BEILSTEIN JOURNAL OF ORGANIC CHEMISTRY

Supporting Information

for

New synthesis of a late-stage tetracyclic key intermediate of lumateperone

Mátyás Milen, Bálint Nyulasi, Tamás Nagy, Gyula Simig and Balázs Volk

Beilstein J. Org. Chem. **2022,** *18,* 653–659. doi:10.3762/bjoc.18.66

General information, synthetic procedures, and spectral data

Table of contents

General information

All melting points were determined on a Büchi–Boëtius microapparatus and are uncorrected. IR spectra were obtained on a Bruker Alpha FT-IR spectrometer in KBr pellets. ^{1}H NMR and ^{13}C NMR spectra were recorded in DMSO-d_6 or $CDCl_3$ solution in 5 mm tubes at room temperature, on a Bruker Avance III HD 600 (600 and 150 MHz for ^{1}H and ^{13}C NMR spectra, respectively) or a Bruker Avance III 400 (400 and 100 MHz for ^{1}H and ^{13}C NMR spectra, respectively) spectrometer with the deuterium signal of the solvent as the lock and TMS as the internal standard. Chemical shifts (δ) and coupling constants (*J*) are given in ppm and in Hz, respectively. The following abbreviations are used to designate multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad. Mass spectra were recorded on a Bruker O-TOF MAXIS Impact mass spectrometer coupled to a Dionex Ultimate 3000 RS HPLC system with a diode array detector. High-resolution mass spectra (HRMS) were recorded on a Bruker O-TOF MAXIS Impact mass spectrometer coupled with a Waters I-Class UPLC system with a diode array detector. Reactions were monitored by thin-layer chromatography (TLC) carried out on silica gel plates (60 F_{254}) using UV light as visualizing agent. Purifications by flash column chromatography were carried out using Merck 107736 silica gel 60 H with a hexane/EtOAc or CH_2Cl_2/MeOH solvent system. All reagents were purchased from commercial sources and were used without further purification. Analytical samples of new compounds were obtained by recrystallization from the solvents or solvent mixtures given after the melting points in parentheses. Compounds (±)-**9**, **25**, **26**, **28**, **29**, **31**, (±)-**36**, and **38** are known from the literature, but insufficiently or not at all [(±)-**36**] characterized [1,2], therefore they are described below. New compounds **27**, **30**, **34**, and (±)-**35** are also characterized below in detail.

Synthesis

1-Methylquinoxalin-1-ium *p*-toluenesulfonate (25). This compound was prepared by a published method [3]. A mixture of quinoxaline (**24**, 10.0 g, 76.8 mmol) and methyl *p*-toluenesulfonate (27.17 g, 22.0 mL, 145.9 mmol, 1.9 equiv) was kept at room temperature for 2 d. The resulting solid was suspended in DEE (30 mL), filtered, and washed with DEE (10 mL). Yield: 19.93 g (82%). Colorless crystals. Mp 156–158 °C (EtOH–Et_2O, lit. mp 150–152 °C [4]). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 9.69–9.65 (m, 1H), 9.61–9.56 (m, 1H), 8.64–8.58 (m, 1H), 8.54–8.48 (m, 1H), 8.36–8.26 (m, 2H), 7.46–7.41 (m, 2H), 7.10–7.04 (m, 2H), 4.73 (s, 3H), 2.27 (s, 3H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ 148.2, 145.9, 145.0, 141.9, 137.7, 135.8, 133.8, 131.2, 131.0, 128.2, 125.6, 119.8, 45.8, 20.9 ppm.

1-Methyl-1,2,3,4-tetrahydroquinoxaline (26). This compound was prepared by a published method [3]. $NaBH_4$ (1.80 g, 47.6 mmol) was dissolved in H_2O (51 mL) and cooled to 0 °C. 1-Methylquinoxalin-1-ium *p*-toluenesulfonate (**25**, 5.00 g, 15.8 mmol) was dissolved in H_2O (10 mL) and added dropwise to the $NaBH_4$ solution over a period of 0.5 h. The mixture was stirred for an additional 10 min at 0 °C, then extracted with CH_2Cl_2 (3 × 30 mL). The combined organic layers were dried over $MgSO_4$, filtered, and the solvent was removed by rotary evaporation. The crude material was purified by Kugelrohr distillation at 145 °C (2 mbar), lit. bp 163–169 °C (20 Hg mm) [4]. Yield: 2.10 g (90%). Pale yellow oil. ^{1}H NMR ($CDCl_3$, 600 MHz): δ 6.68–6.65 (m, 1H), 6.65–6.60 (m, 2H), 6.48–6.45 (m, 1H), 3.48–3.46 (m, 2H), 3.27–3.25 (m, 2H), 2.68 (s, 3H) ppm. ^{13}C NMR ($CDCl_3$, 150 MHz): δ 136.2, 134.2, 118.7, 118.2, 113.5, 111.7, 49.9, 41.3, 39.1 ppm.

1-Methyl-4-nitroso-1,2,3,4-tetrahydroquinoxaline (27). The solution of **26** (9.40 g, 63.42 mmol) in AcOH (70.5 mL) and H_2O (47 mL) was cooled to 0–5 °C and treated dropwise with a solution of $NaNO_2$ (4.60 g, 66.67 mmol, 1.05 equiv) in H_2O (47 mL) at the same temperature. After stirring for 1 h at 0–5 °C, the mixture was basified with 15% NH_3 solution

to pH ≈ 8 and extracted with CH_2Cl_2 (3 × 50 mL), dried over Na_2SO_4, filtered, and evaporated under reduced pressure. Yield: 11.20 g (99%). Yellow oil. IR (film, cm^{-1}): 1605, 1503, 1425, 1306, 1170, 1131, 746. ^1H NMR ($CDCl_3$, 600 MHz): δ 8.04–8.02 (m, 1H), 7.24–7.20 (m, 1H), 6.89–6.85 (m, 1H), 6.80–6.78 (m, 1H), 4.06 (t, J = 5.6 Hz, 2H), 3.22 (t, J = 5.6 Hz, 2H), 2.93 (s, 3H) ppm. ^{13}C NMR ($CDCl_3$, 150 MHz): δ 137.2, 127.5, 126.2, 119.0, 115.7, 113.3, 48.2, 10.9, 39.1 ppm. HRMS calcd. for $C_9H_{12}N_3O$ $[M+H]^+$ 178.0975; found 178.0967.

1-Benzylquinoxalin-1-ium bromide (28). This compound was prepared by a published method [5]. The mixture of quinoxaline (**24**, 42.6 g, 327 mmol) and benzyl bromide (106.2 g, 73.8 mL, 621 mmol, 1.9 equiv) was left standing for 3 days. The resulting solid was filtered and washed extensively with DEE and dried. Yield: 87.1 g (88%). Yellow crystals. Mp 143–146 °C. IR (KBr, cm^{-1}): 3424, 1604, 1511, 1358, 1154, 772, 699. ^1H NMR ($CDCl_3$, 400 MHz): δ 10.73 (d, J = 2.9 Hz, 1H), 9.63 (d, J = 2.8 Hz, 1H), 8.76 (d, J = 8.8 Hz, 1H), 8.44 (dd, J_1 = 1.6 Hz, J_2 = 8.4 Hz, 1H), 8.20–8.16 (m, 1H), 8.13–8.08 (m, 1H), 7.61–7.58 (m, 2H), 7.33–7.31 (m, 3H), 6.91 (s, 2H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ 147.6, 146.1, 140.3, 136.3, 133.5, 132.1, 131.5, 130.1, 129.8, 129.6, 128.5, 119.7, 61.4 ppm.

1-Benzyl-1,2,3,4-tetrahydroquinoxaline (29). An analogous method for the *N*-methyl derivative was described by Beach and coworkers [3]. 1-Benzylquinoxalin-1-ium bromide (**28**, 30.0 g, 99.6 mmol) was dissolved in H_2O (300 mL). To a solution of $NaBH_4$ (11.40 g, 301.3 mmol, 3.0 equiv) in H_2O (150 mL) was added the aqueous solution of **28** over a period of 30 min at 0 °C. The mixture was stirred for further 30 min at 0 °C and then extracted with DCM (3 × 60 mL). The combined organic layers were washed with H_2O (2 × 25 mL), dried over $MgSO_4$, filtered, and the filtrate was concentrated under reduced pressure to afford the title compound **29**. Yield: 22.34 g (86%). Pale yellow crystals. Mp 52–53 °C (hexane, lit. mp 50.5–52.5 °C [6]). ^1H NMR ($CDCl_3$, 400 MHz): δ 7.33–7.29 (m, 4H), 7.26–7.22 (m, 1H), 6.63–6.57 (m, 1H), 6.56–6.52 (m, 3H), 4.44 (s, 2H), 3.73 (br s, 1H), 3.44–3.40 (m, 4H) ppm. ^{13}C NMR

($CDCl_3$, 100 MHz): δ 138.7, 135.4, 133.8, 128.5, 127.0, 126.8, 119.2, 117.6, 114.1, 111.8, 55.2, 48.4, 41.0 ppm [7].

1-Benzyl-4-(trifluoroacetyl)-1,2,3,4-tetrahydroquinoxaline (30). To a stirred solution of amine **29** (8.60 g, 38.34 mmol) and TEA (8.0 mL, 5.81 g, 57.42 mmol, 1.5 equiv) in THF (43 mL) was carefully added TFAA (8.10 mL, 12.06 g, 57.42 mmol, 1.5 equiv) at 0 °C. The reaction mixture was stirred for 30 min at the same temperature and for further 60 min at 25 °C. The mixture was then evaporated under reduced pressure. The residue was dissolved in DCM (50 mL), washed with H_2O (2 × 20 mL), dried over Na_2SO_4, filtered, and evaporated to give the title compound **30**. Yield: 11.95 g (97%). Colorless crystals. Mp 91–92 °C (^{i}PrOH). IR (KBr, cm^{-1}): 3440, 2873, 1688, 1604, 1511, 1350, 1204, 1189, 1146, 751. ^{1}H NMR (DMSO-d_6, 600 MHz): 7.51–7.47 (m, 1H), 7.35–7.31 (m, 2H), 7.28–7.21 (m, 3H), 7.02–6.98 (m, 1H), 6.73–6.68 (m, 1H), 6.62–6.58 (m, 1H), 4.58 (s, 2H), 3.98 (s, 2H), 3.60–3.53 (m, 2H) ppm. ^{13}C NMR (DMSO-d_6, 150 MHz): δ 153.6 (q, J = 35.1 Hz), 139.2, 138.0, 128.8, 127.6, 127.1, 126.8, 124.6, 121.9, 116.6 (q, J = 289.2 Hz), 115.1, 112.2, 53.5, 49.4, 43.2 ppm. At room temperature, the compound consists of a ca. 9:1 mixture of two rotamers. Only the signals of the major species are given here. HRMS calcd. for $C_{17}H_{16}F_3N_2O$ $[M+H]^+$ 321.1215; found 321.1212.

1-(Trifluoroacetyl)-1,2,3,4-tetrahydroquinoxaline (31). *Method A:* To a stirred solution of **30** (2.00 g, 6.24 mmol) in EtOAc (80 mL) were added a saturated HCl solution in EtOAc (1.0 mL) and palladium on carbon (10% Pd, 0.20 g). The reaction mixture was stirred under hydrogen atmosphere (3–5 bar) in an autoclave for 1 d. The catalyst was filtered off and the filtrate was evaporated to dryness. The waxy solid was treated with hexane (5 mL), the crystals were filtered, washed with MTBE (2 × 3 mL) and dried to give title compound **30**. Yield: 1.40 g (97%). Colorless crystals. Mp 162–164 °C (MeCN). IR (KBr, cm^{-1}): 3389, 1674, 1605, 1515, 1283, 1188, 1142, 756. ^{1}H NMR (DMSO-d_6, 600 MHz): 7.49–7.46 (m, 1H), 7.04–6.94 (m, 1H), 6.66–6.63 (m, 1H), 6.53–6.49 (m, 1H), 6.35 (br s, 1H), 3.80 (br s, 2H), 3.42 (br s, 2H) ppm. ^{13}C

NMR (DMSO-d_6, 150 MHz): δ 153.6 (q, J = 34.8 Hz), 139.2, 127.2, 124.4, 120.8, 116.6 (q, J = 288.9 Hz), 114.7, 114.3, 43.0, 41.6 ppm. At room temperature, the compound consists of a ca. 9:1 mixture of two rotamers. Only the signals of the major species are given here. HRMS calcd. for $C_{10}H_{10}F_3N_2O$ $[M+H]^+$ 231.0745; found 231.0742. *Method B:* According to a published method [8], trifluoroacetic anhydride (1.30 mL, 1.94 g, 9.2 mmol) was added dropwise to a solution of 1,2,3,4-tetrahydroquinoxaline (**37**, 1.34 g, 10.0 mmol) and Et_3N (1.53 mL, 1.11 g, 11.0 mmol) in CH_2Cl_2 (10 mL) at 0 °C. After 30 min, the reaction temperature was allowed to warm to 23 °C and the mixture was stirred overnight and evaporated. The residue was separated by flash column chromatography (SiO_2, hexane→hexane–EtOAc 50:50) to afford three compounds: 0.34 g (25%) of the starting material, 0.90 g (39%) of title compound **31** and 0.58 g (25%) of 1,4-bis-(trifluoroacetyl)-1,2,3,4-tetrahydroquinoxaline (**38**). For analytical data of compound **38**, see below.

Ethyl 3-(trifluoroacetyl)-2,3,9,10-tetrahydro-1*H*-pyrido[3',4':4,5]pyrrolo[1,2,3-*de*]quinoxaline-8(7*H*)-carboxylate (34). The solution of **31** (5.10 g, 22.16 mmol) in AcOH (100 mL) and H_2O (50 mL) was cooled to 5–10 °C and treated dropwise with a solution of sodium nitrite (1.80 g, 26.09 mmol, 1.18 equiv) in H_2O (30 mL) at the same temperature. After stirring for 1 h at 0–5 °C, the product was filtered, washed with H_2O (2 × 5 mL) and dried. Yield: 4.80 g (84%). This nitroso intermediate (**32**) was directly used in the next reaction according to the procedure described in the literature for a similar reaction [1]. To a stirred solution of **32** (4.80 g, 18.52 mmol) in AcOH (80 mL) at 10–12 °C was added freshly activated zinc powder [9] (6.40 g, 97.89 mmol, 5.5 equiv) portionwise. The reaction mixture was stirred at 10–20 °C for 2 h. Ethyl 4-oxopiperidine-1-carboxylate (**5**, 2.80 mL, 3.18 g, 18.52 mmol) was added and the mixture was filtered after 5 min of stirring. The insoluble material was washed with acetic acid (4 × 16 mL). To the combined filtrates, a saturated HCl solution in DEE (0.80 mL) was added and heated to 100 °C for 1 h. The dark reaction mixture was cooled and

evaporated under reduced pressure. Flash column chromatography (SiO_2, CH_2Cl_2 → CH_2Cl_2/MeOH 92:8) afforded the desired product. Yield: 2.89 g (41%). Colorless crystals. Mp 126–128 °C (MeCN). IR (KBr, cm^{-1}): 2970, 1699, 1578, 1386, 1199, 1056. ^1H NMR ($CDCl_3$, 400 MHz): 7.83 (br s, 1H), 7.34–7.26 (m, 1H), 7.12–7.08 (m, 1H), 4.71 (s, 2H), 4.23–4.18 (m, 6H), 3.89 (s, 2H), 2.82 (s, 2H) 1.31 (t, J = 7.2 Hz, 3H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ 156.0, 154.8 (q, J = 35.0 Hz), 132.4, 127.3, 124.4, 122.7, 119.6, 116.3 (q, J = 288.3 Hz), 115.8, 114.0, 107.7, 61.6, 44.6, 41.9, 41.3, 40.9, 21.6, 14.7 ppm. At room temperature, the compound consists of a ca. 2:1 mixture of two rotamers. Only the signals of the major species are given here. HRMS calcd. for $C_{18}H_{19}F_3N_3O_3$ $[M+H]^+$ 382.1379; found 382.1372.

***cis*-Ethyl 3-(trifluoroacetyl)-2,3,6b,9,10,10a-hexahydro-1*H*-pyrido[3',4':4,5]pyrrolo[1,2,3-*de*]quinoxaline-8(7*H*)-carboxylate [(±)-35].** A stirred solution of **34** (1.00 g, 2.62 mmol) in AcOH (58 mL) was cooled to 17 °C. The solution was treated portionwise with $NaBH_3CN$ (492 mg, 7.83 mmol, 3.0 equiv) and stirred at room temperature for 24 h. The reaction mixture was diluted with H_2O (87 mL) and basified with 25% NH_4OH (ca. 70 mL) up to pH 8. The precipitated product was filtered, washed with H_2O (2 × 5 mL) and dried. Yield: 960 mg (95%). Colorless crystals. Mp 149–152 °C ($^iPr_2O$, decomp.). IR (KBr, cm^{-1}): 1685, 1202, 1186, 1142, 1121. ^1H NMR (DMSO-d_6, 600 MHz): 7.86–7.84 (m, 1H), 7.01–7.00 (m, 1H), 6.66–6.63 (m, 1H), 4.08–4.06 (m, 1H), 4.04–4.01 (m, 2H), 4.00–3.97 (m, 1H), 3.87–3.77 (m, 1H), 3.67–3.64 (m, 1H), 3.54–3.46 (m, 2H), 3.28–3.24 (m, 1H), 3.22–3.17 (m, 2H), 3.07–3.04 (m, 1H), 1.86–1.79 (m, 2H), 1.17 (t, J = 6.7 Hz, 3H) ppm. ^{13}C NMR (DMSO-d_6, 150 MHz): 154.9, 154.0 (q, J = 34.8 Hz), 142.0, 130.5, 121.9, 121.6, 120.5, 118.0, 116.4 (q, J = 288.5 Hz), 62.8, 60.8, 44.9, 43.9, 42.7, 40.1, 39.3, 23.6/23.5, 14.8 ppm. At room temperature, the compound consists of a ca. 1:1 mixture of two rotamers. HRMS calcd. for $C_{18}H_{21}F_3N_3O_3$ $[M+H]^+$ 384.1535; found 384.1529.

***cis*-Ethyl 2,3,6b,9,10,10a-hexahydro-1*H*-pyrido[3',4':4,5]pyrrolo[1,2,3-*de*]quinoxaline-8(7*H*)-carboxylate [(±)-36]** [1,2]. To a solution of (±)-**35** (500 mg, 1.30 mmol) in MeOH (20 mL) and H_2O (0.8 mL) was added K_2CO_3 (0.36 g, 2.61 mmol, 2.0 equiv). The reaction mixture was stirred at reflux temperature for 2 h and evaporated. The residue was dissolved in the mixture of EtOAc (20 mL) and H_2O (20 mL). After separation, the aqueous phase was extracted with EtOAc (2 × 20 mL). The combined organic layers were washed with brine (20 mL), dried over $MgSO_4$, filtered, and evaporated under reduced pressure to provide the product. Yield: 334 mg (89%). Off-white crystals. Mp 122–125 °C (hexane). IR (KBr, cm^{-1}): 3342, 1678, 1483, 1440, 1122, 730. ^{1}H NMR ($CDCl_3$, 600 MHz): 6.61–6.59 (m, 1H), 6.58–6.56 (m, 1H), 6.41–6.39 (m, 1H), 4.20–4.11 (m, 2H), 4.16/4.05–3.98 (m, 1H), 3.94–3.88/3.88–3.83 (m, 1H), 3.72–3.67 (m, 1H), 3.60 (br s, 1H), 3.51–3.47 (m, 1H), 3.36–3.34 (m, 1H), 3.33–3.31 (m, 1H), 3.22–3.15 (m, 1H), 3.14–3.07 (m, 1H), 2.92–2.86/2.86–2.78 (m, 1H), 2.72–2.67 (m, 1H), 1.95–1.90 (m, 1H), 1.89–1.82 (m, 1H), 1.28 (t, J = 7.1 Hz, 3H) ppm. ^{13}C NMR ($CDCl_3$, 150 MHz): δ 155.5, 138.0, 132.5, 129.7 (br), 120.3, 113.6 (br), 111.9, 65.3, 61.2, 45.6, 44.2, 42.3, 41.0/40.8, 39.7, 24.7/24.4, 14.7 ppm. At room temperature, the compound consists of a ca. 1:1 mixture of two rotamers.

1,4-Bis(trifluoroacetyl)-1,2,3,4-tetrahydroquinoxaline (38). This compound was obtained as a byproduct in the synthesis of compound **31** (see above). Colorless crystals. Mp 121–122.5 °C (EtOH, lit. mp 118–118.5 °C [10]). IR (KBr, cm^{-1}): 1689, 1505, 1206, 1185, 1140, 529. ^{1}H NMR ($CDCl_3$, 600 MHz): 8.00–7.60 (m, 1H), 7.60–7.30 (m, 3H), 4.18–4.02 (m, 4H) ppm. ^{13}C NMR ($CDCl_3$, 150 MHz): δ 156.0, 133.6/128.6, 128.6/127.2, 124.7, 116.0 (q, J = 288.4 Hz), 45.7 ppm. At room temperature, the compound consists of a ca. 1:1 mixture of two rotamers.

***cis*-Ethyl 3-methyl-2,3,6b,9,10,10a-hexahydro-1*H*-pyrido[3',4':4,5]pyrrolo[1,2,3-*de*]quinoxaline-8(7*H*)-carboxylate [(±)-9].** To a solution of (±)-**36** (320 mg, 1.11 mmol) in MeCN (26 mL) was added formaldehyde (0.83 mL of a 37% aqueous solution, 11.12 mmol,

10 equiv). After 20 min, to the dark orange reaction mixture were added $NaBH_3CN$ (176 mg, 2.80 mmol, 2.5 equiv) and AcOH (160 µL, 168 mg, 2.80 mmol, 2.5 equiv) and the mixture was stirred at room temperature for 2 h. The solvents were evaporated, and the residue was taken up in DCM (20 mL) and extracted with saturated $NaHCO_3$ (2 × 15 mL), dried over $MgSO_4$, filtered, and concentrated under reduced pressure to give the product. Yield: 232 mg (69%). Colorless oil. ^{1}H NMR ($CDCl_3$, 600 MHz): 6.68–6.65 (m, 1H), 6.56–6.54 (m, 1H), 6.42–6.41 (m, 1H), 4.17–4.13 (m, 2H), 4.04–3.98 (m, 1H), 3.93–3.81 (m, 1H), 3.62–3.58 (m, 1H), 3.35–3.32 (m, 2H), 3.30–3.26 (m, 1H), 3.19–3.11 (m, 2H), 2.87 (s, 3H), 2.86–2.84 (m, 1H), 2.82–2.78 (m, 1H), 1.92–1.82 (m, 2H) 1.28 (t, 3H, J = 7.2 Hz) ppm. ^{13}C NMR ($CDCl_3$, 150 MHz): δ 155.5, 138.0, 135.0, 128.6 (br), 120.4, 112.9 (br), 109.1, 64.9, 61.2, 50.5, 45.6, 44.2, 41.3/41.0, 39.7, 37.5, 24.6/24.3, 14.7 ppm. At room temperature, the compound consists of a ca. 1:1 mixture of two rotamers.

References

1. Robichaud, A. J.; Lee, T.; Deng, W.; Mitchell, I. S.; Yang, M. G.; Haydar, S.; Chen, W.; MC Clung, Ch. D.; Calvello, E. J. B.; Zawrotny, D. M. Preparation of substituted heterocycle fused gamma-carbolines. WO Patent 2000077002, December 21, 2000.

2. Li, P.; Zhang, Q.; Robichaud, A. J.; Lee, T.; Tomesch, J.; Yao, W.; Beard, J. D.; Snyder, G. L.; Zhu, H.; Peng, Y.; Hendrick, J. P.; Vanover, K. E.; Davis, R. E.; Mates, S.; Wennogle, L. P. *J. Med. Chem.* **2014**, *57*, 2670–2682. doi: 10.1021/jm401958n

3. Smith, R. F.; Rebel, W. J.; Beach, T. N. *J. Org. Chem*. **1959**, *24*, 205–207. doi: 10.1021/jo01084a014

4. Process for preparation of 1-(4-fluorophenyl)-4-((6b*R*,10a*S*)-3-methyl-2,3,6b,9,10,10a-hexahydro-1*H*-pyrido[3',4':4,5]pyrrolo[1,2,3-*de*]quinoxalin-8(7*H*)-yl)butan-1-one. IPCOM000258304D; *Chem. Abstr*. **2019**, *171*, 163843.

5. Leblanc, N.; Sproules, S.; Powell, A. K. *New J. Chem.* **2017**, *41*, 2949–2954. doi: 10.1039/C7NJ00531H

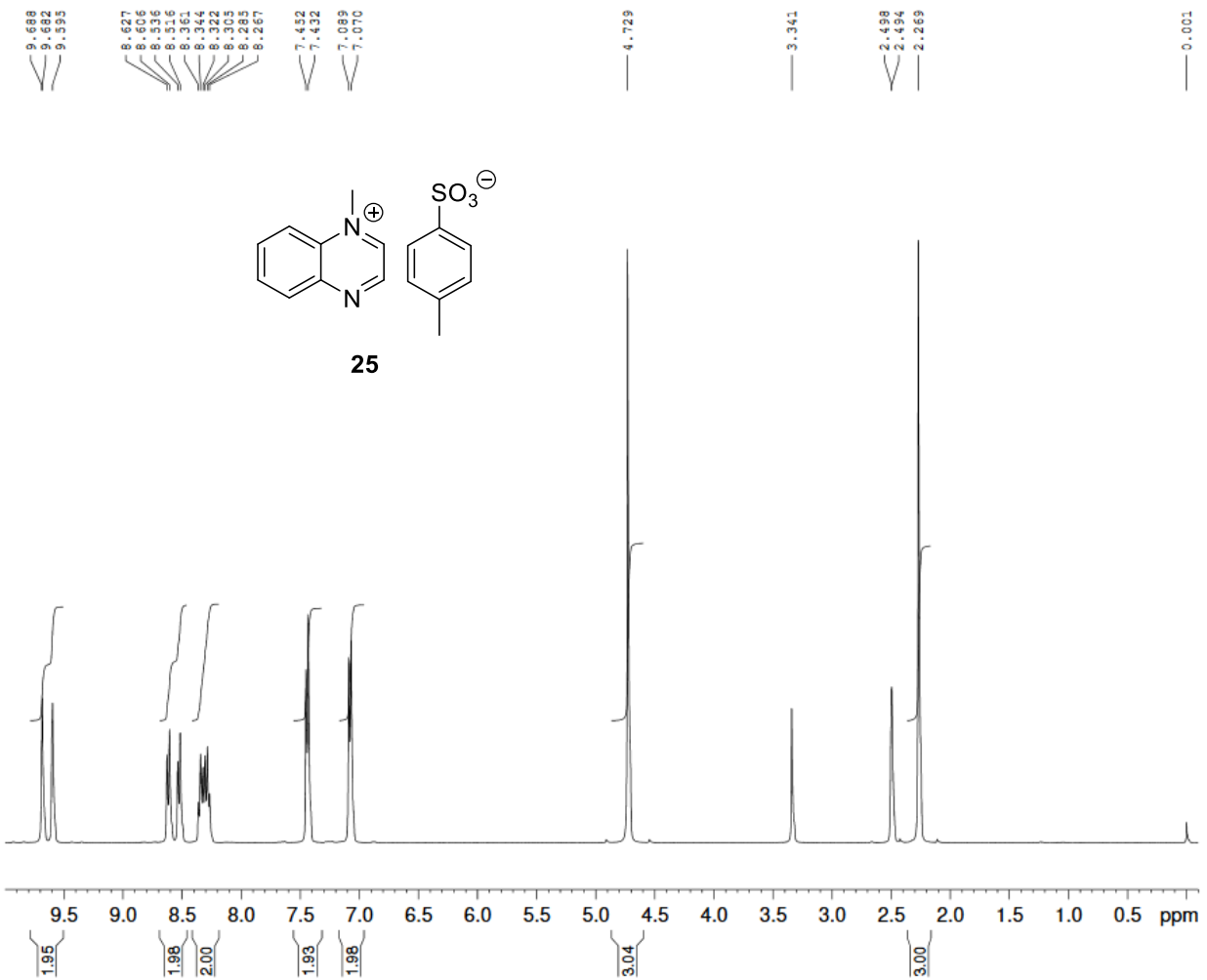
6. Cavagnol, J. C.; Wiselogle, F. Y. *J. Am. Chem. Soc*. **1947**, *69*, 795–799. doi: 10.1021/ja01196a015

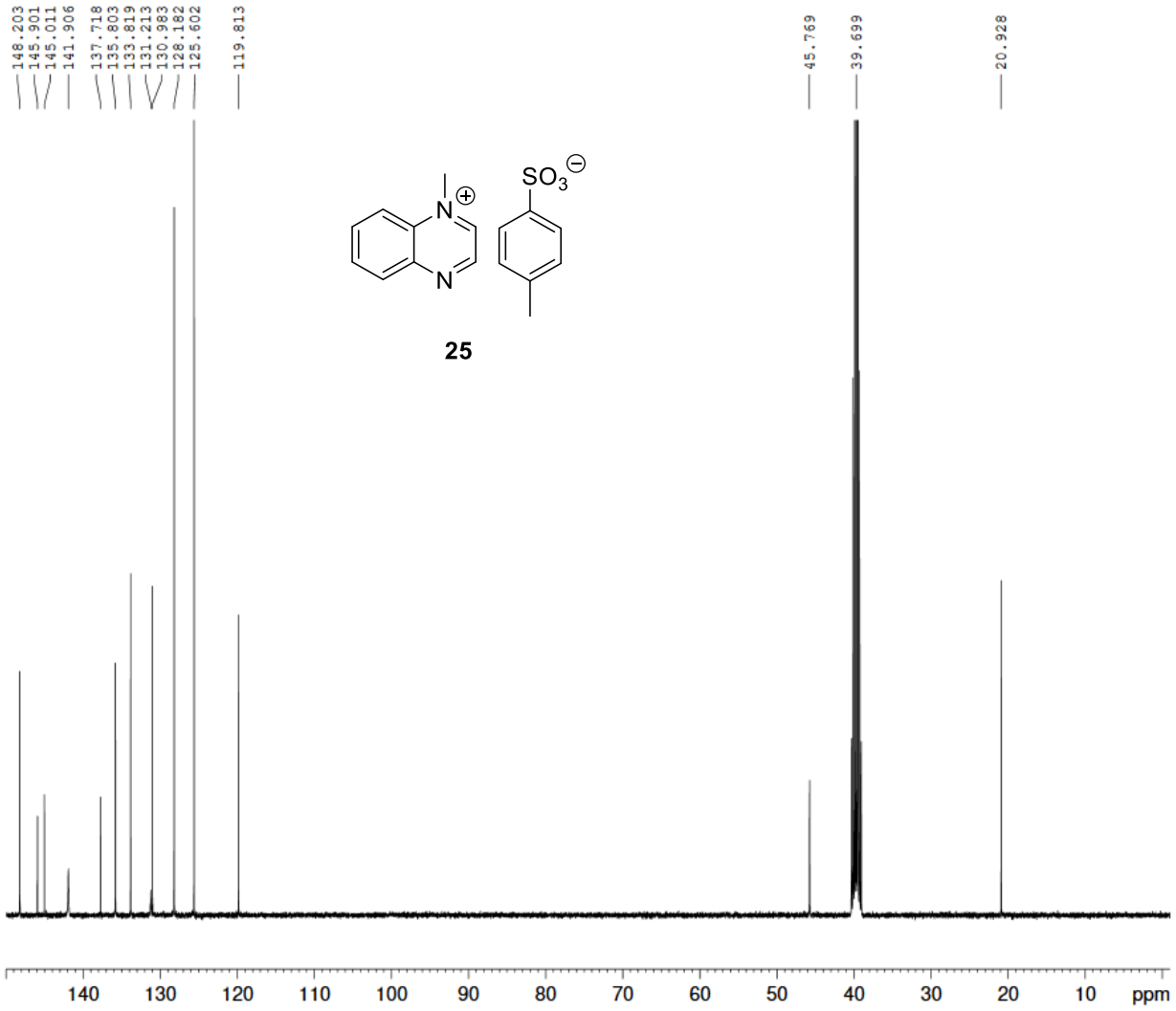
7. Ogata, O.; Nara, H.; Matsumara, K.; Kayaki, Y. *Org. Lett*. **2019**, *10*, 9954–9959. doi: 10.1021/acs.orglett.9b03878

8. Murphy, B. P.; Zhang, G.; Zhao, J. Stable violet-blue to blue imidazolium compounds. WO Patent 2018052725, March 22, 2018.

9. Furniss, B. S.; Hannaford, A. J.; Smith, P. W. G.; Tatchell, A. R. *Vogel's Textbook of Practical Organic Chemistry*, 5th ed.; Longman, Harlow, UK, 1989; pp 467.

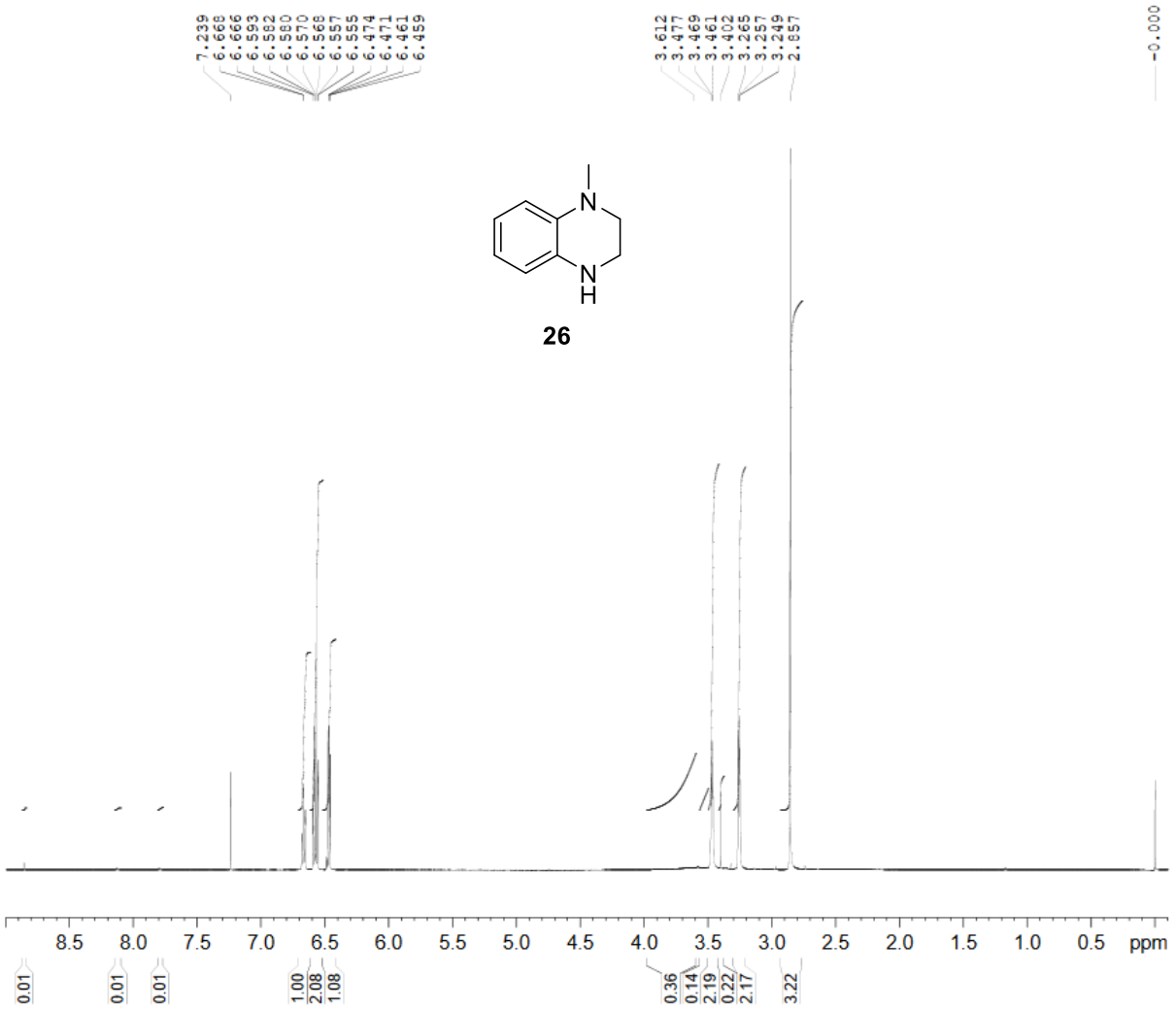
10. De Selms, R. C.; Greaves, R. J.; Schleigh, W. R. *J. Heterocycl. Chem*. **1974**, *11*, 595–597. doi: 10.1002/jhet.5570110425


9.688
9.682
9.595
8.627
8.606
8.536
8.516
8.361
8.344
8.322
8.305
8.285
8.267
7.452
7.432
7.089
7.070
4.729
3.341
2.498
2.494
2.269
0.001
Standard 1H
121202
MIM0341_1
Milen Matyas
2017.02.21. (DA)
SO3
N
N
25
NAME 121202
EXPNO 1
PROCNO 1
Date_ 20170222
Time 15.14
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 64
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 80.6
DW 60.800 usec
DE 6.50 usec
TE 296.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 0.80 dB
PL1W 12.53577709 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300052 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.95
1.98
2.00
1.93
1.98
3.04
3.00

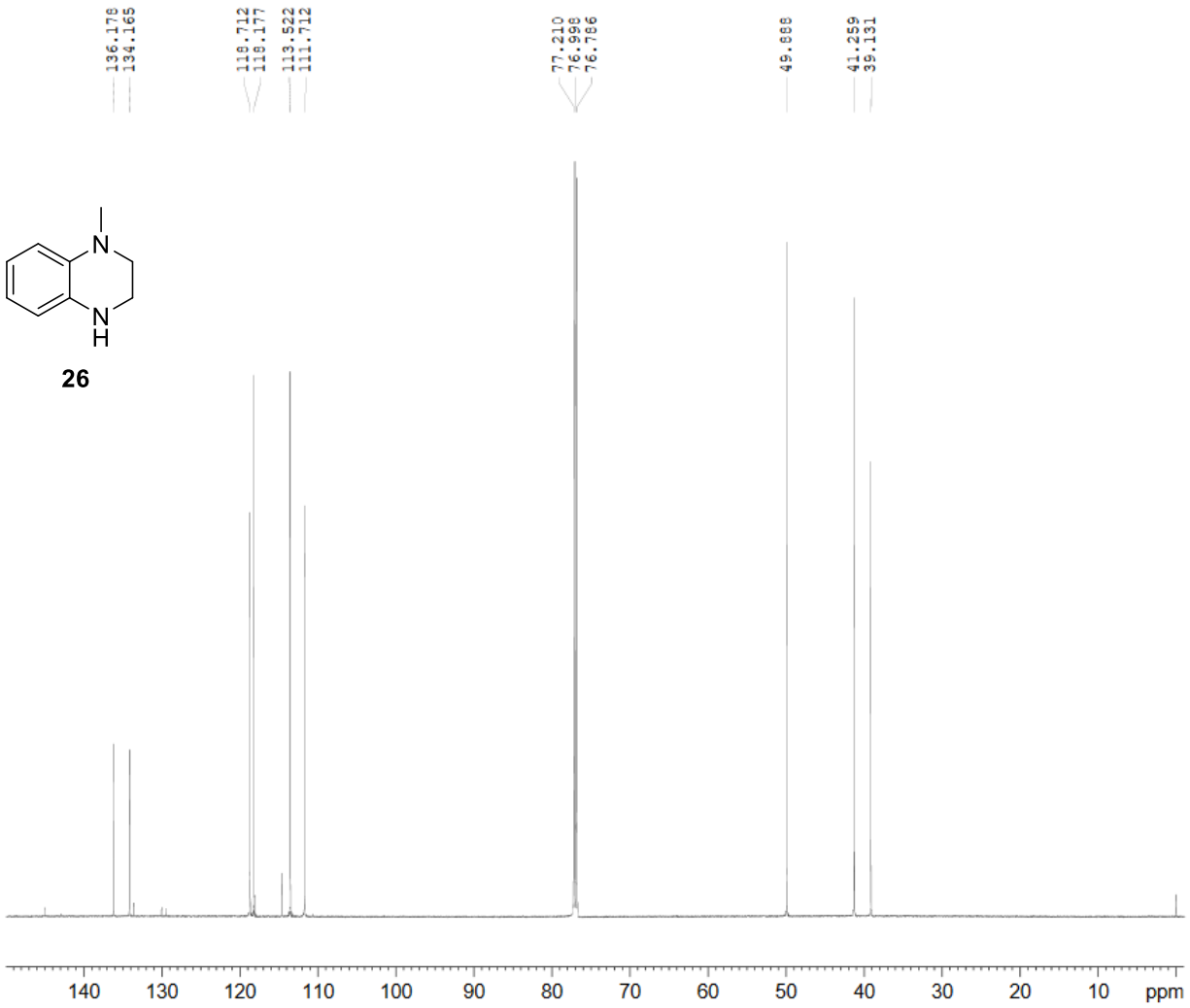


Standard 13C
121202
MIM0341_1
Milen Matyas
2017.02.21. (DA)

NAME	121202	
EXPNO	2	
PROCNO	1	
Date_	20170223	
Time	2.16	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zgpg30	
TD	65536	
SOLVENT	DMSO	
NS	16384	
DS	4	
SWH	24038.461	Hz
FIDRES	0.366798	Hz
AQ	1.3631988	sec
RG	2050	
DW	20.800	usec
DE	6.50	usec
TE	296.8	K
D1	1.00000000	sec
D11	0.03000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	13C	
P1	9.50	usec
PL1	1.44	dB
PL1W	43.99363327	W
SFO1	100.6228298	MHz
======== CHANNEL f2 ========		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	90.00	usec
PL2	0.80	dB
PL12	18.30	dB
PL13	18.40	dB
PL2W	12.53577709	W
PL12W	0.22292118	W
PL13W	0.21784686	W
SFO2	400.1316005	MHz
SI	32768	
SF	100.6128002	MHz
WDW	no	
SSB	0	
LB	0.00	Hz
GB	0	
PC	1.40	



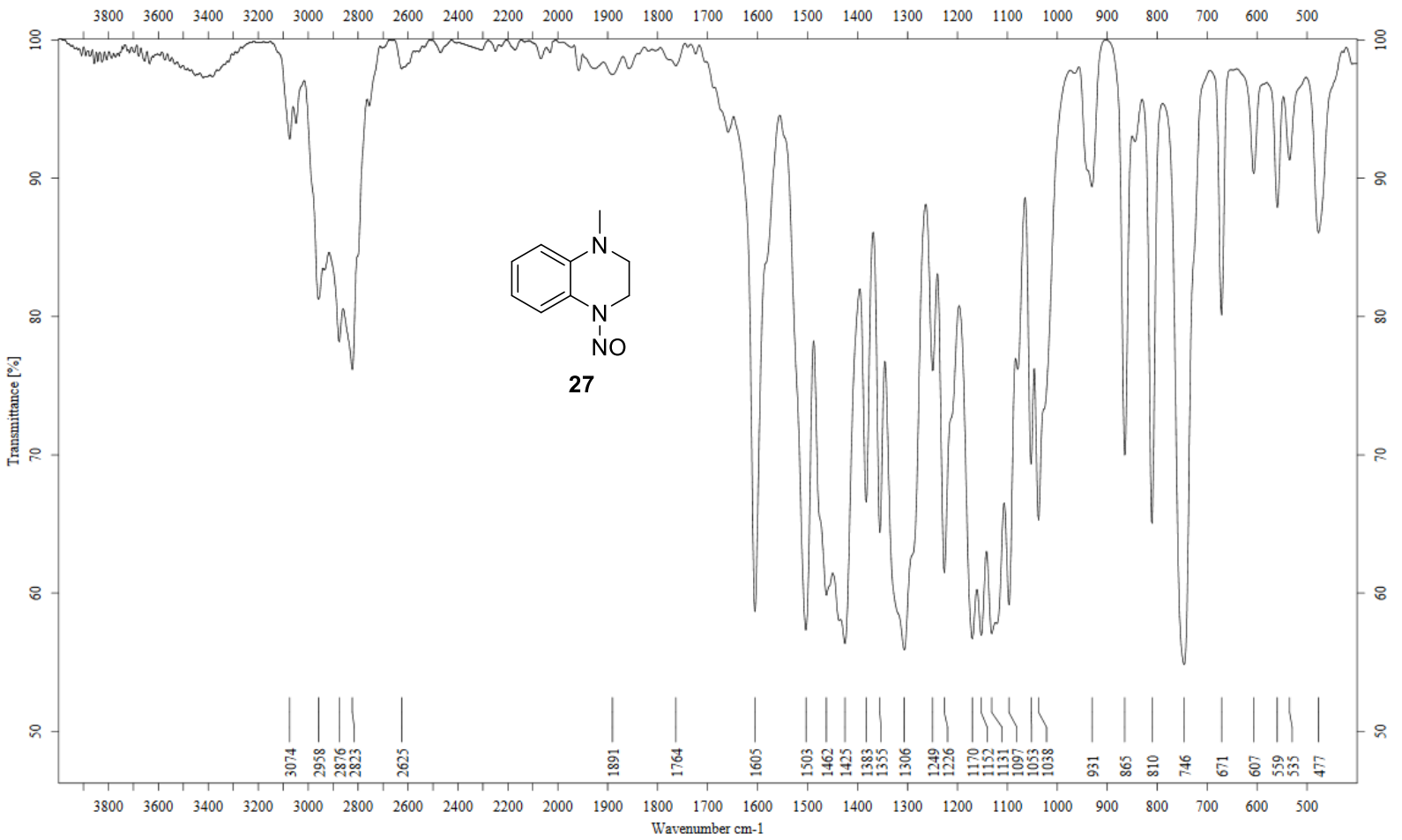

```
Standard 1H
121210
MIM0343_1
Milen Matyas
2017.02.22. (DA)

Current Data Parameters
NAME              121210
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170223
Time_              1.59
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     4
SWH           12626.263 Hz
FIDRES         0.192661 Hz
AQ            2.5952256 sec
RG                 89.22
DW                39.600 usec
DE                25.00 usec
TE                295.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         600.1642011 MHz
NUC1                   1H
P1                 11.50 usec
PLW1         28.00000000 W

F2 - Processing parameters
SI                 65536
SF           600.1600263 MHz
WDW                   no
SSB       0
LB        0 Hz
GB        0
PC                  1.00
```



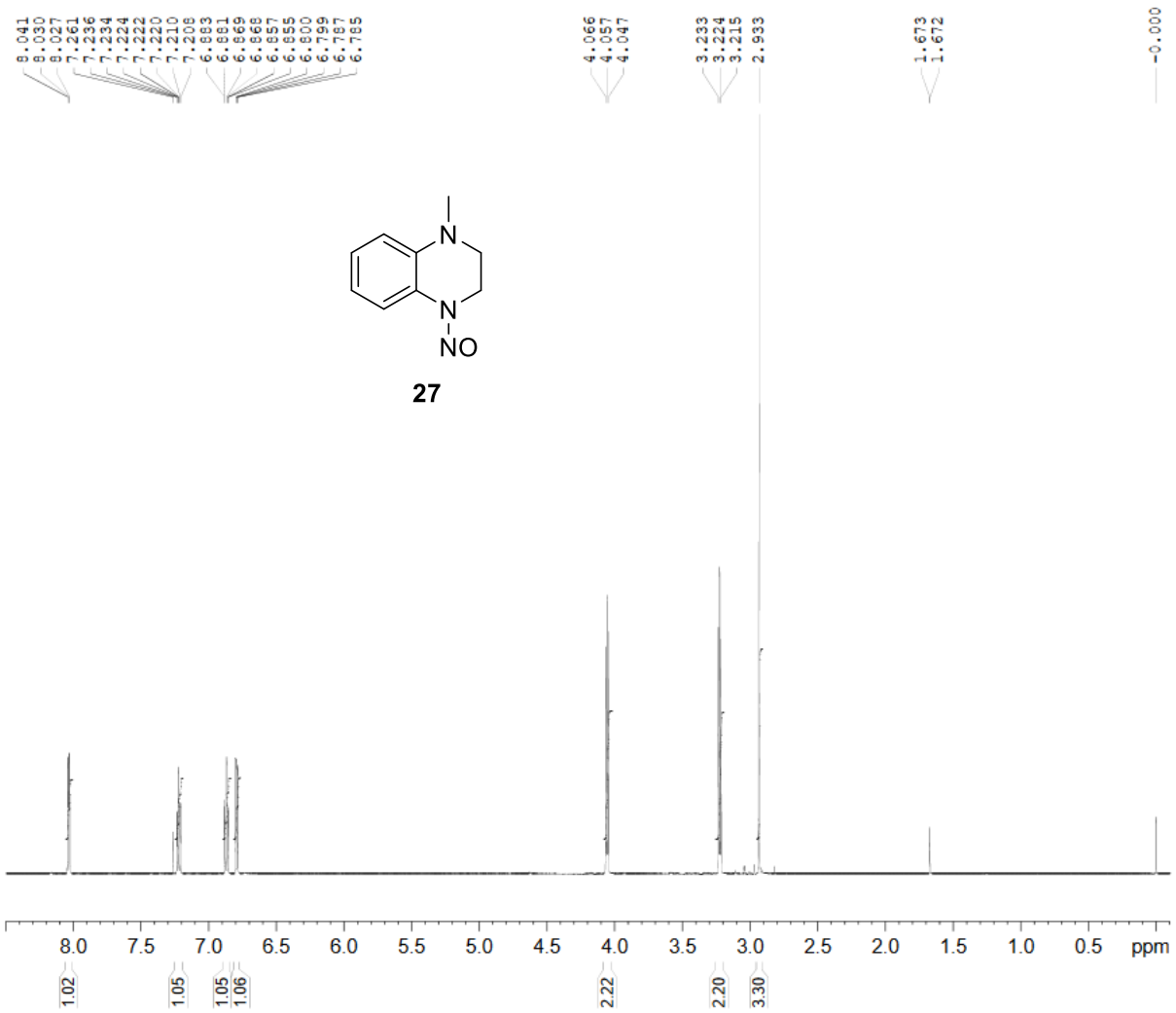

```
Standard 13C
121210
MIM0343_1
Milen Matyas
2017.02.22. (DA)

Current Data Parameters
NAME                  121210
EXPNO                     12
PROCNO                     1

F2 - Acquisition Parameters
Date_               20170223
Time_                  2.43
INSTRUM                spect
PROBHD   5 mm CPPBBO BB
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     1024
DS                        4
SWH               36057.691 Hz
FIDRES             0.550197 Hz
AQ                0.9087659 sec
RG                    196.07
DW                   13.867 usec
DE                    18.00 usec
TE                    295.0 K
D1              1.50000000 sec
D11             0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1           150.9254424 MHz
NUC1                    13C
P1                    10.00 usec
PLW1           65.00000000 W

======== CHANNEL f2 ========
SFO2           600.1624006 MHz
NUC2                    1H
CPDPRG[2           waltz16
PCPD2                 70.00 usec
PLW2           28.00000000 W
PLW12           0.75571001 W
PLW13           0.37029999 W

F2 - Processing parameters
SI                    65536
SF             150.9103658 MHz
WDW                     EM
SSB          0
LB                     1.00 Hz
GB           0
PC                     1.40
```

135068	Milen Matyas	KP
MIM0353_1	Film	2020.10.28.

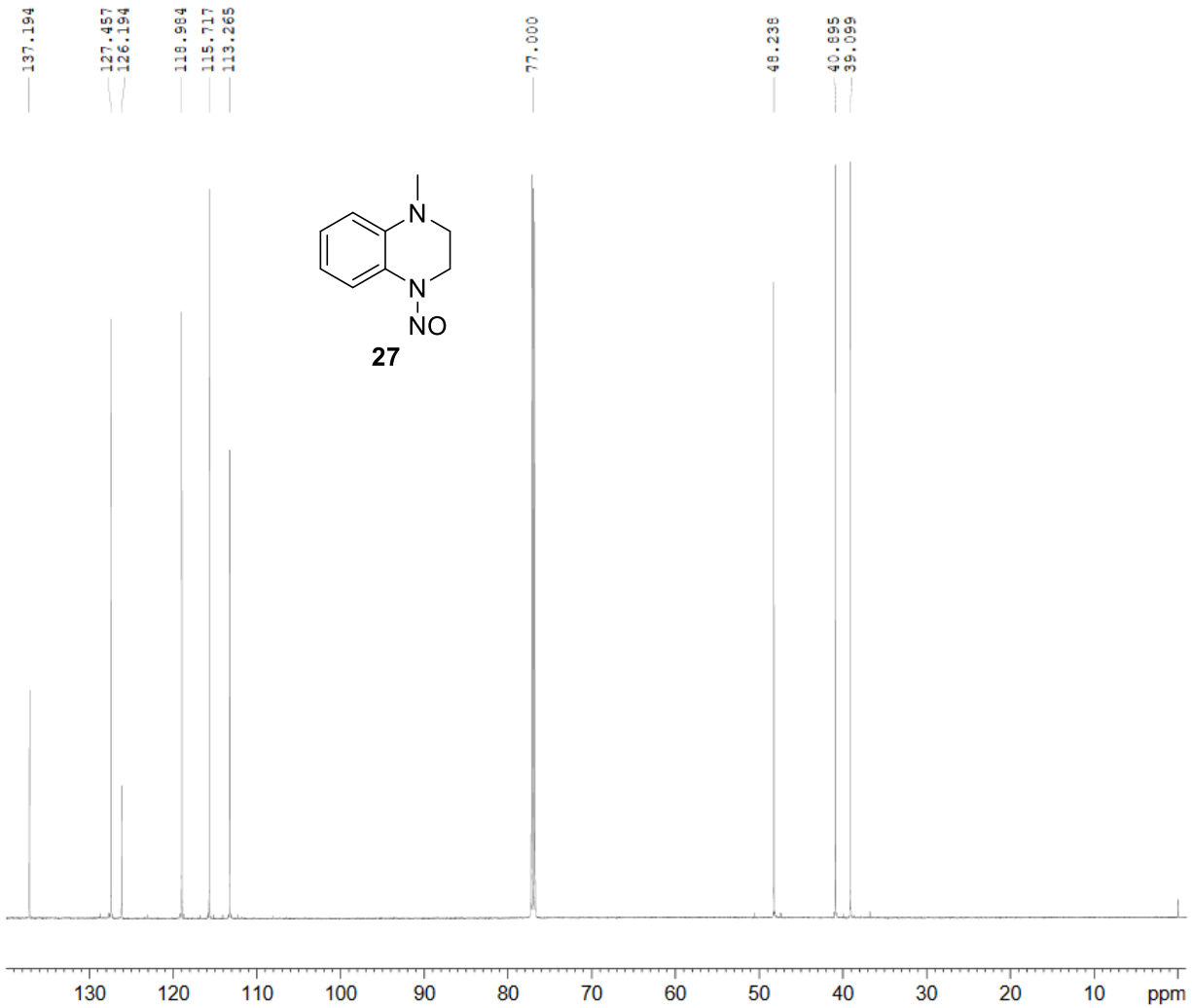
BRUKER Alpha
Resolution: 2 cm-1
Number of Scans: 16



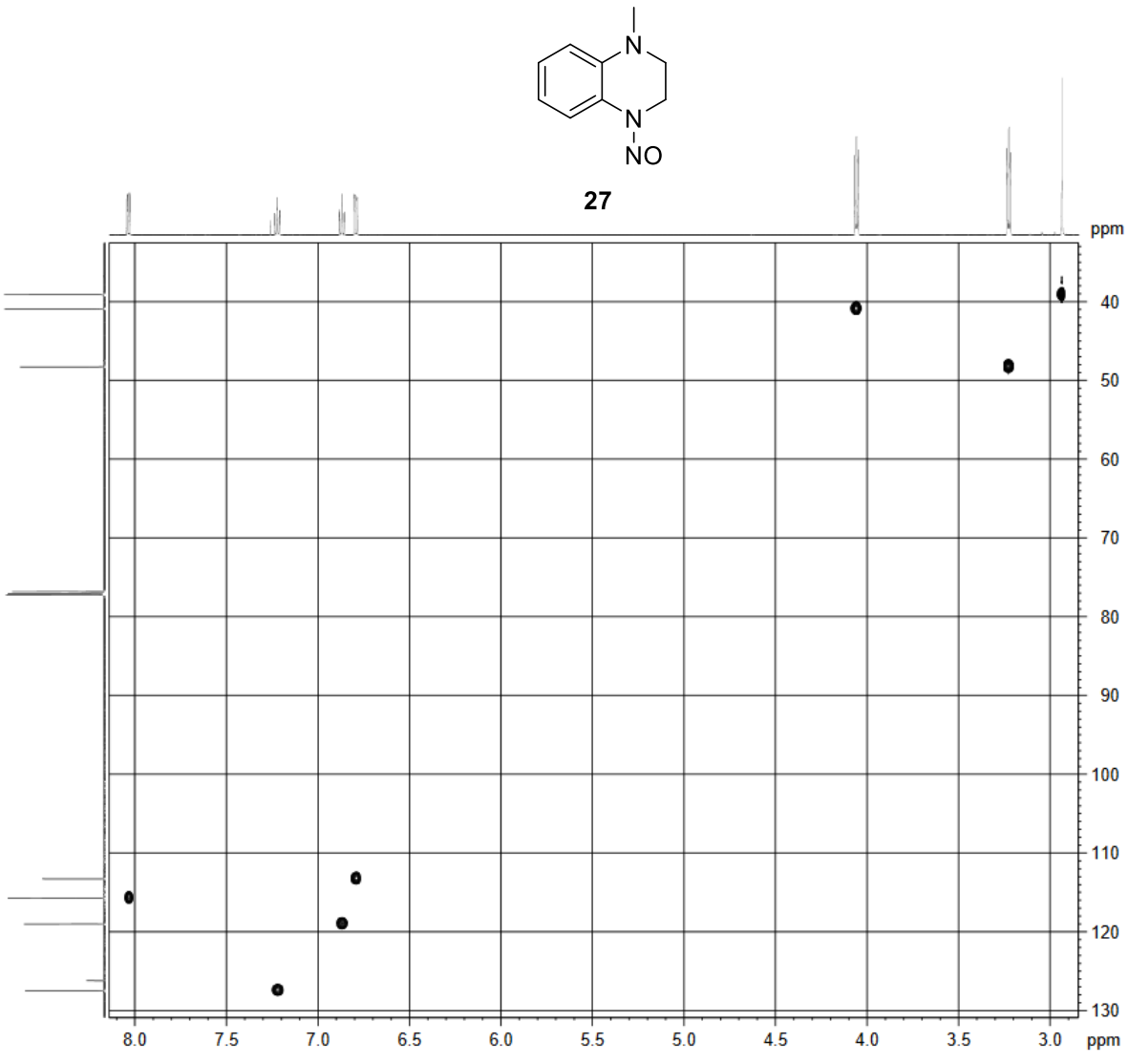
27



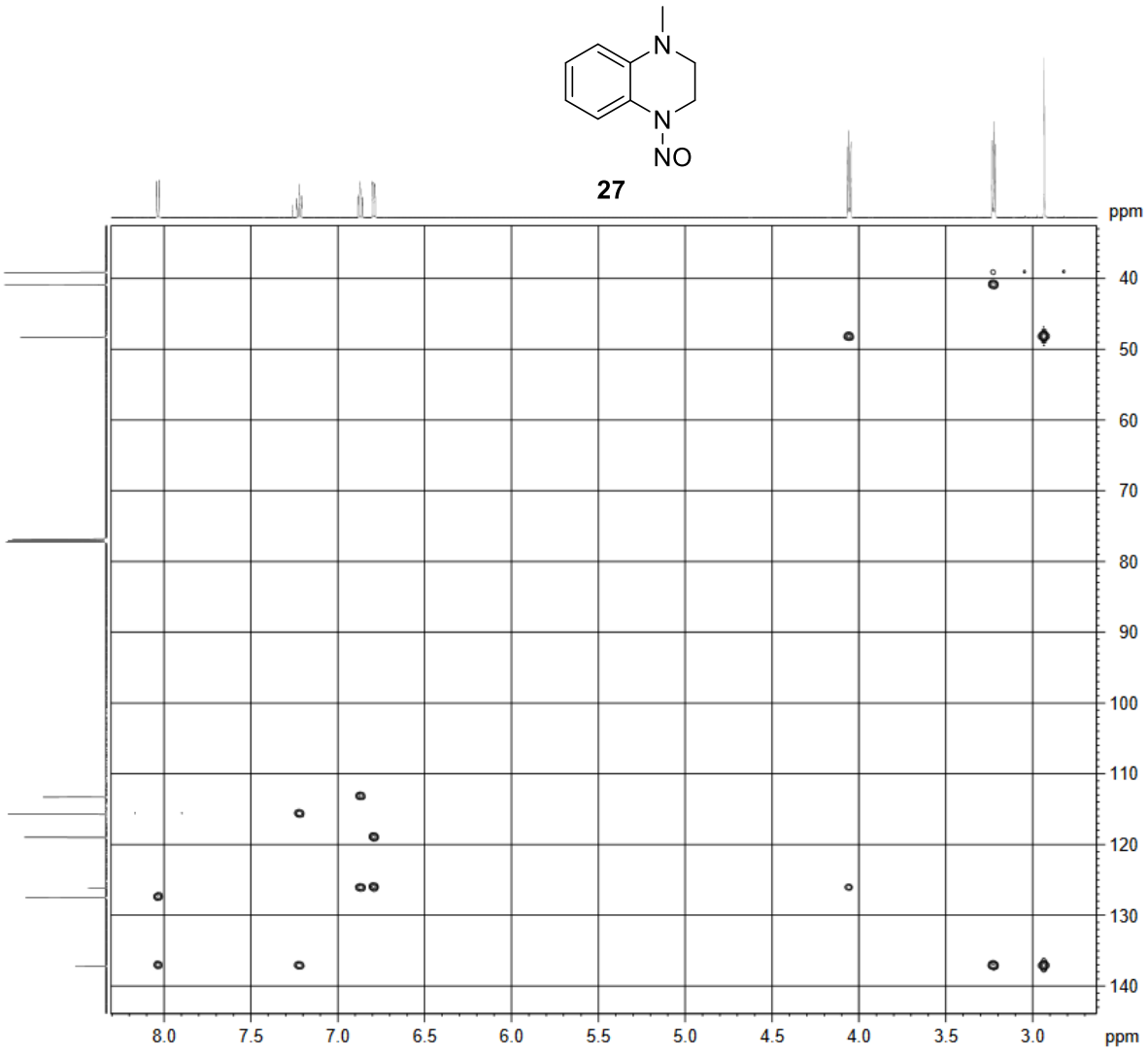

```
Standard 1H
135068
MIM0353_1
Milen Matyas
2020.10.28.(KP)

Current Data Parameters
NAME                 135068
EXPNO                    11
PROCNO                    1

F2 - Acquisition Parameters
Date_              20201028
Time                 19.38 h
INSTRUM                spect
PROBHD   Z145856_0002 (
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH               12019.230 Hz
FIDRES             0.366798 Hz
AQ                2.7262976 sec
RG                    89.22
DW                   41.600 usec
DE                   25.00 usec
TE                   295.0 K
D1              1.00000000 sec
TD0                       1
SFO1            600.1037056 MHz
NUC1                    1H
P1                   11.50 usec
PLW1            28.00000000 W

F2 - Processing parameters
SI                    65536
SF              600.1000142 MHz
WDW                      no
SSB       0
LB        0 Hz
GB        0
PC                    1.00
```




```
Standard 13C
135068
MIM0353_1
Milen Matyas
2020.10.28.(KP)

Current Data Parameters
NAME                135068
EXPNO                   12
PROCNO                   1

F2 - Acquisition Parameters
Date_              20201028
Time                 20.48 h
INSTRUM                spect
PROBHD    Z145856_0002 (
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    2048
DS                       4
SWH              36231.883 Hz
FIDRES            1.105709 Hz
AQ                0.9043968 sec
RG                   196.07
DW                   13.800 usec
DE                   18.00 usec
TE                   295.0 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                      1
SFO1           150.9103545 MHz
NUC1                   13C
P1                   10.00 usec
PLW1           63.09999847 W
SFO2           600.1024004 MHz
NUC2                   1H
CPDPRG[2           waltz16
PCPD2                80.00 usec
PLW2           30.48800087 W
PLW12           0.63000000 W
PLW13           0.31637999 W

F2 - Processing parameters
SI                   32768
SF             150.8952779 MHz
WDW                     EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```

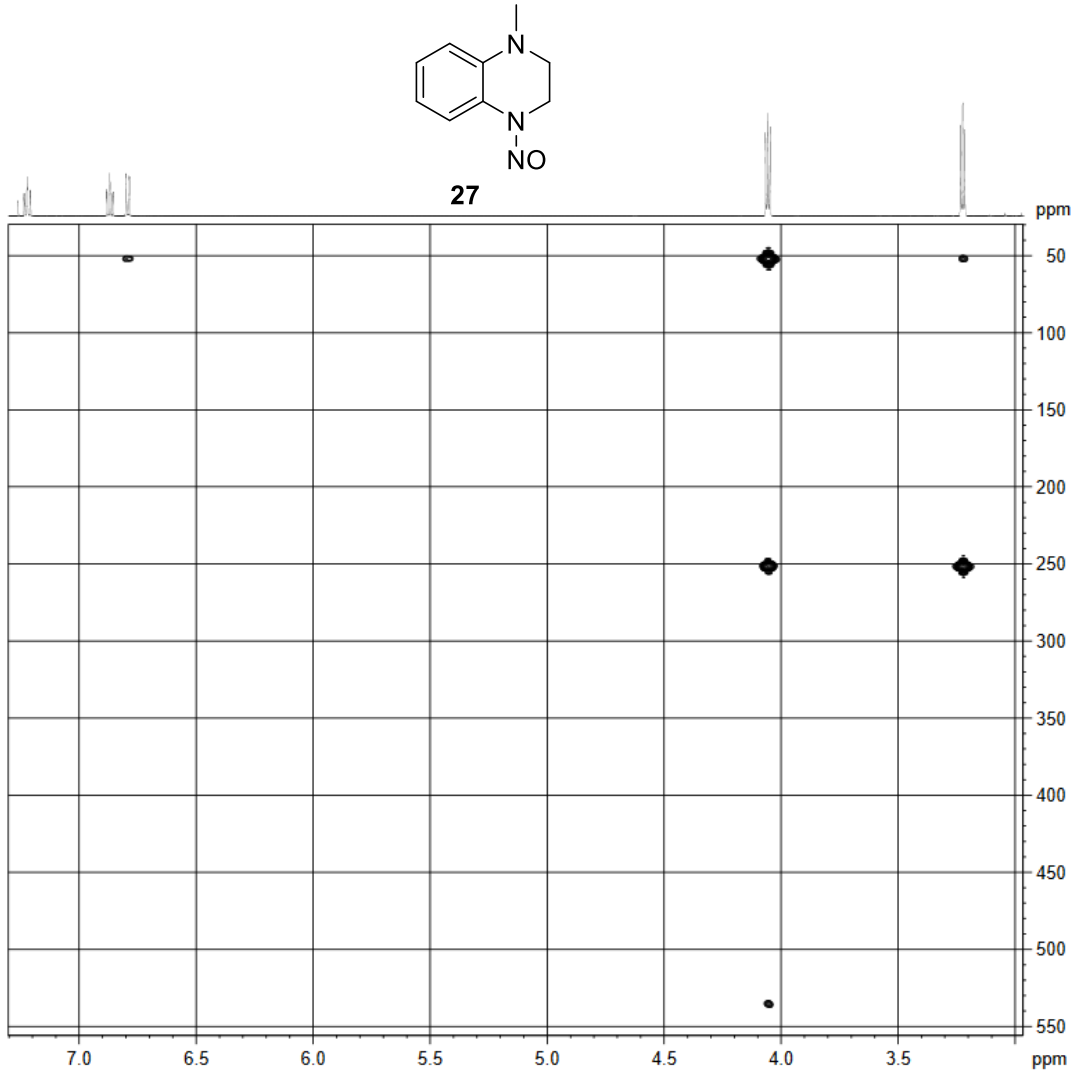



```
HSQC (140Hz)
135068
MIM0353_1
Milen Matyas
2020.10.28.(KP)

Current Data Parameters
NAME                  135068
EXPNO                     13
PROCNO                     1

F2 - Acquisition Parameters
Date_               20201028
Time                   20.50 h
INSTRUM                spect
PROBHD     Z145556_0002 (
PULPROG    hsqcetgpsisp2.2
TD                      2048
SOLVENT              CDCl3
NS                         2
DS                        32
SWH                7812.500 Hz
FIDRES             7.629395 Hz
AQ                 0.13107328 sec
RG                    196.07
DW                    64.000 usec
DE                    25.00 usec
TE                    295.0 K
CNST2             140.0000000
CNST17             -0.5000000
D0                 0.00000300 sec
D1                 1.50000000 sec
D4                 0.00178571 sec
D11                0.03000000 sec
D16                0.00020000 sec
D24                0.00089000 sec
IN0                0.00001810 sec
TDav                       1
SFO1            600.1039007 MHz
NUC1                      1H
P1                    11.50 usec
P2                    23.00 usec
P25          0 usec
PLW1           25.00000000 W
SFO2            150.9118638 MHz
NUC2                      13C
CPDPRG[2 bi_p2m4sp_4sp.2
P3                    10.00 usec
P14                  500.00 usec
P24                 2000.00 usec
P63                 1500.00 usec
PLW0         0 W
PLW2           65.09999847 W
PLW12           2.08599997 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3                0.500
SPOFFS3      0 Hz
SPW3              9.64099979 W
SPNAM[7]     Crp60comp.4
SPOAL7                0.500
SPOFFS7      0 Hz
SPW7              9.64099979 W
SPNAM[14 Crp42,1.5,20.2
SPOAL14               0.500
SPOFFS14     0 Hz
SPW14             3.39900017 W
SPNAM[31 Crp42,1.5,20.2
SPOAL31               0.500
SPOFFS31     0 Hz
SPW31             1.34999997 W
SPNAM[1]      SMSQ10.100
SPI1                  50.00 %
SPNAM[2]      SMSQ10.100
SPI2                  20.10 %
SPNAM[3]      SMSQ10.100
SPI3                  11.00 %
SPNAM[4]      SMSQ10.100
SPI4                  -5.00 %
P16                 1000.00 usec
P19                  600.00 usec

F1 - Acquisition parameters
TD                       256
SFO1            150.9119 MHz
FIDRES         258.692047 Hz
SW              218.417 ppm
FnMODE     Echo-Antiecho

F2 - Processing parameters
SI                      1024
SF           600.1000142 MHz
WDW                   QSINE
```



HMBC (8 Hz, 140Hz)
135068
MIM0353_1
Milen Matyas
2020.10.28.(KP)

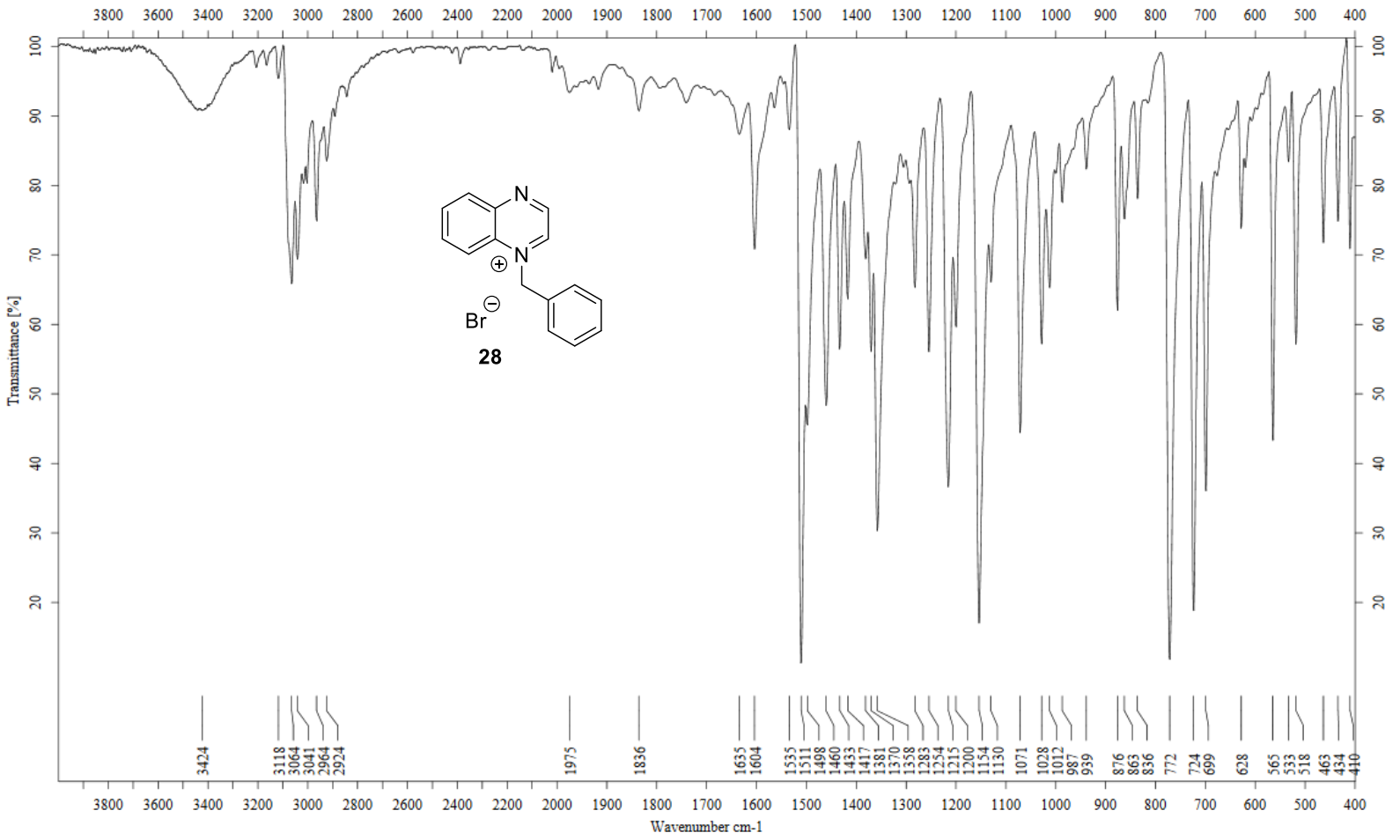
```
Current Data Parameters
NAME                  135068
EXPNO                     14
PROCNO                     1

F2 - Acquisition Parameters
Date_              20201028
Time                   21.05 h
INSTRUM                spect
PROBHD   Z145556_0002 (
PULPROG    hmbcgplpndqf
TD                      2048
SOLVENT              CDCl3
NS                         4
DS                        16
SWH                7812.500 Hz
FIDRES             7.629395 Hz
AQ                 0.1310720 sec
RG                    196.07
DW                   64.000 usec
DE                    25.00 usec
TE                   295.0 K
CNST2             140.0000000
CNST13              8.0000000
D0                  0.00000300 sec
D1                  1.50000000 sec
D2                  0.00357143 sec
D6                  0.06250000 sec
D16                 0.00020000 sec
IN0                 0.00001510 sec
IDay                        7
SFO1               600.1039007 MHz
NUC1                       1H
P1                      11.50 usec
P2                      23.00 usec
PLW1               25.00000000 W
SFO2               150.9118635 MHz
NUC2                       13C
P3                      10.00 usec
PLW2               63.09998547 W
GPNAM[1]           SNSQ10.100
GPZ1                    50.00 %
GPNAM[2]           SNSQ10.100
GPZ2                    30.00 %
GPNAM[3]           SNSQ10.100
GPZ3                    40.10 %
P16                   1000.00 usec

F1 - Acquisition parameters
TD                         256
SFO1               150.9119 MHz
FIDRES             258.692047 Hz
SW                     219.417 ppm
FnMODE                     QF

F2 - Processing parameters
SI                        2048
SF                 600.1000142 MHz
WDW                       SINE
SSB         0
LB          0 Hz
GB          0
PC                        1.40

F1 - Processing parameters
SI                        1024
MC2                         QF
SF                 150.8952779 MHz
WDW                       SINE
SSB         0
LB          0 Hz
GB          0
```



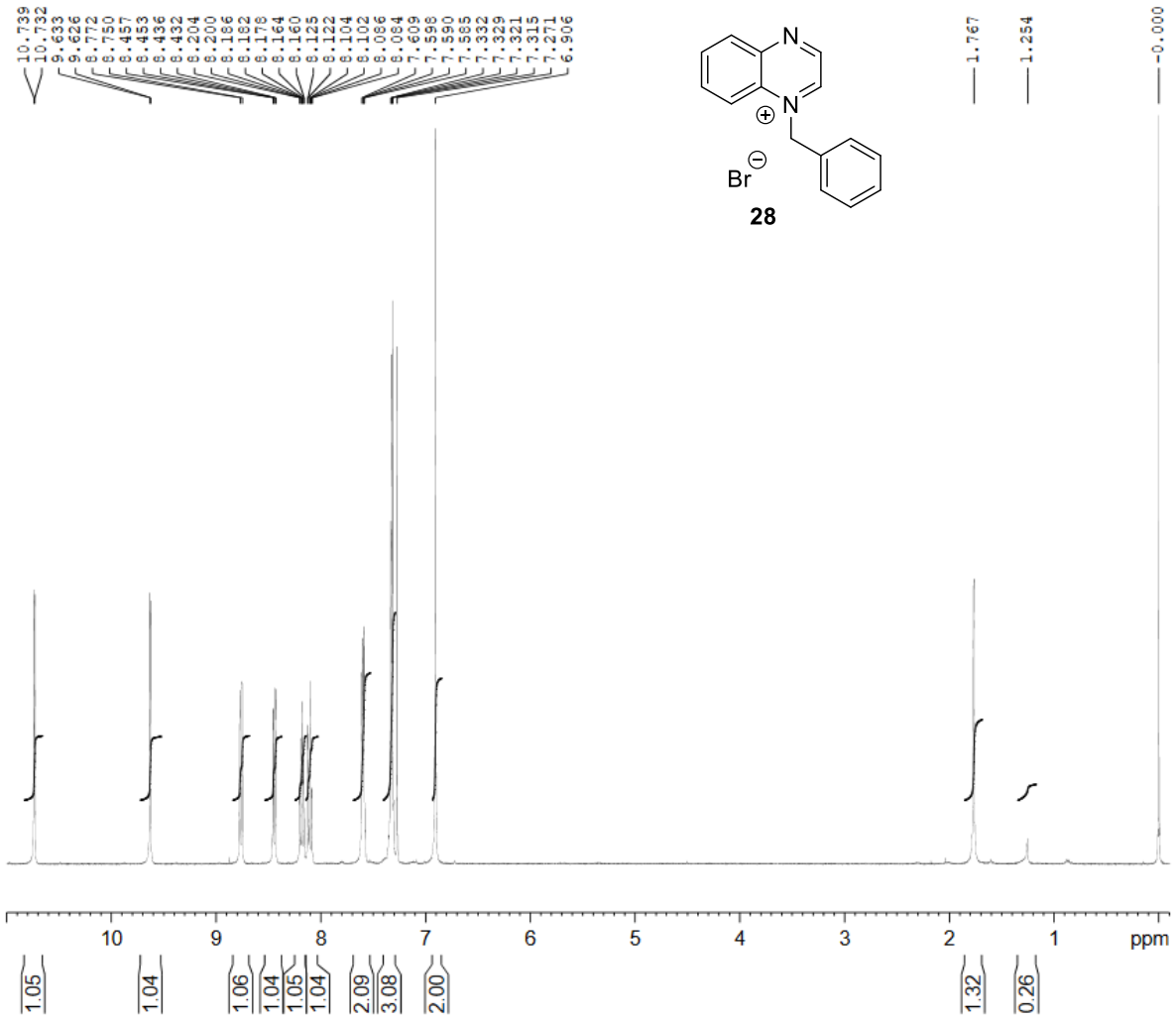

```
NHMBC (5 Hz)
135068
MIM0353_1
Milen Matyas
2020.10.28.(KP)

Current Data Parameters
NAME                  135068
EXPNO                     18
PROCNO                     1

F2 - Acquisition Parameters
Date_              20201028
Time                   21.35 h
INSTRUM                spect
PROBHD   Z145556_0002 (
PULPROG          hmbcgpndqf
TD                       2048
SOLVENT                  CDCl3
NS                         16
DS                         16
SWH                  9615.385 Hz
FIDRES               9.390024 Hz
AQ                   0.1064960 sec
RG                       196.07
DW                       52.000 usec
DE                       25.00 usec
TE                       295.0 K
CNST13               5.0000000
D0                   0.00000300 sec
D1                   2.00000000 sec
D6                   0.10000000 sec
D16                  0.00020000 sec
IN0                  0.00001370 sec
TDav                       1
SFO1              600.1045005 MHz
NUC1                      1H
P1                       11.50 usec
P2                       23.00 usec
PLW1             25.00000000 W
SFO2              60.8258472 MHz
NUC2                     13C
P3                       15.00 usec
PLW2            199.69000244 W
GPNAM[1]          SMSQ10.100
GPZ1                     70.00 %
GPNAM[2]          SMSQ10.100
GPZ2                     30.00 %
GPNAM[3]          SMSQ10.100
GPZ3                     50.10 %
P16                    1000.00 usec

F1 - Acquisition parameters
TD                        128
SFO1              60.82555 MHz
FIDRES           370.255493 Hz
SW                   620.014 ppm
FnMODE                    QF

F2 - Processing parameters
SI                       2048
SF                600.1000142 MHz
WDW                      SINE
SSB          0
LB           0 Hz
GB           0
PC                       1.40

F1 - Processing parameters
SI                       1024
MC2                       QF
SF                60.8076048 MHz
WDW                      SINE
SSB          0
LB           0 Hz
GB           0
```

125082	Milen Matyas	KP
JET0039_1	KBr	2018.03.08.

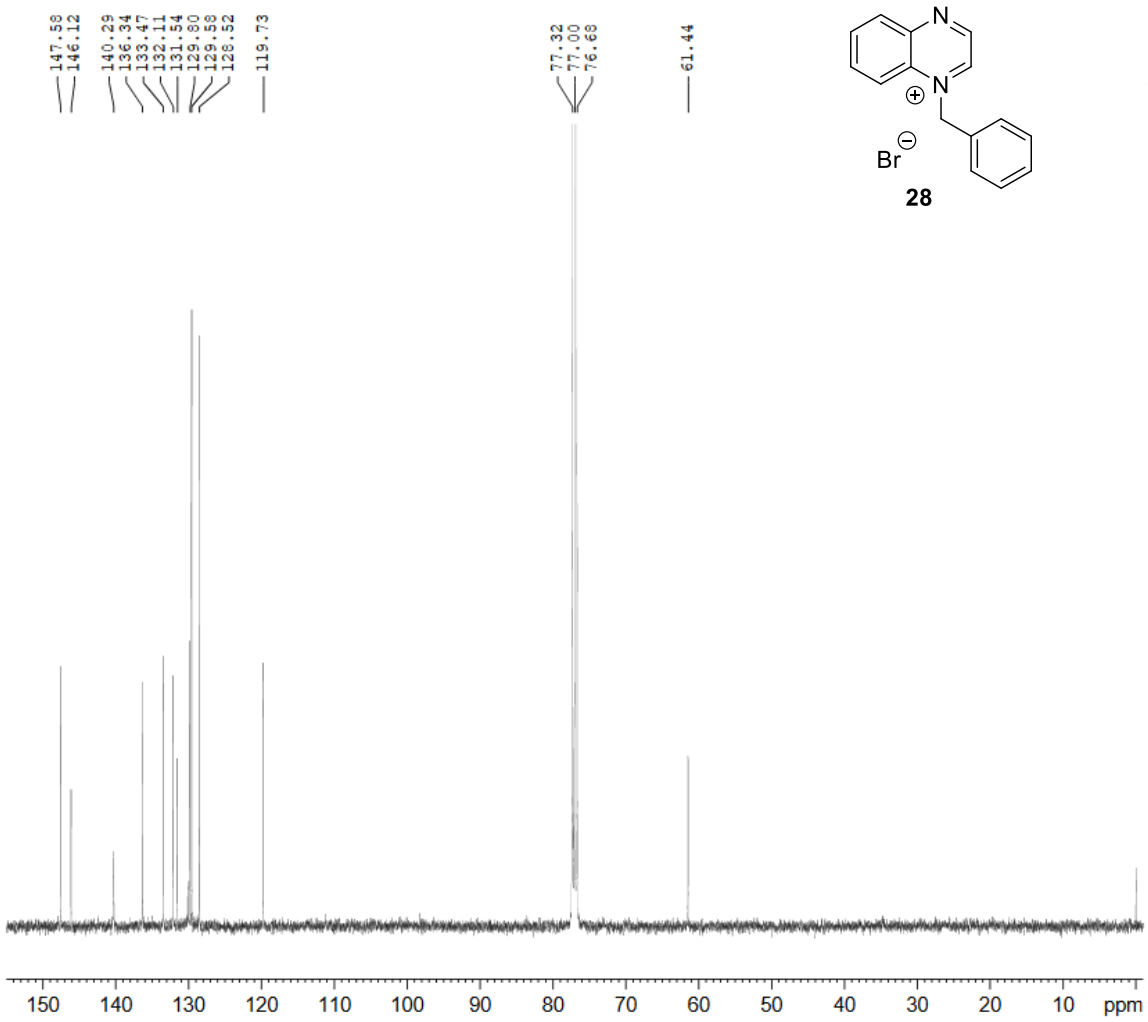
BRUKER Alpha
Resolution: 2 cm-1
Number of Scans: 16



28




```
Standard 1H
125082
JET0039_1
Milen Matyas
2018.03.08. (DA)

Current Data Parameters
NAME                 125082
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20180308
Time                 16.55 h
INSTRUM                spect
PROBHD    Z105190_0010 (
PULPROG                zg30
TD                    65536
SOLVENT               CDC13
NS                       64
DS                        2
SWH                8223.685 Hz
FIDRES             0.250967 Hz
AQ                 3.9845889 sec
RG                      287
DW                    60.800 usec
DE                     6.50 usec
TE                    293.7 K
D1                 1.00000000 sec
TD0                       1
SFO1             400.1324710 MHz
NUC1                     1H
P1                    12.70 usec
PLW1             11.39999962 W

F2 - Processing parameters
SI                    32768
SF               400.1300054 MHz
WDW                      no
SSB          0
LB           0 Hz
GB           0
PC                     1.00
```



28

Standard 13C
125082
JET0039_1
Milen Matyas
2018.03.08. (DA)

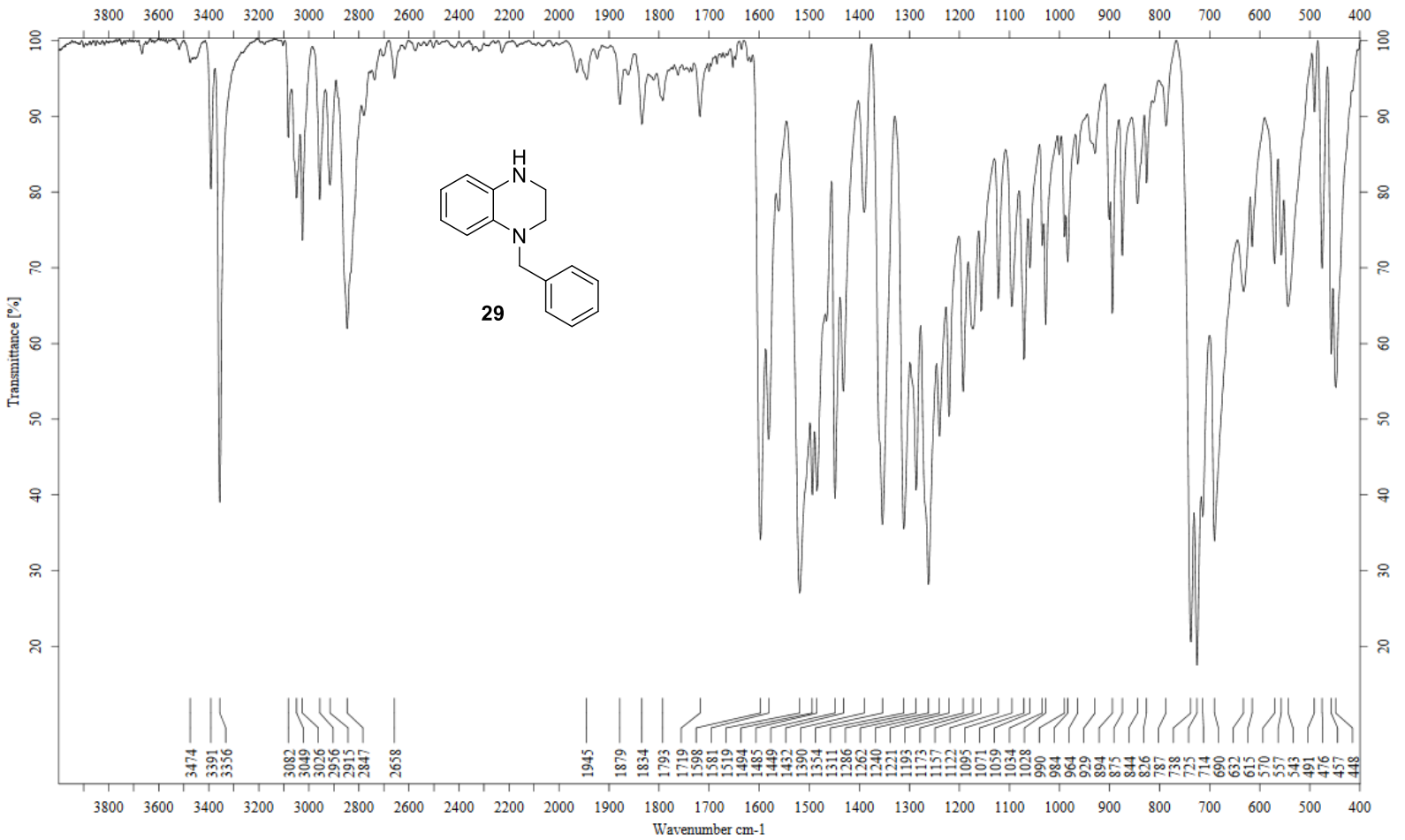
Current Data Parameters
NAME 125082
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180308
Time 21.05 h
INSTRUM spect
PROBHD Z105190_0010 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 6144
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 295.1 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 8.80 usec
PLW1 46.20000076 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 11.39999962 W
PLW12 0.22700000 W
PLW13 0.11418000 W

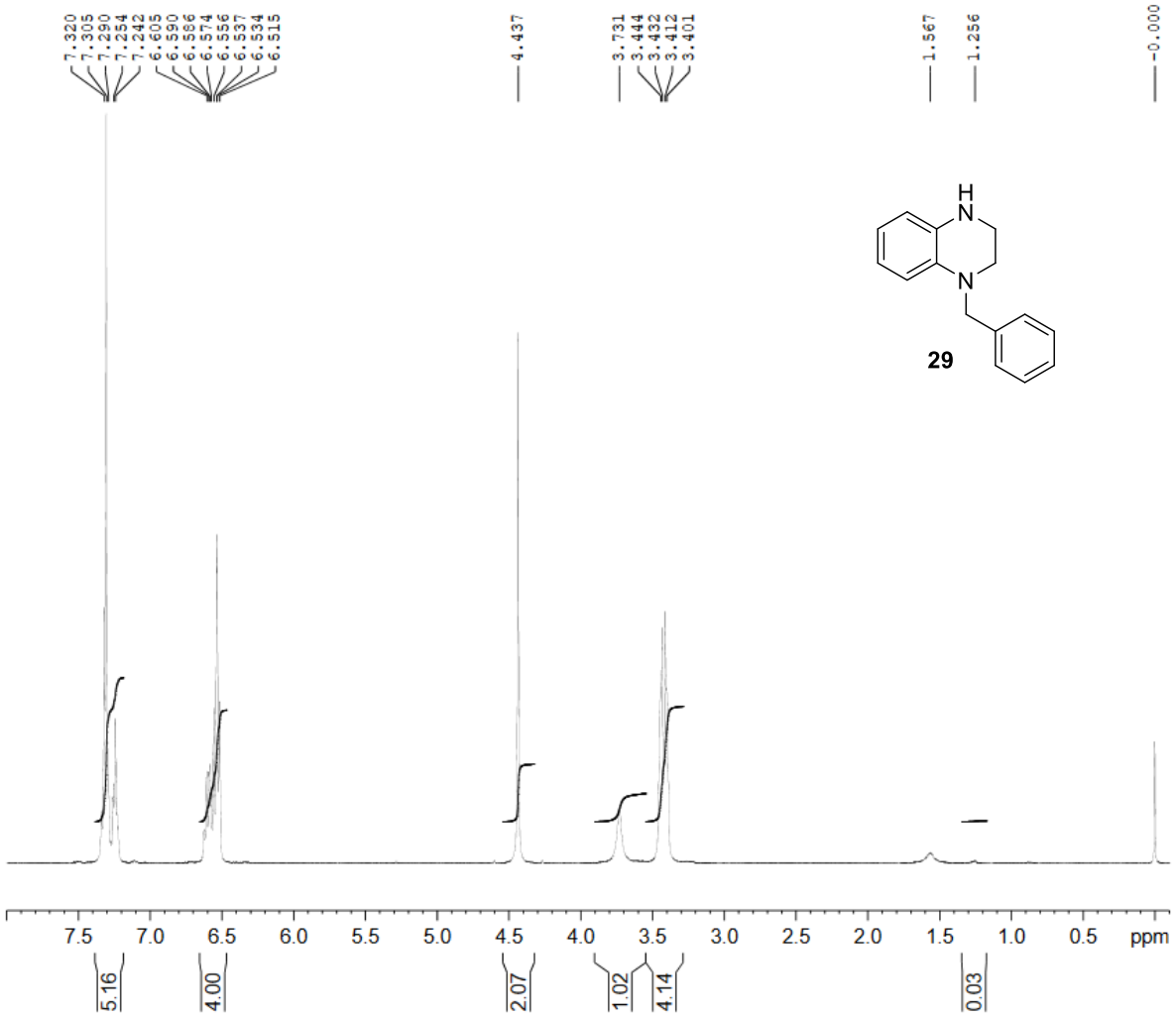
F2 - Processing parameters
SI 32768
SF 100.6127728 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

125191	Milen Matyas	KP
JET0041_1	KBr	2018.03.14.

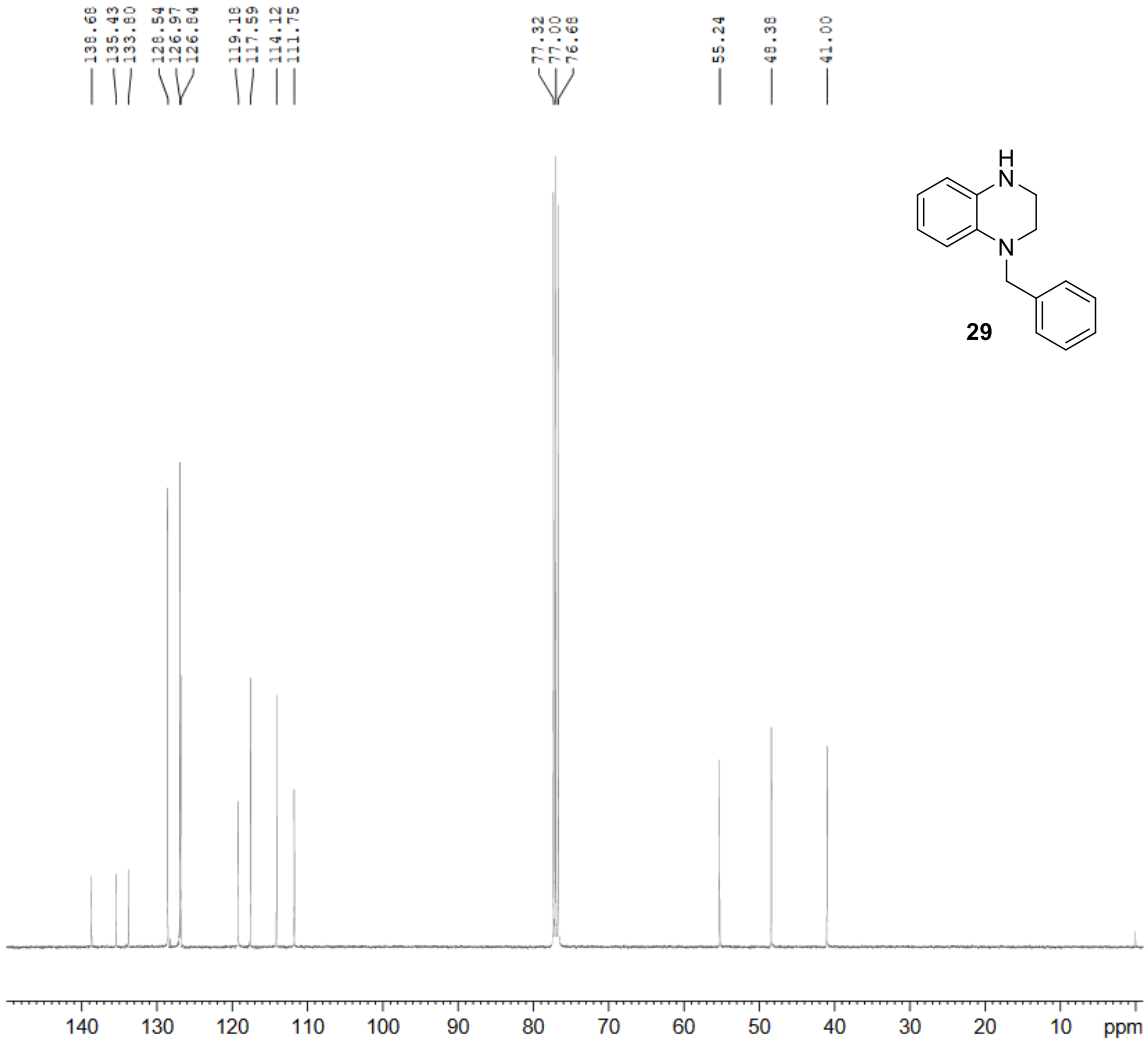
BRUKER Alpha
Resolution: 2 cm-1
Number of Scans: 16



29




```
Standard 1H
125191
JET0041_1
Milen Matyas
2018.03.14. (DA)

Current Data Parameters
NAME                 125191
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20180314
Time                 17.57 h
INSTRUM              spect
PROBHD     Z105190_0010 (
PULPROG              zg30
TD                   65536
SOLVENT              CDCl3
NS                      64
DS                       2
SWH               8223.685 Hz
FIDRES            0.250967 Hz
AQ                3.9845889 sec
RG                     161
DW                  60.800 usec
DE                   6.50 usec
TE                  293.3 K
D1              1.00000000 sec
TD0                      1
SFO1           400.1324710 MHz
NUC1                    1H
P1                  12.70 usec
PLW1           11.39999962 W

F2 - Processing parameters
SI                   32768
SF             400.1300170 MHz
WDW                     no
SSB       0
LB        0 Hz
GB        0
PC                    1.00
```



29

Standard 13C
125191
JET0041_1
Milen Matyas
2018.03.14. (DA)

Current Data Parameters

NAME	125191
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

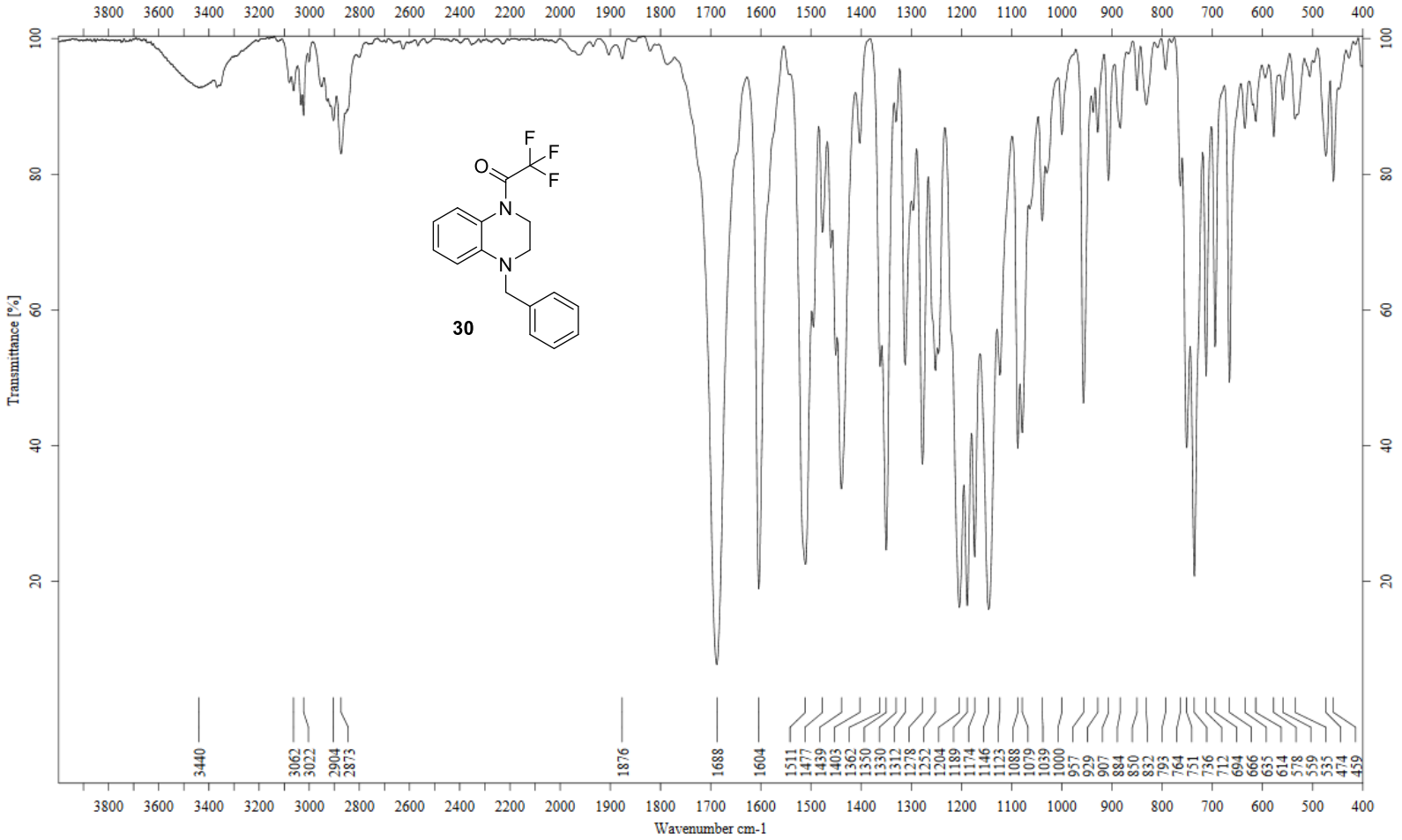
Date_	20180314
Time	23.30 h
INSTRUM	spect
PROBHD	Z105190_0010 (
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	8192
DS	4
SWH	24038.461 Hz
FIDRES	0.733596 Hz
AQ	1.3631488 sec
RG	2050
DW	20.800 usec
DE	6.50 usec
TE	294.1 K
D1	1.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	100.6228298 MHz
NUC1	13C
P1	8.80 usec
PLW1	46.20000076 W
SFO2	400.1316005 MHz
NUC2	1H
CPDPRG[2	waltz16
PCPD2	90.00 usec
PLW2	11.39999962 W
PLW12	0.22700000 W
PLW13	0.11418000 W

F2 - Processing parameters

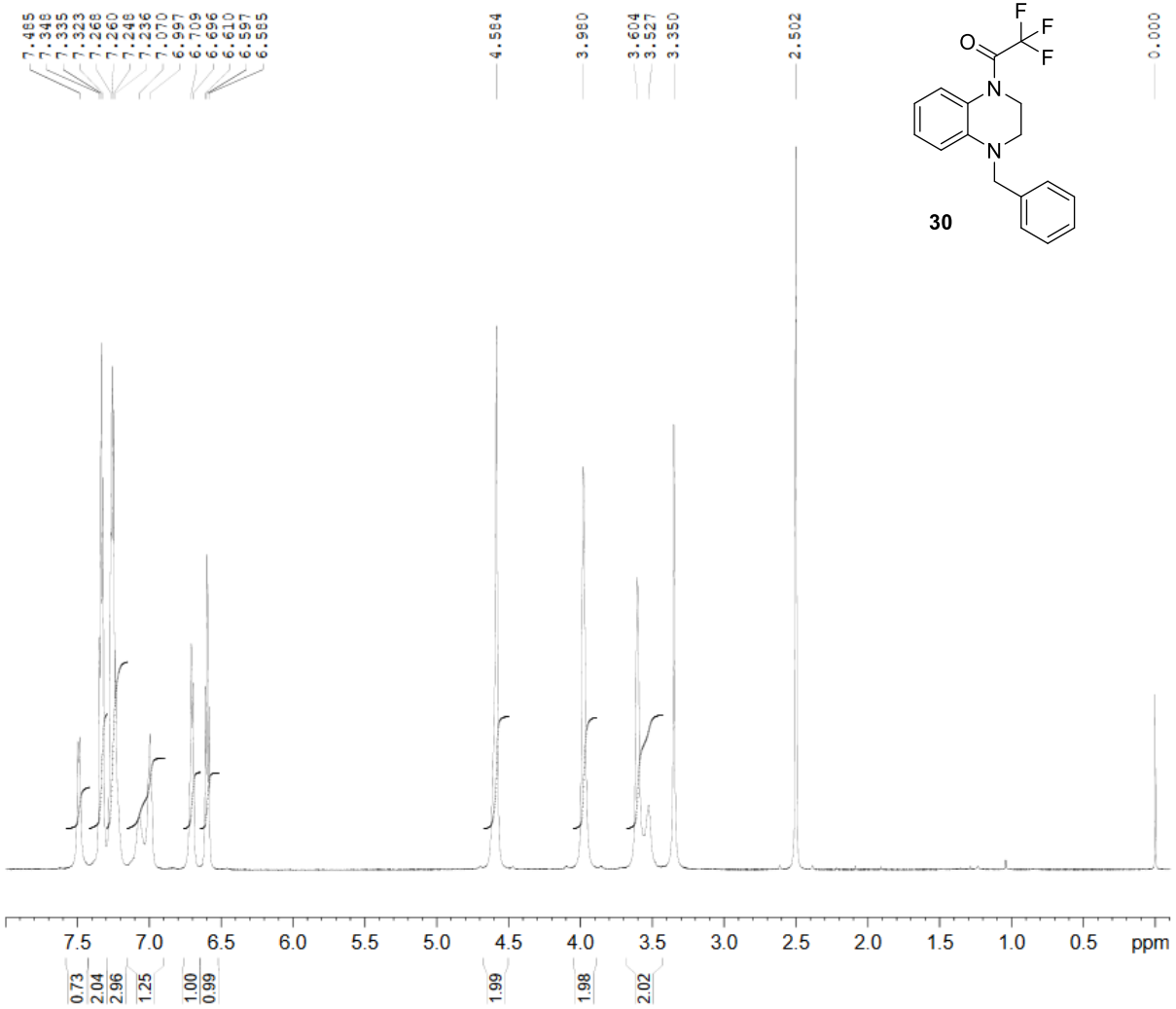
SI	32768
SF	100.6127744 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

125121	Milen Matyas	KP
JET0040_1	KBr	2018.03.09.

BRUKER Alpha
Resolution: 2 cm-1
Number of Scans: 16

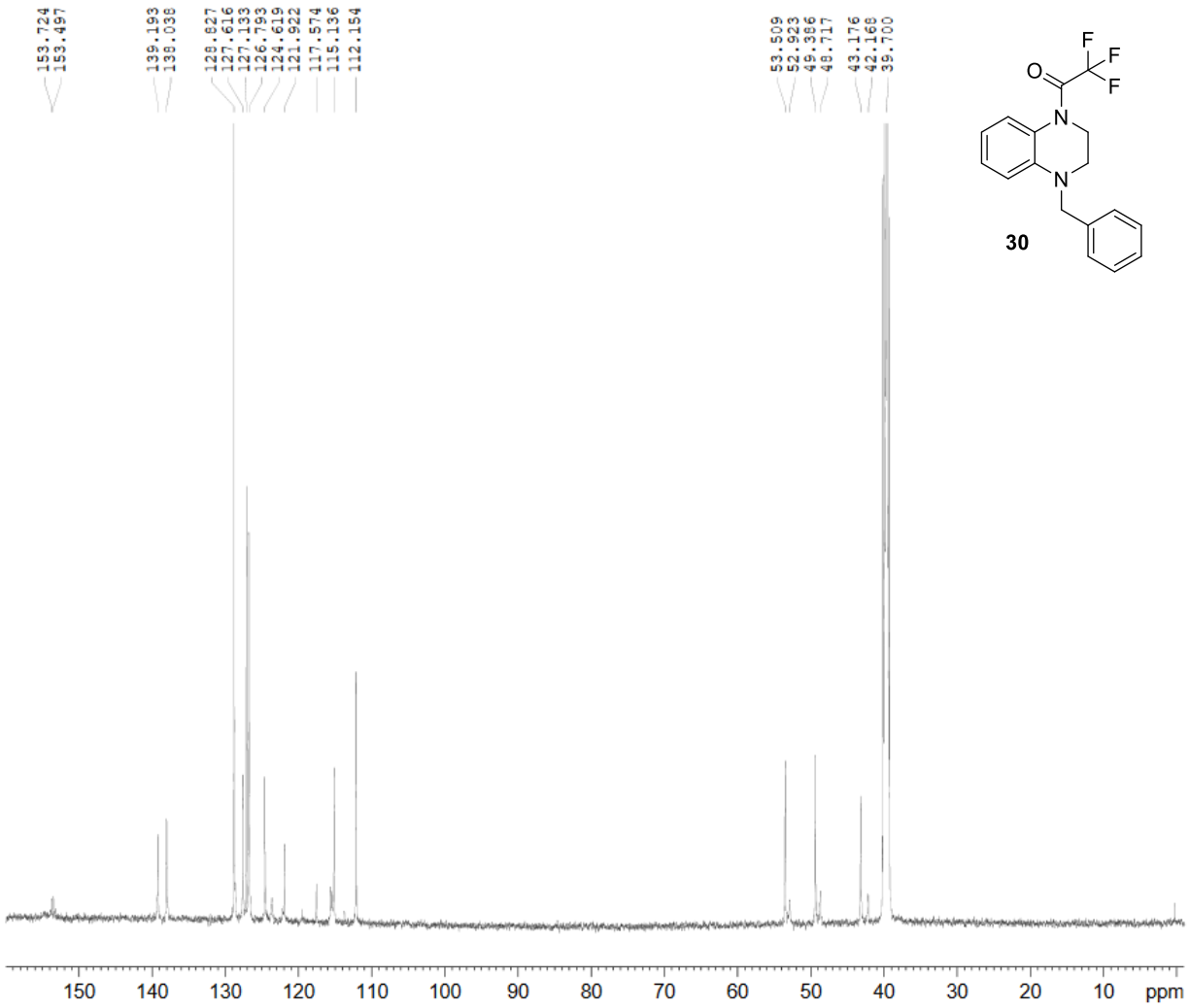


30



30

```
Standard 1H
125121
JET0040_1
Milen Matyas
2018.03.10. (HJ)


Current Data Parameters
NAME                 125121
EXPNO                    21
PROCNO                    1

F2 - Acquisition Parameters
Date_              20180310
Time                 16.40 h
INSTRUM                spect
PROBHD   Z145856_0002 (
PULPROG                zg30
TD                    65536
SOLVENT                DMSO
NS                       16
DS                        4
SWH              12626.263 Hz
FIDRES            0.385323 Hz
AQ                2.5952256 sec
RG                   133.88
DW                   39.600 usec
DE                   25.00 usec
TE                   295.0 K
D1             1.00000000 sec
TD0                       1
SFO1           600.1642011 MHz
NUC1                    1H
P1                   11.50 usec
PLW1           28.00000000 W

F2 - Processing parameters
SI                    65536
SF             600.1600039 MHz
WDW                      no
SSB       0
LB        0 Hz
GB        0
PC                    1.00
```

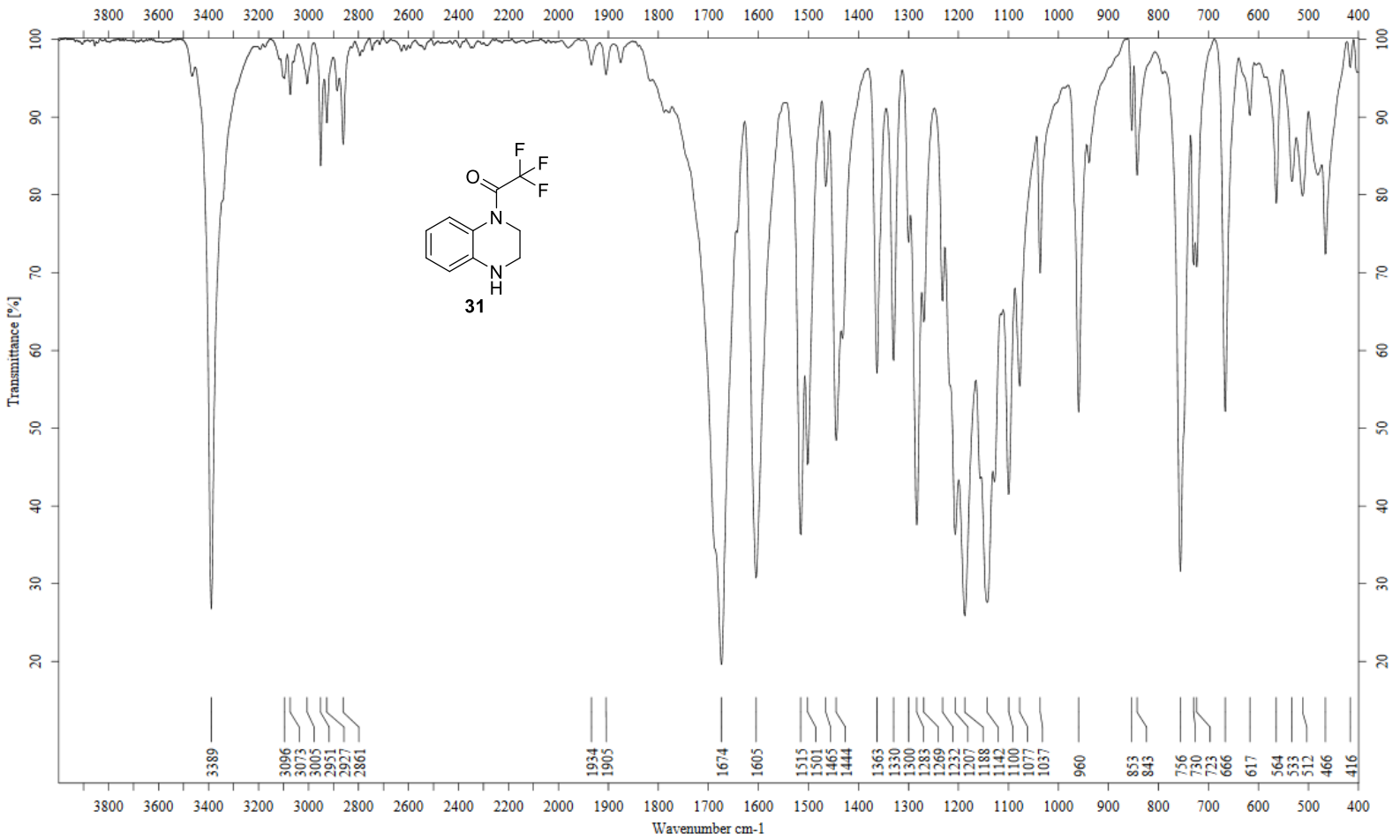


30

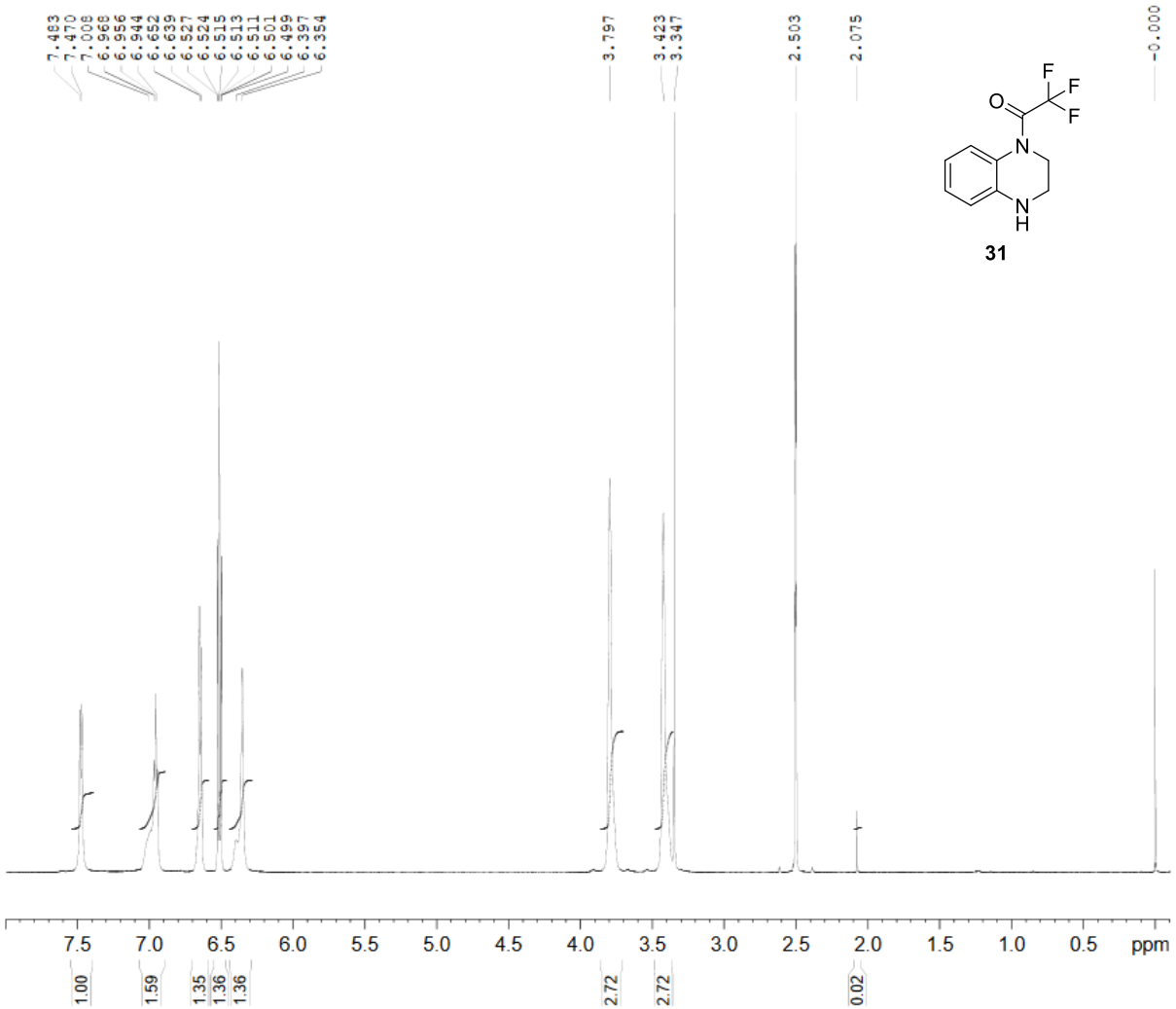
```
Standard 13C
125121
JET0040_1
Milen Matyas
2018.03.10. (HJ)

Current Data Parameters
NAME                125121
EXPNO                   22
PROCNO                   1

F2 - Acquisition Parameters
Date_              20180310
Time                 19.30 h
INSTRUM                spect
PROBHD    Z145856_0002 (
PULPROG             zgpg30
TD                   65536
SOLVENT                DMSO
NS                    4096
DS                       4
SWH              36057.691 Hz
FIDRES            1.100393 Hz
AQ               0.9087659 sec
RG                  196.07
DW                  13.867 usec
DE                  18.00 usec
TE                  295.0 K
D1              1.50000000 sec
D11             0.03000000 sec
TD0                      1
SFO1           150.9254424 MHz
NUC1                   13C
P1                  10.00 usec
PLW1           60.36299896 W
SFO2           600.1624006 MHz
NUC2                   1H
CPDPRG[2            waltz16
PCPD2               80.00 usec
PLW2           26.42600060 W
PLW12           0.59460002 W
PLW13           0.29861000 W

F2 - Processing parameters
SI                   65536
SF             150.9103920 MHz
WDW                     EM
SSB          0
LB                   3.00 Hz
GB           0
PC                   1.40
```

125128	Milen Matyas	KP
MIM0445_1	KBr	2018.03.10.

BRUKER Alpha
Resolution: 2 cm-1
Number of Scans: 16

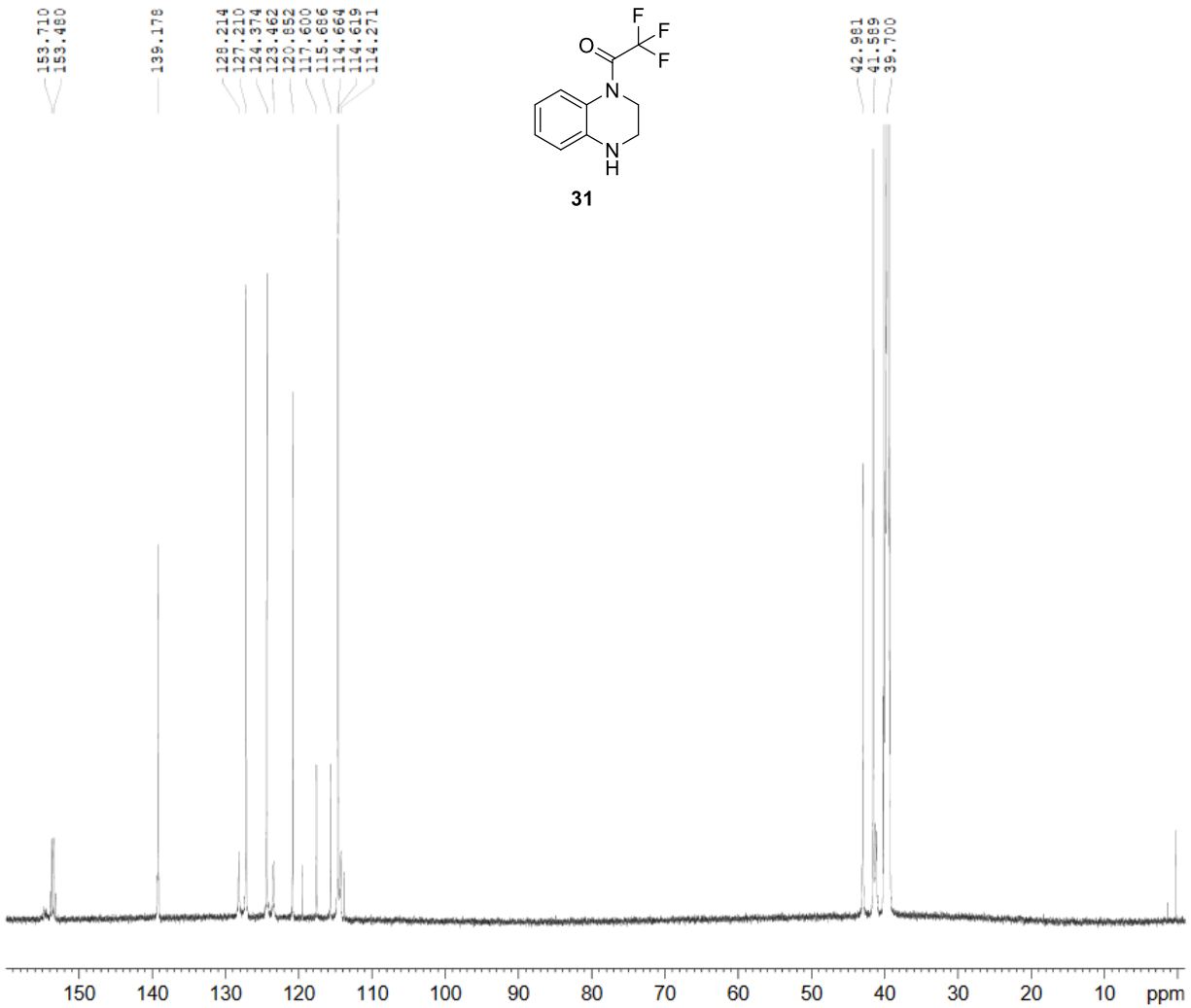


31



31

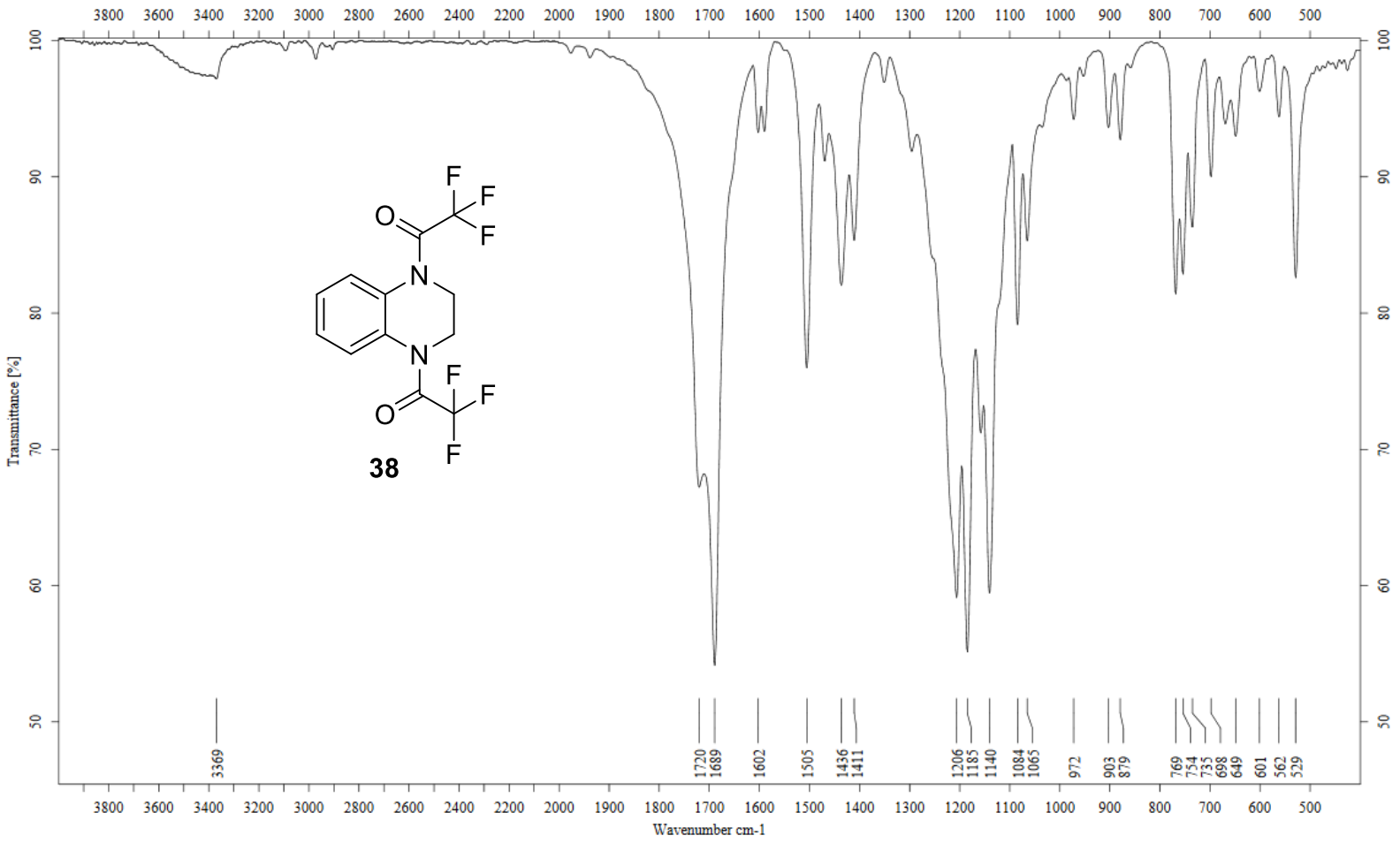
```
Standard 1H
125128
MIM0445_1
Milen Matyas
2018.03.10. (HJ)


Current Data Parameters
NAME                 125128
EXPNO                    11
PROCNO                    1

F2 - Acquisition Parameters
Date_              20180310
Time                 19.36 h
INSTRUM                spect
PROBHD   Z145856_0002 (
PULPROG                zg30
TD                    65536
SOLVENT                DMSO
NS                       16
DS                        4
SWH              12626.263 Hz
FIDRES            0.385323 Hz
AQ                2.5952256 sec
RG                   174.19
DW                   39.600 usec
DE                   25.00 usec
TE                   295.0 K
D1             1.00000000 sec
TD0                       1
SFO1           600.1642011 MHz
NUC1                    1H
P1                   11.50 usec
PLW1           28.00000000 W

F2 - Processing parameters
SI                    65536
SF             600.1600030 MHz
WDW                      EM
SSB       0
LB                    0.10 Hz
GB        0
PC                    1.00
```



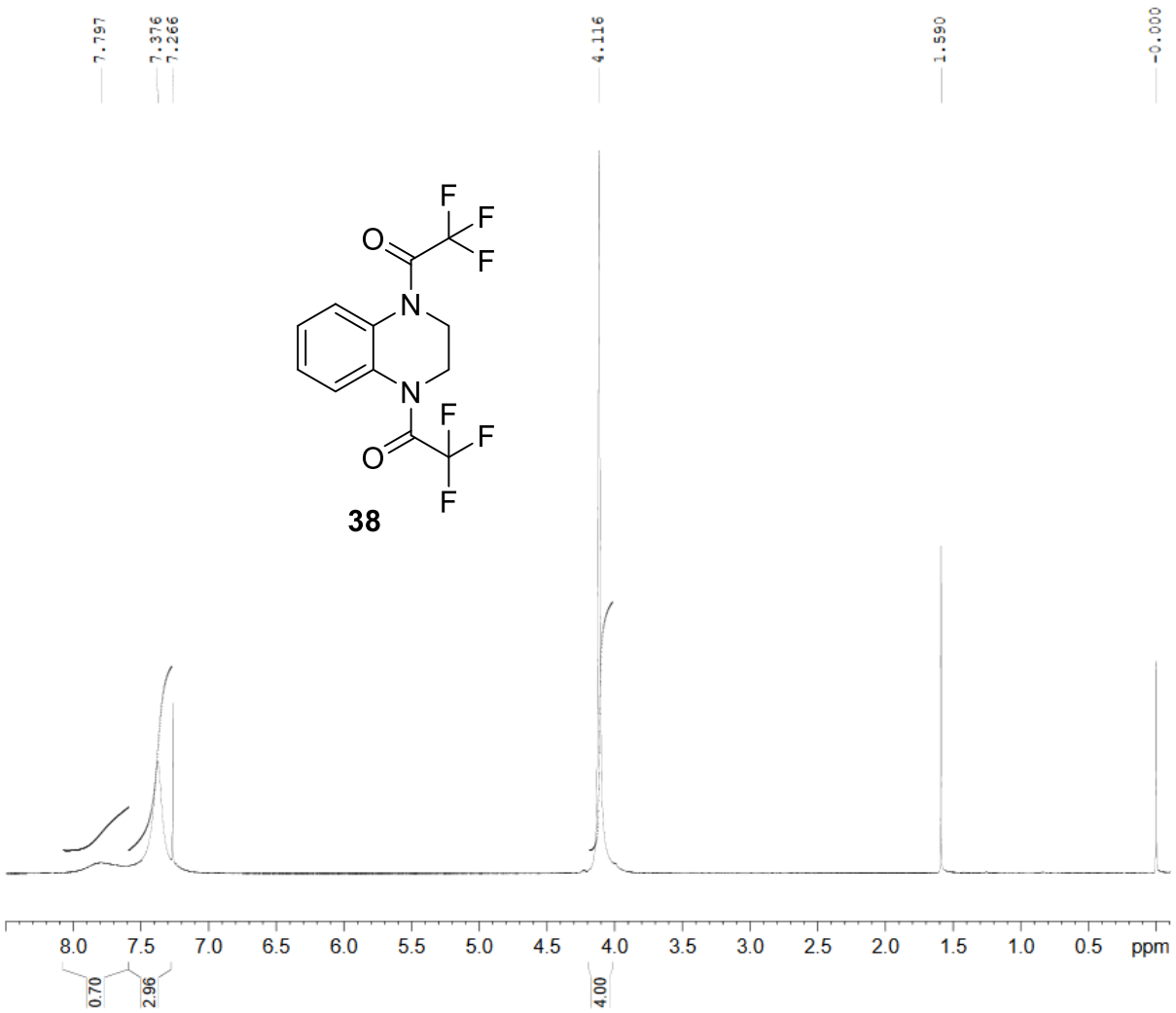

```
Standard 13C
125128
MIM0445_1
Milen Matyas
2018.03.10. (HJ)

Current Data Parameters
NAME                 125128
EXPNO                    12
PROCNO                    1

F2 - Acquisition Parameters
Date_              20180310
Time                 22.26 h
INSTRUM                spect
PROBHD    Z145856_0002 (
PULPROG             zgpg30
TD                   65536
SOLVENT               DMSO
NS                    4096
DS                       4
SWH              36057.691 Hz
FIDRES            1.100393 Hz
AQ               0.9087659 sec
RG                  196.07
DW                  13.867 usec
DE                  18.00 usec
TE                  295.0 K
D1              1.50000000 sec
D11             0.03000000 sec
TD0                      1
SFO1           150.9254424 MHz
NUC1                   13C
P1                  10.00 usec
PLW1           60.36299896 W
SFO2           600.1624006 MHz
NUC2                   1H
CPDPRG[2           waltz16
PCPD2               80.00 usec
PLW2           26.42600060 W
PLW12           0.59460002 W
PLW13           0.29861000 W

F2 - Processing parameters
SI                   65536
SF             150.9103911 MHz
WDW                     EM
SSB       0
LB                   1.00 Hz
GB        0
PC                   1.40
```

137810	Milen Matyas	KP
MIM0517_1	KBr	2021.12.17.

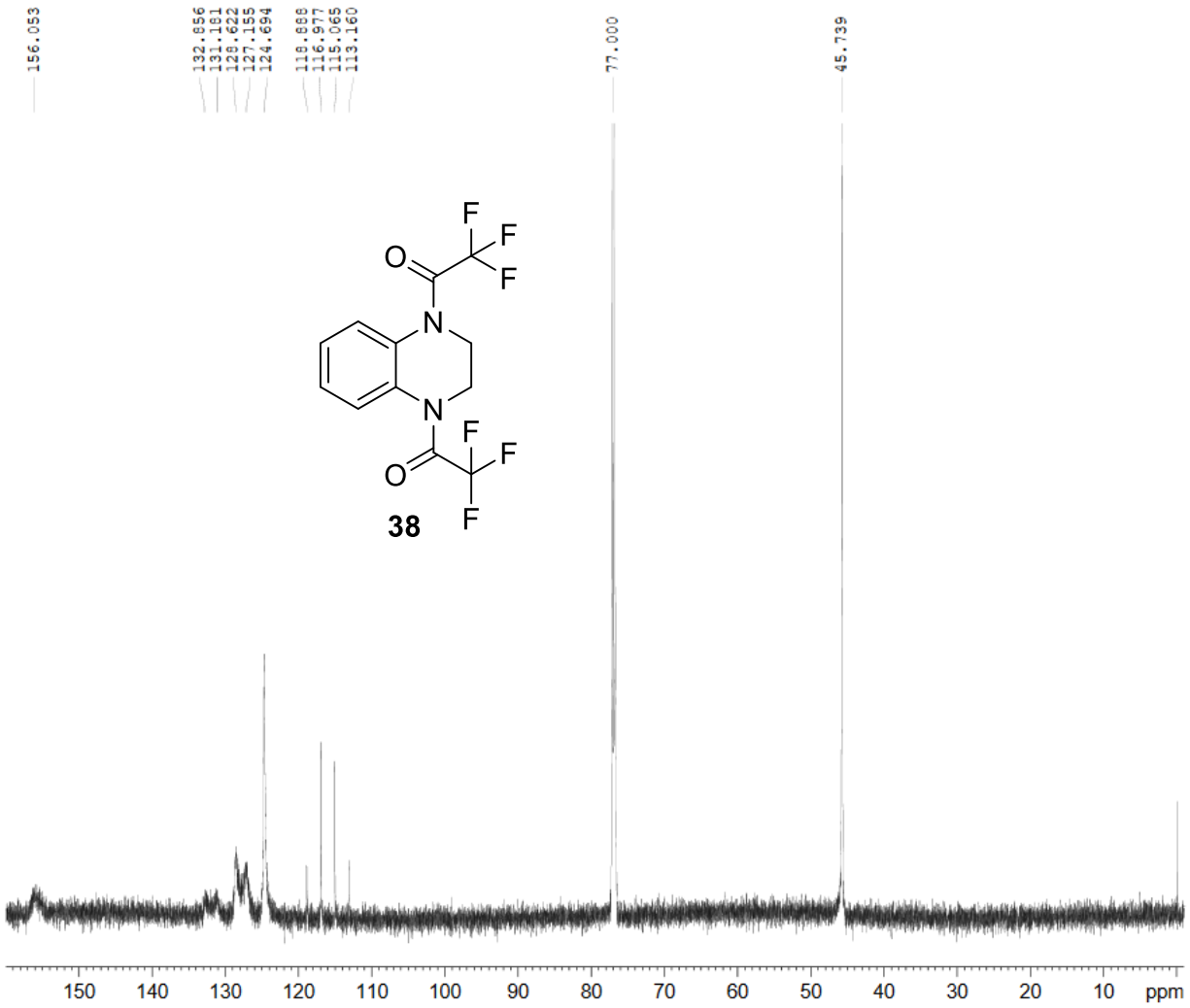
BRUKER Alpha
Resolution: 2 cm-1
Number of Scans: 16



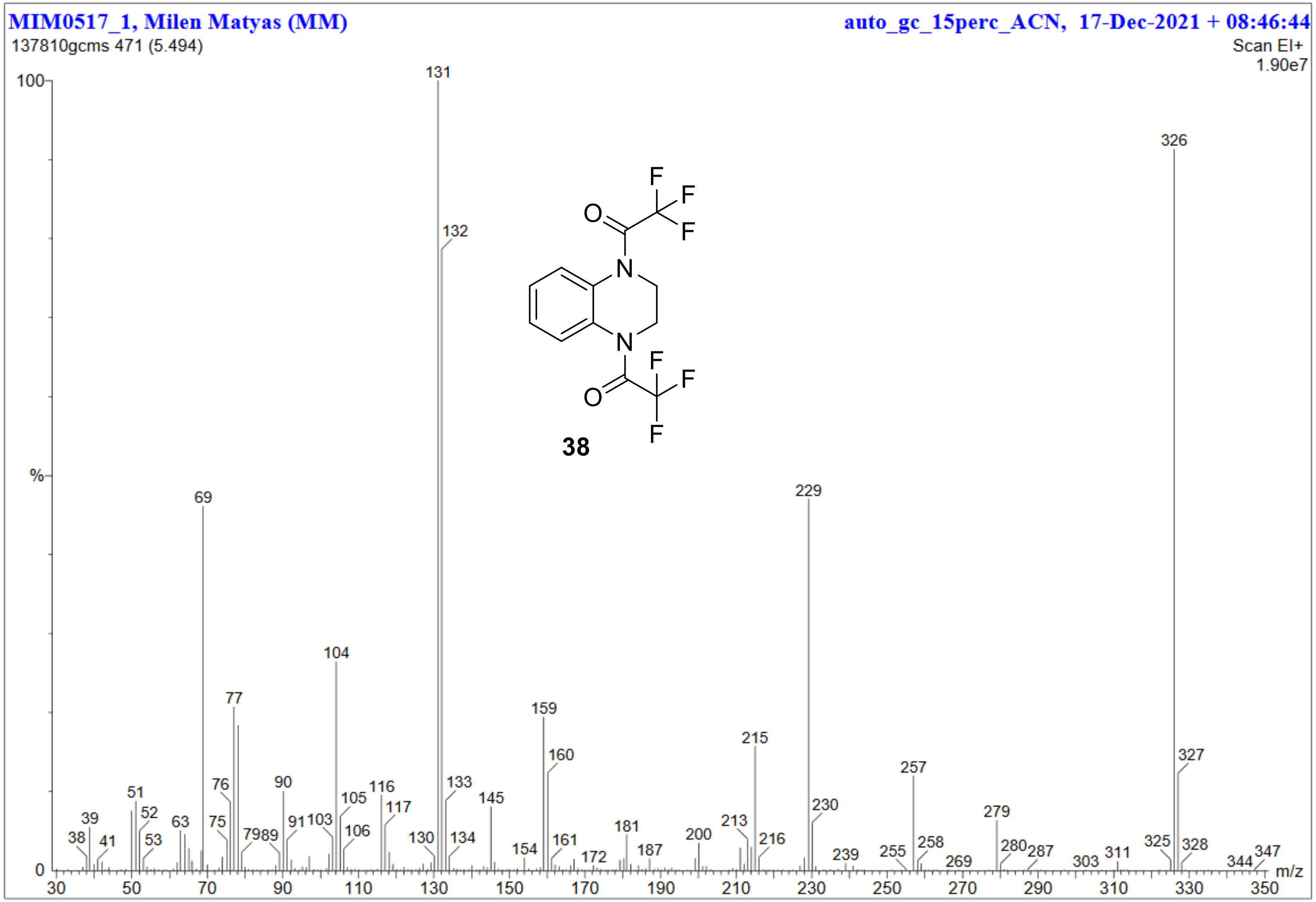
38




```
Standard 1H
137810
MIM0517_1
Milen Matyas
2021.12.17. (KP)

Current Data Parameters
NAME              137810
EXPNO                 13
PROCNO                 1

F2 - Acquisition Parameters
Date_           20211217
Time                12.33 h
INSTRUM             spect
PROBHD   Z145856_0002 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             12019.230 Hz
FIDRES              0.366798 Hz
AQ              2.7262976 sec
RG                196.07
DW                 41.600 usec
DE                 25.00 usec
TE                 295.0 K
D1            1.00000000 sec
TD0                    1
SFO1          600.0537053 MHz
NUC1                  1H
P1                 11.50 usec
PLW1          28.00000000 W

F2 - Processing parameters
SI                 65536
SF            600.0500117 MHz
WDW                   no
SSB       0
LB        0 Hz
GB        0
PC                  1.00
```

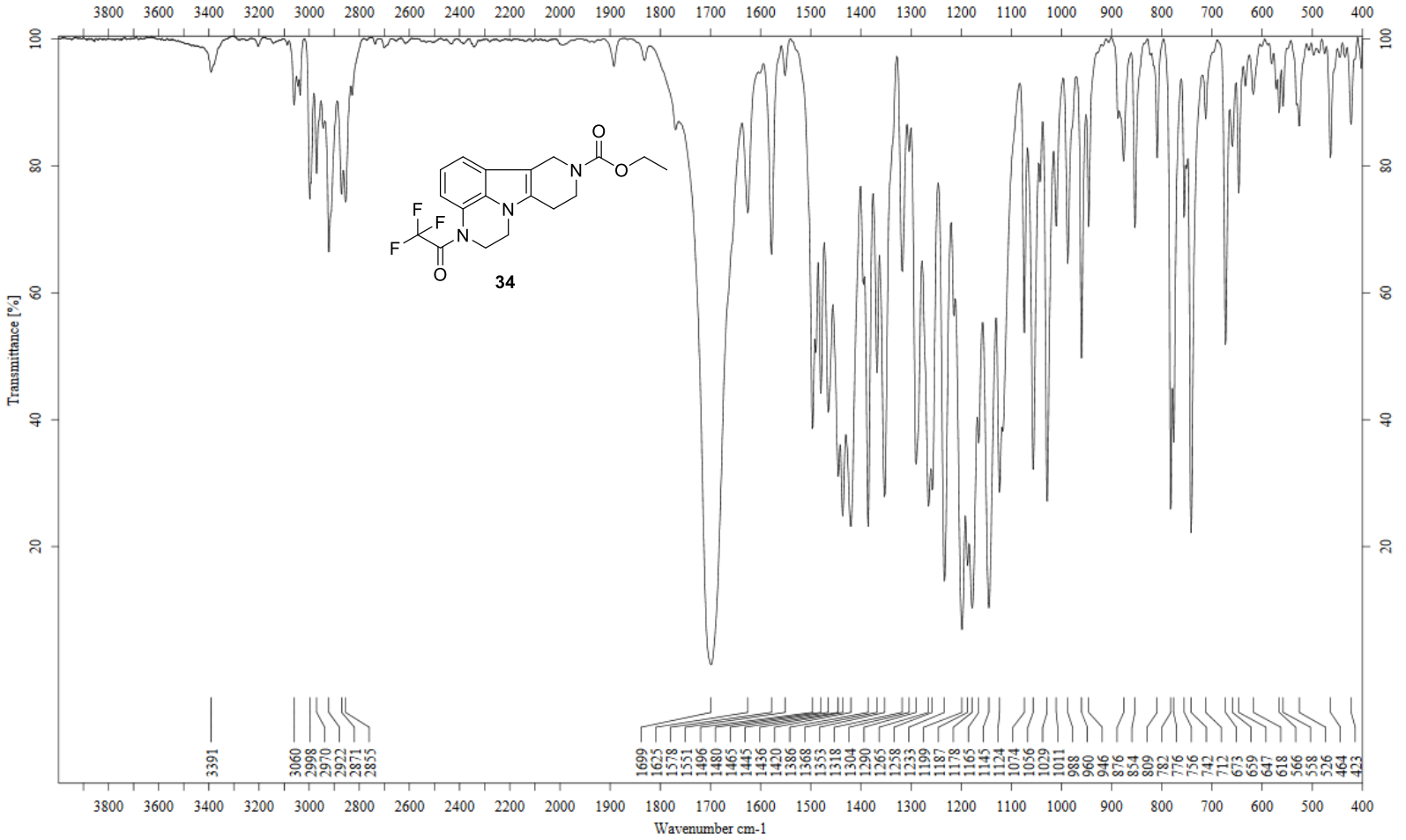



```
Standard 13C
137810
MIM0517_1
Milen Matyas
2021.12.16. (KP)

Current Data Parameters
NAME                137810
EXPNO                   12
PROCNO                   1

F2 - Acquisition Parameters
Date_             20211216
Time                17.28 h
INSTRUM               spect
PROBHD    Z145856_0002 (
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    2048
DS                       4
SWH              36231.883 Hz
FIDRES            1.105709 Hz
AQ               0.9043968 sec
RG                  196.07
DW                  13.800 usec
DE                  18.00 usec
TE                  295.0 K
D1              1.00000000 sec
D11             0.03000000 sec
TD0                      1
SFO1           150.8977808 MHz
NUC1                   13C
P1                  10.00 usec
PLW1           64.59899902 W
SFO2           600.0524002 MHz
NUC2                   1H
CPDPRG[2            waltz16
PCPD2               80.00 usec
PLW2           31.00000000 W
PLW12           0.69749999 W
PLW13           0.35029000 W

F2 - Processing parameters
SI                   32768
SF             150.8826990 MHz
WDW                     no
SSB       0
LB        0 Hz
GB        0
PC                    1.40
```

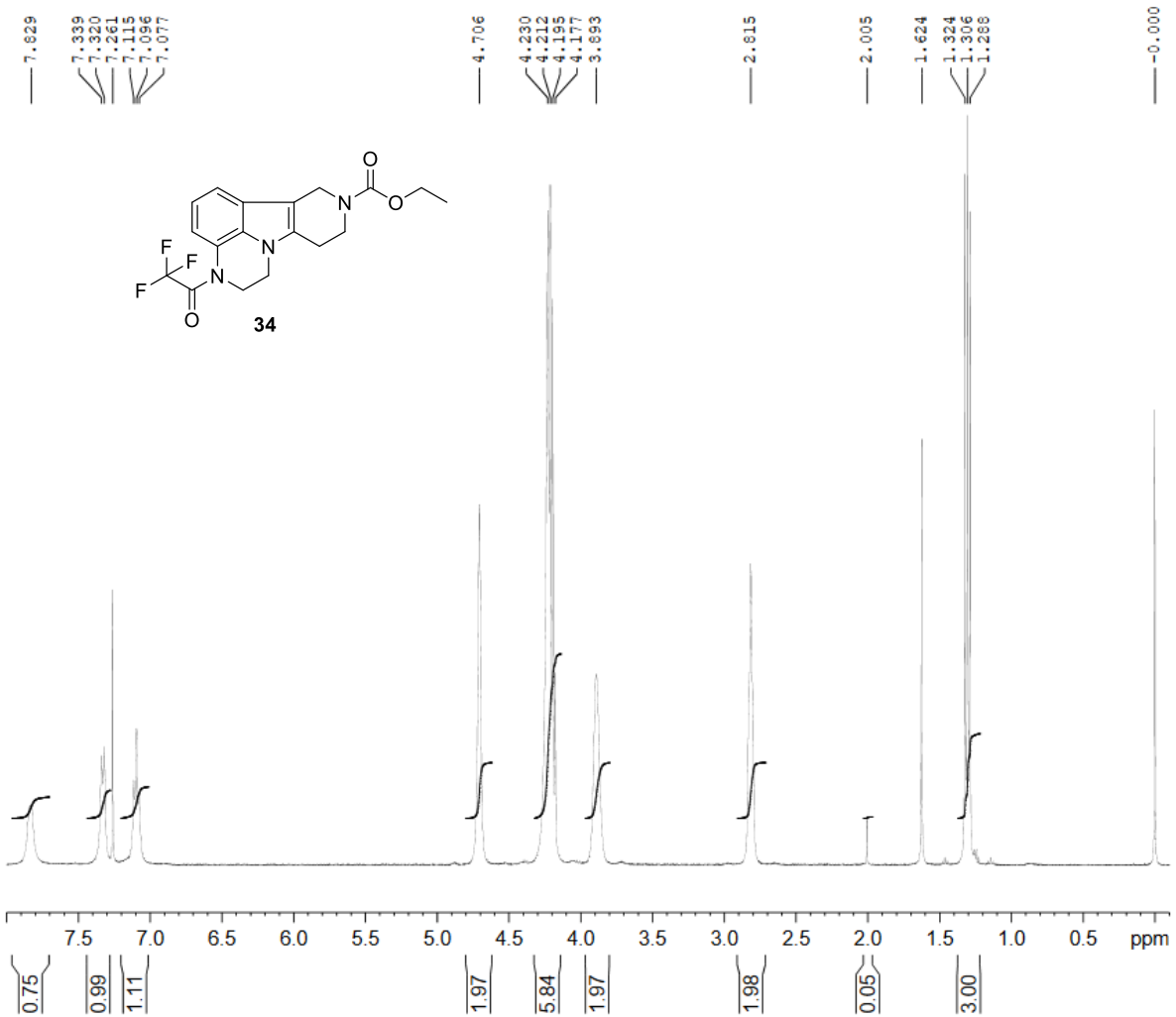

MIM0517_1, Milen Matyas (MM)
auto_gc_15perc_ACN, 17-Dec-2021 + 08:46:44
137810gcms 471 (5.494)
Scan EI+
1.90e7
100
%
0
m/z
38
131
132
326
229
69
104
77
159
160
215
257
327
F
O
N
N
38

125192	Milen Matyas	KP
JET0042_1	KBr	2018.03.14.

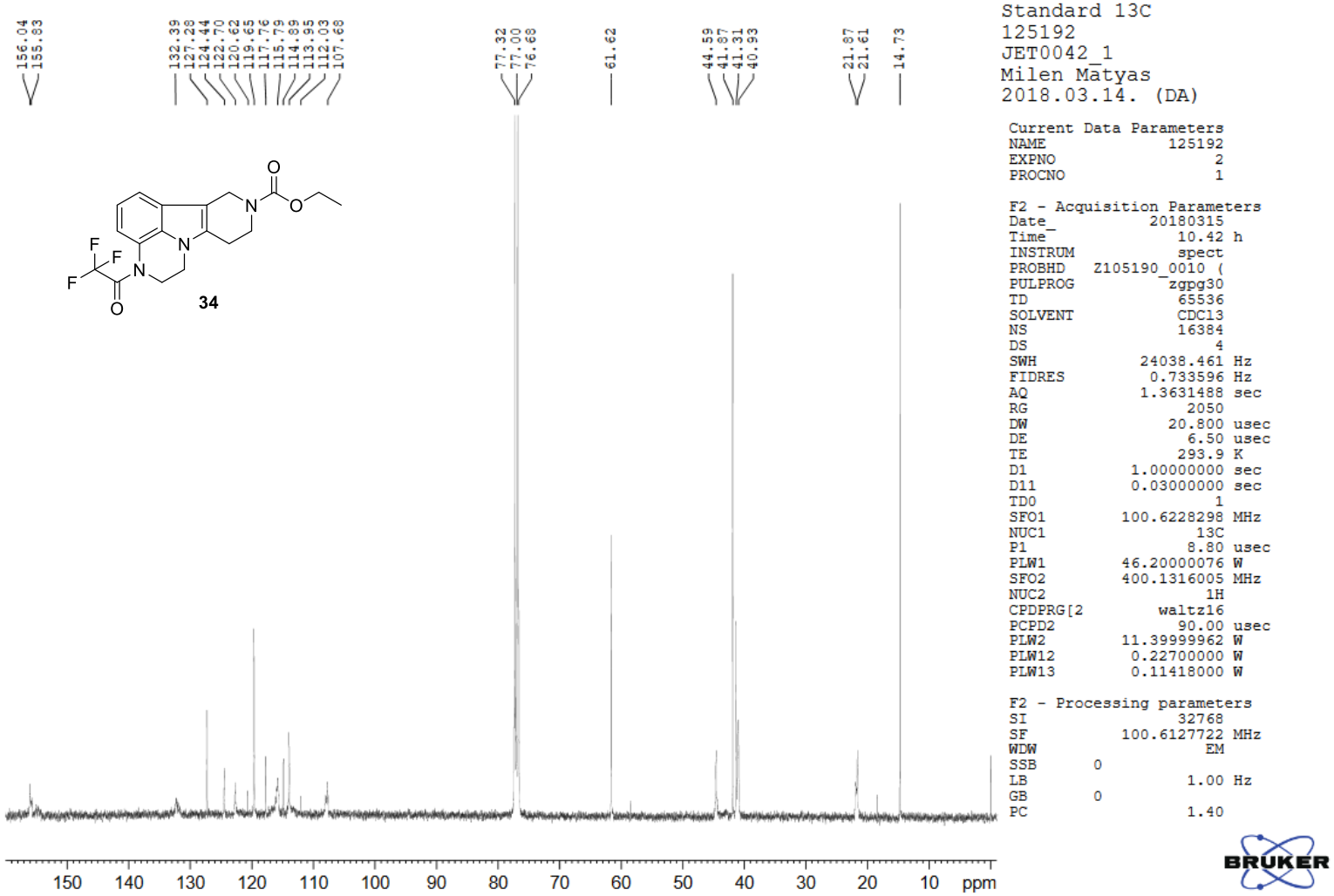
BRUKER Alpha
Resolution: 2 cm-1
Number of Scans: 16



34




```
Standard 1H
125192
JET0042_1
Milen Matyas
2018.03.14. (DA)

Current Data Parameters
NAME                 125192
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20180314
Time                 23.38 h
INSTRUM                spect
PROBHD    Z105190_0010 (
PULPROG                zg30
TD                    65536
SOLVENT               CDC13
NS                       64
DS                        2
SWH                8223.685 Hz
FIDRES             0.250967 Hz
AQ                 3.9845889 sec
RG                      161
DW                   60.800 usec
DE                    6.50 usec
TE                   293.1 K
D1                 1.00000000 sec
TD0                       1
SFO1             400.1324710 MHz
NUC1                     1H
P1                    12.70 usec
PLW1             11.39999962 W

F2 - Processing parameters
SI                    32768
SF               400.1300093 MHz
WDW                      no
SSB          0
LB           0 Hz
GB           0
PC                    1.00
```



Standard 13C
125192
JET0042_1
Milen Matyas
2018.03.14. (DA)

Current Data Parameters

NAME	125192
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

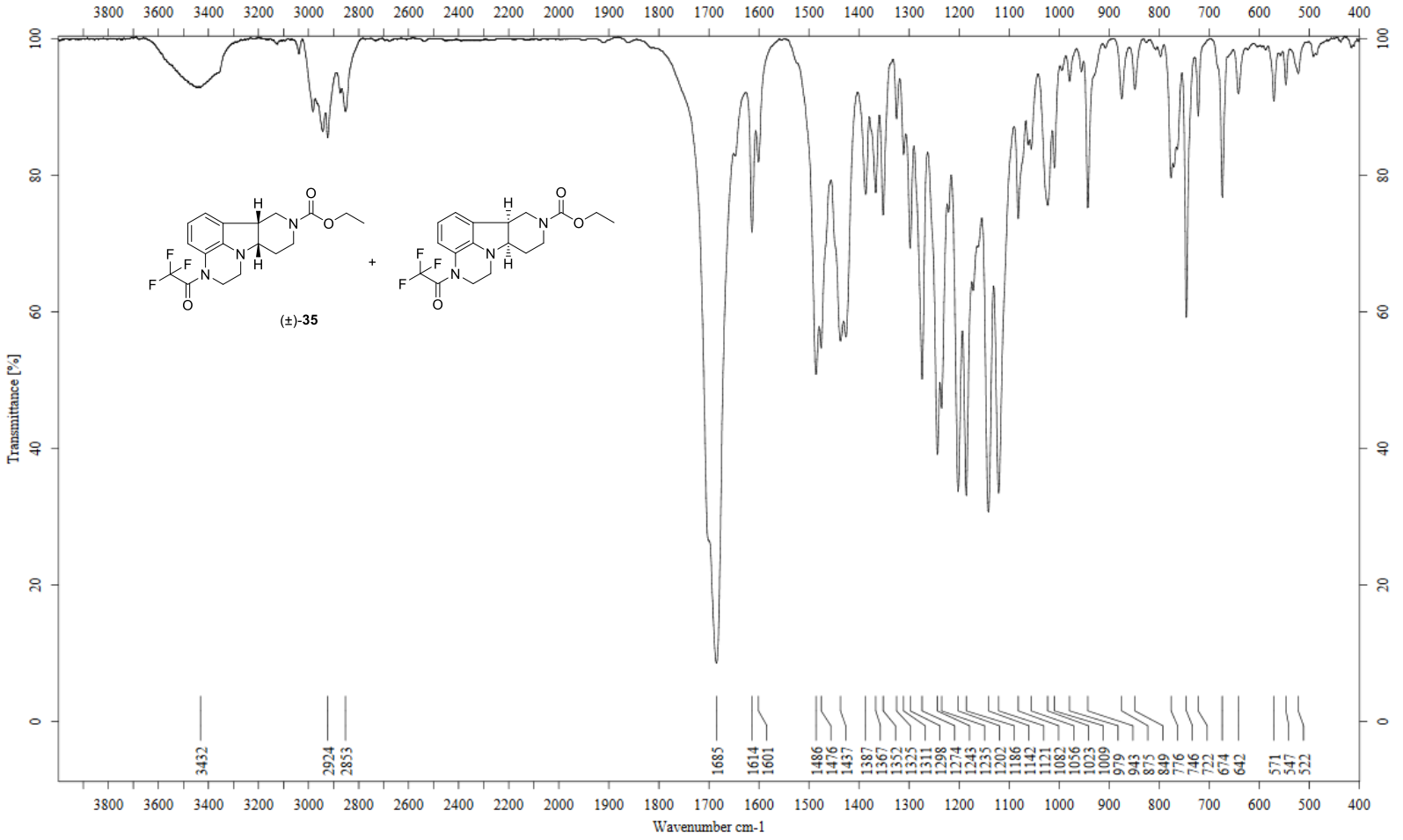
Date_	20180315
Time	10.42 h
INSTRUM	spect
PROBHD	Z105190_0010 (
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	16384
DS	4
SWH	24038.461 Hz
FIDRES	0.733596 Hz
AQ	1.3631488 sec
RG	2050
DW	20.800 usec
DE	6.50 usec
TE	293.9 K
D1	1.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	100.6228298 MHz
NUC1	13C
P1	8.80 usec
PLW1	46.20000076 W
SFO2	400.1316005 MHz
NUC2	1H
CPDPRG[2	waltz16
PCPD2	90.00 usec
PLW2	11.39999962 W
PLW12	0.22700000 W
PLW13	0.11418000 W

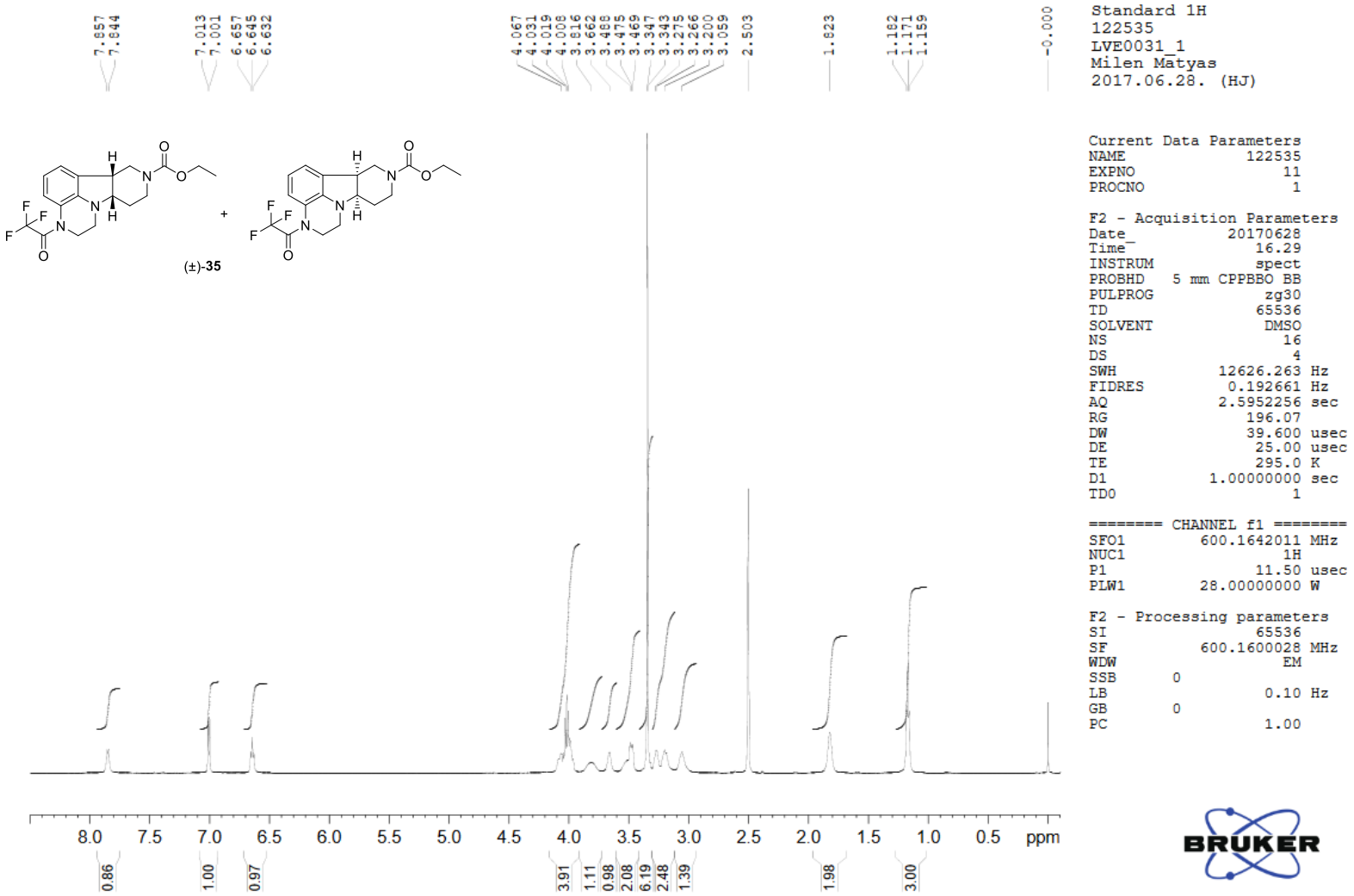
F2 - Processing parameters

SI	32768
SF	100.6127722 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

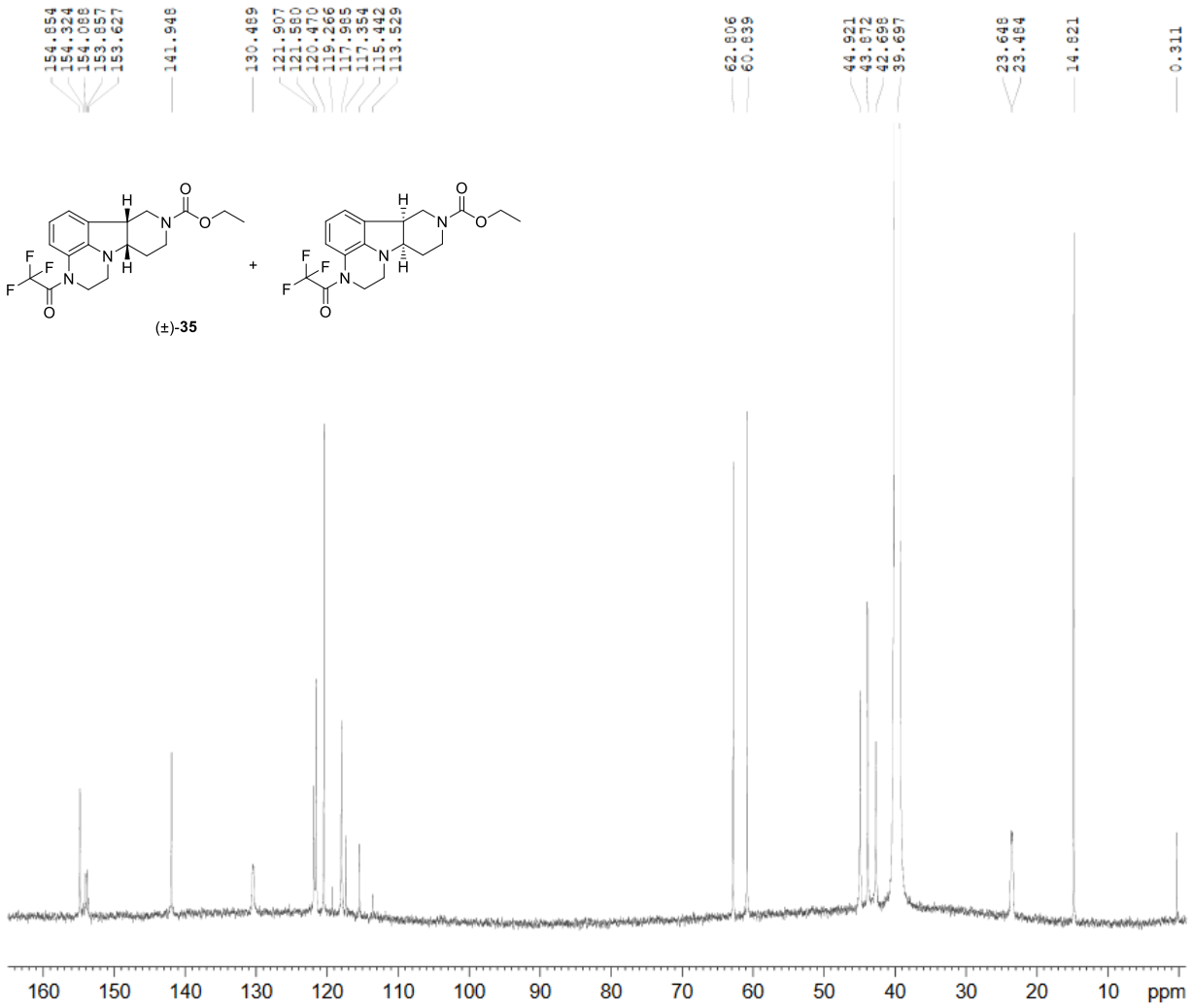
122535	Milen Matyas	MM
LVE0031_1	KBr	27/06/2017

BRUKER Alpha
Resolution: 2 cm-1
Number of Scans: 16





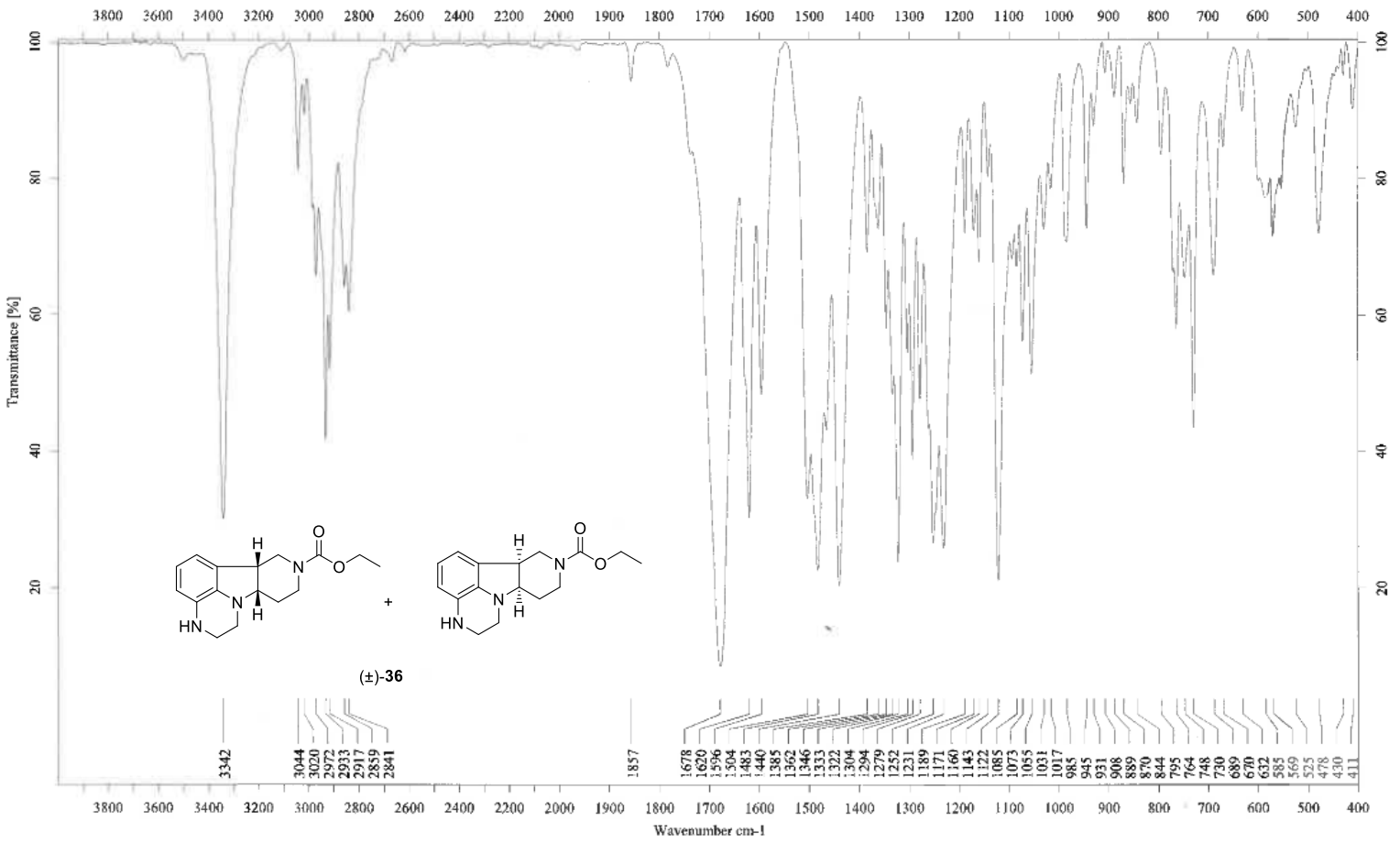

```
Standard 1H
122535
LVE0031_1
Milen Matyas
2017.06.28. (HJ)

Current Data Parameters
NAME              122535
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170628
Time_             16.29
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    16
DS                     4
SWH           12626.263 Hz
FIDRES         0.192661 Hz
AQ            2.5952256 sec
RG               196.07
DW               39.600 usec
DE               25.00 usec
TE               295.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         600.1642011 MHz
NUC1                   1H
P1                 11.50 usec
PLW1         28.00000000 W

F2 - Processing parameters
SI                 65536
SF           600.1600028 MHz
WDW                   EM
SSB       0
LB                 0.10 Hz
GB        0
PC                 1.00
```



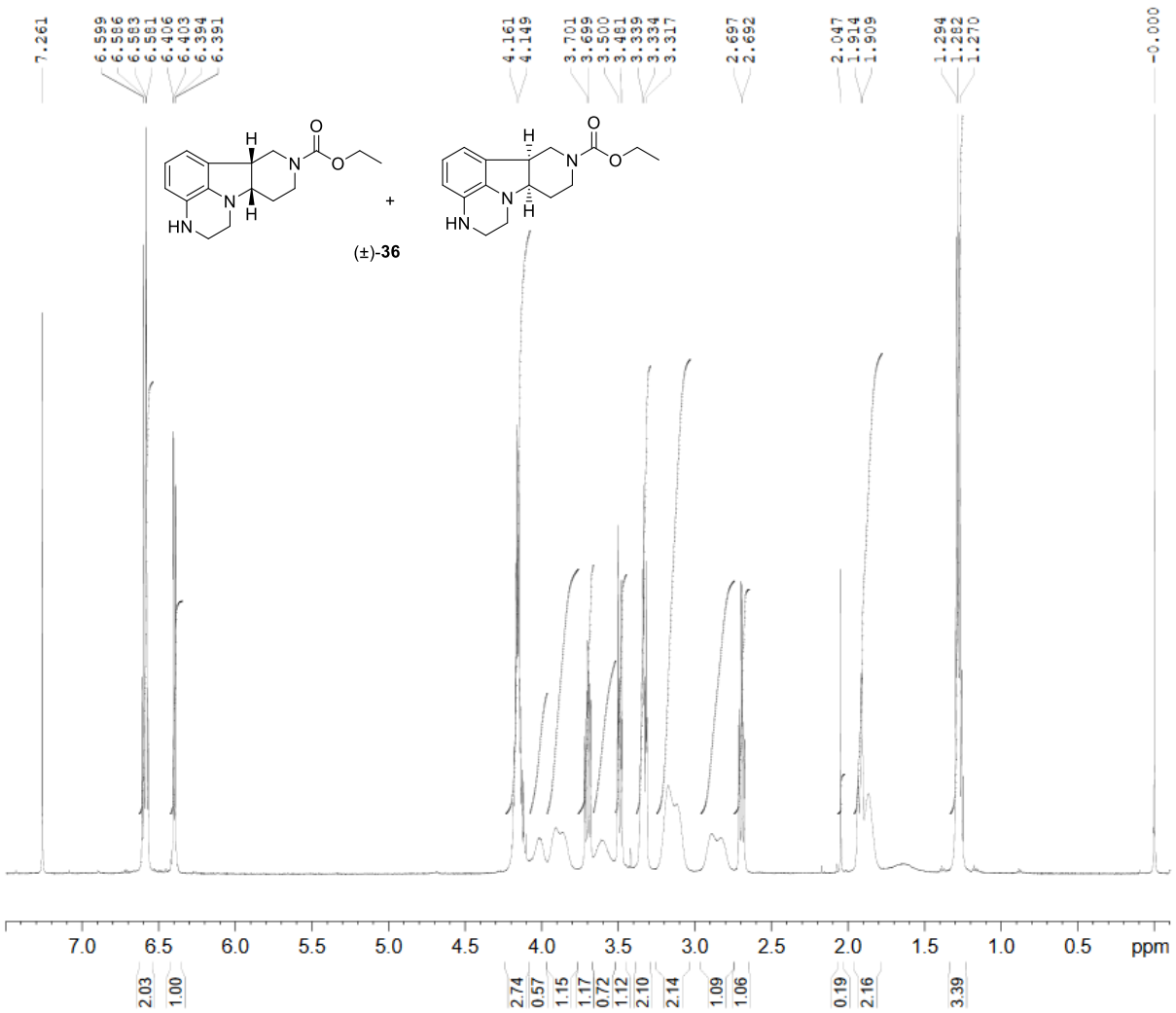

```
Standard 13C
122535
LVE0031_1
Milen Matyas
2017.06.28. (HJ)

Current Data Parameters
NAME                 122535
EXPNO                    12
PROCNO                    1

F2 - Acquisition Parameters
Date_              20170628
Time                  12.40
INSTRUM                spect
PROBHD    5 mm CPPBBO BB
PULPROG              zgpg30
TD                    65536
SOLVENT                DMSO
NS                     4096
DS                        4
SWH              36057.691 Hz
FIDRES            0.550197 Hz
AQ               0.9087659 sec
RG                   196.07
DW                   13.867 usec
DE                   18.00 usec
TE                   295.0 K
D1             1.50000000 sec
D11            0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1          150.9254424 MHz
NUC1                    13C
P1                   10.00 usec
PLW1          65.00000000 W

======== CHANNEL f2 ========
SFO2          600.1624006 MHz
NUC2                    1H
CPDPRG[2          waltz16
PCPD2                70.00 usec
PLW2          28.00000000 W
PLW12          0.75571001 W
PLW13          0.37029999 W

F2 - Processing parameters
SI                    65536
SF            150.9103921 MHz
WDW                      EM
SSB       0
LB                    3.00 Hz
GB        0
PC                    1.20
```

125261	Milen Matyas	KP
JET0044_1	KBr	2018.03.21.

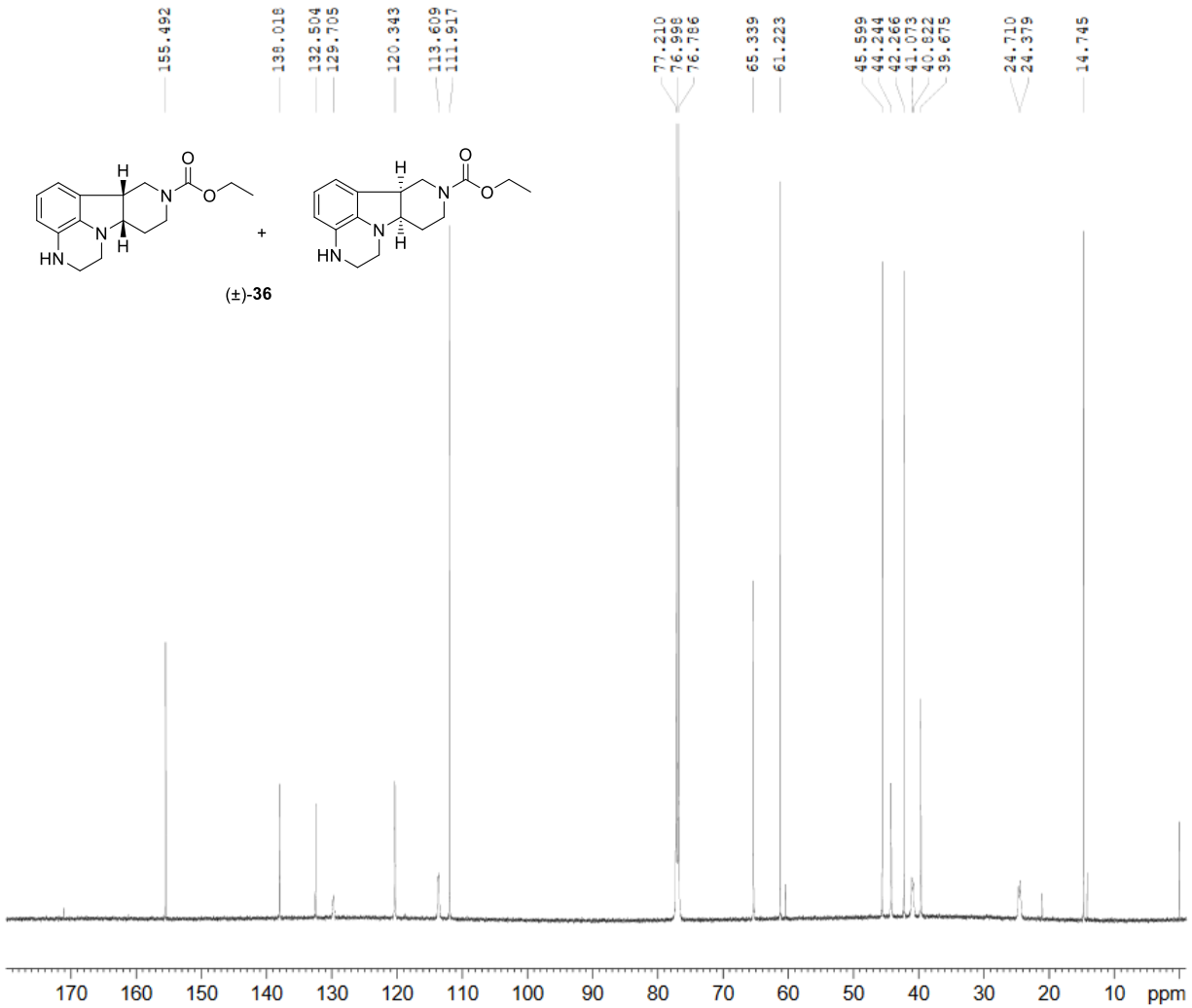
BRUKER Alpha
Resolution: 2 cm-1
Number of Scans: 16



(±)-**36**




```
Standard 1H
125261
JET0044_1
Milen Matyas
2018.03.21. (DA)


Current Data Parameters
NAME                 125261
EXPNO                    11
PROCNO                    1

F2 - Acquisition Parameters
Date_              20180321
Time                 23.06 h
INSTRUM                spect
PROBHD   Z145856_0002 (
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        4
SWH              12626.263 Hz
FIDRES            0.385323 Hz
AQ                2.5952256 sec
RG                   155.77
DW                   39.600 usec
DE                   25.00 usec
TE                   295.0 K
D1              1.00000000 sec
TD0                       1
SFO1            600.1642011 MHz
NUC1                    1H
P1                   11.50 usec
PLW1            28.00000000 W

F2 - Processing parameters
SI                    65536
SF              600.1600144 MHz
WDW                      no
SSB          0
LB           0 Hz
GB           0
PC                    1.00
```



Standard 13C
125261
JET0044_1
Milen Matyas
2018.03.21. (DA)

Current Data Parameters

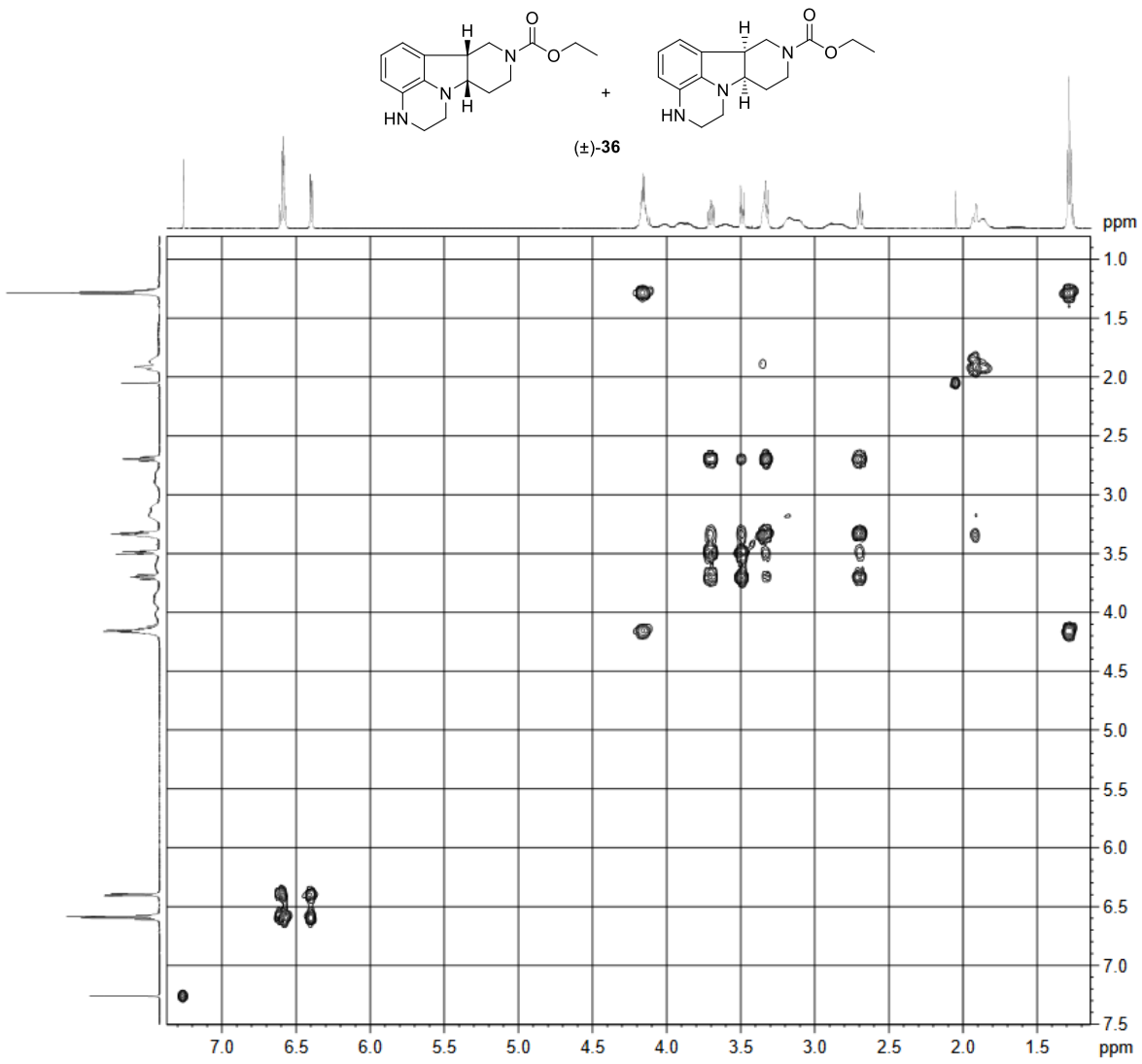
NAME	125261	
EXPNO	12	
PROCNO	1	

F2 - Acquisition Parameters

Date_	20180322	
Time	1.56	h
INSTRUM	spect	
PROBHD	Z145856_0002 (	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	4096	
DS	4	
SWH	36057.691	Hz
FIDRES	1.100393	Hz
AQ	0.9087659	sec
RG	196.07	
DW	13.867	usec
DE	18.00	usec
TE	295.0	K
D1	1.50000000	sec
D11	0.03000000	sec
TD0	1	
SFO1	150.9254424	MHz
NUC1	13C	
P1	10.00	usec
PLW1	60.36299896	W
SFO2	600.1624006	MHz
NUC2	1H	
CPDPRG[2	waltz16	
PCPD2	80.00	usec
PLW2	26.42600060	W
PLW12	0.59460002	W
PLW13	0.29861000	W

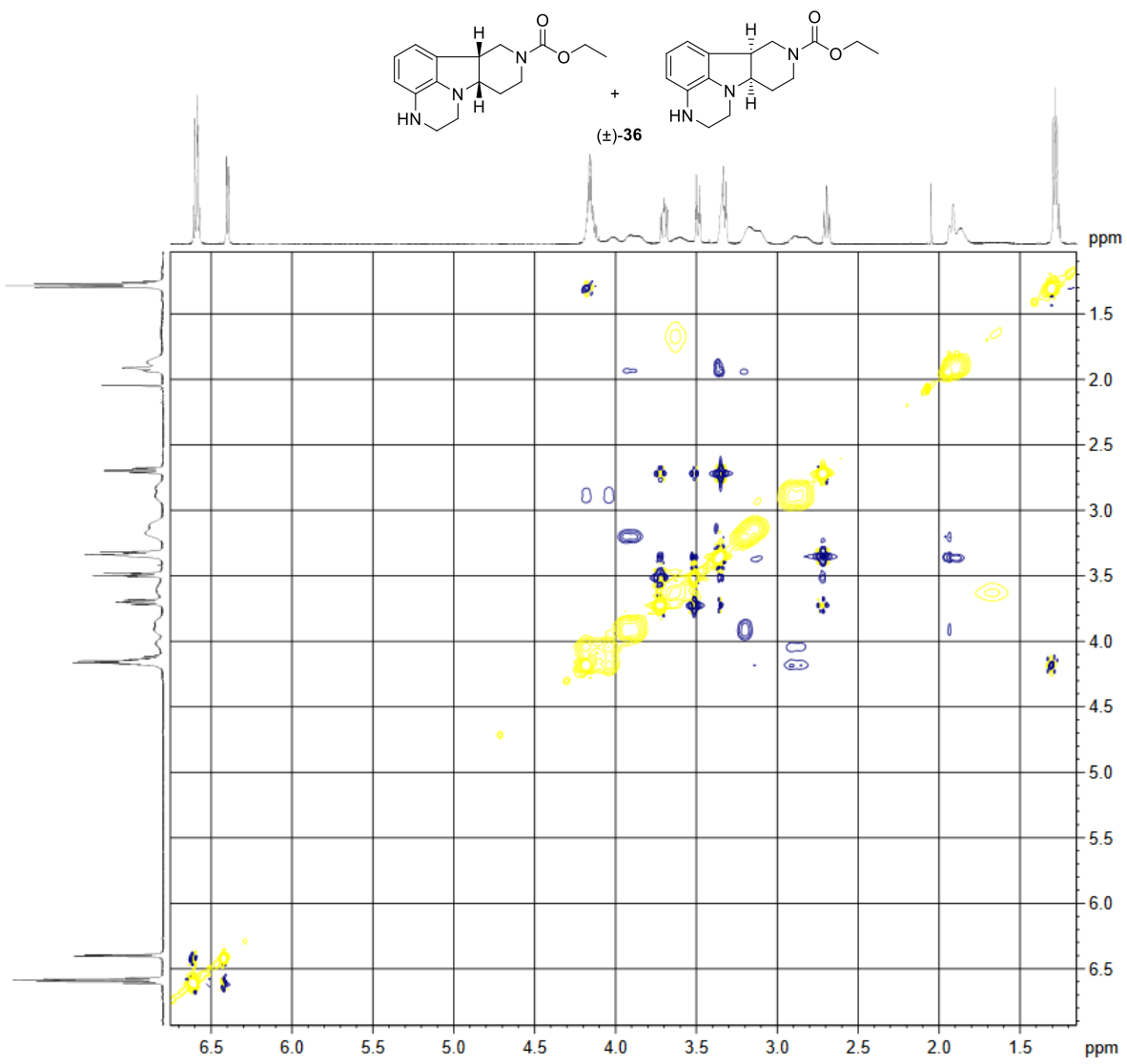
F2 - Processing parameters

SI	65536	
SF	150.9103602	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

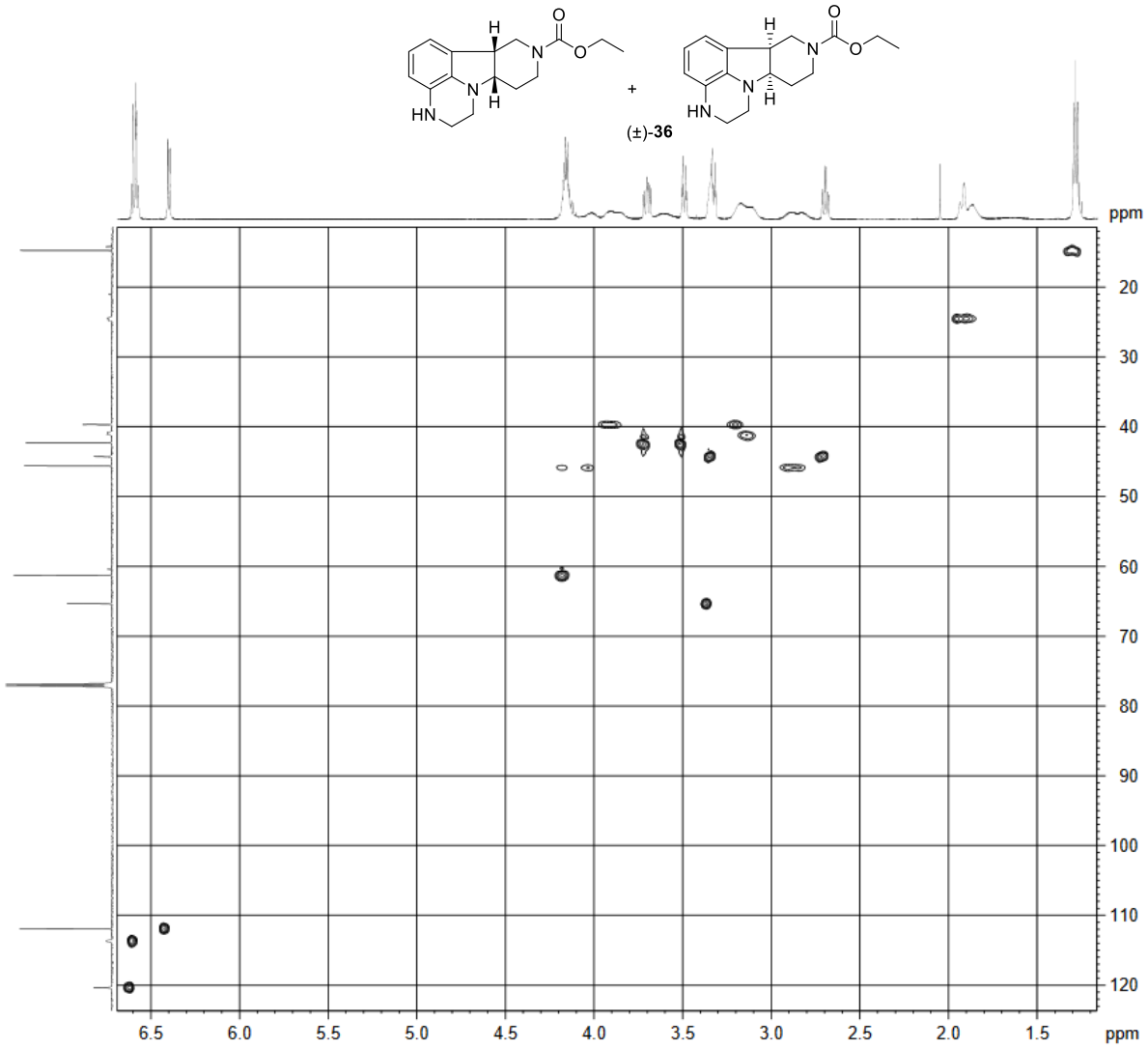


COSY
125261
JET0044_1
Milen Matyas
2018.03.21. (DA)

```
Current Data Parameters
NAME                  125261
EXPNO                     13
PROCNO                     1

F2 - Acquisition Parameters
Date_              20180322
Time                   1.57 h
INSTRUM                spect
PROBHD    Z145556_0002 (
PULPROG          cosygpppqf
TD                       2048
SOLVENT                  CDCl3
NS                          2
DS                          8
SWH                 5012.820 Hz
FIDRES              7.825020 Hz
AQ                  0.12777856 sec
RG                     196.07
DW                     62.400 usec
DE                     25.00 usec
TE                     295.0 K
D0                  0.00000300 sec
D1                  2.00000000 sec
D11                 0.03000000 sec
D12                 0.00002000 sec
D13                 0.00000400 sec
D16                 0.00020000 sec
IN0                 0.00012480 sec
TDav                        1
SFO1               600.1636083 MHz
NUC1                       1H
PO                     11.50 usec
P1                     11.50 usec
P17                  2500.00 usec
PLW1               25.00000000 W
PLW10               5.92479992 W
GPNAM[1]           SNSQ10.100
SPFI                   10.00 %
P16                  1000.00 usec

F1 - Acquisition parameters
TD                        256
SFO1                 600.1636 MHz
FIDRES              62.600159 Hz
SW                     13.351 ppm
FnMODE                     QF

F2 - Processing parameters
SI                        1024
SF                600.1600143 MHz
WDW                      QSINE
SSB          0
LB           0 Hz
GB           0
PC                        1.40

F1 - Processing parameters
SI                        1024
MC2                        QF
SF                600.1600120 MHz
WDW                      QSINE
SSB          0
LB           0 Hz
GB           0
```

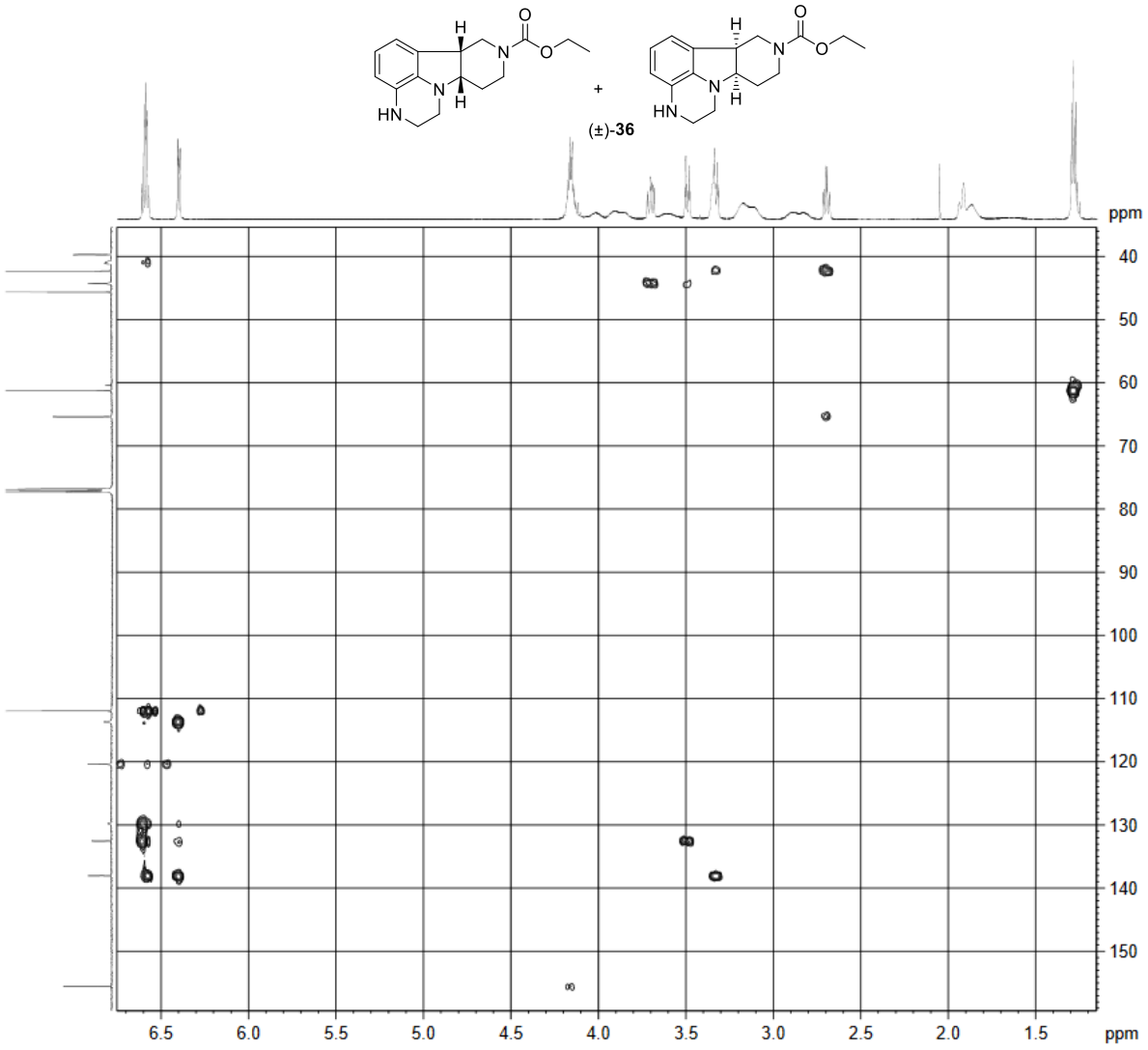



```
NOESY
125261
JET0044_1
Milen Matyas
2018.03.21. (DA)

Current Data Parameters
NAME                  125261
EXPNO                     14
PROCNO                     1

F2 - Acquisition Parameters
Date_              20180322
Time                   3.17 h
INSTRUM                spect
PROBHD    Z145556_0002 (
PULPROG       noesygpppbpp
TD                     2048
SOLVENT               CDCl3
NS                       16
DS                        4
SWH                6009.615 Hz
FIDRES             5.865765 Hz
AQ                 0.1703936 sec
RG                    196.07
DW                    83.200 usec
DE                    25.00 usec
TE                    295.0 K
D0                 0.00006556 sec
D1                 2.00000000 sec
D8                 0.30000001 sec
D11                0.03000000 sec
D12                0.00002000 sec
D16                0.00020000 sec
IN0                0.00016640 sec
TDav                       1
SFO1               600.1627626 MHz
NUC1                      1H
P1                     11.50 usec
P2                     23.00 usec
P17                 2500.00 usec
PLW1               25.00000000 W
PLW10               5.92479992 W
GPNAM[1]          SNSQ10.100
GPZ1                   40.00 %
P16                 1000.00 usec

F1 - Acquisition parameters
TD                       256
SFO1               600.1628 MHz
FIDRES             46.950119 Hz
SW                    10.013 ppm
FnMODE          States-TPPI

F2 - Processing parameters
SI                       1024
SF                600.1600000 MHz
WDW                     QSINE
SSB                        2
LB          0 Hz
GB          0
PC                      1.00

F1 - Processing parameters
SI                       1024
MC2             States-TPPI
SF                600.1600000 MHz
WDW                     QSINE
SSB                        2
LB          0 Hz
GB          0
```



HSQC (140 Hz)
125261
JET0044_1
Milen Matyas
2018.03.21. (DA)

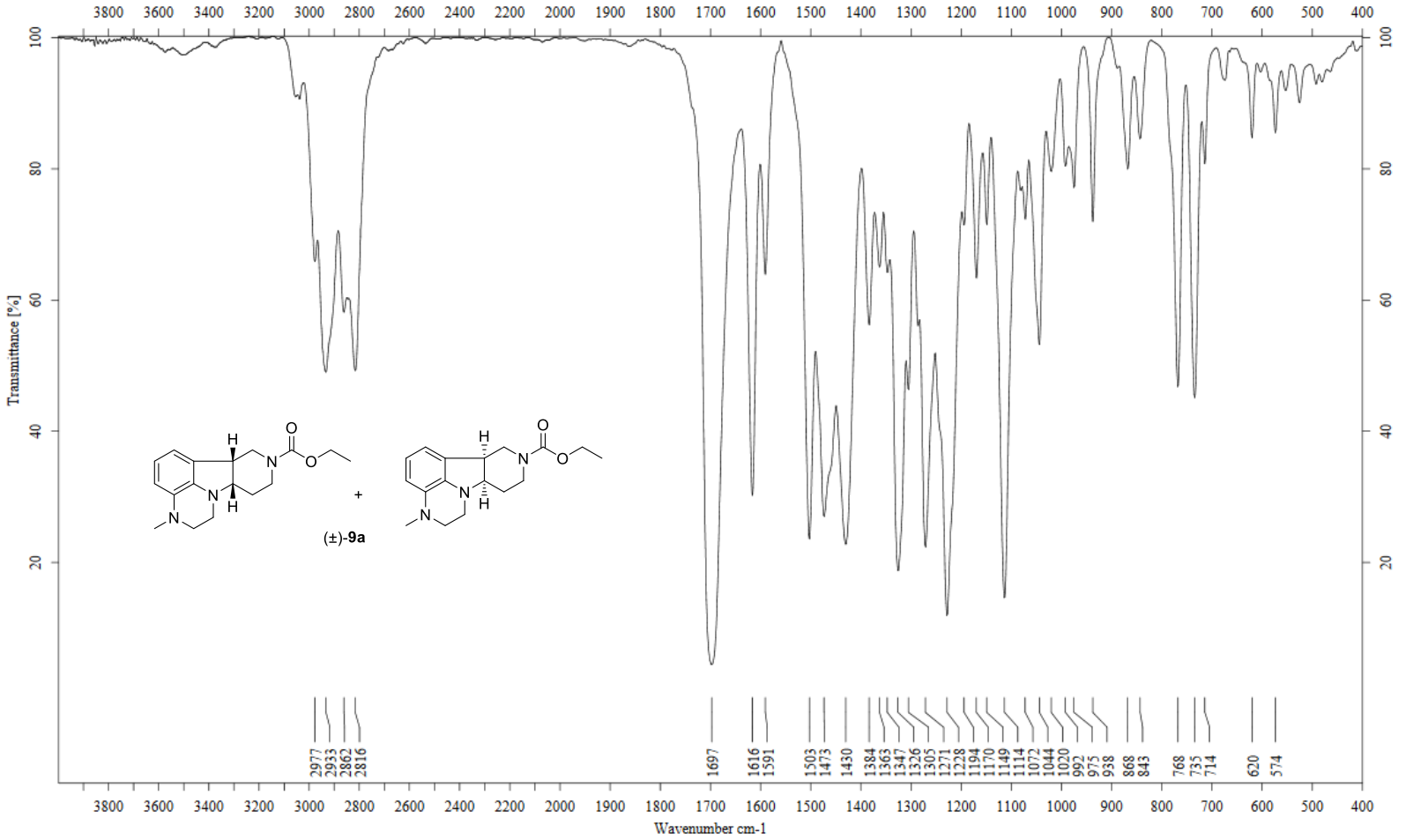
```
Current Data Parameters
NAME                 125261
EXPNO                    14
PROCNO                    1

F2 - Acquisition Parameters
Date_              20180322
Time                   2.17 h
INSTRUM                spect
PROBHD   Z145556_0002 (
PULPROG            hsqcetgp
TD                     2048
SOLVENT              CDCl3
NS                        4
DS                       16
SWH                7812.500 Hz
FIDRES             7.629395 Hz
AQ                 0.1310720 sec
RG                    196.07
DW                   64.000 usec
DE                   25.00 usec
TE                   295.0 K
CNST2          140.0000000
D0               0.00000300 sec
D1               1.80000000 sec
D4               0.00178571 sec
D11              0.03000000 sec
D16              0.00020000 sec
IN0              0.00001531 sec
IDav                      1
ZGOPTNS
SFO1           600.1636083 MHz
NUC1                    1H
P1                    11.50 usec
P2                    23.00 usec
PCPD2      0 usec
PLW1         25.00000000 W
SFO2         150.9269522 MHz
NUC2                   13C
CPDPRG[2               garp
PCPD2                 10.00 usec
P4                    20.00 usec
PCPD2                 55.00 usec
PLW2         60.36299896 W
PLW12         1.99328596 W
GPNAM[1]      SNSQ10.100
GPZ1                  50.00 %
GPNAM[2]      SNSQ10.100
GPZ2                  20.10 %
P16                 1000.00 usec

F1 - Acquisition parameters
TD                     256
SFO1             150.927 MHz
FIDRES         258.692047 Hz
SW                 219.395 ppm
FnMODE     Echo-Antiecho

F2 - Processing parameters
SI                    1024
SF             600.1600000 MHz
WDW                   QSINE
SSB                       2
LB         0 Hz
GB         0
PC                    1.40

F1 - Processing parameters
SI                    1024
MC2         echo-antiecho
SF              150.9103520 MHz
WDW                   QSINE
SSB                       2
LB         0 Hz
GB         0
```

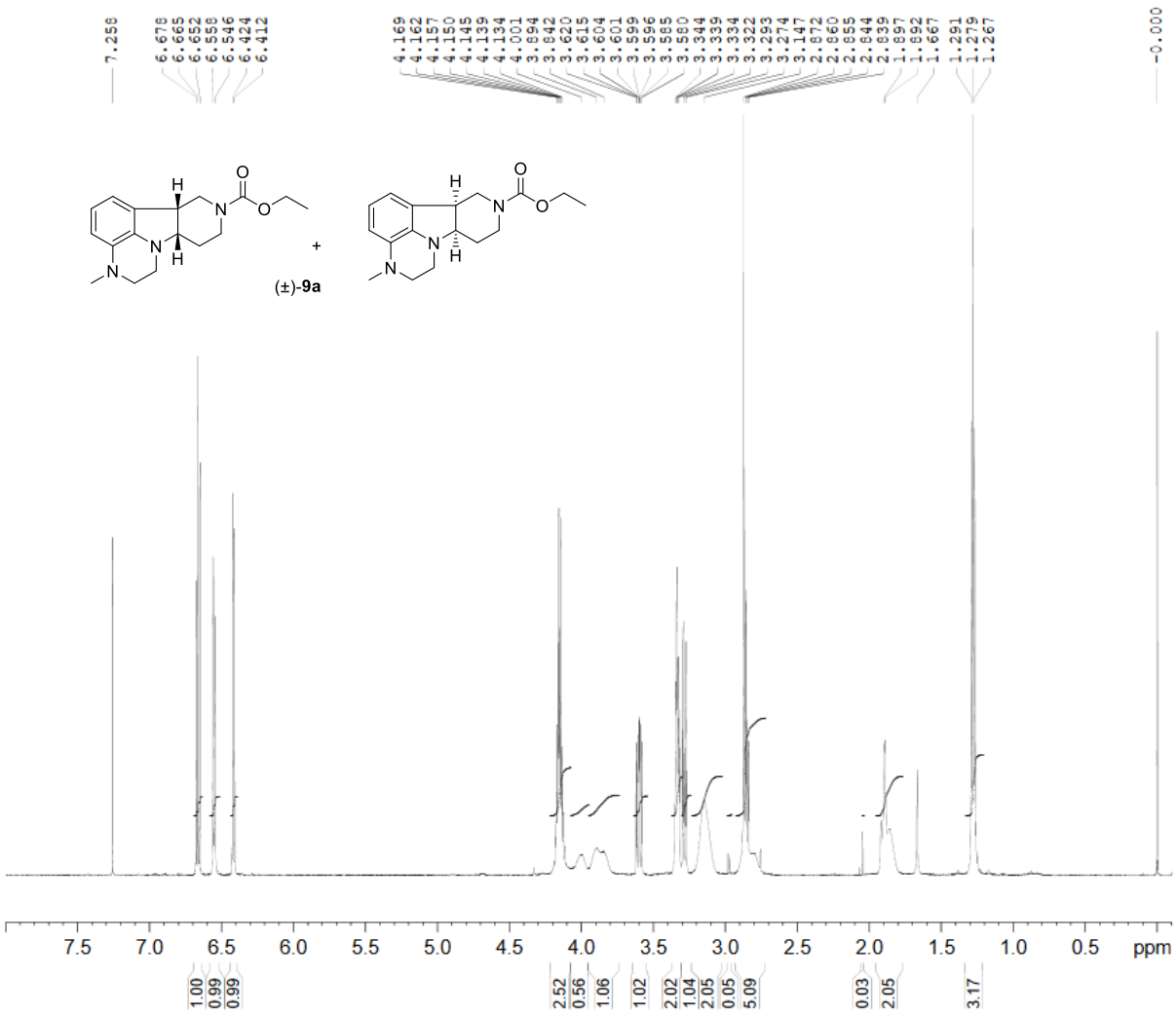



```
HMBC (8 Hz, 140 Hz)
125261
JET0044_1
Milen Matyas
2018.03.21. (DA)

Current Data Parameters
NAME                  125261
EXPNO                     10
PROCNO                     1

F2 - Acquisition Parameters
Date_               20180322
Time                    2.47 h
INSTRUM                 spect
PROBHD   Z145556_0002 (
PULPROG      hmbcgplpndqf
TD                      2048
SOLVENT              CDCl3
NS                         4
DS                        16
SWH                7812.500 Hz
FIDRES             7.629395 Hz
AQ                 0.1310720 sec
RG                    196.07
DW                   64.000 usec
DE                    25.00 usec
TE                   295.0 K
CNST2             140.0000000
CNST13              8.0000000
D0                  0.00000300 sec
D1                  1.50000000 sec
D2                  0.00357143 sec
D6                  0.06250000 sec
D16                 0.00020000 sec
IN0                 0.00001510 sec
IDAY                        1
SFO1               600.1639010 MHz
NUC1                        1H
P1                      11.50 usec
P2                      23.00 usec
PLW1               25.00000000 W
SFO2               150.9269522 MHz
NUC2                       13C
P3                      10.00 usec
PLW2               60.36298596 W
GPNAM[1]           SNSQ10.100
GPZ1                    50.00 %
GPNAM[2]           SNSQ10.100
GPZ2                    30.00 %
GPNAM[3]           SNSQ10.100
GPZ3                    40.10 %
P16                   1000.00 usec

F1 - Acquisition parameters
TD                         256
SFO1               150.927 MHz
FIDRES             258.692047 Hz
SW                 219.395 ppm
FnMODE                     QF

F2 - Processing parameters
SI                        2048
SF                 600.1600136 MHz
WDW                       SINE
SSB         0
LB          0 Hz
GB          0
PC                        1.40

F1 - Processing parameters
SI                         512
MC2                         QF
SF                 150.9103569 MHz
WDW                       SINE
SSB         0
LB          0 Hz
GB          0
```

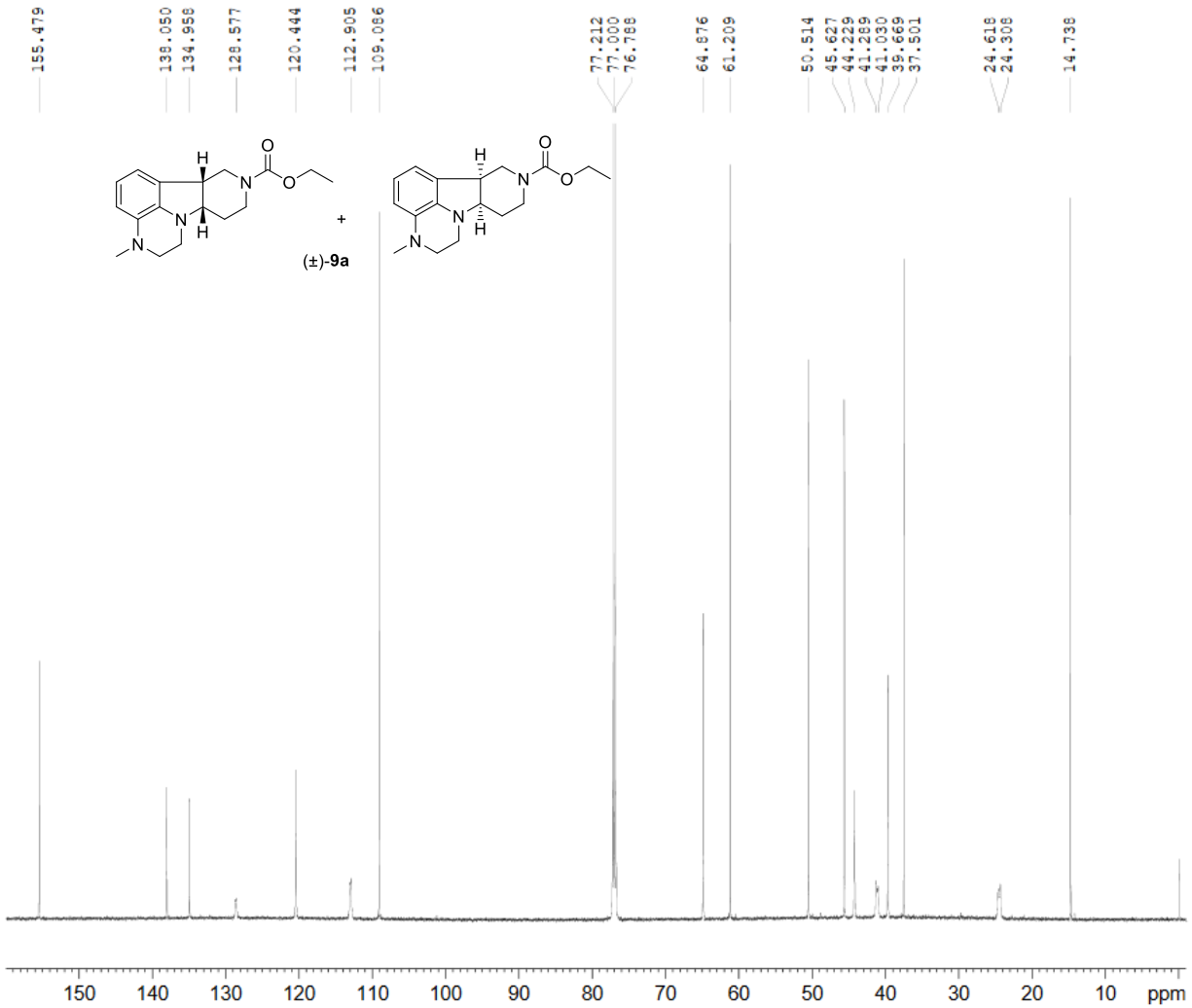
125274	Milen Matyas	KP
JET0043_1	Film	2018.03.23.

BRUKER Alpha
Resolution: 2 cm-1
Number of Scans: 16






```
Standard 1H
125274
JET0043_1
Milen Matyas
2018.03.26. (DA)

Current Data Parameters
NAME                 125274
EXPNO                    11
PROCNO                    1

F2 - Acquisition Parameters
Date_              20180326
Time                 17.37 h
INSTRUM                spect
PROBHD   Z145856_0002 (
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        4
SWH              12626.263 Hz
FIDRES            0.385323 Hz
AQ                2.5952256 sec
RG                    111.4
DW                   39.600 usec
DE                   25.00 usec
TE                   295.0 K
D1              1.00000000 sec
TD0                       1
SFO1            600.1642011 MHz
NUC1                    1H
P1                   11.50 usec
PLW1            28.00000000 W

F2 - Processing parameters
SI                    65536
SF              600.1600159 MHz
WDW                      no
SSB          0
LB           0 Hz
GB           0
PC                    1.00
```




```
Standard 13C
125274
JET0043_1
Milen Matyas
2018.03.26. (DA)

Current Data Parameters
NAME                 125274
EXPNO                    12
PROCNO                    1

F2 - Acquisition Parameters
Date_              20180326
Time                 19.03 h
INSTRUM                spect
PROBHD    Z145856_0002 (
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     2048
DS                        4
SWH              36057.691 Hz
FIDRES            1.100393 Hz
AQ               0.9087659 sec
RG                   196.07
DW                   13.867 usec
DE                   18.00 usec
TE                   295.0 K
D1              1.50000000 sec
D11             0.03000000 sec
TD0                       1
SFO1           150.9254424 MHz
NUC1                    13C
P1                   10.00 usec
PLW1           60.36299896 W
SFO2           600.1624006 MHz
NUC2                    1H
CPDPRG[2           waltz16
PCPD2                80.00 usec
PLW2           26.42600060 W
PLW12           0.59460002 W
PLW13           0.29861000 W

F2 - Processing parameters
SI                    65536
SF             150.9103614 MHz
WDW                      EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```