

Supporting Information

Facile synthesis of 4-vinyl- and 4-fluorovinyl-1,2,3-triazoles via bifunctional “*click-olefination*” reagents

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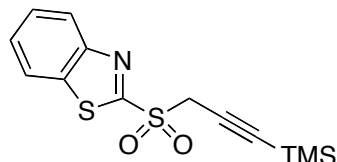
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GENERAL EXPERIMENTAL METHODS

THF was distilled over LiAlH_4 , and then over sodium. Toluene was distilled over sodium. DMF and DMPU were obtained from commercial sources and were used without further purification. For reactions, which were performed under a nitrogen atmosphere, glassware was flame dried under vacuum. LDA (2.0 M solution in heptane/THF/EtPh) and LHMDS (1.0 M in THF) were obtained from commercial sources. Fluorinating reagent *N*-fluorobenzenesulfonimide (NFSI) was a gift from Honeywell (Dr. Andrew Poss), but is also commercially available. Thin layer chromatography was performed on 250 μm silica plates and column chromatographic purifications were performed on 200-300 mesh silica gel. All other reagents were obtained from commercial sources and used without further purification. ^1H NMR spectra were recorded at 500 MHz in CDCl_3 , acetone- d_6 and C_6D_6 . ^{19}F NMR spectra were recorded at 282 MHz using CFCl_3 as internal standard. Chemical shifts (δ) are reported in parts per million and coupling constants (J) are in hertz.

2-[3-(Trimethylsilyl)prop-2-ynylsulfonyl]benzo[*d*]thiazole (1)

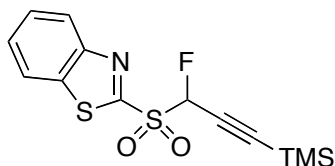


Step 1: Synthesis of 2-[3-(trimethylsilyl)prop-2-ynylthio]benzo[*d*]thiazole. To a solution of (3-bromoprop-1-ynyl)trimethylsilane (2.00 g, 10.5 mmol, 1 molar equiv) in DMF (40.0 mL) at room temperature, the sodium salt of 2-mercapto-1,3-benzothiazole (2.57 g, 13.6 mmol, 1.3 molar equiv) was added and the reaction mixture was stirred for 5 h. The reaction mixture was diluted with water and extracted with EtOAc (3x). The combined organic layer was thoroughly washed with water and then with brine and dried over anhydrous Na_2SO_4 . The solvent was evaporated under reduced pressure to yield 2.81 g (97%) of crude 2-[3-(trimethylsilyl)prop-2-ynylthio]benzo[*d*]thiazole¹ as a light yellow solid, that was subjected to oxidation without further purification. ^1H NMR (500 MHz, CDCl_3): δ 7.90 (d, 1H, Ar-H, $J = 7.8$ Hz), 7.78 (d, 1H, Ar-H, $J = 7.8$ Hz), 7.43 (t, 1H, Ar-H, $J = 7.8$ Hz), 7.32 (t, 1H, Ar-H, $J = 7.6$ Hz), 4.16 (s, 2H, CH_2), 0.15 (s, 9H, SiMe_3).

Step 2: Oxidation of 2-[3-(trimethylsilyl)prop-2-ynylthio]benzo[*d*]thiazole. To a stirring solution of 2-[3-(trimethylsilyl)prop-2-ynylthio]benzo[*d*]thiazole (2.00 g, 7.22 mmol) in CHCl_3

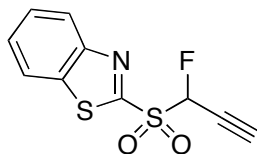
(30.0 mL) at $-10\text{ }^{\circ}\text{C}$ (ice-salt) a solution of *m*-CPBA (3.74 g, 21.7 mmol, 3 molar equiv) in CHCl_3 (55.0 mL) was added dropwise. After complete addition, the mixture was stirred for an additional 10 min at $-10\text{ }^{\circ}\text{C}$, allowed to warm to room temperature and stirred for 15 h. The reaction was quenched with 35 mL of saturated aqueous NaHCO_3 solution, the organic layer was separated and the aqueous layer was extracted with CH_2Cl_2 (3x). The combined organic layer was thoroughly washed with water and finally with brine and dried over anhydrous Na_2SO_4 . The solvent was evaporated under reduced pressure to yield 2.19 g (98%) of 2-[3-(trimethylsilyl)prop-2-ynylsulfonyl]benzo[d]thiazole (**1**)¹ of crude product as an off-white solid that was subjected to fluorination without further purification. ^1H NMR (500 MHz, CDCl_3): δ 8.26 (d, 1H, Ar-H, $J = 8.3$ Hz), 8.03 (d, 1H, Ar-H, $J = 8.3$ Hz), 7.68-7.60 (m, 2H, Ar-H), 4.39 (s, 2H, CH_2), 0.02 (s, 9H, SiMe_3). ^{13}C NMR (125 MHz, CDCl_3): δ 164.1, 152.7, 137.4, 128.4, 127.9, 125.8, 122.4, 95.8, 90.8, 48.7, -0.5 .

2-[1-Fluoro-3-(trimethylsilyl)prop-2-ynylsulfonyl]benzo[d]thiazole (**2**)



To a stirring solution of sulfone **1** (2.00 g, 6.47 mmol, 1 molar equiv) in dry toluene (40.0 mL) cooled to $-78\text{ }^{\circ}\text{C}$ (dry ice/isopropanol) under nitrogen, LDA (1.1 molar equiv of a 2.0 M solution in heptane/THF/EtPh) was added. After 12 min, solid NFSI (2.45 g, 7.76 mmol, 1.2 molar equiv) was added. The reaction mixture was allowed to stir at $-78\text{ }^{\circ}\text{C}$ for 50 min then warmed to room temperature and the stirring was continued for an additional 50 min. Saturated aq NH_4Cl was added to the reaction mixture and the layers were separated. The aqueous layer was extracted with EtOAc (3 x), and the combined organic layer was washed with water, saturated aq NaHCO_3 and brine. The organic layer was dried over anhydrous Na_2SO_4 and the solvent was evaporated under reduced pressure. The crude reaction mixture was rapidly purified by column chromatography (SiO_2 , mesh 200-300, 10% EtOAc in hexanes) to yield 1.33 g (63%) of **2** as a white solid. ^1H NMR (500 MHz, CDCl_3): δ 8.29 (d, 1H, Ar-H, $J = 8.3$ Hz), 8.05 (d, 1H, Ar-H, $J = 7.8$ Hz), 7.69-7.63 (m, 2H, Ar-H), 6.12 (d, 1H, CHF, $^2J_{\text{FH}} = 48.8$ Hz), 0.17 (s, 9H, SiMe_3). ^{13}C NMR (125 MHz, CDCl_3): δ 160.9, 152.6, 137.6, 128.8, 128.0, 125.9, 122.3, 104.5 (d, $^3J_{\text{CF}} = 8.2$ Hz), 91.8 (d, $^1J_{\text{CF}} = 222.0$ Hz), 89.6 (d, $^2J_{\text{CF}} = 23.4$ Hz), 0.9. ^{19}F NMR (282 MHz, CDCl_3): δ -164.6 (d, $^2J_{\text{FH}} = 48.8$ Hz). HRMS (ESI) calcd. for $\text{C}_{13}\text{H}_{15}\text{FNO}_2\text{S}_2\text{Si}$ [$\text{M} + \text{H}$]⁺ 328.0292, found 328.0296.

2-(1-Fluoroprop-2-ynylsulfonyl)benzo[d]thiazole (2a)

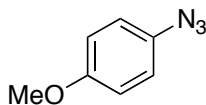


^1H NMR (500 MHz, CDCl_3): δ 8.29 (d, 1H, Ar-H, $J = 8.3$ Hz), 8.05 (d, 1H, Ar-H, $J = 7.4$ Hz), 7.70-7.63 (m, 2H, Ar-H), 6.21 (dd, 1H, CHF, $^2J_{\text{FH}} = 48.1$ Hz, $J_{\text{HH}} = 2.1$ Hz), 3.13 (dd, 1H, $J = 5.8$, 2.1 Hz). ^{13}C NMR (125 MHz, CDCl_3): δ 160.9, 152.9, 137.9, 128.9, 128.2, 126.2, 122.5, 91.4 (d, $^1J_{\text{CF}} = 222.4$ Hz), 84.8 (d, $^3J_{\text{CF}} = 9.6$ Hz), 70.0 (d, $^2J_{\text{CF}} = 24.7$ Hz). ^{19}F NMR (282 MHz, CDCl_3): δ -168.5 (d, $^2J_{\text{FH}} = 45.8$ Hz). HRMS (ESI) calcd. for $\text{C}_{10}\text{H}_7\text{FNO}_2\text{S}_2$ [$\text{M} + \text{H}$] $^+$ 255.9897, found 255.9898.

Synthesis of Azides

4-Methoxyphenyl azide was prepared from the corresponding boronic acid.² Aliphatic azides were obtained from the corresponding halides by nucleophilic substitution.^{3,4}

4-Methoxyphenyl Azide

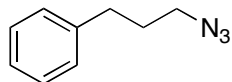


In a 10 mL round bottom flask, boronic acid (76.0 mg, 0.500 mmol, 1 molar equiv) was dissolved in 2.0 mL MeOH. NaN_3 (39.0 mg, 0.600 mmol, 1.2 molar equiv) and $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ (12.5 mg, 0.050 mmol, 0.1 molar equiv) were added and the reaction mixture was stirred at room temperature in an open flask. After 5 h, the reaction mixture was filtered through a short silica plug and the product was eluted with CH_2Cl_2 to yield 73.1 mg (98%) of 4-methoxyphenyl azide² as brownish oil. ^1H NMR (500 MHz, CDCl_3): δ 6.95 (d, 2H, Ar-H, $J = 9.2$ Hz), 6.89 (d, 2H, Ar-H, $J = 8.8$ Hz), 3.80 (s, 3H, CH_3).

General Procedure for Synthesis of Alkyl Azides. To a stirring solution of alkyl halide (1 molar equiv) in DMF (70.0 mL), was added NaN_3 (2 molar equiv). The reaction mixture was stirred at 80 °C for 7 h, cooled to room temperature, 20 mL of water was added and the mixture was poured into separatory funnel containing EtOAc. Organic layer was separated and the aqueous layer was extracted with EtOAc (3x), combined organic layer was washed with water

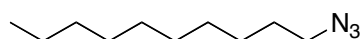
and brine, dried over anhydrous Na_2SO_4 and the solvent was evaporated under reduced pressure. The alkyl azide obtained was used without further purification.

3-Phenylpropyl Azide³



3-Phenylpropyl bromide: 1.00 g (5.02 mmol); NaN_3 : 0.650 g (10.0 mmol). Yield: 700 mg (87%) of 3-phenylpropyl azide as a colorless oil. ^1H NMR (500 MHz, CDCl_3): δ 7.31 (t, 2H, Ar-H, J = 7.6 Hz), 7.23-7.19 (m, 3H, Ar-H), 3.29 (t, 2H, CH_2 , J = 6.9 Hz), 2.72 (t, 2H, CH_2 , J = 7.6 Hz), 1.96-1.90 (m, 2H, CH_2).

Decyl Azide⁴

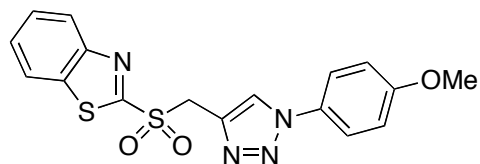


Decyl bromide: 0.500 g (2.26 mmol); NaN_3 : 0.294 g (4.52 mmol). Yield: 172 mg (42%) of decyl azide as a colorless oil. ^1H NMR (500 MHz, CDCl_3): δ 3.25 (t, 2H, CH_2 , J = 6.9 Hz), 1.60 (m, 2H, CH_2 , J = 7.4 Hz), 1.27-1.63 (m, 14H, $(\text{CH}_2)_7$), 0.88 (t, 3H, CH_3 , J = 7.4 Hz).

Synthesis of Triazoles

General Procedure. To a stirring solution of azide (1 molar equiv) in 4:1 (v/v) $\text{CH}_2\text{Cl}_2/\text{MeOH}$ (28.0 mL per mmol of azide), sulfone **1** or **2** (1 to 1.2 molar equiv), $\text{Cu}(\text{CH}_3\text{CN})_4\text{PF}_6$ (0.20 molar equiv) and AgBF_4 (0.20 molar equiv) were added sequentially. The stirring was continued at room temperature until TLC showed disappearance of the azide. The solvents were evaporated under reduced pressure and the crude reaction mixture was purified by column chromatography on silica gel (mesh 200-300). Eluting solvents for chromatography are indicated under the specific compound headings.

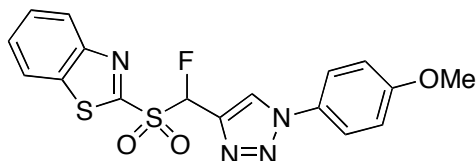
2-[[1-(4-Methoxyphenyl)-1H-1,2,3-triazol-4-yl]methylsulfonyl]benzo[d]thiazole (3)



4-Methoxyphenyl azide: 250 mg (1.68 mmol); sulfone **1**: 624 mg (2.02 mmol). Column chromatography: eluting solvent 20% acetone in hexanes. Yield: 481 mg (74%) of off white solid. ^1H NMR (500 MHz, CDCl_3): δ 8.25 (d, 1H, Ar-H, J = 7.8 Hz), 8.14 (s, 1H), 7.97 (d, 1H, Ar-H, J = 7.8 Hz), 7.65 (t, 1H, Ar-H, J = 7.8 Hz), 7.61-7.58 (m, 3H, Ar-H), 7.02 (d, 2H, Ar-H, J = 8.8

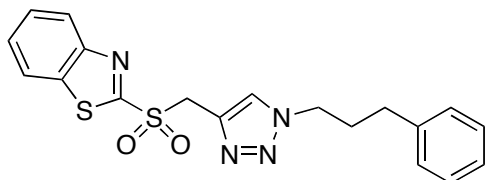
Hz), 5.03 (s, 2H, CH₂), 3.87 (s, 3H, CH₃). ¹³C NMR (125 MHz, CDCl₃): δ 164.4, 160.4, 152.8, 137.3, 135.1, 130.3, 128.4, 128.0, 126.0, 123.3, 122.52, 122.47, 115.1, 55.9, 52.9. HRMS (ESI) calcd. for C₁₇H₁₅N₄O₃S₂ [M + H]⁺ 387.0580, found 387.0582.

2-{Fluoro[1-(4-methoxyphenyl)-1H-1,2,3-triazol-4-yl]methylsulfonyl}benzo[d]-thiazole (4)



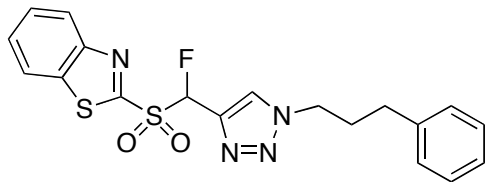
4-Methoxyphenyl azide: 300 mg (2.01 mmol); sulfone **2**: 723 mg (2.21 mmol). Column chromatography: eluting solvent 20% EtOAc in hexanes. Yield: 756 mg (93%) of white solid. ¹H NMR (500 MHz, CDCl₃): δ 8.45 (d, 1H, Ar-H, *J* = 1.4 Hz), 8.32 (d, 1H, Ar-H, *J* = 8.3 Hz), 8.06 (d, 1H, Ar-H, *J* = 7.8 Hz), 7.71-7.64 (m, 4H, Ar-H), 7.05 (d, 2H, Ar-H, *J* = 8.7 Hz), 7.01 (d, 1H, CHF, ²*J*_{FH} = 46.1 Hz), 3.88 (s, 3H, OCH₃). ¹³C NMR (125 MHz, CDCl₃): δ 162.0, 160.6, 153.0, 137.8, 135.7 (d, ²*J*_{CF} = 24.0 Hz), 129.9, 128.8, 128.2, 126.1, 124.1, 122.7, 122.5, 115.1, 96.1 (d, ¹*J*_{CF} = 220.1 Hz), 55.9. ¹⁹F NMR (282 MHz, CDCl₃): δ -165.3 (d, ²*J*_{FH} = 45.8 Hz). HRMS (ESI) calcd. for C₁₇H₁₄FN₄O₃S₂ [M + H]⁺ 405.0486, found 405.0489.

2-[[1-(3-Phenylpropyl)-1H-1,2,3-triazol-4-yl]methylsulfonyl]benzo[d]thiazole (5)



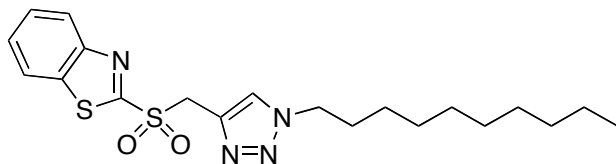
3-Phenylpropyl azide: 300 mg (1.86 mmol); sulfone **1**: 689 mg (2.23 mmol). Column chromatography: eluting solvent 40% EtOAc in hexanes. Yield: 623 mg (84%) of white solid. ¹H NMR (500 MHz, CDCl₃): δ 8.22 (d, 1H, Ar-H, *J* = 8.3 Hz), 7.92 (d, 1H, Ar-H, *J* = 7.8 Hz), 7.73 (s, 1H, Ar-H), 7.62 (t, 1H, Ar-H, *J* = 7.6 Hz), 7.57 (t, 1H, Ar-H, *J* = 7.8 Hz), 7.30 (t, 2H, Ar-H, *J* = 7.6 Hz), 7.23 (t, 1H, Ar-H, *J* = 7.4 Hz), 7.14 (d, 1H, Ar-H, *J* = 7.8 Hz), 4.96 (s, 2H, CH₂), 4.34 (t, 2H, CH₂, *J* = 7.1 Hz), 2.60 (t, 2H, CH₂, *J* = 7.6 Hz), 2.23 (quint, 2H, CH₂, *J* = 7.4 Hz). ¹³C NMR (125 MHz, CDCl₃): δ 164.3, 152.8, 140.1, 137.2, 134.5, 128.8, 128.6, 128.3, 127.9, 126.6, 125.9, 125.0, 122.4, 52.9, 49.9, 32.5, 31.6. HRMS (ESI) calcd. for C₁₉H₁₉N₄O₂S₂ [M + H]⁺ 399.0944, found 399.0943.

2-{Fluoro[1-(3-phenylpropyl)-1H-1,2,3-triazol-4-yl]methylsulfonyl}benzo[d]-thiazole (6)



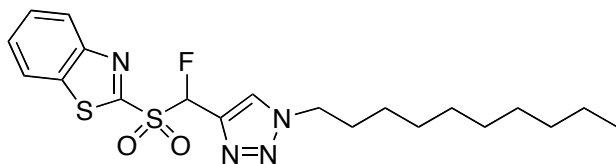
3-Phenylpropyl azide: 500 mg (3.10 mmol); sulfone **2**: 1.01 g (3.10 mmol). Column chromatography: eluting solvent 20% EtOAc in hexanes. Yield: 879 mg (68%) of white solid. ^1H NMR (500 MHz, CDCl_3): δ 8.27 (d, 1H, Ar-H, $J = 8.3$ Hz), 8.08 (d, 1H, Ar-H, $J = 1.8$ Hz), 8.01 (d, 1H, Ar-H, $J = 7.8$ Hz), 7.68-7.61 (m, 2H, Ar-H), 7.30 (t, 2H, Ar-H, $J = 7.6$ Hz), 7.22 (t, 1H, Ar-H, $J = 7.4$ Hz), 7.17 (d, 2H, Ar-H, $J = 7.4$ Hz), 6.94 (d, 1H, CHF, $^2J_{\text{FH}} = 46.5$ Hz), 4.42 (t, 2H, CH_2 , $J = 7.4$ Hz), 2.67 (t, 2H, CH_2 , $J = 7.4$ Hz), 2.29 (quint, 2H, CH_2 , $J = 7.4$ Hz). ^{13}C NMR (125 MHz, CDCl_3): δ 162.0, 153.0, 139.9, 137.7, 135.1 (d, $^2J_{\text{CF}} = 24.2$ Hz), 128.9, 128.8, 128.6, 128.2, 126.7, 126.1, 125.8, 122.5, 96.2 (d, $^1J_{\text{CF}} = 219.7$ Hz), 50.2, 32.5, 31.5. ^{19}F NMR (282 MHz, CDCl_3): δ -165.4 (d, $^2J_{\text{FH}} = 45.8$ Hz). HRMS (ESI) calcd. for $\text{C}_{19}\text{H}_{18}\text{FN}_4\text{O}_2\text{S}_2$ [$\text{M} + \text{H}$] $^+$ 417.0850, found 417.0852.

2-[(1-Decyl-1H-1,2,3-triazol-4-yl)methylsulfonyl]benzo[d]thiazole (**7**)



Decyl azide: 300 mg (1.64 mmol); sulfone **1**: 609 mg (1.97 mmol). Column chromatography: eluting solvent 20% EtOAc in hexanes. Yield: 555 mg (81%) of white solid. ^1H NMR (500 MHz, CDCl_3): δ 8.22 (d, 1H, Ar-H, $J = 8.2$ Hz), 7.95 (d, 1H, Ar-H, $J = 8.2$ Hz), 7.75 (s, 1H, Ar-H), 7.63 (t, 1H, Ar-H, $J = 7.2$ Hz), 7.57 (t, 1H, Ar-H, $J = 7.4$ Hz), 4.95 (s, 2H, CH_2), 4.32 (t, 2H, CH_2 , $J = 7.2$ Hz), 1.89-1.83 (m, 2H, CH_2), 1.28-1.25 (m, 14H, $(\text{CH}_2)_7$), 0.87 (t, 3H, CH_3 , $J = 6.9$ Hz). ^{13}C NMR (125 MHz, CDCl_3): δ 164.4, 152.8, 137.3, 134.4, 128.4, 127.9, 125.9, 124.8, 122.4, 52.9, 50.8, 32.0, 30.3, 29.64, 29.6, 29.4, 29.1, 26.6, 22.8, 14.3. HRMS (ESI) calcd. for $\text{C}_{20}\text{H}_{29}\text{N}_4\text{O}_2\text{S}_2$ [$\text{M} + \text{H}$] $^+$ 421.1726, found 421.173.

2-[(1-Decyl-1H-1,2,3-triazol-4-yl)fluoromethylsulfonyl]benzo[d]thiazole (**8**)



Decyl azide: 91.6 mg (0.500 mmol); sulfone **2**: 164 mg (0.500 mmol). Column chromatography: eluting solvent 20% EtOAc in hexanes. Yield: 178 mg (81%) of white solid. ^1H NMR (500 MHz, CDCl_3): δ 8.30 (d, 1H, Ar-H, $J = 8.3$ Hz), 8.09 (d, 1H, Ar-H, $J = 1.8$ Hz), 8.05 (d, 1H, Ar-H, $J = 7.8$ Hz), 7.70-7.63 (m, 2H, Ar-H), 6.93 (d, 1H, CHF, $^2J_{\text{FH}} = 46.5$ Hz), 4.48-4.38 (m, 2H, CH_2), 1.98-1.93 (m, 2H, CH_2), 1.34-1.26 (m, 14H, $(\text{CH}_2)_7$), 0.88 (t, 3H, CH_3 , $J = 6.9$ Hz). ^{13}C NMR (125 MHz, CDCl_3): δ 162.0, 153.0, 137.7, 135.1 (d, $^2J_{\text{CF}} = 24.3$ Hz), 128.8, 128.2, 126.1, 125.6, 122.5, 96.2 (d, $^1J_{\text{CF}} = 219.7$ Hz), 51.2, 32.0, 30.3, 29.6, 29.5, 29.4, 29.1, 26.6, 22.8, 14.3. ^{19}F NMR (282 MHz, CDCl_3): δ -165.0 (d, $^2J_{\text{FH}} = 46.1$ Hz). HRMS (ESI) calcd. for $\text{C}_{20}\text{H}_{28}\text{FN}_4\text{O}_2\text{S}_2$ [$\text{M} + \text{H}$] $^+$ 439.1632, found 439.1635.

Competitive Click Reaction

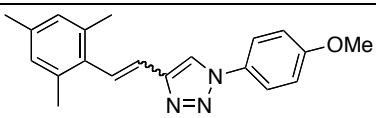
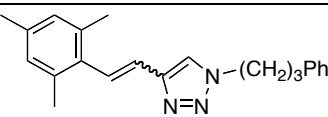
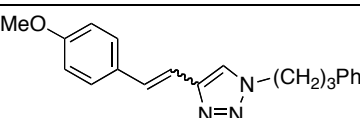
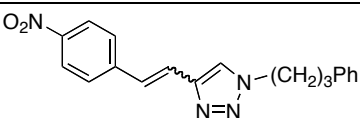
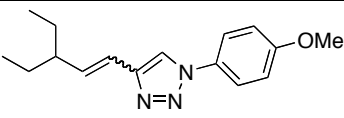
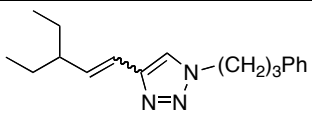
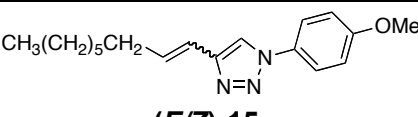
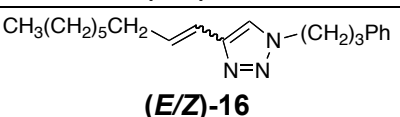
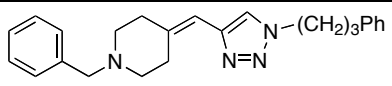
General Procedure. To a stirring solution of azide (0.155 mmol, 1 molar equiv) in 4:1 $\text{CH}_2\text{Cl}_2/\text{MeOH}$ solvent (4.5 mL), sulfones **1** and **2** (0.31 mmol each, 2 molar equiv each), $\text{Cu}(\text{CH}_3\text{CN})_4\text{PF}_6$ (46.0 mg, 0.124 mmol, 0.8 molar equiv) and AgBF_4 (24.1 mg, 0.124 mmol, 0.8 molar equiv) were added sequentially. The stirring was continued at room temperature until TLC showed disappearance of the azide. The solvents were evaporated under reduced pressure and the crude reaction mixture was analyzed by ^1H NMR (500 MHz) using CDCl_3 as solvent.

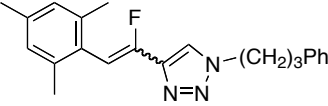
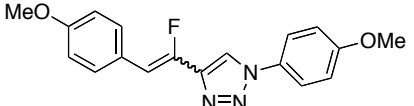
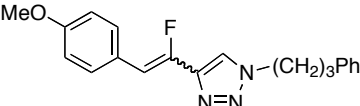
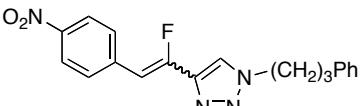
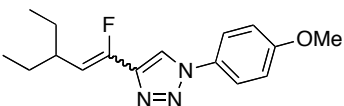
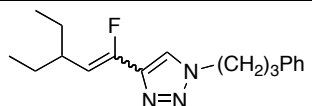
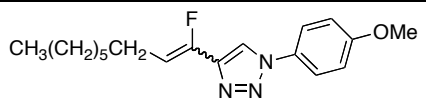
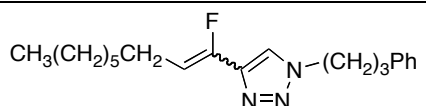
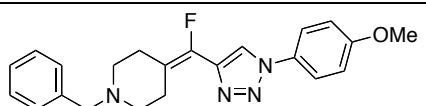
Synthesis of Vinyl Triazoles

General Procedure. A stirring solution of aldehyde (0.5 mmol, 1 molar equiv) and triazole (0.6 mmol, 1.2 molar equiv) in DMF (3.8 mL) and DMPU (3.8 mL) was cooled to -78 °C (dry ice/*iso*-PrOH) and under nitrogen LHMDS (1.2 mmol, 2.4 molar equiv) was added to the reaction mixture. The reaction mixture was stirred at -78 °C for 5 min, saturated aq NH_4Cl was added and the mixture was poured into EtOAc. Organic layer was separated and the aqueous layer was extracted with EtOAc three times. The combined organic layer was washed with water and brine and dried over anhydrous Na_2SO_4 . The solvent was evaporated under reduced pressure and the combined *E/Z* product mixture was isolated by column chromatography using silica gel (mesh 200-300). The product *E/Z* ratio was determined by ^1H NMR for vinyl and by ^{19}F NMR for fluorovinyl derivatives, prior to column chromatography purification. Eluting solvent for chromatography, HRMS and ^{19}F NMR data (for fluorovinyl derivatives) of products are displayed

for each individual substrate in the Table. For all compounds shown in the Table, ^{19}F NMR spectra were recorded in CDCl_3 solvent, at 282 MHz using CFCl_3 as internal standard.

Table 1 Eluting solvents for column chromatography, HRMS data of compounds **9** to **26** and ^{19}F NMR data of compounds **18** to **26**

Compound	Eluting solvent	HRMS and ^{19}F NMR data where applicable
 <p>(E/Z)-9</p>	20% EtOAc in hexanes	HRMS (ESI) calcd. for $\text{C}_{20}\text{H}_{22}\text{N}_3\text{O}$ $[\text{M}+\text{H}]^+$ 320.1757, found 320.176.
 <p>(E/Z)-10</p>	20% EtOAc in hexanes	HRMS (ESI) calcd. for $\text{C}_{22}\text{H}_{26}\text{N}_3$ $[\text{M}+\text{H}]^+$ 332.2121, found 332.2126.
 <p>(E/Z)-11</p>	40% EtOAc in hexanes	HRMS (ESI) calcd. for $\text{C}_{20}\text{H}_{22}\text{N}_3\text{O}$ $[\text{M}+\text{H}]^+$ 320.1757, found 320.1761.
 <p>(E/Z)-12</p>	40% EtOAc in hexanes	HRMS (ESI) calcd. for $\text{C}_{19}\text{H}_{19}\text{N}_4\text{O}_2$ $[\text{M}+\text{H}]^+$ 335.1503, found 335.1506.
 <p>(E/Z)-13</p>	20% EtOAc in hexanes	HRMS (ESI) calcd. for $\text{C}_{16}\text{H}_{22}\text{N}_3\text{O}$ $[\text{M}+\text{H}]^+$ 272.1757, found 272.1757.
 <p>(E/Z)-14</p>	20% EtOAc in hexanes	HRMS (ESI) calcd. for $\text{C}_{18}\text{H}_{26}\text{N}_3$ $[\text{M}+\text{H}]^+$ 284.2121, found 284.2124.
 <p>(E/Z)-15</p>	20% EtOAc in hexanes	HRMS (ESI) calcd. for $\text{C}_{18}\text{H}_{26}\text{N}_3\text{O}$ $[\text{M}+\text{H}]^+$ 300.2070, found 300.2071.
 <p>(E/Z)-16</p>	20% EtOAc in hexanes	HRMS (ESI) calcd. for $\text{C}_{20}\text{H}_{30}\text{N}_3$ $[\text{M}+\text{H}]^+$ 312.2434, found 312.2437.
 <p>17</p>	40% EtOAc in hexanes	HRMS (ESI) calcd. for $\text{C}_{24}\text{H}_{29}\text{N}_4$ $[\text{M}+\text{H}]^+$ 373.2387, found 373.2394.

 (E/Z)-18	20% EtOAc in hexanes	^{19}F NMR: δ -114.2 (d, $^3J_{\text{FH}} = 18.4$ Hz, <i>E</i> isomer), δ -115.5 (d, $^3J_{\text{FH}} = 42.6$ Hz, <i>Z</i> isomer). HRMS (ESI) calcd. for $\text{C}_{22}\text{H}_{25}\text{FN}_3$ [$\text{M} + \text{H}$] $^+$ 350.2027, found 350.2034.
 (E/Z)-19	20% EtOAc in hexanes	^{19}F NMR: δ -108.5 (d, $^3J_{\text{FH}} = 21.4$ Hz, <i>E</i> isomer), δ -121.5 (d, $^3J_{\text{FH}} = 42.7$ Hz, <i>Z</i> isomer). HRMS (ESI) calcd. for $\text{C}_{18}\text{H}_{17}\text{FN}_3\text{O}_2$ [$\text{M} + \text{H}$] $^+$ 326.1299, found 326.1304.
 (E/Z)-20	40% EtOAc in hexanes	^{19}F NMR: δ -108.2 (d, $^3J_{\text{FH}} = 21.4$ Hz, <i>E</i> isomer), δ -121.3 (d, $^3J_{\text{FH}} = 42.7$ Hz, <i>Z</i> isomer). HRMS (ESI) calcd. for $\text{C}_{20}\text{H}_{21}\text{FN}_3\text{O}$ [$\text{M} + \text{H}$] $^+$ 338.1663, found 338.1667.
 (E/Z)-21	20% EtOAc in hexanes	^{19}F NMR: δ -100.6 (d, $^3J_{\text{FH}} = 21.4$ Hz, <i>E</i> isomer), δ -113.4 (d, $^3J_{\text{FH}} = 39.7$ Hz, <i>Z</i> isomer). HRMS (ESI) calcd. for $\text{C}_{19}\text{H}_{18}\text{FN}_4\text{O}_2$ [$\text{M} + \text{H}$] $^+$ 353.1408, found 353.1411.
 (E/Z)-22	20% EtOAc in hexanes	^{19}F NMR: δ -114.3 (d, $^3J_{\text{FH}} = 24.4$ Hz, <i>E</i> isomer), δ -124.7 (d, $^3J_{\text{FH}} = 39.7$ Hz, <i>Z</i> isomer). HRMS (ESI) calcd. for $\text{C}_{16}\text{H}_{21}\text{FN}_3\text{O}$ [$\text{M} + \text{H}$] $^+$ 290.1663, found 290.1666.
 (E/Z)-23	20% EtOAc in hexanes	^{19}F NMR: δ -114.0 (d, $^3J_{\text{FH}} = 24.4$ Hz, <i>E</i> isomer), δ -124.6 (d, $^3J_{\text{FH}} = 39.7$ Hz, <i>Z</i> isomer). HRMS (ESI) calcd. for $\text{C}_{18}\text{H}_{25}\text{FN}_3$ [$\text{M} + \text{H}$] $^+$ 302.2027, found 302.2031.
 (E/Z)-24	20% EtOAc in hexanes	^{19}F NMR: δ -115.6 (d, $^3J_{\text{FH}} = 21.4$ Hz, <i>E</i> isomer), δ -124.6 (d, $^3J_{\text{FH}} = 39.7$ Hz, <i>Z</i> isomer). HRMS (ESI) calcd. for $\text{C}_{18}\text{H}_{25}\text{FN}_3\text{O}$ [$\text{M} + \text{H}$] $^+$ 318.1976, found 318.1983.
 (E/Z)-25	20% EtOAc in hexanes	^{19}F NMR: δ -115.3 (d, $^3J_{\text{FH}} = 21.4$ Hz, <i>E</i> isomer), δ -124.5 (d, $^3J_{\text{FH}} = 36.6$ Hz, <i>Z</i> isomer). HRMS (ESI) calcd. for $\text{C}_{20}\text{H}_{28}\text{FN}_3\text{Na}$ [$\text{M} + \text{Na}$] $^+$ 352.2159, found 352.2161.
 26	40% EtOAc in hexanes	^{19}F NMR: δ -122.0 (s). HRMS (ESI) calcd. for $\text{C}_{22}\text{H}_{24}\text{FN}_4\text{O}$ [$\text{M} + \text{H}$] $^+$ 379.1929, found 379.1924.

Data from DFT Computational Analysis

All quantum mechanical calculations were done using Density Functional Theory (DFT) method based on Becke3-Lee-Yang-Parr (B3LYP) level with 6-311++G(2d,2p) basis sets available with Gaussian 9 programs.⁵ Initial geometries of the protio- and the fluoro-analogue (structures shown in Table 2) were first optimized. The optimized coordinates were then utilized for further calculations to estimate the energies of the HOMO and LUMO. Visualizations of molecular orbitals and estimation of their energy were performed using a personal computer-based GaussView 4.1.2 program.⁶ GaussView 4 generated the MOs from the checkpoint files and extracted the relevant energies in Hartree which was then converted to kcal/mol. Natural bond orbital (NBO) analyses⁷ were performed on the optimized geometries to calculate atom-atom overlap-weighted natural atomic orbital (NAO) bond orders and the natural charges.

Table 2 HOMO and LUMO energies of benzothiazolyl propargyl sulfone and benzothiazolyl fluoropropargyl sulfone

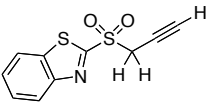
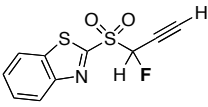
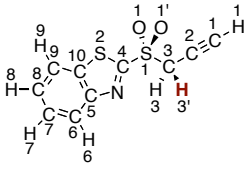
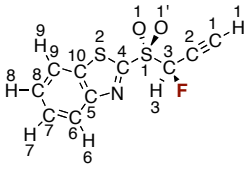
		
	Energy (kcal/mol)	Energy (kcal/mol)
LUMO	-53.1	-56.8
HOMO	-167.6	-167.8

Table 3 NBO analyzed natural charges of benzothiazolyl propargyl sulfone and benzothiazolyl fluoropropargyl sulfone

			
Atom Number	NBO Analyzed Natural Charges	Atom Number	NBO Analyzed Natural Charges
H1	0.230	H1	0.232
C1	-0.162	C1	-0.110
C2	-0.050	C2	-0.101
C3	-0.636	C3	-0.004
H3	0.252	H3	0.228

H3'	0.266	F	-0.353
S1	2.063	S1	2.027
O1	-0.881	O1	-0.878
O1'	-0.908	O1'	-0.889
C4	-0.186	C4	-0.191
N	-0.444	N	-0.436
S2	0.464	S2	0.474
C5	0.101	C5	0.102
C6	-0.175	C6	-0.173
H6	0.223	H6	0.223
C7	-0.200	C7	-0.199
H7	0.211	H7	0.211
C8	-0.182	C8	-0.180
H8	0.211	H8	0.211
C9	-0.211	C9	-0.211
H9	0.218	H9	0.218
C10	-0.205	C10	-0.203

References:

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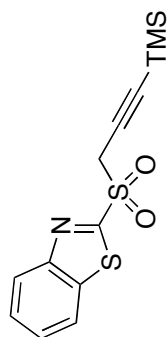
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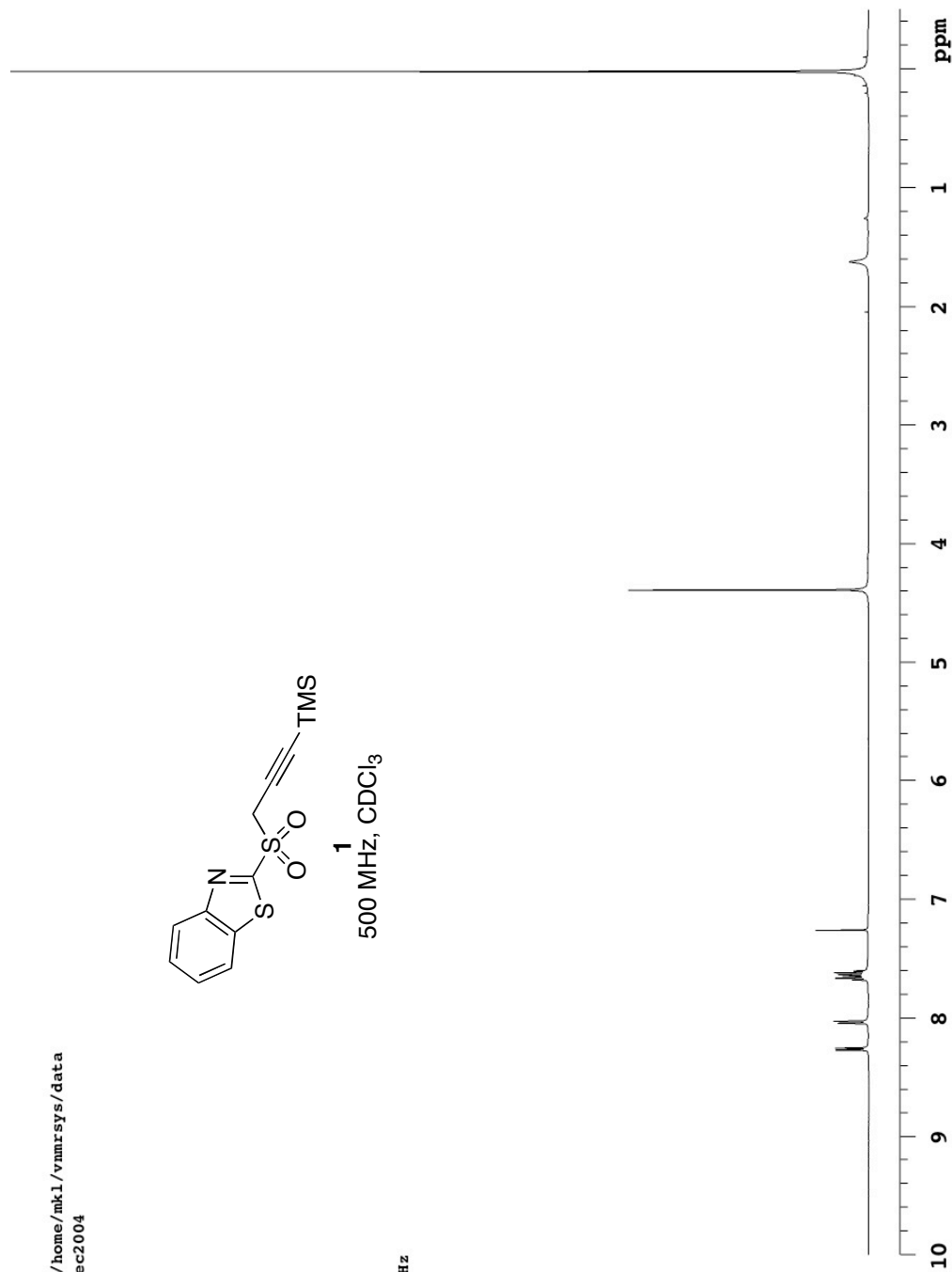
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DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 3 min, 5 sec



1

500 MHz, CDCl₃



1222-RK-02-139-13C

Pulse Sequence: s2pul

Solvent: CDCl₃

Temp. 25.0 C / 298.1 K

Operator: Barbara

INOVA-500 "capella500"

Relax. delay 4.000 sec

Pulse 52.1 degrees

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Width 29996.3 Hz

52 repetitions

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Power 42 dB

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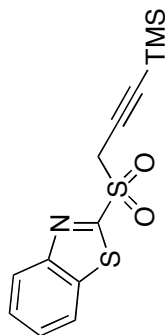
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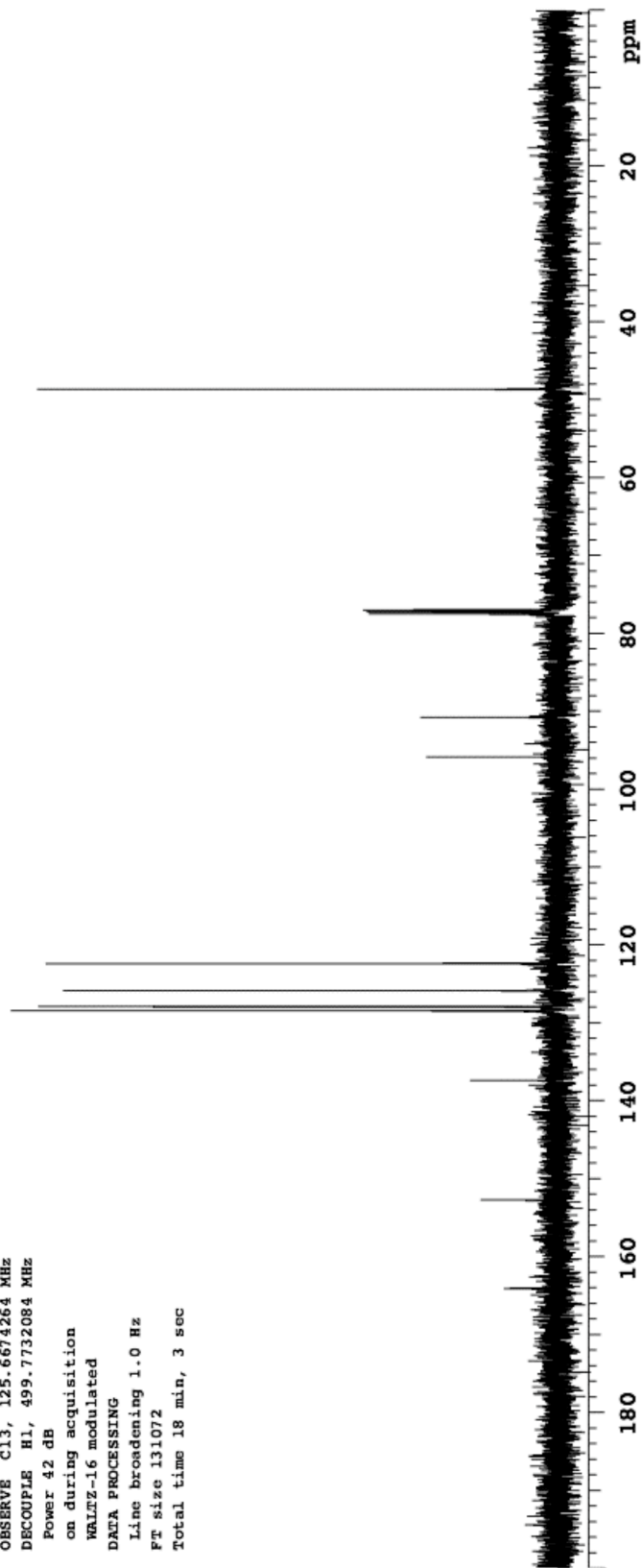
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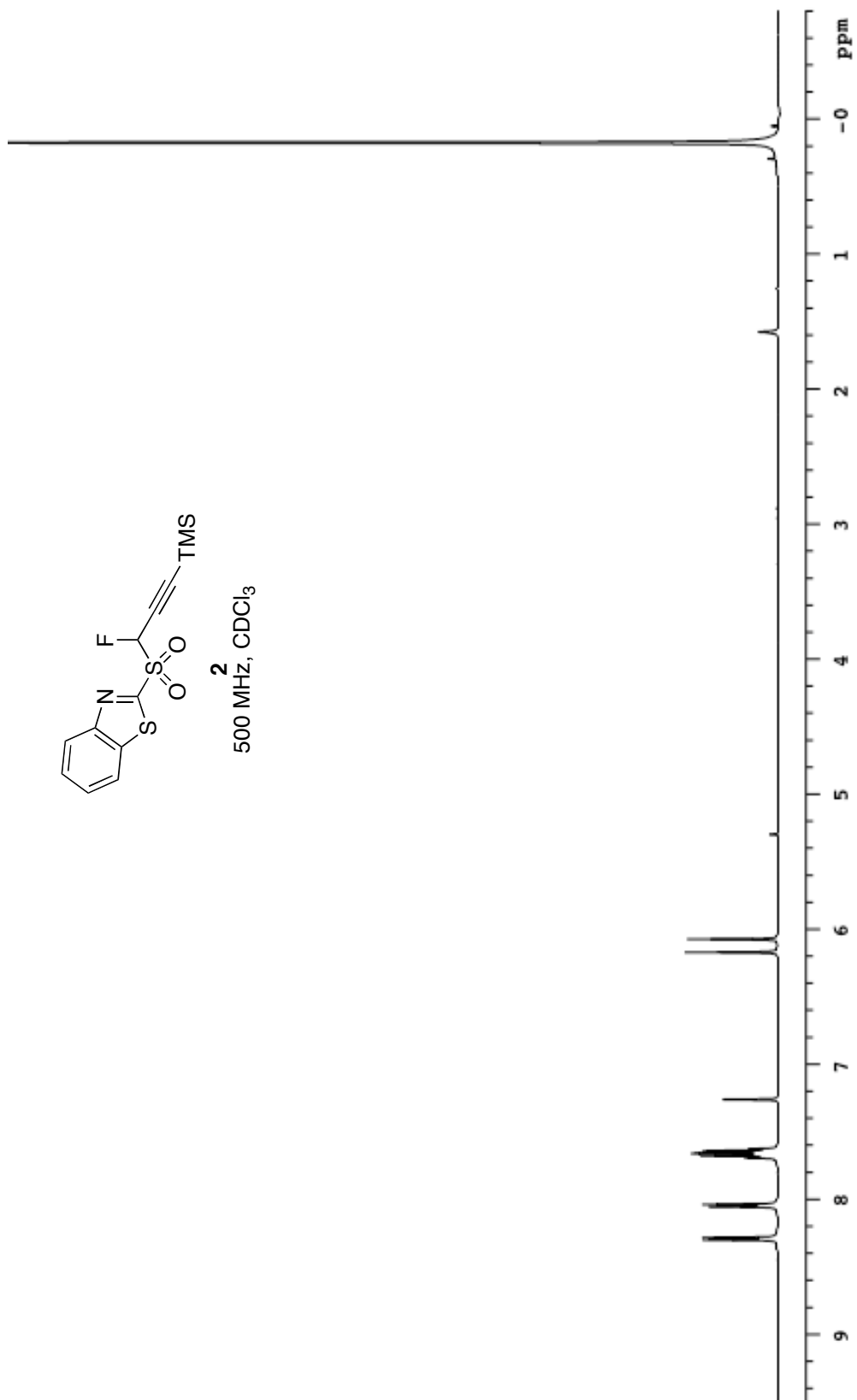
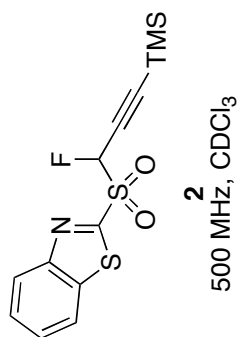
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Operator: Barbara

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INOVA-500 "riga"

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Pulse 52.1 degrees

Acq. time 1.300 sec

Width 29996.3 Hz

100 repetitions

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DECOUPLE H1, 499.7732084 MHz

Power 42 dB

on during acquisition

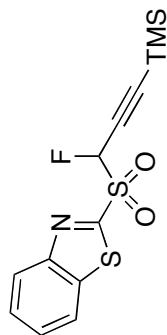
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

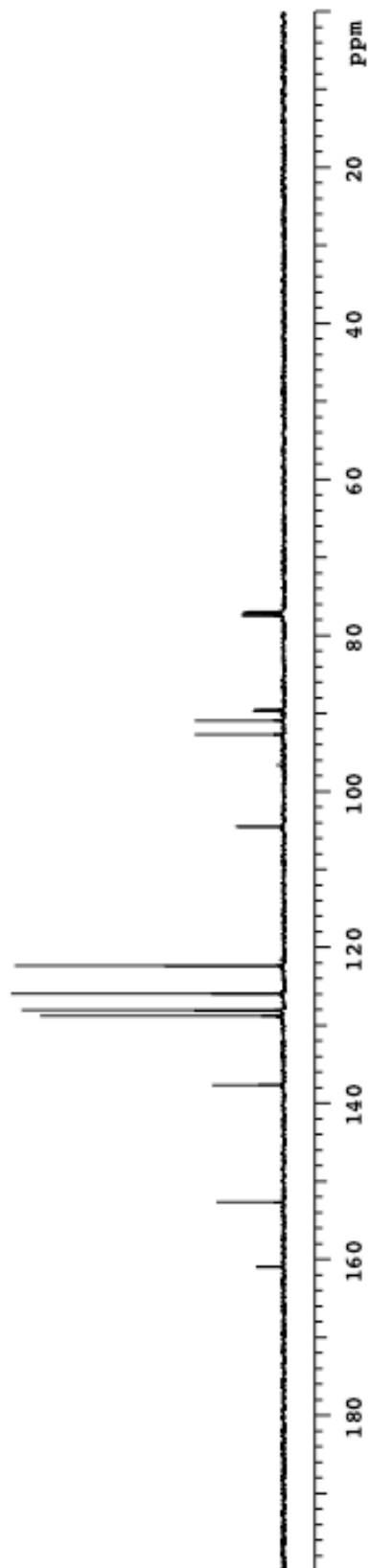
FT size 131072

Total time 9 min, 12 sec



2

125 MHz, CDCl₃



1222-MK-02-117-crude

Archive directory: /export/home/mkl/vmarsys/data
Sample directory: ato_13Dec2004
File: 1222-MK-02-117-crude

Pulse Sequence: s2pul

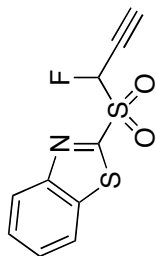
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: Barbara
File: 1222-MK-02-117-crude
INOVA-500 "capella500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7544.3 Hz
64 repetitions

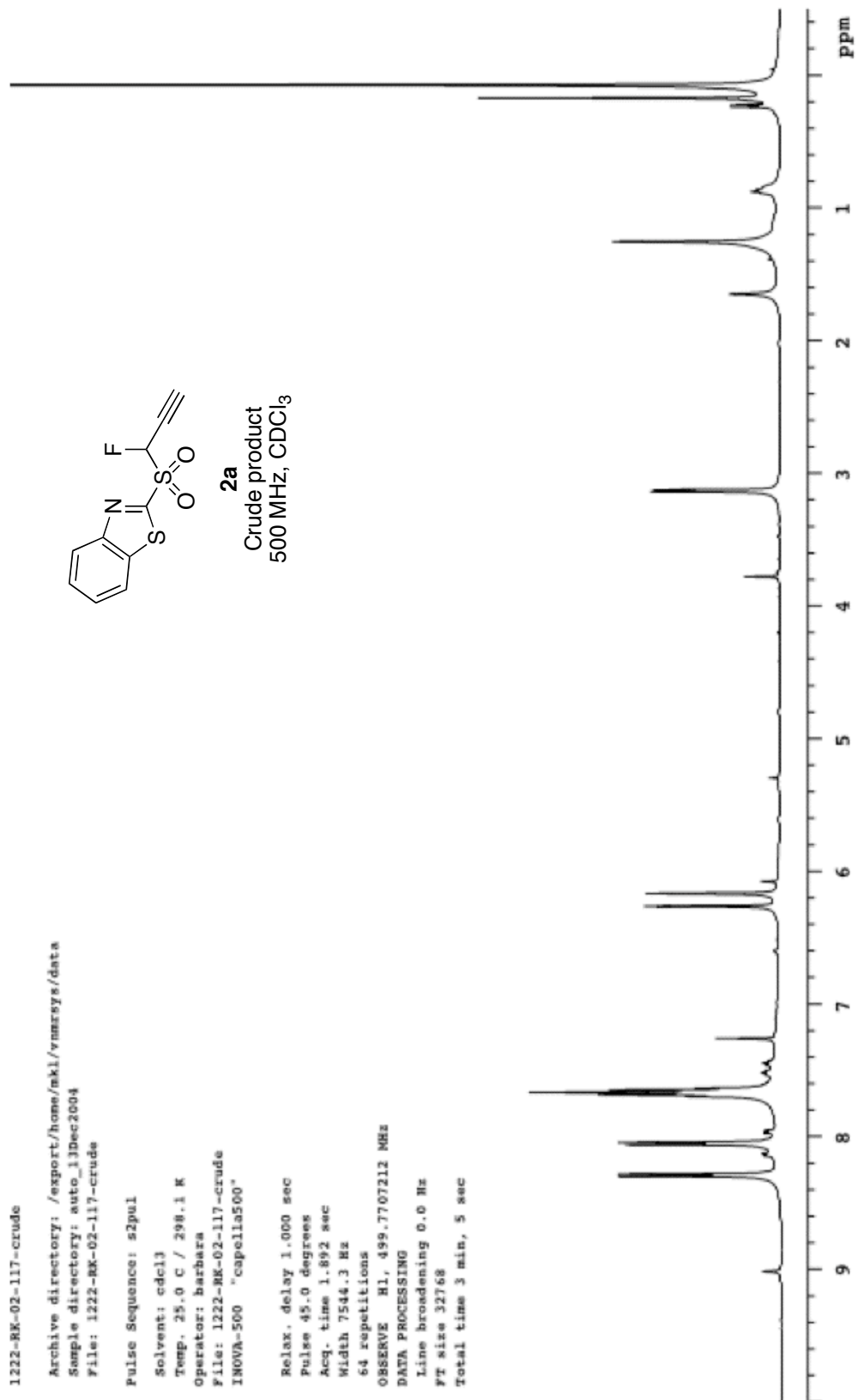
OBSERVE H1, 499.7707212 MHz

DATA PROCESSING

Line broadening 0.0 Hz
FT size 32768
Total time 3 min, 5 sec



2a
Crude product
500 MHz, CDCl₃

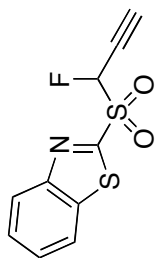


1222-RK-02-117-13C

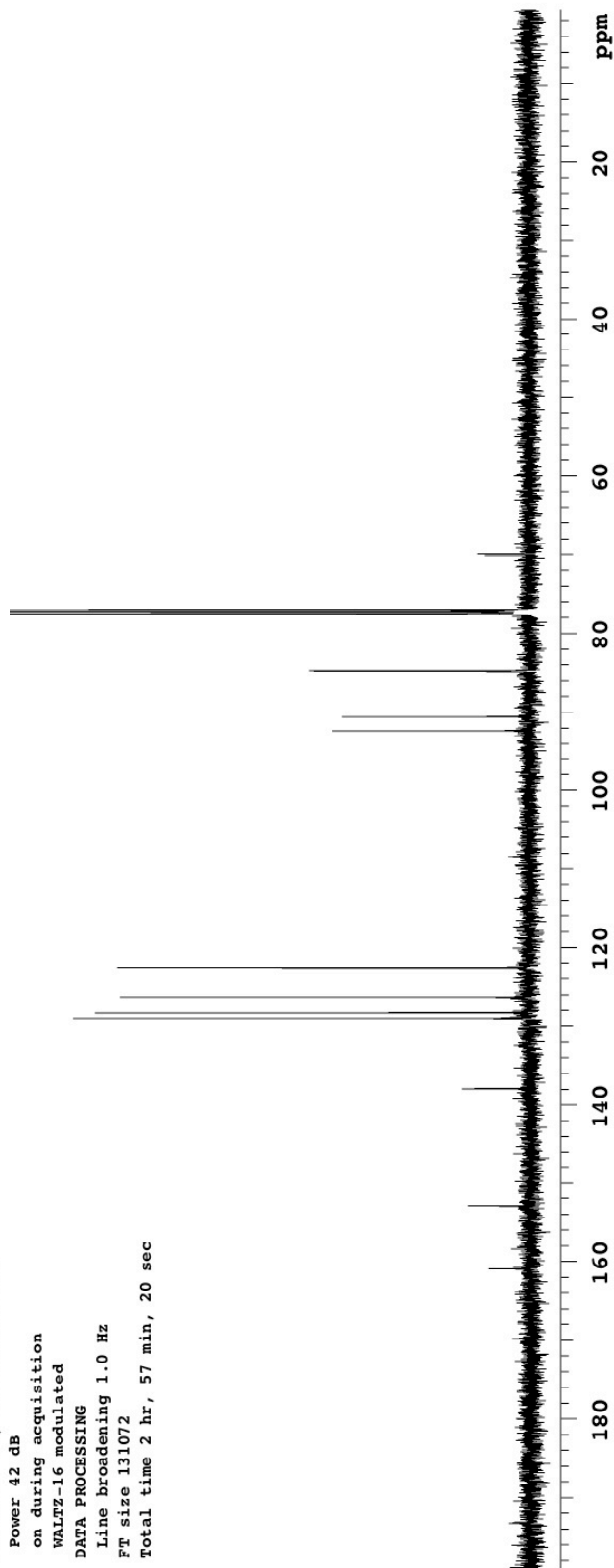
Pulse Sequence: s2pul

Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
Operator: barbara
INOVA-500 "capella500"

Relax. delay 4.000 sec
Pulse 52.1 degrees
Acq. time 1.300 sec
Width 29996.3 Hz
696 repetitions
OBSERVE C13, 125.6674205 MHz
DECOUPLE H1, 499.7732084 MHz
Power 42 dB
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 2 hr, 57 min, 20 sec



2a
Crude product
125 MHz, CDCl₃



1222-MK-06-424-pure

Archive directory: /export/home/mkl/vmrssys/data
Sample directory: auto_13Dec2004

Pulse Sequence: s2pul

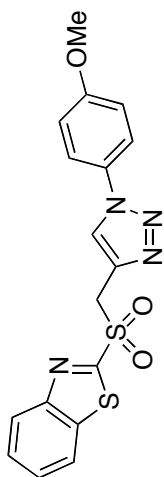
Solvent: cdcl3

Temp. 25.0 C / 298.1 K

Operator: barbara

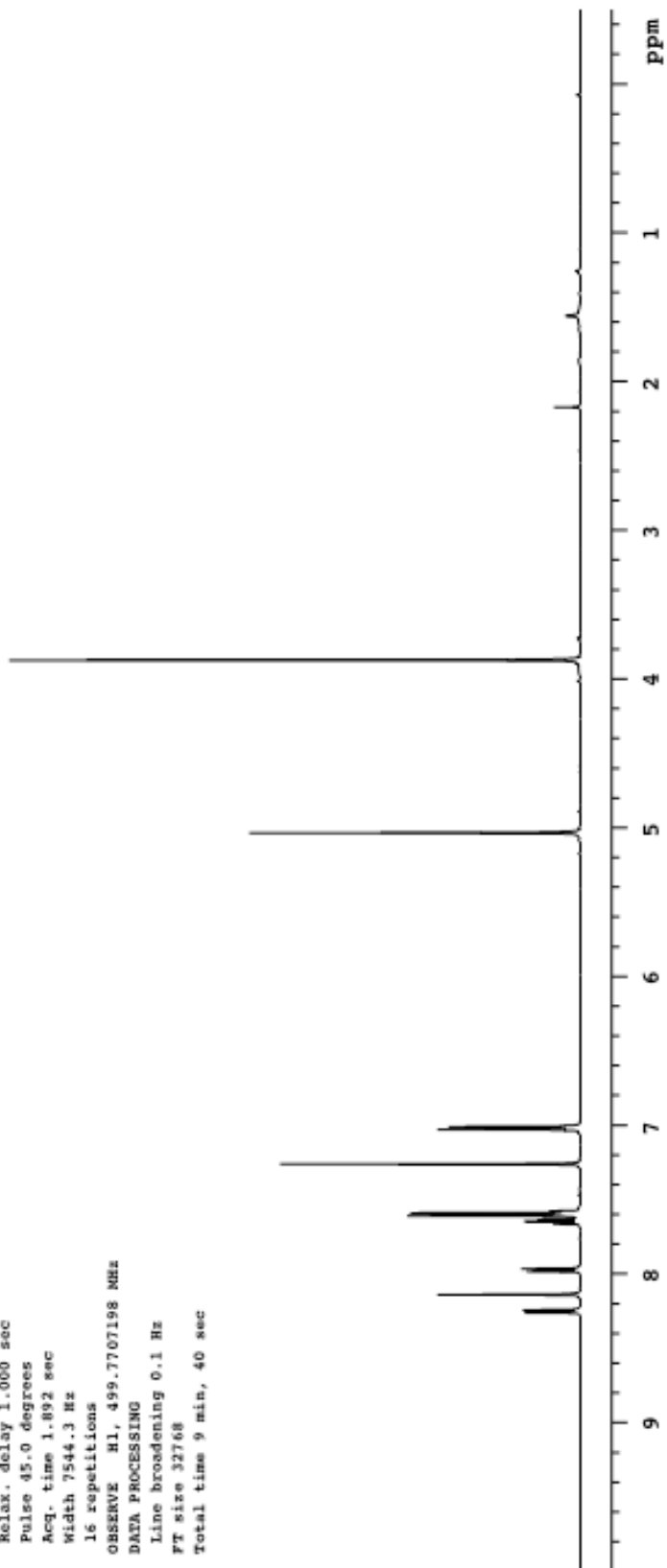
File: 1222-MK-06-424-pure

INOVA-500 "ziga"



3
500 MHz, CDCl₃

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7544.3 Hz
16 repetitions
OBSERVE H1, 499.7707198 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 9 min, 40 sec



1222-RK-06-424-13C

Archive directory: /export/home/barbara/vnarsys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl3

Temp. 25.0 C / 298.1 K

Operator: barbara

File: 1222-RK-06-424-13C

INOVA-500 "ziga"

Relax. delay 4.000 sec

Pulse 52.1 degrees

Acq. time 1.300 sec

Width 29996.3 Hz

1768 repetitions

OBSERVE C13, 125.6674182 MHz

DECOUPLE H1, 499.7732084 MHz

Power 42 dB

on during acquisition

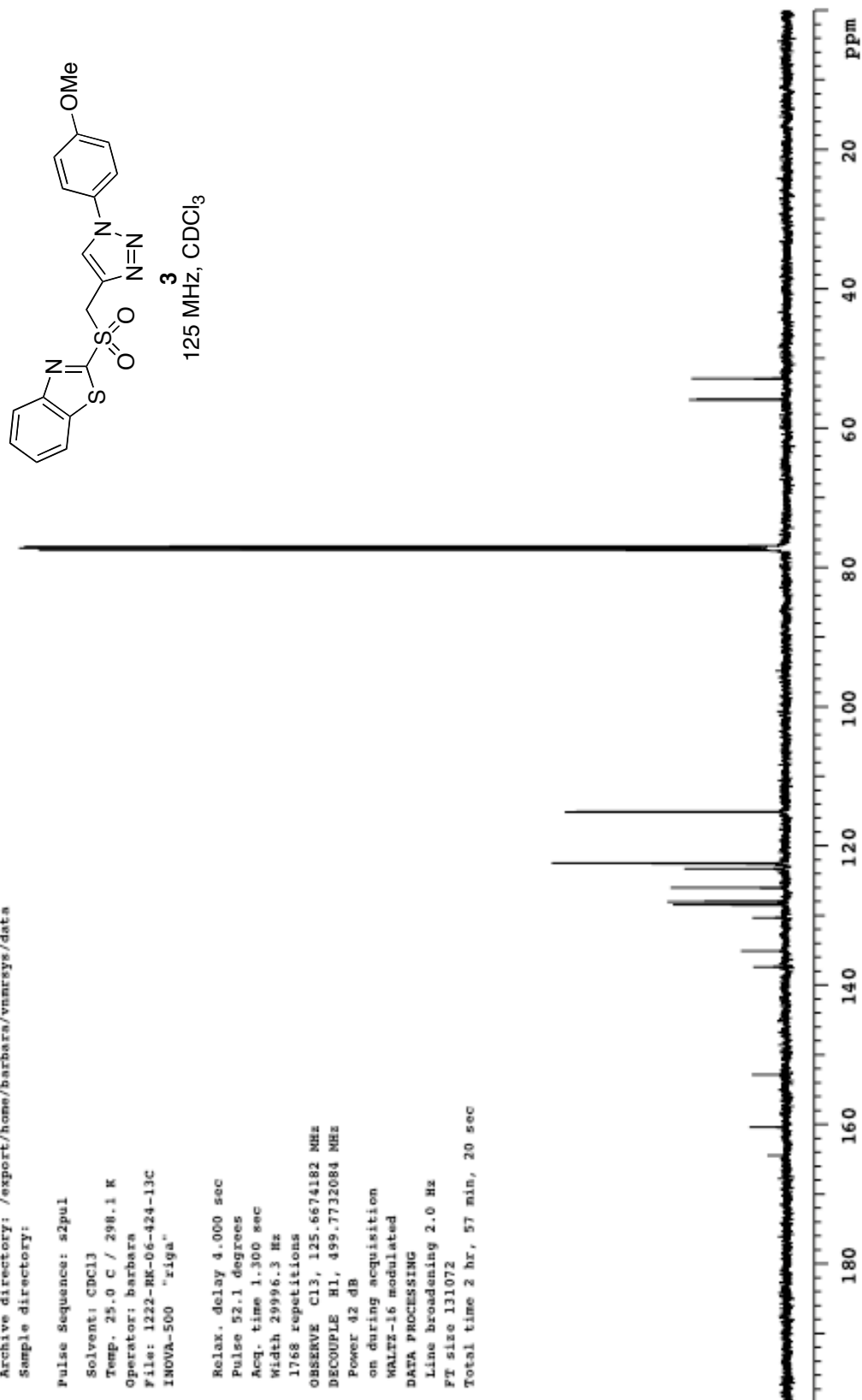
WALTZ-16 modulated

DATA PROCESSING

Line broadening 2.0 Hz

FT size 131072

Total time 2 hr, 57 min, 20 sec

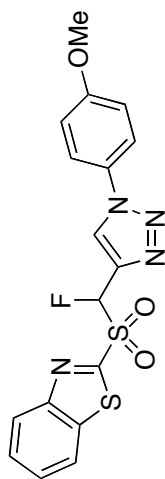


1222-RK-04-246-pure-1H

Archive directory: /export/home/mkl/vnmrsys/data
Sample directory: auto_13Dec2004

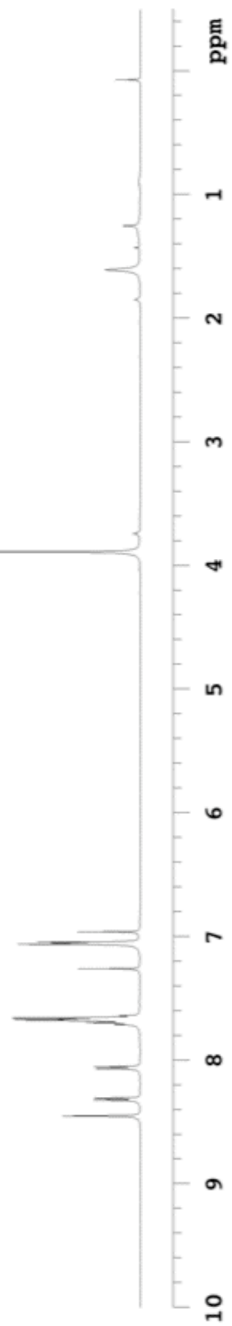
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: barbara
File: 1222-RK-04-246-pure-1H
INOVA-500 "riga"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7544.3 Hz
52 repetitions
OBSERVE H1, 499.7707216 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 32768
Total time 9 min, 40 sec



4

500 MHz, CDCl₃



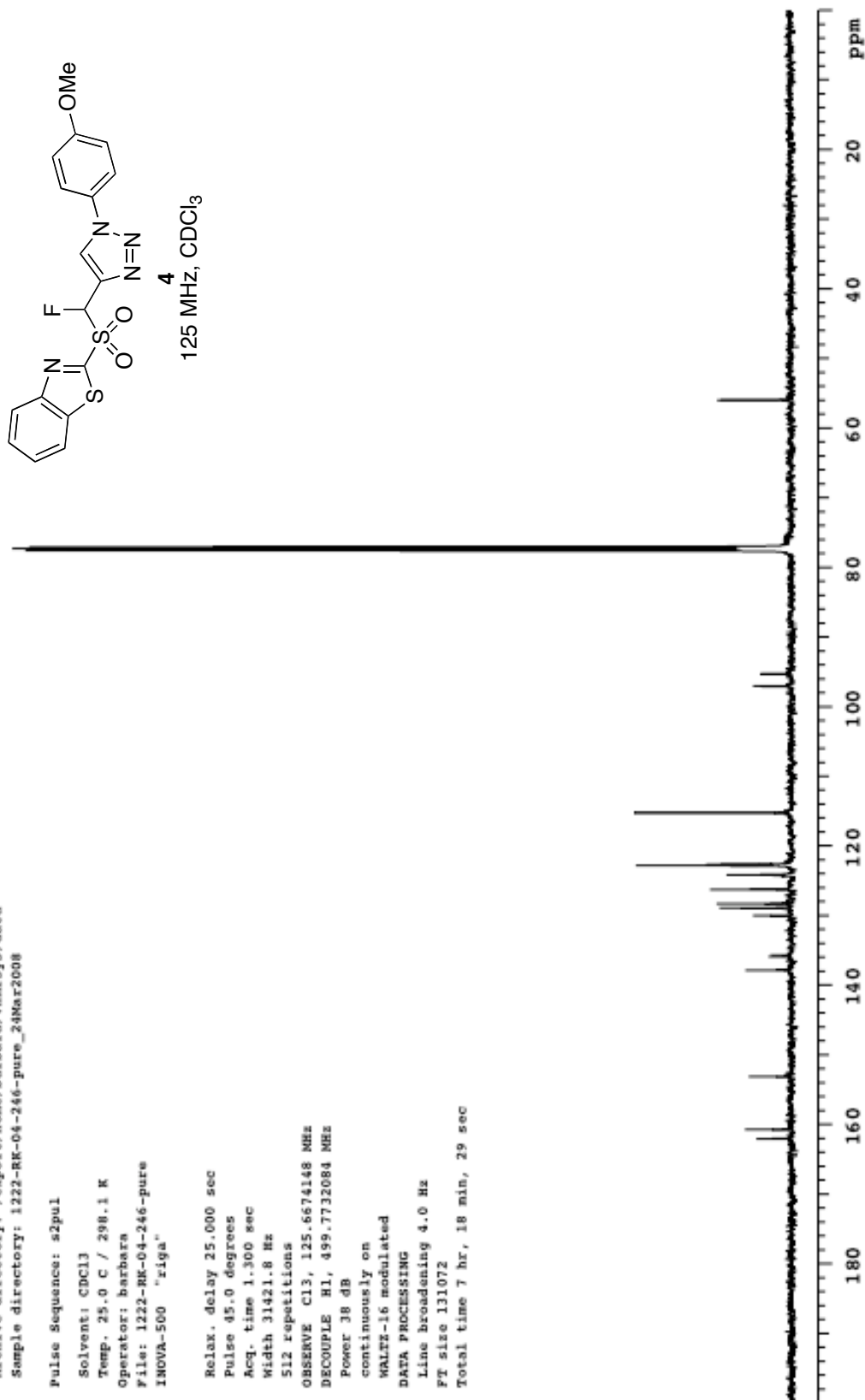
1222-RK-04-246-pure

Archive directory: /export/home/barbara/vnarsys/data
Sample directory: 1222-RK-04-246-pure_24Mar2008

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: barbara
File: 1222-RK-04-246-pure
INOVA-500 "ziga"

Relax. delay 25.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
512 repetitions
OBSERVE C13, 125.6674148 MHz
DECOUPLE H1, 499.7732084 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 131072
Total time 7 hr, 18 min, 29 sec

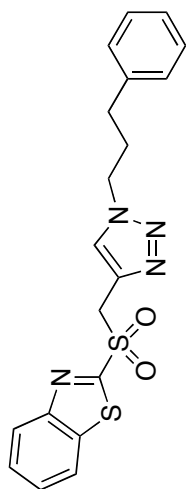


1222-RK-06-423-pure
Archive directory: /export/home/mkl/vmrssys/data
Sample directory: auto_13Dec2004

Pulse Sequence: s2pul

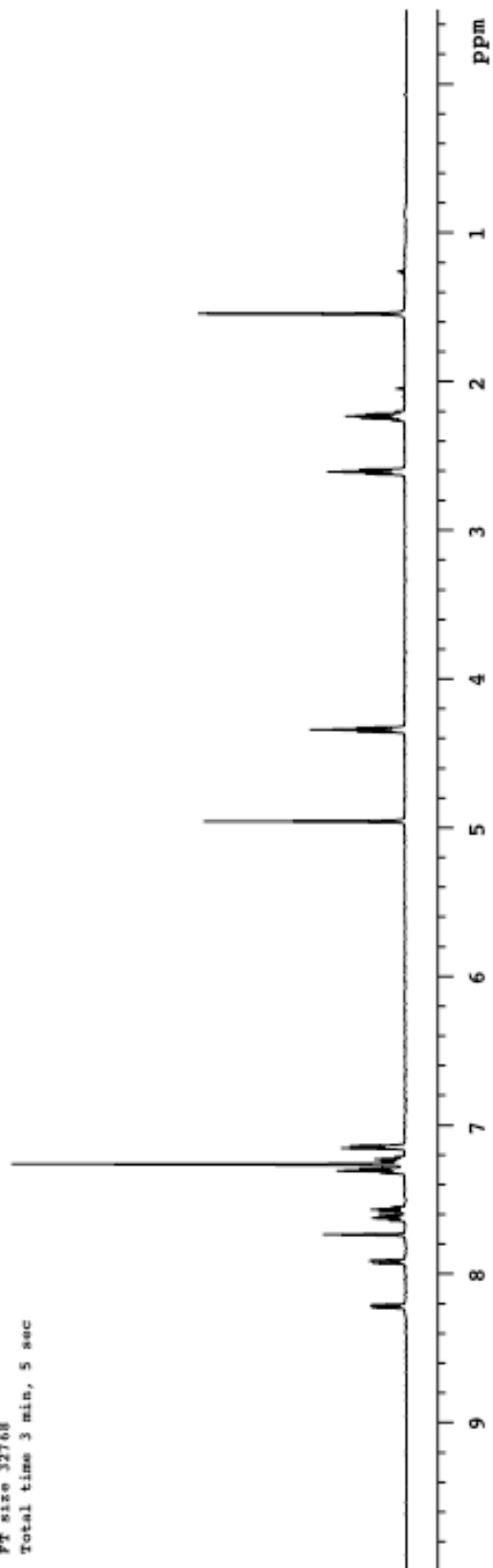
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: barbara
File: 1222-RK-06-423-pure
INOVA-500 "ziga"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7544.3 Hz
20 repetitions
OBSERVE H1, 499.7707184 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 3 min, 5 sec



5

500 MHz, CDCl₃



1222-RK-06-423-pure-13C

Archive directory: /export/home/barbara/vnarsys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl3

Temp. 25.0 C / 298.1 K

Operator: barbara

File: 1222-RK-06-423-pure-13C

INOVA-500 "ziga"

Relax. delay 4.000 sec

Pulse 52.1 degrees

Acq. time 1.300 sec

Width 29996.3 Hz

284 repetitions

OBSERVE C13, 125.6674264 MHz

DECOUPLE H1, 499.7732084 MHz

Power 42 dB

on during acquisition

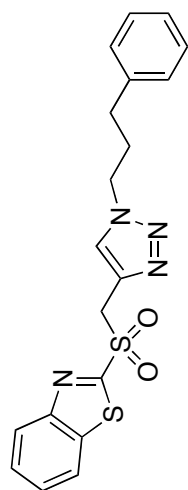
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.1 Hz

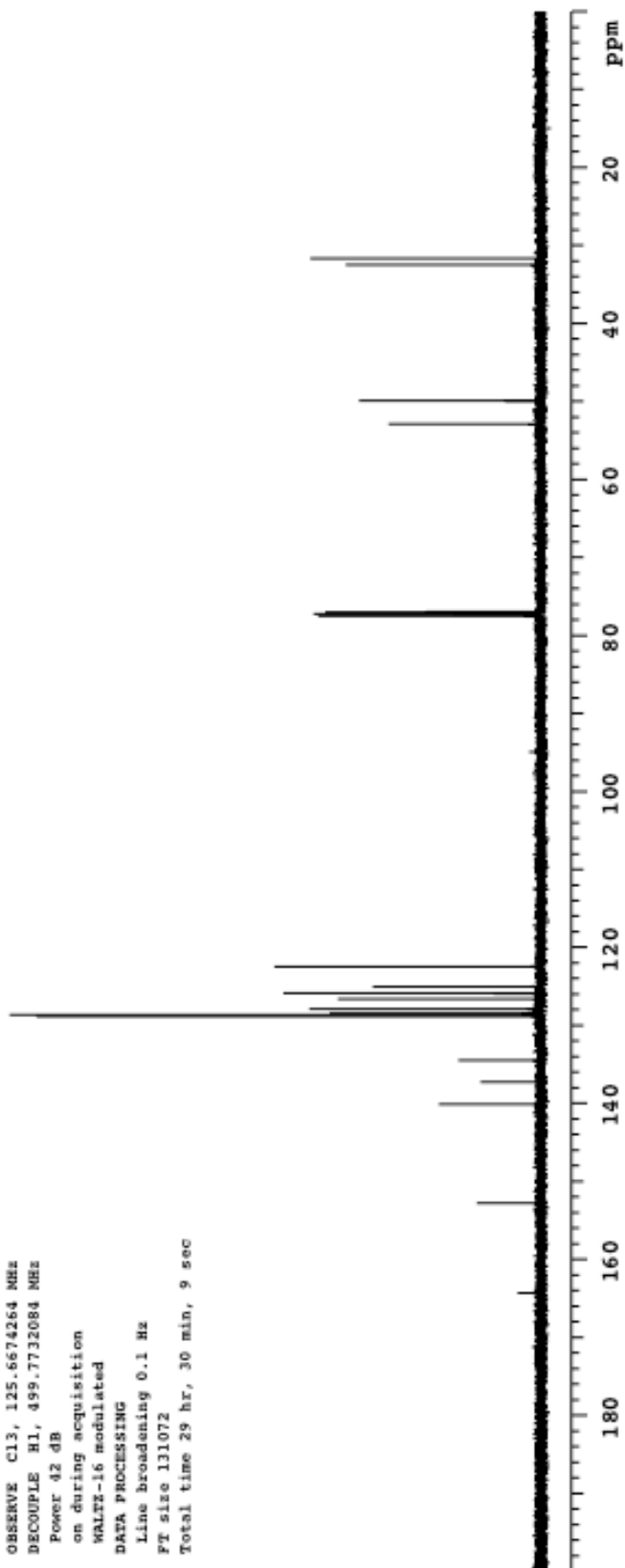
FT size 131072

Total time 29 hr, 30 min, 9 sec



5

125 MHz, CDCl₃



1222-RK-04-258-pure

Archive directory: /export/home/mkl/vmrssys/data
Sample directory: auto_13Dec2004

Pulse Sequence: s2pul

Solvent: cdcl3

Temp. 25.0 C / 298.1 K

Operator: barbara

File: 1222-RK-04-258-pure

INOVA-500 "ziga"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.892 sec

Width 7544.3 Hz

32 repetitions

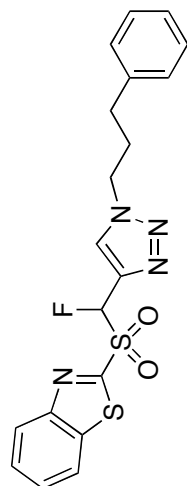
OBSERVE H1, 499.7707212 MHz

DATA PROCESSING

Line broadening 0.1 Hz

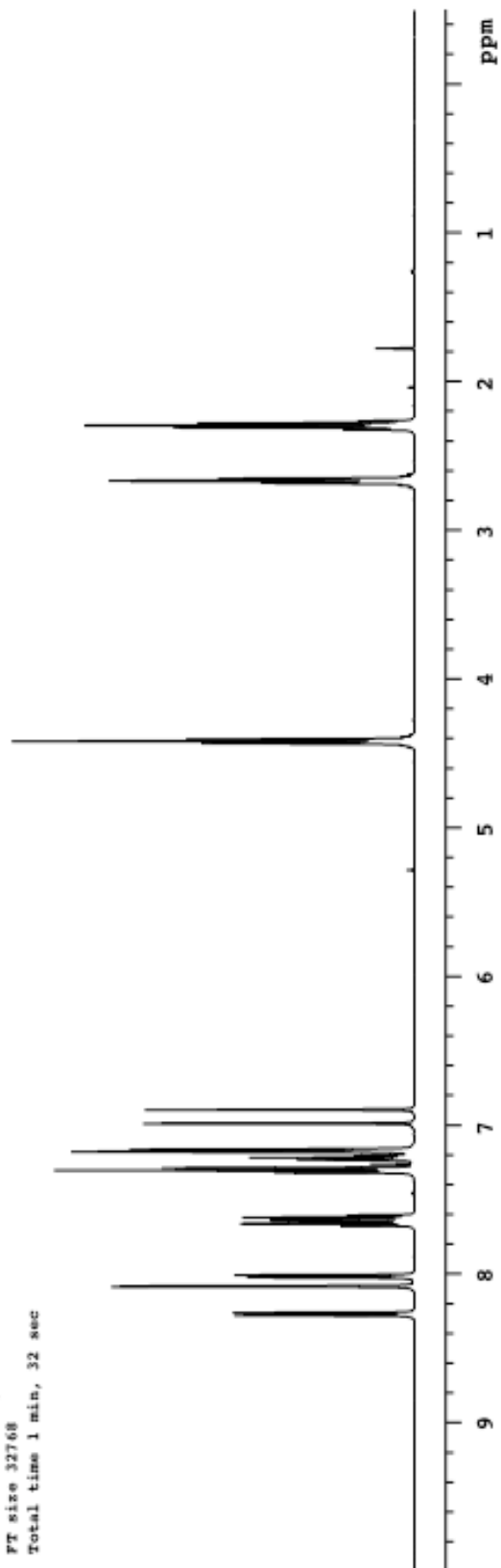
FT size 32768

Total time 1 min, 32 sec



6

500 MHz, CDCl₃



1222-RK-04-258-pure-13C

Pulse Sequence: s2pul

Solvent: CDCl3

Temp. 25.0 C / 298.1 K

Operator: Barbara

File: 1222-RK-04-258-pure-13C

INOVA-500 "riga"

Relax. delay 4.000 sec

Pulse 52.1 degrees

Acq. time 1.300 sec

Width 29996.3 Hz

52 repetitions

OBSERVE C13, 125.6674273 MHz

DECOUPLE H1, 499.7732084 MHz

Power 42 dB

on during acquisition

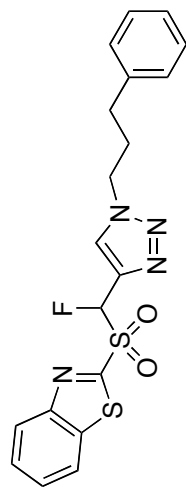
WALTZ-16 modulated

DATA PROCESSING

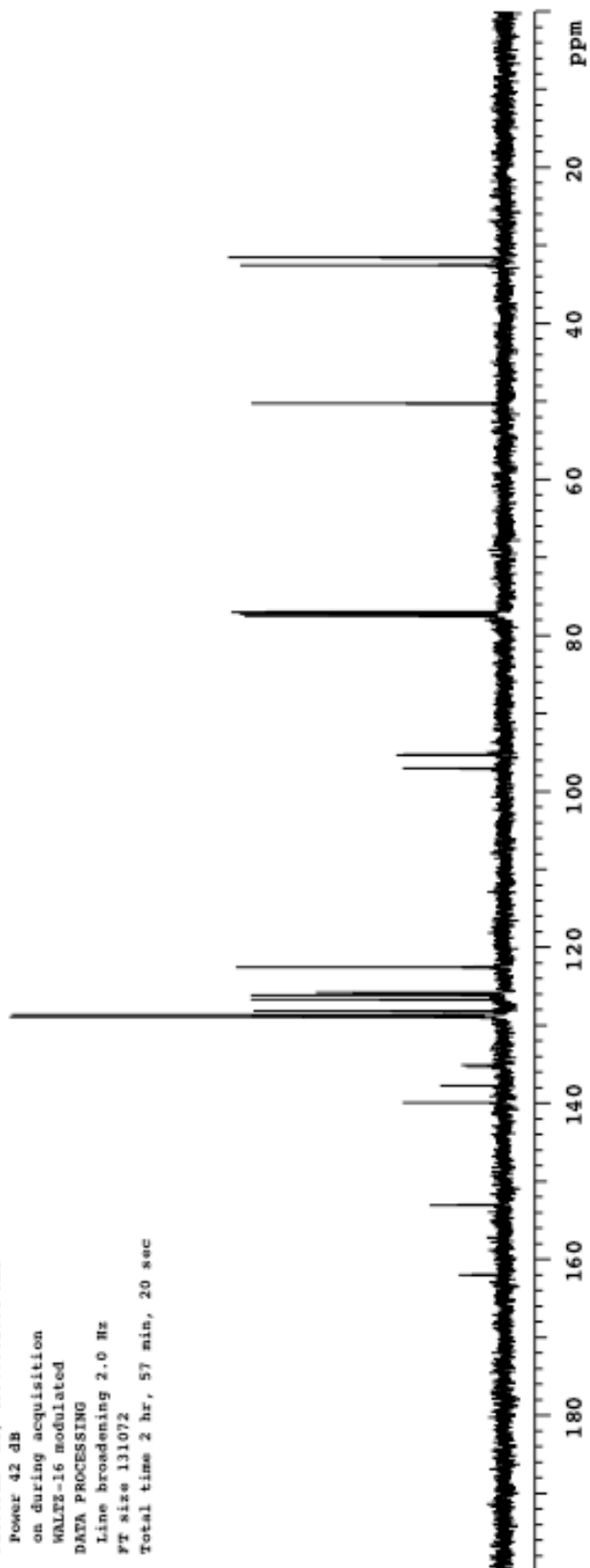
Line broadening 2.0 Hz

FT size 131072

Total time 2 hr, 57 min, 20 sec



6
125 MHz, CDCl₃



1222-RK-09-679

Archive directory: /export/home/mkl/vmrssys/data
Sample directory: auto_13Dec2004

Pulse Sequence: s2pul

Solvent: cdcl3

Temp. 25.0 C / 298.1 K

Operator: barbara

File: 1222-RK-09-679

INOVA-500 "ziga"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.892 sec

Width 10000.0 Hz

129 repetitions

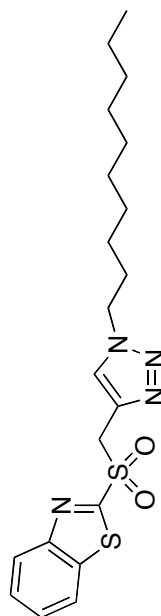
OBSERVE H1, 499.7707215 MHz

DATA PROCESSING

Line broadening 1.0 Hz

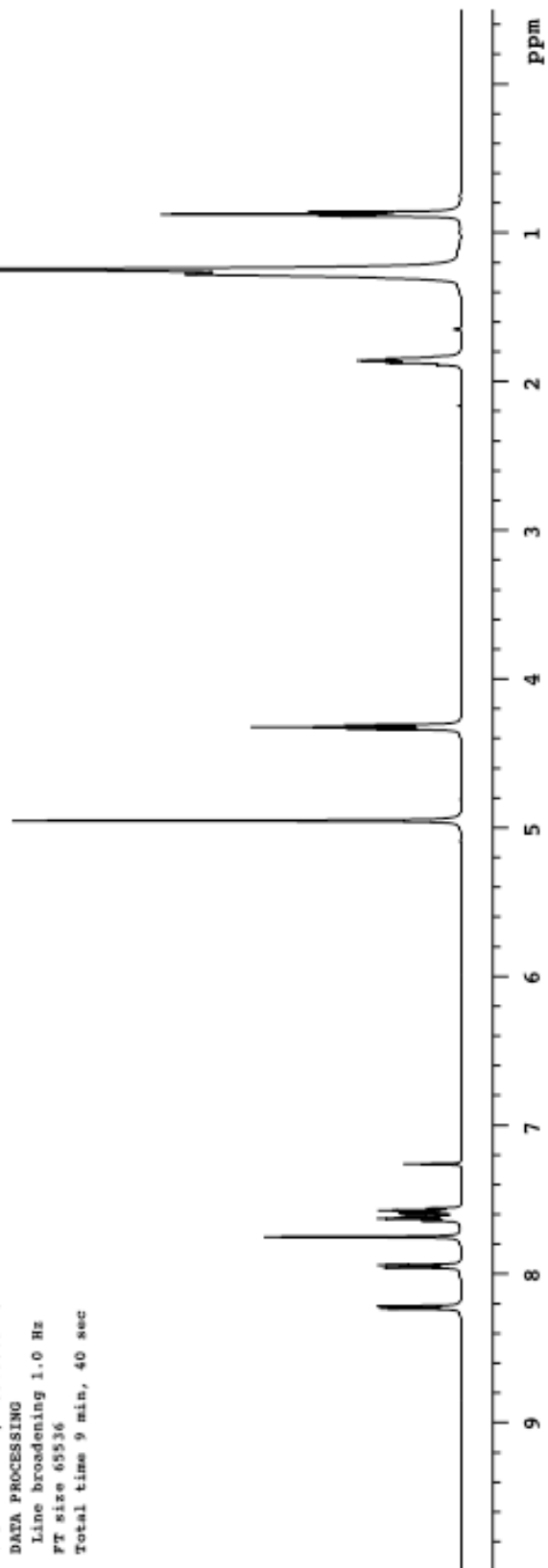
FT size 65536

Total time 9 min, 40 sec



7

500 MHz, CDCl₃



1222-RK-09-679-13C

Pulse Sequence: s2pul

Solvent: CDCl3

Temp. 25.0 C / 298.1 K

Operator: Barbara

File: 1222-RK-09-679-13C

INOVA-500 "riga"

Relax. delay 4.000 sec

Pulse 52.1 degrees

Acq. time 1.300 sec

Width 29996.3 Hz

566 repetitions

OBSERVE C13, 125.6674214 MHz

DECOUPLE H1, 499.7732084 MHz

Power 42 dB

on during acquisition

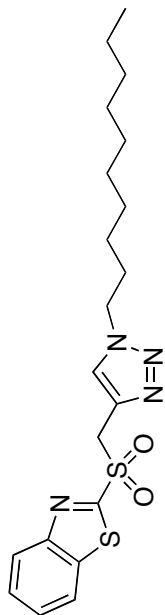
WALTZ-16 modulated

DATA PROCESSING

Line broadening 3.0 Hz

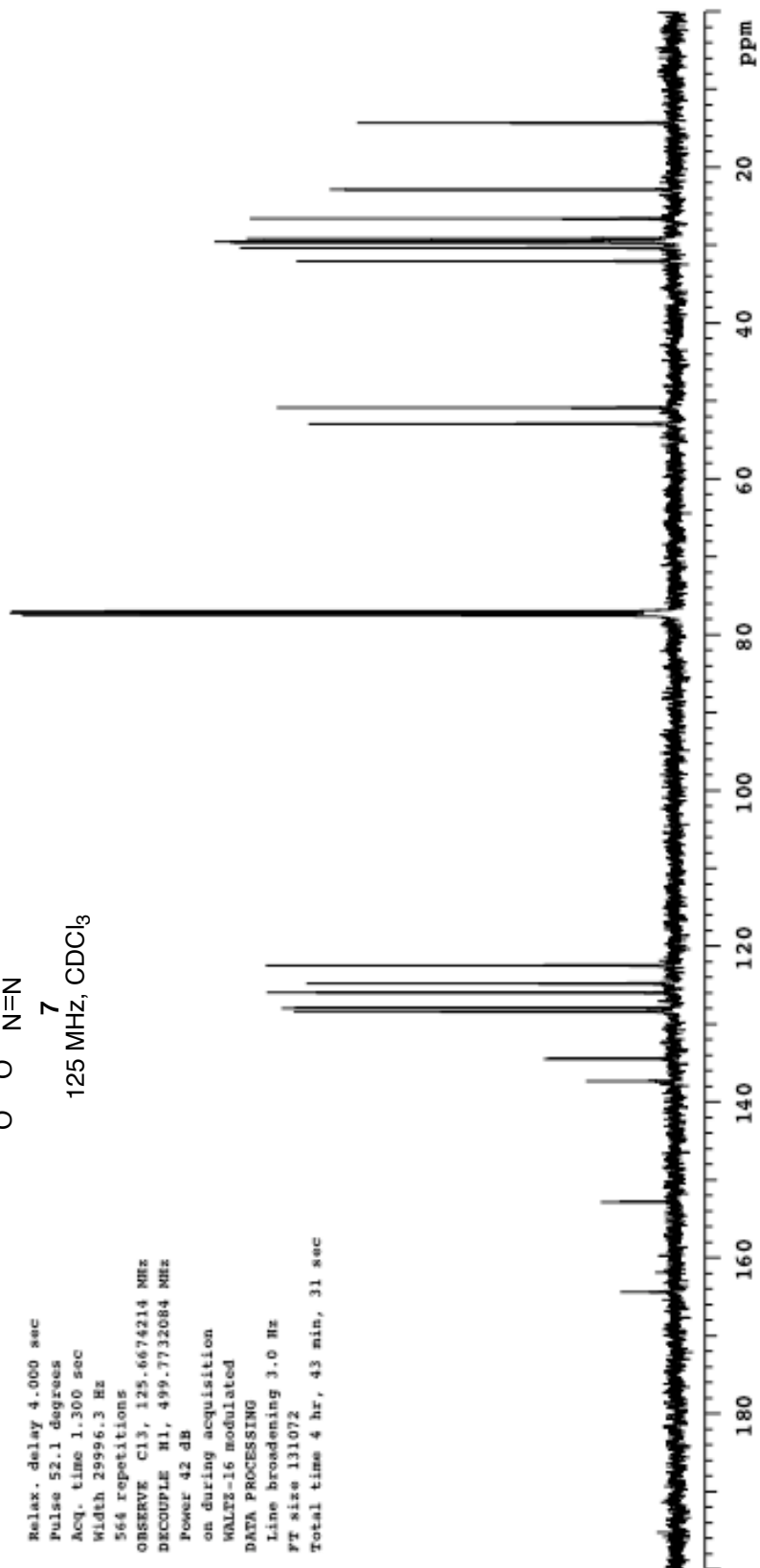
FT size 131072

Total time 4 hr, 43 min, 31 sec



7

125 MHz, CDCl₃



1222-RK-04-270-pure

Archive directory: /export/home/mkl/vmrssys/data
Sample directory: auto_13Dec2004

Pulse Sequence: s2pul

Solvent: cdcl3

Temp. 25.0 C / 298.1 K

Operator: barbara

File: 1222-RK-04-270-pure

INNOVA-500 "ziga"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.892 sec

Width 7544.3 Hz

16 repetitions

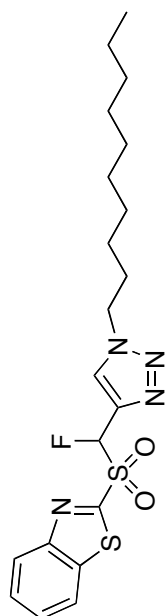
OBSERVE H1, 499.7707212 MHz

DATA PROCESSING

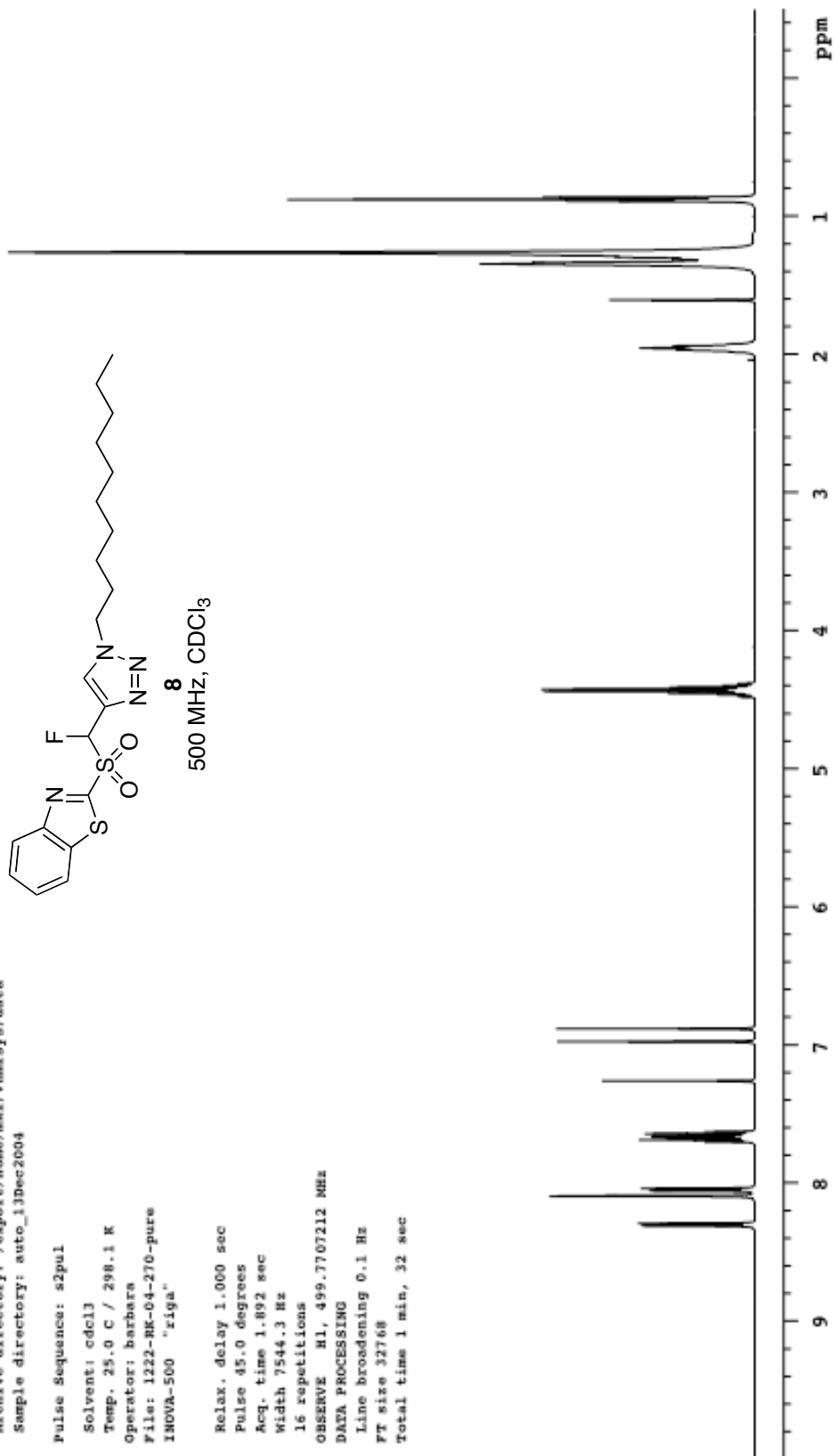
Line broadening 0.1 Hz

FT size 32768

Total time 1 min, 32 sec



8
500 MHz, CDCl₃



1222-RK-04-270-13C

Pulse Sequence: s2pul

Solvent: CDCl3

Temp. 25.0 C / 298.1 K

Operator: Barbara

File: 1222-RK-04-270-13C

INOVA-500 "riga"

Relax. delay 4.000 sec

Pulse 52.1 degrees

Acq. time 1.300 sec

Width 29996.3 Hz

64 repetitions

OBSERVE C13, 125.6674571 MHz

DECOUPLE H1, 499.7732084 MHz

Power 42 dB

on during acquisition

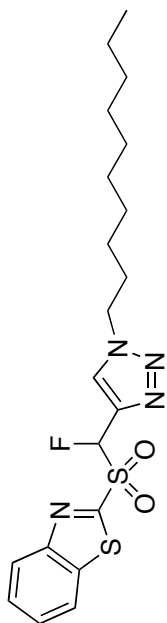
WALTZ-16 modulated

DATA PROCESSING

Line broadening 3.0 Hz

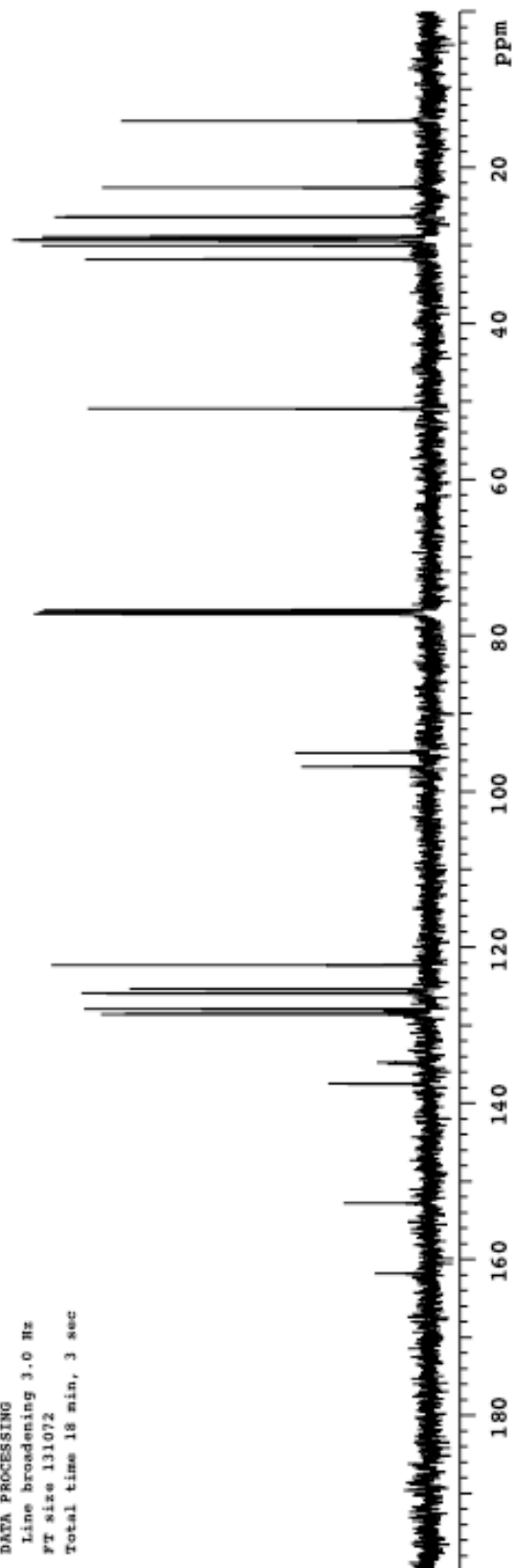
FT size 131072

Total time 18 min, 3 sec



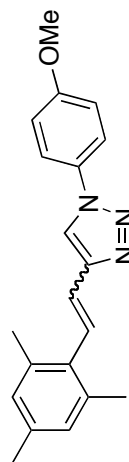
8

125 MHz, CDCl₃

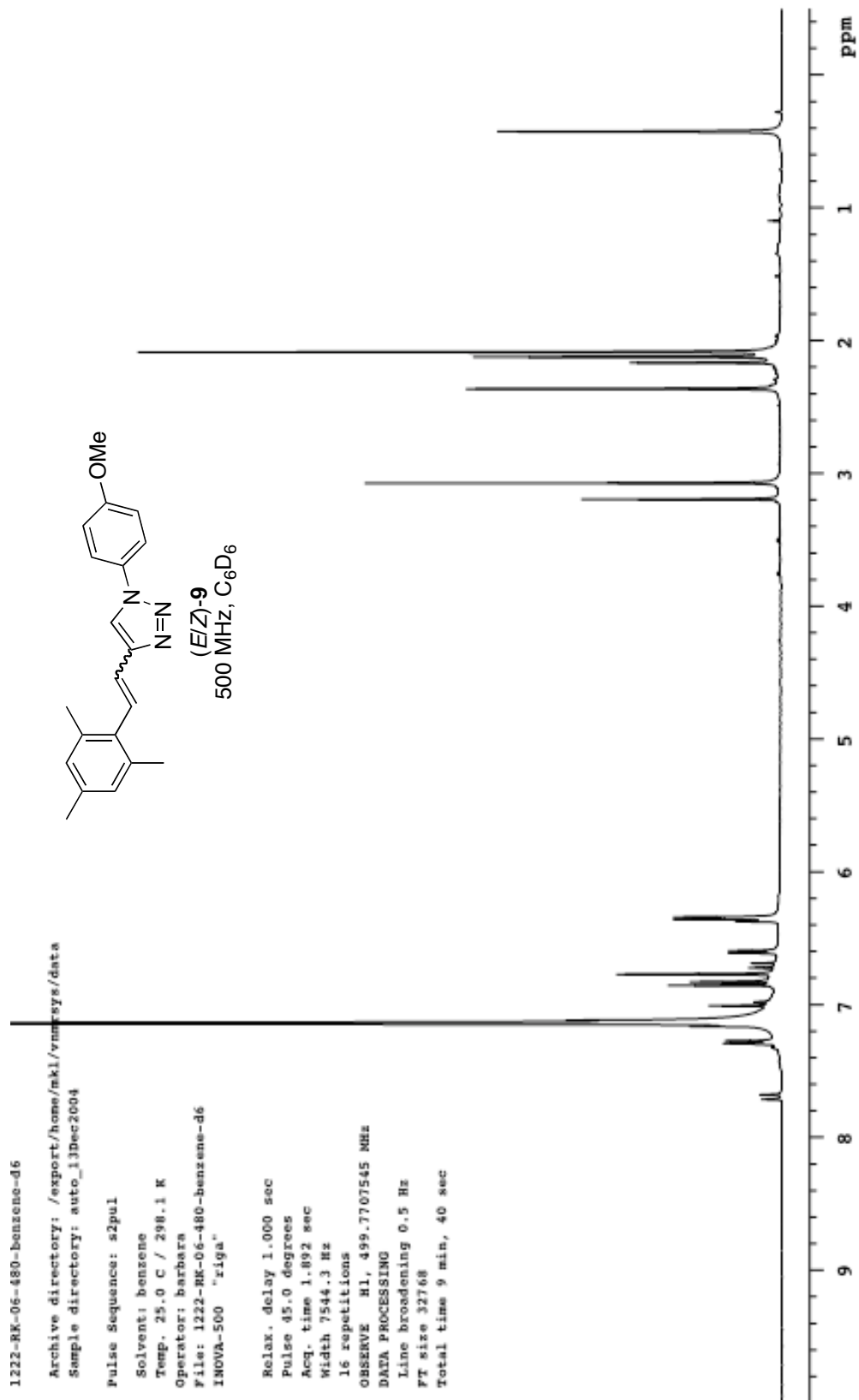


1222-RK-06-480-benzene-d6
Archive directory: /export/home/mkl/vnmrsys/data
Sample directory: auto_13Dec2004
Pulse Sequence: s2pul
Solvent: benzene
Temp. 25.0 C / 298.1 K
Operator: barbara
File: 1222-RK-06-480-benzene-d6
INOVA-500 "ziga"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7544.3 Hz
16 repetitions
OBSERVE H1, 499.7707545 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 32768
Total time 9 min, 40 sec



(E/Z)-9
500 MHz, C₆D₆



1222-MK-06-463-pure-acetone-D6

Archive directory: /export/home/mkl/vmrsvs/data

Sample directory: auto_13Dec2004

Pulse Sequence: s2pul

Solvent: acetone

Temp. 25.0 C / 298.1 K

Operator: barbara

File: 1222-MK-06-463-pure-acetone-D6

INOVA-500 "riga"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.892 sec

Width 7544.3 Hz

32 repetitions

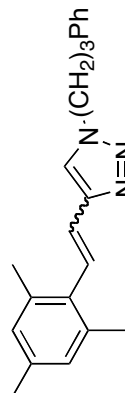
OBSERVE H1, 499.7733168 MHz

DATA PROCESSING

Line broadening 0.1 Hz

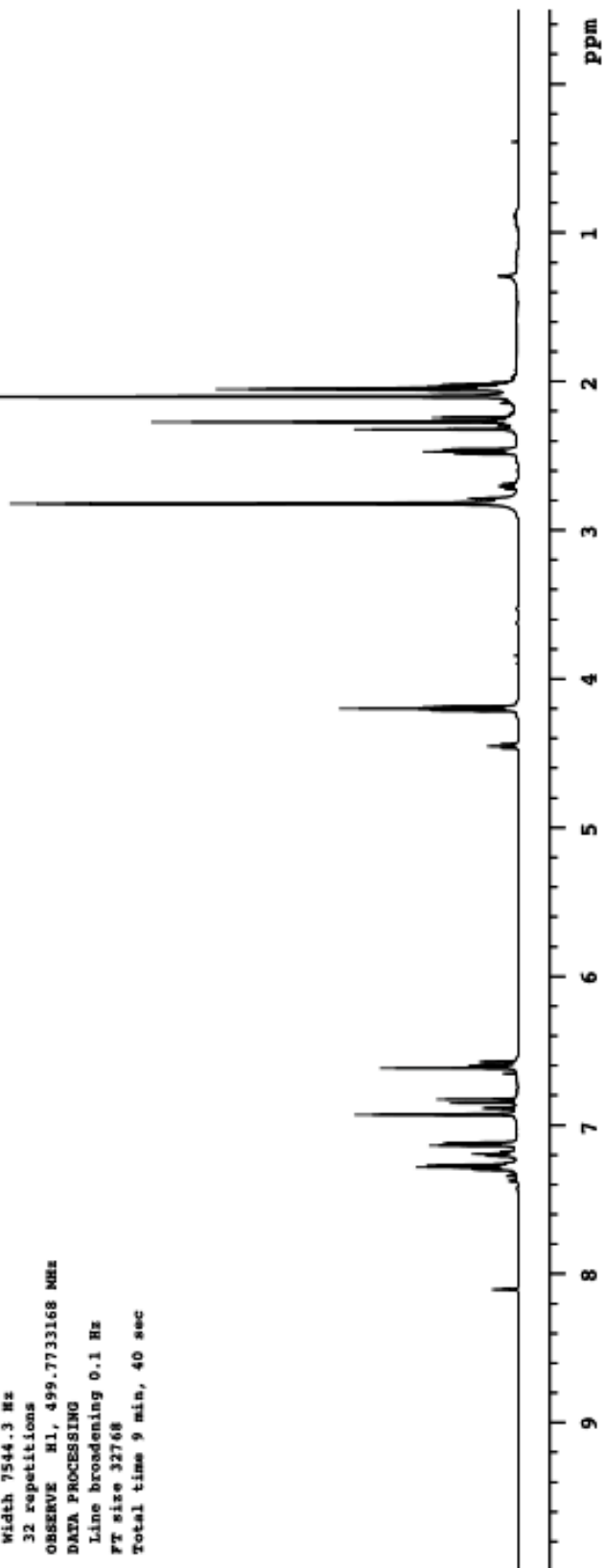
FT size 32768

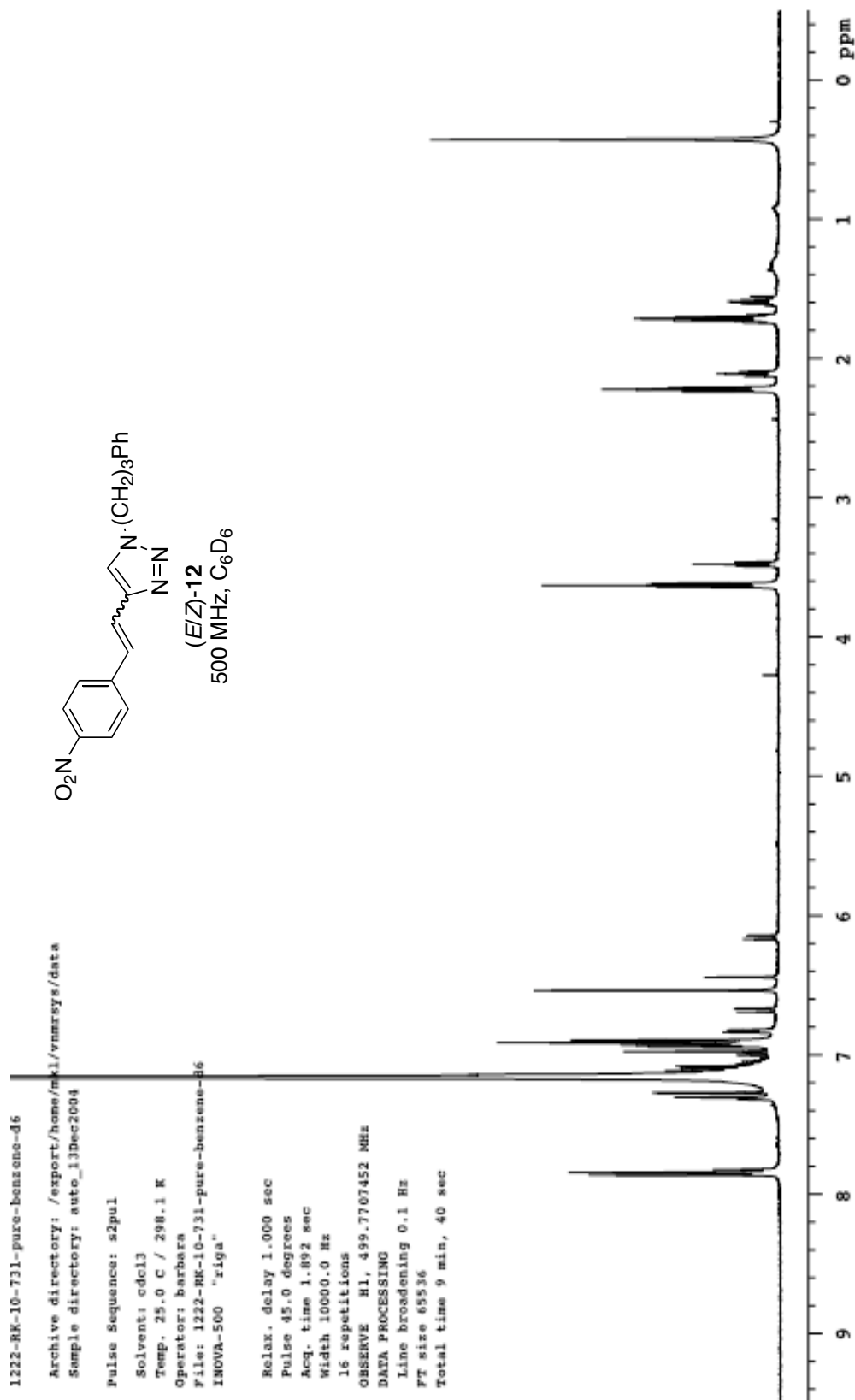
Total time 9 min, 40 sec

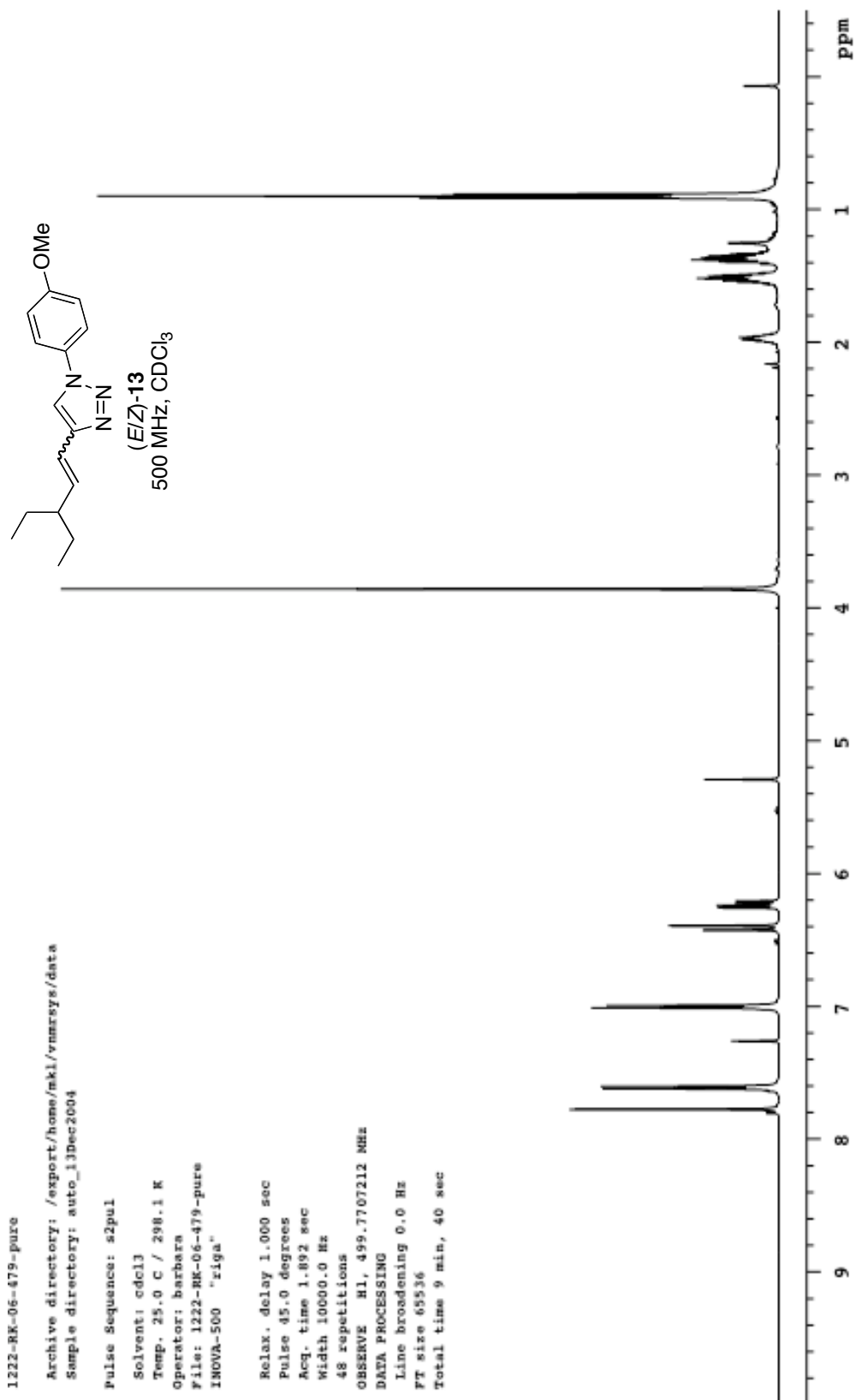


(E/Z)-10

500 MHz, acetone-d₆







1222-RK-06-433-1stfraction

Archive directory: /export/home/mkl/vmrssys/data

Sample directory: auto_13Dec2004

Pulse Sequence: s2pul

Solvent: cdcl3

Temp. 25.0 C / 298.1 K

Operator: barbara

File: 1222-RK-06-433-1stfraction

INOVA-500 "ziga"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.892 sec

Width 7544.3 Hz

16 repetitions

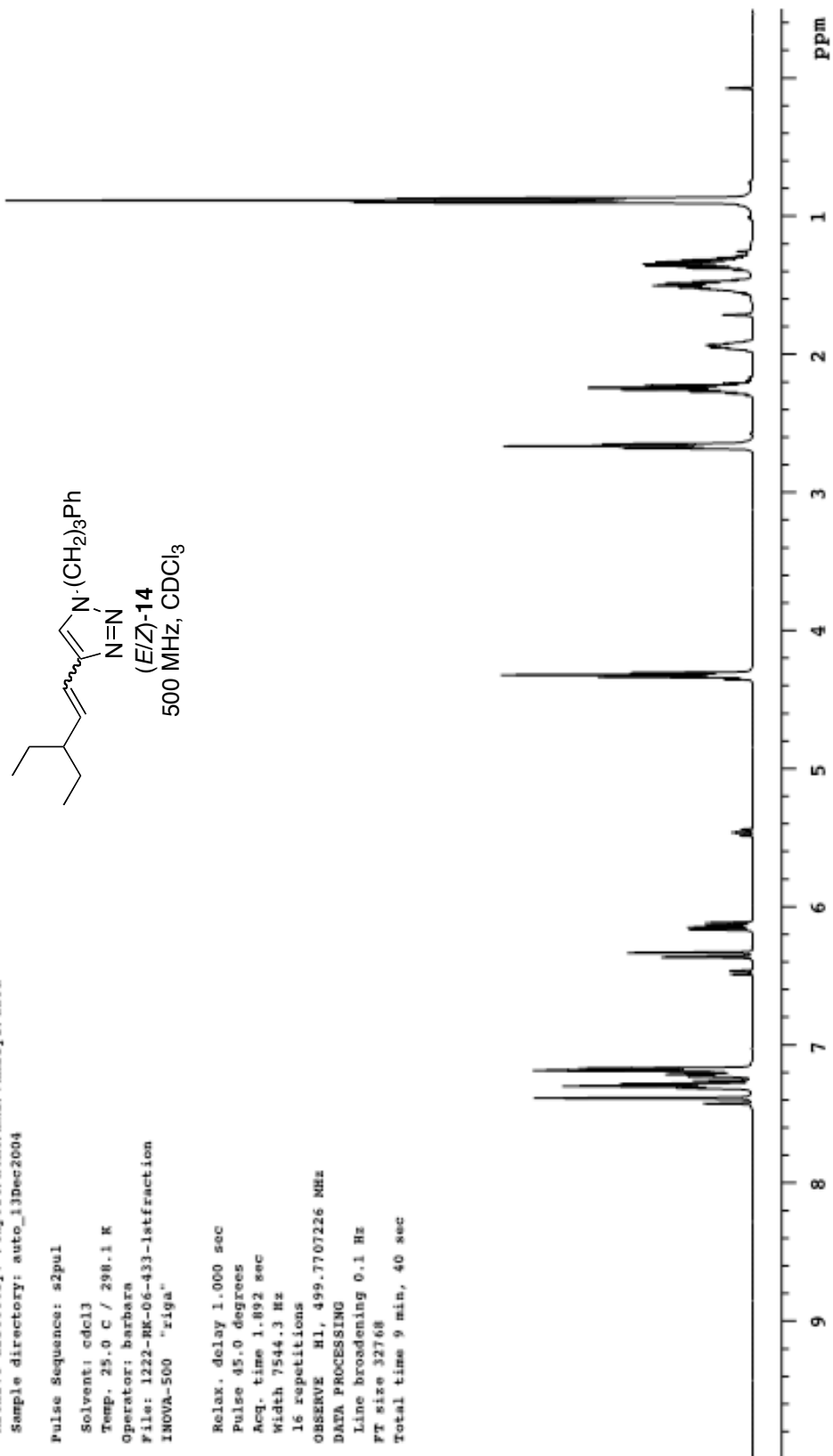
OBSERVE H1, 499.7707226 MHz

DATA PROCESSING

Line broadening 0.1 Hz

FT size 32768

Total time 9 min, 40 sec



1222-Rk-06-481

Archive directory: /export/home/mkl/vmrssys/data
Sample directory: auto_13Dec2004

Pulse Sequence: s2pul

Solvent: cdcl3

Temp. 10.0 C / 283.1 K

Operator: barbara

File: 1222-Rk-06-481

INOVA-500 "ziga"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.892 sec

Width 10000.0 Hz

12 repetitions

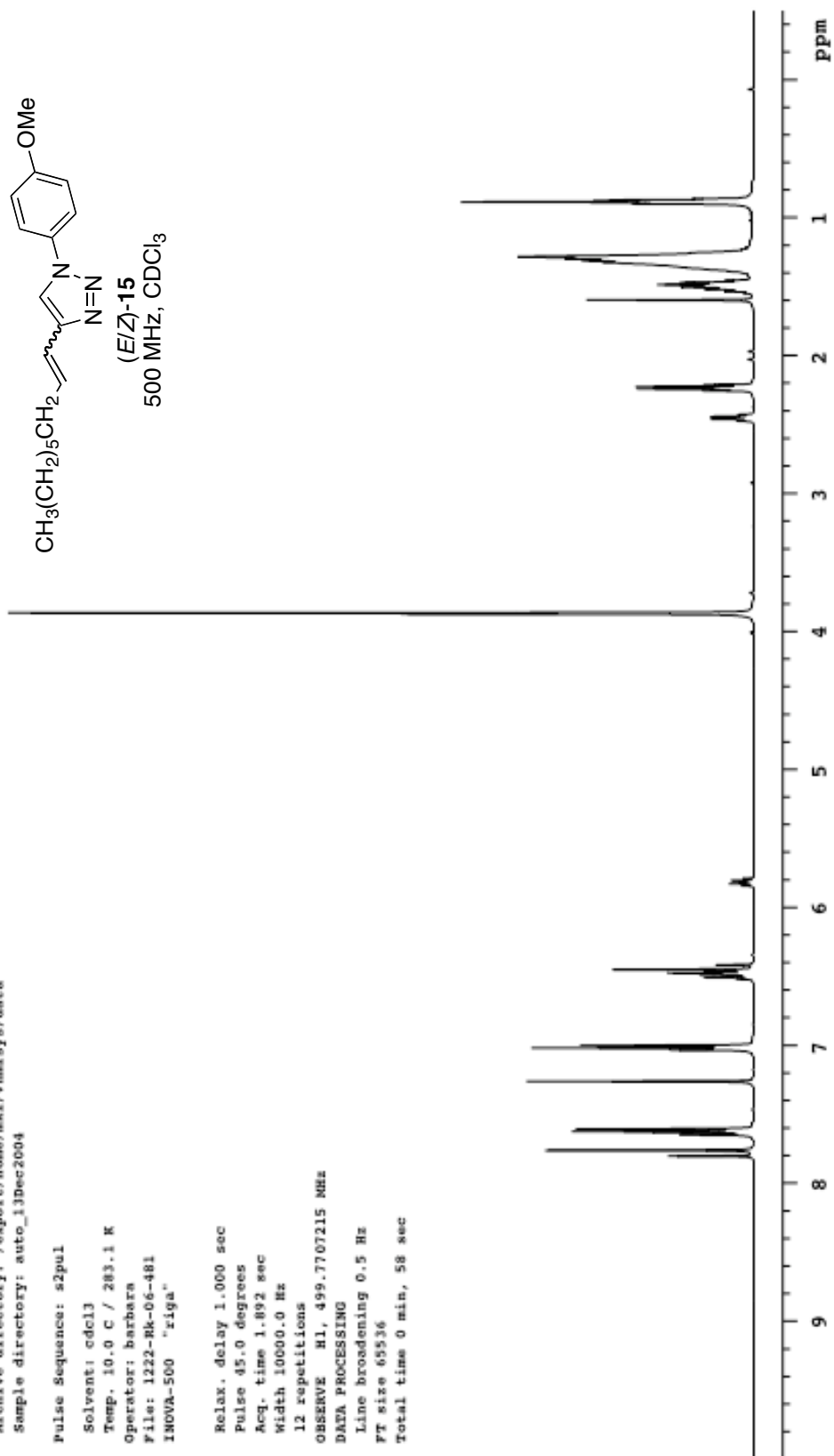
OBSERVE H1, 499.7707215 MHz

DATA PROCESSING

Line broadening 0.5 Hz

FT size 65536

Total time 0 min, 58 sec



1222-RK-10-734-pure

Archive directory: /export/home/mkl/vmrssys/data
Sample directory: auto_13Dec2004

Pulse Sequence: s2pul

Solvent: cdcl3

Temp. 25.0 C / 298.1 K

Operator: barbara

File: 1222-RK-10-734-pure

INOVA-500 "ziga"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.892 sec

Width 10000.0 Hz

20 repetitions

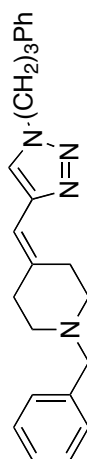
OBSERVE H1, 499.7707452 MHz

DATA PROCESSING

Line broadening 0.1 Hz

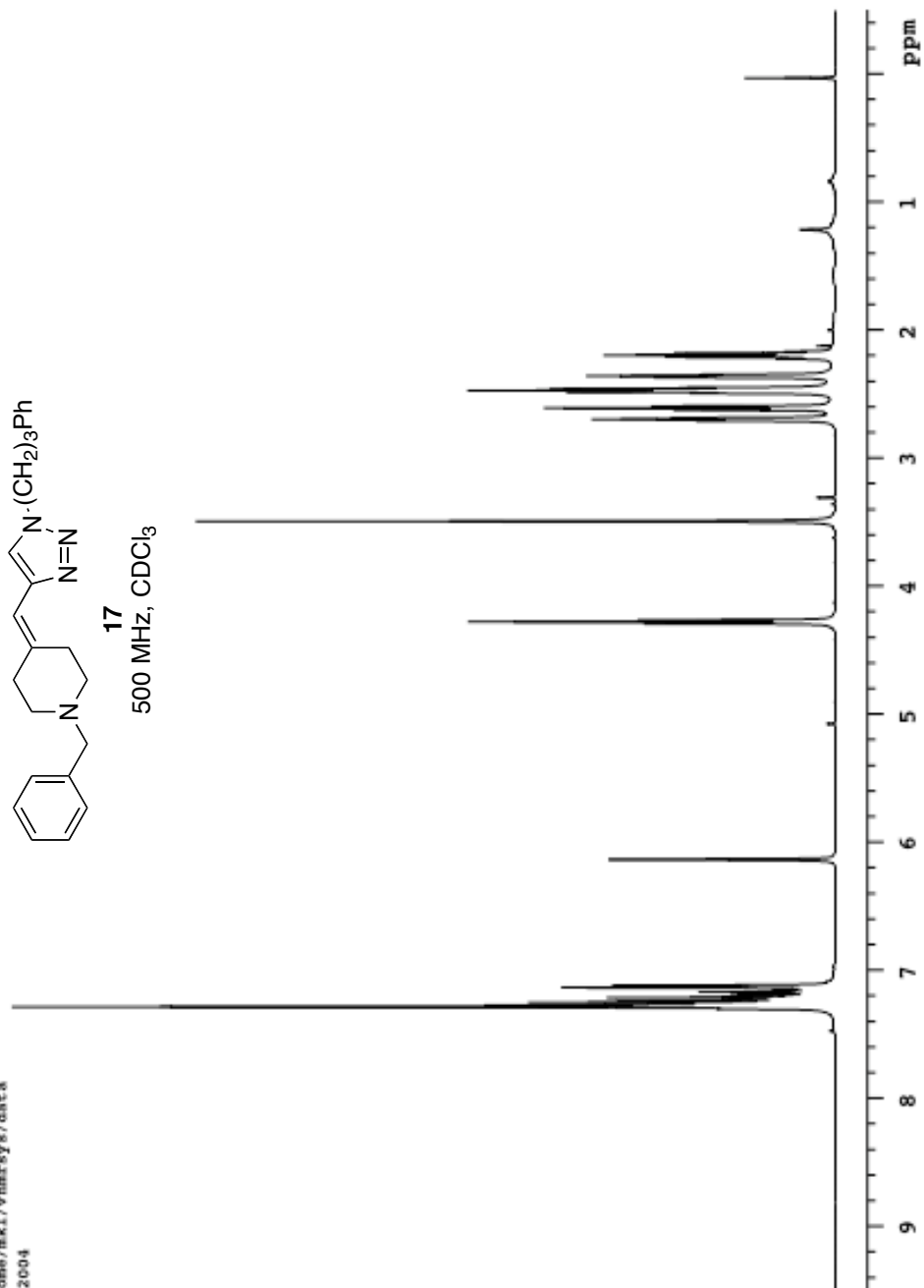
FT size 65536

Total time 9 min, 40 sec



17

500 MHz, CDCl₃



1222-RK-11-851-pure

Pulse Sequence: zgpg30

Solvent: CDCl3

Ambient temperature

Operator: Barbara

File: 1222-RK-11-851-pure

INOVA-500 "riga"

Pulse 48.0 degrees

Acq. time 1.892 sec

Width 8000.0 Hz

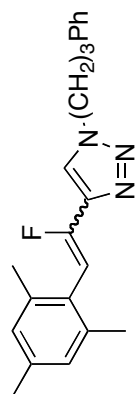
44 repetitions

OBSERVE H1, 499.7707217 MHz

DATA PROCESSING

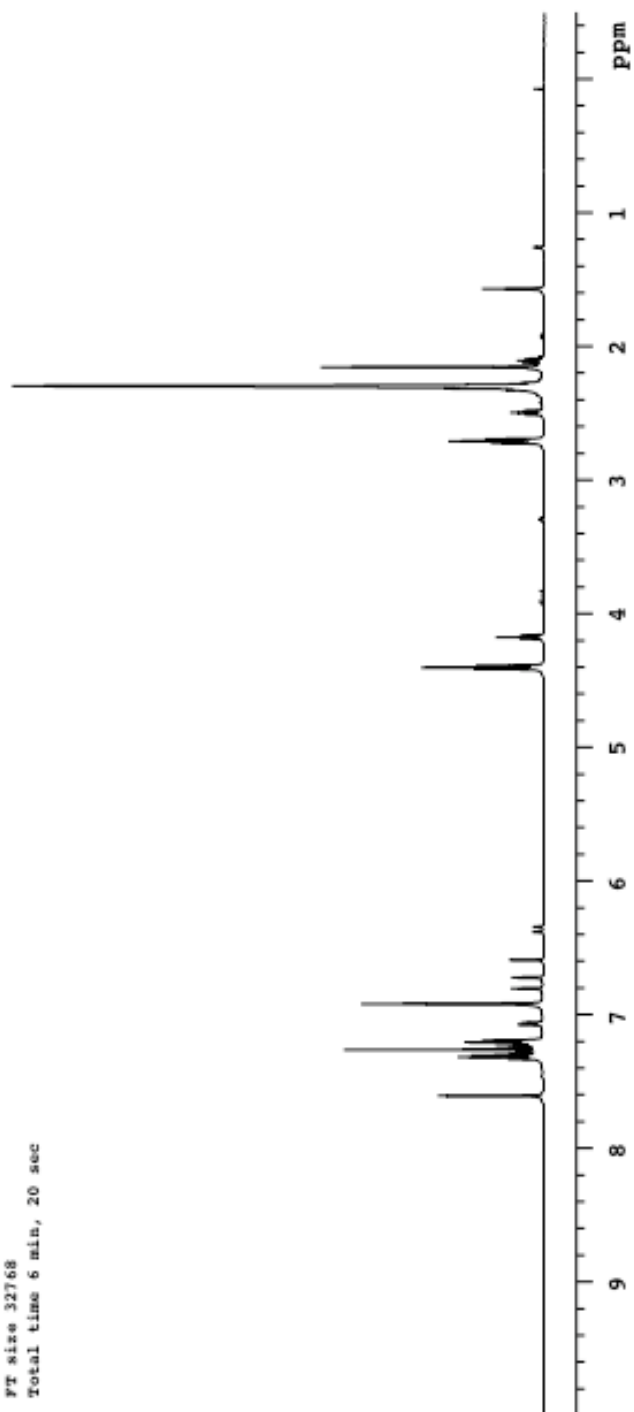
FT size 32768

Total time 6 min, 20 sec



(*E/Z*)-18

500 MHz, CDCl₃



1222-RK-04-306-2ndfraction

Archive directory: /export/home/barbara/vmarsys/data

Sample directory:

Pulse Sequence: s2pul

Solvent: cdcl3

Temp. 25.0 C / 298.1 K

Operator: barbara

File: 1222-RK-04-306-2ndfraction

INOVA-500 "ziga"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.892 sec

Width 7544.3 Hz

60 repetitions

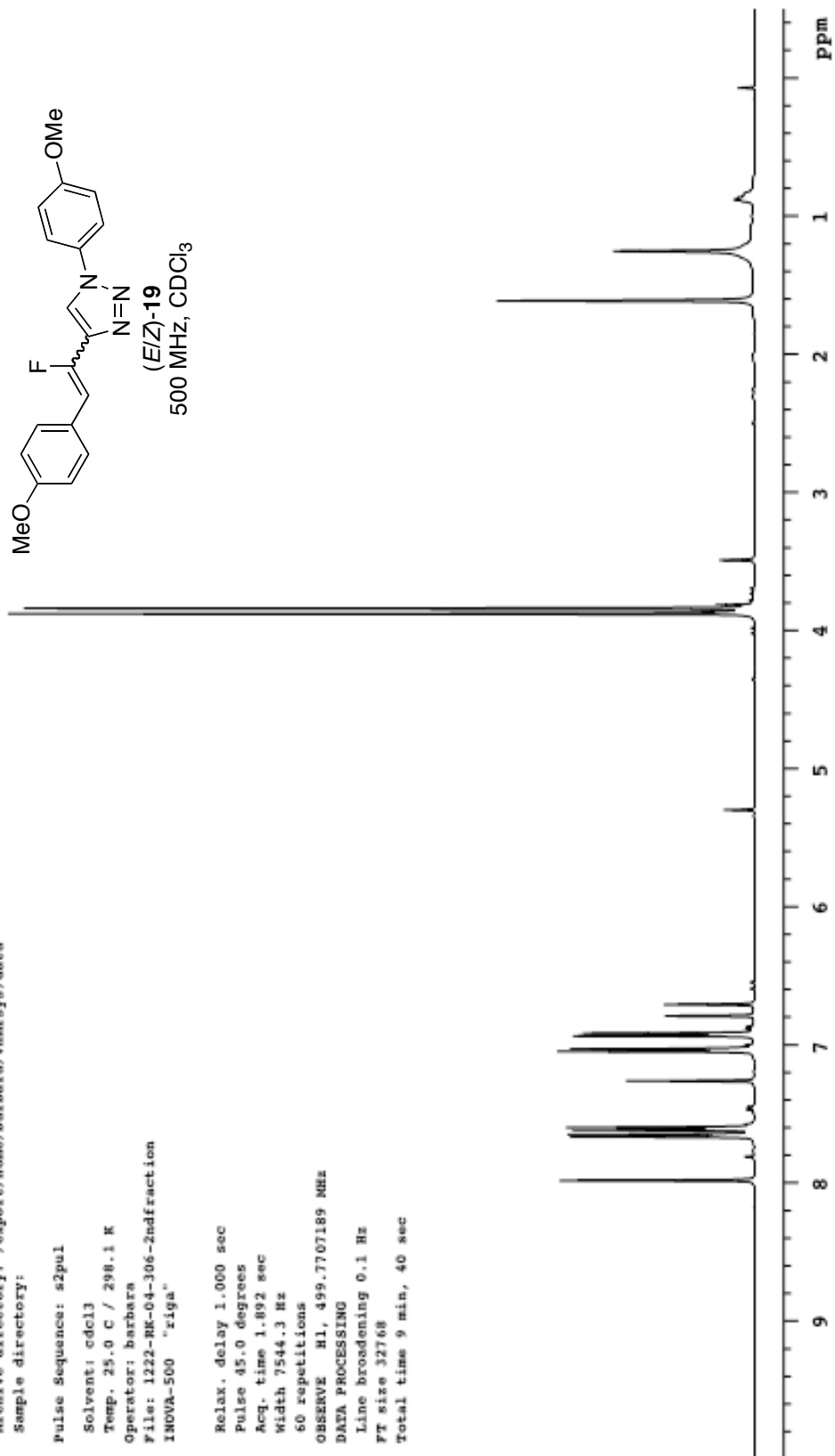
OBSERVE H1, 499.7707189 MHz

DATA PROCESSING

Line broadening 0.1 Hz

FT size 32768

Total time 9 min, 40 sec



1222-RK-04-308-pure

Archive directory: /export/home/mkl/vmrssys/data
Sample directory: auto_13Dec2004

Pulse Sequence: s2pul

Solvent: cdcl3

Temp. 25.0 C / 298.1 K

Operator: barbara

File: 1222-RK-04-308-pure

INOVA-500 "ziga"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.892 sec

Width 7544.3 Hz

48 repetitions

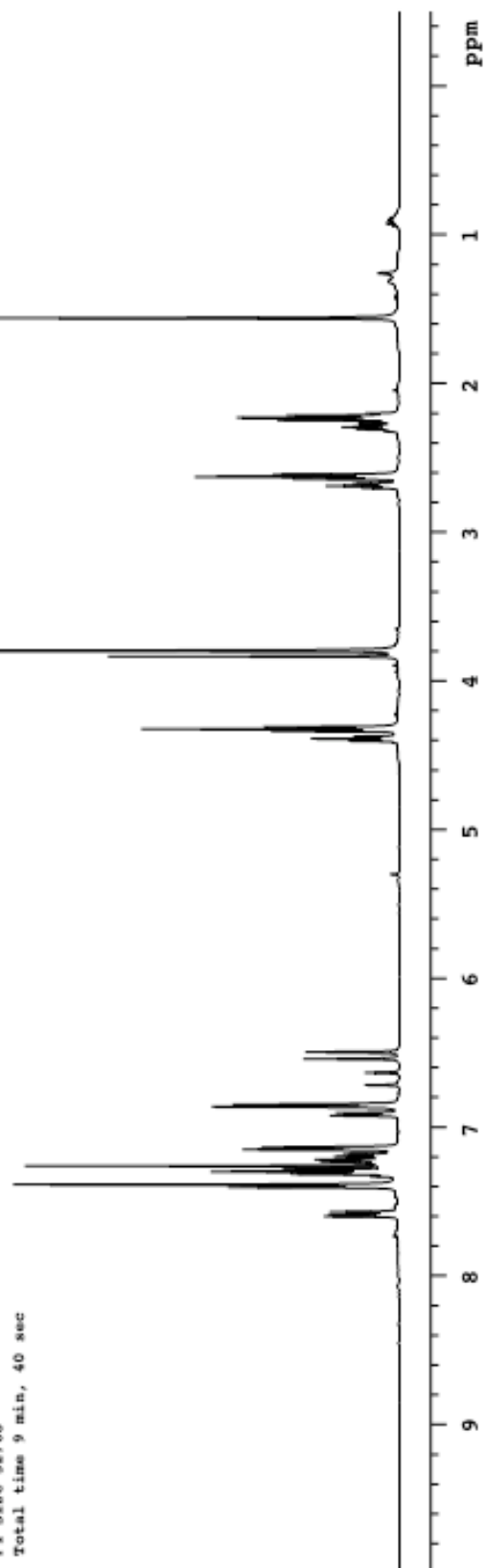
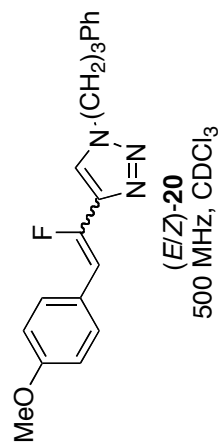
OBSERVE H1, 499.7707198 MHz

DATA PROCESSING

Line broadening 0.1 Hz

FT size 32768

Total time 9 min, 40 sec



1222-RK-04-314-pure

Archive directory: /export/home/barbara/vmarsys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: cdcl3

Temp. 25.0 C / 298.1 K

Operator: barbara

File: 1222-RK-04-314-pure

INOVA-500 "ziga"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.892 sec

Width 7544.3 Hz

64 repetitions

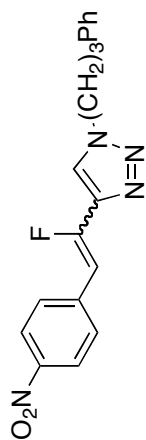
OBSERVE H1, 499.7707189 MHz

DATA PROCESSING

Line broadening 0.1 Hz

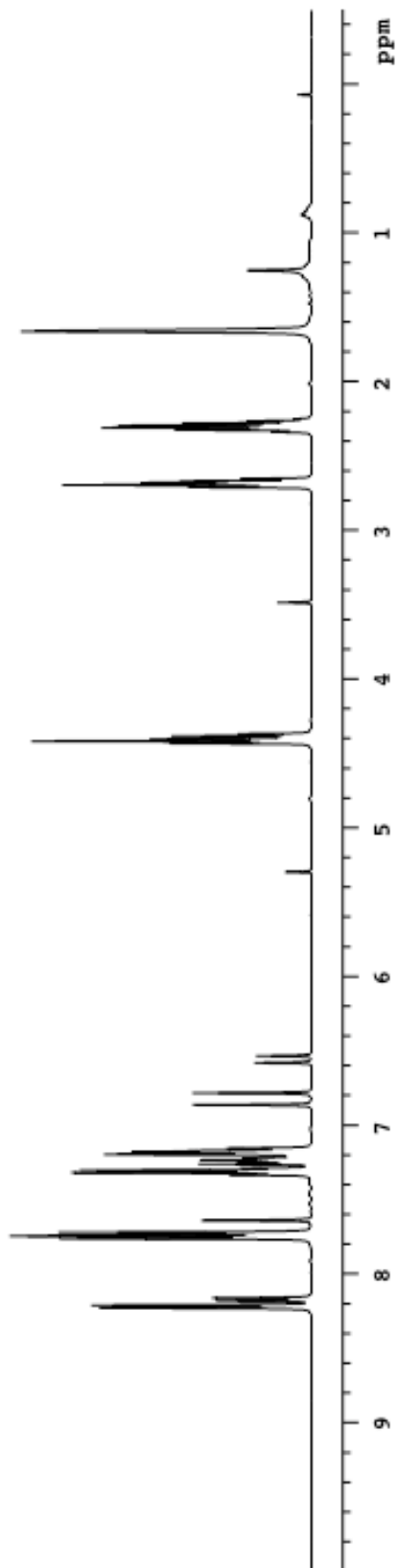
FT size 32768

Total time 9 min, 40 sec



(E/Z)-21

500 MHz, CDCl₃



1222-RK-05-352

Archive directory: /export/home/mkl/vmrssys/data
Sample directory: auto_13Dec2004

Pulse Sequence: s2pul

Solvent: cdcl3

Temp. 25.0 C / 298.1 K

Operator: barbara

File: 1222-RK-05-352

INOVA-500 "ziga"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.892 sec

Width 7544.3 Hz

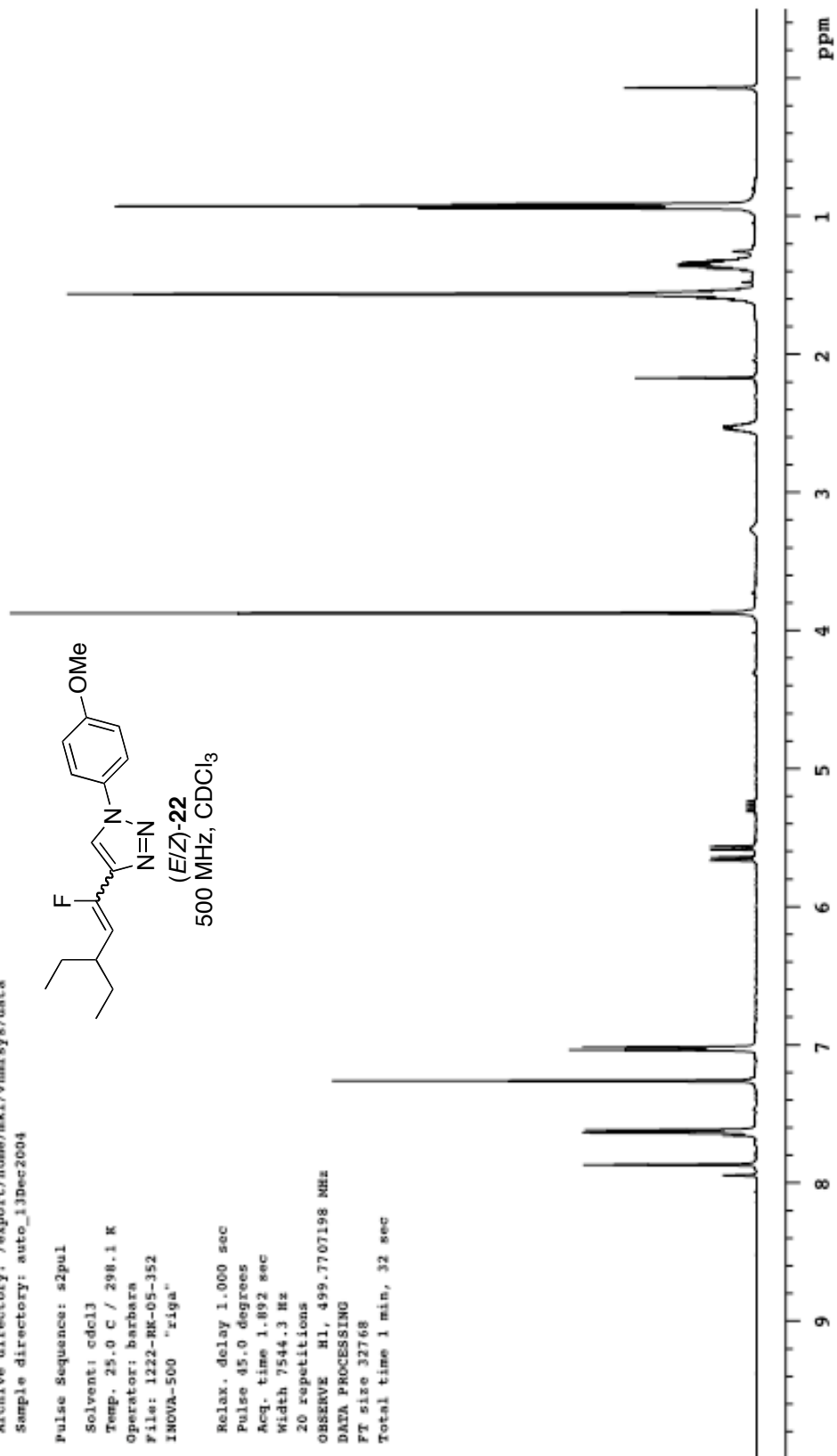
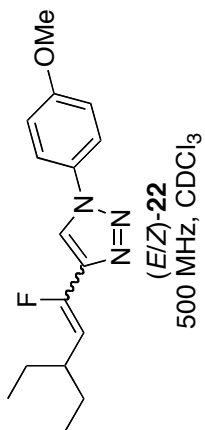
20 repetitions

OBSERVE H1, 499.7707198 MHz

DATA PROCESSING

FT size 32768

Total time 1 min, 32 sec

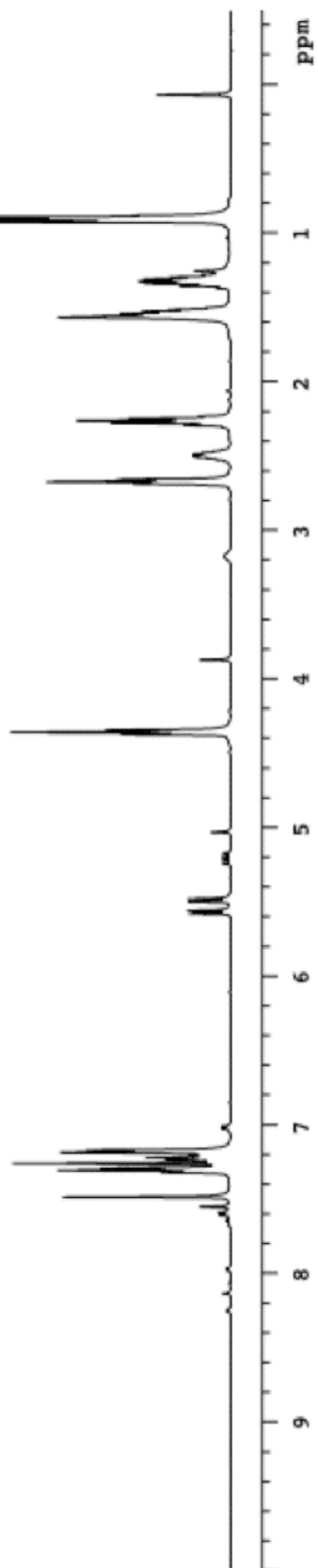
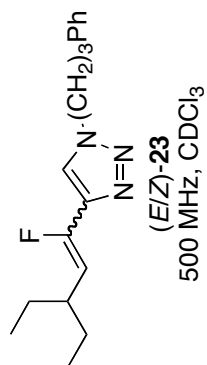


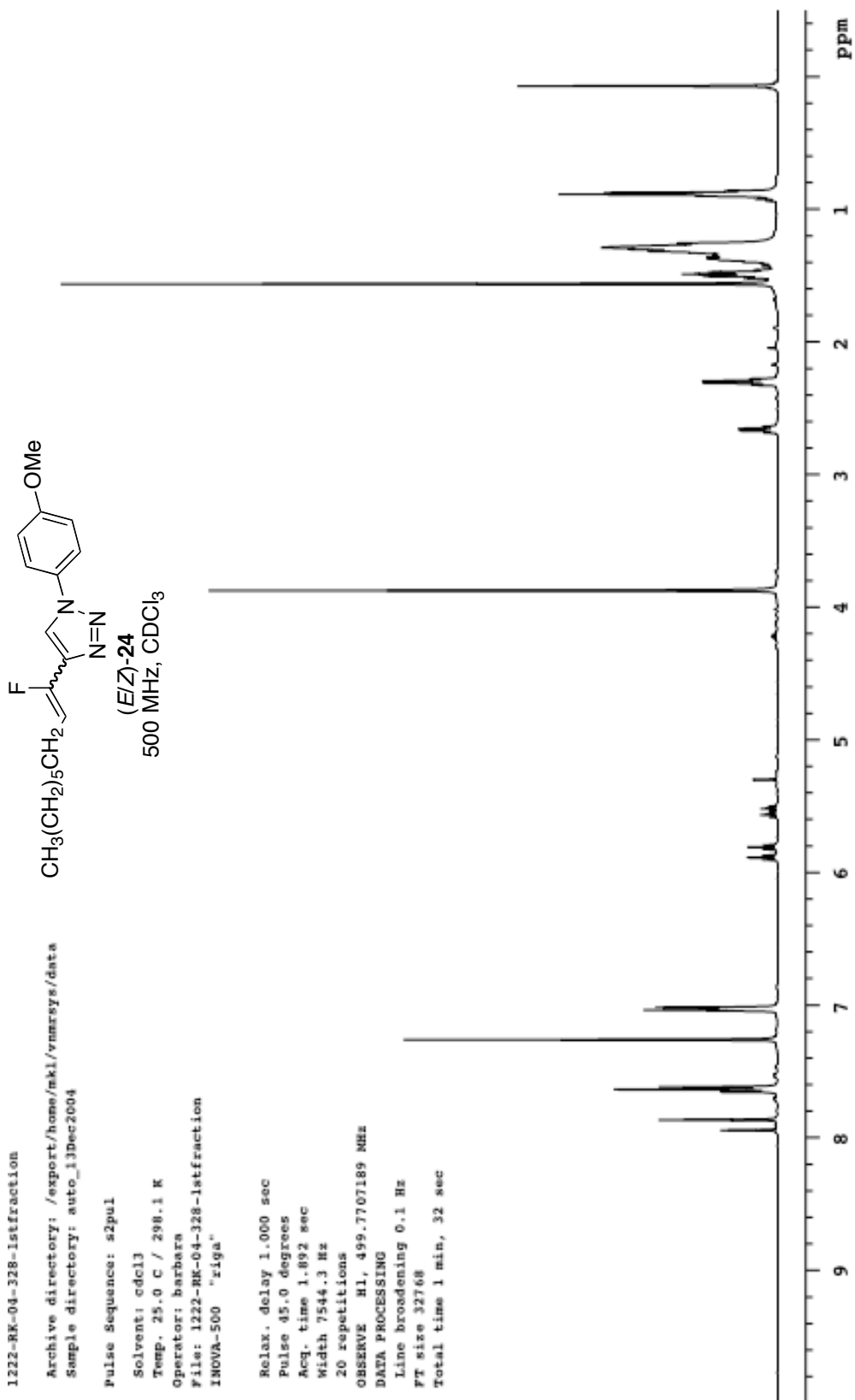
1222-MK-05-336

Archive directory: /export/home/mkl/vnmrsys/data
Sample directory: auto_13Dec2004
File: 1222-MK-05-336

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: Barbara
File: 1222-MK-05-336
INOVA-500 "capella500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7544.3 Hz
16 repetitions
OBSERVE H1, 499.7707226 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 9 min, 40 sec





1222-MK-04-312-pure-2

Archive directory: /export/home/barbara/vnarsys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: cdcl3

Temp. 25.0 C / 298.1 K

Operator: barbara

File: 1222-MK-04-312-pure-2

INOVA-500 "ziga"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.892 sec

Width 7544.3 Hz

36 repetitions

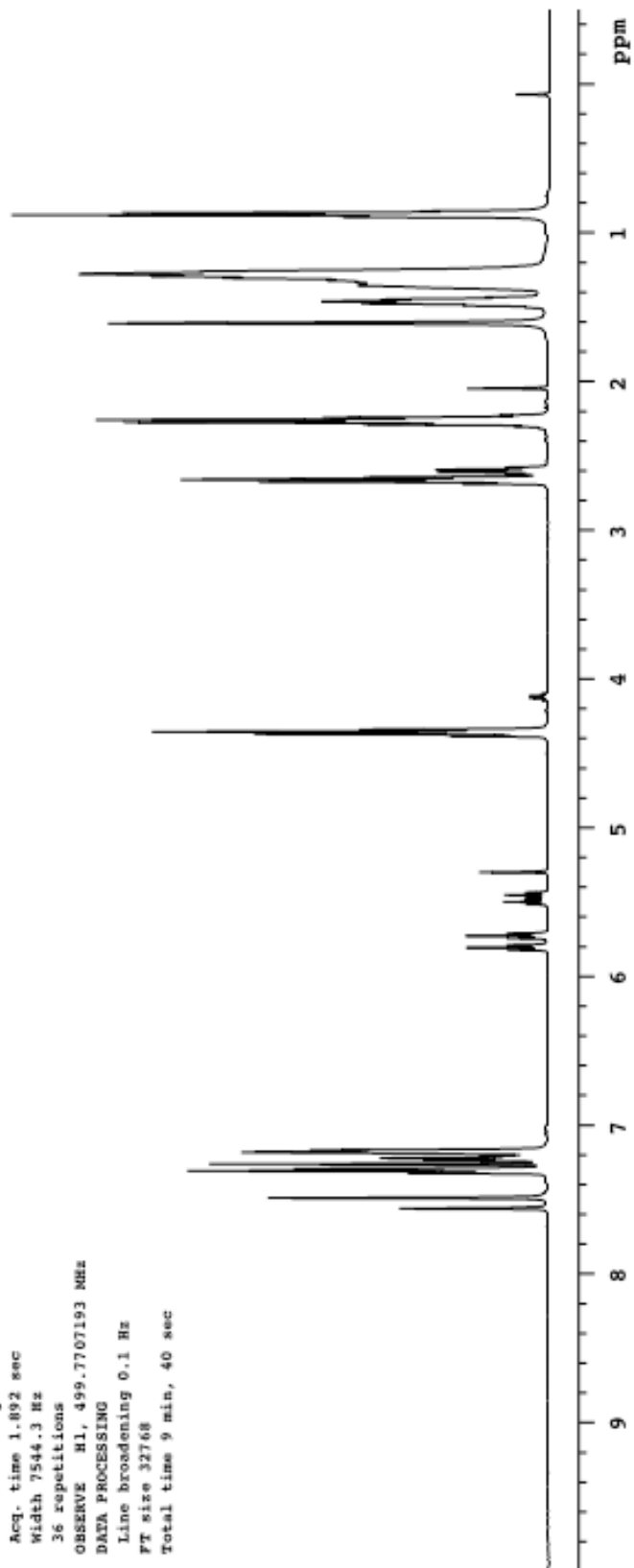
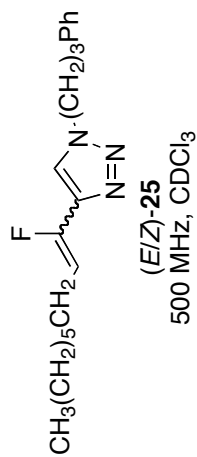
OBSERVE H1, 499.7707193 MHz

DATA PROCESSING

Line broadening 0.1 Hz

FT size 32768

Total time 9 min, 40 sec



1222-RK-05-392-pure

Archive directory: /export/home/mkl/vmrssys/data
Sample directory: auto_13Dec2004

Pulse Sequence: s2pul

Solvent: cdcl3

Temp. 25.0 C / 298.1 K

Operator: barbara

File: 1222-RK-05-392-pure

INOVA-500 "ziga"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.892 sec

Width 10000.0 Hz

16 repetitions

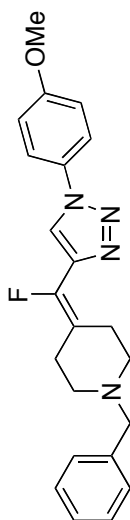
OBSERVE H1, 499.7707212 MHz

DATA PROCESSING

Line broadening 0.1 Hz

FT size 65536

Total time 1 min, 32 sec



26

500 MHz, CDCl₃

