

Supporting Information for **An Isochroman Analog of CD3254 and Allyl-, Isochroman-Analogs of NEt-TMN Prove to be More Potent Retinoid-X-Receptor (RXR) Selective Agonists than Bexarotene**

Synthetic Protocols for compounds made in Schemes 1-10

Representative HPLC Method for compound **36**

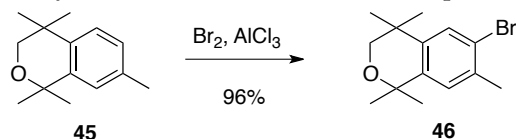
HPLC Traces for compounds **23-44**

^1H -NMR and ^{13}C -NMR for all reported compounds

HPLC. All tested compounds were assessed on a Waters Acquity UPLC with QDA and PDA detectors. Compounds were assayed in ESI-mode on an ACE Excel C18-PFP (1.7 μ m, 50 mm x 2.1 mm) column using a 0.1% formic acid/water:acetonitrile gradient over 5 minutes.

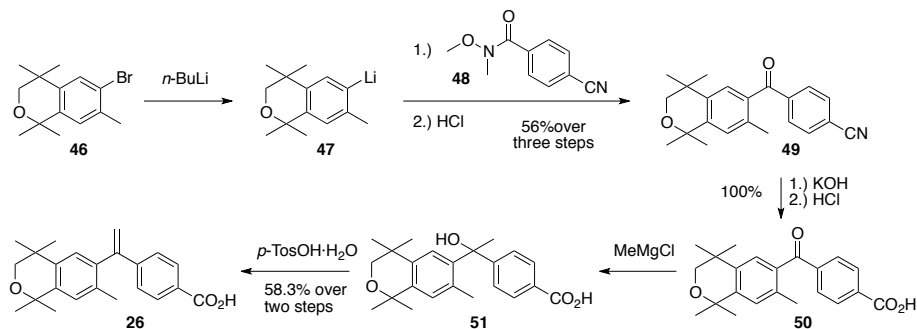
NMR and High Resolution Mass Spectrometry. A 400 MHz Bruker Avance III spectrometer was used to acquire ^1H NMR and ^{13}C NMR spectra. Chemical shifts (δ) are listed in ppm against residual non-deuterated solvent peaks in a given deuterated solvent (e.g. CHCl_3 in CDCl_3) as an internal reference. Coupling constants (J) are reported in Hz, and the abbreviations for splitting include: s, single; d, doublet; t, triplet; q, quartet; p, pentet; m, multiplet; br, broad. All ^{13}C NMR spectra were acquired on a Bruker instrument at 100.6 MHz. Chemical shifts (δ) are listed in ppm against deuterated solvent carbon peaks as an internal reference. High resolution mass spectra were recorded using either a JEOL GCmate(2004), a JEOL LCmate(2002) high resolution mass spectrometer or an ABI Mariner (1999) ESI-TOF mass spectrometer. NMR spectra are available in the Supplementary Information document.

General Procedures. Removal of volatile solvents transpired under reduced pressure using a Büchi rotary evaporator and is referred to as removing solvents in vacuo. Thin layer chromatography was conducted on precoated (0.25 mm thickness) silica gel plates with 60F-254 indicator (Merck). Column chromatography was conducted using 230-400 mesh silica gel (E. Merck reagent silica gel 60). The 1,1,4,4,7-pentamethylisochroman (**45**) was purchased from Ark Pharm Inc., compound **59** was purchased from J&W Pharmed, compound **73** was purchased from Oakwood Chemical, and compound **74** was purchased from Accela. All tested compounds were analyzed for purity by NMR as well as HPLC analysis and were found to be > 95% pure.



Scheme 1.

6-Bromo-1,1,4,4,7-pentamethylisochroman (46). A modified procedure of Pfahl and co-workers was used.[1] To a 100 mL round bottom flask equipped with a stir bar and charged with 1,1,4,4,7-pentamethylisochroman (**45**) (1.8253 g, 8.934 mmols) dissolved in dichloromethane (6.0 mL) and cooled to 0 °C was added aluminum chloride (0.258 g) followed by bromine (0.50 mL, 9.71 mmols) and the reaction was allowed to warm to room temperature and stirred for 2 hours. Then, 20 mL of a 40% ethyl acetate hexanes solution was added to the reaction and the resulting solution was washed with saturated sodium sulfite (50 mL), the organic layer was separated, and the aqueous layer was extracted with ethyl acetate. The combined organic layers were dried over sodium sulfate, filtered, concentrated in vacuo to give a crude product that was purified by column chromatography (250 mL SiO_2) with 2% ethyl acetate in hexanes to give pure **46** (2.4289 g, 96%) as white solid, m.p. 63.0-65.5 °C: ^1H NMR (400 MHz, CDCl_3) δ 7.43 (s, 1H), 6.91 (s, 1H), 3.56 (s, 2H), 2.35 (s, 3H), 1.50 (s, 6H), 1.24 (s, 6H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 142.2, 140.8, 135.0, 129.0, 127.6, 122.7, 74.8, 70.6, 33.7, 29.6, 26.9, 22.6; GC-MS CI (M+H) $^+$ calcd for $\text{C}_{14}\text{H}_{19}\text{BrO}$ 283.0698, found 283.0701.

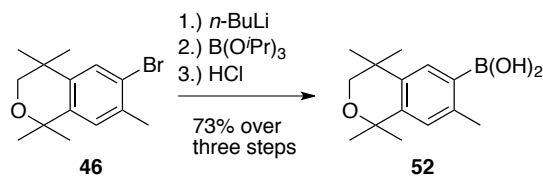


Scheme 2.

4-(1,1,4,4,7-Pentamethylisochroman-6-carbonyl)benzonitrile (49). The method of Faul[2] and co-workers was followed. To a solution of **46** (4.0954 g, 14.46 mmol) in THF (70 mL) at -78°C under nitrogen was added a 1.6 M solution of n-BuLi in hexanes (10.80 mL, 17.20 mmol) over 10 min and the solution was stirred for 20 min at -78°C . This reaction solution was transferred via air-tight syringe to a solution of **48**[2] (2.5239 g, 13.27 mmol) in THF (10 mL) at -78°C , and the combined mixture was stirred for 15 min at -78°C and then warmed to room temperature before 1.0 N HCl (150 mL) was added to quench the reaction. The solution was poured into ethyl acetate, the layers were separated, and the aqueous layer was extracted with ethyl acetate. The combined organic layers were washed with brine, dried over sodium sulfate, and concentrated in vacuo to give a crude product that was purified by column chromatography (150 mL SiO_2 , hexanes:ethyl acetate 95:5) to give **49** (2.7033 g, 56%) as a white crystalline solid, m.p. $136.6\text{--}160.0^{\circ}\text{C}$: ^1H NMR (400 MHz, CDCl_3) δ 7.89 (d, $J = 8.4$, 2H), 7.77 (d, $J = 8.4$, 2H), 7.21 (s, 1H), 6.99 (s, 1H), 3.58 (s, 2H), 2.31 (s, 3H), 1.56 (s, 6H), 1.20 (s, 6H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 196.5, 145.1, 141.3, 139.9, 134.9, 134.8, 132.2, 130.3, 128.3, 126.5, 117.9, 116.1, 70.6, 33.5, 29.5, 26.8, 20.0; ES-MS (M+H) $^{+}$ calcd for $\text{C}_{22}\text{H}_{23}\text{NO}_2\text{Na}$ 356.1627, found 356.1639.

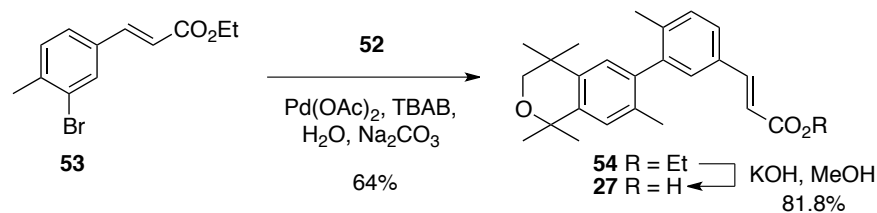
4-(1,1,4,4,7-pentamethylisochroman-6-carbonyl)benzoic acid (50). The method of Faul[2] and co-workers was followed. To a heterogeneous solution of **49** (2.2004 g, 6.60 mmol) in 2-methoxyethanol (27 mL) was added a solution of KOH (2.2338 g, 39.8 mmol) in water (10 mL). The reaction was heated in an oil bath at reflux temperature and stirred under nitrogen for 23 h. The reaction was allowed to cool to room temperature before it was quenched with 1 N HCl (70 mL). The solution was poured into ethyl acetate, the layers were separated, and the aqueous layer was extracted with ethyl acetate. The combined organic layers were washed with brine, dried over sodium sulfate, and concentrated in vacuo with additional toluene to azeotrope off 2-methoxyethanol to give **50** (2.32 g, 100%) as a white powder, m.p. $201.4\text{--}202.6^{\circ}\text{C}$: ^1H NMR (400 MHz, $\text{D}_6\text{-DMSO}$) δ 13.36 (br s, 1H), 8.08 (d, $J = 8.4$, 2H), 7.78 (d, $J = 8.0$, 2H), 7.28 (s, 1H), 7.22 (s, 1H), 3.50 (s, 2H), 2.21 (s, 3H), 1.49 (s, 6H), 1.14 (s, 6H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 197.0, 166.6, 144.3, 140.6, 139.5, 135.6, 134.6, 133.7, 129.8, 129.6, 128.0, 125.8, 74.6, 69.7, 33.2, 29.3, 26.5, 19.4; ES-MS (M+H) $^{+}$ calcd for $\text{C}_{22}\text{H}_{24}\text{O}_4$ 353.1753, found 353.1751.

4-(1-(1,1,4,4,7-pentamethylisochroman-6-yl)vinyl)benzoic acid (26). The procedure of Faul and co-workers was followed.[2] To a 100 mL round bottom flask charged with a 3.0 M solution of MeMgCl (4.35 mL, 13.1 mmol) was added THF (8.5 mL), and the solution was cooled to -10°C in a salt-water ice bath with stirring under nitrogen. To this solution was added a solution of **50** (1.1504 g, 3.26 mmol) in THF (11.3 mL), dropwise, and the reaction was stirred at 0°C for 4 h. The reaction was quenched with 1.0 N HCl (43 mL), the solution was extracted with ethyl acetate, and the combined organic layers were washed with brine, dried over sodium sulfate, filtered and concentrated in vacuo to give an intermediate alcohol that was used without further purification. The intermediate alcohol was dissolved in toluene (85 mL), and to this solution was added p-toluenesulfonic acid monohydrate (0.07 g, 0.406 mmol), and the solution was refluxed into a Dean-Stark apparatus pre-filled with toluene. After the solution was refluxed for 2 h, it was cooled to room temperature and poured into ethyl acetate and water. The aqueous layer was extracted with ethyl acetate, and the combined organic layers were washed with brine, dried over sodium sulfate, filtered and concentrated in vacuo to give a crude product that was column chromatography (150 mL SiO_2 , 30% ethyl acetate:hexanes) to give **26** (0.6672 g, 58.3%) as a white crystalline solid (m.p. $246.7\text{--}249.5^{\circ}\text{C}$): ^1H NMR (400 MHz, CDCl_3) δ 8.90 (br s, 1H), 8.05 (dd, $J = 6.8$, 2.0, 2H), 7.37 (dd, $J = 6.8$, 1.6, 2H), 7.13 (s, 1H), 6.86 (s, 1H), 5.87 (d, $J = 1.2$, 1H), 5.35 (d, $J = 1.2$, 1H), 3.62 (s, 2H), 1.96 (s, 3H), 1.57 (s, 6H), 1.27 (s, 6H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 171.6, 148.7, 146.0, 141.0, 140.0, 138.7, 133.2, 130.3, 128.2, 126.9, 126.6, 126.5, 117.4, 70.9, 33.5, 29.8, 27.0, 19.9; ES-MS (M-H) $^{-}$ calcd for $\text{C}_{23}\text{H}_{25}\text{O}_3$ 349.1804, found 349.1819.



Scheme 3.

(1,1,4,4,7-pentamethylisochroman-6-yl)boronic acid (52). To a 500 mL round bottom flask containing THF (60 mL) was added a 1.6 M solution of *n*-BuLi in hexanes (16.0 mL, 25.6 mmol), and the resulting solution was cooled in a dry-ice acetone bath to -78°C with stirring, under nitrogen. To this solution was added a solution of **46** (4.4727 g, 63.32 mmol) in THF (16 mL) over 20 min and the reaction was stirred at -78°C for 10 min, and a mixture of triisopropylborate (9.8 mL, 42.6 mmol) in THF (10 mL) was added dropwise over 20 min. The reaction was stirred at -78°C for 1 h and then warmed to room temperature and stirred for 2 h. The reaction was then quenched with 3 N HCl (70 mL), and after stirring for 2 h, it was poured into ethyl acetate, the layers were separated, and the aqueous layer was extracted with ethyl acetate. The combined organic layers were washed with brine, dried over sodium sulfate, and concentrated in vacuo to give a crude product that was purified by column chromatography (150 mL SiO₂, ethyl acetate:hexanes 1:3) to give **52** (5.7572 g, 73%) as a white crystalline solid, m.p. $152.5\text{--}168.2^{\circ}\text{C}$: ¹H NMR (400 MHz, CDCl₃) δ 8.25 (s, 1H), 6.99 (s, 1H), 3.64 (s, 2H), 2.82 (s, 3H), 1.58 (s, 6H), 1.33 (s, 6H); ¹³C NMR (100.6 MHz, CDCl₃) δ 146.0, 143.2, 139.1, 134.7, 127.3, 127.1, 75.2, 70.8, 33.5, 29.5, 27.0, 26.9, 22.6.

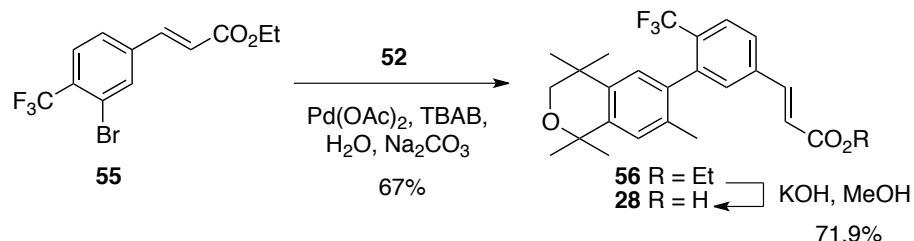


Scheme 4.

(E)-Ethyl 3-(4-methyl-3-(1,1,4,4,7-pentamethylisochroman-6-yl)phenyl)acrylate (54). To a 50 mL Schlenk flask charged with bromide **53** (1.6867 g, 6.2671 mmol), boronic acid **52** (1.5508 g, 6.2501 mmol), TBAB (1.9949 g), Na₂CO₃ (1.9559 g, 18.45 mmol), and water (14.2 mL), was added Pd(OAc)₂ (0.0819 g, 0.36 mmol), and the flask was evacuated and back-filled with nitrogen three times. The reaction was stirred at room temperature for 15 min and then placed in an oil bath pre-heated to 150°C and stirred for 5 min. The reaction was allowed to cool to room temperature, and the black residue was taken up in ethyl acetate and water. The layers were separated, and the aqueous layer was extracted with ethyl acetate. The combined organic layers were washed with brine, dried over sodium sulfate, filtered, and concentrated in vacuo to give a crude product that was purified by column chromatography (150 mL SiO₂, 2% - 4% ethyl acetate:hexanes) to give **54** (1.5781 g, 64.3%) as a white solid, m.p. $133.7\text{--}136.9^{\circ}\text{C}$: ¹H NMR (400 MHz, CDCl₃) δ 7.68 (d, *J* = 16.0, 1H), 7.41 (dd, *J* = 8.0, 2.0, 1H), 7.32 (d, *J* = 2.0, 1H), 7.28 (d, *J* = 8.0, 1H), 7.00 (s, 1H), 6.93 (s, 1H), 6.41 (d, *J* = 16.0, 1H), 4.25 (q, *J* = 7.2, 2H), 3.61 (s, 2H), 2.08 (s, 3H), 2.01 (s, 3H), 1.57 (s, 6H), 1.32 (t, *J* = 7.2, 3H), 1.25 (s, 3H), 1.23 (s, 3H); ¹³C NMR (100.6 MHz, CDCl₃) δ 167.1, 144.4, 142.1, 140.5, 139.8, 138.8, 138.6, 132.8, 131.9, 130.4, 129.1, 126.7, 126.5, 125.9, 117.4, 75.0, 70.9, 60.3, 33.6, 29.9, 29.8, 27.1, 27.0, 20.0, 19.5, 14.3; ES-MS (*M*+Na)⁺ calcd for C₂₆H₃₂O₃Na 415.2249, found 415.2248.

(E)-3-(4-methyl-3-(1,1,4,4,7-pentamethylisochroman-6-yl)phenyl)acrylic acid (27). To a 100 mL round bottom flask containing **54** (1.3285 g, 3.3844 mmol) suspended in methanol (9.0 mL) was added a solution of KOH (0.5744 g, 10.23 mmol) in water (0.48 mL), and the solution was refluxed in an oil-bath pre-heated to 85°C for 1 h. The reaction was allowed to cool to room temperature, and acidified with an aqueous 20% HCl solution (80 mL). The resulting precipitate was filtered and washed with copious amounts of water, and the crude white powder was purified by column chromatography (25 mL SiO₂, ethyl acetate:hexanes 1:3) to give **27** (1.0092 g, 81.8%) as a white crystalline solid, m.p. $219.3\text{--}224.3^{\circ}\text{C}$: ¹H NMR

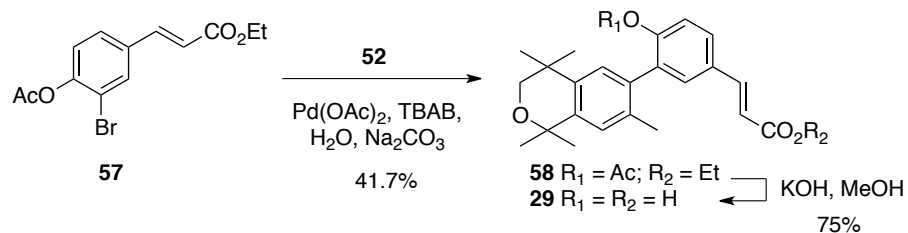
(400 MHz, CDCl₃) δ 8.78 (br s, 1H), 7.78 (d, J = 16.0, 1H), 7.45 (dd, J = 8.0, 1.6, 1H), 7.34 (d, J = 1.6, 1H), 7.30 (d, J = 8.0, 1H), 7.00 (s, 1H), 6.94 (s, 1H), 6.42 (d, J = 16.0, 1H), 3.62 (s, 2H), 2.10 (s, 3H), 2.02 (s, 3H), 1.58 (s, 6H), 1.26 (s, 3H), 1.23 (s, 3H); ¹³C NMR (100.6 MHz, CDCl₃) δ 172.3, 146.9, 142.2, 140.6, 139.8, 139.5, 138.4, 132.8, 131.5, 130.5, 129.4, 127.1, 126.5, 125.8, 116.4, 75.1, 70.8, 33.6, 29.9, 29.8, 27.1, 26.9, 20.1, 19.5; LC-MS (M-H)⁻ calcd for C₂₅H₂₇O₃ 363.1960, found 363.1953.



Scheme 5.

(E)-Ethyl 3-(3-(1,4,4,7-pentamethylisochroman-6-yl)-4-(trifluoromethyl)phenyl)acrylate (56). To a 50 mL Schlenk flask charged with bromide **55**[3] (2.0182 g, 6.246 mmol), boronic acid **52** (1.5545 g, 6.265 mmol), TBAB (2.046 g), Na₂CO₃ (1.9568 g, 18.46 mmol), and water (14.2 mL), was added Pd(OAc)₂ (0.0817 g, 0.36 mmol), and the flask was evacuated and back-filled with nitrogen three times. The reaction was stirred at room temperature for 15 min and then placed in an oil bath pre-heated to 150 °C and stirred for 5 min. The reaction was allowed to cool to room temperature, and the black residue was taken up in ethyl acetate and water. The layers were separated, and the aqueous layer was extracted with ethyl acetate. The combined organic layers were washed with brine, dried over sodium sulfate, filtered, and concentrated in vacuo to give a crude product that was purified by column chromatography (150 mL SiO₂, 2% - 4% ethyl acetate:hexanes) to give **56** (1.8746 g, 67.2%) as a white solid, m.p. 147.9-149.7 °C: ¹H NMR (400 MHz, CDCl₃) δ 7.76 (d, J = 8.4, 1H), 7.68 (d, J = 16.0, 1H), 7.58 (d, J = 8.0, 1H), 7.43 (s, 1H), 7.02 (s, 1H), 6.91 (s, 1H), 6.51 (d, J = 16.0, 1H), 4.26 (q, J = 7.2, 2H), 3.60 (s, 2H), 1.99 (s, 3H), 1.56 (s, 6H), 1.32 (t, J = 7.2, 3H), 1.23 (s, 3H), 1.20 (s, 3H); ¹³C NMR (100.6 MHz, CDCl₃) δ 166.3, 142.5, 141.5, 141.4, 141.2, 139.0, 137.2, 135.9, 132.8, 131.0, 130.1, 129.8, 126.7, 126.6, 126.5, 126.3, 126.2, 125.0, 122.2, 121.0, 75.0, 70.8, 60.7, 33.5, 30.0, 29.6, 27.0, 26.6, 19.8, 14.2; ES-MS (M+Na)⁺ calcd for C₂₆H₂₉F₃O₃Na 469.1967, found 469.1966.

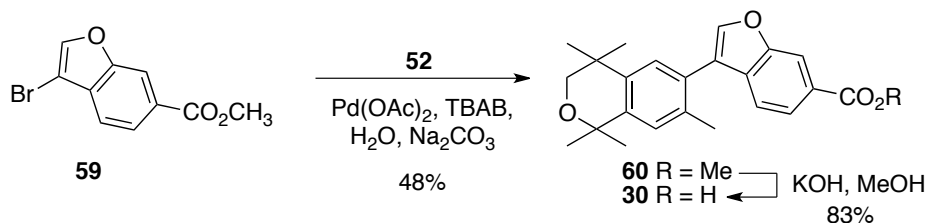
(E)-3-(3-(1,4,4,7-pentamethylisochroman-6-yl)-4-(trifluoromethyl)phenyl)acrylic acid (28). To a 100 mL round bottom flask containing **56** (1.5789 g, 3.536 mmol) suspended in methanol (7.0 mL) was added a solution of KOH (0.6462 g, 11.52 mmol) in water (0.48 mL), and the solution was refluxed in an oil-bath pre-heated to 85 °C for 1 h. The reaction was allowed to cool to room temperature, and acidified with an aqueous 1N HCl solution (80 mL). The resulting precipitate was filtered and washed with copious amounts of water, and the crude white powder was purified by column chromatography (150 mL SiO₂, ethyl acetate:hexanes 1:3) to give **28** (1.0647 g, 72%) as a white crystalline solid, m.p. 225.3-232.6 °C: ¹H NMR (400 MHz, CDCl₃) δ 7.79 (d, J = 16.0, 1H), 7.78 (d, J = 8.4, 1H), 7.62 (d, J = 8.4, 1H), 7.46 (s, 1H), 7.03 (s, 1H), 6.92 (s, 1H), 6.52 (d, J = 16.4, 1H), 3.62 (s, 2H), 2.00 (s, 3H), 1.58 (s, 6H), 1.24 (s, 3H), 1.21 (s, 3H); ¹³C NMR (100.6 MHz, CDCl₃) δ 171.3, 144.9, 141.6, 141.5, 141.2, 139.0, 136.7, 135.8, 132.8, 131.2, 130.6, 130.3, 126.9, 126.8, 126.7, 126.4, 126.3, 126.2, 124.9, 122.2, 120.0, 75.1, 70.8, 33.5, 29.9, 29.6, 27.0, 26.6, 19.8; ES-MS (M-H)⁻ calcd for C₂₄H₂₄F₃O₃ 417.1678, found 417.1697.



Scheme 6.

(E)-Ethyl 3-(4-acetoxy-3-(1,1,4,4,7-pentamethylisochroman-6-yl)phenyl)acrylate (58). To a 50 mL Schlenk flask charged with bromide **57**[3] (1.9557 g, 6.2454 mmol), boronic acid **52** (1.5712 g, 6.332 mmol), TBAB (1.9968 g), Na₂CO₃ (1.9556 g, 18.44 mmol), and water (14.20 mL), was added Pd(OAc)₂ (0.0819 g, 0.3637 mmol), and the flask was evacuated and back-filled with nitrogen three times. The reaction was stirred at room temperature for 15 min and then placed in an oil bath pre-heated to 150 °C and stirred for 5 min. The reaction was allowed to cool to room temperature, and the black residue was taken up in ethyl acetate and water. The layers were separated, and the aqueous layer was extracted with ethyl acetate. The combined organic layers were washed with brine, dried over sodium sulfate, filtered, and concentrated in vacuo to give a crude product that was purified by column chromatography (150 mL SiO₂, 4% - 8% ethyl acetate:hexanes) to give **58** (1.1370 g, 41.7%) as a white solid, m.p. 132.7-136.4 °C: ¹H NMR (400 MHz, CDCl₃) δ 7.68 (d, J = 16.0, 1H), 7.54 (dd, J = 8.4, 2.0, 1H), 7.46 (d, J = 2.0, 1H), 7.16 (d, J = 8.4, 1H), 7.04 (s, 1H), 6.93 (s, 1H), 6.40 (d, J = 16.0, 1H), 4.25 (q, J = 7.2, 2H), 3.59 (m, 2H), 2.11 (s, 3H), 1.92 (s, 3H), 1.57 (s, 6H), 1.32 (t, J = 7.2, 3H), 1.23 (s, 6H); ¹³C NMR (100.6 MHz, CDCl₃) δ 169.0, 166.8, 149.8, 143.4, 141.2, 139.5, 135.3, 134.0, 133.3, 132.3, 130.9, 127.9, 126.7, 126.6, 123.1, 118.6, 75.0, 70.8, 60.5, 33.5, 20.4, 19.6, 14.2; ES-MS (M+H)⁺ calcd for C₂₇H₃₃O₅ 437.2328, found 437.2324.

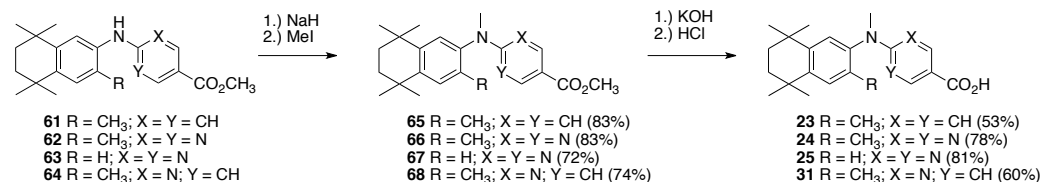
(E)-3-94-hydroxy-3-(1,1,4,4,7-pentamethylisochroman-6-yl)phenyl)acrylic acid (29). To a 100 mL round bottom flask containing **58** (1.0352 g, 2.3714 mmol) suspended in methanol (8.0 mL) was added a solution of KOH (0.9439 g, 16.82 mmol) in water (0.70 mL), and the solution was refluxed in an oil-bath pre-heated to 85 °C for 1.5 h. The reaction was allowed to cool to room temperature, and acidified with an aqueous 1N HCl solution (80 mL). The resulting precipitate was filtered and washed with copious amounts of water, and the crude white powder was purified by column chromatography (25 mL SiO₂, 35% ethyl acetate:hexanes) to give **29** (0.6541 g, 75.2%) as a white crystalline solid, m.p. 175.2-178.0 °C: ¹H NMR (400 MHz, CDCl₃) δ 7.75 (d, J = 15.6, 1H), 7.50 (dd, J = 8.4, 2.0, 1H), 7.35 (d, J = 2.0, 1H), 7.14 (s, 1H), 7.02 (s, 1H), 7.01 (d, J = 8.4, 1H), 6.32 (d, J = 15.6, 1H), 3.62 (s, 2H), 2.13 (s, 3H), 1.58 (s, 6H), 1.26 (s, 6H); ¹³C NMR (100.6 MHz, CDCl₃) δ 172.4, 155.1, 146.6, 142.3, 141.0, 134.4, 132.6, 130.8, 129.6, 128.3, 127.6, 127.1, 126.7, 115.9, 114.8, 75.1, 70.7, 33.7, 29.7, 27.0, 19.4; ES-MS (M+Na)⁺ calcd for C₂₃H₂₆O₄Na 389.1729, found 389.1737.



Scheme 7.

Methyl 3-(1,1,4,4,7-pentamethylisochroman-6-yl)benzofuran-6-carboxylate (60). To a 50 mL Schlenk flask charged with bromide **59** (0.9063 g, 3.553 mmol), boronic acid **52** (0.8888 g, 3.582 mmol), TBAB (1.1366 g), Na₂CO₃ (1.1176 g, 10.54 mmol), and water (8.05 mL), was added Pd(OAc)₂ (0.0507 g, 0.225 mmol), and the flask was evacuated and back-filled with nitrogen three times. The reaction was stirred at room temperature for 15 min and then placed in an oil bath pre-heated to 150 °C and stirred for 5 min. The reaction was allowed to cool to room temperature, and the black residue was taken up in ethyl acetate and water. The layers were separated, and the aqueous layer was extracted with ethyl acetate. The combined organic layers were washed with brine, dried over sodium sulfate, filtered, and concentrated in vacuo to give a crude product that was purified by column chromatography (150 mL SiO₂, 2% - 4% ethyl acetate:hexane) to give **60** (0.6486 g, 48.2%) as a white solid, m.p. 129.8 – 133.0 °C: ¹H NMR (400 MHz, CDCl₃) δ 8.21 (s, 1H), 8.07 (dd, J = 8.8, 1.6, 1H), 7.68 (s, 1H), 7.57 (dd, J = 8.8, 0.4, 1H), 7.30 (s, 1H), 7.03 (s, 1H), 3.91 (s, 3H), 3.64 (s, 2H), 2.26 (s, 3H), 1.60 (s, 6H), 1.29 (s, 6H); ¹³C NMR (100.6 MHz, CDCl₃) δ 167.2, 157.6, 143.2, 141.5, 140.2, 134.1, 128.1, 127.3, 127.2, 126.1, 125.2, 123.2, 121.9, 111.5, 75.0, 70.8, 52.1, 33.6, 29.7, 27.0, 20.4; ES-MS (M+Na)⁺ calcd for C₂₄H₂₆O₄Na 401.1729, found 401.1740.

3-(1,1,4,4,7-pentamethylisochroman-6-yl)benzofuran-6-carboxylic acid (30). To a 100 mL round bottom flask containing **60** (0.5813 g, 1.5359 mmol) suspended in methanol (4.4 mL) was added a solution of KOH (0.2749 g, 5.08 mmol) in water (0.28 mL), and the solution was refluxed in an oil-bath pre-heated to 85 °C for 1.5 h. The reaction was allowed to cool to room temperature, and acidified with an aqueous 1N HCl solution (80 mL). The resulting precipitate was filtered and washed with copious amounts of water, and the crude white powder was purified by column chromatography (25 mL SiO₂, 25% ethyl acetate:hexanes) to give **30** (0.4670 g, 83.4%) as a white crystalline solid, m.p. 251.8-254.8 °C: ¹H NMR (400 MHz, CDCl₃) δ 9.49 (br s, 1H), 8.30 (d, J = 1.6, 1H), 8.15 (dd, J = 8.4, 1.6, 1H), 7.71 (s, 1H), 7.61 (d, J = 8.8, 1H), 7.31 (s, 1H), 7.04 (s, 1H), 3.65 (s, 2H), 2.28 (s, 3H), 1.61 (s, 6H), 1.30 (s, 6H); ¹³C NMR (100.6 MHz, CDCl₃) δ 172.1, 158.1, 143.4, 141.5, 140.2, 134.0, 128.1, 128.0, 127.3, 127.1, 126.7, 124.3, 124.1, 121.9, 111.7, 75.1, 70.8, 33.6, 29.7, 27.0, 20.2; ES-MS (M+Na)⁺ calcd for C₂₃H₂₄O₄Na 387.1572, found 387.1566.



Scheme 8.

Methyl 4-(methyl(3,5,5,8,8-pentamethyl-5,6,7,8-tetrahydronaphthalen-2-yl)amino)benzoate (65). To a flame-dried, 100 mL round-bottomed flask equipped with a magnetic stir bar was added a 60% dispersion of sodium hydride in mineral oil (0.3682 g, 9.206 mmol). The dispersion of sodium hydride was washed with hexanes (3.0 mL, twice) and dried under vacuum and suspended in 4.8 mL of DMF under nitrogen. To this solution of sodium hydride in DMF was added a solution of **61**[4] (1.3285 g, 3.7796 mmol) in DMF (14.0 mL), and the reaction was stirred for 15 min., and then methyl iodide (0.43 mL, 6.91 mmol) was added, and the reaction was stirred for 1 h. The reaction was poured into water and extracted with ethyl acetate. The combined organic layers were washed with brine, dried over sodium sulfate, filtered, and concentrated in vacuo to yield a crude product that was purified by column chromatography (150 mL SiO₂, 6% ethyl acetate:hexanes) to give **65** (1.1502 g, 83.3%) as a white crystalline solid, m.p. 128.5 - 160.0 °C: ¹H NMR (400 MHz, CDCl₃) δ 7.84 (d, J = 8.8, 2H), 7.18 (s, 1H), 7.03 (s, 1H), 6.46 (d, J = 8.8, 1H), 3.84 (s, 3H), 3.26 (s, 3H), 2.03 (s, 3H), 1.68 (s, 4H), 1.30 (s, 6H), 1.22 (s, 6H); ¹³C NMR (100.6 MHz, CDCl₃) δ 167.4, 152.5, 144.5, 143.8, 142.6, 132.7, 131.1, 129.3, 125.8, 117.2, 111.1, 51.4, 39.1, 35.0, 34.9, 34.1, 33.9, 31.8, 17.3; ES-MS (M+H)⁺ calcd for C₂₄H₃₁NO₂ 366.2433, found 366.2418.

4-(Methyl(3,5,5,8,8-pentamethyl-5,6,7,8-tetrahydronaphthalen-2-yl)amino)benzoic acid (23). To a 100 mL round-bottomed flask equipped with a stir bar and charged with methyl ester **65** (1.0449 g, 2.8588 mmol) suspended in methanol (13.0 mL) was added a solution of potassium hydroxide (0.5551 g, 9.89 mmol) in water (0.64 mL). This reaction was stirred at reflux in an oil bath at 87 °C for 1.5h. The reaction was then cooled to r.t. and acidified with 1N HCl (90 mL). The resulting precipitate was filtered and washed with copious amounts of cold water to give a crude product that was purified by column chromatography (25 mL SiO₂, 15% ethyl acetate:hexanes) to give pure **23** (0.5403 g, 53.8%) as a white crystalline solid, m.p. 242.6 - 247.7 °C: ¹H NMR (400 MHz, d₆-DMSO) δ 12.14 (br s, 1H), 7.70 (d, J = 9.2, 2H), 7.27 (s, 1H), 7.07 (s, 1H), 6.42 (d, J = 8.4, 1H), 3.20 (s, 3H), 1.97 (s, 3H), 1.63 (s, 4H), 1.25 (s, 6H), 1.19 (s, 6H); ¹³C NMR (100.6 MHz, d₆-DMSO) δ 167.4, 152.0, 144.1, 143.2, 132.4, 131.0, 129.1, 125.5, 117.6, 111.0, 59.7, 34.6, 34.5, 33.8, 33.7, 31.7, 31.6, 17.0; ES-MS (M+Na)⁺ calcd for C₂₃H₂₉NO₂Na 374.2096, found 374.2097.

Methyl 2-(methyl(3,5,5,8,8-pentamethyl-5,6,7,8-tetrahydronaphthalen-2-yl)amino)pyrimidine-5-carboxylate (66). To a flame-dried, 100 mL round-bottomed flask equipped with a magnetic stir bar was added a 60% dispersion of sodium hydride in mineral oil (0.4425 g, 11.06 mmol). The dispersion of sodium hydride was washed with hexanes (3.7 mL, twice) and dried under vacuum and suspended in 5.8 mL of DMF under nitrogen. To this solution of sodium hydride in DMF was added a solution of **62**[4] (1.6074 g, 4.548 mmol) in DMF (16.9 mL), and the reaction was stirred for 15 min., and then methyl iodide

(0.43 mL, 6.91 mmol) was added, and the reaction was stirred for 1 h. The reaction was poured into water and extracted with ethyl acetate. The combined organic layers were washed with brine, dried over sodium sulfate, filtered, and concentrated in vacuo to yield a crude product that was purified by column chromatography (150 mL SiO₂, 6% ethyl acetate:hexanes) to give **66** (1.4023 g, 83.9%) as a white crystalline solid, m.p. 219.6 - 221.8 °C: ¹H NMR (400 MHz, CDCl₃) δ 8.96 (d, J = 2.8, 1H), 8.81 (d, J = 2.8, 1H), 7.19 (s, 1H), 7.04 (s, 1H), 3.86 (s, 3H), 3.50 (s, 3H), 2.05 (s, 3H), 1.68 (s, 4H), 1.31 (s, 3H), 1.27 (s, 3H), 1.25 (s, 3H), 1.24 (s, 3H); ¹³C NMR (100.6 MHz, CDCl₃) δ 165.1, 161.9, 160.2, 159.1, 144.2, 144.1, 140.4, 131.5, 129.1, 124.7, 112.7, 51.8, 39.0, 35.0, 34.9, 34.1, 34.0, 32.0, 31.9, 31.8, 31.6, 17.4; GC-MS CI (M)+ calcd for C₂₂H₂₉N₃O₂ 367.2260, found 367.2251.

2-(Methyl(3,5,5,8,8-pentamethyl-5,6,7,8-tetrahydronaphthalen-2-yl)amino)pyrimidine-5-carboxylic acid (24). To a 100 mL round-bottomed flask equipped with a stir bar and charged with methyl ester **66** (0.9053 g, 2.464 mmol) suspended in methanol (9.0 mL) was added a solution of potassium hydroxide (0.4726 g, 8.423 mmol) in water (0.54 mL). This reaction was stirred at reflux in an oil bath at 87 °C for 1.5h. The reaction was then cooled to r.t. and acidified with 1N HCl (90 mL). The resulting precipitate was filtered and washed with copious amounts of cold water to give a crude product that was purified by column chromatography (25 mL SiO₂, 60% ethyl acetate:hexanes to 1% methanol:ethyl acetate) to give pure **24** (0.6819 g, 78.3%) as a white crystalline solid, m.p. 323.0 – 326.9 °C: ¹H NMR (400 MHz, d₆-DMSO) δ 12.87 (br s, 1H), 8.87 (d, J = 2.8, 1H), 8.65 (d, J = 2.8, 1H), 7.22 (s, 1H), 7.15 (s, 1H), 3.40 (s, 3H), 1.95 (s, 3H), 1.62 (s, 4H), 1.26 (s, 3H), 1.24 (s, 3H), 1.21 (s, 3H), 1.18 (s, 3H); ¹³C NMR (100.6 MHz, d₆-DMSO) δ 165.6, 162.2, 159.7, 143.5, 143.1, 141.0, 131.8, 128.5, 124.7, 113.2, 38.4, 34.7, 34.6, 33.8, 33.7, 31.6, 31.5, 17.1; ES-MS (M+H)+ calcd for C₂₁H₂₈N₃O₂ 354.2181, found 354.2184.

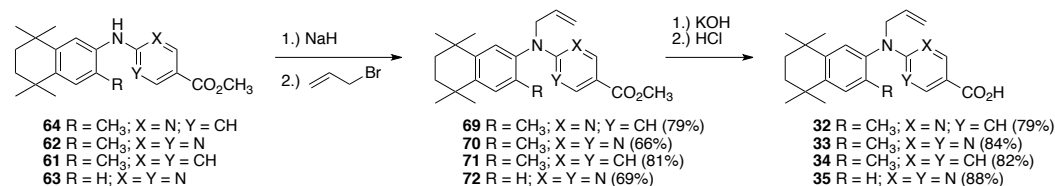
Methyl 2-(methyl(5,5,8,8-tetramethyl-5,6,7,8-tetrahydronaphthalen-2-yl)amino)pyrimidine-5-carboxylate (67). To a flame-dried, 100 mL round-bottomed flask equipped with a magnetic stir bar was added a 60% dispersion of sodium hydride in mineral oil (0.4435 g, 11.09 mmol). The dispersion of sodium hydride was washed with hexanes (3.0 mL, twice) and dried under vacuum and suspended in 5.8 mL of DMF under nitrogen. To this solution of sodium hydride in DMF was added a solution of **63**[4] (1.4193 g, 4.1814 mmol) in DMF (16.4 mL), and the reaction was stirred for 15 min., and then methyl iodide (0.42 mL, 6.74 mmol) was added, and the reaction was stirred for 1 h. The reaction was poured into water and extracted with ethyl acetate. The combined organic layers were washed with brine, dried over sodium sulfate, filtered, and concentrated in vacuo to yield a crude product that was purified by column chromatography (150 mL SiO₂, 5% to 10% ethyl acetate:hexanes) to give **67** (1.0728 g, 72.6%) as a white crystalline solid, m.p. 211.8 - 212.9 °C: ¹H NMR (400 MHz, CDCl₃) δ 8.88 (s, 2H), 7.34 (d, J = 8.4, 1H), 7.19 (d, J = 2.4, 1H), 7.05 (dd, J = 8.4, 2.4, 1H), 3.87 (s, 3H), 3.58 (s, 3H), 1.69 (s, 4H), 1.29 (s, 6H), 1.27 (s, 6H); ¹³C NMR (100.6 MHz, CDCl₃) δ 165.1, 162.2, 159.5, 145.9, 143.1, 141.5, 127.3, 124.0, 123.2, 113.1, 51.8, 39.5, 34.9, 34.9, 34.4, 34.1, 31.7; ES-MS (M+H)+ calcd for C₂₁H₂₈N₃O₂ 354.2181, found 354.2181.

2-(Methyl(5,5,8,8-tetramethyl-5,6,7,8-tetrahydronaphthalen-2-yl)amino)pyrimidine-5-carboxylic acid (25). To a 100 mL round-bottomed flask equipped with a stir bar and charged with methyl ester **67** (0.9075 g, 2.568 mmol) suspended in methanol (8.0 mL) was added a solution of potassium hydroxide (0.4839 g, 8.62 mmol) in water (0.48 mL). This reaction was stirred at reflux in an oil bath at 87 °C for 1.5h. The reaction was then cooled to r.t. and acidified with 1N HCl (80 mL). The resulting precipitate was filtered and washed with copious amounts of cold water to give a crude product that was purified by column chromatography (25 mL SiO₂, 60% ethyl acetate:hexanes to pure ethyl acetate to 1% methanol:ethyl acetate) to give pure **25** (0.7109 g, 81.6%) as a white crystalline solid, m.p. 313.5 – 316.4 °C: ¹H NMR (400 MHz, d₆-DMSO) δ 12.86 (br s, 1H), 8.75 (s, 2H), 7.33 (d, J = 8.4, 2H), 7.26 (d, J = 2.0, 1H), 7.06 (dd, J = 8.4, 2.4, 1H), 3.48 (s, 3H), 1.64 (s, 4H), 1.25 (s, 6H), 1.23 (s, 6H); ¹³C NMR (100.6 MHz, d₆-DMSO) δ 165.5, 162.3, 159.4, 145.3, 142.2, 141.9, 126.9, 124.1, 124.0, 113.7, 34.5, 34.1, 33.8, 31.6, 31.5; ES-MS (M+H)+ calcd for C₂₀H₂₆N₃O₂ 340.2025, found 340.2025.

Methyl 6-(methyl(3,5,5,8,8-pentamethyl-5,6,7,8-tetrahydronaphthalen-2-yl)amino)nicotinate (68). To a flame-dried, 100 mL round-bottomed flask equipped with a magnetic stir bar was added a 60%

dispersion of sodium hydride in mineral oil (0.4232 g, 10.58 mmol). The dispersion of sodium hydride was washed with hexanes (3.7 mL, twice) and dried under vacuum and suspended in 5.8 mL of DMF under nitrogen. To this solution of sodium hydride in DMF was added a solution of **64**[4] (1.6059 g, 4.556 mmol) in DMF (17.3 mL), and the reaction was stirred for 15 min., and then methyl iodide (0.43 mL, 6.91 mmol) was added, and the reaction was stirred for 1 h. The reaction was poured into water and extracted with ethyl acetate. The combined organic layers were washed with brine, dried over sodium sulfate, filtered, and concentrated in vacuo to yield a crude product that was purified by column chromatography (150 mL SiO₂, 5% - 10% ethyl acetate:hexanes) to give **68** (1.2434 g, 74.5%) as a white crystalline solid, m.p. 161.7-163.6 °C: ¹H NMR (400 MHz, CDCl₃) δ 8.86 (dd, J = 2.0, 0.4, 1H), 7.82 (dd, J = 9.2, 2.0, 1H), 7.21 (s, 1H), 7.05 (s, 1H), 6.03 (d, J = 9.2, 1H), 3.85 (s, 3H), 3.46 (s, 3H), 2.05 (s, 3H), 1.68 (s, 4H), 1.30 (s, 6H), 1.23 (s, 6H); ¹³C NMR (100.6 MHz, CDCl₃) δ 166.4, 160.1, 150.4, 145.0, 144.7, 140.6, 137.7, 132.3, 129.5, 114.2, 107.2, 51.6, 38.0, 35.0, 34.9, 34.1, 34.0, 31.8, 17.1; ES-MS (M+H)⁺ calcd for C₂₃H₃₁N₂O₂ 367.2386, found 367.2390.

6-(Methyl(3,5,5,8,8-pentamethyl-5,6,7,8-tetrahydronaphthalen-2-yl)amino)nicotinic acid (31). To a 100 mL round-bottomed flask equipped with a stir bar and charged with methyl ester **68** (0.8203 g, 2.238 mmol) suspended in methanol (8.0 mL) was added a solution of potassium hydroxide (0.4284 g, 7.635 mmol) in water (0.42 mL). This reaction was stirred at reflux in an oil bath at 87 °C for 1.5h. The reaction was then cooled to r.t. and acidified with 1N HCl (80 mL). The solution was extracted with ethyl acetate (2 X 70 mL), and the organic layers were dried over sodium sulfate and concentrated in vacuo to give a crude product that was purified by column chromatography (25 mL SiO₂, 30% ethyl acetate:hexanes to 60% ethyl acetate:hexanes) to give pure **31** (0.4790 g, 60.7%) as a white crystalline solid, m.p. 273.5-274.8 °C: ¹H NMR (400 MHz, CDCl₃) δ 10.37 (br s, 1H), 8.97 (d, J = 2.0, 1H), 7.91 (d, J = 7.6, 1H), 7.22 (s, 1H), 7.06 (s, 1H), 6.10 (d, J = 8.4, 1H), 3.53 (s, 3H), 2.07 (s, 3H), 1.69 (s, 4H), 1.31 (s, 3H), 1.29 (s, 3H), 1.24 (s, 3H), 1.22 (s, 3H); ¹³C NMR (100.6 MHz, CDCl₃) δ 169.6, 161.9, 145.3, 139.9, 139.1, 132.1, 131.9, 129.7, 125.3, 114.1, 112.9, 108.3, 38.9, 34.9, 34.8, 34.2, 34.1, 31.9, 31.8, 17.1; ES-MS (M+H)⁺ calcd for C₂₂H₂₉N₂O₂ 353.2229, found 353.2224.



Scheme 9.

Methyl 6-(allyl(3,5,5,8,8-pentamethyl-5,6,7,8-tetrahydronaphthalen-2-yl)amino)nicotinate (69). To a flame-dried, 100 mL round-bottomed flask equipped with a magnetic stir bar was added a 60% dispersion of sodium hydride in mineral oil (0.4441 g, 11.10 mmol). The dispersion of sodium hydride was washed with hexanes (3.0 mL, twice) and dried under vacuum and suspended in 5.8 mL of DMF under nitrogen. To this solution of sodium hydride in DMF was added a solution of **64**[4] (1.6111 g, 4.571 mmol) in DMF (16.9 mL), and the reaction was stirred for 15 min., and then allyl bromide (0.67 mL, 7.74 mmol) was added, and the reaction was stirred for 1 h. The reaction was poured into water and extracted with ethyl acetate. The combined organic layers were washed with brine, dried over sodium sulfate, filtered, and concentrated in vacuo to yield a crude product that was purified by column chromatography (150 mL SiO₂, 5% ethyl acetate:hexanes) to give **69** (1.4175 g, 79%) as a white crystalline solid, m.p. 88.6 - 90.0 °C: ¹H NMR (400 MHz, CDCl₃) δ 8.86 (d, J = 1.6, 1H), 7.85 (dd, J = 8.8, 1.6, 1H), 7.20 (s, 1H), 7.01 (s, 1H), 6.08-5.98 (m, 2H), 5.21-5.15 (m, 2H), 4.98-4.96 (m, 1H), 4.12 (m, 1H), 3.85 (s, 3H), 2.03 (s, 3H), 1.67 (s, 4H), 1.29 (s, 3H), 1.28 (s, 3H), 1.20 (s, 6H); ¹³C NMR (100.6 MHz, CDCl₃) δ 166.0, 158.9, 149.8, 144.9, 144.6, 138.8, 138.4, 133.0, 132.4, 129.4, 126.7, 117.9, 114.6, 107.6, 53.0, 51.7, 34.9, 34.8, 34.1, 34.0, 31.7, 17.3; ES-MS (M+H)⁺ calcd for C₂₅H₃₃N₂O₂ 393.2542, found 393.2545.

6-(Allyl(3,5,5,8,8-pentamethyl-5,6,7,8-tetrahydronaphthalen-2-yl)amino)nicotinic acid (32). To a 100 mL round-bottomed flask equipped with a stir bar and charged with methyl ester **69** (1.2842 g, 3.272 mmol)

suspended in methanol (8.0 mL) was added a solution of potassium hydroxide (0.6365 g, 11.34 mmol) in water (0.71 mL). This reaction was stirred at reflux in an oil bath at 87 °C for 1.5h. The reaction was then cooled to r.t. and acidified with 1N HCl (80 mL). The resulting precipitate was filtered and washed with copious amounts of cold water to give a crude product that was purified by column chromatography (25 mL SiO₂, 30% ethyl acetate:hexanes) to give pure **32** (0.9875 g, 79.7%) as a white crystalline solid, m.p. 203.4 – 204.4 °C: ¹H NMR (400 MHz, d₆-DMSO) δ 12.50 (br s, 1H), 8.67 (d, J = 2.0, 1H), 7.82 (dd, J = 8.8, 2.4, 2H), 7.30 (s, 1H), 7.08 (s, 1H), 6.02-5.92 (m, 2H), 5.14-5.09 (m, 2H), 4.80-4.77 (m, 1H), 4.12-4.10 (m, 1H), 1.98 (s, 3H), 1.60 (s, 4H), 1.22 (s, 6H), 1.12 (s, 6H); ¹³C NMR (100.6 MHz, d₆-DMSO) δ 166.6, 159.5, 150.6, 143.9, 139.2, 138.1, 134.1, 132.5, 129.2, 126.5, 117.3, 114.9, 106.1, 52.1, 34.6, 34.4, 33.8, 33.7, 31.5, 17.1; ES-MS (M+Na)⁺ calcd for C₂₄H₃₀N₂O₂Na 401.2205, found 401.2202.

Methyl 2-(allyl(3,5,5,8,8-pentamethyl-5,6,7,8-tetrahydronaphthalen-2-yl)amino)pyrimidine-5-carboxylate (70). To a flame-dried, 100 mL round-bottomed flask equipped with a magnetic stir bar was added a 60% dispersion of sodium hydride in mineral oil (0.4412 g, 11.03 mmol). The dispersion of sodium hydride was washed with hexanes (3.0 mL, twice) and dried under vacuum and suspended in 5.8 mL of DMF under nitrogen. To this solution of sodium hydride in DMF was added a solution of **62**[4] (1.6218 g, 4.588 mmol) in DMF (16.9 mL), and the reaction was stirred for 15 min., and then allyl bromide (0.67 mL, 7.74 mmol) was added, and the reaction was stirred for 1 h. The reaction was poured into water and extracted with ethyl acetate. The combined organic layers were washed with brine, dried over sodium sulfate, filtered, and concentrated in vacuo to yield a crude product that was purified by column chromatography (150 mL SiO₂, 6% ethyl acetate:hexanes) to give **70** (1.1987 g, 66.3%) as a white crystalline solid, m.p. 165.1-166.7 °C: ¹H NMR (400 MHz, CDCl₃) δ 8.95 (s, 1H), 8.81 (s, 1H), 7.17 (s, 1H), 7.02 (s, 1H), 6.07-5.97 (dddd, J = 15.6, 11.2, 6.8, 5.6, 1H), 5.20 (d, J = 16.0, 1H), 5.18 (d, J = 11.2, 1H), 4.86-4.80 (dd, J = 15.2, 5.6, 1H), 4.15-4.10 (dd, J = 14.8, 6.8, 1H), 3.86 (s, 3H), 2.04 (s, 3H), 1.67 (s, 4H), 1.31 (s, 3H), 1.26 (s, 3H), 1.23 (s, 3H), 1.21 (s, 3H); ¹³C NMR (100.6 MHz, CDCl₃) δ 165.2, 162.0, 160.1, 159.6, 144.0, 143.7, 139.2, 133.0, 131.9, 128.9, 125.6, 117.9, 112.9, 53.9, 51.7, 35.0, 34.9, 34.0, 33.9, 32.0, 31.9, 31.7, 31.6, 17.6; ES-MS (M+H)⁺ calcd for C₂₄H₃₂N₃O₂ 394.2495, found 394.2486.

2-(Allyl(3,5,5,8,8-pentamethyl-5,6,7,8-tetrahydronaphthalen-2-yl)amino)pyrimidine-5-carboxylic acid (33). To a 100 mL round-bottomed flask equipped with a stir bar and charged with methyl ester **70** (1.0549 g, 2.681 mmol) suspended in methanol (9.0 mL) was added a solution of potassium hydroxide (0.4895 g, 8.72 mmol) in water (0.59 mL). This reaction was stirred at reflux in an oil bath at 87 °C for 1.5h. The reaction was then cooled to r.t. and acidified with 1N HCl (85 mL). The resulting precipitate was filtered and washed with copious amounts of cold water to give a crude product that was purified by column chromatography (25 mL SiO₂, 40% ethyl acetate:hexanes) to give pure **33** (0.8575 g, 84.3%) as a white crystalline solid, m.p. 216.7 – 217.7 °C: ¹H NMR (400 MHz, d₆-DMSO) δ 12.89 (br s, 1H), 8.86 (s, 1H), 8.67 (s, 1H), 7.21 (s, 1H), 7.06 (s, 1H), 6.03-5.93 (dddd, J = 16.0, 11.6, 6.0, 5.6, 1H), 5.16 (d, J = 16.0, 1H), 5.14 (d, J = 11.6, 1H), 4.75-4.70 (dd, J = 15.2, 5.6, 1H), 4.22-4.17 (dd, J = 15.2, 6.4, 1H), 1.95 (s, 3H), 1.61 (s, 4H), 1.26 (s, 3H), 1.24 (s, 3H), 1.18 (s, 6H); ¹³C NMR (100.6 MHz, d₆-DMSO) δ 165.5, 161.8, 159.7, 143.1, 143.0, 139.5, 133.6, 132.2, 128.5, 125.5, 117.8, 113.7, 53.2, 34.6, 34.5, 33.7, 33.6, 31.7, 31.6, 31.5, 17.4; ES-MS (M+Na)⁺ calcd for C₂₃H₂₉N₃O₂Na 402.2158, found 402.2153.

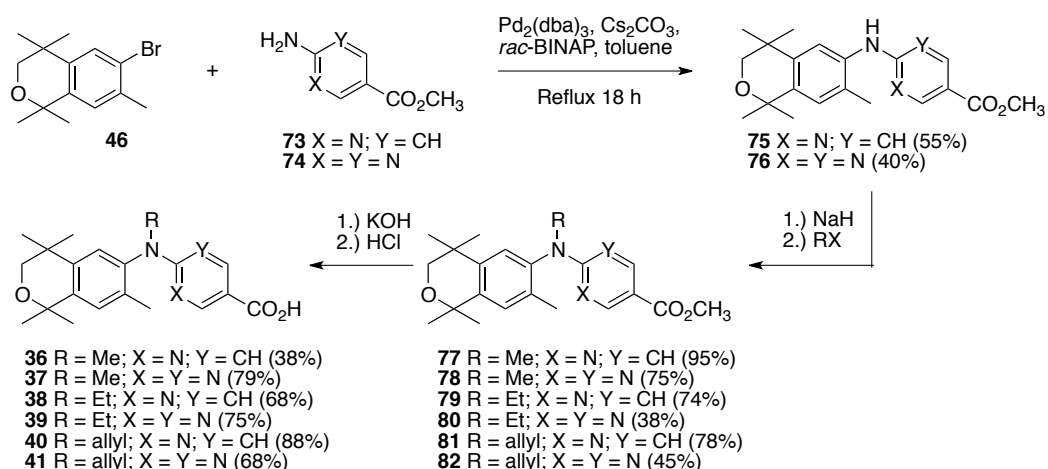
Methyl 4-(allyl(3,5,5,8,8-pentamethyl-5,6,7,8-tetrahydronaphthalen-2-yl)aminobenzoate (71). To a flame-dried, 100 mL round-bottomed flask equipped with a magnetic stir bar was added a 60% dispersion of sodium hydride in mineral oil (0.3564 g, 8.91 mmol). The dispersion of sodium hydride was washed with hexanes (3.0 mL, twice) and dried under vacuum and suspended in 4.8 mL of DMF under nitrogen. To this solution of sodium hydride in DMF was added a solution of **61**[4] (1.3122 g, 3.733 mmol) in DMF (14.0 mL), and the reaction was stirred for 15 min., and then allyl bromide (0.55 mL, 6.40 mmol) was added, and the reaction was stirred for 1 h. The reaction was poured into water and extracted with ethyl acetate. The combined organic layers were washed with brine, dried over sodium sulfate, filtered, and concentrated in vacuo to yield a crude product that was purified by column chromatography (150 mL SiO₂, 2% ethyl acetate:hexanes) to give **71** (1.1947 g, 81.7%) as a white crystalline solid, m.p.

101.9-104.6 °C: ¹H NMR (400 MHz, CDCl₃) δ 7.81 (d, J = 9.2, 2H), 7.19 (s, 1H), 7.04 (s, 1H), 6.46 (d, J = 8.8, 2H), 6.01-5.91 (dddd, J = 17.2, 10.8, 5.6, 5.2, 1H), 5.29 (dd, J = 17.2, 1.6, 1H), 5.22 (dd, J = 10.4, 1.6, 1H), 4.21 (br s, 2H), 3.83 (s, 3H), 2.04 (s, 3H), 1.68 (s, 4H), 1.30 (s, 6H), 1.21 (s, 6H); ¹³C NMR (100.6 MHz, CDCl₃) δ 167.3, 151.9, 144.4, 143.9, 141.6, 133.5, 132.8, 131.1, 129.3, 126.7, 117.4, 117.0, 111.6, 54.5, 51.4, 35.0, 34.9, 34.1, 34.0, 31.9, 31.8, 17.6; ES-MS (M+Na)⁺ calcd for C₂₆H₃₃NO₂Na 414.2409, found 414.2408.

4-(Allyl(3,5,5,8,8-pentamethyl-5,6,7,8-tetrahydronaphthalen-2-yl)amino)benzoic acid (34). To a 100 mL round-bottomed flask equipped with a stir bar and charged with methyl ester **71** (0.6599 g, 1.685 mmol) suspended in methanol (4.0 mL) was added a solution of potassium hydroxide (0.3854 g, 6.87 mmol) in water (0.38 mL). This reaction was stirred at reflux in an oil bath at 87 °C for 1.5h. The reaction was then cooled to r.t. and acidified with 1N HCl (90 mL). The resulting precipitate was filtered and washed with copious amounts of cold water to give a crude product that was purified by column chromatography (25 mL SiO₂, 15% to 30% ethyl acetate:hexanes) to give pure **34** (0.5233 g, 82.2%) as a white crystalline solid, m.p. 235.2 – 238.8 °C: ¹H NMR (400 MHz, d₆-DMSO) δ 12.14 (br s, 1H), 7.68 (d, J = 8.8, 2H), 7.28 (s, 1H), 7.07 (s, 1H), 6.41 (d, J = 8.8, 2H), 5.99-5.89 (dddd, J = 17.2, 10.4, 5.6, 5.2, 1H), 5.27 (dd, J = 17.2, 1.6, 1H), 5.20 (dd, J = 10.4, 1.6, 1H), 4.21 (br s, 2H), 1.98 (s, 3H), 1.62 (s, 4H), 1.25 (s, 6H), 1.12 (s, 6H); ¹³C NMR (100.6 MHz, d₆-DMSO) δ 167.4, 151.3, 143.9, 143.3, 141.4, 133.8, 132.6, 131.0, 129.2, 126.3, 117.8, 116.9, 111.3, 54.0, 34.6, 34.4, 33.8, 33.7, 31.6, 31.6, 17.2; ES-MS (-) (M-H)⁻ calcd for C₂₅H₃₀NO₂ 376.2277, found 376.2270.

Methyl 2-(allyl(5,5,8,8-tetramethyl-5,6,7,8-tetrahydronaphthalen-2-yl)amino)pyrimidine-5-carboxylate (72). To a flame-dried, 100 mL round-bottomed flask equipped with a magnetic stir bar was added a 60% dispersion of sodium hydride in mineral oil (0.3127 g, 7.82 mmol). The dispersion of sodium hydride was washed with hexanes (3.0 mL, twice) and dried under vacuum and suspended in 4.2 mL of DMF under nitrogen. To this solution of sodium hydride in DMF was added a solution of **63**[4] (1.1785 g, 3.472 mmol) in DMF (12.2 mL), and the reaction was stirred for 15 min., and then allyl bromide (0.50 mL, 5.78 mmol) was added, and the reaction was stirred for 1 h. The reaction was poured into water and extracted with ethyl acetate. The combined organic layers were washed with brine, dried over sodium sulfate, filtered, and concentrated in vacuo to yield a crude product that was purified by column chromatography (150 mL SiO₂, 2% to 6% to 10% ethyl acetate:hexanes) to give **72** (0.9160 g, 69.5%) as a white crystalline solid, m.p. 161.6-165.8 °C: ¹H NMR (400 MHz, CDCl₃) δ 8.88 (s, 2H), 7.31 (d, J = 8.4, 1H), 7.20 (d, J = 2.4, 1H), 7.05 (dd, J = 8.4, 2.4, 1H), 6.06-5.96 (dddd, J = 16.8, 10.4, 5.2, 5.2, 1H), 5.23-5.18 (dd, J = 17.2, 1.6, 1H), 5.20-5.17 (dd, J = 10.4, 1.6, 1H), 4.62-4.60 (d, J = 5.6, 1H), 3.86 (s, 3H), 1.68 (s, 4H), 1.29 (s, 6H), 1.23 (s, 6H); ¹³C NMR (100.6 MHz, CDCl₃) δ 165.0, 161.9, 159.6, 145.8, 143.3, 140.5, 133.2, 127.3, 124.4, 123.5, 117.0, 113.4, 54.2, 51.8, 34.9, 34.8, 34.3, 34.0, 31.7; ES-MS (M+H)⁺ calcd for C₂₃H₂₉N₃O₂ 380.2338, found 380.2329.

2-(Allyl(5,6,8,8-tetramethyl-5,6,7,8-tetrahydronaphthalen-2-yl)amino)pyrimidine-5-carboxylic acid (35). To a 100 mL round-bottomed flask equipped with a stir bar and charged with methyl ester **72** (0.8218 g, 2.166 mmol) suspended in methanol (7.6 mL) was added a solution of potassium hydroxide (0.4129 g, 7.36 mmol) in water (0.48 mL). This reaction was stirred at reflux in an oil bath at 87 °C for 1.5h. The reaction was then cooled to r.t. and acidified with 1N HCl (85 mL). The resulting precipitate was filtered and washed with copious amounts of cold water to give a crude product that was purified by column chromatography (25 mL SiO₂, 15% to 60% ethyl acetate:hexanes) to give pure **35** (0.7013 g, 88.6%) as a white crystalline solid, m.p. 212.0 – 213.7 °C: ¹H NMR (400 MHz, d₆-DMSO) δ 12.92 (br s, 1H), 8.75 (s, 2H), 7.32 (d, J = 8.4, 1H), 7.23 (d, J = 2.4, 1H), 7.04 (dd, J = 8.4, 2.0, 1H), 5.99-5.89 (dddd, J = 17.2, 10.4, 5.6, 4.8, 1H), 5.16-5.11 (dd, J = 17.2, 1.6, 1H), 5.13-5.10 (dd, J = 10.4, 1.6, 1H), 4.58 (d, J = 5.2, 2H), 1.62 (s, 4H), 1.23 (s, 6H), 1.18 (s, 6H); ¹³C NMR (100.6 MHz, d₆-DMSO) δ 165.5, 162.1, 159.5, 145.2, 142.2, 140.6, 133.8, 126.9, 124.6, 124.5, 116.7, 114.0, 53.2, 34.5, 34.4, 34.0, 33.7, 31.6, 31.5; ES-MS (M+Na)⁺ calcd for C₂₂H₂₇N₃O₂Na 388.2001, found 388.2011.



Scheme 10.

Methyl 6-((1,1,4,4,7-pentamethylisochroman-6-yl)amino)nicotinate (75). To a solution of **46** (2.2436 g, 7.922 mmol), **73** (1.1979 g, 7.873 mmol), Cs_2CO_3 (6.2394 g, 19.24 mmol), *rac*-BINAP (0.3781 g, 0.6072 mmol) in toluene (9.0 mL) in a 100 mL round-bottomed flask was added $\text{Pd}_2(\text{dba})_3$ (0.3541 g, 0.3867 mmol). The solution was sparged with nitrogen for 5 min., then a reflux condenser was fitted to the flask, the atmosphere was evacuated and back-filled with nitrogen (three times), and the reaction was heated to reflux with stirring in an oil bath (125-120 °C) for 22h. After cooling the reaction to room temperature, excess cesium carbonate and other solid particulates were filtered and washed with ethyl acetate, and the organic filtrate was concentrated in vacuo to give a crude product that was purified by column chromatography (150 mL SiO_2 , 8% ethyl acetate:hexanes to 15% ethyl acetate: hexanes) to give **75** (1.5387 g, 55.1%) as a crystalline solid, m.p. 186.3-188.8 °C: ^1H NMR (400 MHz, CDCl_3) δ 8.77 (dd, J = 2.2, 0.6, 1H), 8.03 (dd, J = 8.8, 2.2, 1H), 7.39 (br s, 1H), 7.26 (s, 1H), 6.96 (s, 1H), 6.51 (dd, J = 8.8, 0.6, 1H), 3.87 (s, 3H), 3.59 (s, 2H), 2.21 (s, 3H), 1.53 (s, 6H), 1.24 (s, 6H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 165.8, 159.3, 141.6, 139.7, 139.3, 134.6, 130.7, 128.0, 121.7, 116.3, 106.0, 74.9, 70.6, 51.8, 33.7, 29.7, 26.9, 17.7; ES-MS ($\text{M}+\text{H}$) $^+$ calcd for $\text{C}_{21}\text{H}_{27}\text{N}_2\text{O}_3$ 355.2022, found 355.2012.

Methyl 2-((1,1,4,4,7-pentamethylisochroman-6-yl)amino)pyrimidine-5-carboxylate (76). To a solution of **46** (2.2669 g, 8.004 mmol), **74** (1.2081 g, 7.889 mmol), Cs_2CO_3 (6.2373 g, 19.14 mmol), *rac*-BINAP (0.3792 g, 0.609 mmol) in toluene (9.0 mL) in a 100 mL round-bottomed flask was added $\text{Pd}_2(\text{dba})_3$ (0.3553 g, 0.388 mmol). The solution was sparged with nitrogen for 5 min., then a reflux condenser was fitted to the flask, the atmosphere was evacuated and back-filled with nitrogen (three times), and the reaction was heated to reflux with stirring in an oil bath (125-120 °C) for 22h. After cooling the reaction to room temperature, excess cesium carbonate and other solid particulates were filtered and washed with ethyl acetate, and the organic filtrate was concentrated in vacuo to give a crude product that was purified by column chromatography (150 mL SiO_2 , 5% to 15% to 20% to 30% ethyl acetate: hexanes) to give **76** (1.1300 g, 40.3%) as a crystalline solid, m.p. 130.7-135.8 °C: ^1H NMR (400 MHz, CDCl_3) δ 8.93 (s, 2H), 7.68 (s, 1H), 7.57 (br s, 1H), 6.93 (s, 1H), 3.90 (s, 3H), 3.59 (s, 2H), 2.26 (s, 3H), 1.53 (s, 6H), 1.27 (s, 6H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 164.6, 161.6, 160.1, 141.0, 139.0, 133.9, 128.6, 127.6, 120.6, 114.8, 74.9, 70.7, 52.0, 33.8, 29.7, 26.9, 17.9; ES-MS ($\text{M}+\text{Na}$) $^+$ calcd for $\text{C}_{20}\text{H}_{25}\text{N}_3\text{O}_3\text{Na}$ 378.1794, found 378.1805.

Methyl 6-(methyl(1,1,4,4,7-pentamethylisochroman-6-yl)amino)nicotinate (77). To a flame-dried, 100 mL round-bottomed flask equipped with a magnetic stir bar was added a 60% dispersion of sodium hydride in mineral oil (0.4325 g, 10.81 mmol). The dispersion of sodium hydride was washed with hexanes (3.8 mL, twice) and dried under vacuum and suspended in 5.8 mL of DMF under nitrogen. To this solution of sodium hydride in DMF was added a solution of **75** (1.5942 g, 4.4978 mmol) in DMF (17.3 mL), and the reaction was stirred for 15 min., and then methyl iodide (0.43 mL, 6.9 mmol) was added, and the reaction was stirred for 1 h. The reaction was poured into water and extracted with ethyl acetate. The combined organic layers were washed with brine, dried over sodium sulfate, filtered, and

concentrated in vacuo to yield a crude product that was purified by column chromatography (150 mL SiO₂, 10% ethyl acetate:hexanes) to give **77** (1.5788 g, 95.26%) as a white crystalline solid, m.p. 166.1-168.3 °C: ¹H NMR (400 MHz, CDCl₃) δ 8.87 (d, J = 2.0, 1H), 7.84 (dd, J = 8.8, 2.0, 1H), 7.06 (s, 1H), 6.99 (s, 1H), 6.04 (d, J = 8.8, 1H), 3.85 (s, 3H), 3.58 (s, 2H), 3.47 (s, 3H), 2.06 (s, 3H), 1.55 (s, 6H), 1.22 (s, 6H); ¹³C NMR (100.6 MHz, CDCl₃) δ 166.2, 142.6, 141.4, 141.3, 137.9, 133.0, 128.4, 124.5, 114.4, 107.1, 75.0, 70.6, 51.6, 38.1, 33.8, 29.7, 27.0, 17.2; ES-MS (M+H)⁺ calcd for C₂₂H₂₉N₂O₃ 369.2178, found 369.2169.

Methyl 2-(methyl(1,1,4,4,7-pentamethylisochroman-6-yl)amino)pyrimidine-5-carboxylate (78). To a flame-dried, 100 mL round-bottomed flask equipped with a magnetic stir bar was added a 60% dispersion of sodium hydride in mineral oil (0.2960 g, 7.401 mmol). The dispersion of sodium hydride was washed with hexanes (3.0 mL, twice) and dried under vacuum and suspended in 3.8 mL of DMF under nitrogen. To this solution of sodium hydride in DMF was added a solution of **76** (1.0752 g, 3.025 mmol) in DMF (11.0 mL), and the reaction was stirred for 15 min., and then methyl iodide (0.28 mL, 4.5 mmol) was added, and the reaction was stirred for 1 h. The reaction was poured into water and extracted with ethyl acetate. The combined organic layers were washed with brine, dried over sodium sulfate, filtered, and concentrated in vacuo to yield a crude product that was purified by column chromatography (150 mL SiO₂, 8.5% to 10% ethyl acetate:hexanes) to give **78** (0.8375 g, 74.9%) as a white crystalline solid, m.p. 216.9-217.8 °C: ¹H NMR (400 MHz, CDCl₃) δ 8.95 (d, J = 2.0, 1H), 8.79 (d, J = 2.4, 1H), 7.05 (s, 1H), 6.96 (s, 1H), 3.86 (s, 3H), 3.57 (q, J = 11.6, 2H), 3.47 (s, 3H), 2.06 (s, 3H), 1.56 (s, 3H), 1.53 (s, 3H), 1.24 (s, 3H), 1.22 (s, 3H); ¹³C NMR (100.6 MHz, CDCl₃) δ 165.2, 162.5, 159.9, 159.7, 141.8, 141.4, 140.7, 132.3, 128.0, 123.6, 112.9, 75.0, 70.7, 51.7, 38.8, 33.7, 30.3, 29.3, 27.4, 26.5, 17.5; ES-MS (M+H)⁺ calcd for C₂₁H₂₈N₃O₃ 370.2131, found 370.2126.

Methyl 6-(ethyl)1,1,4,4,7-pentamethylisochroman-6-yl)amino)nicotinate (79). To a flame-dried, 100 mL round-bottomed flask equipped with a magnetic stir bar was added a 60% dispersion of sodium hydride in mineral oil (0.4132 g, 10.33 mmol). The dispersion of sodium hydride was washed with hexanes (3.0 mL, twice) and dried under vacuum and suspended in 5.7 mL of DMF under nitrogen. To this solution of sodium hydride in DMF was added a solution of **75** (1.5064 g, 4.2501 mmol) in DMF (16.8 mL), and the reaction was stirred for 15 min., and then ethyl iodide (0.54 mL, 6.7 mmol) was added, and the reaction was stirred for 1 h. The reaction was poured into water and extracted with ethyl acetate. The combined organic layers were washed with brine, dried over sodium sulfate, filtered, and concentrated in vacuo to yield a crude product that was purified by column chromatography (150 mL SiO₂, 6% ethyl acetate:hexanes) to give **79** (1.2146 g, 74.7%) as a white crystalline solid, m.p. 106.3-108.3 °C: ¹H NMR (400 MHz, CDCl₃) δ 8.85 (d, J = 2.0, 1H), 7.82 (dd, J = 8.8, 2.0, 1H), 7.01 (s, 1H), 7.00 (s, 1H), 5.94 (d, J = 8.8, 1H), 4.25 (m, 1H), 3.84 (s, 3H), 3.69 (m, 1H), 3.59 (s, 2H), 2.05 (s, 3H), 1.56 (s, 3H), 1.54 (s, 3H), 1.24 (t, J = 7.2, 3H), 1.23 (s, 3H), 1.21 (s, 3H); ¹³C NMR (100.6 MHz, CDCl₃) δ 166.2, 159.4, 150.3, 142.4, 141.4, 139.6, 138.1, 133.4, 128.4, 125.6, 114.3, 107.0, 75.0, 70.6, 51.6, 33.7, 29.8, 27.0, 17.3, 12.9; ES-MS (M+H)⁺ calcd for C₂₃H₃₁N₂O₃ 383.2335, found 383.2338.

Methyl 2-(ethyl(1,1,4,4,7-pentamethylisochroman-6-yl)amino)pyrimidine-5-carboxylate (80). To a flame-dried, 100 mL round-bottomed flask equipped with a magnetic stir bar was added a 60% dispersion of sodium hydride in mineral oil (0.2848 g, 7.12 mmol). The dispersion of sodium hydride was washed with hexanes (3.0 mL, twice) and dried under vacuum and suspended in 3.8 mL of DMF under nitrogen. To this solution of sodium hydride in DMF was added a solution of **76** (1.0788 g, 3.035 mmol) in DMF (11.0 mL), and the reaction was stirred for 15 min., and then ethyl iodide (0.36 mL, 4.5 mmol) was added, and the reaction was stirred for 1 h. The reaction was poured into water and extracted with ethyl acetate. The combined organic layers were washed with brine, dried over sodium sulfate, filtered, and concentrated in vacuo to yield a crude product that was purified by column chromatography (150 mL SiO₂, 8.5% to 10% ethyl acetate:hexanes) to give **80** (0.4435 g, 38.1%) as a white crystalline solid, m.p. 172.1 - 173.5 °C: ¹H NMR (400 MHz, CDCl₃) δ 8.94 (s, 1H), 8.78 (s, 1H), 7.01 (s, 1H), 6.97 (s, 1H), 4.18-4.09 (sext, J = 7.2, 1H), 3.86 (s, 3H), 3.82-3.74 (sext, J = 7.2, 1H), 3.62-3.54 (q, J = 11.6, 2H), 2.05 (s, 3H), 1.57 (s, 3H), 1.53 (s, 3H), 1.26 (t, J = 3.6, 3H), 1.25 (s, 3H), 1.22 (s, 3H); ¹³C NMR (100.6 MHz, CDCl₃) δ 165.3, 162.0, 160.0,

159.8, 141.5, 140.6, 139.8, 132.7, 128.0, 124.6, 112.8, 75.0, 70.7, 51.7, 46.0, 33.7, 30.2, 29.4, 27.5, 26.5, 17.6, 12.7; ES-MS (M+H)⁺ calcd for C₂₂H₃₀N₃O₃ 384.2287, found 384.2274.

Methyl 6-(allyl(1,1,4,4,7-pentamethylisochroman-6-yl)amino)nicotinate (81). To a flame-dried, 100 mL round-bottomed flask equipped with a magnetic stir bar was added a 60% dispersion of sodium hydride in mineral oil (0.4580 g, 4.543 mmol). The dispersion of sodium hydride was washed with hexanes (3.0 mL, twice) and dried under vacuum and suspended in 3.8 mL of DMF under nitrogen. To this solution of sodium hydride in DMF was added a solution of **75** (1.6104 g, 11.45 mmol) in DMF (5.8 mL), and the reaction was stirred for 15 min., and then allyl bromide (0.67 mL, 7.7 mmol) was added, and the reaction was stirred for 1 h. The reaction was poured into water and extracted with ethyl acetate. The combined organic layers were washed with brine, dried over sodium sulfate, filtered, and concentrated in vacuo to yield a crude product that was purified by column chromatography (150 mL SiO₂, 6% ethyl acetate:hexanes) to give **81** (1.4000 g, 78.1%) as a dull yellow crystalline solid, m.p. 92.7 - 94.9 °C: ¹H NMR (400 MHz, CDCl₃) δ 8.86 (d, J = 2.0, 1H), 7.86 (dd, J = 8.8, 1.6, 1H), 7.02 (s, 1H), 6.98 (s, 1H), 6.08-5.98 (m, 2H), 5.20-5.15 (m, 2H), 4.94 (m, 1H), 4.12 (m, 1H), 3.85 (s, 3H), 3.57 (s, 2H), 2.05 (s, 3H), 1.54 (s, 6H), 1.20 (s, 6H); ¹³C NMR (100.6 MHz, CDCl₃) δ 166.0, 158.7, 149.6, 142.3, 141.6, 139.5, 138.5, 133.1, 132.9, 128.4, 125.5, 118.0, 114.8, 107.5, 74.9, 70.6, 53.1, 51.7, 33.7, 29.7, 26.9, 17.4; ES-MS (M+Na)⁺ calcd for C₂₄H₃₀N₂O₃Na 417.2154, found 417.2154.

Methyl 2-(allyl(1,1,4,4,7-pentamethylisochroman-6-yl)amino)pyrimidine-5-carboxylate (82). To a flame-dried, 100 mL round-bottomed flask equipped with a magnetic stir bar was added a 60% dispersion of sodium hydride in mineral oil (0.2269 g, 5.673 mmol). The dispersion of sodium hydride was washed with hexanes (3.0 mL, twice) and dried under vacuum and suspended in 2.8 mL of DMF under nitrogen. To this solution of sodium hydride in DMF was added a solution of **76** (0.8014 g, 2.255 mmol) in DMF (8.1 mL), and the reaction was stirred for 15 min., and then allyl bromide (0.33 mL, 3.8 mmol) was added, and the reaction was stirred for 1 h. The reaction was poured into water and extracted with ethyl acetate. The combined organic layers were washed with brine, dried over sodium sulfate, filtered, and concentrated in vacuo to yield a crude product that was purified by column chromatography (150 mL SiO₂, 6% ethyl acetate:hexanes) to give **82** (0.4061 g, 45.5%) as a crystalline solid, m.p. 167.1 - 172.0 °C: ¹H NMR (400 MHz, CDCl₃) δ 9.01 (s, 1H), 8.88 (s, 1H), 7.02 (s, 1H), 6.98 (s, 1H), 6.06-5.96 (dddd, J = 6.8, 10.4, 14.8, 17.2, 1H), 5.26-5.21 (dd, J = 18.0, 1.2, 1H), 5.24-5.21 (dd, J = 10.4, 1.2, 1H), 4.23-4.18 (dd, J = 6.8, 14.8, 1H), 3.88 (s, 3H), 3.63-3.60 (d, J = 11.2, 1H), 3.55-3.52 (d, J = 11.6, 1H), 2.05 (s, 3H), 1.56 (s, 3H), 1.53 (s, 3H), 1.22 (s, 6H); ¹³C NMR (100.6 MHz, CDCl₃) δ 164.4, 160.1, 160.0, 159.1, 141.7, 141.4, 139.0, 132.3, 132.0, 128.1, 124.4, 118.9, 113.3, 75.0, 70.6, 54.2, 33.7, 30.1, 29.4, 27.4, 26.4, 17.6; ES-MS (M+Na)⁺ calcd for C₂₃H₂₉N₃O₃Na 418.2107, found 418.2101.

6-(Methyl(1,1,4,4,7-pentamethylisochroman-6-yl)amino)nicotinic acid (36). To a 100 mL round-bottomed flask equipped with a stir bar and charged with methyl ester **77** (1.0403 g, 2.823 mmol) suspended in methanol (8.1 mL) was added a solution of potassium hydroxide (0.5223 g, 9.309 mmol) in water (0.52 mL). This reaction was stirred at reflux in an oil bath at 87 °C for 1.5h. The reaction was then cooled to r.t. and acidified with 1N HCl (90 mL). The solution was extracted with ethyl acetate (2 X 70 mL), and the organic layers were dried over sodium sulfate and concentrated in vacuo to give a crude product that was purified by column chromatography (25 mL SiO₂, 30% ethyl acetate:hexanes to 60% ethyl acetate:hexanes) to give pure **36** (0.3879 g, 38.7%) as a white crystalline solid, m.p. 294.5-296.9 °C: ¹H NMR (400 MHz, d₆-DMSO) δ 12.50 (br s, 1H), 8.68 (s, 1H), 7.82 (d, J = 7.6, 1H), 7.19 (d, J = 2.0, 2H), 6.00 (br s, 1H), 3.49 (s, 2H), 3.35 (s, 3H), 1.99 (s, 3H), 1.47 (s, 3H), 1.16 (s, 6H); ¹³C NMR (100.6 MHz, d₆-DMSO) δ 166.7, 159.9, 150.6, 142.2, 141.5, 140.8, 137.9, 132.7, 128.3, 124.4, 114.7, 106.0, 74.5, 69.7, 37.6, 33.4, 29.6, 26.7, 16.9; ES-MS (M+Na)⁺ calcd for C₂₁H₂₆N₂O₃Na 377.1814, found 377.1830.

2-(Methyl(1,1,4,4,7-pentamethylisochroman-6-yl)amino)pyrimidine-5-carboxylic acid (37). To a 100 mL round-bottomed flask equipped with a stir bar and charged with methyl ester **78** (0.7964 g, 2.156 mmol) suspended in methanol (7.0 mL) was added a solution of potassium hydroxide (0.4088 g, 7.286 mmol) in water (0.47 mL). This reaction was stirred at reflux in an oil bath at 87 °C for 1.5h. The reaction was then

cooled to r.t. and acidified with 1N HCl (80 mL). The solution was extracted with ethyl acetate (2 X 70 mL), and the organic layers were dried over sodium sulfate and concentrated in vacuo to give a crude product that was purified by column chromatography (25 mL SiO₂, 60% ethyl acetate:hexanes to pure ethyl acetate to 2% methanol:ethyl acetate) to give pure **37** (0.6112 g, 79.7%) as a white crystalline solid, m.p. 332.0-334.0 °C: ¹H NMR (400 MHz, d₆-DMSO) δ 12.88 (br s, 1H), 8.87 (s, 1H), 8.66 (s, 1H), 7.19 (s, 1H), 7.11 (s, 1H), 3.48 (s, 2H), 3.41 (s, 3H), 1.96 (s, 3H), 1.48 (s, 3H), 1.45 (s, 3H), 1.17 (s, 3H), 1.15 (s, 3H); ¹³C NMR (100.6 MHz, d₆-DMSO) δ 165.6, 162.1, 159.7, 159.6, 141.5, 141.4, 140.2, 132.4, 127.6, 123.6, 113.4, 74.5, 69.8, 33.4, 29.7, 29.5, 26.9, 26.5, 17.1; ES-MS (M+H)⁺ calcd for C₂₀H₂₆N₃O₃ 356.1974, found 356.1962.

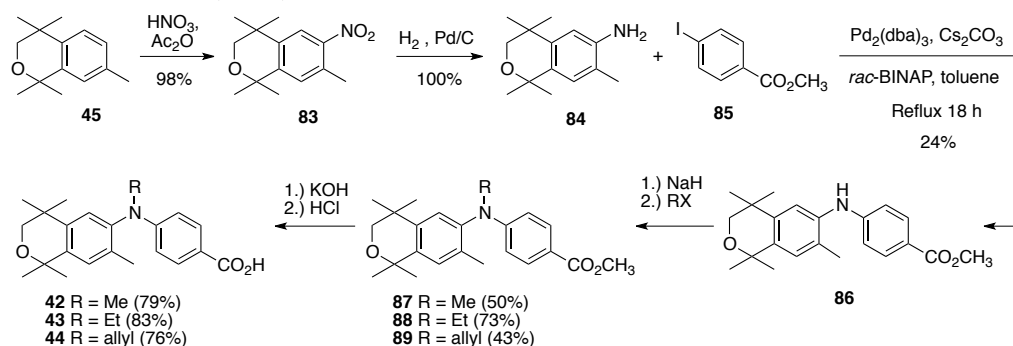
6-(Ethyl(1,1,4,4,7-pentamethylisochroman-6-yl)amino)nicotinic acid (38). To a 100 mL round-bottomed flask equipped with a stir bar and charged with methyl ester **79** (1.0918 g, 2.854 mmol) suspended in methanol (8.2 mL) was added a solution of potassium hydroxide (0.5468 g, 9.656 mmol) in water (0.52 mL). This reaction was stirred at reflux in an oil bath at 87 °C for 1.5h. The reaction was then cooled to r.t. and acidified with 1N HCl (80 mL). The solution was extracted with ethyl acetate (2 X 70 mL), and the organic layers were dried over sodium sulfate and concentrated in vacuo to give a crude product that was purified by column chromatography (25 mL SiO₂, 30% ethyl acetate:hexanes to 60% ethyl acetate:hexanes) to give pure **38** (0.7156 g, 68.0%) as a white crystalline solid, m.p. 280.4-282.1 °C: ¹H NMR (400 MHz, d₆-DMSO) δ 12.48 (br s, 1H), 8.67 (d, J = 2.0, 1H), 7.80 (dd, J = 8.8, 2.0, 1H), 7.21 (s, 1H), 7.12 (s, 1H), 5.91 (br s, 1H), 4.11 (m, 1H), 3.66 (m, 1H), 3.50 (s, 2H), 1.99 (s, 3H), 1.48 (s, 3H), 1.47 (s, 3H), 1.17 (s, 3H), 1.13 (t, J = 7.2, 3H); ¹³C NMR (100.6 MHz, d₆-DMSO) δ 166.7, 159.4, 150.7, 142.0, 140.9, 139.7, 138.1, 133.1, 128.3, 125.4, 114.6, 106.0, 74.5, 69.7, 44.2, 33.4, 29.6, 26.7, 17.0, 12.8; ES-MS (M+H)⁺ calcd for C₂₂H₂₉N₂O₃ 369.2178, found 369.2167.

2-(Ethyl(1,1,4,4,7-pentamethylisochroman-6-yl)amino)pyrimidine-5-carboxylic acid (39). To a 100 mL round-bottomed flask equipped with a stir bar and charged with methyl ester **80** (0.4017 g, 1.048 mmol) suspended in methanol (4.0 mL) was added a solution of potassium hydroxide (0.2080 g, 3.707 mmol) in water (0.24 mL). This reaction was stirred at reflux in an oil bath at 87 °C for 1.5h. The reaction was then cooled to r.t. and acidified with 1N HCl (80 mL). The solution was extracted with ethyl acetate (2 X 70 mL), and the organic layers were dried over sodium sulfate and concentrated in vacuo to give a crude product that was purified by column chromatography (25 mL SiO₂, 40% ethyl acetate:hexanes) to give pure **39** (0.2931 g, 75.7%) as a white crystalline solid, m.p. 249.0-250.3 °C: ¹H NMR (400 MHz, d₆-DMSO) δ 12.86 (br s, 1H), 8.86 (s, 1H), 8.65 (d, J = 2.0, 1H), 7.12 (s, 1H), 7.11 (s, 1H), 4.06-3.98 (sext, J = 6.8, 1H), 3.83-3.74 (sext, J = 6.8, 1H), 3.51 (d, J = 11.6, 1H), 3.48 (d, J = 11.2, 1H), 1.96 (s, 3H), 1.48 (s, 3H), 1.43 (s, 3H), 1.18 (s, 3H), 1.15 (t, J = 6.8, 3H), 1.15 (s, 3H); ¹³C NMR (100.6 MHz, d₆-DMSO) δ 165.6, 161.7, 159.7, 141.1, 140.2, 139.9, 132.8, 127.6, 124.4, 113.4, 74.5, 69.8, 45.4, 33.3, 29.7, 29.6, 26.8, 26.6, 17.3, 12.6; ES-MS (M+H)⁺ calcd for C₂₁H₂₈N₃O₃ 370.2131, found 370.2122.

6-(Allyl(1,1,4,4,7-pentamethylisochroman-6-yl)amino)nicotinic acid (40). To a 100 mL round-bottomed flask equipped with a stir bar and charged with methyl ester **81** (1.1035 g, 2.7972 mmol) suspended in methanol (8.0 mL) was added a solution of potassium hydroxide (0.5389 g, 9.604 mmol) in water (0.61 mL). This reaction was stirred at reflux in an oil bath at 87 °C for 1.5h. The reaction was then cooled to r.t. and acidified with 1N HCl (90 mL). The solution was extracted with ethyl acetate (2 X 70 mL), and the organic layers were dried over sodium sulfate and concentrated in vacuo to give a crude product that was purified by column chromatography (25 mL SiO₂, 30% to 60% ethyl acetate:hexanes) to give pure **40** (0.9411 g, 88.4%) as a white crystalline solid, m.p. 221.3-224.4 °C: ¹H NMR (400 MHz, d₆-DMSO) δ 12.52 (br s, 1H), 8.67 (d, J = 1.6, 1H), 7.84 (dd, J = 9.2, 1.6, 1H), 7.19 (s, 1H), 7.12 (s, 1H), 6.02-5.92 (dddd, J = 16.4, 10.4, 6.0, 6.0, 1H), 5.96-5.94 (d, J = 10.0, 1H), 5.16-5.11 (dd, J = 16.8, 1.6, 1H), 5.12-5.10 (dd, J = 10.0, 1.6, 1H), 4.74 (m, 1H), 4.18 (m, 1H), 3.49 (s, 2H), 1.99 (s, 3H), 1.44 (s, 6H), 1.13 (s, 6H); ¹³C NMR (100.6 MHz, d₆-DMSO) δ 166.6, 159.4, 150.6, 141.8, 139.8, 138.2, 134.1, 133.1, 128.2, 125.3, 117.4, 115.0, 106.1, 74.5, 69.7, 52.2, 33.3, 29.6, 26.6, 17.1; ES-MS (M+H)⁺ calcd for C₂₃H₂₉N₂O₃ 381.2178, found 381.2176.

2-(Allyl(1,1,4,4,7-pentamethylisochroman-6-yl)amino)pyrimidine-5-carboxylic acid (41). To a 100 mL round-bottomed flask equipped with a stir bar and charged with methyl ester **82** (0.3600 g, 0.9034 mmol)

suspended in methanol (3.5 mL) was added a solution of potassium hydroxide (0.1967 g, 3.506 mmol) in water (0.24 mL). This reaction was stirred at reflux in an oil bath at 87 °C for 1.5h. The reaction was then cooled to r.t. and acidified with 1N HCl (80 mL) to give a precipitate. The precipitate was filtered and washed with copious amounts of cold water to give a crude product that was purified by column chromatography (25 mL SiO₂, 40% ethyl acetate:hexanes) to give pure **41** (0.2366 g, 68.1%) as a white crystalline solid, m.p. 203.1-205.1 °C: ¹H NMR (400 MHz, d₆-DMSO) δ 12.90 (br s, 1H), 8.87 (s, 1H), 8.68 (s, 1H), 7.10 (s, 2H), 6.03-5.93 (dddd, J = 16.8, 10.8, 6.4, 6.0, 1H), 5.17-5.12 (dd, J = 16.8, 1.6, 1H), 5.15-5.12 (dd, J = 10.4, 1.2, 1H), 4.73-4.68 (dd, J = 15.2, 6.0, 1H), 4.28-4.23 (dd, J = 15.2, 6.8, 1H), 3.51 (d, J = 11.6, 1H), 3.47 (d, J = 11.6, 1H), 1.99 (s, 3H), 1.49 (s, 3H), 1.46 (s, 3H), 1.12 (s, 6H); ¹³C NMR (100.6 MHz, d₆-DMSO) δ 165.5, 161.8, 159.8, 140.9, 140.2, 140.0, 133.5, 132.8, 127.5, 124.4, 117.9, 113.8, 74.5, 69.7, 53.1, 33.3, 29.7, 29.6, 26.7, 26.6, 17.4; ES-MS (M+H)⁺ calcd for C₂₂H₂₈N₃O₃ 382.2131, found 382.2127.



Scheme 11.

1,1,4,4,7-Pentamethyl-6-nitroisochroman (83). To a 100 mL round bottom flask equipped with a stir bar and containing 1,1,4,4,7-pentamethylisochroman (**45**) (6.0185 g, 29.458 mmol) dissolved in acetic anhydride (30.0 mL) cooled to 0 °C in an ice bath was added concentrated nitric acid (2.40 mL, 57.6 mmol), dropwise. After stirring at 0 °C for 20 min, the solution was poured into water (50 mL) and extracted with ethyl acetate, and the combined organic layers were dried over sodium sulfate, concentrated in vacuo to give a crude oil that was purified by column chromatography (150 mL SiO₂, hexanes to 10% ethyl acetate:hexanes) to give pure **83** (7.2334 g, 98.5%) as a dull yellow crystalline solid, m.p. 89.4-91.3 °C: ¹H NMR (400 MHz, CDCl₃) δ 7.93 (s, 1H), 6.99 (s, 1H), 3.58 (s, 2H), 2.56 (s, 3H), 1.53 (s, 6H), 1.28 (s, 6H); ¹³C NMR (100.6 MHz, CDCl₃) δ 147.4, 147.3, 142.0, 130.9, 129.7, 121.9, 75.0, 70.4, 33.9, 29.4, 26.7, 20.5; ES-MS (M+Na)⁺ calcd for C₁₄H₁₉NO₃Na 272.1263, found 272.1252.

1,1,4,4,7-pentamethylisochroman-6-amine (84). To a 300 mL round bottom flask equipped with a stir bar and containing 1,1,4,4,7-pentamethyl-6-nitroisochroman (**83**) (3.04 g, 12.2 mmol) dissolved in ethyl acetate (20.0 mL) was added 10% Pd/C (0.5975 g). The flask was fitted with a three-way glass stopcock to which one side was connected to a balloon containing hydrogen gas, and the other side was connected to high vacuum. The reaction flask was evacuated and back-filled with hydrogen gas three times and then allowed to stir overnight at room temperature. After stirring overnight, the solution was filtered through cotton to give **84** (2.67 g, 100%) as a dull yellow crystalline solid, m.p. 117.4-123.5 °C: ¹H NMR (400 MHz, CDCl₃) δ 6.82 (s, 1H), 6.79 (s, 1H), 5.37 (br s, 2H), 3.56 (s, 2H), 2.23 (s, 3H), 1.49 (s, 6H), 1.23 (s, 6H); ¹³C NMR (100.6 MHz, CDCl₃) δ 141.4, 139.1, 134.4, 127.5, 122.5, 113.2, 74.8, 70.8, 33.6, 29.8, 26.9, 17.3; ES-MS (M+H)⁺ calcd for C₁₄H₂₂NO 220.1701, found 220.1698.

Methyl 4-((1,1,4,4,7-pentamethylisochroman-6-yl)amino)benzoate (86). To a solution of **84** (5.3327 g, 24.314 mmol), 4-iodo-methylbenzoate **85** (6.5862 g, 25.13 mmol), Cs₂CO₃ (9.7904 g, 30.05 mmol), rac-BINAP (1.2082 g, 1.9404 mmol) in toluene (28.0 mL) in a 250 mL round-bottomed flask was added Pd₂(dba)₃ (1.1442 g, 1.2495 mmol). The solution was sparged with nitrogen for 5 min., then a reflux condenser was fitted to the flask, the atmosphere was evacuated and back-filled with nitrogen (three times), and the reaction was heated to reflux with stirring in an oil bath (125-120 °C) for 22h. After cooling the reaction to room temperature, excess cesium carbonate and other solid particulates were

filtered and washed with ethyl acetate, and the organic filtrate was concentrated in vacuo to give a crude product that was purified by column chromatography (150 mL SiO₂, 5% ethyl acetate:hexanes to 8% ethyl acetate: hexanes) to give **86** (2.0851 g, 24.2%) as a crystalline solid, m.p. 152.3-172.6 °C: ¹H NMR (400 MHz, CDCl₃) δ 7.91 (d, J = 8.4, 2H), 7.68 (br s, 1H), 7.25 (s, 1H), 6.93 (s, 1H), 6.83 (d, J = 8.8, 2H), 3.86 (s, 3H), 3.58 (s, 2H), 2.20 (s, 3H), 1.54 (s, 6H), 1.22 (s, 6H); ES-MS (M+Na)⁺ calcd for C₂₂H₂₇NO₃Na 376.1889, found 376.1888.

Methyl 4-(methyl(1,1,4,4,7-pentamethylisochroman-6-yl)amino)benzoate (87). To a flame-dried, 100 mL round-bottomed flask equipped with a magnetic stir bar was added a 60% dispersion of sodium hydride in mineral oil (0.1994 g, 4.985 mmol). The dispersion of sodium hydride was washed with hexanes (3.0 mL, twice) and dried under vacuum and suspended in 3.0 mL of DMF under nitrogen. To this solution of sodium hydride in DMF was added a solution of **86** (0.6378 g, 1.8045 mmol) in DMF (6.0 mL), and the reaction was stirred for 15 min., and then methyl iodide (0.17 mL, 2.7 mmol) was added, and the reaction was stirred for 1 h. The reaction was poured into water and extracted with ethyl acetate. The combined organic layers were washed with brine, dried over sodium sulfate, filtered, and concentrated in vacuo to yield a crude product that was purified by column chromatography (150 mL SiO₂, 2% to 5% ethyl acetate:hexanes) to give **87** (0.3366 g, 50.76%) as a waxy red crystalline solid: ¹H NMR (400 MHz, CDCl₃) δ 7.85 (d, J = 9.2, 1H), 7.04 (s, 1H), 6.97 (s, 1H), 6.47 (d, J = 8.8, 1H), 3.84 (s, 3H), 3.59 (s, 2H), 3.27 (s, 3H), 2.05 (s, 3H), 1.56 (s, 6H), 1.22 (s, 6H); ¹³C NMR (100.6 MHz, CDCl₃) δ 167.3, 152.4, 143.3, 142.2, 140.5, 133.4, 131.1, 128.2, 124.7, 117.5, 111.2, 75.0, 70.7, 51.4, 39.1, 33.7, 29.8, 27.0, 17.4; ES-MS (M+H)⁺ calcd for C₂₃H₃₀NO₃ 368.2226, found 368.2217.

Methyl 4-(ethyl(1,1,4,4,7-pentamethylisochroman-6-yl)amino)benzoate (88). To a flame-dried, 100 mL round-bottomed flask equipped with a magnetic stir bar was added a 60% dispersion of sodium hydride in mineral oil (0.2025 g, 5.063 mmol). The dispersion of sodium hydride was washed with hexanes (3.0 mL, twice) and dried under vacuum and suspended in 2.6 mL of DMF under nitrogen. To this solution of sodium hydride in DMF was added a solution of **86** (0.7119 g, 2.0141 mmol) in DMF (7.9 mL), and the reaction was stirred for 15 min., and then ethyl iodide (0.25 mL, 3.1 mmol) was added, and the reaction was stirred for 1 h. The reaction was poured into water and extracted with ethyl acetate. The combined organic layers were washed with brine, dried over sodium sulfate, filtered, and concentrated in vacuo to yield a crude product that was purified by column chromatography (150 mL SiO₂, 5% ethyl acetate:hexanes) to give **88** (0.4162 g, 73.4%) as a white crystalline solid, m.p. 95.5-98.4 °C: ¹H NMR (400 MHz, CDCl₃) δ 7.82 (d, J = 9.2, 1H), 7.01 (s, 1H), 6.98 (s, 1H), 6.43 (d, J = 8.8, 1H), 3.83 (s, 3H), 3.66 (m, 2H), 3.59 (s, 2H), 2.04 (s, 3H), 1.56 (s, 6H), 1.25 (t, J = 7.2, 3H), 1.22 (s, 6H); ¹³C NMR (100.6 MHz, CDCl₃) δ 167.3, 151.5, 142.1, 141.7, 140.6, 133.8, 131.3, 128.5, 117.2, 111.0, 75.0, 70.7, 51.4, 45.9, 33.7, 29.8, 26.9, 17.5, 12.5; ES-MS (M+Na)⁺ calcd for C₂₄H₃₁NO₃Na 404.2202, found 404.2201.

Methyl 4-(allyl(1,1,4,4,7-pentamethylisochroman-6-yl)amino)benzoate (89). To a flame-dried, 100 mL round-bottomed flask equipped with a magnetic stir bar was added a 60% dispersion of sodium hydride in mineral oil (0.1983 g, 4.958 mmol). The dispersion of sodium hydride was washed with hexanes (3.0 mL, twice) and dried under vacuum and suspended in 2.0 mL of DMF under nitrogen. To this solution of sodium hydride in DMF was added a solution of **86** (0.6040 g, 1.709 mmol) in DMF (6.0 mL), and the reaction was stirred for 15 min., and then allyl bromide (0.26 mL, 3.0 mmol) was added, and the reaction was stirred for 1 h. The reaction was poured into water and extracted with ethyl acetate. The combined organic layers were washed with brine, dried over sodium sulfate, filtered, and concentrated in vacuo to yield a crude product that was purified by column chromatography (150 mL SiO₂, 5% ethyl acetate:hexanes) to give **89** (0.2935 g, 43.6%) as an oil: ¹H NMR (400 MHz, CDCl₃) δ 7.82 (d, J = 9.2, 1H), 7.05 (s, 1H), 6.97 (s, 1H), 6.46 (d, J = 8.8, 1H), 6.01-5.91 (dddd, J = 17.2, 10.4, 5.6, 5.2, 1H), 5.31-5.26 (dd, J = 17.2, 1.6, 1H), 5.25-5.22 (dd, J = 10.4, 1.6, 1H), 4.22 (m, 2H), 3.83 (s, 3H), 3.58 (s, 2H), 2.05 (s, 3H), 1.56 (s, 6H), 1.21 (s, 6H); ¹³C NMR (100.6 MHz, CDCl₃) δ 167.3, 151.7, 142.4, 142.1, 140.6, 133.5, 133.3, 131.1, 128.2, 125.5, 117.7, 111.6, 75.0, 70.7, 54.5, 51.5, 33.7, 29.8, 26.9, 17.7; ES-MS (M+Na)⁺ calcd for C₂₅H₃₁NO₃Na 416.2202, found 416.2201.

4-(Methyl(1,1,4,4,7-pentamethylisochroman-6-yl)amino)benzoic acid (42). To a 100 mL round-bottomed flask equipped with a stir bar and charged with methyl ester **87** (0.3023 g, 0.8226 mmol) suspended in methanol (3.0 mL) was added a solution of potassium hydroxide (0.1707 g, 3.042 mmol) in water (0.26 mL). This reaction was stirred at reflux in an oil bath at 87 °C for 1.5h. The reaction was then cooled to r.t. and acidified with 1N HCl (85 mL) to give a precipitate. The precipitate was filtered and washed with copious amounts of cold water to give a crude product that was purified by column chromatography (25 mL SiO₂, 30% to 50% ethyl acetate:hexanes) to give pure **42** (0.2323 g, 79.9%) as a white crystalline solid, m.p. 273.3-275.0 °C: ¹H NMR (400 MHz, d₆-DMSO) δ 12.16 (br s, 1H), 7.71 (d, J = 8.8, 2H), 7.16 (s, 1H), 7.12 (s, 1H), 6.43 (d, J = 8.4, 1H), 3.49 (s, 2H), 3.21 (s, 3H), 1.98 (s, 3H), 1.47 (s, 6H), 1.16 (s, 6H); ¹³C NMR (100.6 MHz, d₆-DMSO) δ 167.4, 151.9, 143.1, 142.0, 140.3, 132.9, 131.0, 128.2, 124.3, 117.8, 111.0, 74.5, 69.7, 33.4, 29.6, 26.7, 17.0; ES-MS (M+H)⁺ calcd for C₂₂H₂₇NO₃Na 376.1889, found 376.1893.

4-(Ethyl(1,1,4,4,7-pentamethylisochroman-6-yl)amino)benzoic acid (43). To a 100 mL round-bottomed flask equipped with a stir bar and charged with methyl ester **88** (0.3178 g, 0.8330 mmol) suspended in methanol (2.6 mL) was added a solution of potassium hydroxide (0.1644 g, 2.93 mmol) in water (0.26 mL). This reaction was stirred at reflux in an oil bath at 87 °C for 1.5h. The reaction was then cooled to r.t. and acidified with 1N HCl (85 mL) to give a precipitate. The precipitate was filtered and washed with copious amounts of cold water to give a crude product that was purified by column chromatography (25 mL SiO₂, 30% to 50% ethyl acetate:hexanes) to give pure **43** (0.2562 g, 83.7%) as a white crystalline solid, m.p. 283.9-285.4 °C: ¹H NMR (400 MHz, d₆-DMSO) δ 12.12 (br s, 1H), 7.69 (d, J = 9.2, 2H), 7.18 (s, 1H), 7.07 (s, 1H), 6.40 (d, J = 8.8, 1H), 3.63 (br s, 2H), 3.50 (s, 2H), 1.98 (s, 3H), 1.47 (s, 6H), 1.16 (s, 6H), 1.14 (t, J = 7.2, 3H); ¹³C NMR (100.6 MHz, d₆-DMSO) δ 167.4, 151.1, 141.9, 141.3, 140.4, 133.4, 131.2, 128.3, 125.5, 117.4, 110.8, 74.5, 69.7, 45.6, 33.3, 29.6, 26.7, 17.1, 12.3; ES-MS (M+H)⁺ calcd for C₂₃H₃₀NO₃ 368.2226, found 368.2209.

4-(Allyl(1,1,4,4,7-pentamethylisochroman-6-yl)amino)benzoic acid (44). To a 100 mL round-bottomed flask equipped with a stir bar and charged with methyl ester **89** (0.2611 g, 0.6635 mmol) suspended in methanol (2.6 mL) was added a solution of potassium hydroxide (0.1927 g, 3.43 mmol) in water (0.26 mL). This reaction was stirred at reflux in an oil bath at 87 °C for 1.5h. The reaction was then cooled to r.t. and acidified with 1N HCl (90 mL) to give a precipitate. The precipitate was filtered and washed with copious amounts of cold water to give a crude product that was purified by column chromatography (25 mL SiO₂, 30% to 50% ethyl acetate:hexanes) to give pure **44** (0.1936 g, 76.9%) as a white crystalline solid, m.p. 244.5-246.5 °C: ¹H NMR (400 MHz, d₆-DMSO) δ 12.17 (br s, 1H), 7.69 (d, J = 8.8, 2H), 7.18 (s, 1H), 7.11 (s, 1H), 6.42 (d, J = 8.8, 1H), 5.99-5.90 (dddd, J = 16.8, 10.4, 5.2, 5.2, 1H), 5.31-5.26 (d, J = 17.2, 1.6, 1H), 5.21-5.18 (d, J = 10.4, 1.6, 1H), 4.23 (br s, 2H), 3.49 (s, 2H), 1.99 (s, 3H), 1.47 (s, 6H), 1.15 (s, 6H); ¹³C NMR (100.6 MHz, d₆-DMSO) δ 167.3, 151.2, 142.0, 141.8, 140.4, 133.8, 133.1, 128.3, 125.1, 118.0, 117.0, 111.4, 74.5, 69.7, 54.1, 33.3, 29.6, 26.7, 17.3; ES-MS (M+H)⁺ calcd for C₂₄H₃₀NO₃ 380.2226, found 380.2225.

References

1. Tachdjian, C.G., J.; Boudjelal, M.; Al-Shamma, H.A.; Giachino, A. F.; Jakubowicz-Jaillardon, K.; Chen, Q.; Zapf, J.W.; Pfahl, M., *Preparation of substituted isochroman compounds for the treatment of metabolic disorders, cancer and other diseases*, P.I. Appl, Editor. 2004.
2. Faul, M.M., et al., *Synthesis of Novel Retinoid X Receptor-Selective Retinoids*. J. Org. Chem., 2001. **66**(17): p. 5772-5782.
3. Jurutka, P.W., et al., *Modeling, Synthesis, and Biological Evaluation of Potential Retinoid X Receptor (RXR) Selective Agonists: Novel Analogues of 4-[1-(3,5,5,8,8-Pentamethyl-5,6,7,8-tetrahydro-2-naphthyl)ethynyl]benzoic Acid (Bexarotene) and (E)-3-(3-(1,2,3,4-tetrahydro-1,1,4,4,6-pentamethylnaphthalen-7-yl)-4-hydroxyphenyl)acrylic Acid (CD3254)*. J. Med. Chem., 2013. **56**(21): p. 8432-8454.

4. Heck, M.C., et al., *Modeling, Synthesis, and Biological Evaluation of Potential Retinoid X Receptor (RXR)-Selective Agonists: Analogues of 4-[1-(3,5,5,8,8-Pentamethyl-5,6,7,8-tetrahydro-2-naphthyl)ethynyl]benzoic Acid (Bexarotene) and 6-(Ethyl(5,5,8,8-tetrahydronaphthalen-2-yl)amino)nicotinic Acid (NEt-TMN)*. J. Med. Chem., 2016. **59**(19): p. 8924-8940.

File: C:\MASSLYNX\JULY 21.PRO\DATA\210826_CW-VI-171_POS.RAW

Printed: Friday, August 27, 2021 09:30:35 Pacific Daylight Time

Header

Acquired File Name: 210826_CW-VI-171_pos
Acquired Date: 26-Aug-2021
Acquired Time: 16:52:17
Job Code: July21
Task Code:
User Name:
Laboratory Name:
Instrument: ACQ-QDA#KAD3226
Conditions:
Submitter:
SampleID:
Bottle Number: 2:25
Description: mz not observed

Instrument Calibration:

Calibration File: C:\MassLynx\IntelliStart.pro\AcquDB\AutoCal.cal

Parameters**MS1 Static:**

Mass: 30 Da to 1250 Da.
Resolution: -0.1/-0.0
Ion Energy: -0.3
Reference File: Internal
Acquisition File:

MS1 Scanning:

Mass: 30 Da to 1250 Da.
Resolution: -0.1/-0.0
Ion Energy: -0.3
Reference File: Internal
Acquisition File:

MS1 Scan Speed Compensation:

Scan: 40 to 10000 amu/sec.
Resolution: -0.1/-0.0
Ion Energy: -0.3
Reference File: Internal
Acquisition File:

Calibration Time: 10:54

Calibration Date: 09/13/17

Coefficients

MS1 Static: $-0.000000000000x^4 + 0.000000000656x^3 + -0.000001077025x^2 + 1.000599988917x + -0.085640798905$
Function 1: $-0.000000000000x^4 + 0.000000000378x^3 + -0.000000745481x^2 + 1.000452684494x + -0.068499345191$

Acquisition Experiment Report**Page 2 of 10****File:** C:\MASSLYNX\JULY 21.PRO\DATA\210826_CW-VI-171_POS.RAW**Printed:** Friday, August 27, 2021 09:30:35 Pacific Daylight Time

Function 2: None

Parameters for C:\MassLynx\July 21.PRO\ACQUDB\POS_105-600_C_5mins.EXP

Prescan Statistics:

Initial Average Intensity	379.7540
Initial Average Std Dev	1.1398
Bunch Zero Level	0.0000
Bunch Std Dev	0.0000
Bunch Threshold	0.0000
Spike Removal Std Dev	1.1211
Ion Count Threshold:	25.0000

Data Processing:

Targeted Sampling Frequency	2
Actual Sampling Frequency	2.000
SIR Chromatogram Spike Removal	ON
SIR Smoothing	ON
Smoothing window size (scans)	3
Number of smooths	2

Method Events:

Initial Stop Flow:	No Change
Initial Switch 1:	No Change
Timed Events Enabled	

Event	Time(Sec)	Name	Action
-------	-----------	------	--------

Instrument Parameters - Function 1:

Polarity	ES+
Calibration	Dynamic 2
Capillary (kV)	1.50
Cone (V)	10.00
Source Temperature (°C)	120
Probe Temperature (°C)	450
Calibration Temperature (°C)	0

Engineers Settings:

File: C:\MASSLYNX\JULY 21.PRO\DATA\210826_CW-VI-171_POS.RAW

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LM 1 Resolution	0.00
HM 1 Resolution	0.00
Low Mass Ion Energy 1	-0.91
High Mass Ion Energy 1	0.14
Low Mass Position	0.69
High Mass Position	-1.81
Low Mass Setup	0.63
High Mass Setup	-6.13
Detector Gain Positive	1.0
Detector Gain Negative	1.0
Nominal Rod Polarity	Negative
Dynamic Offset Positive Settings:	
Dynamic Offset Low Mass Resolution	0.00
Dynamic Offset High Mass Resolution	0.00
Dynamic Offset Low Mass Ion Energy	0.00
Dynamic Offset High Mass Ion Energy	-0.02

Dynamic Offset Negative Settings:	
Dynamic Offset Low Mass Resolution	-0.01
Dynamic Offset High Mass Resolution	0.21
Dynamic Offset Low Mass Ion Energy	0.00
Dynamic Offset High Mass Ion Energy	0.19

Dynamic Offset Settings:	
Dynamic Offset Low Mass Position	-0.71
Dynamic Offset High Mass Position	-0.46
Dynamic Offset Low Mass Setup	0.55
Dynamic Offset High Mass Setup	-0.04
Dynamic Offset Linearity Gain	4.80

Set Detector Gain 1.0

Instrument Readbacks	
Capillary (kV)	1.50
Cone (V)	14.26
Source Temperature (°C)	120
Multiplier 375	
Probe Temperature (°C)	450
Calibration Temperature (°C)	29

File: C:\MASSLYNX\JULY 21.PRO\DATA\210826_CW-VI-171_POS.RAW

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Inter-scan delays:

Automatic Mode

MS Delay Table

	R	delay
<=	16.000	0.005
>	16.000	0.006

Health Check Failures:

All Health Checks Passed

Health Checks all enabled unless shown below:

Resolution Setup Required

Recalibration Required

Service Due

ACE Experimental Record

Inlet Method File: c:\masslynx\july 21.pro\acqddb\col4_pfp_fa_acn_5mins

----- Run method parameters -----

Waters ACQUITY QSM

Solvent A Name: Water

Solvent B Name: Methanol

Solvent C Name: 0.1% FA

Solvent D Name: Acetonitrile

Low Pressure Limit: 0 psi

High Pressure Limit: 15000 psi

Seal Wash Period: 5.00 min

[Gradient Table]

	Time(min)	Flow Rate(mL/min)	%A	%B	%C	%D	Curve
1.	Initial	0.500	0.0	0.0	95.0	5.0	Initial
2.	2.00	0.500	0.0	0.0	5.0	95.0	6
3.	3.50	0.500	0.0	0.0	95.0	5.0	11
4.	5.00	0.500	0.0	0.0	95.0	5.0	11

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Comment:

Flow Ramp Rate: 0.45 min
D Solvent Selection (if supported): No Change
System Pressure Data Channel: No
Flow Rate Data Channel: No
%A Data Channel: No
%B Data Channel: No
%C Data Channel: No
%D Data Channel: No
Primary Data Channel: No
Accumulator Data Channel: No
Degasser Data Channel: No
Gradient Start: At Injection
Gradient Start Volume: 0 uL
Gradient Start Time: 0.00 min
Participate in pre-analysis: No

Waters Acquity CM

Target Column Temperature: 50.0 C
Temperature Alarm Band: 5.0 C
: No
Column Valve Position: Column 4
Equilibration Time: 0.1 min
External Valve 1: No Change
External Valve 2: No Change
External Valve 3: No Change
Comment:
Column Temperature Data Channel: No
Preheater Temperature Data Channel: No

Waters Acquity PDA

File: C:\MASSLYNX\JULY 21.PRO\DATA\210826_CW-VI-171_POS.RAW

Printed: Friday, August 27, 2021 09:30:35 Pacific Daylight Time

Run Time: 5.00 min
PDA Detector Type: UPLC LG 500 nm
Lamp: On
Sampling Rate: 20 points/sec
Filter Time Constant: 0.2000 sec
Exposure Time: Auto msec
Interpolate 2nd order filter Region: No
Use UV Blocking Filter: Yes
3D Channel...
Range: 210 - 499
Resolution: 2.4 nm
Channel 1...
Data Mode: Absorbance at 214
Resolution: 2.4 nm
Channel 2...
Data Mode: Absorbance at 260
Resolution: 2.4 nm
Channel 3...
Data Mode: Absorbance at 350
Resolution: 2.4 nm
Initial Switch 1: No Change
Initial Switch 2: No Change

Waters ACQUITY FTN AutoSampler

Run Time: 5.00 min
Comment:
Load Ahead: Disabled
Loop Offline: Automatic min
Wash Solvent Name: Weak Wash
Pre-Inject Wash Time: 0.0 sec
Post-Inject Wash Time: 6.0 sec
Purge Solvent Name: Strong Wash
Dilution: Disabled
Dilution Volume: 0 uL
Delay Time: 0 min
Dilution Needle Placement: Automatic mm
Target Column Temperature: Off C

Acquisition Experiment Report

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File: C:\MASSLYNX\JULY 21.PRO\DATA\210826_CW-VI-171_POS.RAW

Printed: Friday, August 27, 2021 09:30:35 Pacific Daylight Time

Target Sample Temperature: 10.0 C
Sample Temperature Alarm Band: Disabled
Syringe Draw Rate: Automatic
Needle Placement: 2.0 mm
Pre-Aspirate Air Gap: Automatic
Post-Aspirate Air Gap: Automatic
Column Temperature Data Channel: No
Room Temperature Data Channel: No
Sample Temperature Data Channel: No
Sample Organizer Temperature Data Channel: No
Sample Pressure Data Channel: No
Preheater Temperature Data Channel: No
Seal Force Data Channel: No
No Injection Mode Enabled: No
Autoaddition Mix Stroke Cycles: Automatic
Autoaddition Mix Stroke Volume: Automatic uL
Run Events: Yes

Sample Run Injection Parameter

Injection Volume (ul) - 2.00

----- oOo -----

End of experimental record.

----- Waters ACQUITY QSM Postrun Report -----

Firmware Version: 1.60.260 (Jul 9 2013)
Software Version: 1.60.1897
Checksum: 0xa9c8e138
Serial Number: D17QSM962A
Minimum System Pressure: 2958.0 psi
Maximum System Pressure: 5886.0 psi
Average System Pressure: 4651.0 psi

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----- oOo -----

----- Waters ACQUITY FTN Postrun Report -----

Software Version: 1.60.1774
Firmware Version: 1.60.364 (Sep 20 2013)
Checksum: 0x35d5392b
Serial Number: D17SDI835G
Sample Syringe Size: 100.0
Extension Loop Size: 0.0
Needle Size: 30.0
Minimum Sample Temperature: 9.9
Maximum Sample Temperature: 10.2
Average Sample Temperature: 10.1
Minimum Column Temperature: -0.2
Maximum Column Temperature: 0.0
Average Column Temperature: -0.2

----- oOo -----

----- Waters Acquity CM Postrun Report -----

Software Version: 1.60.2072
Firmware Version: 1.65.142 (Apr 02 2015)
Checksum: 0x18bfb4ea
Serial Number: D17CMP604G
Valve Position: 4
Minimum Column Temperature: 50.0
Maximum Column Temperature: 50.0
Average Column Temperature: 50.0

----- oOo -----

Acquisition Experiment Report

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File: C:\MASSLYNX\JULY 21.PRO\DATA\210826_CW-VI-171_POS.RAW

Printed: Friday, August 27, 2021 09:30:35 Pacific Daylight Time

----- Active eCord Data -----

Valve Current Position: 4
Failed to retrieve Active eCord ColumnData Data

----- oOo -----

----- Generic Instrument Postrun Report -----

Software Version: 1.60.1390
Firmware Version: 1.60.6169 (Aug 13 2013)
Checksum: 0xdcfe9340
Serial Number: B17UPD132A
Lamp On/Off Event: No
Lamp Life: 3230.00 hours
Lamp Serial Number: 000133684
Exposure Time: 50.000 msec
Lambda1: 187.360
Lambda512: 502.647
Flow Cell Type: Analytical LG
Flow Cell Path Length: 10.000 mm
Flow Cell Volume: 0.500 microliters
Flow Cell Serial Number: PDA10-15625
Flow Cell Part Number: 205015017
Optics Temperature Stabilization Setting: Normal Temperature

----- oOo -----

-----Failed to get IECordHost2 Interface -----

Function 1

Scans in function: 601
Cycle time (secs): Automatic
Scan duration (secs): 0.495

Acquisition Experiment Report

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File: C:\MASSLYNX\JULY 21.PRO\DATA\210826_CW-VI-171_POS.RAW

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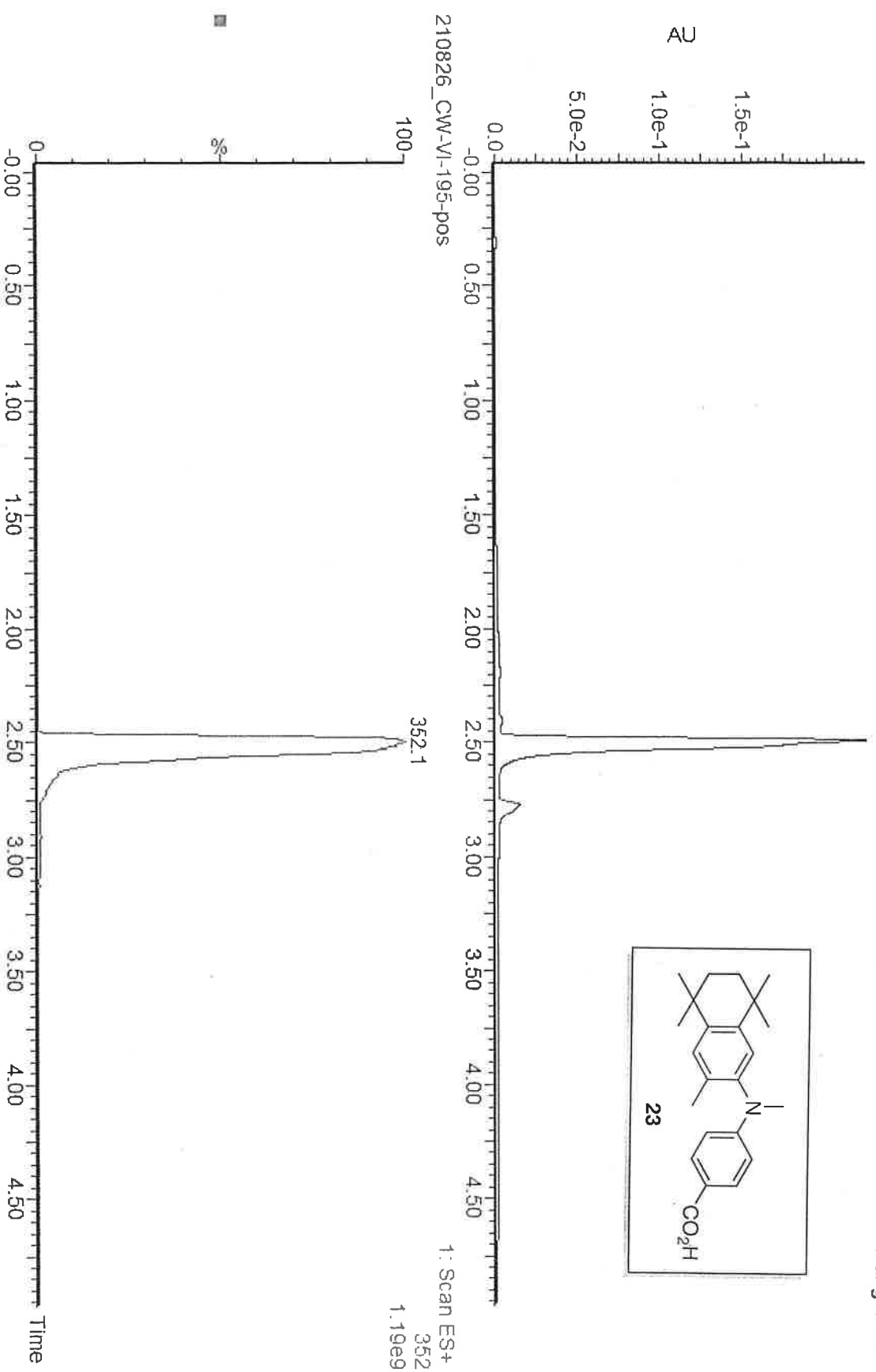
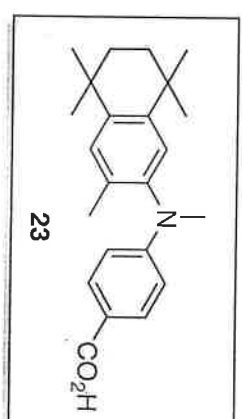
Inter Scan Delay (secs): Automatic
Start and End Time(mins): 0.000 to 5.000
Ionization mode: ES+
Data type: Accurate Mass
Function type: Scan
Mass range: 105 to 600

Function 2

Scans in function: 6001
Function type: Diode Array
Wavelength range (nm): 210 to 499

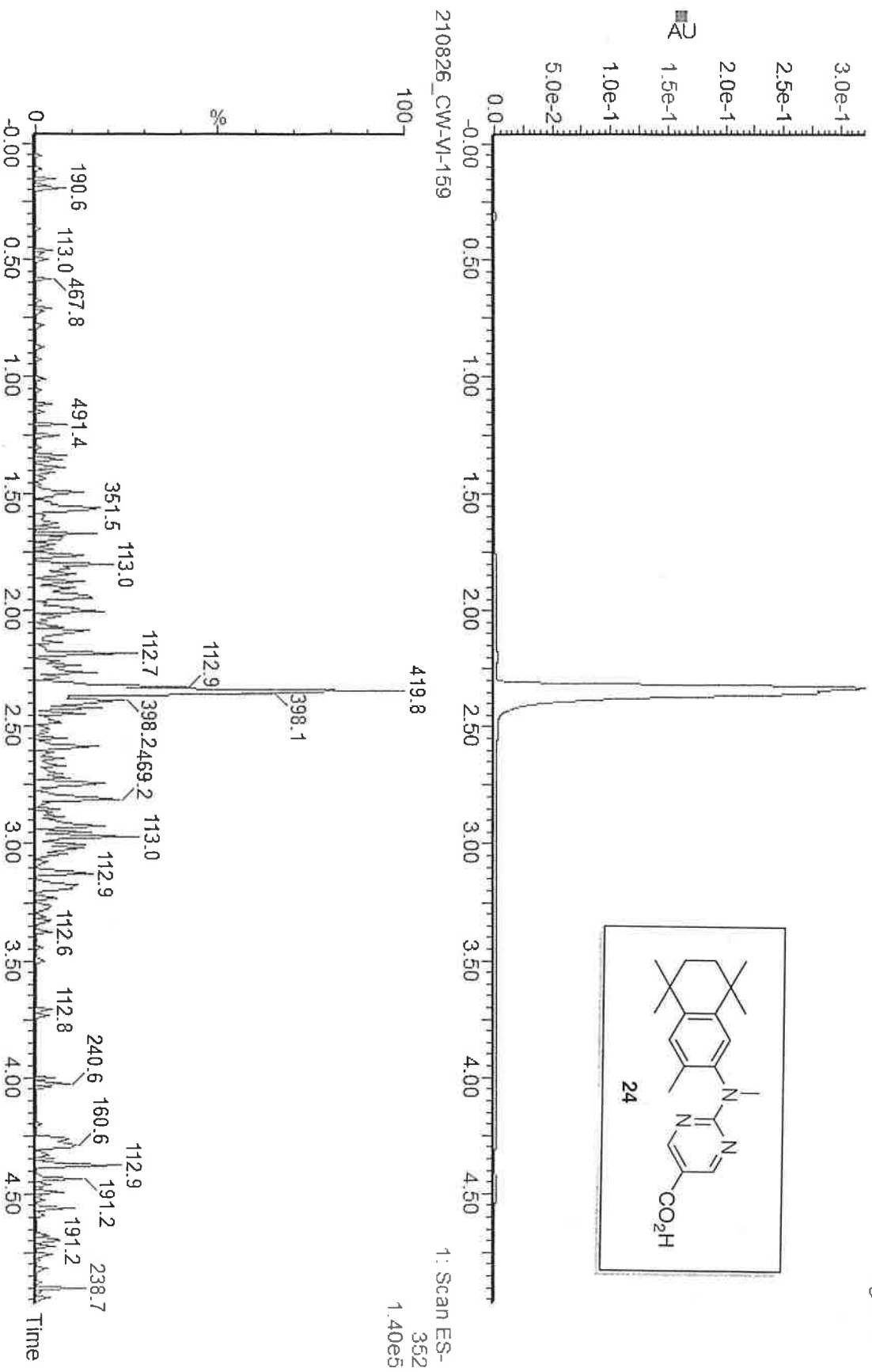
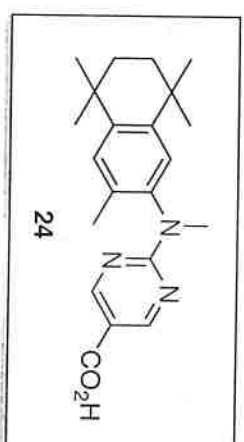
m/z not observed
210826_CW-VI-195-pos

(2) PDA Ch2 260nm@2.4nm
Range: 2e-1



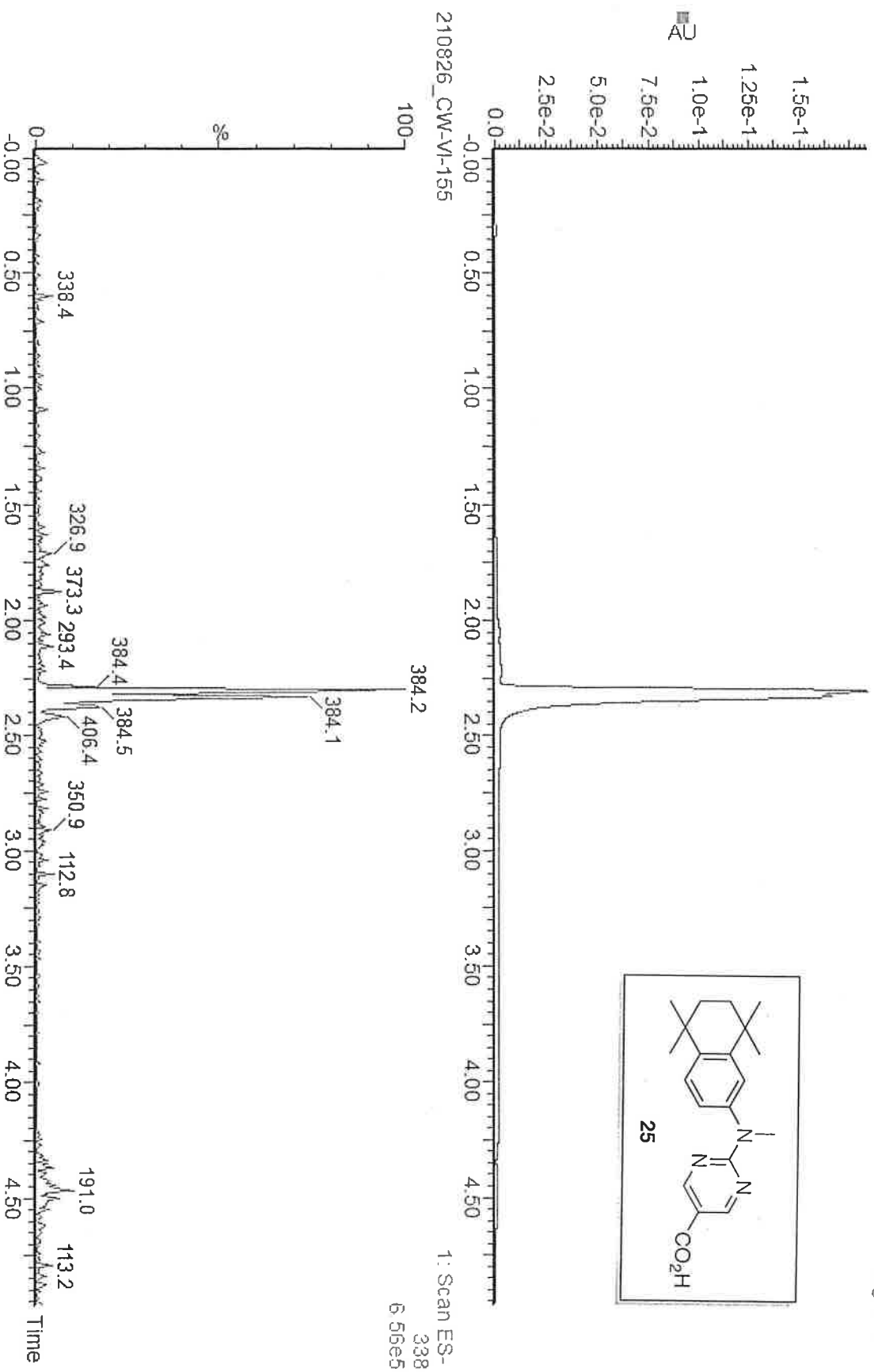
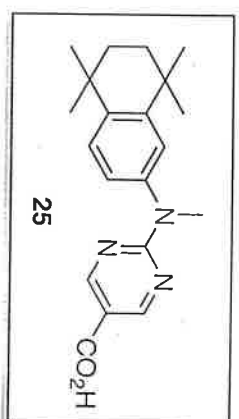
210826_CW-VI-159

(2) PDA Ch2 260nm@2.4nm
Range: 3e-1



210826_CW-VI-155

(2) PDA Ch2 260nm@2.4nm
Range: 2e-1



m/z not observed

210826_CW-VI-141_pos

(2) PDA Ch2 260nm@2.4nm
Range: 6e-1

AU

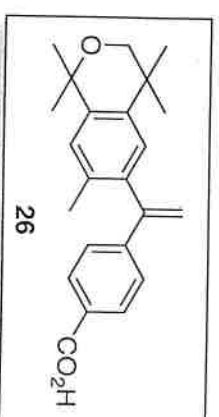
4.0e-1
3.0e-1
2.0e-1
1.0e-1

210826_CW-VI-141_pos

100

351.1

1: Scan ES+
351
6.51e7

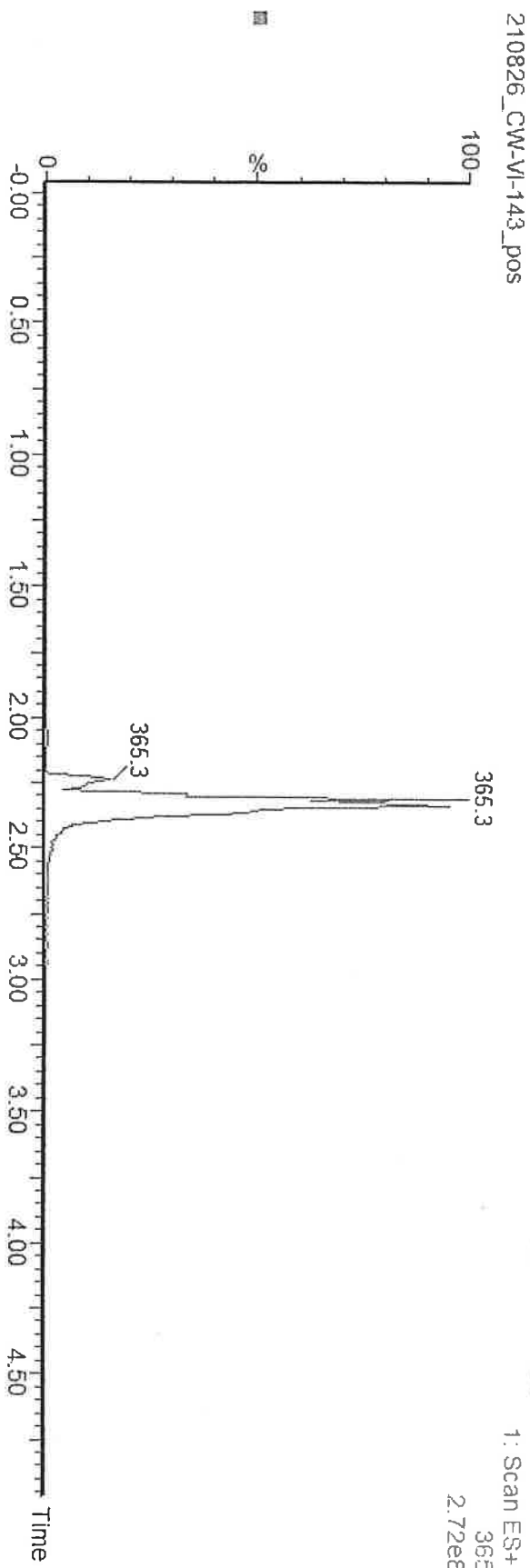
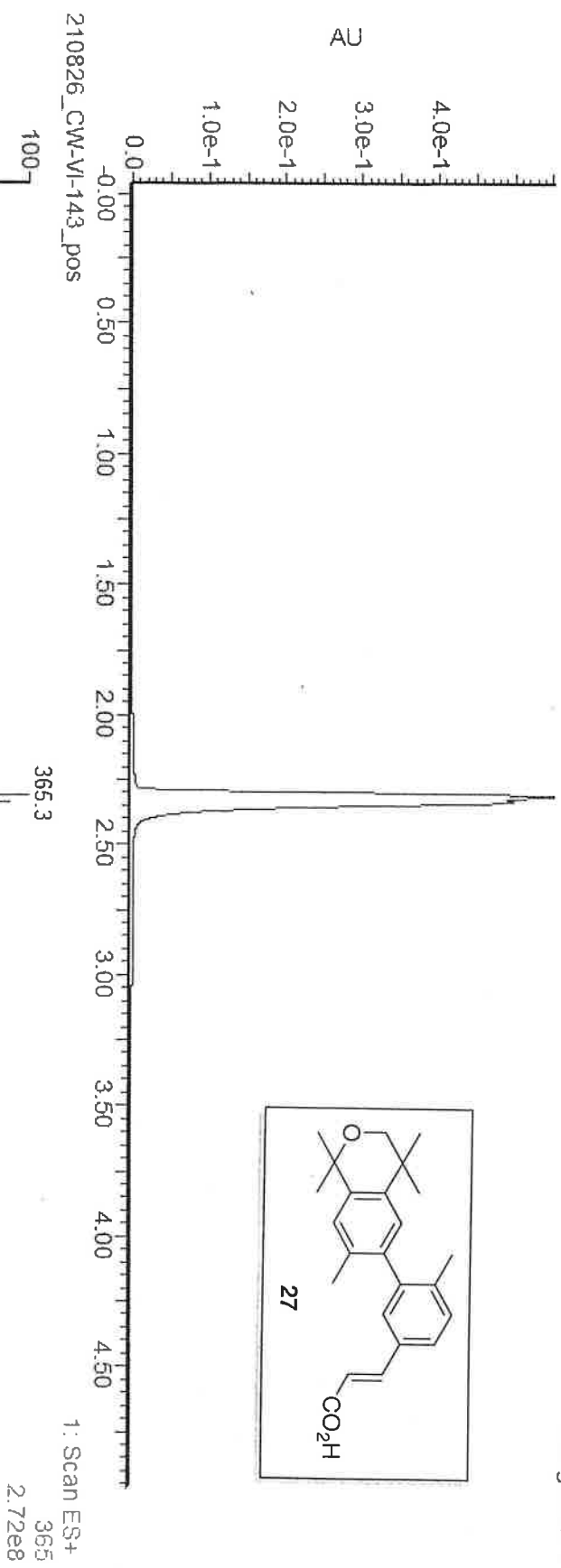
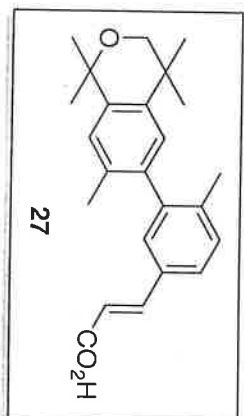


0 -0.00 0.50 1.00 1.50 2.00 2.50 3.00 3.50 4.00 4.50 Time

Br @ 489/491

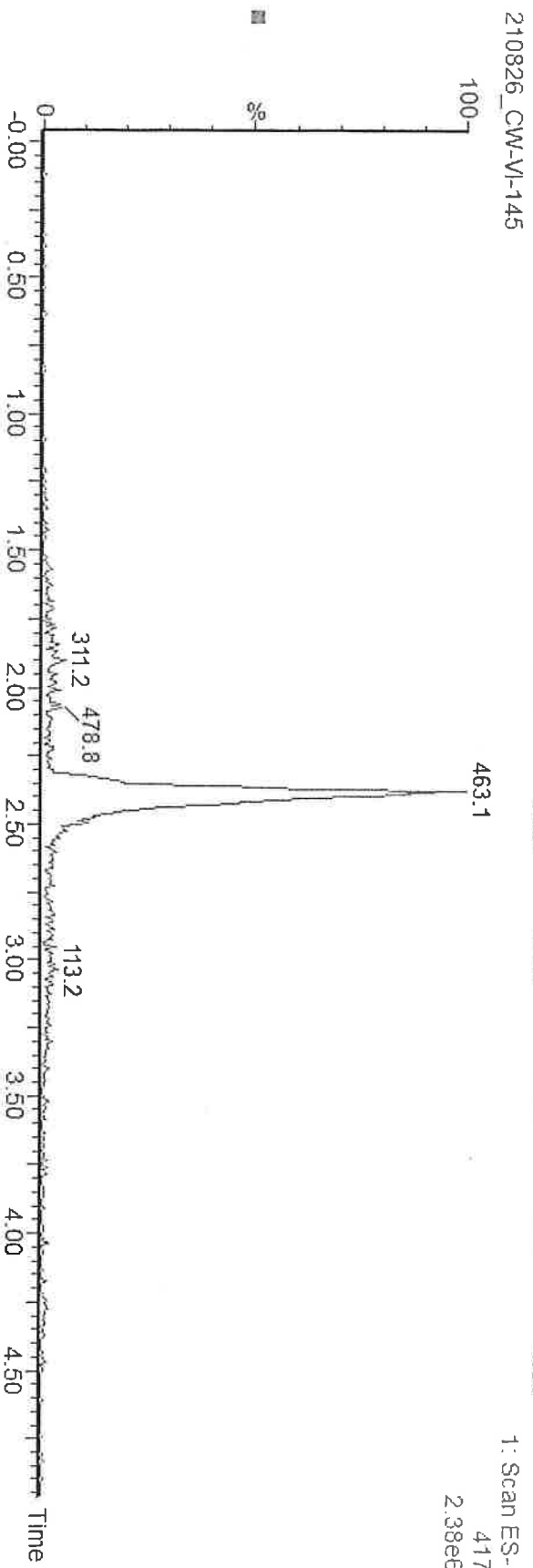
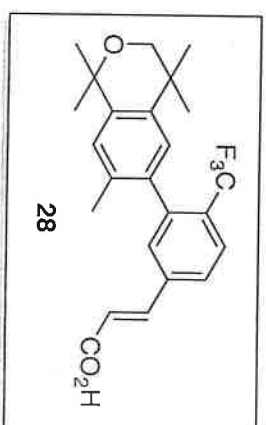
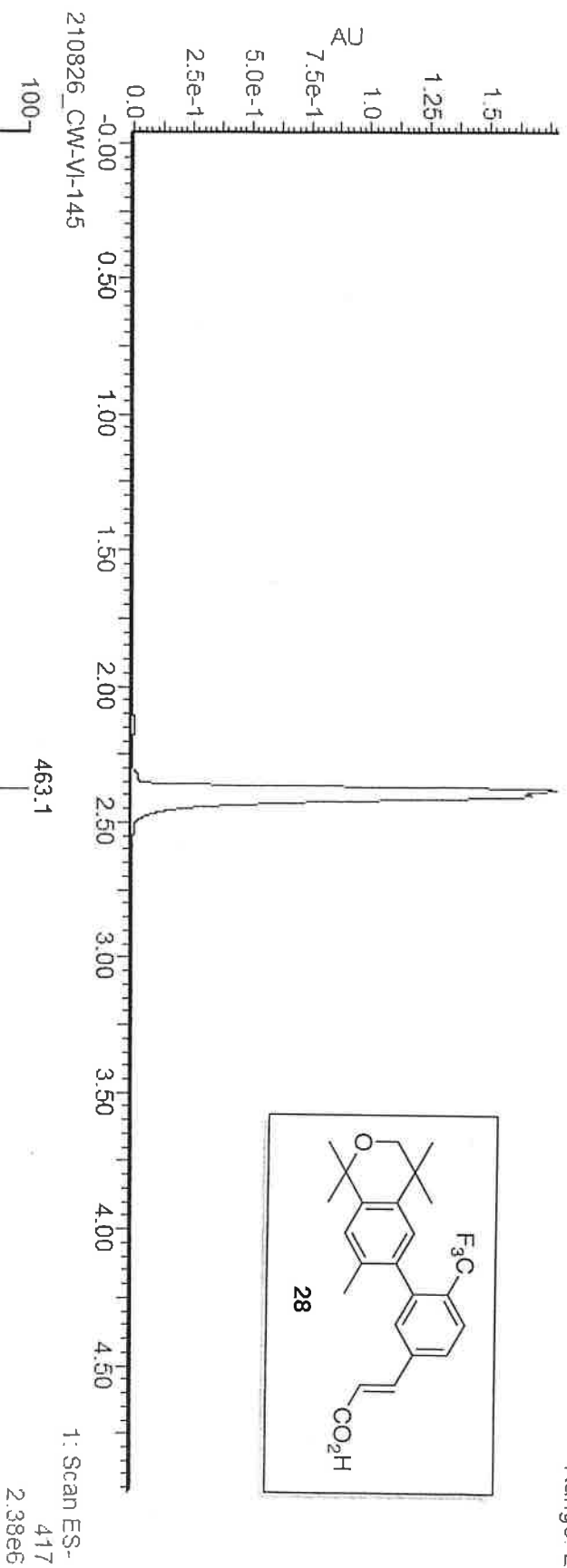
210826_CW-VI-143_pos

(2) PDA Ch2 260nm@2.4nm
Range: 6e-1



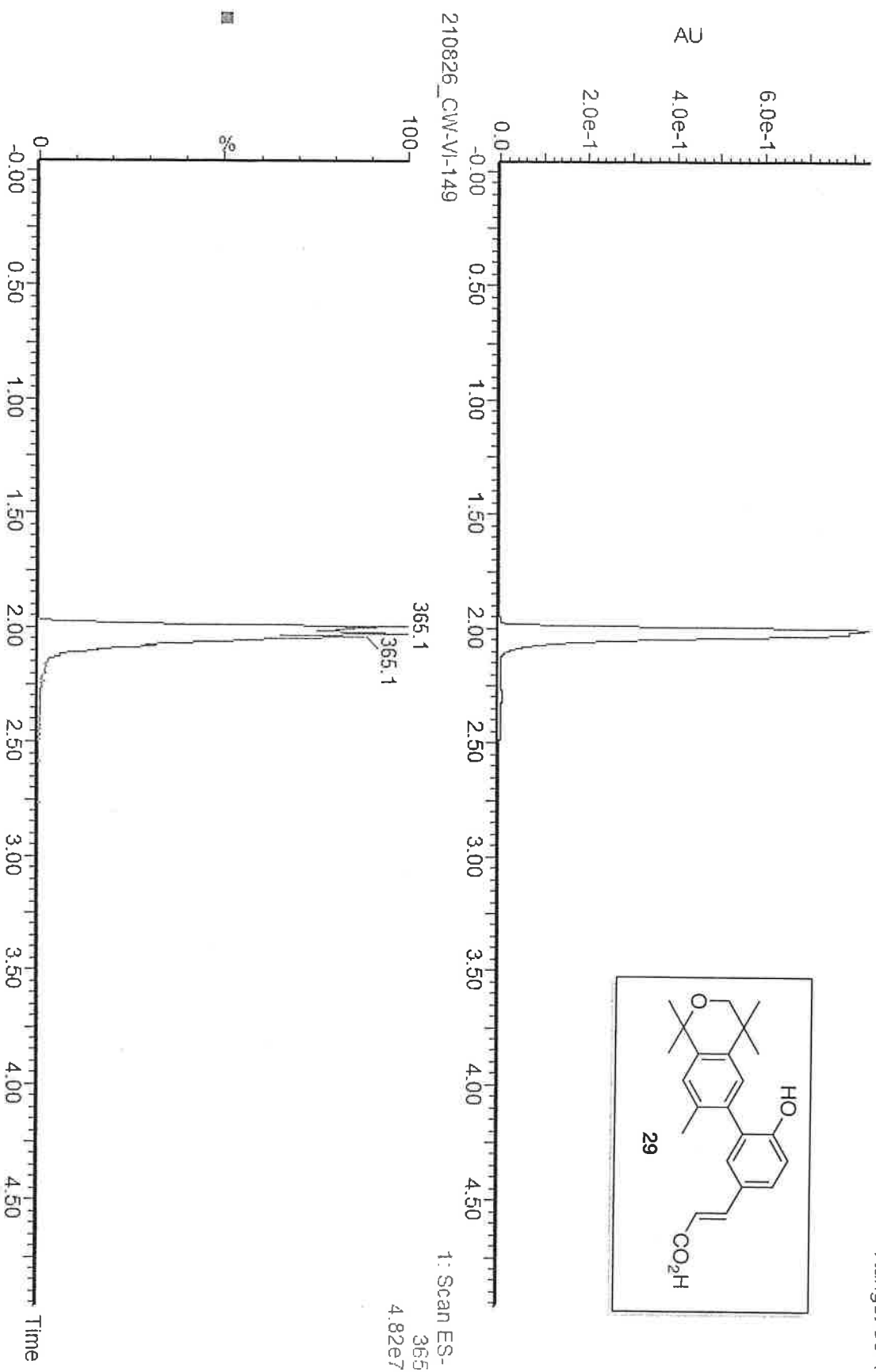
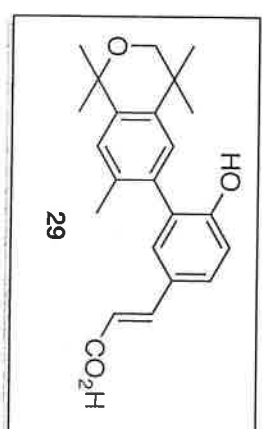
210826_CW-VI-145

(2) PDA Ch2 260nm@2.4nm
Range: 2

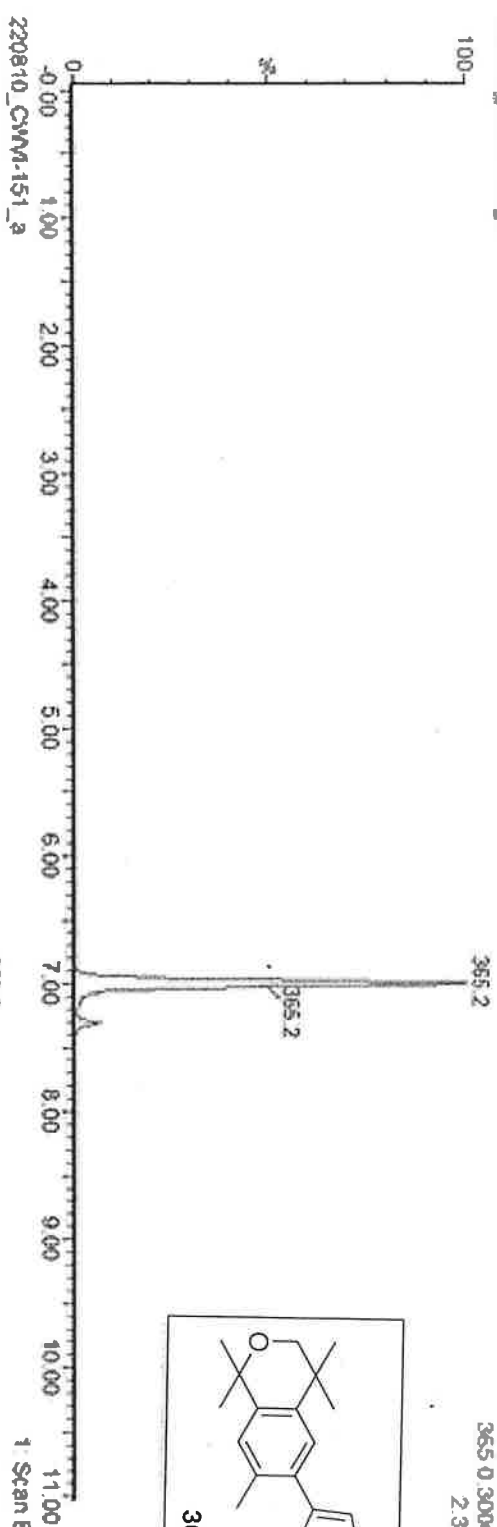


210826_CW-VI-149

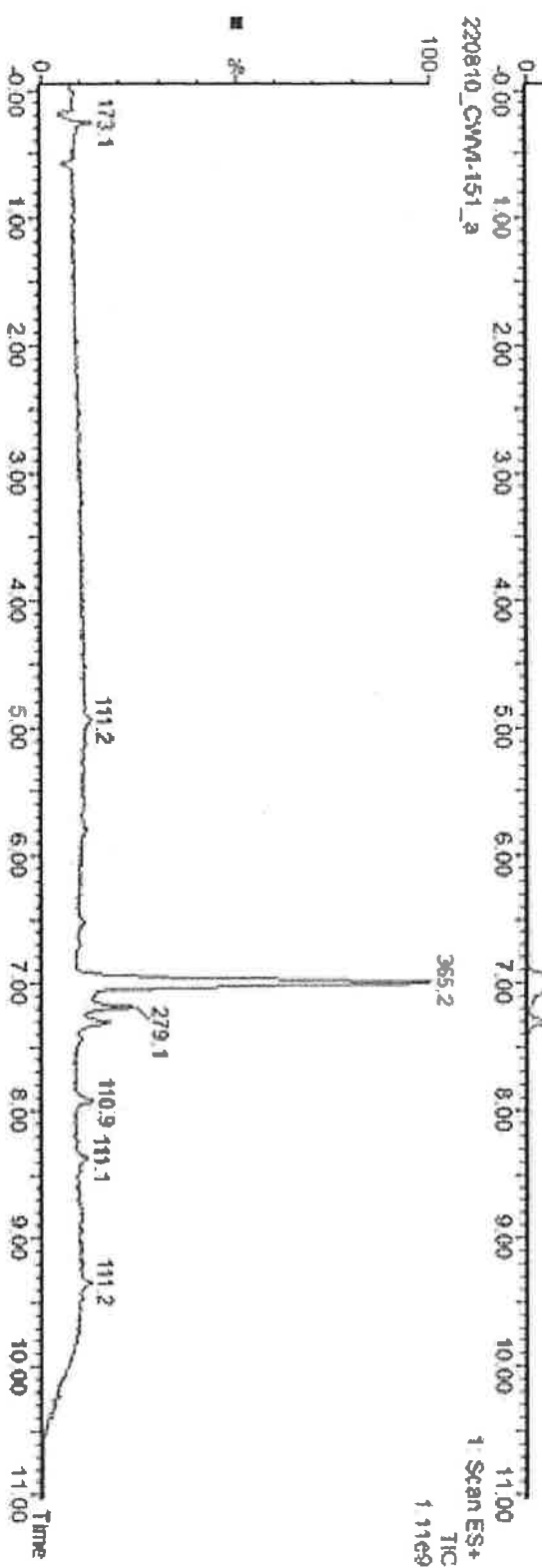
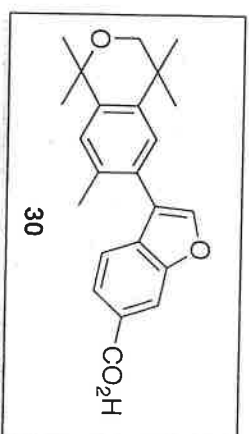
(2) PDA Ch2 260nm@2.4nm
Range: 8e-1



220810_CVM-151_a



1: Scan ES+
365.03000Da
2: 32e8



1: Scan ES+
TIC
1: 11e9

mz not observed

210826_CW-VI-153_pos

(2) PDA Ch2 260nm@2.4nm
Range: 3e-1

3.0e-1
2.5e-1
2.0e-1
1.5e-1
1.0e-1
5.0e-2

210826_CW-VI-153_pos

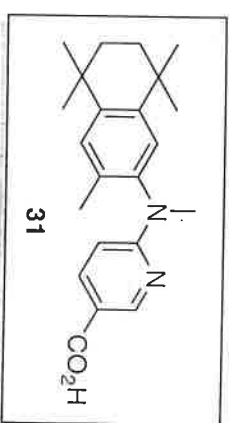
0.0
-0.00
0.50
1.00
1.50
2.00
2.50
3.00
3.50
4.00
4.50

100
0

353.0

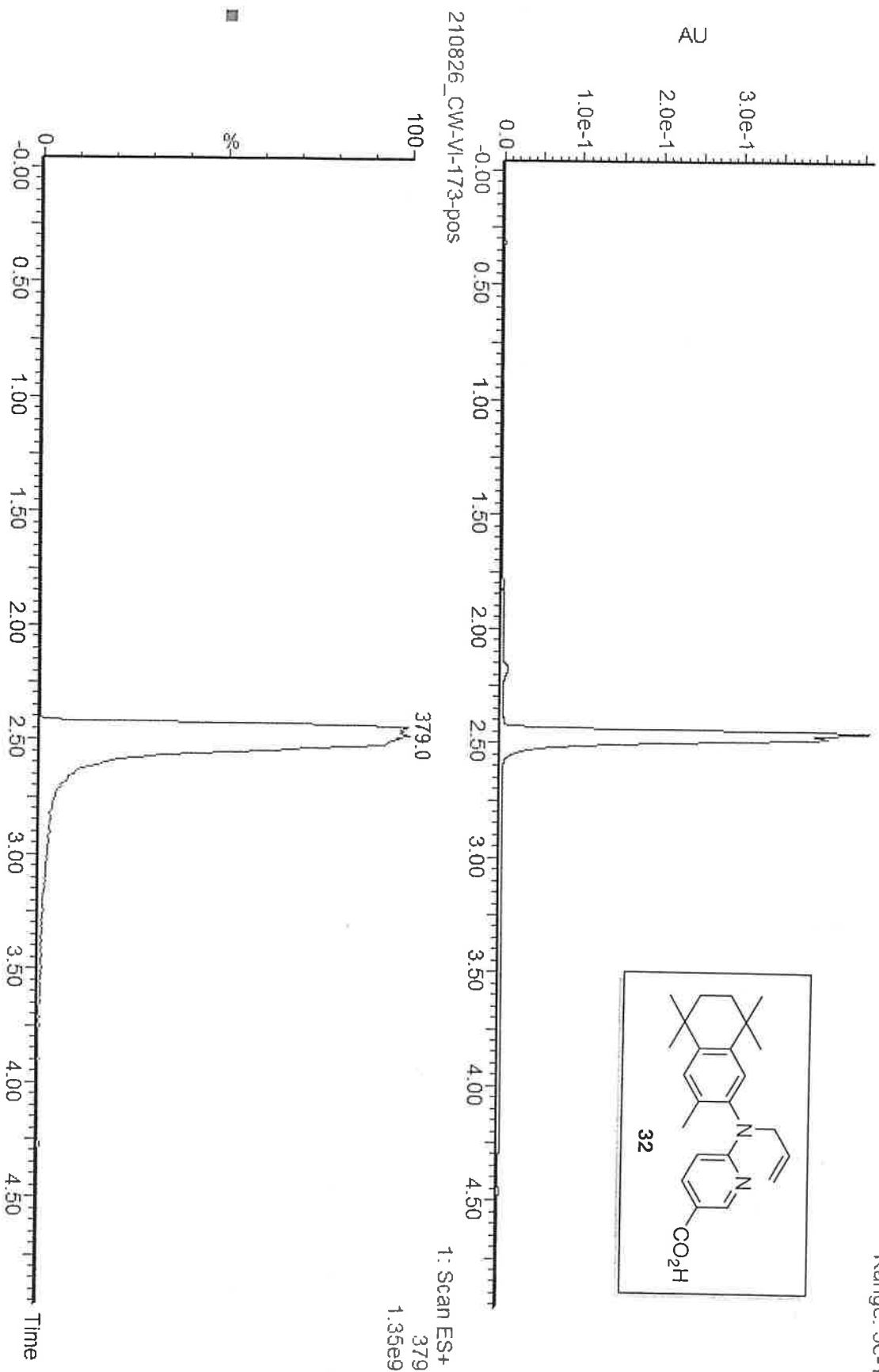
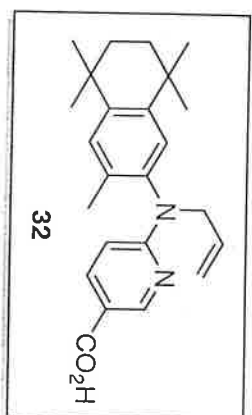
1: Scan ES+
353
1.33e9

0
-0.00
0.50
1.00
1.50
2.00
2.50
3.00
3.50
4.00
4.50
Time



mz not observed
210826_CW-VI-173-pos

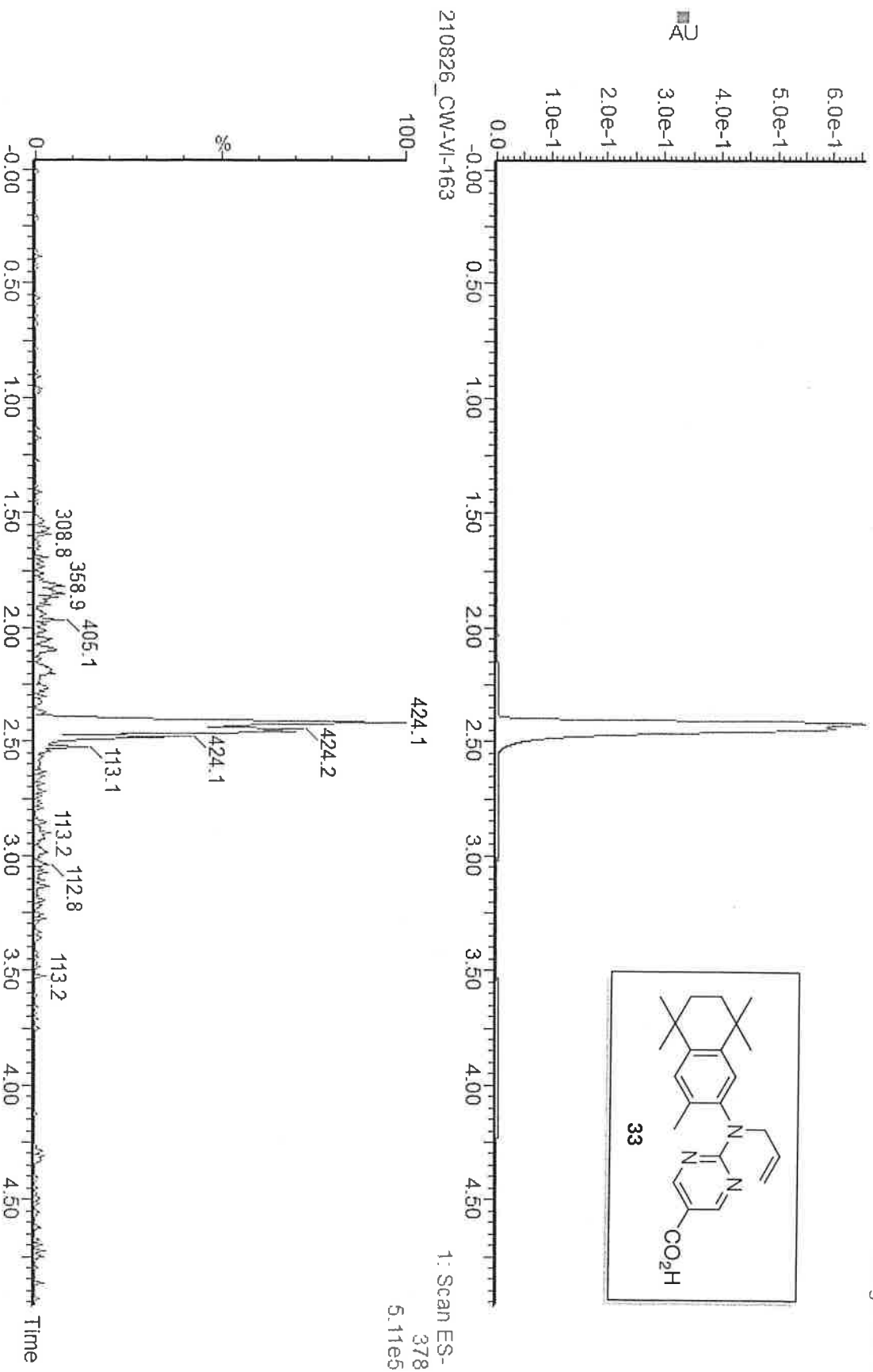
(2) PDA Ch2 260nm@2.4nm
Range: 5e-1



210826_CW-VI-163

(2) PDA Ch2 260nm@2.4nm
Range: 7e-1

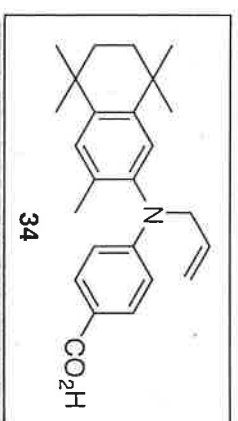
AU



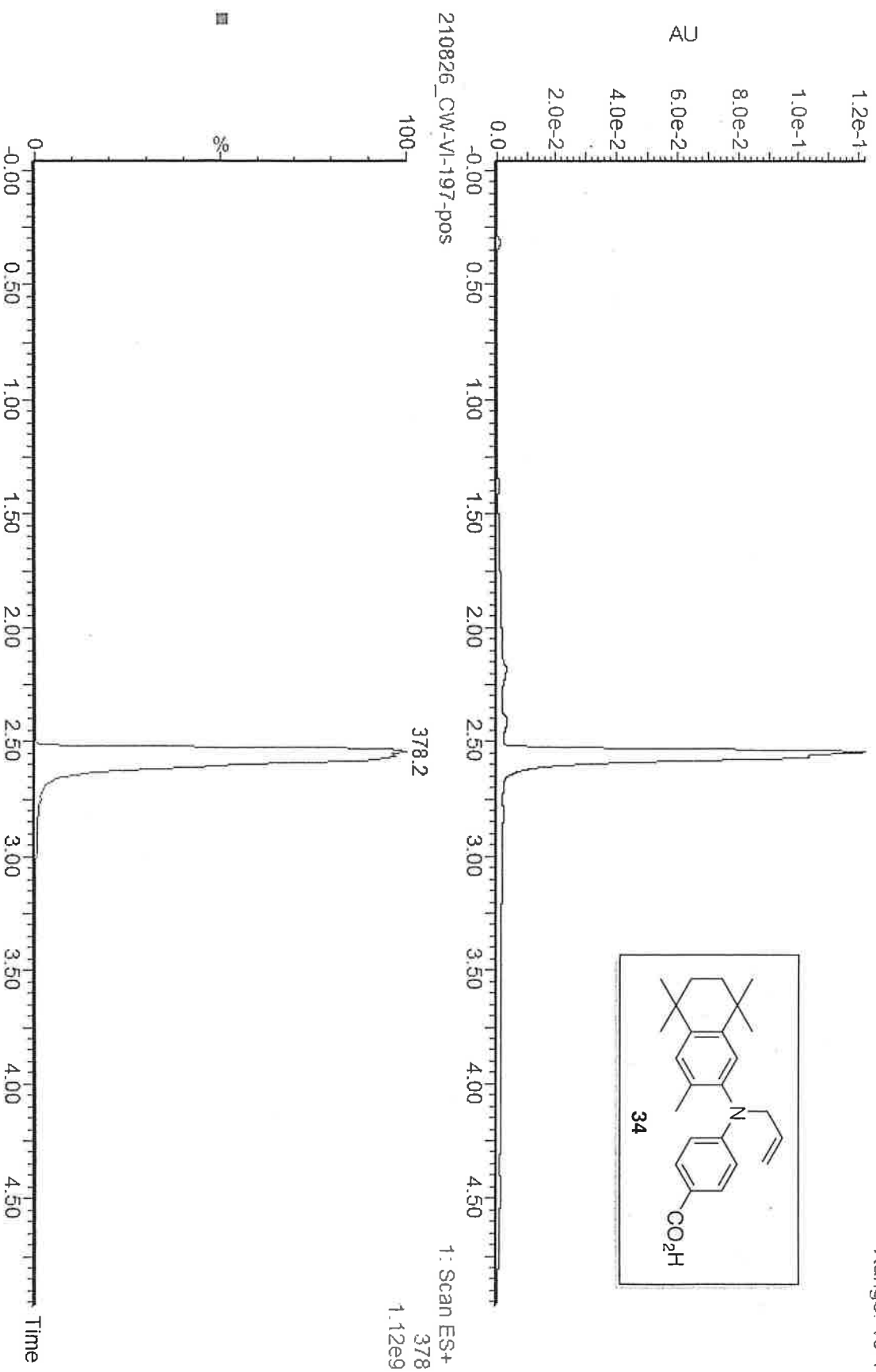
mz not observed

210826_CW-VI-197-pos

(2) PDA Ch2 260nm@2.4nm
Range: 1e-1

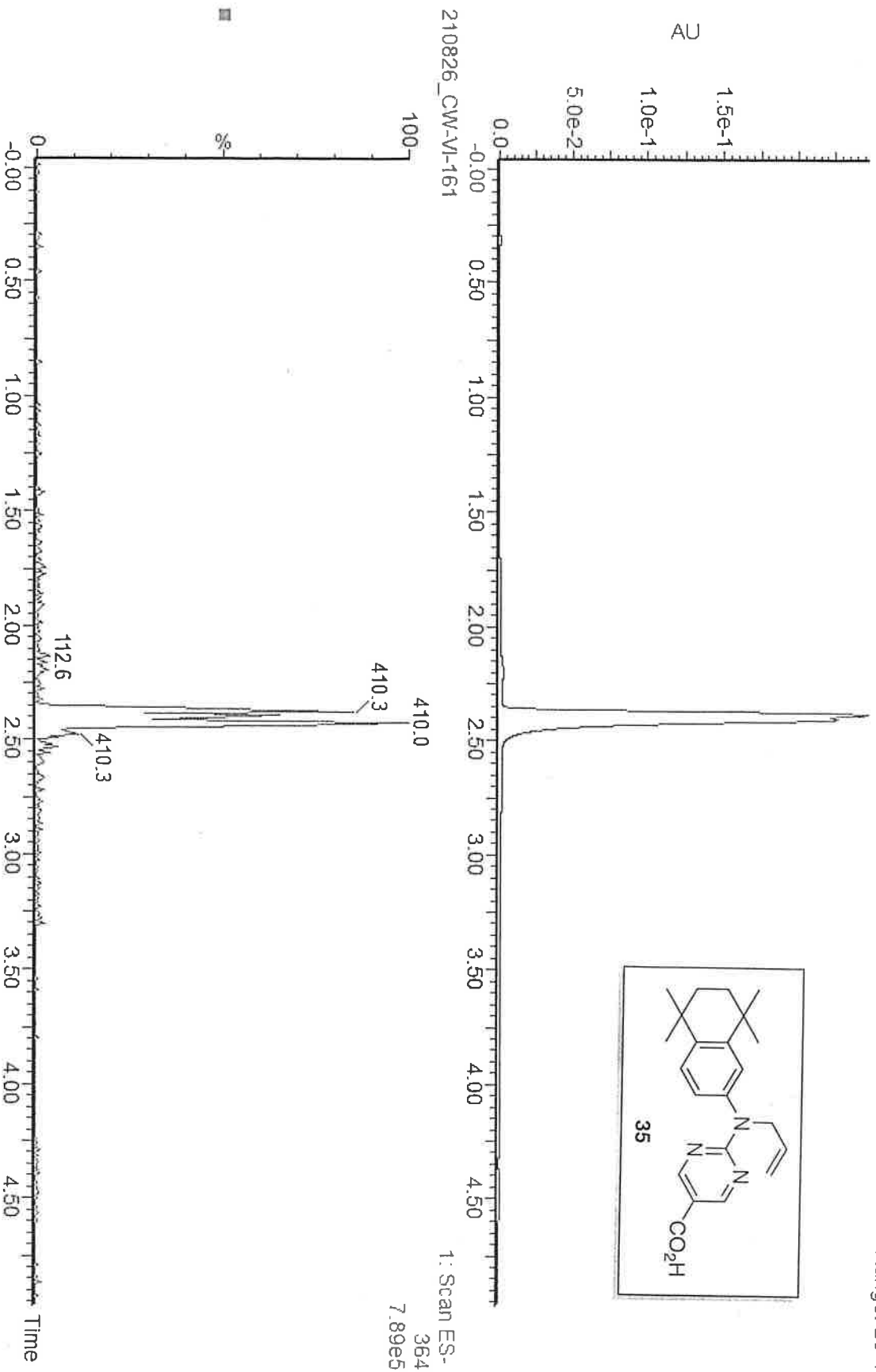
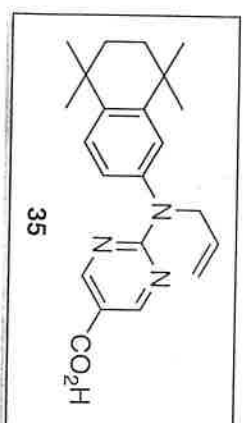


21



210826_CW-VI-161

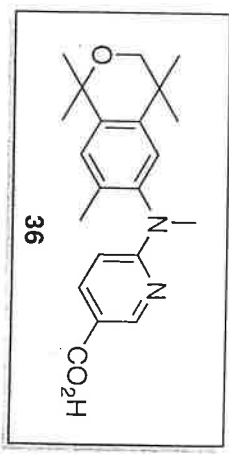
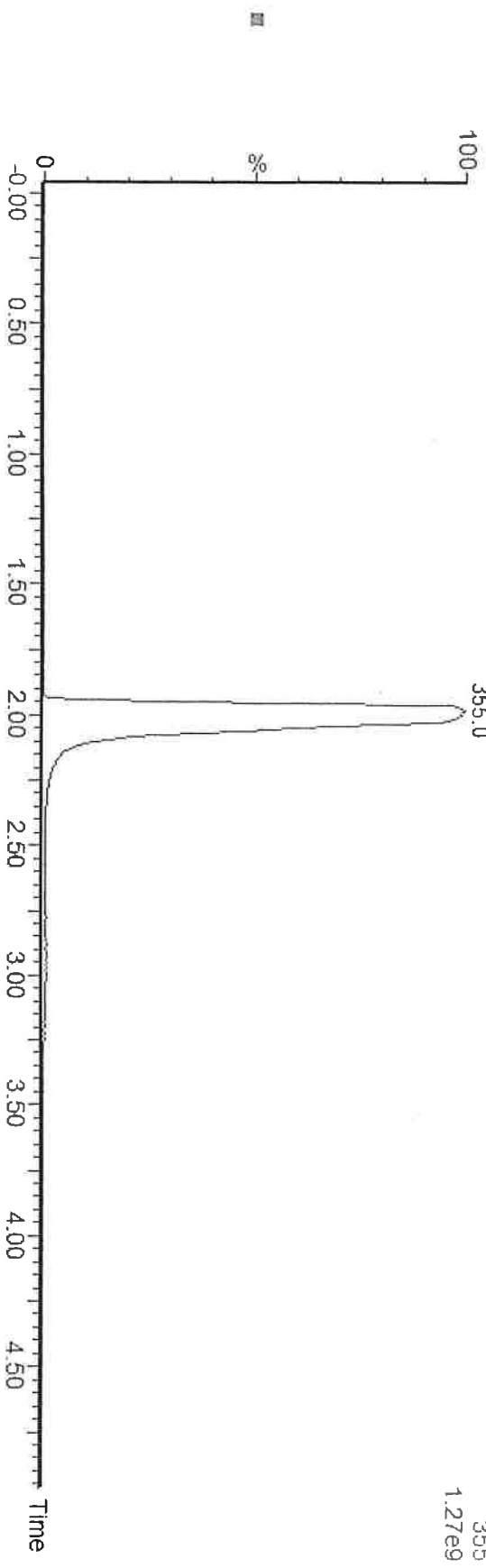
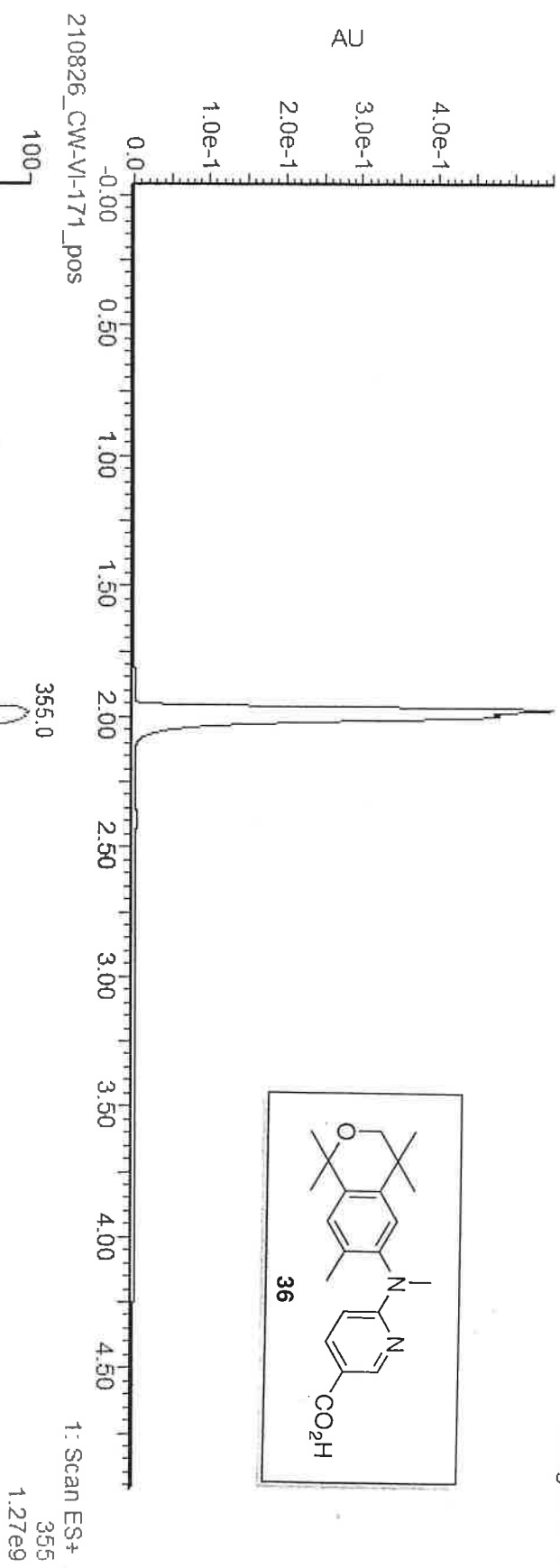
(2) PDA Ch2 260nm@2.4nm
Range: 2e-1



1: Scan ES-
364
7.89e5

mz not observed
210826_CW-VI-171_pos

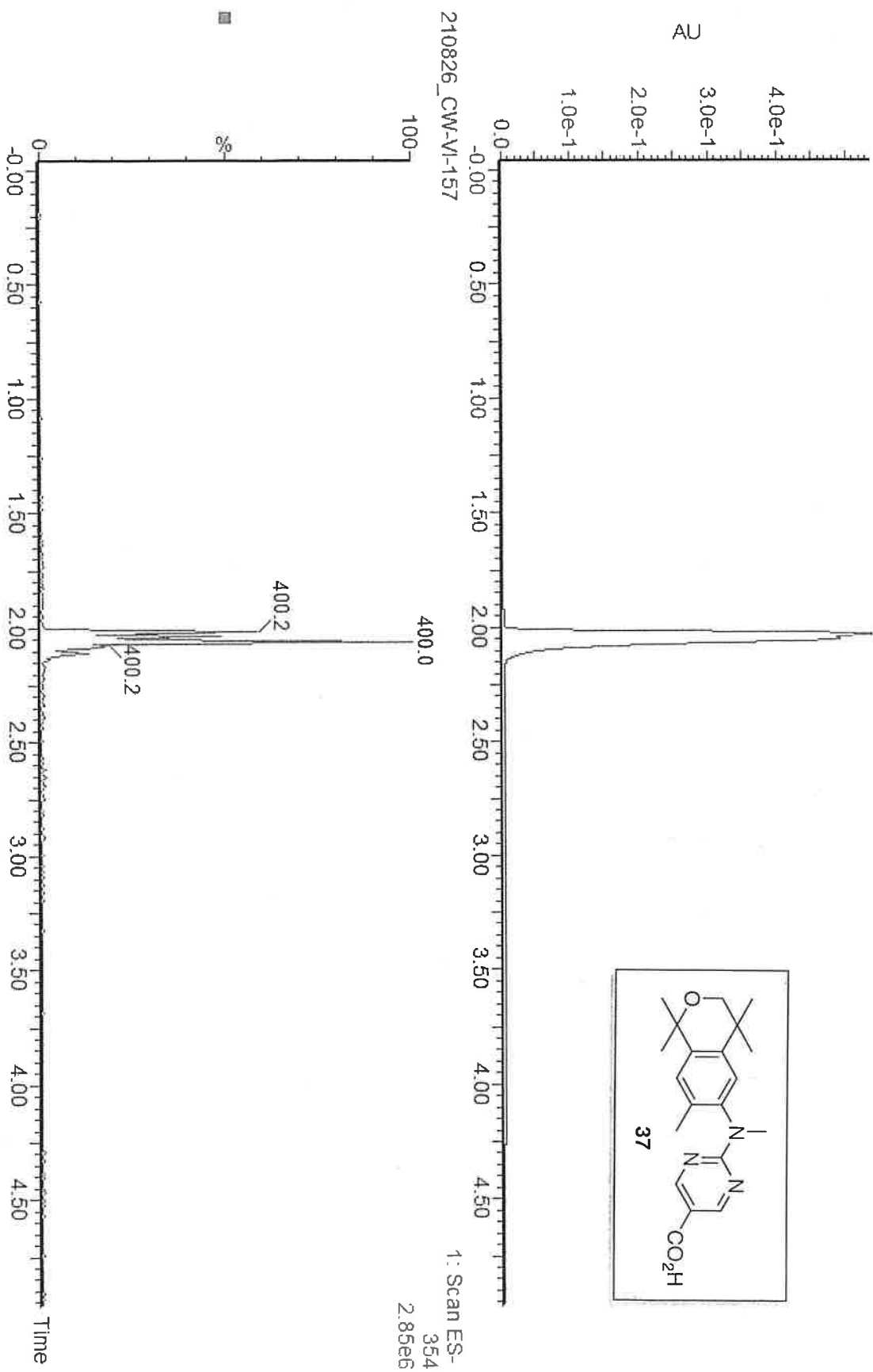
(2) PDA Ch2 260nm@2.4nm
Range: 6e-1



210826_CW-VI-157

(2) PDA Ch2 260nm@2.4nm
Range: 5e-1

21

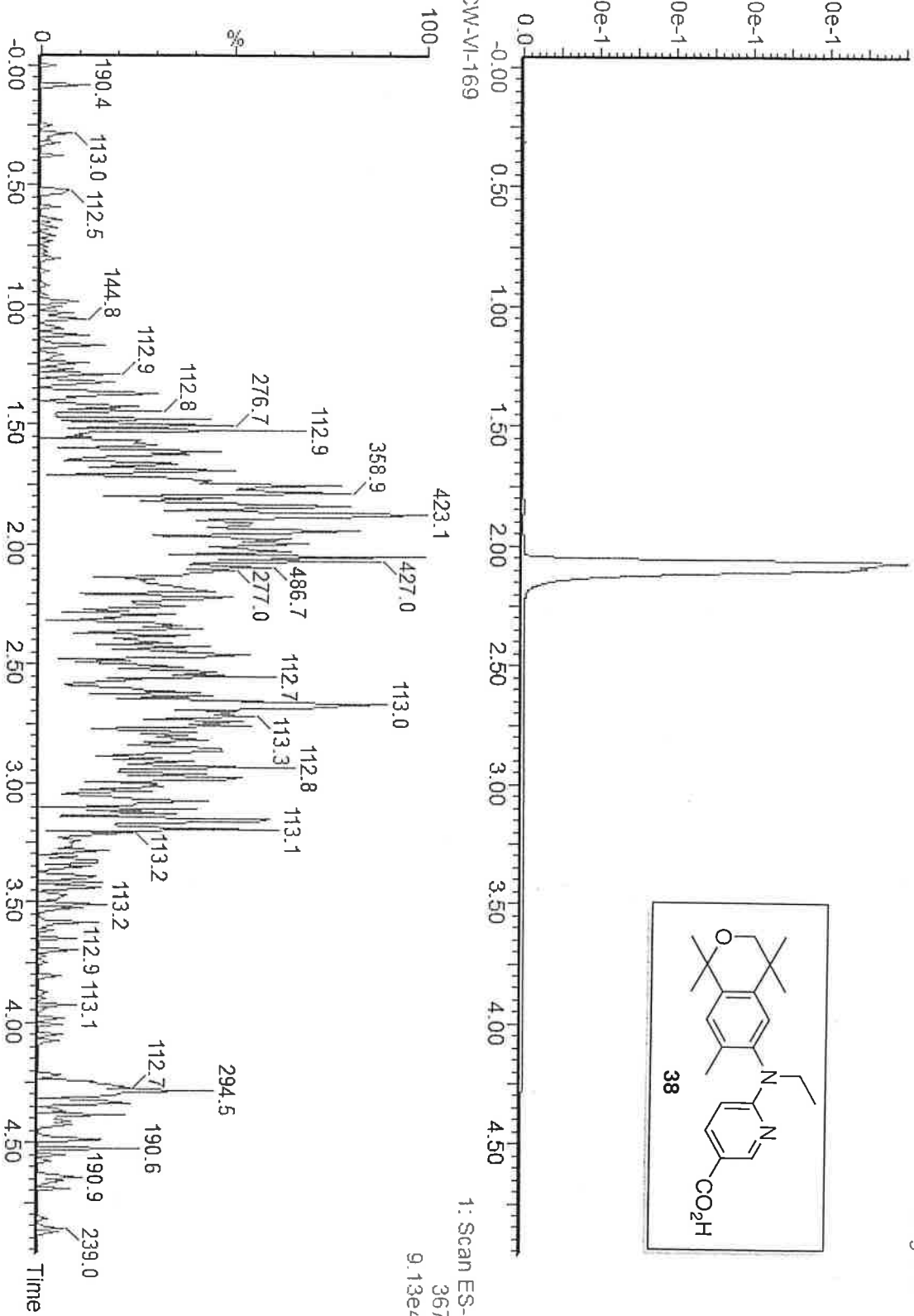


210826_CW-VI-169

(2) PDA Ch2 260nm@2.4nm
Range: 5e-1

AU

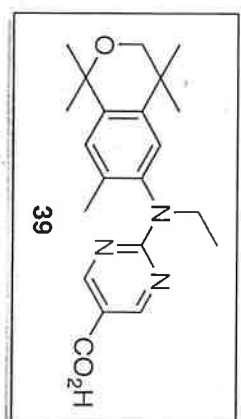
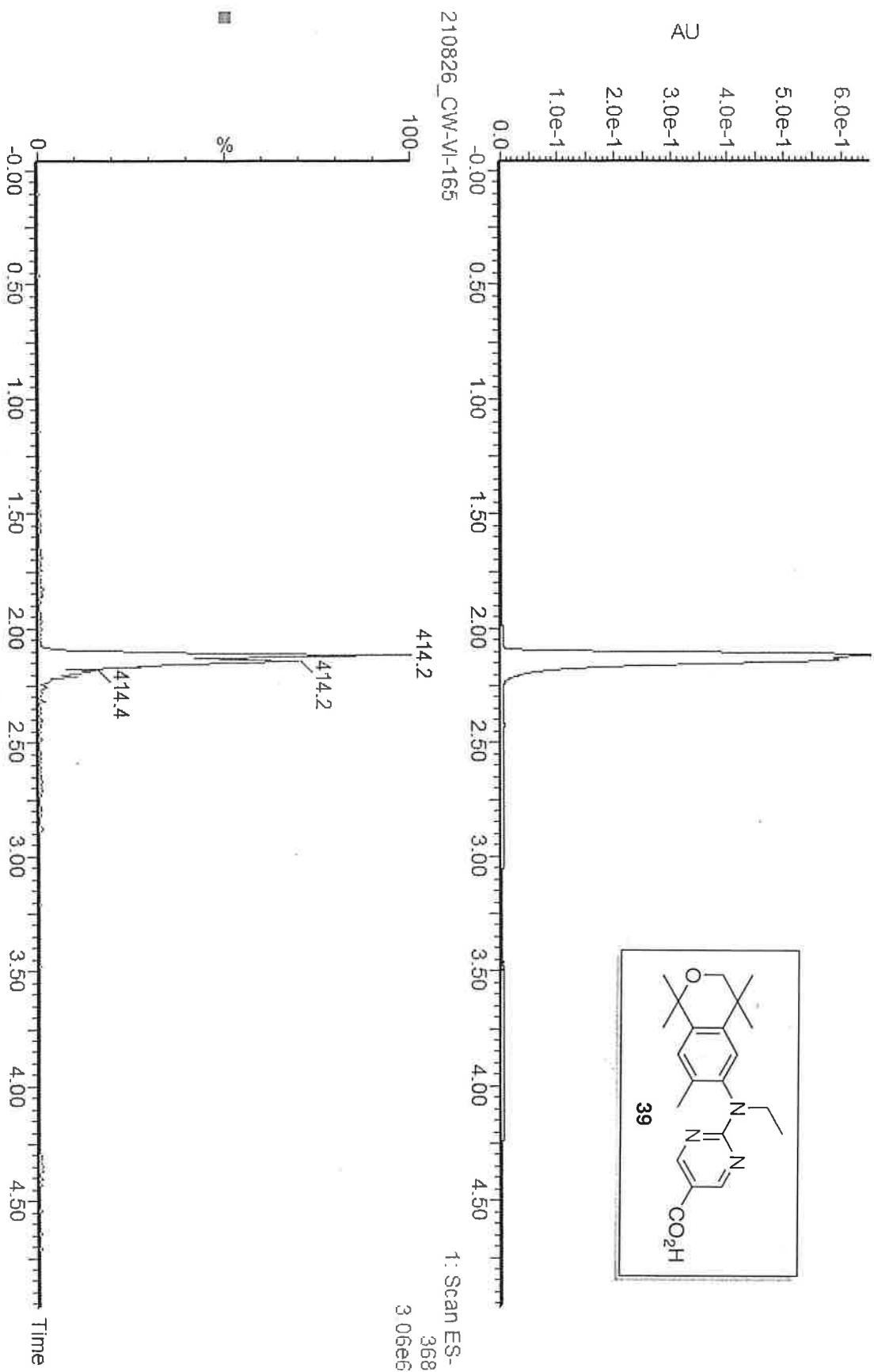
210826_CW-VI-169



210826_CW-VI-165

(2) PDA Ch2 260nm@2.4nm
Range: 7e-1

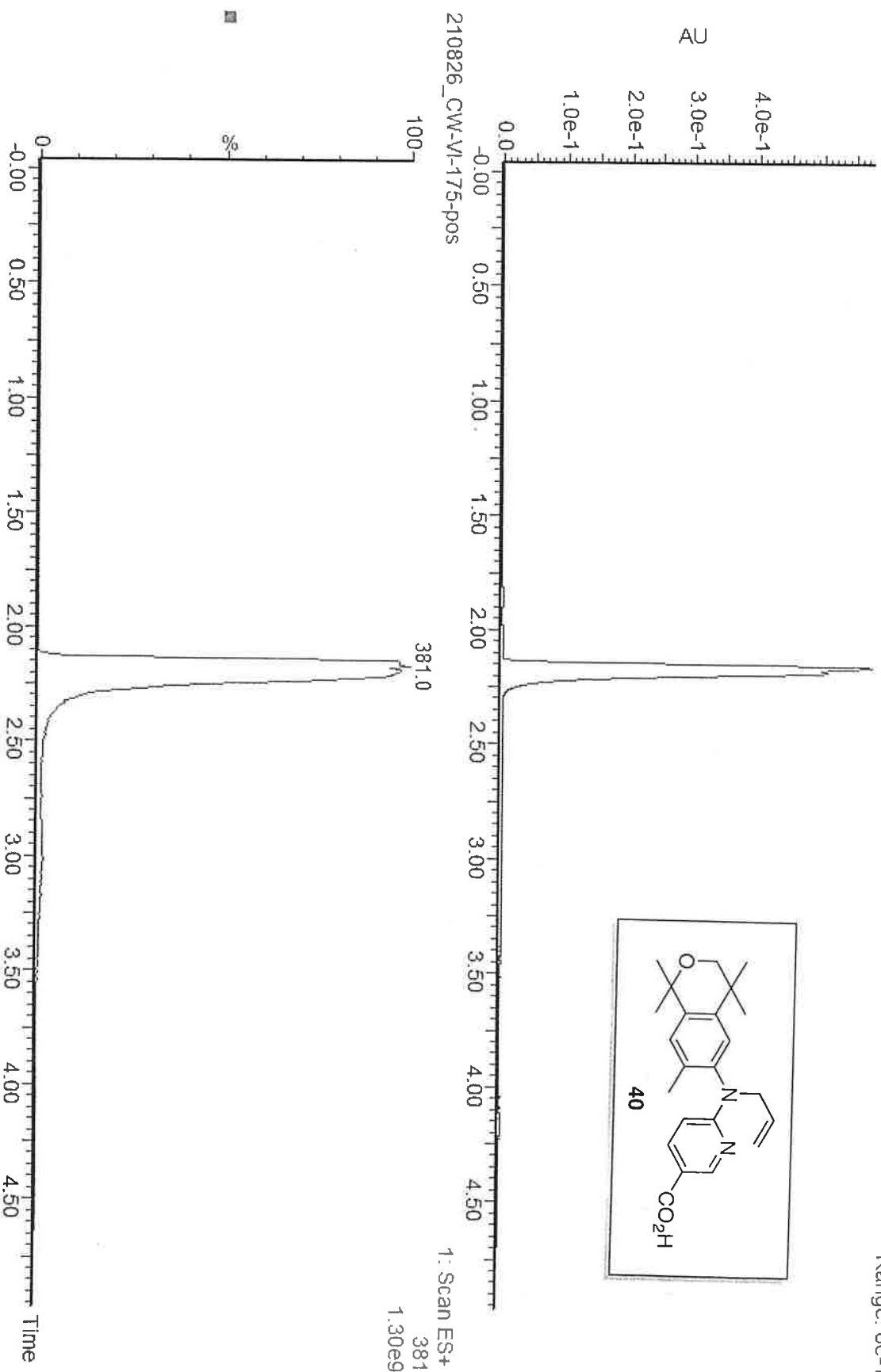
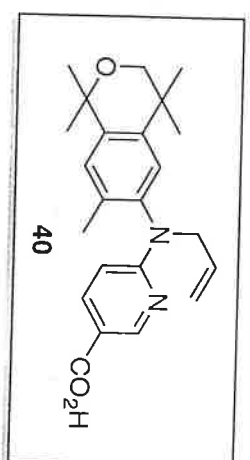
AU



mz not observed

210826_CW-VI-175-pos

(2) PDA Ch2 260nm@2.4nm
Range: 6e-1

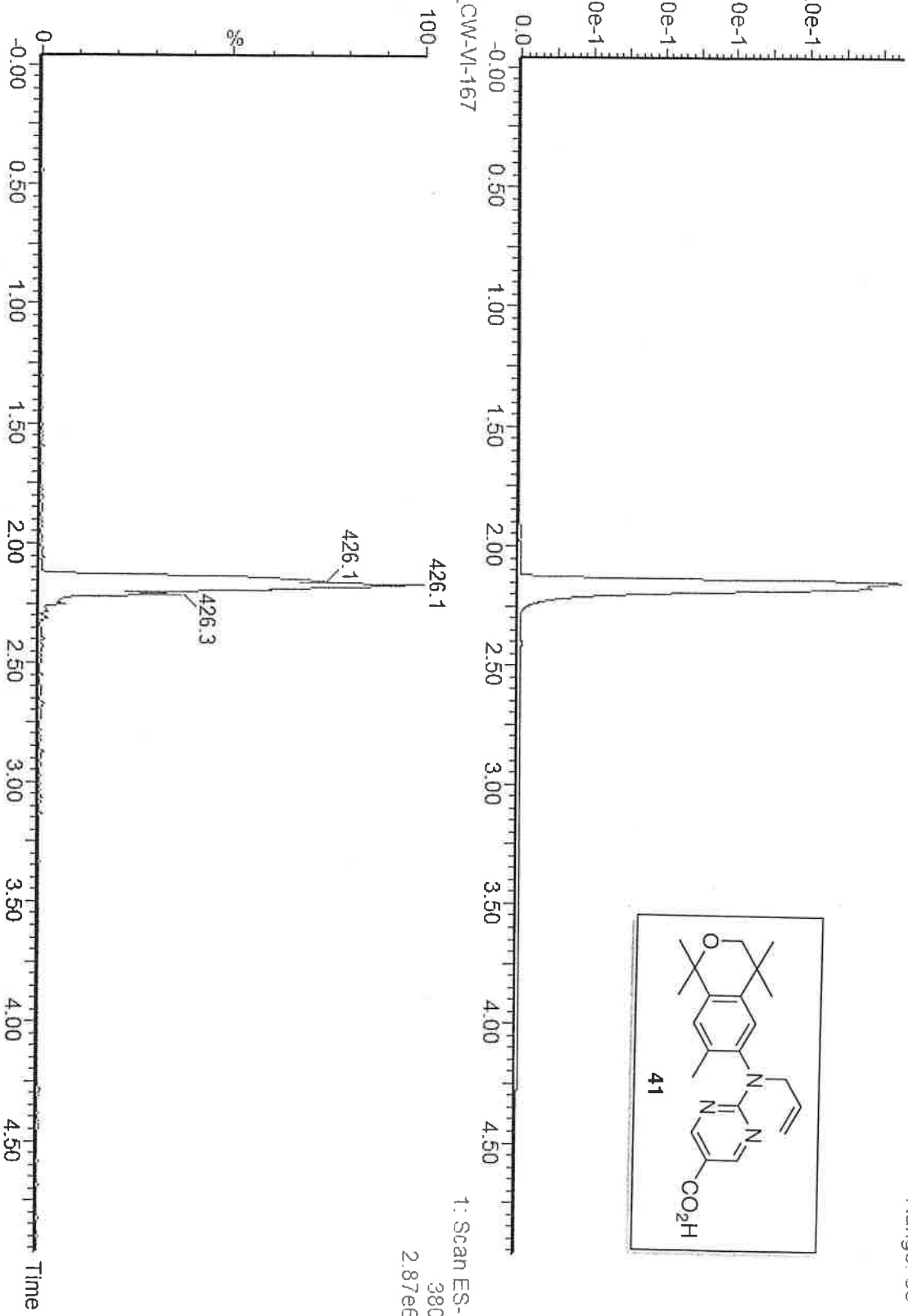


210826_CW-VI-167

(2) PDA Ch2 260nm@2.4nm
Range: 5e-1

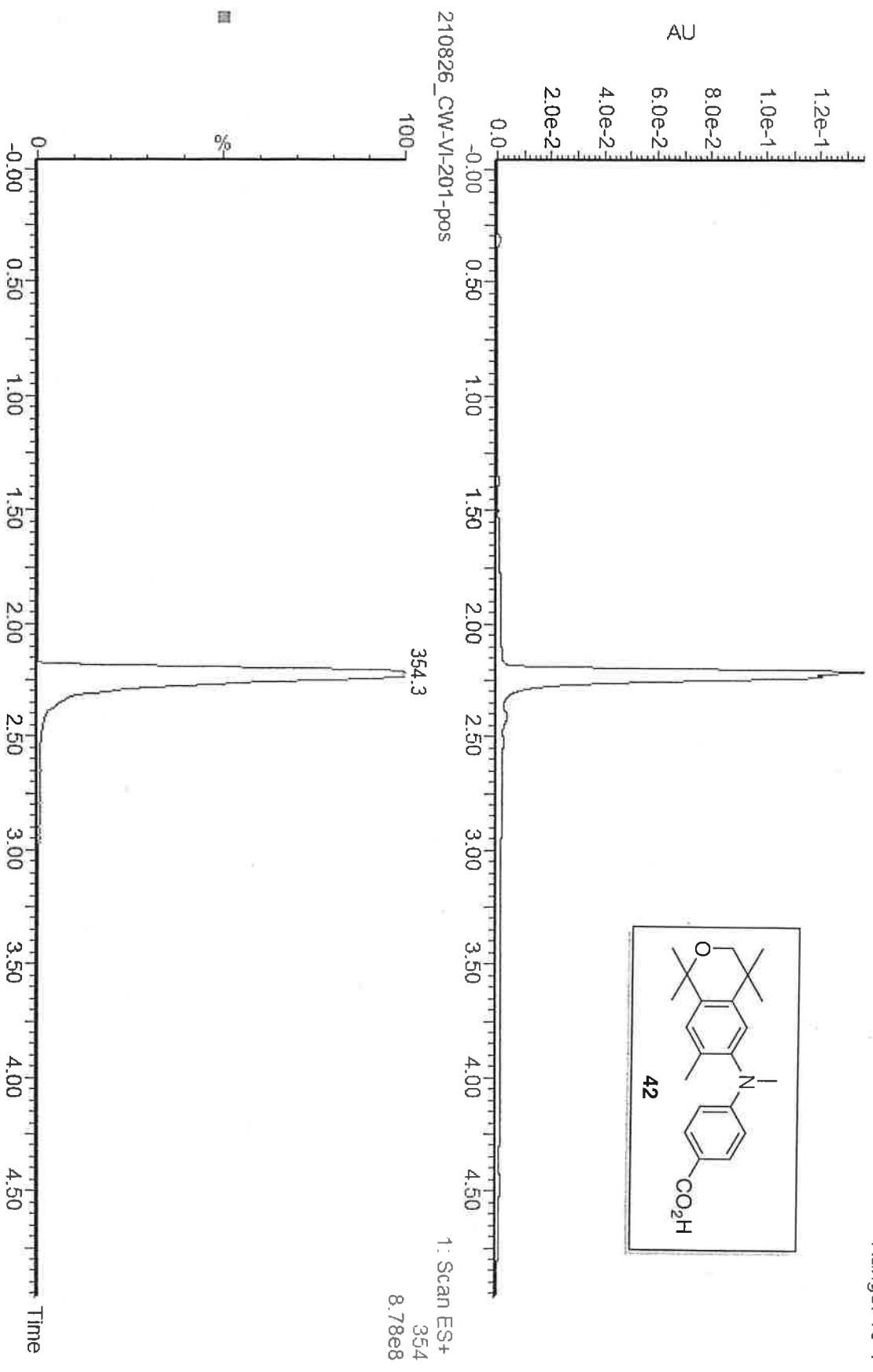
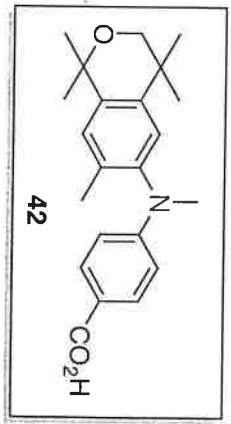
AU

210826_CW-VI-167



mz not observed
210826_CW-VI-201-pos

(2) PDA Ch2 260nm@2.4nm
Range: 1e-1



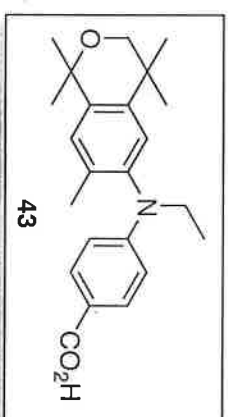
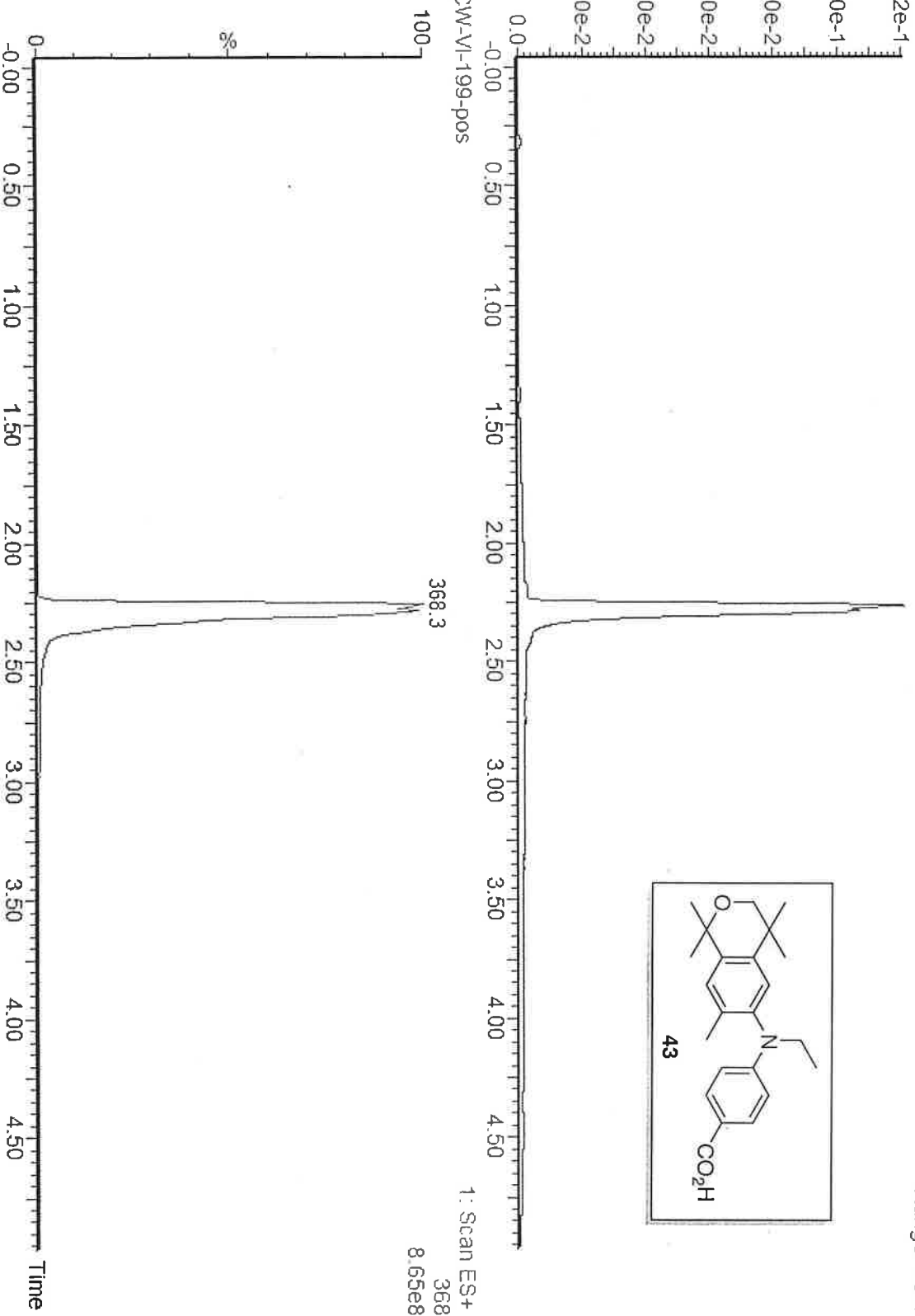
mz not observed

210826_CW-VI-199-pos

(2) PDA Ch2 260nm@2.4nm
Range: 1e-1

AU

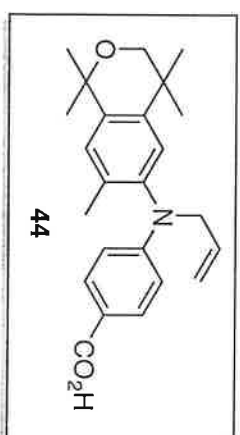
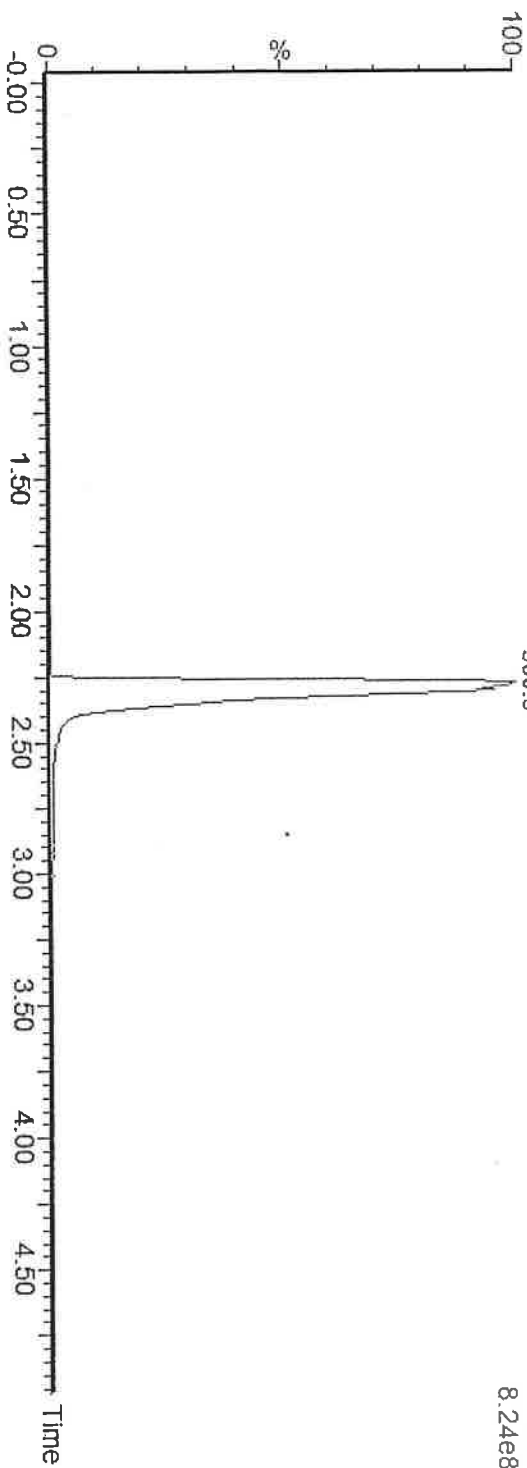
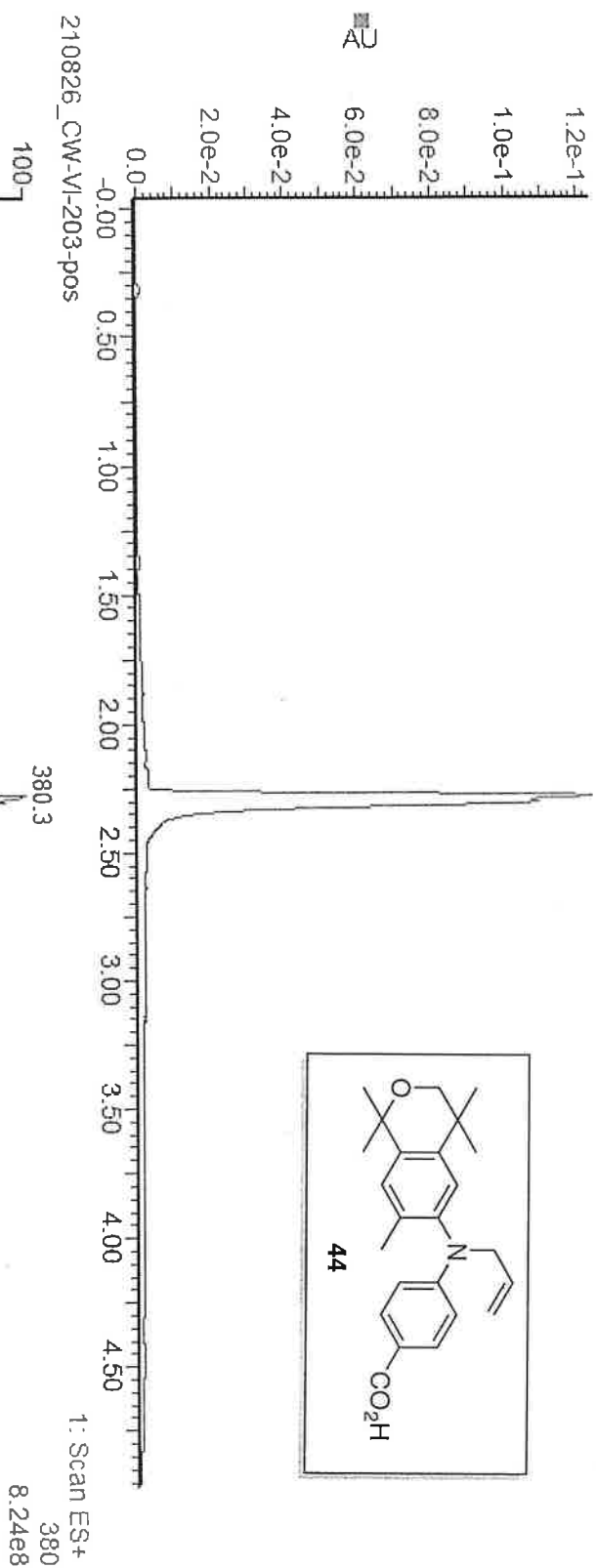
210826_CW-VI-199-pos



mz not observed
210826_CW-VI-203-pos

(2) PDA Ch2 260nm@2.4nm
Range: 1e-1

AU



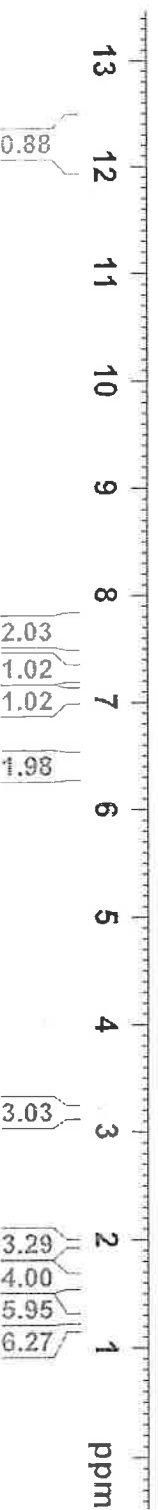
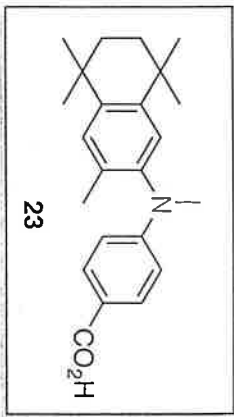
CWVI-195



12.143

7.721
7.698
7.275
7.079
6.439
6.418

3.202
2.509
2.504
2.500
2.495
2.491
1.973
1.631
1.259
1.190



NAME CWVI-195
EXPNO 1
PROCNO 1
Date_ 20181010
Time_ 14.42
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DW 60.800 usec
DE 6.50 usec
TE 292.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300034 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

CWVI-195

167.45
152.09
144.19
143.23
142.54
132.43
131.04
129.19
125.52
117.64
111.00

59.78
40.15
39.94
39.73
39.52
39.31
39.11
38.90
34.64
34.55
33.81
33.72
31.66
31.62
17.07



NAME CWVI-195

EXPNO 2

PROCNO 1

Date 20181010

Time 14.47

INSTRUM spect

PROBHD 5 mm PABBO BB-

PULPROG zg1930

TD 65536

SOLVENT DMSO

NS 6036

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 292.4 K

D1 2.00000000 sec

D11 0.03000000 sec

TD0 1

===== CHANNEL f1 =====

NUC1 13C

P1 8.25 usec

PL1 -2.10 dB

PL1W 60.29227829 W

SFO1 100.6228298 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16

NUC2 1H

PCPD2 90.00 usec

PL2 0.50 dB

PL12 16.21 dB

PL12W 12.76071072 W

PL12W 0.34266910 W

SFO2 400.1316005 MHz

SI 32768

SF 100.6128128 MHz

SE EM

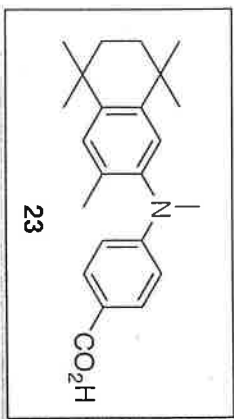
WDW 0

SSB 0

LB 1.00 Hz

GB 0

PC 1.40



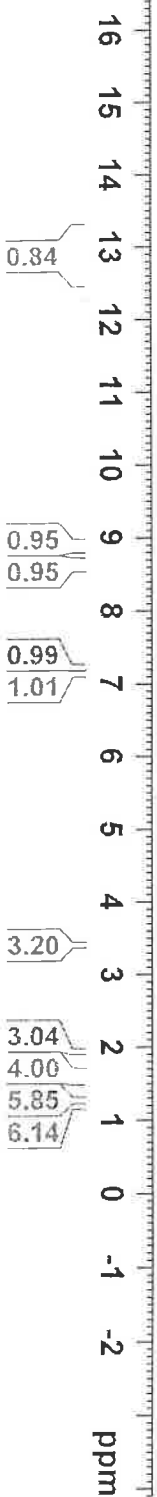
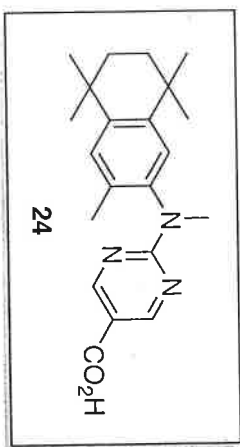
200 180 160 140 120 100 80 60 40 20 0 ppm

CWVI-159

12.871

8.875
8.868
8.658
8.651
7.222
7.150

3.403
2.509
2.504
2.500
2.495
2.491
1.953
1.626
1.269
1.243
1.211
1.187



NAME CWVI-159
EXPNO 1
PROCNO 1
Date_ 20181010
Time_ 10.04
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DW 60.800 usec
DE 6.50 usec
TE 291.9 K
D1 1.0000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300034 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



CWVI-159

165.66
162.20
159.72

143.56
143.16
141.00

131.87
128.59
124.73

113.29

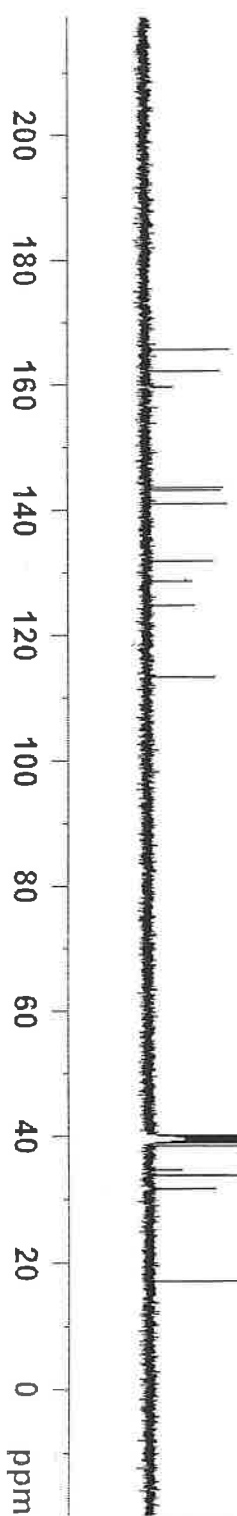
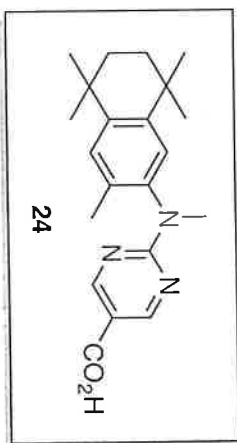
40.15
39.94
39.73
39.52
39.31
39.10
38.89
38.43
34.65
34.60
33.82
33.71
31.68
31.59
17.14



NAME CWVI-159
EXPNO 2
PROCNO 1
Date_ 20181010
Time 10.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 118
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 292.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.25 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL12 16.21 dB
PL12W 12.76071072 W
PL12W 0.34266910 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6128113 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



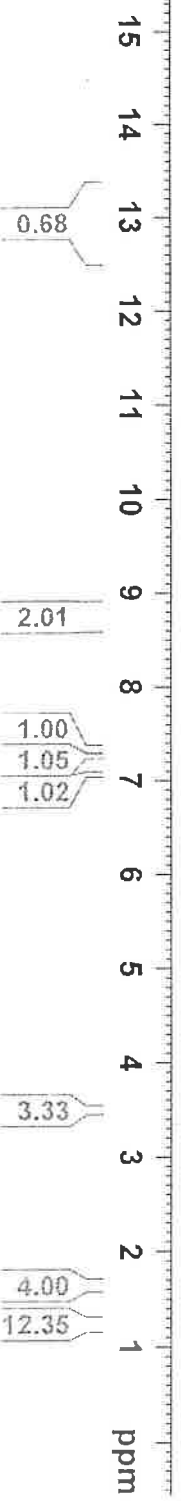
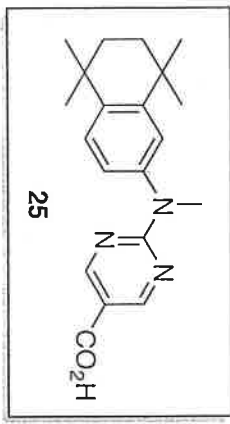
CWVI-155



12.866

8.754
7.349
7.328
7.269
7.264
7.081
7.075
7.060
7.054

3.484
2.504
2.500
2.496
1.649
1.259
1.230



```

NAME CWVI-155
EXPNO 1
PROCNO 1
Date_ 20181010
Time_ 20.50
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DE 60.800 usec
TE 292.6 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300031 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB .0
PC 1.00
  
```

CWVI-155

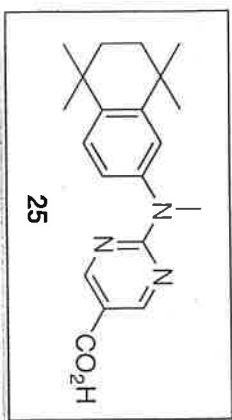
165.58
162.38
159.40

145.36
142.27
141.91

126.96
124.06
124.00

113.72

39.52
38.99
34.56
34.12
33.80
31.62
31.54



200 180 160 140 120 100 80 60 40 20 0 ppm



```

NAME CWVI-155
EXPNO 2
PROCNO 1
Date_ 20181010
Time 20.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 658
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 292.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.25 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL12 16.21 dB
PL12W 12.76071072 W
PL12W 0.34266910 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6128120 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
  
```

CWVI-141 f6-f13 (pure)

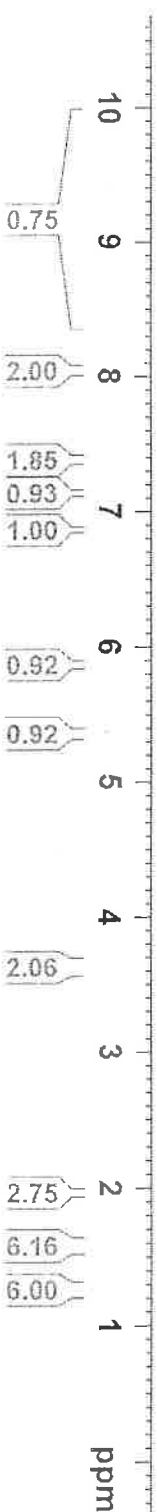
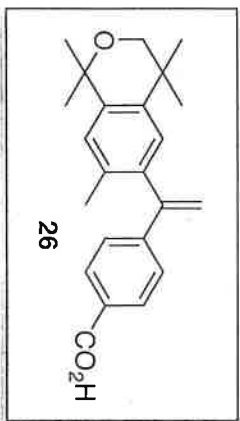
8.902
8.057
8.053
8.040
8.035
7.387
7.383
7.370
7.366
7.260
7.137
6.863
5.870
5.867
5.359
5.356

3.628

1.965

1.572

1.277



NAME CWVI-141
EXPNO 1
PROCNO 1
Date_ 20180711
Time_ 14.27
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DW 60.800 usec
DE 6.50 usec
TE 294.5 K
D1 1.00000000 sec
TD0 1

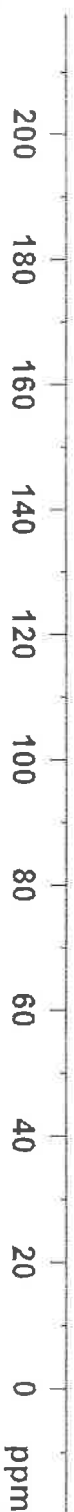
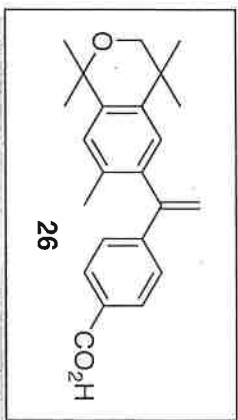
===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

CWVI-141 f6-f13 (pure)

171.66
148.74
146.07
141.09
140.02
138.76
133.20
130.34
128.20
126.90
126.68
126.59
117.46

77.32
77.00
76.68
75.16
70.91

33.59
29.82
27.05
19.97



NAME CWVI-141

EXPNO 2

PROCNO 1

Date_ 20180711

Time_ 14.33

INSTRUM spect

PROBHD 5 mm PABBO BB-

PULPROG zgpg30

TD 65536

SOLVENT CDCl3

NS 129

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 294.8 K

D1 2.00000000 sec

D11 0.03000000 sec

TD0 1

===== CHANNEL f1 =====

NUC1 13C

P1 8.25 usec

PL1 -2.10 dB

PL1W 60.29227829 W

SFO1 100.6228298 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16

NUC2 1H

PCPD2 90.00 usec

PL2 0.50 dB

PL12 16.21 dB

PL12W 12.76071072 W

PL12W 0.34266910 W

SFO2 400.1316005 MHz

SI 32768

SF 100.6127722 MHz

WDW EM

SSB 0

LB 1.00 Hz

GB 0

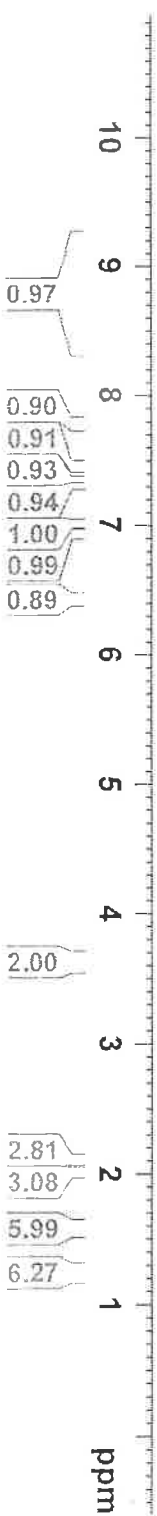
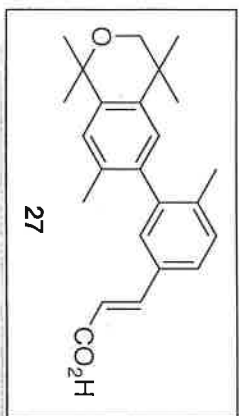
PC 1.40

CWVI-143

8.784
7.803
7.763
7.470
7.465
7.450
7.446
7.349
7.345
7.319
7.299
7.260
7.006
6.943
6.445
6.405

3.626

2.103
2.020
1.587
1.586
1.262
1.238



NAME CWVI-143
EXPNO 1
PROCNO 1
Date_ 20181009
Time_ 10.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DM 60.800 usec
DE 6.50 usec
TE 291.9 K
D1 1.00000000 sec
TD0 1

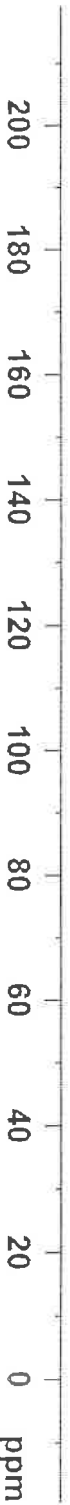
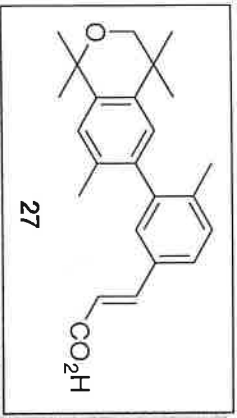
===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

CWVI-143

146.95
142.26
140.60
139.82
139.56
138.48
132.82
131.50
130.54
129.49
127.13
126.56
125.89
116.42

77.32
77.00
76.68
75.17
70.88

33.62
29.86
29.84
27.10
26.99
20.11
19.59



NAME CWVI-143

EXPNO 2

PROCNO 1

Date 20181009

Time 10.43

INSTRUM spect

PROBHD 5 mm PABBO BB-

PULPROG zgpg30

TD 65536

SOLVENT CDCl3

NS 141

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 292.2 K

D1 2.00000000 sec

D11 0.03000000 sec

TD0 1

===== CHANNEL f1 =====

NUC1 13C

P1 8.25 usec

PL1 -2.10 dB

PL1W 60.29227829 W

SFO1 100.6228298 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16

NUC2 1H

PCPD2 90.00 usec

PL2 0.50 dB

PL12 16.21 dB

PL2W 12.76071072 W

PL12W 0.34266910 W

SFO2 400.1316005 MHz

SI 32768

SF 100.6127736 MHz

WDW EM

SSB 0

LB 1.00 Hz

GB 0

PC 1.40

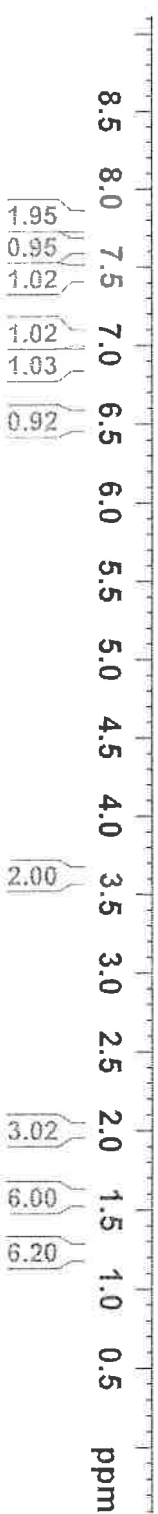
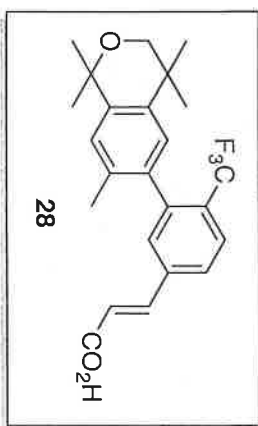
CWVI-145

7.810
7.805
7.784
7.770
7.635
7.614
7.465
7.260
7.037
6.923
6.552
6.511

3.623
3.618

2.002

1.581
1.244
1.215



NAME CWVI-145
EXENO 1
PROCNO 1
Date 20181009
Time 10.52
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG 2930
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DE 60.800 usec
TE 291.8 K
D1 1.00000000 sec
TD0 1

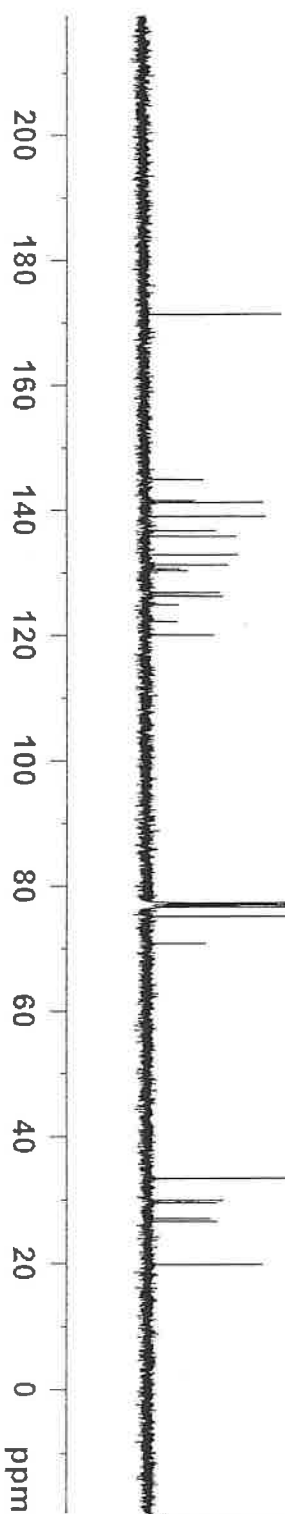
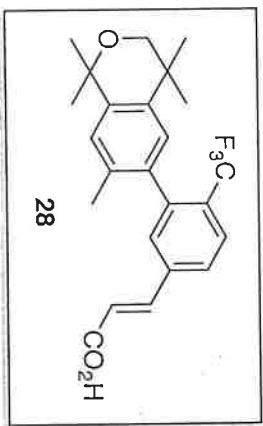
===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SF01 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

CWVI-145

171.37
144.94
141.58
141.56
141.29
139.05
136.77
135.85
132.89
131.28
130.65
130.36
126.87
126.84
126.78
126.38
126.37
126.27
124.92
122.20
120.05

77.32
77.00
76.68
75.17
70.81

33.51
29.98
29.63
27.09
26.65
19.83



NAME CWVI-145

EXPNO 2

PROCNO 1

Date 20181009

Time 11.14

INSTRUM spect

PROBHD 5 mm PABBO BB-

PULPROG zg1930

TD 65536

SOVENT CDC13

NS 478

DS 4

SMH 24038.461 Hz

FIDRES 0.366798 Hz

AQ 1.3631988 sec

RG 2050

DW 20.800 usec

DE 6.50 usec

TE 292.2 K

D1 2.00000000 sec

D11 0.03000000 sec

TD0 1

===== CHANNEL f1 =====

NUC1 13C

P1 8.25 usec

PL1 -2.10 dB

PL1W 60.29227829 W

SFO1 100.6228298 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16

NUC2 1H

PCPD2 90.00 usec

PL2 0.50 dB

PL12 16.21 dB

PL12W 12.76071072 W

PL12W 0.34266910 W

SFO2 400.1316005 MHz

SI 32768

SF 100.6127729 MHz

WDW EM

SSB 0

LB 1.00 Hz

GB 0

PC 1.40

CWVI-149

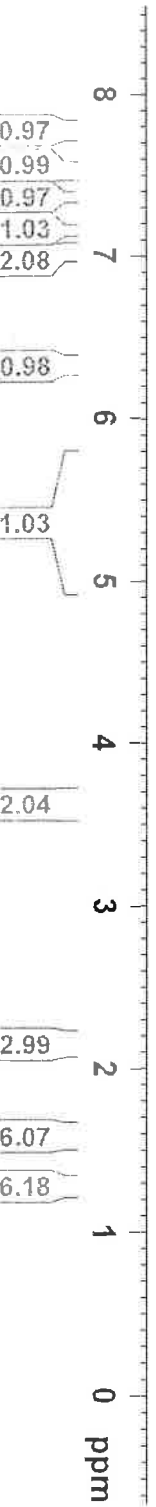
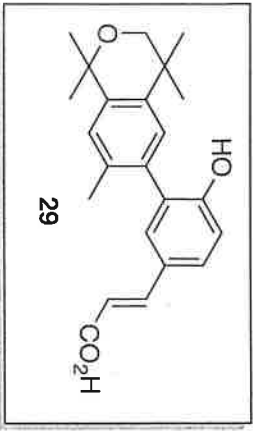
7.520
7.515
7.499
7.493
7.359
7.354
7.260
7.147
7.034
7.023
7.013
6.346
6.307
— 5.338

— 3.620

— 2.130

— 1.586

— 1.263

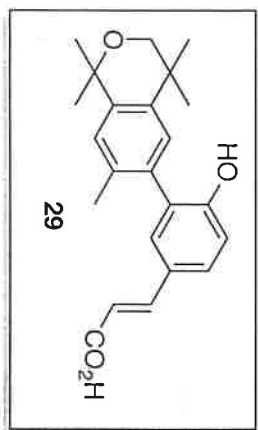


NAME CWVI-149
EXPNO 3
PROCNO 1
Date_ 20181023
Time_ 12.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DM 60.800 usec
DE 6.50 usec
TE 292.1 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

CWVI-149

172.41
155.17
146.67
142.32
141.09
134.40
132.64
130.81
129.64
128.35
127.60
127.14
126.71
115.99
114.82
77.32
77.00
76.68
75.16
70.74
33.70
29.73
27.02
19.46



200 180 160 140 120 100 80 60 40 20 0 ppm

NAME CWVI-149
EXPNO 4
PROCNO 1
Date 20181023
Time 12.43
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg1g30
TD 65536
SOLVENT CDCl3
NS 213
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 292.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.25 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL12 16.21 dB
PL2W 12.76071072 W
PL12W 0.34266910 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127736 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

CWVI-151

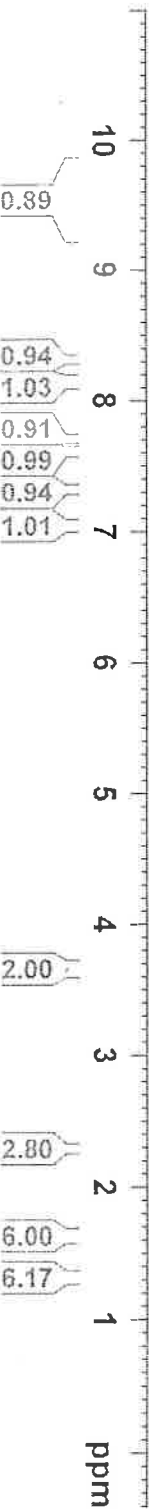
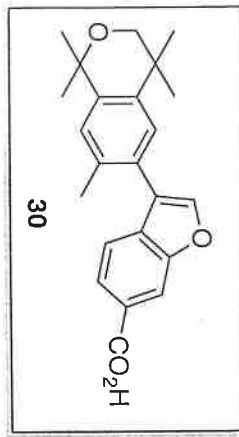
9.499
8.308
8.304
8.164
8.159
8.142
8.138
7.710
7.629
7.607
7.606
7.316
7.260
7.047

— 3.658

— 2.280

— 1.618

— 1.305



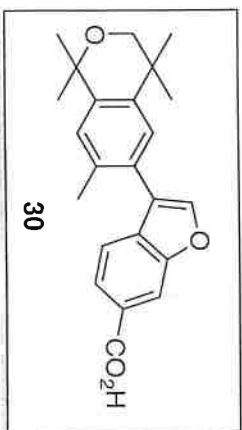
NAME CWVI-151
EXPNO 1
PROCNO 1
Date_ 20181009
Time_ 12.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DW 60.800 usec
DE 6.50 usec
TE 292.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SF01 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



CWVI-151

172.19
158.13
143.47
141.58
140.29
134.09
128.16
128.02
127.39
127.19
126.76
124.33
124.10
121.92
111.71
77.32
77.00
76.69
75.18
70.83
33.63
29.76
27.03
20.26



NAME CWVI-151
EXPNO 2
PROCNO 1
Date_ 20181009
Time_ 12.14
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 117
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 292.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.25 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL12 16.21 dB
PL12W 12.76071072 W
PL12W 0.34266910 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127736 MHz
WDM EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

CWVI-153

— 10.374

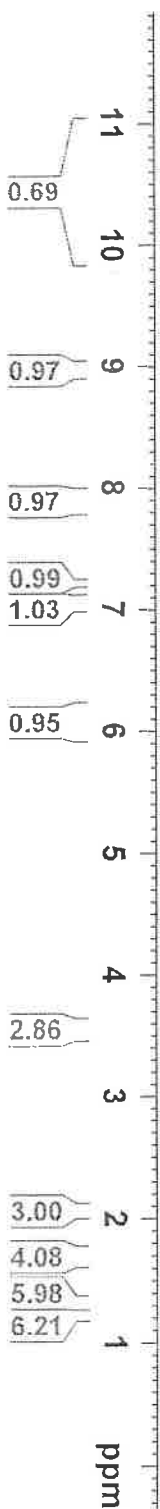
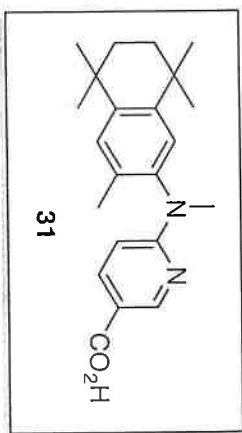
8.971
8.966

7.925
7.906
7.260
7.227
7.062
7.045

6.116
6.095

— 3.535

2.071
1.690
1.312
1.296
1.249
1.236
1.223



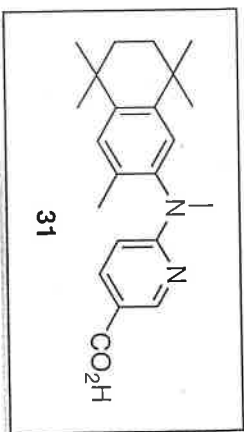
NAME CWVI-153
EXPNO 1
PROCNO 1
Date_ 20181009
Time_ 12.23
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DW 60.800 usec
DE 6.50 usec
TE 292.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



CWVI-153

169.69
161.95
145.32
139.98
139.11
132.18
131.99
129.79
125.33
114.15
112.93
108.33
77.32
77.00
76.69
38.96
34.93
34.81
34.19
34.11
31.94
31.82
17.11



200 180 160 140 120 100 80 60 40 20 0 ppm

NAME CWVI-153
EXPNO 5
PROCNO 1
Date 20181010
Time 5.34
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
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 292.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

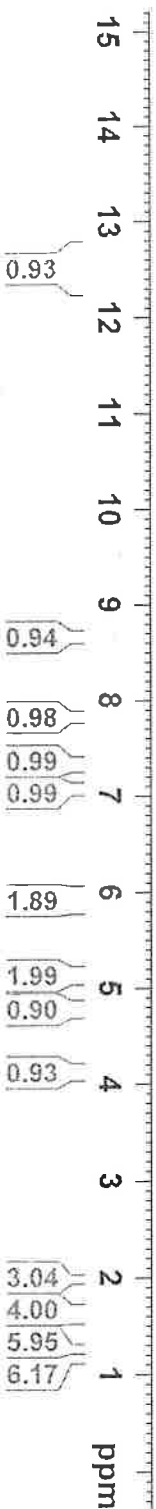
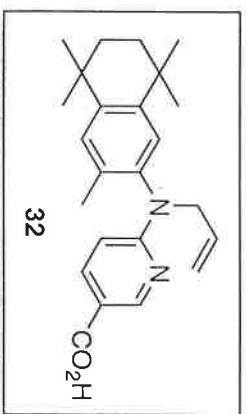
===== CHANNEL f1 =====
NUC1 13C
P1 8.25 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL12 16.21 dB
PL2W 12.76071072 W
PL12W 0.34266910 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127736 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

CWVI-173

12.504

8.674
8.669
7.838
7.832
7.816
7.810
7.301
7.080
6.022
6.007
5.997
5.992
5.982
5.965
5.954
5.949
5.939
5.924
5.148
5.145
5.121
5.102
5.095
4.803
4.773
4.123
4.101
2.509
2.504
2.500
2.495
2.491
1.983



NAME CWVI-173
EXPNO 1
PROCNO 1
Date_ 20181010
Time_ 13.45
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DW 60.800 usec
DE 6.50 usec
TE 292.2 K
D1 1.00000000 sec
TD0 1
===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SF01 400.1324710 MHz
SI 32768
SF 400.1300034 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

CWVI-173

166.69
159.50
150.67
143.95
143.83
139.26
138.18
134.18
132.53
129.20
126.56
117.32
114.95
106.11

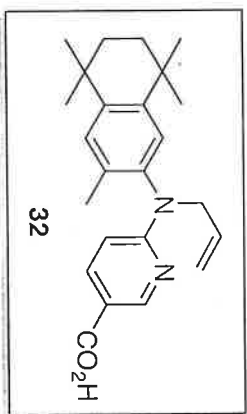
52.16
40.15
39.94
39.73
39.52
39.31
39.10
38.89
34.60
34.44
33.77
33.75
31.57
17.12



NAME CWVI-173
EXPNO 2
PROCNO 1
Date_ 20181010
Time 13.52
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 426
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 292.4 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.25 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL12 16.21 dB
PL2W 12.76071072 W
PL12W 0.34266910 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6128121 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

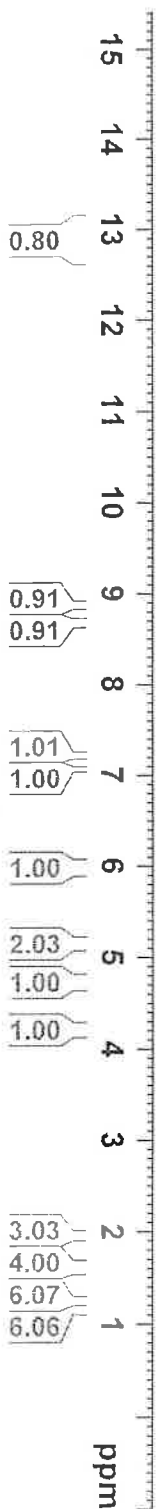
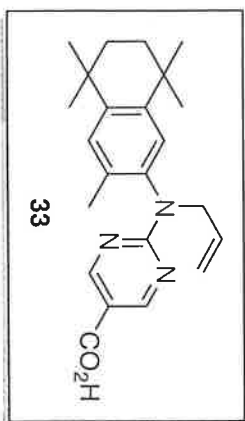


200 180 160 140 120 100 80 60 40 20 0 ppm

CWVI-163

— 12.892

8.868
8.679
7.216
7.065
6.030
6.014
6.006
5.999
5.990
5.986
5.975
5.971
5.961
5.955
5.946
5.931
5.163
5.160
5.149
5.120
4.759
4.744
4.721
4.707
4.226
4.210
4.188
4.172
2.509
2.504
2.500
2.495
2.491
1.952



NAME CWVI-163
EXPNO 1
PROCNO 1
Date 20181010
Time 10.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DW 60.800 usec
DE 6.50 usec
TE 292.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300034 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

CWVI-163

165.58
161.87
159.78

143.14
143.06
139.52
133.60
132.27
128.52
125.57

117.83
113.72

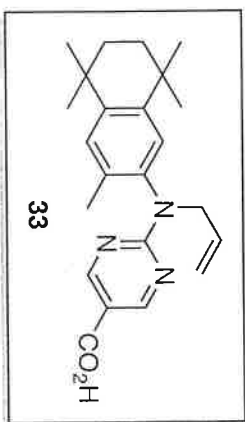
53.20
40.15
39.94
39.73
39.52
39.31
39.10
38.90
34.66
34.51
33.74
33.69
31.69
31.60
31.50
17.40



NAME CWVI-163
EXPNO 2
PROCNO 1
Date_ 20181010
Time 11.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 200
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 292.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.25 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL12 16.21 dB
PL12W 12.76071072 W
PL12W 0.34266910 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6128106 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

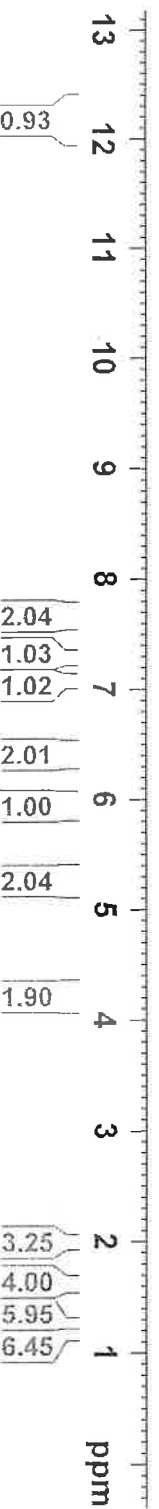
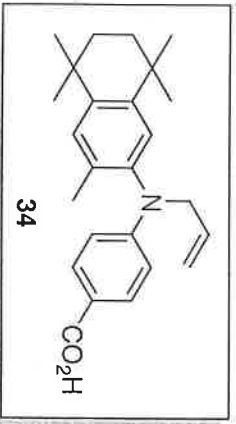


CWVI-197



12.146

7.700
7.678
7.287
7.075
6.426
6.404
5.991
5.978
5.965
5.952
5.948
5.935
5.922
5.909
5.895
5.304
5.300
5.261
5.257
5.210
5.206
5.184
5.180
4.211
2.509
2.504
2.500
2.495
2.491
1.988
1.980
1.628
1.257



NAME CWVI-197
EXPNO 1
PROCNO 1
Date_ 20181010
Time_ 20.29
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DW 60.800 usec
DE 6.50 usec
TE 292.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SF01 400.1324710 MHz
SI 32768
SF 400.1300034 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

CWVI-197

167.40
151.38
143.97
143.33
141.40
133.82
132.64
131.04
129.23
126.39
117.84
116.97
111.32

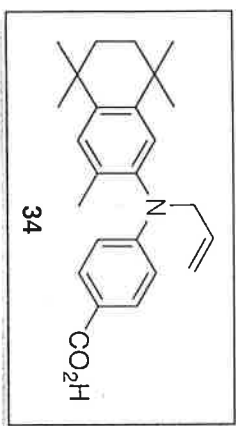
54.06
40.15
39.94
39.73
39.52
39.31
39.10
38.89
34.63
34.48
33.76
33.72
31.64
31.61
17.27



NAME CWVI-197
EXPNO 2
PROCNO 1
Date_ 20181010
Time 20.34
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 230
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 292.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.25 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL12 16.21 dB
PL2W 12.76071072 W
PL12W 0.34266910 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6128121 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

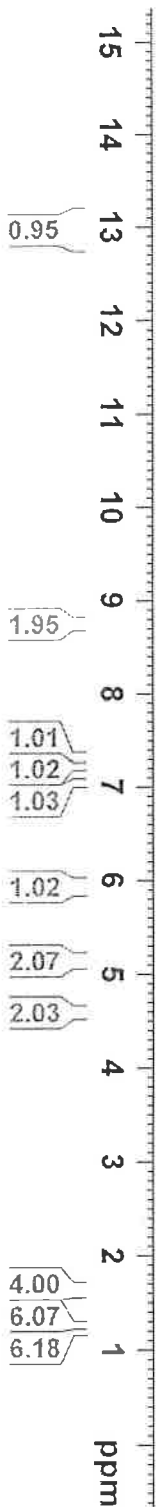
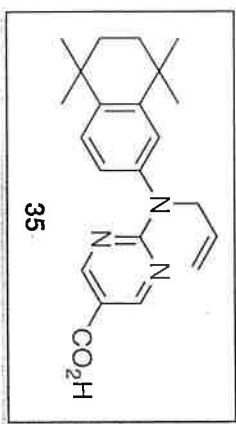


200 180 160 140 120 100 80 60 40 20 0 ppm

CWVI-161

— 12.924

8.759
7.339
7.318
7.231
7.225
7.053
7.048
7.032
7.027
5.991
5.977
5.965
5.952
5.948
5.938
5.934
5.921
5.909
5.895
5.165
5.161
5.132
5.128
5.122
5.117
5.106
5.102
4.588
4.575
2.509
2.504
2.500
2.495
2.491



```

NAME      CWVI-161
EXPNO     1
PROCNO    1
Date_     20181010
Time      10.36
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   DMSO
NS         16
DS         2
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         4
DW         60.800 usec
DE         6.50 usec
TE         292.0 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         14.75 usec
PL1        0.50 dB
PL1W       12.76071072 W
SFO1       400.1324710 MHz
SI         32768
SF         400.1300034 MHz
WDW        EM
SSB        0
GB         0
PC         1.00
  
```


CWVI-161

165.50
162.19
159.52

145.25
142.40
140.66

133.86

126.93
124.69
124.52

116.70
114.07

53.25
40.14
39.94
39.73
39.52
39.31
39.10
38.89
34.53
34.49
34.07
33.79
31.60
31.55



NAME CWVI-161

EXPNO 2

PROCNO 1

Date_ 20181010

Time_ 10.44

INSTRUM spect

PROBHD 5 mm PABBO BB-

PULPROG zgpg30

TD 65536

SOLVENT DMSO

NS 169

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 292.2 K

D1 2.00000000 sec

D11 0.03000000 sec

TD0 1

===== CHANNEL f1 =====

NUC1 13C

P1 8.25 usec

PL1 -2.10 dB

PL1W 60.29227829 W

SFO1 100.6228298 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16

NUC2 1H

PCPD2 90.00 usec

PL2 0.50 dB

PL12 16.21 dB

PL2W 12.76071072 W

PL12W 0.34266910 W

SFO2 400.1316005 MHz

SI 32768

SF 100.6128121 MHz

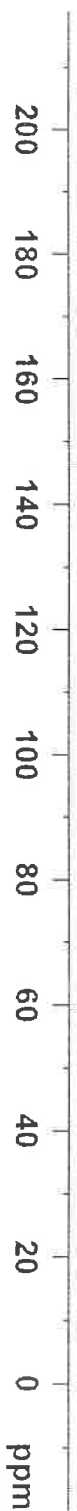
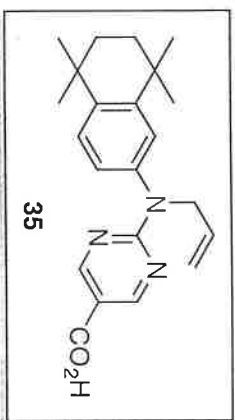
WDM EM

SSB 0

LB 1.00 Hz

GB 0

PC 1.40



CWVI-171

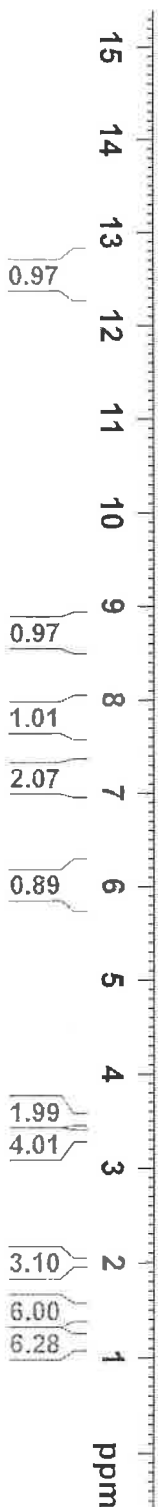
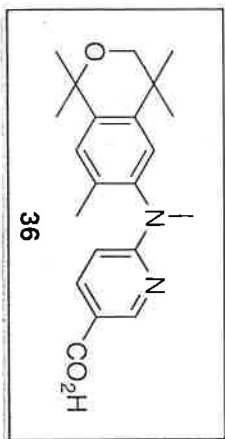
— 12.506

— 8.680

7.837
7.818
7.199
7.194

— 6.006

3.497
3.354
2.509
2.504
2.500
2.495
2.491
1.993
1.478
1.169



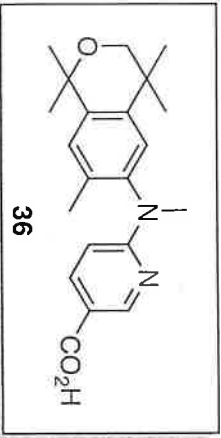
NAME CWVI-171
EXPNO 1
PROCNO 1
Date_ 20181010
Time_ 11.45
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DW 60.800 usec
DE 6.50 usec
TE 292.1 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300034 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



CWVI-171

166.77
159.99
150.69
142.21
141.50
140.88
137.99
132.76
128.31
124.41
114.70
106.04
74.58
69.78
40.15
39.94
39.73
39.52
39.31
39.11
38.90
37.67
33.48
29.63
26.70
16.94



200 180 160 140 120 100 80 60 40 20 0 ppm



NAME CWVI-171
EXPNO 2
PROCNO 1
Date_ 20181010
Time_ 11.50
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 663
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DE 20.800 usec
TE 292.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
===== CHANNEL f1 =====
NUC1 13C
P1 8.25 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.6228298 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL12 16.21 dB
PL12W 12.76071072 W
PL12W 0.34266910 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6128113 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

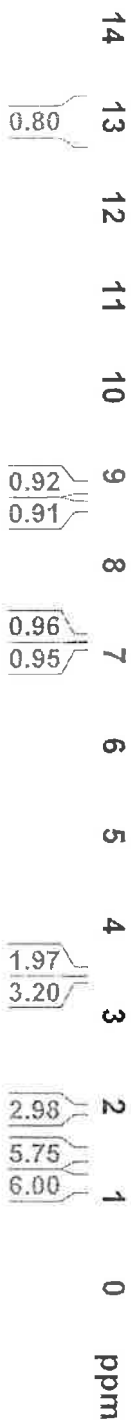
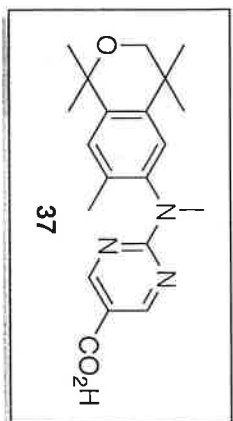
CWVI-157

12.889

8.877
8.664

7.197
7.112

3.498
3.485
3.413
2.500
1.965
1.485
1.458
1.179
1.167
1.153



NAME CWVI-157
EXPNO 1
PROCNO 1
Date_ 20181010
Time 10.18
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DW 60.800 usec
DE 6.50 usec
TE 291.9 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SF01 400.1324710 MHz
SI 32768
SF 400.1300034 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



CWVI-157

165.64
162.17
159.74
159.63

141.55
141.40
140.29
132.41
127.64
123.63
113.42

74.56
69.82

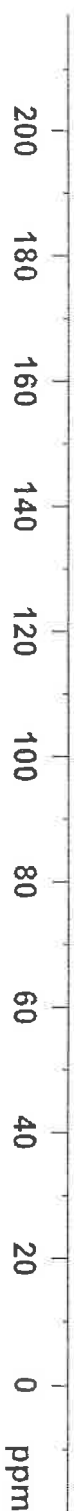
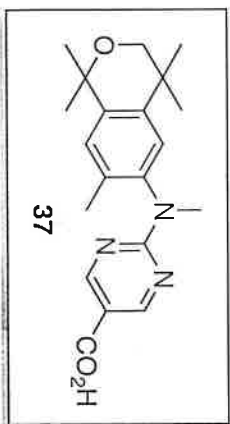
40.15
39.94
39.73
39.52
39.31
39.10
38.89
38.37
33.44
29.78
29.54
26.91
26.57
17.13



NAME CWVI-157
EXPNO 2
PROCNO 1
Date_ 20181010
Time 10.22
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 201
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 292.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.25 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL12 16.21 dB
PL2W 12.76071072 W
PL12W 0.34266910 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6128113 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

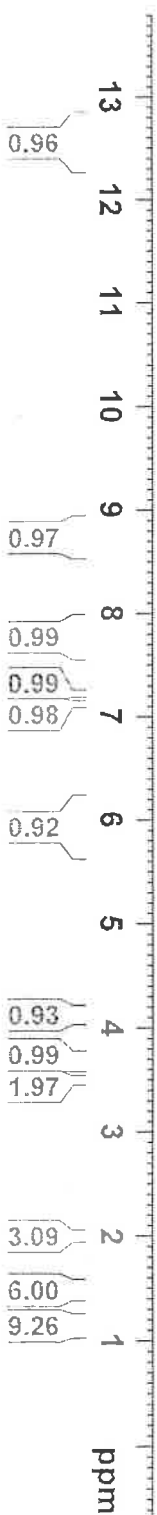
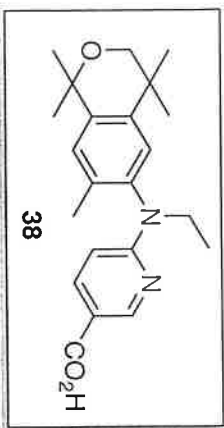


CWVI-169

— 12.480

8.675
8.670
7.819
7.813
7.796
7.791
7.214
7.128

— 5.911
4.113
4.098
3.677
3.661
3.644
3.628
3.501
2.509
2.504
2.500
2.495
2.491
1.995
1.487
1.473
1.171
1.155
1.138
1.120



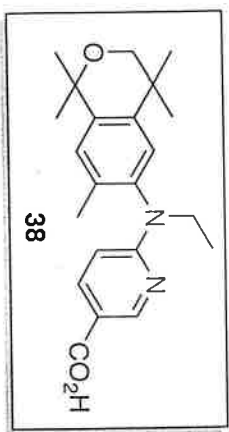
NAME CWVI-169
EXPNO 1
PROCNO 1
Date_ 20181010
Time_ 13.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DW 60.800 usec
DE 6.50 usec
TE 292.1 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300034 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



CWVI-169

166.74
159.48
150.78
142.04
140.94
139.72
138.10
133.18
128.37
125.43
114.65
106.00
74.58
69.76
44.23
40.15
39.94
39.73
39.53
39.32
39.11
38.90
33.40
29.64
26.70
17.05
12.84



200 180 160 140 120 100 80 60 40 20 0 ppm



NAME CWVI-169

EXPNO 2

PROCNO 1

Date 20181010

Time 13.20

INSTRUM spect

PROBHD 5 mm PABBO BB-

PULPROG zgpg30

TD 65536

SOLVENT DMSO

NS 443

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 292.4 K

D1 2.00000000 sec

D11 0.03000000 sec

TD0 1

===== CHANNEL f1 =====

NUC1 13C

P1 8.25 usec

PL1 -2.10 dB

PL1W 60.29227829 W

SFO1 100.6228298 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16

NUC2 1H

PCPD2 90.00 usec

PL2 0.50 dB

PL12 16.21 dB

PL2W 12.76071072 W

PL12W 0.34266910 W

SFO2 400.1316005 MHz

SI 32768

SF 100.6128113 MHz

WDW EM

SSB 0

LB 1.00 Hz

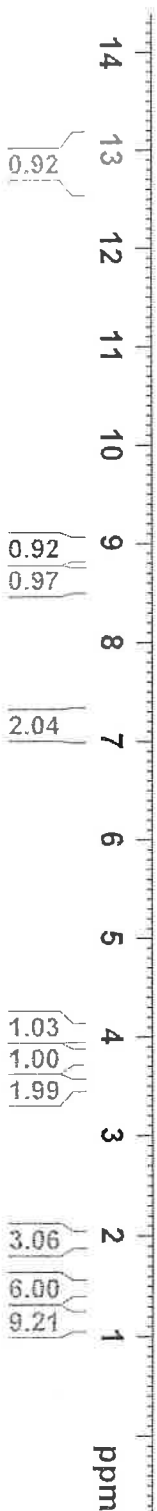
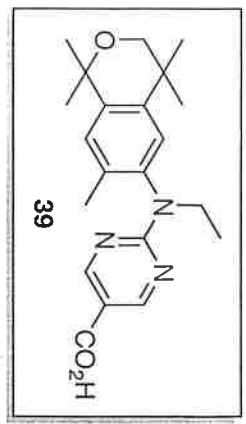
GB 0

PC 1.40

CWVI-165

12.860

- 8.868
- 8.657
- 8.652
- 7.125
- 7.119
- 4.069
- 4.051
- 4.034
- 4.028
- 4.017
- 4.010
- 3.999
- 3.982
- 3.835
- 3.817
- 3.800
- 3.783
- 3.765
- 3.748
- 3.527
- 3.498
- 3.492
- 3.464
- 2.509
- 2.504
- 2.500
- 2.495
- 2.491
- 1.966
- 1.489



```

NAME      CWVI-165
EXPNO     1
PROCNO    1
Date_     20181010
Time      12.28
INSTRUM    spect
PROBHD     5 mm PABBO BB-
PULPROG    zg30
TD         65536
SOLVENT    DMSO
NS         16
DS         2
SWH         8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         4
DW         60.800 usec
DE         6.50 usec
TE         292.1 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         14.75 usec
PL1        0.50 dB
PL1W       12.76071072 W
SFO1       400.1324710 MHz
SI         32768
SF         400.1300034 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```


CWVI-165

165.61
161.77
159.77

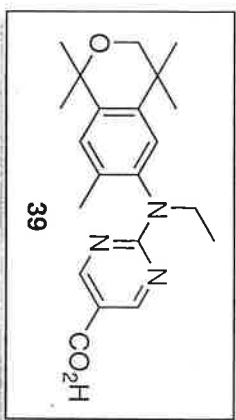
141.17
140.28
139.96
132.87
127.66
124.46

113.46

74.55
69.81

45.44

33.36
29.72
29.61
26.85
26.61
17.32
12.65



200 180 160 140 120 100 80 60 40 20 0 ppm

NAME CWVI-165
EXPNO 2
PROCNO 1
Date_ 20181010
Time_ 12.46
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 354
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 292.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.25 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.6228298 MHz

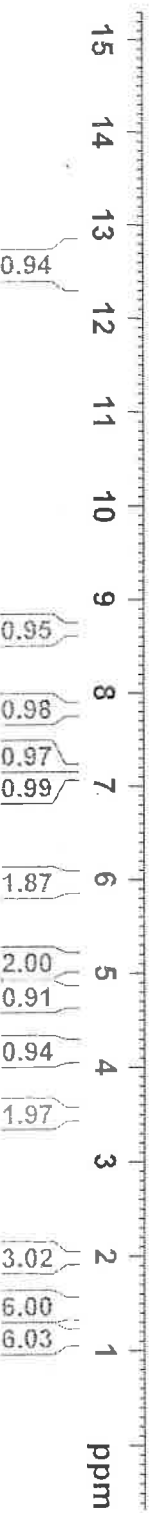
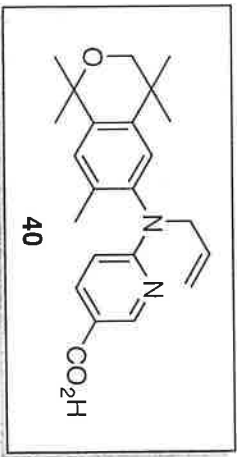
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL12 16.21 dB
PL2W 12.76071072 W
PL12W 0.34266910 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6128113 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

CWVI-175



— 12.529

- 8.675
- 8.671
- 7.856
- 7.851
- 7.834
- 7.828
- 7.194
- 7.125
- 6.024
- 6.009
- 5.998
- 5.994
- 5.983
- 5.966
- 5.956
- 5.951
- 5.941
- 5.925
- 5.162
- 5.159
- 5.129
- 5.125
- 5.120
- 5.116
- 5.104
- 4.744
- 4.182
- 3.494
- 2.509
- 2.504
- 2.500
- 2.495
- 2.491
- 1.994

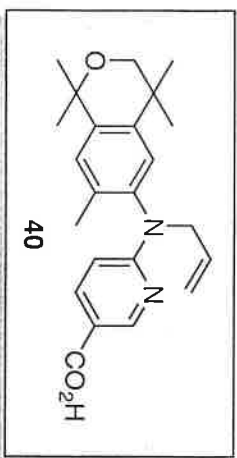


NAME CWVI-175
 EXPNO 1
 PROCNO 1
 Date_ 20181010
 Time_ 14.15
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 4
 DW 60.800 usec
 DE 6.50 usec
 TE 292.2 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 14.75 usec
 PL1 0.50 dB
 PL1W 12.76071072 W
 SFO1 400.1324710 MHz
 SI 32768
 SF 400.1300034 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

CWVI-175

166.68
159.43
150.66
141.81
140.90
139.88
138.27
134.11
133.10
128.25
125.37
117.44
115.09
106.15
74.56
69.71
52.23
40.15
39.94
39.73
39.52
39.31
39.10
38.90
33.39
29.63
26.69
17.16



200 180 160 140 120 100 80 60 40 20 0 ppm



NAME CWVI-175

EXPNO 2

PROCNO 1

Date 20181010

Time 14.22

INSTRUM spect

PROBHD 5 mm PABBO BB-

PULPROG zg1930

TD 65536

SOLVENT DMSO

NS 379

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 292.4 K

D1 2.00000000 sec

D11 0.03000000 sec

TD0 1

===== CHANNEL f1 =====

NUC1 13C

P1 8.25 usec

PL1 -2.10 dB

PL1W 60.29227829 W

SFO1 100.6228298 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16

NUC2 1H

PCPD2 90.00 usec

PL2 0.50 dB

PL12 16.21 dB

PL2W 12.76071072 W

PL12W 0.34266910 W

SFO2 400.1316005 MHz

SI 32768

SF 100.6128121 MHz

WDM EM

SSB 0

LB 1.00 Hz

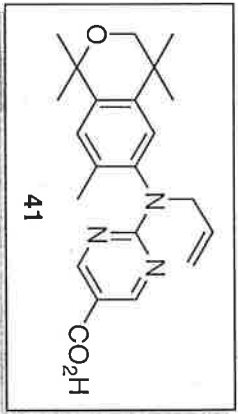
GB 0

PC 1.40

CWVI-167

12.909

8.877
8.686
7.109
6.032
6.016
6.005
6.001
5.990
5.975
5.963
5.959
5.948
5.933
5.174
5.170
5.154
5.151
5.128
4.738
4.723
4.700
4.686
4.285
4.268
4.247
4.230
3.522
3.493
3.483
3.454
2.509
2.504
2.500
2.495



NAME CWVI-167
EXPNO 1
PROCNO 1
Date_ 20181010
Time 12.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DW 60.800 usec
DE 6.50 usec
TE 292.2 K
D1 1.00000000 sec
TD0 1
===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300034 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

CWVI-167

165.55
161.83
159.80

140.93
140.26
140.05
133.53
132.84
127.58
124.40
117.94
113.84

74.54
69.76

53.16
40.15
39.94
39.73
39.52
39.32
39.11
38.90
33.34
29.68
29.63
26.78
26.66
17.41



NAME CWVI-167

EXPNO 2

PROCNO 1

Date 20181010

Time 13.04

INSTRUM spect

PROBHD 5 mm PABBO BB-

PULPROG zgpg30

TD 65536

SOLVENT DMSO

NS 161

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 292.4 K

D1 2.0000000 sec

D11 0.0300000 sec

TD0 1

===== CHANNEL f1 =====

NUC1 13C

P1 8.25 usec

PL1 -2.10 dB

PL1W 60.29227829 W

SFO1 100.6228298 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16

NUC2 1H

PCPD2 90.00 usec

PL2 0.50 dB

PL12 16.21 dB

PL2W 12.76071072 W

PL12W 0.34266910 W

SFO2 400.1316005 MHz

SI 32768

SF 100.6128113 MHz

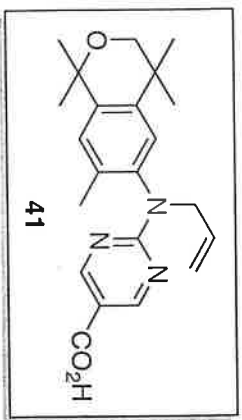
WDW EM

SSB 0

LB 1.00 Hz

GB 0

PC 1.40



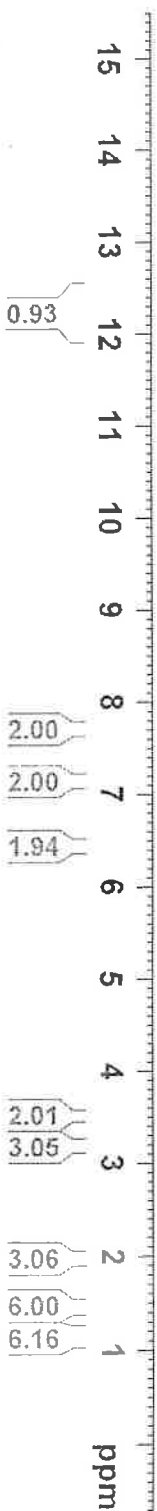
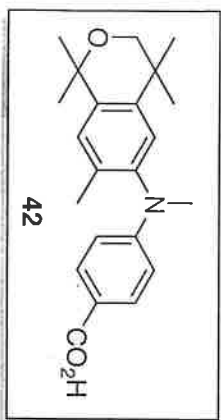
200 180 160 140 120 100 80 60 40 20 0 ppm

CWVI-201

12.165

7.729
7.707
7.166
7.121
6.447
6.426

3.495
3.215
2.509
2.504
2.500
2.495
2.491
1.984
1.476
1.160



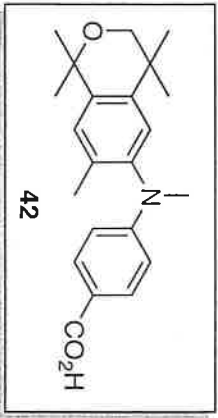
NAME CWVI-201
EXPNO 1
PROCNO 1
Date_ 20181010
Time_ 22.23
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DW 60.800 usec
DE 6.50 usec
TE 292.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300034 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



CWVI-201

167.45
151.99
143.13
142.08
140.34
132.97
131.08
128.26
124.39
117.80
111.08
74.58
69.79
40.15
39.94
39.73
39.52
39.32
39.11
38.90
33.45
29.67
26.73
17.09



200 180 160 140 120 100 80 60 40 20 0 ppm

NAME CWVI-201
EXPNO 2
PROCNO 1
Date_ 20181010
Time_ 22.29
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 14728
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DM 20.800 usec
DE 6.50 usec
TE 292.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.25 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.6228298 MHz

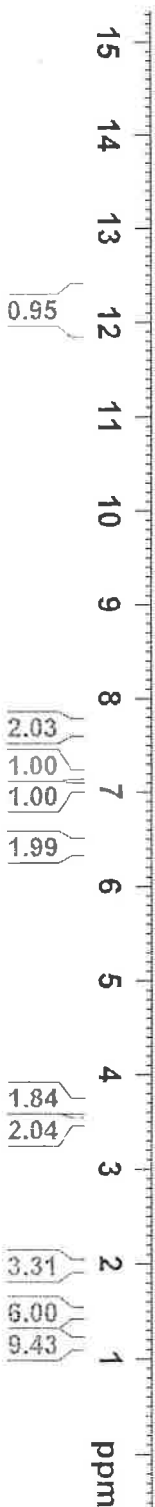
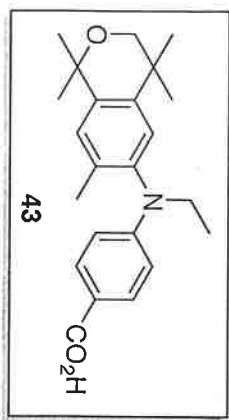
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL12 16.21 dB
PL2W 12.76071072 W
PL12W 0.34266910 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6128120 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

CWVI-199

12.125

7.707
7.684
7.186
7.073
6.418
6.396

3.636
3.501
2.509
2.504
2.500
2.495
2.491
1.982
1.479
1.165
1.148
1.130



NAME CWVI-199
EXPNO 1
PROCNO 1
Date_ 20181010
Time_ 22.03
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DM 60.800 usec
DE .6.50 usec
TE 292.2 K
D1 1.00000000 sec
TD0 1
===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SF01 400.1324710 MHz
SI 32768
SF 400.1300034 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

CWVI-199

167.41
151.10
141.96
141.37
140.43
133.42
131.24
128.30
125.51
117.48
110.84
74.58
69.76
45.62
40.15
39.94
39.73
39.52
39.31
39.10
38.89
33.38
29.67
26.72
17.19
12.36



NAME CWVI-199

EXPNO 2

PROCNO 1

Date_ 20181010

Time_ 22.10

INSTRUM spect

PROBHD 5 mm PABBO BB-

PULPROG zgpg30

TD 65536

SOLVENT DMSO

NS 225

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 292.4 K

D1 2.00000000 sec

D11 0.03000000 sec

TD0 1

===== CHANNEL f1 =====

NUC1 13C

P1 8.25 usec

PL1 -2.10 dB

PL1W 60.29227829 W

SFO1 100.6228298 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16

NUC2 1H

PCPD2 90.00 usec

PL2 0.50 dB

PL12 16.21 dB

PL2W 12.76071072 W

PL12W 0.34266910 W

SFO2 400.1316005 MHz

SI 32768

SF 100.6128121 MHz

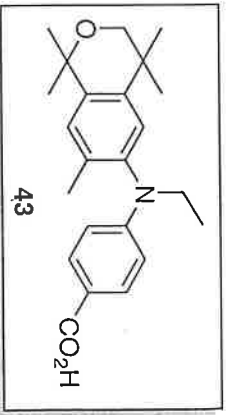
WDW EM

SSB 0

LB 1.00 Hz

GB 0

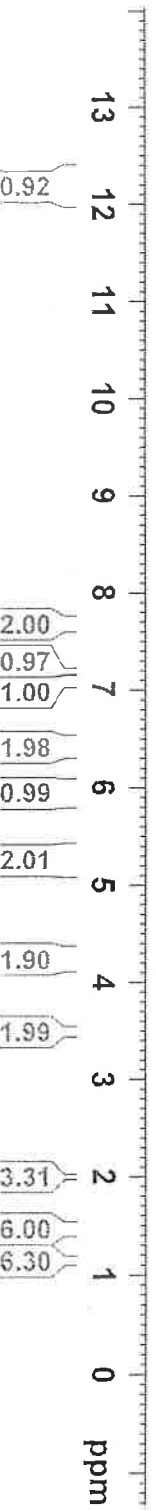
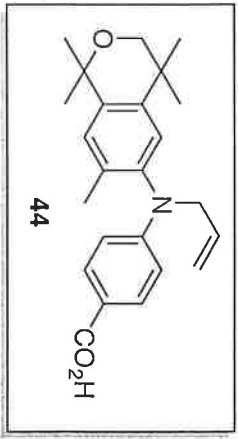
PC 1.40



CWVI-203

— 12.171

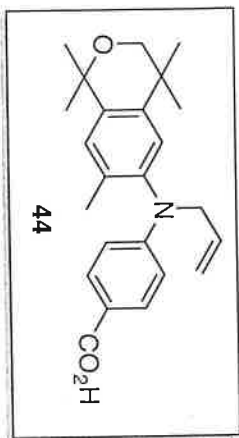
7.708
7.686
7.180
7.114
6.434
6.412
5.997
5.984
5.971
5.958
5.955
5.941
5.928
5.915
5.902
5.315
5.311
5.272
5.268
5.217
5.213
5.191
5.187
4.230
3.495
2.509
2.504
2.500
2.495
2.491
1.999
1.476
1.151



NAME CWVI-203
EXPNO 1
PROCNO 1
Date_ 20181011
Time_ 12.23
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DE 60.800 usec
TE 291.9 K
D1 1.00000000 sec
TD0 1
===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300034 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

CWVI-203

167.38
151.29
142.01
141.87
140.42
133.82
133.19
131.06
128.31
125.19
118.01
117.08
111.42
74.57
69.74
54.11
40.15
39.94
39.73
39.52
39.31
39.10
38.90
33.39
29.66
26.71
17.31



200 180 160 140 120 100 80 60 40 20 0 ppm

NAME CWVI-203
EXENO 2
PROCNO 1
Date 20181011
Time 13.50
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1515
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 292.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.25 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL12 16.21 dB
PL12W 12.76071072 W
PL12W 0.34266910 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6128121 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

CWVI-077 Product (6-bromo-1,1,4,4,7-pentamethyl-1-isochroman)

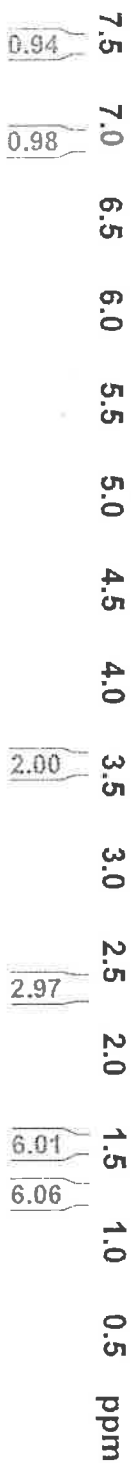
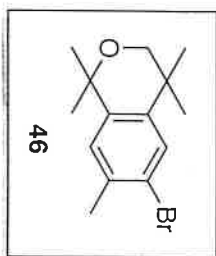
7.433
7.260
6.917

3.557

2.353

1.509

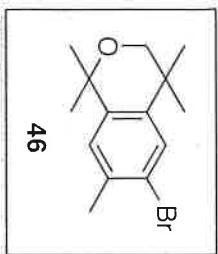
1.246



NAME CWVI-077
EXPNO 5
PROCNO 1
Date 20170721
Time 16.47
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DW 60.800 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SF01 400.1324710 MHz
SI 32768
SF 400.1300101 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

CWVI-077 Product (6-bromo-1,1,4,4,7-pentamethyl-1-isochroman)



142.21
140.89
135.08
129.03
127.65
122.76

77.31
77.00
76.68
74.85
70.66

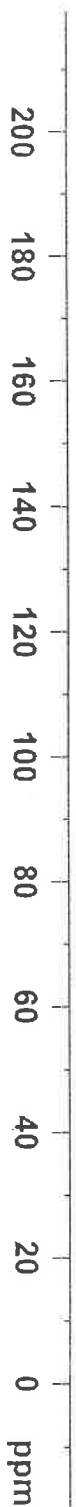
33.73
29.66
26.90
22.60



NAME CWVI-077
EXPNO 6
PROCNO 1
Date_ 20170721
Time_ 16.53
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 91
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 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

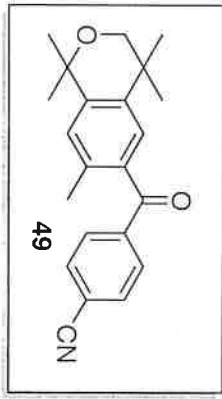
===== CHANNEL f1 =====
NUC1 13C
P1 8.50 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL12 16.21 dB
PL2W 12.76071072 W
PL12W 0.34266910 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127722 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

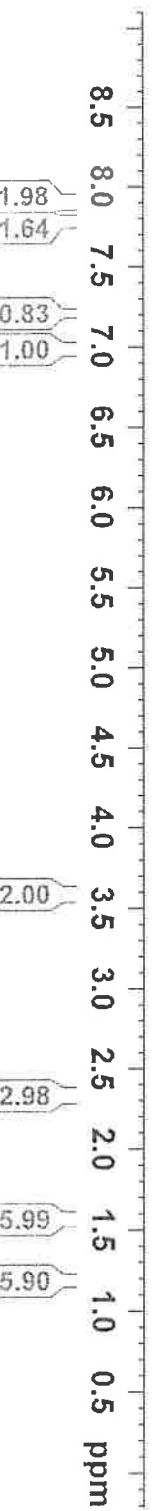


CWVI-111

7.902
7.888
7.886
7.881
7.783
7.762
7.260
7.211
6.990



3.589
2.317
1.558
1.201



NAME CWVI-111
EXPNO 1
PROCNO 1
Date_ 20180706
Time_ 16.16
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DM 60.800 usec
DE 6.50 usec
TE 293.9 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

CWVI-111

196.55

145.14
141.37
139.90
134.99
134.86
132.25
130.35
128.32
126.57
117.97
116.10

77.32
77.00
76.68
75.09
70.61

33.59
29.52
26.86
20.03



CWVI-111

NAME CWVI-111

EXPNO 2

PROCNO 1

Date 20180706

Time 16.23

INSTRUM spect

PROBHD 5 mm PABBO BB-

PULPROG zgpg30

TD 65536

SOLVENT CDCl3

NS 60

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 294.1 K

D1 2.0000000 sec

D11 0.0300000 sec

TD0 1

===== CHANNEL f1 =====

NUC1 13C

P1 8.25 usec

PL1 -2.10 dB

PL1W 60.29227829 W

SFO1 100.6228298 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16

NUC2 1H

PCPD2 90.00 usec

PL2 0.50 dB

PL12 16.21 dB

PL12W 12.76071072 W

PL12W 0.34266910 W

SFO2 400.1316005 MHz

SI 32768

SF 100.6127744 MHz

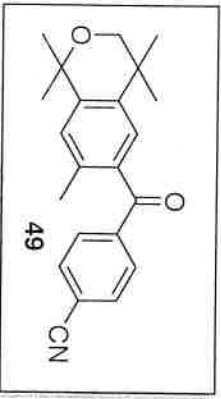
WDW EM

SSB 0

LB 1.00 Hz

GB 0

PC 1.40



49

200 180 160 140 120 100 80 60 40 20 0 ppm

CWVI-139

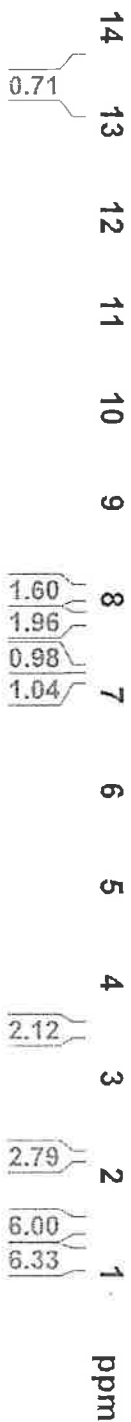
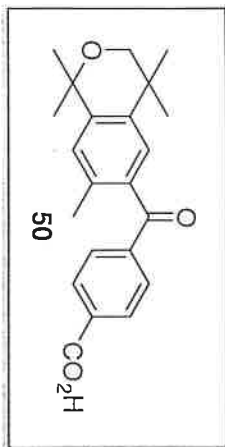
13.356

8.094
8.073
7.840
7.799
7.779
7.297
7.272
7.220
7.210

3.509

2.500
2.212

1.493
1.140



NAME CWVI-139
EXPNO 1
PROCNO 1
Date_ 20181010
Time_ 21.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DW 60.800 usec
DE 6.50 usec
TE 292.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300031 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

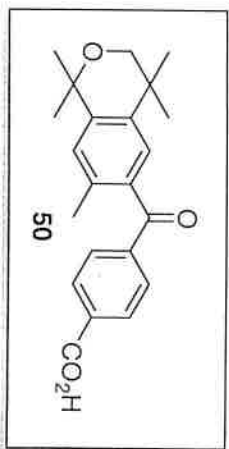


CWVI-139

197.07
166.68
144.37
140.62
139.53
135.63
134.60
133.75
129.80
129.69
128.04
125.87

74.69
69.73

40.15
39.94
39.73
39.52
39.31
39.10
38.90
33.24
29.39
26.52
19.46



200
180
160
140
120
100
80
60
40
20
0
ppm



NAME CWVI-139

EXPNO 2

PROCNO 1

Date 20181010

Time 21.40

INSTRUM spect

PROBHD 5 mm PABBO BB-

PULPROG zgpg30

TD 65536

SOLVENT DMSO

NS 74

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 292.4 K

D1 2.00000000 sec

D11 0.03000000 sec

TD0 1

===== CHANNEL f1 =====

NUC1 13C

P1 8.25 usec

PL1 -2.10 dB

PL1W 60.29227829 W

SFO1 100.6228298 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16

NUC2 1H

PCPD2 90.00 usec

PL2 0.50 dB

PL12 16.21 dB

PL2W 12.76071072 W

PL12W 0.34266910 W

SFO2 400.1316005 MHz

SI 32768

SF 100.6128113 MHz

WDW EM

SSB 0

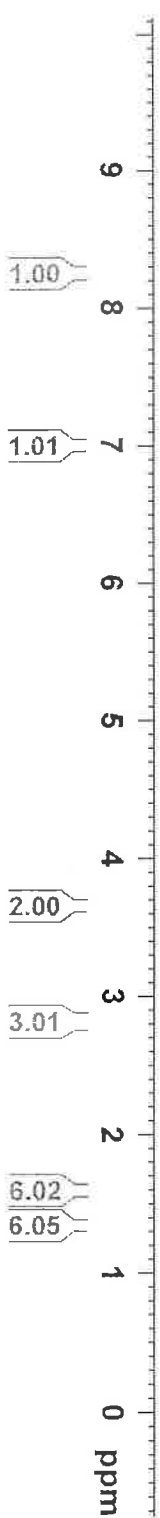
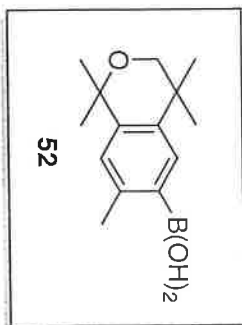
LB 1.00 Hz

GB 0

PC 1.40

CWVI-129

8.254
7.260
6.993
3.645
2.824
1.588
1.335



NAME CWVI-129
EXPNO 1
PROCNO 1
Date_ 20180725
Time_ 14.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DM 60.800 usec
DE 6.50 usec
TE 294.6 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SF01 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



CWVI-129

146.01
143.24
139.18
134.79
127.38
127.19

77.32
77.20
77.00
76.68
75.23
70.89

33.54
29.53
27.03
26.98
22.65



NAME CWVI-129

EXPNO 3

PROCNO 1

Date 20180727

Time 15.19

INSTRUM spect

PROBHD 5 mm PABBO BB-

PULPROG zgpg30

TD 65536

SOLVENT CDCl3

NS 5359

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 295.7 K

D1 2.00000000 sec

D11 0.03000000 sec

TD0 1

===== CHANNEL f1 =====

NUC1 13C

P1 8.25 usec

PL1 -2.10 dB

PL1W 60.29227829 W

SFO1 100.6228298 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16

NUC2 1H

PCPD2 90.00 usec

PL2 0.50 dB

PL12 16.21 dB

PL2W 12.76071072 W

PL12W 0.34266910 W

SFO2 400.1316005 MHz

SI 32768

SF 100.6127729 MHz

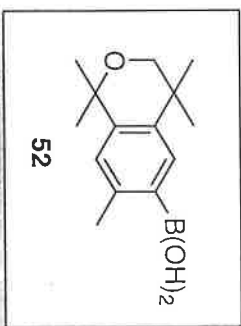
WDW EM

SSB 0

LB 1.00 Hz

GB 0

PC 1.40



52

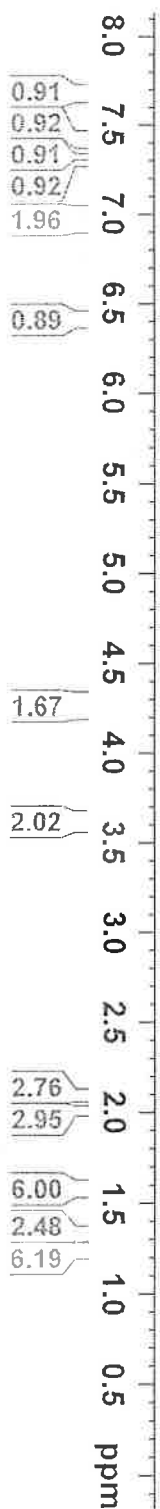
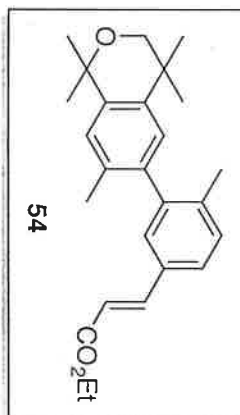


CWVI-131 (pure)

7.434
7.419
7.415
7.322
7.317
7.295
7.275
7.260
7.000
6.935
6.433
6.393

4.275
4.257
4.240
4.222
3.790
3.614

2.089
2.013
1.578
1.338
1.320
1.302
1.257
1.233



NAME CWVI-131
EXPNO 1
PROCNO 1
Date_ 20180712
Time_ 10.23
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DM 60.800 usec
DE 6.50 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1

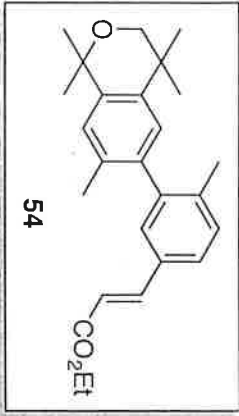
===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



CWVI-131 (pure)



167.14
144.48
142.16
140.58
139.81
138.88
138.60
132.81
131.91
130.44
129.19
126.79
126.53
125.91
117.44
77.32
77.00
76.69
75.07
70.93
60.37
33.61
29.89
29.84
27.10
27.00
20.02
19.57
14.30



200 180 160 140 120 100 80 60 40 20 0 ppm

```

NAME CWVI-131
EXPNO 2
PROCNO 1
Date_ 20180712
Time_ 10.32
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 143
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DM 20.800 usec
DE 6.50 usec
TE 295.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.25 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.6228298 MHz

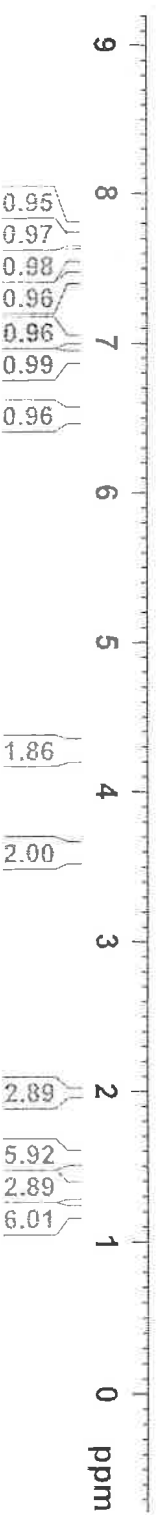
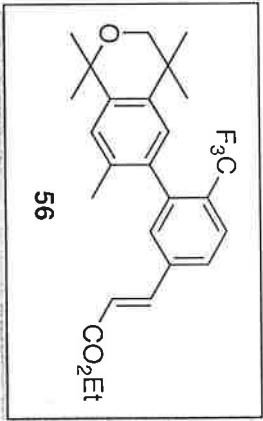
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL12 16.21 dB
PL12W 12.76071072 W
PL12W 0.34266910 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127729 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
  
```

CWVI-135

7.776
7.755
7.706
7.666
7.599
7.579
7.431
7.260
7.026
6.911
6.533
6.493

4.289
4.271
4.253
4.235
3.606
3.600

1.991
1.568
1.341
1.323
1.305
1.236
1.208



NAME CWVI-135
EXPNO 1
PROCNO 1
Date_ 20180712
Time_ 10.40
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DM 60.800 usec
DE 6.50 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

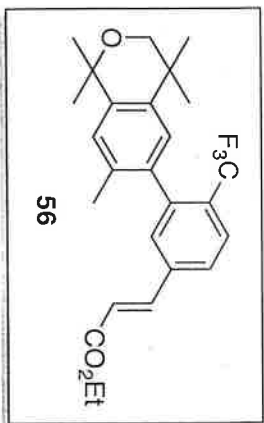


CWVI-135

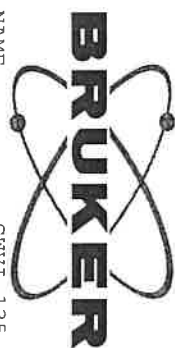
166.37
142.54
141.48
141.45
141.27
139.04
137.26
135.98
132.87
131.03
130.18
129.88
126.73
126.68
126.54
126.37
126.24
125.00
122.28
121.05

77.31
77.00
76.68
75.04
70.88
60.74

33.50
30.00
29.64
27.09
26.65
19.80
14.23



200 180 160 140 120 100 80 60 40 20 0 ppm



NAME CWVI-135
EXPNO 2
PROCNO 1
Date_ 20180712
Time 10.48
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 363
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 295.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.25 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.6228298 MHz

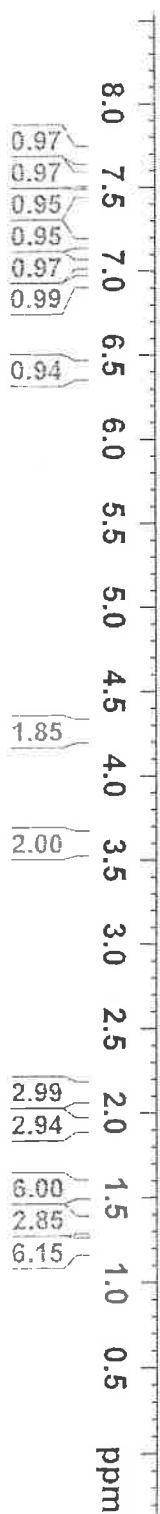
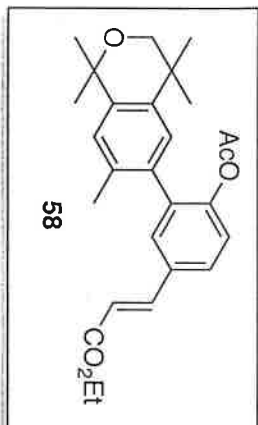
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL12 16.21 dB
PL12W 12.76071072 W
PL12W 0.34266910 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127722 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

CWVI-133

7.555
7.550
7.534
7.529
7.466
7.461
7.260
7.178
7.157
7.044
6.939
6.428
6.388

4.284
4.266
4.249
4.231
3.605
3.593

2.112
1.929
1.557
1.345
1.328
1.310
1.232



NAME CWVI-133
EXPNO 1
PROCNO 1
Date_ 20180717
Time_ 16.24
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DM 60.800 usec
DE 6.50 usec
TE 294.7 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SF01 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

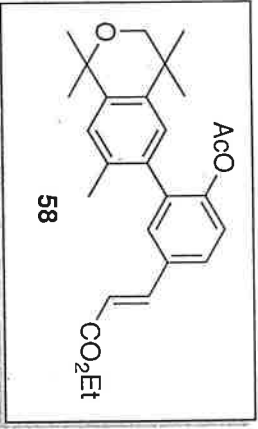


CWVI-133

169.03
166.83
149.80
143.44
141.24
139.57
135.32
134.09
133.38
132.32
130.94
127.97
126.74
126.60
123.18
118.65

77.32
77.00
76.68
75.00
70.88
60.54

33.54
20.49
19.67
14.27



200 180 160 140 120 100 80 60 40 20 0 ppm



NAME CWVI-133

EXPNO 2

PROCNO 1

Date 20180717

Time 16.31

INSTRUM spect

PROBHD 5 mm PABBO BB-

PULPROG zgpg30

TD 65536

SOLVENT CDCl3

NS 128

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 295.0 K

D1 2.0000000 sec

D11 0.03000000 sec

TD0 1

===== CHANNEL f1 =====

NUC1 13C

P1 8.25 usec

PL1 -2.10 dB

PL1W 60.29227829 W

SFO1 100.6228298 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16

NUC2 1H

PCPD2 90.00 usec

PL2 0.50 dB

PL12 16.21 dB

PL2W 12.76071072 W

PL12W 0.34266910 W

SFO2 400.1316005 MHz

SI 32768

SF 100.6127736 MHz

WDW EM

SSB 0

LB 1.00 Hz

GB 0

PC 1.40

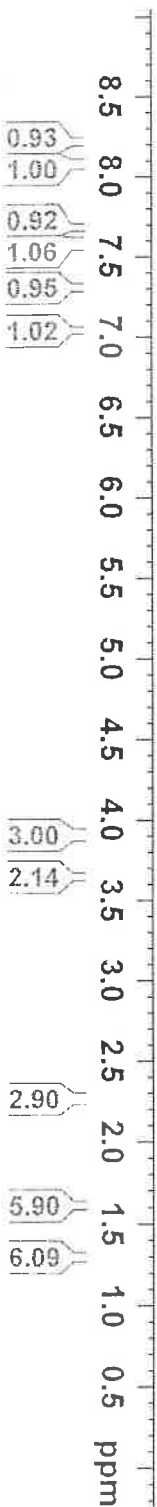
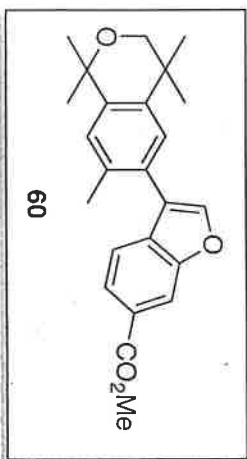
CWVI-137

8.214
8.086
8.082
8.064
8.060
7.684
7.590
7.589
7.568
7.567
7.301
7.260
7.039

3.918
3.642

2.266

1.607
1.297

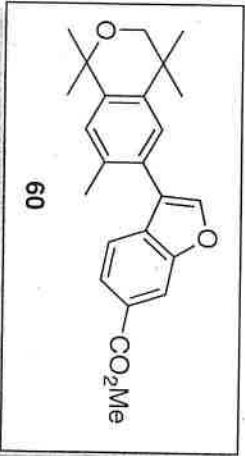


NAME CWVI-137
EXPNO 1
PROCNO 1
Date_ 20180717
Time_ 16.39
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DW 60.800 usec
DE 6.50 usec
TE 294.7 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

CWVI-137

167.21
157.61
143.29
141.57
140.25
134.11
128.15
128.02
127.33
127.21
126.15
125.22
123.28
121.92
111.51
77.32
77.00
76.68
75.07
70.88
52.11
33.62
29.77
27.02
20.24



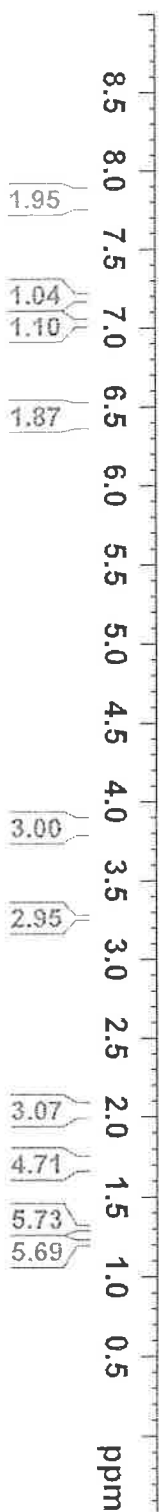
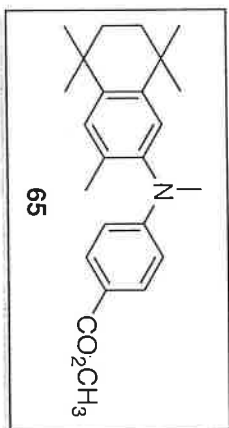
200 180 160 140 120 100 80 60 40 20 0 ppm



NAME CWVI-137
EXPNO 2
PROCNO 1
Date 20180717
Time 16.43
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 82
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DE 20.800 usec
TE 295.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
===== CHANNEL f1 =====
NUC1 13C
P1 8.25 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.6228298 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL12 16.21 dB
PL2W 12.76071072 W
PL12W 0.34266910 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127736 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

CWVI-185

7.852
7.830
7.260
7.189
7.034
6.478
6.456
3.840
3.264
2.036
1.687
1.656
1.308
1.224



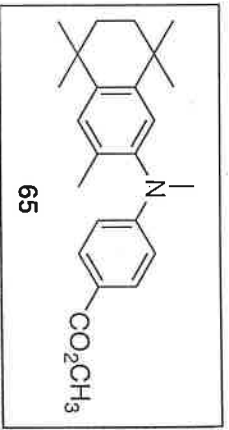
NAME CWVI-185
EXPNO 1
PROCNO 1
Date_ 20180828
Time 14.02
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DW 60.800 usec
DE 6.50 usec
TE 293.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

CWVI-185



167.44
152.59
144.59
143.84
142.60
132.79
131.13
129.30
125.88
117.24
111.15
77.31
77.00
76.68
51.46
39.12
35.07
34.96
34.11
33.99
31.86
17.38



200
180
160
140
120
100
80
60
40
20
0
ppm

NAME CWVI-185
EXPNO 2
PROCNO 1
Date_ 20180828
Time 14.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 102
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 293.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
===== CHANNEL f1 =====
NUC1 13C
P1 8.25 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.6228298 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL12 16.21 dB
PL12W 12.76071072 W
PL12W 0.3426910 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127744 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

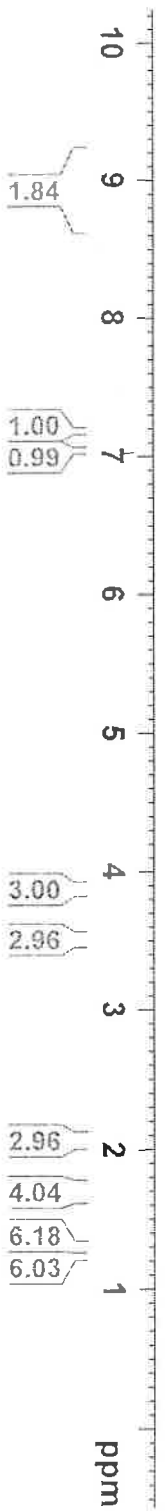
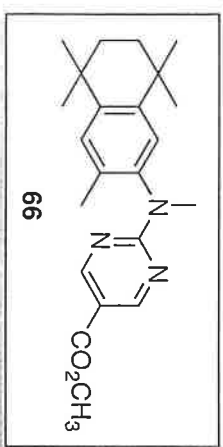
CWVI-125

8.974
8.967
8.817
8.810

7.260
7.191
7.045

3.869
3.500

2.057
1.680
1.674
1.319
1.276
1.252
1.240



```

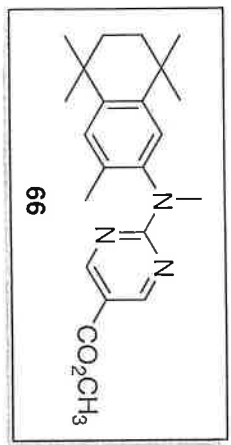
NAME CWVI-125
EXPNO 1
PROCNO 1
Date_ 20180725
Time 20.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DE 60.800 usec
TE 295.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SF01 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

```

CWVI-125

- 165.11
- 161.97
- 160.21
- 159.18
- 144.23
- 144.19
- 140.49
- 131.59
- 129.14
- 124.71
- 112.77
- 77.32
- 77.00
- 76.68
- 51.80
- 39.07
- 35.04
- 34.94
- 34.11
- 34.03
- 32.07
- 31.96
- 31.82
- 31.61
- 17.45



NAME CWVI-125
 EXPNO 2
 PROCNO 1
 Date 20180725
 Time 21.02
 INSTRUM spect
 PROBD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 166
 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 295.2 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.25 usec
 PL1 -2.10 dB
 PL1W 60.29227829 W
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 0.50 dB
 PL12 16.21 dB
 PL2W 12.76071072 W
 PL12W 0.34266910 W
 SFO2 400.1316005 MHz
 SI 32768
 SF 100.6127736 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



CWVI-093

8.884

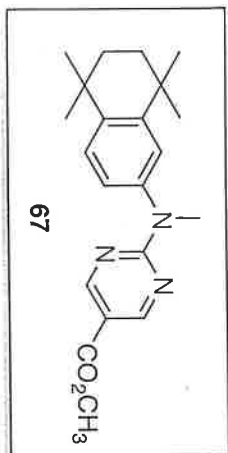
7.351
7.330
7.260
7.200
7.194
7.071
7.065
7.050
7.044

3.875

3.587

1.695

1.299
1.273



9
8
7
6
5
4
3
2
1
ppm

1.90

1.00

0.96

0.91

3.00

2.83

3.78

11.61

NAME CWVI-093
EXPNO 1
PROCNO 1
Date_ 20180725
Time_ 14.24
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG 2930
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DE 60.800 usec
TE 294.6 K
D1 1.00000000 sec
TD0 1
===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

CWVI-093

165.10
162.25
159.54

145.98
143.18
141.51

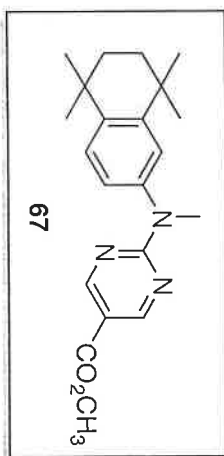
127.36
124.01
123.20

113.15

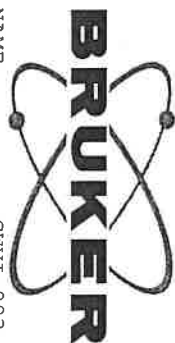
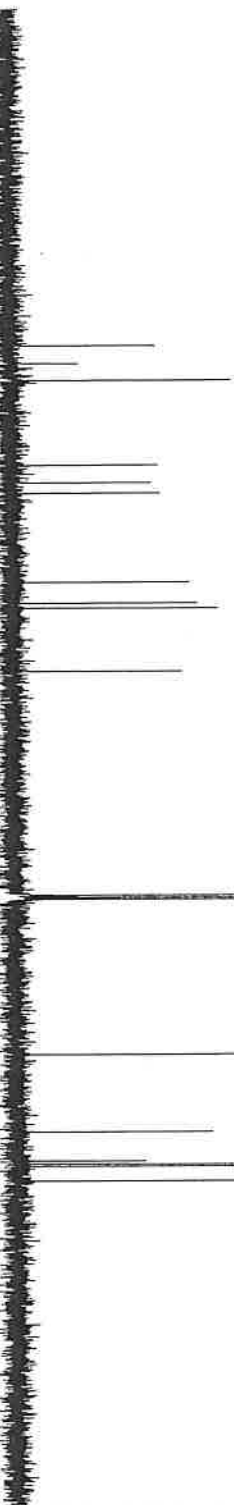
77.32
77.00
76.69

51.81

39.56
34.96
34.93
34.43
34.11
31.79



200 180 160 140 120 100 80 60 40 20 0 ppm



NAME CWVI-093

EXPNO 2

PROCNO 1

Date 20180725

Time 14.27

INSTRUM spect

PROBHD 5 mm PABBO BB-

PULPROG zgpg30

TD 65536

SOLVENT CDCl3

NS 100

DS 4

SWH 24038.461 Hz

FTDRES 0.366798 Hz

AQ 1.3631988 sec

RG 2050

DW 20.800 usec

DE 6.50 usec

TE 294.8 K

D1 2.0000000 sec

D11 0.0300000 sec

TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.25 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL12 16.21 dB
PL12W 12.76071072 W
PL12W 0.34266910 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127736 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

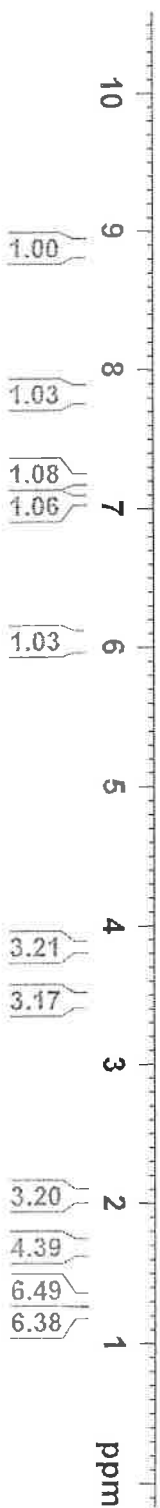
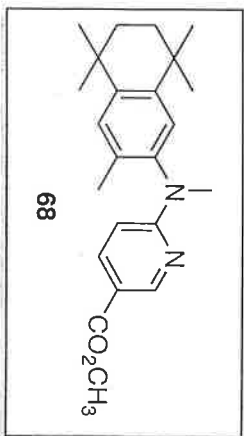
CWVI-079

8.872
8.871
8.867
8.866
7.834
7.828
7.811
7.806
7.260
7.210
7.054

6.048
6.025

3.854
3.462

2.054
1.687
1.304
1.230

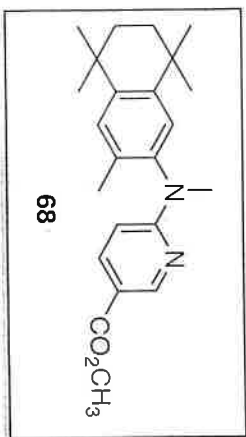


NAME: CWVI-079
EXPNO: 1
PROCNO: 1
Date_ : 20170906
Time: 12.53
INSTRUM: spect
PROBHD: 5 mm PABBO BB-
PULPROG: zg30
TD: 65536
SOLVENT: CDCl3
NS: 16
DS: 2
SWH: 8223.685 Hz
FIDRES: 0.125483 Hz
AQ: 3.9846387 sec
RG: 4
DW: 60.800 usec
DE: 6.50 usec
TE: 298.2 K
D1: 1.00000000 sec
TD0: 1

===== CHANNEL f1 =====
NUC1: 1H
P1: 14.75 usec
PL1: 0.50 dB
PL1W: 12.76071072 W
SFO1: 400.1324710 MHz
SI: 32768
SF: 400.1300101 MHz
WDW: EM
SSB: 0
LB: 0.30 Hz
GB: 0
PC: 1.00

CWVI-079

166.41
160.16
150.47
145.00
144.75
140.67
137.76
132.36
129.56
125.65
114.24
107.21
77.32
77.00
76.68
51.62
38.09
35.03
34.90
34.16
34.06
31.84
17.16



200 180 160 140 120 100 80 60 40 20 0 ppm

NAME CWVI-079
EXPNO 2
PROCNO 1
Date_ 20170906
Time_ 13.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SMH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

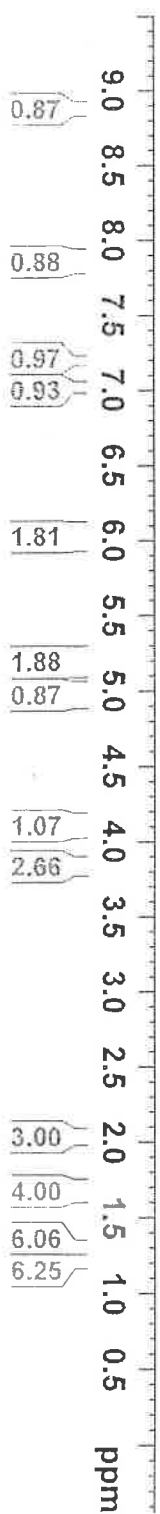
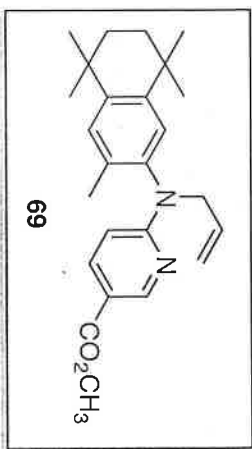
===== CHANNEL f1 =====
NUC1 13C
P1 8.50 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL2W 16.21 dB
PL12W 12.76071072 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127722 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

CWVI-083

8.866
8.862
7.867
7.862
7.844
7.840
7.260
7.202
7.015
6.087
6.072
6.061
6.056
6.046
6.029
6.019
6.013
6.003
5.988
5.218
5.178
5.155
4.987
4.963
4.120
3.852

2.038
1.675
1.298
1.288
1.206

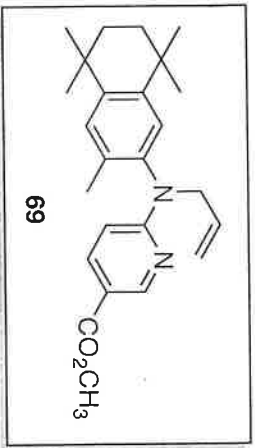


NAME CWVI-083
EXPNO 1
PROCNO 1
Date_ 20180725
Time_ 21.31
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
F2 16
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DW 60.800 usec
DE 6.50 usec
TE 294.8 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

CWVI-083

166.08
158.97
149.85
144.92
144.67
138.83
138.43
133.08
132.41
129.48
126.72
117.94
114.60
107.67
77.31
77.00
76.68
53.09
51.72
34.99
34.81
34.08
34.04
31.79
17.34



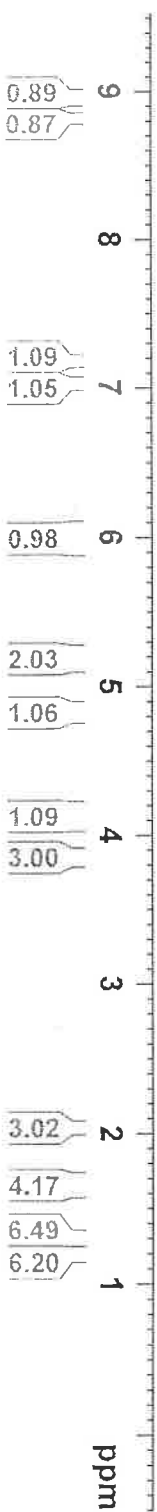
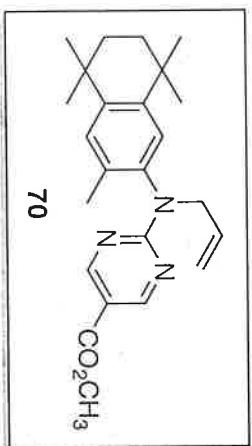
200 180 160 140 120 100 80 60 40 20 0 ppm

NAME CWVI-083
EXPNO 2
PROCNO 1
Date_ 20180725
Time_ 21.38
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg1930
TD 65536
SOLVENT CDCl3
NS 12624
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DE 20.800 usec
TE 295.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
===== CHANNEL f1 =====
NUC1 13C
P1 8.25 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.6228298 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL12 16.21 dB
PL12W 12.76071072 W
PL12W 0.34266910 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127736 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



CWVI-127

- 8.951
- 8.816
- 7.260
- 7.176
- 7.028
- 6.019
- 6.016
- 6.009
- 5.995
- 5.992
- 5.203
- 5.199
- 5.196
- 5.194
- 5.191
- 5.188
- 5.163
- 5.160
- 5.156
- 4.857
- 4.854
- 4.846
- 4.843
- 4.840
- 4.822
- 4.819
- 4.816
- 4.809
- 4.805
- 4.802
- 4.155
- 4.138
- 4.118
- 4.115
- 4.101
- 3.862
- 2.042
- 1.671
- 1.665
- 1.662
- 1.315
- 1.268
- 1.231
- 1.218



===== CHANNEL f1 =====

NUC1	1H
P1	14.75 usec
PL1	0.50 dB
PL1W	12.76071072 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300099 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

NAME CWVI-127

EXPNO 1

PROCNO 1

Date_ 20180725

Time_ 12.08

INSTRUM spect

PROBHD 5 mm PABBO BB-

PULPROG zg30

TD 65536

SOLVENT CDCl3

NS 16

DS 2

SWH 8223.685 Hz

FIDRES 0.125483 Hz

AQ 3.9846387 sec

RG 4

DW 60.800 usec

DE 6.50 usec

TE 294.3 K

D1 1.00000000 sec

TD0 1

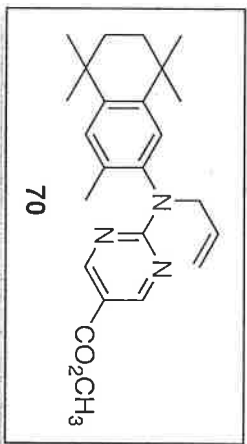


CWVI-127

165.24
162.02
160.15
159.67
144.02
143.70
139.25
133.00
131.92
128.94
125.69
117.99
112.99

77.31
77.00
76.68

53.92
51.74
35.07
34.92
34.04
33.99
32.01
31.93
31.72
31.60
17.68



200 180 160 140 120 100 80 60 40 20 0 ppm

NAME CWVI-127
EXPNO 2
PROCNO 1
Date_ 20180725
Time_ 12.22
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 302
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 294.5 K
D1 2.0000000 sec
D11 0.03000000 sec
TD0 1

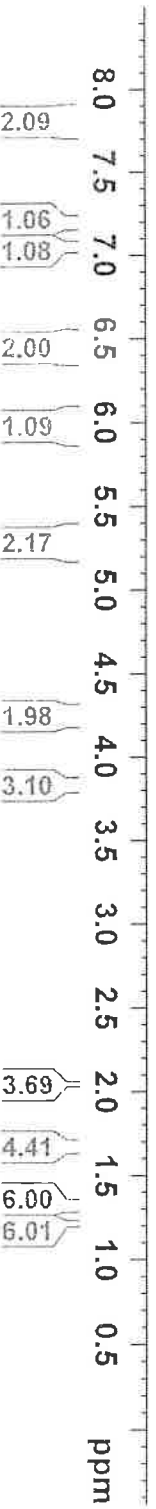
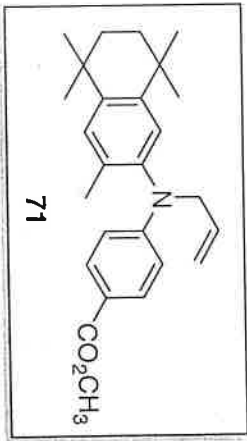
===== CHANNEL f1 =====
NUC1 13C
P1 8.25 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL12 16.21 dB
PL2W 12.76071072 W
PL12W 0.34266910 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127744 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

CWVI-187

7.806
7.260
7.192
7.043
6.473
6.451
6.014
6.001
5.988
5.975
5.971
5.961
5.958
5.945
5.932
5.918
5.316
5.312
5.273
5.269
5.244
5.240
5.218
5.214
4.218
4.211
3.833

2.047
1.684
1.306
1.215

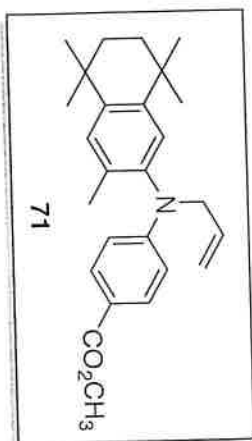


NAME CWVI-187
EXPNO 1
PROCNO 1
Date_ 20180830
Time_ 13.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DM 60.800 usec
DE 6.50 usec
TE 294.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

CWVI-187

167.39
151.94
144.45
143.94
141.68
133.50
132.88
131.12
129.30
126.76
117.46
117.05
111.62
77.32
77.00
76.68
54.56
51.46
35.09
34.94
34.07
34.00
31.86
31.84
17.60



200 180 160 140 120 100 80 60 40 20 0 ppm

NAME CWVI-187
EXPNO 2
PROCNO 1
Date_ 20180830
Time 14.07
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgig30
TD 65536
SOLVENT CDC13
NS 185
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 294.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.25 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL12 16.21 dB
PL12W 12.76071072 W
PL12W 0.34266910 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127729 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



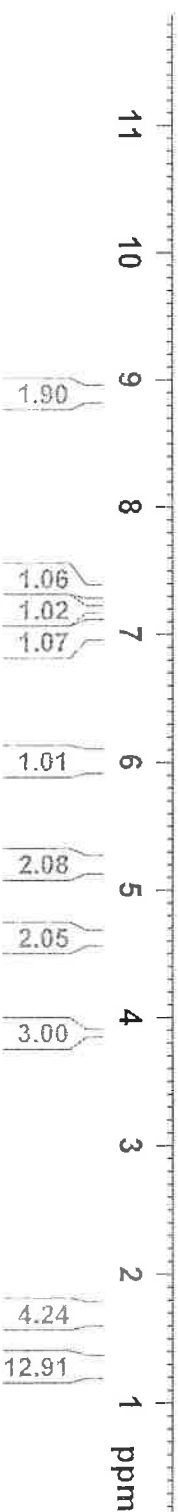
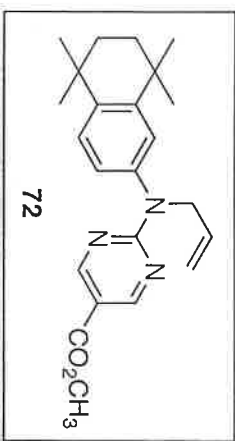
CWVI-105

8.886
7.330
7.309
7.260
7.201
7.195
7.063
7.057
7.042
7.036
6.061
6.048
6.035
6.022
6.019
6.008
6.005
5.992
5.979
5.966
5.230
5.226
5.222
5.200
5.196
5.192
5.187
5.183
5.178
5.174
5.170
4.629
4.625
4.622
4.615
4.612
4.608
3.869
1.687
1.291



NAME CWVI-105
EXPNO 1
PROCNO 1
Date_ 20180725
Time_ 12.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DE 60.800 usec
TE 294.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



CWVI-105

165.04
161.98
159.67

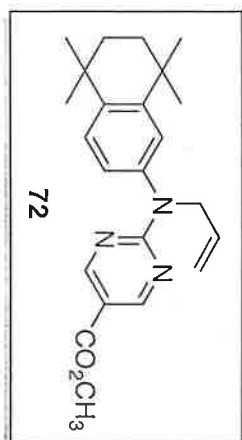
145.89
143.32
140.51

133.26
127.31
124.45
123.59
117.02
113.42

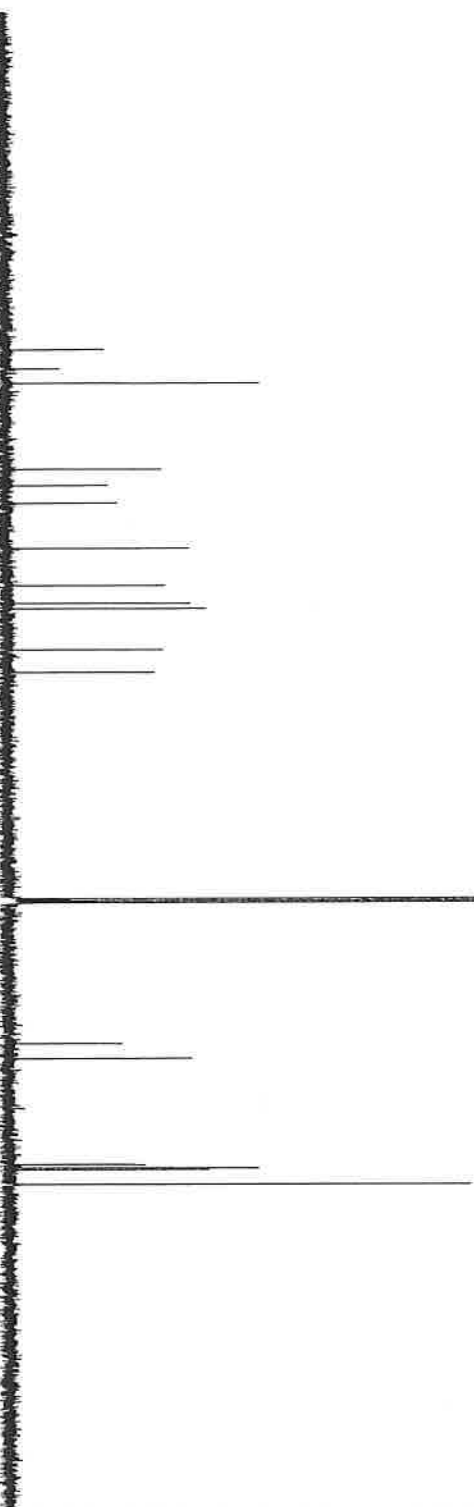
77.32
77.00
76.68

54.21
51.80

34.96
34.89
34.39
34.09
31.77



200 180 160 140 120 100 80 60 40 20 0 ppm



NAME CWVI-105

EXPNO 2

PROCNO 1

Date 20180725

Time 13.14

INSTRUM spect

PROBHD 5 mm PABBO BB-

PULPROG zgpg30

TD 65536

SOLVENT CDCl3

NS 300

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 294.6 K

D1 2.00000000 sec

D11 0.03000000 sec

TD0 1

===== CHANNEL f1 =====

NUC1 13C

P1 8.25 usec

PL1 -2.10 dB

PL1W 60.29227829 W

SFO1 100.6228298 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16

NUC2 1H

PCPD2 90.00 usec

PL2 0.50 dB

PL12 16.21 dB

PL12W 12.76071072 W

PL12W 0.34266910 W

SFO2 400.1316005 MHz

SI 32768

SF 100.6127744 MHz

WDW EM

SSB 0

LB 1.00 Hz

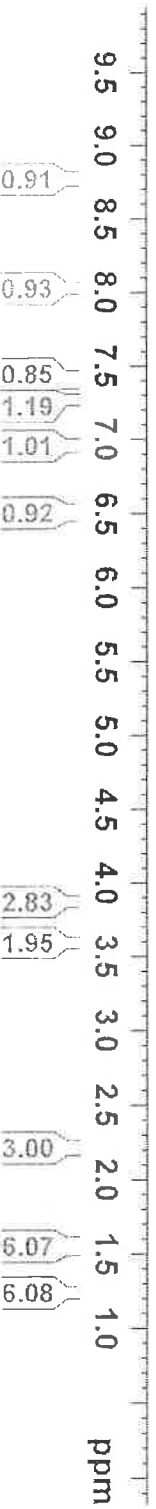
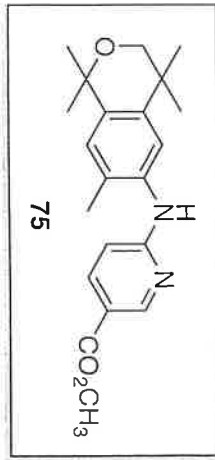
GB 0

PC 1.40

CWVI-109

8.775
8.773
8.769
8.767
8.049
8.043
8.027
8.021
7.391
7.265
7.260
6.963
6.526
6.524
6.503
6.502

3.878
3.590
2.215
1.538
1.241

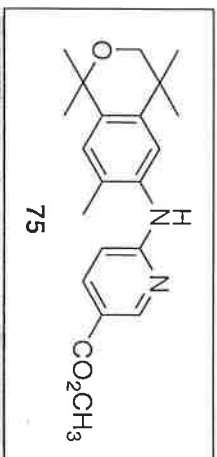


NAME CWVI-109
EXPNO 1
PROCNO 1
Date_ 20180329
Time_ 12.34
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DM 60.800 usec
DE 6.50 usec
TE 292.9 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300104 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

CWVI-109

165.82
159.39
150.36
141.60
139.79
139.38
134.70
130.70
128.05
121.76
116.38
106.06
77.31
77.00
76.68
74.95
70.67
51.85
33.75
29.74
26.98
17.74



200
180
160
140
120
100
80
60
40
20
0
ppm

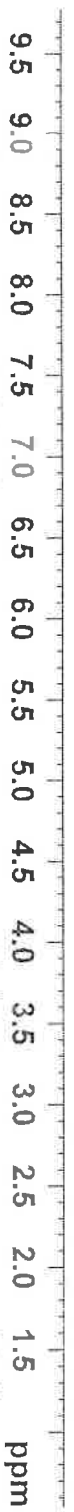
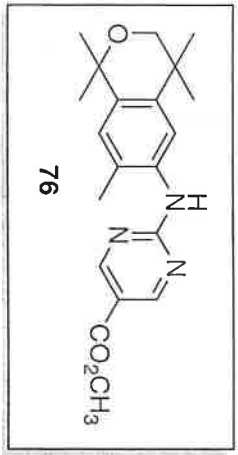
NAME CWVI-109
EXPNO 2
PROCNO 1
Date_ 20180329
Time_ 12.38
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg1930
TD 65536
SOLVENT CDCl3
NS 104
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 293.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
===== CHANNEL f1 =====
NUC1 13C
P1 8.50 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.6228298 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL12 16.21 dB
PL12W 12.76071072 W
PL12W 0.34266910 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127751 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



CWVI-113



8.939
7.689
7.578
7.260
6.939
3.904
3.597
2.262
1.531
1.272



```

NAME CWVI-113
EXPNO 1
PROCNO 1
Date_ 20180420
Time_ 9.26
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DM 60.800 usec
DE 6.50 usec
TE 292.7 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
  
```

CWVI-113

164.61
161.62
160.13

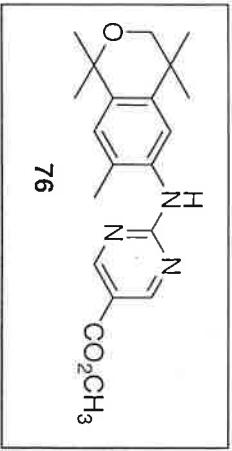
141.09
139.07
133.91
128.64
127.61
120.62
114.87

77.32
77.00
76.68
74.96
70.79

52.02

33.82
29.76
26.97

17.93



200 180 160 140 120 100 80 60 40 20 0 ppm

NAME CWVI-113
EXPNO 2
PROCNO 1
Date_ 20180420
Time_ 9.36
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 250
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 293.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.25 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL12 16.21 dB
PL2W 12.76071072 W
PL12W 0.34266910 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127744 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

CWVI-117

8.872
8.867
8.867

7.860
7.854
7.837
7.832
7.260
7.063
6.991

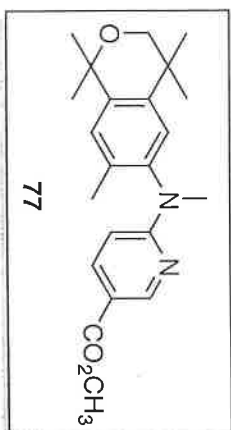
6.053
6.031

3.853
3.584
3.470

2.062

1.552

1.221



0.91

0.93

0.98
0.98

0.92

2.96

1.98

2.84

2.85

6.08

6.00

ppm



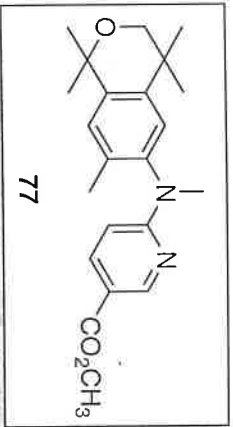
NAME CWVI-117
EXPNO 1
PROCNO 1
Date_ 20180725
Time_ 11.41
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DW 60.800 usec
DE 6.50 usec
TE 294.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

CWVI-117



166.28
142.69
141.47
141.37
137.98
133.00
128.48
124.50
114.49
107.16
77.32
77.00
76.68
75.00
70.65
51.69
38.15
33.82
29.79
27.00
17.24



200 180 160 140 120 100 80 60 40 20 0 ppm

```

NAME CWVI-117
EXPNO 2
PROCNO 1
Date_ 20180725
Time_ 11.46
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 335
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.363198 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 294.5 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.25 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL12 16.21 dB
PL12W 12.76071072 W
PL12W 0.34266910 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127736 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
  
```

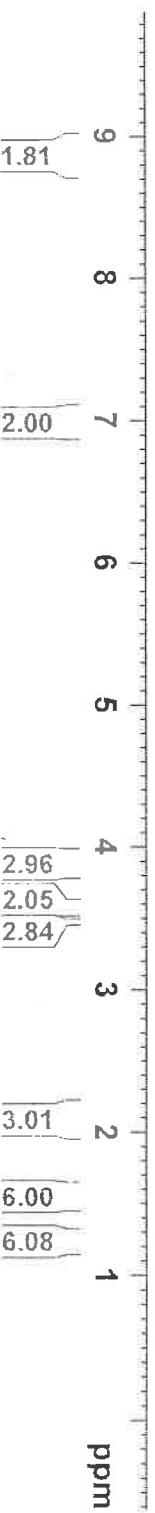
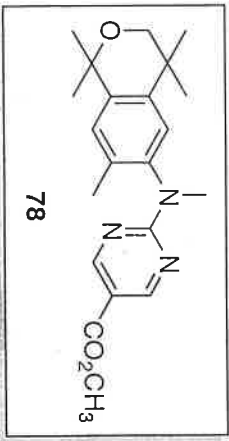
CWVI-123

8.964
8.959
8.796
8.790

7.260
7.054
6.965

3.864
3.622
3.593
3.565
3.536
3.476

2.064
1.564
1.532
1.249
1.220



```

NAME          CWVI-123
EXPNO         2
PROCNO        1
Date_         20180725
Time_         12.32
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8223.685 Hz
FIDRES       0.125483 Hz
AQ           3.9846387 sec
RG            4
DE           60.800 usec
TE           294.2 K
D1           1.00000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1           14.75 usec
PL1          0.50 dB
PL1W         12.76071072 W
SFO1         400.1324710 MHz
SI           32768
SF           400.1300099 MHz
WDW           EM
SSB           0
LB           0.30 Hz
GB           0
PC           1.00
  
```

CWVI-123

165.28
162.53
159.95
159.75

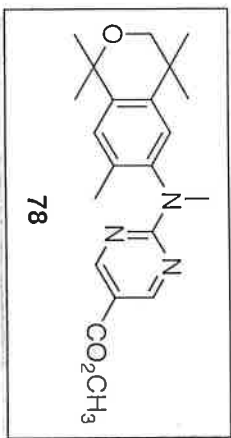
141.83
141.42
140.71
132.34
128.04
123.65

112.94

77.32
77.00
76.69
75.08
70.73

51.77

38.81
33.78
30.30
29.39
27.49
26.52
17.55



200 180 160 140 120 100 80 60 40 20 0 ppm

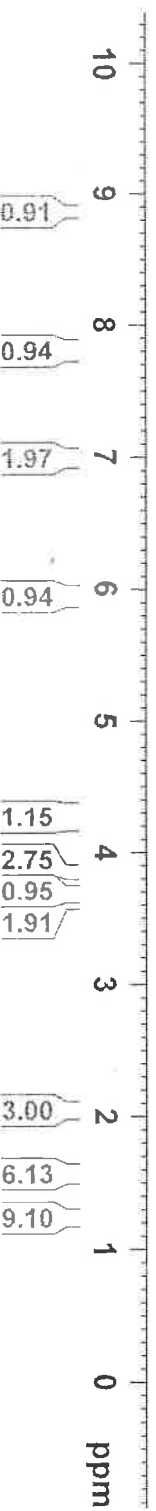
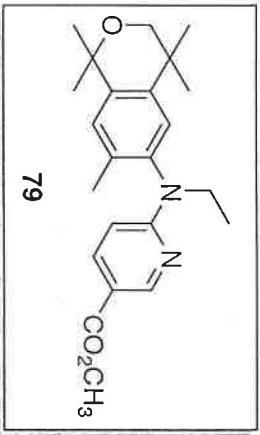
NAME CWVI-123
EXPNO 1
PROCNO 1
Date 20180725
Time 12.45
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 155
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 294.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.25 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.62282298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL12 16.21 dB
PL12W 12.76071072 W
PL12W 0.34266910 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127736 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

CWVI-115

- 8.858
- 8.854
- 8.853
- 7.834
- 7.828
- 7.811
- 7.806
- 7.260
- 7.016
- 7.000
- 5.952
- 5.930
- 4.273
- 4.257
- 3.848
- 3.704
- 3.687
- 3.670
- 3.590
- 2.056
- 1.565
- 1.546
- 1.265
- 1.248
- 1.230
- 1.218



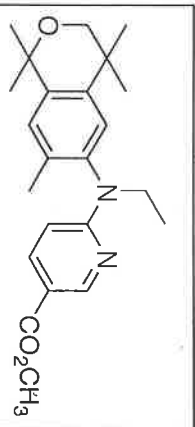
```

NAME CWVI-115
EXPNO 1
PROCNO 1
Date_ 20180725
Time 14.02
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DE 60.800 usec
TE 294.5 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
  
```

166.26
159.43
150.37
142.43
141.48
139.65
138.10
133.42
128.48
125.66
114.37
107.08

77.32
77.00
76.68
75.00
70.65
51.65
44.94
33.75
29.80
27.00
17.35
12.95



200
180
160
140
120
100
80
60
40
20
0
ppm

```

NAME          CWVI-115
EXPNO         3
PROCNO        1
Date_         20180725
Time_         20.42
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            6603
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.363198 sec
RG            2050
DW            20.800 usec
DE            6.50 usec
TE            295.4 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            8.25 usec
PL1           -2.10 dB
PL1W          60.29227829 W
SFO1          100.6228298 MHz

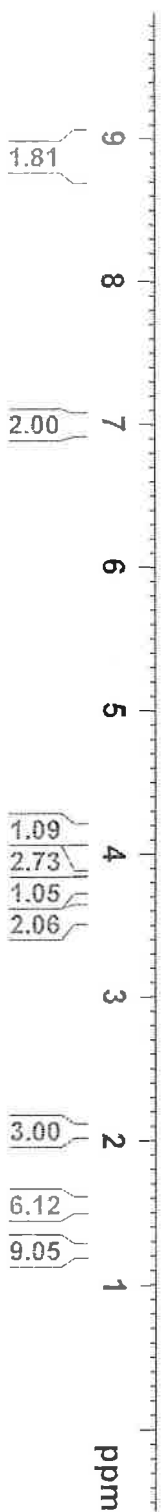
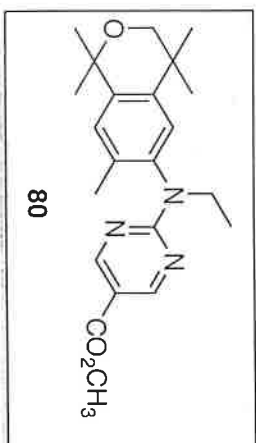
===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         90.00 usec
PL2           0.50 dB
PL12          16.21 dB
PL12W         12.76071072 W
PL12W         0.34266910 W
SFO2          400.1316005 MHz
SI            32768
SF            100.6127729 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

CWVI-121

8.945
8.787

7.260
7.010
6.971

4.182
4.164
4.146
4.130
4.112
4.095
3.862
3.827
3.809
3.792
3.775
3.758
3.740
3.628
3.599
3.572
3.543
2.059
1.570
1.533
1.262
1.253
1.245
1.225



```

NAME          CWVI-121
EXPNO         1
PROCNO        1
Date_         20180725
Time          13.47
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH            8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            4
DW            60.800 usec
DE            6.50 usec
TE            294.3 K
D1            1.00000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1            14.75 usec
PL1           0.50 dB
PL1W          12.76071072 W
SFO1          400.1324710 MHz
SI            32768
SF            400.1300099 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```

CWVI-121

165.30
162.08
160.08
159.81

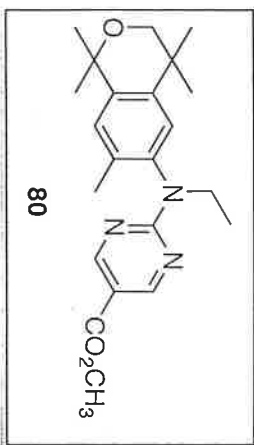
141.53
140.66
139.85
132.77
128.02
124.60

112.86

77.32
77.00
76.68
75.09
70.73

51.74
46.07

33.71
30.28
29.43
27.50
26.52
17.69
12.71



200 180 160 140 120 100 80 60 40 20 0 ppm

```

NAME CWVI-121
EXPNO 2
PROCNO 1
Date_ 20180725
Time_ 13.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg1930
TD 65536
SOLVENT CDCl3
NS 133
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DE 20.800 usec
TE 294.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.25 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.6228298 MHz

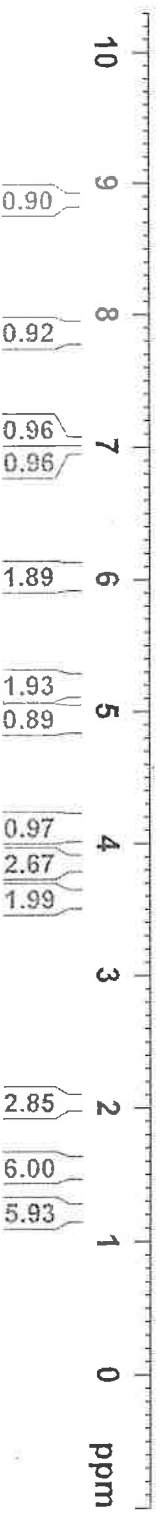
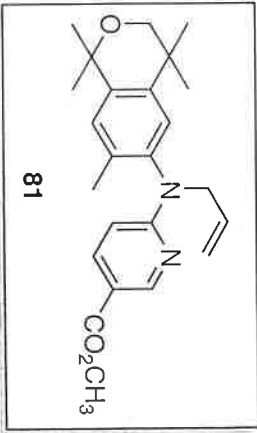
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL12 16.21 dB
PL12W 12.76071072 W
PL12W 0.34266910 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127729 MHz
WDW EM
SSB 0
GB 1.00 Hz
PC 1.40
  
```



CWVI-119

8.867
8.862
7.878
7.874
7.856
7.852
7.260
7.029
6.985
6.084
6.069
6.058
6.053
6.043
6.026
6.016
6.011
6.000
5.985
5.203
5.181
5.156
4.940
4.124
3.852
3.578

— 2.050
— 1.546
— 1.202



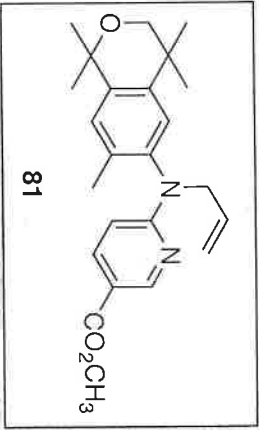
```

NAME CWVI-119
EXPNO 1
PROCNO 1
Date_ 20180725
Time 21.15
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DE 60.800 usec
TE 294.9 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
  
```


CWVI-119

166.01
158.76
149.67
142.37
141.64
139.59
138.57
133.14
132.95
128.41
125.58
118.08
114.86
107.53
77.32
77.00
76.68
74.98
70.61
53.14
51.75
33.76
29.79
26.90
17.44



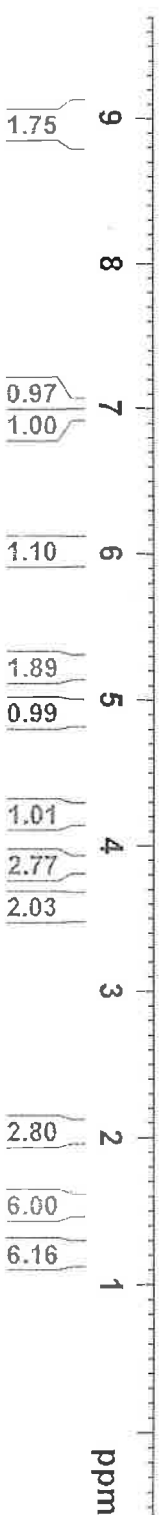
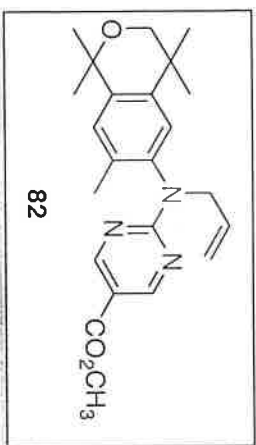
200 180 160 140 120 100 80 60 40 20 0 ppm



NAME CWVI-119
EXPNO 3
PROCNO 1
Date 20180726
Time 12.30
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 8193
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 295.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
===== CHANNEL f1 =====
NUC1 13C
P1 8.25 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.6228298 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL12 16.21 dB
PL12W 12.76071072 W
PL12W 0.34266910 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127736 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

CWVI-147

- 8.882
- 7.260
- 7.026
- 6.981
- 6.065
- 6.050
- 6.048
- 6.039
- 6.033
- 6.023
- 6.008
- 6.006
- 5.997
- 5.991
- 5.982
- 5.980
- 5.965
- 5.267
- 5.264
- 5.245
- 5.242
- 5.219
- 4.931
- 4.917
- 4.894
- 4.879
- 4.238
- 4.220
- 4.200
- 4.183
- 3.880
- 3.632
- 3.604
- 3.558
- 3.529
- 2.058
- 1.569
- 1.531
- 1.222
- 1.216



NAME CWVI-147
 EXPNO 1
 PROCNO 1
 Date_ 20180726
 Time_ 17.28
 INSTRUM spect
 PROBD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 4
 DM 60.800 usec
 DE 6.50 usec
 TE 295.3 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 14.75 usec
 PL1 0.50 dB
 PL1W 12.76071072 W
 SFO1 400.1324710 MHz
 SI 32768
 SF 400.1300099 MHz
 SE EM
 MDM 0
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

CWVI-147

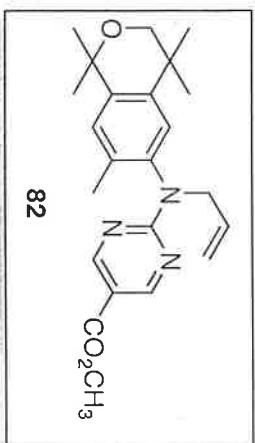
164.42
160.16
160.09
159.19

141.75
141.41
139.09
132.36
132.02
128.18
124.44
118.96
113.33

77.32
77.00
76.68
75.07
70.65

54.21
52.03

33.75
30.13
29.46
27.41
26.49
17.68



200 180 160 140 120 100 80 60 40 20 0 ppm

NAME CWVI-147
EXPNO 2
PROCNO 1
Date_ 20180726
Time_ 17.38
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg1930
TD 65536
SOLVENT CDC13
NS 16384
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DE 20.800 usec
TE 295.5 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

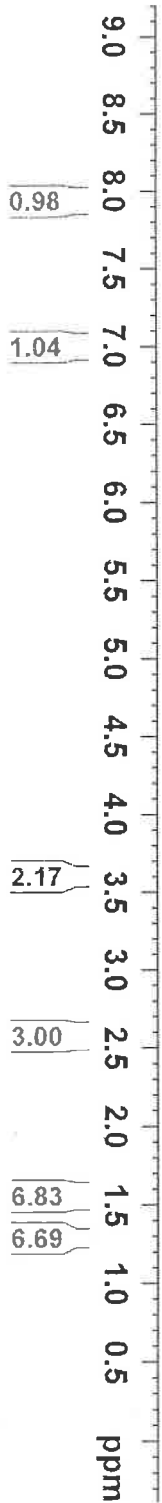
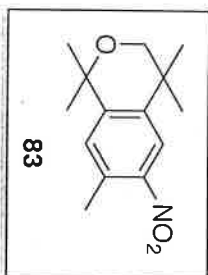
===== CHANNEL f1 =====
NUC1 13C
P1 8.25 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL12 16.21 dB
PL12W 12.76071072 W
PL12W 0.34266910 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127729 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

CWVI-177

7.937
7.260
6.999

3.588
2.561
1.537
1.282



NAME CWVI-177
EXPNO 1
PROCNO 1
Date_ 20180828
Time 13.19
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SMH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DW 60.800 usec
DE 6.50 usec
TE 292.1 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SF01 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

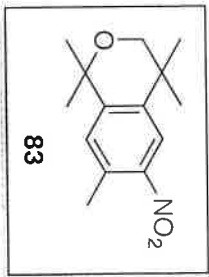
CWVI-177



147.48
147.35
142.06
130.94
129.71
121.97

77.32
77.00
76.68
75.01
70.42

33.94
29.44
26.79
20.50



NAME CWVI-177
EXPNO 2
PROCNO 1
Date 20180828
Time 13.23
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg1g30
TD 65536
SOLVENT CDCl3
NS 66
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 292.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

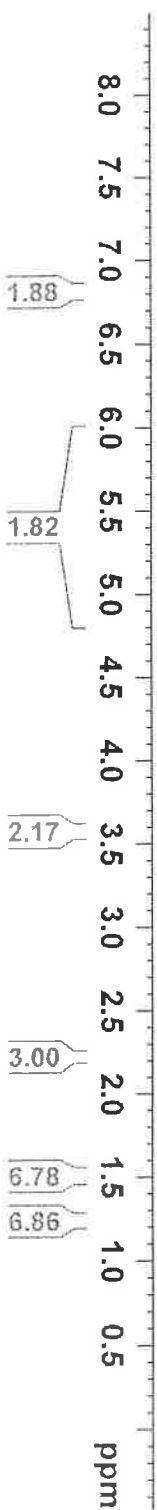
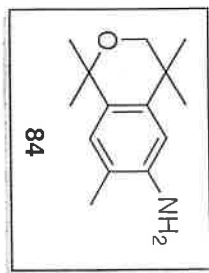
===== CHANNEL f1 =====
NUC1 13C
P1 8.25 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL12 16.21 dB
PL12W 12.76071072 W
PL12W 0.34266910 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127736 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



CWVI-181

7.260
6.822
6.798
5.379
3.563
2.238
1.499
1.239



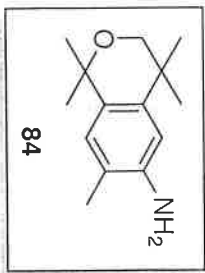
NAME CWVI-181
EXPNO 1
PROCNO 1
Date_ 20180828
Time 13.29
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DE 60.800 usec
TE 292.4 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

CWVI-181



141.44
139.18
134.42
127.52
122.53
113.25
77.32
77.00
76.68
74.83
70.81
33.61
29.87
26.93
17.31



200 180 160 140 120 100 80 60 40 20 0 ppm

```

NAME CWVI-181
EXPNO 2
PROCNO 1
Date_ 20180828
Time 13.33
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 58
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DM 20.800 usec
DE 6.50 usec
TE 292.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.25 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL12 16.21 dB
PL12W 12.76071072 W
PL12W 0.34266910 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127751 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
  
```

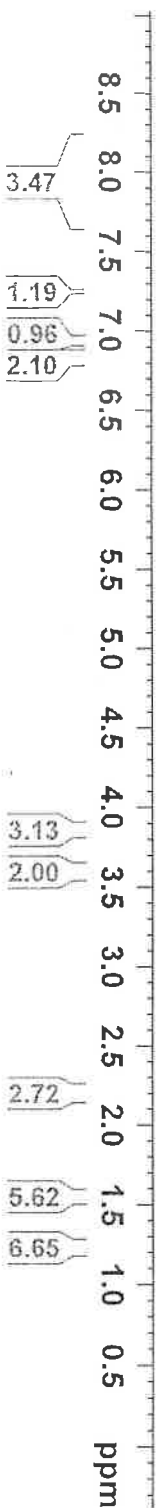
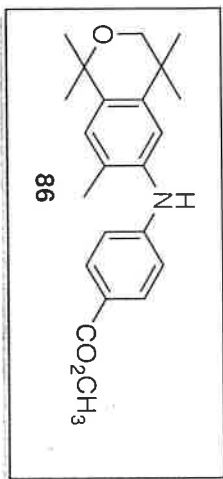
CWVI-183 f 12-16

8.142
8.120
7.916
7.895
7.704
7.682
7.260
7.251
6.932
6.846
6.825

3.868
3.589

2.204

1.540
1.227



NAME CWVI-183
EXNO 3
PROCNO 1
Date_ 20180828
Time_ 13.49
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DW 60.800 usec
DE 6.50 usec
TE 292.8 K
D1 1.00000000 sec
TD0 1

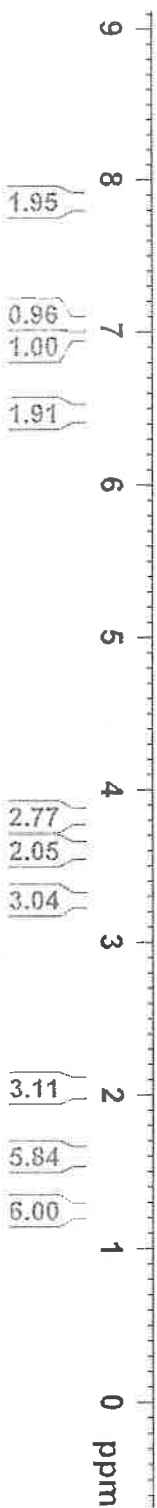
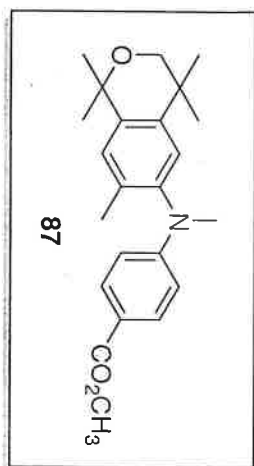
===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SF01 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

CWVI-191

7.864
7.841
7.260
7.045
6.971
6.483
6.461

3.842
3.593
3.271

2.050
1.563
1.220



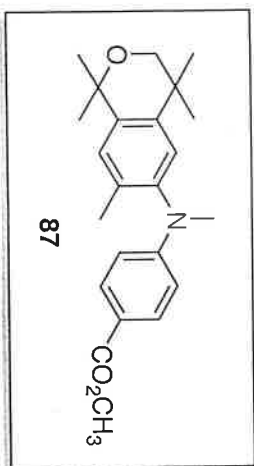
NAME CWVI-191
EXPNO 1
PROCNO 1
Date_ 20180906
Time 12.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DW 60.800 usec
DE 6.50 usec
TE 293.6 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SF01 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



CWVI-191

167.37
152.40
143.37
142.29
140.56
133.44
131.18
128.24
124.72
117.59
111.24
77.31
77.00
76.68
75.03
70.74
51.49
39.12
33.79
29.83
27.00
17.45



200 180 160 140 120 100 80 60 40 20 0 ppm

NAME CWVI-191
EXPNO 2
PROCNO 1
Date_ 20180906
Time_ 12.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 100
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 293.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.25 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.6228298 MHz

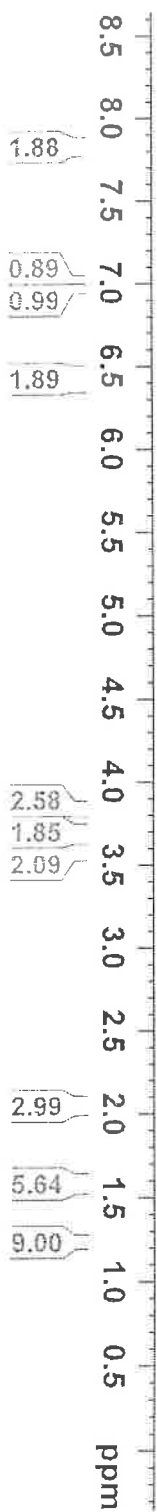
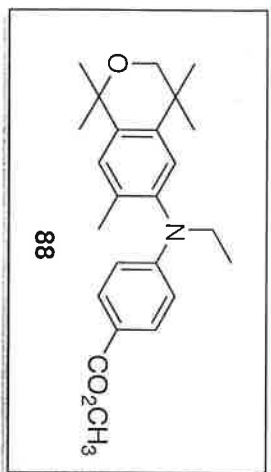
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL12 16.21 dB
PL12W 12.76071072 W
PL12W 0.34266910 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127744 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

CWVI-189

7.839
7.816
7.260
7.012
6.982
6.448
6.426

3.834
3.680
3.663
3.649
3.596

2.043
1.564
1.271
1.253
1.236
1.223



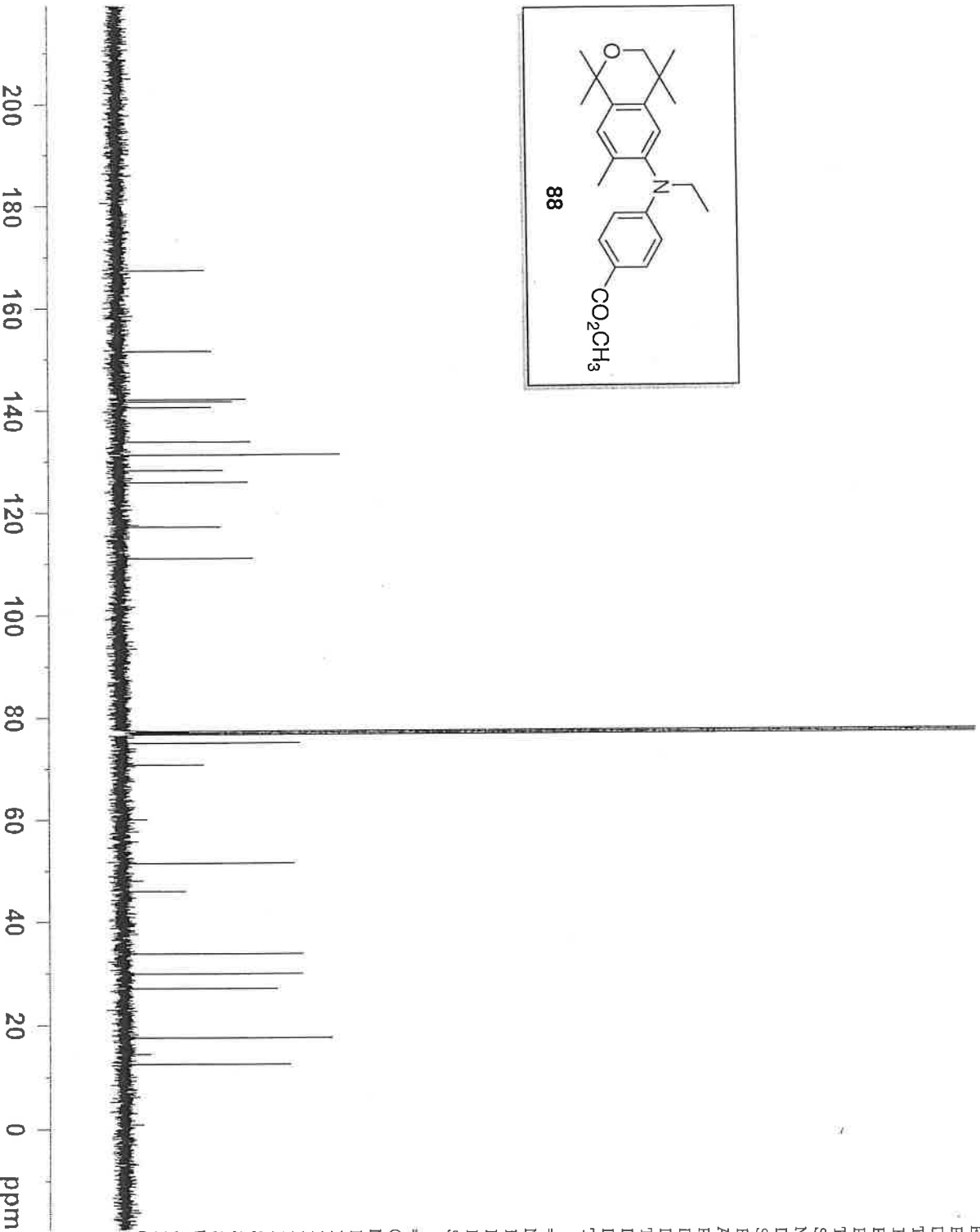
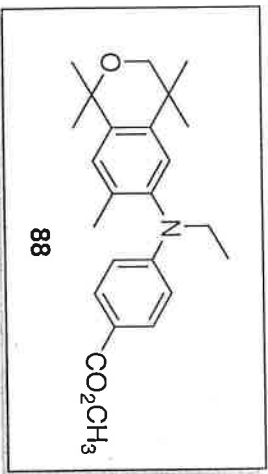
NAME CWVI-189
EXPNO 1
PROCNO 1
Date_ 20180906
Time 12.39
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DW 60.800 usec
DE 6.50 usec
TE 293.5 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

CWVI-189



167.34
151.57
142.13
141.71
140.60
133.85
131.30
128.25
125.91
117.20
111.08
77.31
77.00
76.68
75.03
70.73
51.45
45.93
33.71
29.83
26.99
17.54
12.50



NAME CWVI-189
EXPNO 2
PROCNO 1
Date_ 20180906
Time 12.45
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg1930
TD 65536
SOLVENT CDCl3
NS 151
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 293.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.25 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL12 16.21 dB
PL12W 12.76071072 W
PL12W 0.34266910 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127744 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

CWVI-193

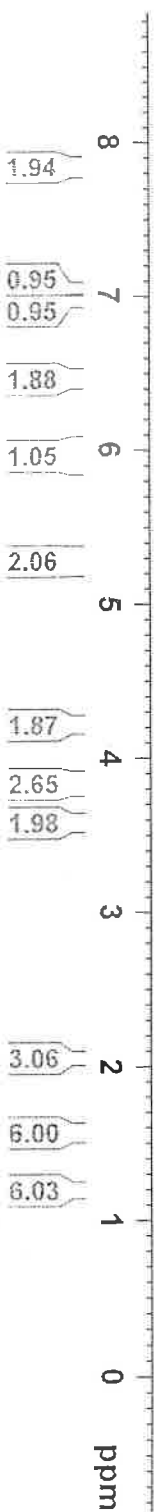
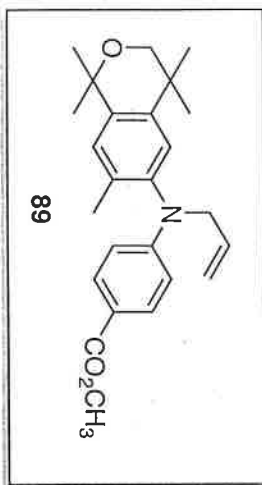
7.815
7.260
7.050
6.973
6.474
6.452
6.014
6.001
5.988
5.975
5.971
5.961
5.958
5.945
5.932
5.918
5.314
5.310
5.306
5.271
5.267
5.252
5.248
5.226
5.223
4.224
4.213
3.835
3.589

2.056
1.560
1.210



NAME CWVI-193
EXPNO 1
PROCNO 1
Date 20180906
Time 13.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DW 60.800 usec
DE 6.50 usec
TE 293.6 K
D1 1.00000000 sec
TD0 1

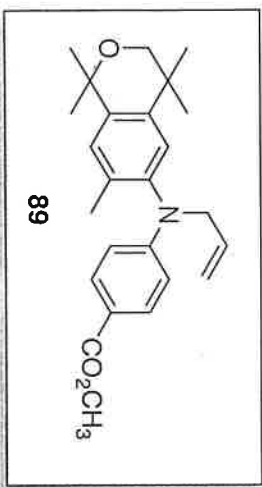
===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 0.50 dB
PL1W 12.76071072 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



CWVI-193



167.32
151.74
142.42
142.15
140.66
133.57
133.35
131.17
128.25
125.58
117.77
117.23
111.68
77.32
77.00
76.68
75.03
70.74
54.56
51.50
33.76
29.83
26.98
17.70



NAME CWVI-193
EXPNO 2
PROCNO 1
Date_ 20180906
Time 13.14
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 200
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 293.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.25 usec
PL1 -2.10 dB
PL1W 60.29227829 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.50 dB
PL12 16.21 dB
PL12W 12.76071072 W
PL12W 0.34266910 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127729 MHz
WDM EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

