Supplementary Material

Three-Component Reactions of Ketene Dithioacetals, Aldehydes and Arenesulfinic Acids: Facile Synthesis of Allylic Sulfones

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I. General

All reagents were purchased from commercial sources and used without treatment, unless otherwise indicated. The products were purified by column chromatography over silica gel. ^1H NMR and ^{13}C NMR spectra were recorded at 25°C on a Varian 500 MHz and 125 MHz, respectively, and TMS as internal standard. High-resolution mass spectra (HRMS) were obtained using a Bruker microTOF II focus spectrometer (ESI). Melting points were uncorrected. Substrates **1** were synthesized according to the literature procedure.^[1,2] Byproduct **A** is a known compound.^[3]

II. Synthesis of arenesulfinic acids **3** (**3a** as example)

The sodium *p*-tolylsulfinate (25.0 g, 140 mmol) was added to water (300 mL), and the mixture was stirred at room temperature for 5 min to fully dissolve the sodium salt. Then, the solution was cooled in an ice bath, and concentrated sulfuric acid (20 mL) was added dropwise. After that, the aqueous layer was extracted with CH_2Cl_2 (3×200 mL). The combined organic extracts were washed with water (3×150 mL), but could not be dried over any desiccant. After removal of the solvent at 35 °C under reduced pressure for several hours, *p*-toluenesulfinic acid **3a** was obtained as a white solid (18.6 g, 85% yield), which should be stored in a dark sealed storage bottle and used within a week.

III. Synthesis and analytical data of **4a-o,p'**

General Procedure for Synthesis of Allylic Sulfones **4a-o,p' (**4a** as Example):** A 25 mL flask, equipped with a magnetic-stirring bar, was charged with ketene dithioacetal **1a** (143 mg, 1.0 mmol) and *p*-toluenesulfinic acid **3a** (469 mg, 3.0 mmol), followed by addition of acetonitrile (5.0 mL). The mixture was stirred at room temperature for 5 min to fully dissolve the substrates. Then benzaldehyde **2a** (281mg, 2.0 mmol) and

1. Fu, Z.; Wang, M.; Ma, Y.; Liu, Q.; Liu, J. Synthesis of functionalized allylic sulfoxides and their use in the construction of 2,3,4-trisubstituted furans via a [3 + 2] annulation. *J. Org. Chem.* **2008**, 73, 7625–7630.

2. Ma, Y.; Wang, M.; Li, D.; Bekturhun, B.; Liu, J.; Liu, Q. α -Alkenoyl ketene S,S-acetal-based multicomponent reaction: an efficient approach for the selective construction of polyfunctionalized cyclohexanones. *J. Org. Chem.* **2009**, 74, 3116–3121.

3. Zhang, Q.; Liu, Y.; Wang, M.; Liu, Q.; Hu, J.; Yin, Y. Highly efficient C–C bond-forming reactions of α -cyanoketene dithioacetal with aldehydes and ketones. *Synthesis* **2006**, 3009–3014.

concentrated sulfuric acid (0.027 mL, 0.5 mmol) were added, and the solution was stirred at ambient temperature for another 5 h. After **1a** was consumed as indicated by TLC, the reaction mixture was quenched with water, neutralized by saturated aqueous NaHCO₃ solution, and extracted with CH₂Cl₂ three times. The extract was dried over anhydrous MgSO₄. After removal of solvents, the residue was purified by column chromatography (silica gel, petroleum ether : dichloromethane : ethyl acetate = 8 : 2 : 1, V/V) to afford allylic sulfone **4a** as a white solid (392 mg, 93 % yield).

Spectral Data

3-(4-chlorophenyl)-2-(1,3-dithiolan-2-ylidene)-3-tosylpropanenitrile **4a**, white solid: mp 218-219 °C. ¹H NMR (500 MHz, CDCl₃) δ = 2.43 (s, 3H), 3.40-3.44 (m, 1H), 3.46-3.53 (m, 3H), 4.92 (s, 1H), 7.28 (d, J = 8.5 Hz, 2H), 7.33 (d, J = 8.5 Hz, 2H), 7.52 (d, J = 8.0 Hz, 2H), 7.68 (d, J = 8.0 Hz, 2H); ¹³C NMR (125 MHz, CDCl₃) δ = 172.1, 145.4, 135.7, 134.0, 131.1 (2C), 129.7 (2C), 129.3 (2C), 129.1 (2C), 128.8, 117.0, 89.7, 73.7, 39.8, 38.7, 21.7; HRMS (ESI-TOF) Calcd for C₁₉H₁₇ClNO₂S₃⁺ ([M+H]⁺) 422.0104. Found 422.0107.

3-(4-chlorophenyl)-2-(1,3-dithiolan-2-ylidene)-3-(phenylsulfonyl)propanenitrile **4b**, white solid: mp 165-166 °C. ¹H NMR (400 MHz, CDCl₃) δ = 3.37-3.52 (m, 4H), 4.93 (s, 1H), 7.34 (dt, J = 8.8, 2.0 Hz, 2H), 7.48-7.55 (m, 4H), 7.63 (t, J = 7.4 Hz, 1H), 7.84 (dd, J = 8.4, 1.2 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃) δ = 171.9, 137.2, 135.9, 134.2, 131.2 (2C), 129.4 (2C), 129.2 (2C), 129.1 (2C), 128.6, 116.9, 89.9, 73.8, 39.8, 38.7; HRMS (ESI-TOF) Calcd for C₁₈H₁₅ClNO₂S₃⁺ ([M+H]⁺) 407.9948. Found 407.9951.

3-(4-chlorophenyl)-3-((4-chlorophenyl)sulfonyl)-2-(1,3-dithiolan-2-ylidene)propanenitrile **4c**, white solid: mp 186-187 °C. ¹H NMR (400 MHz, CDCl₃) δ = 3.41-3.55 (m,

4H), 4.91 (s, 1H), 7.36 (d, $J = 8.8$ Hz, 2H), 7.48 (d, $J = 8.4$ Hz, 2H), 7.54 (d, $J = 8.4$ Hz, 2H), 7.77 (d, $J = 8.8$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) $\delta = 172.2, 141.1, 136.0, 135.7, 131.2$ (2C), 130.9 (2C), 129.4 (2C), 129.3 (2C), 128.3, 116.8, 89.6, 73.8, 39.9, 38.8; HRMS (ESI-TOF) Calcd for $\text{C}_{18}\text{H}_{14}\text{Cl}_2\text{NO}_2\text{S}_3^+$ ($[\text{M}+\text{H}]^+$) 441.9558. Found 441.9552.

2-(1,3-dithiolan-2-ylidene)-3-(4-nitrophenyl)-3-tosylpropanenitrile **4d**, white solid: mp 214-215 °C. ^1H NMR (500 MHz, CDCl_3) $\delta = 2.44$ (s, 3H), 3.45-3.47 (m, 1H), 3.50-3.56 (m, 3H), 5.05 (s, 1H), 7.31 (d, $J = 8.5$ Hz, 2H), 7.71 (d, $J = 8.5$ Hz, 2H), 7.79 (d, $J = 9.0$ Hz, 2H), 8.23 (d, $J = 9.0$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) $\delta = 173.2, 148.1, 145.8, 137.2, 133.6, 130.8$ (2C), 129.7 (2C), 129.1 (2C), 123.7 (2C), 116.7, 88.4, 73.2, 39.8, 38.7, 21.6; HRMS (ESI-TOF) Calcd for $\text{C}_{19}\text{H}_{17}\text{N}_2\text{O}_4\text{S}_3^+$ ($[\text{M}+\text{H}]^+$) 433.0345. Found 433.0346.

2-(1,3-dithiolan-2-ylidene)-3-(2-nitrophenyl)-3-tosylpropanenitrile **4e**, white solid: mp 228-229 °C. ^1H NMR (500 MHz, CDCl_3) $\delta = 2.44$ (s, 3H), 3.32-3.35 (m, 1H), 3.49-3.52 (m, 3H), 6.21 (s, 1H), 7.30 (d, $J = 8.0$ Hz, 2H), 7.57 (dd, $J = 7.5, 8.0$ Hz, 1H), 7.73 (d, $J = 8.0$ Hz, 2H), 7.77 (dd, $J = 7.5, 8.0$ Hz, 1H), 7.93 (d, $J = 8.0$ Hz, 1H), 8.50 (d, $J = 7.0$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) $\delta = 174.2, 149.2, 145.4, 134.0, 133.6, 131.0, 130.1, 129.7, 129.2, 125.2, 124.8, 117.1, 88.3, 65.9, 39.9, 38.6, 21.7$; HRMS (ESI-TOF) Calcd for $\text{C}_{19}\text{H}_{17}\text{N}_2\text{O}_4\text{S}_3^+$ ($[\text{M}+\text{H}]^+$) 433.0345. Found 433.0357.

2-(1,3-dithiolan-2-ylidene)-3-p-tolyl-3-tosylpropanenitrile **4f**, white solid: mp 207-208 °C. ^1H NMR (500 MHz, CDCl_3) $\delta = 2.34$ (s, 3H), 2.42 (s, 3H), 3.40-3.51 (m, 4H), 4.91 (s, 1H), 7.16 (d, $J = 8.0$ Hz, 2H), 7.26 (d, $J = 8.0$ Hz, 2H), 7.45 (d, $J = 8.0$ Hz, 2H), 7.68 (d, $J = 8.5$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) $\delta = 170.9, 145.0, 139.5, 134.3, 129.6$ (2C), 129.5 (2C), 129.44 (2C), 129.40 (2C), 127.1, 117.1, 90.7, 74.4, 39.6, 38.5, 21.7, 21.2; HRMS (ESI-TOF) Calcd for $\text{C}_{20}\text{H}_{20}\text{NO}_2\text{S}_3^+$ ($[\text{M}+\text{H}]^+$) 402.0651. Found 402.0646.

2-(1,3-dithiolan-2-ylidene)-3-(4-methoxyphenyl)-3-tosylpropanenitrile **4g**, white solid: mp 237-238 °C. ^1H NMR (500 MHz, CDCl_3) δ = 2.42 (s, 3H), 3.41-3.51 (m, 4H), 3.81 (s, 3H), 4.90 (s, 1H), 6.87 (d, J = 8.5 Hz, 2H), 7.26 (d, J = 8.5 Hz, 2H), 7.49 (d, J = 8.5 Hz, 2H), 7.67 (d, J = 8.5 Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ = 170.8, 160.4, 144.9, 134.3, 131.1 (2C), 129.5 (2C), 129.4 (2C), 122.1, 117.1, 114.2 (2C), 90.8, 74.1, 55.3, 39.6, 38.6, 21.7; HRMS (ESI-TOF) Calcd for $\text{C}_{20}\text{H}_{20}\text{NO}_3\text{S}_3^+$ ($[\text{M}+\text{H}]^+$) 418.0600. Found 418.0618.

3-(benzo[d][1,3]dioxol-5-yl)-2-(1,3-dithiolan-2-ylidene)-3-tosylpropanenitrile **4h**, white solid: mp 232-233 °C. ^1H NMR (500 MHz, DMSO) δ = 2.40 (s, 3H), 3.38-3.42 (m, 1H), 3.53-3.59 (m, 3H), 5.16 (s, 1H), 6.06 (s, 2H), 6.94 (d, J = 8.0 Hz, 1H), 7.05 (d, J = 7.5 Hz, 1H), 7.22, (s, 1H), 7.42 (d, J = 8.0 Hz, 1H), 7.70 (d, J = 7.5 Hz, 1H); ^{13}C NMR (125 MHz, DMSO) δ = 173.8, 148.6, 147.9, 145.6, 134.8, 130.3 (2C), 129.3 (2C), 124.5, 124.3, 118.0, 109.9, 109.0, 102.1, 89.0, 72.2, 40.6, 39.3, 21.7; HRMS (ESI-TOF) Calcd for $\text{C}_{20}\text{H}_{18}\text{NO}_4\text{S}_3^+$ ($[\text{M}+\text{H}]^+$) 432.0392. Found 432.0376.

2-(1,3-dithiolan-2-ylidene)-3-phenyl-3-tosylpropanenitrile **4i**, white solid: mp 219-220 °C. ^1H NMR (500 MHz, CDCl_3) δ = 2.42 (s, 3H), 3.42-3.53 (m, 4H), 4.95 (s, 1H), 7.26 (d, J = 8.0 Hz, 2H), 7.34-7.38 (m, 3H), 7.56 (dd, J = 2.0, 8.0 Hz, 2H), 7.67 (d, J = 8.5 Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ = 171.6, 145.2, 134.3, 130.3, 129.8 (2C), 129.6 (2C), 129.5, 129.3 (2C), 128.9 (2C), 117.2, 90.2, 74.5, 39.8, 38.7, 21.7; HRMS (ESI-TOF) Calcd for $\text{C}_{19}\text{H}_{18}\text{NO}_2\text{S}_3^+$ ($[\text{M}+\text{H}]^+$) 388.0494. Found 388.0490.

2-(1,3-dithiolan-2-ylidene)-3-(furan-2-yl)-3-tosylpropanenitrile **4j**, yellow solid: mp 208-209 °C. ^1H NMR (500 MHz, CDCl_3) δ = 2.44 (s, 3H), 3.51-3.59 (m, 4H), 5.22 (s,

1H), 6.42 (s, 1H), 6.78 (d, $J = 2.5$ Hz, 1H), 7.31 (d, $J = 8.0$ Hz, 2H), 7.39 (s, 1H), 7.65 (d, $J = 7.5$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) $\delta = 172.5, 145.4, 143.4, 143.1, 133.3, 129.52, 129.49, 116.7, 111.9, 111.3, 88.0, 68.8, 39.8, 38.8, 21.7$; HRMS (ESI-TOF) Calcd for $\text{C}_{17}\text{H}_{16}\text{NO}_3\text{S}_3^+$ ($[\text{M}+\text{H}]^+$) 378.0287. Found 378.0285.

2-(1,3-dithiolan-2-ylidene)-3-tosylpropanenitrile **4k**, white solid: mp > 250 °C. ^1H NMR (500 MHz, CDCl_3) $\delta = 2.47$ (s, 3H), 3.51 (t, $J = 7.0$ Hz, 2H), 3.59 (t, $J = 6.5$ Hz, 2H), 4.02 (s, 2H), 7.39 (d, $J = 8.5$ Hz, 2H), 7.81 (d, $J = 8.0$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) $\delta = 172.1, 145.6, 134.9, 130.1$ (2C), 128.7 (2C), 117.3, 84.3, 60.9, 39.9, 39.0, 21.8; HRMS (ESI-TOF) Calcd for $\text{C}_{13}\text{H}_{14}\text{NO}_2\text{S}_3^+$ ($[\text{M}+\text{H}]^+$) 312.0181. Found 312.0198.

3,3-bis(methylthio)-2-(tosylmethyl)acrylonitrile **4l**, white solid: 149-150 °C. ^1H NMR (500 MHz, CDCl_3) $\delta = 2.31$ (s, 3H), 2.46 (s, 3H), 2.49 (s, 3H), 4.32 (s, 2H), 7.38 (d, $J = 8.0$ Hz, 2H), 7.80 (d, $J = 8.0$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) $\delta = 166.6, 145.5, 135.1, 129.9$ (2C), 128.7 (2C), 116.5, 101.2, 59.4, 21.7, 18.2, 17.8; HRMS (ESI-TOF) Calcd for $\text{C}_{13}\text{H}_{16}\text{NO}_2\text{S}_3^+$ ($[\text{M}+\text{H}]^+$) 314.0338. Found 314.0339.

3,3-bis(ethylthio)-2-(tosylmethyl)acrylonitrile **4m**, white solid: 72-73 °C. ^1H NMR (500 MHz, CDCl_3) $\delta = 1.20$ (t, $J = 7.5$ Hz, 3H), 1.31 (t, $J = 7.5$ Hz, 3H), 2.46 (s, 3H), 2.85 (q, $J = 7.5$ Hz, 2H), 2.94 (q, $J = 7.5$ Hz, 2H), 4.36 (s, 2H), 7.38 (d, $J = 7.5$ Hz, 2H), 7.80 (d, $J = 8.0$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) $\delta = 163.7, 145.6, 135.1, 130.0$ (2C), 128.7 (2C), 116.6, 104.5, 59.5, 30.2, 29.7, 21.7, 15.0; HRMS (ESI-TOF) Calcd for $\text{C}_{15}\text{H}_{20}\text{NO}_2\text{S}_3^+$ ($[\text{M}+\text{H}]^+$) 342.0651. Found 342.0638.

4,4-bis(ethylthio)-3-(tosylmethyl)but-3-en-2-one **4n**, light yellow solid: 82-83 °C. ^1H NMR (500 MHz, CDCl_3) $\delta = 1.11$ (t, $J = 7.5$ Hz, 3H), 1.29 (t, $J = 7.5$ Hz, 3H), 2.46 (s,

3H), 2.60 (s, 3H), 2.68 (q, $J = 7.0$ Hz, 2H), 2.83 (q, $J = 7.5$ Hz, 2H), 4.59 (s, 2H), 7.36 (d, $J = 8.0$ Hz, 2H), 7.76 (d, $J = 8.0$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) $\delta = 202.7$, 145.9, 145.0, 136.8, 135.4, 129.9 (2C), 128.5 (2C), 61.9, 30.7, 28.7, 28.3, 21.7, 14.9, 14.8; HRMS (ESI-TOF) Calcd for $\text{C}_{16}\text{H}_{23}\text{O}_3\text{S}_3^+$ ($[\text{M}+\text{H}]^+$) 359.0804. Found 359.0806.

3,3-bis(ethylthio)-1-phenyl-2-(tosylmethyl)prop-2-en-1-one **4o**, white solid: 133-134 °C. ^1H NMR (500 MHz, CDCl_3) $\delta = 0.98$ (t, $J = 7.5$ Hz, 3H), 1.21 (t, $J = 7.5$ Hz, 3H), 2.40 (s, 3H), 2.67 (q, $J = 7.5$ Hz, 2H), 2.81 (q, $J = 7.5$ Hz, 2H), 4.71 (s, 2H), 7.27 (dd, $J = 4.0, 8.0$ Hz, 2H), 7.43 (dd, $J = 7.5, 7.5$ Hz, 2H), 7.53 (dd, $J = 7.5, 7.5$ Hz, 1H), 7.77 (d, $J = 8.0$ Hz, 2H), 7.92 (d, $J = 8.0$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) $\delta = 195.6$, 148.6, 144.8, 137.1, 136.4, 132.7, 132.5, 129.7 (2C), 129.4 (2C), 128.2 (2C), 128.1 (2C), 62.3, 29.2, 28.3, 21.6, 15.1, 13.9; HRMS (ESI-TOF) Calcd for $\text{C}_{21}\text{H}_{25}\text{O}_3\text{S}_3^+$ ($[\text{M}+\text{H}]^+$) 421.0960. Found 421.0964.

1,1-bis(ethylthio)-5-phenyl-5-tosyl-2-(tosylmethyl)pent-1-en-3-one **4p'**, semisolid. ^1H NMR (500 MHz, CDCl_3) $\delta = 1.07$ (t, $J = 7.5$ Hz, 3H), 1.33 (t, $J = 7.5$ Hz, 3H), 2.38 (s, 3H), 2.46 (s, 3H), 2.63-2.66 (m, 2H), 2.75-2.83 (m, 2H), 4.00-4.06 (m, 1H), 4.22 (d, $J = 17.5$ Hz, 1H), 4.47 (s, 2H), 4.74 (d, $J = 8.5$ Hz, 1H), 7.17 (d, $J = 7.5$ Hz, 2H), 7.23-7.27 (m, 5H), 7.35 (d, $J = 7.5$ Hz, 2H), 7.42 (d, $J = 7.5$ Hz, 2H), 7.72 (d, $J = 7.5$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) $\delta = 199.7$, 147.7, 145.1, 144.5, 135.32, 135.25, 134.1, 132.4, 130.1 (2C), 129.9 (2C), 129.2 (2C), 129.0 (2C), 128.6 (2C), 128.5, 128.1 (2C), 66.8, 61.8, 40.4, 29.1, 28.5, 21.7, 21.6, 14.8, 14.5; HRMS (ESI-TOF) Calcd for $\text{C}_{30}\text{H}_{35}\text{O}_5\text{S}_4^+$ ($[\text{M}+\text{H}]^+$) 603.1362. Found 603.1369.

IV. Synthesis and analytical data of **4p**

To the solution of sulfone **4p'** (301 mg, 0.5 mmol) in N,N-dimethylformamide (2 mL) was added NaOH (20.0 mg, 0.5 mmol). The reaction mixture was stirred at room

temperature for 10 h, then the substrate **4p'** was consumed as indicated by TLC. The resulting mixture was quenched with water and extracted with CH₂Cl₂ three times. The extract was dried over anhydrous MgSO₄. After removal of solvents, the residue was purified by column chromatography (silica gel, petroleum ether : dichloromethane : ethyl acetate = 8 : 3 : 1, V/V) to afford allylic sulfone **4p** as a white solid (219 mg, 98 % yield).

Spectral Data

(*E*)-1,1-bis(ethylthio)-5-phenyl-2-(tosylmethyl)penta-1,4-dien-3-one **4p**, white solid: 126-127 °C. ¹H NMR (500 MHz, CDCl₃) δ = 1.21 (t, *J* = 7.5 Hz, 3H), 1.35 (t, *J* = 7.5 Hz, 3H), 2.20 (s, 3H), 2.82 (q, *J* = 7.5 Hz, 2H), 2.89 (q, *J* = 7.5 Hz, 2H), 4.72 (s, 2H), 6.92 (d, *J* = 15.5 Hz, 1H), 7.23 (d, *J* = 7.5 Hz, 2H), 7.39-7.42 (m, 4H), 7.49 (dd, *J* = 3.5, 7.5 Hz, 2H), 7.73 (d, *J* = 7.5 Hz, 2H); ¹³C NMR (125 MHz, CDCl₃) δ = 192.4, 148.0, 145.0, 140.6, 136.1, 135.8, 134.9, 130.2, 129.7 (2C), 128.9 (2C), 128.6 (2C), 128.2 (2C), 125.9, 61.6, 29.3, 28.6, 21.4, 15.2, 15.1; HRMS (ESI-TOF) Calcd for C₂₃H₂₇O₃S₃⁺ ([M+H]⁺) 447.1117. Found 447.1117.

V. Copies of NMR spectra for all new compounds

4a

STANDARD PROTON PARAMETERS
 Archive directory: /export/home/ouyy/nmr/sys/data
 Sample directory:
 Pulse Sequence: szpu1
 Solvent: CDCl3
 Ambient temperature
 File: c224
 INOVA-500 "NMR500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.692 sec
 Width 7198.0 Hz
 8 repetitions
 OBSERVE H1, 499.8025875 MHz
 DATA PROCESSING
 FT size 65536
 Total time 0 min, 23 sec

STANDARD CARBON PARAMETERS
 Archive directory: /export/home/ouyy/nmr/sys/data
 Sample directory:
 Pulse Sequence: szpu1
 Solvent: cdd
 Ambient temperature
 User: 1-14-87
 File: c231
 INOVA-500 "NMR500"
 Relax. delay 0.500 sec
 Pulse 45.0 degrees
 Acq. time 1.200 sec
 Width 31821.0 Hz
 64 repetitions
 OBSERVE C13, 125.6754664 MHz
 DECOUPLE H1, 499.8051355 MHz
 Power 40.00
 Continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 131072
 Total time 2 hr, 3 min, 31 sec

4b

2008106ph2-H

2008106PH2-C13_CARBON_01
Gradient Shimming

4c

20081064-C11-H

20081064-C11-C13_CARBON_01
Gradient Shimming

4d

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STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouzy/vmr/sys/data
Sample directory:
Pulse Sequence: szpul
Solvent: CDCl3
Ambient temperature
File: c331
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.052 sec
Width 7996.8 Hz
0 repetitions
OBSERVE H1 499.8625073 MHz
DATA PROCESSING
FT size 65536
Total time 0 min. 23 sec

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STANDARD CARBON PARAMETERS
Archive directory: /export/home/rouxy/vnmr51/data
Sample directory:

Pulse Sequence: s2pul
Solvent: C6D6
Ambient temperature
User: j-14-87
File: C553
INSTRUM=508 "MENU508"

Relax. delay 0.500 sec
Pulse 45.0 degree
Acq. time 1.300 sec
Width 31621.8 Hz
120 repetitions
OBSERVE C13 125.6754878 MHz
DECOUPLE M1 499.806535 MHz
Power 42 db
cont inuous 13C on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
F2 size 133972
Total time 3 hr, 56 sec

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4e

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ougy/vnmrsys/data
Sample directory:
Pulse Sequence: szpu1
Solvent: CDCl3
Ambient temperature
File: c897
INOVA-500 "HREUS00"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.002 sec
Width 8578.2 Hz
8 repetitions
OBSERVE H1, 499.8025870 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ougy/vnmrsys/data
Sample directory:
Pulse Sequence: szpu1
Solvent: cdcl3
Ambient temperature
User: j-14-87
File: c898
INOVA-500 "HREUS00"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.380 sec
Width 31421.8 Hz
256 repetitions
OBSERVE C13, 125.6754828 MHz
DECOUPLE H1, 499.8050905 MHz
Power 62 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131372
Total time 2 hr, 3 min, 31 sec

4f

STANDARD PROTON PARAMETERS
 Archive directory: /export/home/ouyy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 File: d051
 INOVA-500 "NMRUS00"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.032 sec
 Width 6576.1 Hz
 0 repetitions
 OBSERVE H1, 499.8025891 MHz
 DATA PROCESSING
 FT size 65336
 Total time 0 min, 23 sec

STANDARD CARBON PARAMETERS
 Archive directory: /export/home/ouyy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
 User: 1-14-87
 File: d052
 INOVA-500 "NMRUS00"
 Relax. delay 0.500 sec
 Pulse 45.0 degrees
 Acq. time 1.350 sec
 Width 31421.8 Hz
 132 repetitions
 OBSERVE C13, 125.8754733 MHz
 DECOUPLE H1, 499.8008985 MHz
 Power 42 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.5 Hz
 FT size 131872
 Total time 2 hr, 3 min, 31 sec

4g

STANDARD PROTON PARAMETERS
Archive directory: /export/home/cuoy/vnmr/sys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 0105
INOVA-500 "NEMUS00"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.412 sec
Width 8578.2 Hz
8 repetitions
OBSERVE H1, 499.8025893 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec

STANDARD CARBON PARAMETERS
Archive directory: /export/home/cuoy/vnmr/sys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-07
File: 0106
INOVA-500 "NEMUS00"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.380 sec
Width 31421.8 Hz
150 repetitions
OBSERVE C13, 125.4754704 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
Continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131972
Total time 2 hr, 3 min, 31 sec

4h

STANDARD PROTON PARAMETERS
 Archive directory: /export/home/ougy/vnmr/sys/data
 Sample directory:
 Pulse Sequence: s2pul
 Solvent: DMSO
 Ambient temperature
 File: c637
 INOVA-500 "HENU500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.852 sec
 Width 7889.7 Hz
 8 repetitions
 OBSERVE H1, 499.8049581 MHz
 DATA PROCESSING
 FI size 65536
 Total time 6 min, 23 sec

STANDARD CARBON PARAMETERS
 Archive directory: /export/home/ougy/vnmr/sys/data
 Sample directory:
 Pulse Sequence: s2pul
 Solvent: DMSO
 Ambient temperature
 User: j-14-87
 File: c637
 INOVA-500 "HENU500"
 Relax. delay 0.500 sec
 Pulse 45.0 degrees
 Acq. time 1.380 sec
 Width 31431.8 Hz
 328 repetitions
 OBSERVE C13, 125.6769473 MHz
 DECOUPLE H1, 499.8074646 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.5 Hz
 FI size 131072
 Total time 3 hr, 56 sec

4i

```
STANDARD PROTON PARAMETERS
Archive directory: /export/home/roxy/vmrsys/data
Sample directory:
Pulse Sequence: szpul
Solvent: CDCl3
Ambient temperature
File: c160
INNOVA-500 "NENUS500"

Relax delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.832 sec
Width 7986.8 Hz
8 repetitions
OBSERVE M1 499.8025982 MHz
DATA PROCESSING
FT size 65536
Total time 9 min, 23 sec
```

```

STANDARD CARBON PARAMETERS
Archive directory: /export/home/rouzy/nmr/sigs/data
Sample directory:

Pulse Sequence: sz2pul
Solvent: c4d6
Solvent temperature
User: 1-14-87
File: c1375
INSTRUM="MUN500"

Relax. delay 0.500 sec
Pulse 45.0 degrees
Pulse 1 2.00 sec
Width 31621.8 Hz
575 repetitions
DECODE F1, 489.805455 MHz
Power 45
continuously on
val72: if enabled
Data PROCESSING
Line broadening 1.5 Hz
FT time 131072
Total time 2 hr, 3 min, 31 sec

```

4j

STANDARD PROTON PARAMETERS
 Archive directory: /export/home/duffy/vnmr/sys/data
 Sample directory:
 Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 File: c895
 INOVA-500 "HENU500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.882 sec
 Width 8578.2 Hz
 OBSERVE F1, 499.8025857 MHz
 8 repetitions
 DATA PROCESSING
 FT size 85536
 Total time 0 min, 23 sec

STANDARD CARBON PARAMETERS
 Archive directory: /export/home/duffy/vnmr/sys/data
 Sample directory:
 Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
 User: j-14-07
 File: c896
 INOVA-500 "HENU500"
 Relax. delay 9.500 sec
 Pulse 45.0 degrees
 Acq. time 1.380 sec
 Width 31421.0 Hz
 128 repetitions
 OBSERVE F1, 125.6754823 MHz
 DECOUPLE H1, 499.8050905 MHz
 Power 42 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 131972
 Total time 2 hr, 3 min, 31 sec

4k

STANDARD PROTON PARAMETERS
Archive directory: /export/home/oupy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: d174
INOVA-500 "HREUS500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.889 sec
Width 8578.2 Hz
8 repetitions
OBSERVE R1 499.8025875 MHz
DATA PROCESSING
FT size 65536
Total time 9 min, 23 sec

STANDARD CARBON PARAMETERS
Archive directory: /export/home/oupy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: j-14-87
File: d175
INOVA-500 "HREUS500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.388 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754666 MHz
DECOUPLE R1 499.8054985 MHz
Power 42 dB
continuous by on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 133872
Total time 2 hr, 3 min, 31 sec

4m

```
STANDARD PROTON PARAMETERS
Archive directory: /export/home/rooy/vnmrsys/data
Sample directory:
Pulse Sequence: szpul
Solvent: CDCl3
Relaxant temperature
File: c480
INOVA-50 "NENU50"

Relax. delay 1.000 sec
Pulse 4.0 degrees
Acq. time 1.832 sec
Width 8578.2 Hz
8 repetitions
GHSFPRF HI, 499.8025836 MHZ
DATA PROCESSING
FT size 65536
Total time 2 min, 23 sec
```

```

STANDARD CARBON PARAMETERS
Archive directory: /export/home/roby/vnmr/sys/data
Sample directory:

Pulse Sequence: szpul
Solvent: cdcl3
Solvent temperature
User: i-14-87
File: c485
INNOVA-500 "HENU509"

Relax. delay 8.500 sec
Pulse 45.0 degrees
Turn 1 388 sec
Width 23.621 8 Hz
Z 320 repetitions
OBSERVE C13 225.4754721 MHz
DECOUPLE M1 499.805125 MHz
Power 42 dB
CONTINUOUSLY ON
PULW12-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
SI 424 133077
Total time 2 hr, 3 min, 31 sec

```

4n

STANDARD PROTON PARAMETERS
 Archive directory: /export/home/ougy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient Temperature
 File: c882
 INOVA-500 "NMR500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.882 sec
 Width 8578.2 Hz
 8 repetitions
 OBSERVE H1: 499.8025757 MHz
 DATA PROCESSING
 FT size 65536
 Total time 0 min, 23 sec

STANDARD CARBON PARAMETERS
 Archive directory: /export/home/ougy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient Temperature
 User: i-14-87
 File: c380
 INOVA-500 "NMR500"
 Relax. delay 0.500 sec
 Pulse 45.0 degrees
 Acq. time 1.350 sec
 Width 31421.8 Hz
 4896 repetitions
 OBSERVE C13: 125.6756685 MHz
 DECOUPLE H1: 499.8025757 MHz
 Power 42 dB
 continuously on
 WALTZ-16 modulated
 Data PROCESSING
 Line broadening 1.5 Hz
 FT size 131072
 Total time 2 hr, 3 min, 31 sec

STANDARD PROTON PARAMETERS
 Archive directory: /export/home/cuy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 File: c384
 INOVA-500 "NENUS00"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 3.000 sec
 Width 6576.2 Hz
 8 repetitions
 OBSERVE H1, 499.8025675 MHz
 DATA PROCESSING
 FT size 65536
 Total time 0 min, 23 sec

STANDARD CARBON PARAMETERS
 Archive directory: /export/home/cuy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
 User: 1-14-87
 File: c385
 INOVA-500 "NENUS00"
 Relax. delay 0.500 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 31421.8 Hz
 64 repetitions
 OBSERVE C13, 125.6754685 MHz
 DECOUPLE H1, 499.8059905 MHz
 Power 42 dB
 Continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line Broadening 1.5 Hz
 FT size 131072
 Total time 2 hr, 3 min, 31 sec

4p'

```

STANDARD PROTON PARAMETERS
Archive directory: /export/home/owuy/vmrs/rs/data
Sample directory:
Pulse sequence: zgpu1
Solvent: CDCl3
Ambient temperature
File: d185
INOVA-500 "NEMUS00"

Relax: delay 1.000 sec
Pulse 45.0 degree
Rise time 1.032 sec
Width 8576.2 HZ
8 repetitions
ORIGIN: HI, 499.6025880 MHz
DATA PROCESSING
F1 size 65336
Total time 5 min, 23 sec

```

```

STANDARD CARBON PARAMETERS
Archive directory: /export/home/buivy/vnmr/sys/data
Sample directory:

Pulse Sequence: szpu1
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: d588
INNOVA-500 "HRENU500"

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.0 HZ
152 repetitions
OBSERVE CH1: 125.4756460 MHz
DECOUPLE M1: 493.8058905 MHz
Power 62.5
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 MZ
F1 size 134972
Total time 2 hr, 3 min, 31 sec

```

4p

STANDARD PROTON PARAMETERS
 Archive directory: /export/home/owgy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 File: d204
 INOVA-500 "NMRUS00"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.002 sec
 Width 6578.2 Hz
 OBSERVE F1, 499.8025986 MHz
 DATA PROCESSING
 FT size 65536
 Total time 0 min, 23 sec

STANDARD CARBON PARAMETERS
 Archive directory: /export/home/owgy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pu1
 Solvent: cdcl3
 Ambient temperature
 User: 1-10-97
 File: d205
 INOVA-500 "NMRUS00"
 Relax. delay 0.500 sec
 Pulse 45.0 degrees
 Acq. time 1.200 sec
 Width 31021.8 Hz
 132 repetitions
 OBSERVE C13, 125.6754661 MHz
 DECOUPLE H1, 499.8050905 MHz
 Power 42 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.5 Hz
 FT size 131072
 Total time 2 hr, 9 min, 31 sec