SUPPORTING INFORMATION SECTION

Vilsmeier reagent mediated synthesis of 6-[(formyloxy)methyl]-pyrazolopyrimidines via one-pot multiple tandem reaction

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General Procedures

All chemicals were reagent grade and used as purchased. All reactions were carried out under nitrogen atmosphere and monitored by TLC analysis. Dichloromethane, ethyl acetate and hexanes were purchased from Mallinckrodt Chemical Company. The following compounds were purchased from Acoros Chemical Company: benzoylchlorid, chloroacetylchloride, formamide, oxalylchloride, phoshophourstribromid and *p*-toluenesulfonyl chloride. Phosphorylchloride was purchased from Ferak Chemical Co. Thionyl chloride was purchased from Merck Reagents. Purification by gravity column chromatography was carried out by use of Merck Reagents Silica Gel 60 (particle size 0.063–0.200 mm, 70–230 mesh ASTM). Cesium formate and iodoacetylchloride were purchased from Sigma-Aldrich Chemical Company. Commercially available reagents were used without further purification unless otherwise noted. ^{1}H NMR were recorded at 200, 400, or 500 MHz and ^{13}C NMR recorded at 50, 100, or 125 MHz, respectively, in $CDCl_3$, CH_3OD, and DMSO-d_6 as solvent. The standard abbreviations s, d, t, q, and m refer to singlet, doublet, triplet, quartet, and multiplet, respectively. Coupling constant (*J*), whenever discernible, have been reported in Hz. Infrared spectra (IR) were recorded as neat solutions or solids; and mass spectra were recorded using electron impact or electrospray ionization techniques. The wavenumbers reported are referenced to the polystyrene 1601 cm^{-1} absorption. High-resolution mass spectra were obtained by means of a JEOL JMS-HX110 mass spectrometer.

Standard procedure for synthesis of 5-(2-haloacetylamino)pyrazoles 1–16 and 33–35.[1] 1-Aryl-3-aryl-5-aminopyrazoles (1.0 equiv) was dissolved in CH_2Cl_2 (10 mL) and stirred in ice-bath. 2-Chloroacetyl chloride, 2-chloroacetyl bromide, or 2-iodoroacetyl chloride (1.2 equiv) in 10 mL of CH_2Cl_2 or THF were slowly added to the reaction mixture at 0 °C under N_2, respectively. The reaction was stirred at 0–10 °C for 3–4 h. The reaction mixture was washed with water (10 mL) and saturated aqueous $NaHCO_3$ (10 mL × 2). The organic layer was dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel to give the corresponding acylation product 5-(2-haloacetylamino)pyrazoles **1**–**6** and **33–35** in 73–87% yields.

5-(2-Chloroacetylamino)-1-phenyl-3-phenylpyrazole (1). ^{1}H NMR ($CDCl_3$, 200 MHz) δ 4.18 (s, 2 H, CH_2), 7.10 (s, 1 H), 7.23–7.55 (m, 8 H, ArH), 7.86 (dd, 2 H, J = 8.0, 4.0 Hz), 8.63 (br, 1 H, N–H).

5-(2-Chloroacetylamino)-1-(2-methylphenyl)-3-phenylpyrazole (2). ^{1}H NMR ($CDCl_3$, 200 MHz) δ 2.38 (d, J = 7.6 Hz, 3 H, CH_3), 4.10 (s, 2 H, CH_2), 6.96 (s, 1 H), 7.38–7.44 (m, 9 H, ArH).

5-(2-Chloroacetylamino)-1-(3-methylphenyl)-3-phenylpyrazole (3). ^{1}H NMR ($CDCl_3$, 200 MHz) δ 2.13 (s, 3 H, CH_3), 4.04 (s, 2 H, CH_2), 7.31–7.47 (m, 8 H, ArH), 7.91 (d, J = 3.0 Hz, 2 H, ArH), 8.82 (br, 1 H, N–H).

5-(2-Chloroacetylamino)-1-(4-methylphenyl)-3-phenylpyrazole (4). ^{1}H NMR ($CDCl_3$, 200 MHz) δ 2.42 (s, 3 H, CH_3), 4.18 (s, 2 H, CH_2), 7.09 (s, 1 H), 7.25–7.43 (m, 7H, ArH), 7.86 (dd, 2 H, J = 9.6, 8.0 Hz), 8.61 (br, 1 H, N–H).

5-(2-Chloroacetylamino)-1-(2-chlorophenyl)-3-phenylpyrazole (5). ^{1}H NMR ($CDCl_3$, 200 MHz) δ 4.13 (s, 2 H, CH_2), 7.04 (s, 1 H), 7.25–7.55 (m, 9H, ArH), 8.29 (br, 1 H, N–H).

5-(2-Chloroacetylamino)-1-(3-chlorophenyl)-3-phenylpyrazole (6). Brown solid;

mp 101–102 °C; ^{1}H NMR ($CDCl_3$, 200 MHz) δ 4.20 (s, 2 H, CH_2), 7.08 (s, 1 H, ArH), 7.31–7.47 (m, 5 H, ArH), 7.61 (s, 2 H, ArH), 7.85 (d, *J* = 7.6 Hz, 2 H, ArH), 8.61 (s, 1 H, N–H); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 42.64, 96.23, 122.00, 124.88, 125.76 (2 × CH), 128.45, 128.66 (3 × CH), 130.84, 132.42, 135.42, 135.81, 138.64, 152.42, 162.50; IR (KBr) 3046, 2955, 2924, 2853, 1721 (s, C=O), 1201, 764; EIMS m/z: 349 (M^+ + 4, 2), 347 (M^+ + 2, 9), 345 (M^+, 16), 194 (100), 165 (11), 133 (79), 105 (42), 91 (74), 77 (30); HRMS Calcd for $C_{17}H_{13}Cl_2ON_3$: 345.0436, Found: 345.0433.

5-(2-Chloroacetylamino)-1-(4-chlorophenyl)-3-phenylpyrazole (7). ^{1}H NMR ($CDCl_3$, 200 MHz) δ 4.18 (s, 2 H, CH_2), 7.04 (s, 1 H), 7.24–7.50 (m, 7H, ArH), 7.79 (dd, 2 H, *J* = 15.7, 7.8 Hz), 7.29 (br, 1 H, N–H).

5-(2-Chloroacetylamino)-1-(3-methoxyphenyl)-3-phenylpyrazole (8). ^{1}H NMR ($CDCl_3$, 200 MHz) δ 3.86 (s, 3 H, CH_3), 4.17 (s, 2 H, CH_2), 7.01–7.07 (m, 3H, ArH), 7.39–7.46 (m, 5H, ArH), 7.83–7.84 (m, 2H, ArH), 8.54 (br, 1 H, N–H).

5-(2-Chloroacetylamino)-1-phenyl-3-methylpyrazole (9). ^{1}H NMR ($CDCl_3$, 200 MHz) δ 2.30 (s, 3 H, CH_3), 4.13 (s, 2 H, CH_2), 6.55 (s, 1 H), 7.36–7.54 (m, 5H, ArH), 8.52 (br, 1 H, N–H).

5-(2-Chloroacetylamino)-1-phenyl-3-tetrabutanyl pyrazole (10). ^{1}H NMR ($CDCl_3$, 200 MHz) δ 1.33 (s, 9 H, CH_3), 4.13 (s, 2 H, CH_2), 6.63 (s, 1 H), 7.46–7.48 (m, 5H, ArH), 8.58 (br, 1 H, N–H).

5-(2-Chloroacetylamino)-1-phenyl-3-(4-methylphenyl) pyrazole (11). ^{1}H NMR ($CDCl_3$, 200 MHz) δ 2.36 (s, 3 H, CH_3), 4.15 (s, 2 H, CH_2), 6.74 (s, 1 H), 7.06–7.54 (m, 7H, ArH), 7.72 (d, *J* = 6.4 Hz, 2 H, ArH), 8.65 (br, 1 H, N–H).

5-**(2-Chloroacetylamino)-1-phenyl-3-(4-methoxyphenyl) pyrazole (12)**. ^{1}H NMR ($CDCl_3$, 200 MHz) δ 3.82 (s, 3 H, CH_3), 4.18 (s, 2 H, CH_2), 6.93 (d, *J* = 8.8 Hz, 2 H, ArH), 7.04 (s, 1 H), 7.43–7.55 (m, 5H, ArH), 7.80 (dd, 2 H, *J* = 8.8, 4.4 Hz), 8.62 (br, 1 H, N–H).

5-(2-Chloroacetylamino)-1-(3-methlyphenyl)-3-(4-methoxyphenyl) pyrazole (13). ^{1}H NMR ($CDCl_3$, 200 MHz) δ 2.42 (s, 3 H, CH_3), 3.82 (s, 3 H, CH_3), 4.17 (s, 2 H, CH_2), 6.92 (dd, 2 H, *J* = 8.8, 6.8 Hz), 7.03 (s, 1 H), 7.26–7.41 (m, 4H, ArH), 7.80 (dd, 2 H, *J* = 8.8, 6.8 Hz), 8.66 (br, 1 H, N–H).

5-(2-Chloroacetylamino)-1-(4-methlyphenyl)-3-(4-methoxyphenyl) pyrazole (14). ^{1}H NMR ($CDCl_3$, 200 MHz) δ 2.39 (s, 3 H, CH_3), 3.82 (s, 3 H, CH_3), 4.17 (s, 2 H, CH_2), 6.92 (dd, 2 H, *J* = 8.8, 4.4 Hz), 7.02 (s, 1 H), 7.30–7.42 (m, 4H, ArH), 7.78 (dd, 2 H, *J* = 8.8, 6.8 Hz), 8.60 (br, 1 H, N–H).

5-(2-Chloroacetylamino)-1-(2-chlorophenyl)-3-tetrabutanyl pyrazole (15). ^{1}H NMR ($CDCl_3$, 200 MHz) δ 1.35 (s, 9 H, CH_3), 4.08 (s, 2 H, CH_2), 6.56 (s, 1 H), 7.38–7.56 (m, 4H, ArH), 8.20 (br, 1 H, N–H); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 30.27 (3 × CH), 32.53, 42.49, 94.85, 128.24, 130.26, 130.58, 130.84, 131.61, 135.07, 135.55, 162.53, 163.29.

5-(2-Chloroacetylamino)-1-(4-chlorophenyl)-3-tetrabutanyl pyrazole (16). ^{1}H NMR ($CDCl_3$, 200 MHz) δ 1.29 (s, 9 H, CH_3), 4.11 (s, 2 H, CH_2), 6.56 (s, 1 H), 7.39–7.46 (m, 4H, ArH), 8.49 (br, 1 H, N–H).

5-(2-Bromoacetylamino)-1-(3-chlorophenyl)-3-phenylpyrazole (33). Yellow solid; mp 106–107 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 4.01 (s, 2 H, CH_2), 7.05 (s, 1 H, ArH), 7.31–7.47 (m, 6 H, ArH), 7.61 (s, 1 H, ArH), 7.85 (d, *J* = 7.6 Hz, 2 H, ArH), 8.48 (s, 1 H, N–H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 42.63, 96.23, 121.99, 124.87, 125.76 (2 × CH), 127.99, 128.66 (3 × CH), 130.84, 132.40, 135.42, 135.81, 138.61, 152.43, 162.50; IR (KBr) 3046, 2955, 2924, 2853, 1721 (s, C=O), 1201, 764; EIMS m/z: 393 (M^+ + 4, 25), 391 (M^+ + 2, 100), 389 (M^+, 76), 296 (11), 271 (30), 269 (93), 233 (35), 102 (69), 77 (16); HRMS Calcd for $C_{17}H_{13}BrClON_3$: 388.9931, Found: 388.9932.

1-(3-Chlorophenyl)-5-(2-iodoacetylamino)-3-phenylpyrazole (34). Yellow solid; mp 103–104 °C; ^{1}H NMR ($CDCl_3$, 200 MHz) δ 4.20 (s, 2 H, CH_2), 7.08 (s, 1 H, ArH),

7.22 (m, 2 H, ArH), 7.42 (m, 4 H, ArH), 7.61 (s, 1 H, ArH), 7.85 (m, 2 H, ArH), 8.62 (s, 1 H, N–H); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 32.88, 90.21, 126.21, 127.26, 129.94 (2 × CH), 128.45, 132.54 (3 × CH), 134.52, 134.77, 135.69, 139.72, 1345.34, 152.43, 165.23; IR (KBr) 3229, 2920, 1674 (s, C=O), 1593, 1558, 1366, 1076, 763, 1201, 764; EIMS m/z: 439 ($M^+ + 2$, 13),437 (M^+, 52), 345 (32), 269 (100), 233 (39), 102 (90), 91 (12), 77 (27); HRMS Calcd for $C_{17}H_{13}ClION_3$: 436.9792, Found: 436.9793.

Standard procedure for synthesis of 1,3-diphenyl-6-[(formyloxy)methyl]-1*H*-pyrazolo-[3,4-*d*]pyrimidine 17 and (1,3-diphenyl-1*H*-pyrazolo[3,4-*d*]pyrimidin-6-yl)methanol 44. The reliable procedure involved the treatment of 5-(2-chloroacetylamino)pyrazoles **1** (203 mg, 0.58 mmol, 1.0 equiv), with PBr_3 (1.77 mmol, ~ 0.2 mL, ~3.0 equiv) in formamide solution (2 mL) at 50–60 °C within 5 h. When the reaction was completed, the reaction mixture was added to saturate sodium bicarbonate (15 mL) and extracted with dichloromethane (15 mL × 2). The combined organic layer was washed sodium bicarbonate (15 mL), dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue solution was purified by column chromatography on silica gel to give the corresponding 6-(formylmethyl)pyrazolo[3,4-*d*]pyrimidines **17** (168 mg, 0.51 mmol) in 88 % yield and by-product (1,3-diphenyl- 1*H*-pyrazolo[3,4-*d*]pyrimidin-6-yl)methanol **44** (17.1 mg, 0.049 mmol) in 9% yield.

1,3-Diphenyl-6-[(formyloxy)methyl]-1*H*-pyrazolo[3,4-*d*]pyrimidine (17). Yellow solid; mp 126–127 °C; ^{1}H NMR ($CDCl_3$, 200 MHz) δ 5.55 (s, 2 H, CH_2), 7.33–7.37 (m, 1 H, ArH), 7.46–7.58 (m, 5 H, ArH), 8.01 (dd, *J* = 1.6, 7.9 Hz, 2 H, ArH), 8.28 (dd, *J* = 1.6, 7.9 Hz, 2 H, ArH), 8.33 (s, 1 H, CHO), 9.43 (s, 1 H, Pyrimidine–H);^{13}C NMR ($CDCl_3$, 50 MHz) δ 65.60, 113.14, 121.16 (2 × CH), 126.73, 127.26 (2 × CH), 129.14, 129.63 (4 × CH), 131.29, 138.39, 144.96, 152.97, 153.59, 160.50, 161.48; IR (KBr) 2955, 1721(s, C=O), 1585, 1497, 1416, 1200, 753; EIMS m/z: 330 (M^+, 100), 301 (38), 286 (79), 273 (15), 271 (12), 180 (7), 142 (10), 91 (14), 77 (51); HRMS Calcd for $C_{19}H_{14}O_2N_4$: 330.1117, Found: 330.1121.

(1,3-Diphenyl-1*H*-pyrazolo[3,4-*d*]pyrimidin-6-yl)methanol (44). Yellow solid; mp 101–102 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 4.99 (s, 2 H, CH_2), 7.34–7.38 (m, 1 H, ArH), 7.48–7.57 (m, 5 H, ArH), 8.04 (d, *J* = 7.2 Hz, 2 H, ArH), 8.26 (d, *J* = 7.2 Hz, 2 H, ArH), 9.44 (s, 1 H, Pyrimidine–H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 64.85, 113.21,

121.49 (2 × CH), 126.92, 127.36 (2 × CH), 129.22 (4 × CH), 129.70, 131.40, 138.39, 145.25, 152.82, 153.55, 166.22; IR (KBr) 3395 (OH, Br), 2916, 1651, 1543, 748; EIMS m/z: 302 (M^+, 100), 301 (53), 273 (25), 273 (15), 271 (20), 91 (16), 77 (49); HRMS Calcd for $C_{18}H_{14}ON_4$: 302.1168, Found: 302.1161.

Standard Procedure for Synthesis of 6-(Formyloxymethyl)pyrazolo[3,4-*d*]pyrimidines 18–32. The reliable procedure involved the treatment of 5-(2-chloroacetylamino)pyrazoles (**2**–**16**, 1.0 equiv), with PBr_3 (~3.0 equiv) in formamide solution (2 mL) at 50–60 °C within 5 h. When the reaction was completed, the reaction mixture was added to saturate sodium bicarbonate (15 mL) and extracted with dichloromethane (15 mL × 2). The combined organic layer was washed sodium bicarbonate (15 mL), dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue solution was purified by column chromatography on silica gel to give the corresponding 6-(formylmethyl)pyrazolo[3,4-*d*]pyrimidines **18–32** in 73–92 % yields.

6-[(Formyloxy)methyl]-1-(2-methylphenyl)-3-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine (18). White solid; mp 133–134 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.22 (s, 3 H, CH_3), 5.47 (s, 2 H, CH_2), 7.37–7.57 (m, 7 H, ArH), 8.03 (d, *J* = 7.6 Hz, 2 H, ArH), 8.23 (s, 1 H, CHO), 9.53 (s, 1 H, Pyrimidine–H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 18.44, 65.69, 111.90, 126.69, 127.29 (2 × CH), 127.61, 129.24 (2 × CH), 129.41, 129.58, 131.43, 131.63, 135.40, 136.11, 145.16, 153.04, 154.55, 160.47, 161.72; IR (KBr) 2920, 2851, 1721 (s, C=O), 1589, 1458, 1265, 1184, 752; EIMS m/z: 344 (M^+, 13), 299 (32) , 298 (100) , 256 (17) , 202 (11), 189 (17), 155 (14), 133 (31), 91 (41), 88 (28), 77 (17); HRMS Calcd for $C_{20}H_{16}O_2N_4$: 344.1273, Found: 344.1274.

6-[(Formyloxy)methyl]-1-(3-methylphenyl)-3-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine (19). White solid; mp 127–128 °C; ^{1}H NMR ($CDCl_3$, 200 MHz) δ 2.22 (s, 3 H, CH_3), 5.47 (s, 2 H, CH_2), 7.34–7.59 (m, 5 H, ArH), 7.53 (d, *J* = 7.5 Hz, 2 H, ArH), 8.04 (dd, *J* = 1.4, 7.8 Hz, 2 H, ArH), 8.23 (s, 1 H, CHO), 9.49 (s, 1 H, Pyrimidine–H); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 18.43, 65.66, 111.88, 126.68, 127.27 (2 × CH), 127.60, 129.23 (2 × CH), 129.39, 129.57, 131.42, 131.60, 135.38, 136.08, 145.14, 153.03, 154.53, 160.46, 161.70; IR (KBr) 3059 (m),2916, 2847, 1724 (s,

C=O), 1589, 1504, 1458, 1184, 1134, 760; EIMS m/z: 344 (M^+, 15), 298 (100), 256 (19), 163 (20), 146 (47), 134 (35), 120 (55), 115 (58), 99 (84), 77 (30); HRMS Calcd for $C_{20}H_{16}O_2N_4$: 344.1273, Found: 344.1268.

6-[(Formyloxy)methyl]-1-(4-methylphenyl)-3-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine (20). White solid; mp 107–108 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.42 (s, 3 H, CH_3), 5.55 (s, 2 H, CH_2), 7.34 (d, J = 8.0 Hz, 2 H, ArH), 7.56 (s, 1 H, ArH), 8.04 (d, J = 8.0 Hz, 2 H, ArH), 8.14 (d, J = 8.0 Hz, 2 H, ArH), 8. 23 (s, 1 H, CHO), 9.46 (s, 1 H, Pyrimidine–H); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 21.10, 65.68, 113.06, 121.35 (2 × CH), 127.34 (2 × CH), 129.22 (2 × CH), 129.71, 129.73 (2 × CH), 131.46, 135.97, 136.79, 144.87, 152.99, 153.45, 160.53, 161.42; IR (KBr) 2922, 1726 (s, C=O), 1514, 1454, 1184, 1172, 957, 816, 766; EIMS m/z: 344 (M^+, 100), 301 (37), 208 (11),91 (19), 77 (10); HRMS Calcd for $C_{20}H_{16}O_2N_4$: 344.1273, Found: 344.1276.

1-(2-Chlorophenyl)-6-[(formyloxy)methyl]-3-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine (21). Yellow solid; mp 81–82 °C; ^{1}H NMR ($CDCl_3$, 200MHz) δ 5.47 (s, 2 H, CH_2), 7.45–7.64 (m, 7 H, ArH), 8.02 (dd, J = 1.4, 7.7 Hz, 2 H, ArH), 8.24 (s, 1 H, CHO), 9.48 (s, 1 H, Pyrimidine–H); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 65.61, 112.02, 127.37 (2 × CH), 127.59, 129.24 (2 × CH), 129.61, 129.72, 130.69, 130.78, 131.35, 132.09, 134.74, 145.79, 153.03, 154.99, 160.45, 161.92; IR (KBr) 3059,2922, 2851, 1722 (s, C=O), 1591, 1503, 1410, 1184, 1132, 980, 756; EIMS m/z: 366 (M^+ + 2, 35), 364 (M^+, 100), 335 (34), 329 (13), 320 (83), 307 (15), 271 (13), 133 (29), 91 (24), 77 (27); HRMS Calcd for $C_{19}H_{13}ClO_2N_4$: 364.0721, Found: 364.0725.

1-(3-Chlorophenyl)-6-[(formyloxy)methyl]-3-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine (22). Yellow solid; mp 128–129 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 5.58 (s, 2 H, CH_2), 7.32 (d, J = 7.2 Hz, 1 H, ArH), 7.44–7.59 (m, 3 H, ArH), 7.57 (s, 1 H, ArH), 8.04 (d, J = 8.0, 2 H, ArH), 8.31 (d, J = 8.0 Hz, 1 H, ArH), 8.34 (s, 1 H, ArH), 8.44 (s, 1 H, Pyrimidine–H), 9.46 (s, 1 H, CHO); ^{13}C NMR ($CDCl_3$, 100 MHz) δ

65.46, 113.42, 118.81, 121.05, 126.58, 127.37 (2 × CH), 129.24 (2 × CH), 129.88, 130.15, 131.09, 134.90, 139.52, 145.51, 153.12, 153.97, 160.38, 161.88; IR (KBr) 3059,2922, 2851, 1722 (s, C=O), 1591, 1503, 1410, 1184, 1132, 980, 756; EIMS m/z: 366 (M^+ + 2, 34), 364 (M^+, 100), 355 (16), 335 (53), 320 (77), 307 (22), 281 (18), 221 (18), 111 (12), 77 (18); HRMS Calcd for $C_{19}H_{13}ClO_2N_4$: 364.0721, Found: 364.0725.

1-(4-Chlorophenyl)-6-[(formyloxy)methyl]-3-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine (23). Yellow solid; mp 127–128 °C; ^{1}H NMR ($CDCl_3$, 200MHz) δ 5.56 (s, 2 H, CH_2), 7.48–7.57 (m, 5 H, ArH), 8.03 (dd, *J* = 1.5, 8.4, 2 H, ArH), 8.31 (d, *J* = 8.4, 2 H, ArH), 8.33(s, 1 H, CHO), 9.46 (s, 1 H, Pyrimidine–H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 65.61, 113.36, 122.26, 125.75, 127.40, 129.32, 129.90 (2 × CH), 131.18, 132.21, 137.08, 145.41, 153.19, 153.78, 160.50, 161.81; IR (KBr) 3059,2922, 2851, 1722 (s, C=O), 1591, 1503, 1410, 1184, 1132, 980, 756; EIMS m/z: 366 (M^+ + 2, 2), 364 (M^+, 7), 284 (21), 217 (11), 135 (100), 91 (16), 77 (9); HRMS Calcd for $C_{19}H_{13}ClO_2N_4$: 364.0721, Found: 364.0718.

6-[(Formyloxy)methyl]-1-(3-methoxyphenyl)-3-phenyl-1*H*-pyrazolo-[3,4-*d*]pyrimidine (24). Yellow solid; mp 90–91 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.87 (s, 3 H, CH_3), 5.54 (s, 2 H, CH_2), 7.06 (d, *J* = 8.0 Hz, 2 H, ArH), 7.48–7.57 (m, 2 H, ArH), 7.55 (s, 1 H, ArH), 8.03 (d, *J* = 8.0 Hz, 2 H, ArH), 8.14 (dd, *J* = 2.0, 8.0 Hz, 2 H, ArH), 8.32 (s, 1 H, CHO), 9.45 (s, 1 H, Pyrimidine–H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ55.58, 65.73, 112.91, 114.38 (2 × CH), 123.01 (2 × CH), 127.32 (2 × CH), 129.22 (2 × CH), 129.57, 131.55, 131.72, 144.71, 153.01, 153.29, 158.45, 160.51, 161.46; IR (KBr) 2924, 1728 (s, C=O), 1585, 1512, 1458, 1250, 1173, 1030, 829, 764; EIMS m/z: 360 (M^+, 100), 331 (13), 316 (12), 77 (10); HRMS Calcd for $C_{20}H_{16}O_3N_4$: 360.1222, Found: 360.1224.

6-[(Formyloxy)methyl]-3-methyl-1-phenyl-1*H*-pyrazolo[3,4-*d*]-pyrimidine (25). Brown solid; mp 85–86 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.69 (s, 3 H, CH_3), 5.52 (s,

2 H, CH_2), 7.29–7.33 (m, 1 H, ArH), 7.48–7.52 (m, 1 H, ArH), 7.50 (s, 1 H, ArH), 8.19 (d, *J* = 8.0 Hz, 2 H, ArH), 8.30 (s, 1 H, CHO), 9.12 (s, 1 H, Pyrimidine–H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 12.58, 65.74, 114.77, 120.87 (2 × CH), 126.41, 129.11 (2 × CH), 138.44, 143.41, 151.95, 153.12, 160.45, 161.58; IR (KBr) 2924, 2854, 1732 (s, C=O), 1589, 1508, 1458, 1161, 752; EIMS m/z: 268 (M^+, 89), 239 (46), 224 (100), 211 (20), 142 (12), 91 (13), 77 (53); HRMS Calcd for $C_{14}H_{12}O_2N_4$: 268.0960, Found: 268.0963.

3-*tetra*-Butanyl-6-[(formyloxy)methyl]-1-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine (26). Brown solid; mp 91–92 °C; ^{1}H NMR ($CDCl_3$, 200 MHz) δ 1.56 (s, 9 H, CH3), 5.51 (s, 2 H, CH2), 7.25–7.33 (m, 1 H, ArH), 7.46–7.53 (m, 2 H, ArH), 7.50 (s, 1 H, ArH), 8.22 (d, *J* = 8.0 Hz, 2 H, ArH), 8.30 (s, 1 H, CHO), 9.29 (s, 1 H, Pyrimidine–H); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 30.02 (3 × CH), 65.74, 113.02, 120.99 (2 × CH), 126.28, 129.08 (2 × CH), 138.62, 153.07, 153.53, 154.98, 160.54, 160.90; IR (KBr) 2963, 2924, 1732 (s, C=O), 1582, 1508, 1420, 1366, 1157, 756; EIMS m/z: 310 (M^+, 21), 295 (30), 265 (12), 159 (15), 113 (12), 98 (13), 91 (100), 77 (20); HRMS Calcd for $C_{17}H_{18}O_2N_4$: 310.1434, Found: 310.1430.

6-[(Formyloxy)methyl]-3-(4-methylphenyl)-1-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine (27). Yield: 87%; yellow liquid; ^{1}H NMR ($CDCl_3$, 200 MHz) δ 2.44 (s, 3 H, CH_3), 5.55 (s, 2 H, CH_2), 7.36 (d, *J* = 8.1 Hz, 2 H, ArH), 7.53 (dd, *J* = 8.0, 7.4 Hz, 2 H, ArH), 7.94 (d, *J* = 8.1 Hz, 2 H, ArH), 8.30 (d, *J* = 8.1 Hz, 2 H, ArH), 8.32 (s, 1 H, CHO), 9.44 (s, 1 H, Pyrimidine–H); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 29.69, 65.70, 113.28, 121.27 (2 × CH), 126.72, 127.24 (2 × CH), 128.58, 129.16 (2 × CH), 129.93 (2 × CH), 138.53, 139.89, 145.19, 153.05, 153.68, 160.51, 160.51, 161.51; IR (KBr) 2920, 2851, 1717 (s, C=O), 1589, 1501, 1411, 1200, 1134, 980, 756 cm^{-1}; EIMS m/z: 344 (M^+, 100), 315 (36), 300 (51), 287 (11), 91 (13),77 (17); HRMS Calcd for $C_{20}H_{16}O_2N_4$: 344.1273, Found: 344.1275.

6-[(Formyloxy)methyl]-3-(4-methoxyphenyl)-1-phenyl-1*H*-pyrazolo-[3,4-*d*]pyrimidine (28). Yellow solid; mp 165–167 °C; ^{1}H NMR ($CDCl_3$, 200MHz) δ 3.88 (s, 3 H, CH_3), 5.55 (s, 2 H, CH_2), 7.07 (d, *J* = 8.8 Hz, 2 H, ArH), 7.33–7.37(m, 1 H, ArH), 7.49–7.57 (m, 2 H, ArH), 8.29 (dd, *J* = 1.0, 8.8 Hz, 2 H, ArH), 8.32 (s, 1 H, CHO), 9.41 (s, 1 H, Pyrimidine–H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 55.42, 65.69, 113.19, 114.66 (2 × CH), 121.19 (2 × CH), 123.96, 126.24, 128.66 (2 × CH), 129.14 (2 × CH), 138.52, 144.92, 152.97, 153.61, 160.51, 160.83, 161.46; IR (KBr) 2922, 1715 (s, C=O), 1585, 1504, 1412, 1199, 1030, 982, 787 cm^{-1}; EIMS m/z: 360 (M^+, 100), 331 (24), 316 (29), 77 (15); HRMS Calcd for $C_{20}H_{16}O_3N_4$: 360.1222, Found: 360.1217.

6-[(Formyloxy)methyl]-1-(3-methlyphenyl)-3-(4-methoxyphenyl)-1*H*-pyrazolo[3,4-*d*]pyrimidine (29). Yellow solid; mp 138–139 °C; ^{1}H NMR ($CDCl_3$, 400MHz) δ 2.47 (s, 3 H, CH_3), 3.89 (s, 3 H, OCH_3), 5.55 (s, 2 H, CH_2), 7.08 (d, *J* = 8.4 Hz, 2 H, ArH), 7.17 (m, 1 H, ArH), 7.41 (m, 1 H, ArH), 7.99 (d, *J* = 8.4 Hz, 2 H, ArH), 8.08 (m, 2 H, ArH), 8.32 (s, 1 H, CHO), 9.42 (s, 1 H, Pyrimidine–H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 21.62, 55.45, 65.73, 113.19, 114.72 (2 × CH), 118.50, 121.94, 124.10, 127.54, 128.72 (2 × CH), 128.97, 138.46, 139.19, 144.89, 152.97, 153.65, 160.49, 160.88, 161.46; IR (KBr) 2953, 1717 (s, C=O), 1585, 1503, 1408, 1175, 1134, 837, 787; EIMS m/z: 374 (M^+, 100), 345 (19), 330 (24), 91 (8); HRMS Calcd for $C_{21}H_{18}O_3N_4$: 374.1379, Found: 374.1383.

6-[(Formyloxy)methyl]-1-(4-methlyphenyl)-3-(4-methoxyphenyl)-1*H*-pyrazolo[3,4-*d*]pyrimidine (30). Yellow solid; mp 152–154 °C; ^{1}H NMR ($CDCl_3$, 200 MHz) δ 2.42 (s, 3 H, CH_3), 3.89 (s, 3 H, OCH_3), 5.54 (s, 2 H, CH_2), 7.07 (d, *J* = 8.0 Hz, 2 H, ArH), 7.33 (d, *J* = 8.0 Hz, 2 H, ArH), 7.98 (d, *J* = 8.0 Hz, 2 H, ArH), 8.13 (d, *J* = 8.0 Hz, 2 H, ArH), 8.31 (s, 1 H, CHO), 9.41 (s, 1 H, Pyrimidine–H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 21.62, 55.45, 65.73, 113.19, 114.72 (2 × CH), 118.50,

121.94, 124.10, 127.54, 128.72 (2 × CH), 128.97, 138.46, 139.19, 144.89, 152.97, 153.65, 160.49, 160.88, 161.46; IR (KBr) 2922, 1732 (s, C=O), 1585, 1504, 1412, 1199, 1136, 839, 754; EIMS m/z: 374 (M^+, 100), 345 (19), 330 (25), 226 (22), 210 (13), 180 (15), 167 (36), 135 (18), 125 (20), 111 (40), 97 (51), 91 (17); HRMS Calcd for $C_{21}H_{18}O_3N_4$: 374.1379, Found: 374.1381.

3-tetra-Butyl-1-(2-chlorophenyl)-6-[(formyloxy)methyl]-1*H*-pyrazolo[3,4-*d*]pyrimidine (31). Yellow solid; mp 138–139 °C; ^{1}H NMR ($CDCl_3$, 200 MHz) δ 1.56 (s, 9 H, CH_3), 5.51 (s, 2 H, CH_2), 7.25–7.33 (m, 1 H, ArH), 7.46–7.53 (m, 2 H, ArH), 8.22 (d, *J* = 8.0 Hz, 2 H, ArH), 8.23 (s, 1 H, CHO), 9.46 (s, 1 H, Pyrimidine–H); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 30.01 (3 × CH), 67.54, 112.03, 121.97 (2 × CH), 128.72, 129.08 (2 × CH), 139.63, 153.07, 153.53, 154.98, 156.35, 161.93; IR (KBr) 2916, 1721 (s, C=O), 1578, 1558, 1458, 1261, 1165, 760, 694; EIMS m/z: 346 (M^+ + 2, 30), 344 (M^+, 100), 345 (19), 330 (25), 226 (22), 210 (13), 180 (15), 167 (36), 135 (18), 125 (20), 111 (40), 97 (51), 91 (17); HRMS Calcd for $C_{17}H_{17}ClO_4N_2$: 344.1040, Found: 344.1044.

3-Tetrabutanyl-1-(4-chlorophenyl)-6-[(formyloxy)methyl]-1*H*-pyrazolo[3,4-*d*]pyrimidine (32). Yellow solid; mp 147–148 °C; ^{1}H NMR ($CDCl_3$, 200 MHz) δ 1.55 (s, 9 H, CH_3), 5.51 (s, 2 H, CH_2), 7.49 (m, 2 H, ArH), 8.25 (m, 2H, ArH), 8.30 (s, 1 H, CHO), 9.29 (s, 1 H, Pyrimidine–H); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 30.27 (3 × CH), 65.70, 128.24 (2 × CH), 130.26, 130.58, 130.84 (2 × CH), 131.61, 135.07, 135.55, 145.19, 153.05, 162.53, 163.29; IR (KBr) 2922, 1721 (s, C=O), 1585, 1252, 1175, 1026, 781; EIMS m/z: 346 (M^+ + 2, 37), 344 (M^+, 94), 345 (19), 330 (25), 226 (22), 210 (13), 180 (15), 167 (36), 135 (18), 125 (20), 111 (40), 97 (51), 91 (17); HRMS Calcd for $C_{17}H_{17}ClO_4N_2$: 344.1040, Found: 344.1043. (100), 55 (44); HRMS calcd. for $C_{17}H_{22}N_4O_3$, 330.1692; found 330.1696.

1-(3-Chlorophenyl)-3phenyl-5-(2-hydroxyacetylamino)pyrazole (35)[29] A solution

of 5-(2-chloroacetylamino)pyrazole (**6**, 1.0 equiv) and cesium formate (HCO_2Cs, 3.0 equiv) in dry MeOH (10 mL) was heated at reflux for >2.0 h. When the reaction was completed, the solution was filtered to remove the excess amount of HCO_2Cs and the filtrate was concentrated under reduced pressure. The residue was purified by column chromatography on silica gel or re-crystallization to give the corresponding 5-(2-hydroxyacetylamino)pyrazole **35** in 84% yield. yellow solid; mp 98–99 °C; ^{1}H NMR ($CDCl_3$, 200 MHz) δ 4.15 (s, 2 H, CH_2), 7.06 (s, 1 H, ArH), 7.32–7.44 (m, 7 H, ArH), 7.56 (m, 1 H, ArH), 7.83 (dd, *J* = 4.5, 8.2 Hz, 2 H, ArH), 8.79 (s, 1 H, NH); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 62.12, 95.94, 122.08, 124.87, 125.78 (2 × CH), 128.46, 128.54, 128.66 (2 × CH), 130.74, 132.44, 135.61, 136.03, 138.75, 152.51, 168.37; IR (KBr) 3429, 2359, 1591, 1184, 966, 756; EIMS m/z: 329 (M^+ + 2, 33), 327 (M^+, 73), 329 (33), 270 (32), 235 (20), 220 (39), 207 (25), 195 (58), 180 (97), 162 (24), 135 (94), 121 (100), 102 (36), 91 (56), 81 (77); HRMS Calcd for $C_{17}H_{14}ClO_2N_3$: 327.0775, Found: 327.0776.

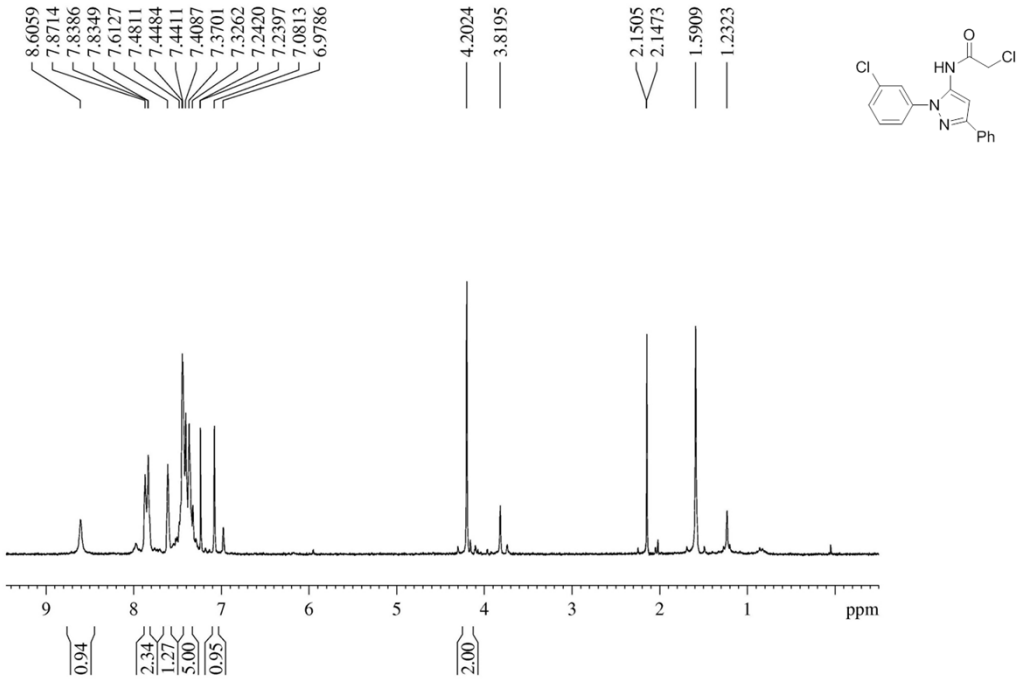
6-(Chloromethyl)pyrazolo[3,4-*d*]pyrimidine (40). The treatment of 1,3-diphenyl-1*H*-pyrazolo[3,4-*d*]pyrimidin-6-yl)methanol (**44**, 1.0 equiv), with $SOCl_2$ (~3.0 equiv) in formamide solution (2 mL) at 50–60 °C within 3 h. When the reaction was completed, the reaction mixture was added to saturate sodium bicarbonate (15 mL) and extracted with dichloromethane (15 mL × 2). The combined organic layer was washed sodium bicarbonate (15 mL), dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue solution was purified by column chromatography on silica gel to give the corresponding 6-(chloromethyl)pyrazolo[3,4-*d*]pyrimidine **40** in 80 % yields: yellow solid; mp 133–134 °C; ^{1}H NMR ($CDCl_3$, 200 MHz) δ 4.89 (s, 2 H, CH_2), 7.32–7.40 (m, 1 H, ArH), 7.49–7.61 (m, 5 H, ArH), 8.05 (dd, *J* = 7.2, Hz, 2 H, ArH), 8.26 (dd, *J* = 7.2 Hz, 2 H, ArH), 9.49 (s, 1 H, Pyrimidine–H); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 47.33, 112.95, 121.33 (2 × CH), 126.87, 127.37 (2 × CH), 129.24

(3 × CH), 129.73, 131.38, 138.50, 145.07, 153.37, 153.88, 162.99; IR (KBr) 2920, 1581, 1501, 1226, 1134, 976, 756, 691; EIMS m/z: 320 (M^+, 100), 319 (38), 294 (35), 251 (24), 221 (20), 207 (48), 140 (15), 91 (39), 77 (56); HRMS Calcd for $C_{18}H_{13}ClN_4$: 320.0829; Found: 320.0828.

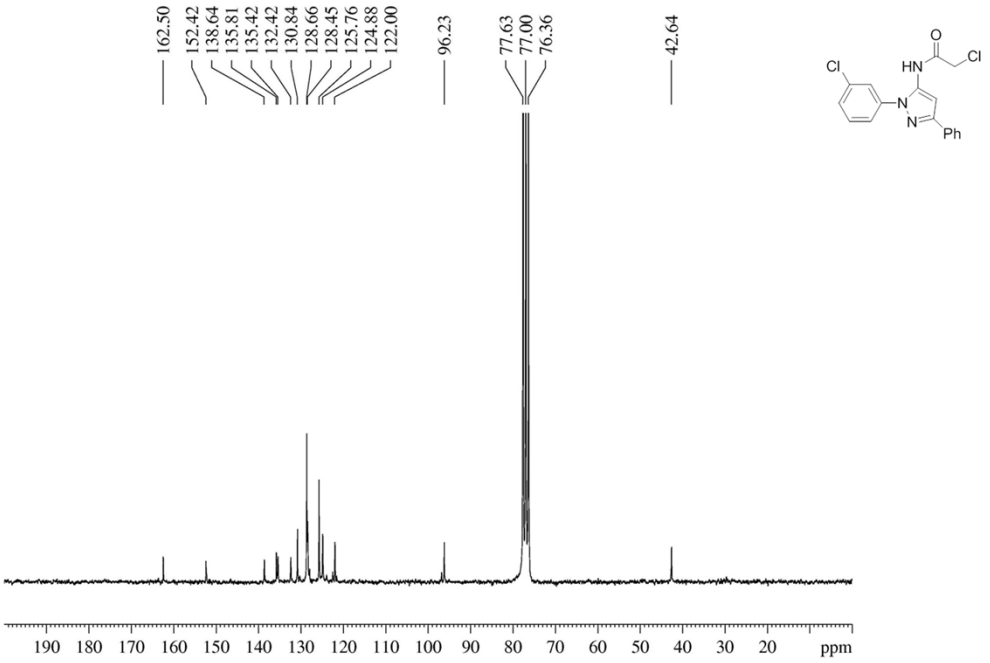
^{1}H-NMR and ^{13}C-NMR of 5-(2-haloacetylamino)pyrazoles 6 and 33–35

5-(2-Chloroacetylamino)-1-(3-chlorophenyl)-3-phenylpyrazole **6**

^{1}H-NMR

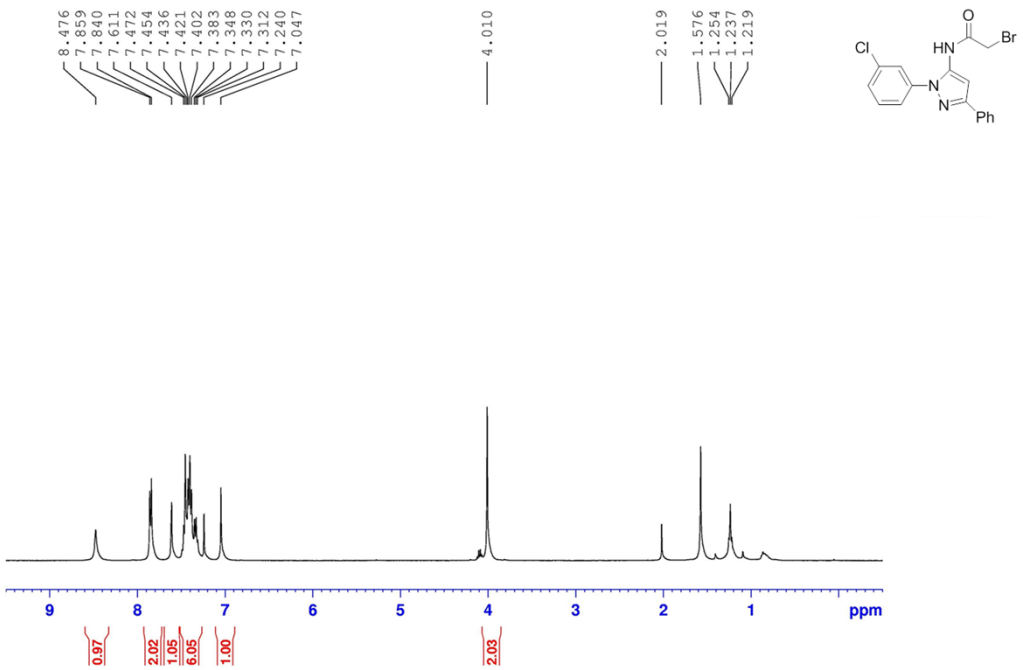


^{13}C-NMR

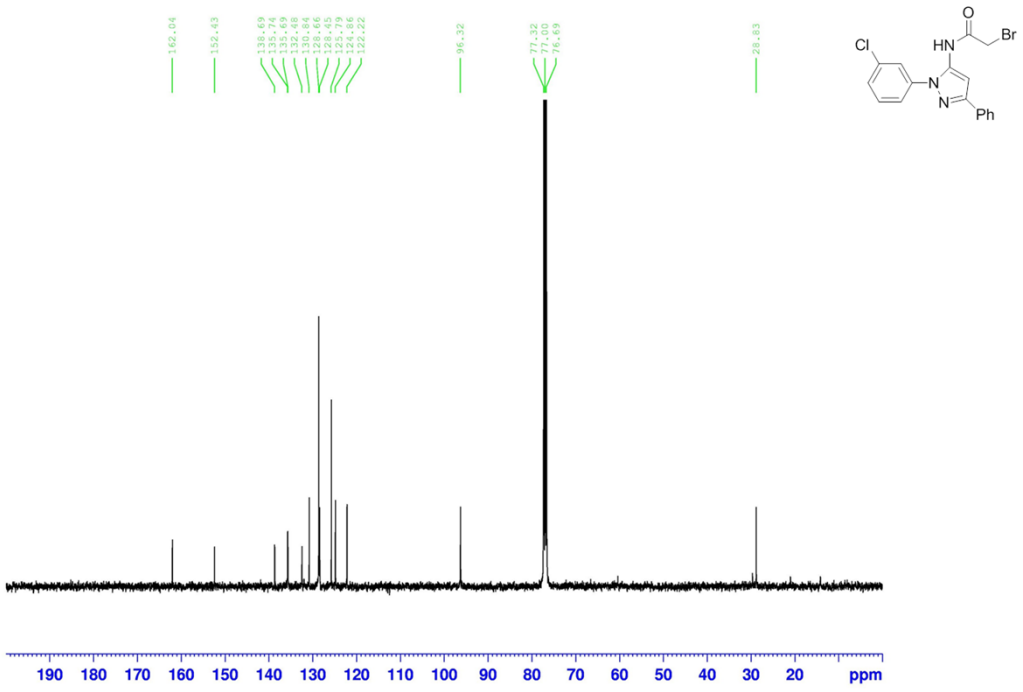


5-(2-Bromoacetylamino)-1-(3-chlorophenyl)-3-phenylpyrazole **33**

^{1}H-NMR

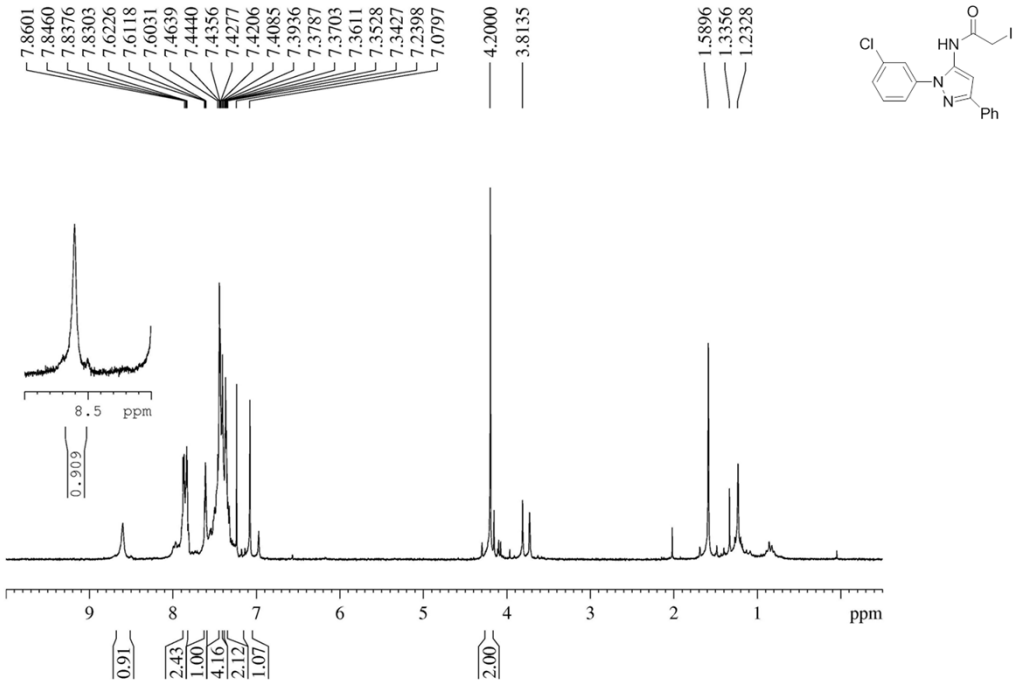


^{13}C-NMR

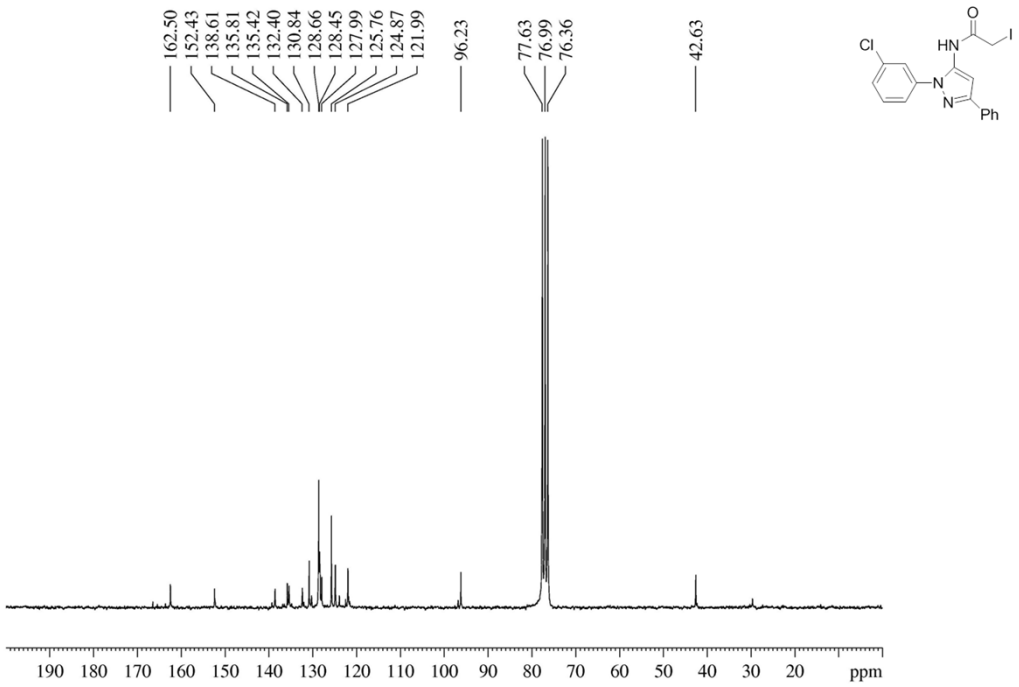


1-(3-Chlorophenyl)-5-(2-iodoacetylamino)-3-phenylpyrazole **34**

^{1}H-NMR

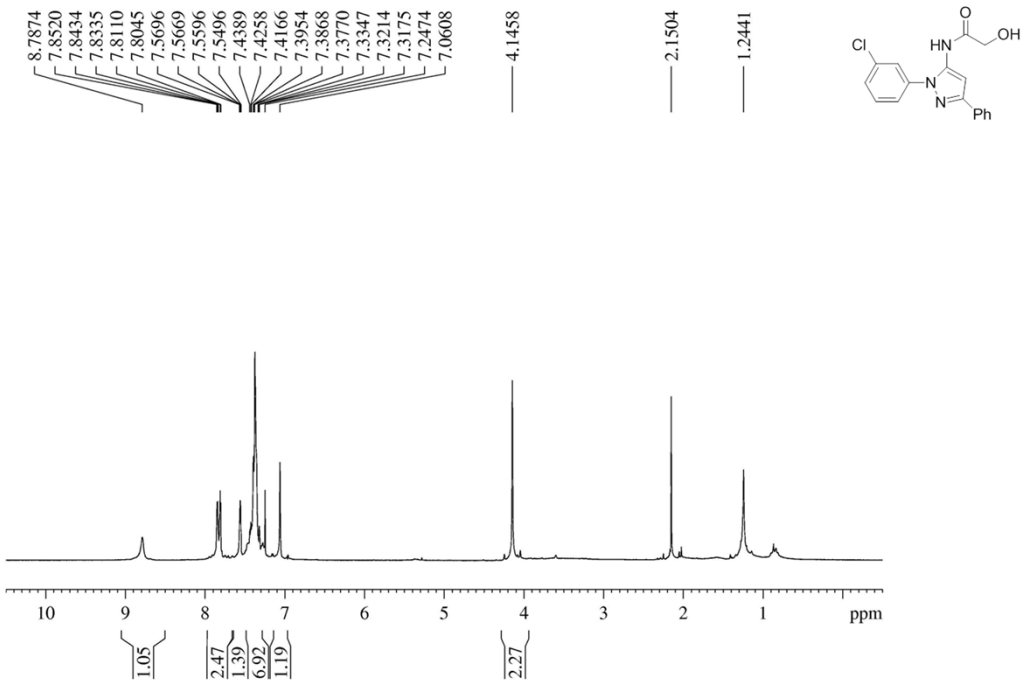


^{13}C-NMR

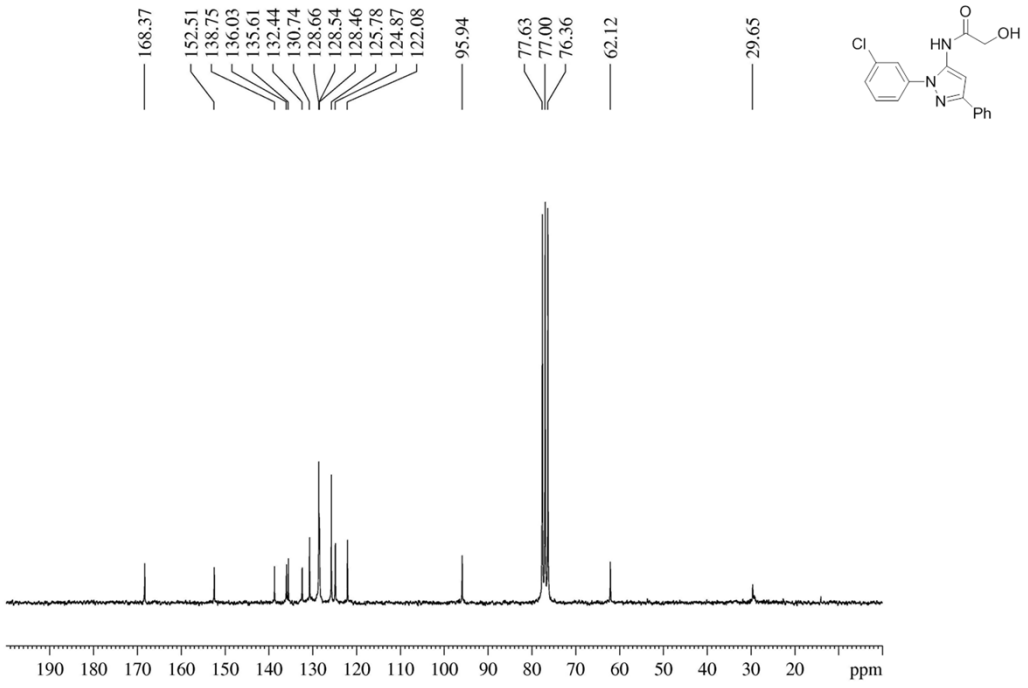


1-(3-Chlorophenyl)-5-(2-hydroxyacetylamino)-3-phenylpyrazole **35**

^{1}H-NMR



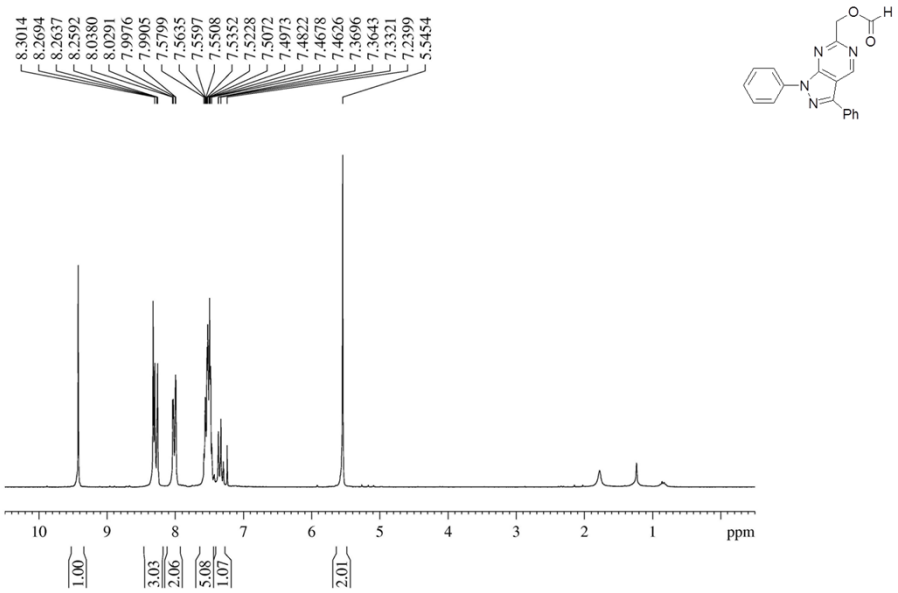
^{13}C-NMR



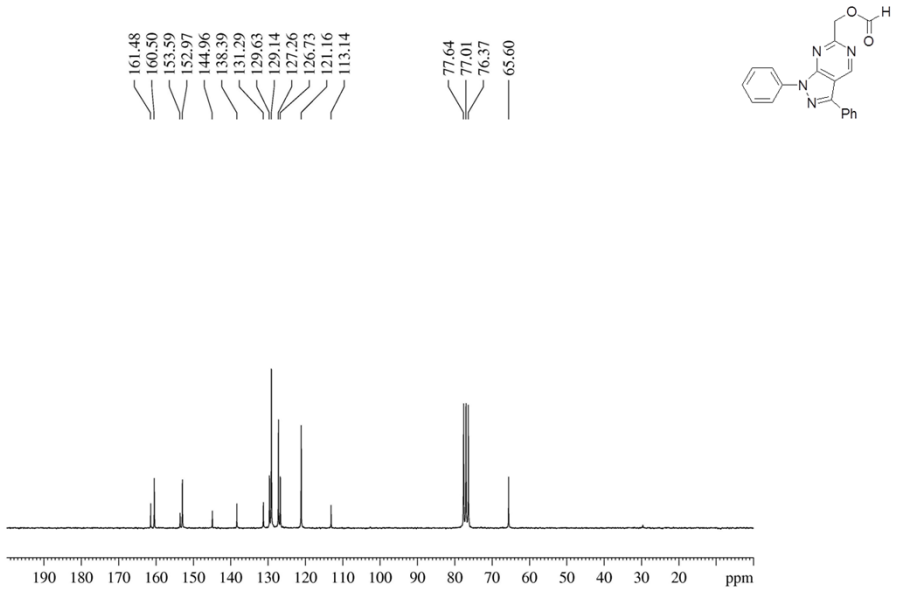
^{1}H and ^{13}C-NMR Spectra of Reported Compounds **17**, **18–32**, and **44**

1,3-Diphenyl-6-[(formyloxy)methyl]-1*H*-pyrazolo[3,4-*d*] pyrimidine **17**

^{1}H-NMR

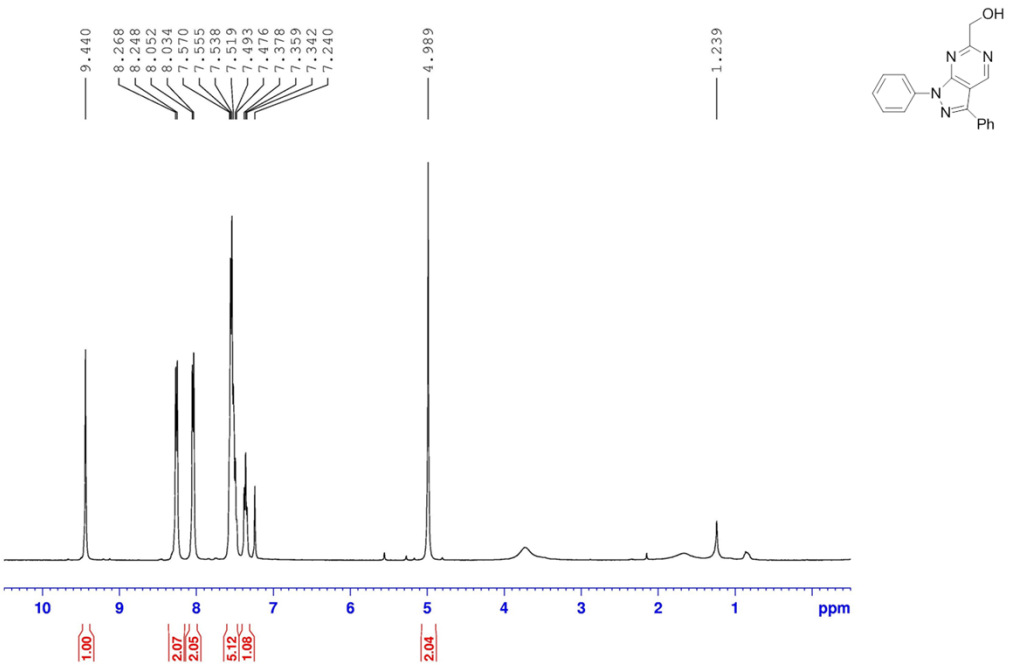


^{13}C-NMR

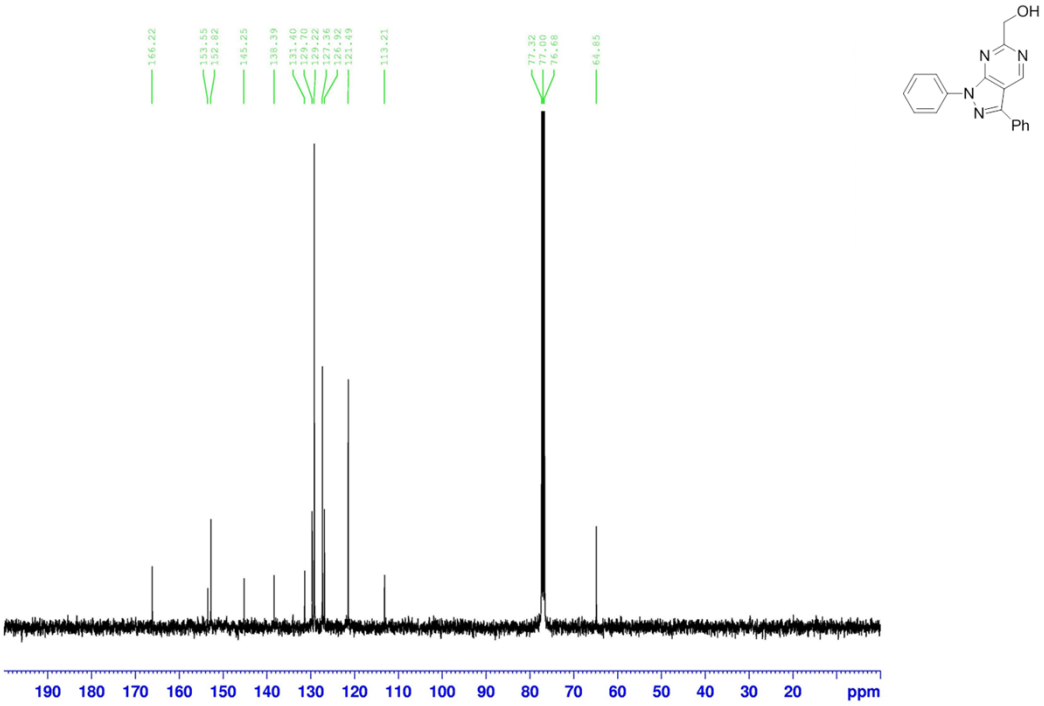


(1,3-Diphenyl-1*H*-pyrazolo[3,4-*d*]pyrimidin-6-yl)methanol **44**

^{1}H-NMR

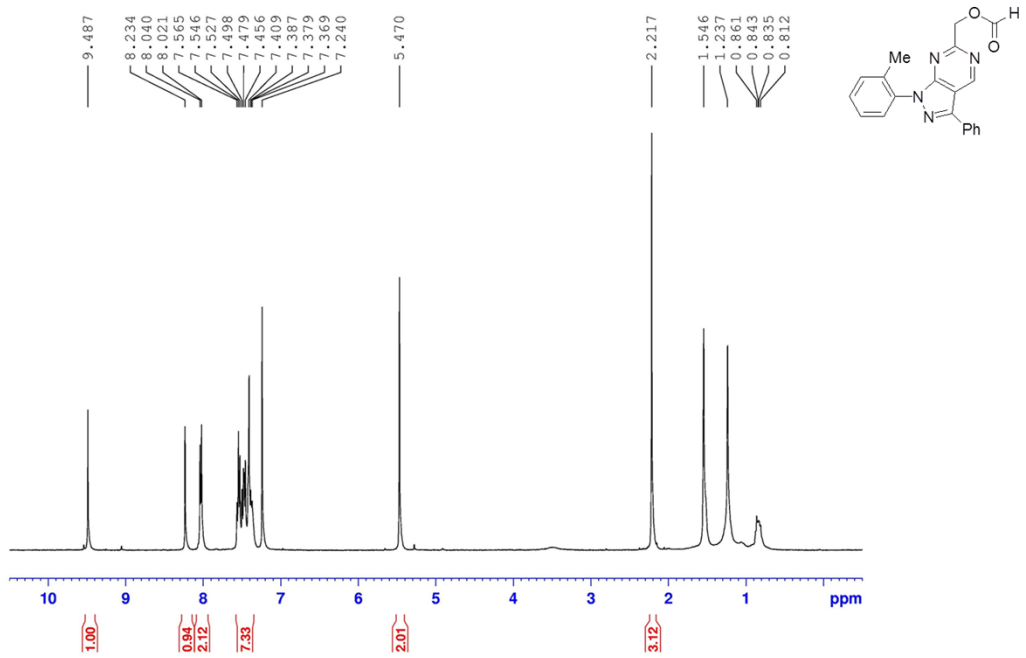


^{13}C-NMR

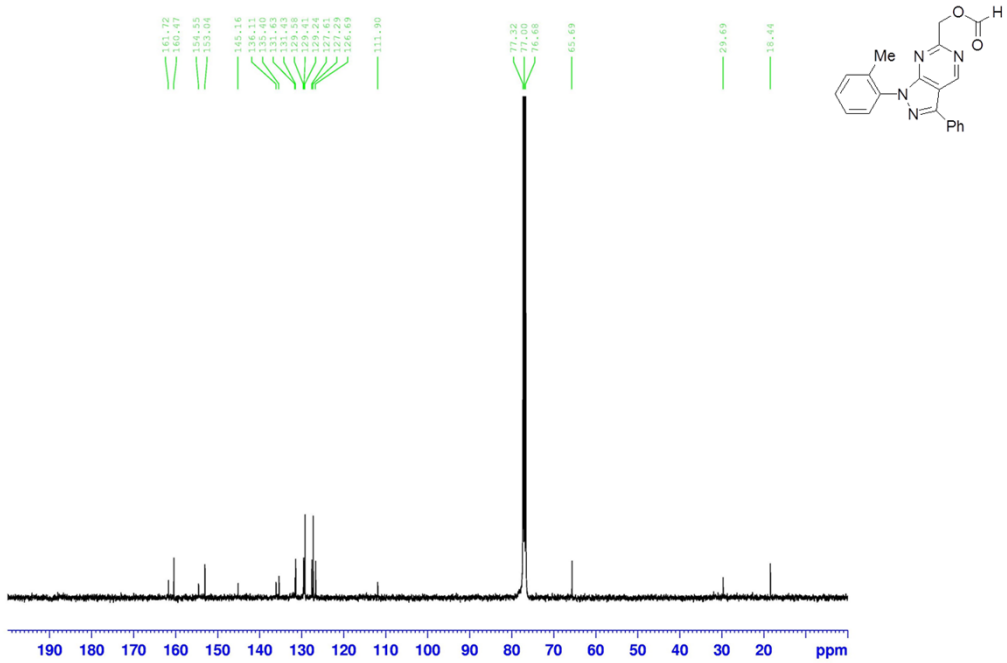


6-[(Formyloxy)methyl]-1-(2-methylphenyl)-3-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine **18**

^{1}H-NMR

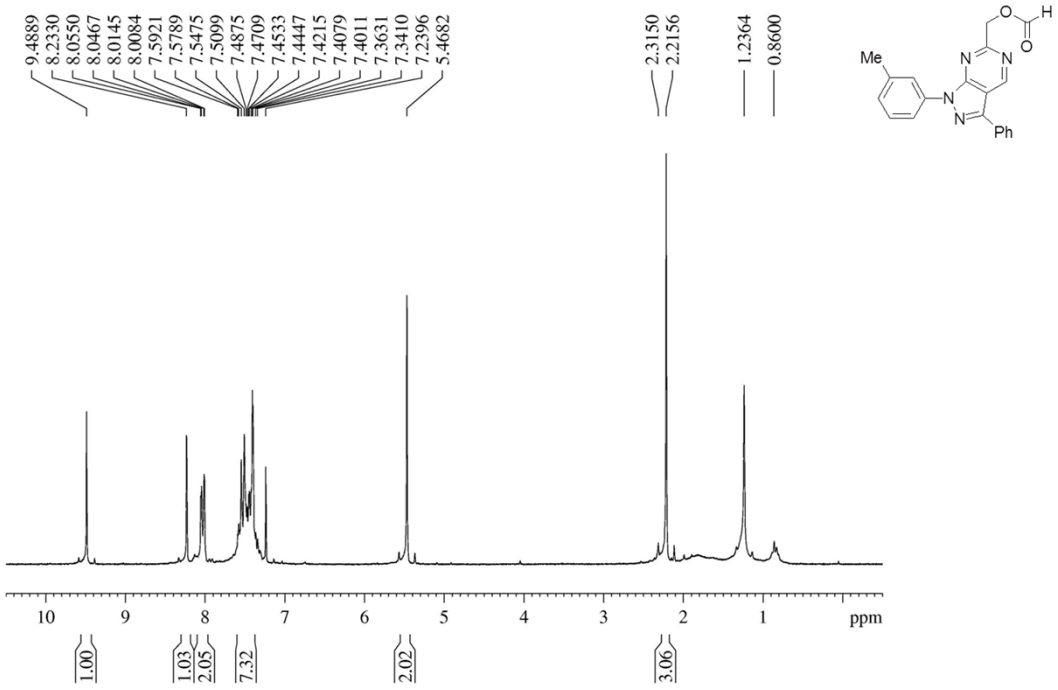


^{13}C-NMR

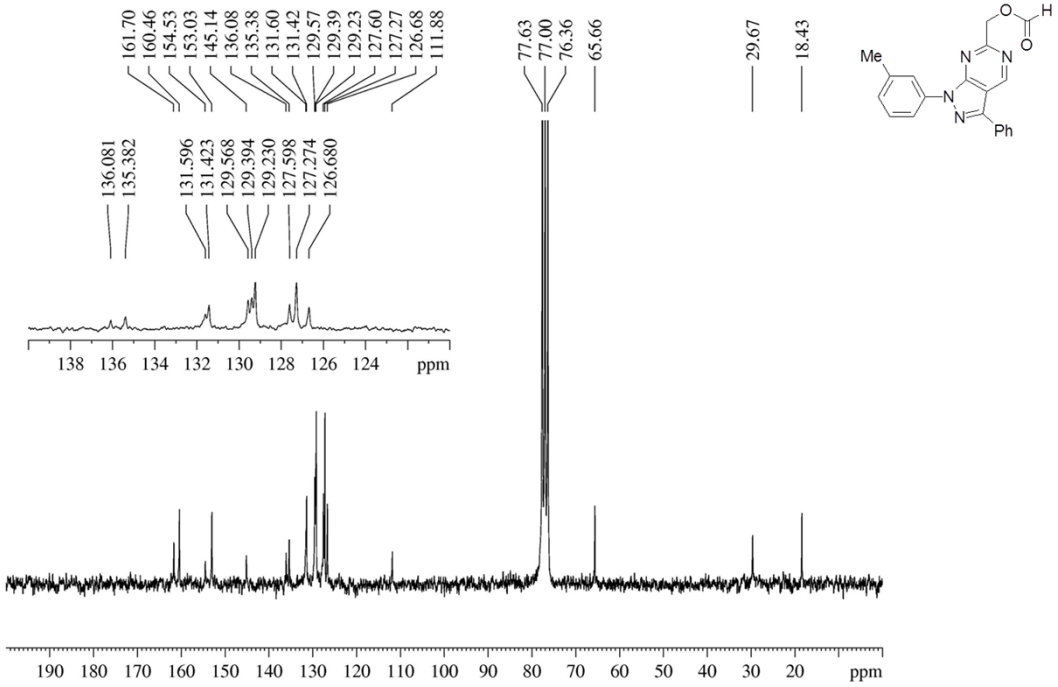


6-[(Formyloxy)methyl]-1-(3-methylphenyl)-3-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine **19**

^{1}H-NMR

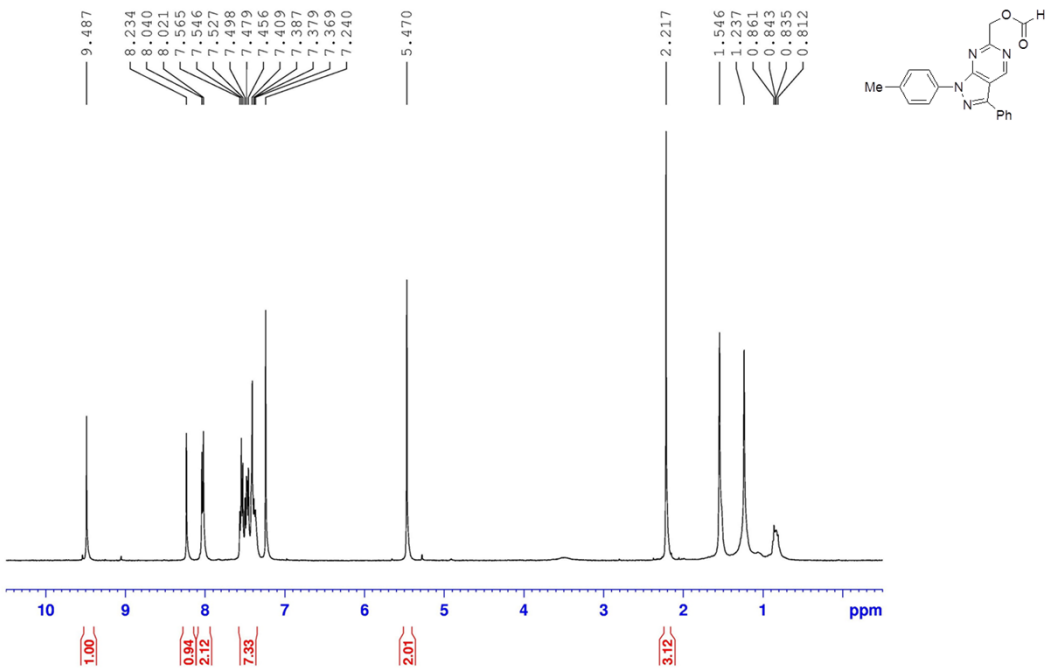


^{13}C-NMR

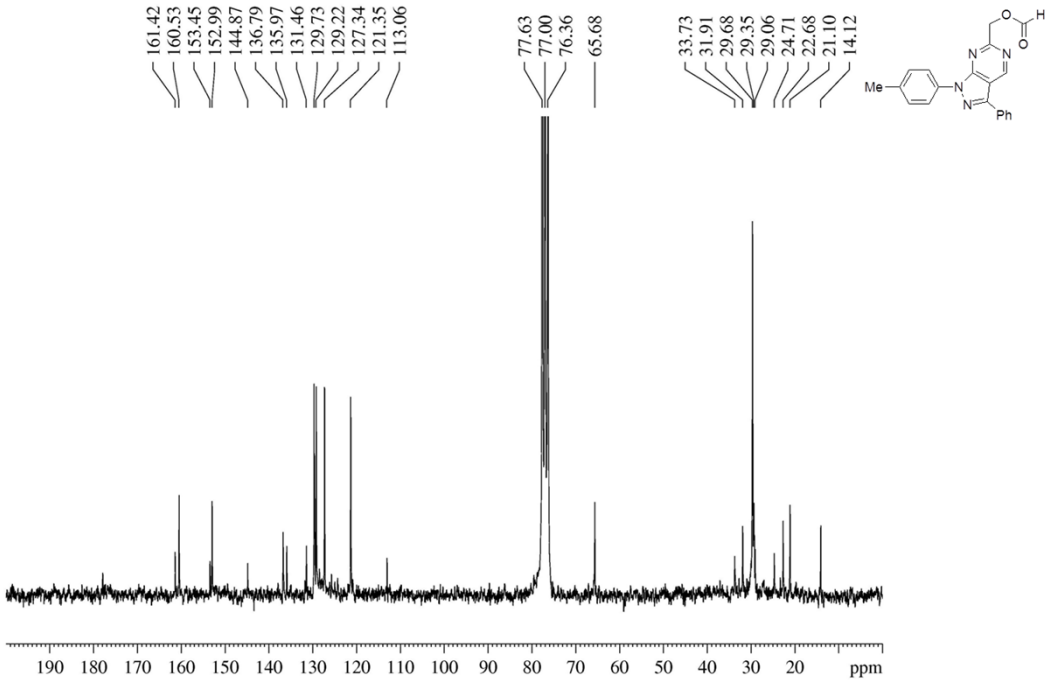


6-[(Formyloxy)methyl]-1-(4-methylphenyl)-3-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine **20**

^{1}H-NMR

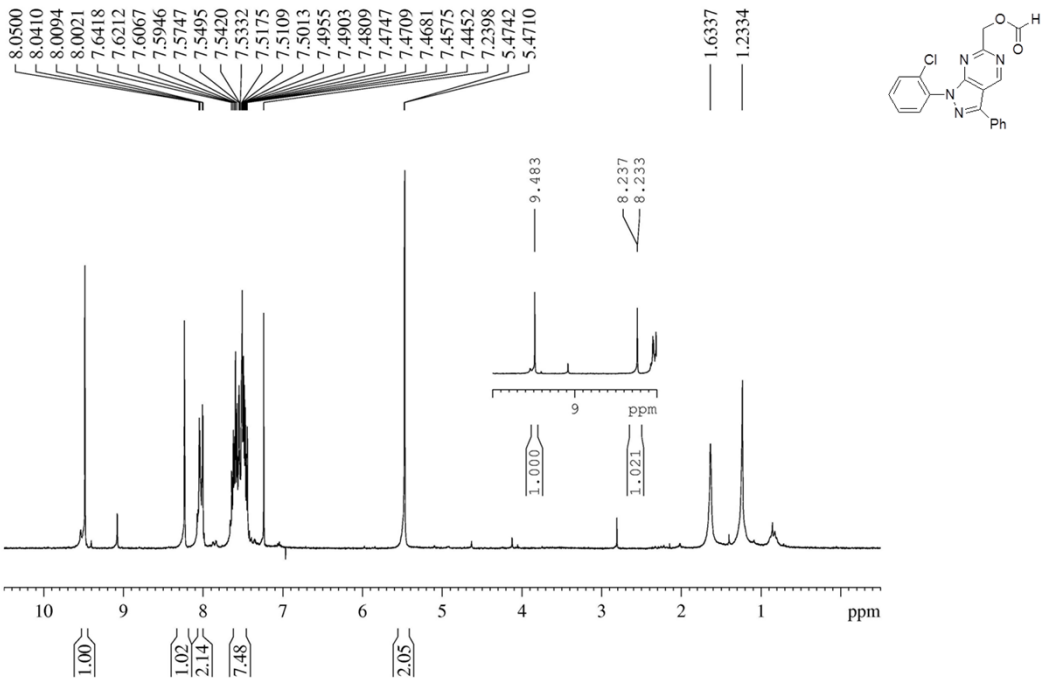


^{13}C-NMR

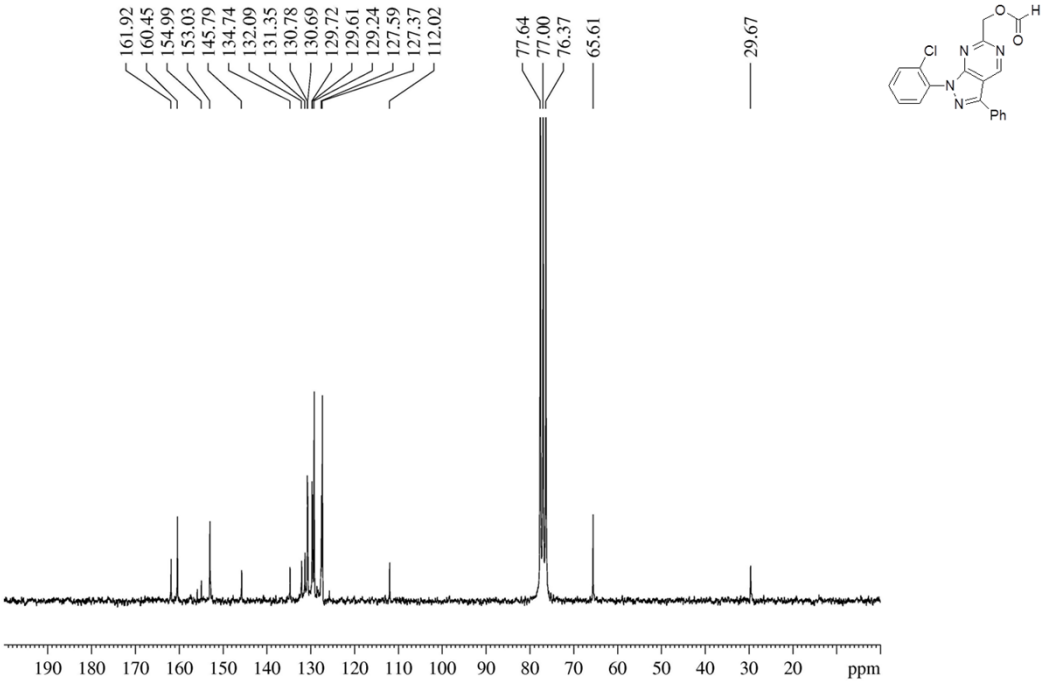


1-(2-Chlorophenyl)-6-[(formyloxy)methyl]-3-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine **21**

^{1}H-NMR

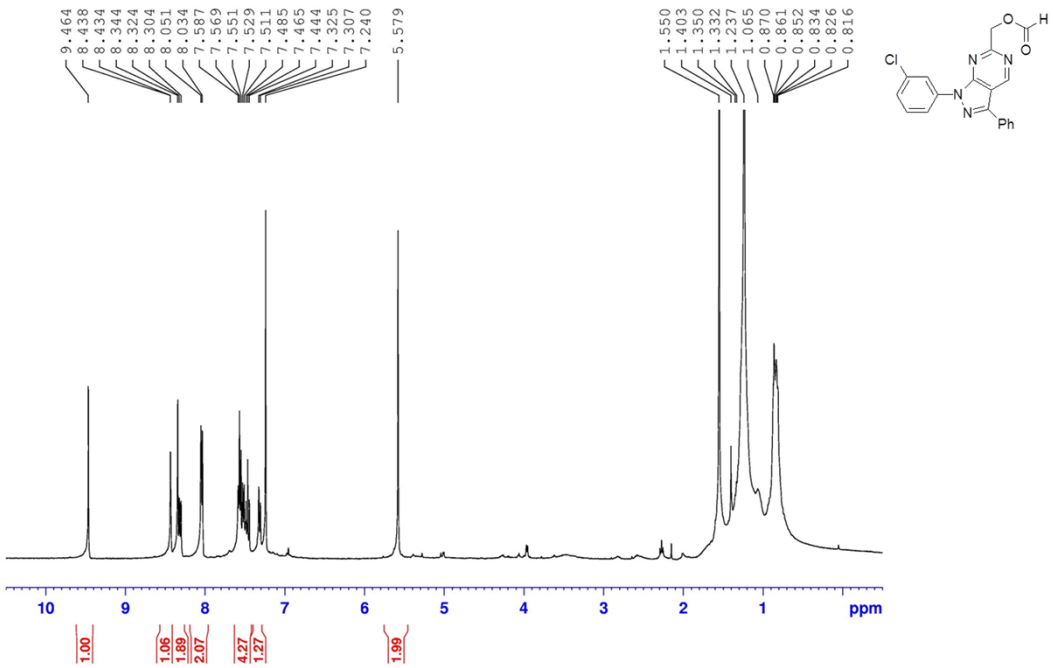


^{13}C-NMR

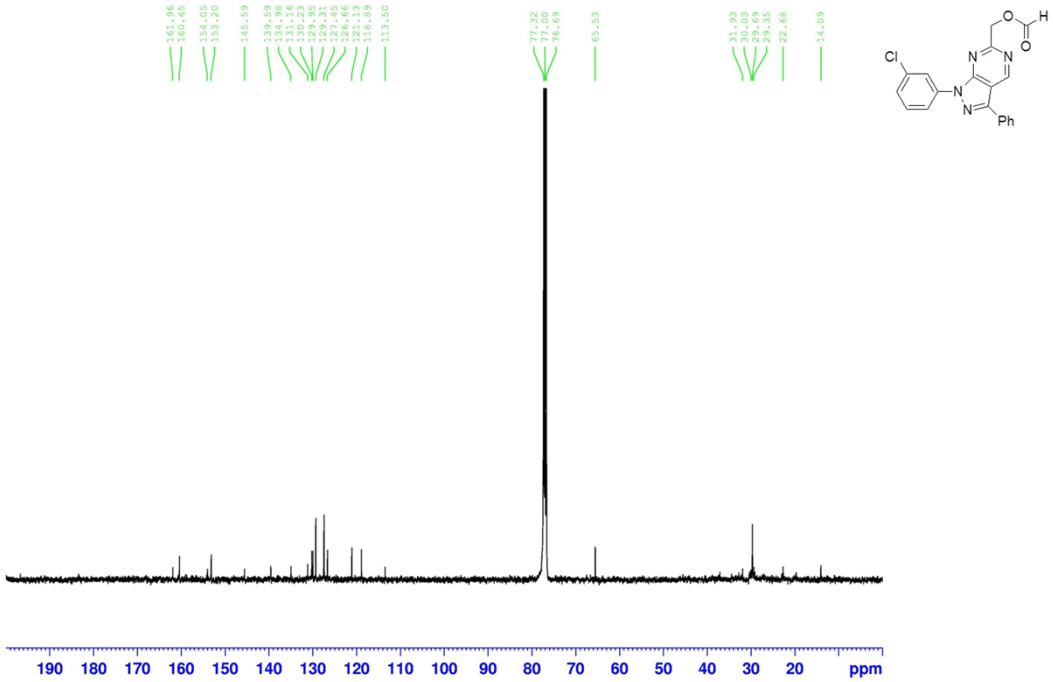


1-(3-Chlorophenyl)-6-[(formyloxy)methyl]-3-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine **22**

^{1}H-NMR

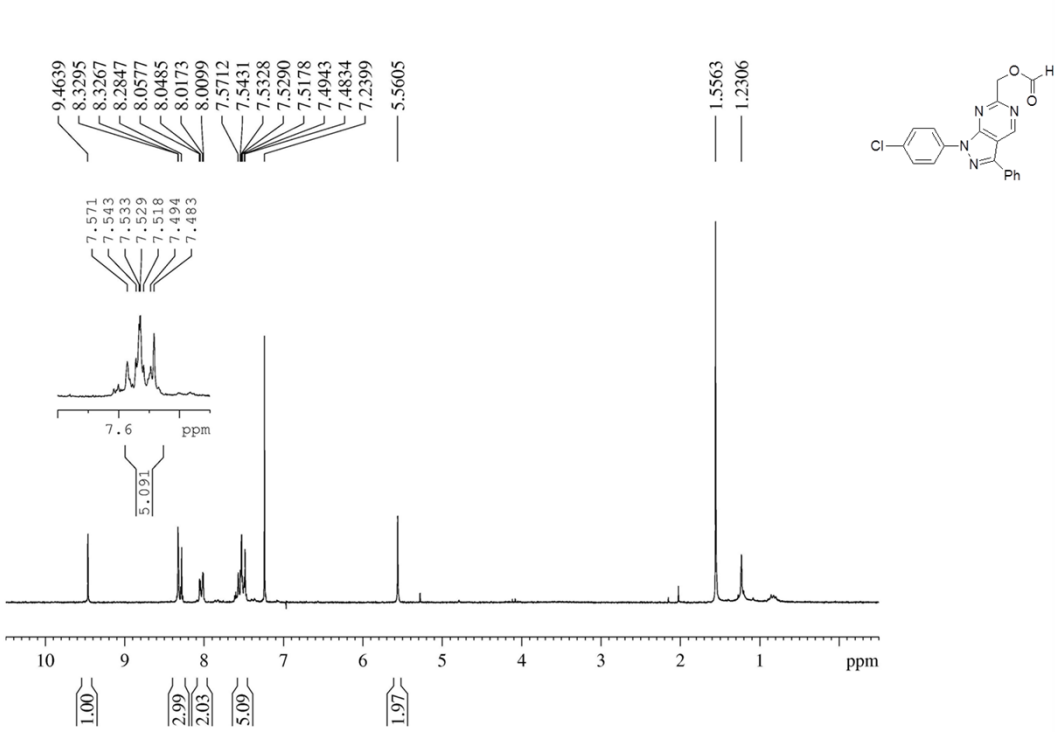


^{13}C-NMR

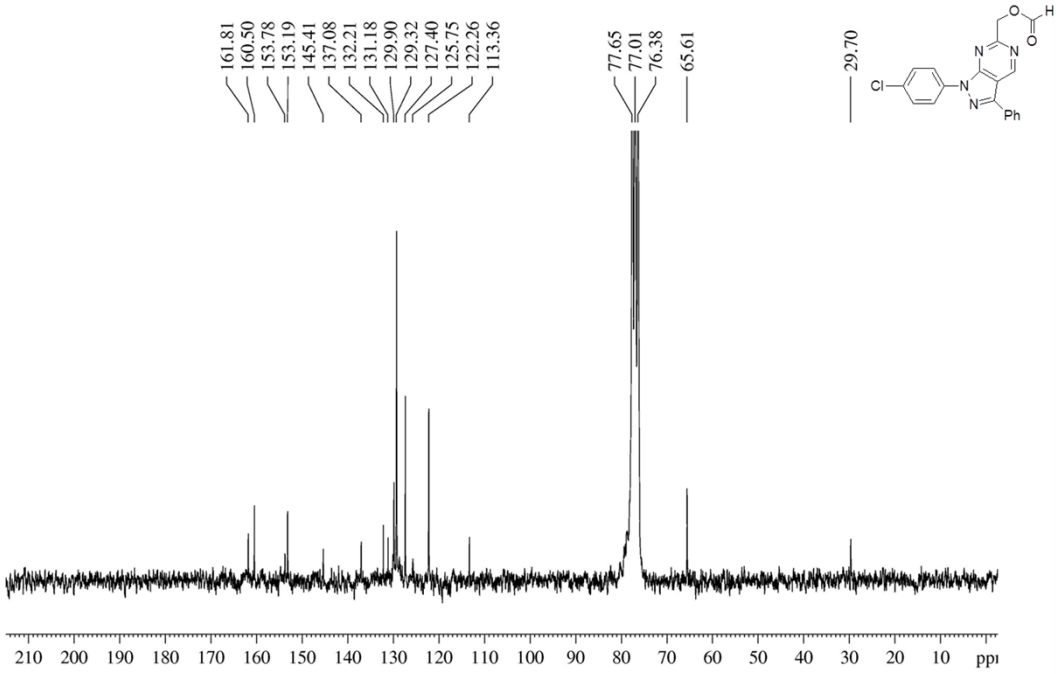


1-(4-Chlorophenyl)-6-[(formyloxy)methyl]-3-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine **23**

[1]H-NMR

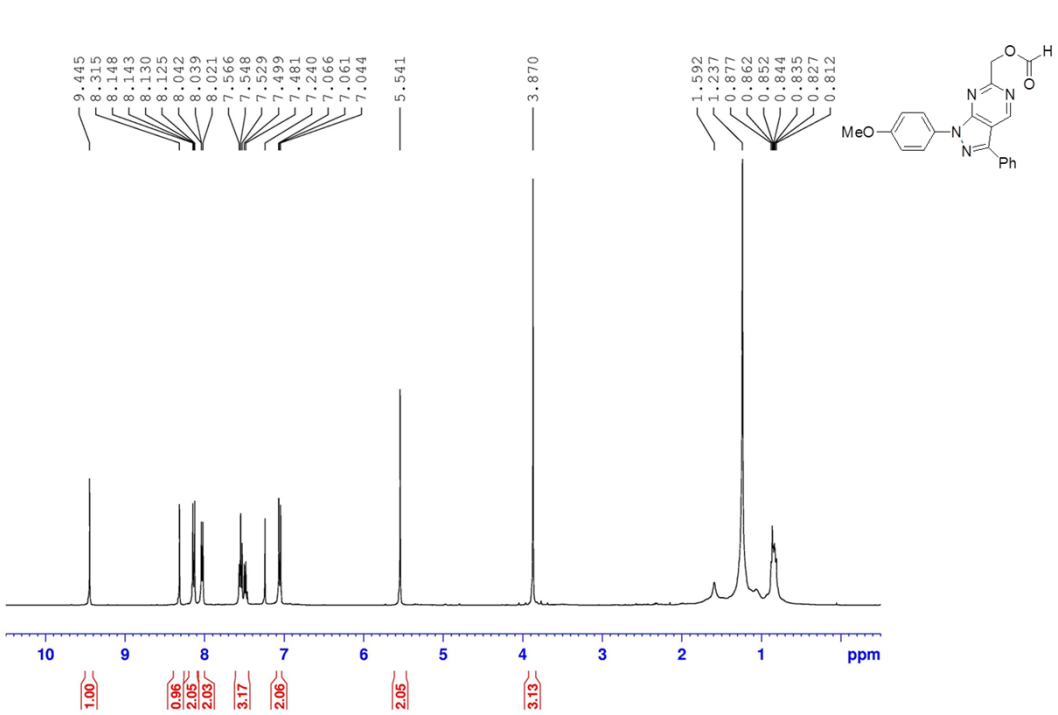


[13]C-NMR

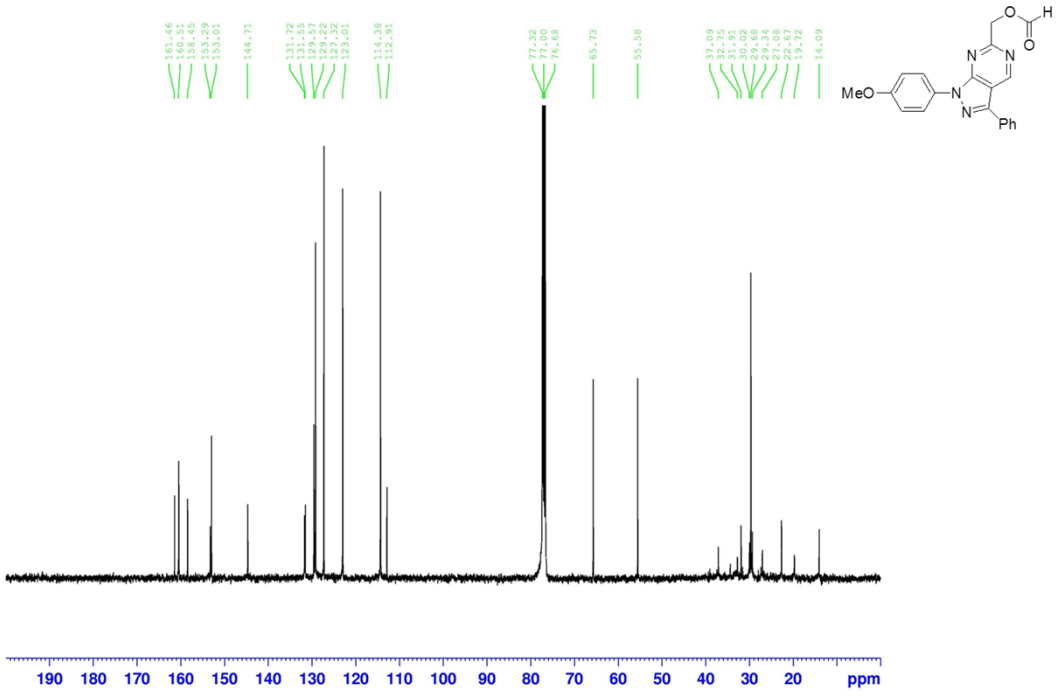


6-[(Formyloxy)methyl]-1-(3-methoxyphenyl)-3-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine **24**

^{1}H-NMR

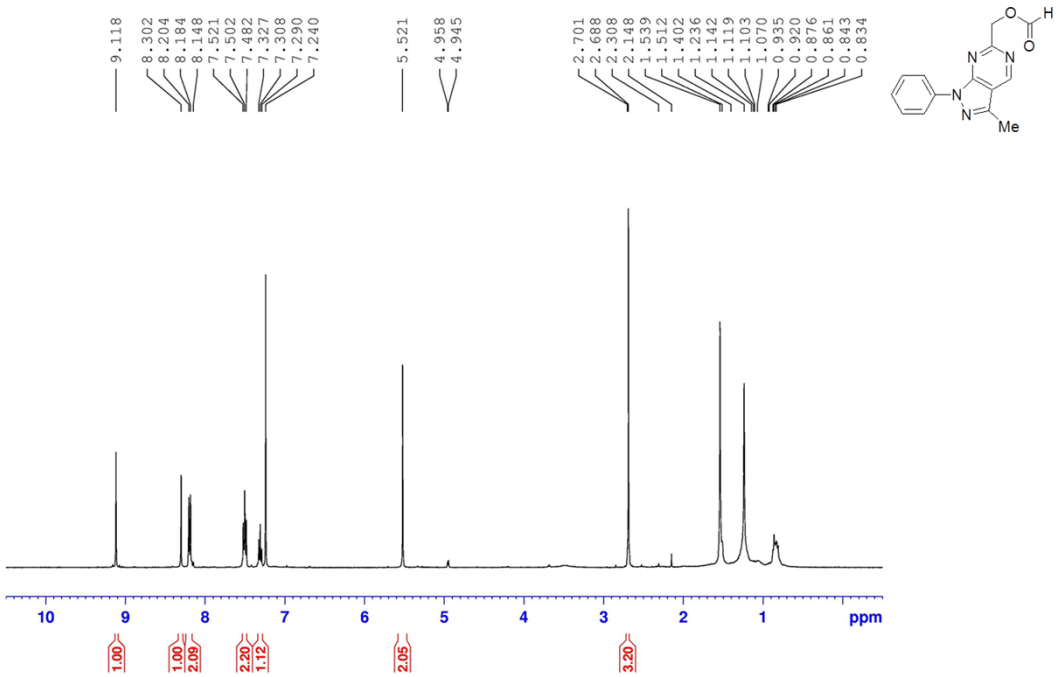


^{13}C-NMR

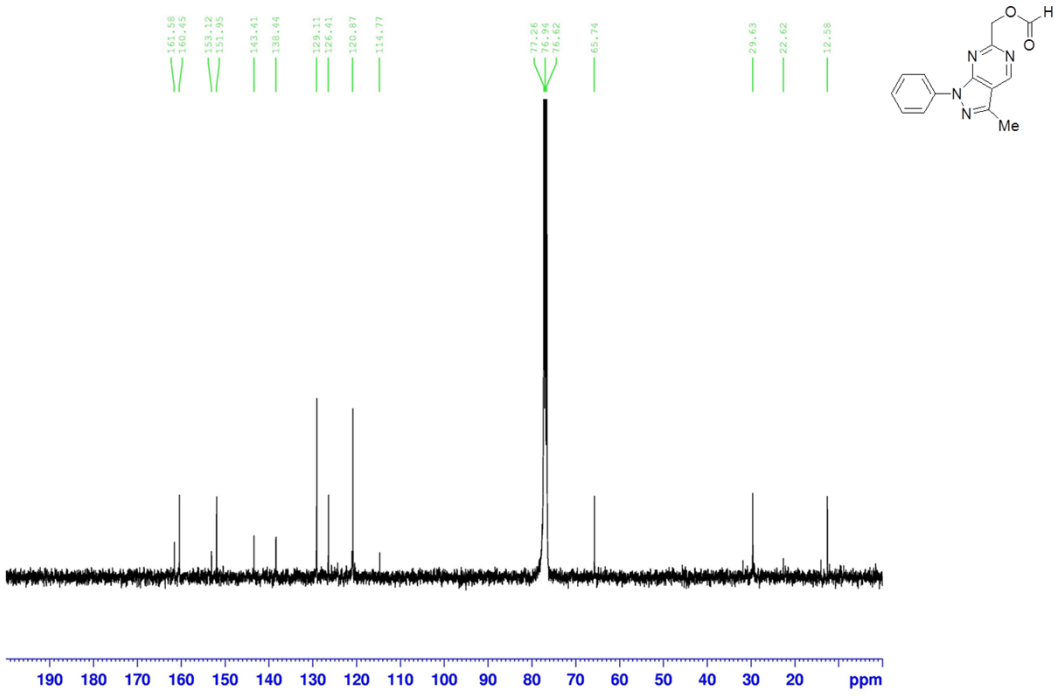


6-[(Formyloxy)methyl]-3-methyl-1-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine **25**

^{1}H-NMR

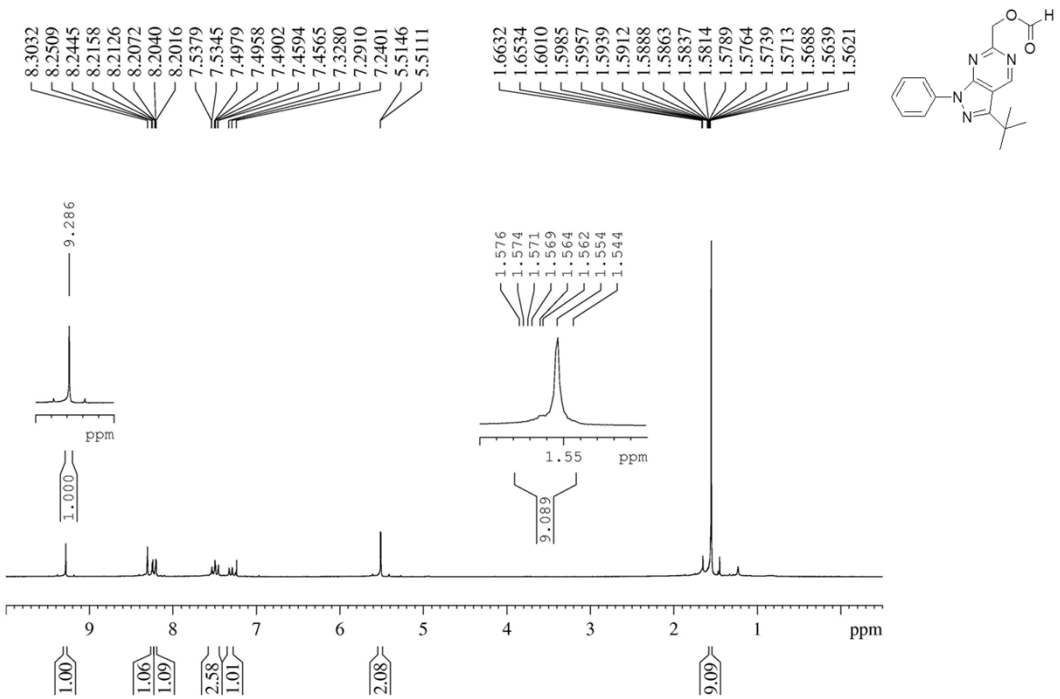


^{13}C-NMR

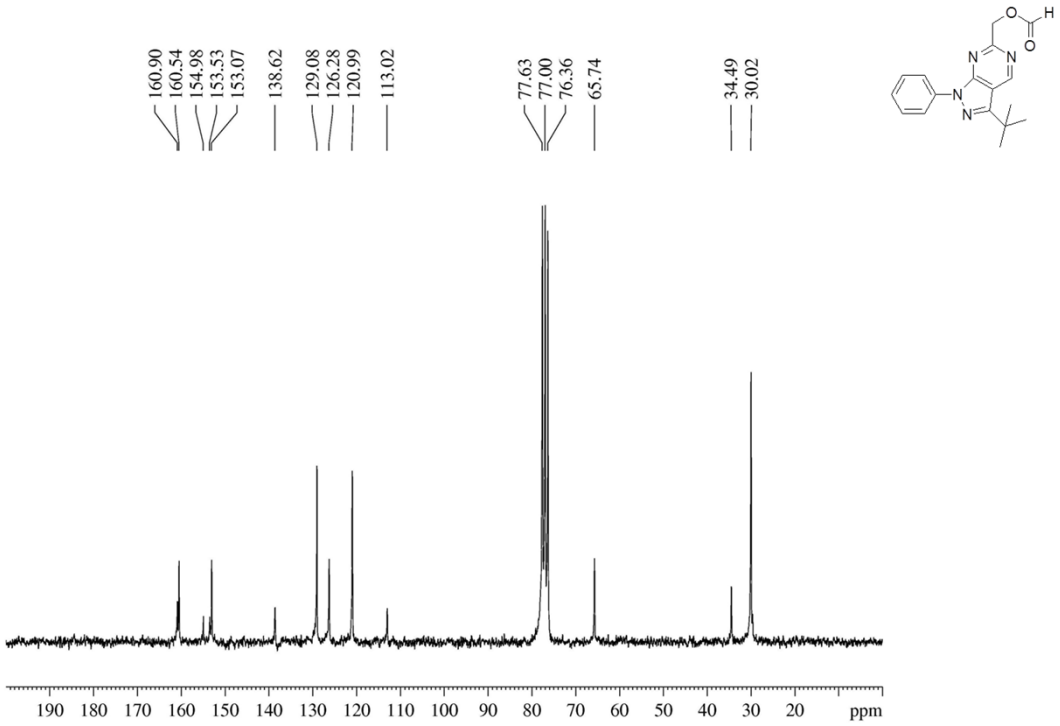


3-Tetrabutanyl-6-[(formyloxy)methyl]-1-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine **26**

^{1}H-NMR

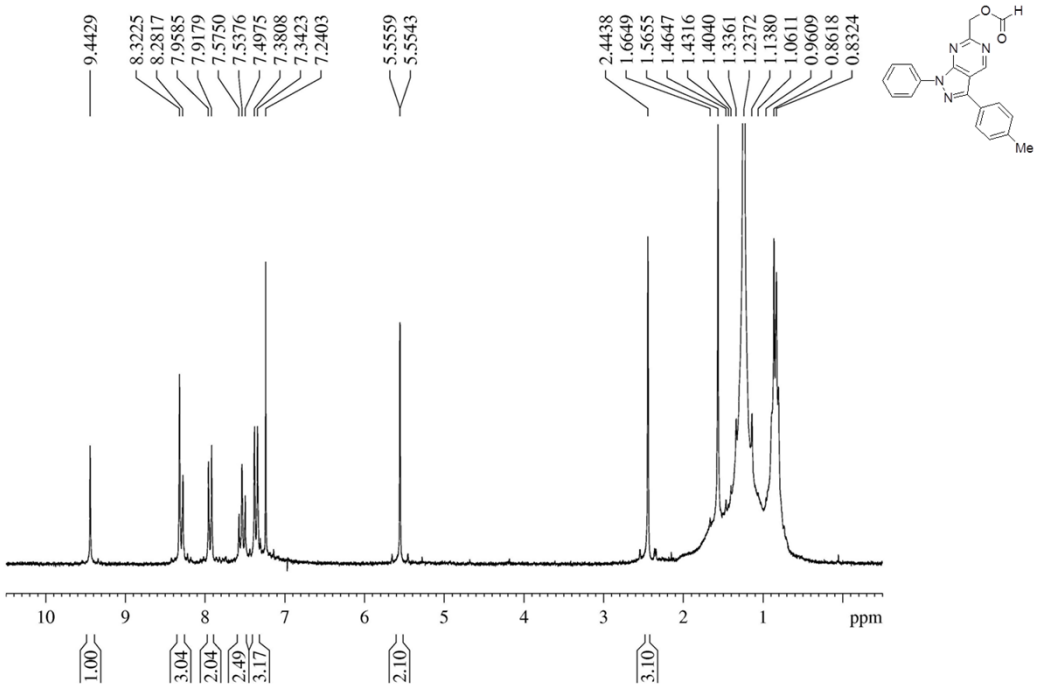


^{13}C-NMR

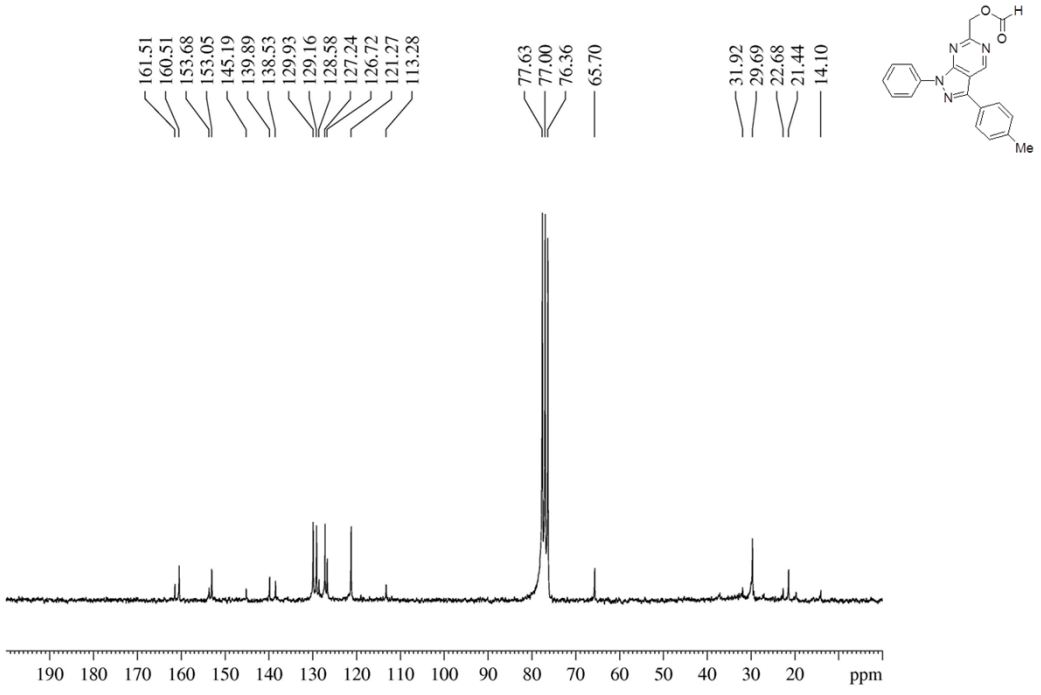


6-[(Formyloxy)methyl]-3-(4-methylphenyl)-1-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine **27**

^{1}H-NMR

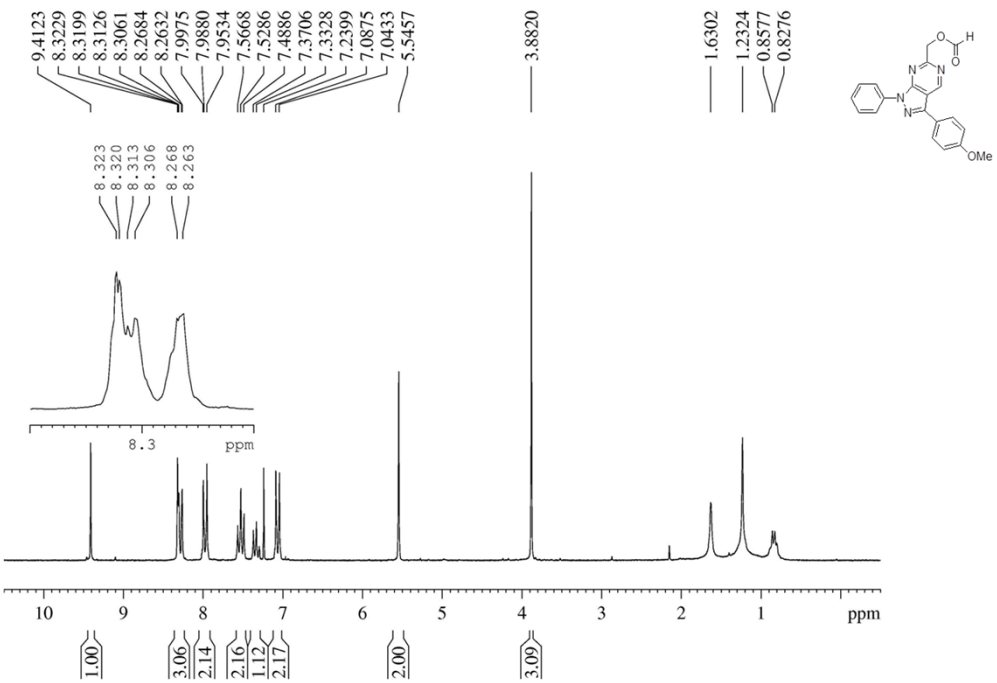


^{13}C-NMR

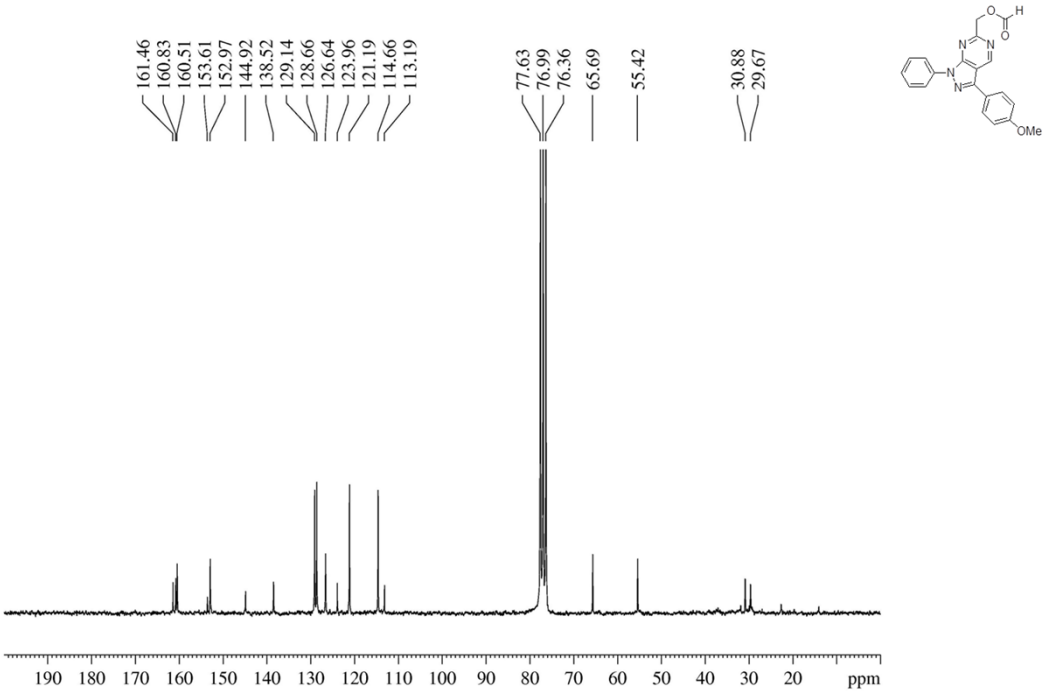


6-[(Formyloxy)methyl]-3-(4-methoxyphenyl)-1-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine **28**

^{1}H-NMR

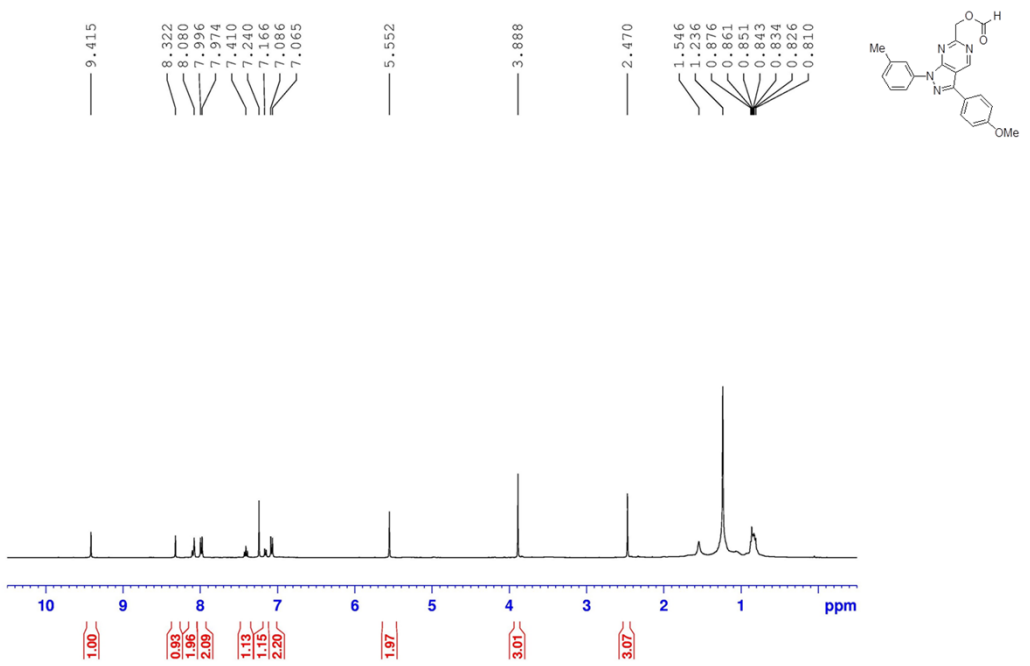


^{13}C-NMR

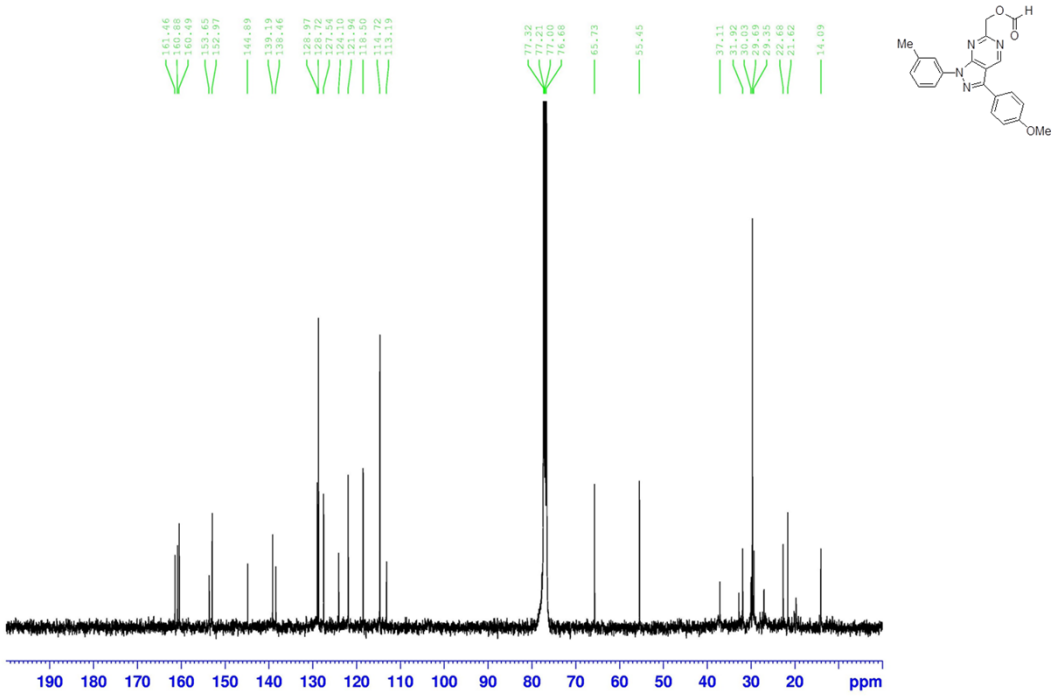


6-[(Formyloxy)methyl]-1-(3-methlyphenyl)-3-(4-methoxyphenyl)-1*H*-pyrazolo[3,4-*d*]pyrimidine **29**

^{1}H-NMR

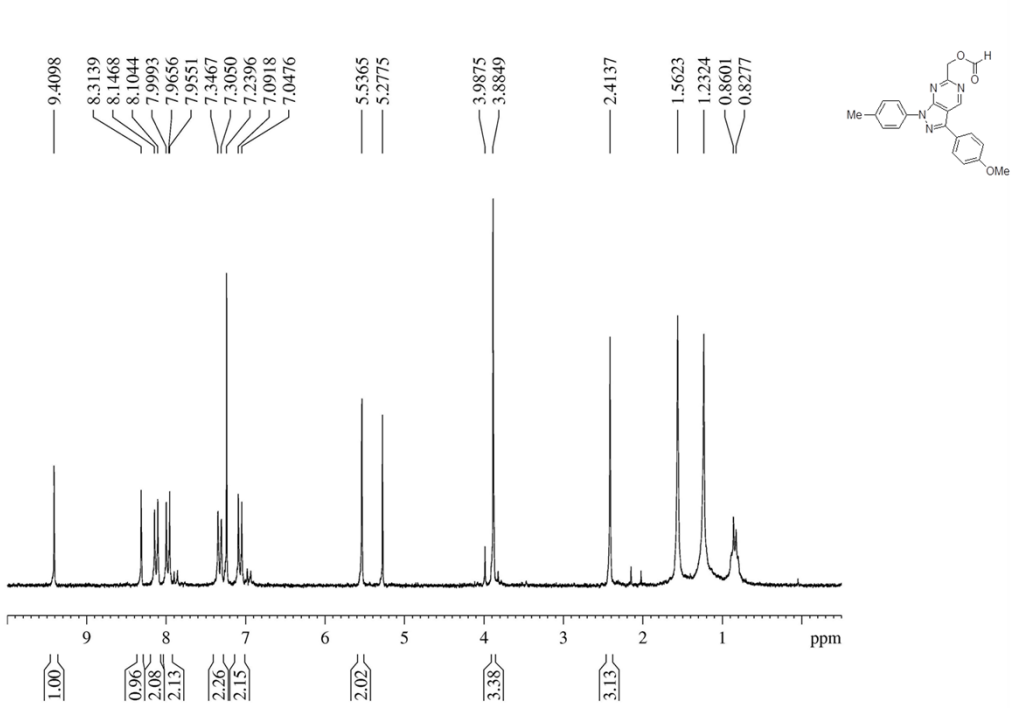


^{13}C-NMR

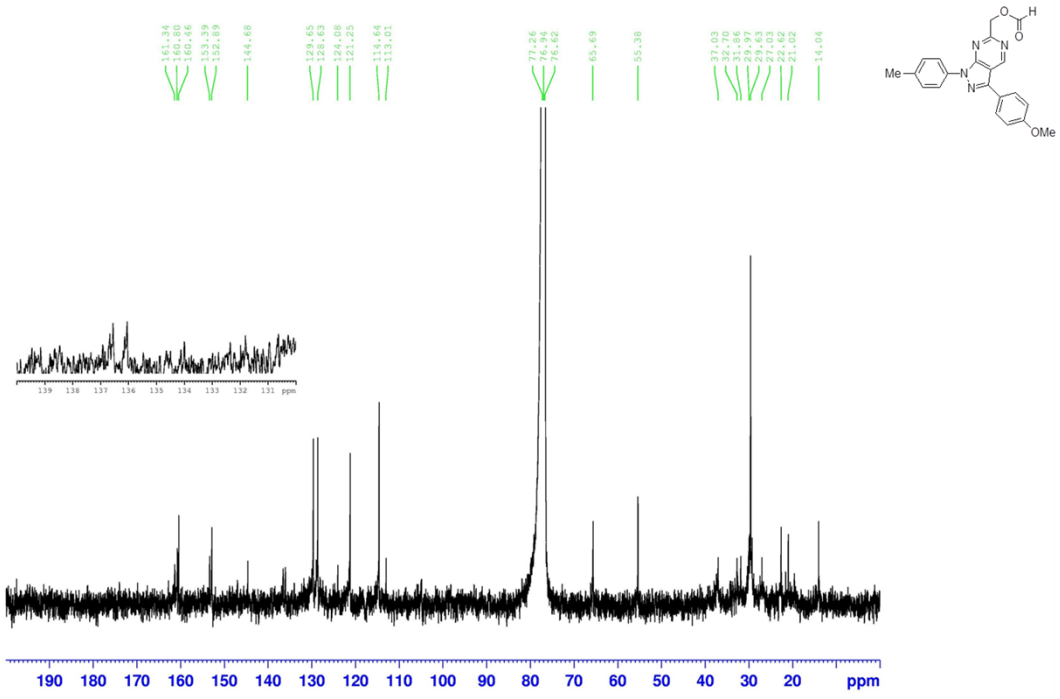


6-[(Formyloxy)methyl]-1-(4-methlyphenyl)-3-(4-methoxyphenyl)-1*H*-pyrazolo[3,4-*d*]pyrimidine **30**

^{1}H-NMR

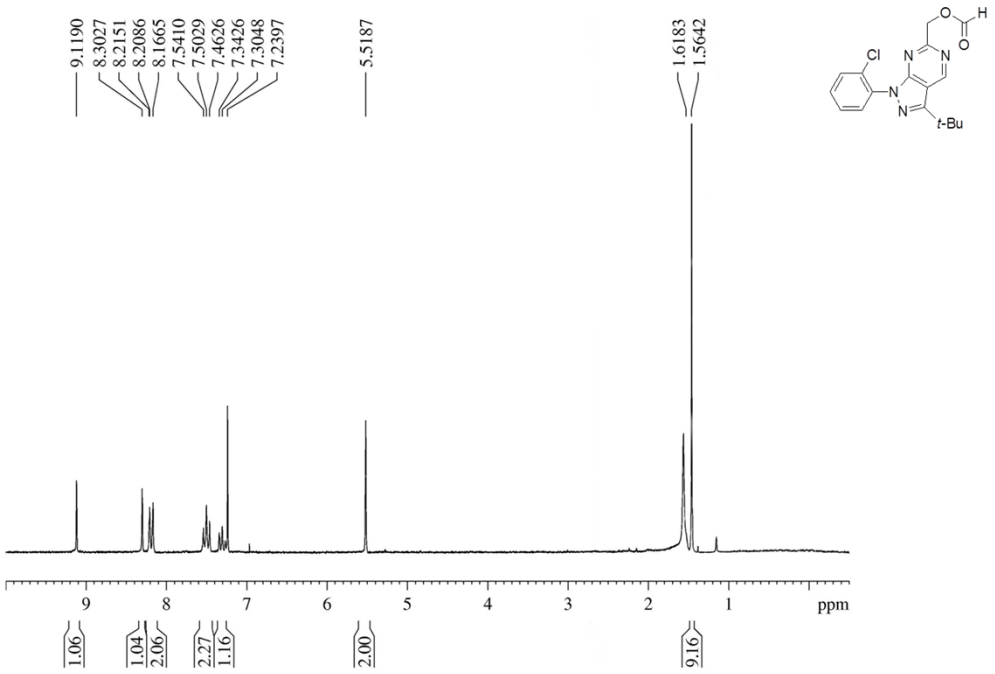


^{13}C-NMR

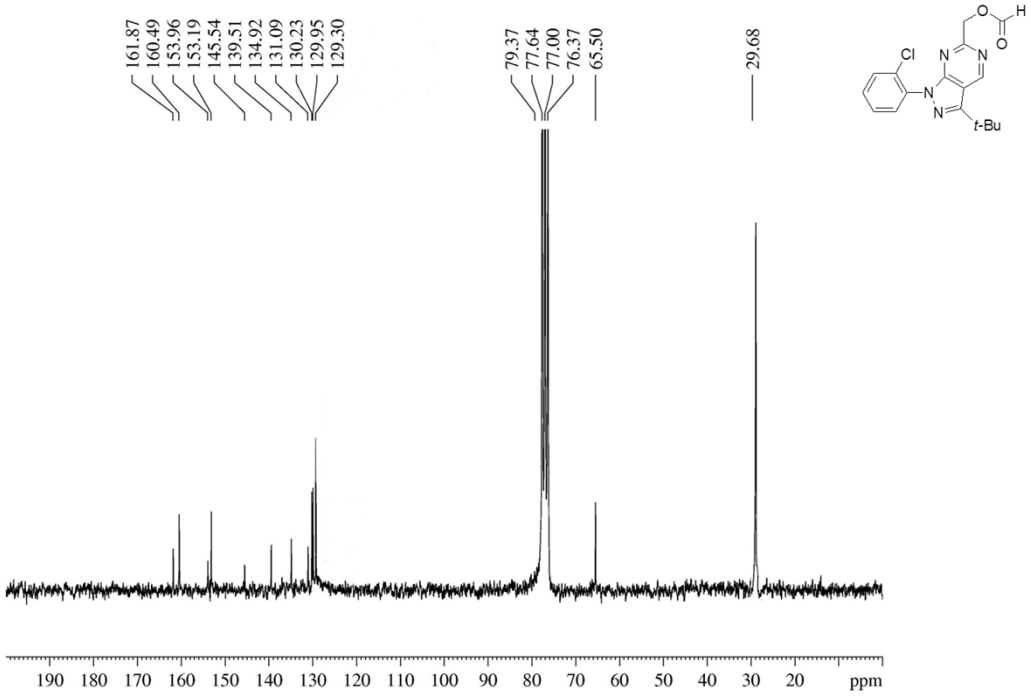


6-[(Formyloxy)methyl]-1-(2-methlyphenyl)-3-(4-methoxyphenyl)-1*H*-pyrazolo[3,4-*d*]pyrimidine **31**

^{1}H-NMR

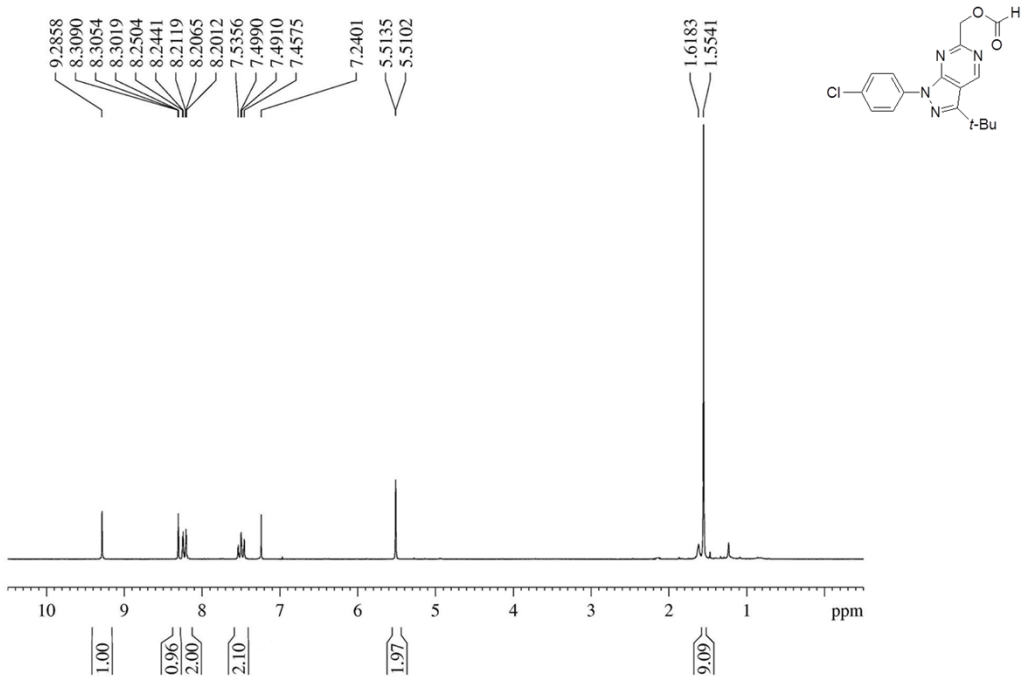


^{13}C-NMR

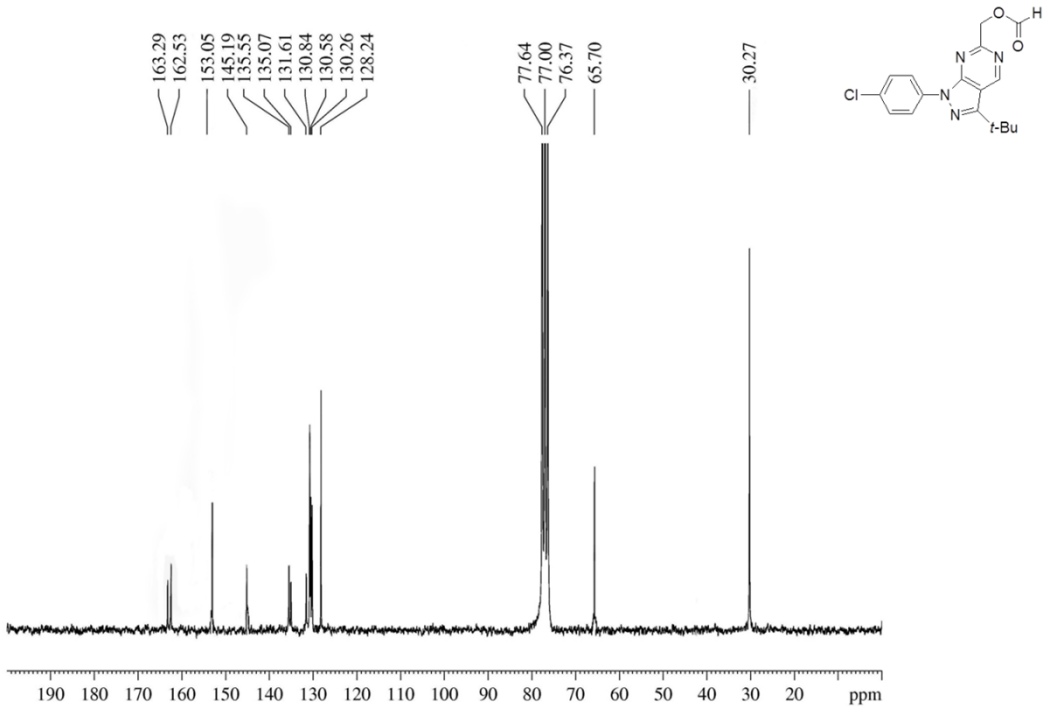


6-[(Formyloxy)methyl]-1-(4-methlyphenyl)-3-(4-methoxyphenyl)-1*H*-pyrazolo[3,4-*d*]pyrimidine **32**

^{1}H-NMR



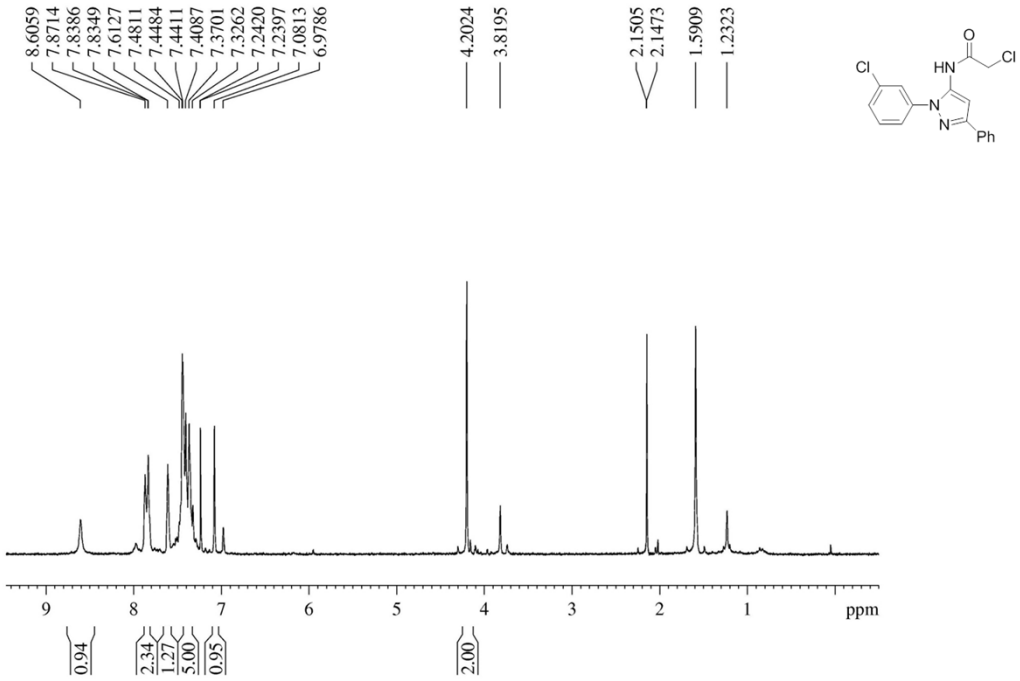
^{13}C-NMR



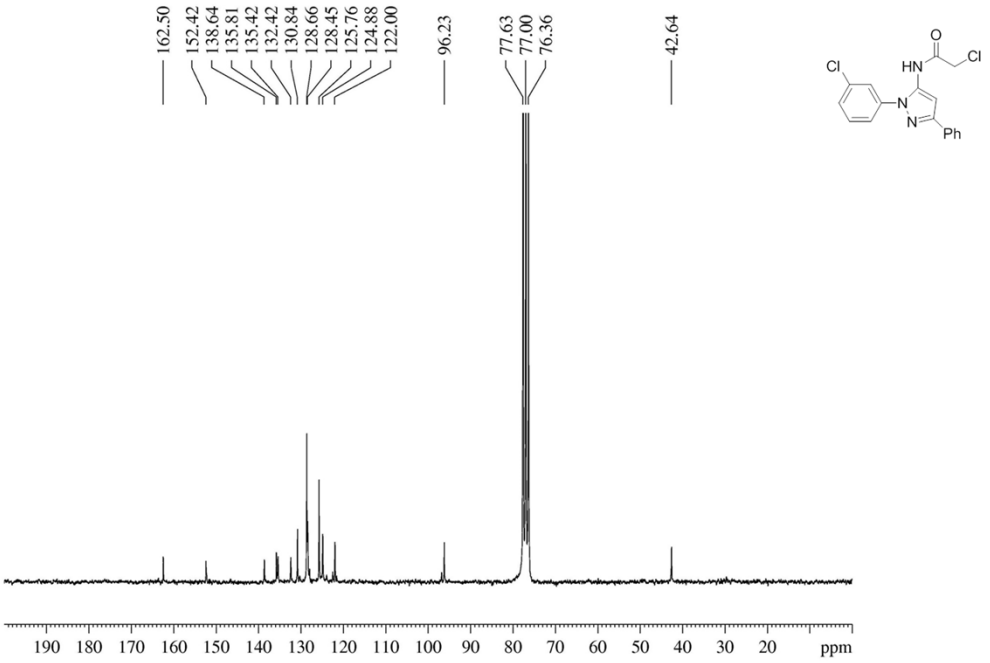
^{1}H-NMR and ^{13}C-NMR of 5-(2-haloacetylamino)pyrazoles 6 and 33–35

5-(2-Chloroacetylamino)-1-(3-chlorophenyl)-3-phenylpyrazole **6**

^{1}H-NMR

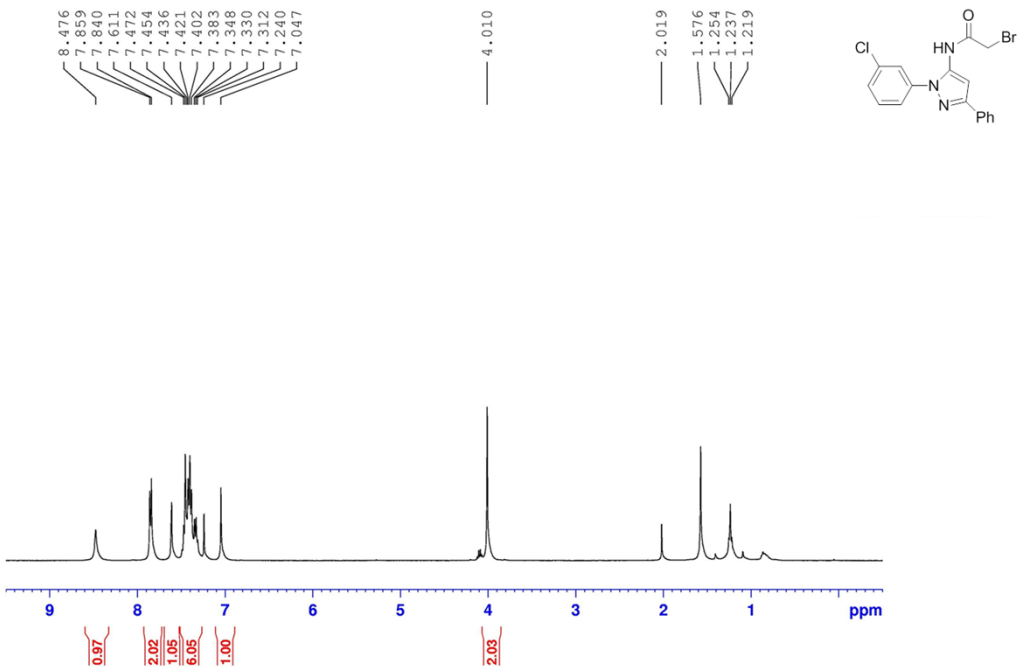


^{13}C-NMR

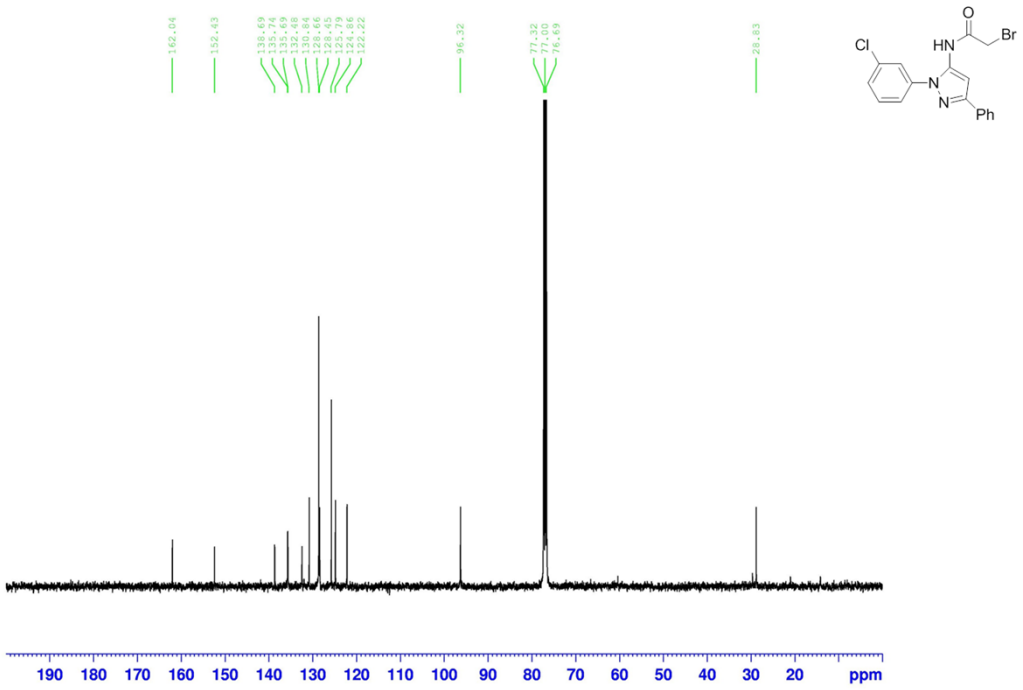


5-(2-Bromoacetylamino)-1-(3-chlorophenyl)-3-phenylpyrazole **33**

^{1}H-NMR

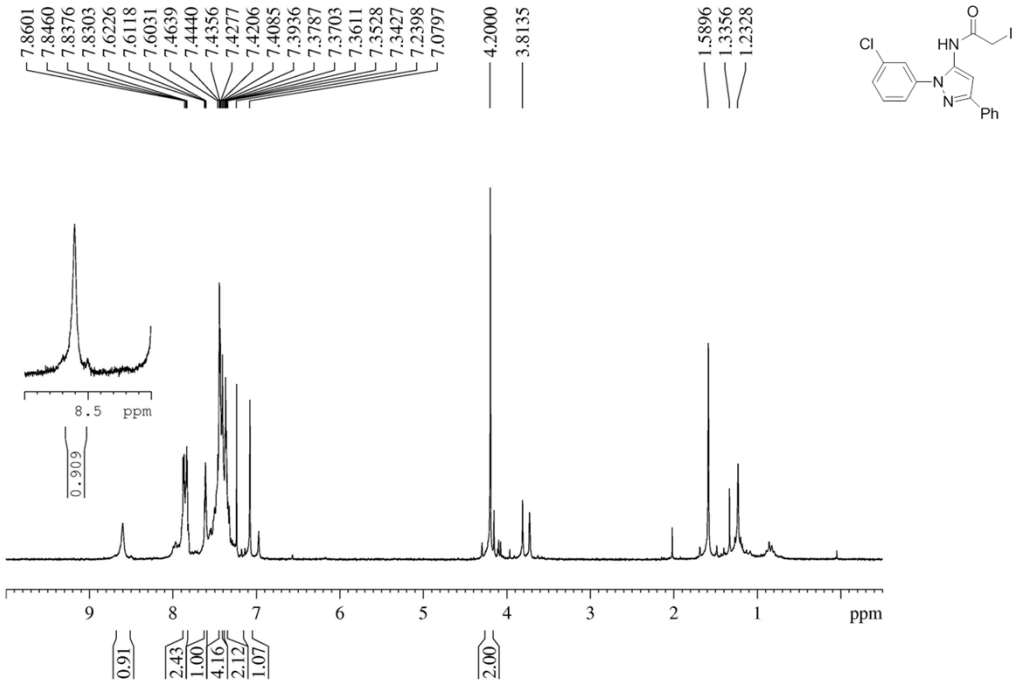


^{13}C-NMR

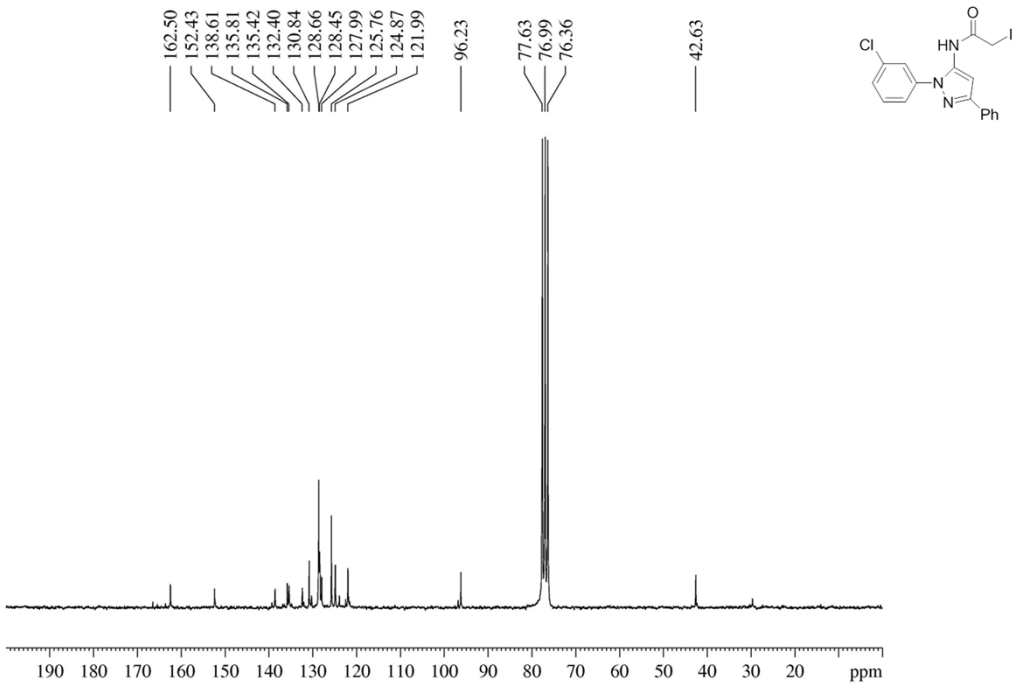


1-(3-Chlorophenyl)-5-(2-iodoacetylamino)-3-phenylpyrazole **34**

^{1}H-NMR

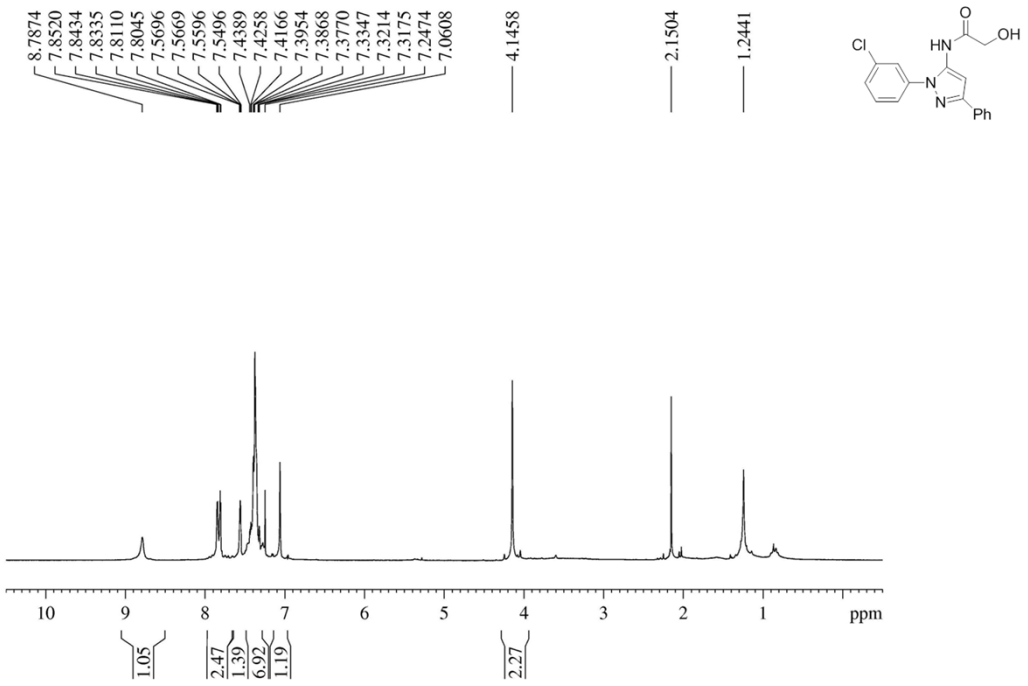


^{13}C-NMR

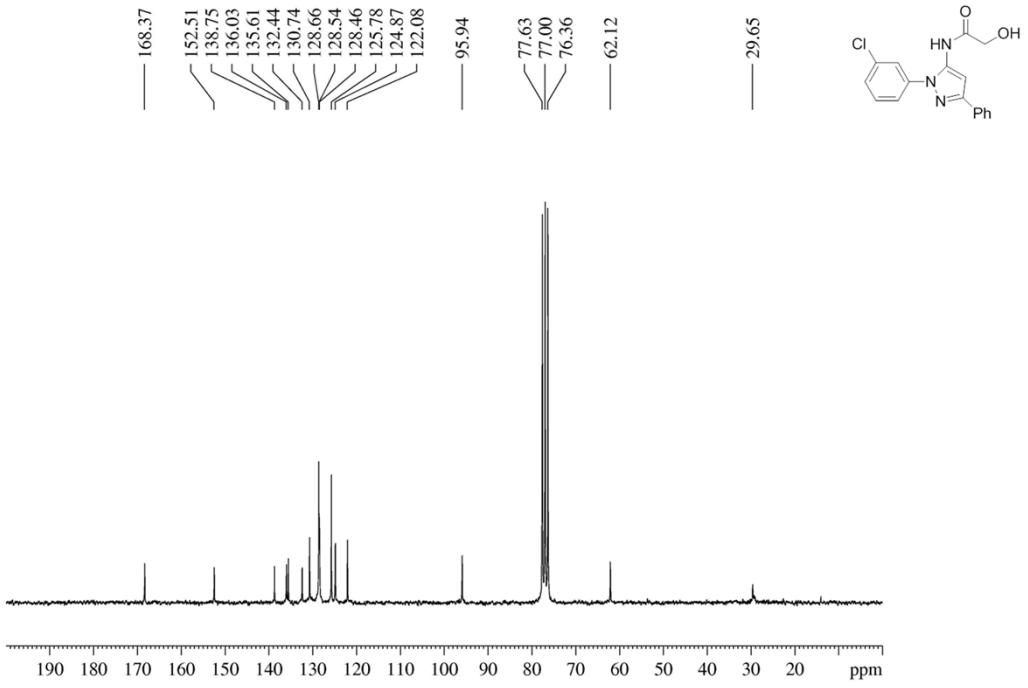


1-(3-Chlorophenyl)-5-(2-hydroxyacetylamino)-3-phenylpyrazole **35**

^{1}H-NMR

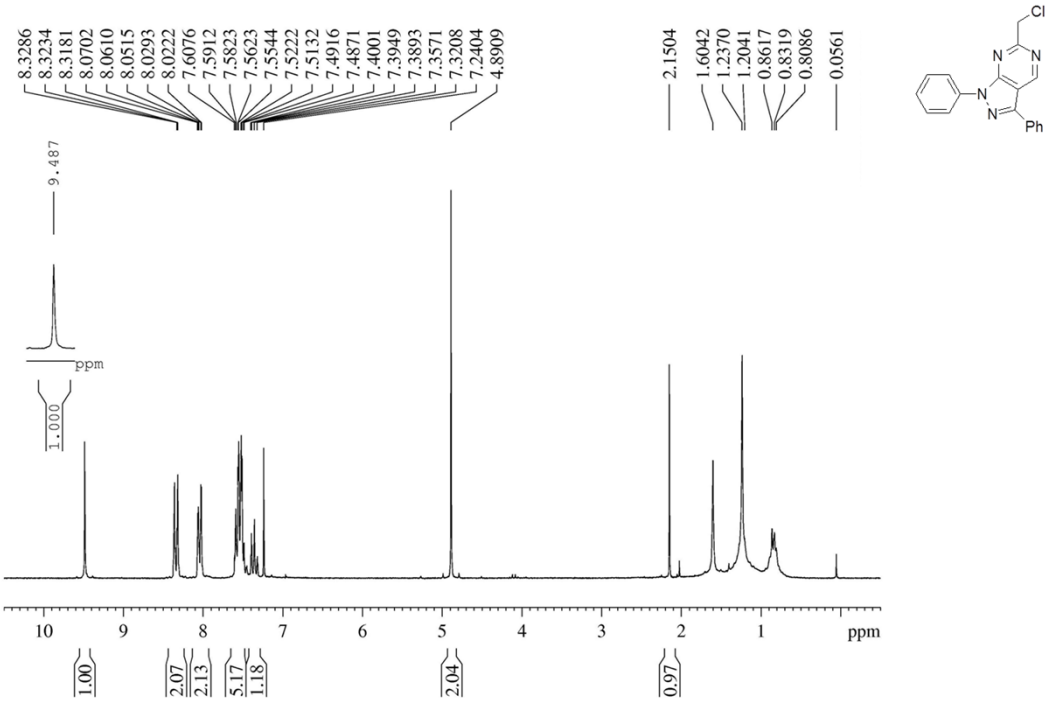


^{13}C-NMR

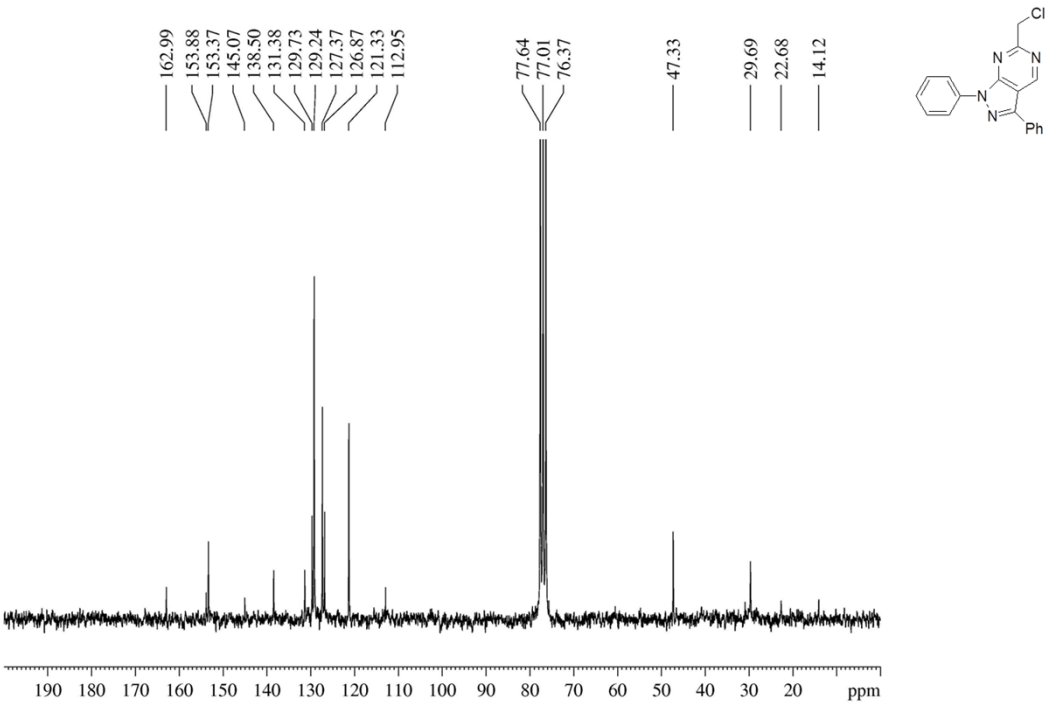


6-(Chloromethyl)pyrazolo[3,4-*d*]pyrimidine **40**

^{1}H-NMR



^{13}C-NMR



X-Ray data of 6-[(Formyloxy)methyl]-1-(3-methoxyphenyl)-3-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine 24

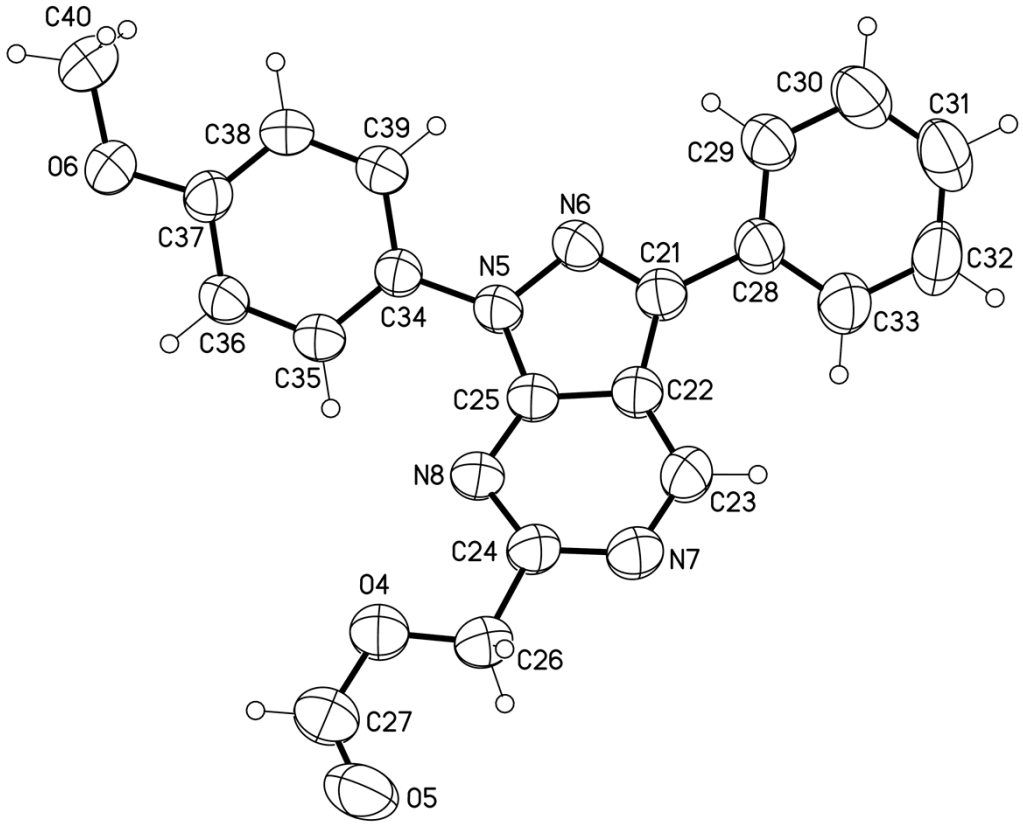


Table 1. Crystal data and structure refinement for 130527 (CCDC 1027455).

Identification code	130527	
Empirical formula	C20 H16 N4 O3	
Formula weight	360.37	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	P 1 21/n 1	
Unit cell dimensions	a = 14.1926(3) Å	□= 90°.
	b = 7.2123(2) Å	□= 98.900(2)°.
	c = 34.1402(8) Å	□ = 90°.
Volume	3452.56(15) Å^3	
Z	8	
Density (calculated)	1.387 Mg/m^3	
Absorption coefficient	0.791 mm^{-1}	
F(000)	1504	
Crystal size	0.12 x 0.10 x 0.10 mm^3	

Theta range for data collection	3.22 to 65.78°.
Index ranges	-15<=h<=16, -3<=k<=8, -40<=l<=40
Reflections collected	18171
Independent reflections	5703 [R(int) = 0.0460]
Completeness to theta = 65.78°	95.2 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9492 and 0.8180
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5703 / 111 / 526
Goodness-of-fit on F^2	1.039
Final R indices [I>2sigma(I)]	R1 = 0.0586, wR2 = 0.1431
R indices (all data)	R1 = 0.0978, wR2 = 0.1682
Largest diff. peak and hole	0.309 and -0.235 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 130527. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	9412(2)	3209(5)	731(1)	52(1)
C(2)	8917(2)	3084(5)	336(1)	54(1)
C(3)	9112(3)	2877(6)	-48(1)	68(1)
C(4)	7510(2)	2956(5)	-281(1)	64(1)
C(5)	7950(2)	3225(4)	369(1)	52(1)
C(6)	6810(3)	2821(7)	-652(1)	90(1)
C(7)	5249(3)	2246(9)	-885(1)	109(2)
C(8)	10439(2)	3146(5)	879(1)	57(1)
C(9)	10805(2)	3923(5)	1242(1)	63(1)
C(10)	11766(3)	3888(6)	1381(1)	72(1)
C(11)	12386(3)	3088(6)	1161(1)	78(1)
C(12)	12038(3)	2302(7)	800(1)	84(1)
C(13)	11068(2)	2328(6)	657(1)	73(1)
C(14)	7088(2)	3568(5)	962(1)	50(1)
C(15)	6218(2)	2822(5)	799(1)	63(1)
C(16)	5459(2)	2919(5)	1005(1)	63(1)
C(17)	5563(2)	3757(5)	1373(1)	56(1)
C(18)	6427(2)	4506(5)	1536(1)	56(1)
C(19)	7186(2)	4404(5)	1328(1)	54(1)
C(20)	4806(3)	4587(7)	1926(1)	86(1)
C(21)	10094(2)	8433(4)	1688(1)	51(1)
C(22)	9619(2)	8440(4)	1288(1)	51(1)
C(23)	9845(2)	8232(5)	909(1)	61(1)
C(24)	8252(2)	8375(5)	660(1)	56(1)
C(25)	8654(2)	8589(4)	1314(1)	49(1)
C(28)	11113(2)	8291(5)	1846(1)	55(1)
C(29)	11412(2)	7508(5)	2215(1)	62(1)
C(30)	12375(3)	7383(6)	2363(1)	73(1)
C(31)	13036(3)	8015(6)	2141(1)	80(1)
C(32)	12748(3)	8777(7)	1773(1)	83(1)
C(33)	11789(2)	8919(6)	1626(1)	69(1)
C(34)	7750(2)	8738(4)	1896(1)	46(1)
C(35)	6909(2)	9534(5)	1715(1)	53(1)

C(36)	6136(2)	9600(5)	1914(1)	53(1)
C(37)	6206(2)	8886(4)	2295(1)	50(1)
C(38)	7041(2)	8084(5)	2472(1)	54(1)
C(39)	7814(2)	8005(4)	2274(1)	52(1)
C(40)	5413(3)	8379(7)	2854(1)	82(1)
N(1)	7895(2)	3429(4)	762(1)	51(1)
N(2)	8794(2)	3423(4)	981(1)	57(1)
N(3)	8417(2)	2799(5)	-354(1)	75(1)
N(4)	7222(2)	3189(4)	66(1)	57(1)
N(5)	8570(2)	8666(4)	1703(1)	51(1)
N(6)	9459(2)	8557(4)	1933(1)	54(1)
N(7)	9163(2)	8197(4)	594(1)	63(1)
N(8)	7939(2)	8580(4)	1004(1)	54(1)
O(1)	5856(2)	2883(4)	-588(1)	82(1)
O(2)	5442(3)	1660(7)	-1180(1)	138(2)
O(3)	4762(2)	3738(4)	1552(1)	75(1)
O(6)	5395(2)	9068(4)	2465(1)	65(1)
O(4)	6612(5)	8303(18)	360(2)	82(2)
O(5)	6063(13)	8768(19)	-266(4)	89(3)
C(26)	7538(11)	8210(50)	274(8)	73(3)
C(27)	5920(8)	8500(20)	42(3)	85(2)
O(4')	6769(5)	7279(13)	335(2)	88(2)
O(5')	6014(13)	8050(20)	-238(3)	123(4)
C(26')	7552(10)	8410(40)	299(7)	69(2)
C(27')	6046(7)	7306(19)	46(3)	91(2)

Table 3. Bond lengths [Å] and angles [°] for 130527.

C(1)-N(2)	1.325(4)
C(1)-C(2)	1.423(4)
C(1)-C(8)	1.468(4)
C(2)-C(3)	1.389(4)
C(2)-C(5)	1.398(4)
C(3)-N(3)	1.322(4)
C(3)-H(3)	0.9500
C(4)-N(4)	1.323(4)
C(4)-N(3)	1.354(4)
C(4)-C(6)	1.487(5)
C(5)-N(4)	1.344(4)
C(5)-N(1)	1.363(4)
C(6)-O(1)	1.406(4)
C(6)-H(6A)	0.9900
C(6)-H(6B)	0.9900
C(7)-O(2)	1.165(5)
C(7)-O(1)	1.309(5)
C(7)-H(7)	0.9500
C(8)-C(9)	1.388(4)
C(8)-C(13)	1.388(4)
C(9)-C(10)	1.373(5)
C(9)-H(9)	0.9500
C(10)-C(11)	1.369(5)
C(10)-H(10)	0.9500
C(11)-C(12)	1.376(5)
C(11)-H(11)	0.9500
C(12)-C(13)	1.387(5)
C(12)-H(12)	0.9500
C(13)-H(13)	0.9500
C(14)-C(19)	1.374(4)
C(14)-C(15)	1.384(4)
C(14)-N(1)	1.425(4)
C(15)-C(16)	1.377(4)
C(15)-H(15)	0.9500
C(16)-C(17)	1.383(4)
C(16)-H(16)	0.9500

C(17)-O(3)	1.371(4)
C(17)-C(18)	1.376(4)
C(18)-C(19)	1.381(4)
C(18)-H(18)	0.9500
C(19)-H(19)	0.9500
C(20)-O(3)	1.406(4)
C(20)-H(20A)	0.9800
C(20)-H(20B)	0.9800
C(20)-H(20C)	0.9800
C(21)-N(6)	1.323(4)
C(21)-C(22)	1.429(4)
C(21)-C(28)	1.467(4)
C(22)-C(23)	1.387(4)
C(22)-C(25)	1.390(4)
C(23)-N(7)	1.330(4)
C(23)-H(23)	0.9500
C(24)-N(8)	1.326(4)
C(24)-N(7)	1.352(4)
C(24)-C(26')	1.46(2)
C(24)-C(26)	1.54(3)
C(25)-N(8)	1.348(4)
C(25)-N(5)	1.354(3)
C(28)-C(33)	1.384(4)
C(28)-C(29)	1.385(4)
C(29)-C(30)	1.385(5)
C(29)-H(29)	0.9500
C(30)-C(31)	1.371(5)
C(30)-H(30)	0.9500
C(31)-C(32)	1.376(6)
C(31)-H(31)	0.9500
C(32)-C(33)	1.380(5)
C(32)-H(32)	0.9500
C(33)-H(33)	0.9500
C(34)-C(35)	1.382(4)
C(34)-C(39)	1.385(4)
C(34)-N(5)	1.424(4)
C(35)-C(36)	1.379(4)
C(35)-H(35)	0.9500

C(36)-C(37)	1.388(4)
C(36)-H(36)	0.9500
C(37)-O(6)	1.372(3)
C(37)-C(38)	1.373(4)
C(38)-C(39)	1.376(4)
C(38)-H(38)	0.9500
C(39)-H(39)	0.9500
C(40)-O(6)	1.413(4)
C(40)-H(40A)	0.9800
C(40)-H(40B)	0.9800
C(40)-H(40C)	0.9800
N(1)-N(2)	1.376(3)
N(5)-N(6)	1.381(3)
O(4)-C(27)	1.353(10)
O(4)-C(26)	1.394(15)
O(5)-C(27)	1.117(12)
C(26)-H(26A)	0.9900
C(26)-H(26B)	0.9900
C(27)-H(27)	0.9500
O(4')-C(27')	1.310(8)
O(4')-C(26')	1.399(13)
O(5')-C(27')	1.102(10)
C(26')-H(26C)	0.9900
C(26')-H(26D)	0.9900
C(27')-H(27')	0.9500

N(2)-C(1)-C(2)	109.9(3)
N(2)-C(1)-C(8)	120.2(3)
C(2)-C(1)-C(8)	130.0(3)
C(3)-C(2)-C(5)	115.2(3)
C(3)-C(2)-C(1)	139.4(3)
C(5)-C(2)-C(1)	105.4(3)
N(3)-C(3)-C(2)	121.1(3)
N(3)-C(3)-H(3)	119.4
C(2)-C(3)-H(3)	119.4
N(4)-C(4)-N(3)	127.5(3)
N(4)-C(4)-C(6)	120.9(3)
N(3)-C(4)-C(6)	111.5(3)

N(4)-C(5)-N(1)	127.2(3)
N(4)-C(5)-C(2)	125.7(3)
N(1)-C(5)-C(2)	107.1(3)
O(1)-C(6)-C(4)	113.4(3)
O(1)-C(6)-H(6A)	108.9
C(4)-C(6)-H(6A)	108.9
O(1)-C(6)-H(6B)	108.9
C(4)-C(6)-H(6B)	108.9
H(6A)-C(6)-H(6B)	107.7
O(2)-C(7)-O(1)	125.8(5)
O(2)-C(7)-H(7)	117.1
O(1)-C(7)-H(7)	117.1
C(9)-C(8)-C(13)	118.4(3)
C(9)-C(8)-C(1)	120.7(3)
C(13)-C(8)-C(1)	120.9(3)
C(10)-C(9)-C(8)	121.0(3)
C(10)-C(9)-H(9)	119.5
C(8)-C(9)-H(9)	119.5
C(11)-C(10)-C(9)	120.5(3)
C(11)-C(10)-H(10)	119.8
C(9)-C(10)-H(10)	119.8
C(10)-C(11)-C(12)	119.5(3)
C(10)-C(11)-H(11)	120.3
C(12)-C(11)-H(11)	120.3
C(11)-C(12)-C(13)	120.6(4)
C(11)-C(12)-H(12)	119.7
C(13)-C(12)-H(12)	119.7
C(12)-C(13)-C(8)	120.0(3)
C(12)-C(13)-H(13)	120.0
C(8)-C(13)-H(13)	120.0
C(19)-C(14)-C(15)	119.7(3)
C(19)-C(14)-N(1)	119.5(3)
C(15)-C(14)-N(1)	120.8(3)
C(16)-C(15)-C(14)	119.6(3)
C(16)-C(15)-H(15)	120.2
C(14)-C(15)-H(15)	120.2
C(15)-C(16)-C(17)	120.4(3)
C(15)-C(16)-H(16)	119.8

C(17)-C(16)-H(16)	119.8
O(3)-C(17)-C(18)	124.9(3)
O(3)-C(17)-C(16)	115.0(3)
C(18)-C(17)-C(16)	120.1(3)
C(17)-C(18)-C(19)	119.3(3)
C(17)-C(18)-H(18)	120.4
C(19)-C(18)-H(18)	120.4
C(14)-C(19)-C(18)	120.9(3)
C(14)-C(19)-H(19)	119.6
C(18)-C(19)-H(19)	119.6
O(3)-C(20)-H(20A)	109.5
O(3)-C(20)-H(20B)	109.5
H(20A)-C(20)-H(20B)	109.5
O(3)-C(20)-H(20C)	109.5
H(20A)-C(20)-H(20C)	109.5
H(20B)-C(20)-H(20C)	109.5
N(6)-C(21)-C(22)	109.6(3)
N(6)-C(21)-C(28)	120.2(3)
C(22)-C(21)-C(28)	130.2(3)
C(23)-C(22)-C(25)	116.1(3)
C(23)-C(22)-C(21)	138.5(3)
C(25)-C(22)-C(21)	105.3(3)
N(7)-C(23)-C(22)	120.7(3)
N(7)-C(23)-H(23)	119.6
C(22)-C(23)-H(23)	119.6
N(8)-C(24)-N(7)	128.1(3)
N(8)-C(24)-C(26')	117.9(7)
N(7)-C(24)-C(26')	114.0(7)
N(8)-C(24)-C(26)	120.0(8)
N(7)-C(24)-C(26)	111.8(8)
C(26')-C(24)-C(26)	6(2)
N(8)-C(25)-N(5)	127.0(3)
N(8)-C(25)-C(22)	125.4(3)
N(5)-C(25)-C(22)	107.6(2)
C(33)-C(28)-C(29)	119.2(3)
C(33)-C(28)-C(21)	120.2(3)
C(29)-C(28)-C(21)	120.6(3)
C(28)-C(29)-C(30)	120.2(3)

C(28)-C(29)-H(29)	119.9
C(30)-C(29)-H(29)	119.9
C(31)-C(30)-C(29)	119.9(4)
C(31)-C(30)-H(30)	120.0
C(29)-C(30)-H(30)	120.0
C(30)-C(31)-C(32)	120.4(3)
C(30)-C(31)-H(31)	119.8
C(32)-C(31)-H(31)	119.8
C(31)-C(32)-C(33)	119.9(4)
C(31)-C(32)-H(32)	120.0
C(33)-C(32)-H(32)	120.0
C(32)-C(33)-C(28)	120.4(4)
C(32)-C(33)-H(33)	119.8
C(28)-C(33)-H(33)	119.8
C(35)-C(34)-C(39)	120.2(3)
C(35)-C(34)-N(5)	121.2(3)
C(39)-C(34)-N(5)	118.5(2)
C(36)-C(35)-C(34)	119.4(3)
C(36)-C(35)-H(35)	120.3
C(34)-C(35)-H(35)	120.3
C(35)-C(36)-C(37)	120.2(3)
C(35)-C(36)-H(36)	119.9
C(37)-C(36)-H(36)	119.9
O(6)-C(37)-C(38)	125.3(3)
O(6)-C(37)-C(36)	114.7(3)
C(38)-C(37)-C(36)	120.1(3)
C(37)-C(38)-C(39)	120.0(3)
C(37)-C(38)-H(38)	120.0
C(39)-C(38)-H(38)	120.0
C(38)-C(39)-C(34)	120.0(3)
C(38)-C(39)-H(39)	120.0
C(34)-C(39)-H(39)	120.0
O(6)-C(40)-H(40A)	109.5
O(6)-C(40)-H(40B)	109.5
H(40A)-C(40)-H(40B)	109.5
O(6)-C(40)-H(40C)	109.5
H(40A)-C(40)-H(40C)	109.5
H(40B)-C(40)-H(40C)	109.5

C(5)-N(1)-N(2)	110.2(2)
C(5)-N(1)-C(14)	130.8(2)
N(2)-N(1)-C(14)	119.0(2)
C(1)-N(2)-N(1)	107.5(2)
C(3)-N(3)-C(4)	117.8(3)
C(4)-N(4)-C(5)	112.6(3)
C(25)-N(5)-N(6)	110.1(2)
C(25)-N(5)-C(34)	131.2(2)
N(6)-N(5)-C(34)	118.6(2)
C(21)-N(6)-N(5)	107.3(2)
C(23)-N(7)-C(24)	117.4(3)
C(24)-N(8)-C(25)	112.4(3)
C(7)-O(1)-C(6)	113.7(3)
C(17)-O(3)-C(20)	118.4(3)
C(37)-O(6)-C(40)	117.9(2)
C(27)-O(4)-C(26)	115.2(13)
O(4)-C(26)-C(24)	109.4(18)
O(4)-C(26)-H(26A)	109.8
C(24)-C(26)-H(26A)	109.8
O(4)-C(26)-H(26B)	109.8
C(24)-C(26)-H(26B)	109.8
H(26A)-C(26)-H(26B)	108.2
O(5)-C(27)-O(4)	123.9(13)
O(5)-C(27)-H(27)	118.0
O(4)-C(27)-H(27)	118.0
C(27')-O(4')-C(26')	117.7(11)
O(4')-C(26')-C(24)	111.2(15)
O(4')-C(26')-H(26C)	109.4
C(24)-C(26')-H(26C)	109.4
O(4')-C(26')-H(26D)	109.4
C(24)-C(26')-H(26D)	109.4
H(26C)-C(26')-H(26D)	108.0
O(5')-C(27')-O(4')	126.3(11)
O(5')-C(27')-H(27')	116.8
O(4')-C(27')-H(27')	116.8

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 130527. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	48(2)	65(2)	45(2)	-5(1)	12(1)	-3(1)
C(2)	49(2)	69(2)	44(2)	-9(1)	10(1)	-4(2)
C(3)	58(2)	99(3)	51(2)	-7(2)	16(2)	-6(2)
C(4)	62(2)	87(3)	42(2)	-3(2)	8(2)	-10(2)
C(5)	52(2)	61(2)	44(2)	-4(1)	8(1)	-2(1)
C(6)	67(3)	155(4)	46(2)	-5(2)	5(2)	-17(3)
C(7)	72(3)	191(6)	62(3)	-5(3)	3(2)	-22(3)
C(8)	48(2)	75(2)	48(2)	-4(2)	10(1)	-3(2)
C(9)	54(2)	77(2)	57(2)	-8(2)	5(2)	3(2)
C(10)	60(2)	92(3)	62(2)	-10(2)	0(2)	-7(2)
C(11)	50(2)	109(3)	74(3)	1(2)	2(2)	-5(2)
C(12)	53(2)	130(4)	71(3)	-7(2)	18(2)	8(2)
C(13)	52(2)	113(3)	53(2)	-14(2)	10(2)	2(2)
C(14)	43(2)	67(2)	40(2)	-1(1)	7(1)	1(1)
C(15)	48(2)	94(3)	44(2)	-14(2)	2(2)	-4(2)
C(16)	41(2)	93(3)	54(2)	-11(2)	2(2)	-8(2)
C(17)	44(2)	77(2)	46(2)	-1(2)	6(1)	5(2)
C(18)	49(2)	74(2)	45(2)	-11(2)	7(1)	-3(2)
C(19)	47(2)	68(2)	45(2)	-8(1)	5(1)	-7(2)
C(20)	70(3)	126(4)	67(2)	-20(2)	26(2)	3(2)
C(21)	48(2)	60(2)	46(2)	-4(1)	6(1)	2(1)
C(22)	48(2)	60(2)	45(2)	-3(1)	6(1)	0(1)
C(23)	52(2)	81(2)	51(2)	-4(2)	13(2)	2(2)
C(24)	54(2)	73(2)	41(2)	-1(1)	8(2)	-3(2)
C(25)	48(2)	58(2)	40(2)	-2(1)	4(1)	0(1)
C(28)	44(2)	71(2)	50(2)	-8(2)	5(1)	2(2)
C(29)	52(2)	78(2)	55(2)	-4(2)	2(2)	2(2)
C(30)	60(2)	85(3)	69(2)	-6(2)	-6(2)	5(2)
C(31)	49(2)	105(3)	82(3)	-24(2)	-4(2)	7(2)
C(32)	51(2)	117(4)	86(3)	-18(2)	22(2)	-10(2)
C(33)	51(2)	94(3)	62(2)	-4(2)	9(2)	-8(2)
C(34)	40(2)	61(2)	37(2)	-4(1)	4(1)	0(1)
C(35)	48(2)	71(2)	39(2)	1(1)	3(1)	4(2)

C(36)	44(2)	71(2)	41(2)	-2(1)	0(1)	4(1)
C(37)	45(2)	65(2)	41(2)	-7(1)	8(1)	-4(1)
C(38)	53(2)	70(2)	39(2)	6(1)	5(1)	3(2)
C(39)	48(2)	63(2)	44(2)	3(1)	3(1)	5(1)
C(40)	73(3)	123(4)	54(2)	11(2)	24(2)	6(2)
N(1)	42(1)	74(2)	38(1)	-4(1)	6(1)	-3(1)
N(2)	47(2)	78(2)	44(1)	-6(1)	7(1)	0(1)
N(3)	62(2)	122(3)	43(2)	-9(2)	10(1)	-7(2)
N(4)	56(2)	76(2)	39(1)	-4(1)	4(1)	-7(1)
N(5)	43(1)	70(2)	37(1)	-3(1)	2(1)	4(1)
N(6)	44(1)	71(2)	43(1)	-3(1)	1(1)	2(1)
N(7)	61(2)	87(2)	42(2)	-5(1)	9(1)	-1(2)
N(8)	51(2)	72(2)	38(1)	0(1)	3(1)	-1(1)
O(1)	65(2)	127(2)	49(1)	-5(1)	-1(1)	4(2)
O(2)	113(3)	216(4)	82(2)	-47(3)	10(2)	-50(3)
O(3)	47(1)	117(2)	64(2)	-18(1)	17(1)	-4(1)
O(6)	52(1)	96(2)	50(1)	3(1)	14(1)	5(1)
O(4)	63(3)	137(5)	45(3)	-2(4)	4(2)	3(4)
O(5)	86(5)	102(8)	70(4)	17(4)	-18(4)	-9(6)
C(26)	64(4)	110(6)	46(5)	7(5)	9(4)	-2(4)
C(27)	71(4)	125(6)	54(4)	-1(5)	-6(4)	-6(5)
O(4')	83(3)	99(4)	71(3)	9(3)	-19(3)	-23(3)
O(5')	118(6)	162(10)	75(5)	14(5)	-32(4)	-41(8)
C(26')	64(4)	100(5)	39(4)	-4(4)	-1(3)	-18(4)
C(27')	79(4)	126(6)	61(4)	3(5)	-14(3)	-24(4)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for 130527.

	x	y	z	U(eq)
H(3)	9756	2789	-91	82
H(6A)	6923	3853	-830	108
H(6B)	6918	1645	-788	108
H(7)	4593	2264	-857	131
H(9)	10384	4487	1398	75
H(10)	12003	4423	1631	87
H(11)	13051	3076	1257	94
H(12)	12465	1737	648	101
H(13)	10835	1785	408	87
H(15)	6144	2247	545	75
H(16)	4860	2407	894	76
H(18)	6500	5088	1789	67
H(19)	7784	4918	1440	64
H(20A)	5276	3943	2119	129
H(20B)	4178	4521	2011	129
H(20C)	4993	5888	1907	129
H(23)	10493	8113	874	73
H(29)	10954	7056	2367	75
H(30)	12578	6859	2617	88
H(31)	13697	7927	2243	96
H(32)	13209	9205	1619	100
H(33)	11591	9451	1372	83
H(35)	6864	10031	1455	64
H(36)	5554	10136	1791	63
H(38)	7086	7583	2732	65
H(39)	8392	7448	2397	62
H(40A)	5559	7050	2859	123
H(40B)	4789	8575	2936	123
H(40C)	5904	9035	3036	123
H(26A)	7645	9220	90	87
H(26B)	7633	7010	143	87
H(27)	5275	8400	83	102

H(26C)	7332	9696	242	83
H(26D)	7855	7971	73	83
H(27')	5496	6621	85	110

Notes and references

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