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Organocatalytic One-Pot Asymmetric Synthesis of Functionalized Spiropyrazolones *via* a Michael-Aldol Sequential Reaction

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Experimental section

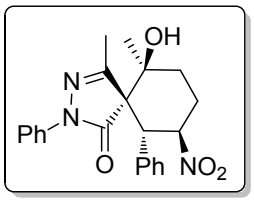
General remarks

All reagents were used as purchased from commercial suppliers without further purification. IR spectra were recorded on a Perkin-Elmer 500 spectrometer. NMR spectra were recorded on a Bruker Avance 400 NMR spectrometer (400 MHz for ^{1}H and 100 MHz for ^{13}C). Chemical shifts are reported in δ parts per million referenced to an internal TMS standard for ^{1}H NMR and chloroform-d (δ 77.2 ppm) for ^{13}C NMR. Optical rotations were measured on a JASCO P-2000 polarimeter HRMS spectra were recorded on Waters Xevo G2-S Tof. The X-ray diffraction measurements were carried out at 296 K on a KAPPA APEX II CCD area detector system equipped with a graphite monochromator and a Mo-Ka fine-focus sealed tube (k = 0.71073 A^{o}). Routine monitoring of reactions was performed using silica gel, glass-backed TLC plates (Merck Kieselgel $60F_{254}$) and visualized by UV light (254 nm). Solutions were evaporated to dryness under reduced pressure on a rotary evaporator and the residues purified by flash column chromatography on silica gel (230-400 mesh) with the indicated eluents.

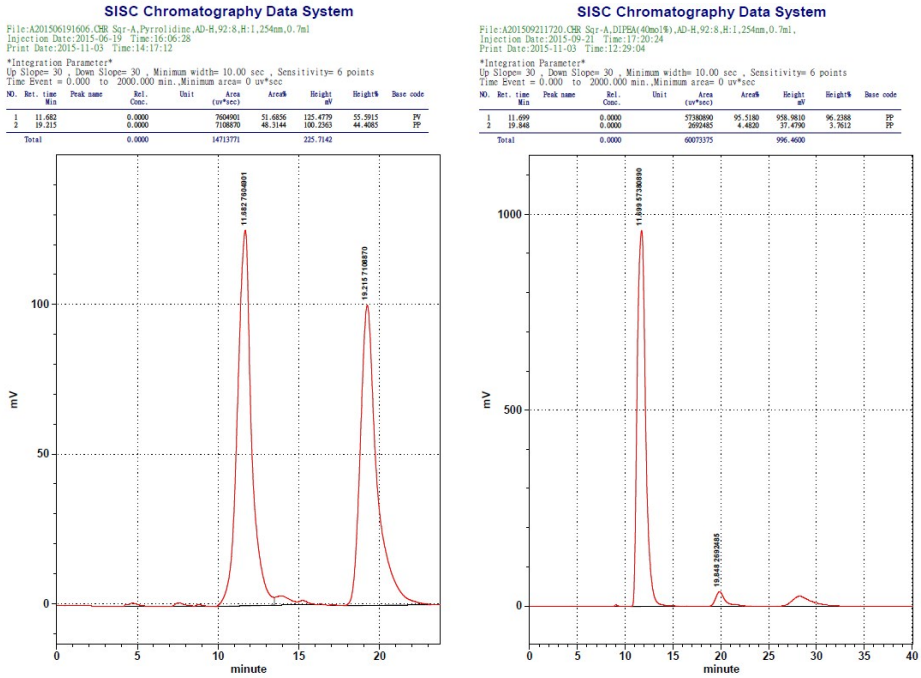
General procedure: To a stirred solution of 3-methyl-1-phenyl-2-pyrazolin-5-one (**1**) (0.2 mmol) and catalyst (2 mol %) in CH_2Cl_2 (0.6 mL) solvent was added (E)-5-nitro-6-aryl-hex-5-en-2-one (**2**) (0.2 mmol). The reaction mixture was stirred at room temperature (25-30 ^{o}C) until the completion of 3-methyl-1-phenyl-2-pyrazolin-5-one and monitored by TLC. After completion of pyrazolinone, DIPEA were added subsequently and further stirred for indicated time at room temperature and CH_2Cl_2 was evaporated off. After removal of the solvent, a crude residue was purified by flash column chromatography (hexanes/ethyl acetate = 4:1~3:1) to afford the pure spirocyclohexane pyrazolone derivatives (**3a**-**3p**).

The diastereomeric ratios were determined from the crude reaction mixture measured in ^{1}H NMR spectra. Some compounds were isolated as mixture of diastereomers after flash column chromatography. The ^{1}H and ^{13}C NMR data of the major isomers were given.

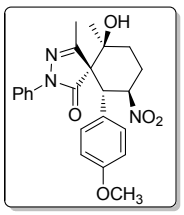
(5*R*,6*S*,9*R*,10*R*)-6-hydroxy-4,6-dimethyl-9-nitro-2,10-diphenyl-2,3-diazaspiro[4.5]dec-3-en-1-one (3a):



White solid, Yield: 61%, mp 140-142 °C; [α]25.6 D= -155.6 (c = 0.5 in CH_2Cl_2); IR (KBr): ν 3568, 2918, 2850. 2378, 1773, 1694, 911, 759, 691 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.58 (d, 2H, *J* = 7.8 Hz), 7.35(t, 2H, *J* = 7.8 Hz), 7.20-7.14 (m, 6H), 5.84 (td, 1H, *J* = 11.7, 4.3 Hz), 4.12 (d, 1H, *J* = 11.7 Hz), 2.85 (td, 1H, *J* = 14.1, 4.3 Hz), 2.59-2.48 (m, 1H), 2.38-2.35 (m, 1H), 2.24 (s, 3H), 1.73 (s, 1H), 1.65 (m, 1H), 1.16 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.6, 161.0, 137.2, 134.0, 129.0, 128.9, 128.8, 126.0, 120.0, 84.5, 72.1, 66.4, 46.1, 33.0, 27.1, 26.3, 17.7. HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{22}H_{24}N_3O_4$ 394.1767; found 394.1765. The ee value of **3a** was 92% determined by HPLC with chiralpak AD-H column (i-PrOH/hexanes: 8/92; flow rate: 0.7 mL/min; λ 254 nm); t_R (major) 11.69 min; t_R (minor) 19.84 min.

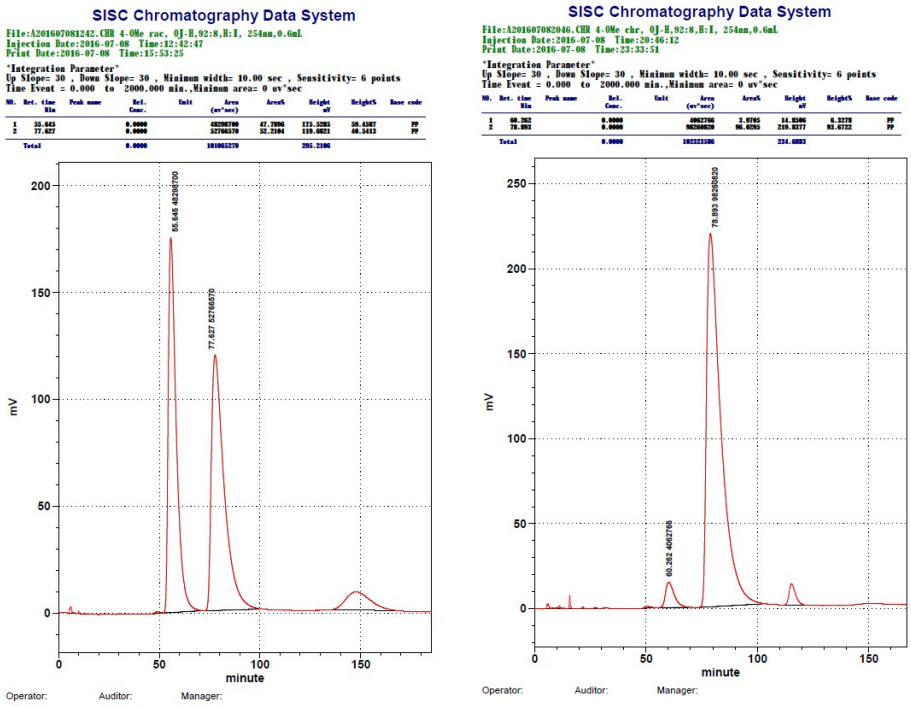


(5*R*,6*S*,9*R*,10*R*)-6-hydroxy-10-(4-methoxyphenyl)-4,6-dimethyl-9-nitro-2-phenyl-2,3-diazaspiro[4.5]dec-3-en-1-one (3b):

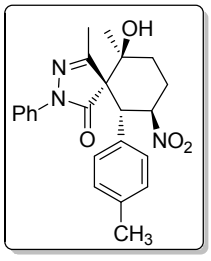


White solid, Yield: 53%, mp 74-76 °C; [α]25.6 D= -106.9 (*c* = 0.5 in CH_2Cl_2); IR (KBr): ν 3543, 2916, 2849, 2352, 1715, 1695, 1371, 1032, 757 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.63-7.61 (m, 2H), 7.36 (t, 2H, *J* = 7.7 Hz), 7.19 (t, 1H, *J* = 7.7 Hz), 7.11 (d, 2H, *J* = 8.7 Hz), 6.67 (d, 2H, *J* = 8.7 Hz) 5.79 (td, 1H, *J* = 11.8, 4.4 Hz), 4.06 (d, 1H, *J* = 11.8 Hz), 3.67 (s, 3H), 2.83 (td, 1H, *J* =

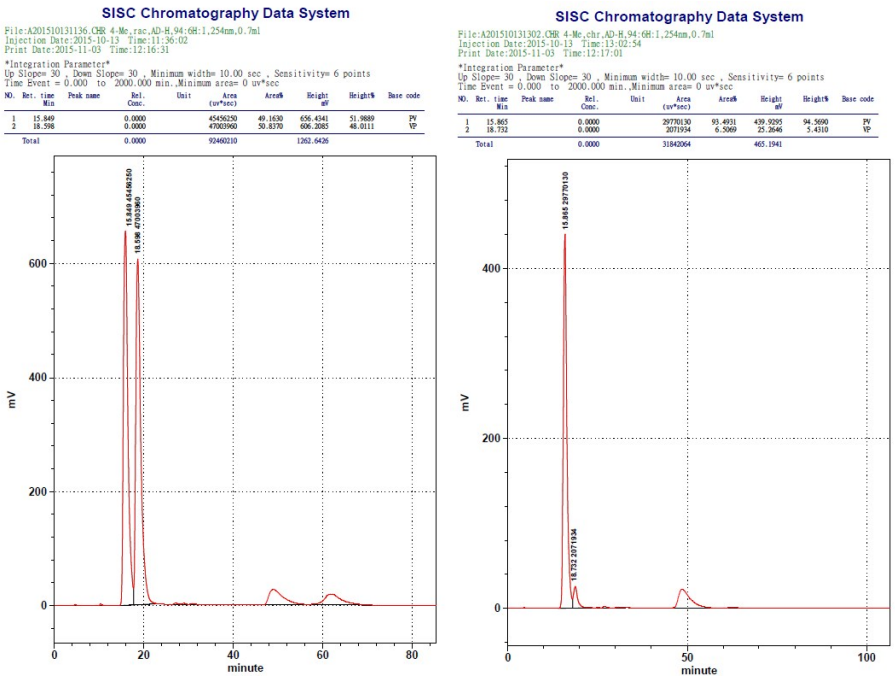
14.2, 4.4 Hz), 2.58-2.48 (m, 1H), 2.38-2.32 (m, 1H), 2.23 (s, 3H), 1.69 (s, 1H), 1.66-1.62 (m, 1H), 1.16 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.7, 161.1, 159.7, 137.3, 129.0, 125.9 (2C), 119.9, 114.3, 84.8, 72.2, 66.5, 55.3, 45.4, 33.0, 27.1, 26.3, 17.6. HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{23}H_{26}N_3O_5$ 424.1872; found 424.1874. The ee value of **3b** was 92% determined by HPLC with chiralpak OJ-H column (i-PrOH/hexanes: 8/92; flow rate: 0.6 mL/min; λ 254 nm); t_R (minor) 60.26 min; t_R (major) 78.89 min.



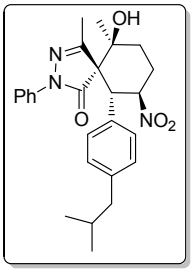
(5*R*,6*S*,9*R*,10*R*)-6-hydroxy-4,6-dimethyl-9-nitro-2-phenyl-10-(p-tolyl)-2,3-diazaspiro[4.5]dec-3-en-1-one (3c):



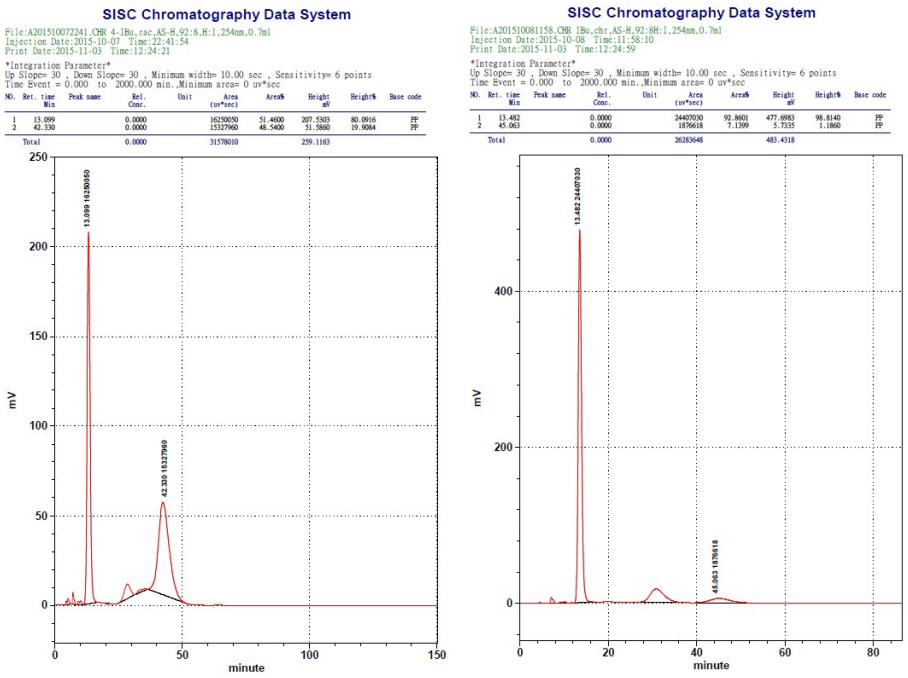
White solid, Yield: 55%, mp 88-90 °C; [α]25.6 D= -129.8 (*c* = 0.5 in CH_2Cl_2); IR (KBr): ν 3588, 2916, 2849, 1698, 1654, 1551, 1293 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.63-7.61 (m, 2H), 7.38-7.34 (m, 2H), 7.21-7.17 (m, 1H), 7.07 (d, 2H, *J* = 8.2 Hz), 6.96-6.93 (m, 2H), 5.82 (td, 1H, *J* = 11.8, 4.4 Hz), 4.07 (d, 1H, *J* = 11.8 Hz), 2.85 (td, 1H, *J* = 14.2, 4.4 Hz), 2.58-2.48 (m, 1H), 2.39-2.33 (m, 1H), 2.24 (s, 3H), 2.19 (s, 3H), 1.66-1.64 (m, 2H), 1.16 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.7, 161.1, 138.5, 137.3, 130.9, 129.6, 129.0, 125.9, 120.0, 84.7, 72.2, 66.4, 45.8, 33.0, 27.1, 26.3, 21.1, 17.7. HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{23}H_{26}N_3O_4$ 408.1923; found 408.1923. The ee value of **3c** was 86% determined by HPLC with chiralpak AD-H column (i-PrOH/hexanes: 6/94; flow rate: 0.7 mL/min; λ 254 nm); t_R (minor) 56.34 min; t_R (major) 75.17 min.



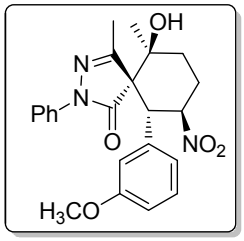
(5*R*,6*S*,9*R*,10*R*)-6-hydroxy-10-(4-isobutylphenyl)-4,6-dimethyl-9-nitro-2-phenyl-2,3-diazaspiro[4.5]dec-3-en-1-one (3d):



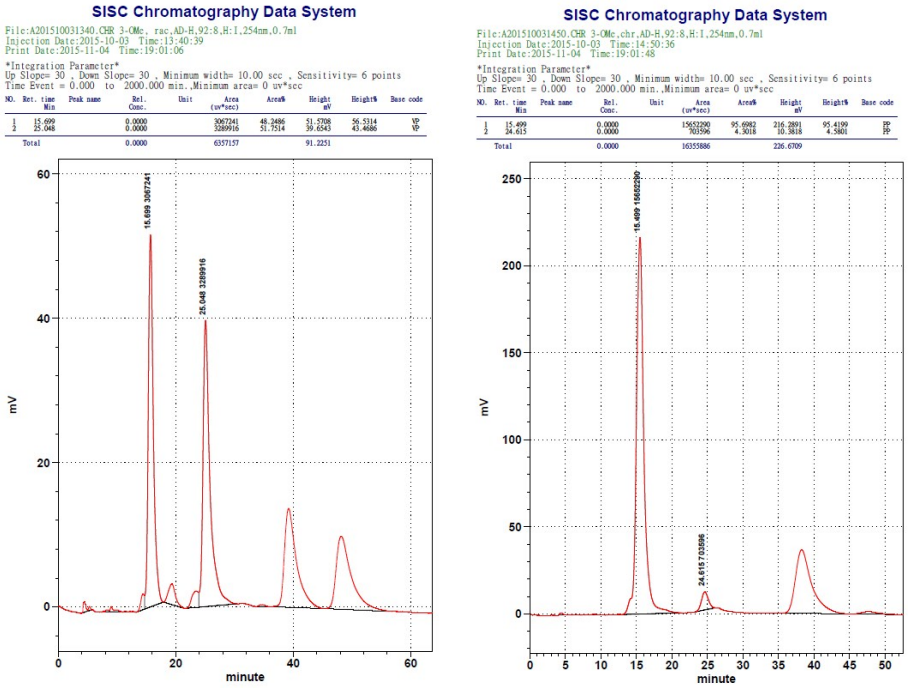
White solid, Yield: 50%, mp 76-78 ^{o}C; [α]25.6 D= -134.0 (*c* = 0.5 in CH_2Cl_2); IR (KBr): ν 3585, 2955, 2924, 2356, 1715, 1551, 1368, 1249, 865, 757, 690 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.55-7.53 (m, 2H), 7.36-7.31 (m, 2H), 7.20-7.15 (m, 1H), 7.09-7.07 (m, 1H), 6.91- 6.88 (m, 2H), 5.82 (td, 1H, *J* = 11.8, 4.4 Hz), 4.08 (d, 1H, *J* = 11.8 Hz), 2.85 (td, 1H, *J* = 14.2, 4.4 Hz), 2.59-2.48 (m, 1H), 2.39-2.34 (m, 1H), 2.32-2.29 (m, 2H), 2.24 (s, 3H), 1.74-1.67 (m, 2H), 1.66-1.61 (m, 2H), 1.16 (s, 3H), 0.77 (q, 6H, *J* = 6.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.7, 161.0, 142.2, , 137.3, 131.1, 129.5, 128.9, 125.9, 120.1, 84.5, 72.1, 66.5, 45.8, 45.0, 33.0, 30.1, 27.1, 26.3, 22.5, 22.4, 17.7. HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{26}H_{32}N3O_4$ 450.2393; found 450.2394. The ee value of **3d** was 86% determined by HPLC with chiralpak AS-H column (i-PrOH/hexanes: 8/92; flow rate: 0.7 mL/min; λ 254 nm); t_R (major) 13.48 min; t_R (minor) 45.06 min.



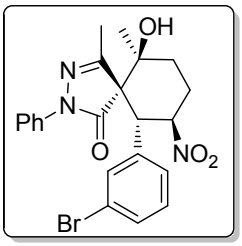
(5*R*,6*S*,9*R*,10*R*)-6-hydroxy-10-(3-methoxyphenyl)-4,6-dimethyl-9-nitro-2-phenyl-2,3-diazaspiro[4.5]dec-3-en-1-one (3e):



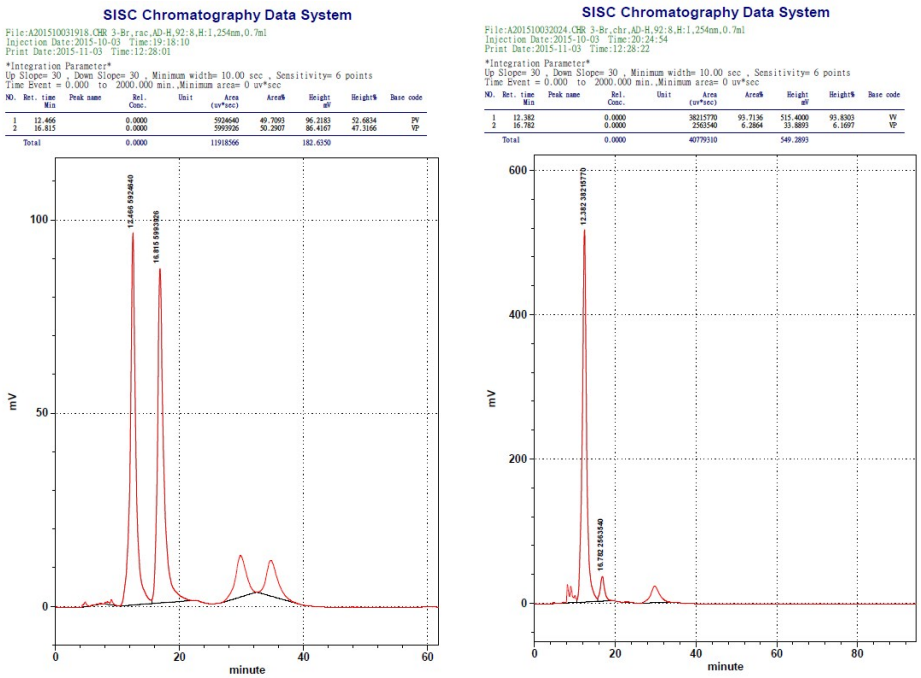
White solid, Yield: 58%, mp 138-140 °C; [α]25.6 D= -219.0 (*c* = 0.5 in CH_2Cl_2); IR (KBr): ν 3655, 3593, 2917, 2850, 2358, 1715, 1651, 1495 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.62 (d, 2H, *J* = 7.7 Hz), 7.35 (t, 2H, *J* = 7.9 Hz), 7.18 (t, 1H, *J* = 7.8 Hz), 7.06 (t, 1H, *J* = 7.8 Hz), 6.78 (d, 1H, *J* = 7.8 Hz), 6.78 (d, 1H, *J* = 7.8 Hz), 6.71- 6.68 (m, 2H), 5.83 (td, 1H, *J* = 11.8, 4.5 Hz), 4.09 (d, 1H, *J* = 11.8 Hz), 3.54 (s, 3H), 2.87 (td, 1H, *J* = 14.1, 4.4 Hz), 2.59-2.49 (m, 1H), 2.41-2.35 (m, 1H), 2.25 (s, 3H), 1.68-1.62 (m, 1H), 1.59 (s, 1H), 1.19 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.7, 161.0, 159.8, 137.3, 135.5, 129.9, 129.0, 125.9, 119.8, 114.7, 84.5, 72.2, 66.3, 55.1, 46.2, 33.0, 27.1, 26.3, 17.7. HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{23}H_{26}N_3O_5$ 424.1872; found 424.1874. The ee value of **3e** was 92% determined by HPLC with chiralpak AD-H column (i-PrOH/hexanes: 8/92; flow rate: 0.7 mL/min; λ 254 nm); t_R (major) 15.49 min; t_R (minor) 24.61 min.



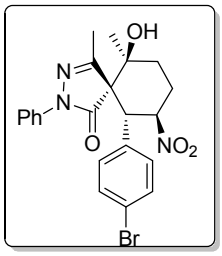
(5*R*,6*S*,9*R*,10*R*)-10-(3-bromophenyl)-6-hydroxy-4,6-dimethyl-9-nitro-2-phenyl-2,3-diazaspiro[4.5]dec-3-en-1-one (3f):



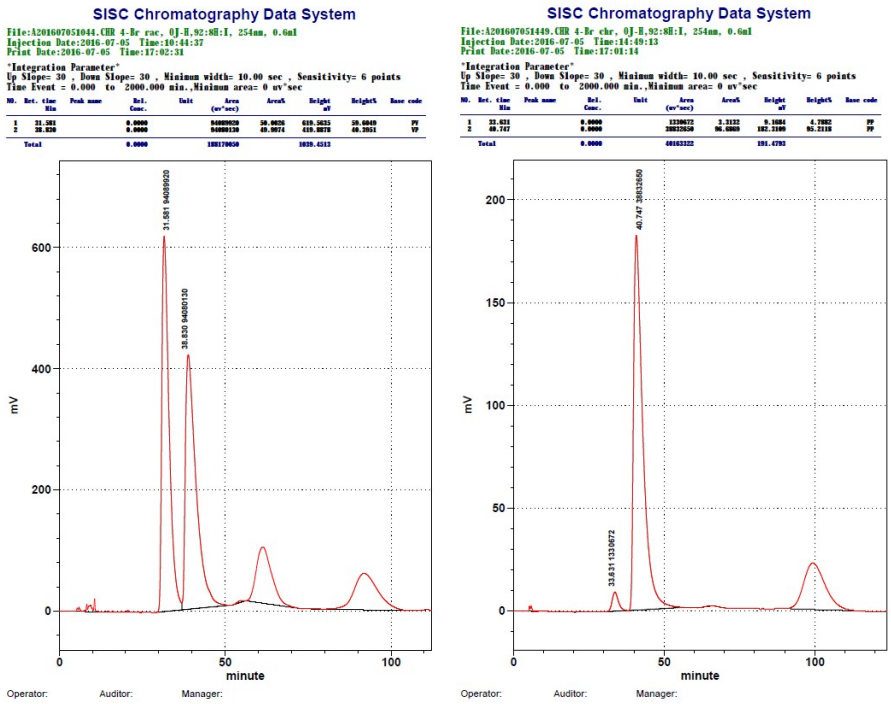
White solid, Yield: 37%, mp 108-110 ^{o}C; [α]25.6 D= -109.7 (*c* = 0.5 in CH_2Cl_2); IR (KBr): ν 3543, 2919, 2850, 2357, 1715, 1557, 1496, 758 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.64-7.61 (m, 2H), 7.41-7.34 (m, 3H), 7.34-7.31 (m, 1H), 7.25-7.21 (m, 1H), 7.18-7.16 (m, 1H), 7.04 (t, 1H, *J* = 7.8 Hz), 5.82 (td, 1H, *J* = 11.8, 4.5 Hz), 4.12 (d, 1H, *J* = 11.8 Hz), 2.88 (td, 1H, *J* = 14.1, 4.4 Hz), 2.61-2.50 (m, 1H), 2.44-2.38 (m, 1H), 2.27 (s, 3H), 1.70-1.65 (m, 2H), 1.21 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.3, 160.6, 136.5, 132.0, 130.5, 129.0, 126.2, 120.2, 84.2, 72.2, 66.2, 45.7, 33.0, 27.1, 26.3, 17.7. HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{22}H_{23}N_3O_4Br$ 472.0872; found 472.0874. The ee value of **3f** was 88% determined by HPLC with chiralpak AD-H column (i-PrOH/hexanes: 8/92; flow rate: 0.7 mL/min; λ 254 nm); t_R (major) 12.38 min; t_R (minor) 16.78 min.



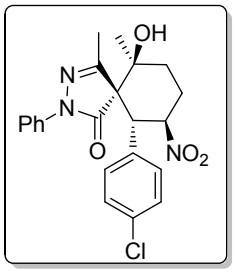
(5*R*,6*S*,9*R*,10*R*)-10-(4-bromophenyl)-6-hydroxy-4,6-dimethyl-9-nitro-2-phenyl-2,3-diazaspiro[4.5]dec-3-en-1-one (3g):



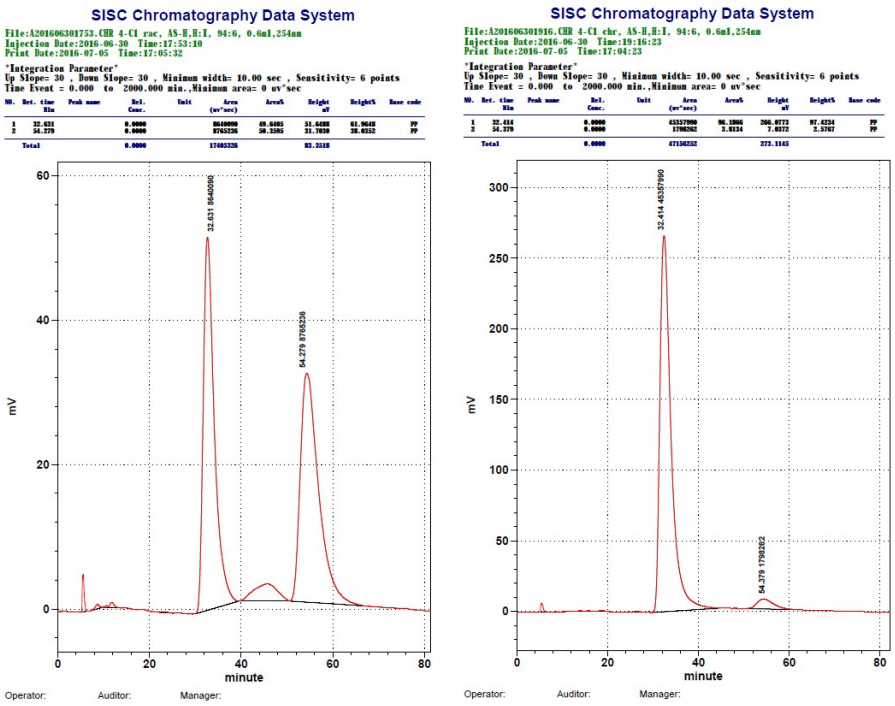
White solid, Yield:42%, mp 105-107 ^{o}C; [α]25.6 D= -130.0 (*c* = 0.5 in CH_2Cl_2); IR (KBr): ν 3566, 2920, 2341, 2359, 1715, 1496, 1076, 756, 690 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.63-7.60 (m, 2H), 7.44-7.35 (m, 3H), 7.30-7.28 (m, 2H), 7.23-7.19 (m, 1H), 7.08 (d, 2H, *J* = 8.5 Hz), 5.79 (td, 1H, *J* = 11.8, 4.4 Hz), 4.10 (d, 1H, *J* = 11.8 Hz), 2.85 (td, 1H, *J* = 14.2, 4.4 Hz), 2.59-2.48 (m, 1H), 2.41-2.35 (m, 1H), 2.23 (s, 3H), 1.68-1.63 (m, 1H), 1.59 (s, 1H), 1.18 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.4, 160.7, 137.2, 133.2, 132.2, 129.1, 126.1, 123.0, 119.8, 84.3, 72.2, 66.2, 45.5, 33.0, 27.0, 26.3, 17.7. HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{22}H_{23}N_3O_4Br$ 472.0872; found 472.0874. The ee value of **3g** was 94 % determined by HPLC with chiralpak OJ-H column (i-PrOH/hexanes: 8/92; flow rate: 0.6 mL/min; λ 254 nm); t_R (minor) 33.63 min; t_R (major) 40.74 min.



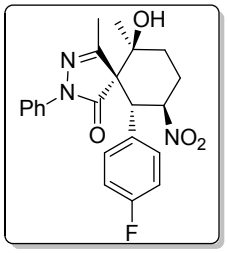
(5*R*,6*S*,9*R*,10*R*)-10-(4-chlorophenyl)-6-hydroxy-4,6-dimethyl-9-nitro-2-phenyl-2,3-diazaspiro[4.5]dec-3-en-1-one (3h):



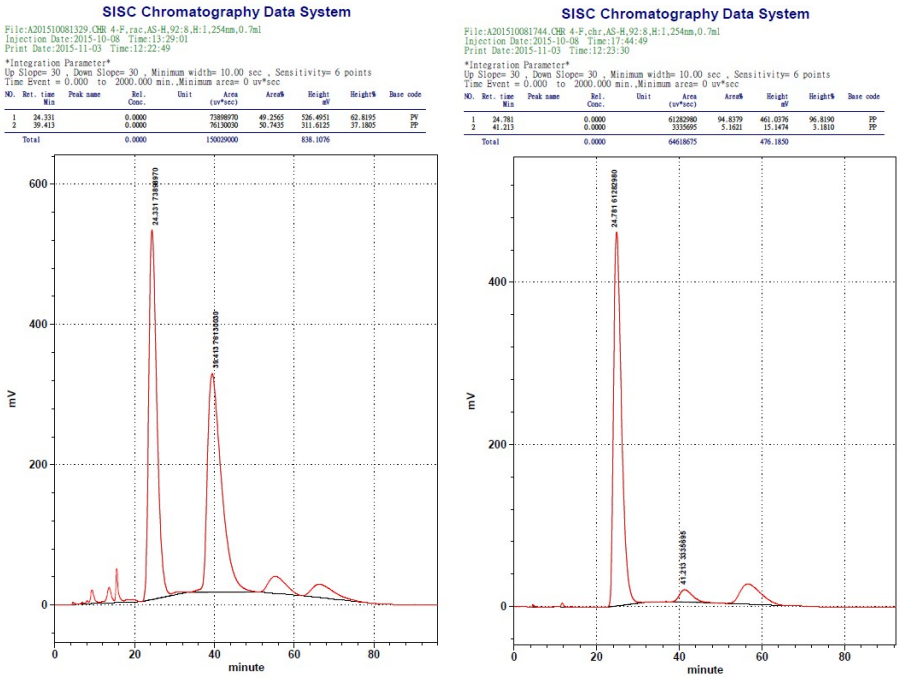
White solid, Yield: 54%, mp 100-102 °C; [α]25.6 D= -101.5 (*c* = 0.5 in CH_2Cl_2); IR (KBr): ν 3566, 2916, 2850, 2357, 1715, 1595, 1093, 863, 690 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.65-7.62 (m, 2H), 7.42-7.37 (m, 2H), 7.25-7.19 (m, 1H), 7.16-7.14 (m, 4H), 5.82 (td, 1H, *J* = 11.8, 4.4 Hz), 4.14 (d, 1H, *J* = 11.8 Hz), 2.86 (td, 1H, *J* = 14.2, 4.4 Hz), 2.61-2.50 (m, 1H), 2.43-2.37 (m, 1H), 2.26 (s, 3H), 1.75 (s, 1H), 1.70-1.65 (m, 1H), 1.19 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.4, 160.7, 137.1, 134.8, 132.7, 129.2, 129.1, 126.1, 119.8, 84.4, 72.2, 66.3, 45.5, 32.9, 27.0, 26.2, 17.7. HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{22}H_{23}N_3O_4Cl$ 428.1377; found 428.1381. The ee value of **3h** was 92% determined by HPLC with chiralpak AS-H column (i-PrOH/hexanes: 6/94; flow rate: 0.6 mL/min; λ 254 nm); t_R (major) 32.41 min; t_R (minor) 54.37 min.



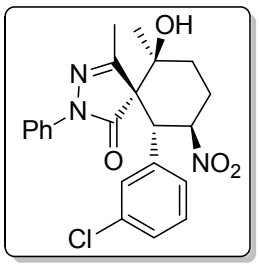
(5*R*,6*S*,9*R*,10*R*)-10-(4-fluorophenyl)-6-hydroxy-4,6-dimethyl-9-nitro-2-phenyl-2,3-diazaspiro[4.5]dec-3-en-1-one (**3i**)**:**



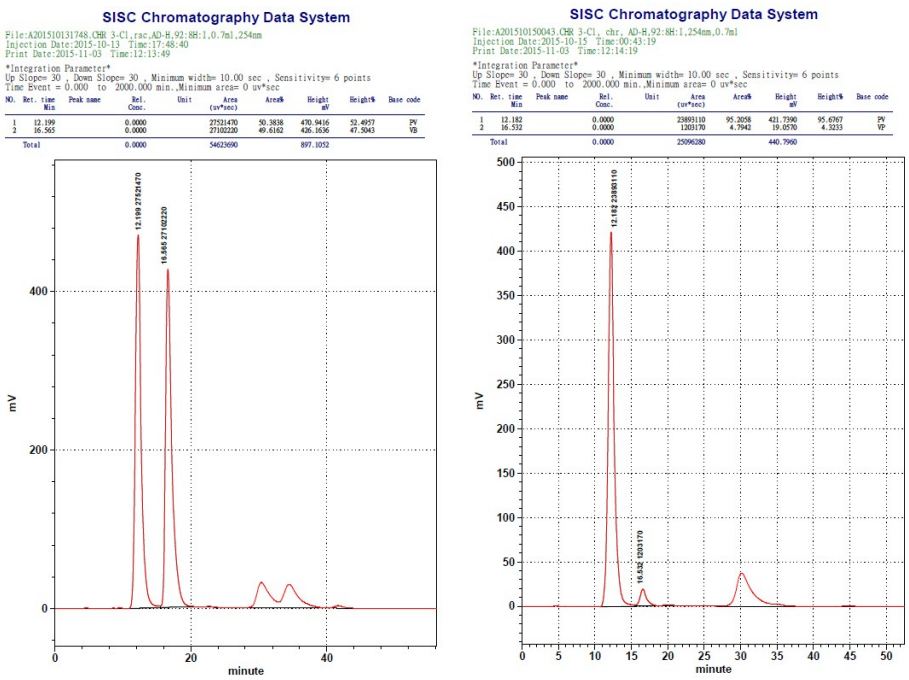
White solid, Yield: 80%, mp 81-83 ^{o}C; [α]25.6 D= -118.3 (*c* = 0.5 in CH_2Cl_2); IR (KBr): ν 3546, 2916, 2850, 2358, 1715, 1695, 1371, 1163, 1034, 915, 691 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.61-7.59 (m, 2H), 7.38-7.34 (m, 2H), 7.22-7.17 (m, 3H), 6.87-6.83 (m, 2H), 5.80 (td, 1H, *J* = 11.8, 4.4 Hz), 4.12 (d, 1H, *J* = 11.8 Hz), 2.84 (td, 1H, *J* = 14.1, 4.4 Hz), 2.59-2.48 (m, 1H), 2.40-2.35 (m, 1H), 2.24 (s, 3H), 1.69 (s, 1H), 1.67-1.62 (m, 1H), 1.17 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.5, 162.7 (d, $^{1}J_{C\text{-}F}$ = 248.0 Hz) 160.9, 137.1, 129.9 (d, $^{4}J_{C\text{-}F}$ = 3.26 Hz), 129.1, 126.1, 119.9, 116.0 (d, $^{2}J_{C\text{-}F}$ = 22.0 Hz), 115.9, 84.5, 72.1, 66.4, 45.3, 32.9, 27.0, 26.3, 17.7. HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{22}H_{23}N_3O_4F$ 412.1673; found 412.1672. The ee value of **3i** was 90% determined by HPLC with chiralpak AS-H column (i-PrOH/hexanes: 8/92; flow rate: 0.7 mL/min; λ 254 nm); t_R (major) 24.78 min; t_R (minor) 41.21 min.



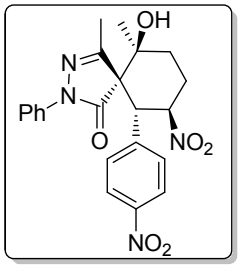
(5*R*,6*S*,9*R*,10*R*)-10-(3-chlorophenyl)-6-hydroxy-4,6-dimethyl-9-nitro-2-phenyl-2,3-diazaspiro[4.5]dec-3-en-1-one (**3j**)**:**



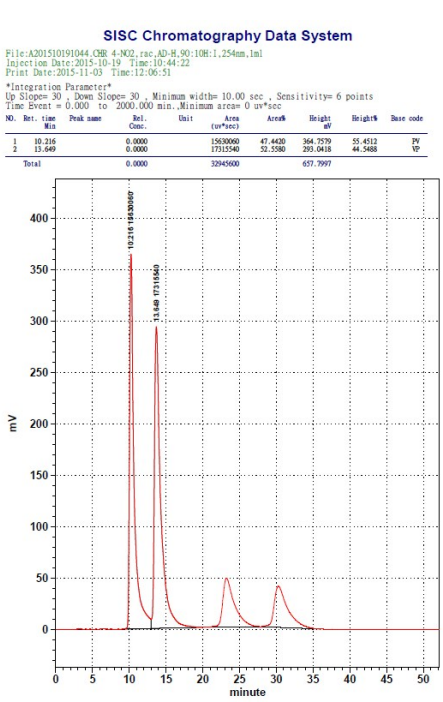
White solid, Yield: 54%, mp 95-97 ^{o}C; [α]25.6 D= -131.2 (*c* = 0.5 in CH_2Cl_2); IR (KBr): ν 3587, 2916, 2850, 2356, 1715, 1694, 1455, 1371, 1034, 738, 696 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60-7.58 (m, 2H), 7.39-7.35 (m, 2H), 7.22-7.18 (m, 2H), 7.17-7.14 (m, 1H), 7.10-7.08 (m, 2H), 5.81 (td, 1H, *J* = 11.8, 4.5 Hz), 4.11 (d, 1H, *J* = 11.8 Hz), 2.85 (td, 1H, *J* = 14.2, 4.4 Hz), 2.58-2.48 (m, 1H), 2.42-2.37 (m, 1H), 2.25 (s, 3H), 1.68-1.63 (m, 2H), 1.18 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.3, 160.7, 137.1, 136.2, 134.8, 130.2, 129.1, 129.0, 126.2, 120.1, 84.2, 72.2, 66.1, 45.7, 33.0, 27.1, 26.3, 17.7. HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{22}H_{23}N_3O_4Cl$ 428.1377; found 428.1377. The ee value of **3j** was 90% determined by HPLC with chiralpak AD-H column (i-PrOH/hexanes: 8/92; flow rate: 0.7 mL/min; λ 254 nm); t_R (major) 12.18 min; t_R (minor) 16.53 min.

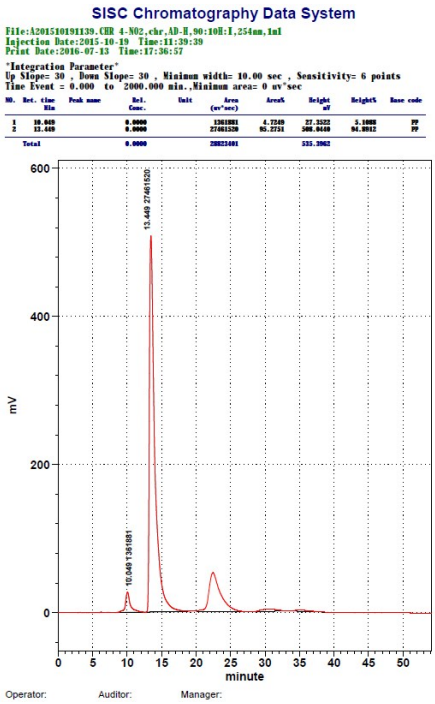


(5*R*,6*S*,9*R*,10*R*)-6-hydroxy-4,6-dimethyl-9-nitro-10-(4-nitrophenyl)-2-phenyl-2,3-diazaspiro[4.5]dec-3-en-1-one (3k):

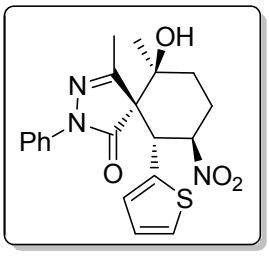


White solid, Yield: 53%, mp 159-161 ^{o}C; [α]25.6 D= -117.0 (*c* = 0.5 in CH_2Cl_2); IR (KBr): ν 3566, 2916, 2849, 1715, 1695, 1348, 740 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.03 (d, 2H, *J* = 8.8 Hz), 7.61-7.59 (m, 2H), 7.42-7.35 (m, 4H), 7.23-7.20 (m, 1H), 5.86 (td, 1H, *J* = 11.7, 4.5 Hz), 4.29 (d, 1H, *J* = 11.7 Hz), 2.86 (td, 1H, *J* = 14.1, 4.5 Hz), 2.62-2.51 (m, 1H), 2.45-2.40 (m, 1H), 2.25 (s, 3H), 1.80 (s, 1H), 1.73-1.67 (m, 1H), 1.20 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.0, 160.4, 148.1, 141.8, 136.9, 129.2, 126.3, 124.1, 119.6, 84.0, 72.1, 66.1, 45.7, 32.9, 27.0, 26.2, 17.7.. HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{22}H_{23}N_4O_6$ 439.1618; found 439.1621. The ee value of **3k** was 90% determined by HPLC with chiralpak AD-H column (i-PrOH/hexanes: 10/90; flow rate: 1 mL/min; λ 254 nm); t_R (minor) 10.04 min; t_R (major) 13.44 min.

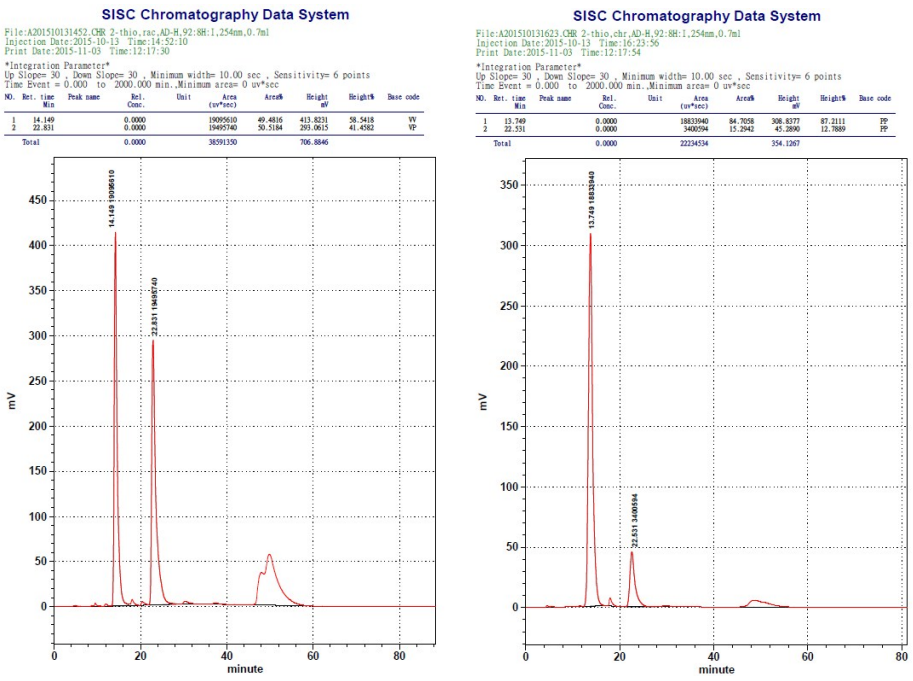




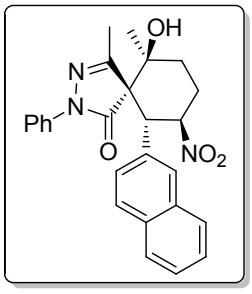
(5*R*,6*S*,9*R*,10*R*)-6-hydroxy-4,6-dimethyl-9-nitro-2-phenyl-10-(thiophen-2-yl)-2,3-diazaspiro[4.5]dec-3-en-1-one (3l):



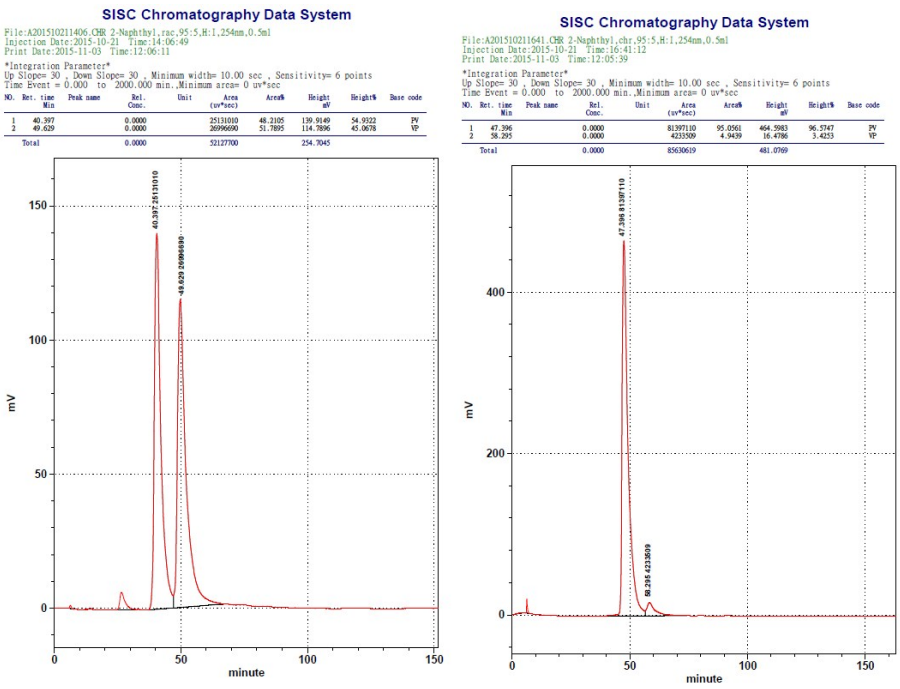
White solid, Yield: 53%, mp 72-74 ^{o}C; [α]25.6 D= -100.3 (*c* = 0.5 in CH_2Cl_2); IR (KBr): ν 3567, 2916, 2850, 2427, 1696, 1552, 1295, 739 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.69-7.67 (m, 2H), 7.39-7.35 (m, 2H), 7.21-7.19 (m, 1H), 7.08-7.07 (m, 1H), 6.90-6.89 (m, 1H), 6.79-6.77 (m, 1H), 5.77 (td, 1H, *J* = 11.7, 4.5 Hz), 4.43 (d, 1H, *J* = 11.7 Hz), 2.78 (td, 1H, *J* = 14.1, 4.4 Hz), 2.57-2.46 (m, 1H), 2.37-2.31 (m, 1H), 2.27 (s, 3H), 1.81 (s, 1H), 1.65-1.60 (m, 1H), 1.18 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.3, 160.9, 137.4, 136.1, 129.0, 127.1, 125.9, 125.7, 119.8, 85.8, 72.2, 66.7, 41.2, 32.7, 27.0, 26.3, 17.6. HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{20}H_{22}N_3O_4S$ 400.1331; found 400.1333. The ee value of **3l** was 70% determined by HPLC with chiralpak AD-H column (i-PrOH/hexanes: 8/92; flow rate: 0.7 mL/min; λ 254 nm); t_R (major) 13.74 min; t_R (minor) 22.53 min.



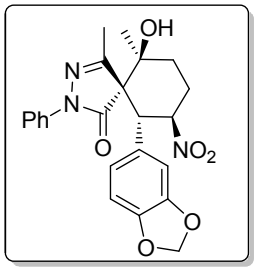
(5*R*,6*S*,9*R*,10*R*)-6-hydroxy-4,6-dimethyl-10-(naphthalen-2-yl)-9-nitro-2-phenyl-2,3-diazaspiro[4.5]dec-3-en-1-one (3m):



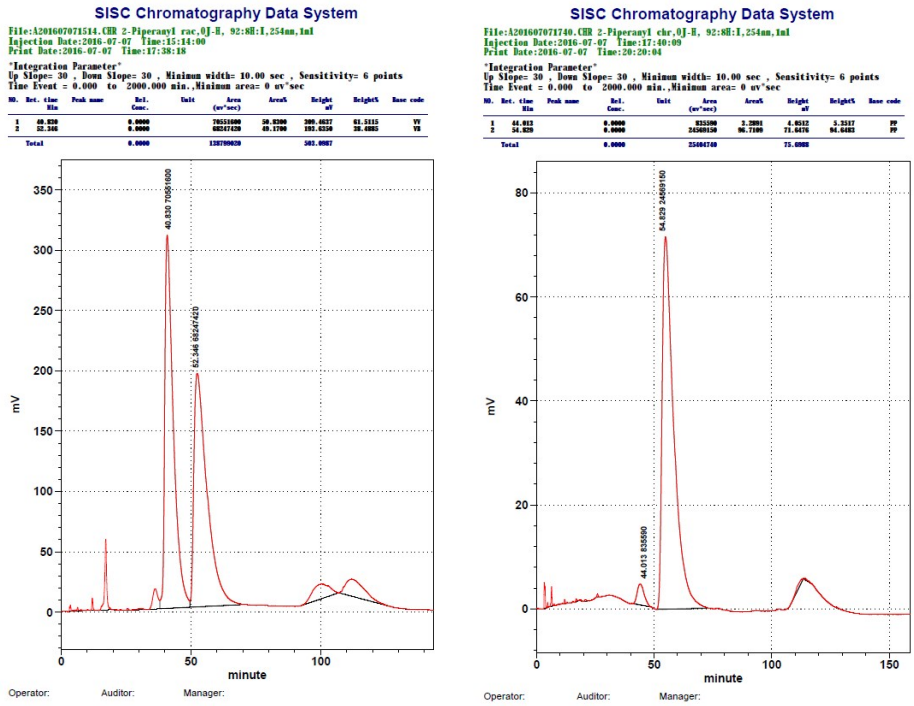
White solid, Yield: 56%, mp 100-102 ^{o}C; [α]25.6 D= -122.8 (*c* = 0.5 in CH_2Cl_2); IR (KBr): ν 3565, 2919, 2356, 2329, 1715, 1695, 1496, 750 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.70-7.62 (m, 4H), 7.55 (d, 2H, *J* = 7.8 Hz), 7.40-7.38 (m, 2H), 7.33-7.29 (m, 3H), 7.19-7.15 (m, 1H), 5.95 (td, 1H, *J* = 11.8, 4.4 Hz), 4.30 (d, 1H, *J* = 11.8 Hz), 2.90 (td, 1H, *J* = 14.2, 4.4 Hz), 2.65-2.54 (m, 1H), 2.43-2.37 (m, 1H), 2.25 (s, 3H), 1.68 (s, 1H), 1.66-1.64 (m, 1H), 1.19 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.8, 161.0, 137.2, 133.4, 133.3, 131.6, 129.0, 128.1, 127.7, 126.5, 126.5, 126.0, 120.0, 84.7, 72.3, 66.4, 46.4, 33.0, 27.2, 26.3, 17.7. HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{26}H_{26}N_3O_4$ 444.1923; found 444.1924. The ee value of **3m** was 90% determined by HPLC with chiralpak AD-H column (i-PrOH/hexanes: 5/95; flow rate: 0.5 mL/min; λ 254 nm); t_R (major) 47.39 min; t_R (minor) 58.29 min.



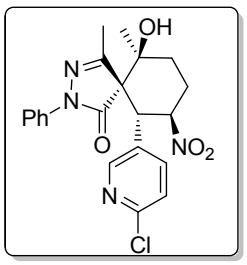
(5*R*,6*S*,9*R*,10*R*)-10-(benzo[d][1,3]dioxol-5-yl)-6-hydroxy-4,6-dimethyl-9-nitro-2-phenyl-2,3-diazaspiro[4.5]dec-3-en-1-one (3n):



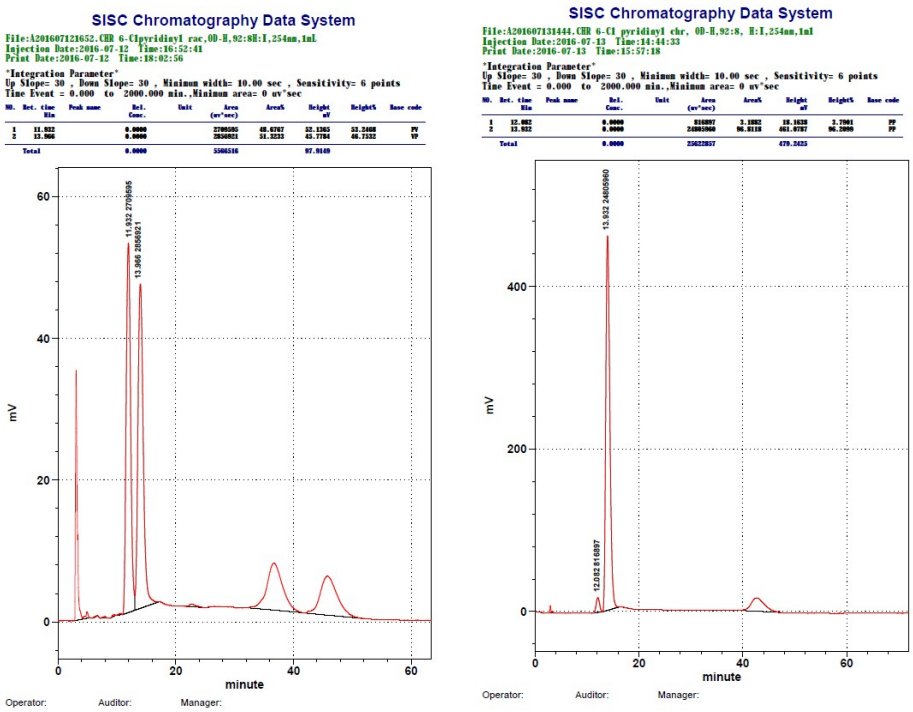
White solid, Yield: 51%, mp 99-101 ^{o}C; [α]25.6 D= -107.4 (*c* = 0.5 in CH_2Cl_2); IR (KBr): ν 3586, 2916, 2849, 1695, 1715, 1495, 1244, 1038, 928, 756 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.63 (d, 2H, *J* = 7.6 Hz), 7.36 (t, 2H, *J* = 7.9 Hz), 7.19 (t, 1H, *J* = 7.4 Hz), 6.70-6.66 (m, 2H), 6.56 (d, 1H, *J* = 8.0 Hz), 5.84-5.84 (d, 1H, *J* = 1.2 Hz), 5.81 (s, 1H), 5.71 (td, 1H, *J* = 11.8, 4.4 Hz), 4. 03 (d, 1H, *J* = 11.8 Hz), 2.81 (td, 1H, *J* = 14.2, 4.4 Hz), 2.56-2.46 (m, 1H), 2.37-2.31 (m, 1H), 2.24 (s, 3H), 1.68 (s, 1H), 1.64-1.60 (m, 1H), 1.15 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.6, 161.1, 147.8, 137.3, 129.0, 127.6, 126.0, 120.0, 108.6, 101.3, 84.8, 72.2, 66.5, 45.8, 32.9, 27.1, 26.3, 17.7. HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{23}H_{24}N_3O_6$ 438.1665; found 438.1664. The ee value of **3n** was 94% determined by HPLC with chiralpak OJ-H column (i-PrOH/hexanes: 8/92; flow rate: 1 mL/min; λ 254 nm); t_R (minor) 44.01 min; t_R (major) 54.89 min.



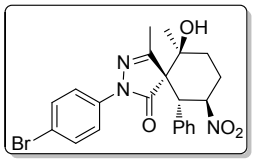
(5*R*,6*S*,9*R*,10*R*)-10-(6-chloropyridin-3-yl)-6-hydroxy-4,6-dimethyl-9-nitro-2-phenyl-2,3-diazaspiro[4.5]dec-3-en-1-one (3o):



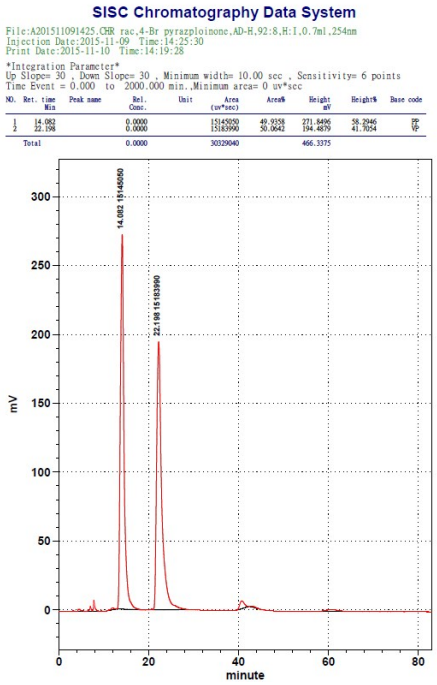
White solid, Yield: 40%, mp 98-100 ^{o}C; [α]25.6 D= -219.0 (*c* = 0.5 in CH_2Cl_2); IR (KBr): ν 3547, 3058, 2916, 2850, 2363, 2344, 1715, 1456, 1024, 838, 739, 692 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.24 (d, 1H, *J* = 2.5 Hz), 7.64-7.62 (m, 2H), 7.53-7.50 (m, 1H), 7.40-7.36 (m, 2H), 7.24-7.20 (m, 1H), 7.10 (d, 1H, *J* = 8.3 Hz), 5.78 (td, 1H, *J* = 11.8, 4.5 Hz), 4. 18 (d, 1H, *J* = 11.8 Hz), 2.84 (td, 1H, *J* = 14.2, 4.4 Hz), 2.61-2.54 (m, 1H), 2.43-2.39 (m, 1H), 2.26 (s, 3H), 1.72-1.66 (m, 2H), 1.20 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.0, 160.4, 152.1, 150.3, 137.0, 129.2, 126.3, 124.6, 119.6, 83.8, 72.1, 66.6, 45.7, 33.0, 30.0, 27.0, 26.3, 17.7. HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{21}H_{22}N_4O_4Cl$ 429.1330; found 429.1333. The ee value of **3o** was 94% determined by HPLC with chiralpak OD-H column (i-PrOH/hexanes: 8/92; flow rate: 1 mL/min; λ 254 nm); t_R (minor) 12.08 min; t_R (major) 13.93 min.

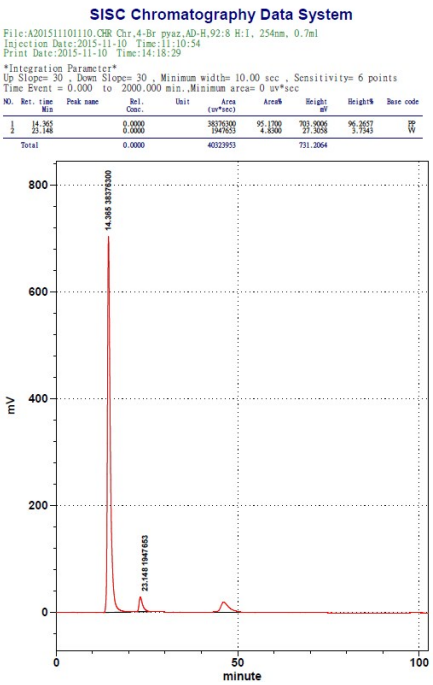


(5*R*,6*S*,9*R*,10*R*)-2-(4-bromophenyl)-6-hydroxy-4,6-dimethyl-9-nitro-10-phenyl-2,3-diazaspiro[4.5]dec-3-en-1-one (3p):



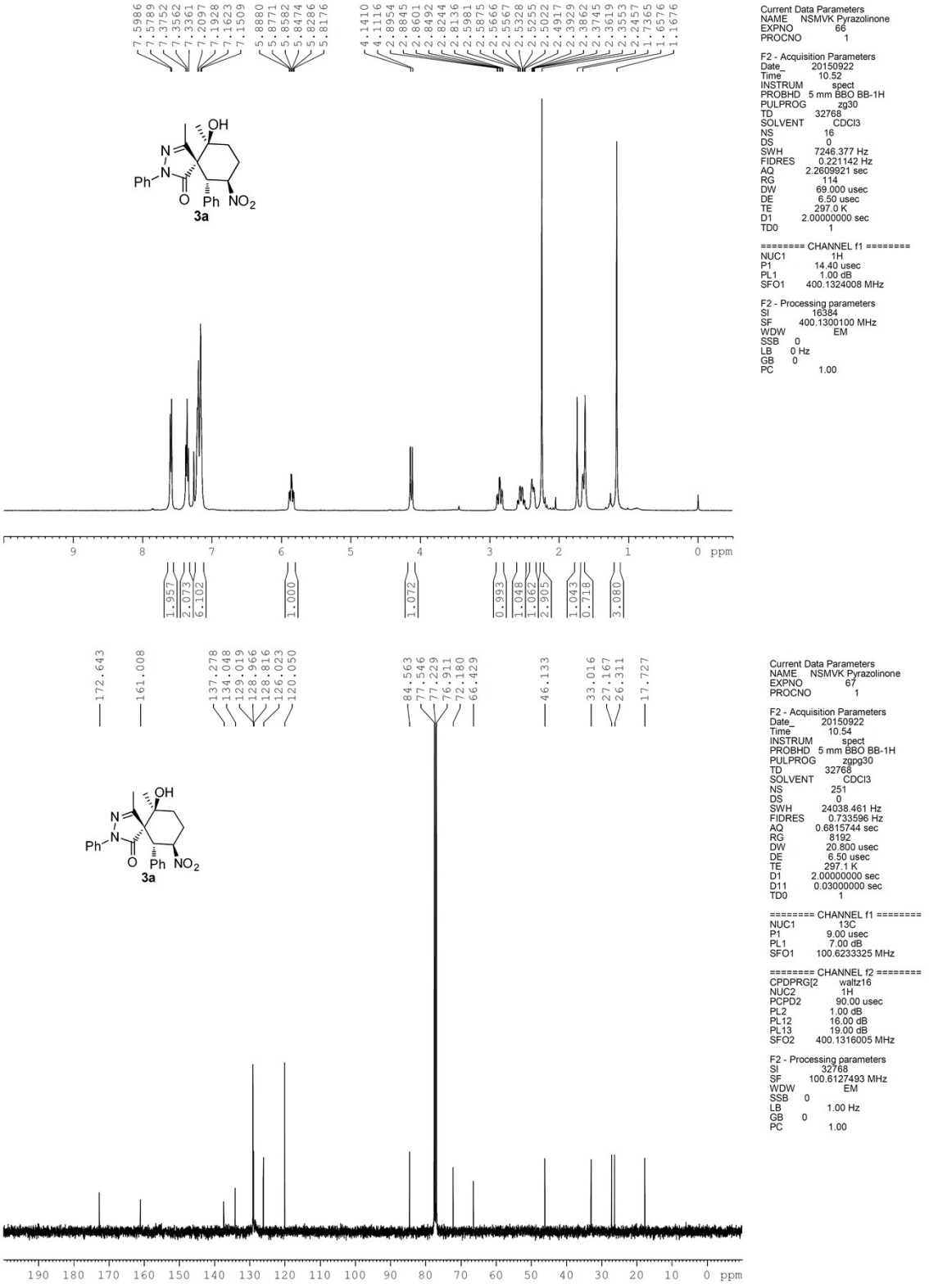
White solid, Yield: 67%, mp 79-81 ^{o}C; [α]25.6 D= -156.0 (*c* = 0.5 in CH_2Cl_2); IR (KBr): ν 3590, 2917, 2850, 1698, 1548, 1490, 1364, 703 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.55-7.53 (m, 2H), 7.47-7.44 (m, 2H), 7.15-7.15 (m, 5H), 5.81 (td, 1H, *J* = 11.8, 4.4 Hz), 4. 11 (d, 1H, *J* = 11.8 Hz), 2.82 (td, 1H, *J* = 14.2, 4.4 Hz), 2.59-2.48 (m, 1H), 2.40-2.35 (m, 1H), 2.24 (s, 3H), 1.75 (s, 1H), 1.66-1.63 (m, 1H), 1.15 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.6, 161.3, 136.3, 133.9, 132.0, 128.9, 128.8, 121.1, 118.8, 84.4, 72.1, 66.5, 46.1, 33.0, 27.1, 26.3, 17.7. HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{22}H_{23}N_3O_4Br$ 472.0872; found 472.0875. The ee value of **3p** was 90% determined by HPLC with chiralpak AD-H column (i-PrOH/hexanes: 8/92; flow rate: 0.7 mL/min; λ 254 nm); t_R (major) 14.36 min; t_R (minor) 23.14 min.



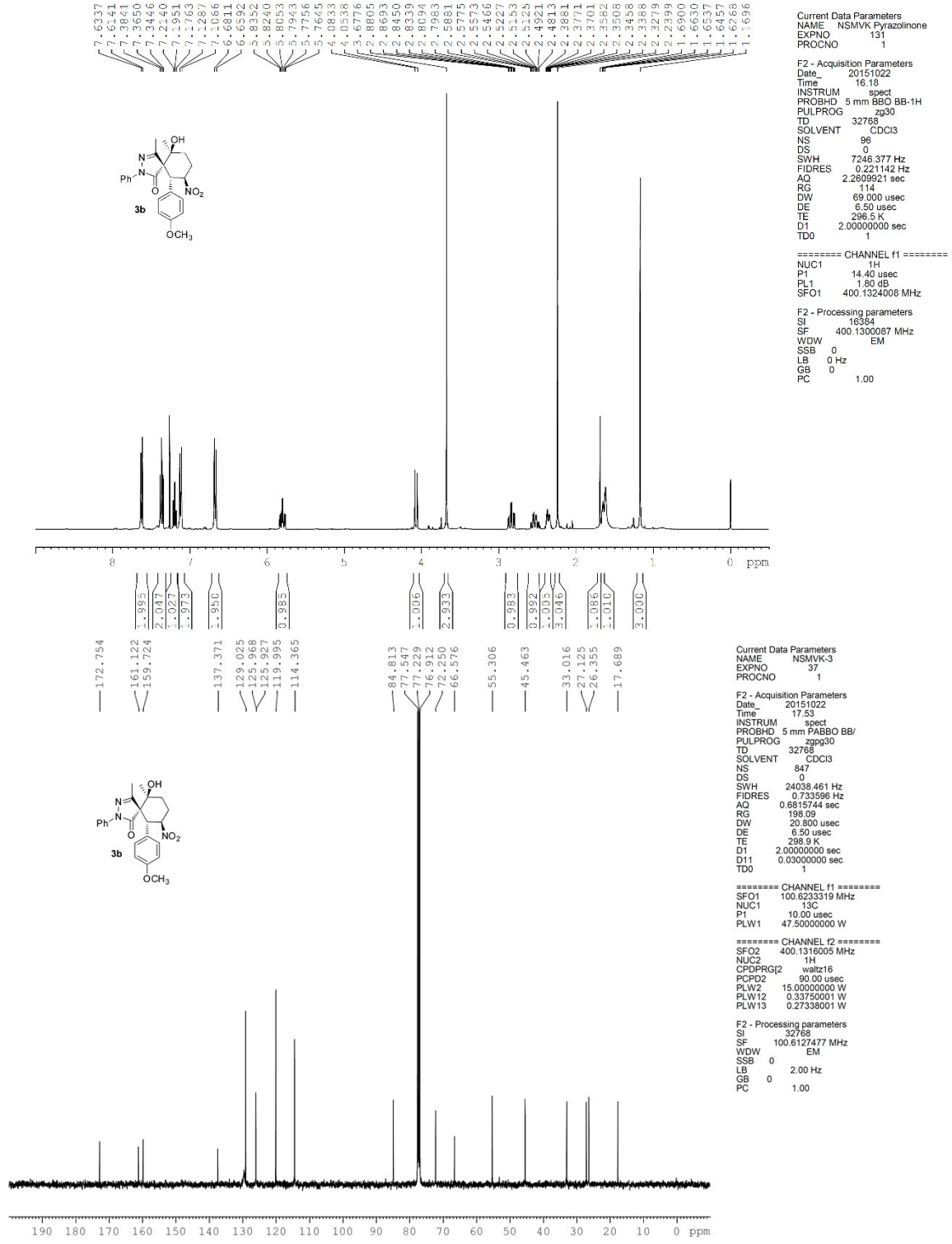


^{1}H and ^{13}C NMR spectral copies:

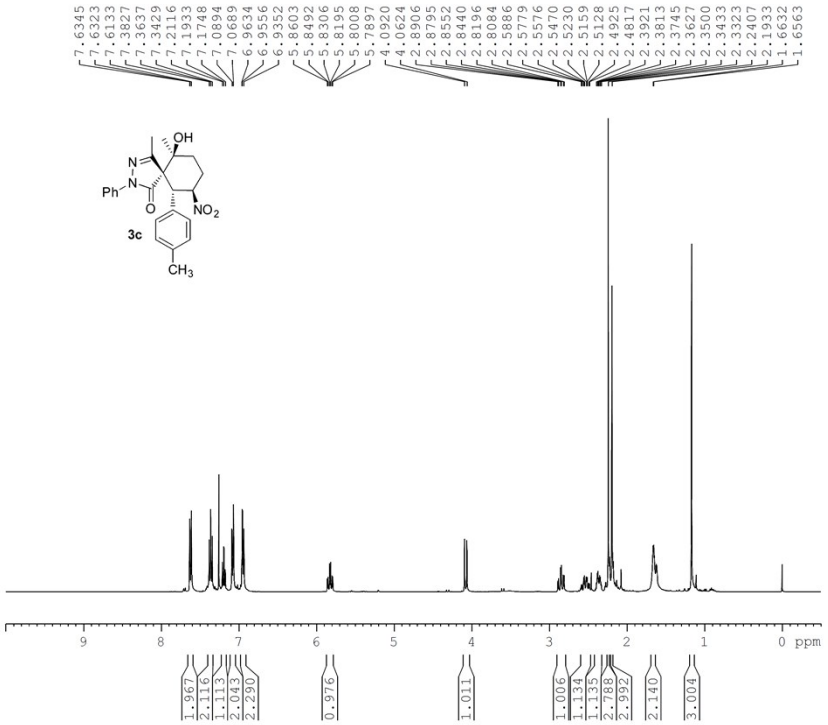
(5*R*,6*S*,9*R*,10*R*)-6-hydroxy-4,6-dimethyl-9-nitro-2,10-diphenyl-2,3-diazaspiro[4.5]dec-3-en-1-one (3a):



(5*R*,6*S*,9*R*,10*R*)-6-hydroxy-10-(4-methoxyphenyl)-4,6-dimethyl-9-nitro-2-phenyl-2,3-diazaspiro[4.5]dec-3-en-1-one (3b):



(5*R*,6*S*,9*R*,10*R*)-6-hydroxy-4,6-dimethyl-9-nitro-2-phenyl-10-(p-tolyl)-2,3-diazaspiro[4.5]dec-3-en-1-one (3c):

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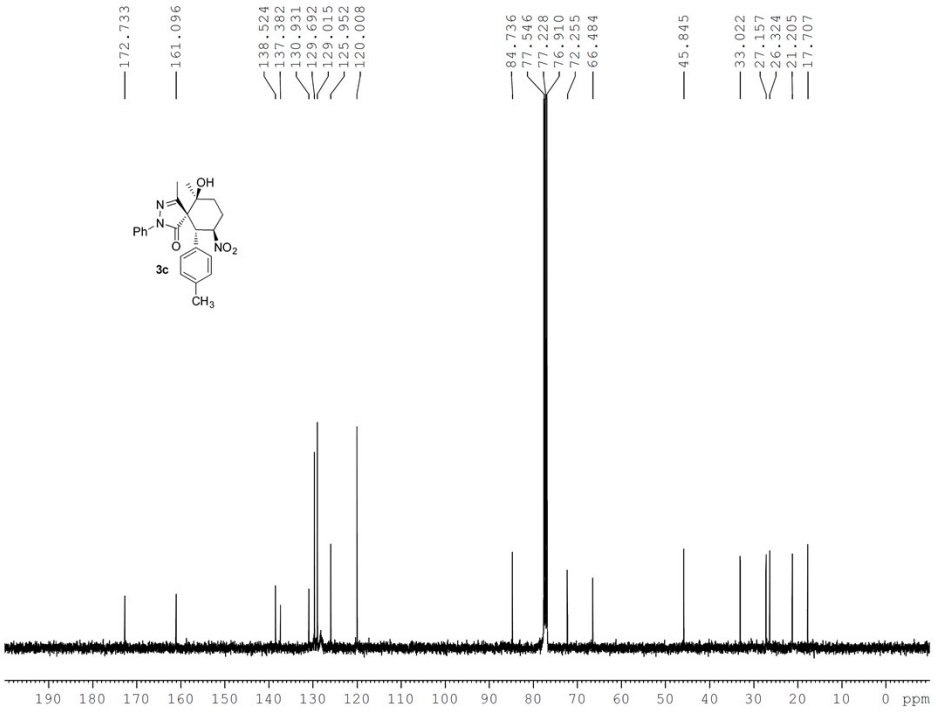


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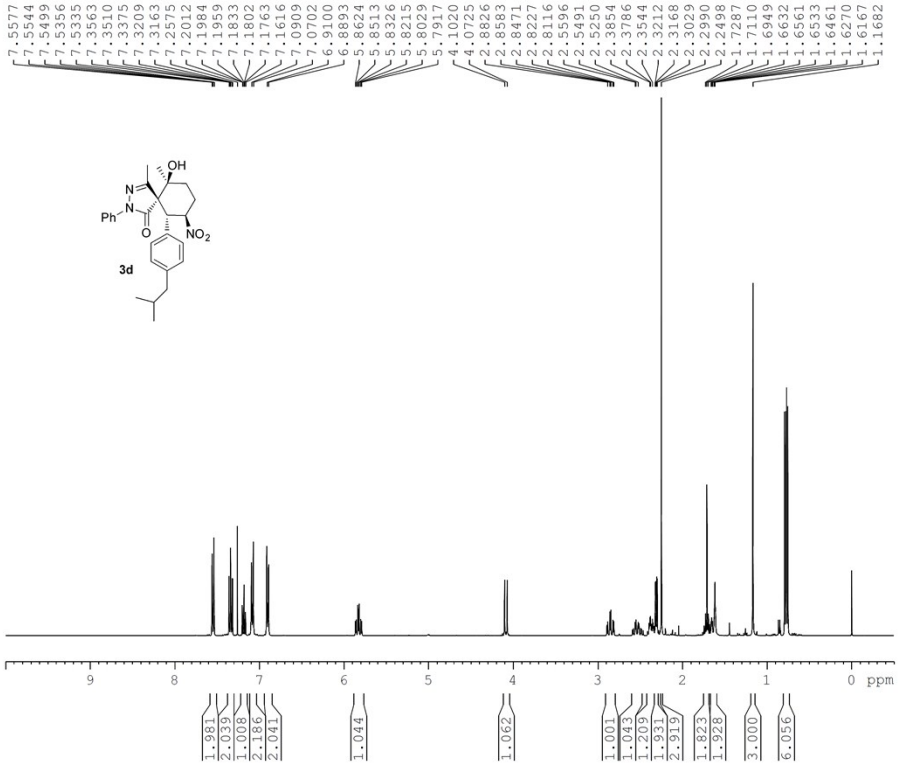
F2 - Acquisition Parameters
Date_              20160315
Time                  12.17
INSTRUM               spect
PROBHD    5 mm BBO BB-1H
PULPROG             zgpg30
TD                    32768
SOLVENT               CDCl3
NS                     1181
DS                        0
SWH               24038.461 Hz
FIDRES            0.733596 Hz
AQ                0.6815744 sec
RG                     8192
DW                  20.800 usec
DE                   6.50 usec
TE                   299.2 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                    9.00 usec
PL1                   7.00 dB
SFO1         100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                    1H
PCPD2                90.00 usec
PL2                   1.80 dB
PL12                 17.00 dB
PL13                 20.00 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                       32768
SF            100.6127476 MHz
WDW                         EM
SSB          0
LB                    1.00 Hz
GB           0
PC                       1.00
```

(5*R*,6*S*,9*R*,10*R*)-6-hydroxy-10-(4-isobutylphenyl)-4,6-dimethyl-9-nitro-2-phenyl-2,3-diazaspiro[4.5]dec-3-en-1-one (3d):

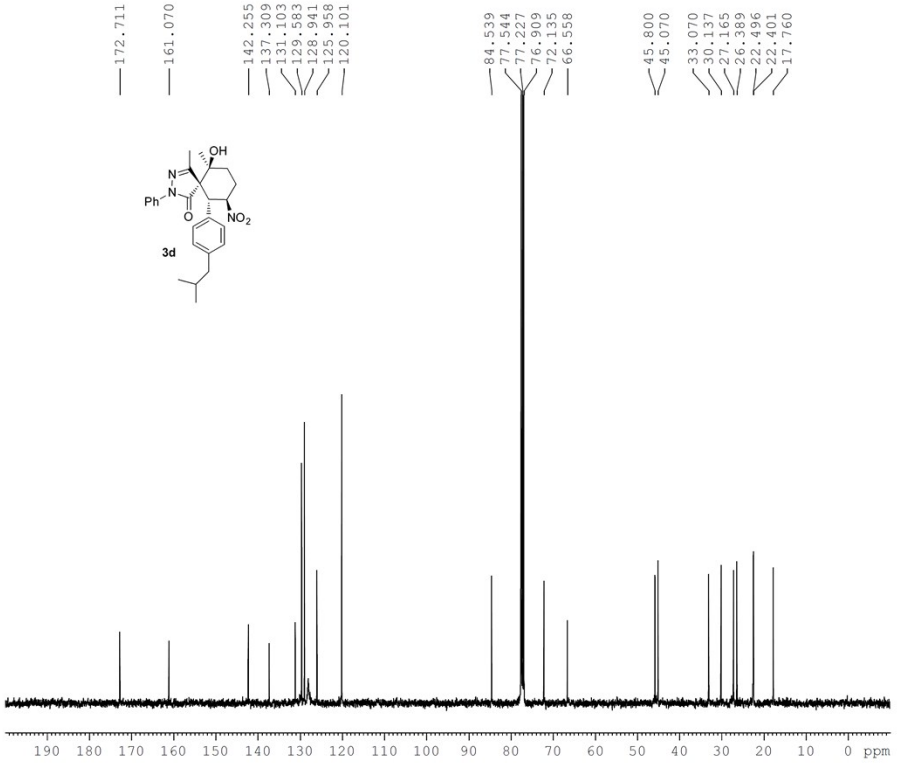


Current Data Parameters
NAME NSMVK-3
EXPNO 33
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151021
Time 15.38
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 64
DS 0
SWH 7211.539 Hz
FIDRES 0.220079 Hz
AQ 2.2719147 sec
RG 71.42
DW 69.333 usec
DE 10.50 usec
TE 297.9 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 12.90 usec
PLW1 15.00000000 W

F2 - Processing parameters
SI 16384
SF 400.1300108 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00



Current Data Parameters
NAME NSMVK-3
EXPNO 34
PROCNO 1

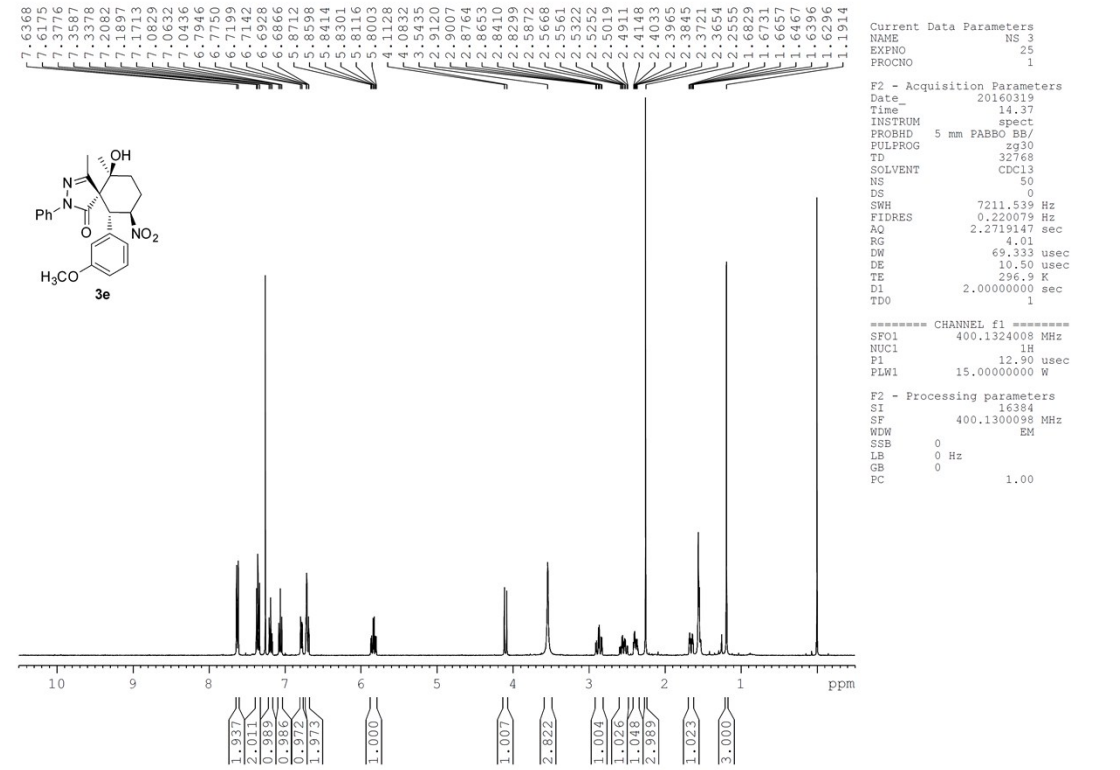
F2 - Acquisition Parameters
Date_ 20151021
Time 15.45
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 509
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 198.09
DW 20.800 usec
DE 6.50 usec
TE 298.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6233319 MHz
NUC1 13C
P1 10.00 usec
PLW1 47.50000000 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 15.00000000 W
PLW12 0.33750001 W
PLW13 0.27338001 W

F2 - Processing parameters
SI 32768
SF 100.6127488 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

(5*R*,6*S*,9*R*,10*R*)-6-hydroxy-10-(3-methoxyphenyl)-4,6-dimethyl-9-nitro-2-phenyl-2,3-diazaspiro[4.5]dec-3-en-1-one (3e):

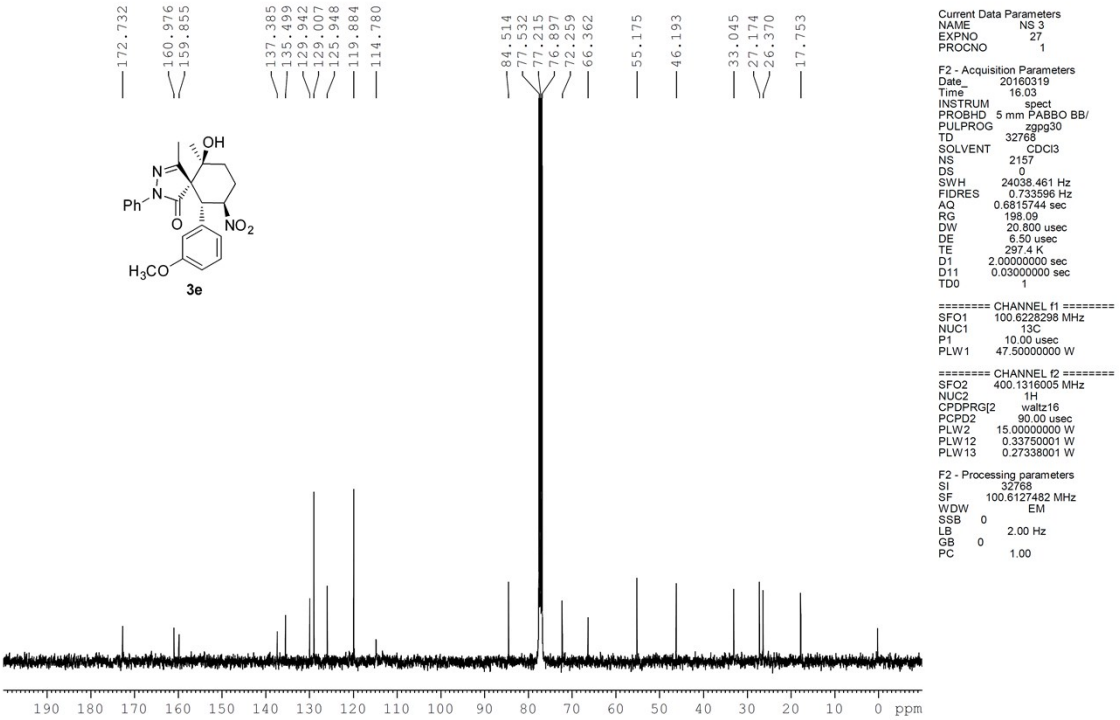


Current Data Parameters
NAME NS 3
EXPNO 25
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160319
Time 14.37
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 50
DS 0
SWH 7211.539 Hz
FIDRES 0.220079 Hz
AQ 2.2719147 sec
RG 4.01
DW 69.333 usec
DE 10.50 usec
TE 296.9 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 12.90 usec
PLW1 15.00000000 W

F2 - Processing parameters
SI 16384
SF 400.1300098 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00



Current Data Parameters
NAME NS 3
EXPNO 27
PROCNO 1

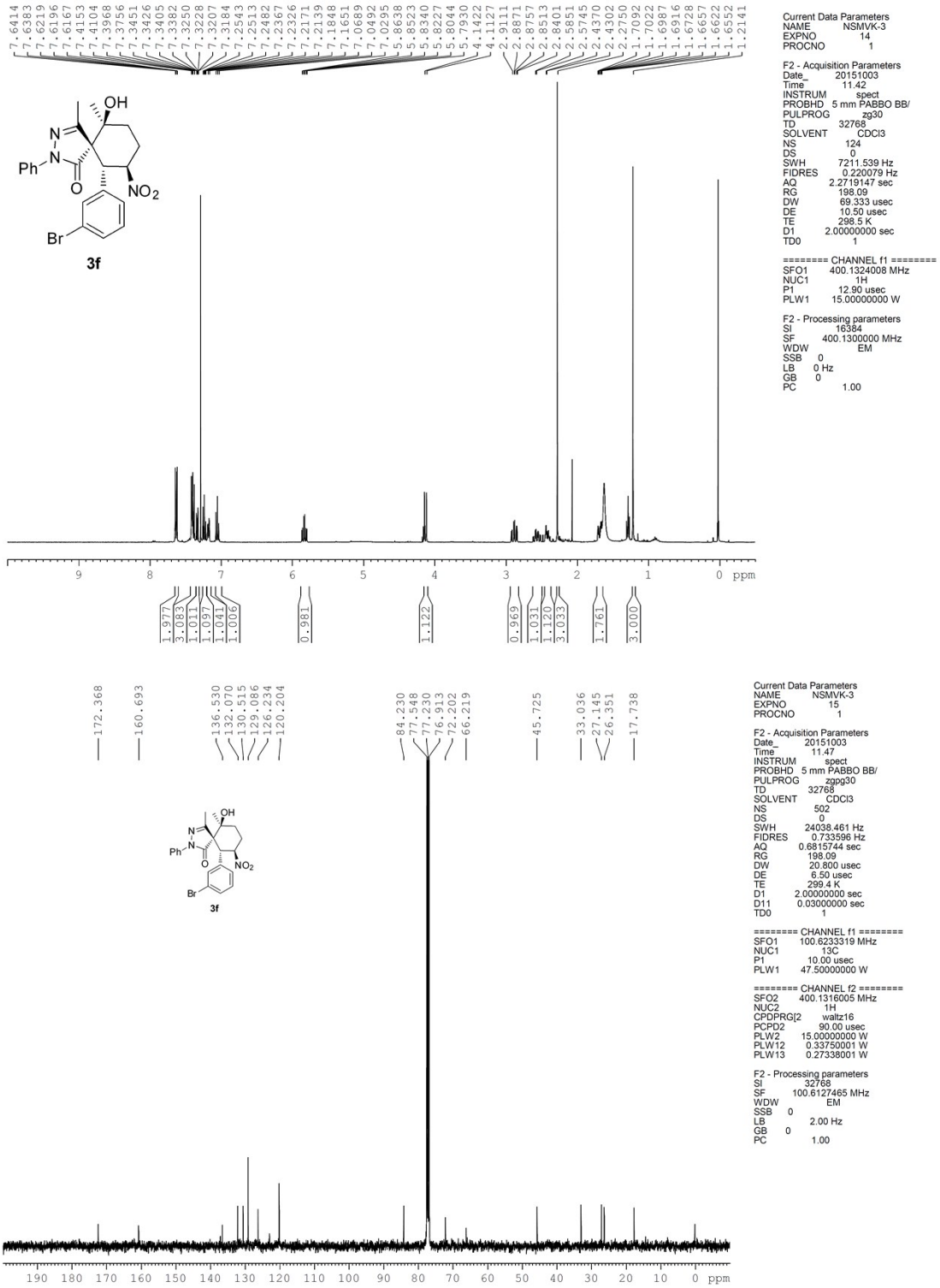
F2 - Acquisition Parameters
Date_ 20160319
Time 16.03
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 2157
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 198.09
DW 20.800 usec
DE 6.50 usec
TE 297.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 47.50000000 W

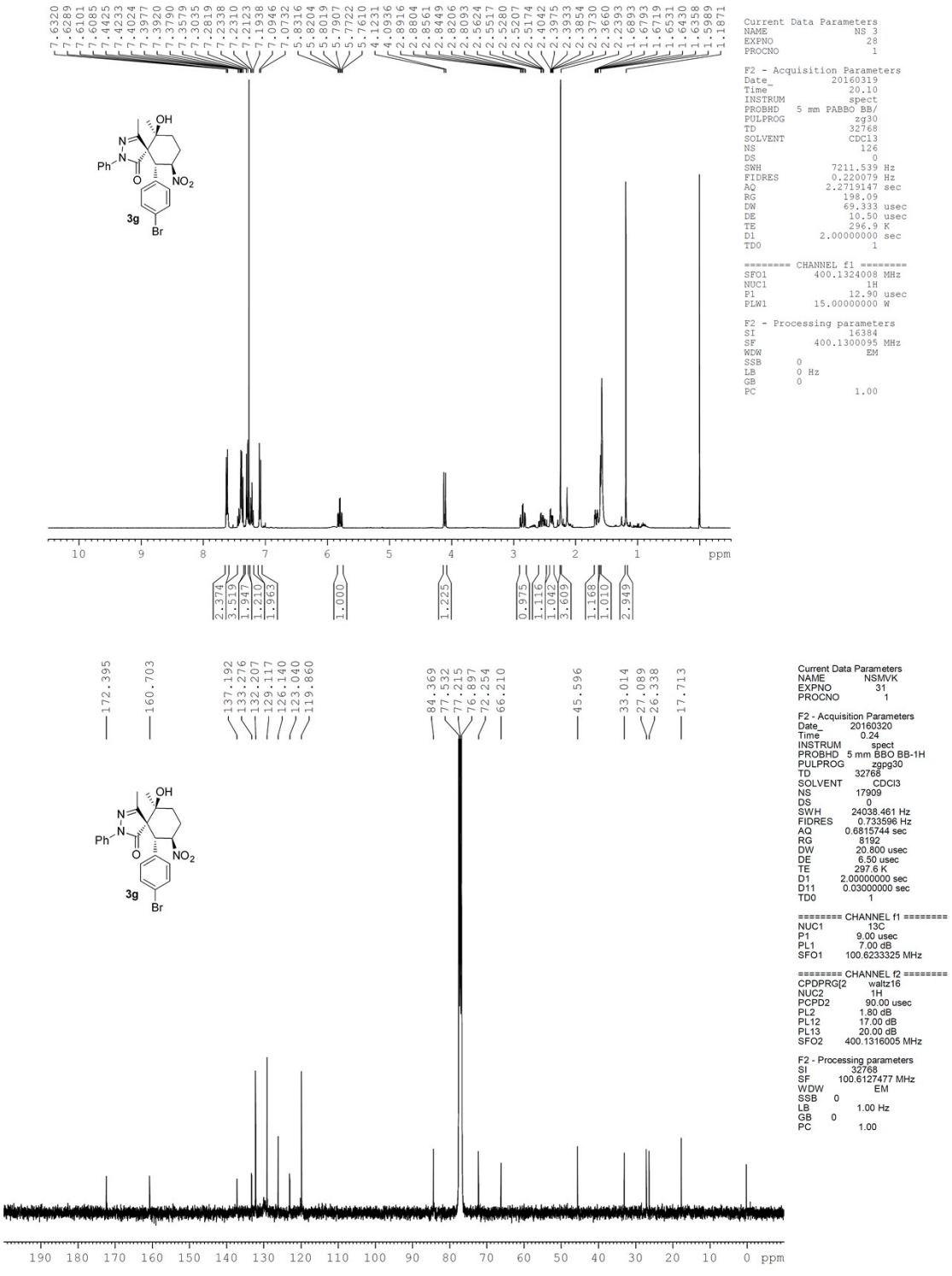
======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 15.00000000 W
PLW12 0.33750001 W
PLW13 0.27338001 W

F2 - Processing parameters
SI 32768
SF 100.6127482 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

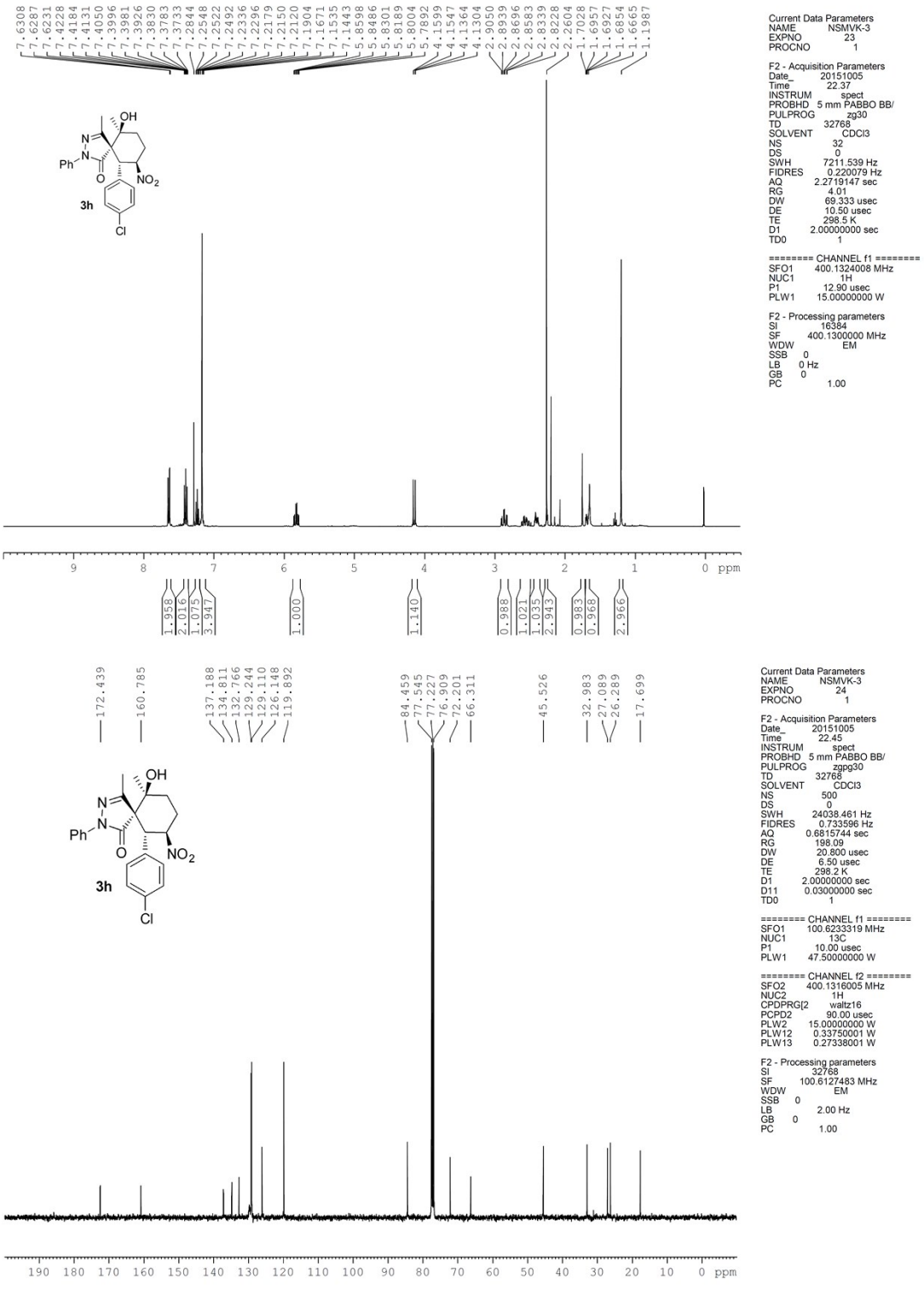
(5*R*,6*S*,9*R*,10*R*)-10-(3-bromophenyl)-6-hydroxy-4,6-dimethyl-9-nitro-2-phenyl-2,3-diazaspiro[4.5]dec-3-en-1-one (3f):



(5*R*,6*S*,9*R*,10*R*)-10-(4-bromophenyl)-6-hydroxy-4,6-dimethyl-9-nitro-2-phenyl-2,3-diazaspiro [4.5] dec-3-en-1-one (3g):



(5*R*,6*S*,9*R*,10*R*)-10-(4-chlorophenyl)-6-hydroxy-4,6-dimethyl-9-nitro-2-phenyl-2,3-diazaspiro[4.5]dec-3-en-1-one (3h):



Current Data Parameters
NAME NSMVK-3
EXPNO 23
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151005
Time 22.37
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 7211.539 Hz
FIDRES 0.220079 Hz
AQ 2.2719147 sec
RG 4.01
DW 69.333 usec
DE 10.50 usec
TE 298.5 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 12.90 usec
PLW1 15.00000000 W

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

Current Data Parameters
NAME NSMVK-3
EXPNO 24
PROCNO 1

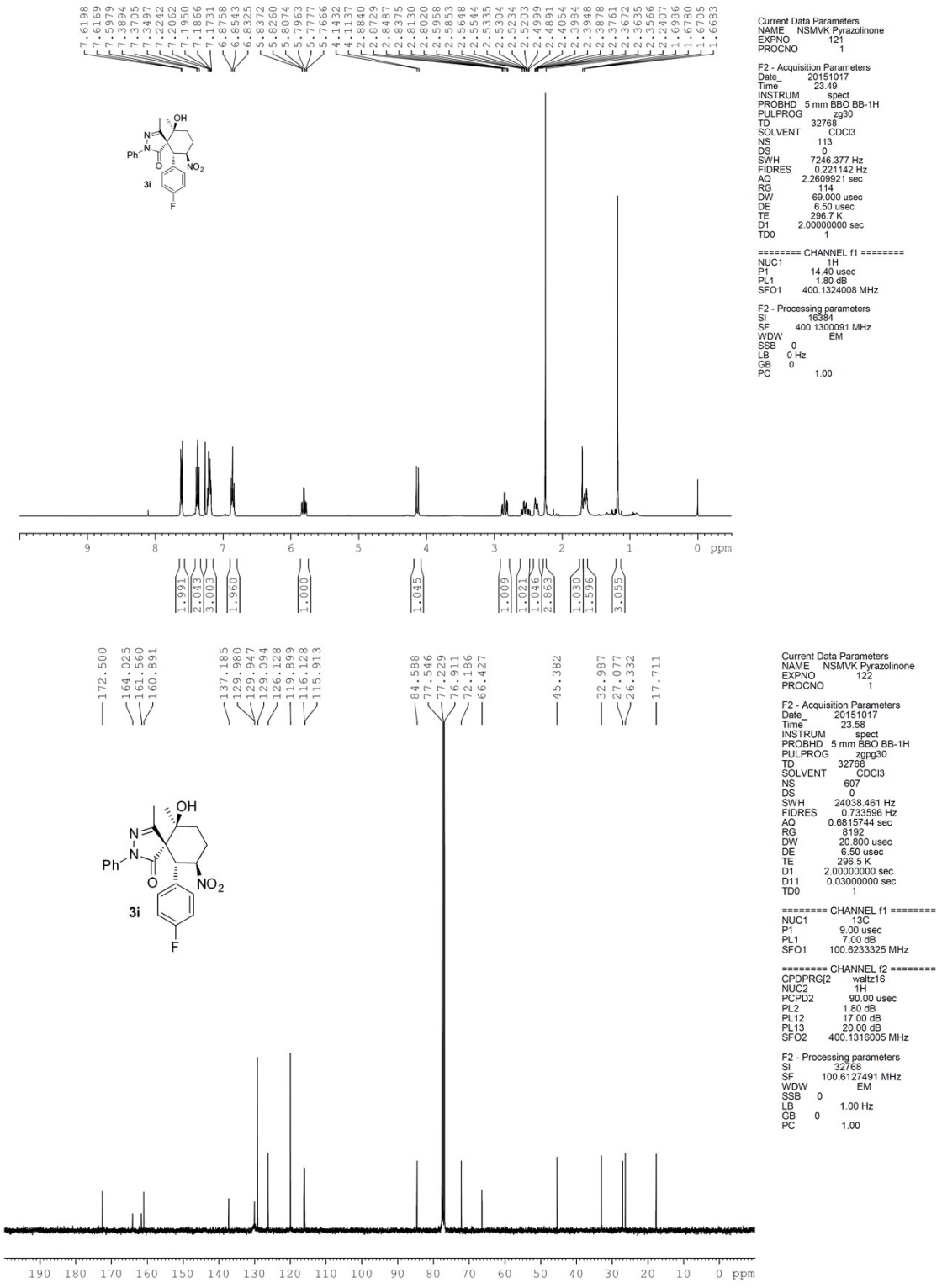
F2 - Acquisition Parameters
Date_ 20151005
Time 22.45
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 500
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 198.09
DW 20.800 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6233319 MHz
NUC1 13C
P1 10.00 usec
PLW1 47.50000000 W

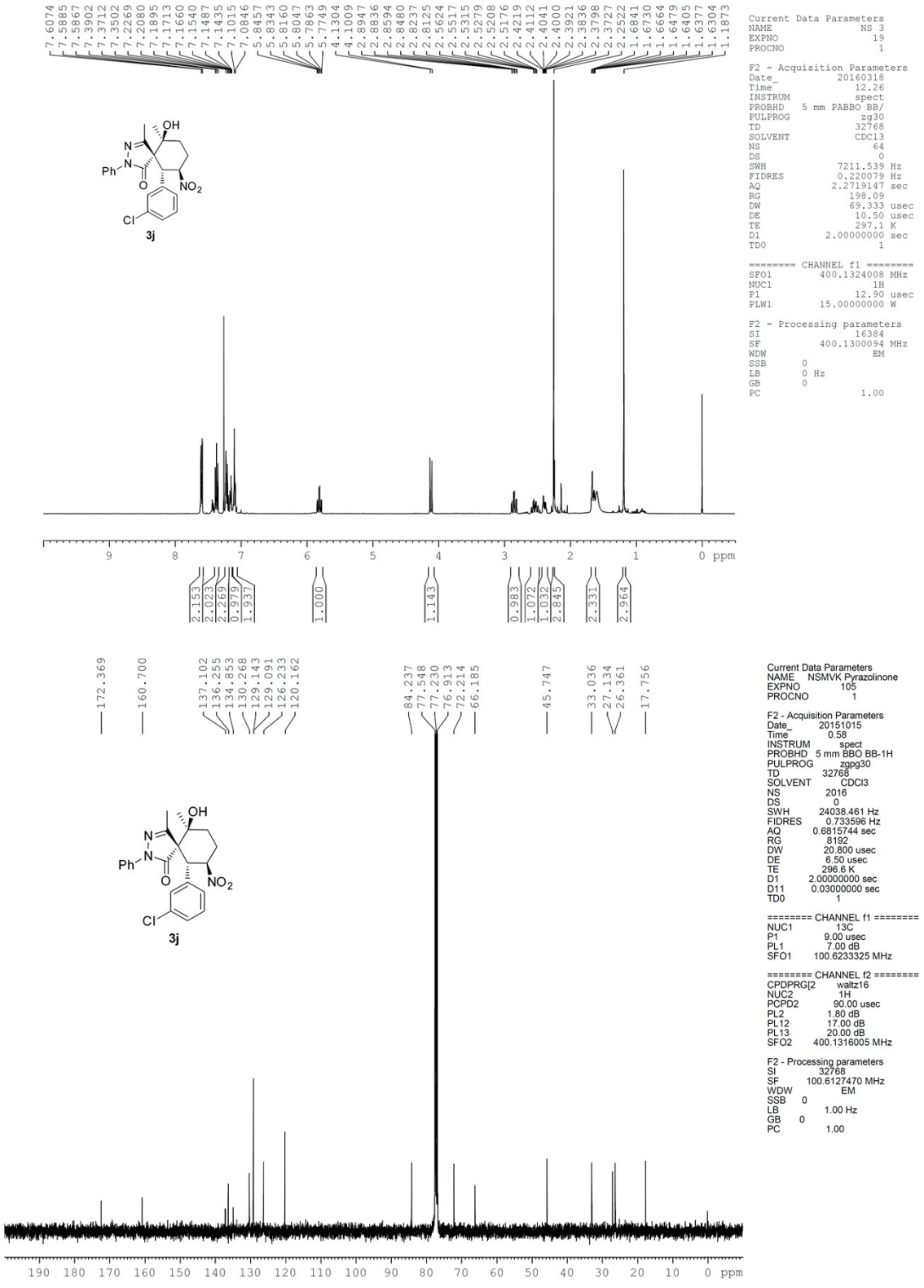
======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 15.00000000 W
PLW12 0.33750001 W
PLW13 0.27338001 W

F2 - Processing parameters
SI 32768
SF 100.6127483 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

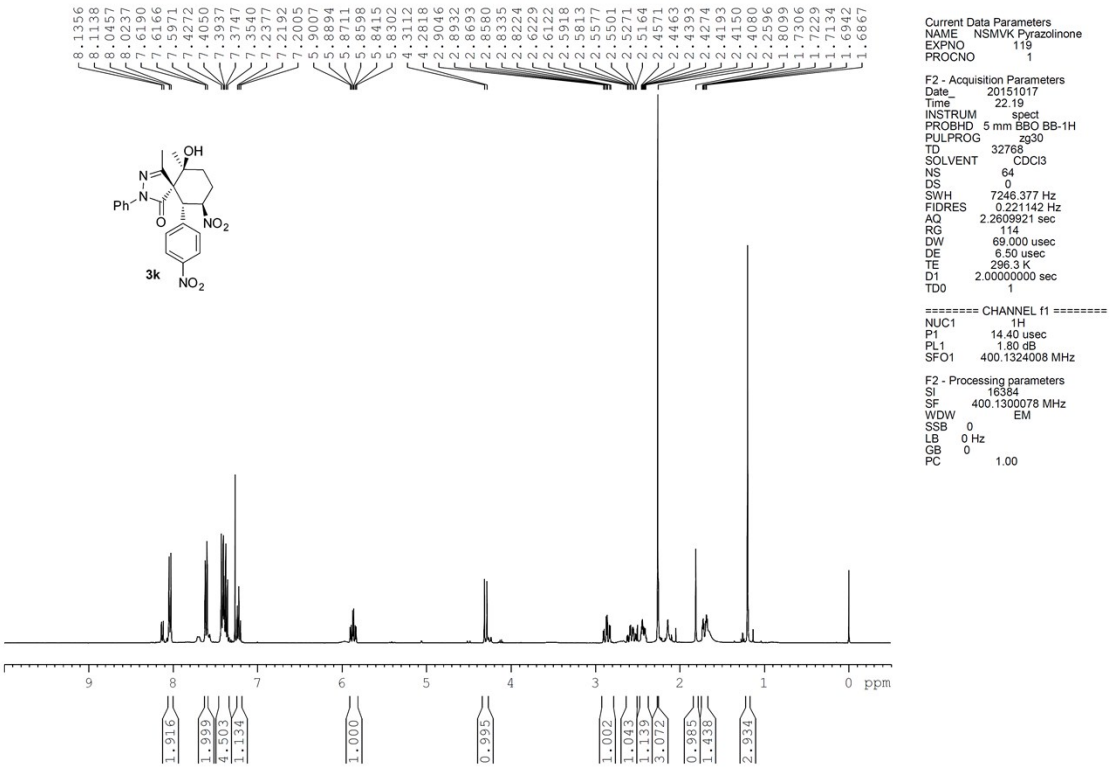
(5*R*,6*S*,9*R*,10*R*)-10-(4-fluorophenyl)-6-hydroxy-4,6-dimethyl-9-nitro-2-phenyl-2,3-diazaspiro[4.5]dec-3-en-1-one (3i):



(5*R*,6*S*,9*R*,10*R*)-10-(3-chlorophenyl)-6-hydroxy-4,6-dimethyl-9-nitro-2-phenyl-2,3-diazaspiro[4.5]dec-3-en-1-one (3j):



(5*R*,6*S*,9*R*,10*R*)-6-hydroxy-4,6-dimethyl-9-nitro-10-(4-nitrophenyl)-2-phenyl-2,3-diazaspiro[4.5]dec-3-en-1-one (3k):

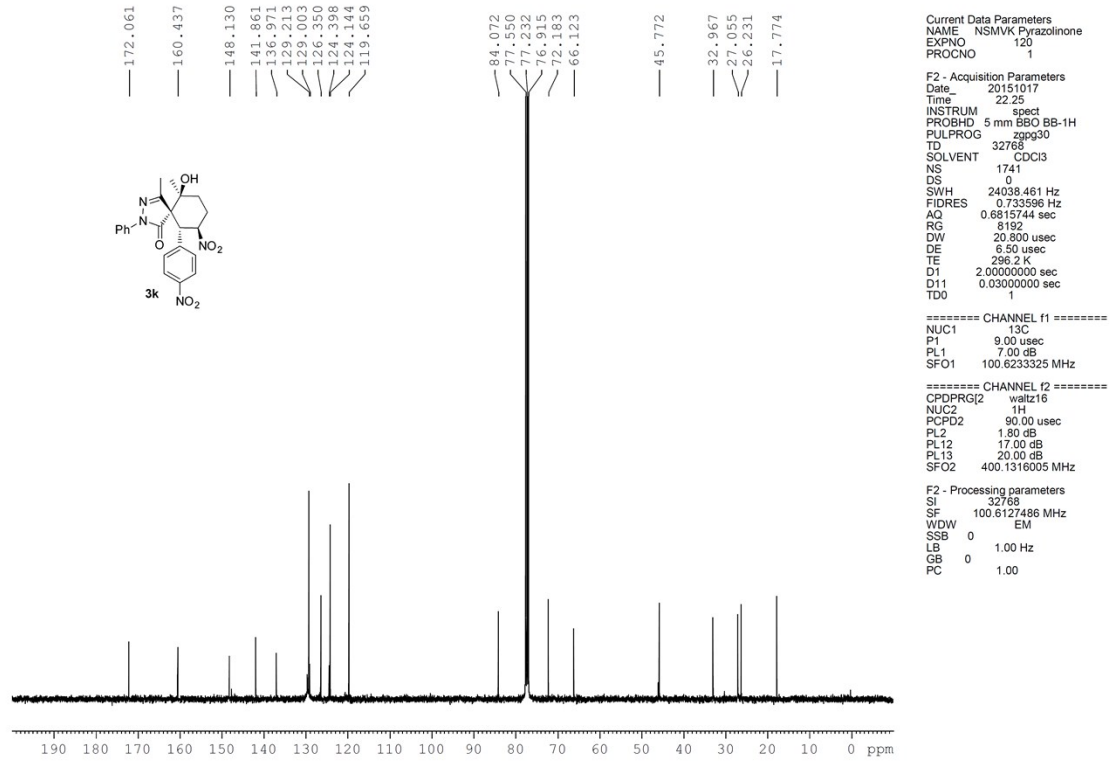



```
Current Data Parameters
NAME      NSMVK Pyrazolinone
EXPNO              119
PROCNO               1

F2 - Acquisition Parameters
Date_          20151017
Time             22.19
INSTRUM          spect
PROBHD  5 mm BBO BB-1H
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                  64
DS                   0
SWH           7246.377 Hz
FIDRES        0.221142 Hz
AQ            2.2609921 sec
RG                 114
DW              69.000 usec
DE               6.50 usec
TE              296.3 K
D1            2.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               1H
P1              14.40 usec
PL1              1.80 dB
SFO1       400.1324008 MHz

F2 - Processing parameters
SI                16384
SF         400.1300078 MHz
WDW                EM
SSB     0
LB      0 Hz
GB      0
PC              1.00
```




```
Current Data Parameters
NAME      NSMVK Pyrazolinone
EXPNO              120
PROCNO               1

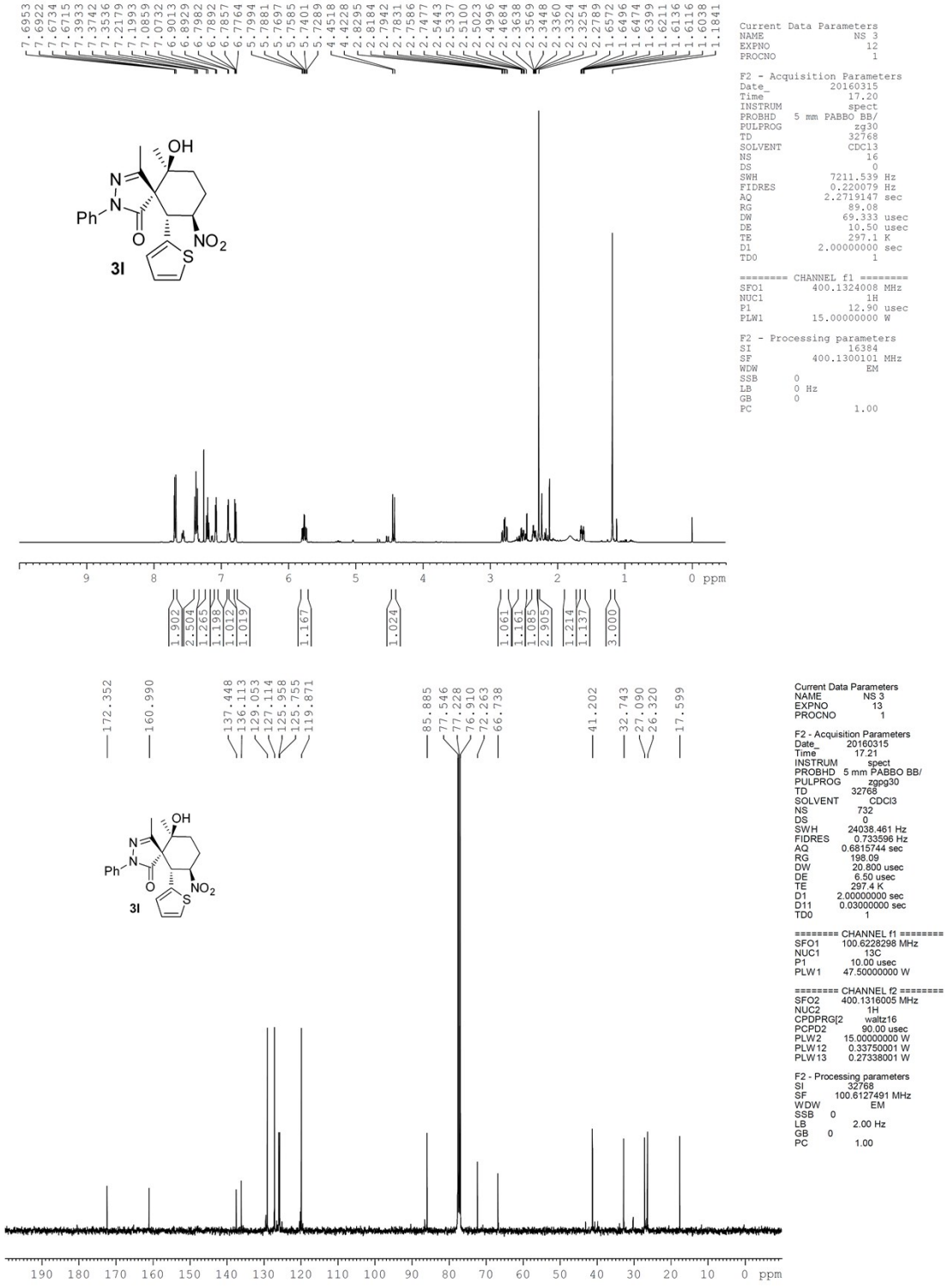
F2 - Acquisition Parameters
Date_          20151017
Time             22.25
INSTRUM          spect
PROBHD  5 mm BBO BB-1H
PULPROG         zgpg30
TD               32768
SOLVENT          CDCl3
NS                1741
DS                   0
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            0.6815744 sec
RG                8192
DW              20.800 usec
DE               6.50 usec
TE              296.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1              9.00 usec
PL1              7.00 dB
SFO1       100.6233325 MHz

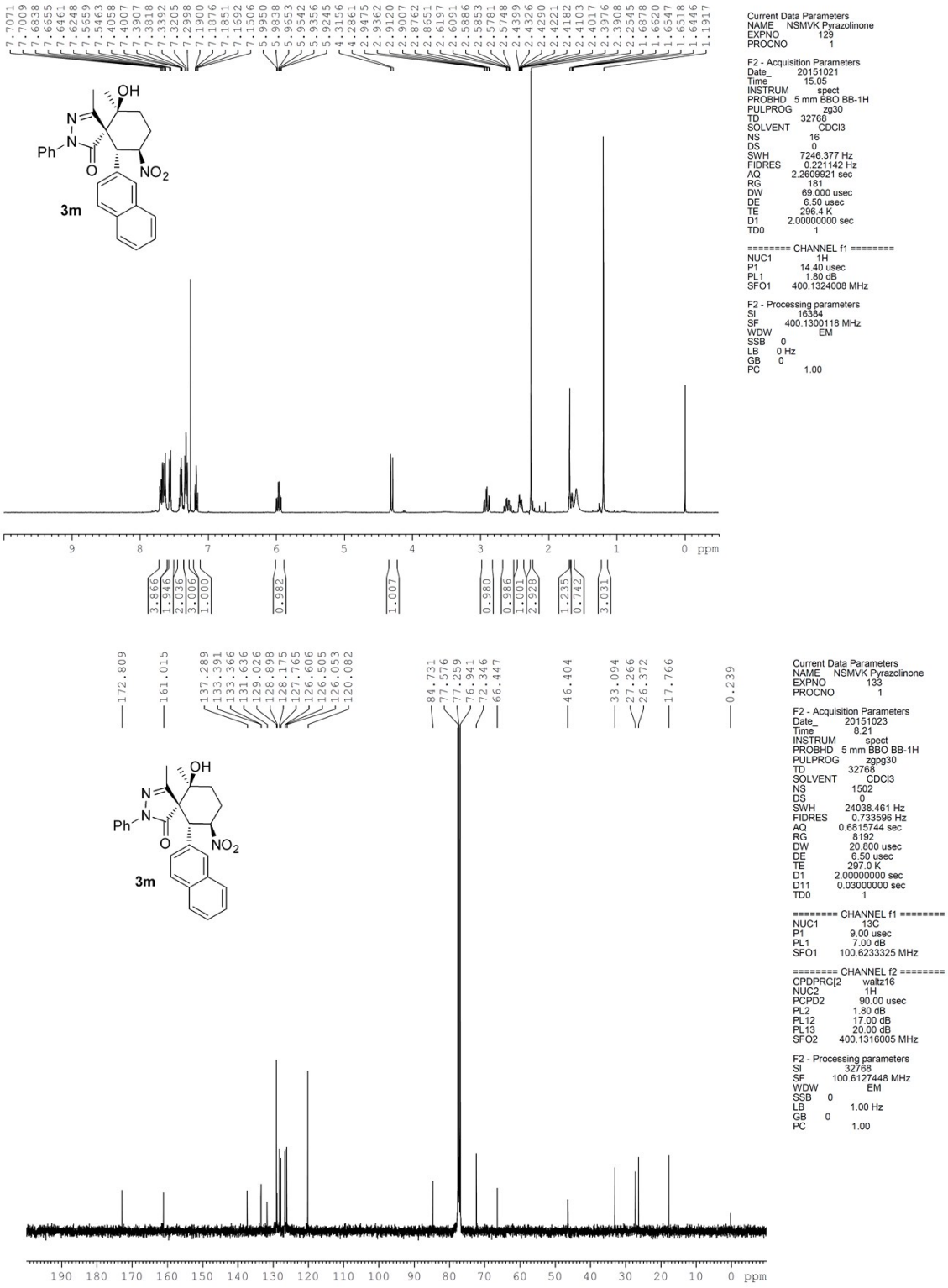
======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2               1H
PCPD2           90.00 usec
PL2              1.80 dB
PL12            17.00 dB
PL13            20.00 dB
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                32768
SF         100.6127486 MHz
WDW                EM
SSB     0
LB              1.00 Hz
GB      0
PC              1.00
```

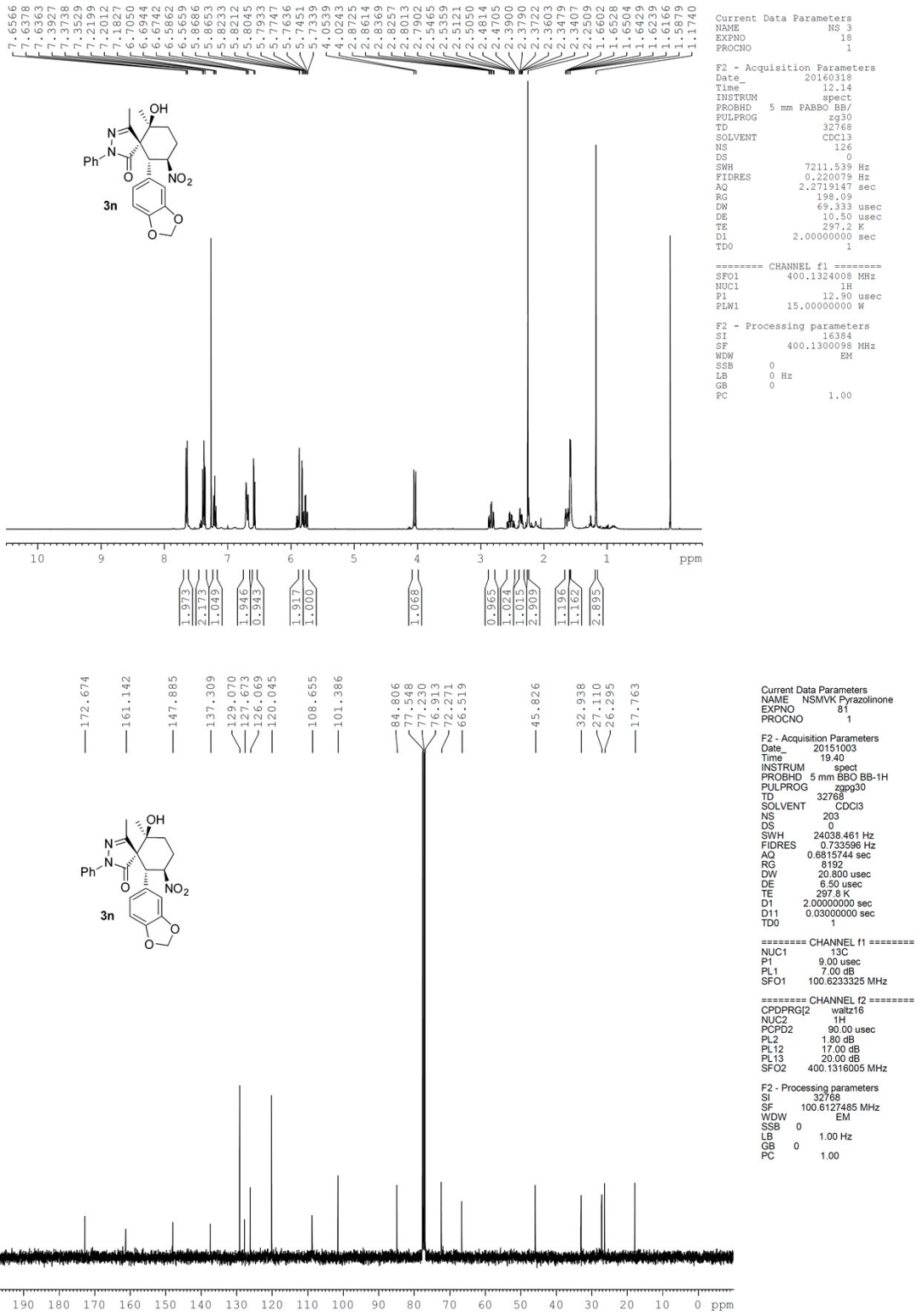
(5*R*,6*S*,9*R*,10*R*)-6-hydroxy-4,6-dimethyl-9-nitro-2-phenyl-10-(thiophen-2-yl)-2,3-diazaspiro[4.5]dec-3-en-1-one (3l):



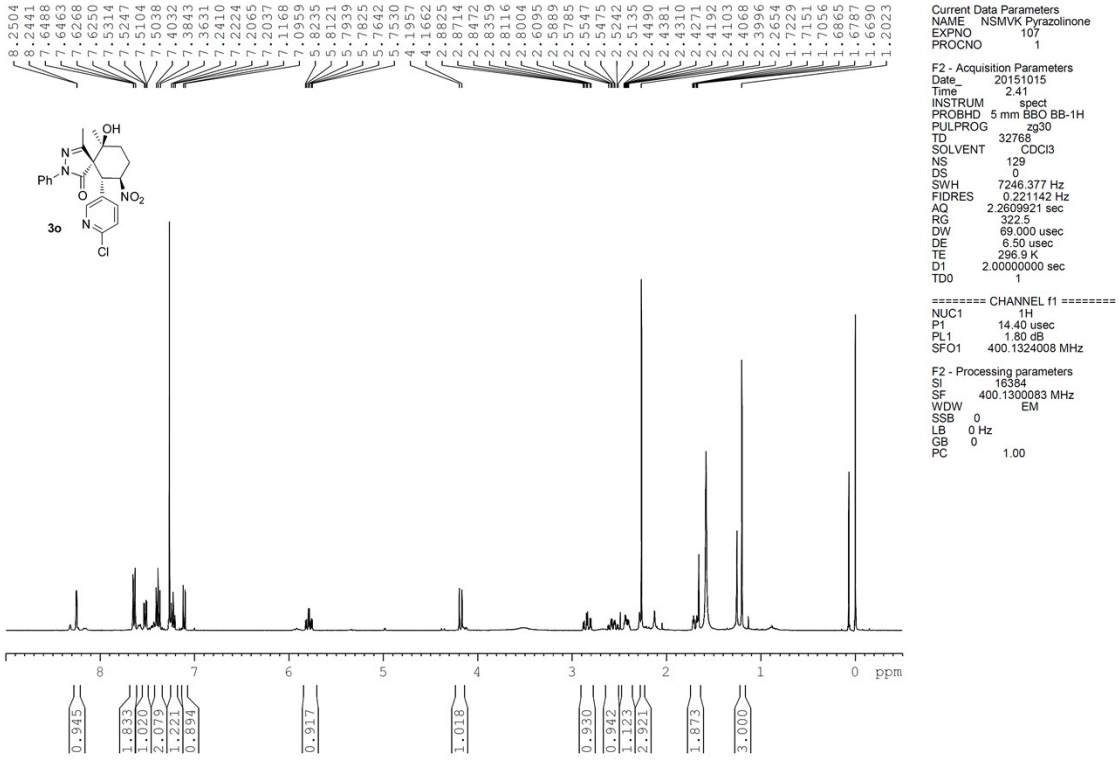
(5*R*,6*S*,9*R*,10*R*)-6-hydroxy-4,6-dimethyl-10-(naphthalen-2-yl)-9-nitro-2-phenyl-2,3-diazaspiro[4.5]dec-3-en-1-one (3m):



(5*R*,6*S*,9*R*,10*R*)-10-(benzo[d][1,3]dioxol-5-yl)-6-hydroxy-4,6-dimethyl-9-nitro-2-phenyl-2,3-diazaspiro[4.5]dec-3-en-1-one (3n):



(5*R*,6*S*,9*R*,10*R*)-10-(6-chloropyridin-3-yl)-6-hydroxy-4,6-dimethyl-9-nitro-2-phenyl-2,3-diazaspiro[4.5]dec-3-en-1-one (3o):

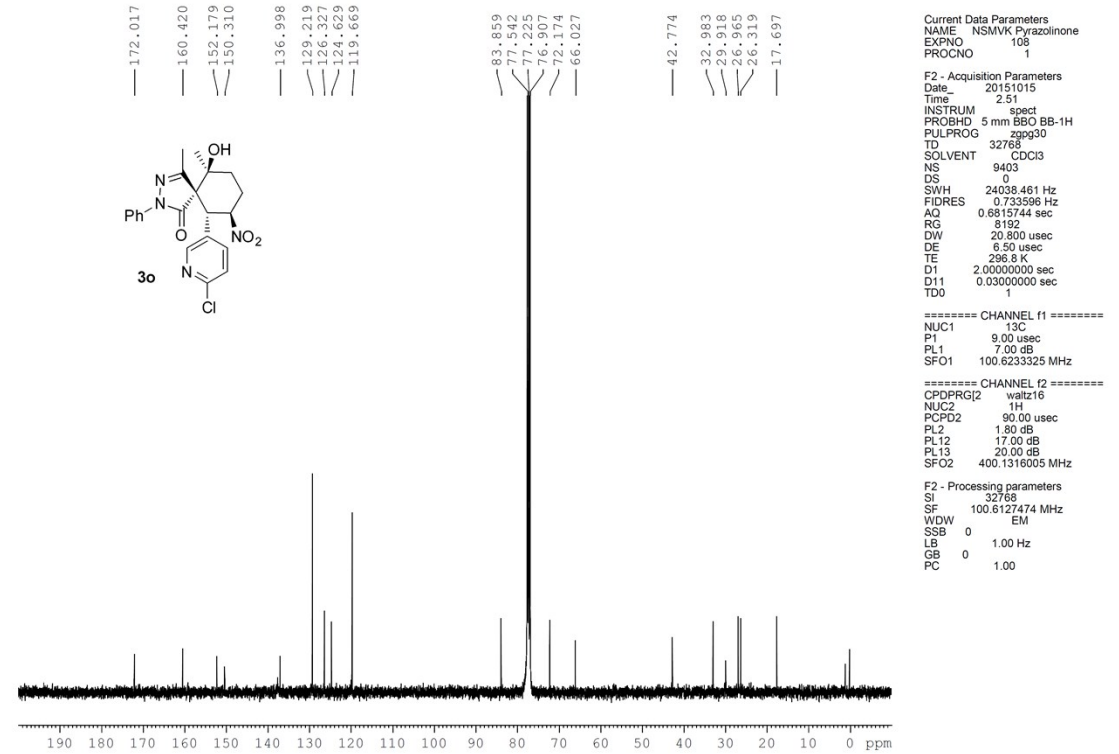


Current Data Parameters
NAME NSMVK Pyrazolinone
EXPNO 107
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151015
Time 2.41
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 129
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.26099921 sec
RG 322.5
DW 69.000 usec
DE 6.50 usec
TE 296.9 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 14.40 usec
PL1 1.80 dB
SFO1 400.1324008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300083 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00



Current Data Parameters
NAME NSMVK Pyrazolinone
EXPNO 108
PROCNO 1

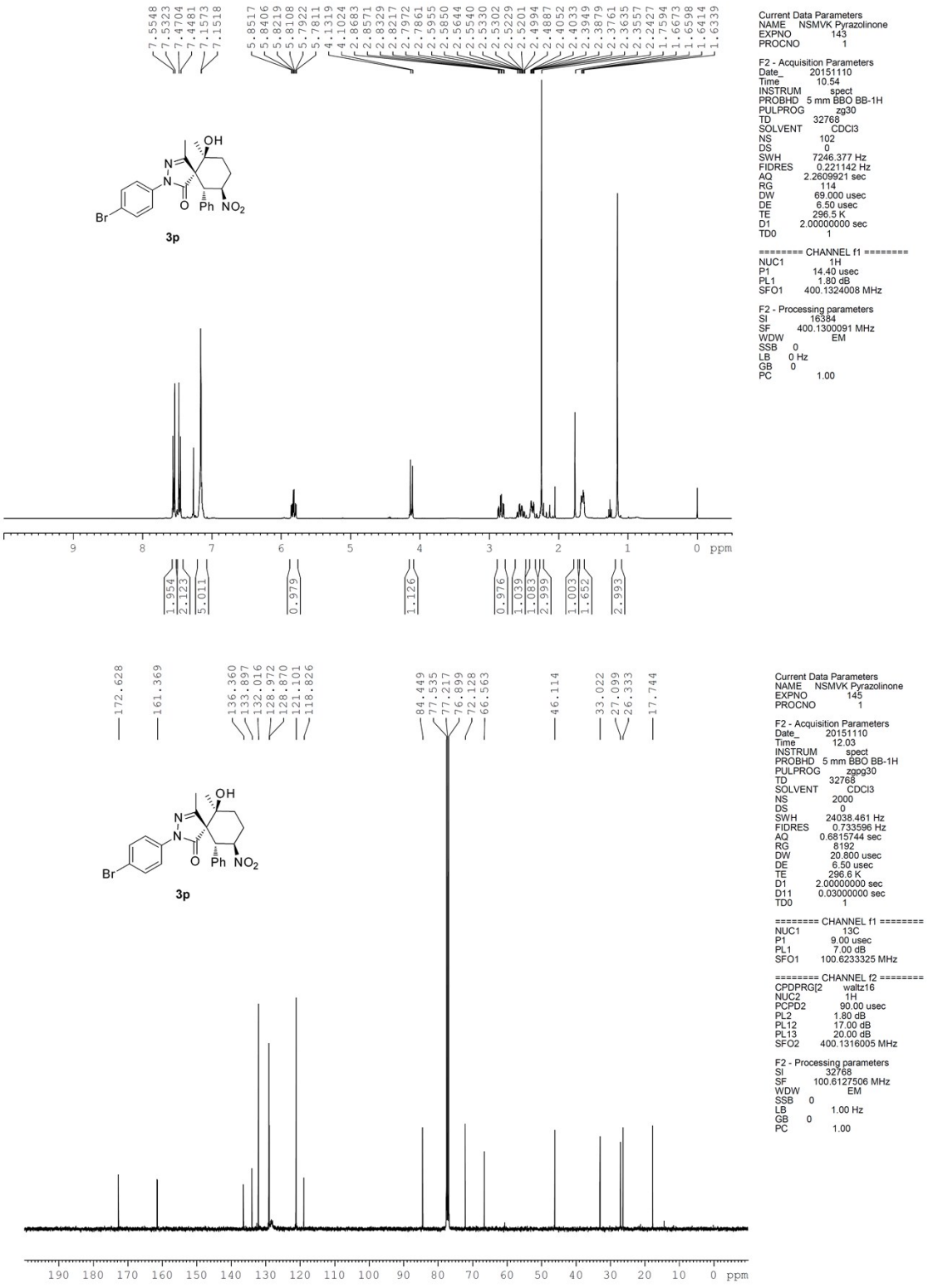
F2 - Acquisition Parameters
Date_ 20151015
Time 2.51
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 9403
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 8192
DW 20.800 usec
DE 6.50 usec
TE 296.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 7.00 dB
SFO1 100.6233325 MHz

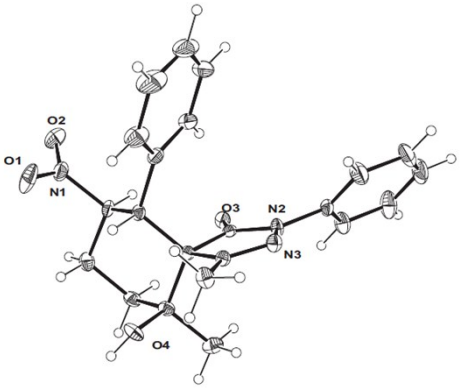
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.80 dB
PL12 17.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127474 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

(5*R*,6*S*,9*R*,10*R*)-2-(4-bromophenyl)-6-hydroxy-4,6-dimethyl-9-nitro-10-phenyl-2,3-diazaspiro[4.5]dec-3-en-1-one (3p):



X-ray crystallographic structure and crystal data for compound (3a): [CCDC: 1492783]



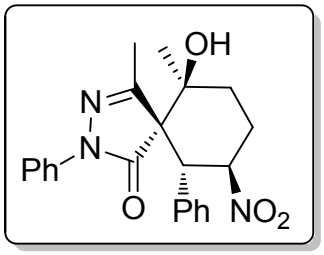


Table 1. Crystal data and structure refinement for a18203.

Identification code	a18203	
Empirical formula	C22 H23 N3 O4	
Formula weight	393.43	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P 21 21 21	
Unit cell dimensions	a = 8.2009(3) Å	α= 90°.
	b = 11.6802(4) Å	β= 90°.
	c = 21.3811(8) Å	γ = 90°.
Volume	2048.06(13) $Å^3$	
Z	4	
Density (calculated)	1.276 Mg/m^3	
Absorption coefficient	0.089 mm^{-1}	

F(000)	832
Crystal size	0.13 x 0.10 x 0.06 mm^3
Theta range for data collection	1.90 to 25.10°.
Index ranges	-9<=h<=9, -13<=k<=11, -25<=l<=22
Reflections collected	14052
Independent reflections	3569 [R(int) = 0.0556]
Completeness to theta = 25.10°	99.6 %
Absorption correction	multi-scan
Max. and min. transmission	0.9947 and 0.9885
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3569 / 0 / 263
Goodness-of-fit on F^2	0.968
Final R indices [I>2sigma(I)]	R1 = 0.0468, wR2 = 0.0791
R indices (all data)	R1 = 0.1164, wR2 = 0.0975
Absolute structure parameter	0.01
Extinction coefficient	0.0239(19)
Largest diff. peak and hole	0.169 and -0.161 $e.Å^{-3}$