Supporting Information for

Reactions of α-Haloacroleins with Azides: Highly Regioselective Synthesis of Formyl Triazoles

Dongsheng Zhang, Yingzhu Fan, Zhongliang Yan, Yi Nie, Xingquan Xiong, and Lizhu Gao*

College of Materials Science & Engineering, Huaqiao University, Xiamen 361021, China

E-mail: lizhugao@hqu.edu.cn

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1. General Information

Thin-layer chromatography (TLC) was performed on Merck silica gel 60 F254. Flash chromatography was performed using E. Merck silica gel (40-60 μm partical size). ^{1}H and ^{13}C NMR spectra were recorded on a Bruker AVIII-500M spectrometers at 500 and 126 MHz. Chemical shift values are reported in ppm from tetramethylsilane as the internal standard (TMS: δ 0 for ^{1}H and δ 77.16 for ^{13}C). Data are reported as follows: chemical shifts, integration, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, dq = doublet of quartets, m = multiplet), and coupling constants (Hz). Infrared spectra were recorded on Thermo Scientific Nicolet iS50. HRMS were recorded on Agilent 1290 UHPLC/ 6545 Q-TOF mass spectrometer. Commercial grade reagents and solvents were used without further purification except as indicated below. Data for the single crystal structure determination were collected with an Agilent Gemini/Xcalibur X-ray diffractometer equipped with a CCD area Atlas detector and a mirror monochromator by utilizing Cu-K α radiation ($\lambda = 1.5418$ Å).

2. Preparation of α-Bromoacroleins [1]

To a stirred solution of acroleins (23.1 mmol) in CH_2Cl_2 (35 mL) was added Br_2 (1.18 mL, 23.1 mmol) over 30 min at −78 °C under argon. After an additional 30 min at −78 °C, Et_3N (3.2 mL, 23.1 mmol) was added and the mixture was warmed to room temperature over 2 h. The reaction was then quenched by the addition of H_2O (40 mL), the layers were separated, and the aqueous layer was extracted with CH_2Cl_2 (1 × 40 mL). The combined organic layers were washed with a 9:1 mixture of brine and 1 M HCl (40 mL), dried (Na_2SO_4), filtered and concentrated at room temperature. The residue was purified by vacuum distillation or by silica gel chromatography to give the corresponding α-bromoacroleins.

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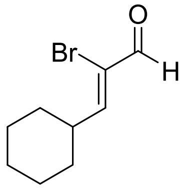
[1] R. H. Baker, S. W. Tinsley, D. Butler, B. Riegel, *J. Am. Chem. Soc.*, **1950**, *72*, 393.

2-Bromo-3-cyclohexylacrylaldehyde



A light yellow oil; *R_f* 0.20 (ethyl acetate/hexane = 1:20); ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.18 (s, 1H), 6.96 (d, *J* = 9.1 Hz, 1H), 2.87 – 2.72 (m, 1H), 1.88 – 1.67 (m, 5H), 1.44 – 1.31 (m, 2H), 1.32 – 1.19 (m, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 186.5, 160.0, 126.8, 41.2, 30.8, 25.8, 25.3 ppm; IR (neat) 2917, 2849, 1462, 1260, 1081, 1020, 800 cm^{-1}; HRMS (APCI) exact mass calcd. for $C_9H_{13}BrO$: m/z 217.0223 ([M + H]$^+$), found: m/z 217.0224 ([M + H]$^+$).

3. Preparation of Alkyl Azides [2]

To a solution of NaN_3 (0.715 g, 11 mmol) in DMSO (22 mL) was added alkyl bromide (10 mmol) at room temperature, then the reaction mixture was stirred at 60 °C until alkyl bromide had been consumed. The reaction was quenched with H_2O (50 mL) and stirred until it cooled to room temperature. The mixture was extracted with Et_2O (3 × 30 mL), and Et_2O extracts were washed with H_2O (2 × 50 mL) and brine (50 mL). The organic layer was dried over $MgSO_4$. The solvent was removed under reduced pressure and the residue was purified by short column chromatography on silica gel with hexane/dichloromethane (5:1) as the eluent to give the corresponding organic azide as a colorless oil.

4. General Procedure for Preparation of 4-Formyl-1,2,3-Triazoles

To a solution of α-bromoacrolein (0.20 mmol) in 200 μL of solvent was added organic azide (0.13 mmol) at room temperature. The reaction mixture was stirred at same temperature for 36 h, then triethyl amine (21μL, 0.15 mmol) was added. 5 Min later, solvent was removed under reduced pressure, and the residue was purified by silica gel chromatography to afford the desired

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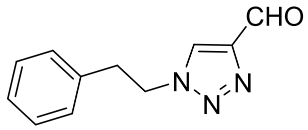
[2] S. Alvarez, M. Alvarez, *Synthesis*, **1997**, *4*, 413.

corresponding 4-Formyl-1,2,3- Triazoles.

5. Synthesis and Characterization of 4-Formyl-1,2,3-Triazoles



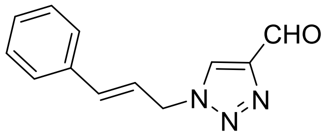
1-Phenethyl-1*H*-1,2,3-triazole-4-carbaldehyde (1)

The compound was prepared according to general procedure with α-bromoacrolein (0.2 mmol, 27.0 mg) and (2-azidoethyl) benzene (0.13 mmol, 19.1 mg) in 200 μL of DMSO/H_2O (7:3 vol/vol) over a course of 36 h at room temperature. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [95% yield (25.0 mg)] as a white solid (mp: 79-80 °C).

R_f 0.20 (ethyl acetate/hexane = 1:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.08 (s, 1H), 7.85 (s, 1H), 7.35–7.20 (m, 3H), 7.11–7.03 (m, 2H), 4.68 (t, *J* = 7.1 Hz, 2H), 3.25 (t, *J* = 7.1 Hz, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.0, 147.4, 136.3, 129.0, 128.6, 127.4, 125.7, 52.1, 36.4 ppm; IR (neat) 3118, 2951, 2855, 1694, 1533, 1459, 1163, 1049, 760, 703 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{11}H_{11}N_3O$: m/z 202.0975 ([M + H]$^+$), found: m/z 202.0977 ([M + H]$^+$).

Gram-scale reaction:

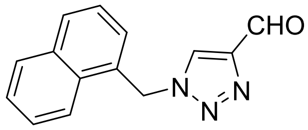
To a stirred solution of α-bromoacrolein (1.22 g, 9 mmol) in 10 mL of DMSO/H_2O (7:3 vol/vol) was added phenethyl azide (1.03 g, 7 mmol) dropwise at 0℃ over 20 min. Then the mixture was slowly warmed to room temperature and stirred for another 36 h. The reaction mixture was extracted with ether (3 × 20 mL), and combined organic phase was washed with brine (2× 5 mL). Then it was condensed under reduced pressure and the resulting solid was washed with hot hexane (3 mL). The product was got in 89% yield (1.256 g) and pure enough for analysis.



1-Cinnamyl-1*H*-1,2,3-triazole-4-carbaldehyde (2)

The compound was prepared according to general procedure with α-bromoacrolein (0.2 mmol, 27.0 mg) and (3-azidoprop-1-en-1-yl) benzene (0.13 mmol, 20.7 mg) in 200 μL of DMSO/H_2O (7:3 vol/vol) over a course of 36 h at room temperature. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [88% yield (24.5 mg)] as a white solid (mp: 74-76 °C).

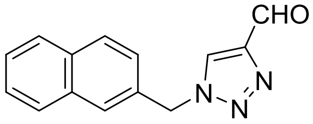
R_f 0.20 (ethyl acetate/hexane = 1:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.14 (s, 1H), 8.20 (s, 1H), 7.43 – 7.25 (m, 5H), 6.73 (d, *J* = 15.7, 1.3 Hz, 1H), 6.34 (dt, *J* = 15.8, 6.8 Hz, 1H), 5.20 (d, *J* = 6.8, 1.4 Hz, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.0, 148.0, 133.3, 133.2, 130.8, 129.5, 128.0, 128.0, 127.9, 127.1, 127.0, 125.4, 125.3, 54.8 ppm; IR (neat) 3024, 2926, 2838, 1691, 1533, 1239, 1166, 1048, 969, 748, 696 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{12}H_{11}N_3O$: m/z 214.0975 ([M + H]$^+$), found: m/z 214.0976 ([M + H]$^+$).



1-(Naphthalen-1-ylmethyl)-1*H*-1,2,3-triazole-4-carbaldehyde (3)

The compound was prepared according to general procedure with α-bromoacrolein (0.2 mmol, 27.0 mg) and 1-(azidomethyl)naphthalene (0.13 mmol, 23.8 mg) in 200 μL of DMSO/H_2O (7:3 vol/vol) over a course of 36 h at room temperature. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [90% yield (27.8 mg)] as a white solid (mp: 99-100 °C, decomposed).

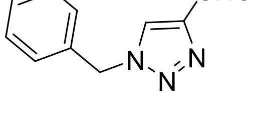
R$_f$ 0.25 (ethyl acetate/hexane = 1:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.04 (s, 1H), 7.96 – 7.85 (m, 3H), 7.84 (s, 1H), 7.56 – 7.45 (m, 4H), 6.01 (s, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.0, 147.9, 134.1, 131.0, 130.7, 129.2, 128.7, 128.5, 127.7, 126.7, 125.5, 125.3, 122.5, 52.6 ppm; IR (neat) 3067, 2922, 2842, 1686, 1526, 1249, 1169, 1040, 794, 779 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{14}H_{11}N_3O$: m/z 238.0975 ([M + H]$^+$), found: m/z 238.0974 ([M + H]$^+$).



1-(Naphthalen-2-ylmethyl)-1*H*-1,2,3-triazole-4-carbaldehyde (4)

The compound was prepared according to general procedure with α-bromoacrolein (0.2 mmol, 27.0 mg) and 2-(azidomethyl)naphthalene (0.13 mmol, 23.8 mg) in 200 μL of DMSO/H_2O (7:3 vol/vol) over a course of 36 h at room temperature. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [99% yield (30.4 mg)] as a white solid (mp: 86-88 °C).

R$_f$ 0.25 (ethyl acetate/hexane = 1:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.10 (s, 1H), 8.05 (s, 1H), 7.87 – 7.79 (m, 3H), 7.77 (s, 1H), 7.55 – 7.49 (m, 2H), 7.36 – 7.31 (m, 1H), 5.71 (s, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.0, 148.0, 133.3, 133.2, 130.8, 129.5, 128.0, 127.9, 127.9, 127.1, 127.0, 125.4, 125.3, 54.8 ppm; IR (neat) 3057, 2930, 2844, 1707, 1535, 1557, 1175, 1050, 865, 759 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{14}H_{11}N_3O$: m/z 238.0975 ([M + H]$^+$), found: m/z 238.0976 ([M + H]$^+$).



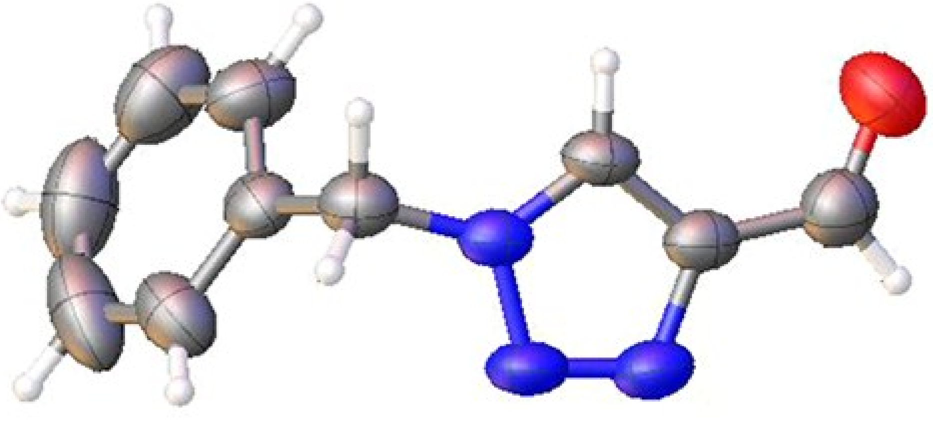
1-Benzyl-1*H*-1,2,3-triazole-4-carbaldehyde (5)

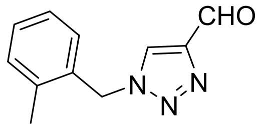
The compound was prepared according to general procedure with α-bromoacrolein (0.2 mmol, 27.0 mg) and (azidomethyl)benzene (0.13 mmol, 17.3 mg) in 200 μL of DMSO/H_2O (7:3 vol/vol) over a course of 36 h at room temperature. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [96% yield (23.3 mg)] as a colorless oil as a white solid (mp: 71-73 °C).

R_f 0.20 (ethyl acetate/hexane = 1:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.10 (s, 1H), 8.05 (s, 1H), 7.44 – 7.36 (m, 3H), 7.34 – 7.29 (m, 2H), 5.60 (s, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.0, 148.0, 133.5, 129.4, 129.3, 128.4, 125.3, 54.6 ppm; IR (neat) 3036, 2922, 2851, 1691, 1532, 1446, 1236, 1163, 1050, 765, 699 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{10}H_9N_3O$: m/z 188.0818 ([M + H]$^+$), found: m/z 188.0820 ([M + H]$^+$).

The configuration of the compound was assigned based on single-crystal X-ray analysis.

CCDC 1864100 contains the supplementary crystallographic data for this compound. These data can be obtained free of charge at www.ccdc.cam.ac.uk/conts/retrieving.html [or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (internat.) +44(1223)336-033, E-mail: deposit@ccdc.cam.ac.uk].

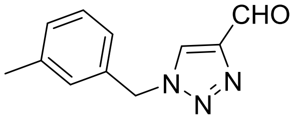




1-(2-Methylbenzyl)-1*H*-1,2,3-triazole-4-carbaldehyde (6)

The compound was prepared according to general procedure with α-bromoacrolein (0.2 mmol, 27.0 mg) and 1-(azidomethyl)-2-methylbenzene (0.13 mmol, 19.1 mg) in 200 μL of DMSO/H_2O (7:3 vol/vol) over a course of 36 h at room temperature. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [96% yield (25.1 mg)] as a white solid (mp: 58-60 °C).

R_f 0.25 (ethyl acetate/hexane = 1:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.10 (s, 1H), 7.89 (s, 1H), 7.40 – 7.12 (m, 4H), 5.60 (s, 2H), 2.28 (s, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.0, 147.8, 137.0, 131.3, 131.3, 129.8, 129.7, 126.9, 125.0, 77.4, 77.1, 76.8, 52.7, 18.9 ppm; IR (neat) 3038, 2930, 2853, 1693, 1522, 1451, 1355, 1119, 775, 747, 738cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{11}H_{11}N_3O$: m/z 202.0975 ($[M + H]^+$), found: m/z 202.0976 ($[M + H]^+$).

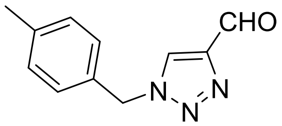


1-(3-Methylbenzyl)-1*H*-1,2,3-triazole-4-carbaldehyde (7)

The compound was prepared according to general procedure with α-bromoacrolein (0.2 mmol, 27.0 mg) and 1-(azidomethyl)-3-methylbenzene (0.13 mmol, 19.1 mg) in 200 μL of DMSO/H_2O (7:3 vol/vol) over a course of 36 h at room temperature. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [97% yield (25.4 mg)] as a white solid (mp:44-45 °C).

R_f 0.20 (ethyl acetate/hexane = 1:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.10 (s, 1H), 8.04 (s, 1H), 7.31 – 7.28 (m, 1H), 7.20 (d, *J* = 7.7 Hz, 1H), 7.11 (d, *J* = 8.0 Hz, 2H), 5.55 (s, 2H), 2.35 (s, 3H)

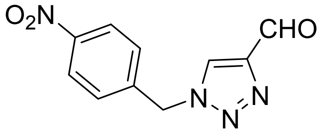
ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.1, 148.0, 139.3, 133.4, 130.1, 129.3, 129.1, 125.5, 125.3, 54.6, 21.3 ppm; IR (neat) 3028, 2922, 1686, 1608, 1531, 1344, 1234, 1162, 1048, 779, 728 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{11}H_{11}N_3O$: m/z 202.0975 ([M + H]$^+$), found: m/z 202.0975 ([M + H]$^+$).



1-(4-Methylbenzyl)-1*H*-1,2,3-triazole-4-carbaldehyde (8)

The compound was prepared according to general procedure with α-bromoacrolein (0.2 mmol, 27.0 mg) and 1-(azidomethyl)-4-methylbenzene (0.13 mmol,19.1 mg) in 200 μL of DMSO/H_2O (7:3 vol/vol) over a course of 36 h at room temperature. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [98% yield (25.7 mg)] as a white solid (mp: 66-67 °C).

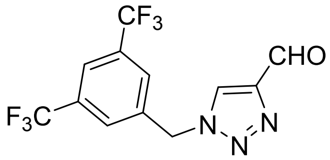
R$_f$ 0.20 (ethyl acetate/hexane = 1:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.10 (s, 1H), 8.01 (s, 1H), 7.20 (s, 4H), 5.55 (s, 2H), 2.36 (s, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.1, 148.0, 139.4, 130.4, 130.1, 128.5, 125.2, 54.4, 21.2 ppm; IR (neat) 3032, 2924, 2853, 1685, 1524, 1459, 1430, 1354, 1240, 1045, 772, 756 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{11}H_{11}N_3O$: m/z 224.0794 ([M + Na]$^+$), found: m/z 224.0796 ([M + Na]$^+$).



1-(4-Nitrobenzyl)-1*H*-1,2,3-triazole-4-carbaldehyde (9)

The compound was prepared according to general procedure with α-bromoacrolein (0.2 mmol, 27.0 mg) and 1-(azidomethyl)-4-nitrobenzene (0.13 mmol, 23.2 mg) in 200 μL of DMSO/H_2O (7:3 vol/vol) over a course of 36 h at room temperature. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [88% yield (26.6 mg)] as a light yellow solid (mp:124-126 °C).

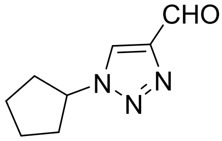
R_f 0.25 (ethyl acetate/hexane = 1:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.15 (s, 1H), 8.26 (d, J = 8.7 Hz, 2H), 8.15 (s, 1H), 7.48 (d, J = 8.7 Hz, 2H), 5.74 (s, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 184.9, 148.5, 148.4, 140.5, 129.1, 125.5, 124.6, 53.6 ppm; IR (neat) cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{10}H_8N_4O_3$: m/z 255.0489 ([M + Na]$^+$), found: m/z 255.0493 ([M + Na]$^+$).



1-(3,5-Bis(trifluoromethyl)benzyl)-1*H*-1,2,3-triazole-4-carbaldehyde (10)

The compound was prepared according to general procedure with α-bromoacrolein (0.2 mmol, 27.0 mg) and 1-(azidomethyl)-3,5-bis(trifluoromethyl)benzene (0.13 mmol, 35.0 mg) in 200 μL of DMSO/H_2O (7:3 vol/vol) over a course of 36 h at room temperature. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [84% yield (35.1 mg)] as a colorless oil as a white solid (mp: 105-106 °C).

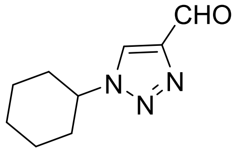
R_f 0.20 (ethyl acetate/hexane = 1:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.15 (s, 1H), 8.24 (s, 1H), 7.92 (s, 1H), 7.81 (s, 2H), 5.77 (s, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.0, 148.4, 136.2, 133.0 (q, J_{CF} = 34.0 Hz), 128.5 (q, J_{CF} = 3.8 Hz), 125.6, 123.4 (m, J_{CF} = 3.8 Hz), 122.9 (q, J_{CF} = 274.3 Hz), 53.5, 29.8 ppm; IR (neat) 3038, 2930, 2857, 1700, 1686, 1283, 1170, 1118, 784, 702 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{12}H_7F_6N_3O$: m/z 324.0566 ([M + H]$^+$), found: m/z 324.0570 ([M + H]$^+$).



1-Cyclopentyl-1*H*-1,2,3-triazole-4-carbaldehyde (11)

The compound was prepared according to general procedure with α-bromoacrolein (0.2 mmol, 27.0 mg) and azidocyclopentane (0.13 mmol, 14.4 mg) in 200 μL of DMSO/H_2O (7:3 vol/vol) over a course of 36 h at room temperature. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [90% yield (19.4 mg)] as a colorless oil.

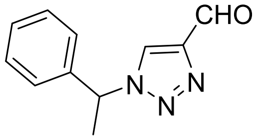
R_f 0.20 (ethyl acetate/hexane = 1:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.14 (s, 1H), 8.16 (s, 1H), 5.06 – 4.98 (m, 1H), 2.41 – 2.25 (m, 2H), 2.13 – 2.03 (m, 2H), 1.99 – 1.88 (m, 2H), 1.87 – 1.75 (m, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.3, 147.6, 123.9, 62.5, 33.5, 24.0 ppm; IR (neat) 2923, 2853, 1724, 1459, 1373, 1100 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_8H_{11}N_3O$: m/z 166.0975 ([M + H]$^+$), found: m/z 166.0976 ([M + H]$^+$).



1-Cyclohexyl-1*H*-1,2,3-triazole-4-carbaldehyde (12)

The compound was prepared according to general procedure with α-bromoacrolein (0.2 mmol, 27.0 mg) andazidocyclohexane (0.13 mmol, 16.3 mg) in 200 μL of DMSO/H_2O (7:3 vol/vol) over a course of 36 h at room temperature. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [82% yield (19.1 mg)] as a white solid (mp: 79-80 °C).

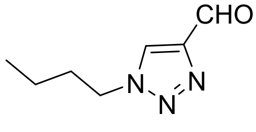
R_f 0.20 (ethyl acetate/hexane = 1:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.14 (s, 1H), 8.17 (s, 1H), 4.59 – 4.49 (m, 1H), 2.33 – 2.19 (m, 2H), 2.01 – 1.91 (m, 2H), 1.84 – 1.73 (m, 3H), 1.56 – 1.44 (m, 2H), 1.38 – 1.24 (m, 1H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.3, 147.5, 123.2, 60.7, 33.4, 25.0, 25.0 ppm; IR (neat) 3026, 2925, 2849, 1682, 1448, 1247, 1187, 1048, 779 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_9H_{13}N_3O$: m/z 180.1131 ([M + H]$^+$), found: m/z 180.1132 ([M + H]$^+$).



1-(1-Phenylethyl)-1*H*-1,2,3-triazole-4-carbaldehyde (13)

The compound was prepared according to general procedure with α-bromoacrolein (0.2 mmol, 27.0 mg) and (1-azidoethyl) benzene (0.13 mmol, 19.1 mg) in 200 μL of DMSO/H_2O (7:3 vol/vol) over a course of 36 h at room temperature. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [77% yield (20.2 mg)] as a colorless oil.

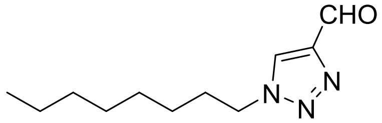
R_f 0.20 (ethyl acetate/hexane = 1:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.12 (s, 1H), 8.00 (s, 1H), 7.43 – 7.35 (m, 3H), 7.33 – 7.28 (m, 2H), 5.88 (q, *J* = 7.1 Hz, 1H), 2.03 (d, *J* = 7.1 Hz, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.3, 147.7, 138.7, 129.4, 129.2, 126.8, 124.2, 61.1, 21.3 ppm; IR (neat) 3097, 2990, 2875, 1691, 1532, 1443, 1190, 1146, 1048, 1018, 759, 696 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{11}H_{11}N_3O$: m/z 224.0994 ([M + Na]$^+$), found: m/z 224.0995 ([M + Na]$^+$).



1-Butyl-1*H*-1,2,3-triazole-4-carbaldehyde (14)

The compound was prepared according to general procedure with α-bromoacrolein (0.2 mmol, 27.0 mg) and 1-azidobutane (0.13 mmol, 12.9 mg) in 200 μL of DMSO/H_2O (7:3 vol/vol) over a course of 36 h at room temperature. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [88% yield (17.5 mg)] as a colorless oil.

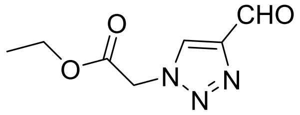
R_f 0.25 (ethyl acetate/hexane = 1:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.13 (s, 1H), 8.13 (s, 1H), 4.44 (t, J = 7.2 Hz, 2H), 1.97 – 1.89 (m, 2H), 1.42 – 1.32 (m, 2H), 0.97 (t, J = 7.4 Hz, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.3, 147.8, 125.1, 50.6, 32.1, 19.7, 13.4 ppm; IR (neat) 3047, 2963, 2876, 1695, 1531, 1465, 1237, 1140, 1042, 780 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_7H_{11}N_3O$: m/z 154.0975 ([M + H]$^+$), found: m/z 154.0977 ([M + H]$^+$).



1-Octyl-1*H*-1,2,3-triazole-4-carbaldehyde (15)

The compound was prepared according to general procedure with α-bromoacrolein (0.2 mmol, 27.0 mg) and 1-azidooctane (0.13 mmol, 20.2 mg) in 200 μL of DMSO/H_2O (7:3 vol/vol) over a course of 36 h at room temperature. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [82% yield (22.4 mg)] as a white solid (mp: 46-47 °C).

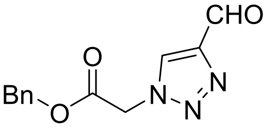
R_f 0.25 (ethyl acetate/hexane = 1:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.14 (s, 1H), 8.16 (s, 1H), 4.45 (t, J = 7.2 Hz, 2H), 2.01 – 1.89 (m, 2H), 1.40 – 1.18 (m, 10H), 0.87 (t, J = 6.8 Hz, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.2, 147.8, 125.2, 50.9, 31.7, 30.1, 29.0, 28.9, 26.4, 22.6, 14.1 ppm; IR (neat) 3042, 2917, 1851, 1685, 1470, 1438, 1354, 1248, 1046, 777 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{11}H_{11}N_3O$: m/z 210.1601 ([M + H]$^+$), found: m/z 210.1601 ([M + H]$^+$).



Ethyl 2-(4-formyl-1*H*-1,2,3-triazol-1-yl)acetate (16)

The compound was prepared according to general procedure with α-bromoacrolein (0.2 mmol, 27.0 mg) and ethyl 2-azidoacetate (0.13 mmol, 16.8 mg) in 200 μL of anhydrous DMSO over a course of 36 h at room temperature. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [86% yield (20.4 mg)] as a white solid (mp: 43-45 °C).

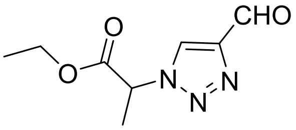
R_f 0.20 (ethyl acetate/hexane = 1:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.16 (s, 1H), 8.31 (s, 1H), 5.26 (s, 2H), 4.30 (q, J = 7.1 Hz, 2H), 1.32 (t, J = 7.1 Hz, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 184.9, 165.6, 148.1, 127.0, 63.0, 51.1, 29.8, 14.1 ppm; IR (neat) 3061, 2961, 2846, 1742, 1701, 1543, 1382, 1215, 1048, 1016, 795cm^{-1}; HRMS (ESI) exact mass calcd. for $C_7H_9N_3O_3$: m/z 184.0717 ($[M + H]^+$), found: m/z 184.0719 ($[M + H]^+$).



Benzyl 2-(4-formyl-1*H*-1,2,3-triazol-1-yl)acetate (17)

The compound was prepared according to general procedure with α-bromoacrolein (0.2 mmol, 27.0 mg) and benzyl 2-azidoacetate (0.13 mmol, 24.9 mg) in 200 μL of anhydrous DMSO over a course of 36 h at room temperature. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [75% yield (24.0 mg)] as a white solid (mp: 83-85 °C).

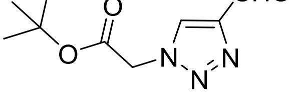
R_f 0.20 (ethyl acetate/hexane = 1:1); ^{1}H NMR (500 MHz, CDCl$_3$) δ 10.14 (s, 1H), 8.28 (s, 1H), 7.43 – 7.31 (m, 5H), 5.28 (s, 2H), 5.24 (s, 2H) ppm; ^{13}C NMR (126 MHz, CDCl$_3$) δ ^{13}C NMR (126 MHz, CDCl$_3$) δ ^{13}C NMR (126 MHz, CDCl$_3$) δ 184.9, 165.5, 148.1, 134.3, 129.1, 128.9, 128.8, 127.0, 68.6, 51.1 ppm; IR (neat) 3059, 2935, 2851, 1752, 1683, 1538, 1457, 1200, 1046, 731, 688 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{12}H_{11}N_3O_3$: m/z 246.0873 ([M + H]$^+$), found: m/z 246.0871 ([M + H]$^+$).



Ethyl 2-(4-formyl-1*H*-1,2,3-triazol-1-yl)propanoate (18)

The compound was prepared according to general procedure with α-bromoacrolein (0.2 mmol, 27.0 mg) and ethyl 2-azidopropanoate (0.13 mmol, 18.6 mg) in 200 μL of anhydrous DMSO over a course of 36 h at room temperature. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [82% yield (21.1 mg)] as a colorless oil.

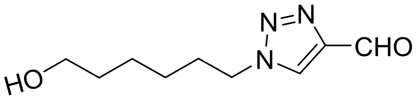
R_f 0.20 (ethyl acetate/hexane = 1:1); ^{1}H NMR (500 MHz, CDCl$_3$) δ 10.16 (s, 1H), 8.38 (s, 1H), 5.55 (q, *J* = 7.4 Hz, 1H), 4.26 (q, *J* = 7.1 Hz, 2H), 1.91 (d, *J* = 7.5 Hz, 3H), 1.29 (t, *J* = 7.2 Hz, 3H) ppm; ^{13}C NMR (126 MHz, CDCl$_3$) δ 185.0, 168.6, 147.9, 125.1, 62.8, 58.6, 18.3, 14.1 ppm; IR (neat) 3049, 2988, 2847, 1742, 1697, 1533, 1453, 1186, 1067, 1043, 771 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_8H_{11}N_3O_3$: m/z 220.0693 ([M + Na]$^+$), found: m/z 220.0695 ([M + Na]$^+$).



Tert-butyl 2-(4-formyl-1*H*-1,2,3-triazol-1-yl)acetate (19)

The compound was prepared according to general procedure with α-bromoacrolein (0.2 mmol, 27.0 mg) and *tert*-butyl 2-azidoacetate (0.13 mmol, 20.4 mg) in 200 μL of anhydrous DMSO over a course of 36 h at room temperature. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [60% yield (16.5 mg)] as a colorless oil.

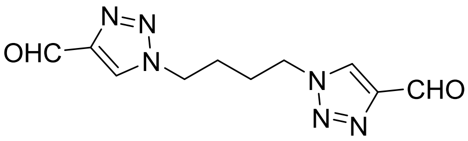
R_f 0.20 (ethyl acetate/hexane = 1:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.16 (s, 1H), 8.27 (s, 1H), 5.15 (s, 2H), 1.50 (s, 9H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.0, 164.5, 148.1, 126.9, 84.7, 51.8, 28.1 ppm; IR (neat) 2961, 2871, 1740, 1683, 1369, 1236, 1048, 768 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_9H_{13}N_3O_3$: m/z 212.1030 ([M + H]$^+$), found: m/z 212.1031 ([M + H]$^+$).



1-(6-Hydroxyhexyl)-1*H*-1,2,3-triazole-4-carbaldehyde (20)

The compound was prepared according to general procedure with α-bromoacrolein (0.2 mmol, 27.0 mg) and 6-azidohexan-1-ol (0.13 mmol, 18.6 mg) in 200 μL of DMSO/H_2O (7:3 vol/vol) over a course of 36 h at room temperature. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [91% yield (23.4 mg)] as a colorless oil.

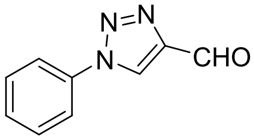
R_f 0.20 (ethyl acetate/hexane = 3:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.14 (s, 1H), 8.13 (s, 1H), 4.45 (t, J = 7.2 Hz, 2H), 3.64 (t, J = 6.4 Hz, 2H), 2.02 – 1.92 (m, 2H), 1.81 (s, 1H), 1.61 – 1.52 (m, 2H), 1.50 – 1.34 (m, 4H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.3, 147.9, 125.2, 62.6, 50.8, 32.4, 30.1, 26.2, 25.2 ppm; IR (neat) 3382, 2937, 2859, 1694, 1528, 1044, 780 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_9H_{15}N_3O_2$: m/z 198.1237 ([M + H]$^+$), found: m/z 198.1238 ([M + H]$^+$).



1,1'-(Butane-1,4-diyl)bis(1*H*-1,2,3-triazole-4-carbaldehyde) (21)

The compound was prepared according to general procedure with α-bromoacrolein (0.39 mmol, 52.6 mg) and 1,4-diazidobutane (0.13 mmol, 18.2 mg) in 300 μL of DMSO/H_2O (7:3 vol/vol) over a course of 36 h at room temperature. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [83% yield (26.9 mg)] as a white solid (mp: 169-170 °C).

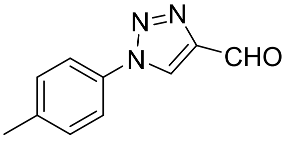
R_f 0.20 (ethyl acetate/hexane = 1:1); ^{1}H NMR (500 MHz, Acetone) δ 10.05 (s, 2H), 8.63 (s, 2H), 4.64 – 4.62 (m, 5H), 2.25 – 1.76 (m, 13H) ppm; ^{13}C NMR (126 MHz, Acetone) δ 206.1, 185.3, 127.4, 50.4, 27.7 ppm; IR (neat) 3110, 2953, 2842, 1683, 1435, 1347, 1242, 1044, 1011, 799 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{10}H_{12}N_6O_2$: m/z 271.0914 ([M + Na]$^+$), found: m/z 271.0916 ([M + Na]$^+$).



1-Phenyl-1*H*-1,2,3-triazole-4-carbaldehyde (22)

The compound was prepared according to general procedure with α-bromoacrolein (0.26 mmol, 35.1 mg) and phenyl azide (0.13 mmol, 15.5 mg) in 200 μL of DMSO/H_2O (7:3 vol/vol) over a course of 48 h at 40℃. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [80% yield (18.0 mg)] as a light yellow solid (mp: 89-91 °C).

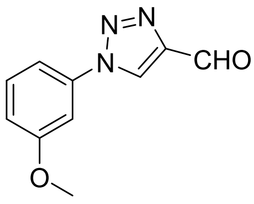
R_f 0.20 (ethyl acetate/hexane = 1:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.24 (s, 1H), 8.54 (s, 1H), 7.81 – 7.73 (m, 2H), 7.63 – 7.56 (m, 2H), 7.56 – 7.50 (m, 1H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.2, 148.3, 136.3, 130.2, 130.0, 123.2, 121.0 ppm; IR (neat) 3135, 2922, 2841, 1686, 1528, 1500, 1210, 758 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_9H_7N_3O$: m/z 174.0662 ([M + H]$^+$), found: m/z 174.0633 ([M + H]$^+$).



1-(*p*-tolyl)-1H-1,2,3-triazole-4-carbaldehyde (23)

The compound was prepared according to general procedure with α-bromoacrolein (0.26 mmol, 35.1 mg) and *p*-tolyl azide (0.13 mmol, 17.3 mg) in 200 μL of DMSO/H_2O (7:3 vol/vol) over a course of 48 h at 40℃. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [76% yield (18.6 mg)] as a white solid (mp: 104-105 °C).

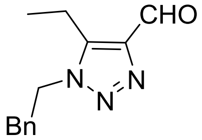
R_f 0.20 (ethyl acetate/hexane = 1:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.22 (s, 1H), 8.51 (s, 1H), 7.64 (d, *J* = 8.4 Hz, 2H), 7.37 (d, *J* = 8.1 Hz, 2H), 2.45 (s, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.2, 148.2, 140.3, 134.0, 130.7, 123.2, 120.9, 21.3 ppm; IR (neat) 3098, 2922, 2837, 1703, 1513, 1254, 1206, 1050, 987, 816, 770 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{10}H_9N_3O$: m/z 188.0818 ([M + H]$^+$), found: m/z 188.0819 ([M + H]$^+$).



1-(3-Methoxyphenyl)-1*H*-1,2,3-triazole-4-carbaldehyde (24)

The compound was prepared according to general procedure with α-bromoacrolein (0.26 mmol, 35.1 mg) and *m*-methoxy benzyl azide (0.13 mmol, 19.4 mg) in 200 μL of DMSO/H_2O (7:3 vol/vol) over a course of 48 h at 40℃. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [81% yield (21.5 mg)] as a light yellow solid (mp: 95-96 °C).

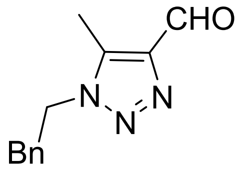
R_f 0.20 (ethyl acetate/hexane = 1:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.23 (s, 1H), 8.55 (s, 1H), 7.51 – 7.43 (m, 1H), 7.39 – 7.34 (m, 1H), 7.32 – 7.25 (m, 1H), 7.08 – 7.02 (m, 1H), 3.91 (s, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.2, 160.8, 148.1, 137.2, 131.0, 123.4, 115.7, 112.8, 106.9, 55.8 ppm; IR (neat) 3094, 2920, 1689, 1598, 1504, 1254, 1019, 774 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{10}H_9N_3O_2$: m/z 204.0768 ($[M + H]^+$), found: m/z 204.0769 ($[M + H]^+$).



5-Ethyl-1-phenethyl-1*H*-1,2,3-triazole-4-carbaldehyde (25)

The compound was prepared according to general procedure with 2-bromopent-2-enal (0.26 mmol, 42.4 mg) and (2-azidoethyl) benzene (0.13 mmol, 19.1 mg) in 200 μL of $CHCl_3$ over a course of 48 h at room temperature. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [80% yield (23.8 mg)] as a colorless oil.

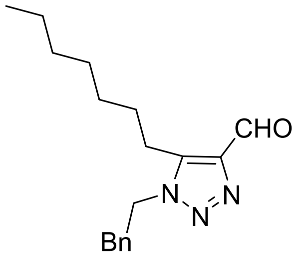
R_f 0.20 (ethyl acetate/hexane = 1:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.16 (s, 1H), 7.33 – 7.17 (m, 3H), 7.09 – 6.97 (m, 2H), 4.49 (t, J = 7.1 Hz, 2H), 3.27 (t, J = 7.1 Hz, 2H), 2.66 (q, J = 7.6 Hz, 2H), 0.99 (t, J = 7.6 Hz, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 186.04, 143.14, 142.78, 136.67, 128.93, 128.68, 127.31, 48.97, 36.52, 16.23, 12.51 ppm; IR (neat) 3030, 2936, 2832, 1690, 1556, 1473, 1454, 1262, 751, 699 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{13}H_{15}N_3O$: m/z 230.1288 ([M + H]$^+$), found: m/z 230.1288 ([M + H]$^+$).



5-methyl-1-phenethyl-1H-1,2,3-triazole-4-carbaldehyde (26)

The compound was prepared according to general procedure with 2-bromobut-2-enal (0.26 mmol, 38.7 mg) and (2-azidoethyl) benzene (0.13 mmol, 19.1 mg) in 200 μL of $CHCl_3$ over a course of 48 h at room temperature. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [78% yield (21.8 mg)] as a colorless oil.

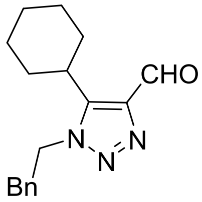
R$_f$ 0.20 (ethyl acetate/hexane = 1:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.16 (s, 1H), 7.37 – 7.16 (m, 3H), 7.09 – 6.93 (m, 2H), 4.49 (t, *J* = 6.8 Hz, 2H), 3.22 (t, *J* = 6.9 Hz, 2H), 2.12 (s, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ ^{13}C NMR (126 MHz, $CDCl_3$) δ 186.5, 143.6, 137.6, 136.7, 129.1, 128.8, 127.5, 49.2, 36.5, 8.3 ppm; IR (neat) 2957, 2924, 2852, 1683, 1566, 1456, 1273, 1133, 723, 697 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{11}H_{11}N_3O$: m/z 202.0975 ([M + H]$^+$), found: m/z 202.0977 ([M + H]$^+$).



5-*H*eptyl-1-phenethyl-1*H*-1,2,3-triazole-4-carbaldehyde (27)

The compound was prepared according to general procedure with 2-bromodec-2-enal (0.26 mmol, 60.6 mg) and (2-azidoethyl) benzene (0.13 mmol, 19.1 mg) in 200 µL of $CHCl_3$ over a course of 72 h at room temperature. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [62% yield (24.1 mg)] as a light yellow solid (mp: 51-52 °C).

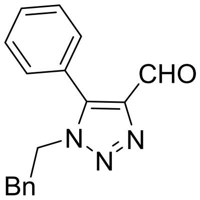
R_f 0.20 (ethyl acetate/hexane = 1:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.15 (s, 1H), 7.26 (d, J = 7.4 Hz, 2H), 7.12 – 6.97 (m, 3H), 4.47 (t, J = 7.2 Hz, 2H), 3.27 (t, J = 7.2 Hz, 2H), 2.68 – 2.54 (m, 2H), 1.42 – 1.12 (m, 10H), 0.87 (t, J = 7.0 Hz, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 186.0, 143.4, 141.7, 136.7, 128.9, 128.7, 127.3, 49.1, 36.5, 31.6, 29.2, 28.7, 28.2, 22.6, 22.5, 14.0 ppm; IR (neat) 3028, 2922, 2851, 1686, 1557, 1467, 1453, 1249, 749, 697 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{18}H_{25}N_3O$: m/z 300.2070 ($[M + H]^+$), found: m/z 300.2071 ($[M + H]^+$).



5-Cyclohexyl-1-phenethyl-1*H*-1,2,3-triazole-4-carbaldehyde (28)

The compound was prepared according to general procedure with 2-bromo-3-cyclohexylacrylaldehyde (0.26 mmol, 56.4 mg) and (2-azidoethyl) benzene (0.13 mmol, 19.1 mg) in 200 µL of $CHCl_3$ over a course of 72 h at room temperature. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [60% yield (22.1 mg)] as a white solid (mp: 71-73 °C).

R_f 0.20 (ethyl acetate/hexane = 1:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.15 (s, 1H), 7.34 – 7.20 (m, 3H), 7.09 – 7.04 (m, 2H), 4.57 (t, J = 7.1 Hz, 2H), 3.23 (t, J = 7.0 Hz, 2H), 2.59 – 2.49 (m, 1H), 1.92 – 1.80 (m, 2H), 1.79 – 1.65 (m, 3H), 1.38 – 1.30 (m, 1H), 1.20 – 1.13 (m, 4H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.9, 145.5, 143.7, 136.7, 129.1, 128.8, 127.4, 49.8, 37.0, 35.3, 29.2, 26.4, 25.2 ppm; IR (neat) 3059, 2924, 2851, 1686, 1536, 1461, 1448, 1253, 814, 697 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{17}H_{21}N_3O$: m/z 284.1757 ([M + H]$^+$), found: m/z 284.1759 ([M + H]$^+$).

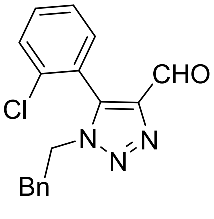


1-Phenethyl-5-phenyl-1*H*-1,2,3-triazole-4-carbaldehyde (29)

The compound was prepared according to general procedure with 2-bromo-3-phenylacrylaldehyde (0.26 mmol, 54.9 mg) and (2-azidoethyl) benzene (0.13 mmol, 19.1 mg) in 200 μL of $CHCl_3$ over a course of 48 h at 40 ℃. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [80% yield (28.8 mg)] as a white solid (mp: 60-61 °C).

R

R_f 0.20 (ethyl acetate/hexane = 1:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.04 (s, 1H), 7.52 – 7.46 (m, 1H), 7.45 – 7.39 (m, 2H), 7.23 – 7.16 (m, 3H), 7.02 – 6.96 (m, 2H), 6.91 – 6.85 (m, 2H), 4.48 (t, J = 7.1 Hz, 2H), 3.19 (t, J = 7.1 Hz, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 184.37, 143.27, 141.02, 136.51, 130.41, 129.42, 128.89, 128.83, 128.68, 127.18, 124.67, 77.16, 49.40, 36.25 ppm; IR (neat) 3061, 2922, 2855, 1684, 1488, 1450, 1434, 1236, 758, 696 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{17}H_{15}N_3O$: m/z 278.1288 ([M + H]$^+$), found: m/z 278.1290 ([M + H]$^+$).

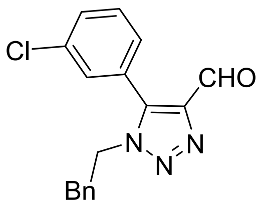


5-(2-Chlorophenyl)-1-phenethyl-1*H*-1,2,3-triazole-4-carbaldehyde (30)

The compound was prepared according to general procedure with 2-bromo-3-(2-

chlorophenyl)acrylaldehyde (0.26 mmol, 63.8 mg) and (2-azidoethyl) benzene (0.13 mmol, 19.1 mg) in 200 μL of $CHCl_3$ over a course of 48 h at 40 ℃. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [66% yield (26.7 mg)] a colorless oil.

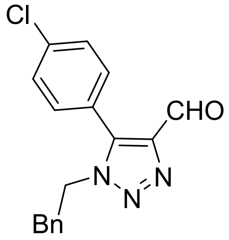
R_f 0.25 (ethyl acetate/hexane = 1:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.06 (t, J = 1.2 Hz, 1H), 7.54 – 7.48 (m, 1H), 7.48 – 7.41 (m, 1H), 7.29 – 7.15 (m, 4H), 6.92 – 6.82 (m, 2H), 6.66 – 6.58 (m, 1H), 4.64 – 4.51 (m, 1H), 4.23 – 4.12 (m, 1H), 3.29 – 3.20 (m, 1H), 3.18 – 3.05 (m, 1H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 184.3, 144.0, 137.7, 136.5, 133.6, 131.9, 131.7, 130.0, 128.9, 128.7, 127.2, 124.4, 49.7, 36.1 ppm; IR (neat) 3026, 2930, 1697, 1608, 1561, 1471, 1455, 1432, 1083, 730, 698 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{17}H_{14}ClN_3O$: m/z 312.0898 ([M + H]$^+$), found: m/z 312.0901 ([M + H]$^+$).



5-(3-Chlorophenyl)-1-phenethyl-1*H*-1,2,3-triazole-4-carbaldehyde (31)

The compound was prepared according to general procedure with 2-bromo-3-(3-chlorophenyl)acrylaldehyde (0.26 mmol, 63.8 mg) and (2-azidoethyl) benzene (0.13 mmol, 19.1 mg) in 200 μL of $CHCl_3$ over a course of 72 h at 40 ℃. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [81% yield (32.8 mg)] as a colorless oil.

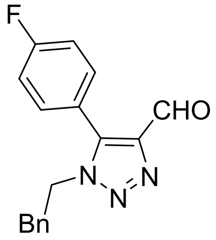
R_f 0.20 (ethyl acetate/hexane = 1:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.05 (s, 1H), 7.45 – 7.41 (m, 1H), 7.33 (t, J = 7.9 Hz, 1H), 7.25 – 7.17 (m, 3H), 6.87 – 6.80 (m, 3H), 6.75 (t, J = 1.9 Hz, 1H), 4.47 (t, J = 6.8 Hz, 2H), 3.21 (t, J = 6.7 Hz, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ ^{13}C NMR (126 MHz, $CDCl_3$) δ 184.4, 143.3, 139.4, 136.3, 134.8, 130.5, 130.1, 129.4, 128.9, 128.7, 127.6, 127.3, 126.4, 49.6, 36.3 ppm; IR (neat) 3026, 2959, 2847, 1697, 1555, 1455, 1298, 825, 730, 698 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{17}H_{14}ClN_3O$: m/z 312.0898 ([M + H]$^+$), found: m/z 312.0897 ([M + H]$^+$).



5-(4-Chlorophenyl)-1-phenethyl-1*H*-1,2,3-triazole-4-carbaldehyde (32)

The compound was prepared according to general procedure with 3-bromo-3-(4-chlorophenyl)acrylaldehyde (0.26 mmol, 63.8 mg) and (2-azidoethyl) benzene (0.13 mmol, 19.1 mg) in 200 μL of $CHCl_3$ over a course of 72 h at 40 ℃. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [82% yield (33.2 mg)] as a white solid (mp: 135-136 °C, decomposed).

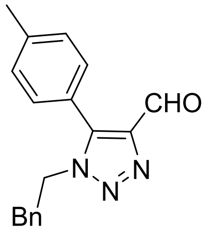
R$_f$ 0.20 (ethyl acetate/hexane = 1:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.06 (s, 1H), 7.36 (d, *J* = 8.4 Hz, 1H), 7.26 – 7.14 (m, 4H), 6.96 – 6.74 (m, 4H), 4.47 (t, *J* = 6.8 Hz, 2H), 3.21 (t, *J* = 6.8 Hz, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 184.55, 143.27, 139.69, 136.76, 136.45, 130.80, 129.18, 128.91, 128.72, 127.30, 123.09, 77.41, 77.16, 76.91, 49.50, 36.29 ppm; IR (neat) 3034, 2928, 2853, 1690, 1604, 1487, 1453, 1259, 1234, 1091, 839, 751, 703 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{17}H_{14}ClN_3O$: m/z 312.0898 ([M + H]$^+$), found: m/z 312.0900 ([M + H]$^+$).



5-(4-Fluorophenyl)-1-phenethyl-1*H*-1,2,3-triazole-4-carbaldehyde (33)

The compound was prepared according to general procedure with 3-bromo-3-(4-fluorophenyl)acrylaldehyde (0.26 mmol, 59.5 mg) and (2-azidoethyl) benzene (0.13 mmol, 19.1 mg) in 200 μL of $CHCl_3$ over a course of 72 h at 40 ℃. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [61% yield (23.4 mg)] as a light yellow solid (mp: 96-98 ^oC).

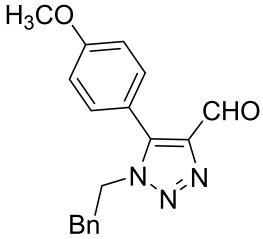
R_f 0.25 (ethyl acetate/hexane = 1:1); ^1H NMR (500 MHz, $CDCl_3$) δ 10.08 (s, 1H), 7.26 – 7.18 (m, 3H), 7.13 – 7.06 (m, 2H), 6.94 – 6.90 (m, 2H), 6.89 – 6.86 (m, 2H), 4.48 (t, J = 6.9 Hz, 2H), 3.22 (t, J = 6.9 Hz, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 184.6, 163.8 (d, J_{CF} = 252 Hz), 143.3, 139.9, 136.5, 131.6 (d, J_{CF} = 8.8 Hz), 128.9, 128.7, 127.3, 120.7 (d, J_{CF} = 2.5 Hz), 116.2 (d, J_{CF} = 22.7 Hz), 49.5, 36.3 ppm; IR (neat) 3034, 2924, 2853, 1690, 1604, 1499, 1453, 1221, 1160, 842, 755, 704 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{17}H_{14}FN_3O$: m/z 296.1194 ($[M + H]^+$), found: m/z 296.1194 ($[M + H]^+$).



1-Phenethyl-5-(p-tolyl)-1*H*-1,2,3-triazole-4-carbaldehyde (34)

The compound was prepared according to general procedure with 3-bromo-3-(p-tolyl)acrylaldehyde (0.26 mmol, 58.5 mg) and (2-azidoethyl) benzene (0.13 mmol, 19.1 mg) in 200 μL of $CHCl_3$ over a course of 72 h at room temperature. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [76% yield (28.8 mg)] as a white solid (mp: 90-92 ^oC).

R_f 0.20 (ethyl acetate/hexane = 1:1); ^1H NMR (500 MHz, $CDCl_3$) δ 10.04 (s, 1H), 7.27 – 7.18 (m, 5H), 6.94 – 6.85 (m, 4H), 4.48 (t, *J* = 7.2 Hz, 2H), 3.19 (t, *J* = 7.2 Hz, 2H), 2.41 (s, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 184.4, 143.3, 141.2, 140.8, 136.6, 129.6, 129.3, 128.8, 128.7, 127.2, 121.6, 49.3, 36.2, 21.5 ppm; IR (neat) 3030, 2922, 2859, 1691, 1502, 1450, 1435, 1300, 1236, 832, 752, 701 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{18}H_{17}N_3O$: m/z 292.1444 ([M + H]$^+$), found: m/z 292.1446 ([M + H]$^+$).



5-(4-Methoxyphenyl)-1-phenethyl-1*H*-1,2,3-triazole-4-carbaldehyde (35)

The compound was prepared according to general procedure with 2-bromo-3-cyclohexylacrylaldehyde (0.26 mmol, 62.7 mg) and (2-azidoethyl) benzene (0.13 mmol, 19.1 mg) in 200 µL of $CHCl_3$ over a course of 48 h at 50 ℃. The crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [44% yield (17.6 mg)] as a colorless oil.

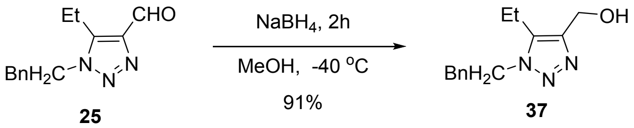
R_f 0.25 (ethyl acetate/hexane = 1:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.06 (s, 1H), 7.24 – 7.19 (m, 3H), 7.00 – 6.88 (m, 6H), 4.49 (t, *J* = 7.1 Hz, 2H), 3.86 (s, 3H), 3.20 (t, *J* = 7.1 Hz, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 184.7, 161.2, 143.3, 141.0, 136.7, 131.0, 128.9, 128.8, 127.3, 116.5, 114.5, 55.5, 49.4, 36.3 ppm; IR (neat) 3030, 2936, 1696, 1611, 1500, 1455, 1295, 1251, 1177, 1024, 839, 698 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{18}H_{17}N_3O_2$: m/z 303.1213 ([M + Na$^+$), found: m/z 303.1213 ([M + Na]$^+$).

6. Synthetic Transformations of 4-Formyl-1,2,3-Triazole

Gram-scale synthesis of compound **25**.

To a stirred solution of α-brom-β-ethyl acrolein (1.63 g, 10 mmol) in 10 mL of $CHCl_3$, was added phenethyl azide (1.03 g, 7 mmol) dropwise at RT in room temperature water bath over a course of 20 min. The mixture was slowly warmed to room temperature and stirred for another 48 h, then Et_3N (1.11 mL, 8 mmol) was added slowly. Five min later, reaction mixture was condensed under reduced pressure and crude material was purified by column chromatography on silica gel (eluting with ethyl acetate/hexane = 1:1) to give the desired product [79% yield (1.268 g)] as a colorless oil.

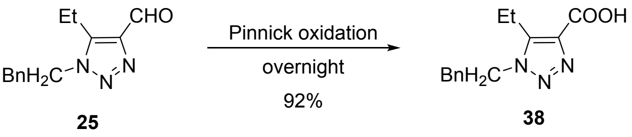
(a) Preparation of (5-ethyl-1-phenethyl-1*H*-1,2,3-triazol-4-yl)methanol (37)



To a stirred solution of compound **25** (22.93 mg, 0.1 mmol) in methanol (2 mL) was added $NaBH_4$ (5.7 mg, 0.15 mmol) at -40 °C. The reaction mixture was warmed to 0 °C and stirred for 1 h. The reaction solution was then quenched with aqueous NH_4Cl and extracted with CH_2Cl_2. The organic layer was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel to give the title alcohol **37** as a colorless oil [95% yield (21.9 mg)] a colorless oil.

R_f 0.20 (ethyl acetate/hexane = 1:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.32 – 7.20 (m, 3H), 7.11 – 7.05 (m, 2H), 4.70 (s, 2H), 4.41 (t, J = 7.5 Hz, 2H), 3.31 - 3.17 (m, 3H), 2.48 (q, J = 7.6 Hz, 2H), 1.07 (t, J = 7.6 Hz, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 143.8, 137.4, 136.5, 128.9, 128.8, 127.2, 55.9, 49.3, 36.9, 15.8, 13.7 ppm; IR (neat) 3318, 3030, 2934, 1604, 1495, 1454, 1232, 1016, 751, 699 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{13}H_{17}N_3O$: m/z 232.1444 ($[M + H]^+$), found: m/z 232.1444 ($[M + H]^+$).

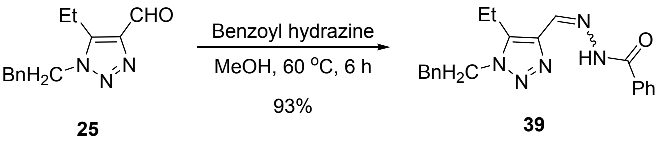
(b) Preparation of 5-ethyl-1-phenethyl-1*H*-1,2,3-triazole-4-carboxylic acid (38)



A mixture of compound **25** (50.0 mg, 0.22 mmol), $NaH_2PO_4.H_2O$ (207 mg, 1.5 mmol), sodium chlorite (80% purity, 62 mg, 0.55 mmol), THF (4 mL), water (6.0 mL), *t*-BuOH (1.5 mL), and 2-methyl-2-butene (2.0 mL of 2.0 M in THF) was stirred vigorously for 1 hour at -10 ^oC. Then the mixture was warmed to room temperature and stirred for another 2 h. The mixture was then diluted with water (10 mL) and extracted with CH_2Cl_2 (4 × 20 mL). The combined organic extract was dried over anhydrous Na_2SO_4 and concentrated *in vacuo*. The residue was purified by flash column chromatography on silica gel to give product **38** [91% yield (49.3 mg)] as a white solid (mp: 80-82 ^oC).

R_f 0.15 (ethyl acetate/hexane = 1:1); ^1H NMR (500 MHz, $CDCl_3$) δ 8.83 (br, 1H), 7.33 – 7.17 (m, 3H), 7.11 – 6.97 (m, 2H), 4.49 (t, *J* = 7.2 Hz, 2H), 3.27 (t, *J* = 7.2 Hz, 2H), 2.72 (q, *J* = 7.6 Hz, 2H), 1.03 (t, *J* = 7.6 Hz, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 164.8, 144.8, 136.8, 135.0, 129.0, 128.8, 127.3, 49.5, 36.6, 16.4, 12.9 ppm; IR (neat) 3461, 3026, 2935, 1694, 1569, 1467, 1274, 1255, 1217, 1023, 760, 703 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{13}H_{15}N_3O_2$: m/z 246.1237 ($[M + H]^+$), found: m/z 246.1241 ($[M + H]^+$).

(c) Preparation of N'-((5-ethyl-1-phenethyl-1*H*-1,2,3-triazol-4-yl)methylene)benzohydrazide (39)



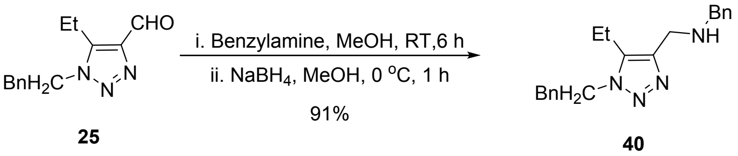
To a stirred solution of compound **25** (22.9 mg, 0.10 mmol) in methanol (1.0 mL) was added benzoyl hydrazine (13.6 mg, 0.10 mmol). The reaction mixture was warmed to 60 ^oC and stirred at this temperature for 6 h. Solvent was removed under reduced pressure, and the residue was washed with cold ethanol (1.0 mL) and cold diethyl ether (1.0 mL) to give the product **39** [93%

yield (32.4 mg)] as a white solid. It was a mixtures of isomers with the *E/Z* in 2.3:1 ratio.

E isomer: R_f 0.70 (ethyl acetate/methanol = 20:1), ^{1}H NMR (500 MHz, DMSO) δ 12.09 (s, 1H), 8.72 (s, 1H), 8.06 – 7.85 (m, 2H), 7.63 – 7.48 (m, 3H), 7.31 – 7.12 (m, 5H), 4.57 (t, *J* = 7.1 Hz, 2H), 3.19 (t, *J* = 7.1 Hz, 2H), 2.80 (q, *J* = 7.4 Hz, 2H), 1.01 (t, *J* = 7.5 Hz, 3H) ppm; ^{13}C NMR (126 MHz, DMSO) δ 162.9, 141.2, 138.9, 138.2, 137.6, 133.5, 131.7, 128.8, 128.5, 128.4, 127.5, 126.6, 48.3, 35.7, 15.8, 12.3 ppm; IR (neat) 3220, 3030, 2936, 1648, 1549, 1271, 1147, 1025, 693 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{20}H_{21}N_5O$: m/z 348.1819 ($[M + H]^+$), found: m/z 348.1820 ($[M + H]^+$).

Z isomer: R_f 0.50 (ethyl acetate/methanol = 20:1), ^{1}H NMR (500 MHz, DMSO) δ13.43 (s, 1H), 7.98 – 7.89 (m, 2H), 7.69 – 7.59 (m, 4H), 7.32 – 7.15 (m, 5H), 4.66 (t, *J* = 7.1 Hz, 2H), 3.23 (t, *J* = 7.1 Hz, 2H), 2.79 (q, *J* = 7.6 Hz, 2H), 1.00 (t, *J* = 7.6 Hz, 3H) ppm; ^{13}C NMR (126 MHz, DMSO) δ 162.7, 140.7, 138.3, 137.3, 132.9, 132.3, 131.5, 129.1, 128.8, 128.5, 127.0, 126.8, 48.9, 35.4, 14.7, 13.7 ppm; IR (neat) 3224, 3029, 2975, 1681, 1557, 1486, 1277, 1027, 696, 688 cm^{-1}.

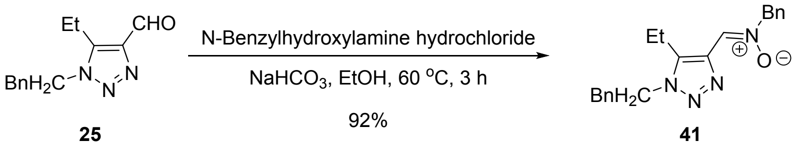
(d) Preparation of N-benzyl-1-(5-ethyl-1-phenethyl-1*H*-1,2,3-triazol-4-yl)methanamine (40)



To a stirred solution of compound **25** (22.9 mg, 0.10 mmol) in anhydrous methanol (1.0 mL) was added benzylamine (15.0 mg, 0.14 mmol) under argon at room temperature. After stirring for 6 h at RT, the reaction mixture was allowed to cool to 0 °C and $NaBH_4$ (5 mg, 0.14 mmol) was added. After an additional 1 h at 0 °C, reaction was quenched with aqueous NH_4Cl (3.0 mL). The mixture was then diluted with water (15 mL) and extracted with CH_2Cl_2 (3 × 20 mL). The combined organic extracts were dried over anhydrous Na_2SO_4, and the solvent was removed under reduced pressure. T he residue was purified by flash column chromatography on silica gel to gave product **40** [91% yield (29.0 mg)] as a light yellow solid.

R_f 0.30 (ethyl acetate/methanol = 20:1), ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37 – 7.14 (m, 8H), 7.12 – 7.00 (m, 2H), 4.40 (t, *J* = 7.6 Hz, 2H), 3.82 (s, 2H), 3.80 (s, 2H), 3.55 (br, 1H), 3.22 (t, *J* = 7.4 Hz, 2H), 2.41 (q, *J* = 7.6 Hz, 2H), 1.00 (t, *J* = 7.6 Hz, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 142.4, 140.0, 137.5, 136.0, 128.9, 128.5, 128.4, 127.1, 127.1, 53.2, 49.3, 43.5, 37.0, 15.8, 13.6 ppm; IR (neat) 3318, 3028, 2928, 1735, 1453, 1241, 1167, 1045, 1028, 731, 698 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{20}H_{24}N_4O$: m/z 321.2074 ([M + H]$^+$), found: m/z 321.2077 ([M + H]$^+$).

(e) Preparation of N-benzyl-1-(5-ethyl-1-phenethyl-1*H*-1,2,3-triazol-4-yl)methanimine oxide (41)

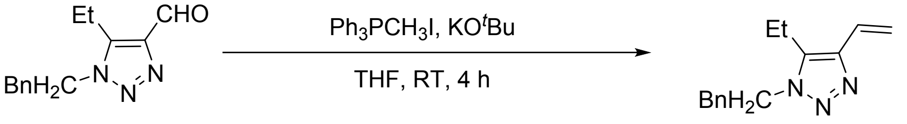


To a stirred solution of compound **25** (22.9 mg, 0.10 mmol) in absolute ethanol (5 mL) was added N-benzylhydroxylamine hydrochloride (23.8 mg, 1.5 mmol) and sodium bicarbonate (12.6 mg, 1.5 mmol). The reaction mixture was heated to 60 °C and stirred for 3 h. Solvent was removed under vacuum, and the residue was diluted with H_2O (10 mL) and extracted with CH_2Cl_2 (3 × 20 mL). The combined organic extracts were dried over anhydrous Na_2SO_4, and the solvent was removed under reduced pressure. The residue was purified by flash column chromatography on silica gel to give product **41** [91% yield (30.8 mg)] as a light yellow solid.

R_f 0.50 (ethyl acetate/methanol = 20:1), ^{1}H NMR (500 MHz, DMSO) δ 8.20 (s, 1H), 7.53 – 7.47 (m, 2H), 7.43 – 7.33 (m, 3H), 7.29 – 7.23 (m, 2H), 7.23 – 7.18 (m, 1H), 7.17 – 7.13 (m, 2H), 5.09 (s, 2H), 4.53 (t, *J* = 7.2 Hz, 2H), 3.14 (t, *J* = 7.2 Hz, 2H), 2.89 (q, *J* = 7.5 Hz, 2H), 0.78 (t, *J* = 7.4 Hz, 3H) ppm; ^{13}C NMR (126 MHz, DMSO) δ 139.0, 137.5, 135.8, 134.6, 129.0, 128.8, 128.4, 128.3, 128.3, 126.6, 126.5, 68.6, 48.4, 35.6, 17.0, 14.2 ppm; IR (neat) 3029, 2930, 1610, 1496, 1455, 1143, 755, 700 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{20}H_{22}N_4O$: m/z 335.1866 ([M +

H]$^+$), found: m/z 335.1864 ([M + H]$^+$).

(f) Preparation of 5-ethyl-1-phenethyl-4-vinyl-1H-1,2,3-triazole (42)[3]

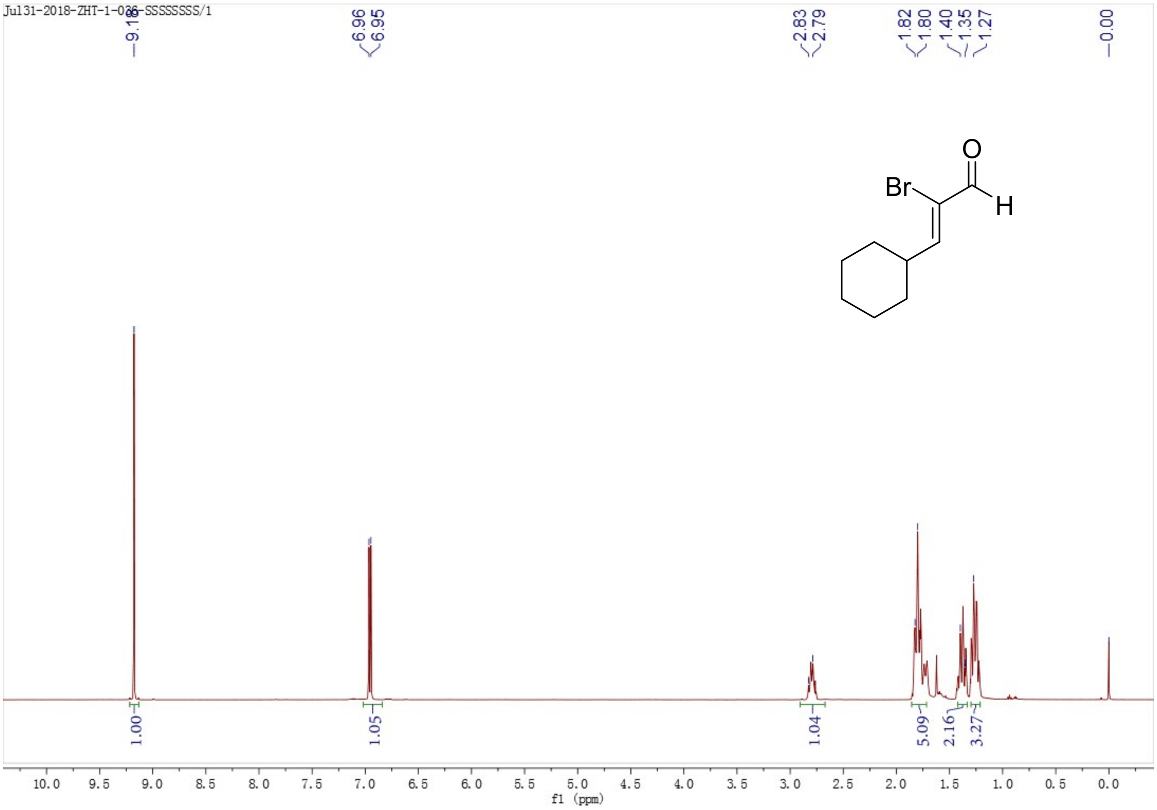


25 **42**

A solution of methyltriphenylphosphonium iodide (53.8 mg, 0.13 mmol) and KO^{t}Bu (33.3 mg, 0.30 mmol) in dry THF (0.3 mL) was stirred at 0 °C for 2 h. Then aldehyde **25** (22.9 mg, 0.10 mmol) in dry THF (1 mL) was added dropwise. Then the mixture was allowed warm to room temperature and stirred for 4 h. The reaction was terminated with diethyl ether (10 mL)and poured temperature and stirred for 4 h. The reaction was terminated with diethyl ether (10 mL)and poured into ice-water (10 mL) and extracted with diethyl ether twice. The organic layers were dried over anhydrous Na_2SO_4 and evaporated under reduced pressure. The crude product was purified by silica gel column chromatography, to give product **42** [88% yield (20.0 mg)] as a light yellow liquid.

R_f 0.20 (ethyl acetate/hexane = 1:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33 – 7.18 (m, 3H), 7.13 – 7.04 (m, 2H), 6.59 (dd, *J* = 17.6, 11.3 Hz, 1H), 5.97 (dd, *J* = 17.6, 1.5 Hz, 1H), 5.31 (dd, *J* = 11.3, 1.5 Hz, 1H), 4.41 (t, *J* = 7.4 Hz, 2H), 3.23 (t, *J* = 7.4 Hz, 2H), 2.45 (q, *J* = 7.6 Hz, 2H), 1.01 (t, *J* = 7.6 Hz, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 141.9, 137.5, 135.2, 128.9, 128.9, 127.2, 125.1, 115.1, 77.2, 49.2, 37.0, 15.8, 13.4 ppm; IR (neat) 3028, 2973, 2932, 1454, 1239, 1011, 985, 722, 699 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{14}H_{17}N_3$: m/z 228.1495 ([M + H]$^+$), found: m/z 228.1497 ([M + H]$^+$).

7. ^{1}H NMR and ^{13}C NMR Spectra



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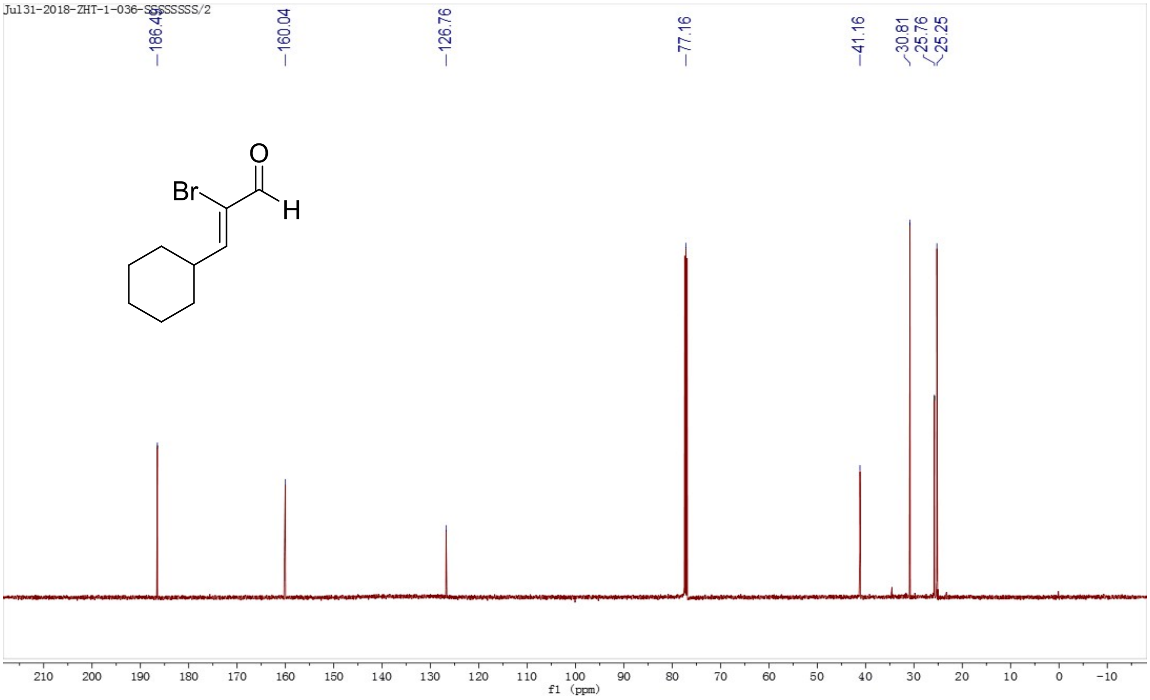
[3] H-C. Tsai, Y-H. Huang, C-M. Chou, *Org. Lett.*, **2018**, *20*, 1328.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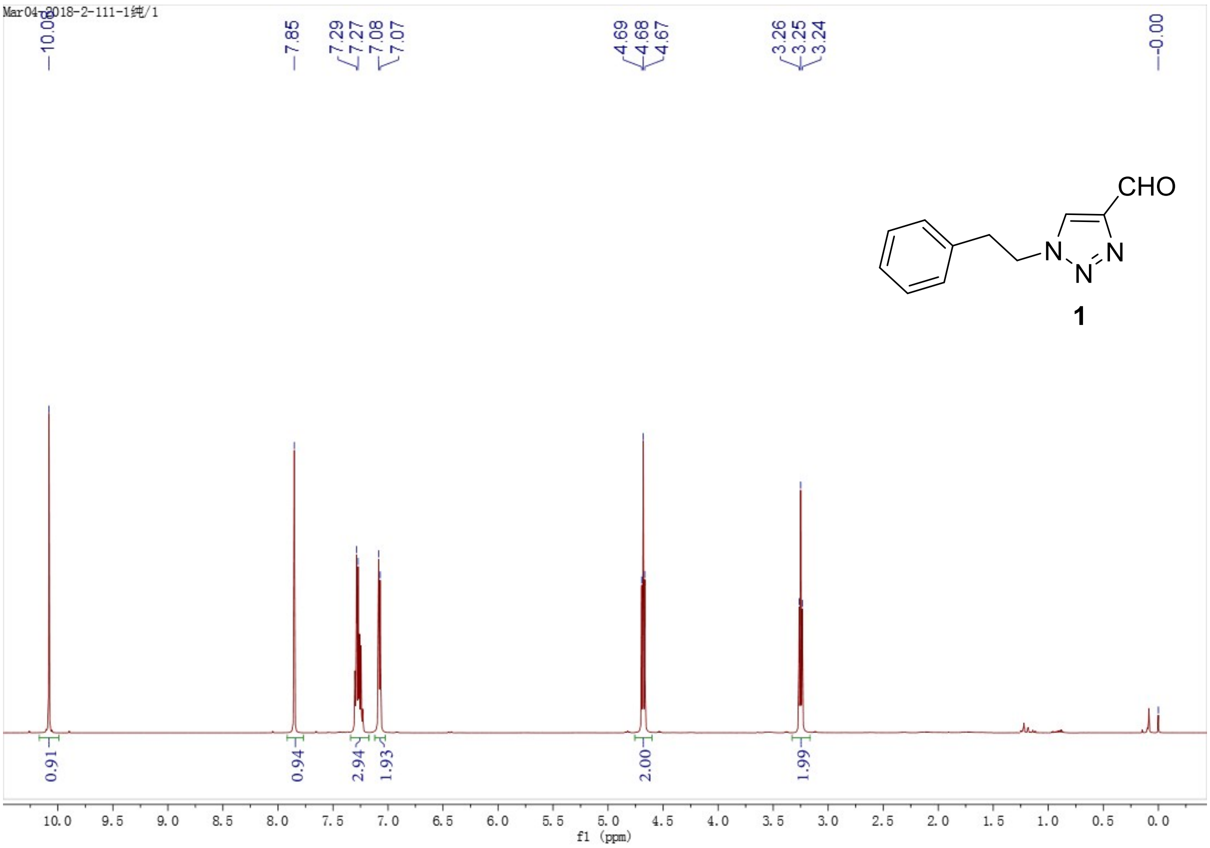
186.49
160.04
126.76
77.16
41.16
30.81
25.76
25.25
Br
O
H
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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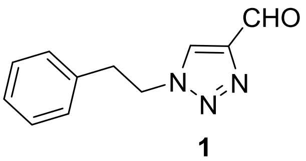
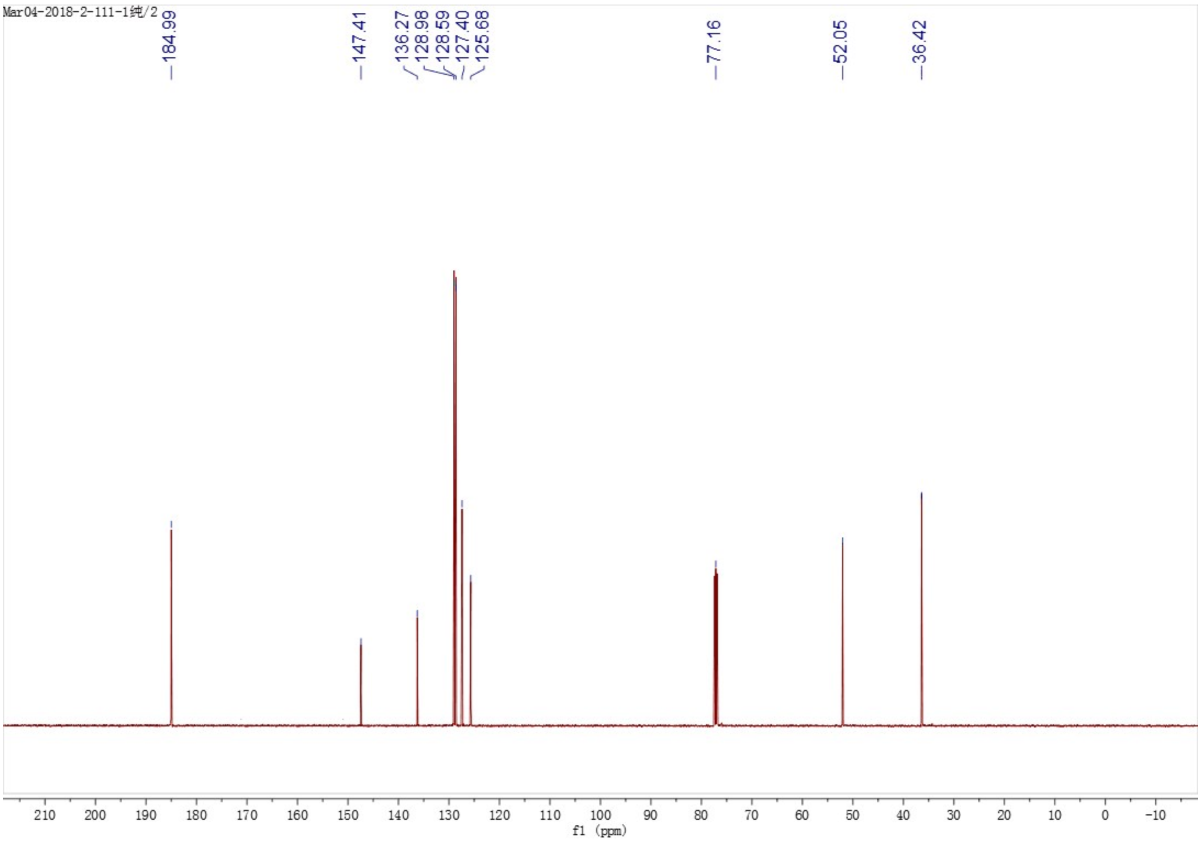
7.85
7.29
7.27
7.08
7.07
4.69
4.68
4.67
3.26
3.25
3.24
0.00
CHO
N
N
N
1
0.91
0.94
2.94
1.93
2.00
1.99
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


CHO
N
N
N

1


Mar04-2018-2-111-1纯/2
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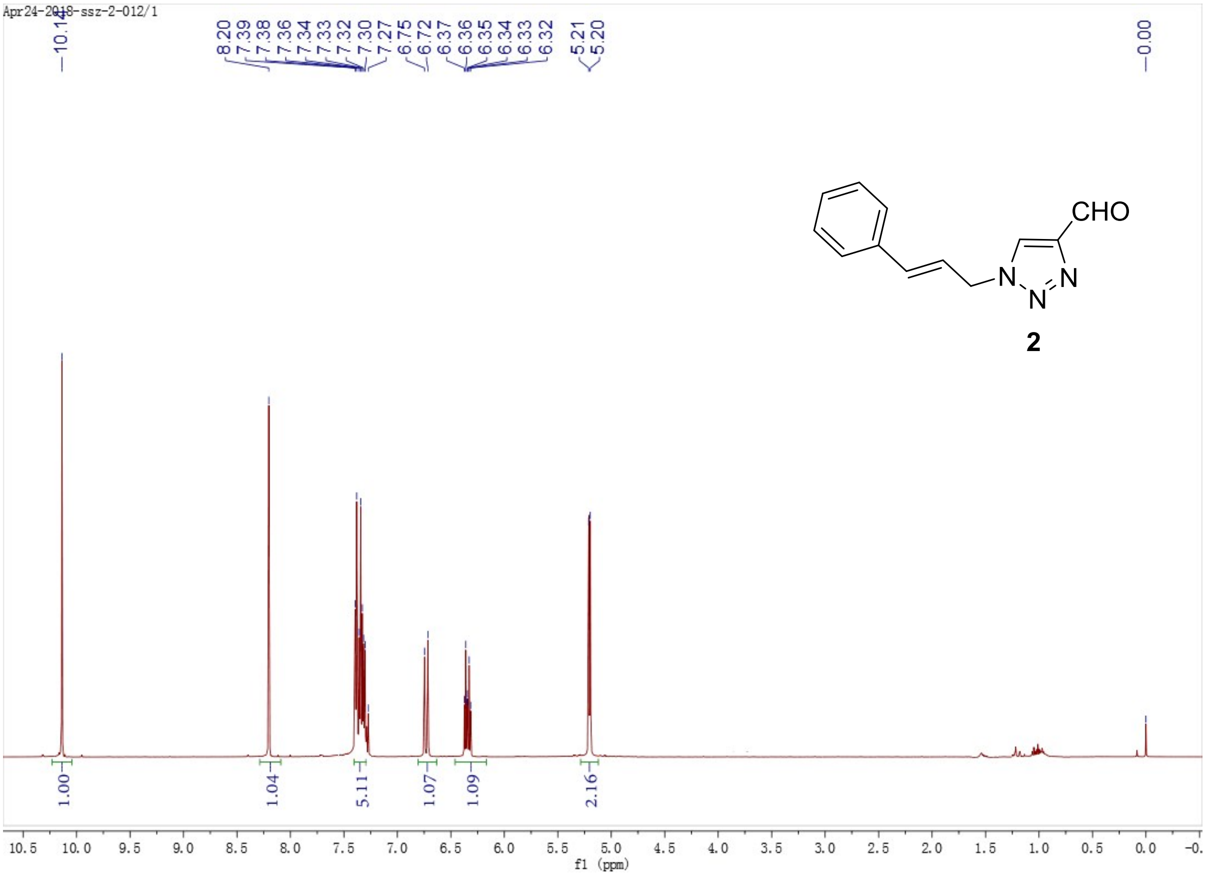
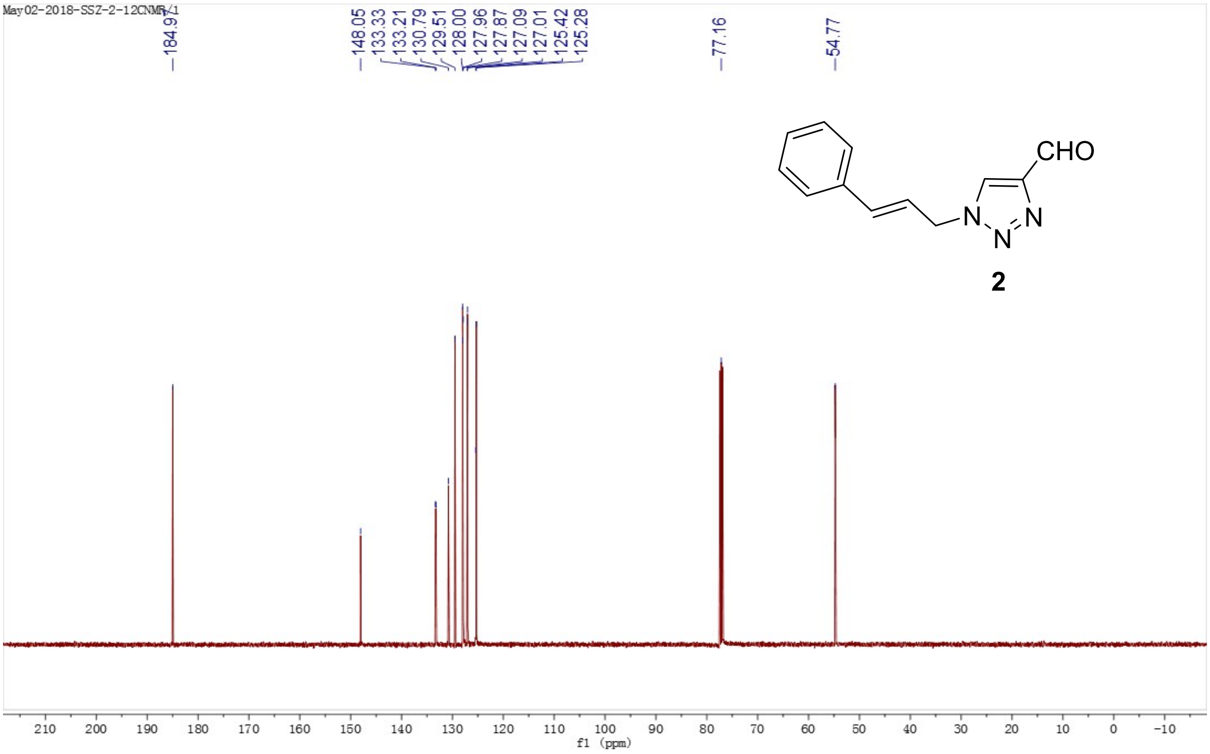
136.27
128.98
128.59
127.40
125.68
77.16
52.05
36.42
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)


Apr24-2018-ssz-2-012/1
10.14
8.20
7.39
7.38
7.36
7.34
7.33
7.32
7.30
7.27
6.75
6.72
6.37
6.36
6.35
6.34
6.33
6.32
5.21
5.20
0.00
CHO
N
N
N
1.00
1.04
5.11
1.07
1.09
2.16
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.
f1 (ppm)

2


May02-2018-SSZ-2-12CNMR/1
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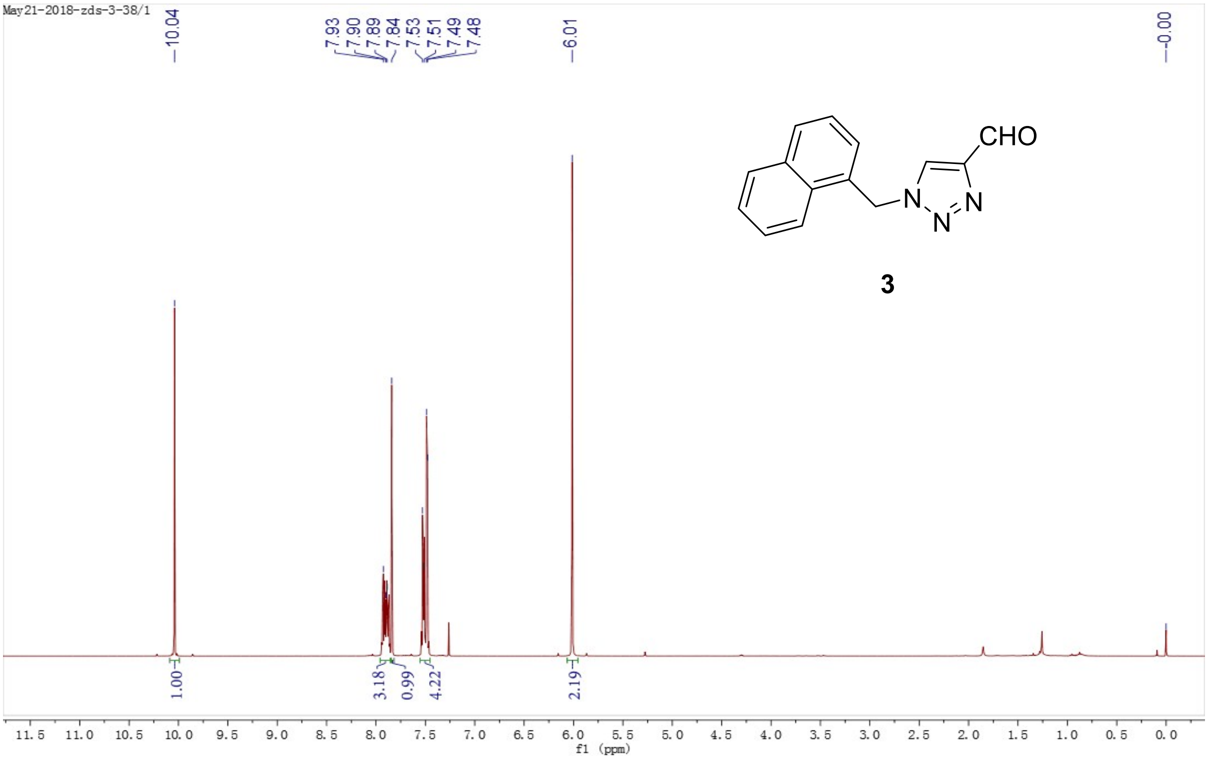
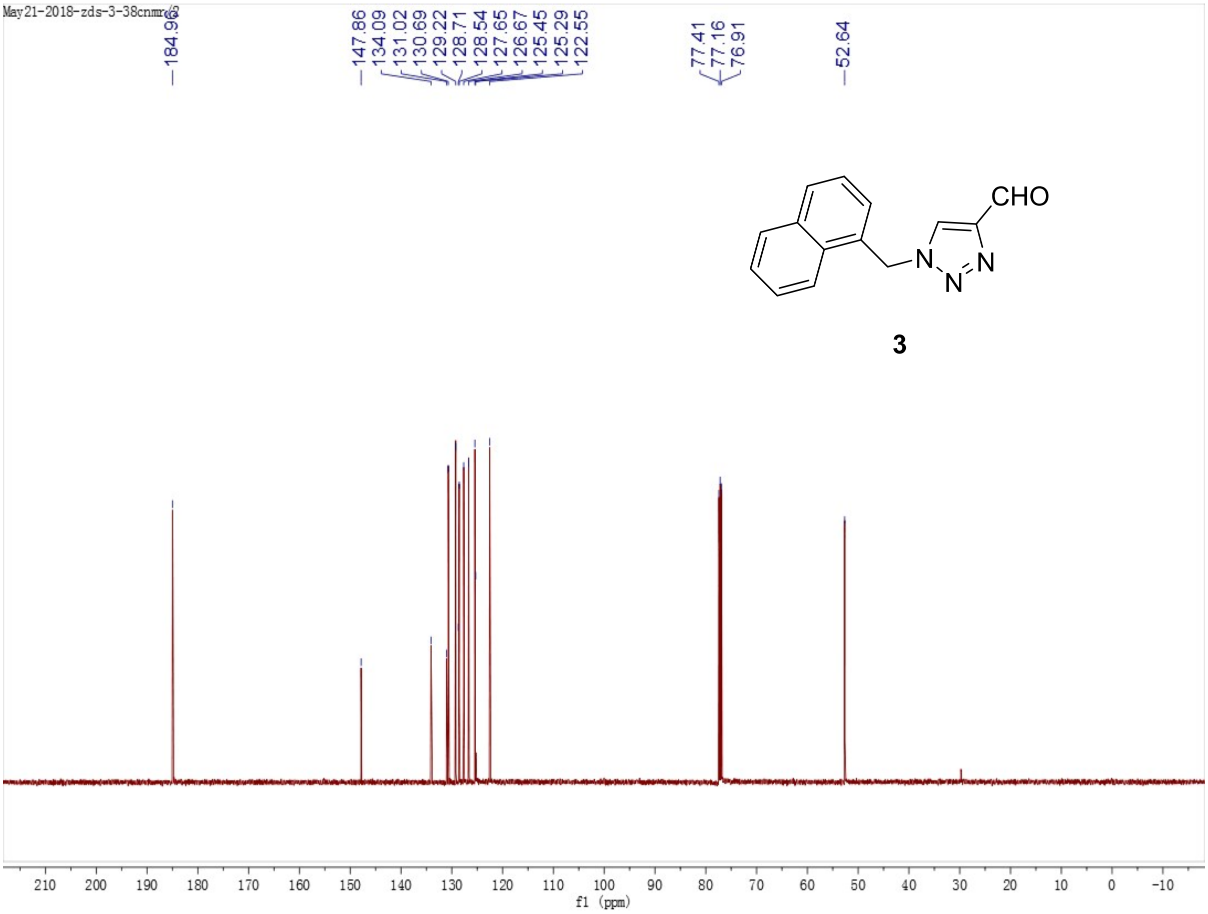
148.05
133.33
133.21
130.79
129.51
128.00
127.96
127.87
127.09
127.01
125.42
125.28
77.16
54.77
CHO
N
N
N
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

2



May21-2018-zds-3-38/1
10.04
7.93
7.90
7.89
7.84
7.53
7.51
7.49
7.48
6.01
0.00
CHO
N
N
N
3
1.00
3.18
0.99
4.22
2.19
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)



May21-2018-zds-3-38cnmr/2
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134.09
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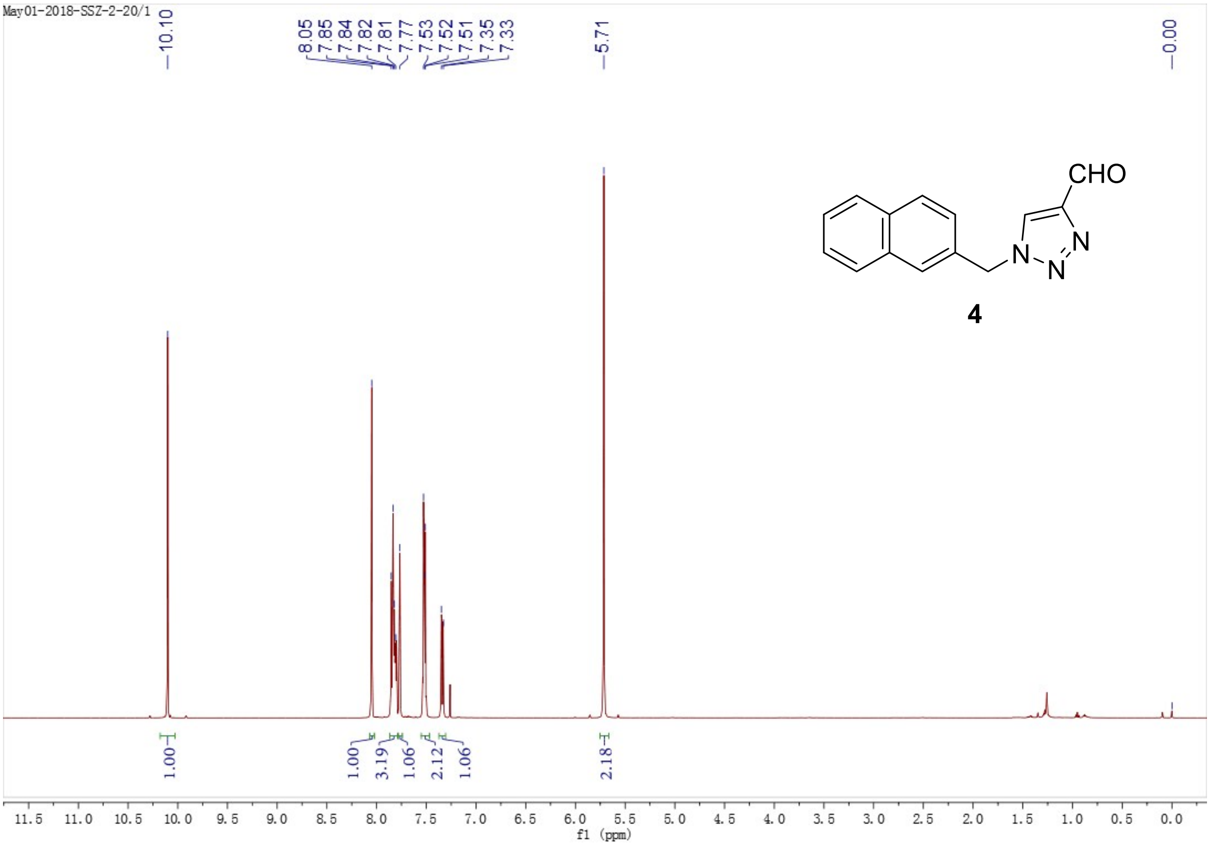
129.22
128.71
128.54
127.65
126.67
125.45
125.29
122.55
77.41
77.16
76.91
52.64
CHO
N
N
N
3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


May01-2018-SSZ-2-20/1
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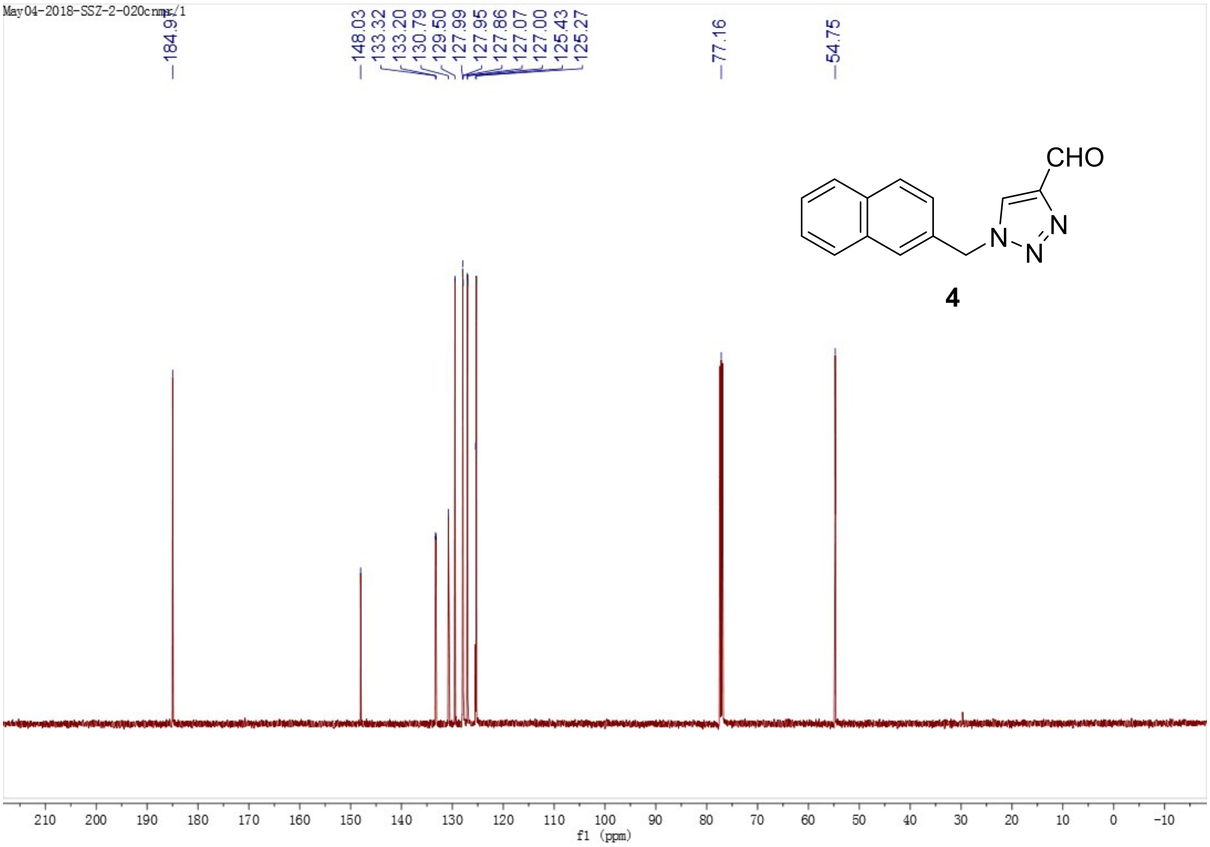
8.05
7.85
7.84
7.82
7.81
7.77
7.53
7.52
7.51
7.35
7.33
5.71
0.00
CHO
N
N
N
4
1.00
1.00
3.19
1.06
2.12
1.06
2.18
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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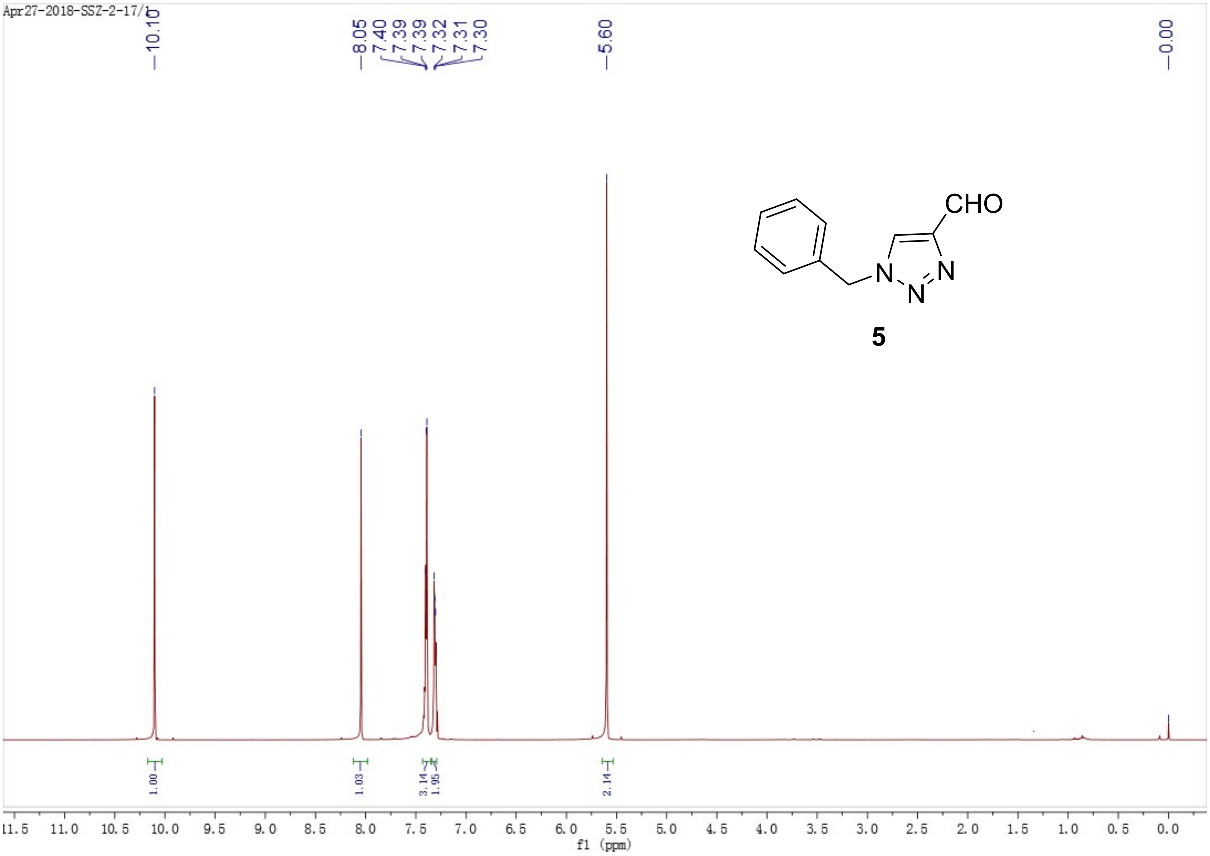
148.03
133.32
133.20
130.79
129.50
127.99
127.95
127.86
127.07
127.00
125.43
125.27
77.16
54.75
CHO
N
N
N
4
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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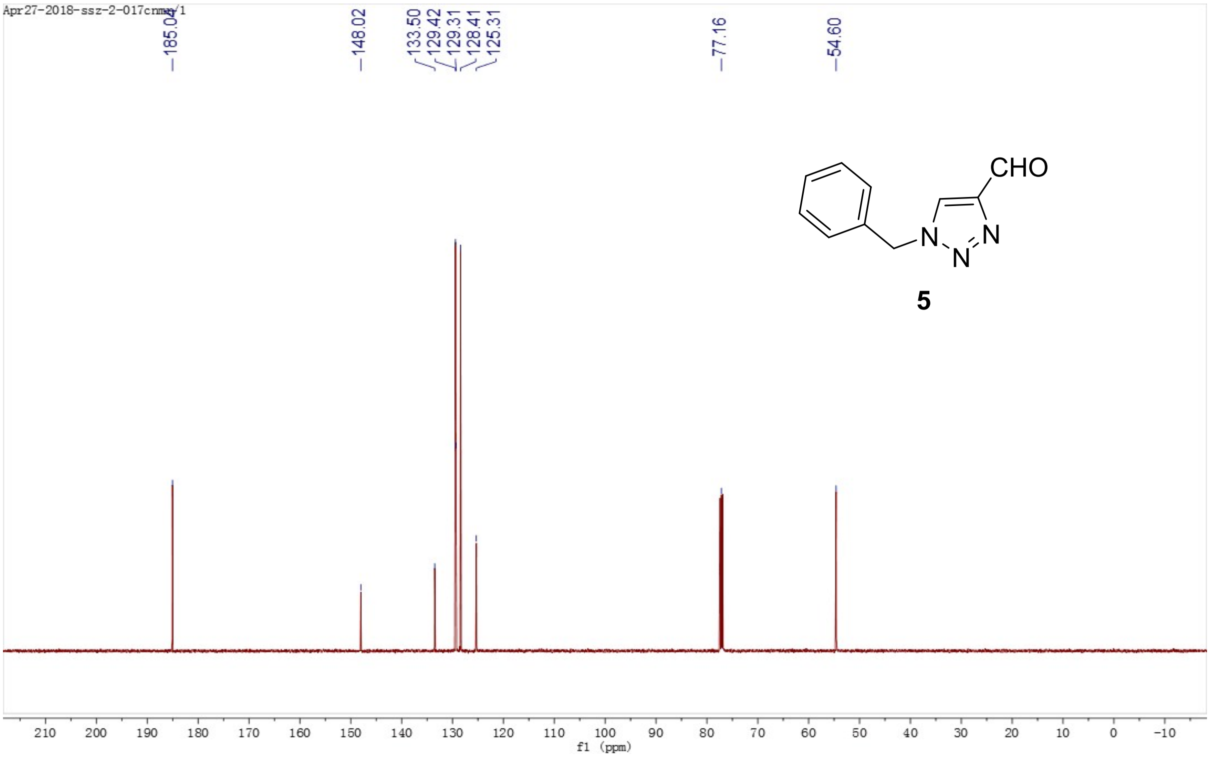
7.39
7.39
7.32
7.31
7.30
5.60
0.00
CHO
N
N
N
5
1.00
1.03
3.14
1.95
2.14
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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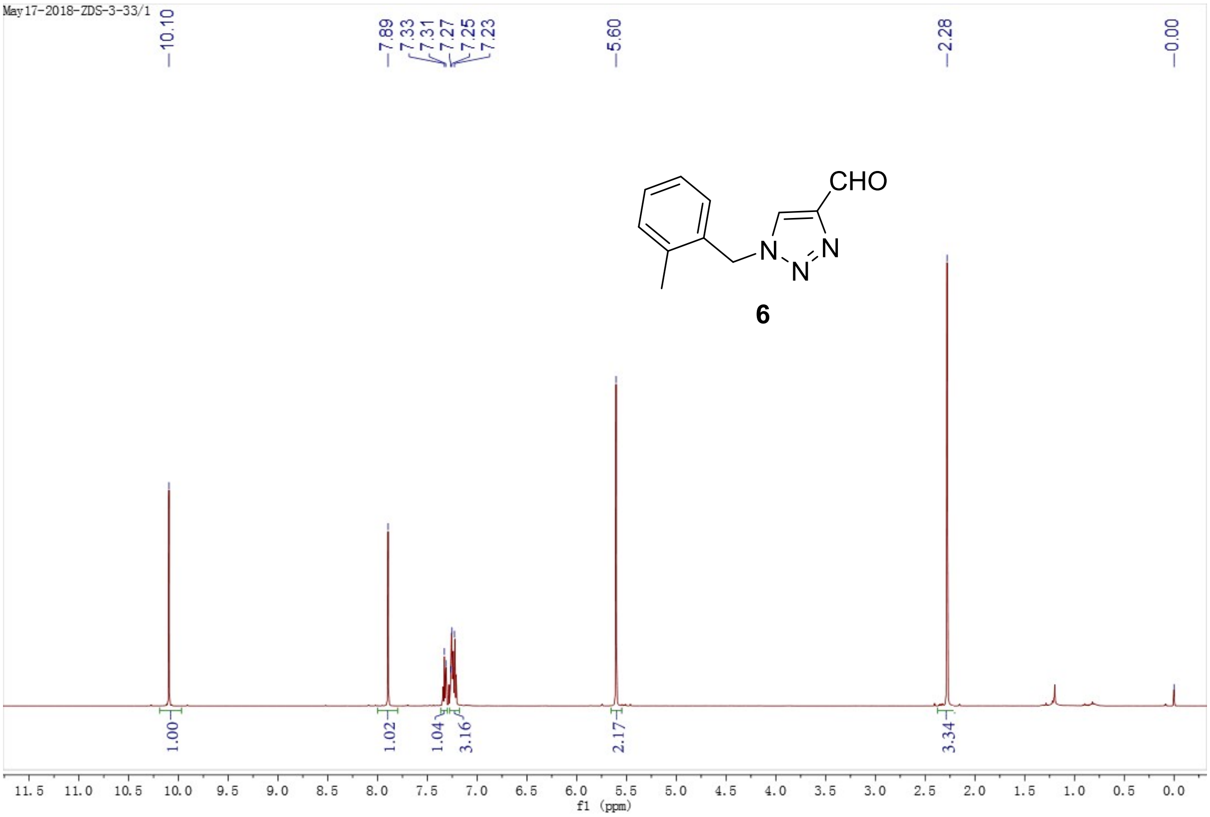
148.02
133.50
129.42
129.31
128.41
125.31
77.16
54.60
CHO
N
N
N
5
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


May17-2018-ZDS-3-33/1
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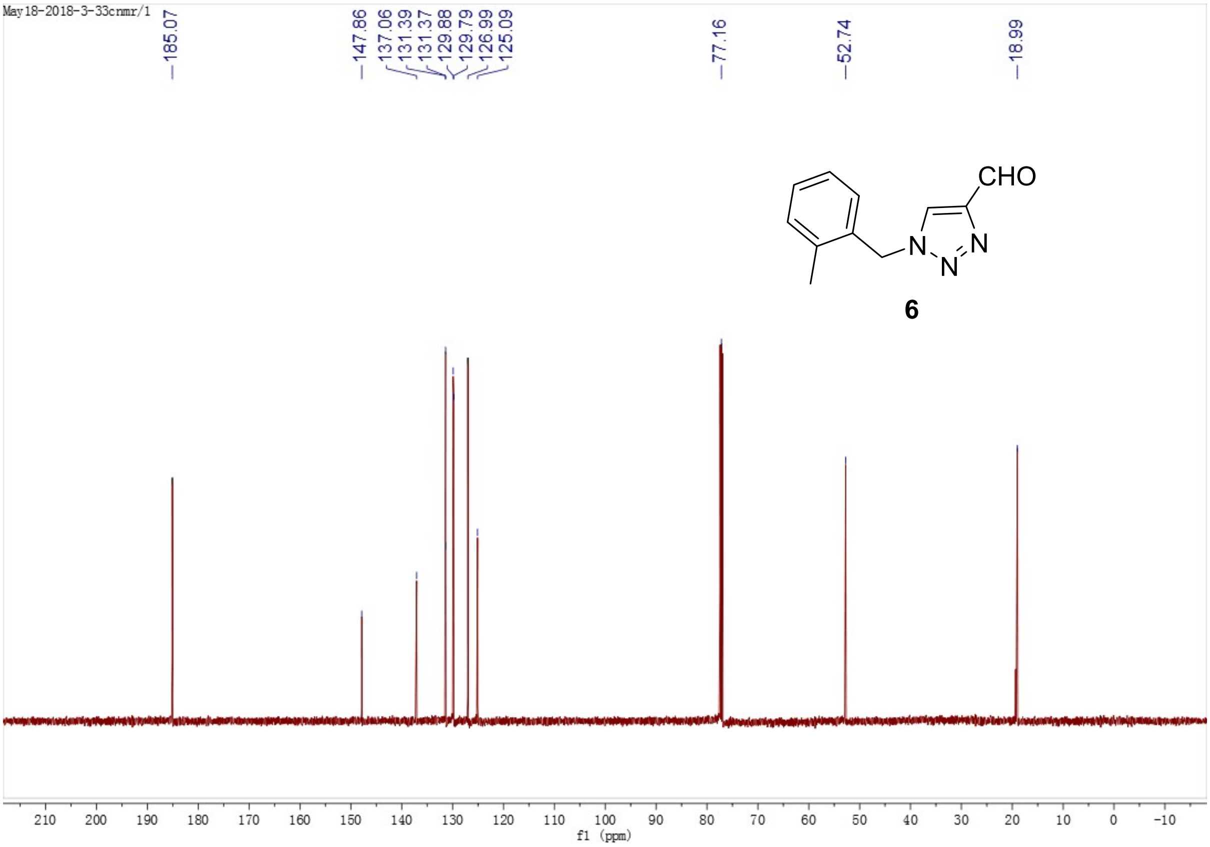
7.33
7.31
7.27
7.25
7.23
5.60
2.28
0.00
CHO
N
N
N
6
1.00
1.02
1.04
3.16
2.17
3.34
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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137.06
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125.09
77.16
52.74
18.99
CHO
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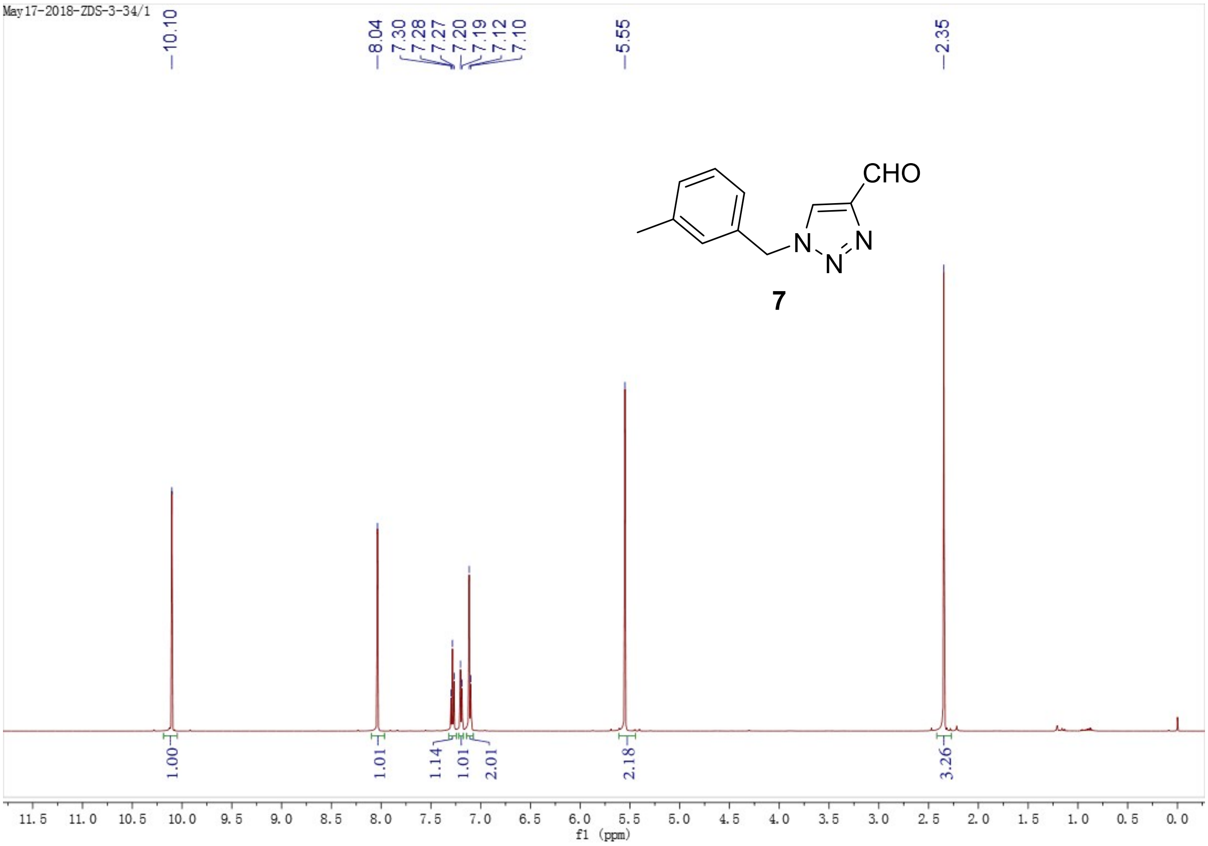
N
N
6
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


May17-2018-ZDS-3-34/1
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7.30
7.28
7.27
7.20
7.19
7.12
7.10
5.55
2.35
CHO
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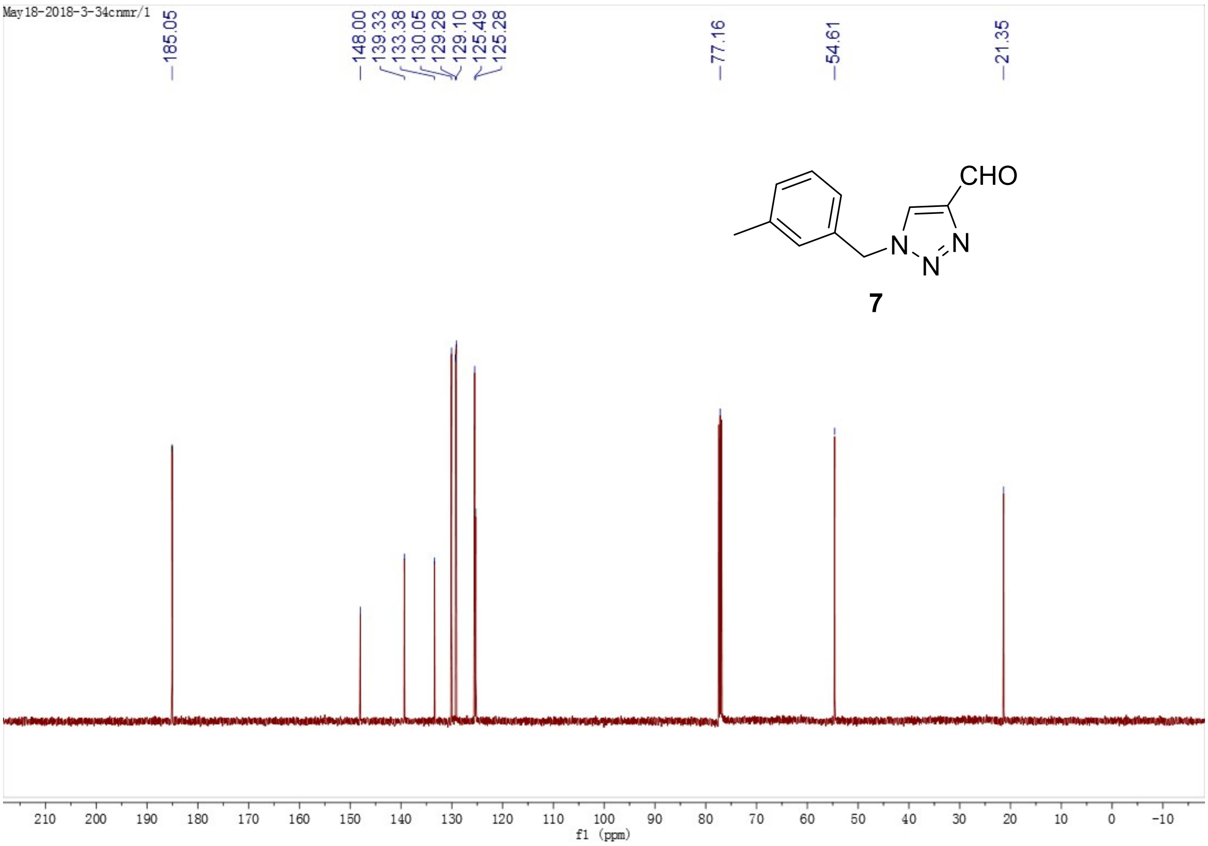
N
N
7
1.00
1.01
1.14
1.01
2.01
2.18
3.26
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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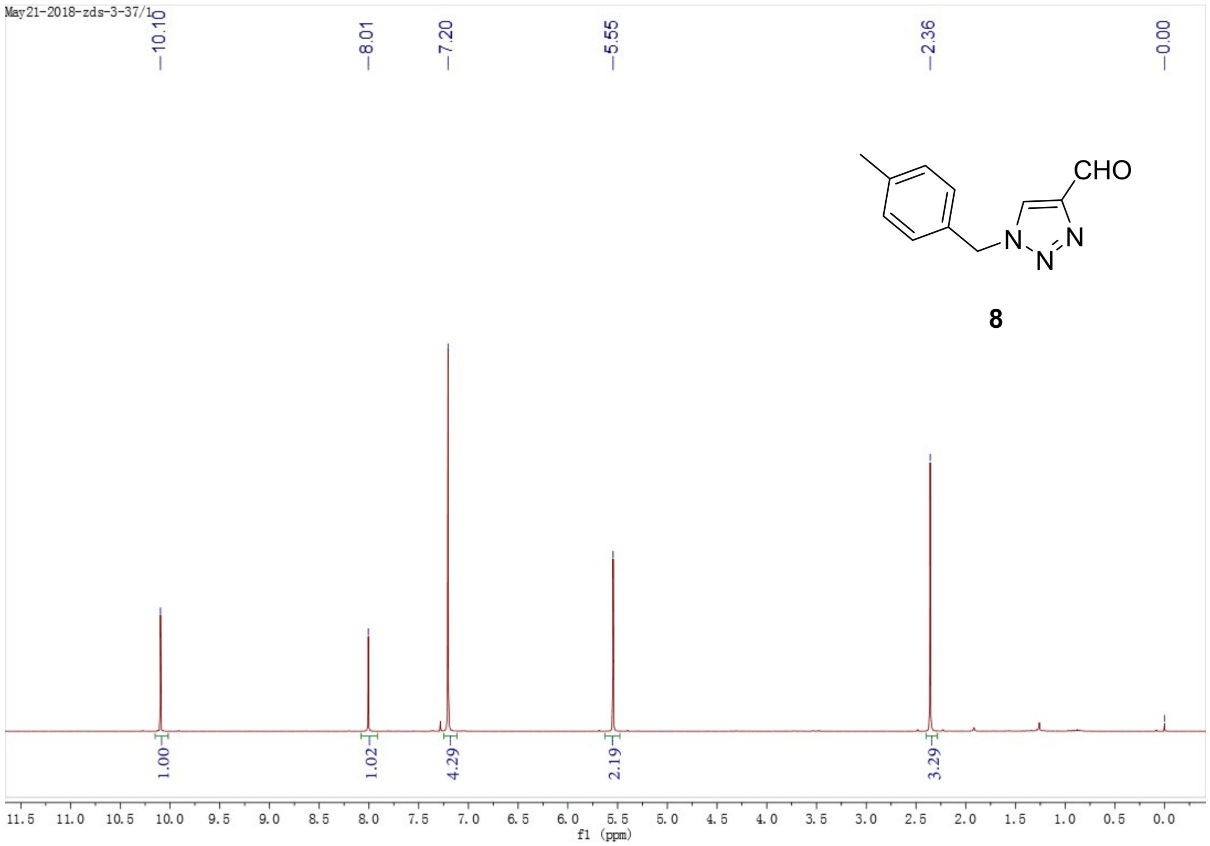
133.38
130.05
129.28
129.10
125.49
125.28
77.16
54.61
21.35
CHO
N
N
N
7
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


May21-2018-zds-3-37/1
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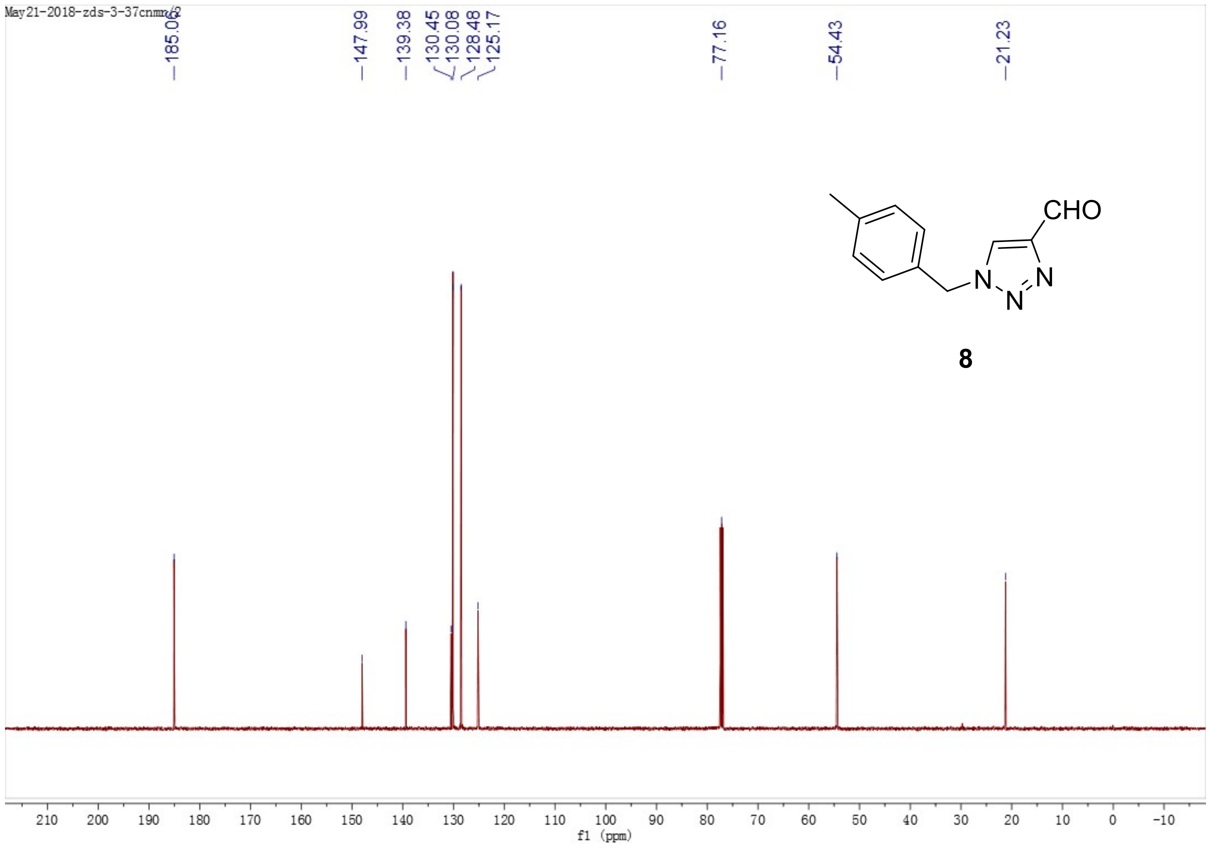
8.01
7.20
5.55
2.36
0.00
CHO
N
N
N
8
1.00
1.02
4.29
2.19
3.29
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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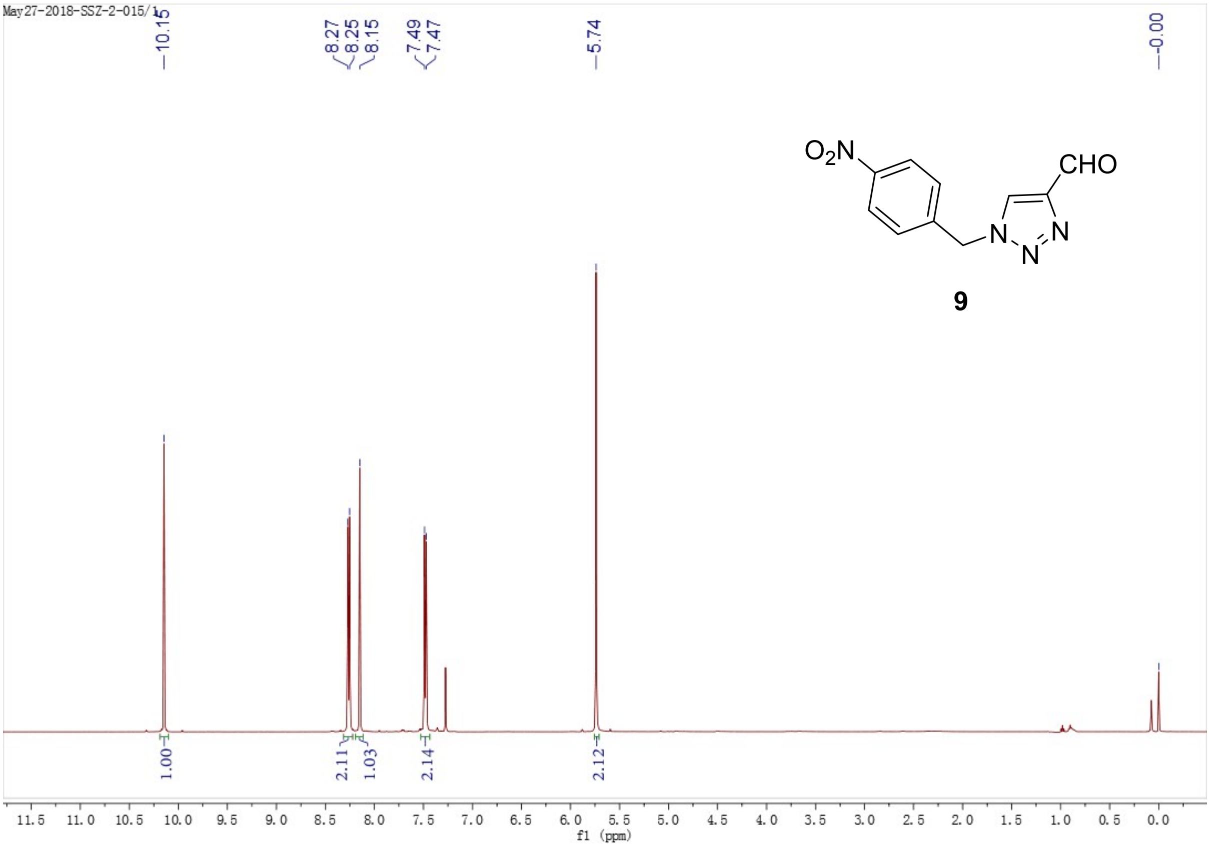
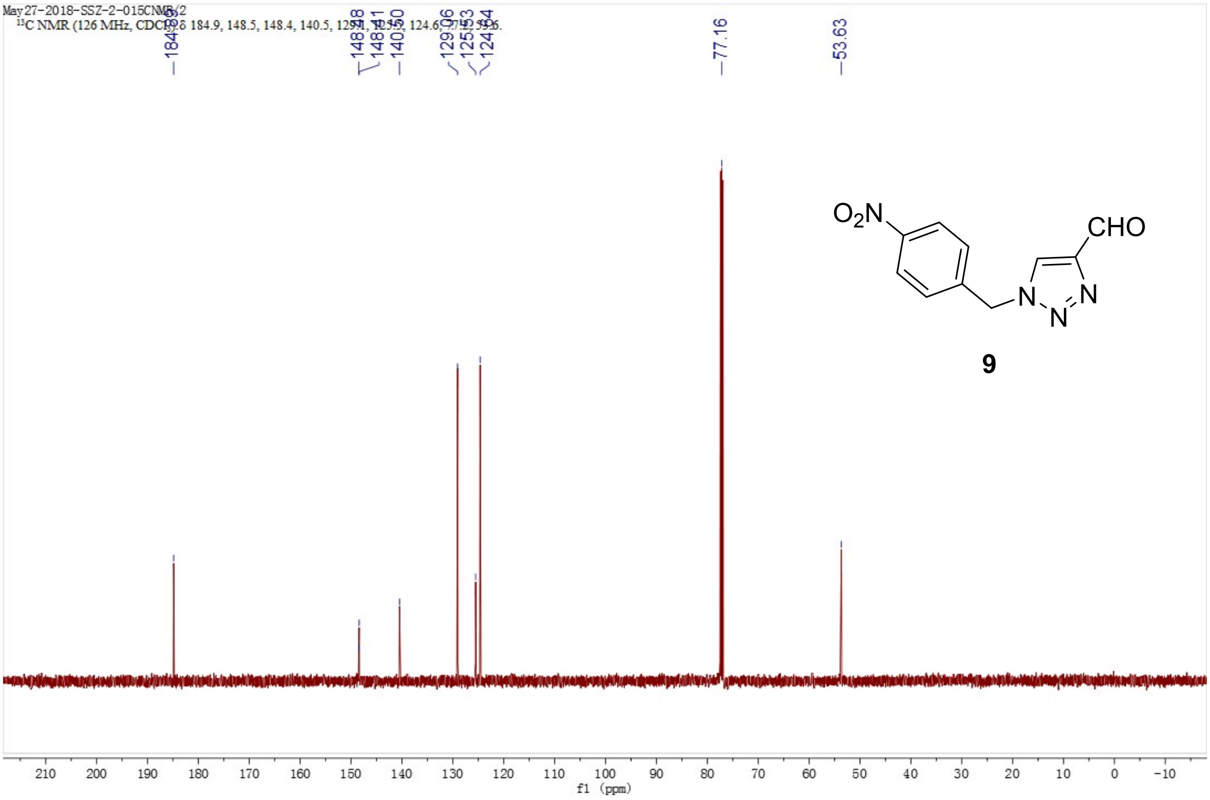
147.99
139.38
130.45
130.08
128.48
125.17
77.16
54.43
21.23
CHO
N
N
N
8
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


May27-2018-SSZ-2-015/1
10.15
8.27
8.25
8.15
7.49
7.47
5.74
0.00
O$_2$N
CHO
N
N
N
9
1.00
2.11
1.03
2.14
2.12
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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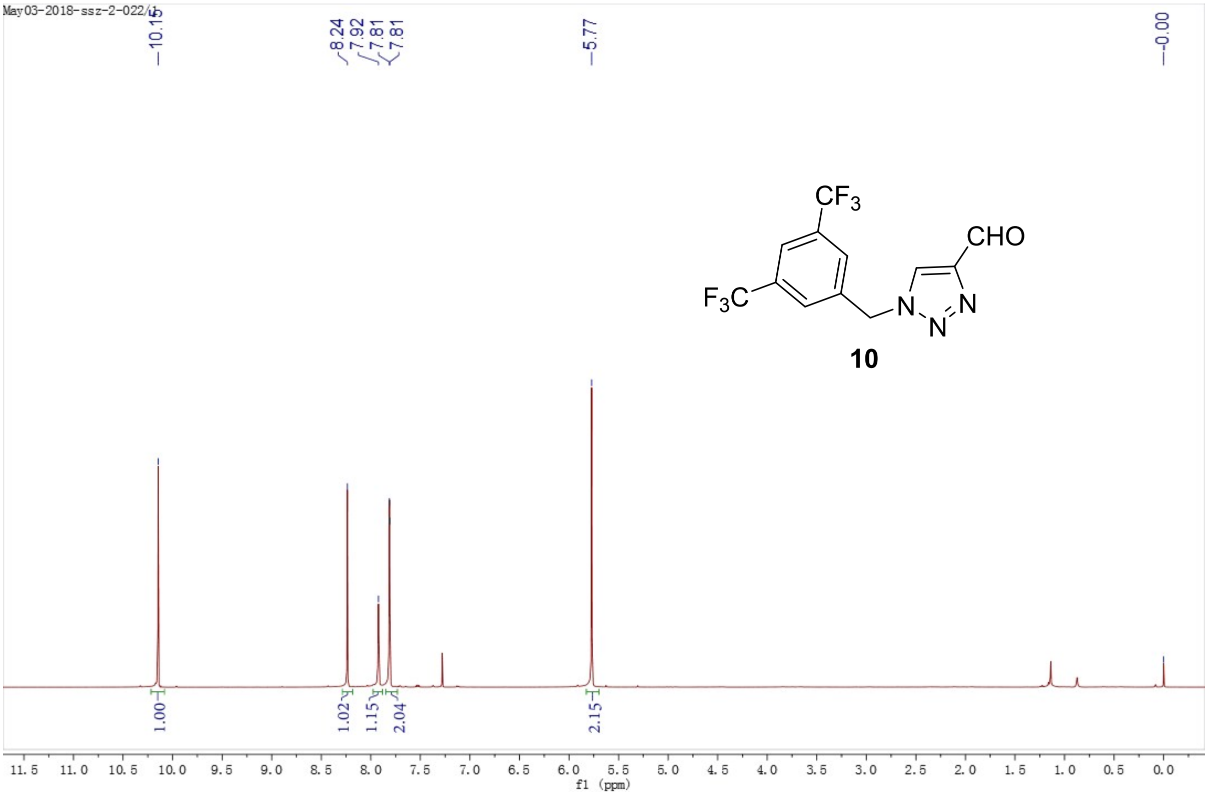
184.89
148.48
148.41
140.50
129.06
125.53
124.54
77.16
53.63
O$_2$N
CHO
N
N
N
9
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)


May03-2018-ssz-2-022/1
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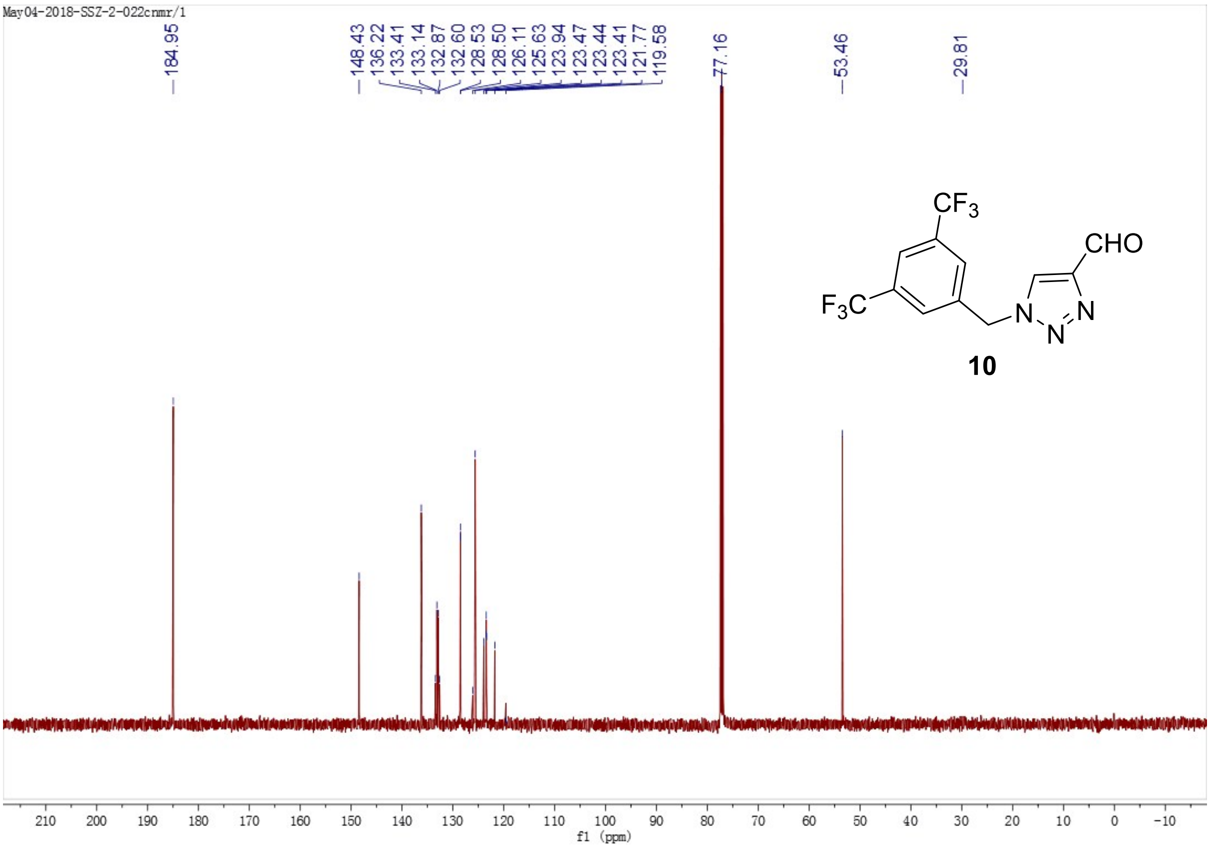
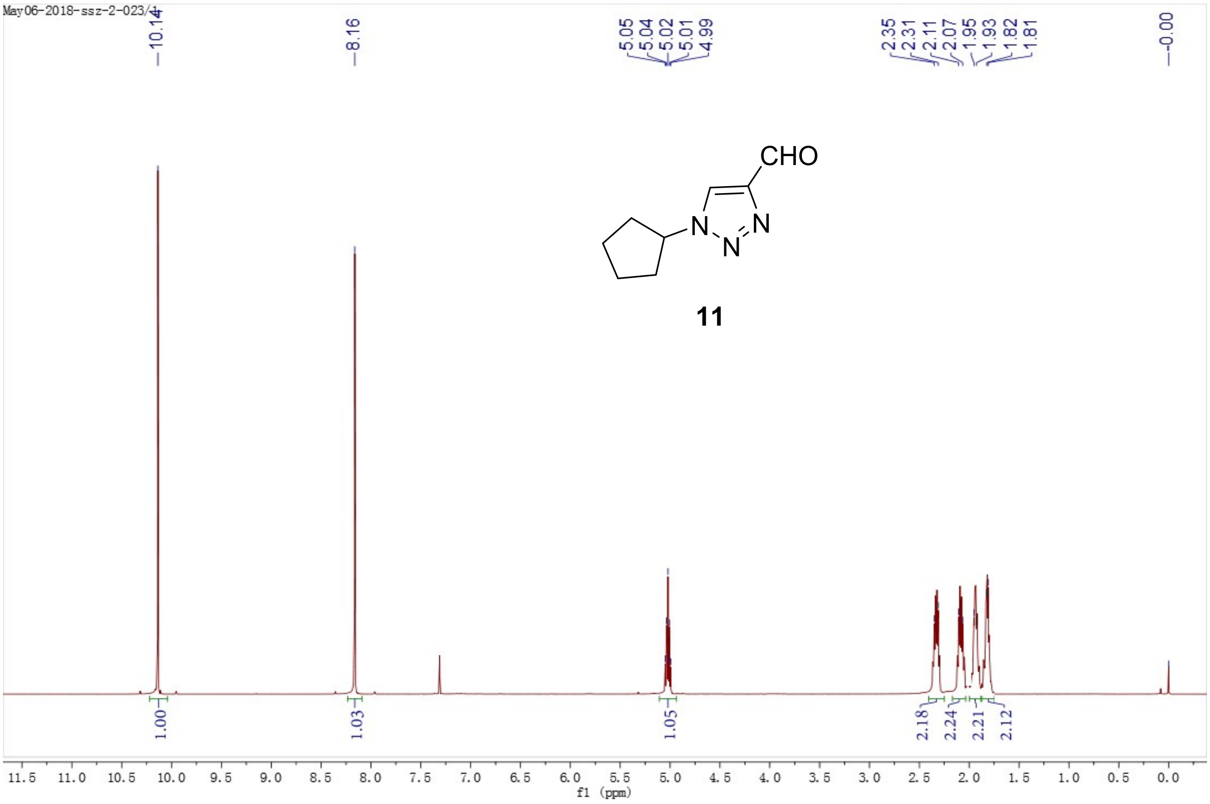
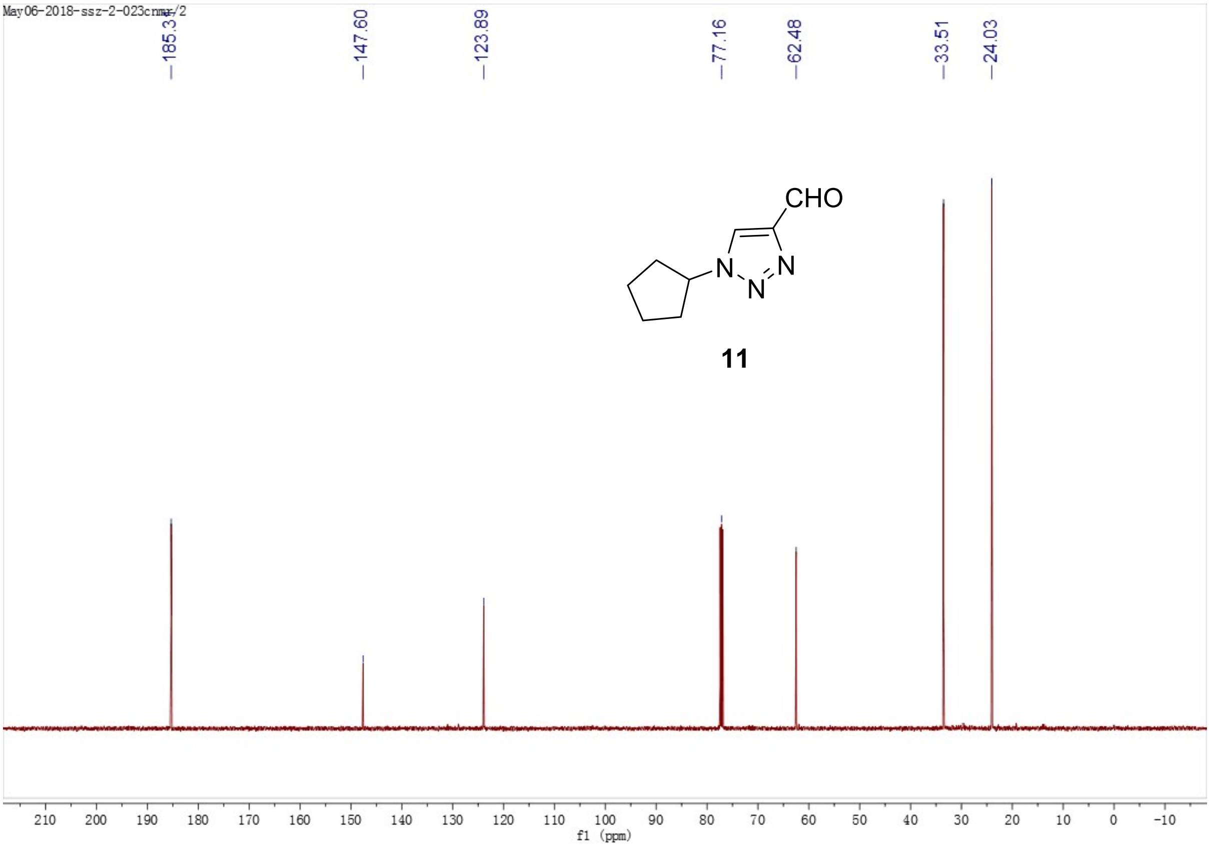
8.24
7.92
7.81
7.81
5.77
0.00
1.00
1.02
1.15
2.04
2.15
CF3
CHO
F3C
N
N
N
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

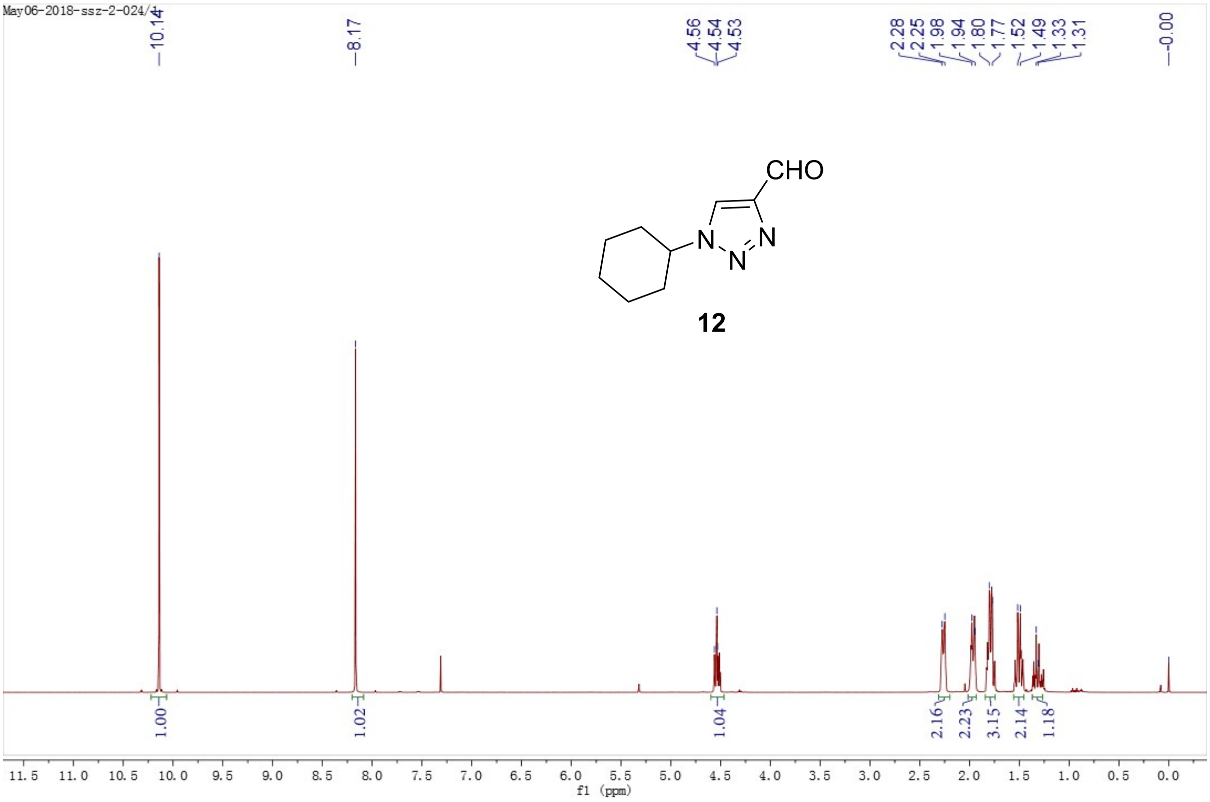
10

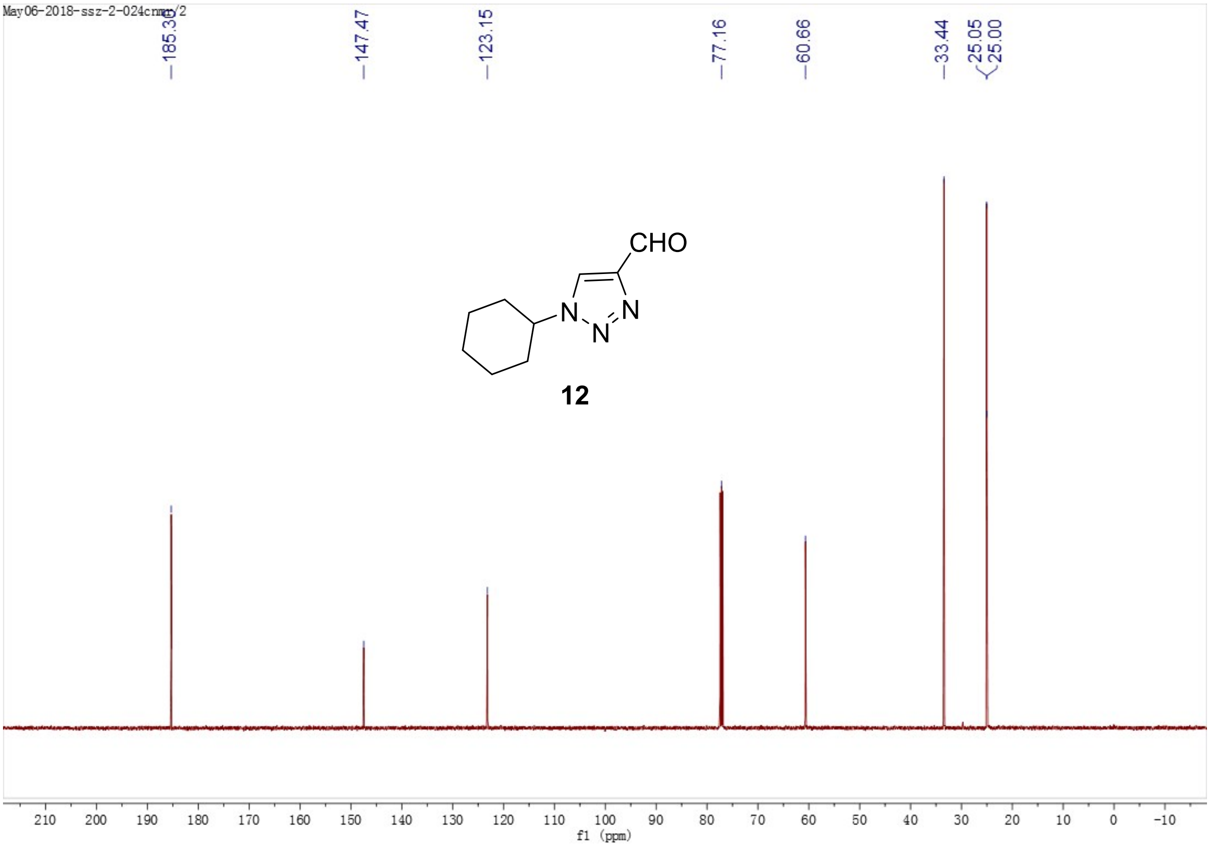

May04-2018-SSZ-2-022cnmr/1
184.95
148.43
136.22
133.41
133.14
132.87
132.60
128.53
128.50
126.11
125.63
123.94
123.47
123.44
123.41
121.77
119.58
77.16
53.46
29.81
CF3
CHO
F3C
N
N
N
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

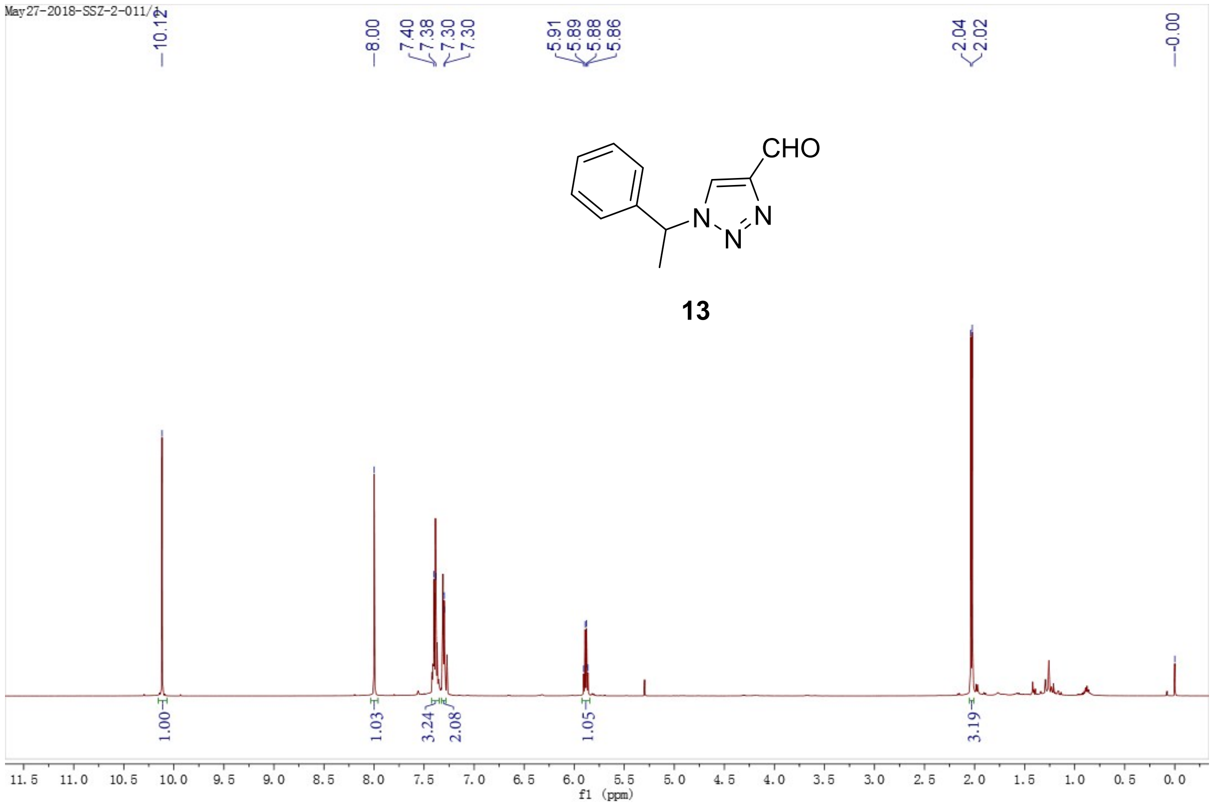
10

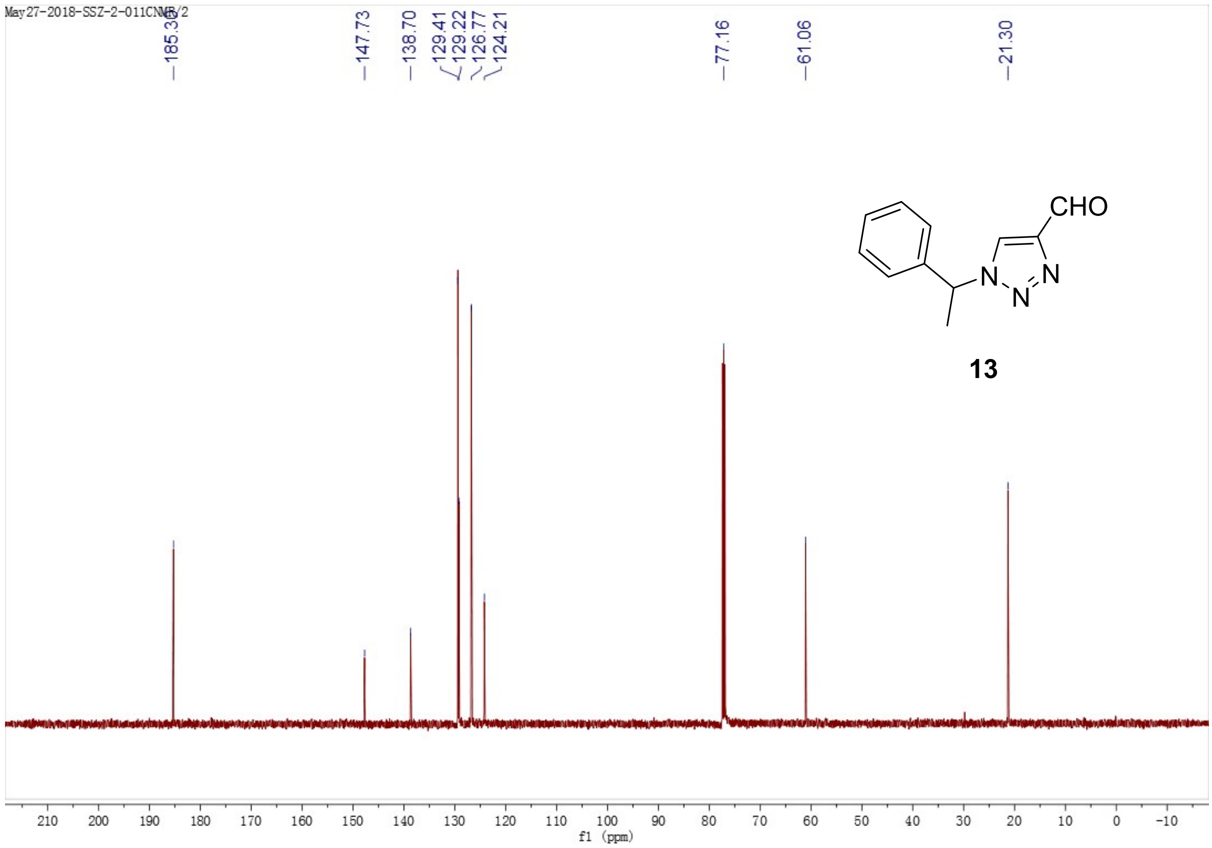

May06-2018-ssz-2-023/1
10.14
8.16
5.05
5.04
5.02
5.01
4.99
2.35
2.31
2.11
2.07
1.95
1.93
1.82
1.81
0.00
CHO
N
N
N
11
1.00
1.03
1.05
2.18
2.24
2.21
2.12
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

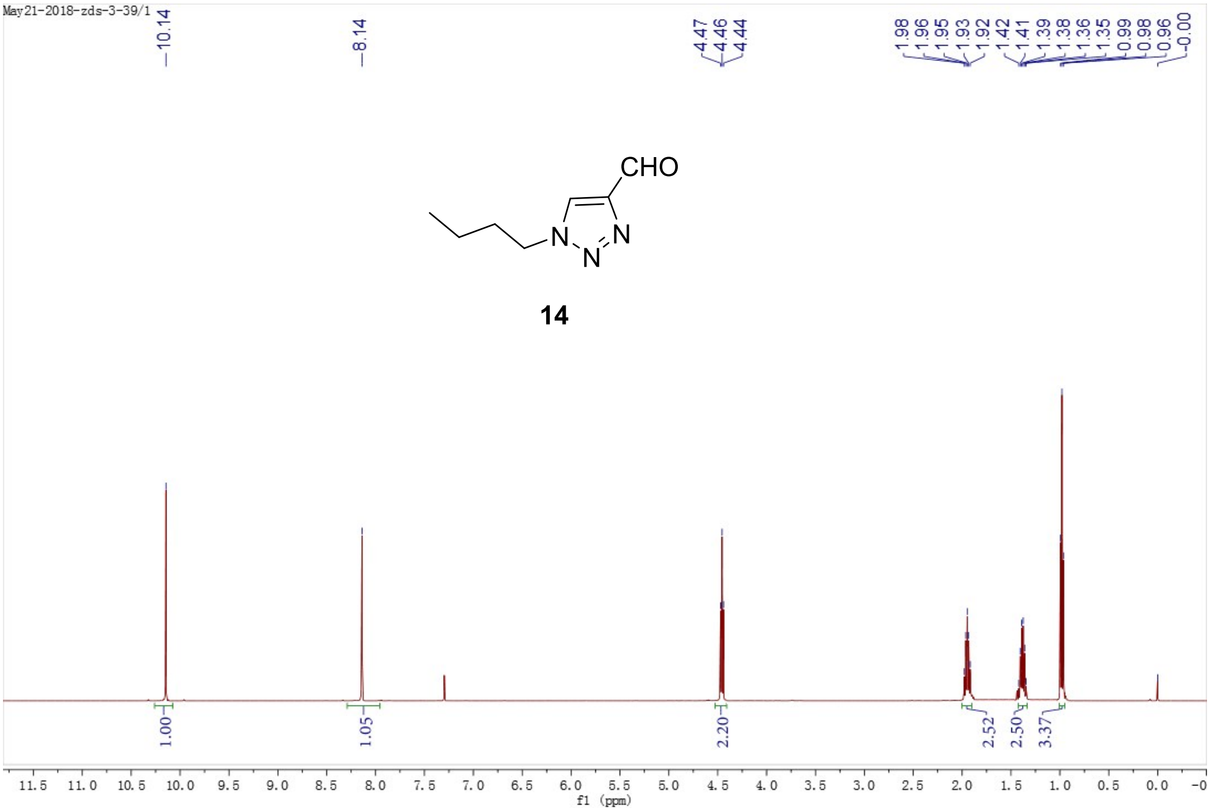

May06-2018-ssz-2-023cnmr/2
185.34
147.60
123.89
77.16
62.48
33.51
24.03
CHO
N
N
N
11
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

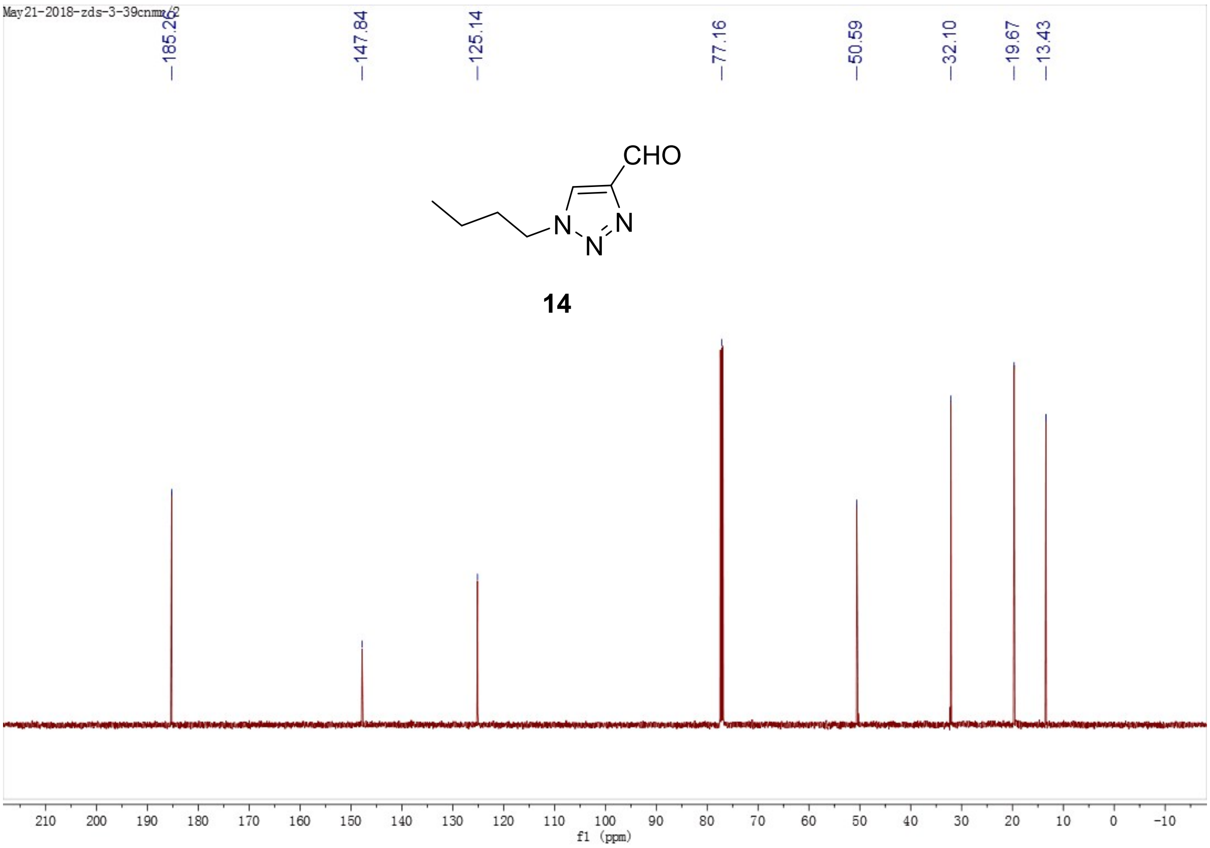

May06-2018-ssz-2-024/1
10.14
8.17
4.56
4.54
4.53
2.28
2.25
1.98
1.94
1.80
1.77
1.52
1.49
1.33
1.31
-0.00
CHO
N
N
N
12
1.00
1.02
1.04
2.16
2.23
3.15
2.14
1.18
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

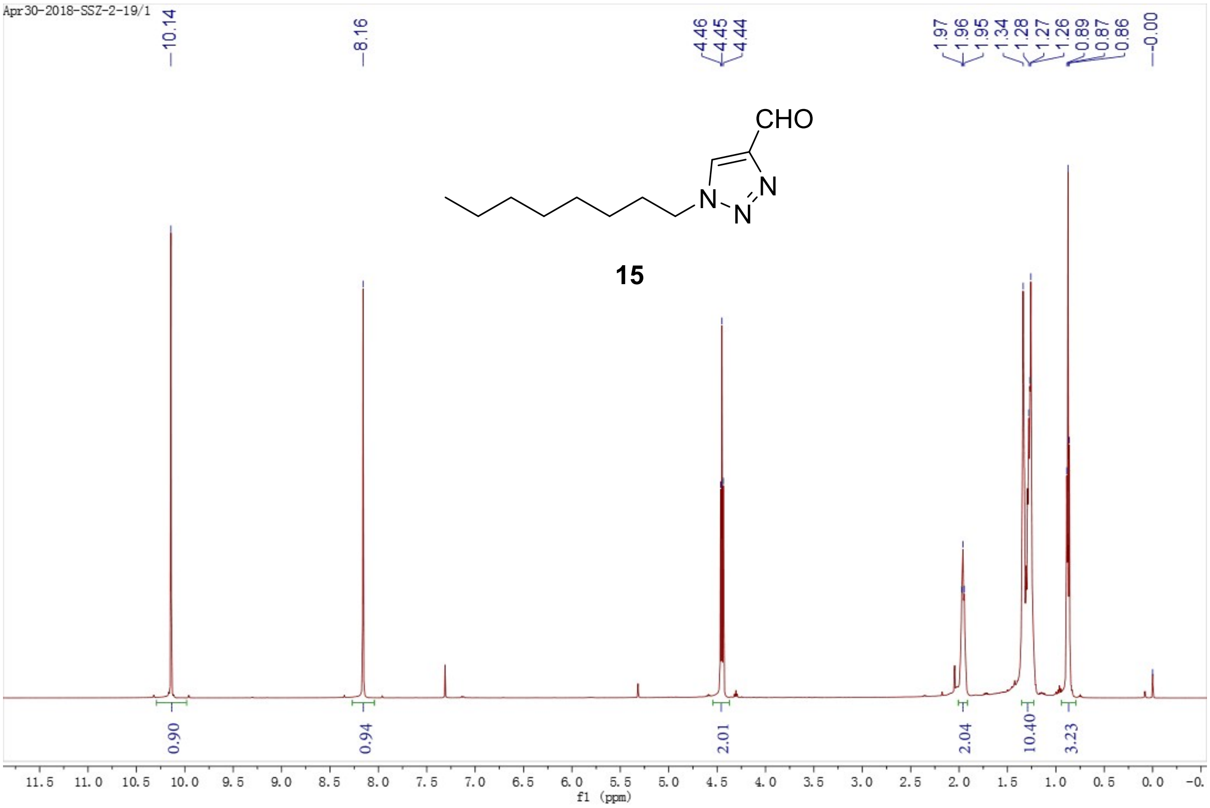

May06-2018-ssz-2-024cnmr/2
185.30
147.47
123.15
77.16
60.66
33.44
25.05
25.00
CHO
N
N
N
12
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


May27-2018-SSZ-2-011/1
10.12
8.00
7.40
7.38
7.30
7.30
5.91
5.89
5.88
5.86
2.04
2.02
0.00
CHO
N
N
N
13
1.00
1.03
3.24
2.08
1.05
3.19
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


May27-2018-SSZ-2-011CNMR/2
185.30
147.73
138.70
129.41
129.22
126.77
124.21
77.16
61.06
21.30
CHO
N
N
N
13
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

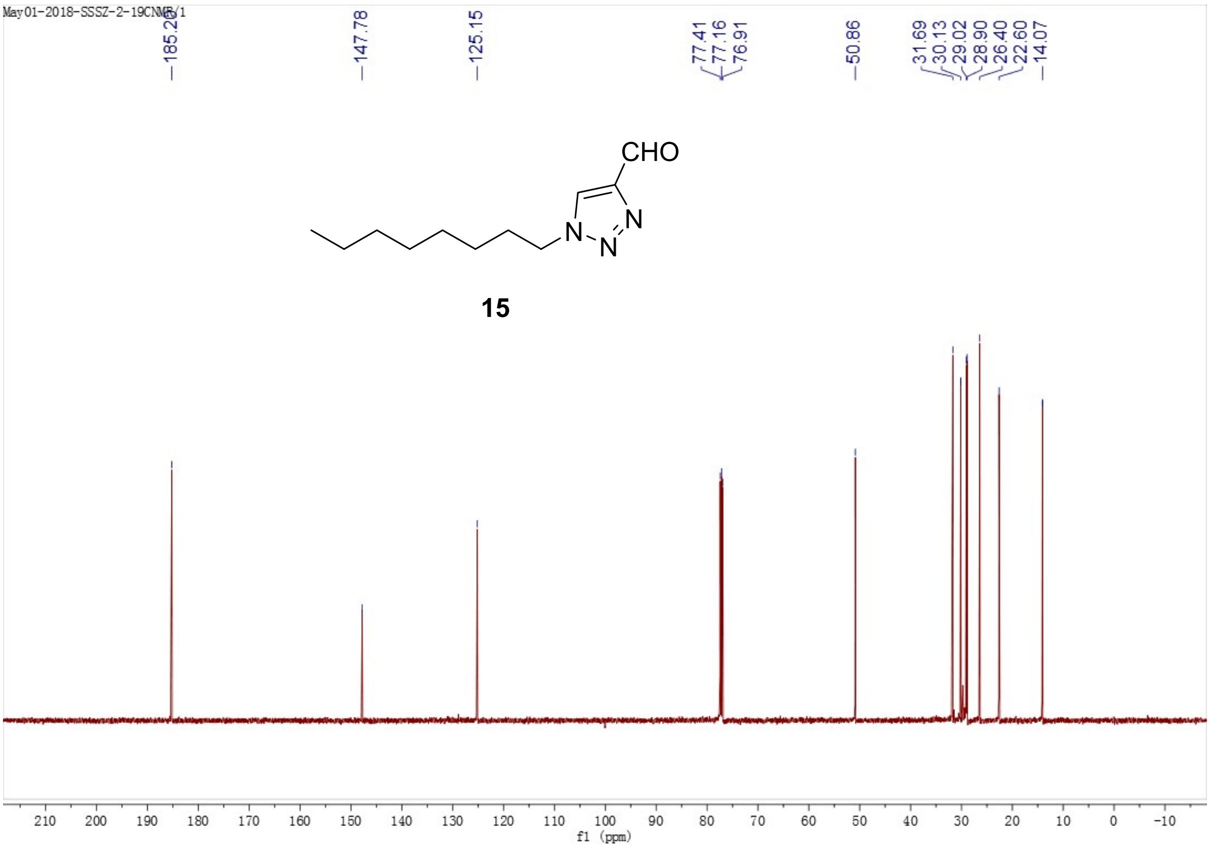

May21-2018-zds-3-39/1
10.14
8.14
4.47
4.46
4.44
1.98
1.96
1.95
1.93
1.92
1.42
1.41
1.39
1.38
1.36
1.35
0.99
0.98
0.96
-0.00
CHO
N
N
N
14
1.00
1.05
2.20
2.52
2.50
3.37
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0
f1 (ppm)


May21-2018-zds-3-39cnmr/2
185.26
147.84
125.14
77.16
50.59
32.10
19.67
13.43
CHO
N
N
N
14
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



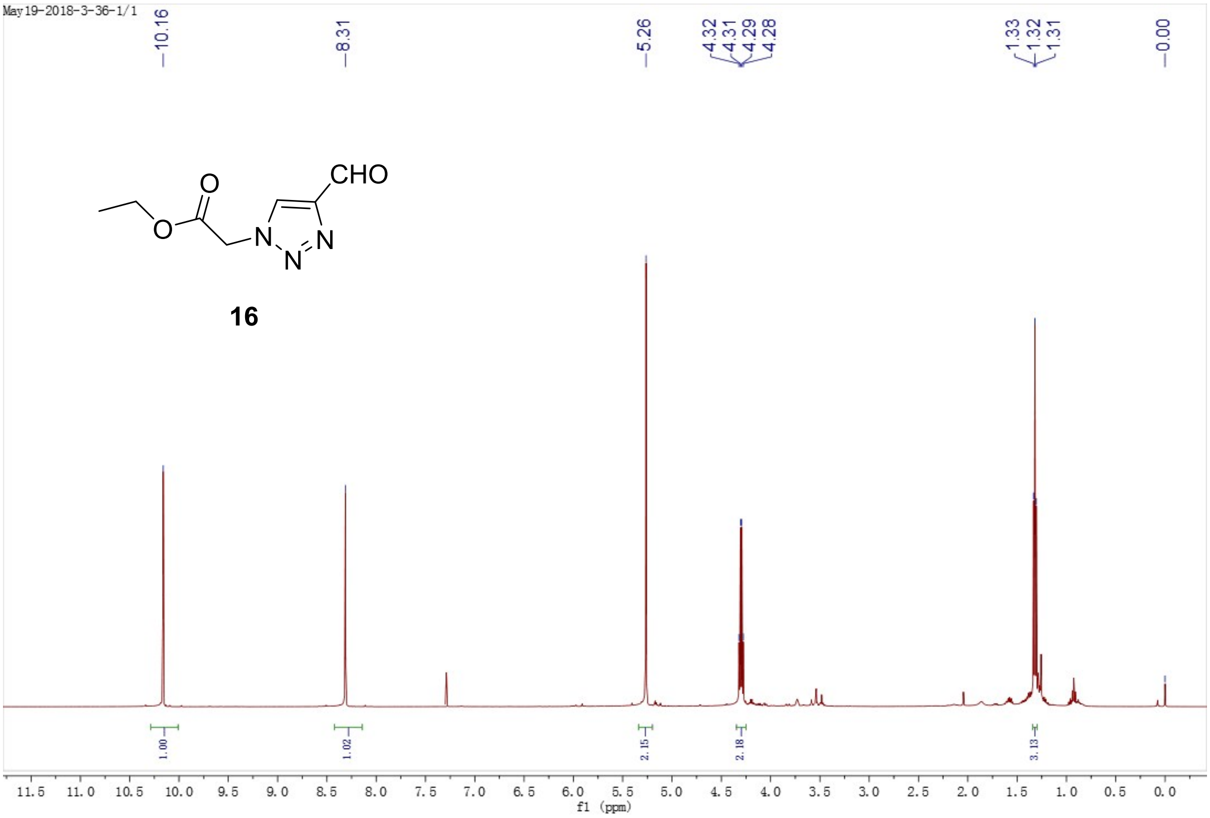
Apr30-2018-SSZ-2-19/1
10.14
8.16
4.46
4.45
4.44
1.97
1.96
1.95
1.34
1.28
1.27
1.26
0.89
0.87
0.86
-0.00
CHO
N
N
N
0.90
0.94
2.01
2.04
10.40
3.23
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.
f1 (ppm)

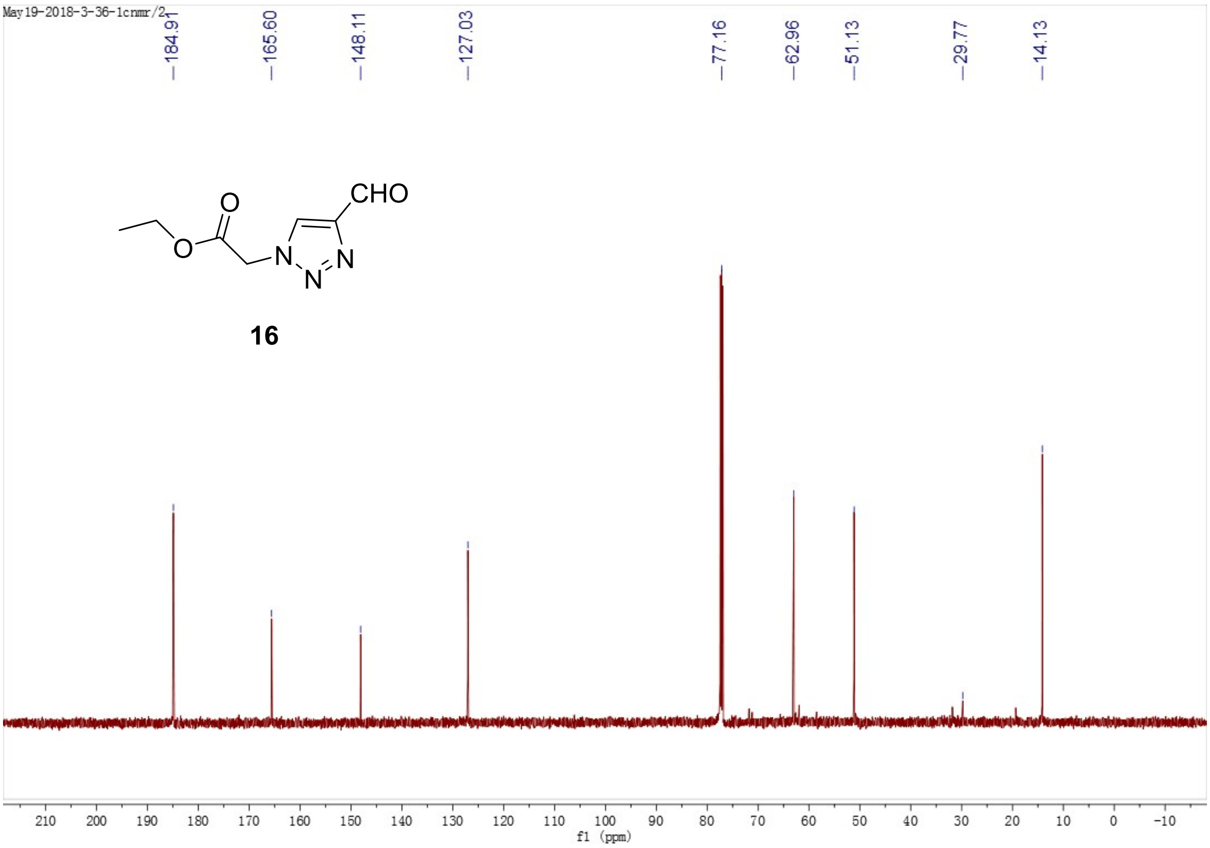
15

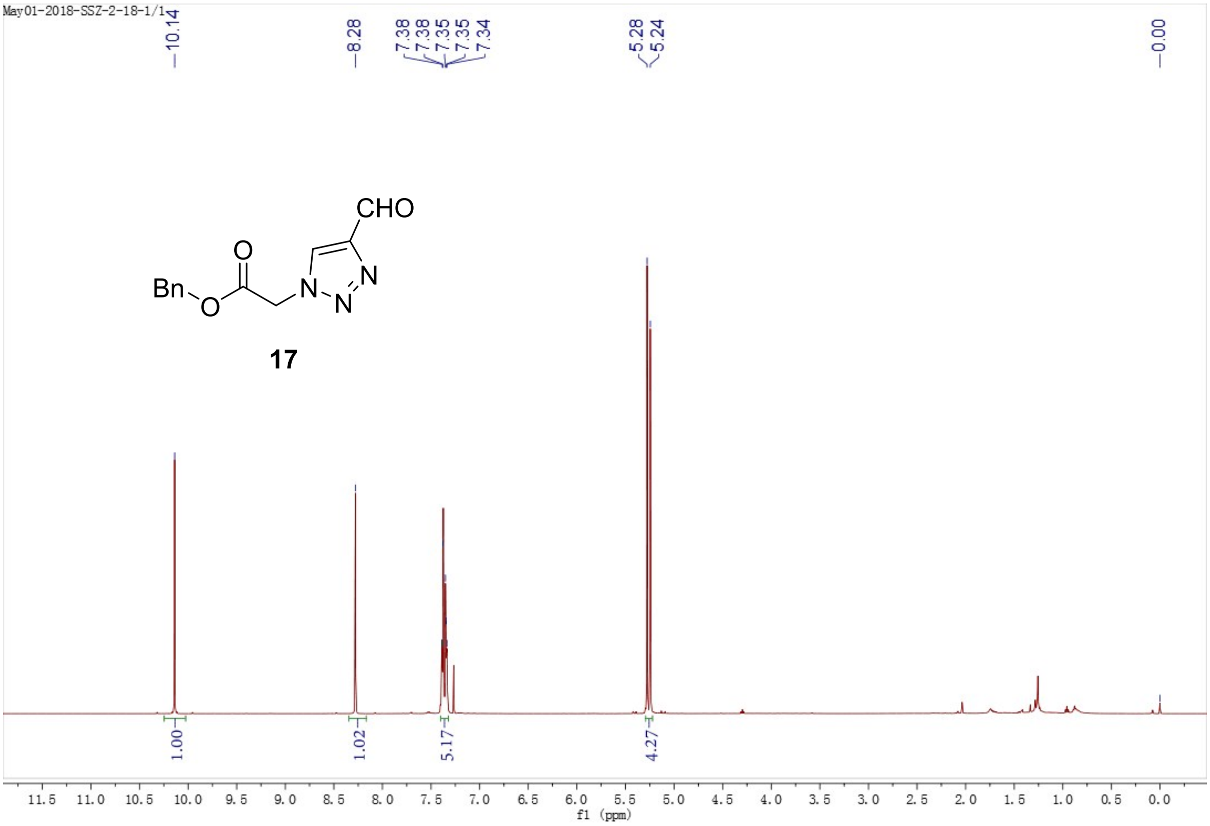


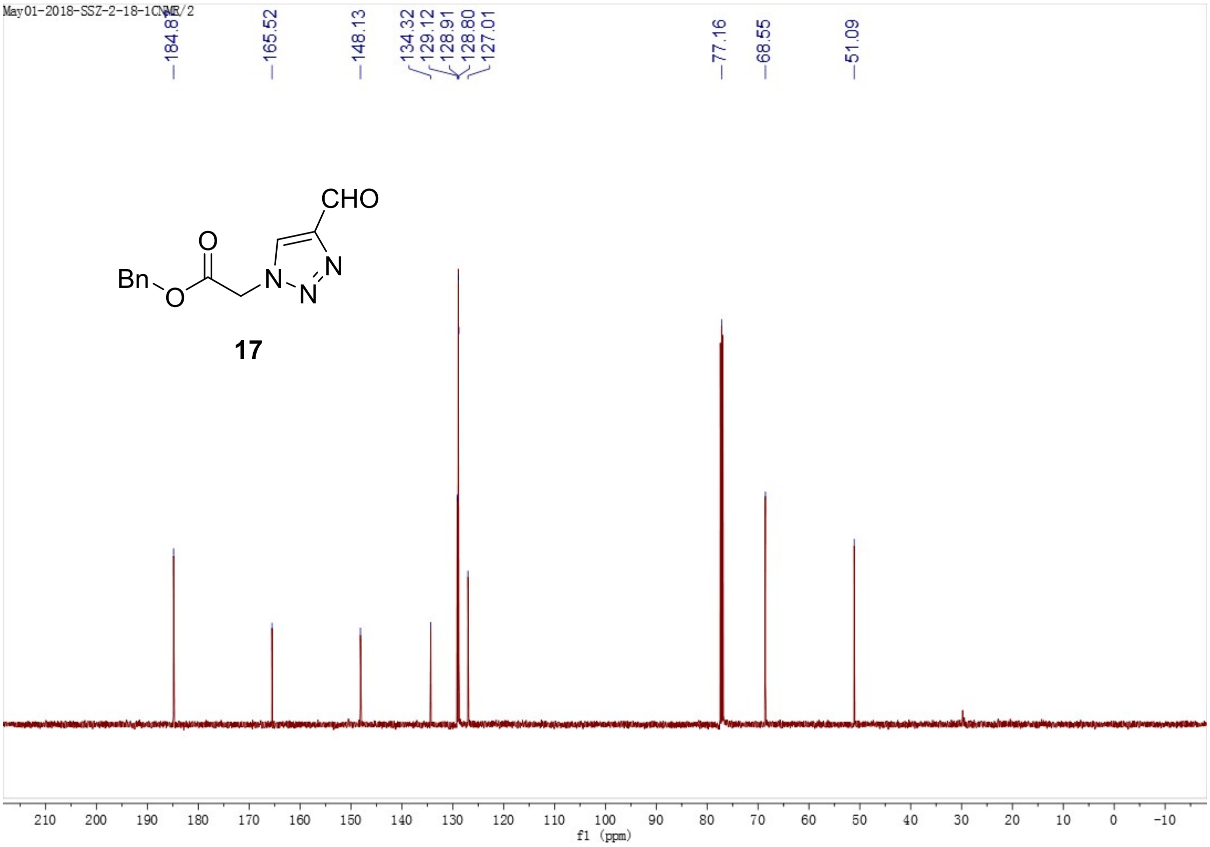
May01-2018-SSSZ-2-19CNMR/1
185.26
147.78
125.15
77.41
77.16
76.91
50.86
31.69
30.13
29.02
28.90
26.40
22.60
14.07
CHO
N
N
N
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

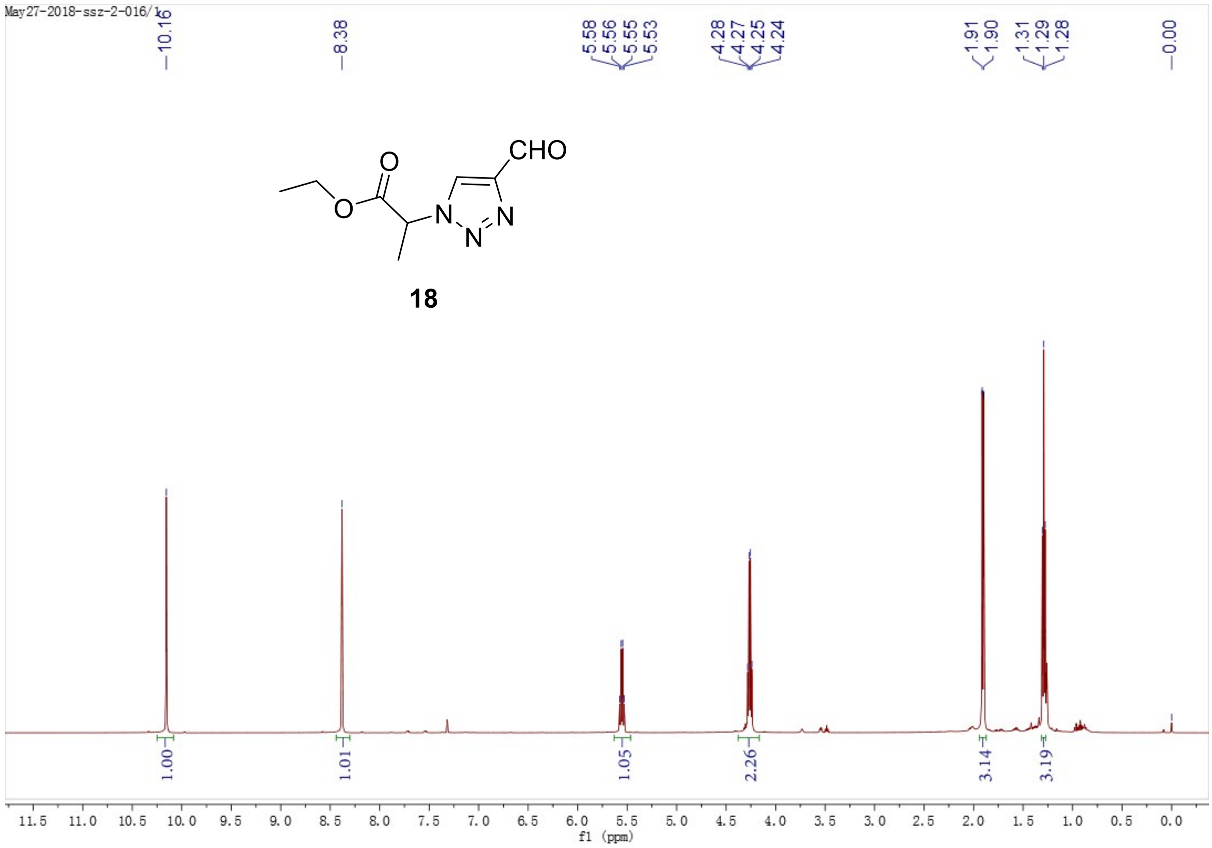
15


16
May19-2018-3-36-1/1
10.16
8.31
5.26
4.32
4.31
4.29
4.28
1.33
1.32
1.31
0.00
CHO
O
O
N
N
N
1.00
1.02
2.15
2.18
3.13
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

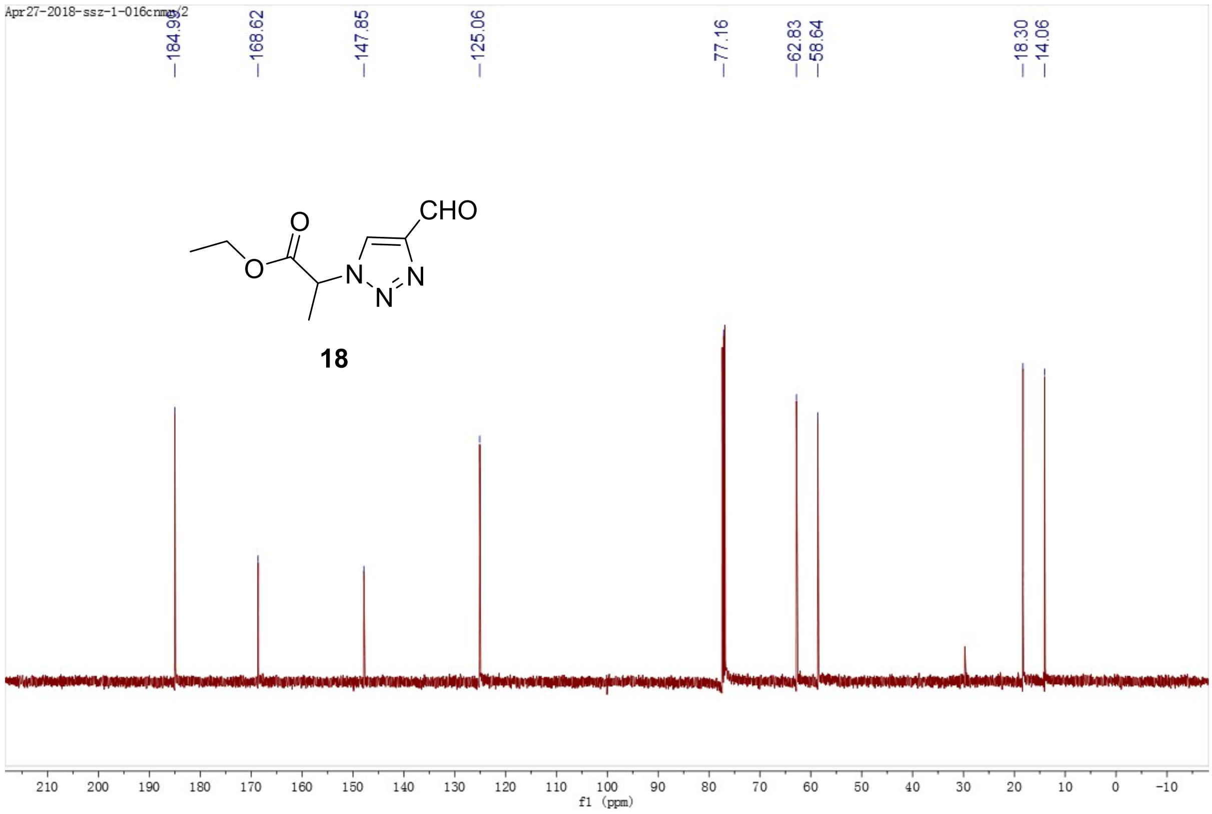

16
May19-2018-3-36-1cnmr/2
184.91
165.60
148.11
127.03
77.16
62.96
51.13
29.77
14.13
CHO
O
O
N
N
N
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


May01-2018-SSZ-2-18-1/1
10.14
8.28
7.38
7.38
7.35
7.35
7.34
5.28
5.24
0.00
CHO
O
Bn
O
N
N
N
17
1.00
1.02
5.17
4.27
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

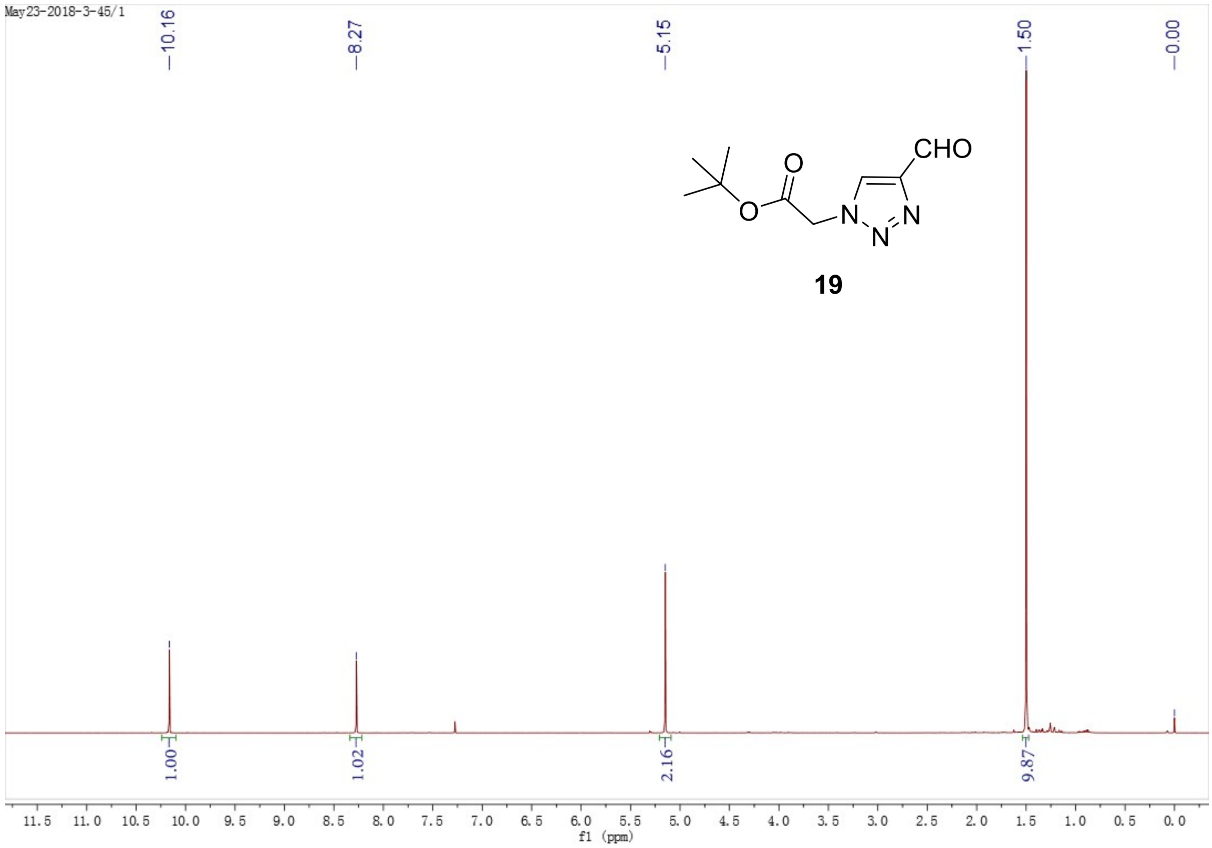

May01-2018-SSZ-2-18-1CNMR/2
184.87
165.52
148.13
134.32
129.12
128.91
128.80
127.01
77.16
68.55
51.09
CHO
O
Bn
O
N
N
N
17
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

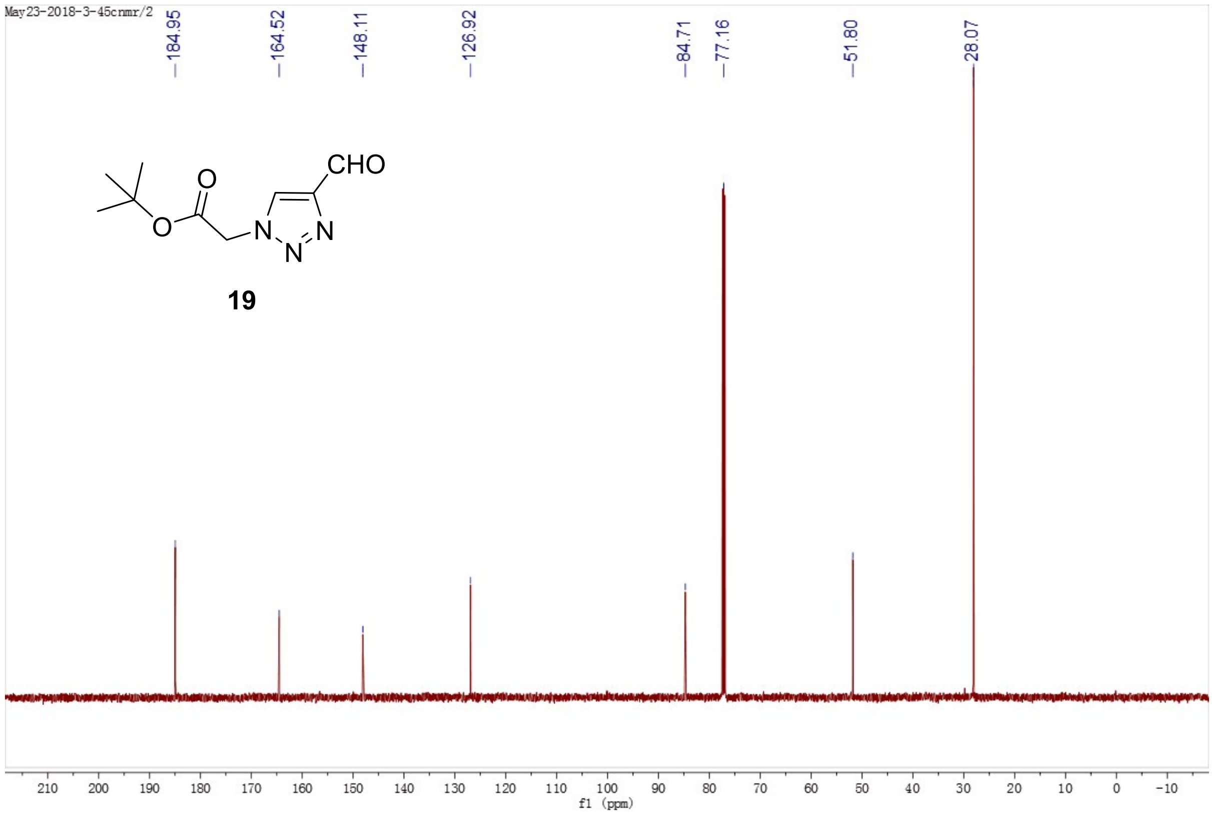

May27-2018-ssz-2-016/1
10.16
8.38
5.58
5.56
5.55
5.53
4.28
4.27
4.25
4.24
1.91
1.90
1.31
1.29
1.28
0.00
CHO
O
O
N
N
N
1.00
1.01
1.05
2.26
3.14
3.19
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

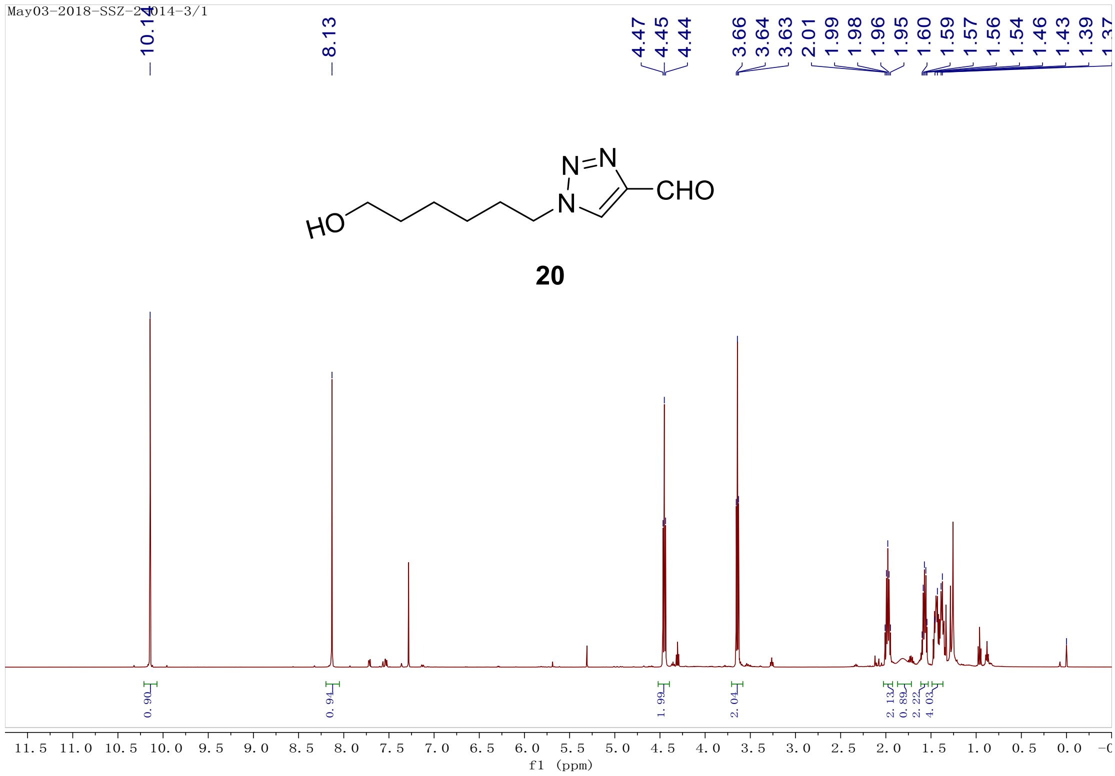
18

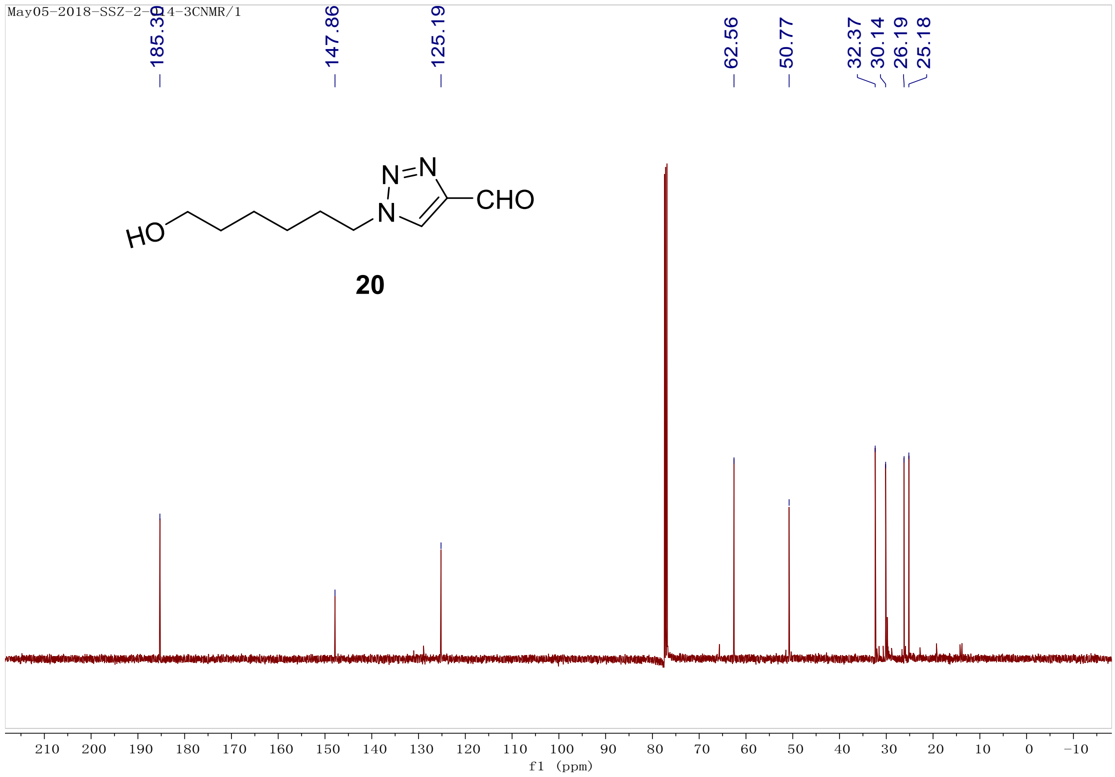

Apr27-2018-ssz-1-016cnmr/2
184.96
168.62
147.85
125.06
77.16
62.83
58.64
18.30
14.06
CHO
O
O
N
N
N
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

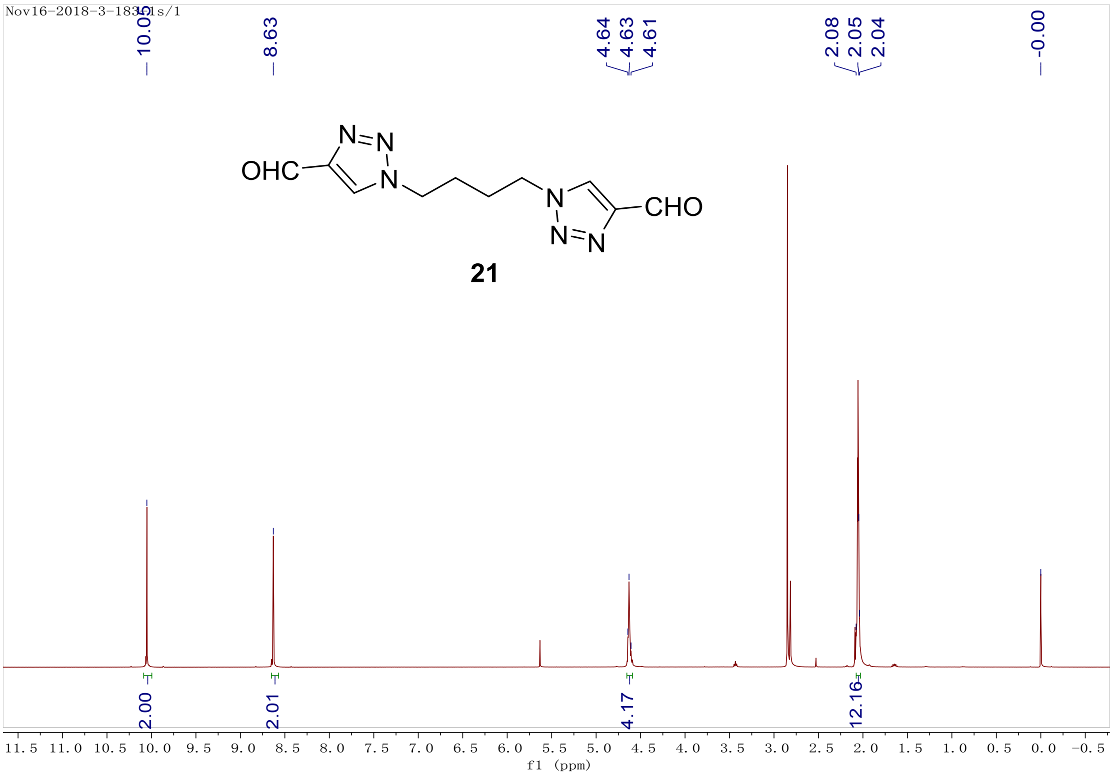
18

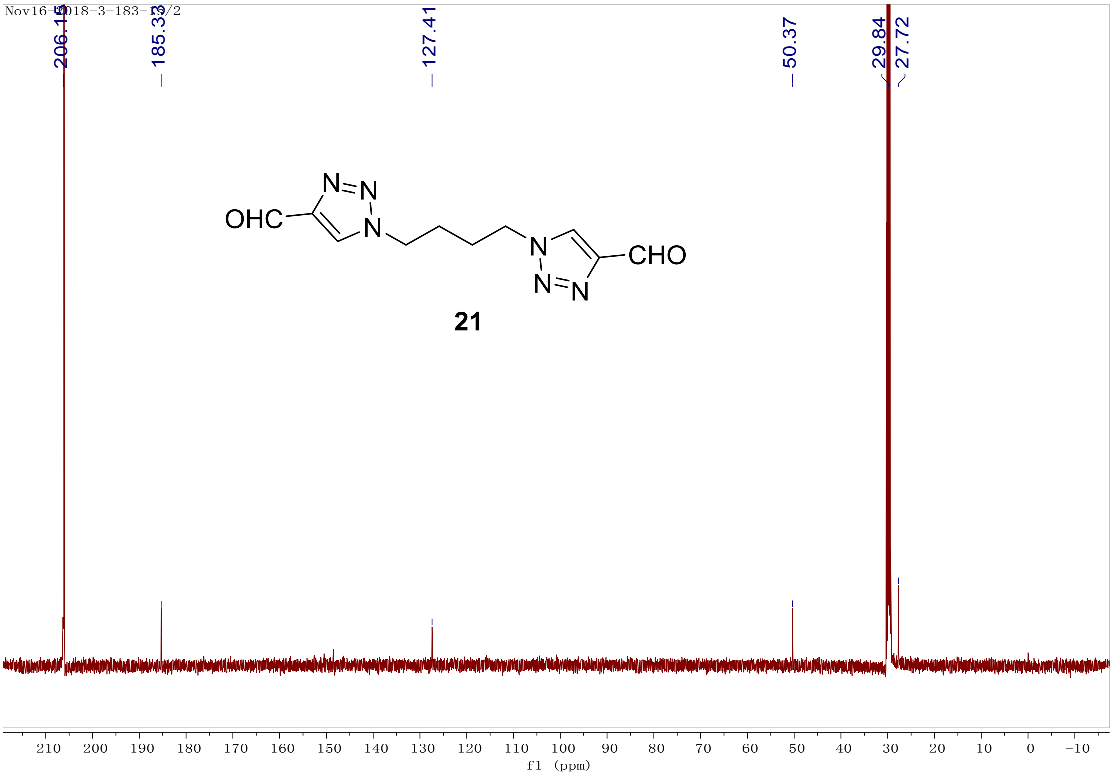

May23-2018-3-45/1
10.16
8.27
5.15
1.50
0.00
CHO
O
O
N
N
N
19
1.00
1.02
2.16
9.87
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

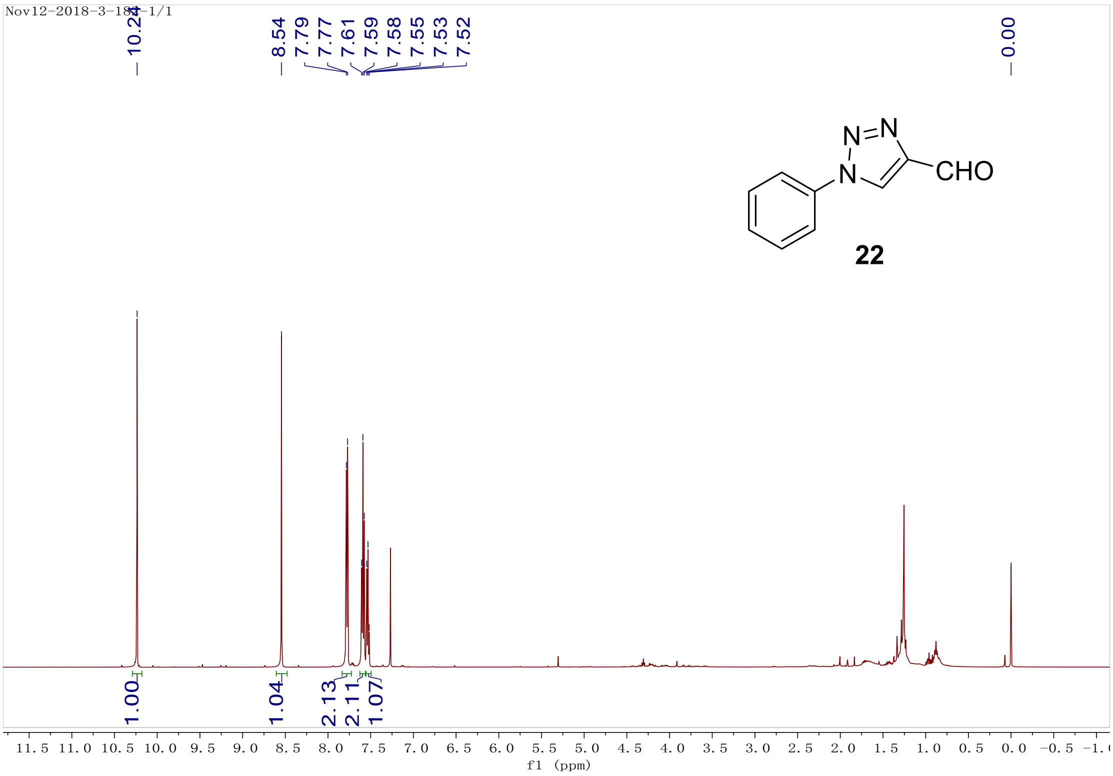

May23-2018-3-45cnmr/2
184.95
164.52
148.11
126.92
84.71
77.16
51.80
28.07
CHO
O
O
N
N
N
19
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

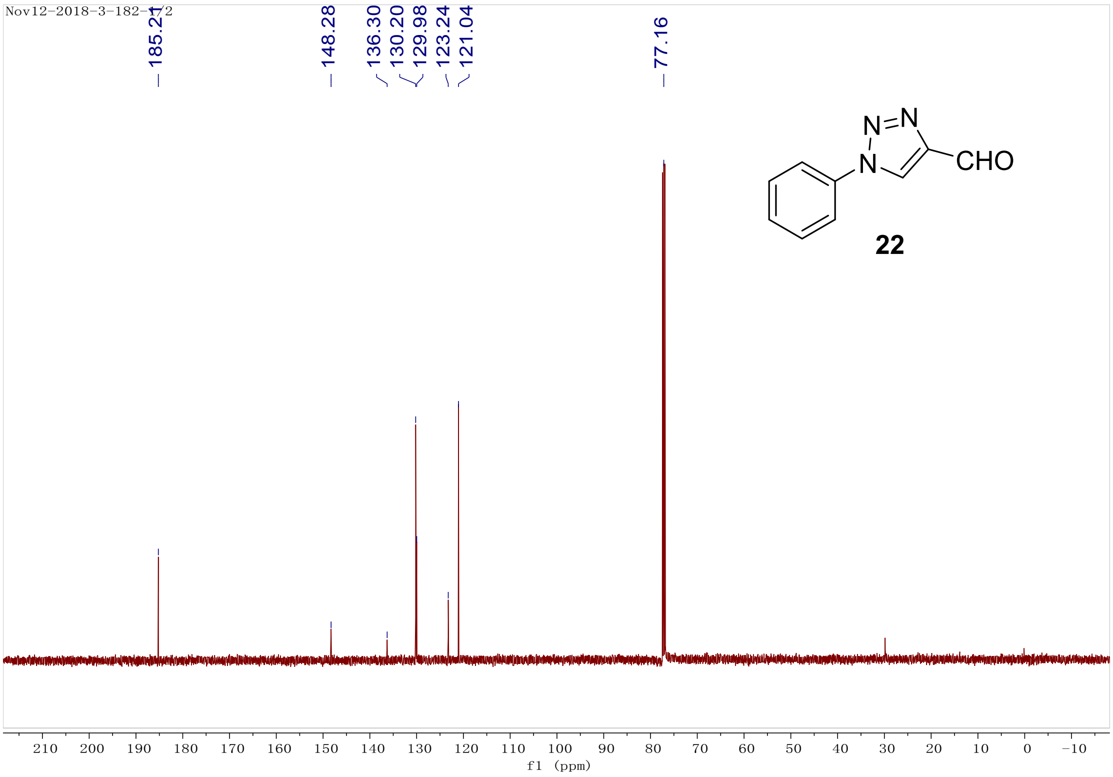

May03-2018-SSZ-2-014-3/1
10.14
8.13
4.47
4.45
4.44
3.66
3.64
3.63
2.01
1.99
1.98
1.96
1.95
1.60
1.59
1.57
1.56
1.54
1.46
1.43
1.39
1.37
N=N
N
CHO
HO
20
0.90
0.94
1.99
2.04
2.13
0.89
2.22
4.03
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

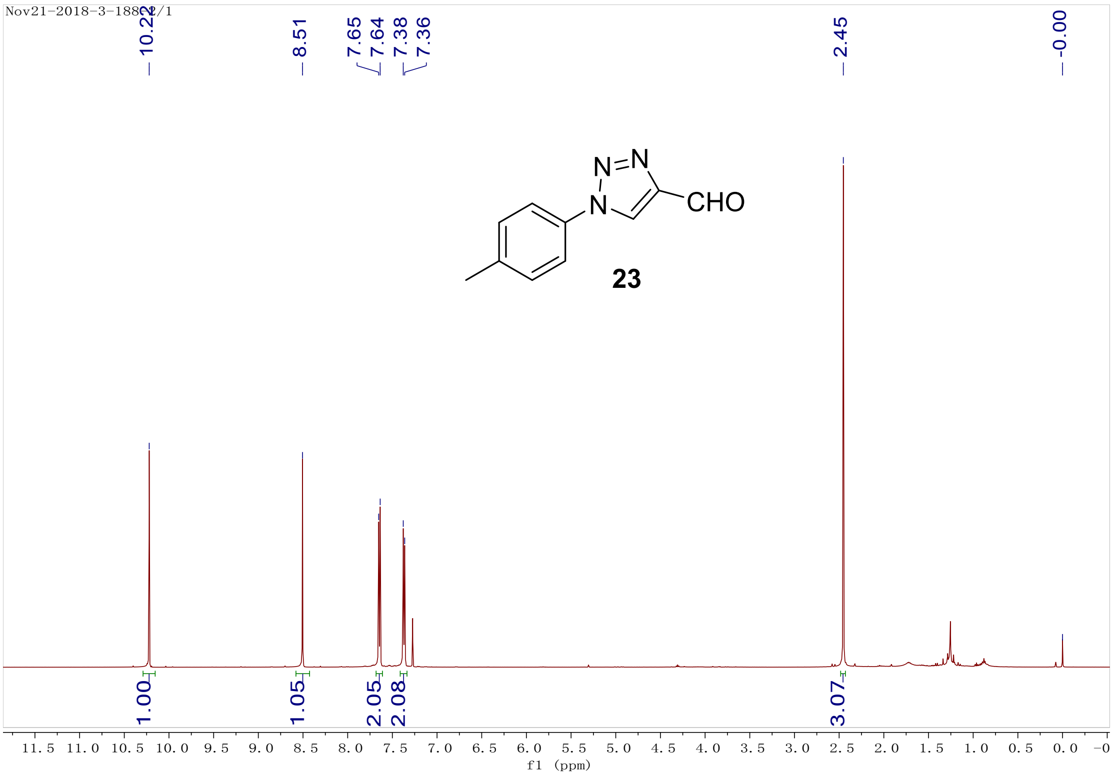

May05-2018-SSZ-2-014-3CNMR/1
185.30
147.86
125.19
62.56
50.77
32.37
30.14
26.19
25.18
N=N
N
CHO
HO
20
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

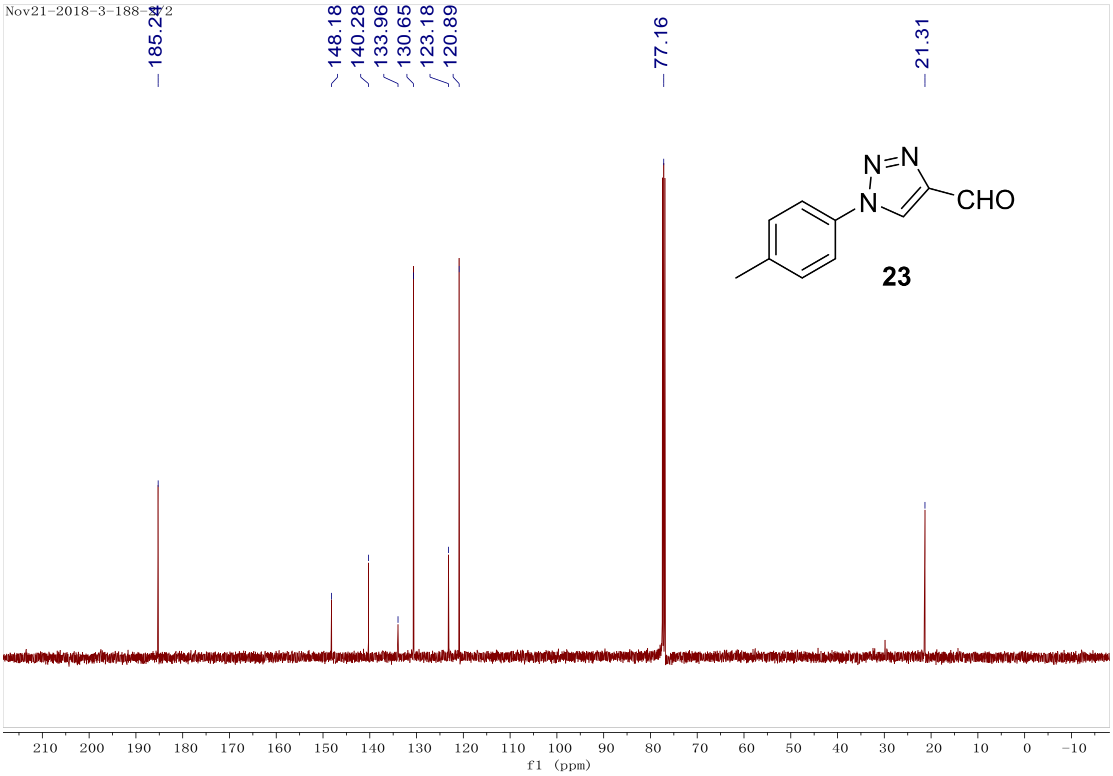

Nov16-2018-3-183-1s/1
10.05
8.63
4.64
4.63
4.61
2.08
2.05
2.04
-0.00
OHC
N
N
N
N
N
N
CHO
21
2.00
2.01
4.17
12.16
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

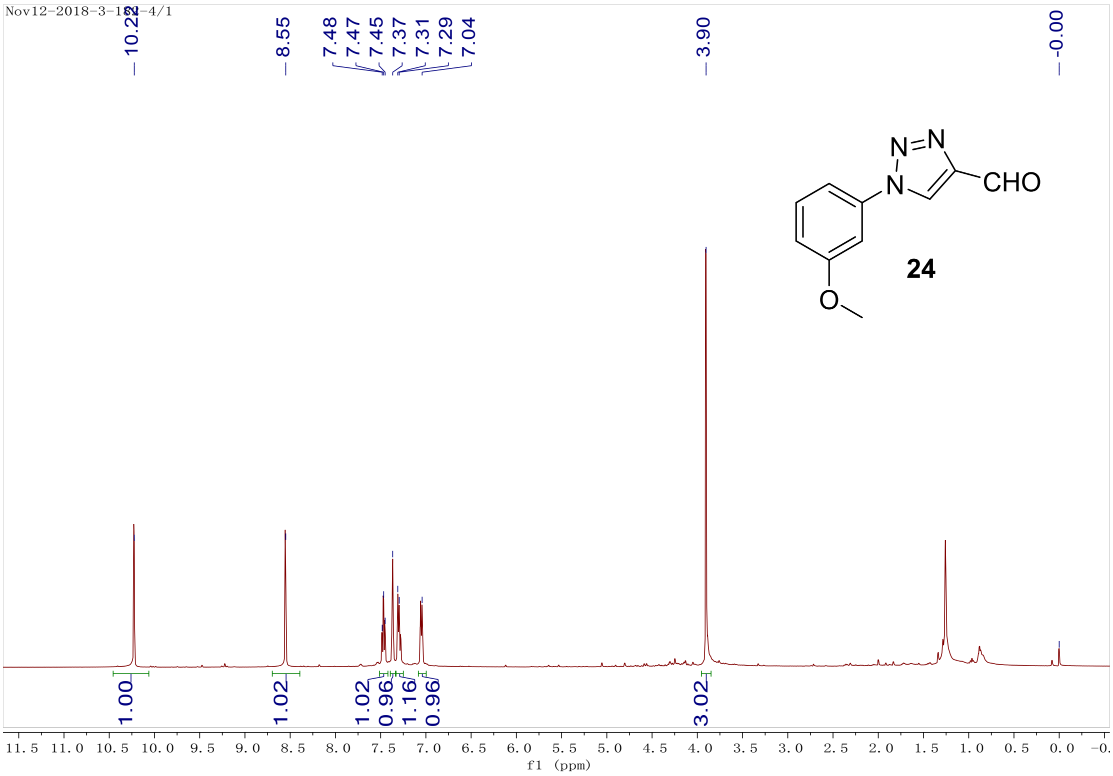

Nov16-2018-3-183-1s/2
206.15
185.37
127.41
50.37
29.84
27.72
OHC
N
N
N
N
N
N
CHO
21
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

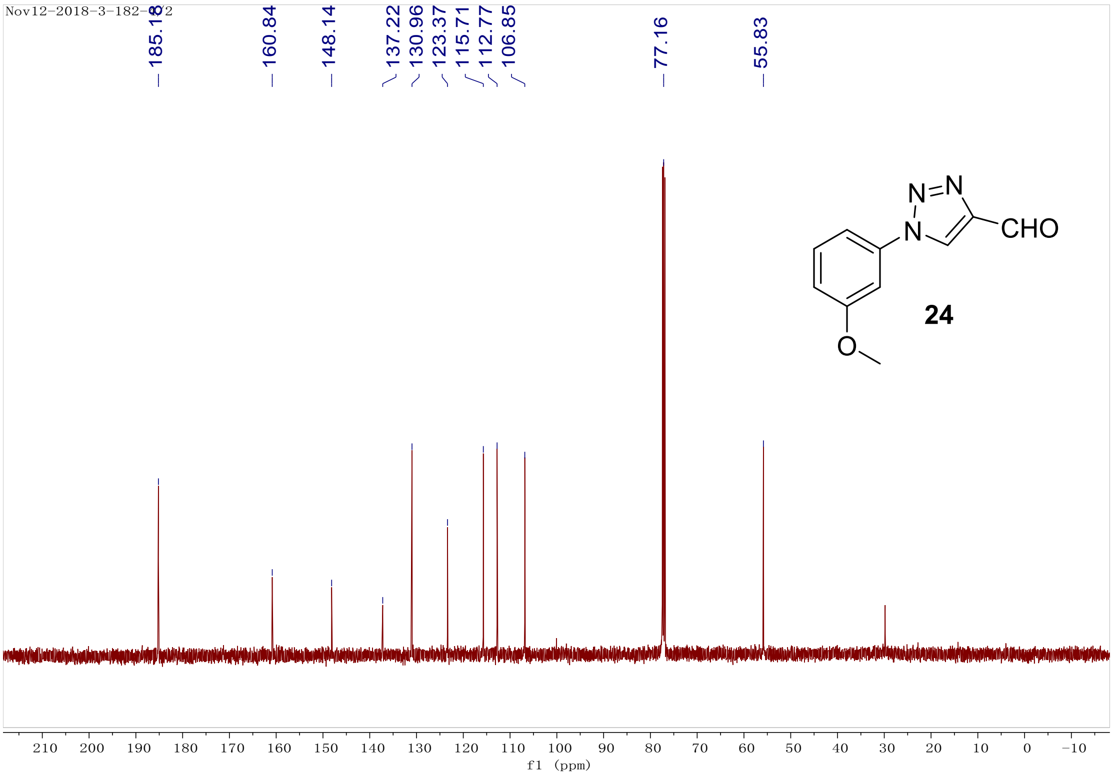

Nov12-2018-3-182-1/1
10.24
8.54
7.79
7.77
7.61
7.59
7.58
7.55
7.53
7.52
0.00
N=N
N
CHO
22
1.00
1.04
2.13
2.11
1.07
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

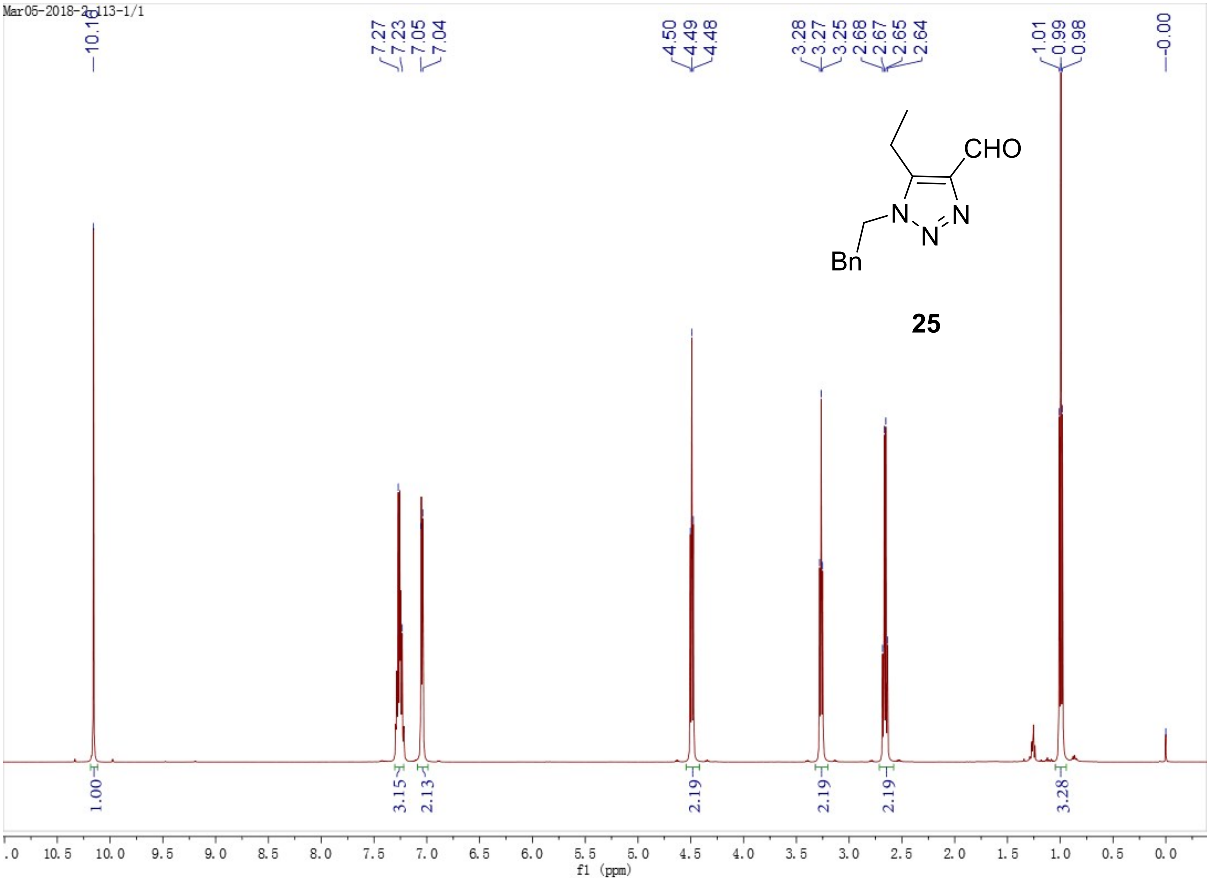

Nov12-2018-3-182-1/2
185.21
148.28
136.30
130.20
129.98
123.24
121.04
77.16
N=N
N
CHO
22
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

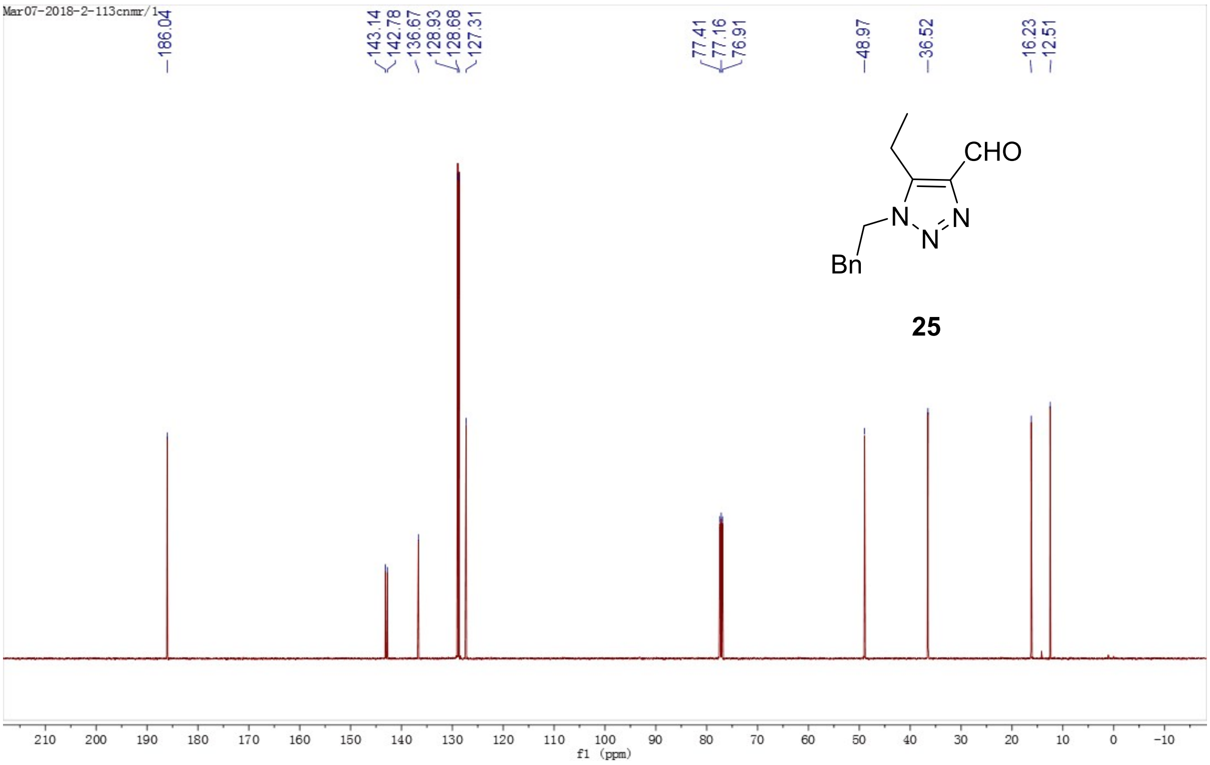

Nov21-2018-3-188-2/1
10.22
8.51
7.65
7.64
7.38
7.36
2.45
-0.00
CHO
23
1.00
1.05
2.05
2.08
3.07
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0
f1 (ppm)

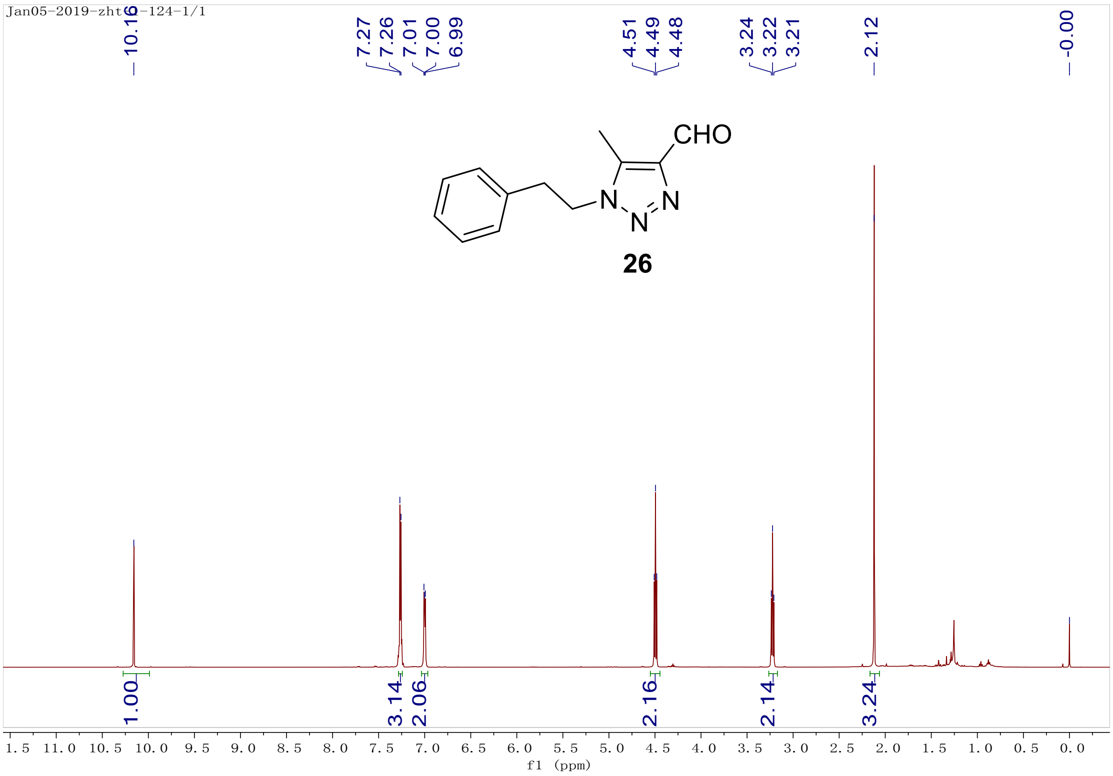

Nov21-2018-3-188-2/2
185.24
148.18
140.28
133.96
130.65
123.18
120.89
77.16
21.31
CHO
23
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

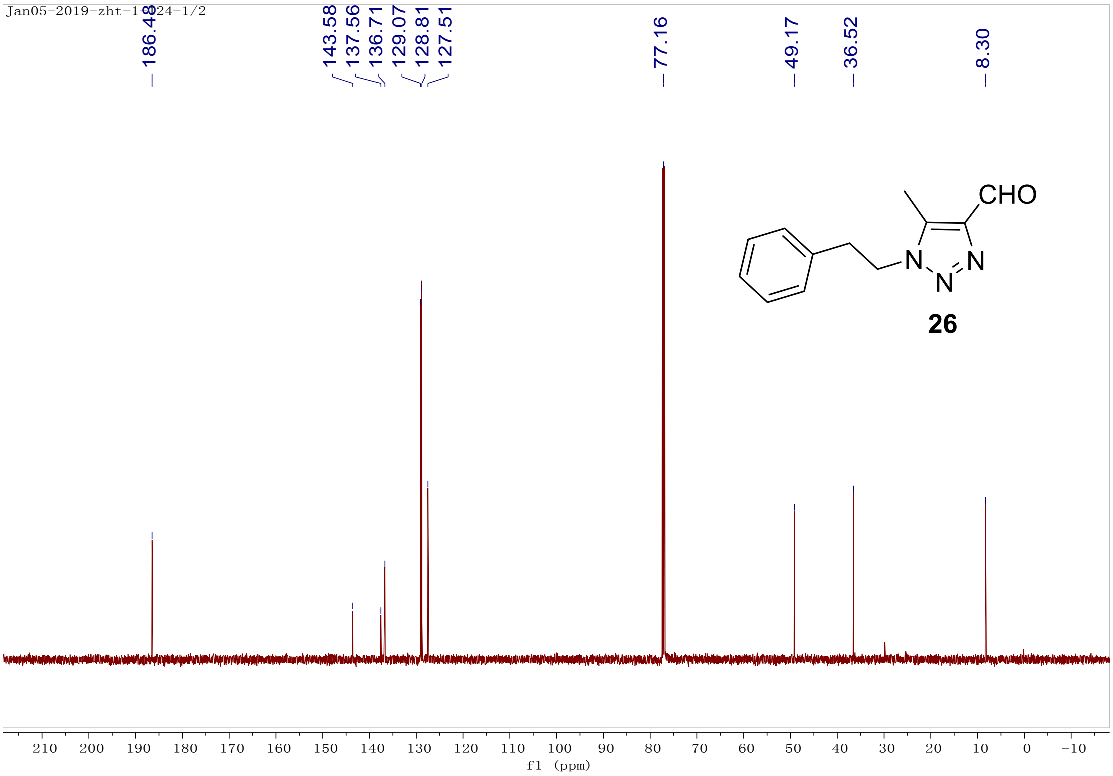

Nov12-2018-3-182-4/1
10.22
8.55
7.48
7.47
7.45
7.37
7.31
7.29
7.04
3.90
-0.00
1.00
1.02
1.02
0.96
1.16
0.96
3.02
CHO
N
N
N
O
24
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.
f1 (ppm)

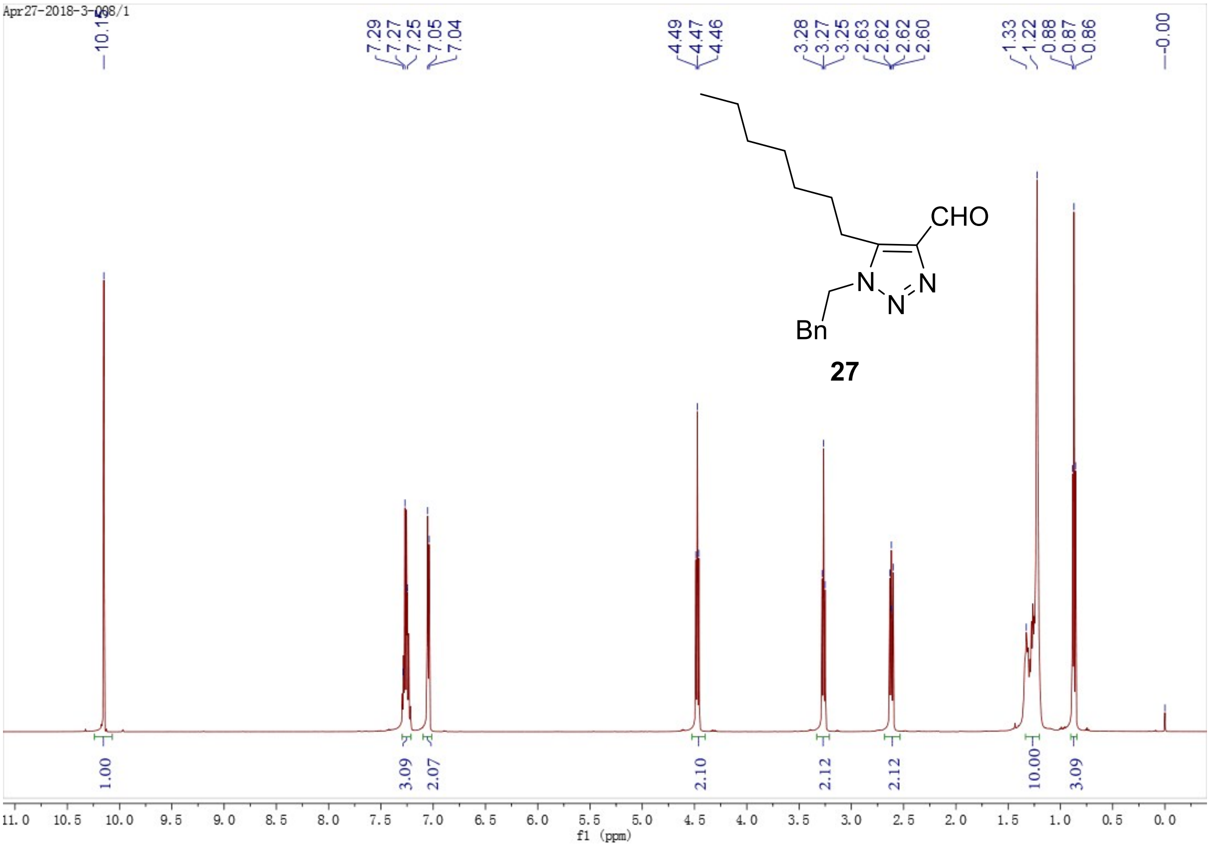

Nov12-2018-3-182-4/2
185.18
160.84
148.14
137.22
130.96
123.37
115.71
112.77
106.85
77.16
55.83
CHO
N
N
N
O
24
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

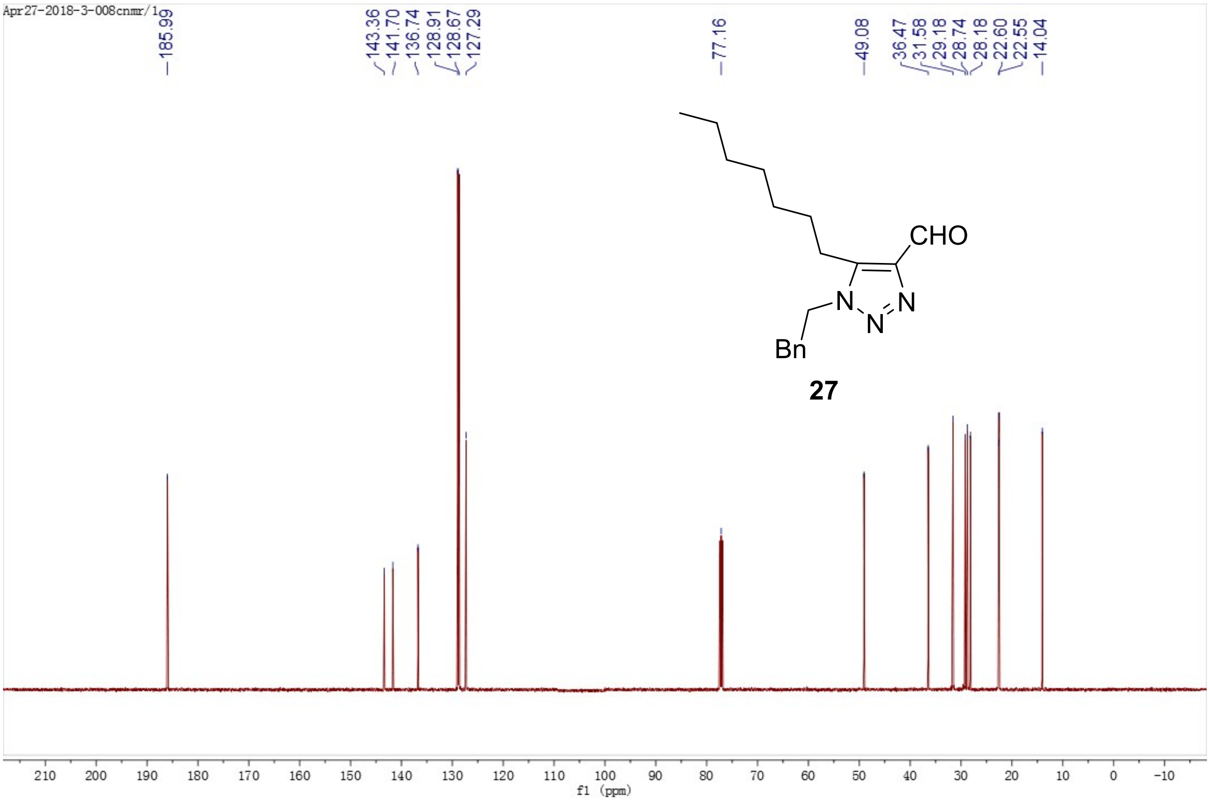

Mar05-2018-2-113-1/1
10.16
7.27
7.23
7.05
7.04
4.50
4.49
4.48
3.28
3.27
3.25
2.68
2.67
2.65
2.64
1.01
0.99
0.98
-0.00
CHO
N
N
N
Bn
25
1.00
3.15
2.13
2.19
2.19
2.19
3.28
.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

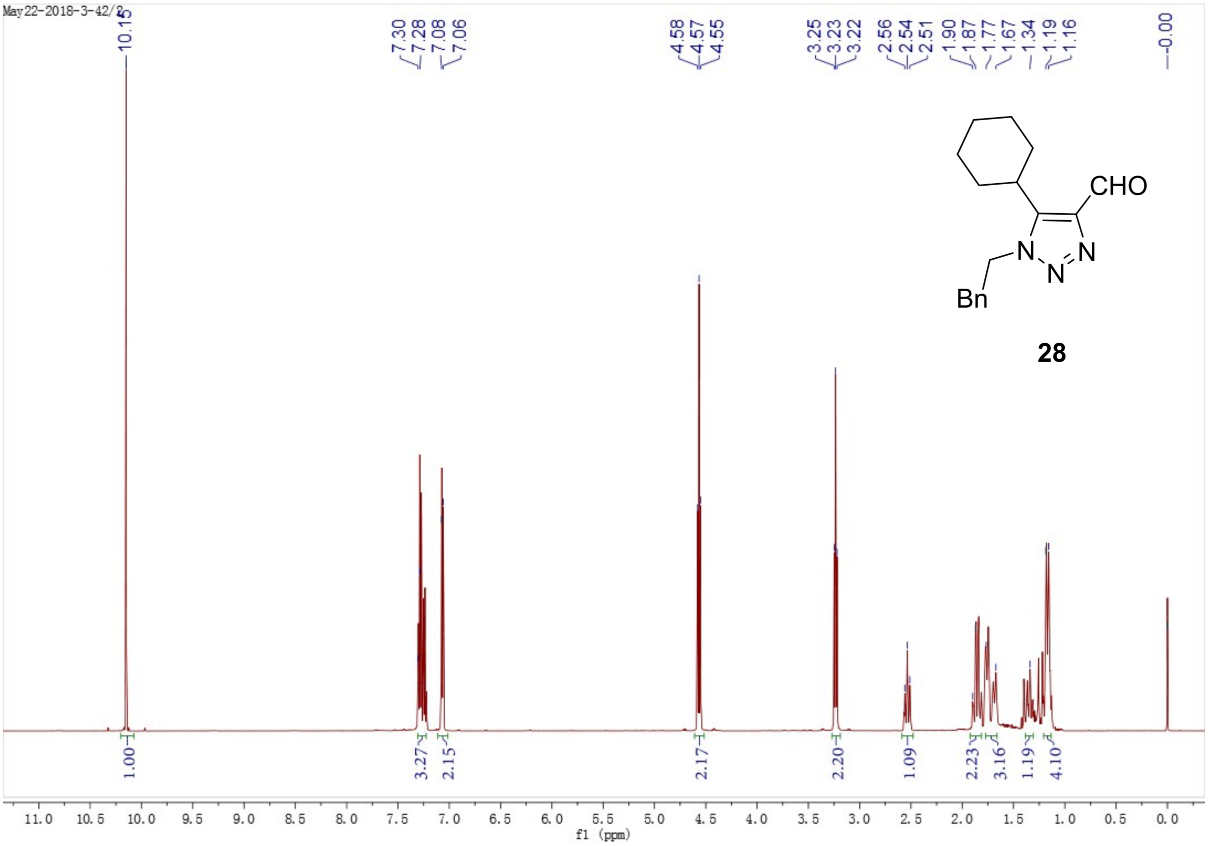

Mar07-2018-2-113cnmr/1
186.04
143.14
142.78
136.67
128.93
128.68
127.31
77.41
77.16
76.91
48.97
36.52
16.23
12.51
CHO
N
N
N
Bn
25
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

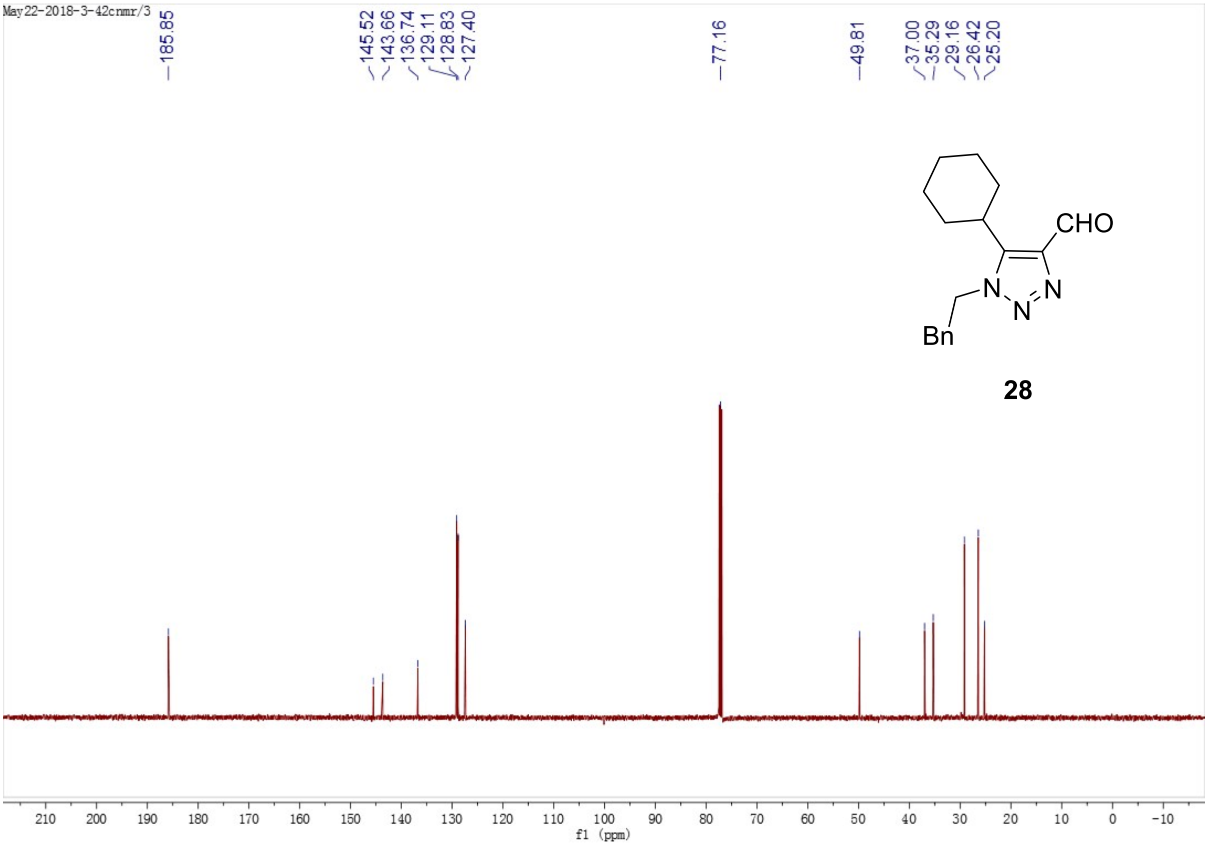

Jan05-2019-zht-1-124-1/1
10.16
7.27
7.26
7.01
7.00
6.99
4.51
4.49
4.48
3.24
3.22
3.21
2.12
-0.00
CHO
N
N
N
26
1.00
3.14
2.06
2.16
2.14
3.24
1.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

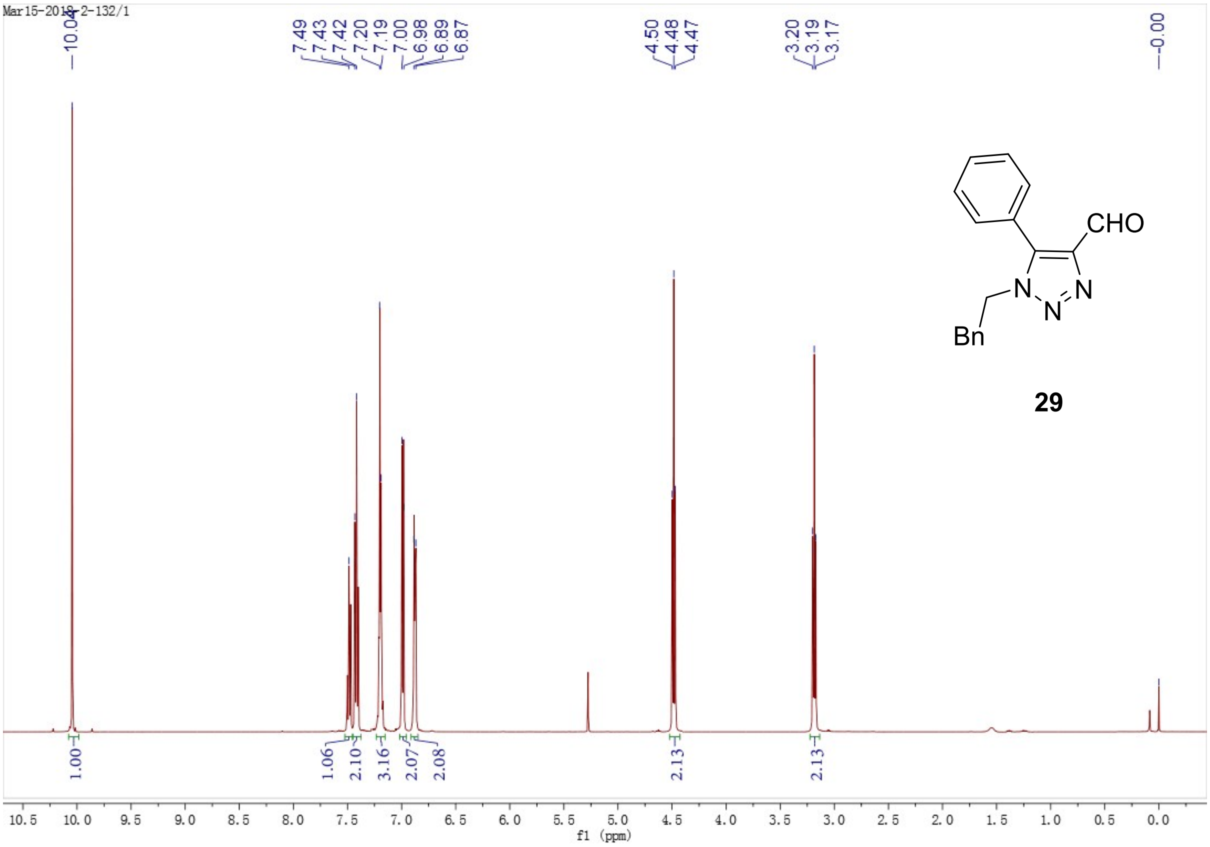

Jan05-2019-zht-1-124-1/2
186.43
143.58
137.56
136.71
129.07
128.81
127.51
77.16
49.17
36.52
8.30
CHO
N
N
N
26
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


Apr27-2018-3-008/1
10.15
7.29
7.27
7.25
7.05
7.04
4.49
4.47
4.46
3.28
3.27
3.25
2.63
2.62
2.62
2.60
1.33
1.22
0.88
0.87
0.86
-0.00
CHO
N
N
N
Bn
27
1.00
3.09
2.07
2.10
2.12
2.12
10.00
3.09
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

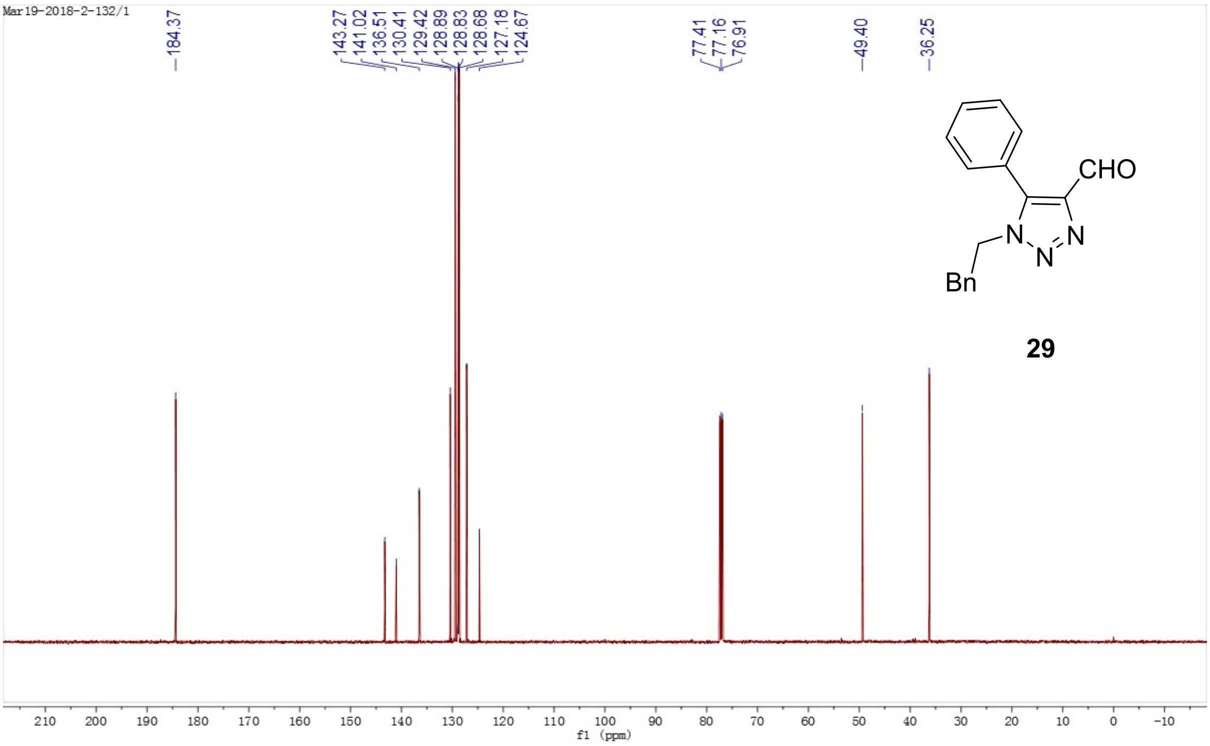

Apr27-2018-3-008cnmr/1
185.99
143.36
141.70
136.74
128.91
128.67
127.29
77.16
49.08
36.47
31.58
29.18
28.74
28.18
22.60
22.55
14.04
CHO
N
N
N
Bn
27
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


May22-2018-3-42/2
10.15
7.30
7.28
7.08
7.06
4.58
4.57
4.55
3.25
3.23
3.22
2.56
2.54
2.51
1.90
1.87
1.77
1.67
1.34
1.19
1.16
-0.00
CHO
N
N
N
Bn
28
1.00
3.27
2.15
2.17
2.20
1.09
2.23
3.16
1.19
4.10
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

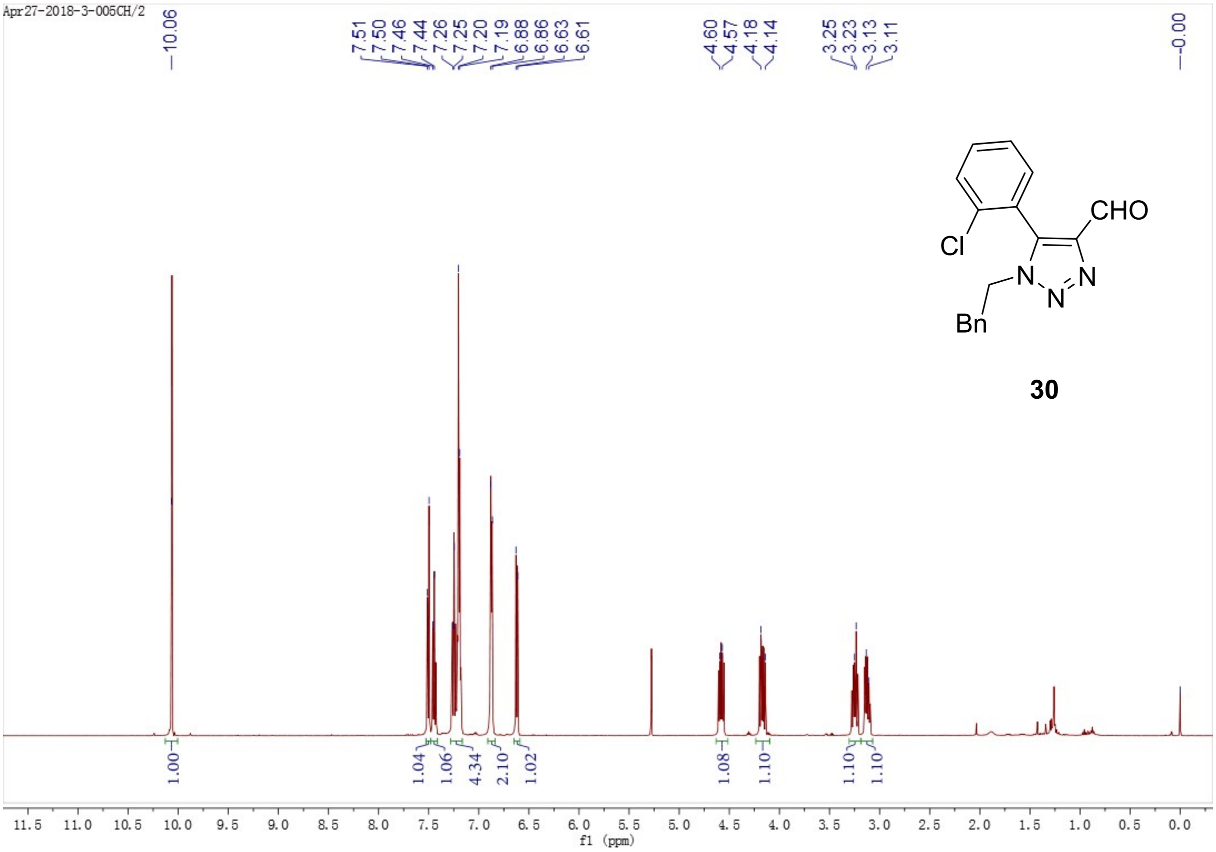

May22-2018-3-42crnmr/3
185.85
145.52
143.66
136.74
129.11
128.83
127.40
77.16
49.81
37.00
35.29
29.16
26.42
25.20
CHO
N
N
N
Bn
28
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

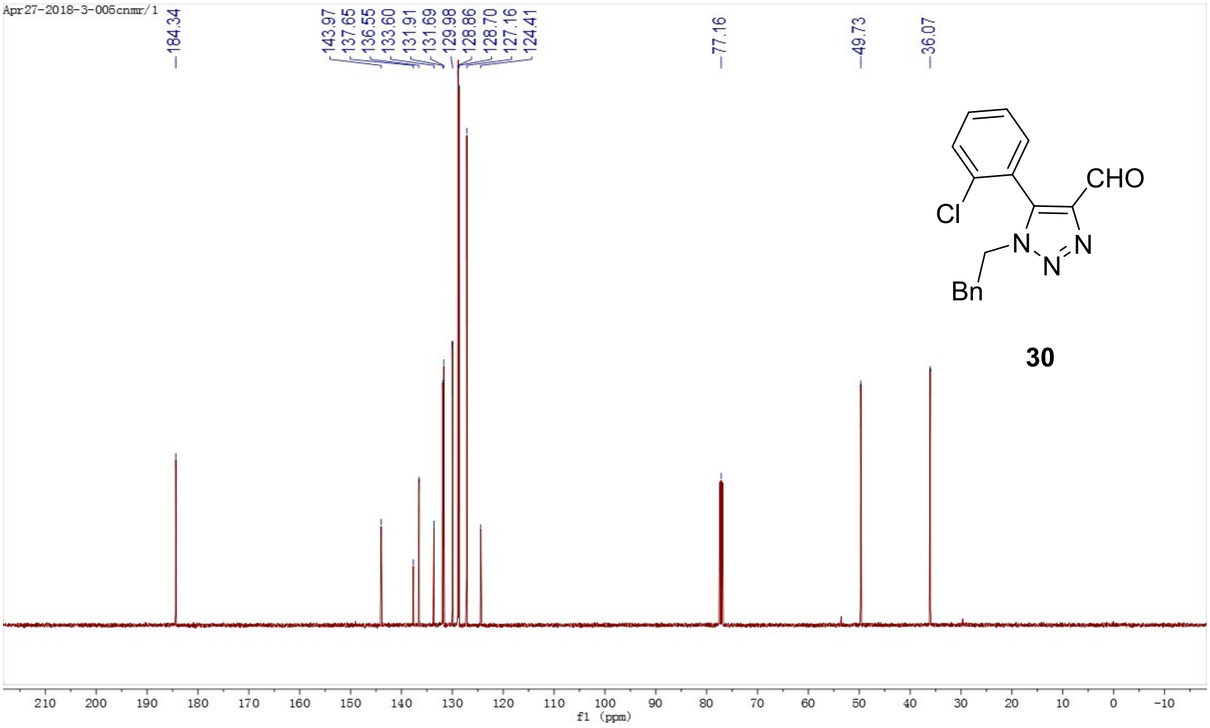

Mar15-2018-2-132/1
10.04
7.49
7.43
7.42
7.20
7.19
7.00
6.98
6.89
6.87
4.50
4.48
4.47
3.20
3.19
3.17
0.00
CHO
N
N
N
Bn
1.00
1.06
2.10
3.16
2.07
2.08
2.13
2.13
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

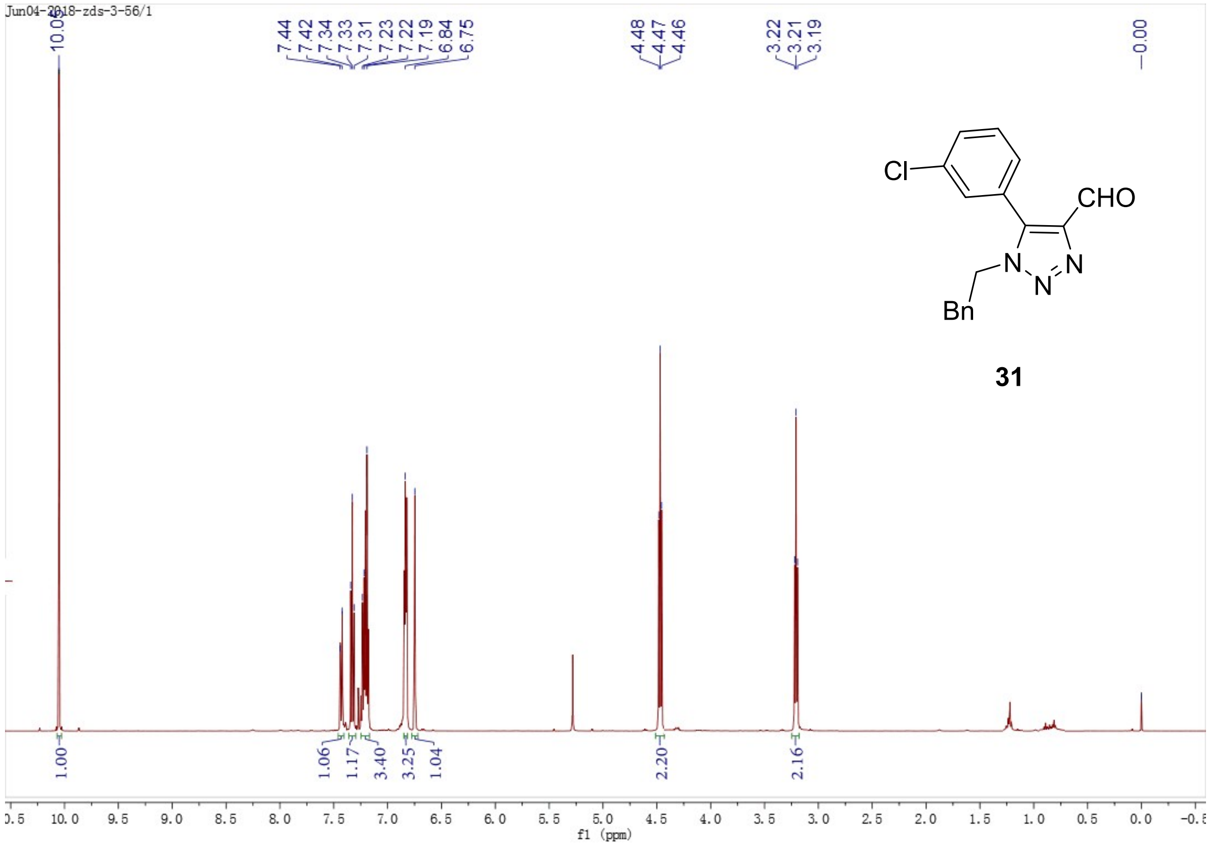
29

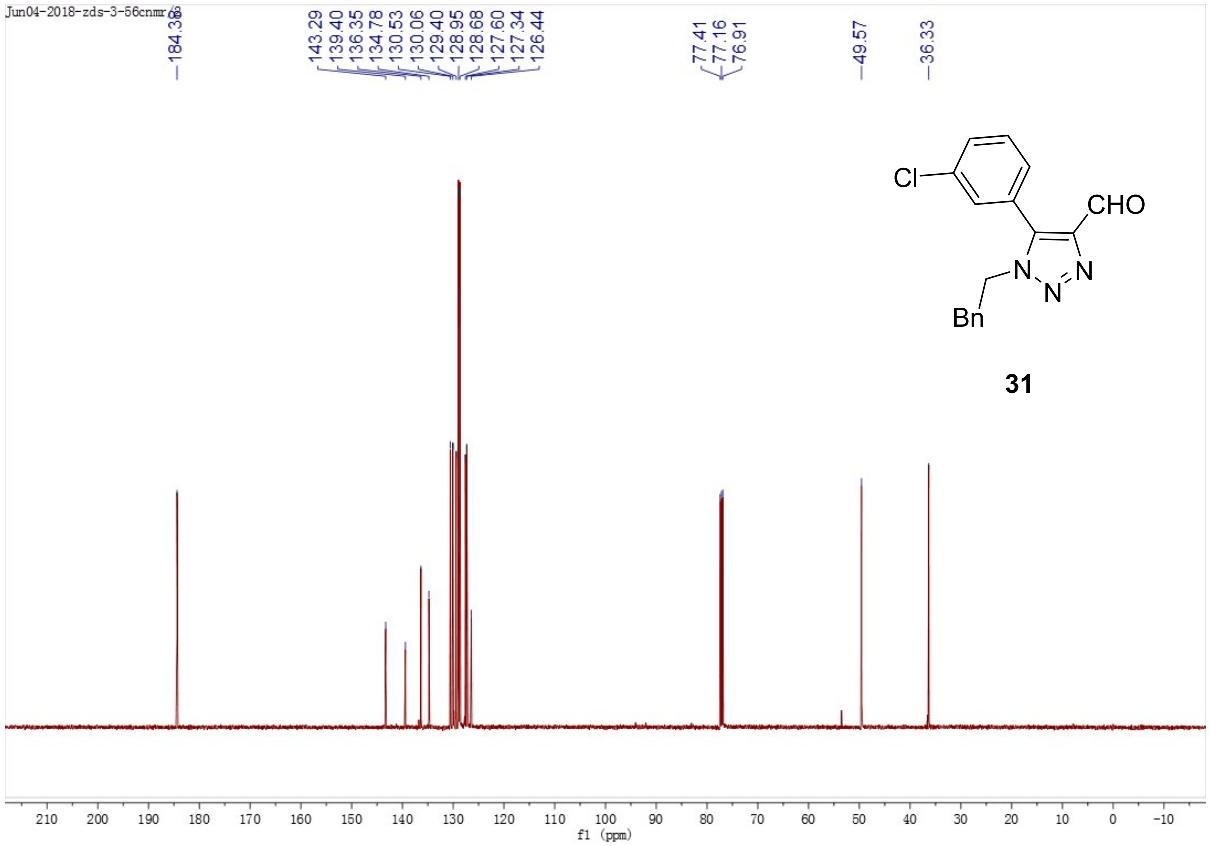

Mar19-2018-2-132/1
184.37
143.27
141.02
136.51
130.41
129.42
128.89
128.83
128.68
127.18
124.67
77.41
77.16
76.91
49.40
36.25
CHO
N
N
N
Bn
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

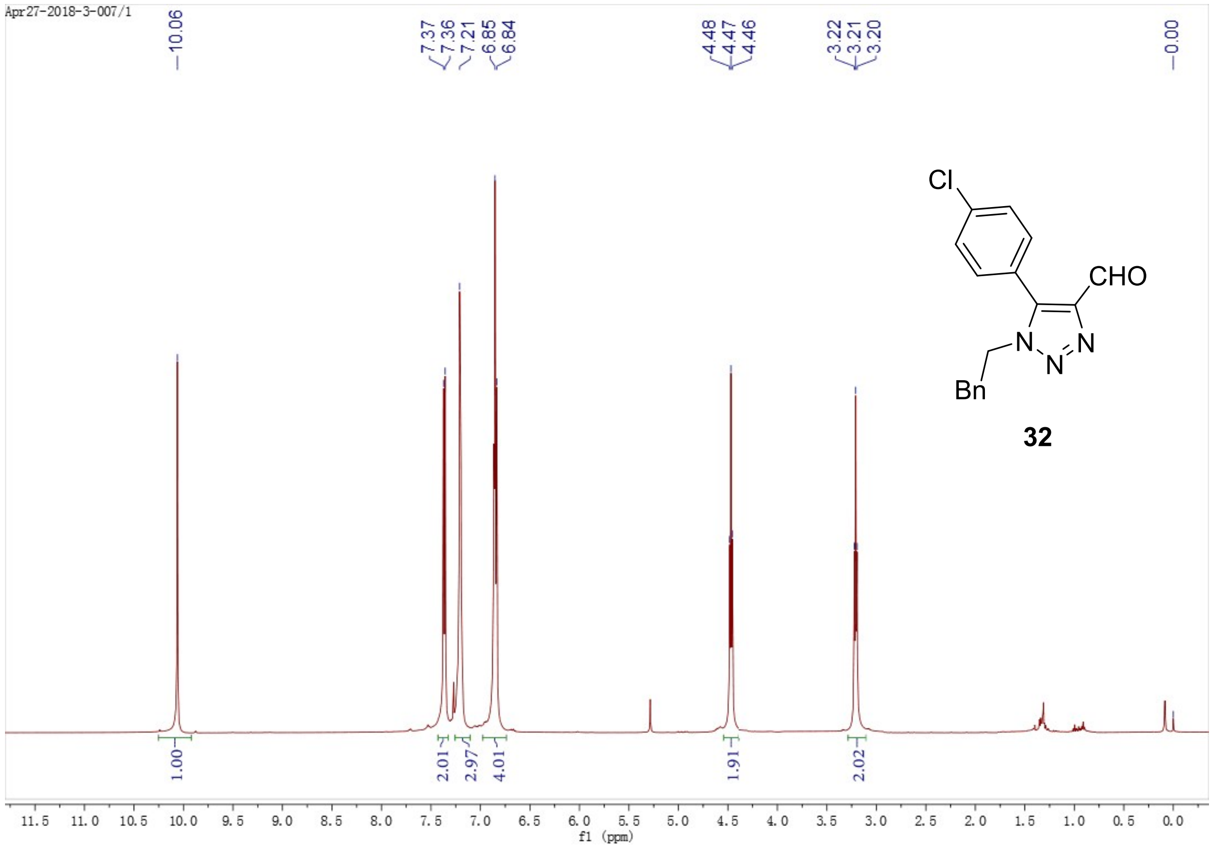
29

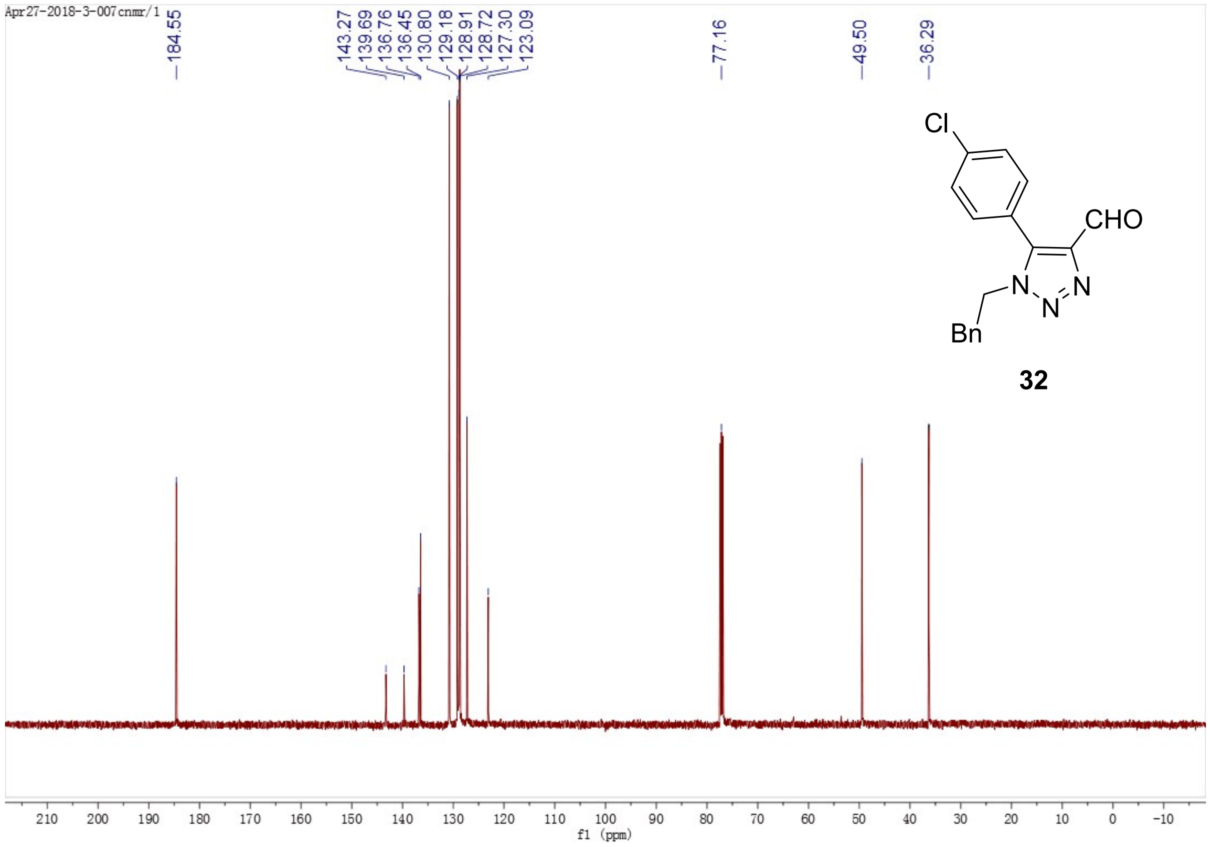

Apr27-2018-3-005CH/2
10.06
7.51
7.50
7.46
7.44
7.26
7.25
7.20
7.19
6.88
6.86
6.63
6.61
4.60
4.57
4.18
4.14
3.25
3.23
3.13
3.11
0.00
CHO
Cl
N
N
N
Bn
30
1.00
1.04
1.06
4.34
2.10
1.02
1.08
1.10
1.10
1.10
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

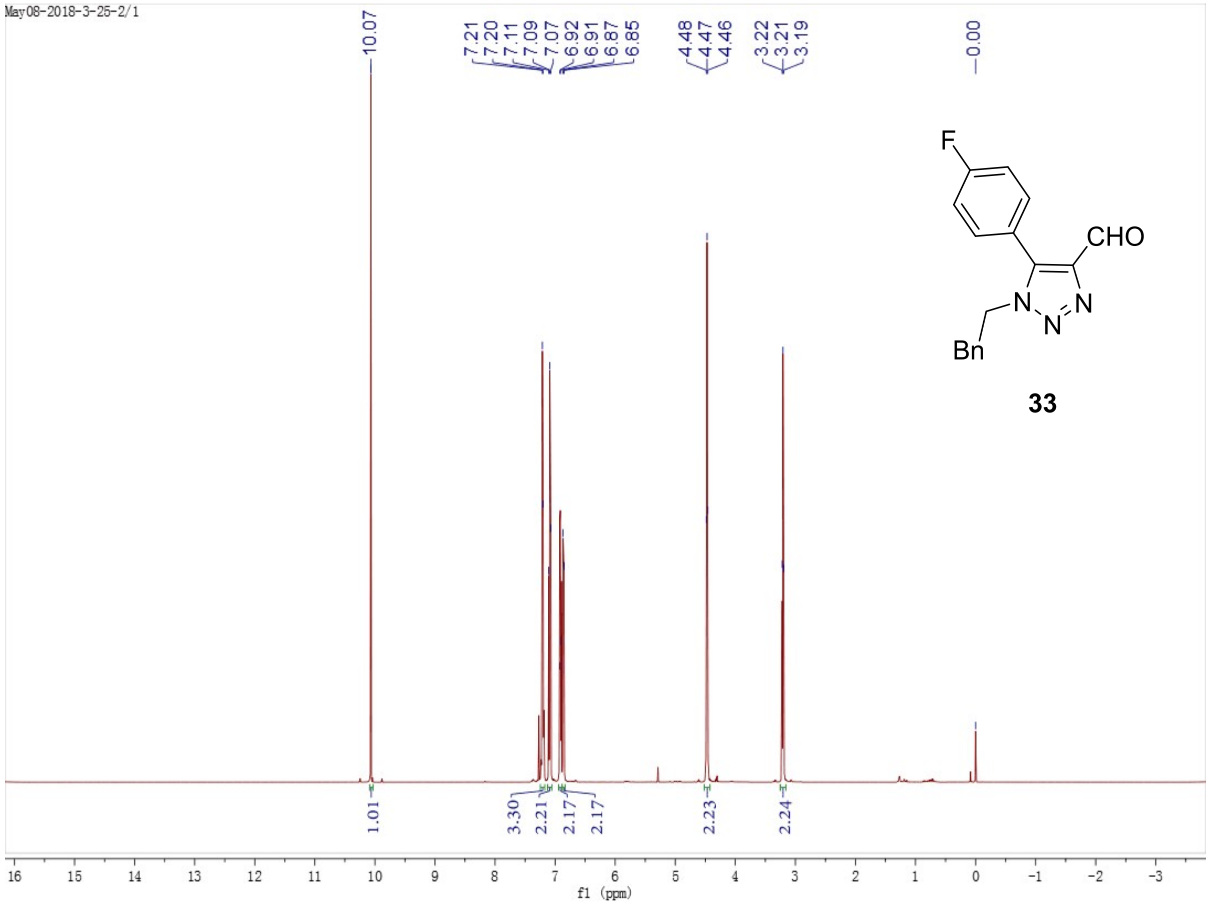

Apr27-2018-3-005cnmr/1
184.34
143.97
137.65
136.55
133.60
131.91
131.69
129.98
128.86
128.70
127.16
124.41
77.16
49.73
36.07
CHO
Cl
N
N
N
Bn
30
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

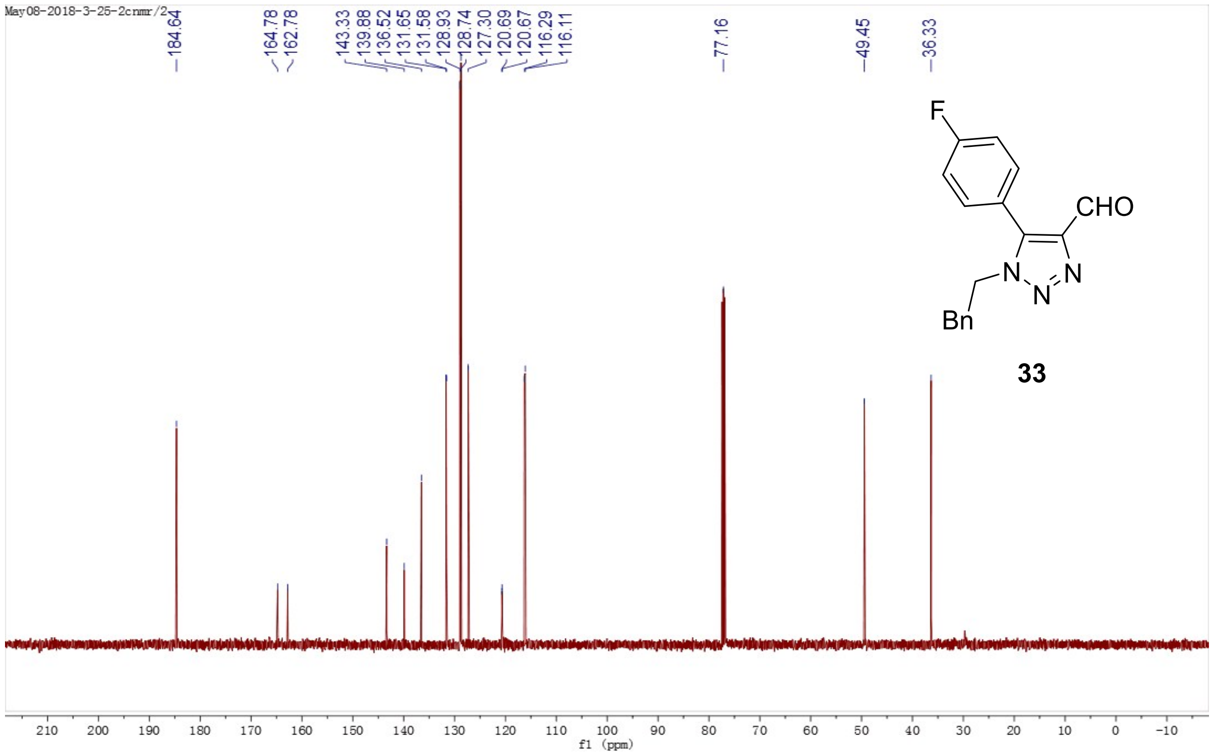

Jun04-2018-zds-3-56/1
10.05
7.44
7.42
7.34
7.33
7.31
7.23
7.22
7.19
6.84
6.75
4.48
4.47
4.46
3.22
3.21
3.19
0.00
Cl
CHO
N
N
N
Bn
31
1.00
1.06
1.17
3.40
3.25
1.04
2.20
2.16
0.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

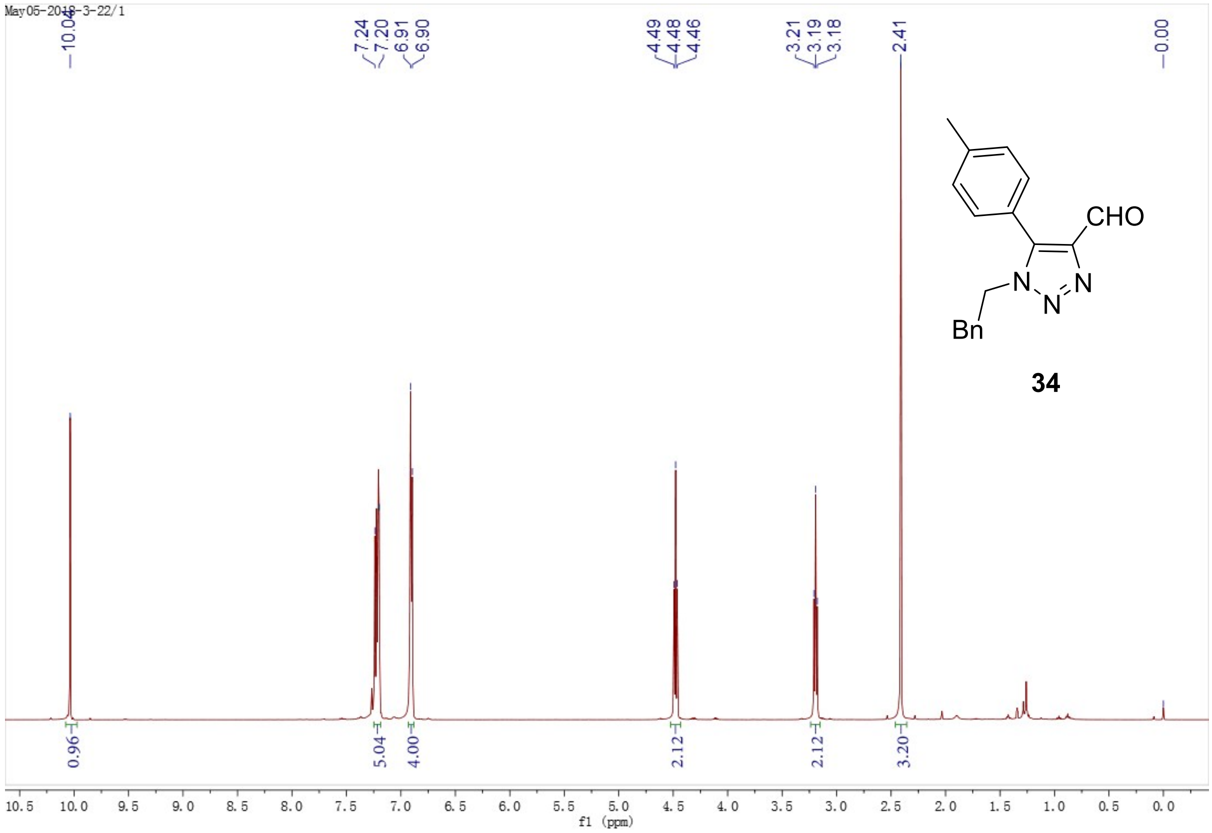

Jun04-2018-zds-3-56cnmr/2
184.36
143.29
139.40
136.35
134.78
130.53
130.06
129.40
128.95
128.68
127.60
127.34
126.44
77.41
77.16
76.91
49.57
36.33
Cl
CHO
N
N
N
Bn
31
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


Apr27-2018-3-007/1
10.06
7.37
7.36
7.21
6.85
6.84
4.48
4.47
4.46
3.22
3.21
3.20
0.00
Cl
CHO
N
N
N
Bn
32
1.00
2.01
2.97
4.01
1.91
2.02
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

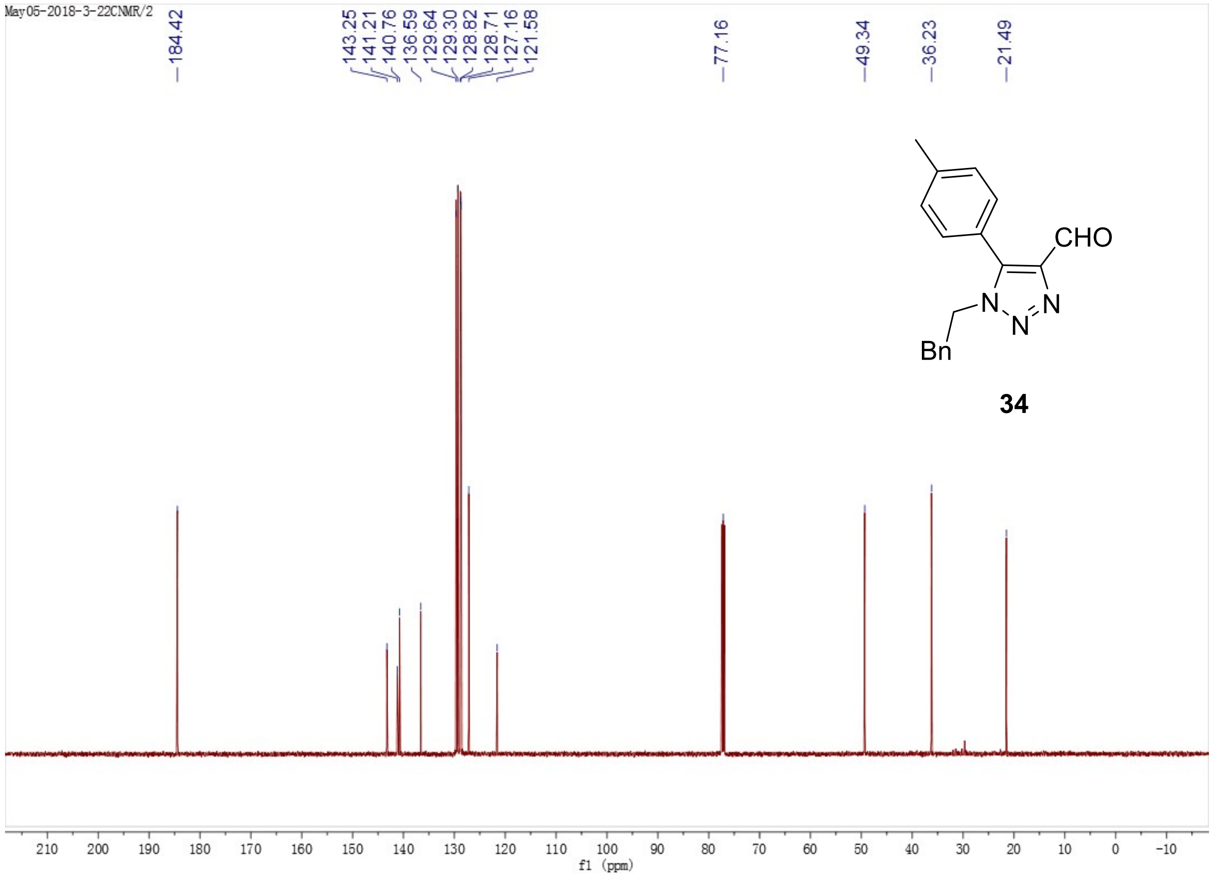

Apr27-2018-3-007cnmr/1
184.55
143.27
139.69
136.76
136.45
130.80
129.18
128.91
128.72
127.30
123.09
77.16
49.50
36.29
Cl
CHO
N
N
N
Bn
32
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


May08-2018-3-25-2/1
10.07
7.21
7.20
7.11
7.09
7.07
6.92
6.91
6.87
6.85
4.48
4.47
4.46
3.22
3.21
3.19
0.00
F
CHO
N
N
N
Bn
33
1.01
3.30
2.21
2.17
2.17
2.23
2.24
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

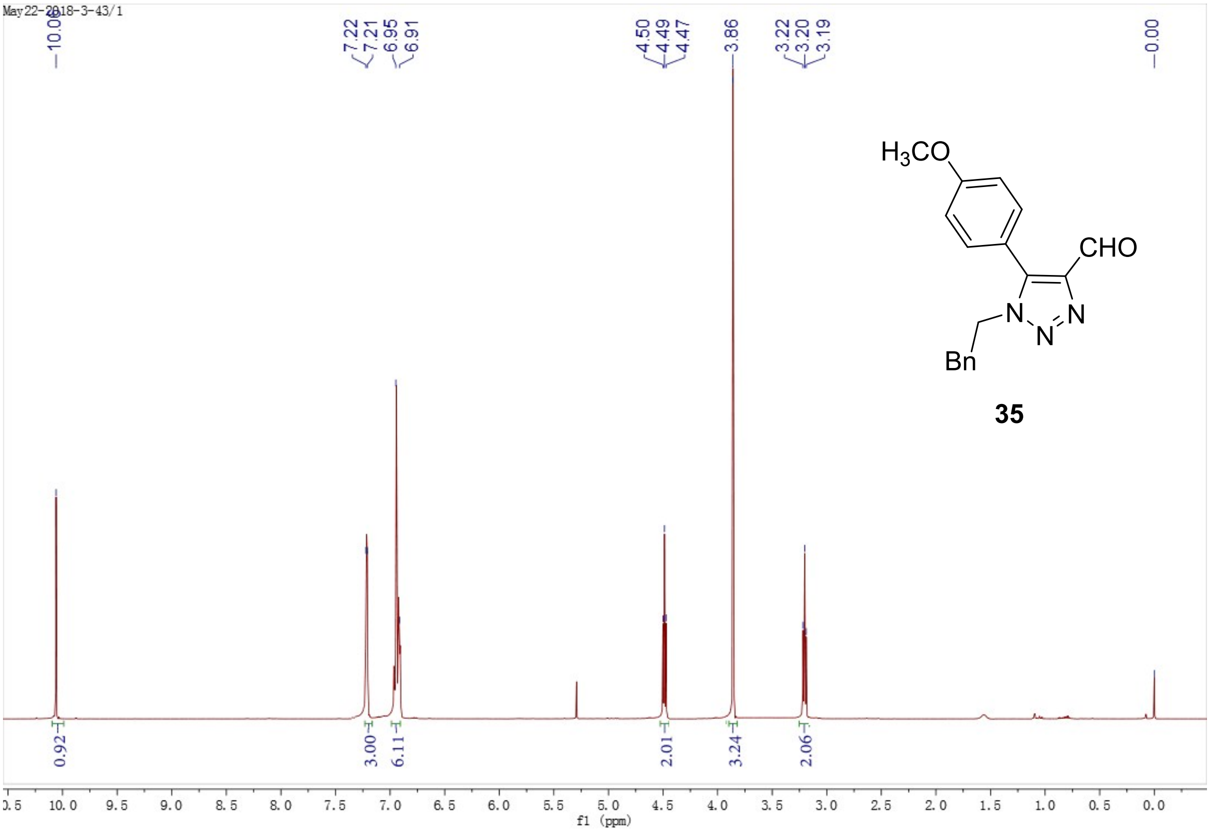

May08-2018-3-25-2cnmr/2
184.64
164.78
162.78
143.33
139.88
136.52
131.65
131.58
128.93
128.74
127.30
120.69
120.67
116.29
116.11
77.16
49.45
36.33
F
CHO
N
N
N
Bn
33
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

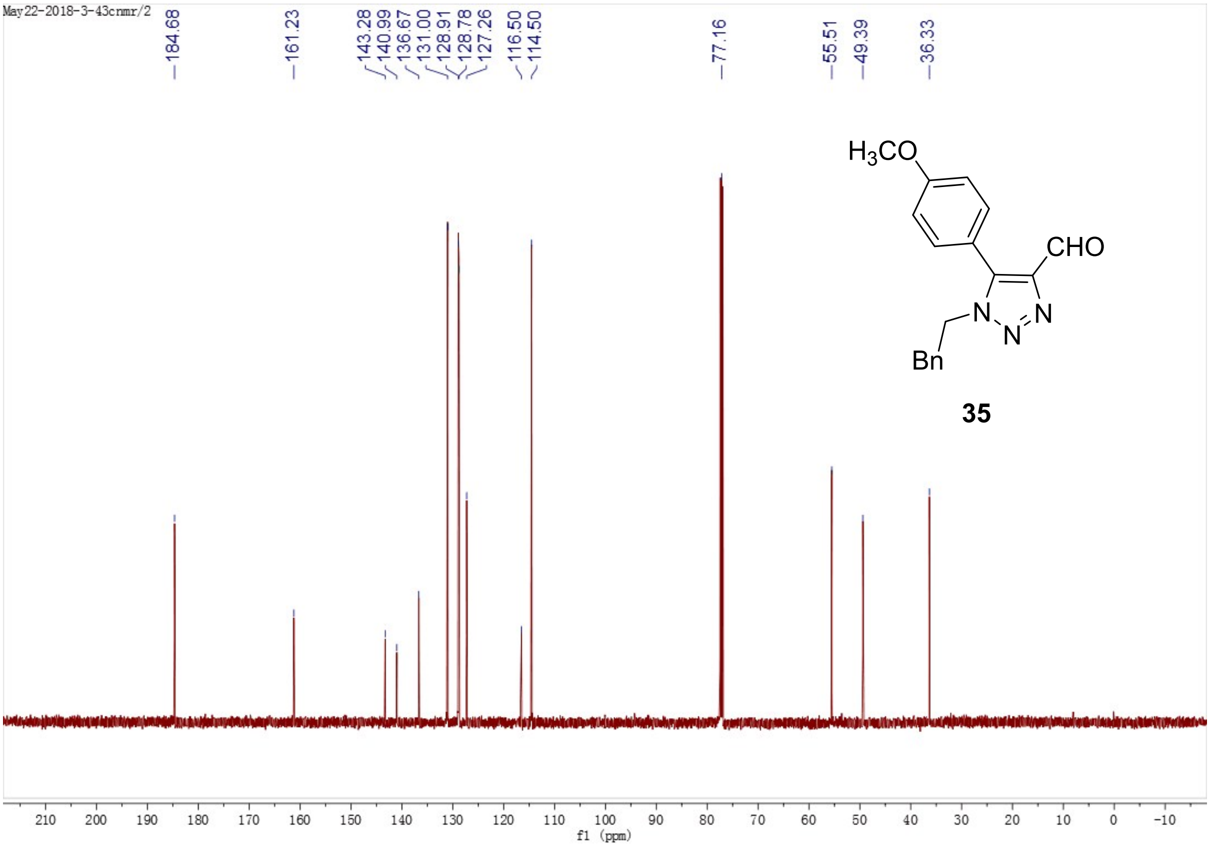

May05-2018-3-22/1
10.04
7.24
7.20
6.91
6.90
4.49
4.48
4.46
3.21
3.19
3.18
2.41
0.00
CHO
N
N
N
Bn
0.96
5.04
4.00
2.12
2.12
3.20
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

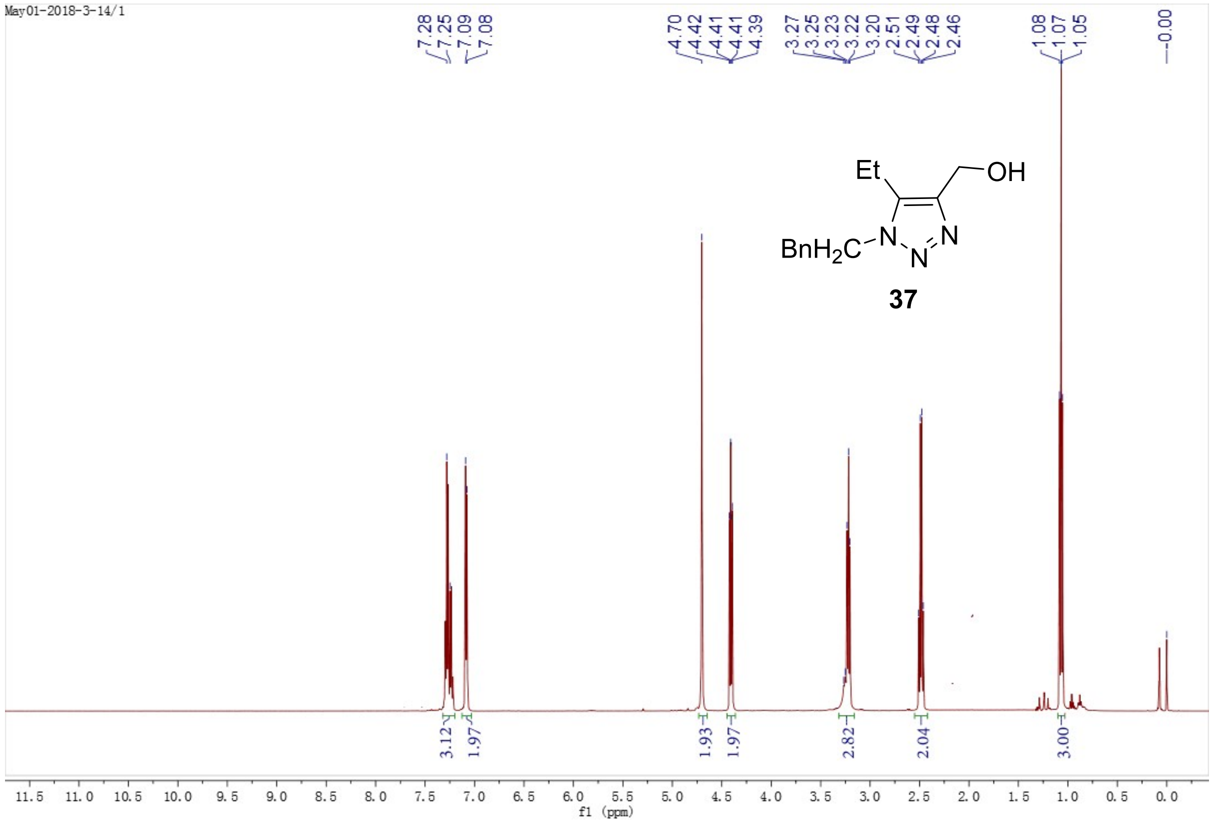
34

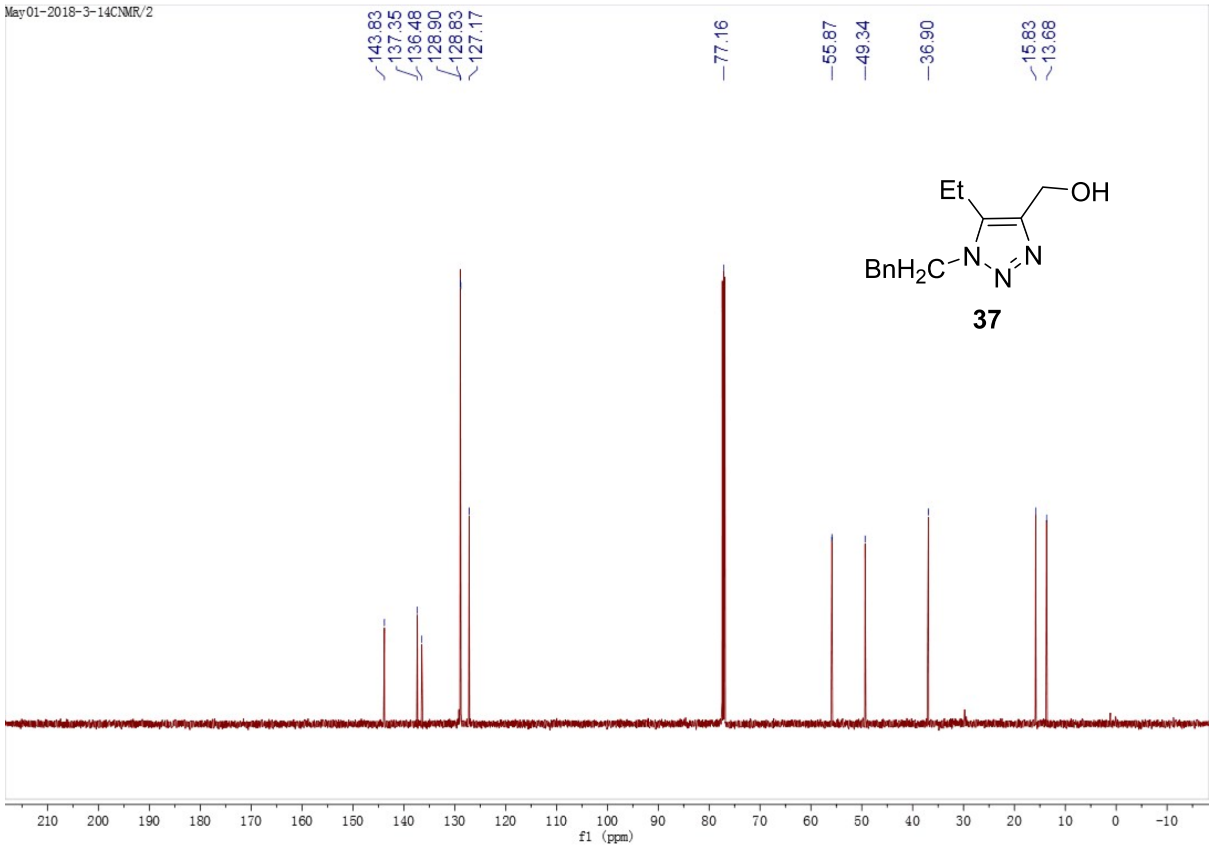

May05-2018-3-22CNMR/2
184.42
143.25
141.21
140.76
136.59
129.64
129.30
128.82
128.71
127.16
121.58
77.16
49.34
36.23
21.49
CHO
N
N
N
Bn
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

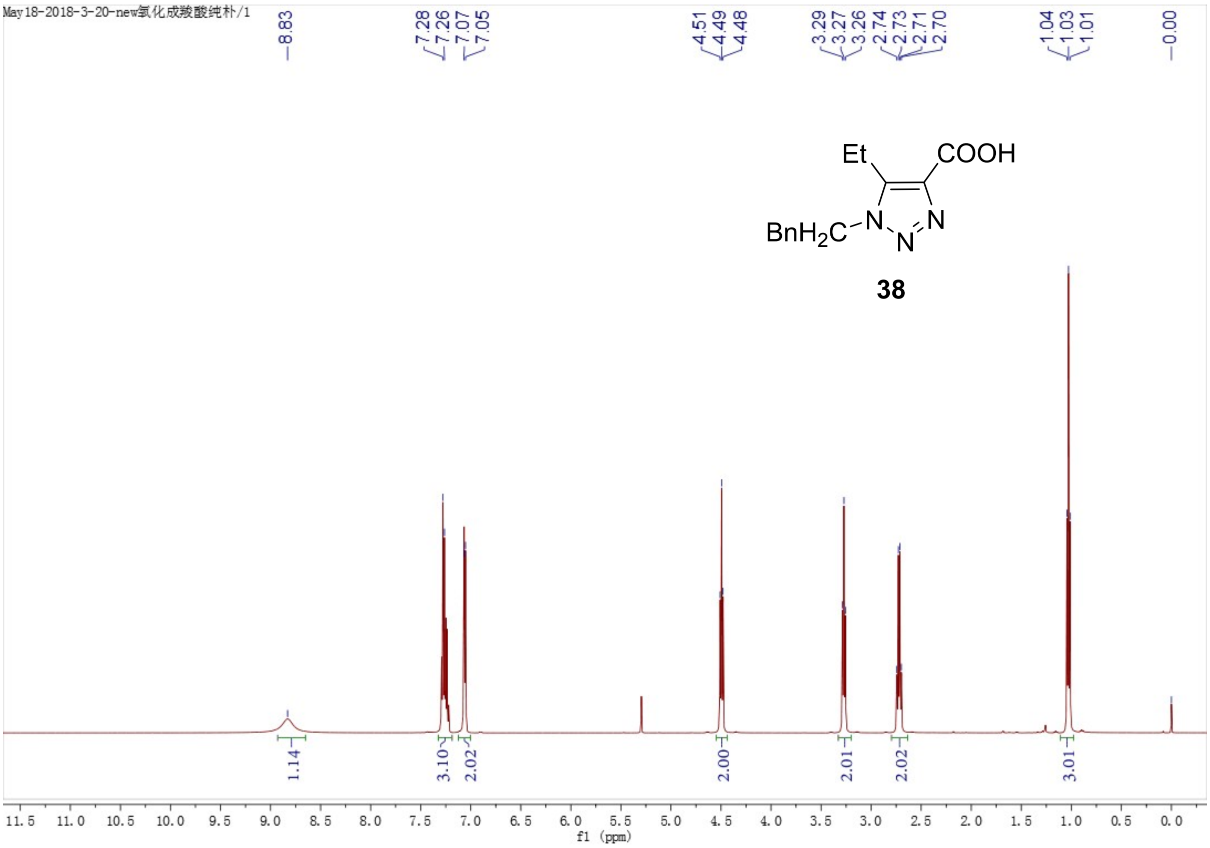
34

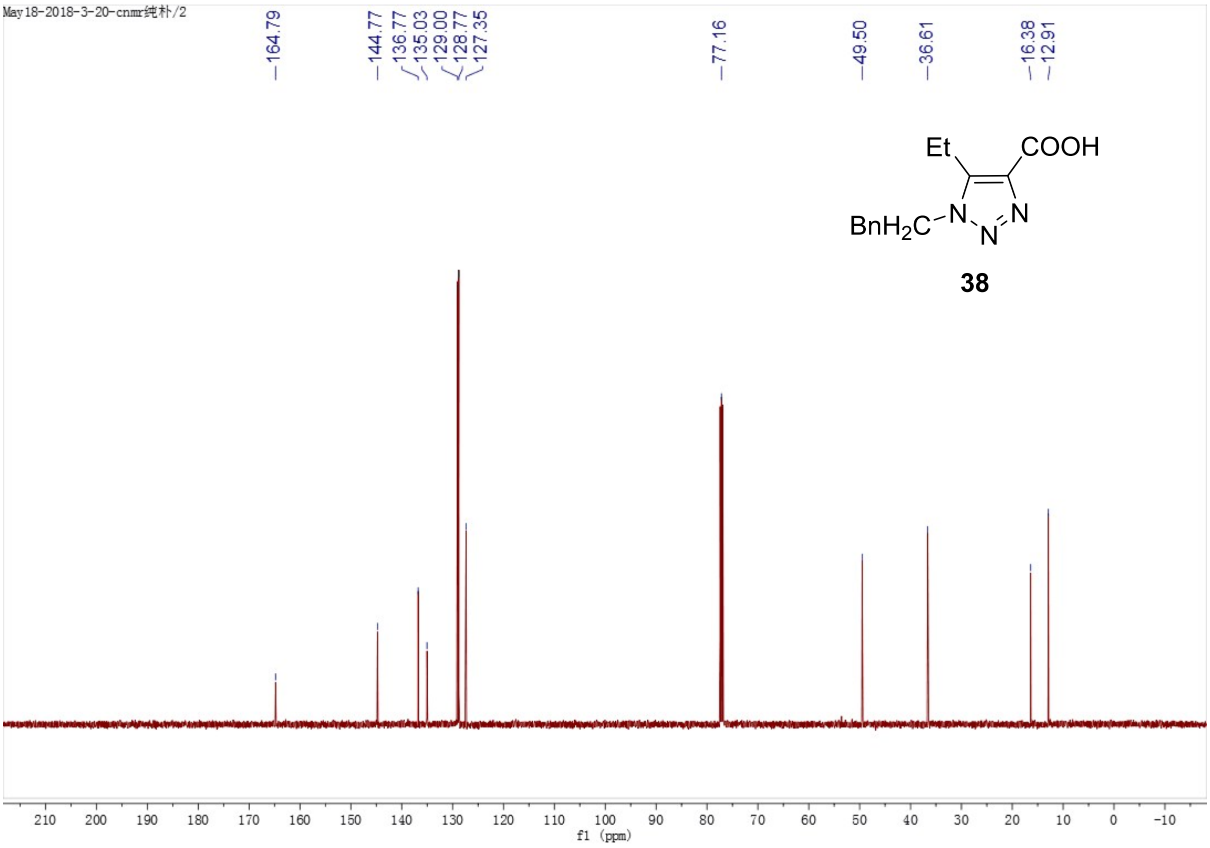

May22-2018-3-43/1
10.06
7.22
7.21
6.95
6.91
4.50
4.49
4.47
3.86
3.22
3.20
3.19
0.00
0.92
3.00
6.11
2.01
3.24
2.06
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
H3CO
CHO
N
N
N
Bn
35

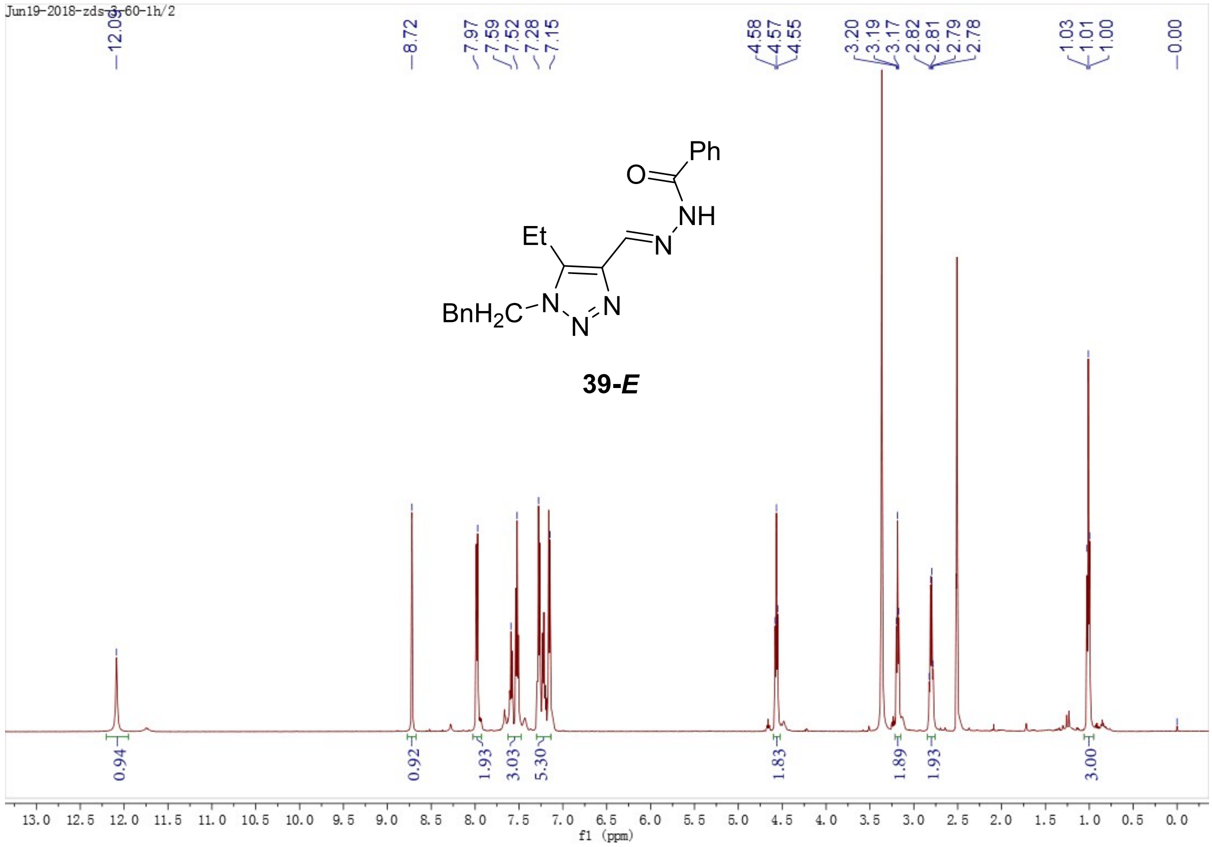

May22-2018-3-43cnmr/2
184.68
161.23
143.28
140.99
136.67
131.00
128.91
128.78
127.26
116.50
114.50
77.16
55.51
49.39
36.33
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)
H3CO
CHO
N
N
N
Bn
35

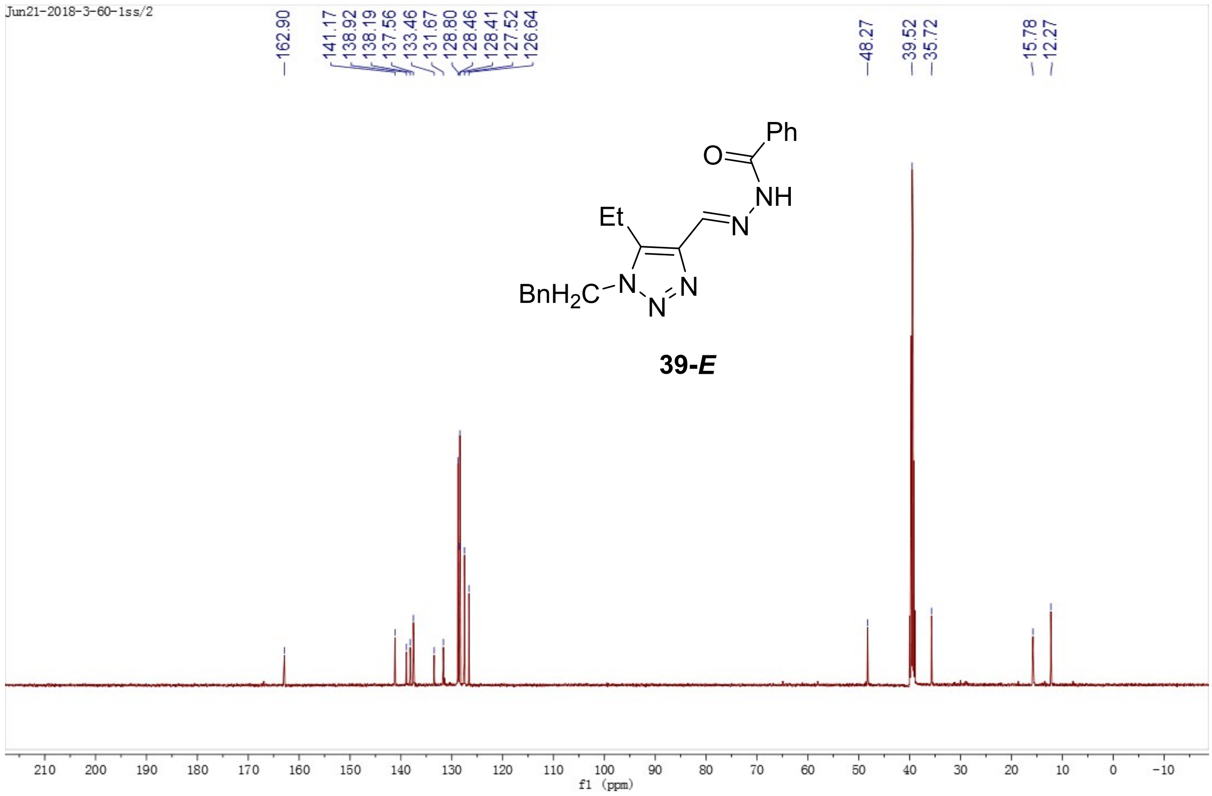

May01-2018-3-14/1
7.28
7.25
7.09
7.08
4.70
4.42
4.41
4.41
4.39
3.27
3.25
3.23
3.22
3.20
2.51
2.49
2.48
2.46
1.08
1.07
1.05
0.00
Et
OH
BnH2C
N
N
N
37
3.12
1.97
1.93
1.97
2.82
2.04
3.00
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

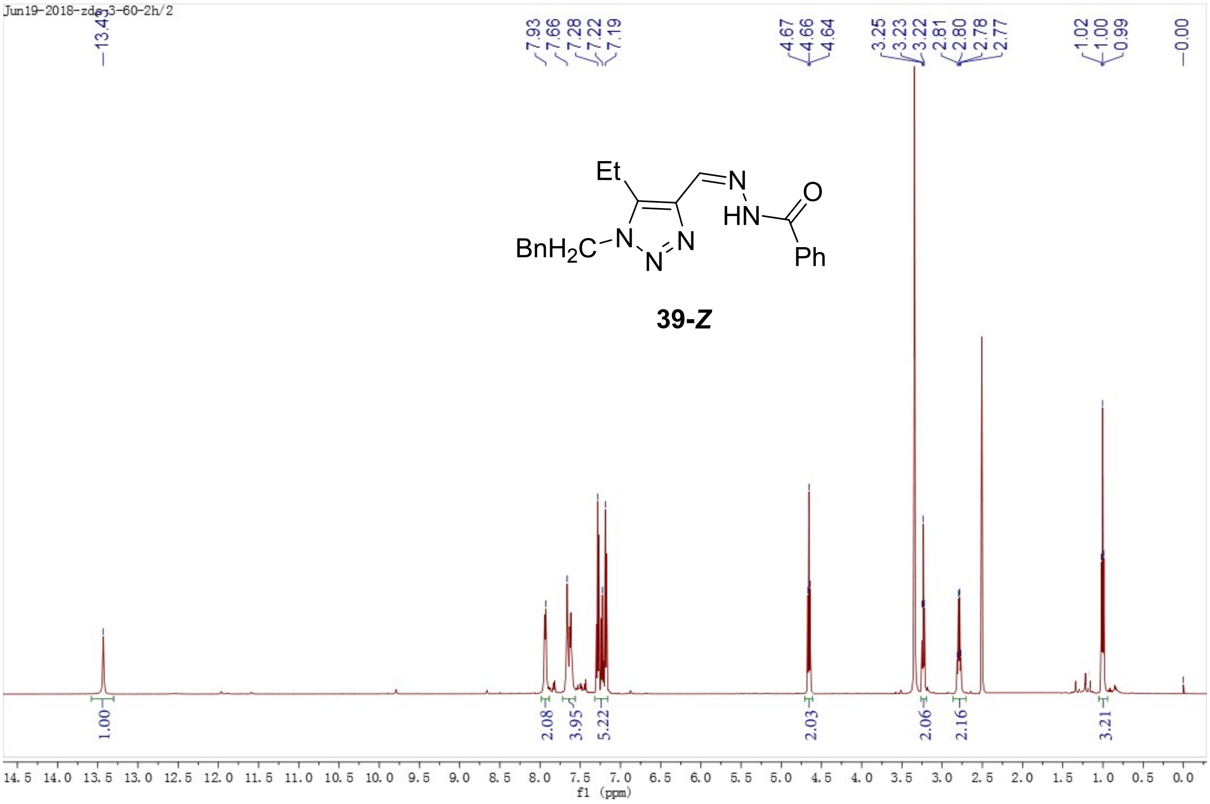

May01-2018-3-14CNMR/2
143.83
137.35
136.48
128.90
128.83
127.17
77.16
55.87
49.34
36.90
15.83
13.68
Et
OH
BnH2C
N
N
N
37
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

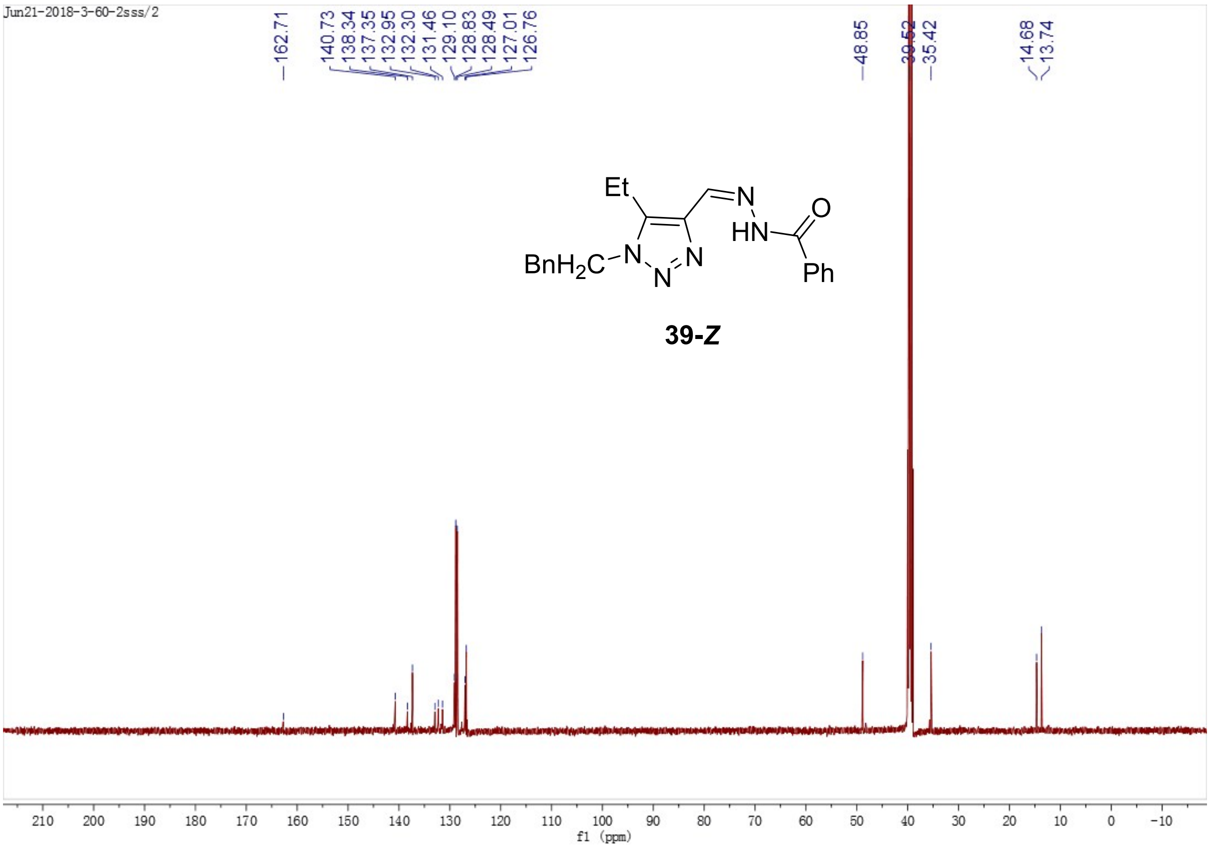

May18-2018-3-20-new氢化成羧酸纯朴/1
8.83
7.28
7.26
7.07
7.05
4.51
4.49
4.48
3.29
3.27
3.26
2.74
2.73
2.71
2.70
1.04
1.03
1.01
0.00
Et
COOH
BnH$_2$C
N
N
N
38
1.14
3.10
2.02
2.00
2.01
2.02
3.01
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

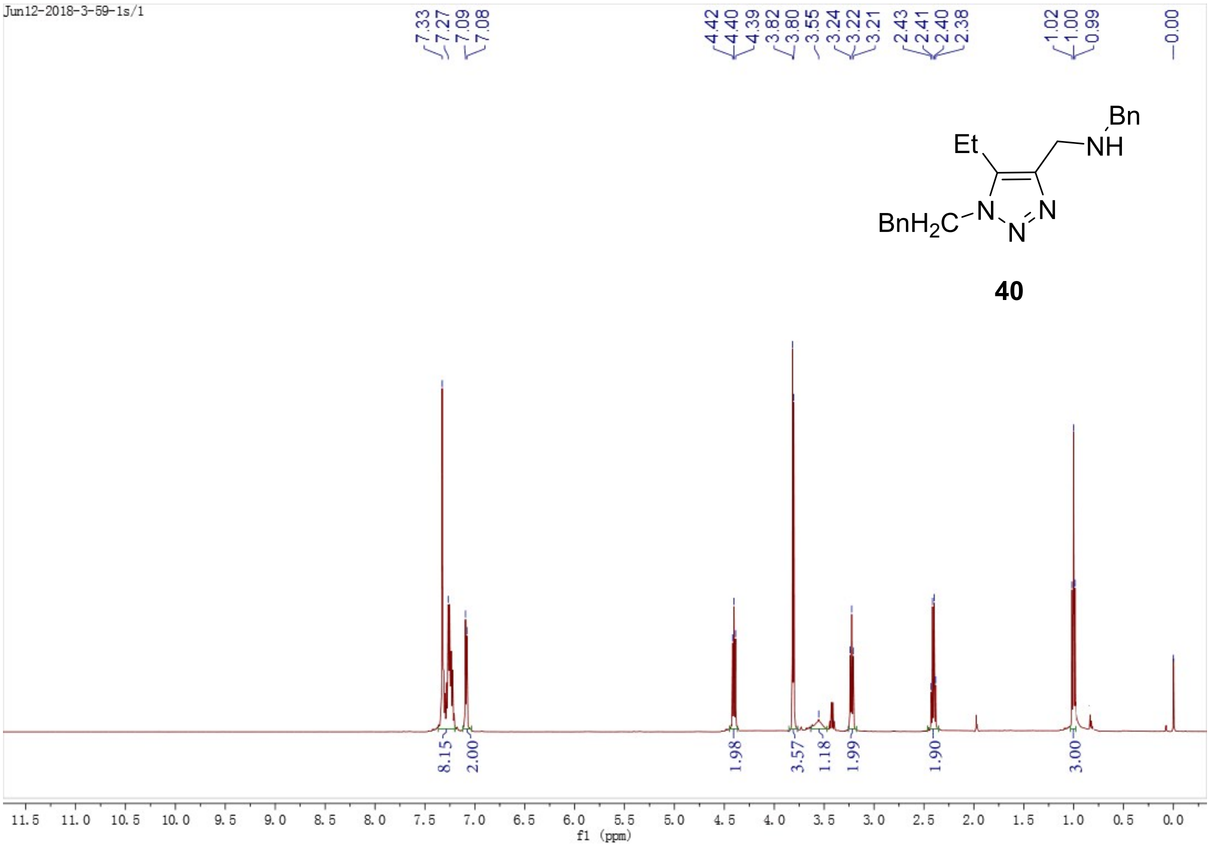

May18-2018-3-20-cnmr纯朴/2
164.79
144.77
136.77
135.03
129.00
128.77
127.35
77.16
49.50
36.61
16.38
12.91
Et
COOH
BnH$_2$C
N
N
N
38
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

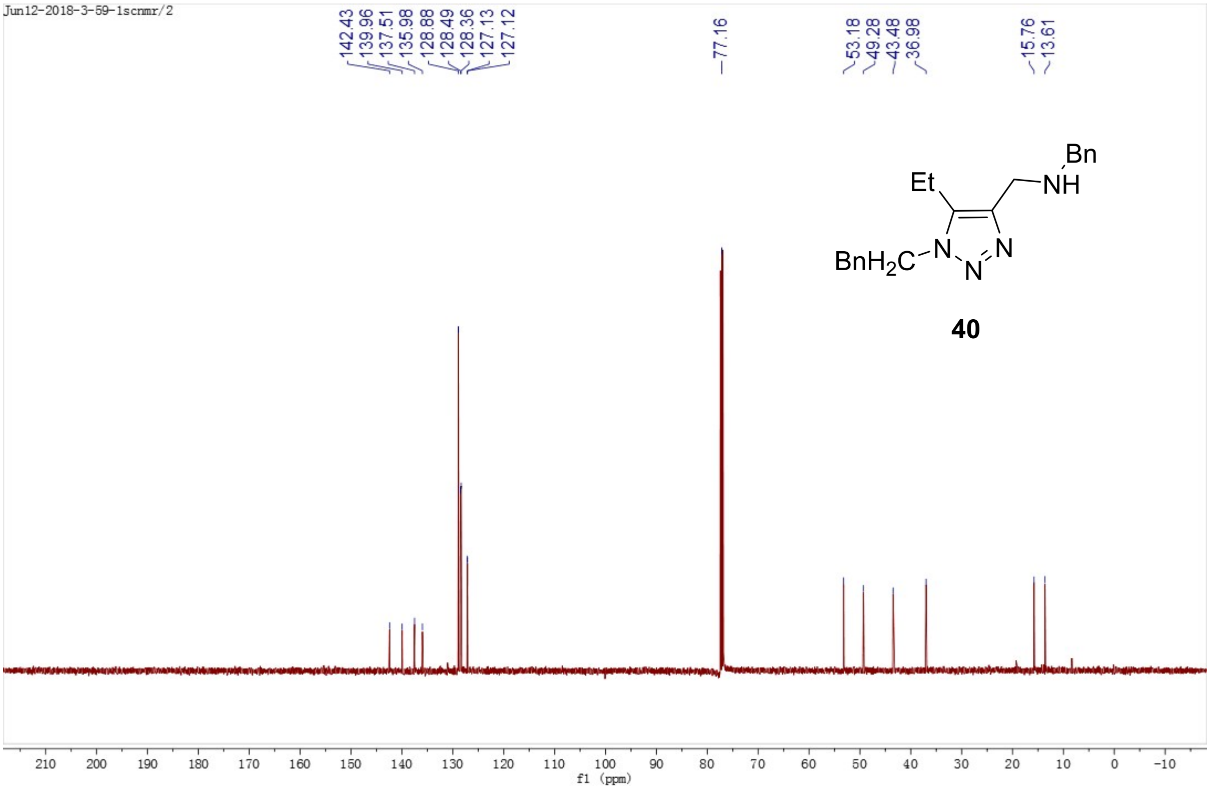

Jun19-2018-zds-3-60-1h/2
12.09
8.72
7.97
7.59
7.52
7.28
7.15
4.58
4.57
4.55
3.20
3.19
3.17
2.82
2.81
2.79
2.78
1.03
1.01
1.00
0.00
Ph
O
NH
N
Et
BnH2C
N
N
N
39-E
0.94
0.92
1.93
3.03
5.30
1.83
1.89
1.93
3.00
13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

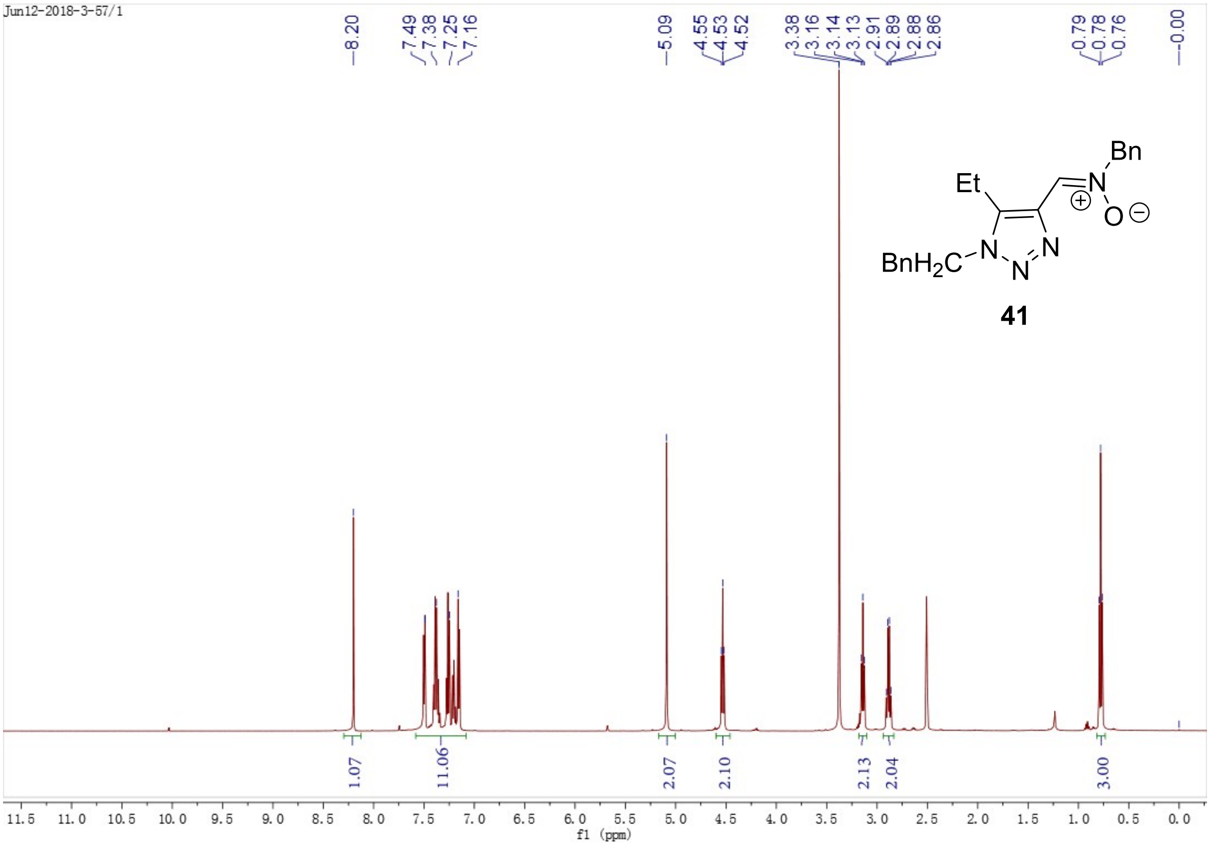

Jun21-2018-3-60-1ss/2
162.90
141.17
138.92
138.19
137.56
133.46
131.67
128.80
128.46
128.41
127.52
126.64
48.27
39.52
35.72
15.78
12.27
Ph
O
NH
N
Et
BnH2C
N
N
N
39-E
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

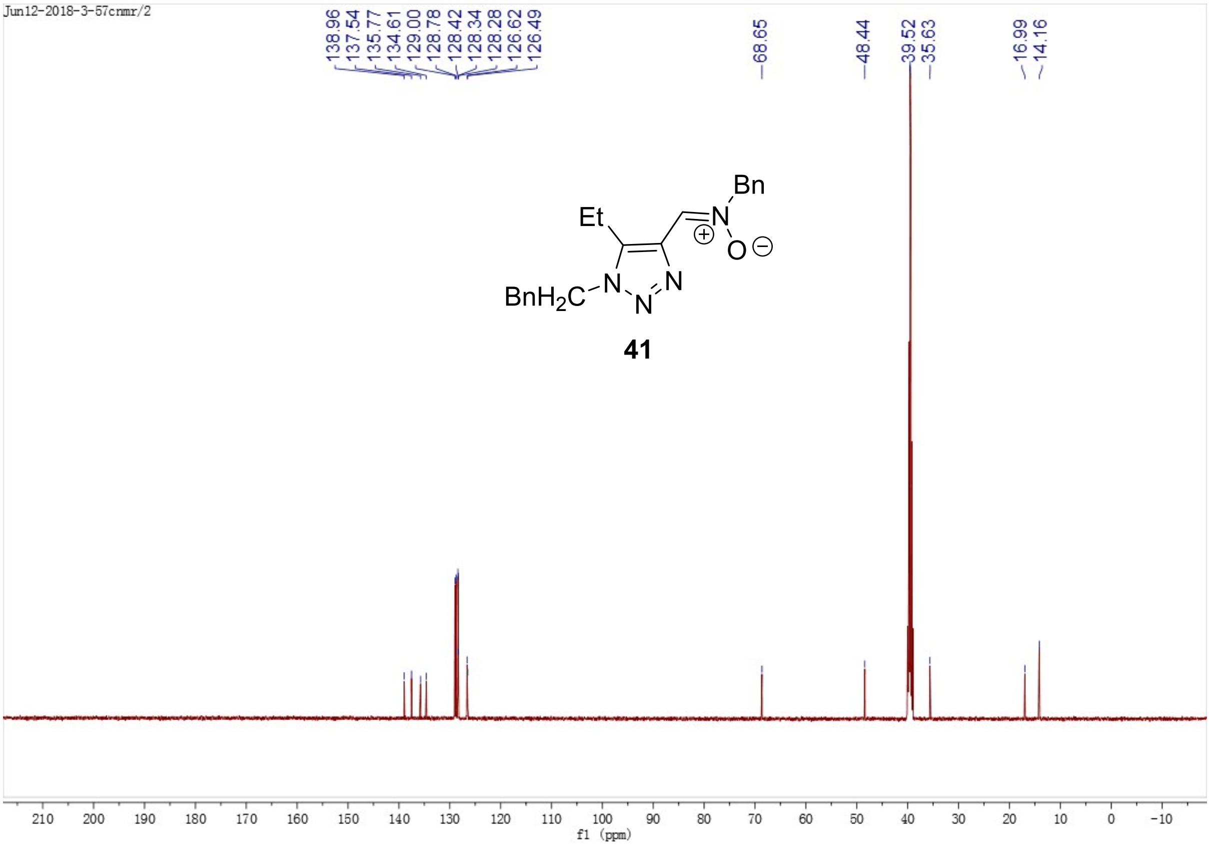

Jun19-2018-zds-3-60-2h/2
13.43
7.93
7.66
7.28
7.22
7.19
4.67
4.66
4.64
3.25
3.23
3.22
2.81
2.80
2.78
2.77
1.02
1.00
0.99
0.00
Et
N
HN
O
BnH2C
N
N
N
Ph
39-Z
1.00
2.08
3.95
5.22
2.03
2.06
2.16
3.21
14.5 14.0 13.5 13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

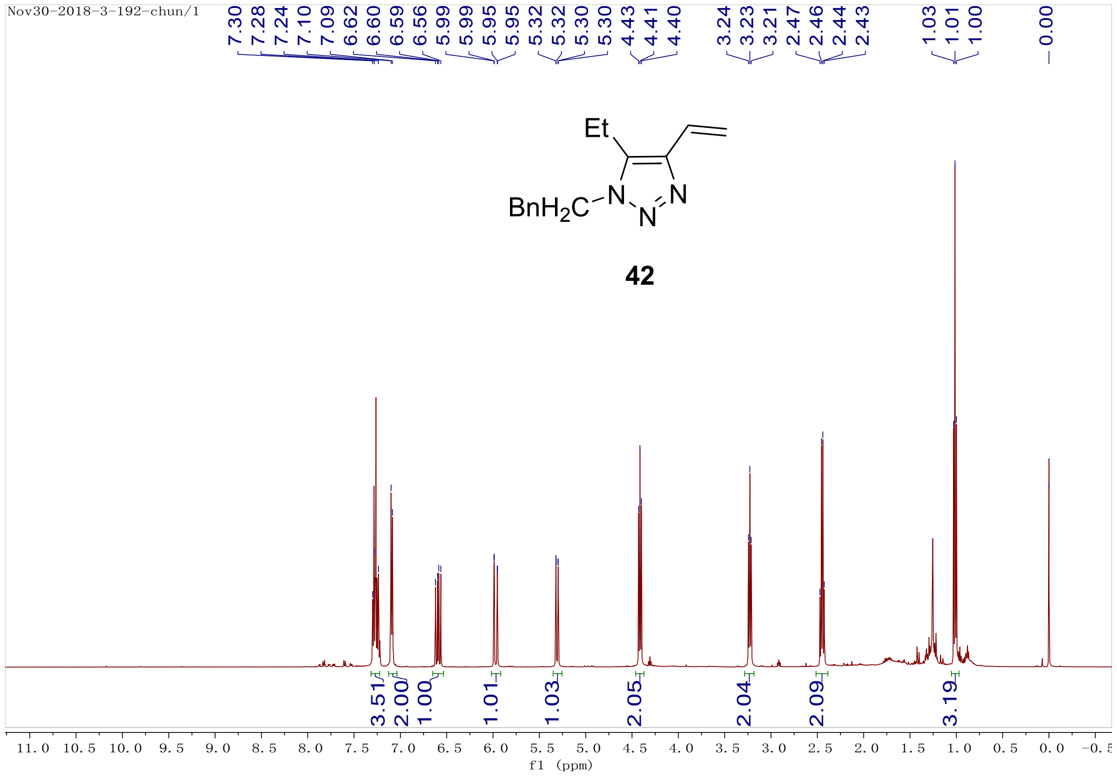

Jun21-2018-3-60-2sss/2
162.71
140.73
138.34
137.35
132.95
132.30
131.46
129.10
128.83
128.49
127.01
126.76
48.85
39.52
35.42
14.68
13.74
Et
N
HN
O
BnH2C
N
N
N
Ph
39-Z
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

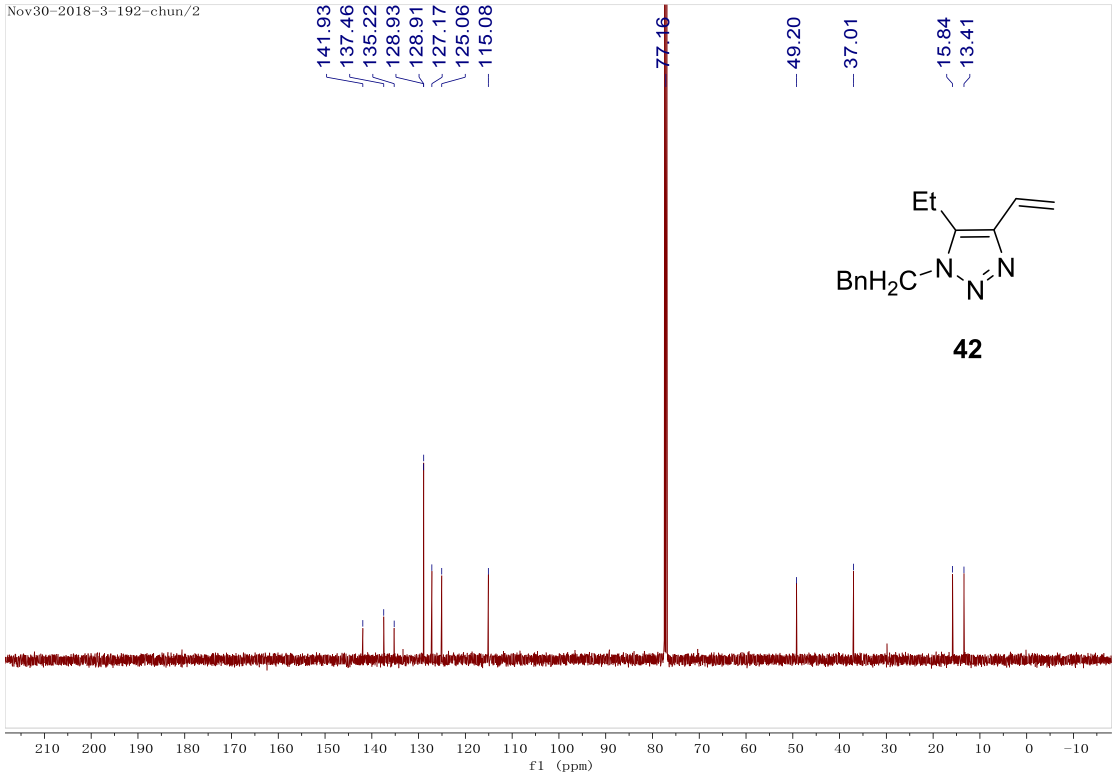

Jun12-2018-3-59-1s/1
7.33
7.27
7.09
7.08
4.42
4.40
4.39
3.82
3.80
3.55
3.24
3.22
3.21
2.43
2.41
2.40
2.38
1.02
1.00
0.99
0.00
Et
NH
Bn
BnH2C
N
N
N
40
8.15
2.00
1.98
3.57
1.18
1.99
1.90
3.00
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


Jun12-2018-3-59-1scnmr/2
142.43
139.96
137.51
135.98
128.88
128.49
128.36
127.13
127.12
77.16
53.18
49.28
43.48
36.98
15.76
13.61
Et
NH
Bn
BnH2C
N
N
N
40
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


Jun12-2018-3-57/1
8.20
7.49
7.38
7.25
7.16
5.09
4.55
4.53
4.52
3.38
3.16
3.14
3.13
2.91
2.89
2.88
2.86
0.79
0.78
0.76
0.00
1.07
11.06
2.07
2.10
2.13
2.04
3.00
Et
Bn
N
O
BnH2C
N
N
N
41
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


Jun12-2018-3-57cnmr/2
138.96
137.54
135.77
134.61
129.00
128.78
128.42
128.34
128.28
126.62
126.49
68.65
48.44
39.52
35.63
16.99
14.16
Et
Bn
N
O
BnH2C
N
N
N
41
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


Nov30-2018-3-192-chun/1
7.30
7.28
7.24
7.10
7.09
6.62
6.60
6.59
6.56
5.99
5.99
5.95
5.95
5.32
5.32
5.30
5.30
4.43
4.41
4.40
3.24
3.23
3.21
2.47
2.46
2.44
2.43
1.03
1.01
1.00
0.00
Et
BnH2C
N
N
N
42
3.51
2.00
1.00
1.01
1.03
2.05
2.04
2.09
3.19
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


Nov30-2018-3-192-chun/2
141.93
137.46
135.22
128.93
128.91
127.17
125.06
115.08
77.16
49.20
37.01
15.84
13.41
Et
BnH2C
N
N
N
42
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)