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Synthesis of new tetraalkoxyquinolines from parsley and dill secondary metabolites

Victor E. Kalugin, Olga I. Adaeva, Dmitry V. Demchuk and Victor V. Semenov

Table of Content

General Experimental Details S2

Synthesis of nitro aldehydes 4 and 5 ..... S2

Synthesis of nitro derivatives of the Knoevenagel reaction products 7a–c and 8a–c (general procedure) S2

Synthesis of Knoevenagel reaction products 9a–c and 10a–c with aldehydes 1 and 2 (general procedure) S4

Synthesis of Knoevenagel reaction products 9d and 10d of aldehydes 1 and 2 with dimethyl malonate (general procedure) S5

Nitration of the Knoevenagel reaction products 9a–d and 10a–d..... S6

Synthesis of quinoline-3-carboxylic acid derivatives 11-15 ..... S6

Synthesis of quinolines 16a–c S8

Synthesis of acetanilides 18a–d starting from carboxylic acids 17a–d (general procedure) S9

Synthesis of 2-chloroquinoline-3-carbaldehyde derivatives 19a-d (general procedure).... S10

Synthesis of methyl chloroquinolinecarboxylates 20a-c (general procedure)..... S11

Synthesis of chloro(hydroxyiminomethyl)quinoline derivatives 21a-c (general procedure) S12

Synthesis of chloroquinolinemethanol derivatives 22a,b (general procedure) S14

Synthesis of 2-chloro-5,6,7,8-tetramethoxyquinoline-3-methanol 22c..... S14

Synthesis of 6-chloro-4,9-dimethoxy [1,3]dioxolo[4,5-g]quinoline-7-carbonitrile 23a..... S15

Synthesis of 2-chloro-5,6,7,8-tetramethoxyquinoline-3-carbonitrile 23c S15

References..... S15

General Experimental Details

The melting points were determined on a Nagema PHMK 05 polarizing microscope with a Boetius heating stage. ^{1}H and ^{13}C NMR spectra were recorded in DMSO-d_6 and $CDCl_3$ on Bruker DRX500 and Bruker AM300 spectrometers (operating frequency 500.13 and 300.13 MHz for ^{1}H and 125.76 and 75.47 MHz for ^{13}C, respectively). Chemical shifts in the ^{1}H NMR spectra are given relative to the residual proton signal of the solvent ($CHCl_3$ – δ_H 7.27 ppm, DMSO – δ_H 2.50 ppm, D_2O – δ_H 4.75 ppm), in the ^{13}C NMR spectra – relative to the solvent signal ($CDCl_3$ – δ_C 77.0 ppm, DMSO – δ_C 39.5 ppm). Mass spectra (m/z) were recorded on a Finnigan MAT/INCOS 50 mass spectrometer at 70 eV using direct input. High resolution mass spectra (HRMS) were obtained on a Bruker micrOTOF 10248 instrument by electrospray ionization (ESI). Elemental microanalyses were performed on a Perkin-Elmer 2400 CHN analyzer.

Synthesis of nitro aldehydes 4 and 5

Nitro aldehydes **4** and **5** were obtained by nitration of apiol aldehyde **1** and dilapiol aldehyde **2** with concentrated nitric acid according to the described procedure [S1].

Synthesis of nitro derivatives of the Knoevenagel reaction products 7a–c and 8a–c (general procedure). ***Method A.*** A mixture of nitro aldehyde **4** or **5** (0.51 g, 2 mmol) of and malonic acid derivative **6b,c** (2.3 mmol) in methanol (5 ml) was heated until the substances were completely dissolved, and a few drops of triethylamine were added. The reaction mixture was refluxed for 2 h for methyl cyanoacetate **6b** and 4 h for cyanoacetamide **6c**, then it was cooled to 0 °C. The precipitated crystals were filtered off, washed with cold methanol, and dried on air.

Method B. To a solution of nitro aldehyde **4** or **5** (0.51 g, 2 mmol) and malononitrile **6a** (0.15 g, 2.3 mmol) in CH_2Cl_2 (5 ml), aluminum oxide (0.6 g) was added. The reaction mass was stirred for 4 h at 20–25 °C, then the aluminum oxide was filtered off, the precipitate was washed with CH_2Cl_2, and after evaporation of the filtrate, the product was crystallized from methanol.

[(4,7-Dimethoxy-6-nitro-1,3-benzodioxol-5-yl)methylene]malononitrile (7a)

Yield 0.52 g (86%), m.p. 156–158 °C. **^{1}H NMR** (DMSO-d_6, δ): 4.04 (s, 3H, CH_3O), 4.15 (s, 3H, CH_3O), 6.20 (s, 2H, OCH_2O), 7.55 (s, 1H, CH=). **^{13}C NMR** (DMSO-d_6, δ): 60.56 (CH_3O), 61.45 (CH_3O), 89.71 (=$\underline{C}(CN)_2$), 104.70 (OCH_2O), 111.78 (CN), 112.09 (C_{Ar}), 112.99 (CN), 132.74 (C_{Ar}), 135.72 (C_{Ar}), 137.18 (C_{Ar}), 141.54 (C_{Ar}), 142.76 (C_{Ar}), 154.88 (=CH). Anal. Calcd for $C_{13}H_9N_3O_6$ (%): C, 51.49; H, 2.99; N, 13.86. Found (%): C, 51.63; H, 2.91; N, 13.77.

Methyl (2*E*)-2-cyano-3-(4,7-dimethoxy-6-nitro-1,3-benzodioxol-5-yl)acrylate (7b)

Yield 0.56 g (83%), m.p. 155–157 °C. **^{1}H NMR** (DMSO-d_6, δ): 3.86 (s, 3H, $COOCH_3$), 3.96 (s, 3H, CH_3O), 3.97 (s, 3H, CH_3O), 6.20 (s, 2H, CH_{Ar}), 8.13 (s, 1H, CH=). **^{13}C NMR** (DMSO-d_6, δ): 53.73 ($COO\underline{C}H_3$), 60.46 (CH_3O), 61.41 (CH_3O), 104.42 (OCH_2O), 111.08 (=$\underline{C}$(CN)$COOCH_3$), 113.85 (C_{Ar}), 113.04 (CN), 132.42 (C_{Ar}), 135.60 (C_{Ar}), 137.28 (C_{Ar}), 141.40 (C_{Ar}), 141.78 (C_{Ar}), 151.97 (=CH), 162.84 ($\underline{C}OOCH_3$). Anal. Calcd for $C_{14}H_{12}N_2O_8$ (%): C, 50.01; H, 3.60; N, 8.33. Found (%): C, 49.83; H, 3.68; N, 8.21.

(2*E*)-2-Cyano-3-(4,7-dimethoxy-6-nitro-1,3-benzodioxol-5-yl)acrylamide (7c)

Yield 0.58 g (90%), m.p. 234–236 °C. **^{1}H NMR** (DMSO-d_6, δ): 3.94 (s, 3H, CH_3O), 3.96 (s, 3H, CH_3O), 6.30 (s, 2H, OCH_2O), 7.89 (s, 2H, NH_2), 8.01 (s, 1H, CH=). **^{13}C NMR** (DMSO-d_6, δ): 60.00 (CH_3O), 61.17 (CH_3O), 103.65 (OCH_2O), 114.80 (=$\underline{C}$(CN)$CONH_2$), 116.50 (C_{Ar}), 116.51 (CN), 133.50 (C_{Ar}), 138.82 (C_{Ar}), 139.18 (C_{Ar}), 139.50 (C_{Ar}), 142.56 (C_{Ar}), 150.23 (=CH), 163.46 ($CONH_2$). Anal. Calcd for $C_{13}H_{11}N_3O_7$. (%): C, 48.61; H, 3.45; N, 13.08. Found (%): C, 48.51; H, 3.53; N, 13.17.

[(6,7-Dimethoxy-4-nitro-1,3-benzodioxol-5-yl)methylene]malononitrile (8a)

Yield 0.50 g (83%), m.p. 144–146 °C. **^{1}H NMR** (DMSO-d_6, δ): 3.72 (s, 3H, CH_3O), 4.13 (s, 3H, CH_3O), 6.37 (s, 2H, OCH_2O), 8.67 (s, 1H, CH=). **^{13}C NMR** (DMSO-d_6, δ): 60.44 (CH_3O), 62.08 (CH_3O), 88.03 (=$\underline{C}(CN)_2$), 105.08 (OCH_2O), 111.97 (CN), 113.24 (CN), 113.28 (C_{Ar}), 124.16 (C_{Ar}), 140.98 (C_{Ar}), 141.20 (C_{Ar}), 143.95 (C_{Ar}), 144.81 (C_{Ar}), 157.99 (CH=). Anal. Calcd for $C_{13}H_9N_3O_6$ (%): C, 51.49; H, 2.99; N, 13.86. Found (%): C, 51.39; H, 3.07; N, 13.93.

Methyl (2*E*)-2-cyano-3-(6,7-dimethoxy-4-nitro-1,3-benzodioxol-5-yl)acrylate (8b)

Yield 0.57 g (85%), m.p. 159–161 °C. **^{1}H NMR** (DMSO-d_6, δ): 3.68 (s, 3H, CH_3O), 3.88 (s, 3H, $COOCH_3$), 4.12 (s, 3H, CH_3O), 6.34 (s, 2H, OCH_2O), 8.49 (s, 1H, CH=). **^{13}C NMR** (DMSO-d_6, δ): 53.55 ($COOCH_3$), 60.38 (CH_3O), 61.84 (CH_3O), 104.77 (OCH_2O), 109.06 (=$\underline{C}$(CN)$COOCH_3$), 114.21 (CN), 114.52 (C_{Ar}), 124.59 (C_{Ar}), 140.12 (C_{Ar}), 141.28 (C_{Ar}), 143.55 (C_{Ar}), 144.38 (C_{Ar}), 151.43 (CH=), 161.47 ($\underline{C}OOCH_3$). Anal. Calcd for $C_{14}H_{12}N_2O_8$. (%): C, 50.01; H, 3.60; N, 8.33. Found (%): C, 50.13; H, 3.55; N, 8.44.

(2*E*)-2-Cyano-3-(6,7-dimethoxy-4-nitro-1,3-benzodioxol-5-yl)acrylamide (8c)

Yield 0.56 g (88%), m.p. 213–215 °C. **^{1}H NMR** (DMSO-d_6, δ): 3.67 (s, 3H, CH_3O), 4.12 (s, 3H, CH_3O), 6.33 (s, 2H, OCH_2O), 7.84 (br.s, 1H, NH), 7.99 (br.s, 1H, NH), 8.22 (s, 1H, CH=). **^{13}C NMR** (DMSO-d_6, δ): 60.32 (CH_3O), 61.64 (CH_3O), 104.60 (OCH_2O), 113.68 (=$\underline{C}$(CN)$CONH_2$), 115.01 (CN), 115.11 (C_{Ar}), 124.57 (C_{Ar}), 139.55 (C_{Ar}), 141.32 (C_{Ar}), 143.33 (C_{Ar}), 144.02 (C_{Ar}),

145.84 (CH=), 161.47 (<u>C</u>ONH$_2$). Anal. Calcd for $C_{13}H_{11}N_3O_7$ (%): C, 48.61; H, 3.45; N, 13.08. Found (%): C, 48.72; H, 3.51; N, 13.01.

Synthesis of Knoevenagel reaction products 9a–c and 10a–c with aldehydes 1 and 2 (general procedure)

A few drops of triethylamine were added to a solution of aldehyde **1** or **2** (0.63 g, 3 mmol) and compound **6a–c** (4 mmol) in methanol (5 ml). The reaction mixture was refluxed for 30 min for malononitrile **6a**, 2 h for methyl cyanoacetate **6b**, and 4 h for cyanoacetamide **6c**, then cooled to -5 °C. The precipitated yellow crystals were filtered off, washed with cold methanol and dried in air.

[4,7-Dimethoxy-1,3-benzodioxol-5-yl)methylene]malononitrile (9a)

Yield 0.72 g (92%), m.p. 170–172 °C. **^{1}H NMR** ($CDCl_3$, δ): 3.92 (s, 3H, CH_3O), 4.07 (s, 3H, CH_3O), 6.13 (s, 2H, OCH_2O), 7.61 (s, 1H, CH_{Ar}), 8.15 (s, 1H, CH=). **^{13}C NMR** ($CDCl_3$, δ): 60.23 (CH_3O), 62.74 (CH_3O), 78.93 (=<u>C</u>$(CN)_2$), 99.71 (C_{Ar}), 102.77 (OCH_2O), 113.21 (CN), 114.72 (C_{Ar}), 118.13(CN), 137.24 (C_{Ar}), 144.29 (C_{Ar}), 145.58 (C_{Ar}), 150.55 (C_{Ar}), 153.15 (CH=). Anal. Calcd for $C_{13}H_{10}N_2O_4$ (%): C, 60.47; H, 3.90; N, 10.85. Found (%): C, 60.56; H, 3.95; N, 10.77.

Methyl (2*E*)-2-cyano-3-(4,7-dimethoxy-1,3-benzodioxol-5-yl)acrylate (9b)

Yield 0.76 g (87%), m.p. 204–205 °C. **^{1}H NMR** (DMSO-d_6, δ): 3.84 (s, 3H, CH_3O), 3.86 (s, 3H, $COOCH_3$), 3.99 (s, 3H, CH_3O), 6.17 (s, 2H, OCH_2O), 7.59 (s, 1H, CH_{Ar}), 8.42 (s, 1H, CH=). **^{13}C NMR** ($CDCl_3$, δ): 53.10 (COO<u>C</u>H_3), 60.17 (CH_3O), 62.63 (CH_3O), 99.87 (=<u>C</u>(CN)$COOCH_3$), 100.67 (C_{Ar}), 102.43 (OCH_2O), 116.02 (CN), 118.48 (C_{Ar}), 137.31 (C_{Ar}), 143.12 (C_{Ar}), 145.37 (C_{Ar}), 148.94 (CH=), 150.56 (C_{Ar}), 163.55 (<u>C</u>$OOCH_3$). Anal. Calcd for $C_{14}H_{13}NO_6$ (%): C, 57.73; H, 4.50; N, 4.81. Found (%): C, 57.64; H, 4.57; N, 4.88.

(2*E*)-2-Cyano-3-(4,7-dimethoxy-1,3-benzodioxol-5-yl)acrylamide (9c)

Yield 0.68 g (82%), m.p. 266–267 °C. **^{1}H NMR** (DMSO-d_6, δ): 3.83 (s, 3H, CH_3O), 3.94 (s, 3H, CH_3O), 6.15 (s, 2H, OCH_2O), 7.48 (s, 1H, CH_{Ar}), 7.65 (br. s, 2H, NH_2), 8.26 (s, 1H, CH=). **^{13}C NMR** (DMSO-d_6, δ): 56.48 (CH_3O), 60.60 (CH_3O), 102.77 (C_{Ar}), 102.92 (OCH_2O), 113.21 (CN), 114.72 (C_{Ar}), 118.13(CN), 137.24 (C_{Ar}), 144.29 (C_{Ar}), 145.58 (C_{Ar}), 150.55 (C_{Ar}), 153.15 (CH=), 162.81 ($CONH_2$). Anal. Calcd for $C_{13}H_{12}N_2O_5$ (%): C, 56.52; H, 4.38; N, 10.14. Found (%): C, 56.70; H, 4.31; N, 10.21.

[(6,7-Dimethoxy-1,3-benzodioxol-5-yl)methylene]malononitrile (10a)

Yield 0.62 g (80%), m.p. 136–138 °C. **^{1}H NMR** ($CDCl_3$, δ): 3.92 (s, 3H, CH_3O), 4.07 (s, 3H, CH_3O), 6.13 (s, 2H, OCH_2O), 7.61 (s, 1H, CH_{Ar}), 8.15 (s, 1H, CH=). **^{13}C NMR** ($CDCl_3$, δ): 60.22 (7-CH_3O), 62.74 (6-CH_3O), 78.94 (=<u>C</u>$(CN)_2$), 99.72 (OCH_2O), 102.76 (C_{Ar}), 113.21 (CN),

114.72 (C_{Ar}), 118.13(CN), 137.23 (C_{Ar}), 144.28 (C_{Ar}), 145.58 (C_{Ar}), 150.55 (C_{Ar}), 153.15 (CH=). Anal. Calcd for $C_{13}H_{10}N_2O_4$ (%): C, 60.47; H, 3.90; N, 10.85. Found (%): C, 60.34; H, 3.97; N, 10.94.

Methyl (2*E*)-2-cyano-3-(6,7-dimethoxy-1,3-benzodioxol-5-yl)acrylate (10b)

Yield 0.78 g (89%), m.p. 132–134 °C. **^{1}H NMR** (DMSO-d_6, δ): 3.83 (s, 3H, CH_3O), 3.85 (s, 3H, $COOCH_3$), 3.98 (s, 3H, CH_3O), 6.18 (s, 2H, OCH_2O), 7.43 (s, 1H, CH_{Ar}), 8.40 (s, 1H, CH=). **^{13}C NMR** ($CDCl_3$, δ): 53.10 ($COO\underline{C}H_3$), 60.17 (CH_3O), 62.58 (CH_3O), 99.83 (=$\underline{C}$(CN)$COOCH_3$), 100.62 (OCH_2O), 102.45 (C_{Ar}), 116.02 (CN), 118.46 (C_{Ar}), 118.13(CN), 137.23 (C_{Ar}), 144.28 (C_{Ar}), 145.37 (C_{Ar}), 148.92 (CH=), 150.56 (C_{Ar}), 163.55 ($\underline{C}OOCH_3$). Anal. Calcd for $C_{14}H_{13}NO_6$ (%): C, 57.73; H, 4.50; N, 4.81. Found (%): C, 57.84; H, 4.43; N, 4.96.

(2*E*)-2-Cyano-3-(6,7-dimethoxy-1,3-benzodioxol-5-yl)acrylamide (10c)

Yield 0.72 g (87%), m.p. 232–234 °C. **^{1}H NMR** (DMSO-d_6, δ): 3.79 (s, 3H, CH_3O), 3.97 (s, 3H, CH_3O), 6.14 (s, 2H, OCH_2O), 7.35 (s, 1H, CH_{Ar}), 7.67 (br. s, 1H, NH), 7.83 (br. s, 1H, NH), 8.22 (s, 1H, CH=). **^{13}C NMR** (DMSO-d_6, δ): 59.69 (CH_3O), 62.10 (CH_3O), 102.82 (OCH_2O), 103.16 (=$\underline{C}$(CN)$CONH_2$), 107.07 (C_{Ar}), 117.16 (C_{Ar}), 117.38 (CN), 140.09 (C_{Ar}), 142.84 (C_{Ar}), 145.27 (C_{Ar}), 147.99 (C_{Ar}), 144.56 (=CH), 162.64 ($\underline{C}ONH_2$). Anal. Calcd for $C_{13}H_{12}N_2O_5$ (%): C, 56.52; H, 4.38; N, 10.14. Found (%): C, 56.61; H, 4.44; N, 10.05.

Synthesis of Knoevenagel reaction products 9d and 10d of aldehydes 1 and 2 with dimethyl malonate (general procedure)

To a solution of aldehyde **1** or **2** (0.63 g, 3 mmol) and dimethyl malonate (0.52 g, 4 mmol) in toluene (10 ml) was added piperidine-AcOH (0.05 g). The reaction mixture was refluxed for 3 h with off-distillation of water, then the reaction mixture was cooled to room temperature, poured into a separating funnel, washed with water (3×5 ml), dried over $MgSO_4$, filtered, the solvent was distilled off in a vacuum. The residue was dissolved in hot methanol and cooled to -10 °C. The precipitated crystals were filtered off, washed with cold methanol, and dried on air.

Dimethyl [(4,7-dimethoxy-1,3-benzodioxol-5-yl)methylene]malonate (9d)

Yield 0.72 g (74%), m.p. 120–122 °C. **^{1}H NMR** ($CDCl_3$, δ): 3.85 (s, 6H, 2×$COOCH_3$), 3.86 (s, 3H, CH_3O), 3.97 (s, 3H, CH_3O), 6.05 (s, 2H, OCH_2O), 6.65 (s, 1H, CH_{Ar}), 7.97 (s, 1H, CH=). **^{13}C NMR** ($CDCl_3$, δ): 52.41; 52.55 ($COO\underline{C}H_3$), 60.11 (CH_3O), 62.27 (CH_3O), 100.42 (C_{Ar}), 102.26 (OCH_2O), 119.54 (C_{Ar}), 124.44 (=$\underline{C}$($COOCH_3$)$_2$), 137.42 (C_{Ar}), 143.81 (C_{Ar}), 145.34 (C_{Ar}), 138.23 (=CH), 150.48 (C_{Ar}), 164.83; 167.35 ($\underline{C}OOCH_3$). Anal. Calcd for $C_{15}H_{16}O_8$ (%): C, 55.56; H, 4.97. Found (%): C, 55.41; H, 5.04.

Dimethyl [(6,7-dimethoxy-1,3-benzodioxol-5-yl)methylene]malonate (10d)

Yield 0.80 g (82%), m.p. 95–97 °C. **^{1}H NMR** ($CDCl_3$, δ): 3.82 (s, 3H, CH_3O), 3.85 (s, 6H, 2×$COOCH_3$), 4.03 (s, 3H, CH_3O), 5.98 (s, 2H, OCH_2O), 6.55 (s, 1H, CH_{Ar}), 7.98 (s, 1H, CH=). **^{13}C NMR** ($CDCl_3$, δ): 52.46; 52.53 ($COO\underline{C}H_3$), 60.09 (CH_3O), 62.15 (CH_3O), 101.86 (OCH_2O), 100.75 (C_{Ar}), 119.66 (C_{Ar}), 124.31 (=$\underline{C}(COOCH_3)_2$), 137.51 (C_{Ar}), 137.88 (=CH), 140.33 (C_{Ar}), 145.07 (C_{Ar}), 147.96 (C_{Ar}), 164,78; 167.25 ($\underline{C}OOCH_3$). Anal. Calcd for $C_{15}H_{16}O_8$ (%): C, 55.56; H, 4.97. Found (%): C, 55.64; H, 4.92.

Nitration of the Knoevenagel reaction products 9a–d and 10a–d

To a solution of compound **9a–d** or **10a–d** (3 mmol) in CH_2Cl_2 (10 ml) was added dropwise at 0–2°C a solution of concentrated nitric acid (0.5 g) and acetic anhydride (0.5 ml) in CH_2Cl_2 (10 ml). Then the reaction mass was stirred for 20 min at 0–2 °C and poured into water (20 ml). The organic layer was separated, washed with $NaHCO_3$ solution, and dried over $MgSO_4$. After evaporation of the solvent, the substance was crystallized from methanol. Thus were obtained:

Compound **7a.** Yield 0.84 g (92%)

Compound **7b.** Yield 0.73 g (73%)

Compound **7c.** Yield 0.73 g (76%)

Compound **8a.** Yield 0.65 g (71%)

Compound **8b.** Yield 0.72 g (72%)

The nitration products of compounds **10c** and **10d** appeared as yellow oils and were mixtures with less than 10% (NMR) of the target substances.

Dimethyl [(4,7-dimethoxy-6-nitro-1,3-benzodioxol-5-yl)methylene]malonate (7d)

Yield 0.84 g (76%), m.p. 120–122 °C. **^{1}H NMR** ($CDCl_3$, δ): 3.75 (s, 3H, $COOCH_3$), 3.85 (s, 3H, $COOCH_3$), 3.87 (s, 3H, CH_3O), 4.01 (s, 3H, CH_3O), 6.12 (s, 2H, OCH_2O), 7.49 (s, 1H, CH=). **^{13}C NMR** ($CDCl_3$, δ): 54.53; 54.71 ($COO\underline{C}H_3$), 60.67 (CH_3O), 61.27 (CH_3O), 103.45 (OCH_2O), 112.02 (C_{Ar}), 126.47 (=$\underline{C}(COOCH_3)_2$), 133.66 (C_{Ar}), 136.59 (C_{Ar}), 138.69 (C_{Ar}), 139.64 (C_{Ar}), 142.07 (C_{Ar}), 144.39 (=CH), 159.74; 160.00 ($\underline{C}OOCH_3$). Anal. Calcd for $C_{15}H_{15}NO_{10}$ (%): C, 48.79; H, 4.09; N, 3.79. Found (%): C, 48.65; H, 4.16; N, 3.68.

Synthesis of quinoline-3-carboxylic acid derivatives 11-15

6-Amino-4,9-dimethoxy[1,3]dioxolo[4,5-*g*]quinoline-7-carbonitrile (11)

To a suspension of nitro compound **7a** (0.46 g, 1.5 mmol) in acetic acid (5 ml) was added zinc dust (0.40 g, 6 mmol) with stirring. The reaction mixture was stirred at 50-70 °C for 30 min and then filtered hot. Then 15% Na_2CO_3 solution was added to the filtrate until pH = 8–9. After some time, the precipitate was filtered off and dried. Then ethyl acetate (10 ml) was added to the

precipitate, heated to boiling and filtered. This operation was repeated additional two times. The combined extract was evaporated and the product was crystallized from DMF-methanol. Yield 0.22 g (48%), m.p. 239–241 °C. **^{1}H NMR** (DMSO-d_6, δ): 3.91 (s, 3H, CH_3O), 4.01 (s, 3H, CH_3O), 6.11 (s, 2H, OCH_2O), 6.80 (s, 2H, NH_2), 8.38 (s, 1H, CH_{Ar}). **^{13}C NMR** (DMSO-d_6, δ): 60.18 (CH_3O), 60,52 (CH_3O), 90.84 (C_{Ar}), 102.29 (OCH_2O), 111.67 (C_{Ar}), 116.98 (CN), 130.19 (C_{Ar}), 131.11 (C_{Ar}), 131.94 (C_{Ar}), 138.11 (C_{Ar}), 141.40 (C_{Ar}), 144,17 (C_{Ar}), 155.18 (C_{Ar}). Anal. Calcd for $C_{13}H_{11}N_3O_4$ (%): C, 57.14; H, 4.06; N, 15.38. Found (%): C, 57.27; H, 4.13; N, 15.22.

Synthesis of compounds 12, 13, and 14

To a suspension of nitro compound **7b** or **7c** (1.5 mmol) in acetic acid (5 ml), zinc dust (0.40 g, 6 mmol) was added with stirring. The reaction mass was stirred at 50–70°C for 30 min and then poured into 10% hydrochloric acid solution (15 ml), then stirred for 30 min. The precipitate that formed, which was mainly compound **12**, was filtered off, washed with water, dried, and crystallized from a DMF-methanol mixture. Then 15% Na_2CO_3 solution was added to the filtrate until pH = 8–9, and then 2-aminoquinolines **13** and **14** were isolated as described for compound **11**.

4,9-Dimethoxy-6-oxo-5,6-dihydro[1,3]dioxolo[4,5-*g*]quinoline-7-carbonitrile (12)

Yield 0.06 g (13%) from **7b** and 0.09 g (20%) from **7c**, m.p. 312–314 °C (decomp.) (DMF–MeOH). **^{1}H NMR** (DMSO-d_6, δ): 3.87 (s, 3H, CH_3O), 4.04 (s, 3H, CH_3O), 6.18 (s, 2H, OCH_2O), 8.53 (s, 1H, CH_{Ar}), 11.86 (br.s., 1H, NH). **^{13}C NMR** (DMSO-d_6, δ): 60.18 (CH_3O), 60.52 (CH_3O), 102.29 (OCH_2O), 90.84 (C_{Ar}), 111.67 (C_{Ar}), 116.98 (CN), 130.19 (C_{Ar}), 131.11 (C_{Ar}), 131.94 (C_{Ar}), 138.11 (C_{Ar}), 141.40 (C_{Ar}), 144.17 (C_{Ar}), 155.17 (C_{Ar}). Anal. Calcd for $C_{13}H_{10}N_2O_5$ (%): C, 56.94; H, 3.68; N, 10.22. Found (%): C, 56.82; H, 3.73; N, 10.12.

Methyl 6-amino-4,9-dimethoxy[1,3]dioxolo[4,5-*g*]quinoline-7-carboxylate (13)

Yield 0.19 g (40%), m.p. 253–255 °C (decomp.) (DMF–MeOH). **^{1}H NMR** (DMSO-d_6, δ): 3.88 (s, 3H, $COOCH_3$), 3.94 (s, 3H, CH_3O), 4.04 (s, 3H, CH_3O), 6.11 (s, 2H, OCH_2O), 7.15 (s, 2H, NH_2), 8.67 (s, 1H, CH_{Ar}). **^{13}C NMR** (DMSO-d_6, δ): 52.17 ($COO\underline{C}H_3$), 59.93 (CH_3O), 60.47 (CH_3O), 102.02 (OCH_2O), 105.74 (C_{Ar}), 111.88 (C_{Ar}), 135.30 (C_{Ar}), 129.83 (C_{Ar}), 131.09 (C_{Ar}), 131.57 (C_{Ar}), 142.37 (C_{Ar}), 144.01 (C_{Ar}), 155.45 (C_{Ar}), 166.69 ($\underline{C}OOCH_3$). Anal. Calcd for $C_{14}H_{14}N_2O_6$ (%): C, 54.90; H, 4.61; N, 9.15. Found (%): C, 54.97; H, 4.53; N, 9.22.

6-Amino-4,9-dimethoxy[1,3]dioxolo[4,5-g]quinoline-7-carboxamide (14)

Yield, 0.16 g (33%), m.p. 251–253 °C (DMF–MeOH). **^{1}H NMR** (DMSO-d_6, δ): 3.91 (s, 3H, CH_3O), 4.02 (s, 3H, CH_3O), 6.08 (s, 2H, OCH_2O), 7.17 (br.s, 2H, NH_2), 7.42 (br. s, 1H, NH), 8.23 (br. s, 1H, NH), 8.47 (s, 1H, CH_{Ar}). Anal. Calcd for $C_{13}H_{13}N_3O_5$ (%): C, 53.61; H, 4.50; N,

14.43. Found (%): C, 53.52; H, 4.57; N, 14.51. **^{13}C NMR** was not recorded due to the low solubility of **14**.

Methyl dimethoxy-6-oxo-5,6-dihydro[1,3]dioxolo[4,5-*g*]quinoline-7-carboxylate (15)

To a solution of compound **7d** (0.37 g, 1 mmol) in acetic acid (4 ml), zinc dust (0.4 g) was added with stirring. The reaction mixture was stirred at 50–70°C for 25 min and then poured into water (20 ml). The precipitate formed was filtered off, washed with water and dried. Then it was dissolved in hot DMF and filtered. Methanol was added to the filtrate and cooled to 0°C. The precipitate formed was filtered off and washed with methanol. Yield 0.25 g (81%), m.p. 282–284 °C (decomp., DMF–MeOH). **^{1}H NMR** (DMSO-d_6, δ): 3.81 (s, 3H, $COOCH_3$), 3.92 (s, 3H, CH_3O), 4.06 (s, 3H, CH_3O), 6.14 (s, 2H, OCH_2O), 8.46 (s, 1H, CH_{Ar}), 10.87 (br.s., 1H, NH). Anal. Calcd for $C_{14}H_{13}NO_7$ (%): C, 54.73; H, 4.26; N, 4.56. Found (%): C, 54.88; H, 4.21; N, 4.48. **^{13}C NMR** was not recorded due to the low solubility of **15**.

Synthesis of quinolines 16a–c

These compounds were obtained according to the procedure described for the synthesis of aminoquinoline **11** (see above).

8-Amino-4,5-dimethoxy[1,3]dioxolo[4,5-*h*]quinoline-7-carbonitrile (16a)

Yield 0.26 g (56%), m.p. 248–250 °C decomp. (DMF-MeOH). **^{1}H NMR** (DMSO-d_6, δ): 3.91 (s, 3 H, 4-CH_3O), 3.94 (s, 3H, CH_3O), 6.18 (s, 2 H, OCH_2O), 6.80 (s, 2H, NH_2), 8.49 (s,, 1H, CH_{Ar}). **^{13}C NMR** (DMSO-d_6, δ): 60.62 (CH_3O), 62.29 (CH_3O), 92.78 (C_{Ar}), 102.51 (OCH_2O), 111.91 (C_{Ar}), 116.82 (CN), 130.68 (C_{Ar}), 133.46 (C_{Ar}), 135.43 (C_{Ar}), 140.22 (C_{Ar}), 142.14 (C_{Ar}), 143.26 (C_{Ar}), 155.18 (C_{Ar}). Anal. Calcd for $C_{13}H_{11}N_3O_4$ (%): C, 57.14; H, 4.06; N, 15.38. Found (%): C, 57.05; H, 4.01; N, 15.50.

Methyl amino-4,5-dimethoxy[1,3]dioxolo[4,5-*h*]quinoline-7-carboxylate (16b)

Yield 0.17 g (34%), m.p. 243–246 °C (decomp.) (DMF – MeOH). **^{1}H NMR** (DMSO-d_6, δ): 3.89 (s, 3H, $COOCH_3$), 3.93 (s, 6H, 2×CH_3O), 6.17 (s, 2H, OCH_2O), 7.19 (s, 2H, NH_2), 8.71 (s, 1H, CH_{Ar}). **^{13}C NMR** (DMSO-d_6, δ): 52.36 (COO<u>C</u>H_3), 60.64 (CH_3O), 62.11 (CH_3O), 102.35 (OCH_2O), 107.72 (C_{Ar}), 109.48 (C_{Ar}), 131.76 (C_{Ar}), 133.85 (C_{Ar}), 134.80 (C_{Ar}), 136.97 (C_{Ar}), 142.36 (C_{Ar}), 143.85 (C_{Ar}), 155.81 (C_{Ar}), 166.62 (<u>C</u>$OOCH_3$). Anal. Calcd for $C_{14}H_{14}N_2O_6$ (%): C, 54.90; H, 4.61; N, 9.15. Found (%): C, 54.78; H, 4.68; N, 9.09.

8-Amino-4,5-dimethoxy[1,3]dioxolo[4,5-*h*]quinoline-7-carboxamide (16c)

Yield 0.23 g (48%), m.p. 214–216 °C (EtOAc). **^{1}H NMR** (DMSO-d_6, δ): 3.89 (s, 3H, CH_3O), 3.92 (s, 3H, CH_3O), 6.14 (s, 2H, OCH_2O), 7.24 (br. s, 2H, NH_2), 7.49 (br. s, 1H, NH), 8.32 (br. s, 1H,

NH), 8.46 (s, 1H, CH_{Ar}). Anal. Calcd for $C_{13}H_{13}N_3O_5$ (%): C, 53.61; H, 4.50; N, 14.43. Found (%): C, 53.74; H, 4.42; N, 14.36. **^{13}C NMR** was not recorded due to the low solubility of **16c**

The action of diphenylphosphoryl azide on acids **17a-d** in the presence of triethylamine under mild conditions at 0°C gave acyl azides. Their rearrangement in a neutral medium often leads to the formation up to 30% of symmetrical diarylureas as by-products, which reduce the yield of anilines. We have found that this rearrangement proceeded efficiently in an acidic medium and led to isocyanates, which, under the reaction conditions, rapidly hydrolyzed and give aniline hydrochlorides. The latter can be acetylated without isolation with acetic anhydride in the presence of triethylamine, resulting in the formation of the corresponding acetanilides **18a–d**

Synthesis of acetanilides 18a–d starting from carboxylic acids 17a–d (general procedure)

Triethylamine (30 mmol, 3.0 g, 4.2 ml) was added to a mixture of substituted carboxylic acids **17a–d** (20 mmol) and acetonitrile (40 ml). Diphenylphosphoryl azide (21 mmol, 5.78 g, 4.5 ml) was added dropwise to the resulting solution while cooling in an ice bath. The mixture was stirred for 1 h in an ice bath until complete disappearance of the starting material (TLC monitoring). Then, while cooling (2–3 °C), a solution of hydrochloric acid (60 mmol, 11M, 5.5 ml) was quickly added in one portion, thereafter the reaction mixture was heated for 1 h at 80 °C. After heating was completed, the mixture was cooled, a solution of sodium hydroxide (180 mmol, 7.2 g) in water (40 ml) was added, then acetic anhydride (100 mmol, 10.2 g, 9.5 ml) was added dropwise. The mixture was stirred at room temperature (20 °C) until the initial product completely disappeared (TLC control), then the solvents were evaporated, and the residue was dissolved in dichloromethane (50 ml), washed 3 times with a solution of sodium hydroxide (5 mmol, 0.20 g) in water (20 ml), dried over $MgSO_4$, and evaporated *in vacuo*. The product was isolated by column chromatography, eluting with ethyl acetate-cyclohexane or dichloromethane-methanol.

***N*-(4,7-Dimethoxy-1,3-benzodioxol-5-yl)acetamide (18a)**

Yield 4.31 g (90%), m.p. 128–130 °C **^{1}H NMR** (DMSO-d_6, δ) 2.05 (s, 3H, $COCH_3$) 3.74 (s, 3H, CH_3O) 3.80 (s, 3H, CH_3O) 5.98 (s, 2H, OCH_2O) 7.15 (s, 1H, CH_{Ar}) 9.15 (br. s., 1 H, NH). **^{13}C NMR** (126 MHz, DMSO-d_6, δ): 23.64 ($\underline{CH_3}CO$), 56.56 (CH_3O), 60.04 (CH_3O), 101.55 (OCH_2O), 102.82 (C_{Ar}), 124.91 (C_{Ar}), 130.27 (C_{Ar}), 132.63 (C_{Ar}), 137.87 (C_{Ar}), 138.46 (C_{Ar}), 168.60 ($CH_3\underline{C}O$). **MS** $C_{11}H_{13}NO_5$ M.w.239.23: 239, 208, 197, 182, 166, 152, 137, 124, 110, 94. Anal. Calcd for $C_{11}H_{13}NO_5$ (%): C, 55.23; H, 5.48; N, 5.86. Found (%):.C, 55.37; H, 5.40; N, 5.95.

***N*-(6,7-Dimethoxy-1,3-benzodioxol-5-yl)acetamide (18b)**

Yield 3.78 g (79%), m.p. 140–141 °C **^{1}H NMR** (DMSO-d_6, *δ*) 2.06 (s, 3H, $COCH_3$) 3.67 (s, 3H, CH_3O) 3.93 (s, 3H, CH_3O) 5.94 (s, 2H, OCH_2O) 7.22 (s, 1H, CH_{Ar}) 9.11 (br. s., 1 H, NH). **^{13}C NMR** (126 MHz, DMSO-d_6, *δ*) 23.73 ($\underline{C}H_3CO$), 59.83 (CH_3O), 60.97 (CH_3O), 97.54 (OCH_2O), 101.20 (C_{Ar}), 125.60 (C_{Ar}), 133.51 (C_{Ar}), 137.02 (C_{Ar}), 137.14 (C_{Ar}), 143.54 (C_{Ar}), 168.73 ($CH_3\underline{C}O$). **MS** $C_{11}H_{13}NO_5$ M.w. 239.23: 239, 224, 208, 197, 182, 167, 138, 124, 110, 93. Anal. Calcd for $C_{11}H_{13}NO_5$. (%): C, 55.23; H, 5.48; N, 5.86. Found (%): C, 55.08; H, 5.30; N, 5.71.

***N*-(2,3,4,5-Tetramethoxyphenyl)acetamide (18c)**

Yield 3.83 g (75%), m.p. 75–76 °C. **^{1}H NMR** (DMSO-d_6, *δ*) 2.09 (s, 3H, $COCH_3$) 3.71 (s, 6H, 2×CH_3O), 3.72 (s, 3H, CH_3O), 3.82 (s, 3H, CH_3O), 7.46 (s, 1H, CH_{Ar}), 9.19 (br. s., 1 H, NH). **^{13}C NMR** (126 MHz, DMSO-d_6, *δ*) 23.83 ($\underline{C}H_3CO$), 55.95 (CH_3O), 60.72 (CH_3O), 60.95 (CH_3O), 101.87 (C_{Ar}), 127.30 (C_{Ar}), 137.77 (C_{Ar}), 138.56 (C_{Ar}), 146.48 (C_{Ar}), 148.47 (C_{Ar}), 168.74 ($CH_3\underline{C}O$) **MS** $C_{12}H_{17}NO_5$ M.w. 255.11: 255, 230, 225, 213, 208, 198, 183, 170, 155, 140, 127, 112, 99. Anal. Calcd for $C_{12}H_{17}NO_5$ (%): C, 56.46; H, 6.71; N, 5.49 Found (%):C, 56.33; H, 6.81; N, 5.30.

***N*-(2,4,5-Trimethoxyphenyl)acetamide (18d)**

Yield 3.83 g (85%), m.p. 106–108 °C. **^{1}H NMR** (DMSO-d_6, *δ*) 2.05 (s, 3H, COMe) 3.67 (s, 3H, CH_3O) 3.78 (s, 3H, CH_3O) 3.80 (s, 3H, CH_3O) 6.74 (s, 1H, CH_{Ar}) 7.54 (s, 1H, CH_{Ar}) 9.05 (s, 1H, NH) **^{13}C NMR** (126 MHz, DMSO-d_6, *δ*): 23.65 ($\underline{C}H_3CO$), 56.05 (CH_3O), 56.28 (CH_3O), 56.42 (CH_3O), 98.78 (C_{Ar}), 108.98 (C_{Ar}), 119.86 (C_{Ar}), 141.83 (C_{Ar}), 144.46 (C_{Ar}), 145.63 (C_{Ar}), 168.22 ($CH_3\underline{C}O$) **MS** $C_{11}H_{15}NO_4$ M.w. 225.24: 225, 210, 194, 183, 168, 154, 140, 125, 108, 94. Anal. Calcd for $C_{11}H_{15}NO_4$ (%): C, 58.66; H, 6.71; N, 6.22. Found (%): C, 58.74; H, 6.87; N, 6.30.

Synthesis of 2-chloroquinoline-3-carbaldehyde derivatives 19a-d (general procedure)

According to the general procedure [S2], acetanilide **18a–c** (1.3 mmol) was mixed with DMF (3.9 mmol, 0.3 g, 0.3 ml), then $POCl_3$ (9.1 mmol, 1.4 g, 0.9 ml) was added dropwise while cooling in an ice bath. The reaction mixture was stirred in an ice bath for 5 min, then heated for 1–2 hours at 90°C. Upon completion of heating, the reaction mixture was cooled to room temperature, poured onto ice (25 g), and $NaHCO_3$ (4.6 g, 55 mmol) and dichloromethane (20 ml) were added. The organic layer was separated, the aqueous layer was extracted with dichloromethane (3×20 ml), the organic phases were combined, dried over $MgSO_4$, and evaporated *in vacuo*. The products were purified by column chromatography on 30 g of silica gel, eluting with dichloromethane-methanol.

6-Chloro-4,9-dimethoxy[1,3]dioxolo[4,5-*g*]quinoline-7-carbaldehyde (19a)

Yield 0.250 g (65%), m.p. 238–240 °C **^{1}H NMR** (DMSO-d_6,*δ*) 4.01 (s, 3H, CH_3O) 4.11 (s, 3H, CH_3O) 6.28 (s, 2H, OCH_2O) 8.64 (s, 1H, CH_{Ar}) 10.29 (s, 1H, CHO). **^{13}C NMR** (126 MHz, DMSO-d_6,*δ*) 60.11 (CH_3O), 60.68 (CH_3O), 103.13 (OCH_2O), 117.81 (C_{Ar}), 123.66 (C_{Ar}), 131.24 (C_{Ar}), 131.44 (C_{Ar}), 134.02 (C_{Ar}), 136.03 (C_{Ar}), 140.38 (C_{Ar}), 144.78 (C_{Ar}), 146.67 (C_{Ar}), 188.71 (CHO). **HRMS** $C_{13}H_{10}ClNO_5$ M+H. Anal. Calcd for 296.0311, Found 296.0320.

7-Chloro-4,5-dimethoxy[1,3]dioxolo[4,5-*f*]quinoline-8-carbaldehyde (19b)

Yield 0.192 g (50%), m.p. 154–156 °C **^{1}H NMR** (DMSO-d_6, *δ*) 3.92 (s, 3H, CH_3O) 4.16 (s, 3H, CH_3O) 6.34 (s, 2H, OCH_2O) 8.55 (s, 1H, CH_{Ar}) 10.29 (s, 1H, CHO). **^{13}C NMR** (126 MHz, DMSO-d_6, *δ*) 60.48 (CH_3O), 62.07 (CH_3O), 103.50 (OCH_2O), 107.68 (C_{Ar}), 124.27 (C_{Ar}), 133.68 (C_{Ar}), 136.96 (C_{Ar}), 137.36 (C_{Ar}), 139.48 (C_{Ar}), 139.70 (C_{Ar}), 143.36 (C_{Ar}), 146.35 (C_{Ar}), 188.77 (CHO). **MS** $C_{13}H_{10}ClNO_5$ M.w. 295.68: 295, 280, 266, 250, 236, 222, 216, 207, 194, 188, 179, 167, 154.. Anal. Calcd for $C_{13}H_{10}ClNO_5$ (%): C, 52.81; H, 3.41; N, 4.74. Found (%):C, 52.90; H, 3.50; N, 4.91.

2-Chloro-5,6,7,8-tetramethoxyquinoline-3-carbaldehyde (19c)

Yield 0.182 g (45%), m.p. 84–87 °C. **^{1}H NMR** (DMSO-d_6, *δ*) 3.95 (s, 3H, CH_3O), 3.99 (s, 3H, CH_3O), 4.01 (s, 3H, CH_3O), 4.07 (s, 3H, CH_3O), 8.78 (s, 1H, CH_{Ar}), 10.32 (s, 1H, CHO). **^{13}C NMR** (126 MHz, DMSO-d_6, *δ*) 61.38 (CH_3O), 61.59 (CH_3O), 62.05 (CH_3O), 62.13 (CH_3O), 118.39 (C_{Ar}), 124.86 (C_{Ar}), 135.64 (C_{Ar}), 140.18 (C_{Ar}), 142.64 (C_{Ar}), 144.12 (C_{Ar}), 144.73 (C_{Ar}), 147.77 (C_{Ar}), 151.86 (C_{Ar}), 189.16 (CHO). **MS** $C_{14}H_{14}ClNO_5$ M.w. 311.72: 311, 296, 282, 268, 253, 238, 224, 210, 194, 182, 167, 160, 154. Anal. Calcd for $C_{14}H_{14}ClNO_5$ (%): C, 53.94; H, 4.53; N, 4.49. Found (%): C, 53.86; H, 4.65; N, 4.40.

2-Chloro-5,6,8-trimethoxyquinoline-3-carbaldehyde (19d)

Yield 0.007 g (2%), m.p. 158–159 °C. **^{1}H NMR** (DMSO-d_6, *δ*) 3.89 (s, 3H, CH_3O), 4.03 (s, 3H, CH_3O), 4.04 (s, 3H, CH_3O), 7.33 (s, 1H, CH_{Ar}), 8.75 (s, 1H, CH_{Ar}), 10.35 (s, 1H, CHO). **^{13}C NMR** (126 MHz, DMSO-d_6, *δ*): 56.34 (CH_3O), 56.91 (CH_3O), 61.33 (CH_3O), 103.42 (C_{Ar}), 122.25 (C_{Ar}), 126.52 (C_{Ar}), 134.21 (C_{Ar}), 135.08 (C_{Ar}), 135.51 (C_{Ar}), 145.13 (C_{Ar}), 149.75 (C_{Ar}), 151.31 (C_{Ar}), 189.45 (CHO). **MS** $C_{13}H_{12}ClNO_4$ M.w. 281.69: 281, 266, 252, 238, 223, 208, 195, 167, 136, 129, 123, 116, 100. Anal. Calcd for $C_{13}H_{12}ClNO_4$ (%): C, 55.43; H, 4.29; N, 4.97. Found (%): C, 55.51; H, 4.14; N, 4.80.

Synthesis of methyl chloroquinolinecarboxylates 20a-c (general procedure)

Crystalline iodine (6 mmol, 1.5 g) was added to a solution of corresponding aldehyde **19a–c** (1.5 mmol), K_2CO_3 (9 mmol, 1.2 g) in MeOH (9 ml). The reaction mixture was stirred at room temperature for 72 h. After stirring, $Na_2S_2O_3{\cdot}5H_2O$ (7 mmol, 1.7 g) in water (20 ml) and

dichloromethane (20 ml) were added to the reaction mixture. The organic layer was separated, the aqueous layer was extracted with dichloromethane (3×20 ml), the organic phases were combined, dried over $MgSO_4$, and evaporated *in vacuo*. Products **20a-c** were purified by recrystallization from methanol.

Methyl 6-chloro-4,9-dimethoxy[1,3]dioxolo[4,5-*g*]quinoline-7-carboxylate (20a)

Yield 0.420 g (86%), m.p. 179–180 °C **^{1}H NMR** (DMSO-d_6, δ) 3.91 (s, 3H, $COOCH_3$), 4.01 (s, 3H, CH_3O), 4.08 (s, 3H, CH_3O), 6.25 (s, 2H, OCH_2O), 8.68 (s, 1H, CH_{Ar}). **^{13}C NMR** (126 MHz, DMSO-d_6, δ) 52.73 ($COO\underline{C}H_3$), 60.19 (CH_3O), 60.80 (CH_3O), 103.19 (OCH_2O), 117.10 (C_{Ar}), 120.64 (C_{Ar}), 130.68 (C_{Ar}), 131.01 (C_{Ar}), 135.21 (C_{Ar}), 136.03 (C_{Ar}), 139.38 (C_{Ar}), 143.88 (C_{Ar}), 144.23 (C_{Ar}), 164.20 ($\underline{C}OOCH_3$). **MS** $C_{14}H_{12}ClNO_6$ M.w.325.70: 325, 310, 296, 282, 264, 252, 237, 230, 222, 206, 184, 150, 138. Anal. Calcd for $C_{14}H_{12}ClNO_6$ (%): C, 51.63; H, 3.71; N, 4.30. Found (%): C, 51.78; H, 3.63; N, 4.50.

Methyl 7-chloro-4,5-dimethoxy [1,3]dioxolo[4,5-*f*]quinoline-8-carboxylate (20b)

Yield 0.425 g (87%), m.p. 143–144 °C. **^{1}H NMR** (DMSO-d_6, δ) 3.90 (s, 3H, $COOCH_3$), 3.90 (c 3H, CH_3O), 4.12 (s, 3H, CH_3O), 6.30 (s, 2H, OCH_2O), 8.51 (s, 1H, CH_{Ar}) **^{13}C NMR** (126 MHz, DMSO-d_6, δ) 52.88 ($COO\underline{C}H_3$), 60.48 (CH_3O), 62.10 (CH_3O), 103.36 (OCH_2O), 107.24 (C_{Ar}), 121.70 (C_{Ar}), 134.08 (C_{Ar}), 136.75 (C_{Ar}), 137.13 (C_{Ar}), 138.41 (C_{Ar}), 139.76 (C_{Ar}), 142.50 (C_{Ar}), 143.95 (C_{Ar}), 164.10 ($\underline{C}OOCH_3$). **MS** $C_{14}H_{12}ClNO_6$ M.w.325.70: 325, 310, 296, 280, 266, 252, 237, 230, 206, 184, 169, 138, 124. Anal. Calcd for $C_{14}H_{12}ClNO_6$ (%): C, 51.63; H, 3.71; N, 4.30. Found (%): C, 51.81; H, 3.62; N, 4.21.

Methyl 2-chloro-5,6,7,8-tetramethoxyquinoline-3-carboxylate (20c)

Yield 0.502 g (98%), m.p. 54–57 °C. **^{1}H NMR** (DMSO-d_6, δ) 3.93 (s, 3H, $COOCH_3$), 3.95 (s, 3H, CH_3O), 3.99 (s, 3H, CH_3O), 4.00 (s, 3H, CH_3O), 4.04 (s, 3H, CH_3O), 8.80 (s, 1H, CH_{Ar}). **^{13}C NMR** (126 MHz, DMSO-d_6, δ) 52.89 ($COO\underline{C}H_3$), 61.32 (CH_3O), 61.52 (CH_3O), 61.94 (CH_3O), 62.05 (CH_3O), 117.77 (C_{Ar}), 122.36 (C_{Ar}), 136.13 (C_{Ar}), 139.19 (C_{Ar}), 142.64 (C_{Ar}), 143.19 (C_{Ar}), 144.82 (C_{Ar}), 145.07 (C_{Ar}), 150.85 (C_{Ar}), 164.34 ($\underline{C}OOCH_3$). **MS** $C_{15}H_{16}ClNO_6$ M.w. 341.74: 341, 326, 312, 298, 283, 268, 254, 240, 224, 212, 206, 197, 184. Anal. Calcd for $C_{15}H_{16}ClNO_6$. (%): C, 52.72; H, 4.72; N, 4.10. Found (%): C, 52.90; H, 4.88; N, 4.33.

Synthesis of chloro(hydroxyiminomethyl)quinoline derivatives 21a-c (general procedure)

Triethylamine (0.65 mmol, 0.066 g) was added to a mixture of 2-chloroquinoline-3-carbaldehyde **19a–c** (0.5 mmol) and $NH_2OH{\cdot}HCl$ (0.65 mmol, 0.045 g) in ethanol (5 ml) at 20 °C. The reaction mixture was stirred at room temperature until complete disappearance of the starting

mixture (TLC monitoring). After stirring, the reaction mixture was diluted with water (5 ml), the precipitate was filtered off, washed with water, and dried in air.

6-Chloro-4,9-dimethoxy[1,3]dioxolo[4,5-*g*]quinoline-7-carbaldehyde oxime (21a)

Yield 0.143 g (92%), m.p. 202–203 °C (decomp.). **^{1}H NMR** (DMSO-d_6, δ) 4.00 (s, 3H, CH_3O) 4.06 (s, 3H, CH_3O) 6.22 (s, 2H, OCH_2O) 8.33 (s, 1H, CH_{Ar}) 8.55 (s, 1H, CHN) 11.82 (s, 1H, OH) **^{13}C NMR** (126 MHz, DMSO-d_6, δ) 60.20 (CH_3O), 60.76 (CH_3O), 102.91 (OCH_2O), 118.45 (C_{Ar}), 122.26 (C_{Ar}), 128.67 (C_{Ar}), 130.39 (C_{Ar}), 131.12 (C_{Ar}), 138.73 (C_{Ar}), 139.81 (C_{Ar}), 142.40 (C_{Ar}), 143.84 (C_{Ar}), 145.50 (CNHOH). **MS** $C_{13}H_{11}ClN_2O_5$ M.w. 310.69: 310, 295, 281, 267, 251, 237, 221, 207, 195, 189, 182, 165, 158.. Anal. Calcd for $C_{13}H_{11}ClN_2O_5$ (%): C, 50.26; H, 3.57; N, 9.02. Found (%): C, 50.38; H, 3.69; N, 9.25.

7-Chloro-4,5-dimethoxy[1,3]dioxolo[4,5-*f*]quinoline-8-carbaldehyde oxime (21b) (mixture of *E/Z* isomers)

Yield 0.134 g (86%), m.p. 207–209 °C (decomp.).

***E*-21b ^{1}H NMR** (DMSO-d_6, δ) 3.90 (s, 3H, CH_3O), 4.09 (s, 3H, CH_3O), 6.28 (s, 2H, OCH_2O), 8.31 (s, 1H, CH_{Ar}) 8.35 (s, 1H, CHN) 11.91 (s, 1H, OH).

***Z*-21b ^{1}H NMR** (DMSO-d_6, δ) 4.00 (s, 3H, CH_3O), 4.10 (s, 3H, CH_3O), 6.21 (s, 2H, OCH_2O), 8.28 (s, 1H, CH_{Ar}) 8.35 (s, 1H, CHN) 11.89 (s, 1H, OH).

^{13}C NMR (126 MHz, DMSO-d_6, δ) 60.51 (CH_3O), 62.08 (CH_3O), 102.58 (OCH_2O), 103.10 (C_{Ar}), 108.76 (C_{Ar}), 123.12 (C_{Ar}), 127.00 (C_{Ar}), 136.46 (C_{Ar}), 136.99 (C_{Ar}), 137.73 (C_{Ar}), 139.99 (C_{Ar}), 143.89 (C_{Ar}), 145.82 (CNHOH). **MS** $C_{13}H_{11}ClN_2O_5$ M.w. 310.69: 310, 295, 281, 265, 251, 237, 221, 215, 207, 195, 182, 176. Anal. Calcd for $C_{13}H_{11}ClN_2O_5$ (%): C, 50.26; H, 3.57; N, 9.02. Found (%): C, 50.19; H, 3.39; N, 9.17.

2-Chloro-5,6,8-tetramethoxyquinoline-3-carbaldehyde oxime (21c)

Yield 0.155 g (95%), m.p. 123–126 °C. **^{1}H NMR** (DMSO-d_6, δ) 3.94 (s, 3H, CH_3O) 3.96 (s, 3H, CH_3O), 3.98 (s, 3H, CH_3O), 4.01 (s, 3H, CH_3O), 8.39 (s, 1H, CH_{Ar}) 8.66 (s, 1H, CHN) 11.93 (s, 1H, OH). **^{13}C NMR** (126 MHz, DMSO-d_6, δ) 61.36 (CH_3O), 61.56 (CH_3O), 61.88 (CH_3O), 62.06 (CH_3O), 119.11 (C_{Ar}), 123.64 (C_{Ar}), 129.58 (C_{Ar}), 138.72 (C_{Ar}), 142.87 (C_{Ar}), 142.93 (C_{Ar}), 143.94 (C_{Ar}), 143.98 (C_{Ar}), 146.79 (C_{Ar}), 149.57 (CNHOH). **MS** $C_{14}H_{15}ClN_2O_5$ M.w. 326.73, 385, 370, 340, 326, 311, 297, 283, 267, 253, 239, 225, 217Anal. Calcd for $C_{14}H_{15}ClN_2O_5$. (%): C, 51.47; H, 4.63; N, 8.57. Found (%):C, 51.55; H, 4.51; N, 8.43.

Synthesis of chloroquinolinemethanol derivatives 22a,b (general procedure)

To a suspension of 2-chloroquinoline-3-carbaldehyde **19a,b** (1.7 mmol) in ethanol (35 ml) was added $NaBH_4$ (3.4 mmol, 1.3 g for **19a**, or 6.6 mmol, 0.2 g for **19b**) at 20 °C. The reaction mixture was stirred at room temperature for 72 h. After stirring, the precipitate was filtered off, the filtrate was evaporated, then the residue was diluted with water (10 ml), the precipitate that formed again was filtered off, washed with water, the precipitates were combined, and dried on air.

6-Chloro-4,9-dimethoxy[1,3]dioxolo[4,5-*g*]quinoline-7-methanol (22a)

Yield 0.430 g (85%), m.p. 157–158 °C **^{1}H NMR** (DMSO-d_6, *δ*) 4.00 (s, 3H, CH_3O) 4.05 (s, 3H, CH_3O) 4.63 (s, 2H, CH_2OH) 5.63 (br. s., 1H, OH) 6.19 (s, 2H, OCH_2O) 8.38 (s, 1H, CH_{Ar}). **^{13}C NMR** (126 MHz, DMSO-d_6, *δ*) 59.81 (CH_2OH), 60.20 (CH_3O), 60.74 (CH_3O), 102.58 (OCH_2O), 118.78 (C_{Ar}), 129.82 (C_{Ar}), 130.27 (C_{Ar}), 131.29 (C_{Ar}), 131.41 (C_{Ar}), 135.89 (C_{Ar}), 137.63 (C_{Ar}), 140.94 (C_{Ar}), 145.87 (C_{Ar}). **MS** $C_{13}H_{12}ClNO_5$ M.w.297.69: 297, 282, 268, 254, 236, 224, 208, 194, 156, 138, 126, 102, 90Anal. Calcd for $C_{13}H_{12}ClNO_5$ (%): C, 52.45; H, 4.06; N, 4.71. Found (%): C, 52.37; H, 4.18; N, 4.64.

7-Chloro-4,5-dimethoxy[1,3]dioxolo[4,5-*f*]quinoline-8-methanol (22b)

Yield 0.420 g (83%), m.p. 189–191 °C. **^{1}H NMR** (DMSO-d_6, *δ*, *J*/Hz) 3.90 (s, 3H, CH_3O), 4.06 (s, 3H, CH_3O), 4.62 (d, 2H, CH_2, J=5.2), 5.68 (t, 1H, OH, J=5.2), 6.26 (s, 2H, OCH_2O), 8.11 (s, 1H, CH_{Ar}). **^{13}C NMR** (126 MHz, DMSO-d_6, *δ*) 59.81 (CH_2OH), 60.49 (CH_3O), 62.05 (CH_3O), 102.80 (OCH_2O), 109.26 (C_{Ar}), 127.58 (C_{Ar}), 132.48, (C_{Ar}) 135.69 (C_{Ar}), 136.69 (C_{Ar}), 137.09 (C_{Ar}), 140.04 (C_{Ar}), 140.19 (C_{Ar}), 146.44 (C_{Ar}). **MS** $C_{13}H_{12}ClNO_5$ M.w.297.69: 297, 282, 268, 252, 238, 226, 208, 194, 180, 169, 156, 142, 126. Anal. Calcd for $C_{13}H_{12}ClNO_5$ (%): C, 52.45; H, 4.06; N, 4.71. Found (%): C, 52.53; H, 4.15; N, 4.82.

Synthesis of 2-chloro-5,6,7,8-tetramethoxyquinoline-3-methanol (22c)

To a solution of 2-chloroquinoline-3-carbaldehyde **19c** (0.45 mmol, 0.140 g) in ethanol (3 ml), $NaBH_4$ (1.4 mmol, 0.053 g) was added in portions while cooling in an ice bath. The reaction mixture was stirred for 30 min while cooling, then 30 min at room temperature. After stirring, the solvent was distilled off *in vacuo*, the residue was diluted with water (10 ml) and dichloromethane (10 ml), the organic layer was separated, the aqueous layer was extracted with dichloromethane (2 × 10 ml), the organic phases were combined, dried over $MgSO_4$, evaporated into vacuum to give 0.135 g of product **22c** as colorless solid. Yield 96%, m.p. 70–73 °C. **^{1}H NMR** (DMSO-d_6, *δ*) 3.94 (s, 3H, CH_3O), 3.95 (s, 3H, CH_3O), 3.98 (s, 6H, 2×CH_3O), 4.66 (s, 2H, CH_2), 5.72 (br.s., 1H, OH), 8.44 (s, 1H, CH_{Ar}). **^{13}C NMR** (126 MHz, DMSO-d_6, *δ*) 59.84 (CH_2OH), 61.30 (CH_3O), 61.48 (CH3O), 61.74 (CH_3O), 61.95 (CH_3O), 119.46 (C_{Ar}), 130.22 (C_{Ar}), 132.72 (C_{Ar}), 137.73 (C_{Ar}), 142.44 (C_{Ar}), 143.10 (C_{Ar}), 144.58 (C_{Ar}), 147.24 (C_{Ar}), 148.14 (C_{Ar}). **MS** $C_{14}H_{16}ClNO_5$ M.w.313.73: 313, 298, 284, 270, 264, 255, 240, 226, 220, 212, 194, 184. Anal. Calcd for $C_{14}H_{16}ClNO_5$ (%): C, 53.60; H, 5.14; N, 4.46. Found (%): C, 53.73; H, 5.25; N, 4.38.

Synthesis of 6-chloro-4,9-dimethoxy[1,3]dioxolo[4,5-*g*]quinoline-7-carbonitrile (23a)

To a suspension of 2-chloroquinoline-3-carbaldehyde **19a** (1 mmol, 0.296 g) in THF (5 ml) an aqueous solution of NH_3 (30%, 8 ml, 111 mmol) and I_2 (1.2 mmol, 0.305 g) were added at 20 °C. The reaction mixture was stirred at same temperature for 9.5 h until the starting aldehyde completely disappeared (TLC monitoring). Then, Na_2SO_3 (1.5 mmol, 0.225 g) and concentrated HCl solution (11 M, 10 ml, 111 mmol) were added to the mixture while cooling in an ice bath. The precipitate was filtered off, washed with water, and dried in air to give 0.187 g of product **23a** as light yellow solid. Yield 64% m.p. 227–228 °C. **^{1}H NMR** (DMSO-d_6, δ) 4.00 (s, 3H, CH_3O) 4.07 (s, 3H, CH_3O), 6.30 (s, 2H, OCH_2O) 8.81 (s, 1H, CH_{Ar}). **^{13}C NMR** (126 MHz, DMSO-d_6, δ) 60.55 (CH_3O), 60.95 (CH_3O), 103.71 (OCH_2O), 103.94 (C_{Ar}), 115.66 (C_{Ar}), 117.13 (CN), 130.68 (C_{Ar}), 131.33 (C_{Ar}), 136.99 (C_{Ar}), 139.25 (C_{Ar}), 139.41 (C_{Ar}), 145.04 (C_{Ar}), 145.33 (C_{Ar}). **MS** $C_{13}H_{19}ClN_2O_4$ M.w.292.67: 292, 277, 263, 249, 231, 219, 204, 197, 189, 176, 164, 151. Anal. Calcd for $C_{13}H_{19}ClN_2O_4$ (%): C, 53.35; H, 3.10; N, 9.57. Found (%): C, 53.48; H, 3.23; N, 9.69.

Synthesis of 2-chloro-5,6,7,8-tetramethoxyquinoline-3-carbonitrile (23c)

To a suspension of 2-chloroquinoline-3-carbaldehyde **19c** (0.7 mmol, 0.218 g) in THF (3.5 ml) was added an aqueous solution of NH_3 (30%, 5.6 ml, 78 mmol) and I_2 (1.3 mmol, 0.330 g) at 20 °C. The reaction mixture was stirred at room temperature for 1 h until the starting aldehyde completely disappeared (TLC monitoring). After stirring, a solution of Na_2SO_3 (2 mmol, 0.250 g) in water (2 ml) and a concentrated solution of HCl (11 M, 7.1 ml, 78 mmol) were added dropwise to the mixture while cooling in an ice bath. The mixture was diluted with water (5 ml) and dichloromethane (10 ml), the organic layer was separated, the aqueous one was extracted with dichloromethane (2×10 ml), the organic phases were combined, dried over $MgSO_4$, and evaporated in vacuum to give 0.197 g of product **23c** as light yellow solid. Yield 91%, m.p. 103–105 °C. **^{1}H NMR** (DMSO-d_6, δ) 3.96 (s, 3H, CH_3O) 3.98 (s, 6H, 2× CH_3O), 4.07 (s, 3H, CH_3O), 9.05 (s, 1H, CH_{Ar}). **^{13}C NMR** (126 MHz, DMSO-d_6, δ) 61.36 (CH_3O), 61.59 (CH_3O), 62.17 (CH_3O), 62.25 (CH_3O), 105.20 (C_{Ar}), 115.61 (C_{Ar}), 117.56 (CN), 139.12 (C_{Ar}), 141.04 (C_{Ar}), 142.70 (C_{Ar}), 143.28 (C_{Ar}), 145.34 (C_{Ar}), 146.11 (C_{Ar}), 151.95 (C_{Ar}). **MS** $C_{14}H_{13}ClN_2O_4$ M.w.308.72: 308, 293, 279, 265, 250, 235, 221, 207, 198, 189, 179, 172, 164. Anal. Calcd for $C_{14}H_{13}ClN_2O_4$ (%): C, 54.47; H, 4.24; N, 9.07. Found (%): C, 54.59; H, 4.32; N, 9.15.

References

S1 I. B. Karmanova, S. I. Firgang, L. D. Konyushkin, V. N. Khrustalev, A. V. Ignatov, L. A. Kuznetsov, Y. A. Pinchuk, I. A. Kozlov, V. V Semenov, *Mend. Comm.*, 2016, **26**, 66.

S2 O. Meth-Cohn, B. Narine, B. Tarnowski, *J. Chem. Soc., Perkin Trans. 1*, 1981, 1520.

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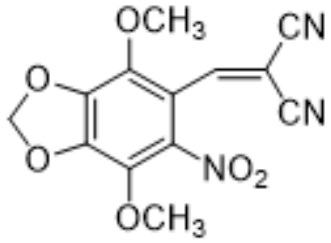
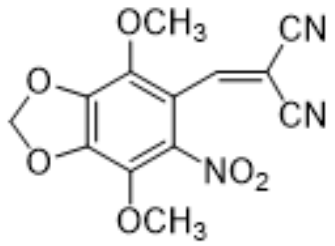


7a

NMR/50164189



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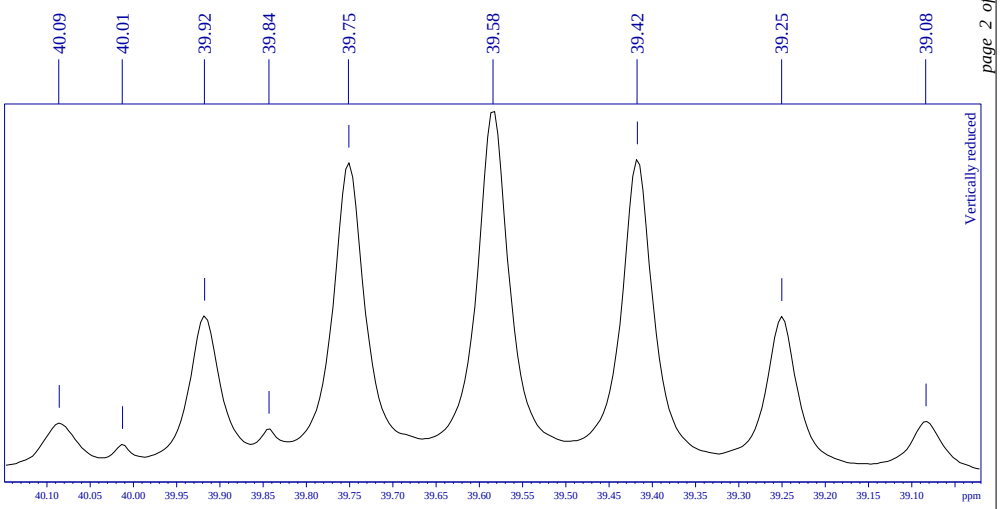

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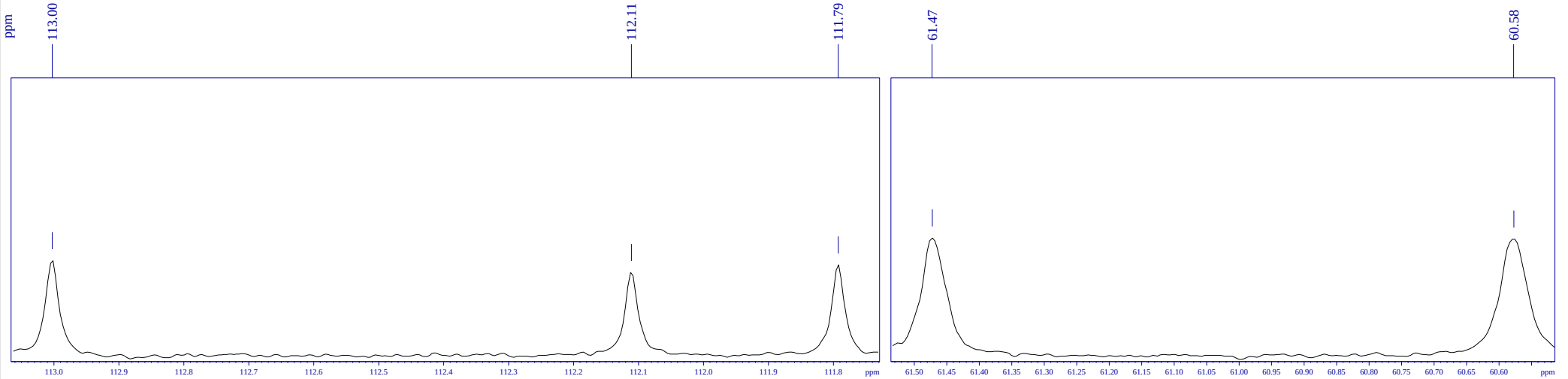

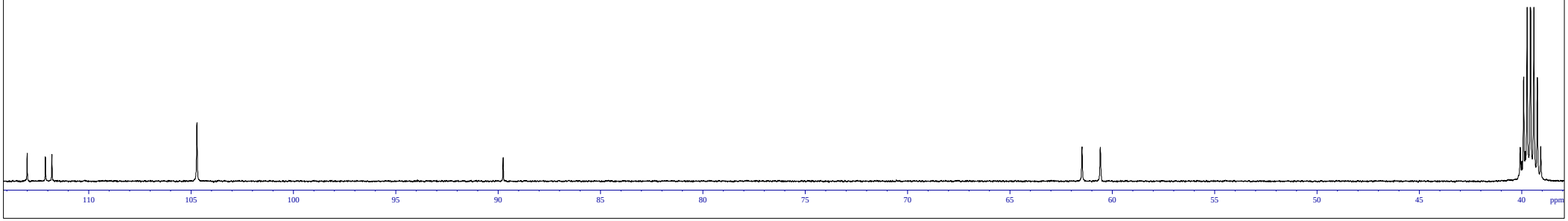

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3	23187.0	17801.887	141.5569	0.95
4	24323.7	17256.480	137.2199	0.70
5	24705.6	17073.189	135.7624	0.94
6	25495.1	16694.389	132.7503	0.93
7	30670.6	14210.967	113.0026	1.64
8	30904.3	14098.838	112.1110	1.44
9	30987.8	14058.798	111.7926	1.56
10	32844.1	13168.048	104.7096	3.41
11	36764.4	11286.963	89.7516	1.39
12	44176.0	7730.620	61.4723	2.01
13	44410.6	7618.056	60.5772	2.00
14	49781.1	5041.102	40.0858	1.97
15	49800.2	5031.902	40.0126	1.17
16	49825.1	5019.962	39.9177	6.08
17	49844.6	5010.589	39.8432	1.76
18	49868.8	4998.992	39.7509	11.95
19	49912.5	4978.008	39.5841	14.00
20	49956.3	4957.032	39.4173	12.09
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22	50043.7	4915.060	39.0835	2.03

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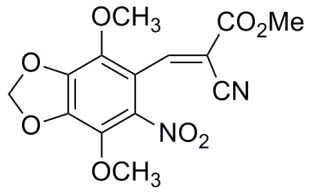

7b

The Best Applied NMR!

NMR/50164190

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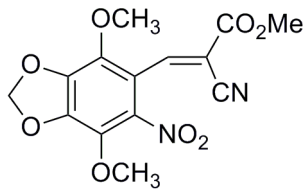

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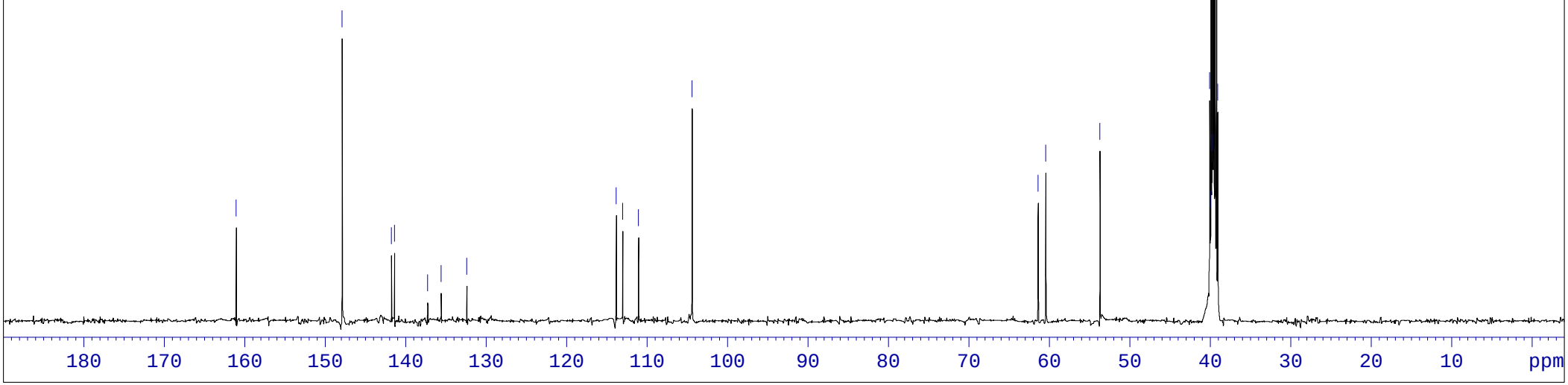

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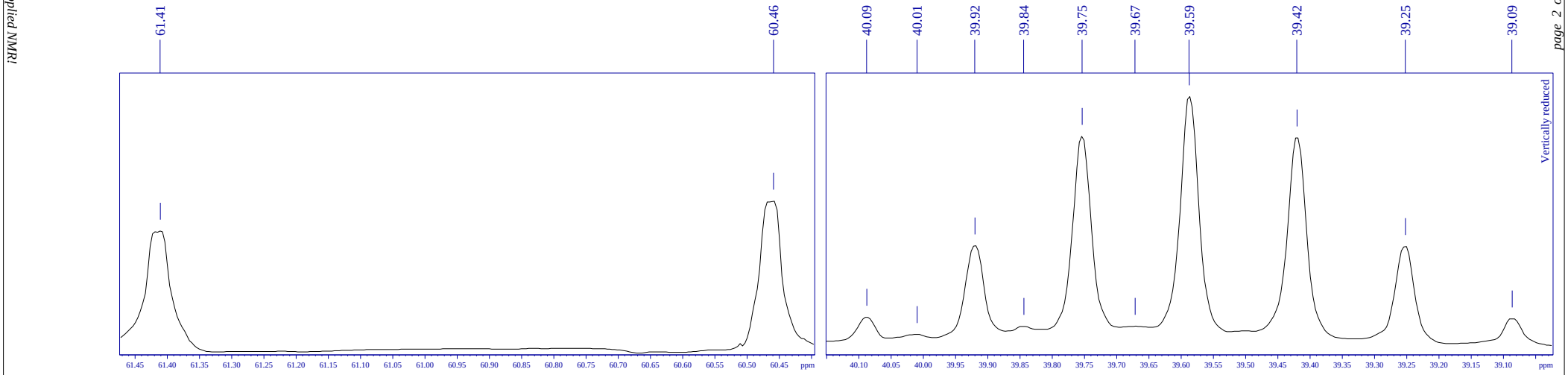

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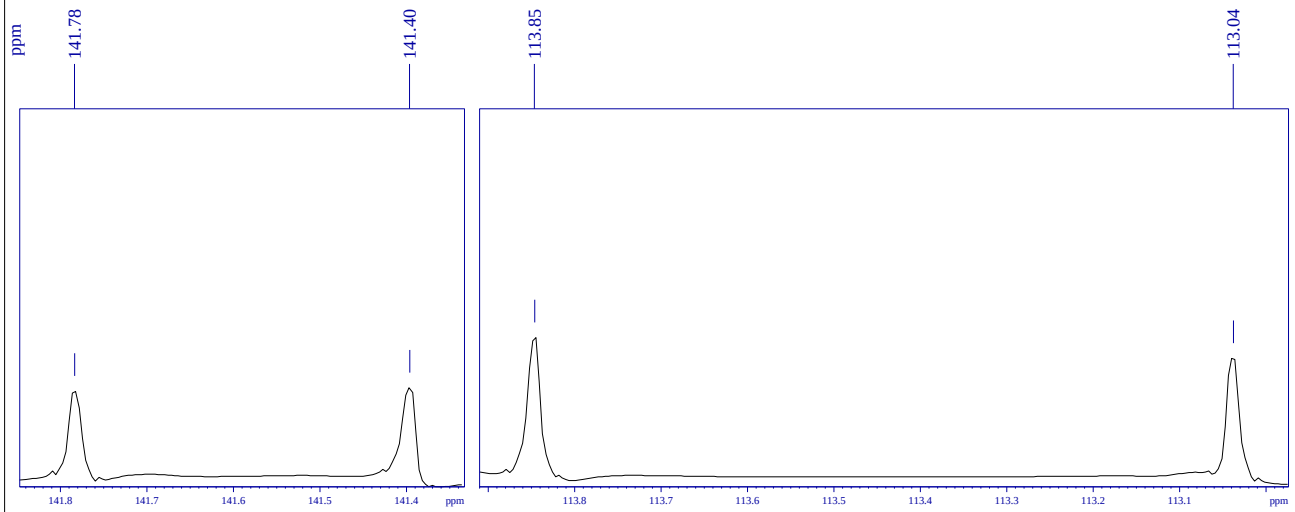

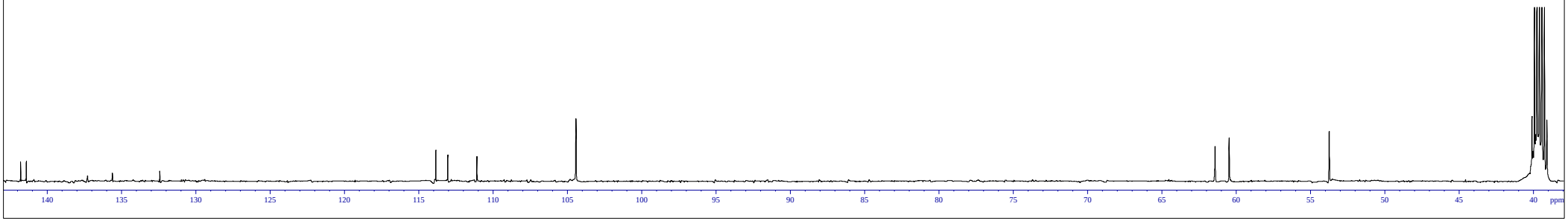

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3	23127.6	17830.393	141.7835	1.23
4	23229.1	17781.686	141.3962	1.26
5	24307.2	17264.385	137.2828	0.34
6	24747.4	17053.174	135.6033	0.52
7	25581.7	16652.836	132.4199	0.66
8	30449.6	14317.051	113.8462	2.01
9	30661.4	14215.383	113.0377	1.69
10	31174.0	13969.422	111.0819	1.54
11	32921.2	13131.055	104.4154	3.93
12	44192.1	7722.890	61.4108	2.18
13	44441.6	7603.188	60.4590	2.75
14	46204.1	6757.466	53.7340	3.14
15	49780.5	5041.347	40.0877	4.09
16	49801.0	5031.523	40.0096	1.89
17	49824.5	5020.236	39.9199	13.15
18	49844.4	5010.690	39.8440	2.92
19	49868.1	4999.331	39.7536	26.93
20	49889.6	4988.994	39.6714	2.93
21	49911.7	4978.414	39.5873	32.00
22	49955.5	4957.402	39.4202	26.96
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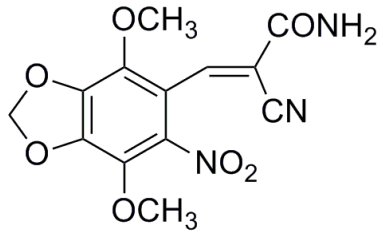
/KVEN 745



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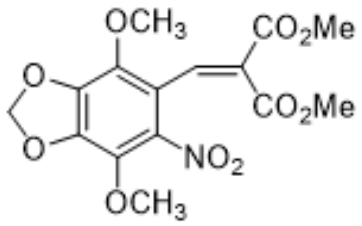

7d

NMR/50160119

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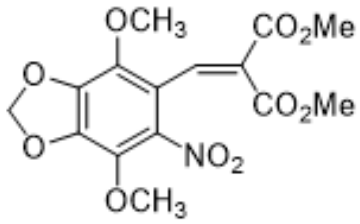
52.46



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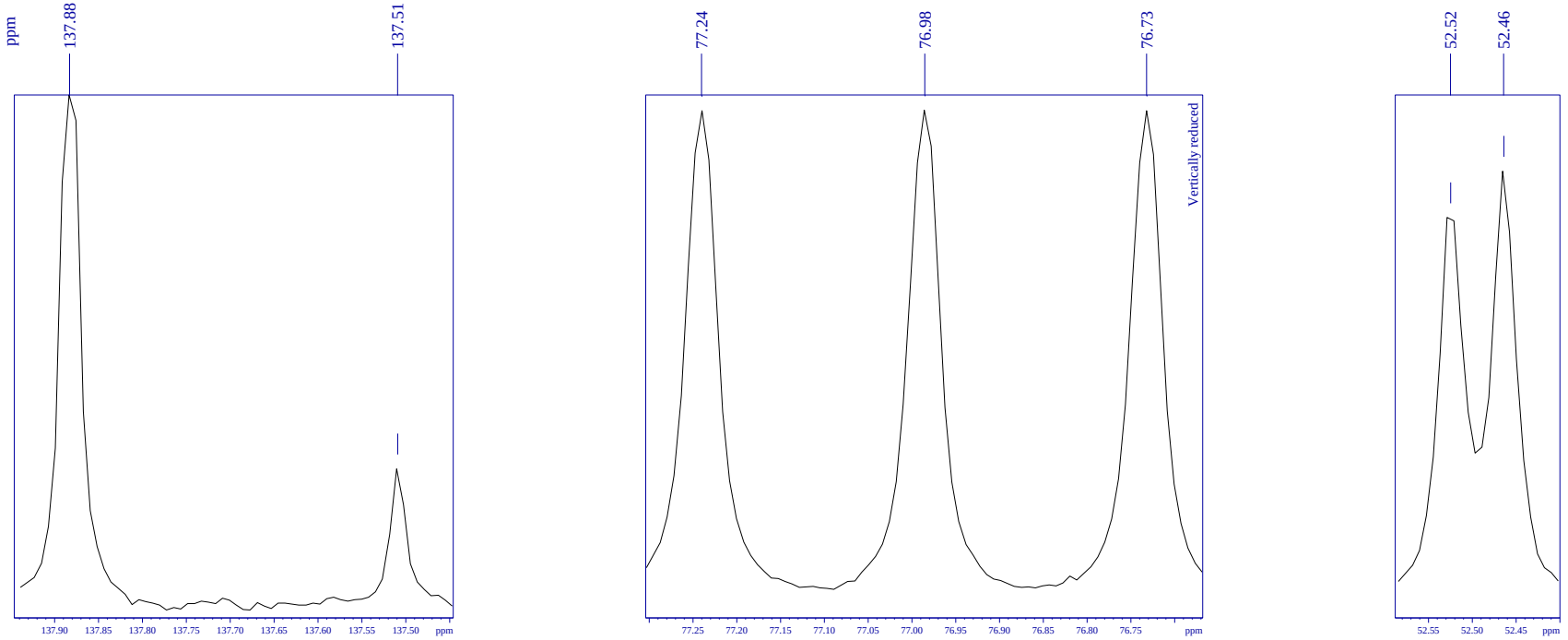

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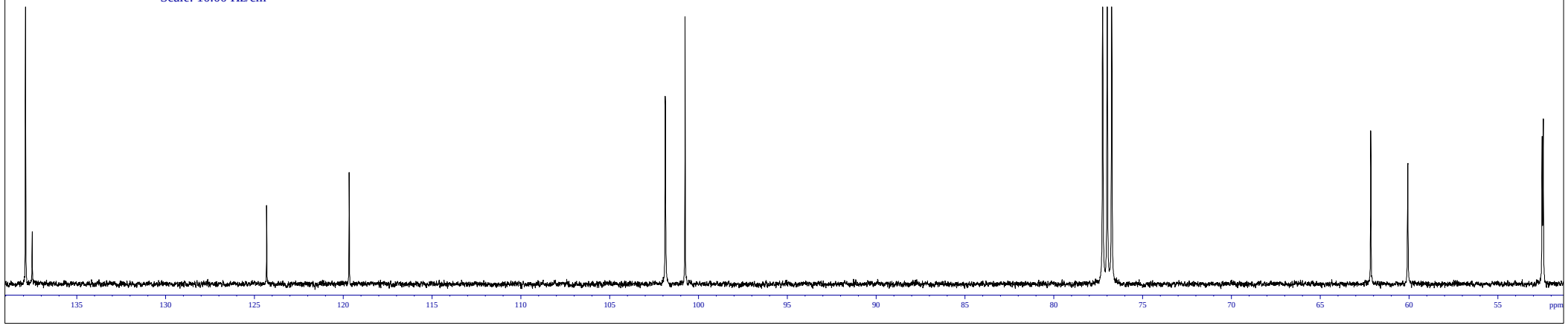

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4	11961.5	18243.930	145.0720	2.96
5	12559.6	17647.447	140.3288	1.92
6	12868.0	17339.834	137.8828	11.07
7	12915.1	17292.861	137.5092	1.97
8	14579.8	15632.655	124.3076	2.96
9	15165.9	15048.159	119.6599	4.17
10	17410.3	12809.800	101.8609	7.07
11	17550.3	12670.150	100.7504	10.31
12	20515.0	9713.514	77.2399	11.98
13	20547.1	9681.468	76.9850	12.00
14	20579.0	9649.606	76.7317	11.99
15	22417.9	7815.656	62.1485	5.70
16	22677.6	7556.730	60.0896	4.59
17	23631.5	6605.395	52.5247	5.71
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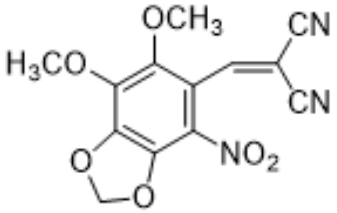
The Best Applied NMR!

NMR/50751271

Found protons = 9 impurity* < 0.1 %



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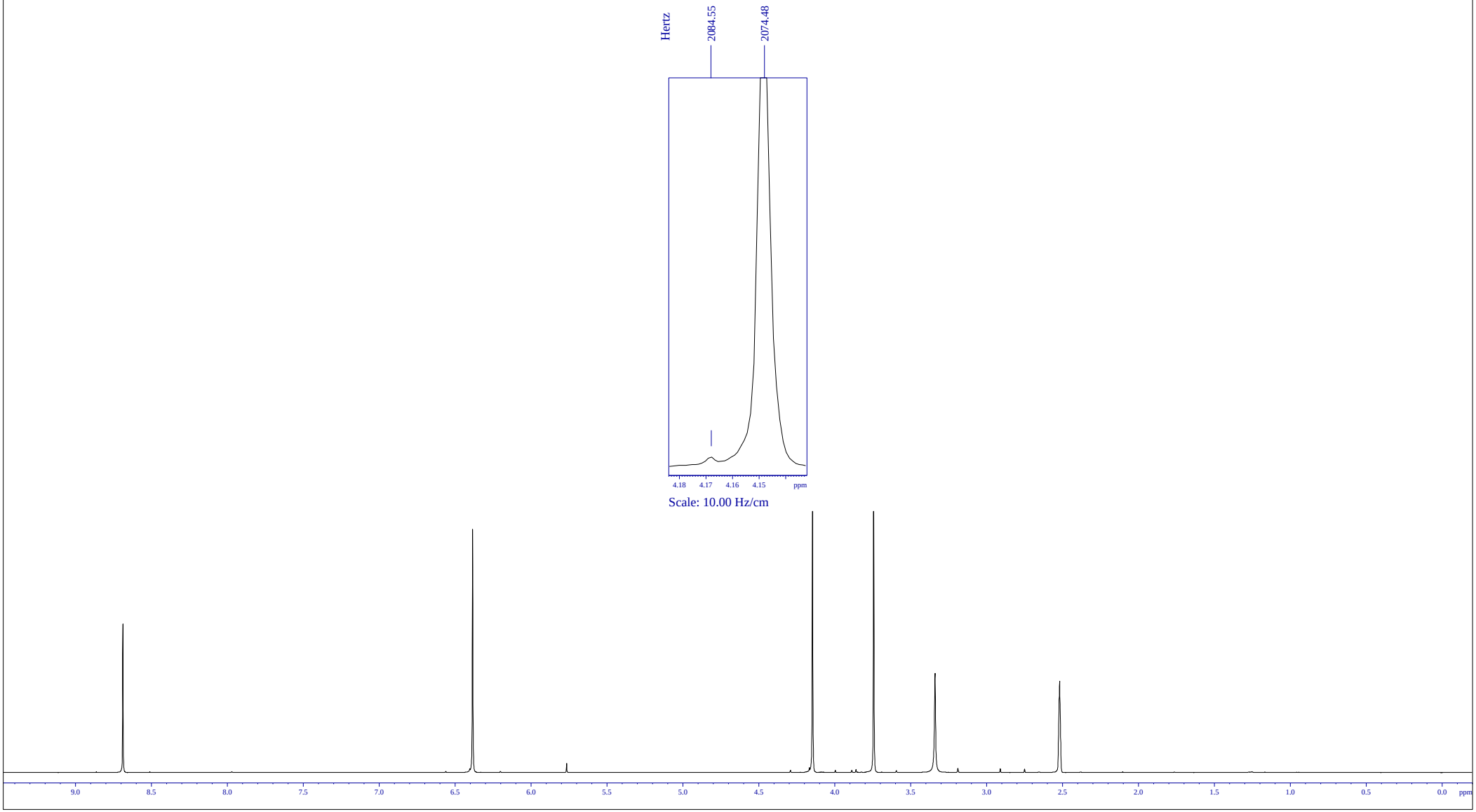
NMR/50751271



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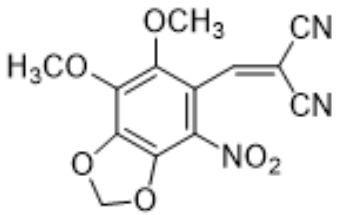
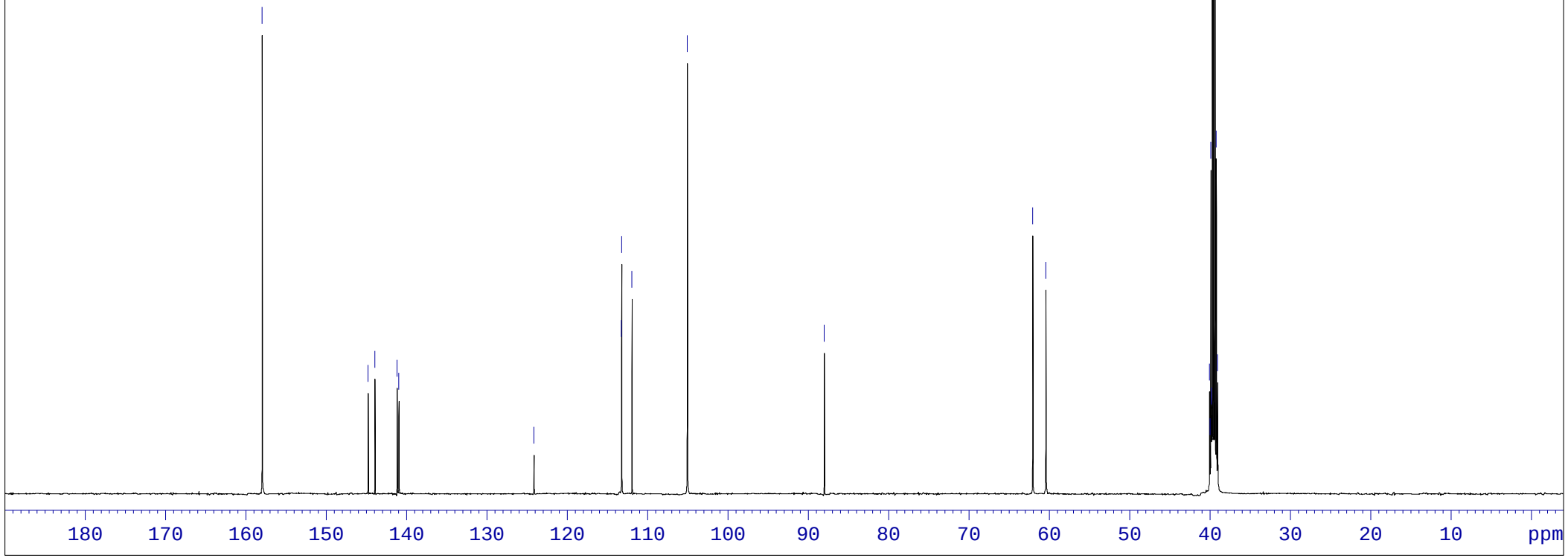
3	10029.5	2884.292	5.7671	0.39
4	11339.8	2084.554	4.1680	0.20
5	11356.3	2074.484	4.1479	16.00
6	11686.2	1873.114	3.7453	15.26
7	12016.9	1671.297	3.3417	3.93
8	12689.8	1260.601	2.5205	3.64

NMR/50751148

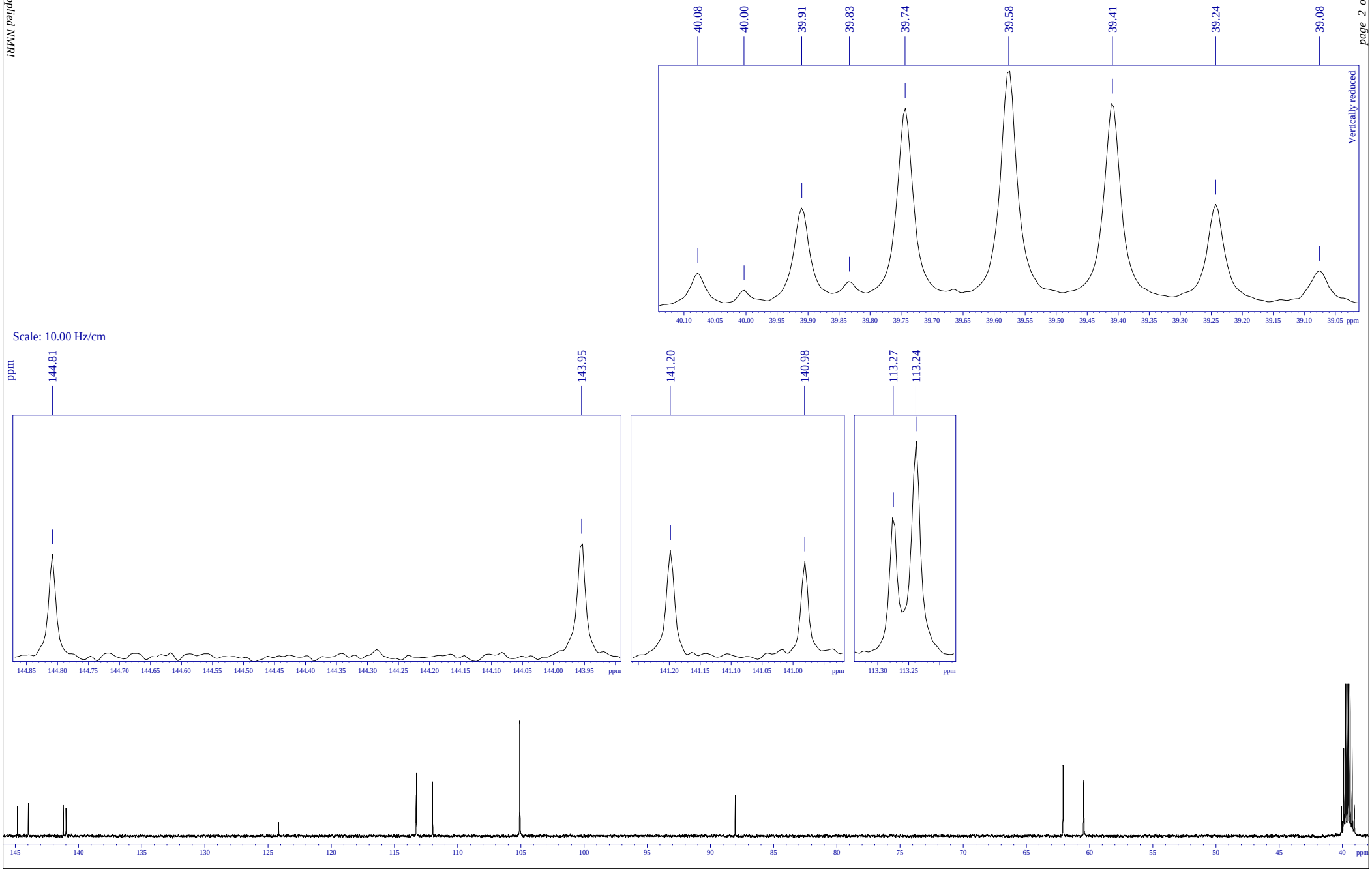
157.99
144.81
143.95
141.20
140.98
124.16
113.27
113.24
111.97
105.08
88.03
62.08
60.45
40.08
40.00
39.91
39.83
39.74
39.58
39.41
39.24
39.08



8a



NMR/50751148



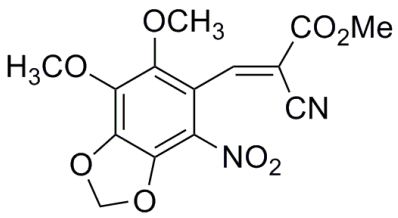
NMR/50751148

Peaks List

#	Address [points]	Frequency [Hz]	[ppm]	Intensity [cm]
1	18879.3	19868.871	157.9931	8.25
2	22334.9	18210.762	144.8082	2.01
3	22558.6	18103.424	143.9546	2.29
4	23281.1	17756.756	141.1980	2.10
5	23337.9	17729.465	140.9810	1.87
6	27745.8	15614.409	124.1625	0.93
7	30599.3	14245.187	113.2747	2.80
8	30608.9	14240.584	113.2381	4.23
9	30940.7	14081.392	111.9723	3.69
10	32748.3	13214.049	105.0753	7.72
11	37215.4	11070.580	88.0309	2.77
12	44016.3	7807.241	62.0815	4.73
13	44443.8	7602.118	60.4504	3.76
14	49783.2	5040.080	40.0777	1.99
15	49802.7	5030.708	40.0031	0.98
16	49827.1	5018.993	39.9100	5.83
17	49847.2	5009.350	39.8333	1.50
18	49870.8	4998.040	39.7434	11.72
19	49914.6	4977.038	39.5764	14.00
20	49958.4	4956.026	39.4093	12.04
21	50002.0	4935.095	39.2428	6.02
22	50045.9	4914.040	39.0754	2.13

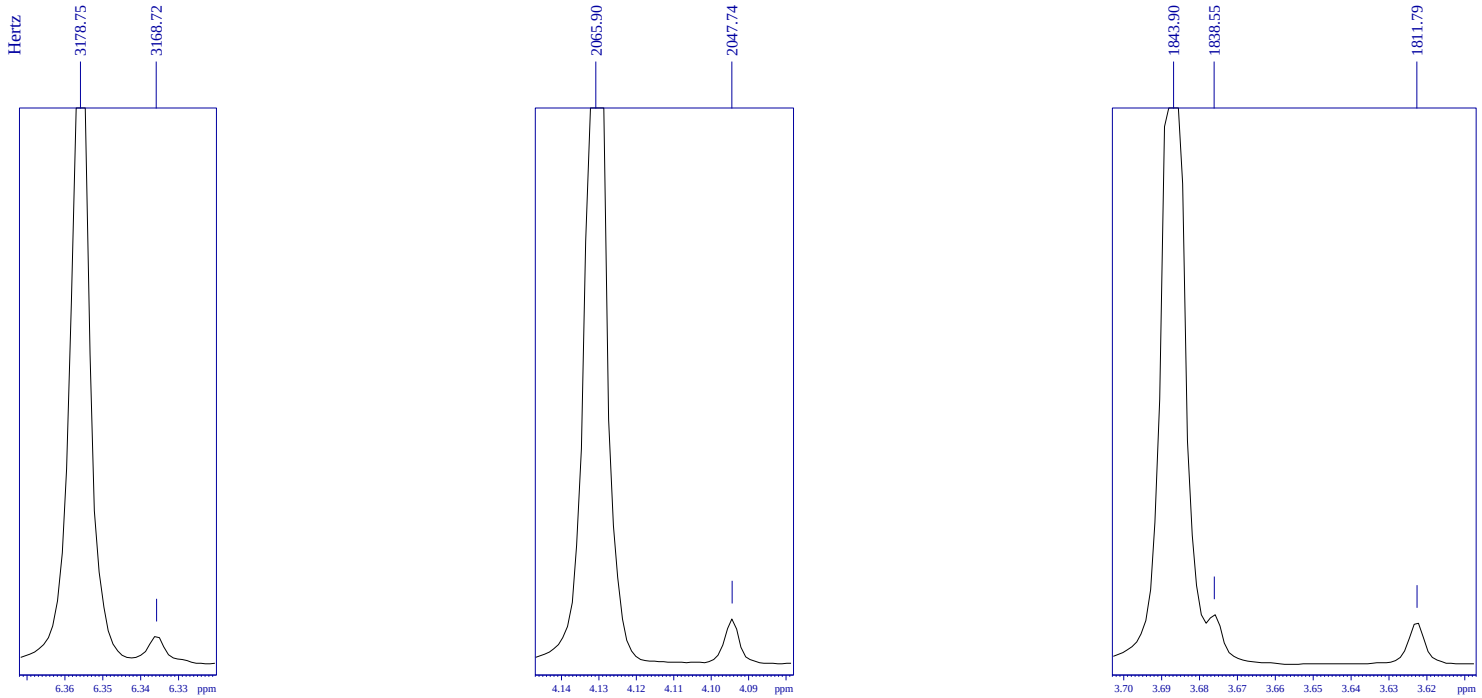
NMR/50751270

Found protons = 12 impurity* = 0.7 %



8b

NMR/50751270



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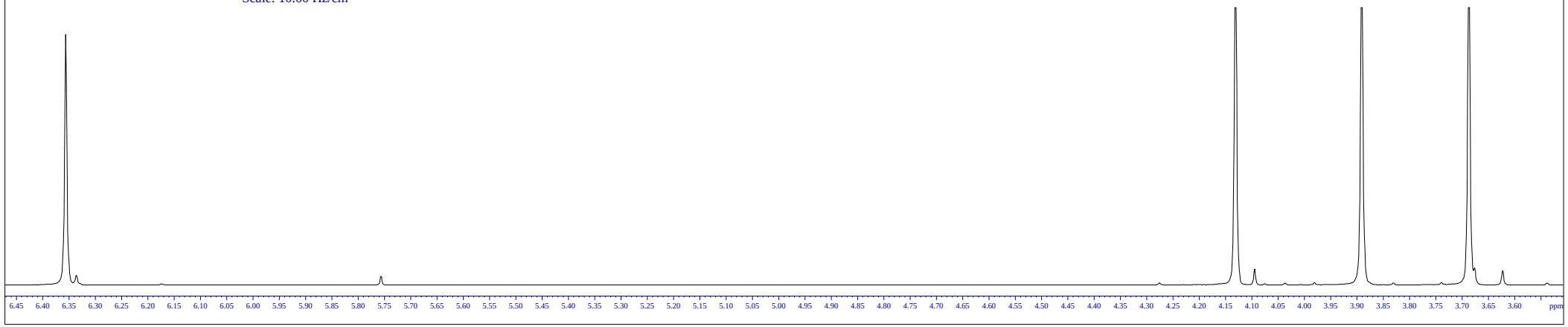


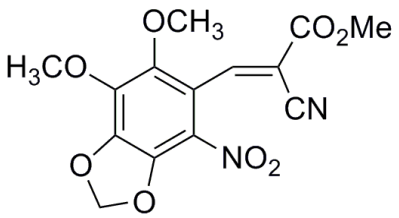
NMR/50751270

Peaks List

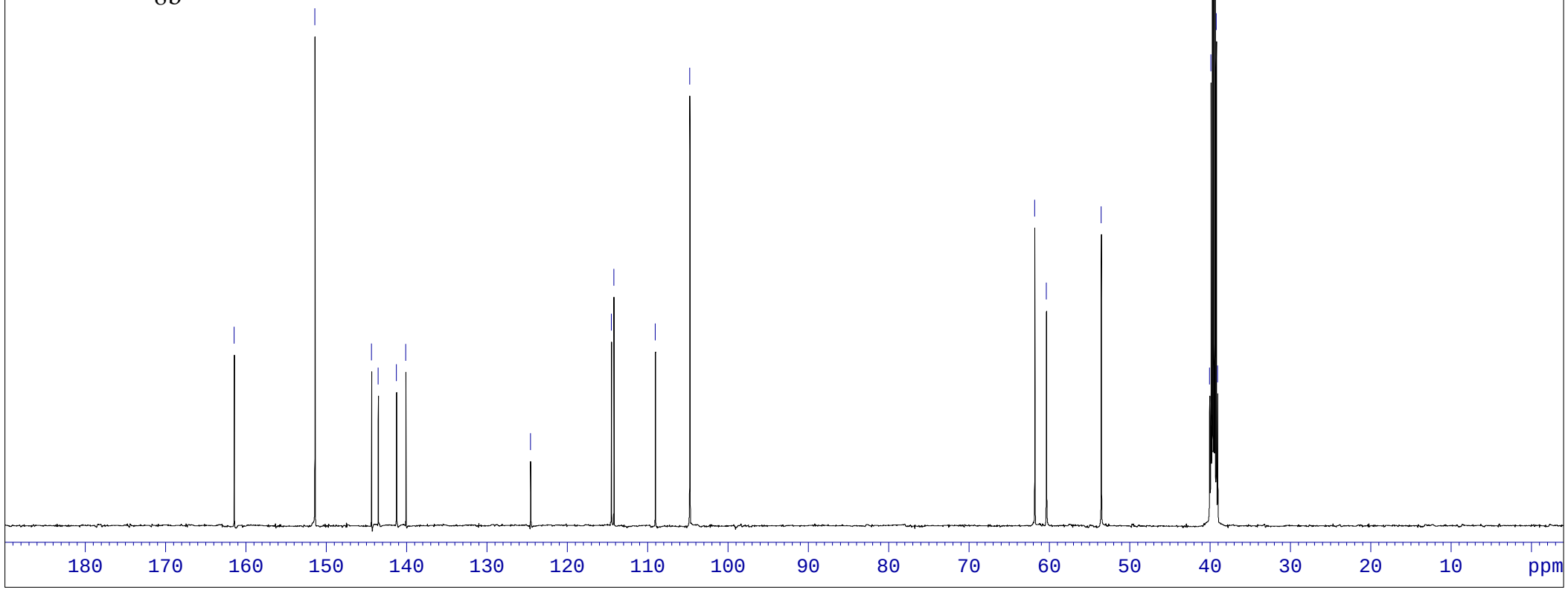
#	Address [points]	Frequency [Hz]	[ppm]	Intensity [cm]
1	7779.9	4251.160	8.5001	5.63
2	7972.1	4133.850	8.2655	0.20
3	9536.9	3178.752	6.3559	9.37
4	9553.4	3168.715	6.3358	0.36
5	10028.2	2878.884	5.7563	0.33
6	11360.2	2065.902	4.1307	16.00
7	11390.0	2047.741	4.0944	0.59
8	11557.1	1945.746	3.8905	14.41
9	11724.0	1843.900	3.6868	15.36
10	11732.7	1838.550	3.6761	0.65
11	11776.6	1811.790	3.6226	0.55
12	12016.9	1665.079	3.3293	6.41
13	12689.6	1254.503	2.5084	4.24

NMR/50164183

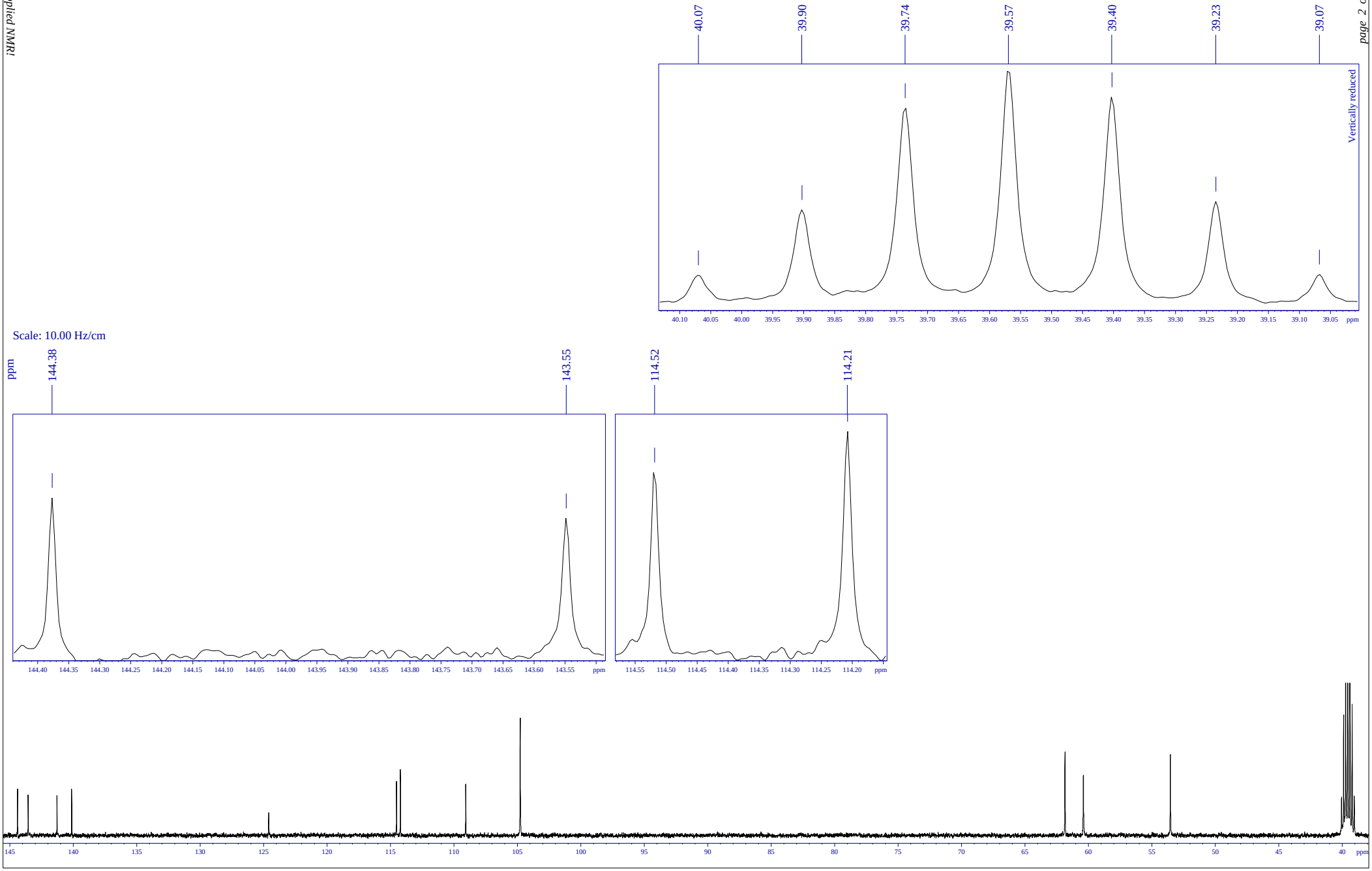
161.47 151.43 144.38 143.55 141.28 140.12 124.58 114.52 114.21 109.06 104.77 61.83 60.38 53.56 40.07 39.90 39.74 39.57 39.40 39.23 39.07



8b



NMR/50164183

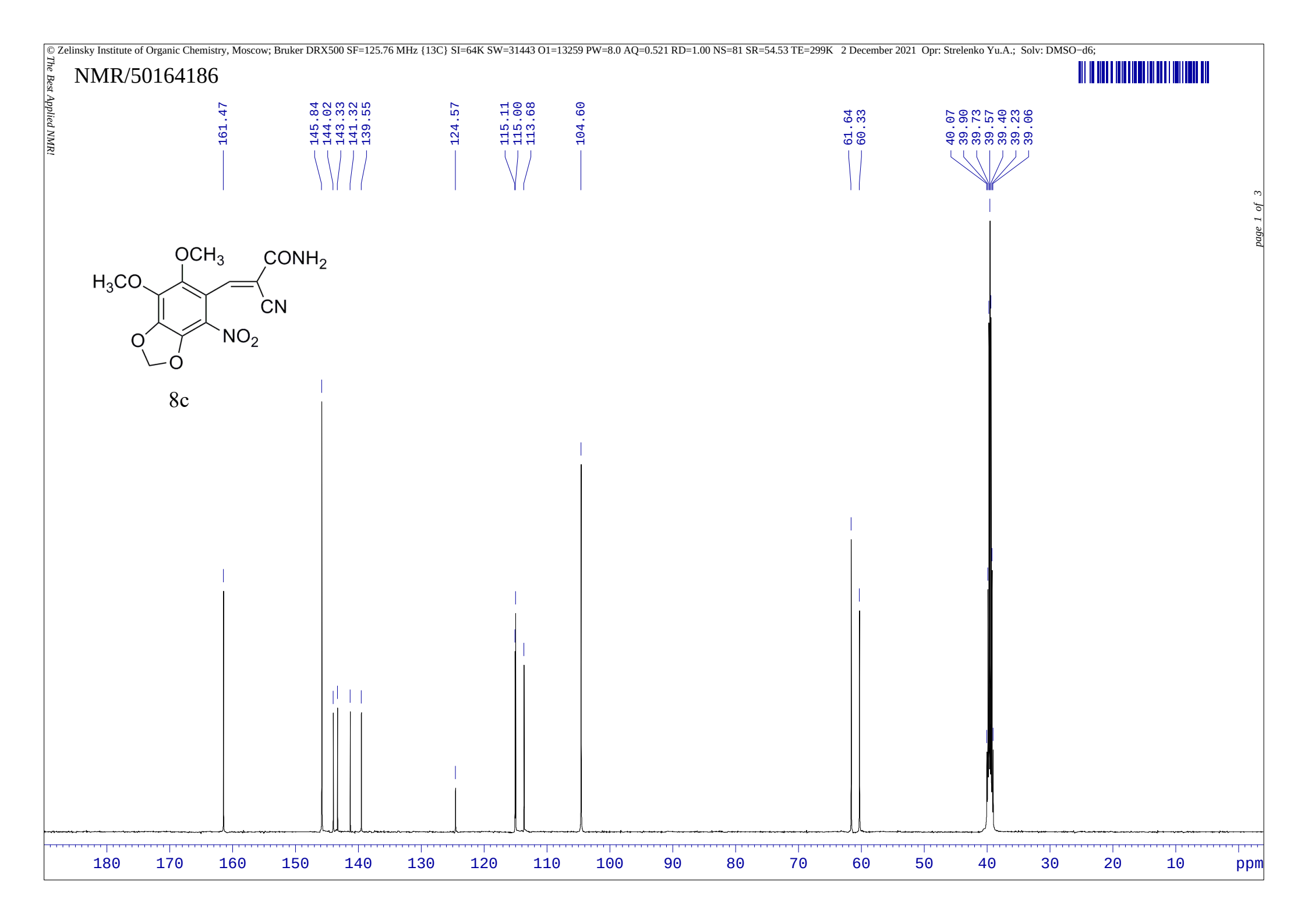


NMR/50164183

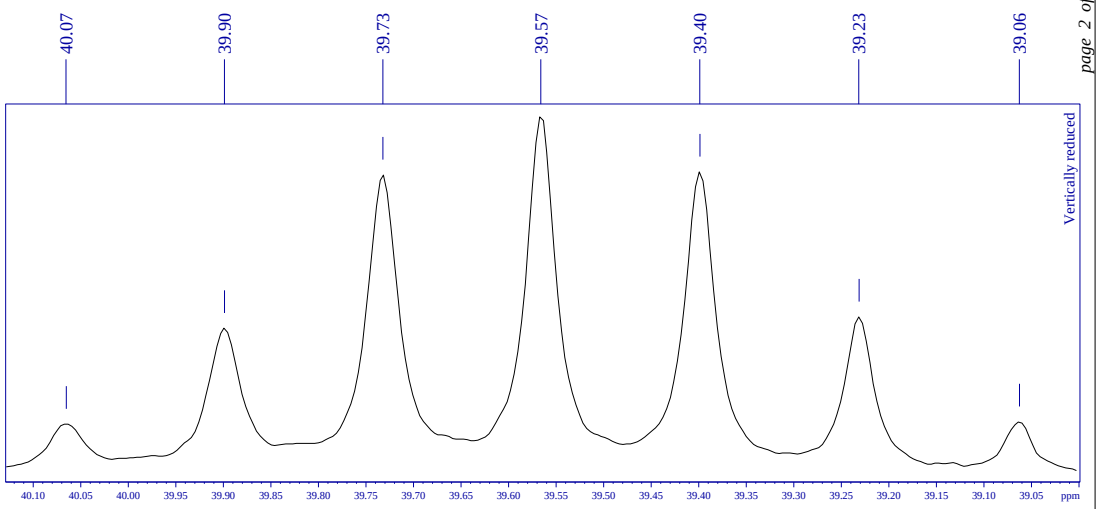
Peaks List

#	Address [points]	Frequency [Hz]	[ppm]	Intensity [cm]
1	17967.6	20306.334	161.4717	3.48
2	20600.0	19043.221	151.4277	8.91
3	22448.0	18156.492	144.3766	3.11
4	22665.1	18052.309	143.5482	2.71
5	23260.5	17766.633	141.2765	2.79
6	23564.9	17620.566	140.1151	3.12
7	27635.1	15667.531	124.5849	1.52
8	30273.3	14401.610	114.5186	3.68
9	30354.8	14362.504	114.2076	4.43
10	31703.7	13715.282	109.0611	3.49
11	32829.3	13175.149	104.7660	7.91
12	44081.0	7776.214	61.8348	5.61
13	44461.4	7593.681	60.3834	4.04
14	46249.7	6735.568	53.5598	5.42
15	49785.2	5039.109	40.0699	2.58
16	49829.0	5018.077	39.9027	8.07
17	49872.7	4997.134	39.7362	16.75
18	49916.5	4976.132	39.5692	20.00
19	49960.1	4955.169	39.4025	17.59
20	50004.1	4934.079	39.2348	8.79
21	50047.8	4913.086	39.0678	2.64

NMR/50164186



NMR/50164186



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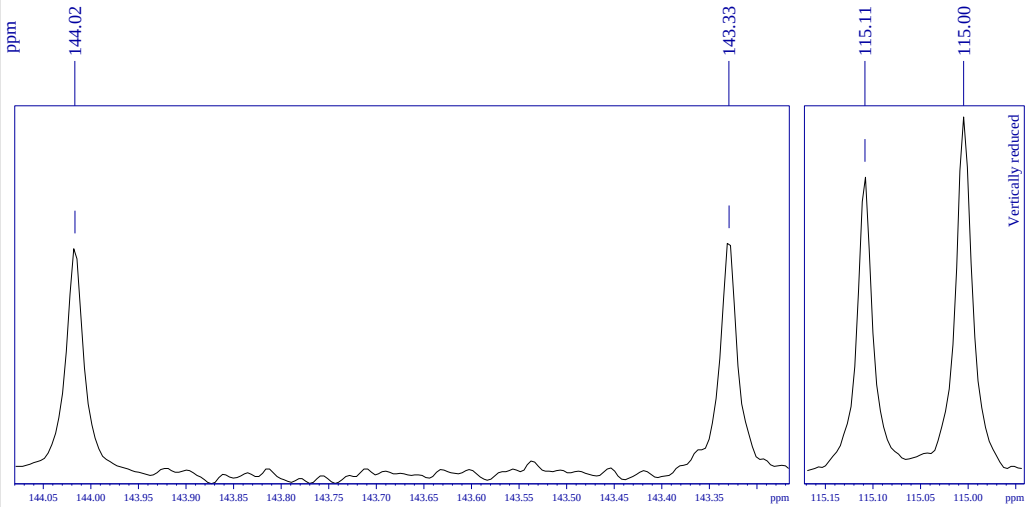
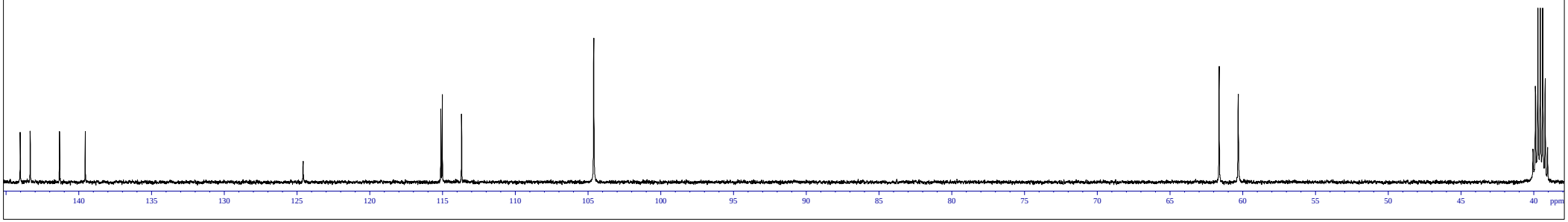


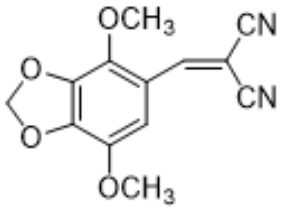


NMR/50164186

Peaks List

#	Address [points]	Frequency [Hz]	[ppm]	Intensity [cm]
1	17968.4	20305.980	161.4689	5.80
2	22064.7	18340.387	145.8389	9.97
3	22542.3	18111.230	144.0167	2.99
4	22722.5	18024.789	143.3293	3.10
5	23250.1	17771.596	141.3160	3.01
6	23713.7	17549.148	139.5471	3.02
7	27638.7	15665.790	124.5711	1.23
8	30118.8	14475.784	115.1084	4.37
9	30146.0	14462.703	115.0044	5.20
10	30493.1	14296.173	113.6802	4.05
11	32874.0	13153.706	104.5955	8.52
12	44132.0	7751.720	61.6401	6.84
13	44476.7	7586.341	60.3250	5.22
14	49786.5	5038.511	40.0652	1.97
15	49830.1	5017.585	39.8988	5.69
16	49873.7	4996.632	39.7322	11.69
17	49917.4	4975.690	39.5656	14.00
18	49961.1	4954.695	39.3987	11.78
19	50005.0	4933.639	39.2313	6.13
20	50049.2	4912.430	39.0626	2.06

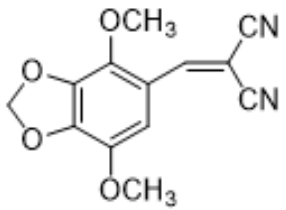
/KVEN 747



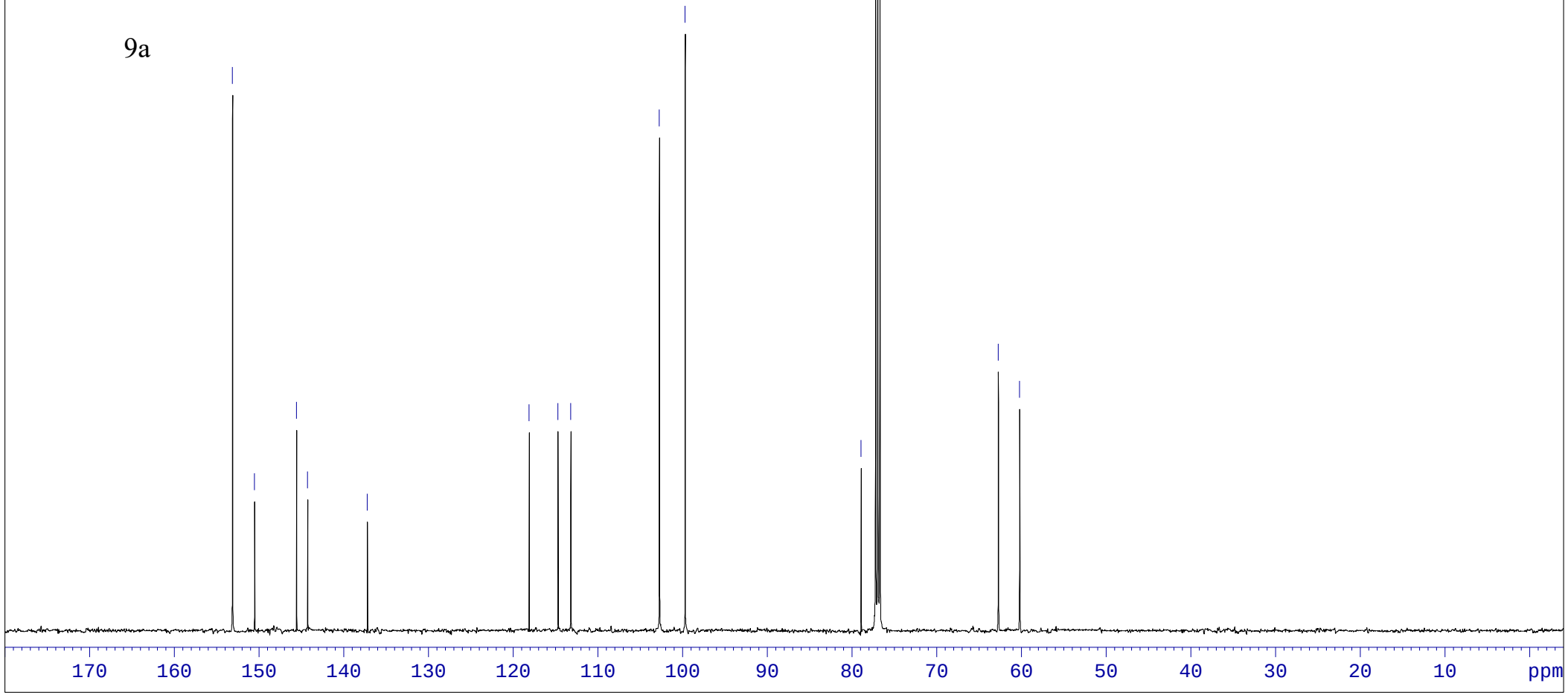
9a

NMR/50160931

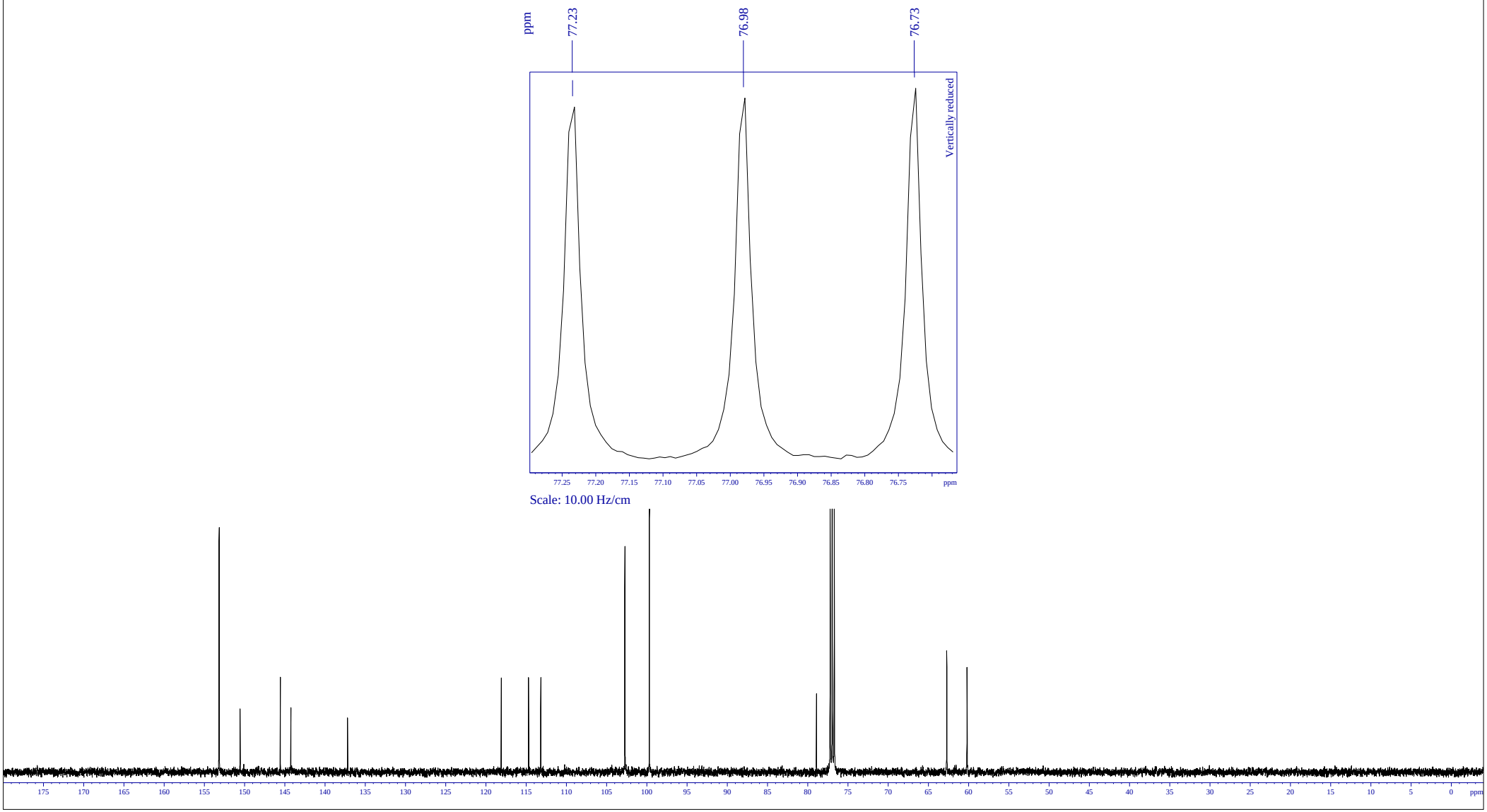
153.15 150.55 145.58 144.28 137.23 118.13 114.72 113.21 102.76 99.72 78.94 77.23 76.98 76.73 62.74 60.23



9a



NMR/50160931

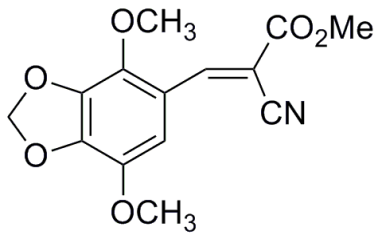


NMR/50160931

Peaks List

#	Address [points]	Frequency [Hz]	[ppm]	Intensity [cm]
1	10942.6	19260.105	153.1524	10.00
2	11271.3	18932.318	150.5459	2.54
3	11897.0	18308.307	145.5839	3.70
4	12061.1	18144.633	144.2824	2.50
5	12950.1	17257.951	137.2317	2.14
6	15358.8	14855.764	118.1300	3.72
7	15788.4	14427.325	114.7231	3.93
8	15978.8	14237.413	113.2130	3.72
9	17296.8	12922.996	102.7610	8.85
10	17680.6	12540.247	99.7175	11.16
11	20300.1	9927.764	78.9435	3.06
12	20515.6	9712.845	77.2345	22.14
13	20547.7	9680.884	76.9804	22.54
14	20579.7	9648.922	76.7262	23.00
15	22343.1	7890.271	62.7418	4.72
16	22660.0	7574.222	60.2286	4.06

/KVEN 740

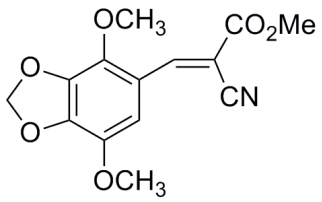


9b

The Best Applied NMR!

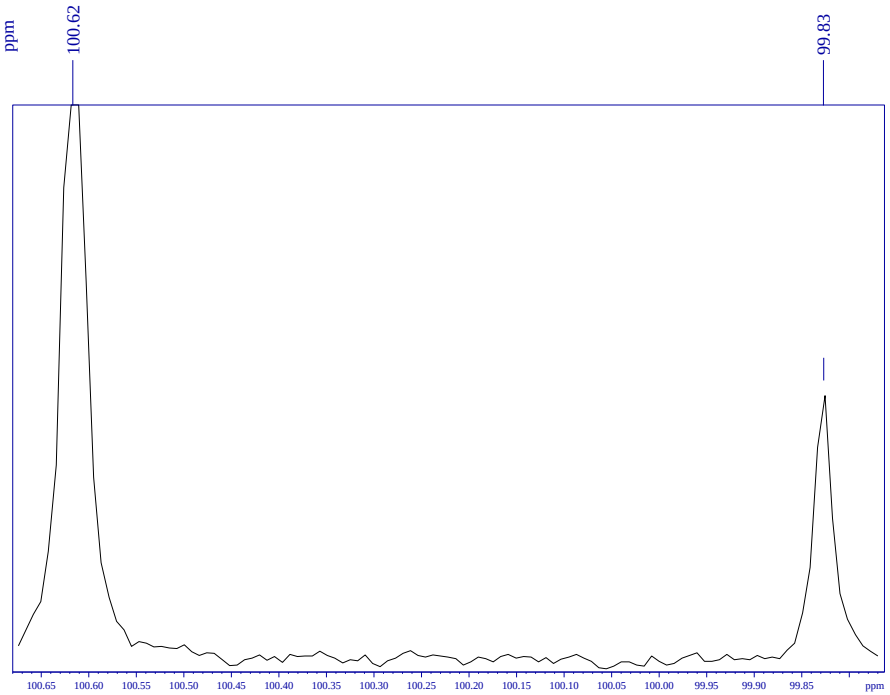
NMR/50160929

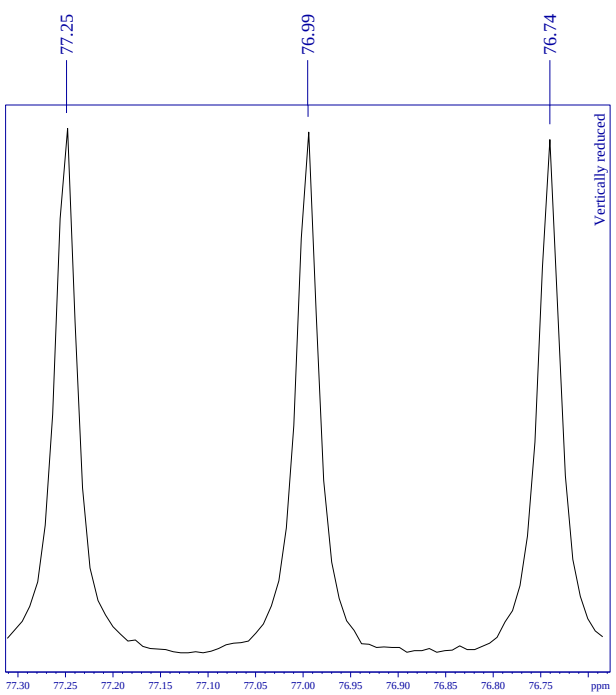
163.55
150.54
148.92
145.37
143.12
137.30
118.46
116.02
102.44
100.62
99.83
77.25
76.99
76.74
62.68
60.17
53.10



9b

NMR/50160929





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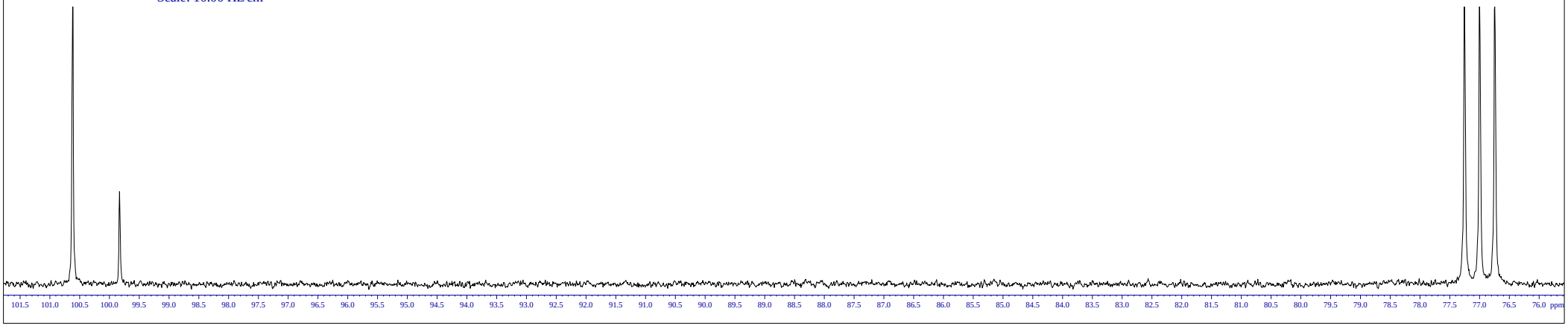
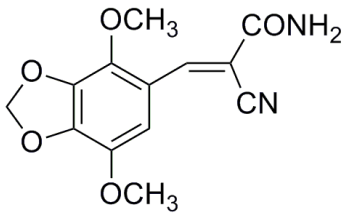


NMR/50160929

Peaks List

#	Address [points]	Frequency [Hz]	[ppm]	Intensity [cm]
1	9631.9	20567.262	163.5466	4.70
2	11271.4	18932.164	150.5446	3.11
3	11476.2	18727.938	148.9207	11.68
4	11924.4	18280.965	145.3665	4.66
5	12207.4	17998.686	143.1218	2.95
6	12941.5	17266.627	137.3006	2.66
7	15317.6	14896.847	118.4566	4.47
8	15625.5	14589.840	116.0154	4.48
9	17336.7	12883.226	102.4447	10.43
10	17567.2	12653.296	100.6164	13.32
11	17666.8	12554.009	99.8269	3.50
12	20513.8	9714.655	77.2489	14.00
13	20545.9	9682.691	76.9948	13.84
14	20577.9	9650.705	76.7404	13.60
15	22350.6	7882.845	62.6828	6.43
16	22667.0	7567.260	60.1733	5.45
17	23559.1	6677.571	53.0987	7.32

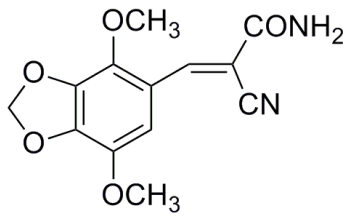
/KVEN 741



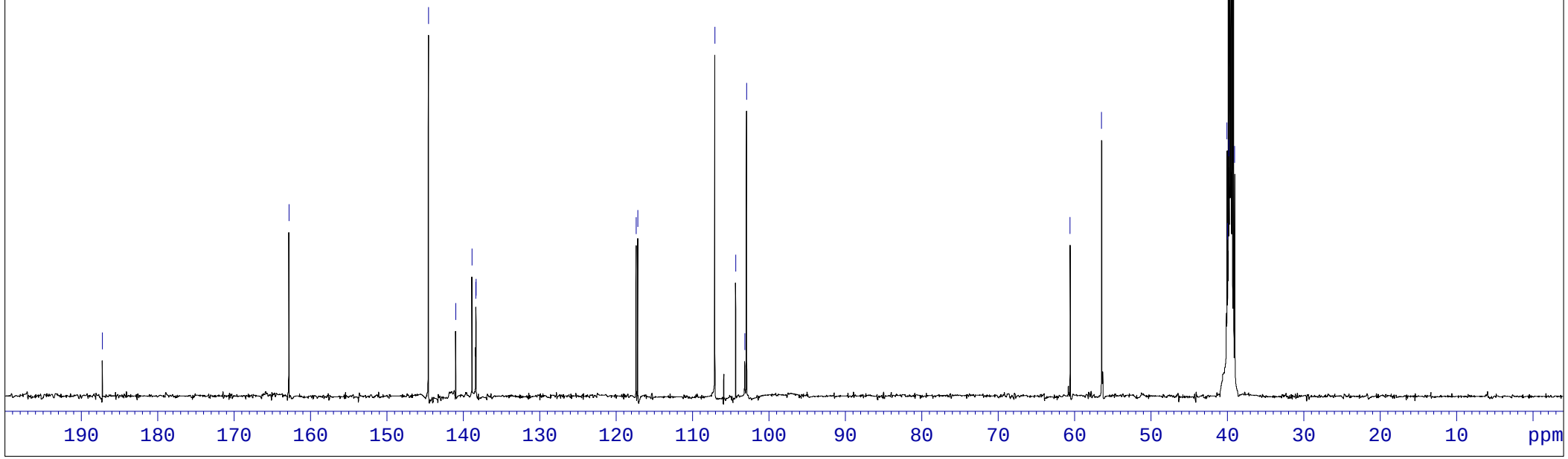
9c

NMR/50164193

187.24
162.81
144.56
140.99
138.86
138.37
138.33
117.38
117.16
107.07
104.35
103.16
102.92
60.60
56.48
40.06
39.99
39.90
39.82
39.73
39.56
39.40
39.23
39.06

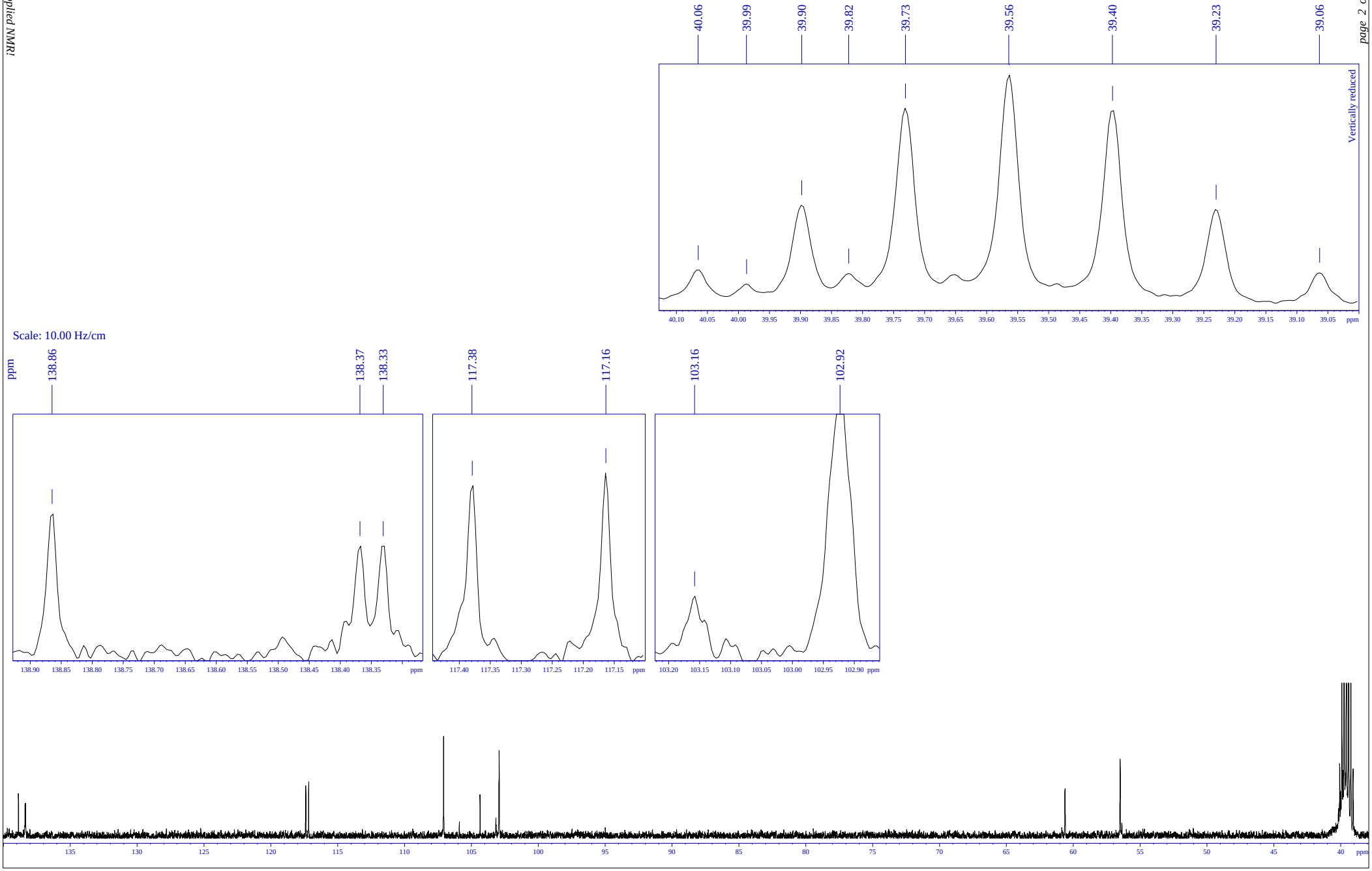


9c



The Best Applied NMR!

NMR/50164193

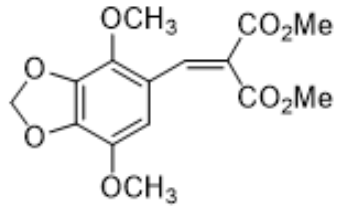


NMR/50164193

Peaks List

#	Address [points]	Frequency [Hz]	[ppm]	Intensity [cm]
1	11213.8	23547.076	187.2414	1.15
2	17616.0	20475.053	162.8133	3.85
3	22401.0	18179.061	144.5561	7.40
4	23336.1	17730.357	140.9881	1.93
5	23892.6	17463.328	138.8647	2.94
6	24022.7	17400.896	138.3683	2.25
7	24032.5	17396.184	138.3308	2.29
8	29523.6	14761.347	117.3791	3.51
9	29580.1	14734.227	117.1635	3.71
10	32224.6	13465.333	107.0735	6.90
11	32939.2	13122.434	104.3468	2.83
12	33250.7	12972.942	103.1581	1.22
13	33312.3	12943.384	102.9231	5.90
14	44404.0	7621.224	60.6024	3.23
15	45483.7	7103.148	56.4827	5.29
16	49786.5	5038.468	40.0648	4.99
17	49807.0	5028.671	39.9869	3.02
18	49830.3	5017.484	39.8980	13.98
19	49850.1	5007.957	39.8222	4.49
20	49874.1	4996.469	39.7309	27.38
21	49917.8	4975.475	39.5639	32.00
22	49961.6	4954.489	39.3971	27.24
23	50005.3	4933.498	39.2301	13.39
24	50049.0	4912.517	39.0633	4.58

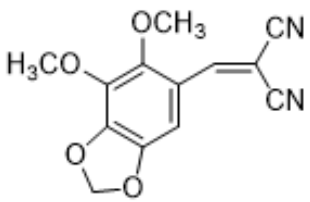
/KVEN 770



9d

The Best Applied NMR!

NMR/50160120

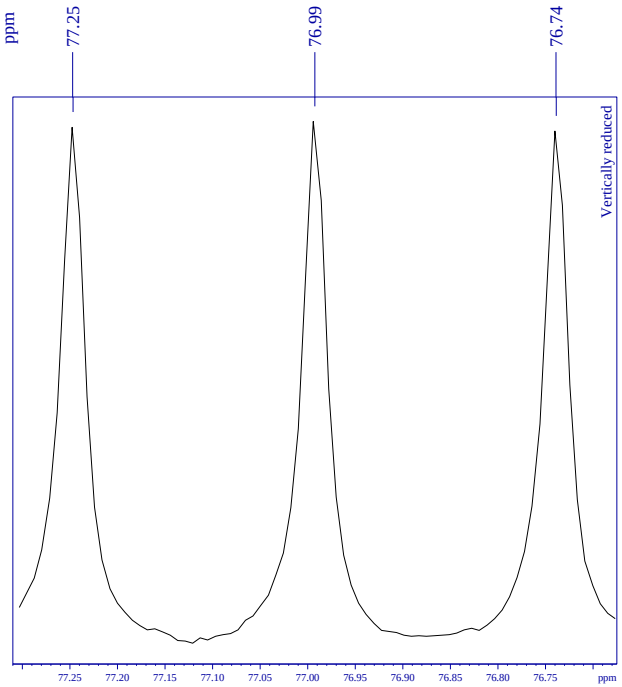


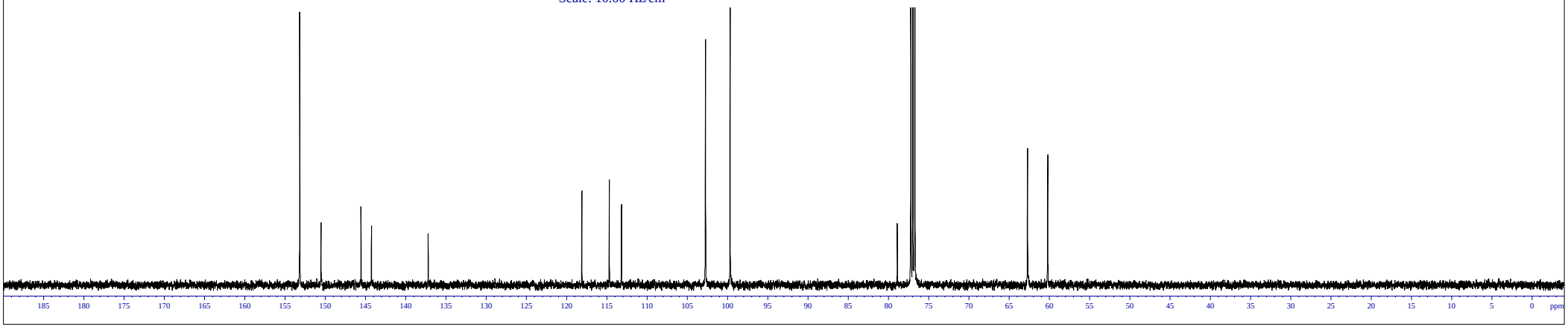
10a

153.15
150.55
145.58
144.29
137.24
118.13
114.72
113.22
102.77
99.71
78.93
77.25
76.99
76.74
62.74
60.23

180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

NMR/50160120



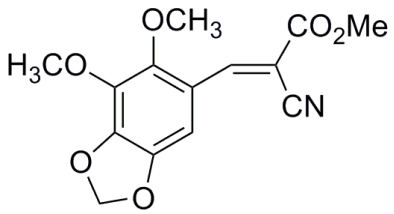


NMR/50160120

Peaks List

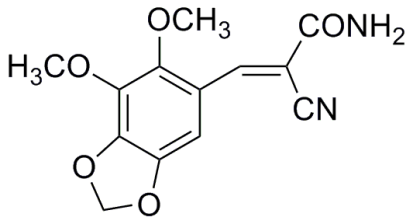
#	Address [points]	Frequency [Hz]	[ppm]	Intensity [cm]
1	10942.8	19259.920	153.1509	10.37
2	11270.5	18933.055	150.5517	2.46
3	11896.9	18308.412	145.5847	2.97
4	12059.9	18145.758	144.2913	2.23
5	12949.6	17258.496	137.2360	2.01
6	15359.2	14855.390	118.1270	3.59
7	15788.6	14427.120	114.7215	4.13
8	15978.6	14237.666	113.2150	3.19
9	17295.9	12923.912	102.7683	9.24
10	17681.7	12539.111	99.7084	11.61
11	20301.8	9926.056	78.9299	2.33
12	20514.1	9714.381	77.2467	16.76
13	20546.2	9682.402	76.9925	17.00
14	20578.2	9650.485	76.7387	16.70
15	22343.7	7889.657	62.7369	5.17
16	22660.1	7574.160	60.2281	4.89

/KVEN 778



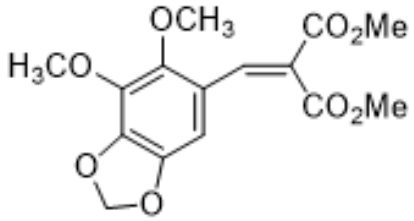
10b

/KVEN 769



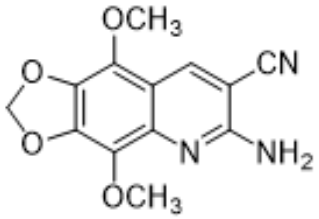
10c

/KVEN 762



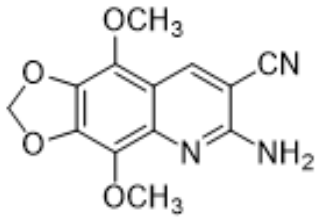
10d

/KVEN 737



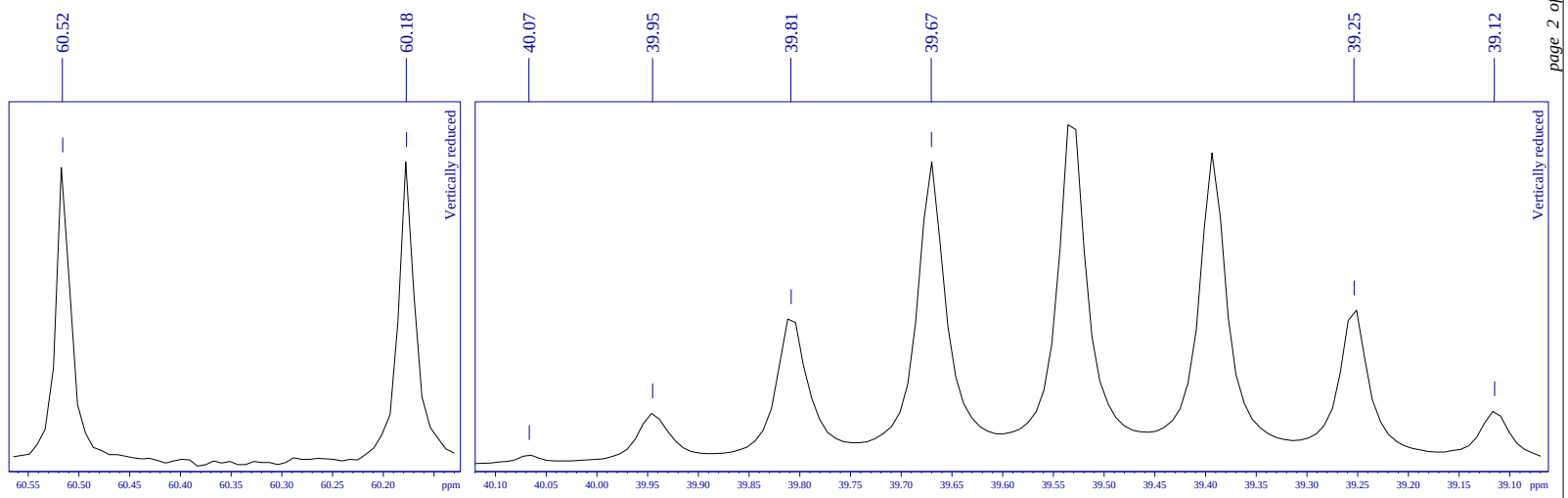
11

NMR/50164187

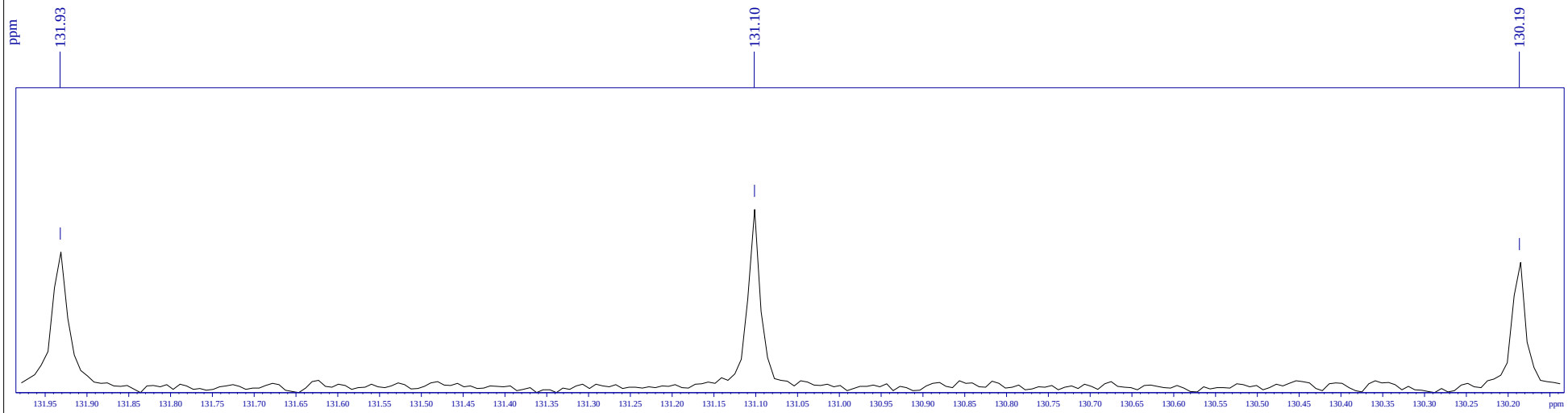


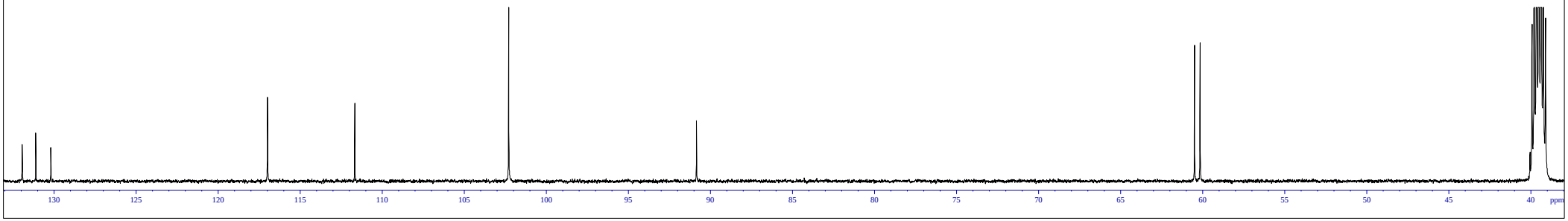
11

NMR/50164187



Scale: 11.00 Hz/cm



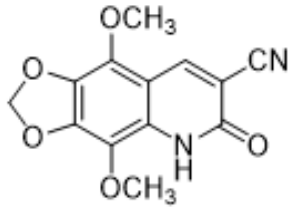


NMR/50164187

Peaks List

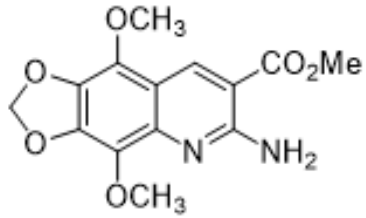
#	Address [points]	Frequency [Hz]	[ppm]	Intensity [cm]
1	11760.3	23415.461	155.1691	5.04
2	13153.2	21755.043	144.1659	2.85
3	13503.9	21336.938	141.3952	3.47
4	13920.1	20840.777	138.1072	9.64
5	14701.9	19908.906	131.9319	2.18
6	14807.0	19783.596	131.1015	2.84
7	14922.8	19645.521	130.1865	2.03
8	16594.9	17652.246	116.9775	5.00
9	17267.0	16850.963	111.6676	4.62
10	18454.9	15434.889	102.2836	11.46
11	19903.6	13707.958	90.8396	3.81
12	23742.1	9132.041	60.5160	8.11
13	23785.0	9080.896	60.1771	8.18
14	26330.8	6046.176	40.0667	1.74
15	26346.1	6027.829	39.9451	9.29
16	26363.4	6007.216	39.8085	27.16
17	26380.9	5986.355	39.6702	54.82
18	26433.7	5923.472	39.2535	28.42
19	26451.2	5902.566	39.1150	9.72

/KVEN 785



12

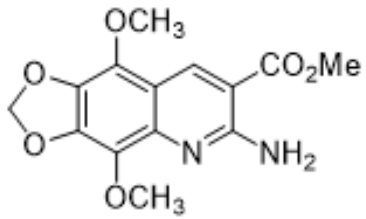
/KVEN 782



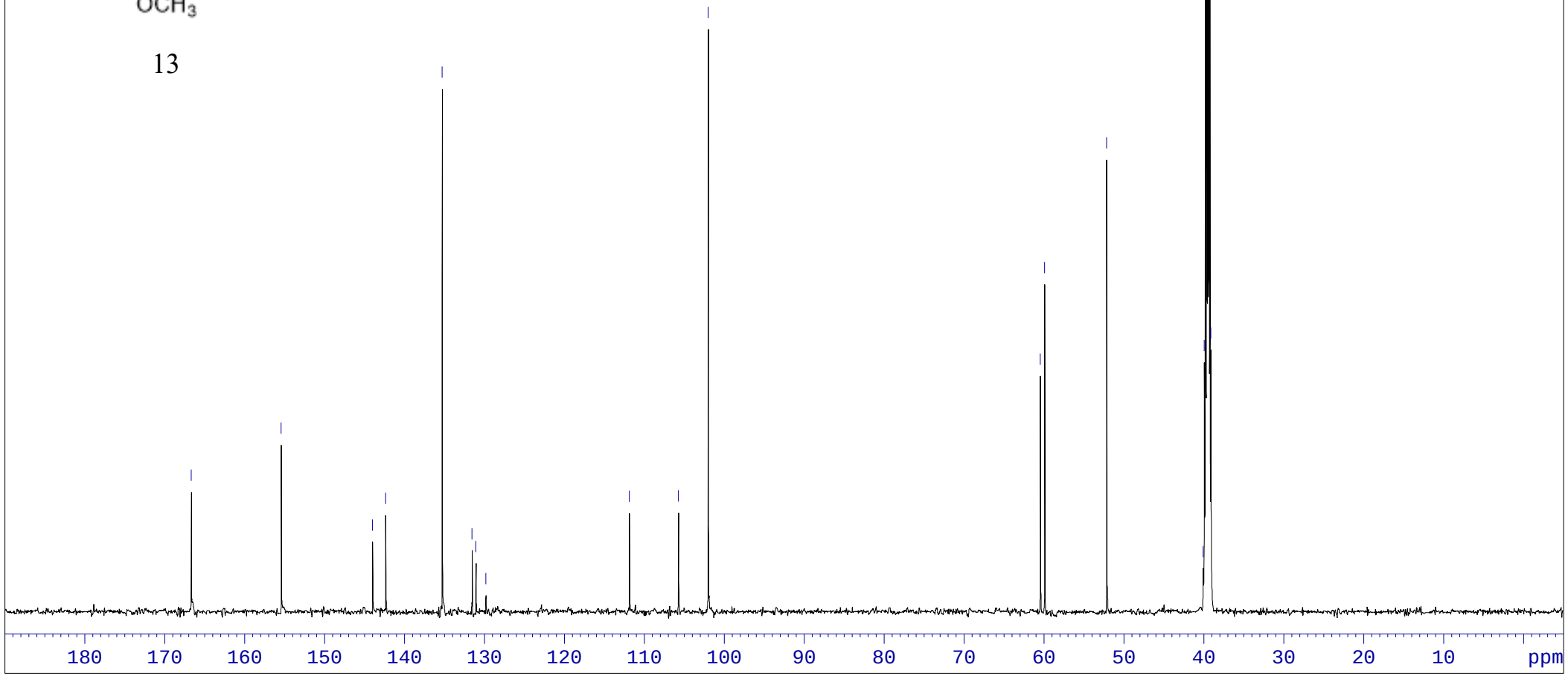
13

NMR/50164191

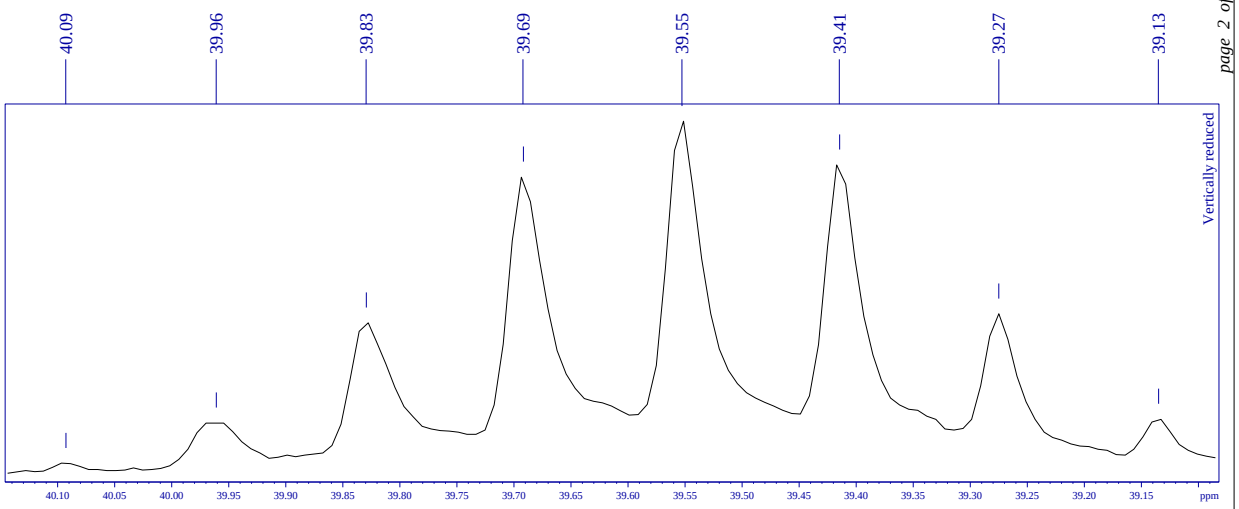
166.69 155.45 144.01 142.37 135.30 131.57 131.09 129.83 111.88 105.74 102.02 60.47 59.93 52.17 40.09 39.96 39.83 39.69 39.55 39.41 39.27 39.13



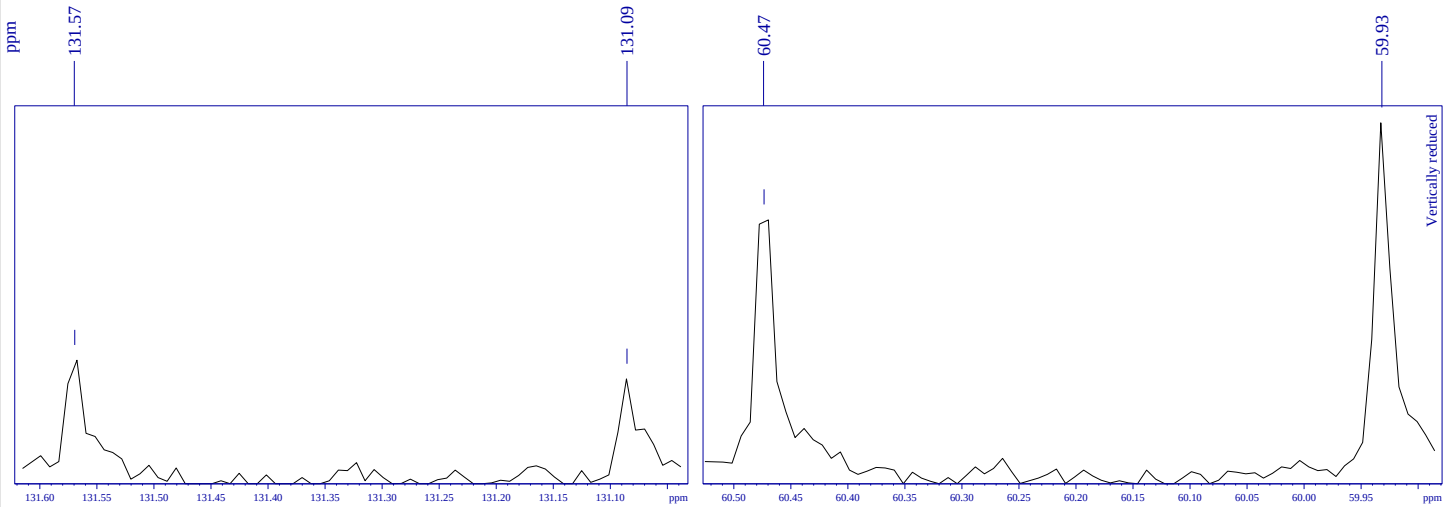
13

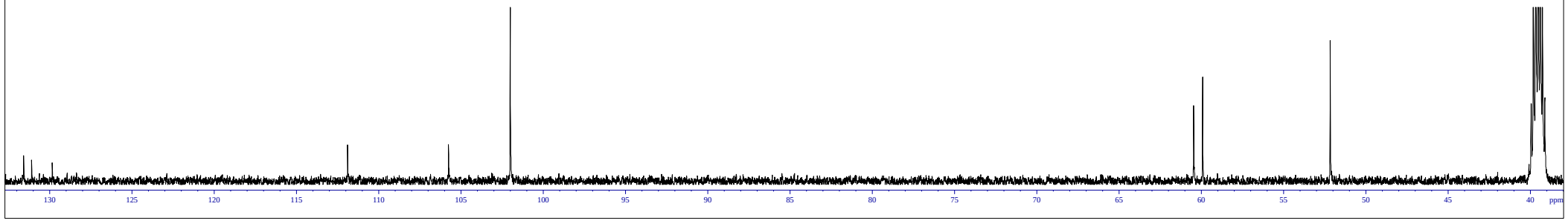


NMR/50164191



Scale: 10.00 Hz/cm



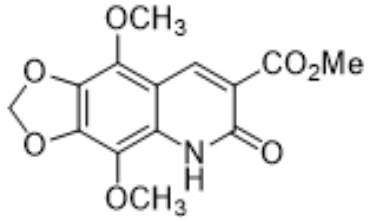


NMR/50164191

Peaks List

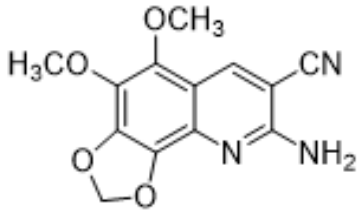
#	Address [points]	Frequency [Hz]	[ppm]	Intensity [cm]
1	10301.8	25154.225	166.6915	2.65
2	11724.9	23457.654	155.4487	3.42
3	13172.9	21731.543	144.0101	1.70
4	13380.7	21483.846	142.3687	2.25
5	14275.0	20417.721	135.3037	9.73
6	14747.7	19854.197	131.5694	1.57
7	14809.0	19781.160	131.0854	1.28
8	14967.7	19591.982	129.8317	1.14
9	17239.6	16883.633	111.8841	2.28
10	18017.2	15956.706	105.7416	2.23
11	18488.3	15395.115	102.0200	11.08
12	23747.5	9125.620	60.4735	4.87
13	23816.1	9043.875	59.9318	6.28
14	24798.0	7873.312	52.1747	8.47
15	26327.5	6050.108	40.0927	1.06
16	26344.1	6030.220	39.9609	4.67
17	26360.8	6010.369	39.8294	13.74
18	26378.2	5989.586	39.6917	27.03
19	26395.8	5968.616	39.5527	32.00
20	26413.3	5947.759	39.4145	28.36
21	26431.0	5926.713	39.2750	14.49
22	26448.7	5905.575	39.1349	5.07

/KVEN 784



15

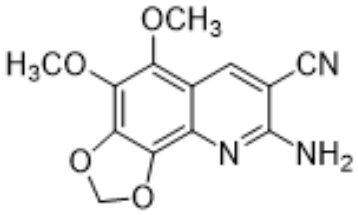
/KVEN 754



16a

NMR/50164184

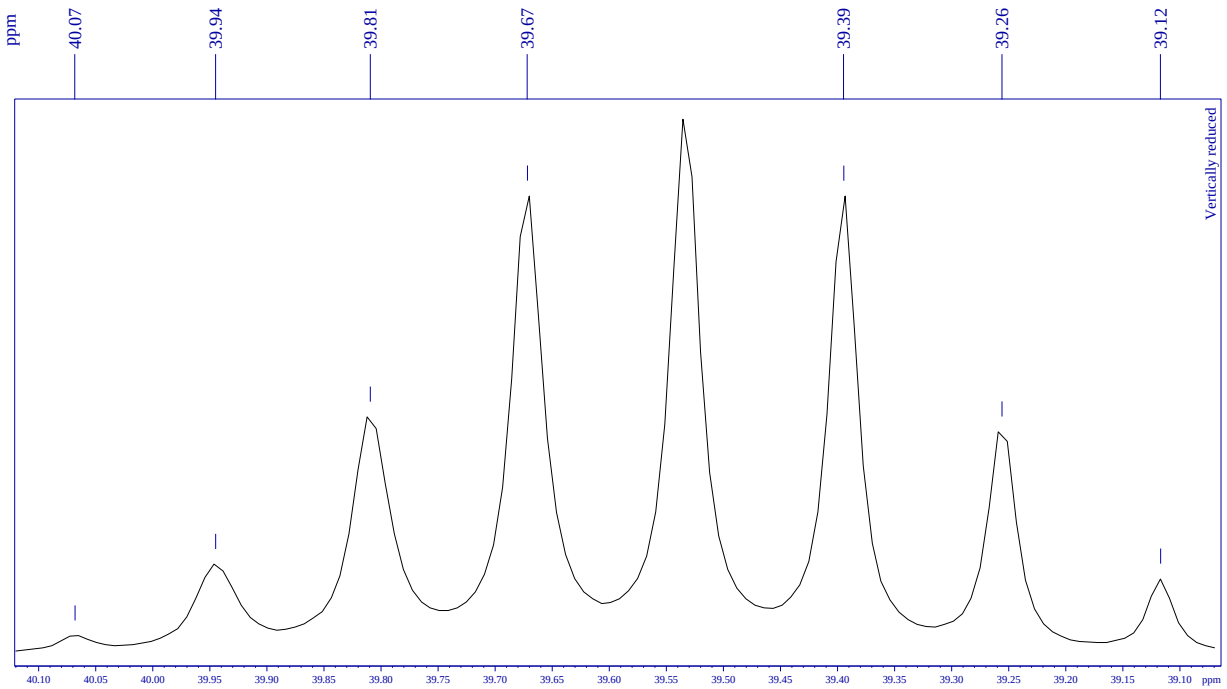
155.18
143.25
142.14
140.21
135.43
133.44
130.67
116.82
111.91
102.50
92.78
62.28
60.62
40.07
39.94
39.81
39.67
39.39
39.26
39.12

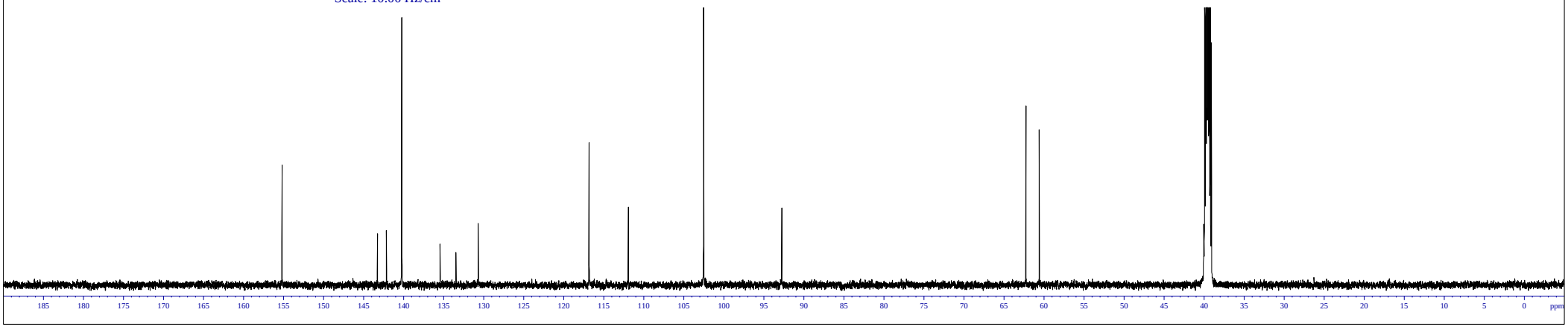


16a

180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

NMR/50164184



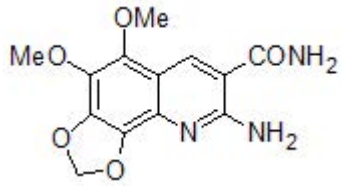


NMR/50164184

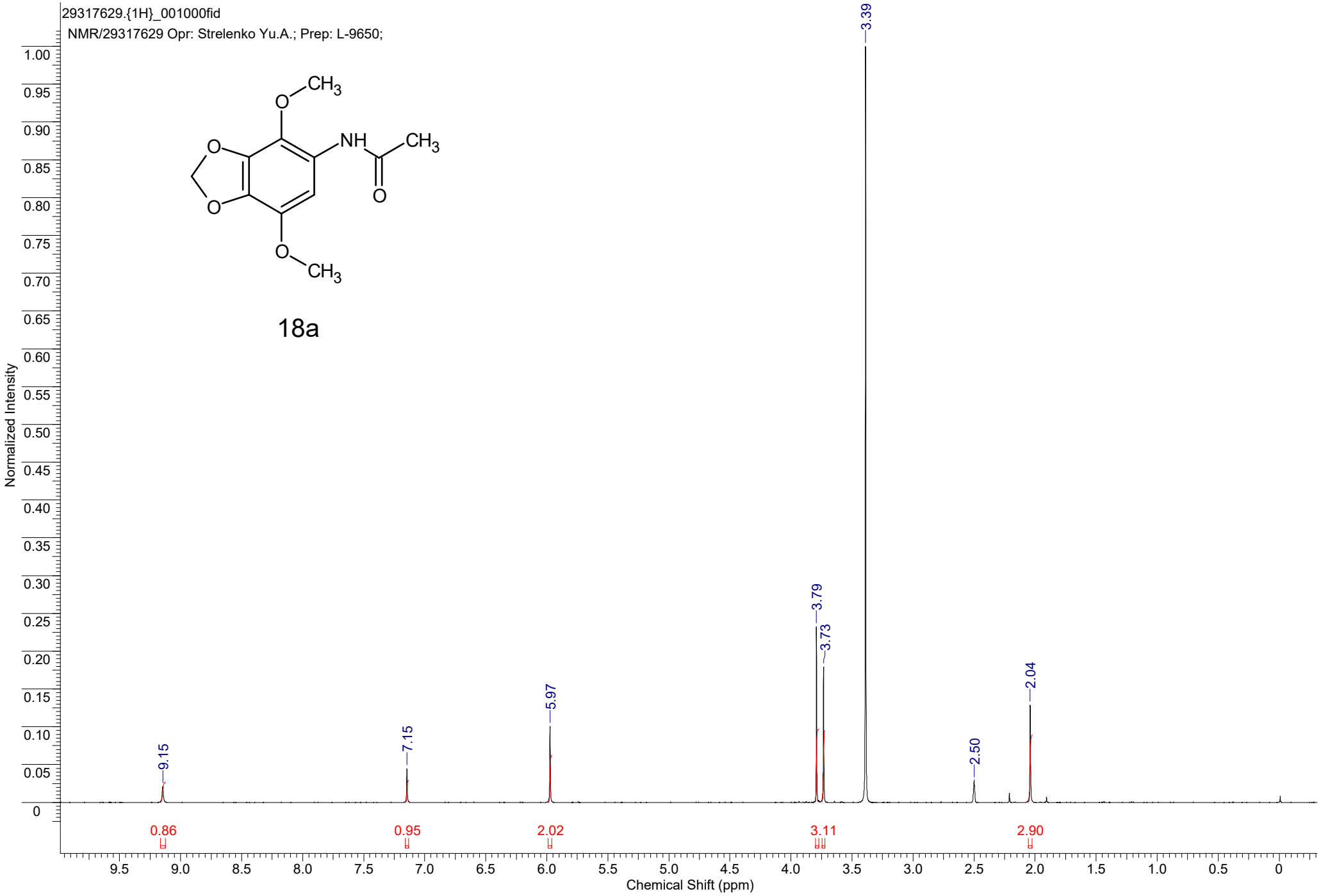
Peaks List

#	Address [points]	Frequency [Hz]	[ppm]	Intensity [cm]
1	11759.2	23416.830	155.1782	4.52
2	13268.7	21617.328	143.2533	1.97
3	13410.3	21448.588	142.1351	2.10
4	13653.4	21158.779	140.2146	10.35
5	14259.6	20436.121	135.4257	1.63
6	14510.5	20137.039	133.4437	1.34
7	14861.5	19718.623	130.6710	2.48
8	16615.0	17628.301	116.8189	5.28
9	17236.7	16887.186	111.9076	3.02
10	18427.2	15467.973	102.5028	12.34
11	19658.0	14000.717	92.7797	2.86
12	23518.7	9398.367	62.2809	6.87
13	23729.0	9147.683	60.6197	5.75
14	26330.6	6046.386	40.0681	2.30
15	26346.2	6027.806	39.9449	10.75
16	26363.3	6007.349	39.8094	28.54
17	26380.8	5986.566	39.6717	54.87
18	26415.8	5944.746	39.3945	54.59
19	26433.4	5923.826	39.2559	27.14
20	26451.0	5902.857	39.1169	8.98

/KVEN 777

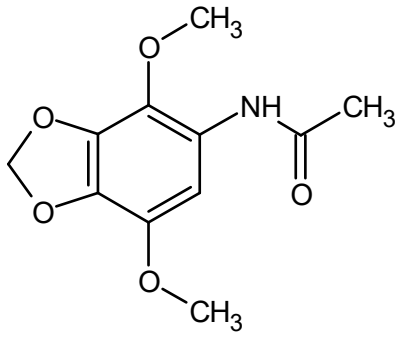


16c

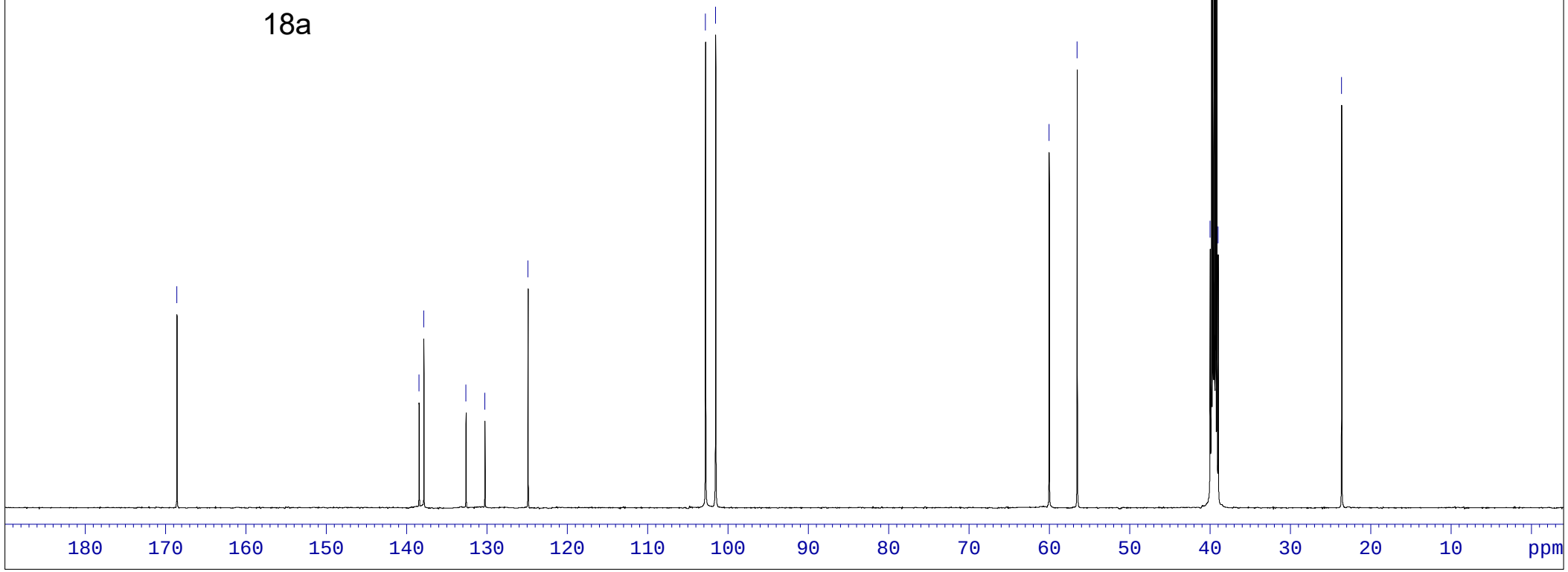

29317629.{1H}_001000fid
NMR/29317629 Opr: Strelenko Yu.A.; Prep: L-9650;
18a
Normalized Intensity
Chemical Shift (ppm)
3.39
3.79
3.73
2.50
2.04
9.15
7.15
5.97
0.86
0.95
2.02
3.11
2.90

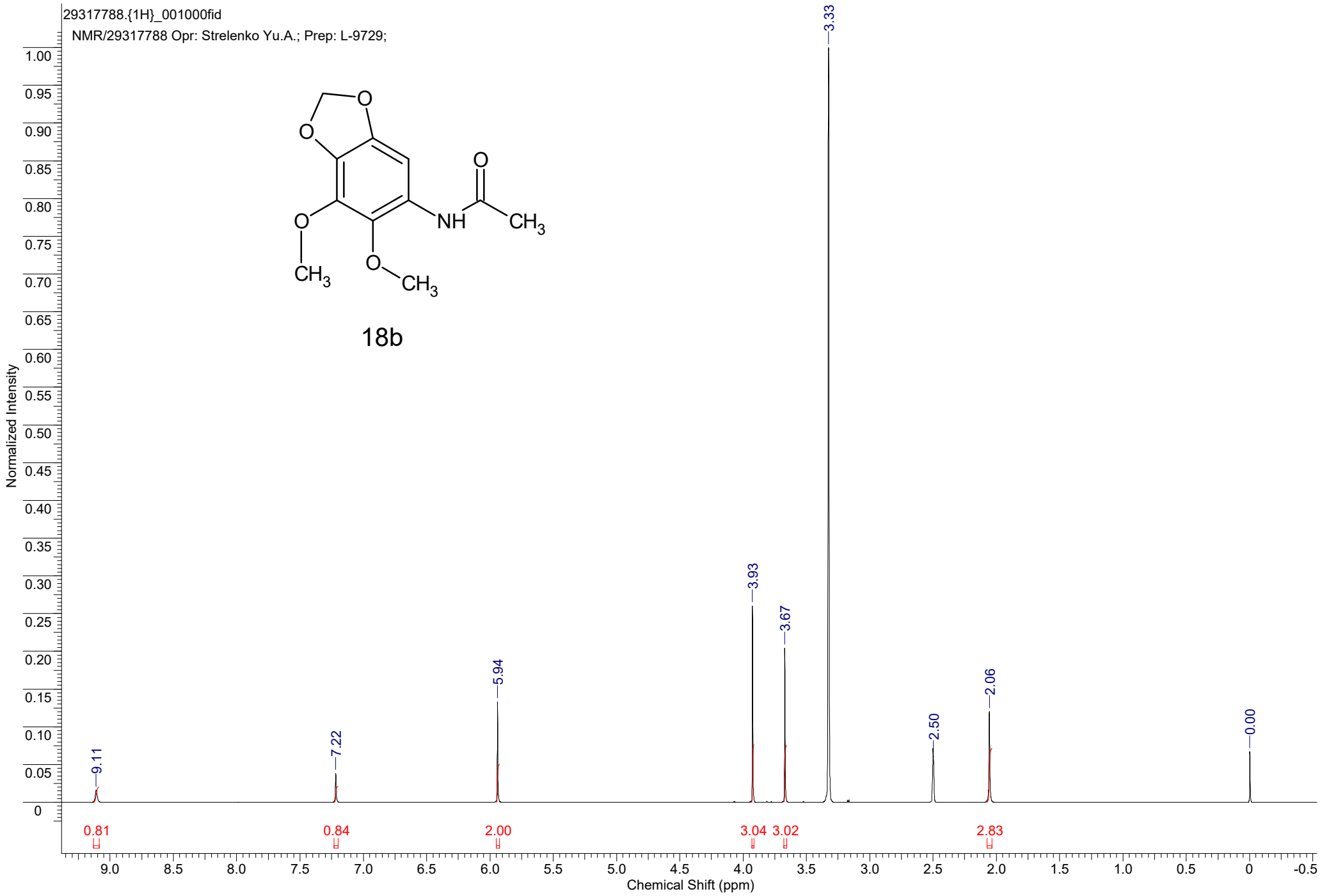
NMR/29317629

168.60
138.46
137.87
132.63
130.27
124.91
102.82
101.55
60.04
56.56
40.01
39.84
39.67
39.50
39.34
39.17
39.00
23.64



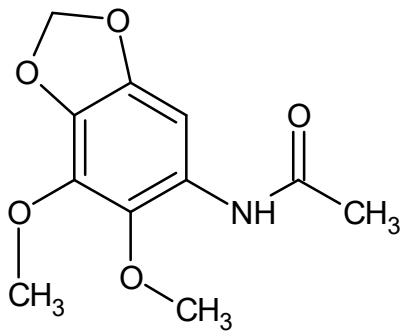
18a




29317788.{1H}_001000fid
NMR/29317788 Opr: Strelenko Yu.A.; Prep: L-9729;
18b
Normalized Intensity
Chemical Shift (ppm)
9.11
7.22
5.94
3.93
3.67
3.33
2.50
2.06
0.00
0.81
0.84
2.00
3.04
3.02
2.83

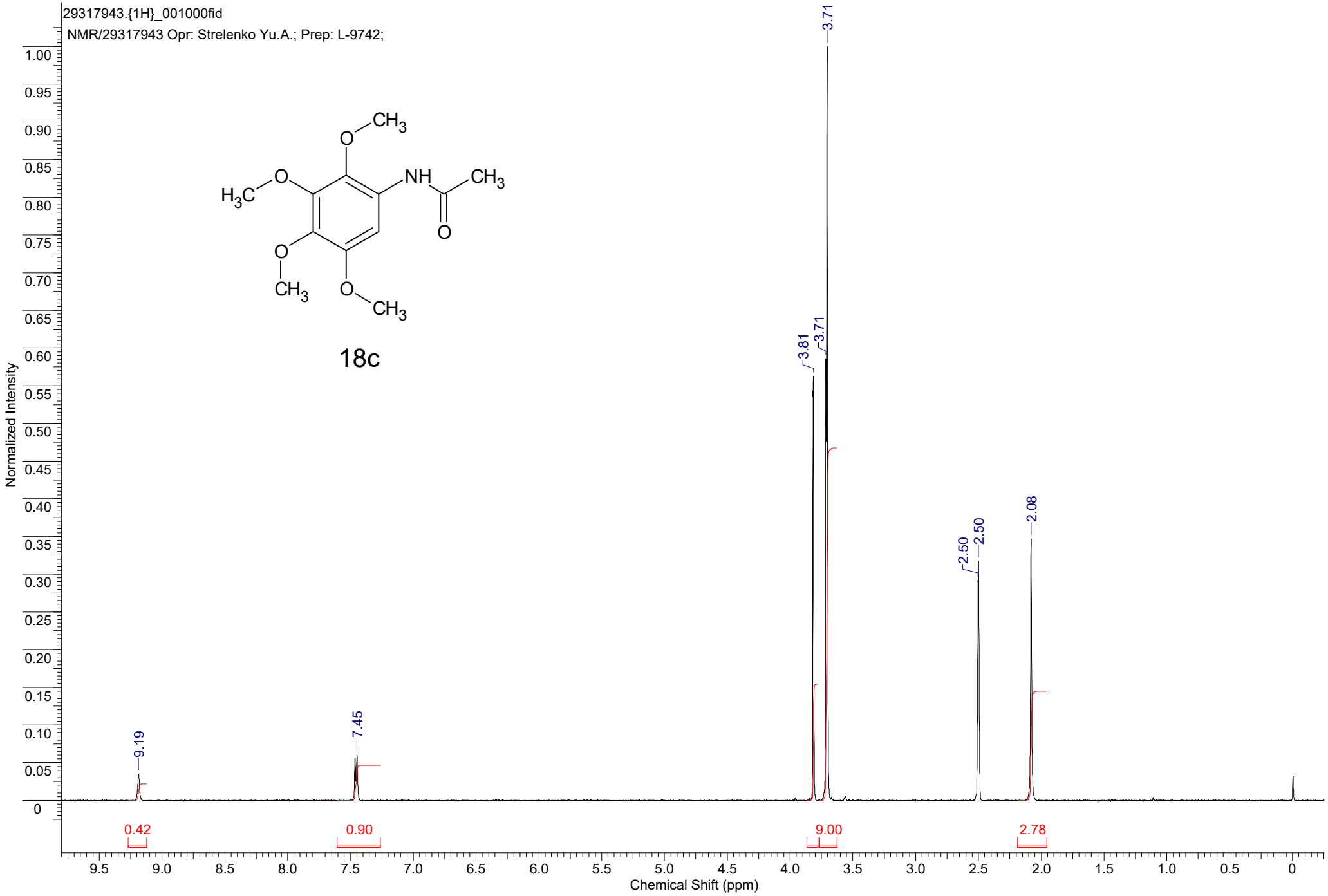
NMR/29317788

168.73
143.54
137.14
137.02
133.51
125.60
101.20
97.54
60.97
59.83
40.02
39.86
39.69
39.52
39.36
39.19
39.02
23.73



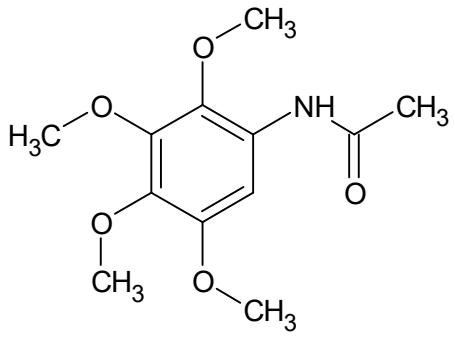
18b

180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


29317943.{1H}_001000fid
NMR/29317943 Opr: Strelenko Yu.A.; Prep: L-9742;
18c
Normalized Intensity
Chemical Shift (ppm)
9.19
7.45
3.81
3.71
3.71
2.50
2.50
2.08
0.42
0.90
9.00
2.78

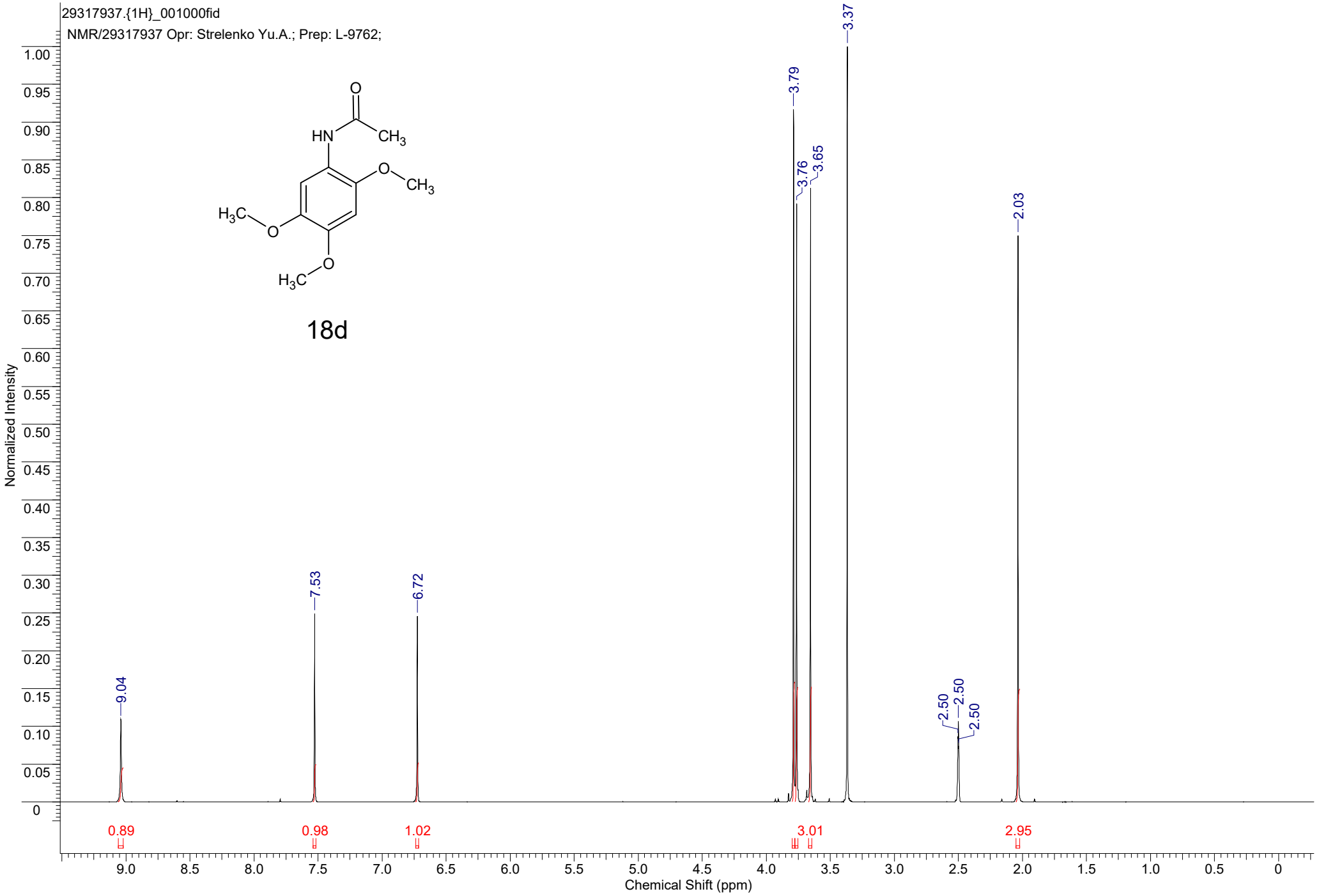
NMR/29317943

168.82
168.74
148.47
146.48
138.56
137.77
127.40
127.30
101.99
101.87
60.95
60.72
55.95
40.12
40.03
39.95
39.86
39.79
39.70
39.62
39.53
39.36
39.20
39.03
23.83



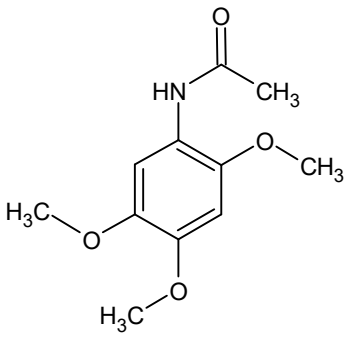
18c

180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


29317937.{1H}_001000fid
NMR/29317937 Opr: Strelenko Yu.A.; Prep: L-9762;
18d
Normalized Intensity
Chemical Shift (ppm)
9.04
7.53
6.72
3.79
3.76
3.65
3.37
2.50
2.50
2.50
2.03
0.89
0.98
1.02
3.01
2.95

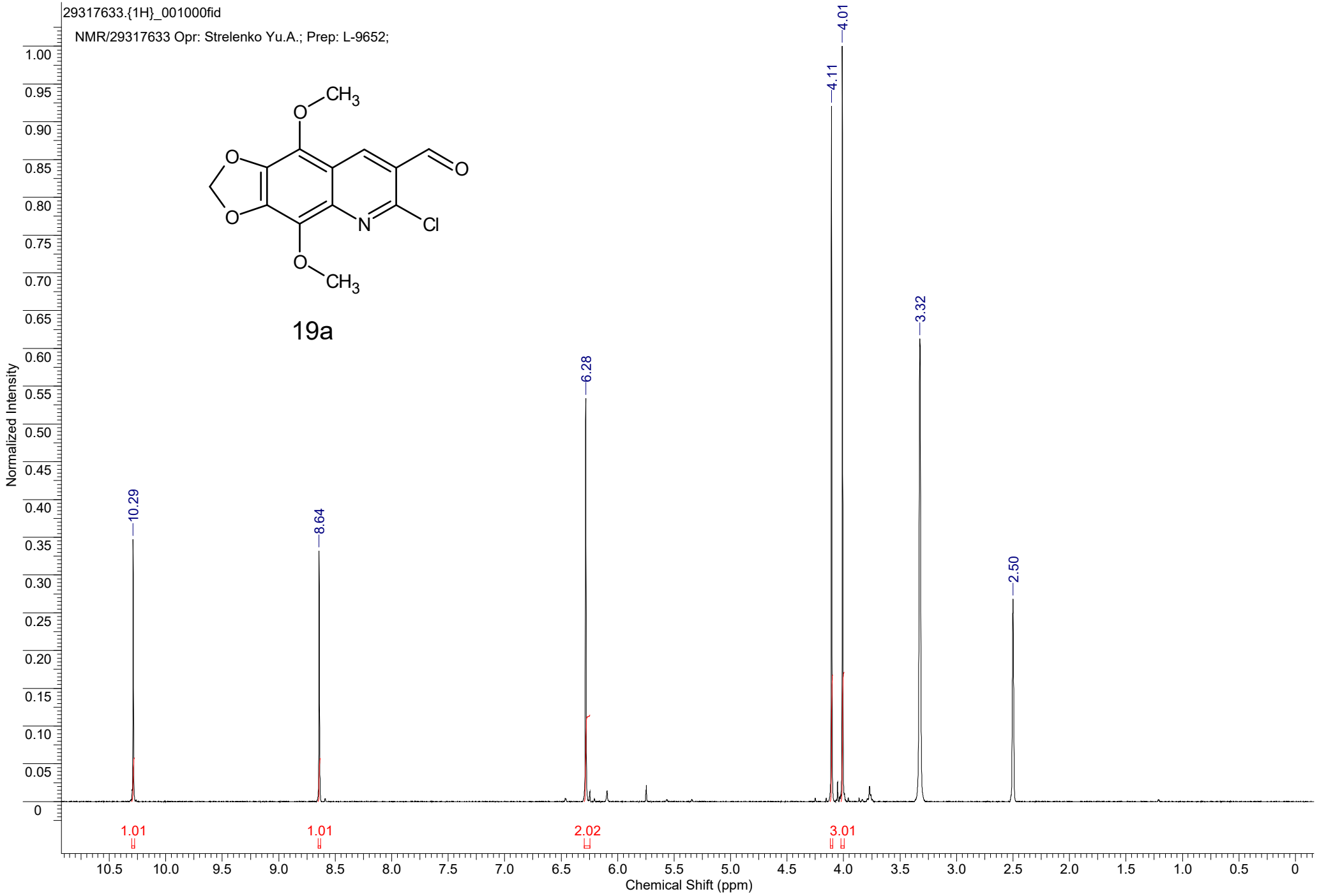
NMR/29317937

168.23
145.64
144.47
141.84
119.86
108.98
98.79
56.43
56.28
56.05
40.01
39.93
39.84
39.67
39.51
39.34
39.17
39.00
23.65



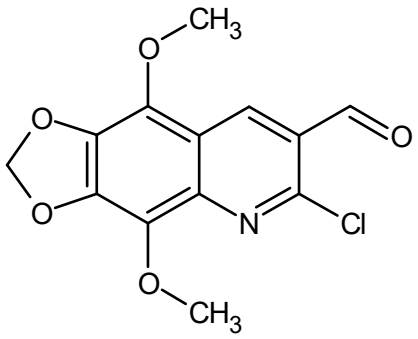
18d

180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


29317633.{1H}_001000fid
NMR/29317633 Opr: Strelenko Yu.A.; Prep: L-9652;
19a
Normalized Intensity
Chemical Shift (ppm)
10.29
8.64
6.28
4.11
4.01
3.32
2.50
1.01
1.01
2.02
3.01

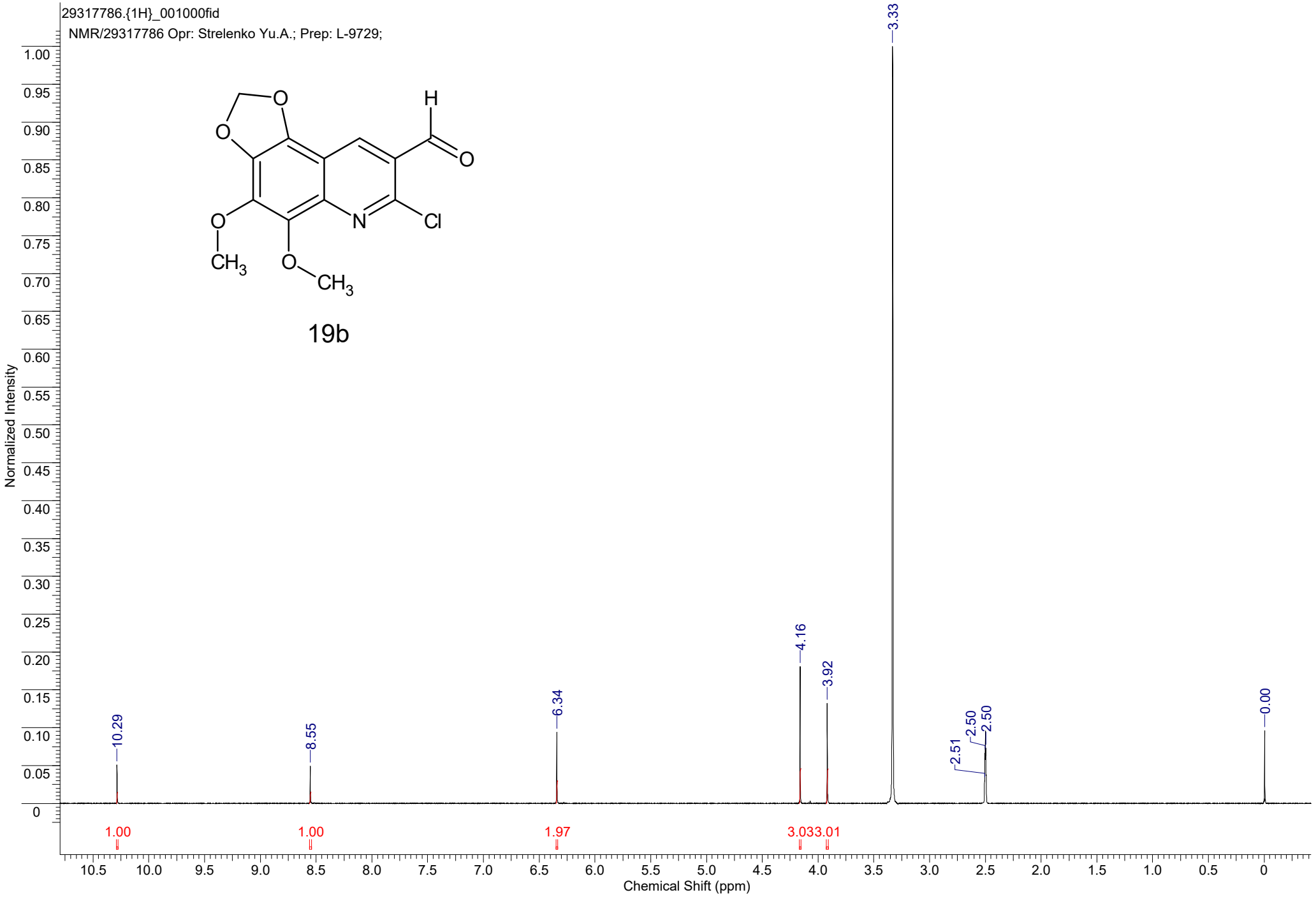
NMR/29317633

189.03
146.99
145.09
140.69
136.34
134.33
131.75
131.55
123.98
118.13
103.45
60.99
60.42
40.32
40.15
39.99
39.82
39.65
39.49
39.32



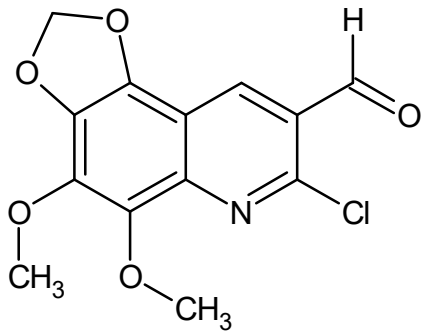
19a

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


29317786.{1H}_001000fid
NMR/29317786 Opr: Strelenko Yu.A.; Prep: L-9729;
O
O
H
O
O
N
Cl
CH3
O
CH3
19b
Normalized Intensity
Chemical Shift (ppm)
10.29
8.55
6.34
4.16
3.92
3.33
2.51
2.50
2.50
0.00
1.00
1.00
1.97
3.03
3.01

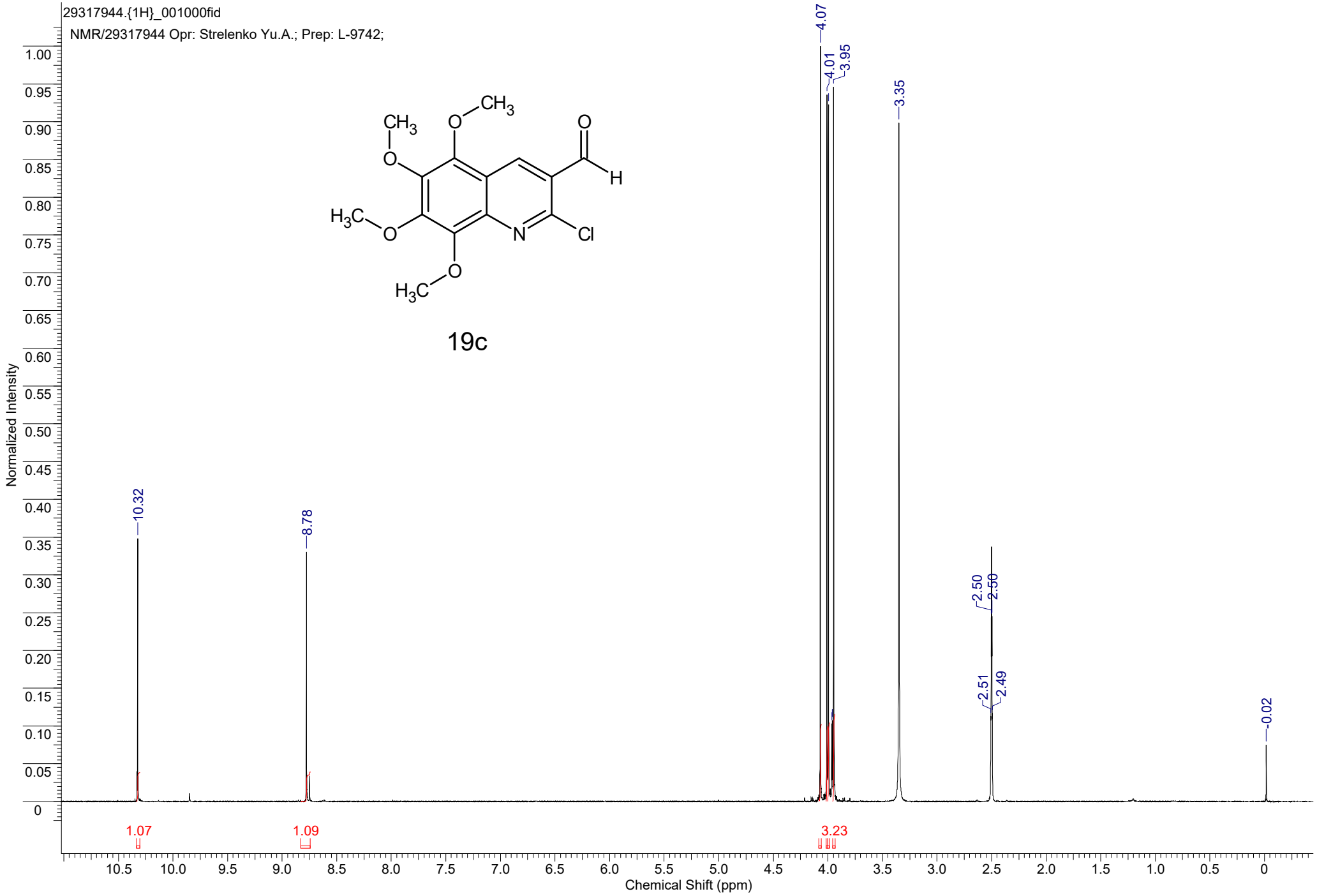
NMR/29317786

188.82
146.39
143.40
139.73
139.53
137.40
137.01
133.72
124.30
107.72
103.54
62.10
60.52
40.04
39.87
39.80
39.71
39.54
39.37
39.21
39.04



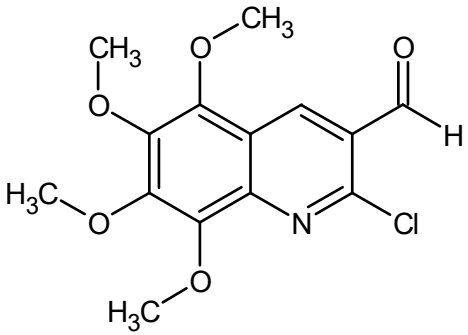
19b

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


29317944.{1H}_001000fid
NMR/29317944 Opr: Strelenko Yu.A.; Prep: L-9742;
19c
Normalized Intensity
Chemical Shift (ppm)
10.32
8.78
4.07
4.01
3.95
3.35
2.50
2.50
2.51
2.49
0.02
1.07
1.09
3.23

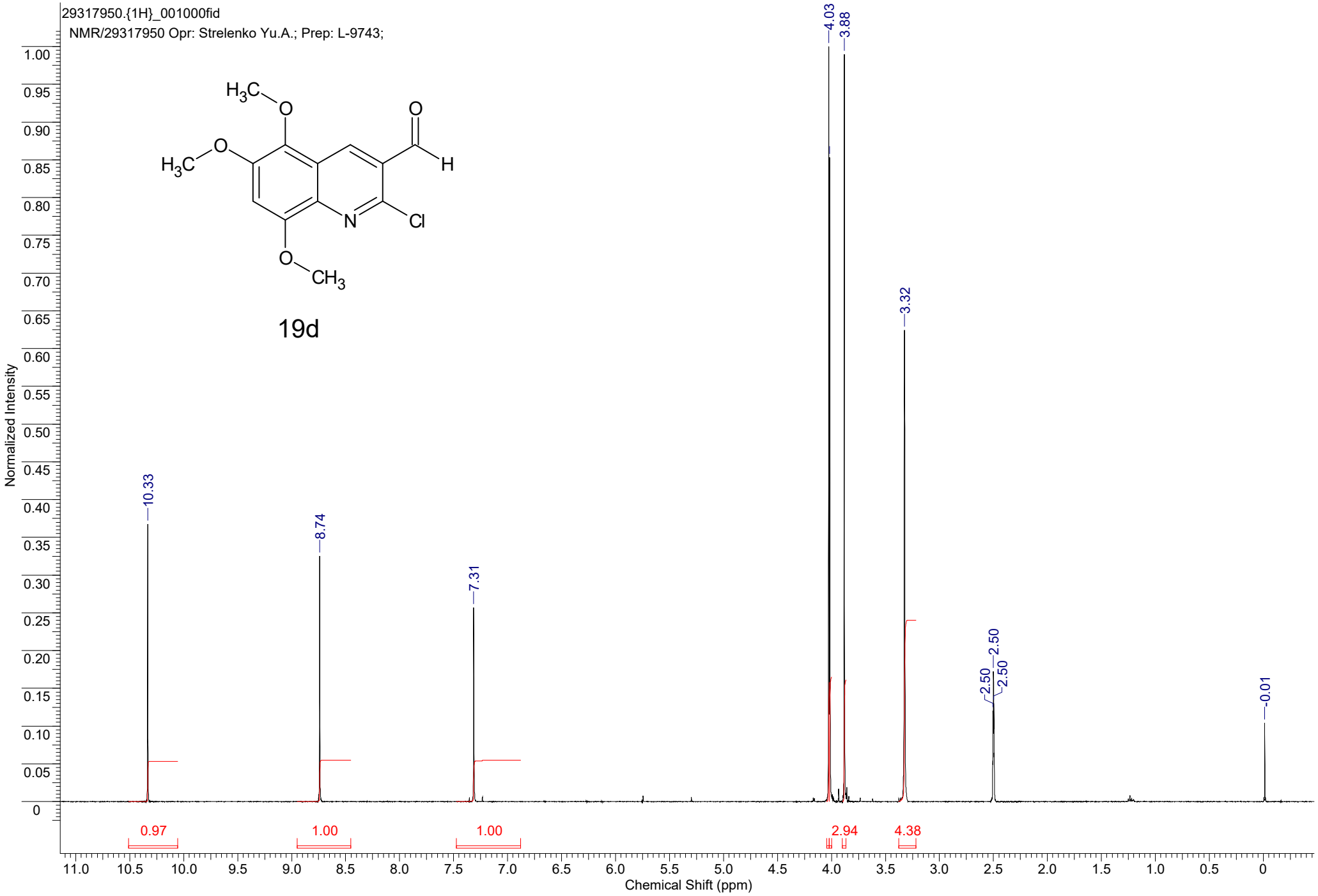
NMR/29317944

189.30
189.20
151.90
147.81
144.78
144.17
142.68
140.22
135.67
135.56
124.90
118.43
118.15
62.16
62.09
61.63
61.41
61.31
40.13
40.04
39.96
39.87
39.79
39.70
39.62
39.54
39.37
39.20
39.04



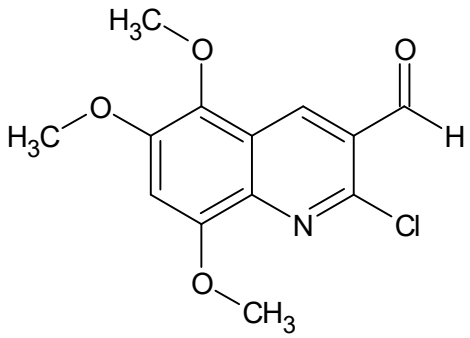
19c

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

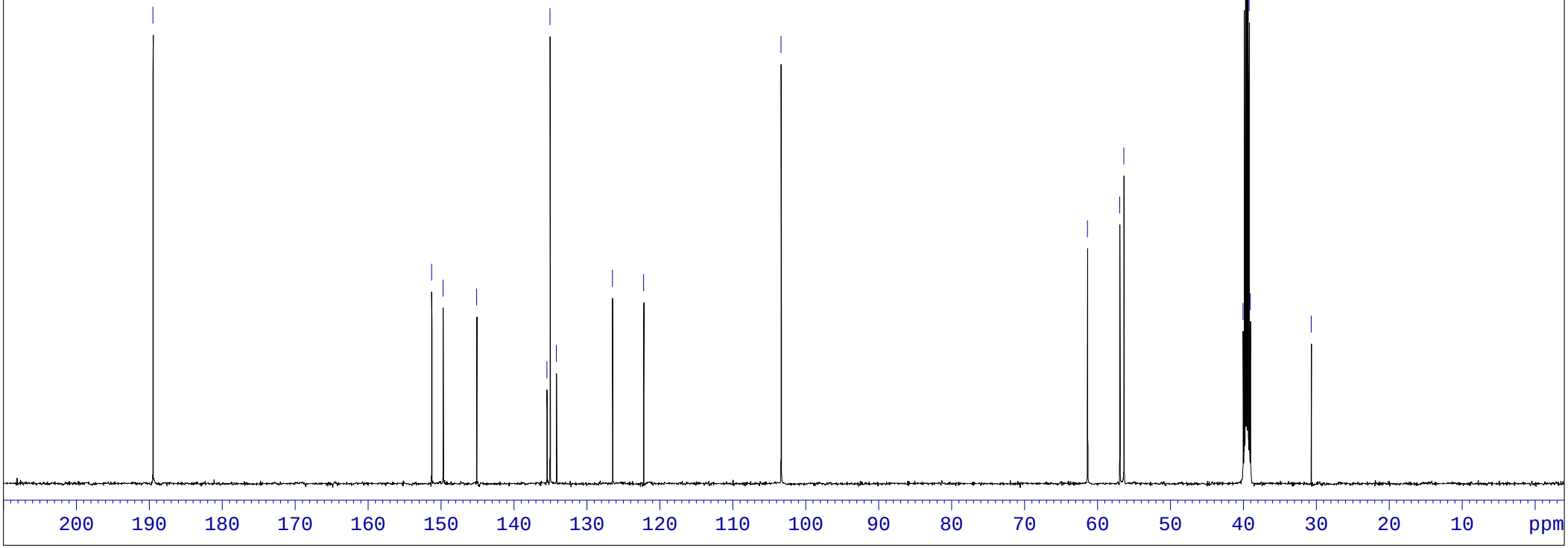

29317950.{1H}_001000fid
NMR/29317950 Opr: Strelenko Yu.A.; Prep: L-9743;
19d
Normalized Intensity
Chemical Shift (ppm)
10.33
8.74
7.31
4.03
3.88
3.32
2.50
2.50
2.50
-0.01
0.97
1.00
1.00
2.94
4.38

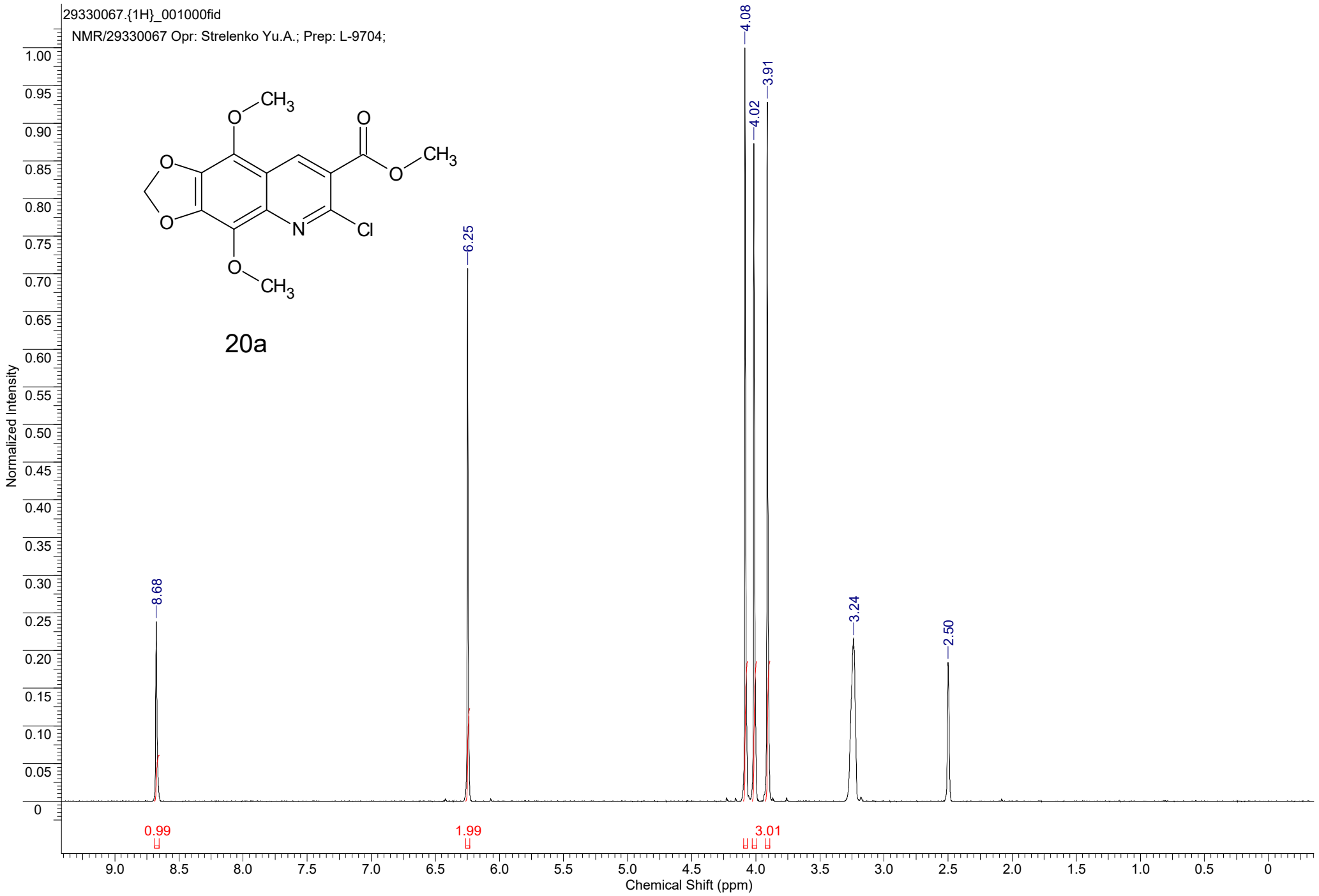
NMR/29317950

189.50
151.30
149.73
145.12
135.49
135.07
134.20
126.51
122.23
103.40
61.38
56.96
56.39
40.06
39.89
39.72
39.56
39.39
39.22
39.05
30.70



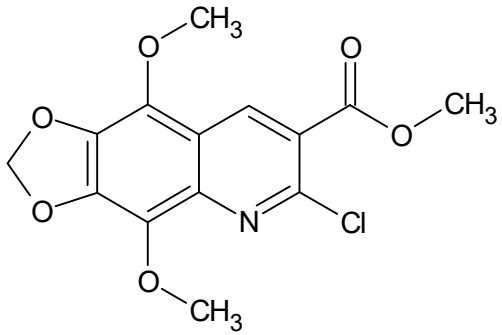
19d



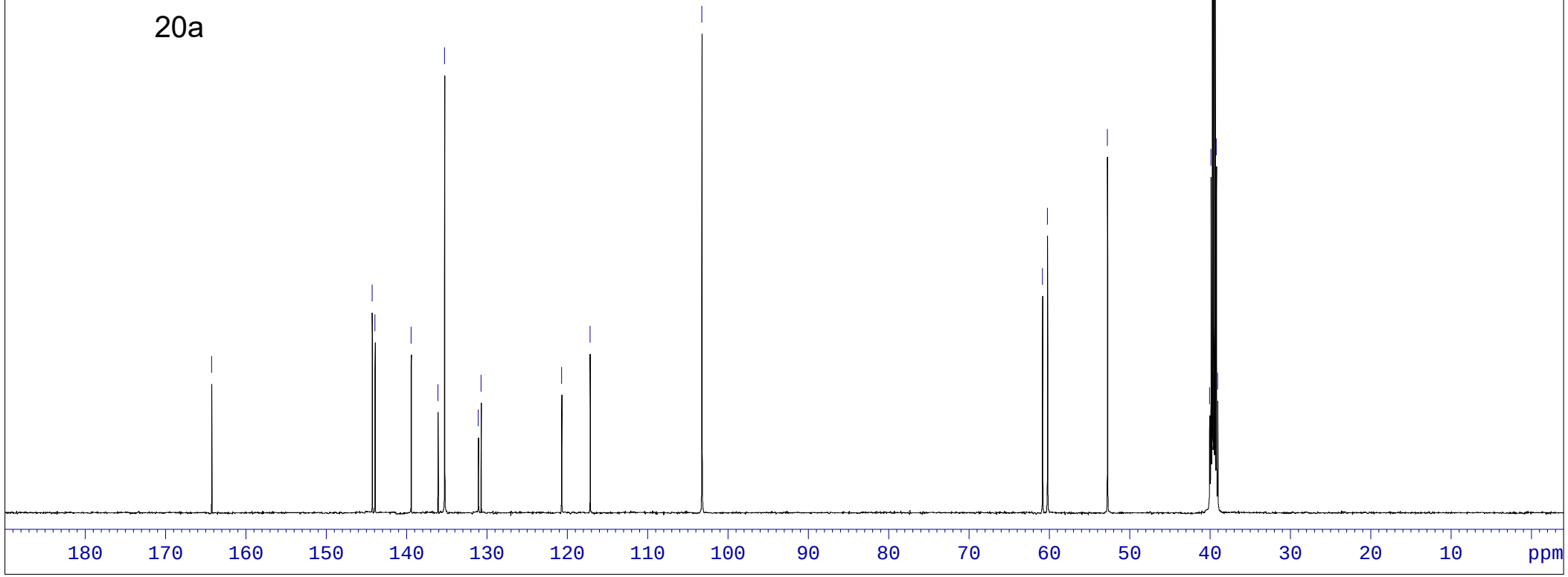

29330067.{1H}_001000fid
NMR/29330067 Opr: Strelenko Yu.A.; Prep: L-9704;
20a
8.68
6.25
4.08
4.02
3.91
3.24
2.50
0.99
1.99
3.01
Normalized Intensity
Chemical Shift (ppm)

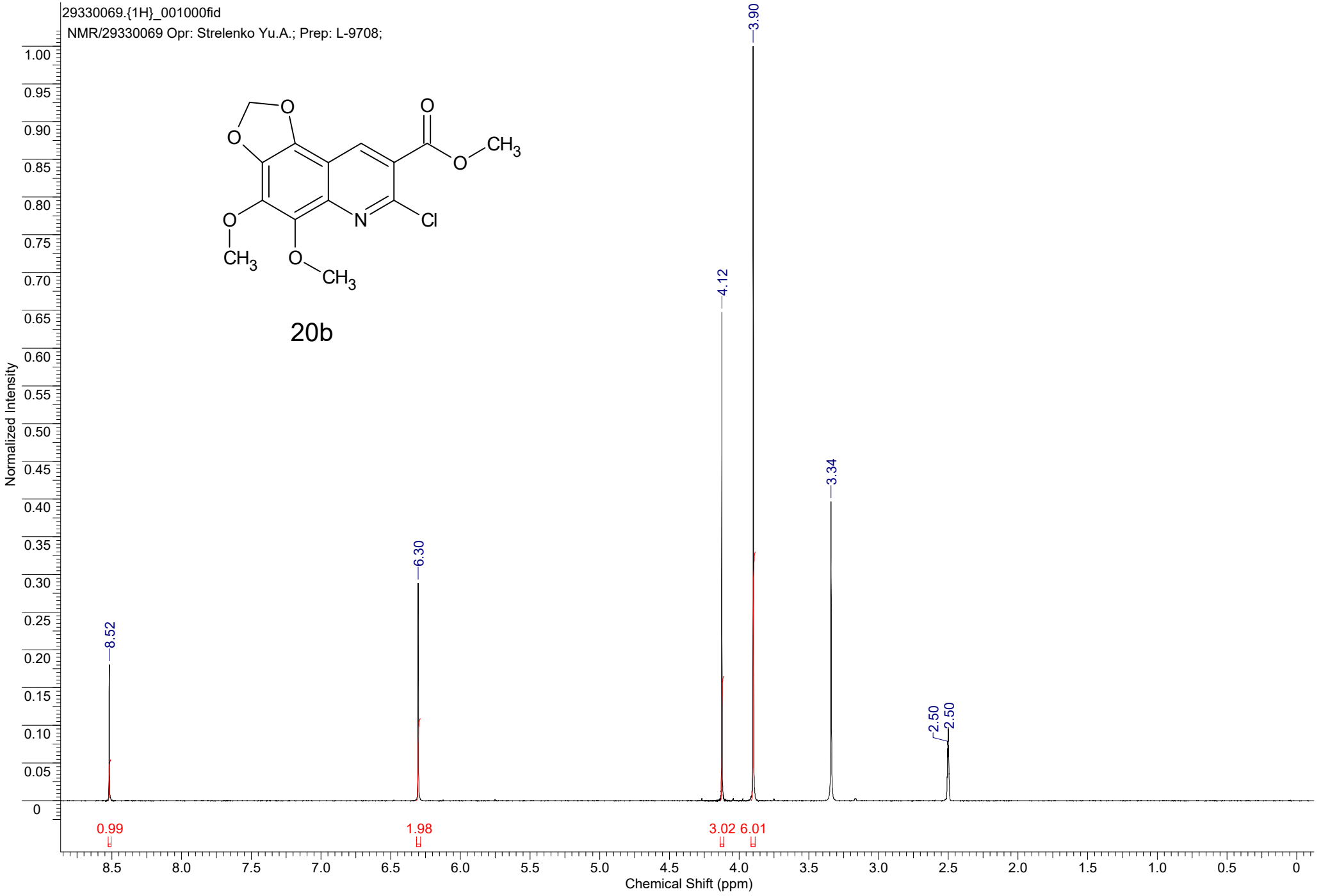
NMR/29330067

164.27
144.29
143.95
139.45
136.11
135.29
131.09
130.74
120.71
117.17
103.26
60.87
60.25
52.79
40.07
39.90
39.73
39.57
39.40
39.23
39.06



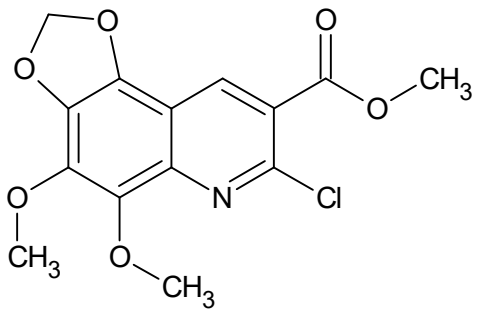
20a




29330069.{1H}_001000fid
NMR/29330069 Opr: Strelenko Yu.A.; Prep: L-9708;
20b
Normalized Intensity
Chemical Shift (ppm)
8.52
6.30
4.12
3.90
3.34
2.50
2.50
0.99
1.98
3.02
6.01

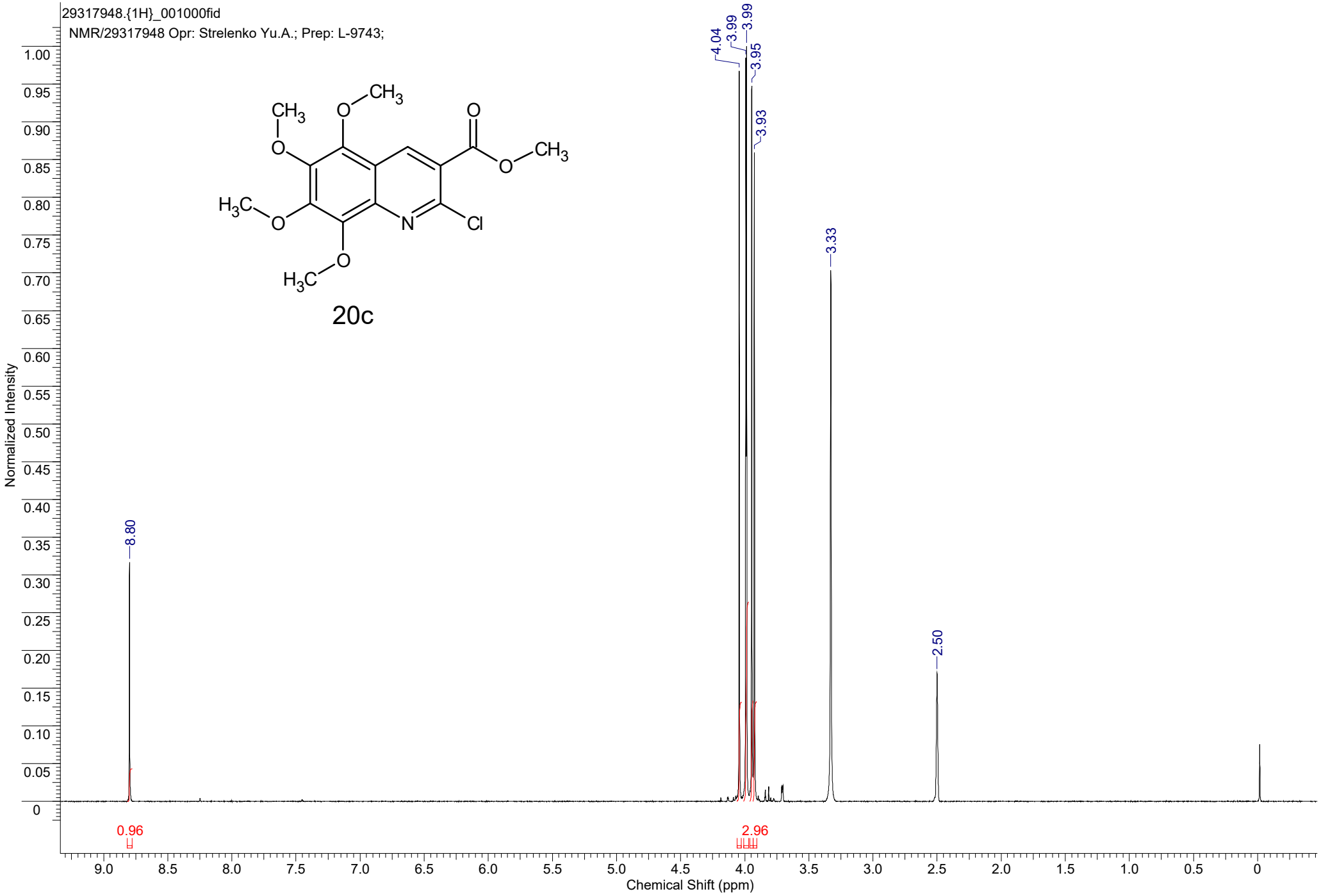
NMR/29330069

164.14
144.00
142.55
139.79
138.46
137.16
136.79
134.12
121.75
107.27
103.39
62.12
60.51
52.92
40.04
39.88
39.71
39.54
39.38
39.21
39.04



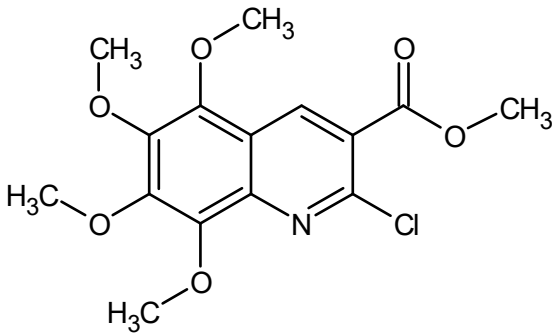
20b

180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


29317948.{1H}_001000fid
NMR/29317948 Opr: Strelenko Yu.A.; Prep: L-9743;
20c
8.80
4.04
3.99
3.99
3.95
3.93
3.33
2.50
0.96
2.96
Normalized Intensity
Chemical Shift (ppm)

NMR/29317948

164.34 150.84 145.06 144.82 143.17 142.64 139.19 136.12 122.34 117.76 62.04 61.92 61.50 61.30 52.87 45.75 40.00 39.92 39.83 39.75 39.66 39.59 39.50 39.33 39.16 38.99 30.64 8.61

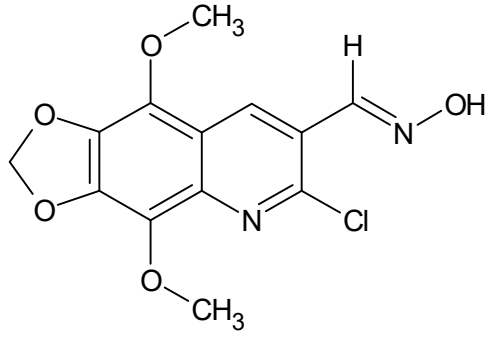


20c

180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

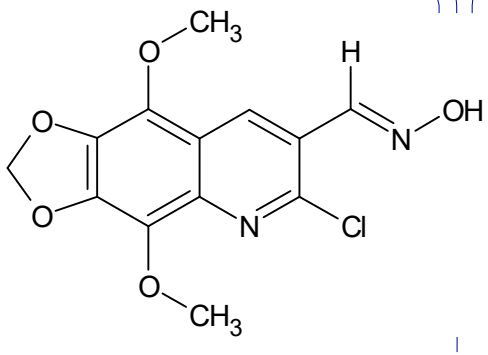
29317664.{1H}_001000fid

NMR/29317664 Opr: Strelenko Yu.A.; Prep: L-9710;



21a

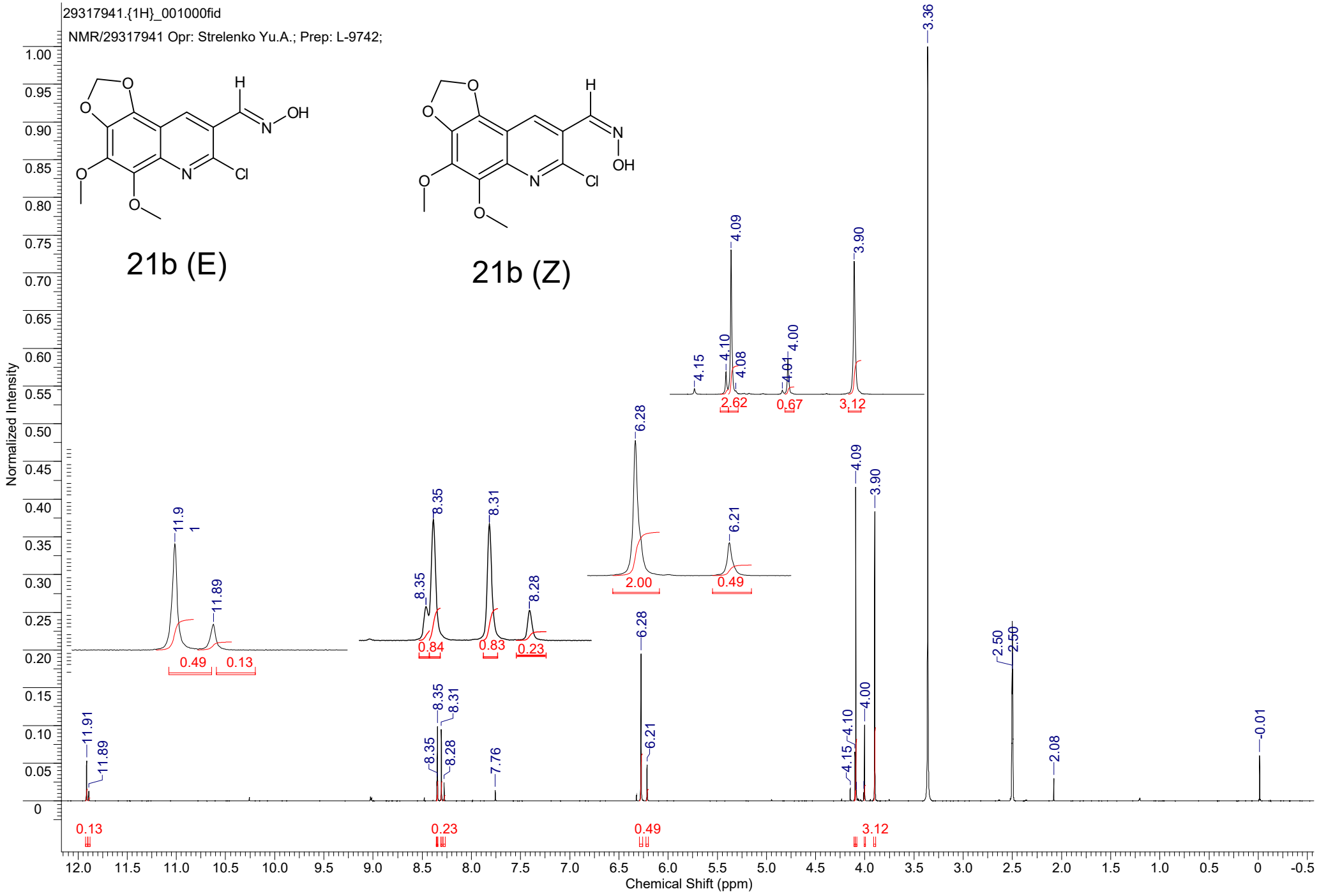
NMR/29317664



21a

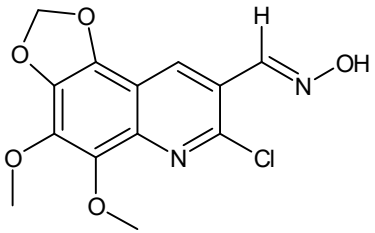
145.53
143.87
142.43
139.84
138.77
136.00
131.16
130.43
128.71
122.29
118.48
102.94
60.78
60.23
39.94
39.80
39.67
39.53
39.39
39.25
39.11

180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

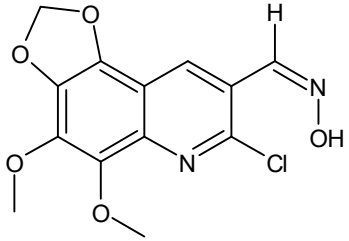

29317941.{1H}_001000fid
NMR/29317941 Opr: Strelenko Yu.A.; Prep: L-9742;
21b (E)
21b (Z)
Normalized Intensity
Chemical Shift (ppm)
11.91
11.89
8.35
8.31
8.28
7.76
6.28
6.21
4.15
4.10
4.09
4.08
4.00
3.90
3.36
2.50
2.08
-0.01
0.13
0.23
0.49
3.12

NMR/29317941

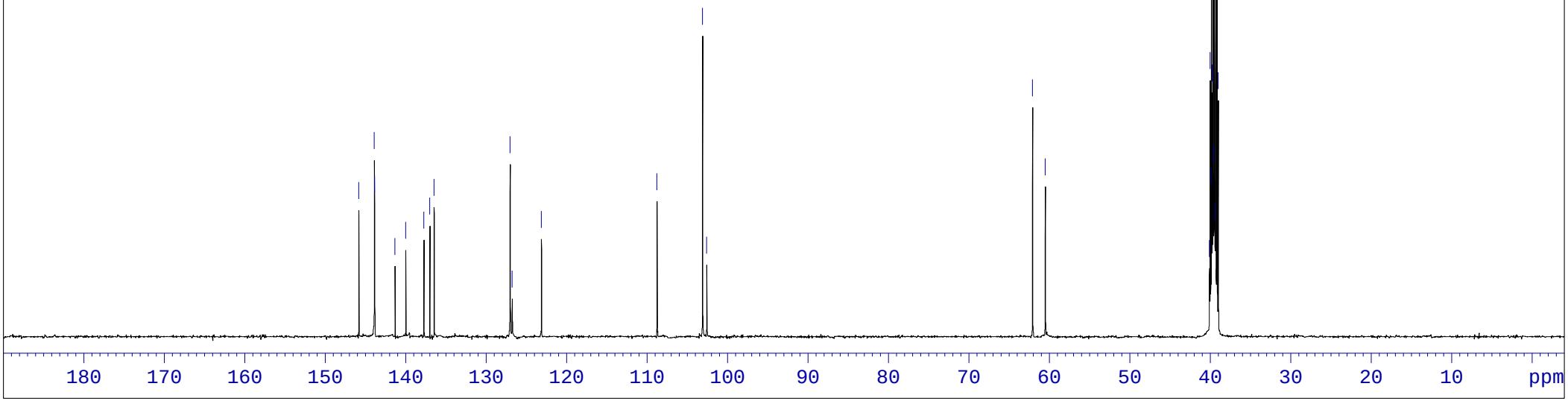
145.84
143.92
143.87
141.35
140.01
137.75
137.02
136.49
127.04
126.78
123.15
108.79
103.12
102.61
62.11
60.53
40.12
40.03
39.95
39.86
39.78
39.69
39.61
39.53
39.44
39.36
39.19
39.03



21b (E)

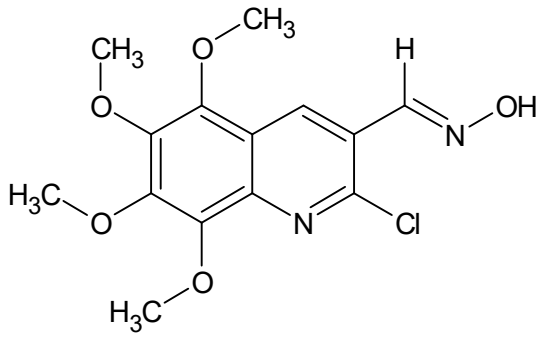


21b (Z)



29317946.{1H}_001000fid

NMR/29317946 Opr: Strelenko Yu.A.; Prep: L-9742;



21c

Normalized Intensity

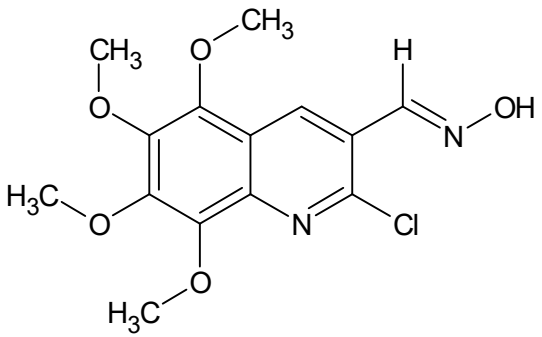
Chemical Shift (ppm)

11.93
8.66
8.39
4.01
3.98
3.94
3.38
2.50
2.50

0.54
1.00
0.97
3.60

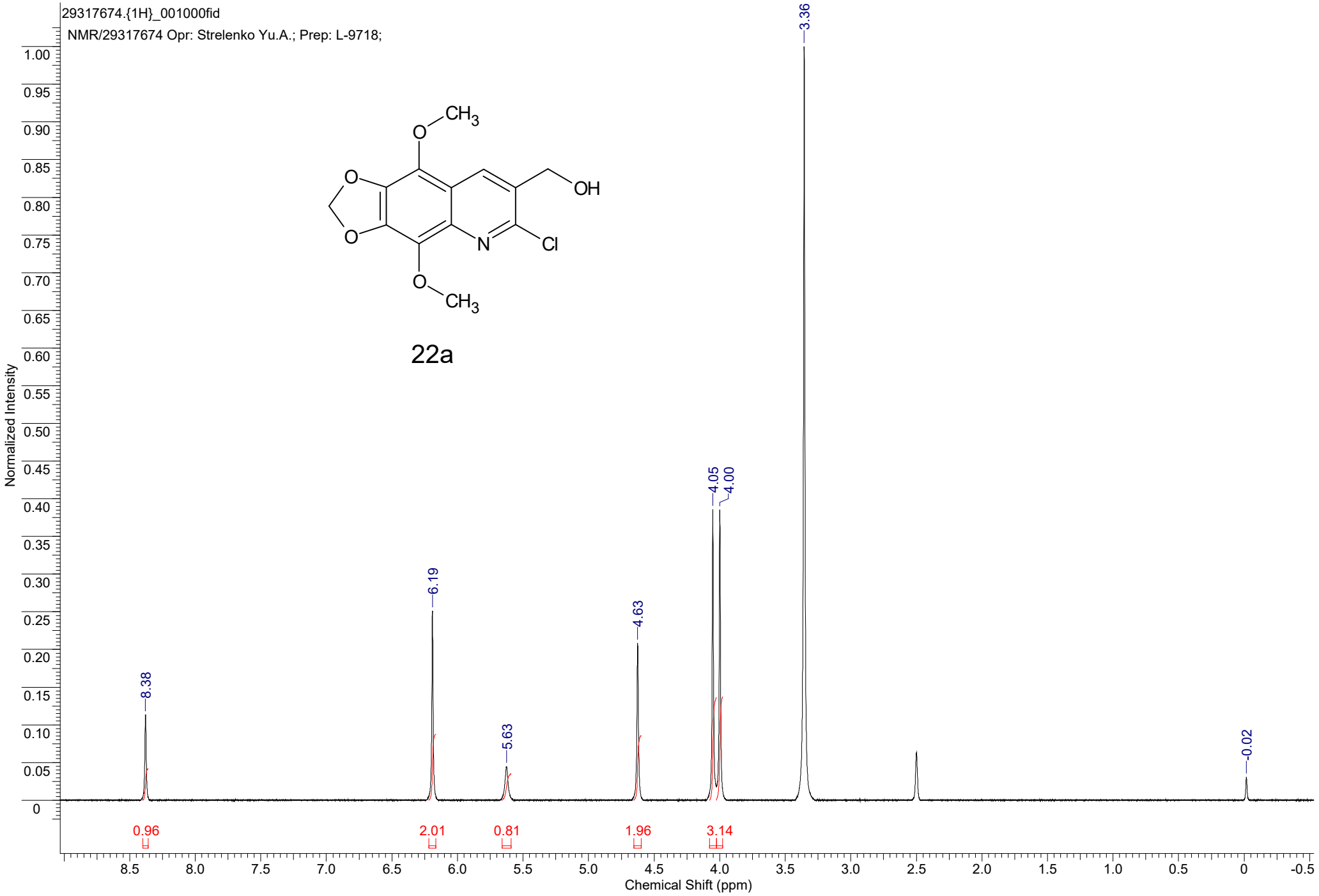
NMR/29317946

149.55
146.78
144.78
143.97
143.92
142.91
142.85
138.70
135.33
131.02
129.56
129.17
123.62
119.08
118.72
98.47
62.32
62.03
61.85
61.54
61.34
45.73
40.12
40.03
39.95
39.86
39.78
39.69
39.62
39.53
39.36
39.19
39.03
15.15



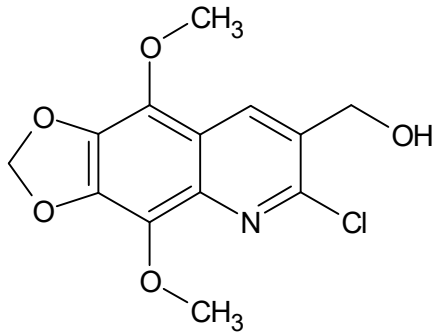
21c

180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

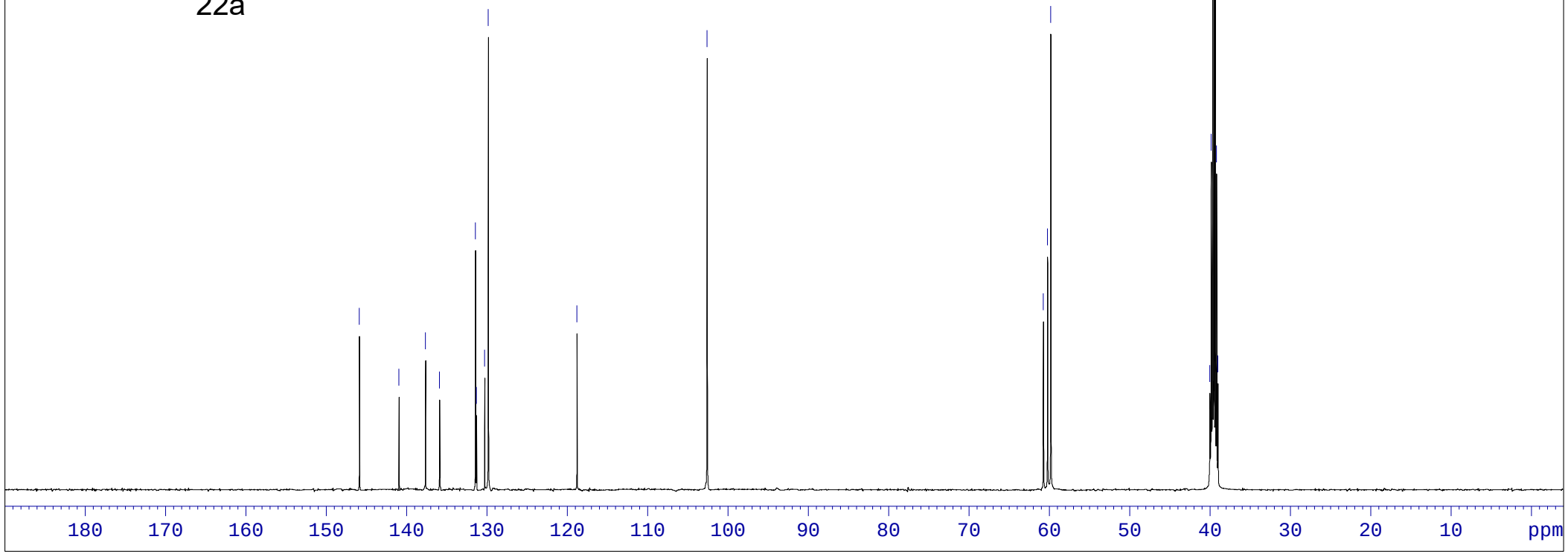

29317674.{1H}_001000fid
NMR/29317674 Opr: Strelenko Yu.A.; Prep: L-9718;
O
CH3
O
O
OH
N
Cl
O
CH3
22a
3.36
4.05
4.00
6.19
4.63
8.38
5.63
-0.02
0.96
2.01
0.81
1.96
3.14
Normalized Intensity
Chemical Shift (ppm)

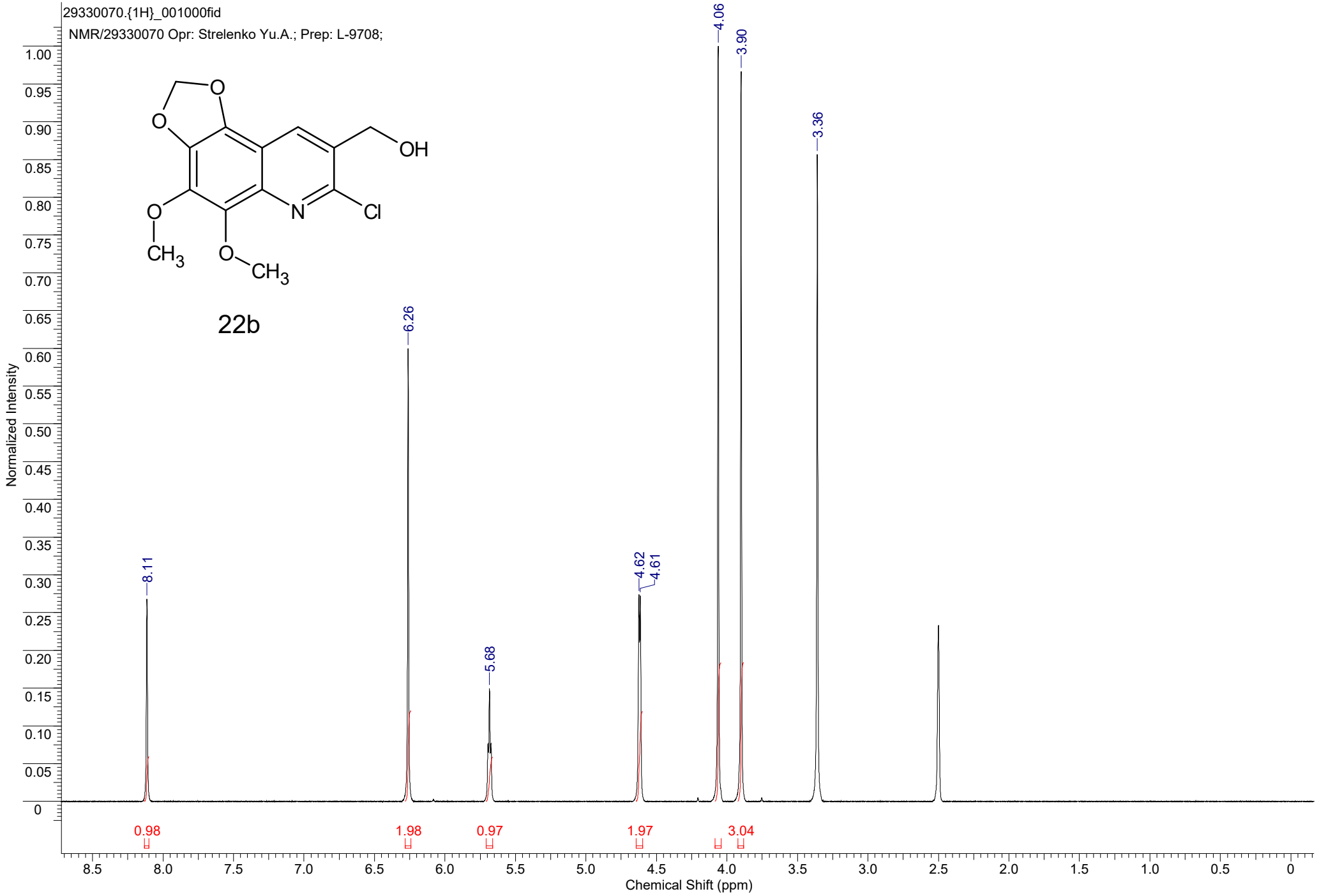
NMR/29317674

145.90 140.97 137.66 135.92 131.44 131.32 130.30 129.85 118.81 102.61 60.76 60.24 59.84 40.04 39.87 39.71 39.54 39.37 39.21 39.04



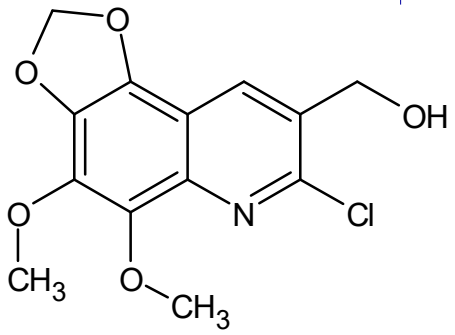
22a



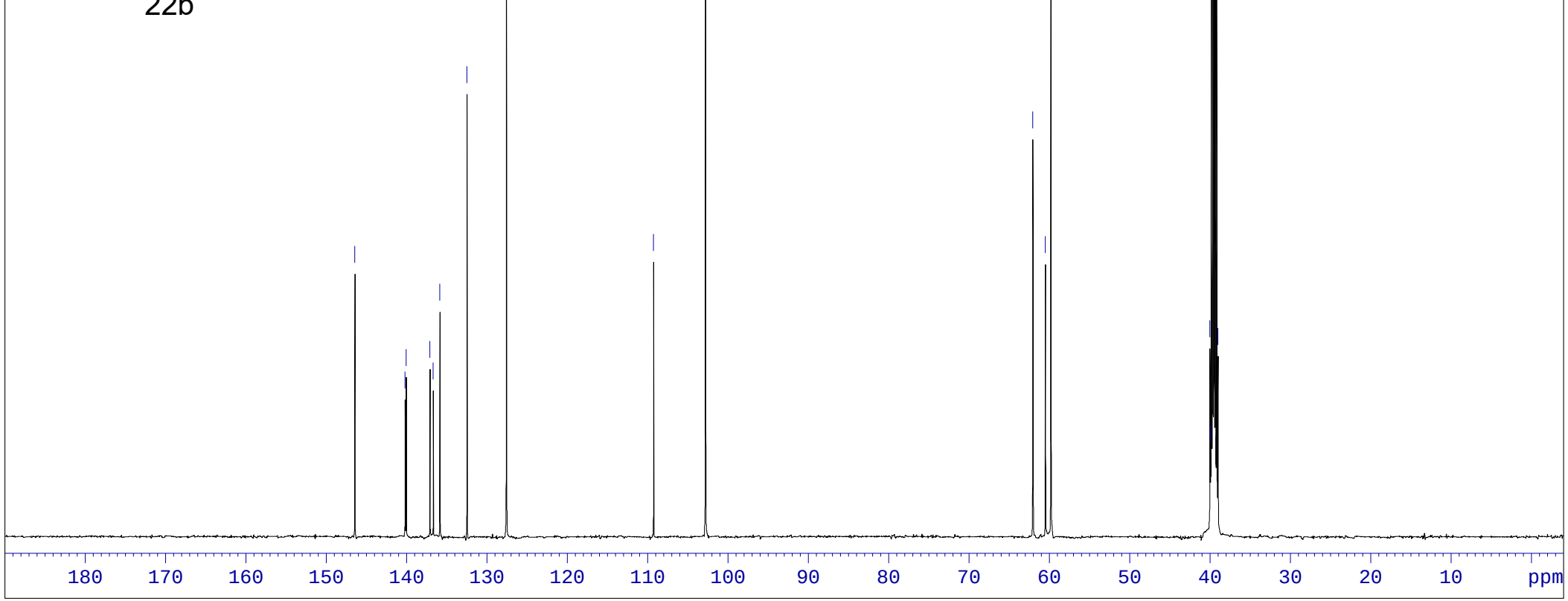

29330070.{1H}_001000fid
NMR/29330070 Opr: Strelenko Yu.A.; Prep: L-9708;
O
O
OH
O
N
Cl
CH3
O
CH3
22b
8.11
6.26
5.68
4.62
4.61
4.06
3.90
3.36
0.98
1.98
0.97
1.97
3.04
Normalized Intensity
Chemical Shift (ppm)

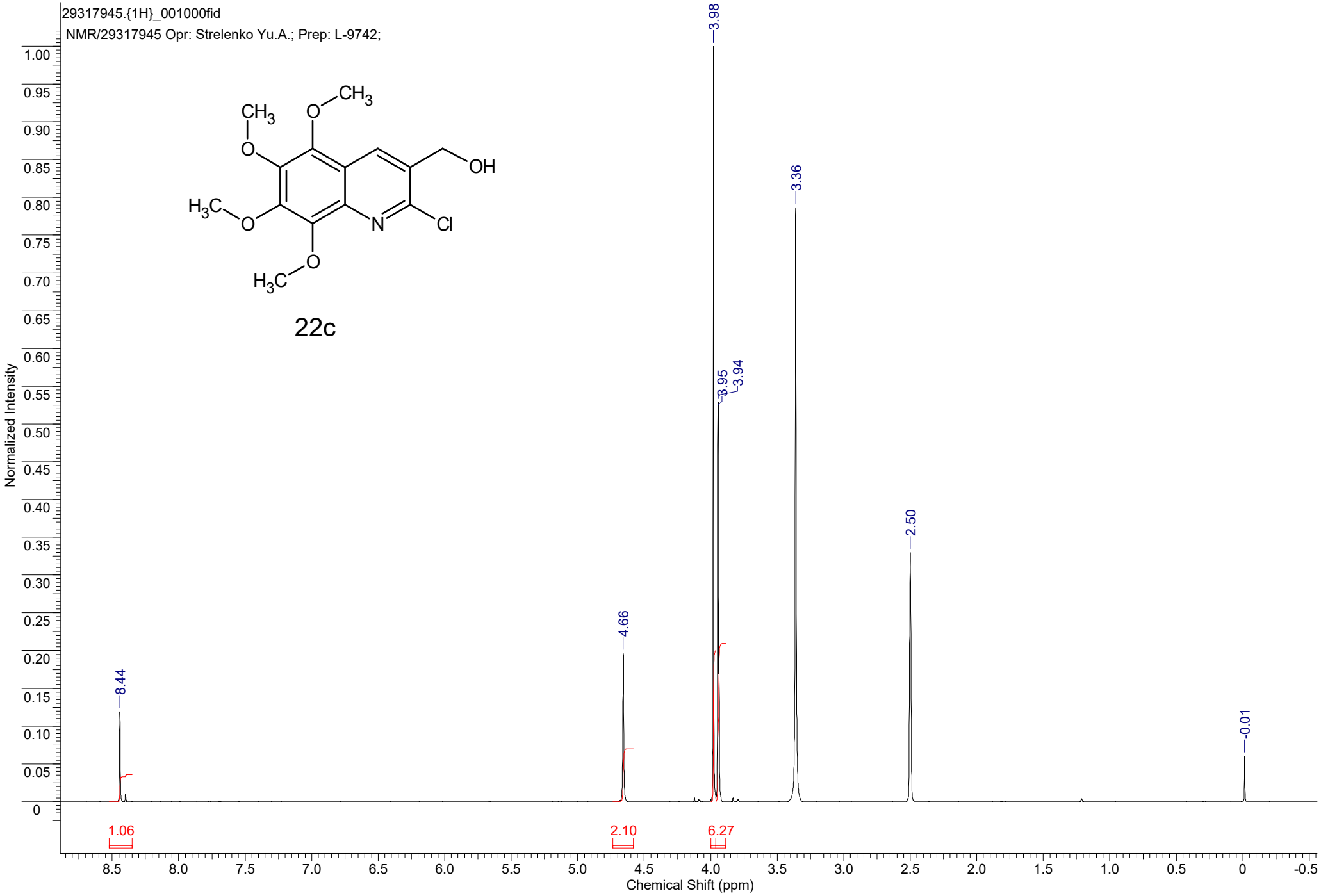
NMR/29330070

146.46 140.21 140.06 137.11 136.71 135.88 132.50 127.60 109.28 102.82 62.07 60.51 59.83 40.03 39.96 39.87 39.70 39.53 39.36 39.20 39.03



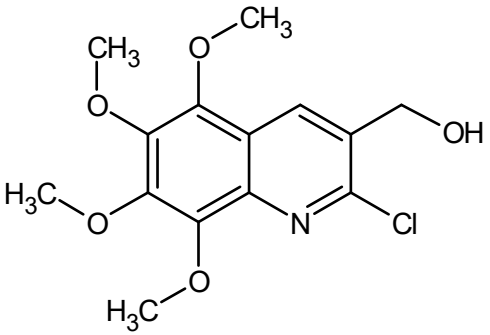
22b




29317945.{1H}_001000fid
NMR/29317945 Opr: Strelenko Yu.A.; Prep: L-9742;
CH3
O
CH3
O
OH
H3C
O
N
Cl
O
H3C
22c
3.98
3.36
3.95
3.94
2.50
4.66
8.44
-0.01
1.06
2.10
6.27
Normalized Intensity
Chemical Shift (ppm)

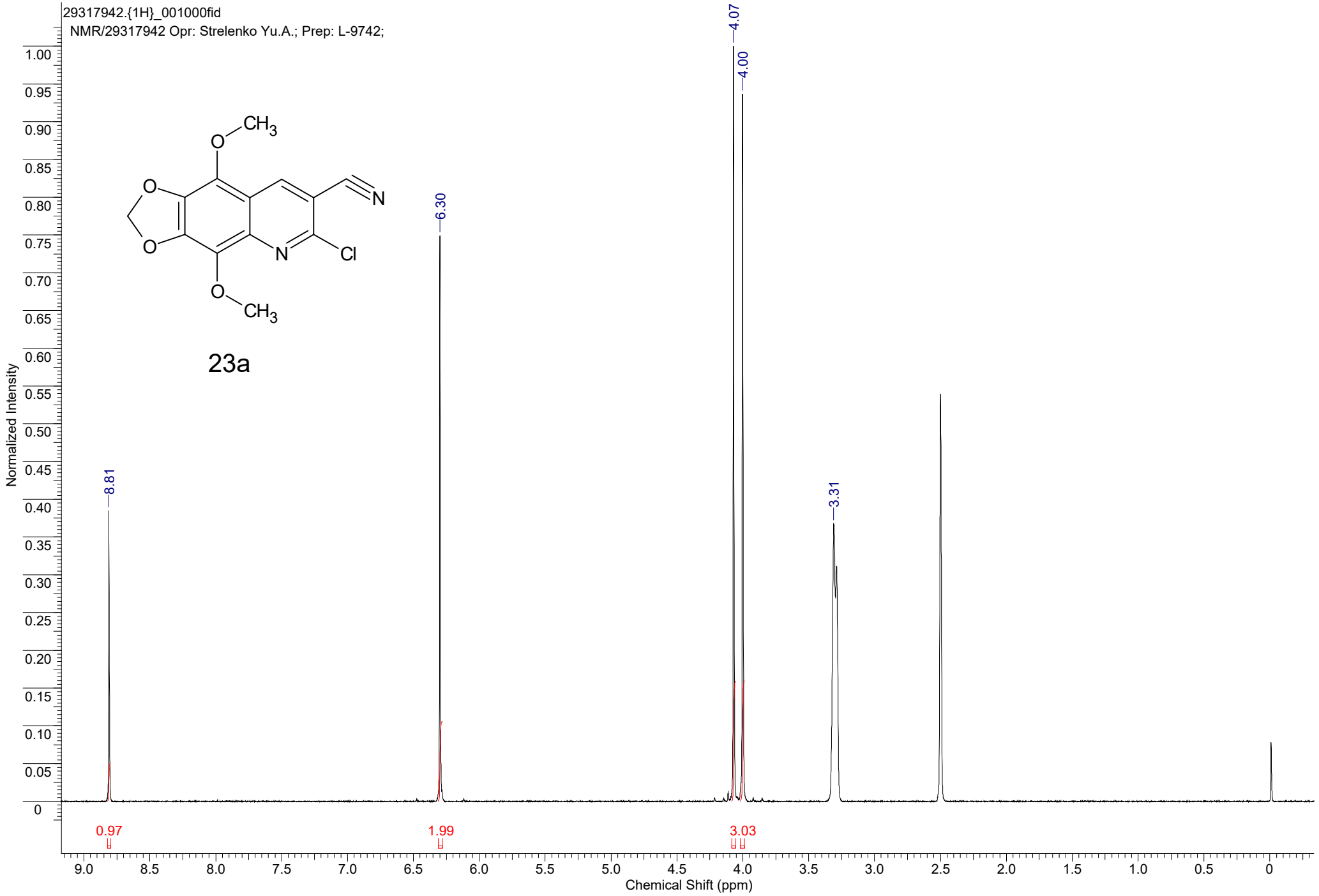
NMR/29317945

148.16
147.26
144.60
143.12
142.46
137.75
132.74
130.24
119.48
61.97
61.76
61.50
61.32
59.86
40.03
39.95
39.86
39.79
39.70
39.62
39.53
39.36
39.19
39.03



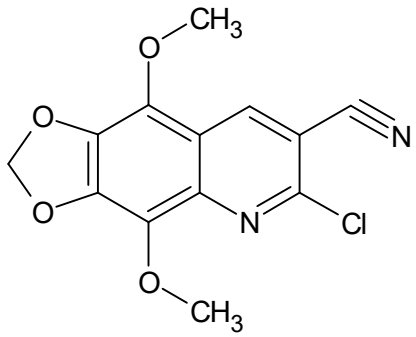
22c

180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


29317942.{1H}_001000fid
NMR/29317942 Opr: Strelenko Yu.A.; Prep: L-9742;
O
CH3
O
O
N
Cl
O
CH3
23a
8.81
6.30
4.07
4.00
3.31
0.97
1.99
3.03
Normalized Intensity
Chemical Shift (ppm)

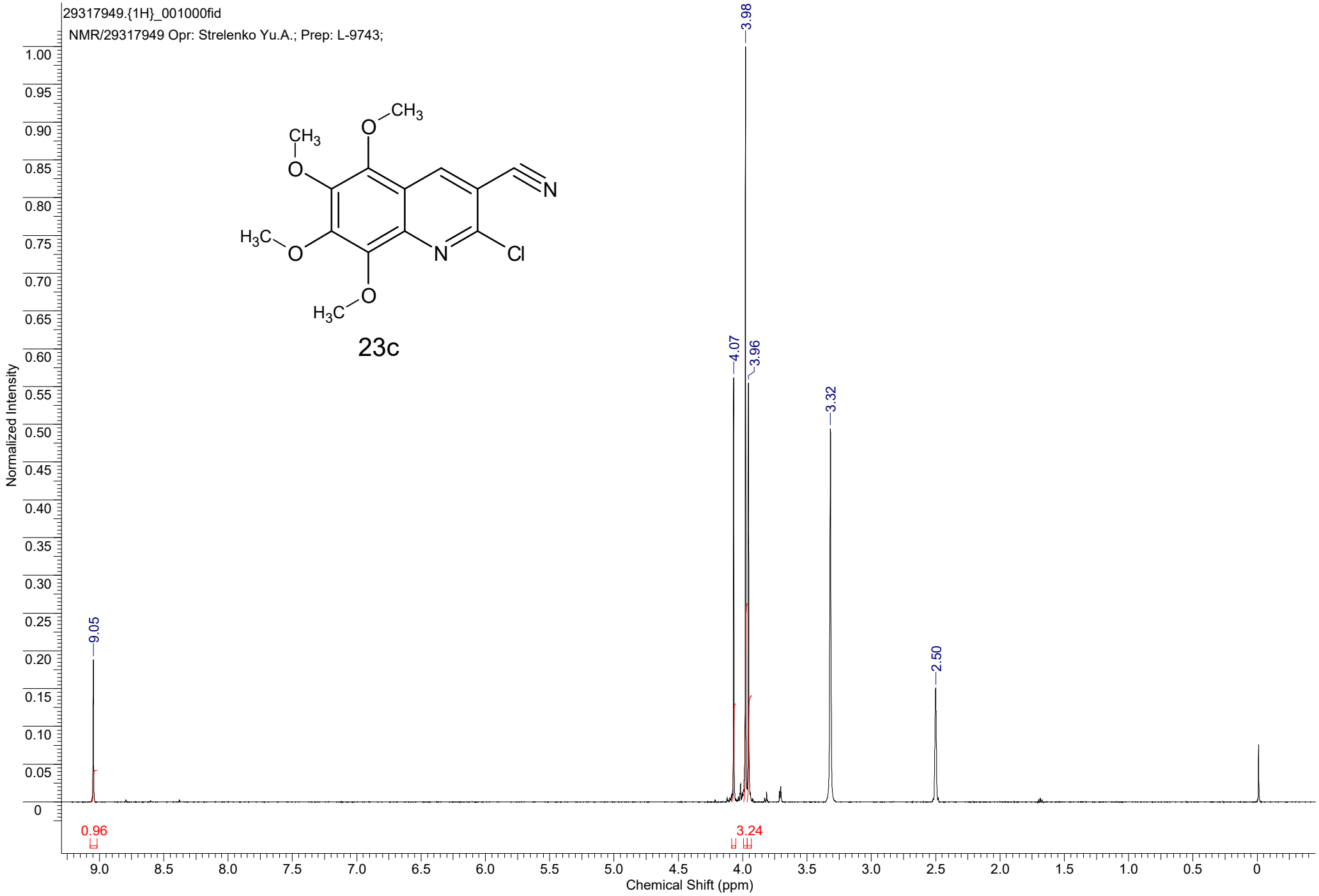
NMR/29317942

145.36
145.08
139.44
139.29
137.03
131.38
130.71
117.17
115.69
103.97
103.75
60.98
60.58
40.04
39.97
39.88
39.80
39.71
39.63
39.54
39.37
39.21
39.04



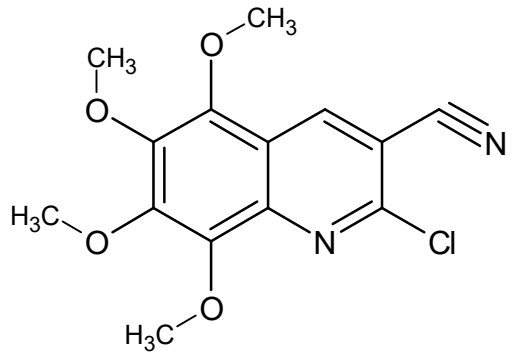
23a

180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


29317949.{1H}_001000fid
NMR/29317949 Opr: Strelenko Yu.A.; Prep: L-9743;
23c
Normalized Intensity
Chemical Shift (ppm)
9.05
4.07
3.98
3.96
3.32
2.50
0.96
3.24

NMR/29317949

151.94
146.10
145.33
143.27
142.69
141.03
139.11
117.55
115.60
105.19
62.22
62.16
61.58
61.35
45.58
40.00
39.92
39.83
39.75
39.66
39.59
39.50
39.33
39.16
39.00
30.65
8.52



23c

180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm