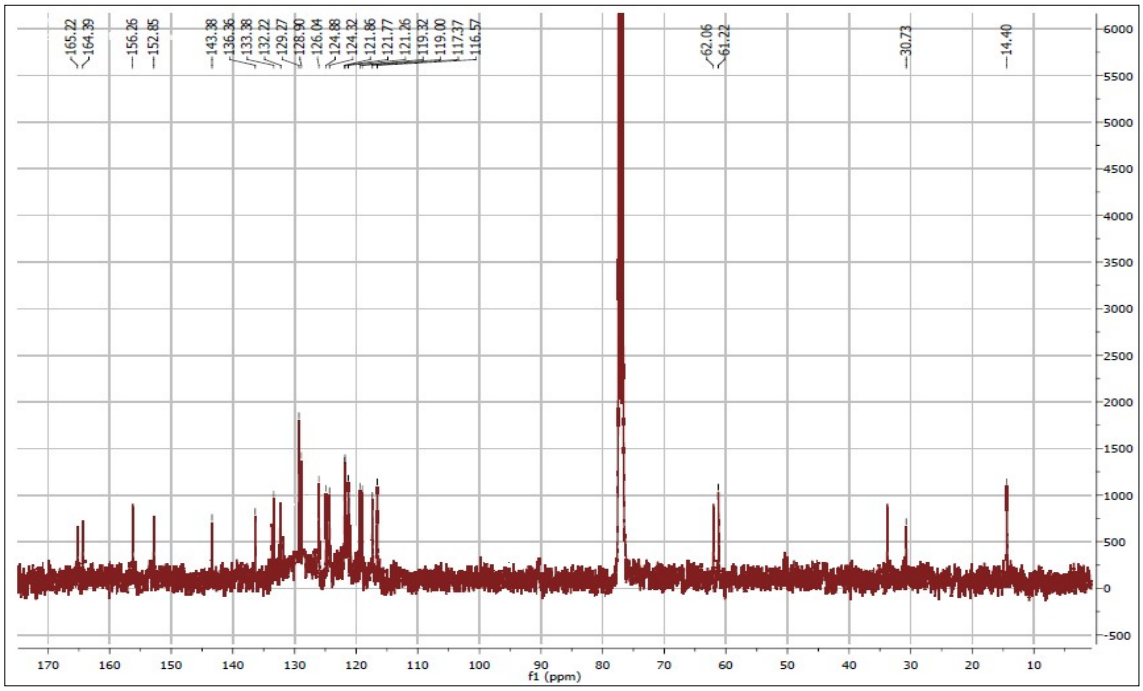
The FT-IR of Ethyl-2-methyl-4-(2-hydroxynaphthalen-1-yl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate



The ^{1}H NMR of Ethyl-2-methyl-4-(2-hydroxynaphthalen-1-yl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate



The ^{1}H NMR of Ethyl-2-methyl-4-(2-hydroxynaphthalen-1-yl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate



The ^{13}C NMR of Ethyl-2-methyl-4-(2-hydroxynaphthalen-1-yl)-4*H*-pyrimido[2,1-*b*][1,3]benzothiazole-3-carboxylate