

Advanced
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Supporting Information

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Supporting Information

Chiral Brønsted Acid-Mediated Highly Enantioselective Organocatalytic Three-component Reaction for the Construction of Trifluoromethyl-containing Molecules

Guang-Wu Zhang,^a Lian Wang,^a Jing Nie,^a Jun-An Ma*^{a,b}

^a *Department of Chemistry, School of Science,*

Tianjin University, Tianjin 300072,

majun_an68@tju.edu.cn

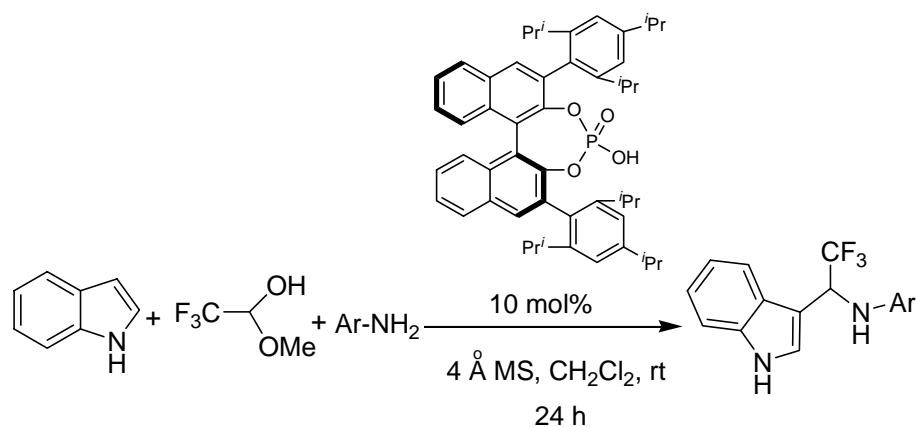
^b *State Key Laboratory of Elemento-Organic Chemistry,*

Nankai University, Tianjin 300071, China

General information: ^1H , ^{13}C , ^{19}F NMR were recorded on Varian Mercury Plus 500 and 400 instruments at 500 MHz and 400 MHz (^1H NMR), 125 MHz (^{13}C NMR), as well as 376 MHz (^{19}F NMR), or a Bruker AV 300 Spectrometer at 300 MHz (^1H NMR), 75 MHz (^{13}C NMR), or a Bruker AV 600 Spectrometer at 150 MHz (^{13}C NMR). Chemical shifts were reported in ppm down field from internal Me_4Si and external CCl_3F , respectively. Data are reported as follows: chemical shift, multiplicity (s = singlet, d= doublet, q = quartet, m = multiplet), coupling constants (Hz) and integration. MS were recorded on a VG-7070E or HP 5988A spectrometer using the ESI method. HPLC analyses were carried out on a Hewlett Packard Model HP 1200 instrument. All of the reactions were carried out under an argon atmosphere with the exclusion of moisture.

Materials: Unless otherwise noted, all commercially available compounds and solvents were used as provided without further purification. Analytical thin layer chromatography was performed on 0.20 mm Qingdao Haiyang silica gel plates. Silica gel (200-300 mesh) (from Qingdao Haiyang Chem. Company, Ltd.) was used for flash chromatography. All of chiral phosphoric acids were prepared according to the literature procedure.^[1-3]

The influence of the structure of amine component in three-component reaction ^a



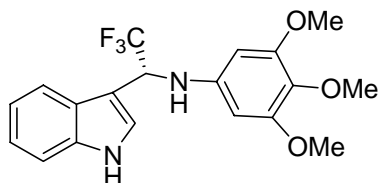
Entry	Ar	Yield (%) ^b	Ee (%) ^c
1	Ph	> 99	53
2	4-MeOC ₆ H ₄	97	64
3	2-MeOC ₆ H ₄	38	65
4	3-MeOC ₆ H ₄	92	79
5	3- ⁱ PrOC ₆ H ₄	> 99	67
6	3,4,5-(MeO) ₃ C ₆ H ₂	> 99	94

^a Reaction conditions: a solution of ArNH_2 (0.25 mmol), Trifluoro-acetaldehyde methyl hemiacetal (0.375 mmol), indole (0.375 mmol), powdered 4 Å molecular sieves (50 mg), and catalyst (0.025 mmol) in solvent (1.0 mL) was stirred at room temperature for 24 h. ^b Isolated yield. ^c Enantiomeric excess was determined by chiral HPLC analysis.

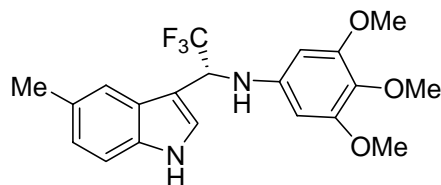
Representative procedure of three-component reaction:

Brønsted acid catalyst **1** (0.025 mmol) was added to a mixture of indole (43.8 mg, 0.37 mmol), trifluoroacetaldehyde methyl hemiacetal (48.8 mg, 0.37 mmol), 3, 4,

5-trimethoxyaniline (45.7 mg, 0.25 mmol) and powdered 4 Å molecular sieves (50 mg) in dichloromethane (1.0 mL). The resulting mixture was stirred at room temperature for the stated time. Then, the reaction solution was concentrated *in vacuo*, and the residue was purified by silica gel flash column chromatography (petroleum ether / AcOEt: 15:1) to give the desired product **5**.

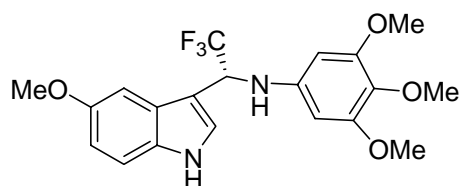


N-((R)-2,2,2-trifluoro-1-(1H-indol-3-yl)ethyl)-3,4,5-trimethoxybenzenamine (5a), yield > 99%, ee > 99.9%(After recrystallization), [Daicel Chiralcel AD-H, Hexanes / *i*PrOH = 80 / 20, 0.5 mL /min, λ = 254 nm, t (minor) = 31.05 min, t (major) = 44.14 min]; $[\alpha]_D^{20}$ = -44.7 (c = 1.0, CH₂Cl₂). **M. p.** 139-141 °C. **¹⁹F-NMR** (376MHz, CDCl₃) δ [ppm] -74.1 (d, *J* = 7.1 Hz, 3F). **¹H-NMR** (400MHz, CDCl₃) δ [ppm] 8.28 (br, 1H), 7.61 (d, *J* = 10.0 Hz, 1H), 7.22 (d, *J* = 10.0 Hz, 1H), 7.33 (d, *J* = 2.0 Hz, 1H), 7.28-7.24 (m, 1H), 7.19-7.15 (m, 1H), 5.22-5.15 (m, 1H), 3.77 (s, 9H). **¹³C-NMR** (125MHz, CDCl₃) δ [ppm] 154.1, 143.4, 136.3, 131.4, 126.1, 126.1 (q, ¹J_{C-F} = 280.8 Hz), 123.4 (d, *J* = 1.3 Hz), 123.1, 120.7, 119.1, 111.8, 109.7, 90.8, 60.3, 56.2, 55.1 (q, ²J_{C-F} = 31.7 Hz). **IR** (KBr) ν (cm⁻¹) 1609, 1511, 1465, 1282, 1244, 1206, 1108, 1134, 996, 737. **MS** (EI) *m/z* (%) 381.41 [M+H]⁺ (100%).

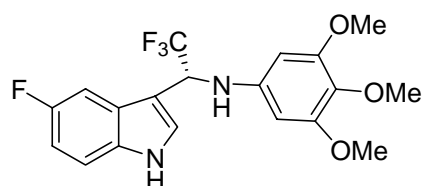


N-((R)-2,2,2-trifluoro-1-(5-methyl-1H-indol-3-yl)ethyl)-3,4,5-trimethoxybenzenamine (5b), yield 83%, ee 95%, [Daicel Chiralcel AD-H, Hexanes / *i*PrOH = 80 / 20, 0.5 mL /min, λ = 254 nm, t (minor) = 24.32 min, t (major) = 29.81 min]; $[\alpha]_D^{20}$ = -225.0 (c = 1.0, CH₂Cl₂). **M. p.** 188-191 °C. **¹⁹F-NMR** (376MHz, CDCl₃) δ [ppm] -74.1 (d, *J* = 7.1 Hz, 3F). **¹H-NMR** (400MHz, CDCl₃) δ [ppm] 8.19 (br, 1H), 7.39

(1H), 7.31-7.28 (m, 2H), 7.08(d, $J = 8.4$ Hz, 1H), 5.98 (s, 2H), 5.14 (d, $J = 2.4$, 1H), 3.77 (s, 9H), 2.44 (s, 3H). $^{13}\text{C-NMR}$ (125MHz, CDCl_3) δ [ppm] 154.1, 143.4, 134.7, 131.4, 130.1, 126.3, 126.1 (q, $^1J_{\text{C-F}} = 280.7$ Hz), 124.7, 123.5 (d, $J = 0.1$ Hz), 118.6, 111.5, 109.3, 90.9, 61.3, 56.2, 55.2(q, $^2J_{\text{C-F}} = 31.6$ Hz), 21.8. **IR** (KBr) ν (cm^{-1}) 1610, 1510, 1457, 1243, 1133, 998, 796, 649. **MS** (EI) m/z (%): 395.32 $[\text{M}+\text{H}]^+$ (100%).

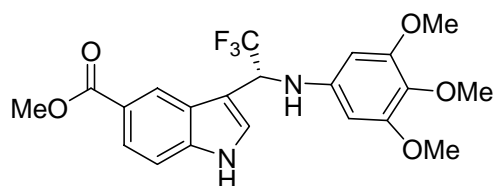


N-((R)-2,2,2-trifluoro-1-(5-methoxy-1H-indol-3-yl)ethyl)-3,4,5-trimethoxybenzenamine (5c), yield >99%, ee 92%, [Daicel Chiralcel AD-H, Hexanes / i PrOH = 80 / 20, 0.5 mL /min, $\lambda = 254$ nm, t (minor) = 38.77 min, t (major) = 48.03 min]; $[\alpha]_{\text{D}}^{20} = -40.0$ ($c = 1.0$, CH_2Cl_2). **M. p.** 125-127 °C. $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ [ppm] -74.1 (d, $J = 7.1$ Hz, 3F). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ [ppm] 8.21 (br, 1H), 7.31-7.25 (m, 2H), 6.97 (d, $J = 1.2$ Hz, 1H), 6.91-6.89 (m, 1H), 5.98 (s, 2H), 5.15-5.10 (m, 1H), 3.77 (s, 9H), 3.75 (s, 3H). $^{13}\text{C NMR}$ (125MHz, CDCl_3) δ [ppm] 154.8, 154.1, 143.7, 131.5, 126.6, 126.1 (q, $^1J_{\text{C-F}} = 280.5$ Hz), 125.0, 124.0, 113.2, 112.6, 100.9, 92.0, 61.3, 56.2, 56.0, 55.5 (q, $^2J_{\text{C-F}} = 31.5$ Hz). **IR** (KBr) ν (cm^{-1}) 1610, 1510, 1489, 1458, 1243, 1166, 1114, 996, 799. **MS** (ESI) m/z (%): 411.09 $[\text{M}+\text{H}]^+$ (100%).

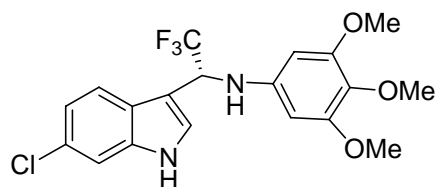


N-((R)-2,2,2-trifluoro-1-(5-fluoro-1H-indol-3-yl)ethyl)-3,4,5-trimethoxybenzenamine (5d), yield >99%, ee 94 %, [Daicel Chiralcel AD-H, Hexanes / i PrOH = 80 / 20, 0.5 mL /min, $\lambda = 254$ nm, t (minor) = 23.836 min, t (major) = 27.426 min]; $[\alpha]_{\text{D}}^{20} = -29.2$ ($c = 1.0$, CH_2Cl_2). **M. p.** 150-153 °C. $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ [ppm] -74.3 (d, $J = 7.1$ Hz, 3F), -123.3-123.4 (m, 1F). $^1\text{H NMR}$ (500MHz, CDCl_3) δ [ppm] 8.29 (br, 1H), 7.38-7.33 (m, 2H), 7.29-7.26 (m, 1H), 7.03-6.99 (m, 1H), 5.96 (s, 2H),

5.14-5.10 (m, 2H), 3.77 (s, 6H), 3.77 (s, 3H). ^{13}C NMR (125 MHz, CDCl_3) δ [ppm] 158.4 (d, $J = 234.9$ Hz), 154.1, 143.1, 132.9, 131.5, 126.5 (d, $J = 9.8$ Hz), 125.9 (q, $^1J_{\text{C-F}} = 280.7$ Hz), 125.2, 112.6 (d, $J = 9.6$ Hz), 111.7 (d, $J = 26.2$ Hz), 110.0, 104.2 (d, $J = 24.2$ Hz), 91.9, 61.3, 56.2, 55.1 (q, $^2J_{\text{C-F}} = 31.7$ Hz). IR (KBr) ν (cm^{-1}) 1610, 1511, 1453, 1243, 1206, 1133, 1109, 999, 942, 798. MS (ESI) m/z : 399.22 $[\text{M}+\text{H}]^+$ (100%).

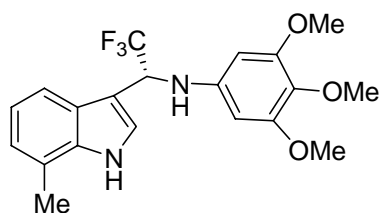


Methyl 3-((R)-1-(3,4,5-trimethoxyphenylamino)-2,2,2-trifluoroethyl)-1H-indole-5-carboxylate (5e), yield 90%, ee 92 %, [Daicel Chiralcel AD-H, Hexanes / i PrOH = 80 / 20, 0.5 mL /min, $\lambda = 254$ nm, t (major) = 24.201 min, t (minor) = 37.336 min]; $[\alpha]_{\text{D}}^{20} = +11.5$ ($c = 1.0$, CH_2Cl_2). **M. p.** 142-143 °C. ^{19}F NMR (376 MHz, CDCl_3) δ [ppm] -74.4 (d, $J = 6.7$ Hz, 3F). ^1H NMR (400 MHz, CDCl_3) δ [ppm] 8.54 (br, 1H), 8.42 (s, 1H), 7.96 (d, $J = 8.8$ Hz, 1H), 7.43-7.40 (m, 2H), 5.96 (s, 2H), 5.25-5.20 (m, 1H), 3.92 (d, $J = 1.2$ Hz, 3H), 3.74 (s, 9H). ^{13}C NMR (125 MHz, CDCl_3) δ [ppm] 168.1, 154.1, 143.1, 139.0, 131.5, 125.8, 125.1, 124.4, 124.3 (q, $^1J_{\text{C-F}} = 280.6$ Hz), 122.8, 121.9, 111.6, 92.1, 61.3, 56.2, 54.9 (q, $^2J_{\text{C-F}} = 30.8$ Hz), 52.2. IR (KBr) ν (cm^{-1}) 1728, 1611, 1513, 1235, 1134, 989, 771, 751. MS (ESI) m/z (%): 439.28 $[\text{M}+\text{H}]^+$ (100%).

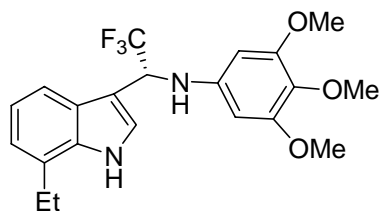


N-((R)-1-(6-chloro-1H-indol-3-yl)-2,2,2-trifluoroethyl)-3,4,5-trimethoxybenzamine (5f), yield 95%, ee 90%, [Daicel Chiralcel AD-H, Hexanes / i PrOH = 80 / 20, 0.5 mL /min, $\lambda = 254$ nm, t (minor) = 24.201 min, t (major) = 37.336 min]; $[\alpha]_{\text{D}}^{20} = -23.8$ ($c = 1.0$, CH_2Cl_2). **M. p.** 159-162 °C. ^{19}F NMR (376 MHz, CDCl_3) δ [ppm] -74.3 (d, $J = 7.1$ Hz, 3F). ^1H NMR (500 MHz, CDCl_3) δ [ppm] 8.31 (br, 1H), 7.51 (d,

$J = 8.5$ Hz, 1H), 7.42 (d, $J = 1.5$ Hz, 1H), 7.32 (d, $J = 2.0$ Hz, 1H), 7.15-7.13 (m, 1H), 5.96 (s, 2H), 5.18-5.13 (m, 1H), 3.77 (s, 9H). ^{13}C NMR (125 MHz, CDCl_3) δ [ppm] 154.2, 143.1, 136.7, 131.5, 129.2, 124.7, 124.4 (q, $^1J_{\text{C-F}} = 280.2$ Hz), 124.1, 121.6, 120.0, 111.7, 110.0, 61.3, 56.2, 55.1 (q, $^2J_{\text{C-F}} = 31.3$ Hz). IR (KBr) ν (cm^{-1}) 1610, 1515, 1452, 1269, 1236, 1125, 1004, 849. MS (ESI) m/z (%): 413.28 [M-H] (100%).

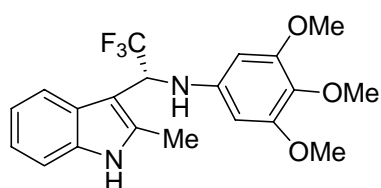


N-((R)-2,2,2-trifluoro-1-(7-methyl-1H-indol-3-yl)ethyl)-3,4,5-trimethoxybenzenamine (5g), yield 97%, ee 96%, [Daicel Chiralcel AD-H, Hexanes / i PrOH = 80 / 20, 0.5 mL / min, $\lambda = 254$ nm, t (minor) = 23.615 min, t (major) = 28.242 min]; $[\alpha]_{\text{D}}^{20} = +3.0$ ($c = 1.0$, CH_2Cl_2). **M. p.** 174-178 °C. ^{19}F NMR (376 MHz, CDCl_3) δ [ppm] -74.2 (d, $J = 6.8$ Hz, 3F). ^1H NMR (500 MHz, CDCl_3) δ [ppm] 8.22 (br, 1H), 7.46 (d, $J = 7.5$ Hz, 1H), 7.34 (s, 1H), 7.12-7.06 (m, 2H), 6.00 (s, 2H), 5.20-5.16 (m, 1H), 3.77 (s, 9H), 2.51 (s, 3H). ^{13}C NMR (125 MHz, CDCl_3) δ [ppm] 154.1, 143.4, 136.0, 131.4, 126.1 (q, $^1J_{\text{C-F}} = 280.6$ Hz), 125.6, 123.6, 123.1 (d, $J = 1.2$ Hz), 121.1, 120.9, 116.7, 110.2, 91.8, 61.3, 56.2, 55.2 (q, $^2J_{\text{C-F}} = 31.7$ Hz), 16.7. IR (KBr) ν (cm^{-1}) 1614, 1512, 1451, 1264, 1126, 1005, 783, 629. MS (ESI) m/z : 393.27 [M-H] $^+$ (100%).

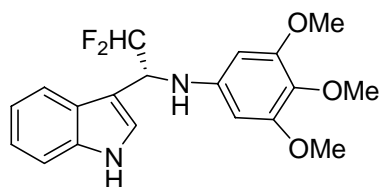


N-((R)-1-(7-ethyl-1H-indol-3-yl)-2,2,2-trifluoroethyl)-3,4,5-trimethoxybenzenamine (5h), yield >99%, ee 98%, [Daicel Chiralcel AD-H, Hexanes / i PrOH = 80 / 20, 0.5 mL / min, $\lambda = 254$ nm, t (minor) = 24.082min, t (major) = 25.981 min]; $[\alpha]_{\text{D}}^{20} = -65.9$ ($c = 1.0$, CH_2Cl_2). **M. p.** 137-140 °C. ^{19}F NMR (376 MHz, CDCl_3) δ [ppm] -74.1 (d, $J = 7.1$ Hz, 3F). ^1H NMR (400 MHz, CDCl_3) δ [ppm] 8.26 (br, 1H), 7.46 (d, $J = 9.6$ Hz,

1H), 7.32 (s, 1H), 7.18-7.08 (m, 2H), 5.97 (s, 2H), 5.20-5.16 (m, 1H), 3.77 (s, 9H), 2.91-2.84 (m, 2H), 1.37 (t, $J = 10.4$ Hz, 3H). ^{13}C NMR (125MHz, CDCl_3) δ [ppm] 154.1, 143.5, 135.2, 131.4, 127.3, 126.1 (q, $^1J_{\text{C-F}} = 280.6$ Hz), 125.9, 123.0 (d, $J = 1.6$ Hz), 121.6, 121.1, 116.8, 110.2, 91.8, 61.3, 56.2, 55.2 (q, $^2J_{\text{C-F}} = 31.3$ Hz), 24.1, 14.0. IR (KBr) ν (cm^{-1}) 1609, 1514, 1448, 1417, 1349, 1234, 1121, 998, 812, 747. MS (ESI) m/z (%): 409.07 $[\text{M}+\text{H}]^+$ (100%).

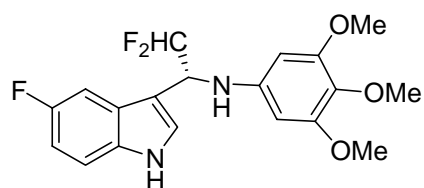


N-((R)-2,2,2-trifluoro-1-(2-methyl-1H-indol-3-yl)ethyl)-3,4,5-trimethoxybenzenamine (5i), yield 80%, ee 79 %, [Daicel Chiralcel AD-H, Hexanes / i PrOH = 80 / 20, 0.5 mL /min, $\lambda = 254$ nm, t (minor) = 22.332 min, t (major) = 35.116 min]; $[\alpha]_{\text{D}}^{20} = -5.4$ ($c = 1.0$, CH_2Cl_2). ^{19}F NMR (376 MHz, CDCl_3) δ [ppm] -73.9 (d, $J = 7.5$ Hz, 3F). ^1H NMR (500 MHz, CDCl_3) δ [ppm] 8.04 (br, 1H), 7.74-7.73 (m, 1H), 7.34-7.32 (m, 1H), 7.21-7.16 (m, 2H), 5.94 (s, 2H), 5.05-5.01 (m, 1H), 3.74 (s, 9H), 2.50 (s, 3H). ^{13}C NMR (75MHz, CDCl_3) δ [ppm] 153.8, 143.2, 135.3, 134.0, 131.2, 128.5 (q, $^1J_{\text{C-F}} = 270.7$ Hz), 126.5, 121.8, 120.3, 118.5, 110.8, 104.7, 91.8, 61.0, 55.9, 55.3 (q, $^2J_{\text{C-F}} = 32.6$ Hz), 12.3. IR (KBr) ν (cm^{-1}) 1770, 1714, 1609, 1478, 1313, 1271, 1032, 936. MS(ESI) m/z : 393.12 $[\text{M}-\text{H}]^+$ (100%).



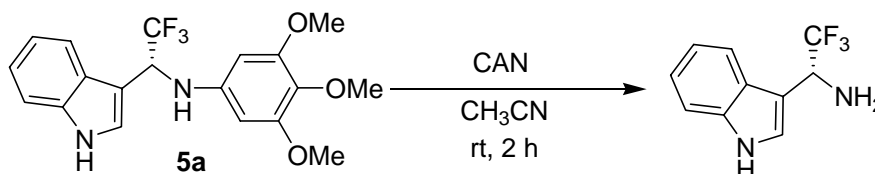
N-((R)-2,2-difluoro-1-(1H-indol-3-yl)ethyl)-3,4,5-trimethoxybenzenamine (6a), yield >99%, ee 93.5 %, [Daicel Chiralcel AD-H, Hexanes / i PrOH = 80 / 20, 0.5 mL /min, $\lambda = 254$ nm, t (minor) = 42.529 min, t (major) = 44.684 min]; $[\alpha]_{\text{D}}^{20} = +21.0$ ($c = 1.0$, CH_2Cl_2). **M. p.** 140-142 °C. ^{19}F NMR (376 MHz, CDCl_3) δ [ppm] -123.3 ~ -126.6 (m, 2F). ^1H NMR (500 MHz, DMSO) δ [ppm] 11.08 (br, 1H), 7.75 (d, $J = 8.0$

Hz, 1H), 7.43 (d, $J = 2.5$ Hz, 1H), 7.35 (d, $J = 8.0$ Hz, 1H), 7.08-7.05 (m, 1H), 6.99-6.96 (m, 1H), 6.39-6.16 (m, 1H), 6.09 (s, 2H), 5.92 (d, $J = 9.0$ Hz, 1H), 5.20-5.12 (m, 1H), 3.63 (s, 6H), 3.46 (s, 3H). ^{13}C NMR (150MHz, $\text{CDCl}_3 + \text{DMSO}$) δ [ppm] 153.7, 144.7, 136.5, 129.5, 127.0, 125.0, 121.6, 119.5, 119.2, 116.7 (t, $^1J_{\text{C-F}} = 241.2$ Hz), 112.0, 110.4, 91.5, 60.5, 56.0, 52.5 (t, $^2J_{\text{C-F}} = 22.5$ Hz). IR (KBr) ν (cm^{-1}) 1611, 1513, 1448, 1234, 1125, 1028, 790, 741. MS(ESI) m/z : 361.30 $[\text{M-H}]^+(100\%)$.



N-((R)-2,2-difluoro-1-(5-fluoro-1H-indol-3-yl)ethyl)-3,4,5-trimethoxybenzenamine (6b), yield >99%, ee 93 %, [Daicel Chiralcel AD-H, Hexanes / *i*PrOH = 80 / 20, 0.5 mL /min, $\lambda = 254$ nm, t (major) = 35.943 min, t (minor) = 39.148 min]; $[\alpha]_{\text{D}}^{20} = +28.0$ ($c = 1.0$, CH_2Cl_2). **M. p.** 158-160 °C. ^{19}F NMR (376 MHz, CDCl_3) δ [ppm] -123.68 ~ -123.74 (m, 1F), -124.1 ~ -126.2(m, 2F). ^1H NMR (500 MHz, CDCl_3) δ [ppm] 8.31 (br, 1H), 7.34 -7.32 (m, 3H), 6.24-6.01 (m, 1H), 5.94 (s, 2H), 4.96-4.91 (m, 1H), 4.08 (br, 1H), 3.75(s, 3H), 3.74 (s, 6H). ^{13}C NMR (150MHz, $\text{CDCl}_3 + \text{DMSO}$) δ [ppm] 157.0 (d, $^1J_{\text{C-F}} = 232.2$ Hz), 153.1, 143.3, 132.6, 129.6, 126.0 (d, $J = 9.9$ Hz), 125.3 (d, $J = 8.4$ Hz) , 115.4 (t, $^1J_{\text{C-F}} = 251.7$ Hz), 112.0, 109.6, 109.4 (d, $J = 20.1$ Hz), 103.1 (d, $J = 29.8$ Hz), 90.9, 60.3, 55.3, 53.3 (t, $^2J_{\text{C-F}} = 24.3$ Hz). IR (KBr) ν (cm^{-1}) 1611, 1599, 1444, 1234, 1207, 1123, 1029, 1006, 785. MS (ESI) m/z (%): 379.03 $[\text{M-H}]^+(100\%)$.

General procedure for the removal of the trimethoxyphenyl group of (*R*)-**5a**.^[4]



A CH₃CN (mL) solution of **5a** (0.1 mmol) was cooled to 0 °C and a solution of ammonium cerium nitrate (CAN, 0.44 mmol, in 1.5 mL water) was directly added. The resulting mixture was stirred under room temperature for 2 h. The aqueous phase was extracted with dichloromethane (15 mL × 3). The combined organic phase was dried over anhydrous MgSO₄ and concentrated by evaporation under reduced pressure. The residue was isolated on a silica gel column eluted with petroleum ether / AcOEt (5/1-1/2) to give the desired product: colorless needles, $[\alpha]_{\text{D}}^{20} = -1.0$ (*c* 1.0, EtOH). Mp 129-130 °C (lit.^[5] (*S*)-product, $[\alpha]_{\text{D}}^{20} = +0.7$ (*c* 1.1, EtOH). Mp 130-131 °C). ¹H NMR (CDCl₃): 2.88 (br, 2H, NH₂), 5.60 (m, 1H, CF₃CH), 7.80-7.89 (m, 4H, Indole-H), 7.92-7.95 (m, 1H, Indole-H), 8.15 (br, 1H, NH). ¹⁹F NMR (CDCl₃): 76.6 (d, *J* = 7.5 Hz, 3F, CF₃).

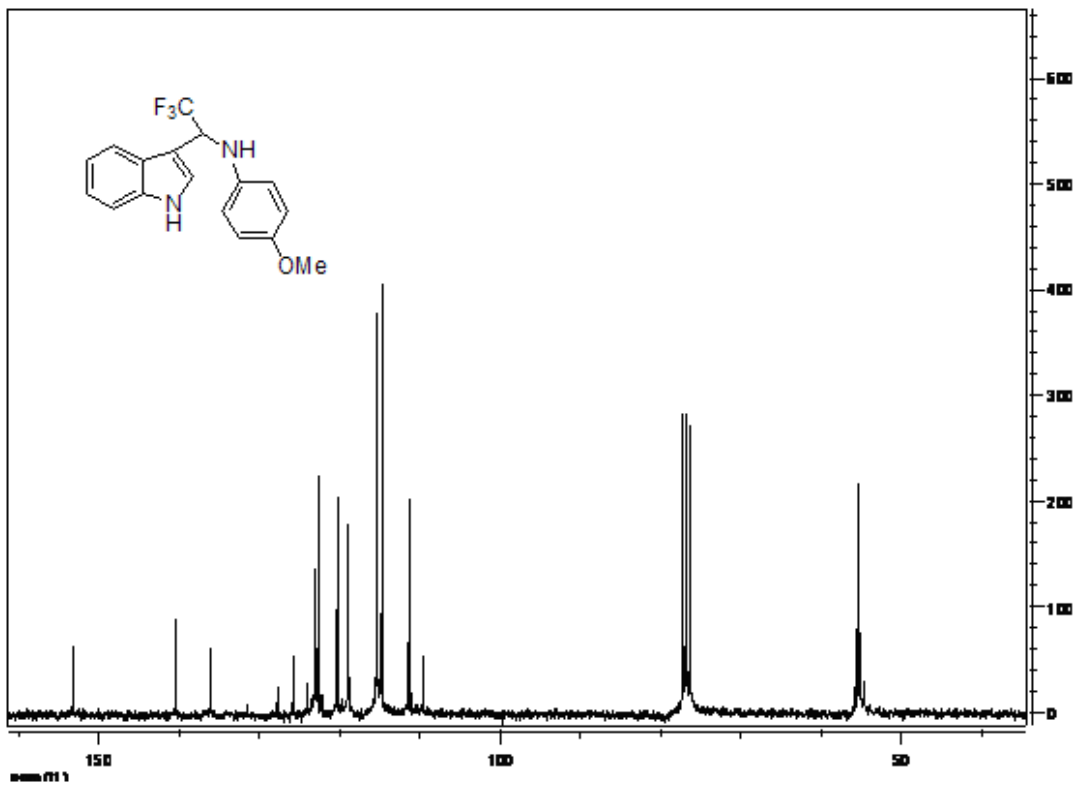
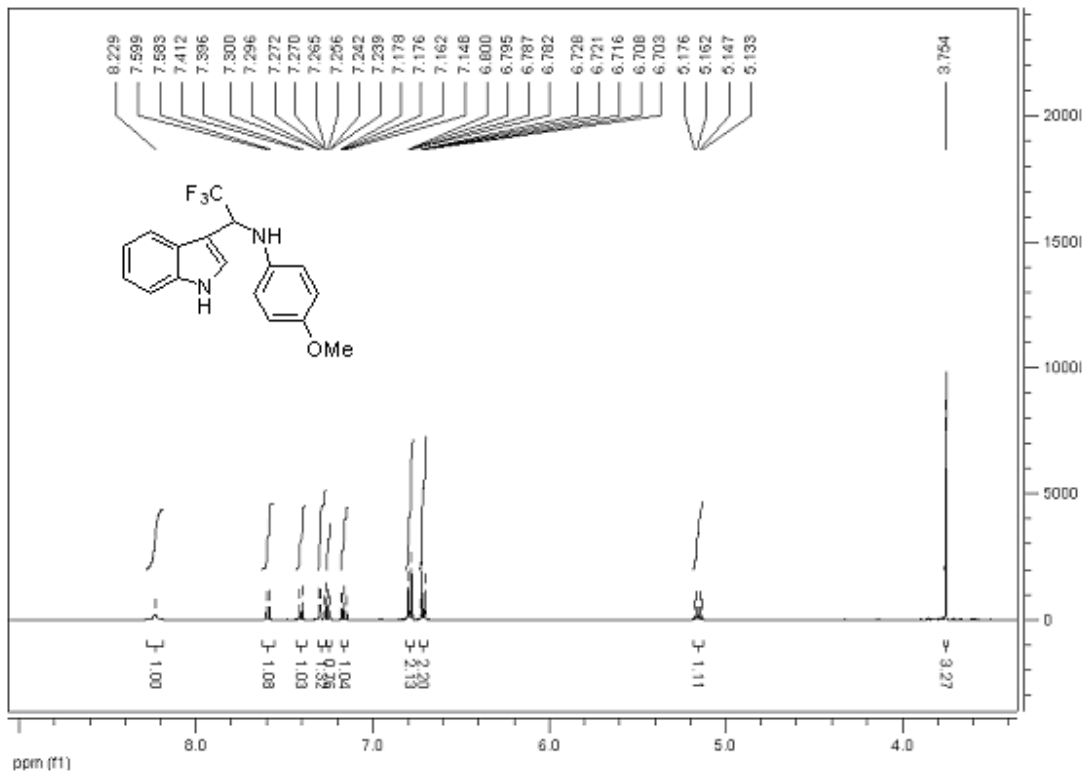
[1] Uraguchi, D.; Terada, M. *J. Am. Chem. Soc.* **2004**, *126*, 5356.

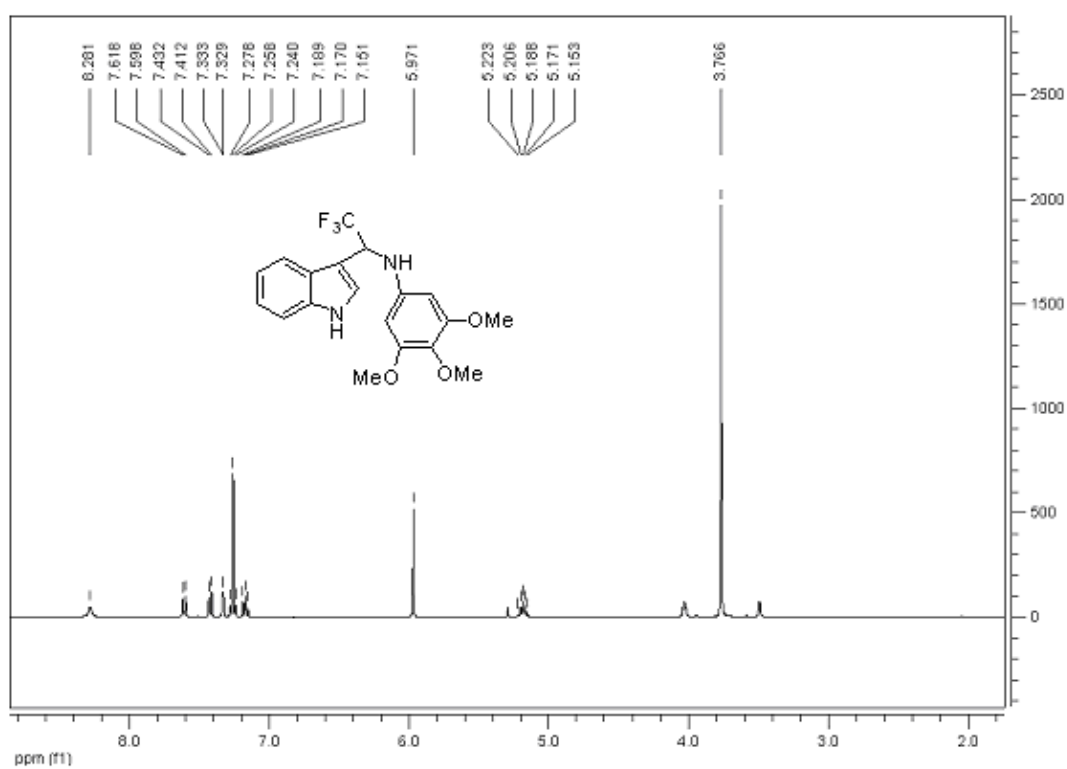
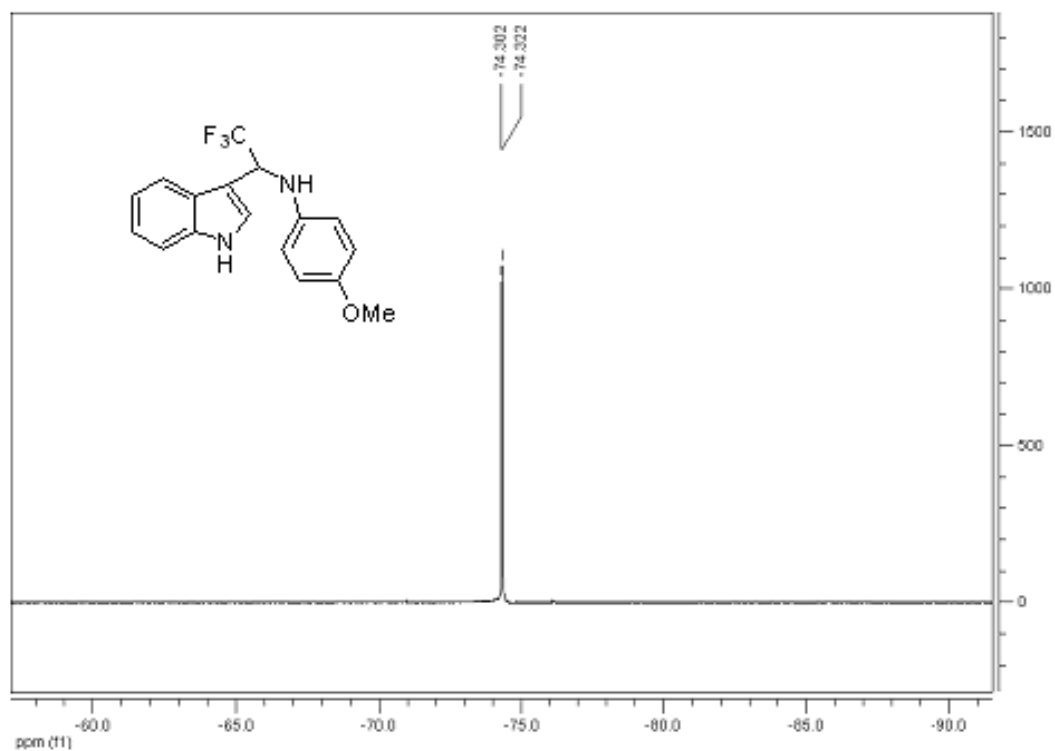
[2] Akiyama, T.; Morita, H.; Itoh, J.; Fuchibe, K. *Org. Lett.* **2005**, *7*, 2583.

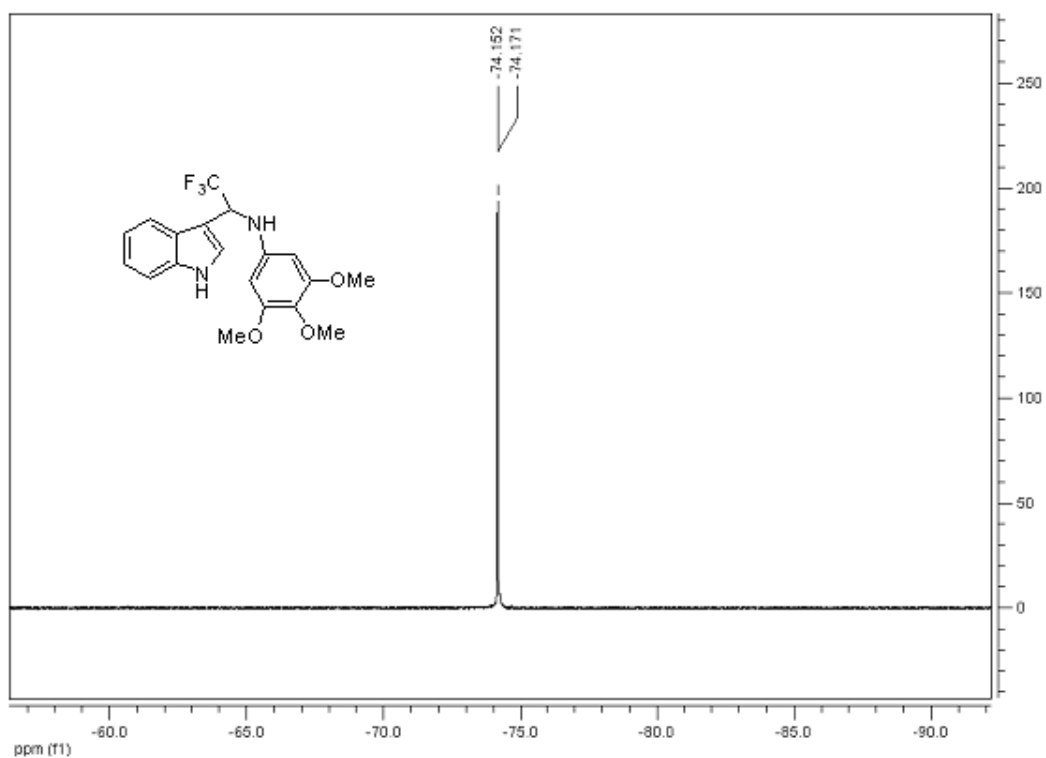
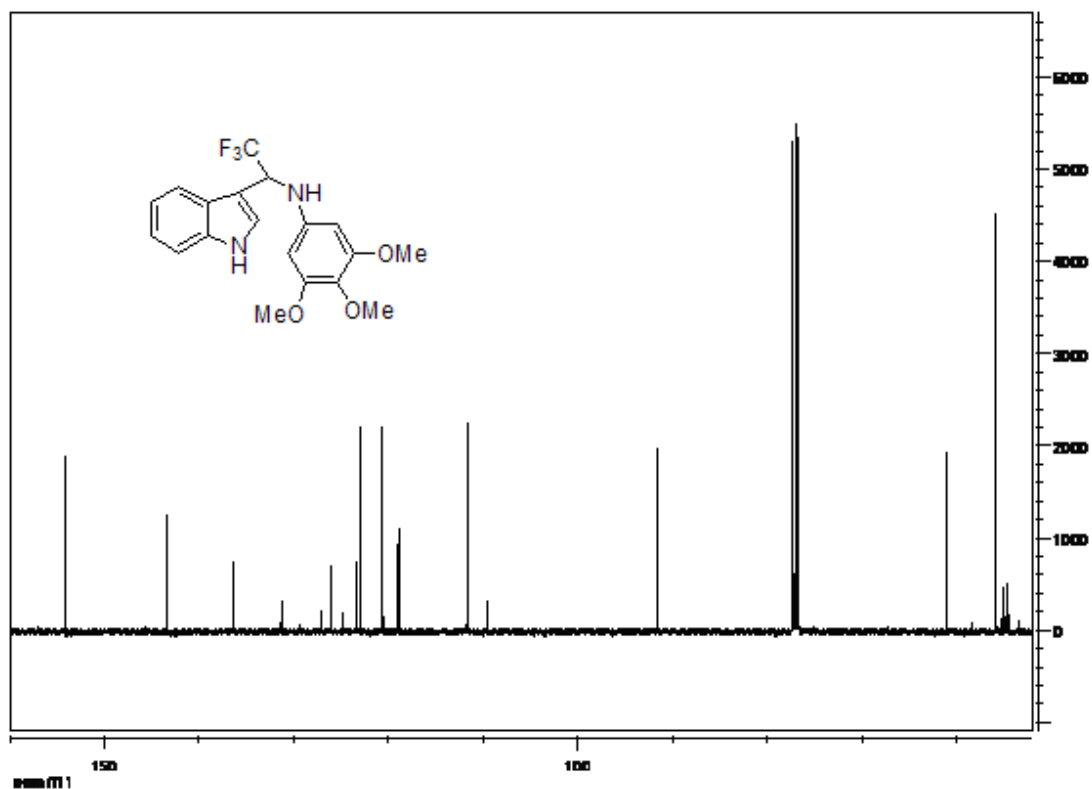
[3] Storer, R. I.; Carrera, D. E.; Ni, Y.; MacMillan, D. W. C. *J. Am. Chem. Soc.* **2006**, *128*, 84.

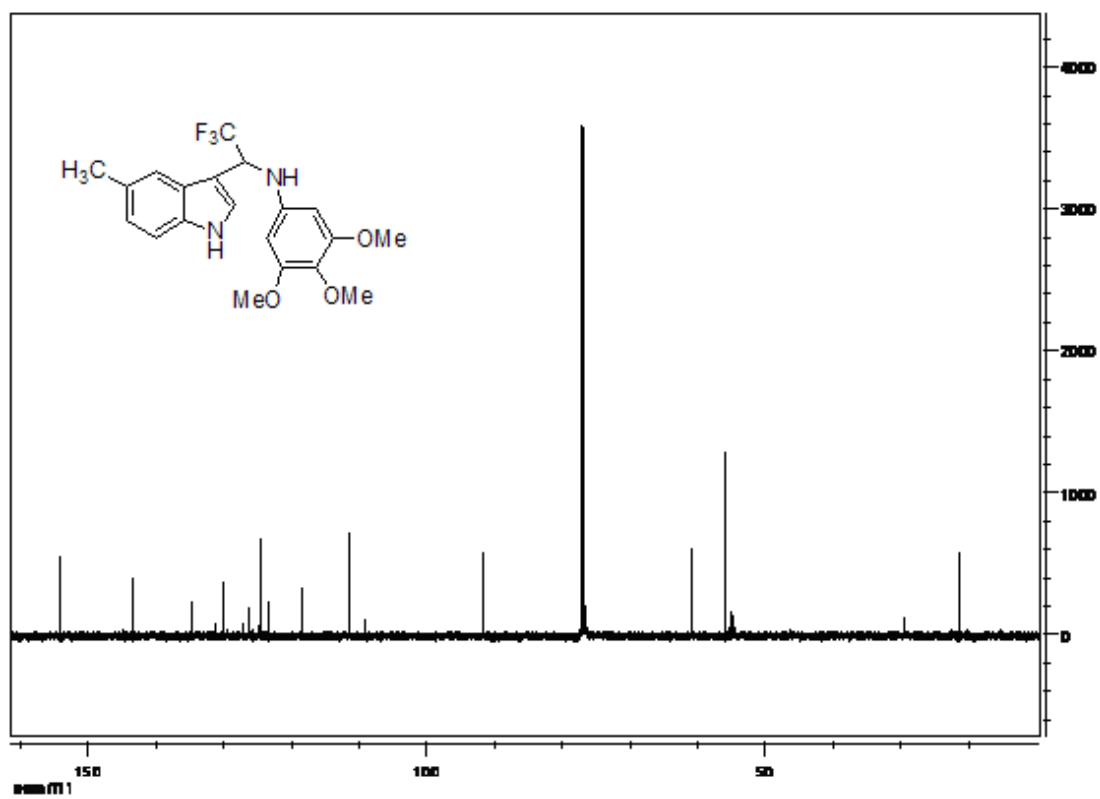
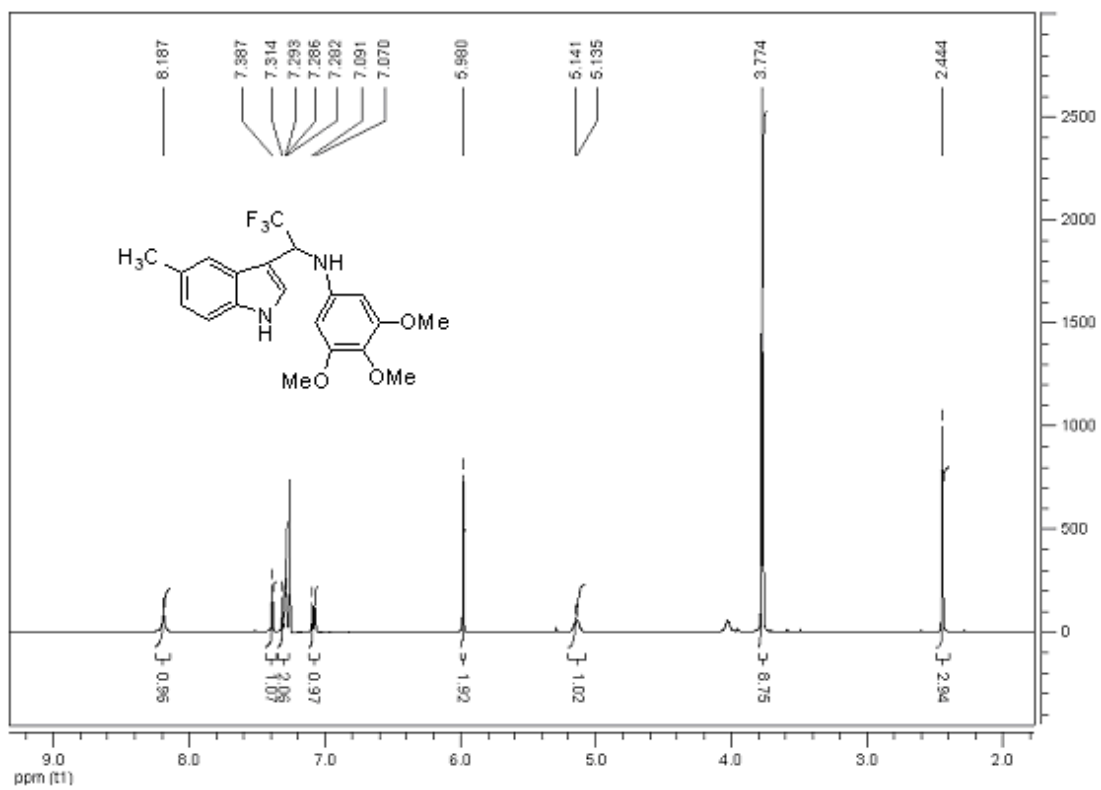
[4] (a) Niwa, Y.; Shimizu, M. *J. Am. Chem. Soc.* **2003**, *125*, 3720. (b) Shang, G.; Yang, Q.; Zhang, X. *Angew. Chem. Int. Ed.* **2006**, *45*, 6360.

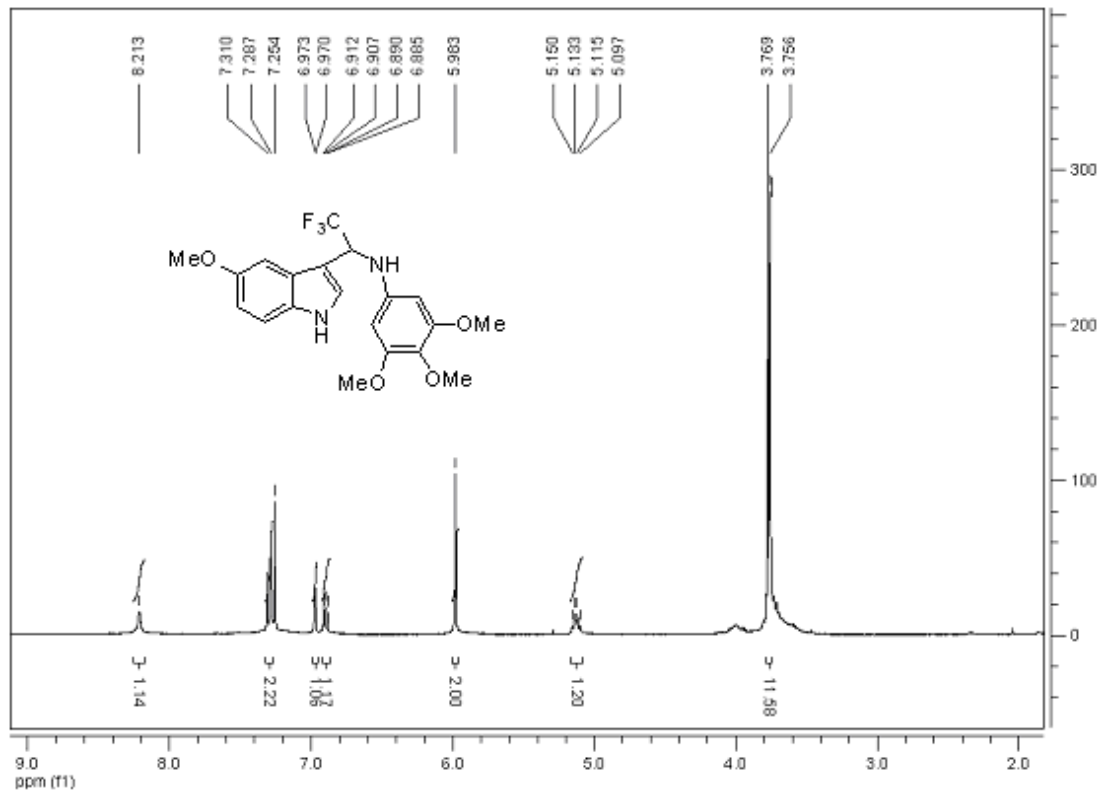
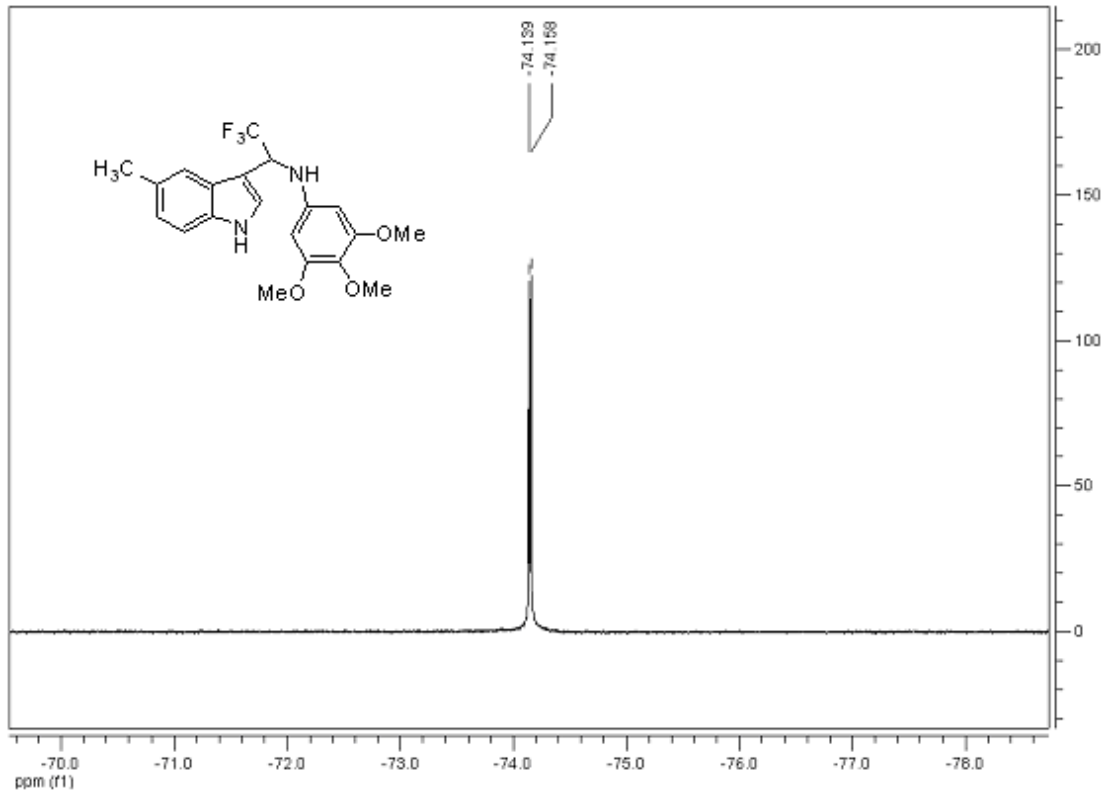
[5] Gong, Y.; Kato, K. *Tetrahedron: Asymmetry* **2001**, *12*, 2121.

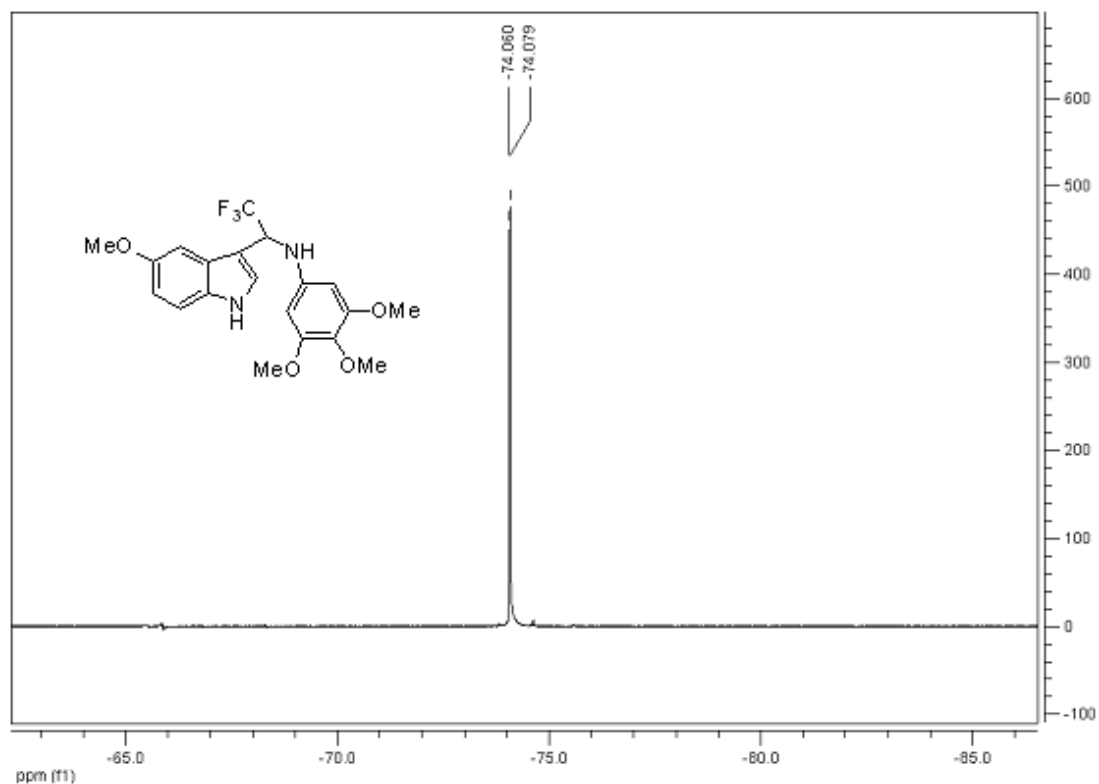
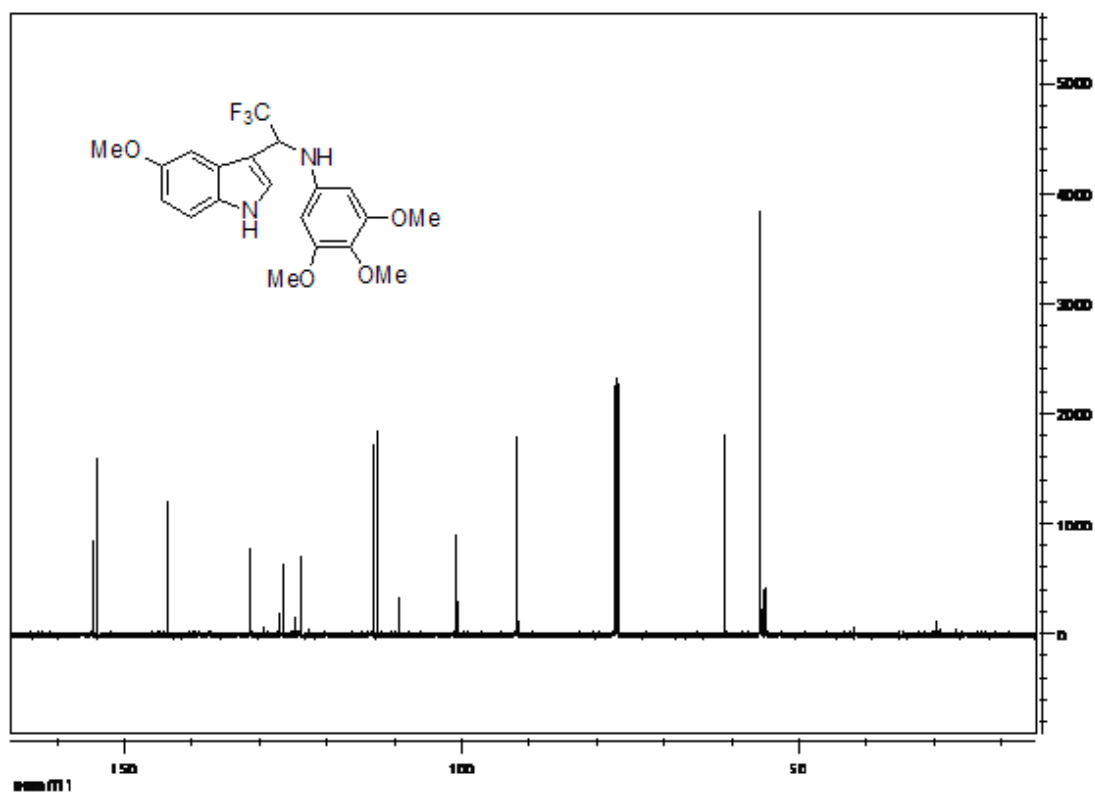


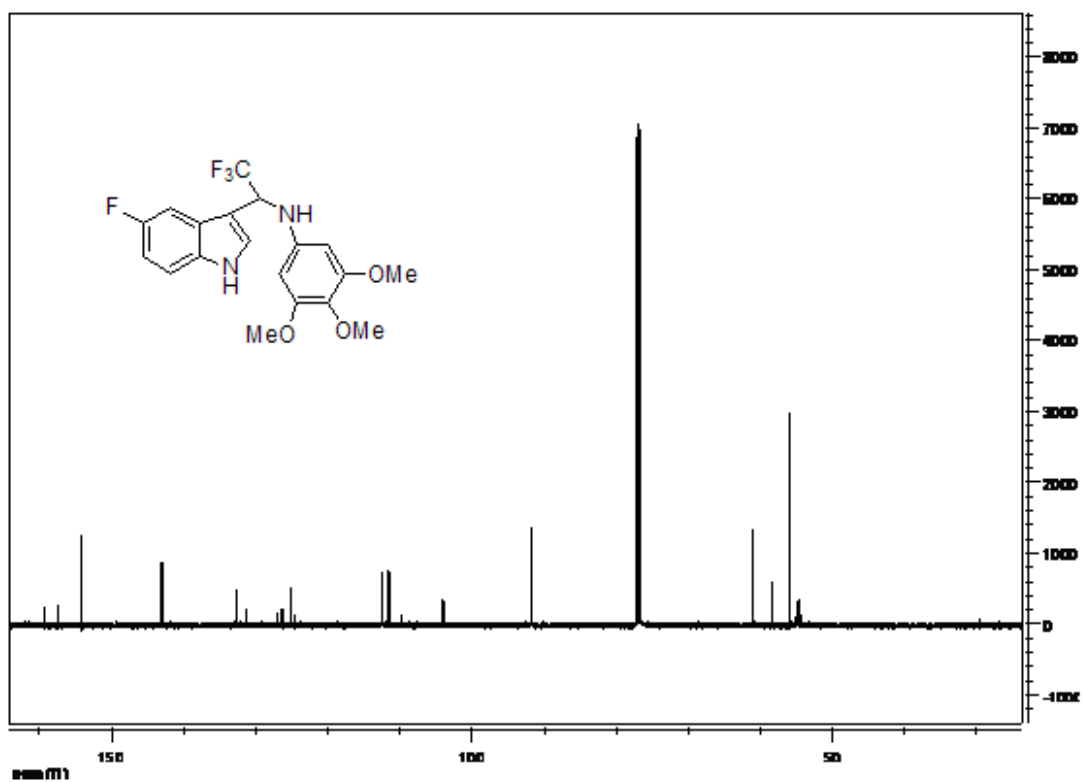
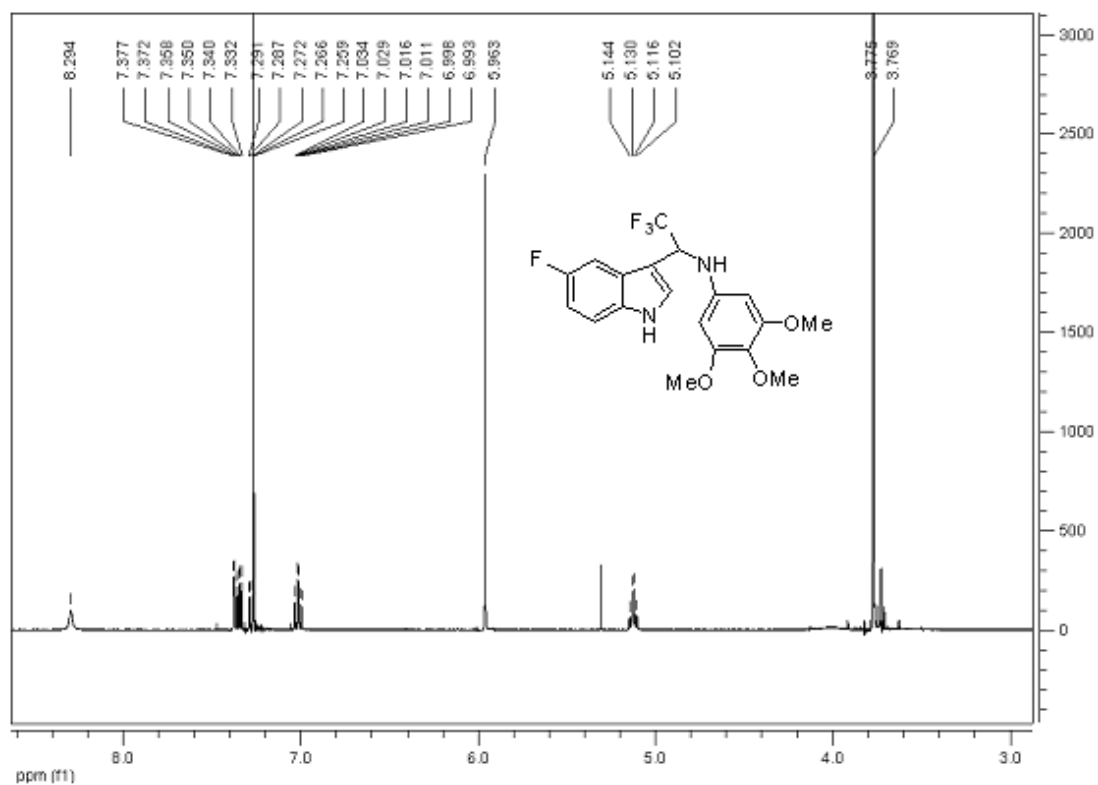


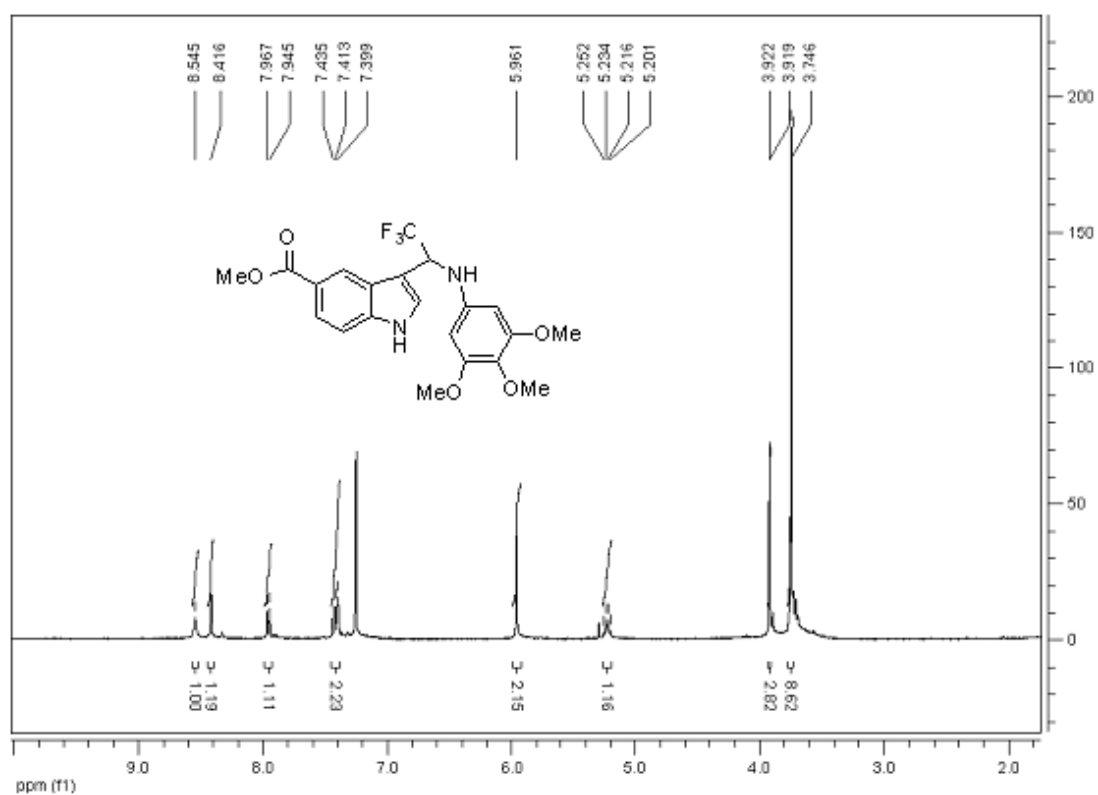
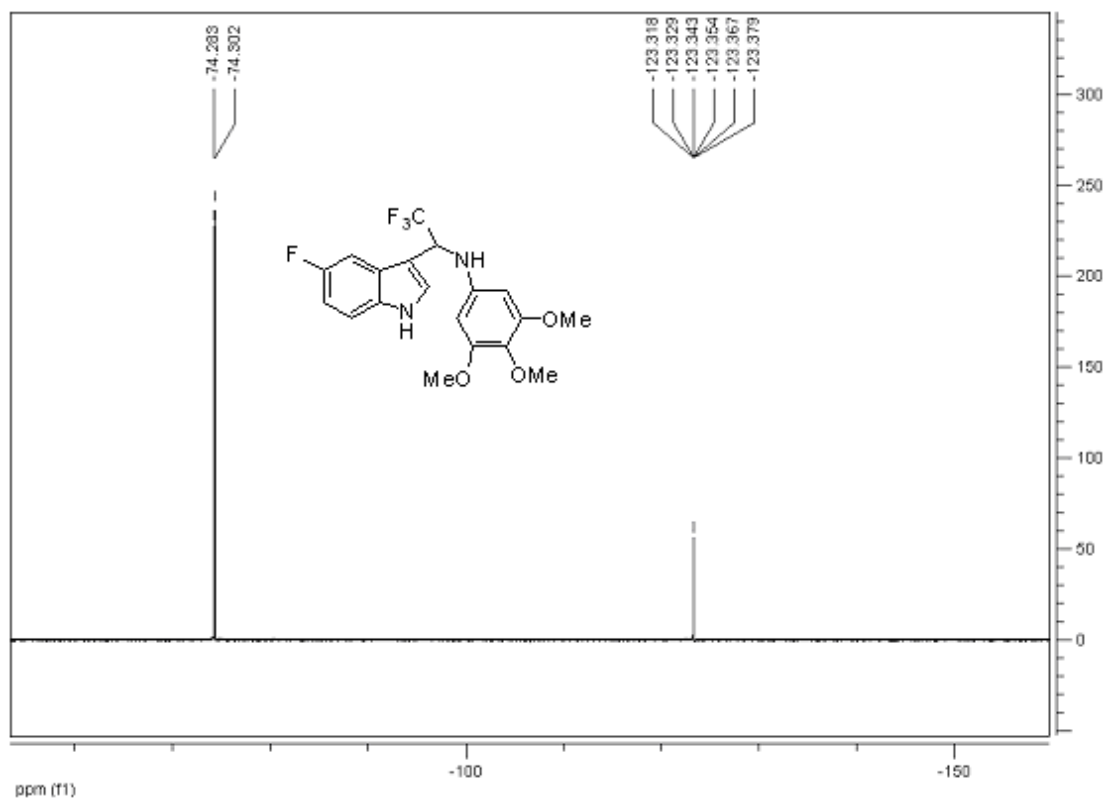


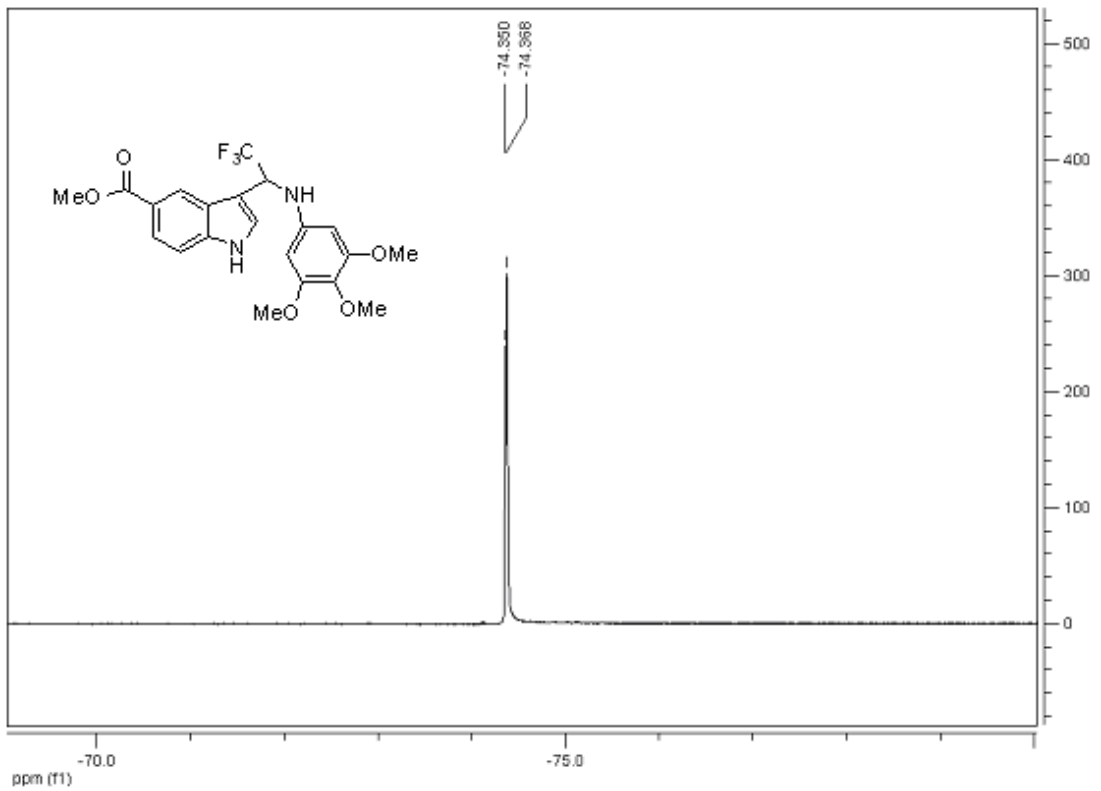
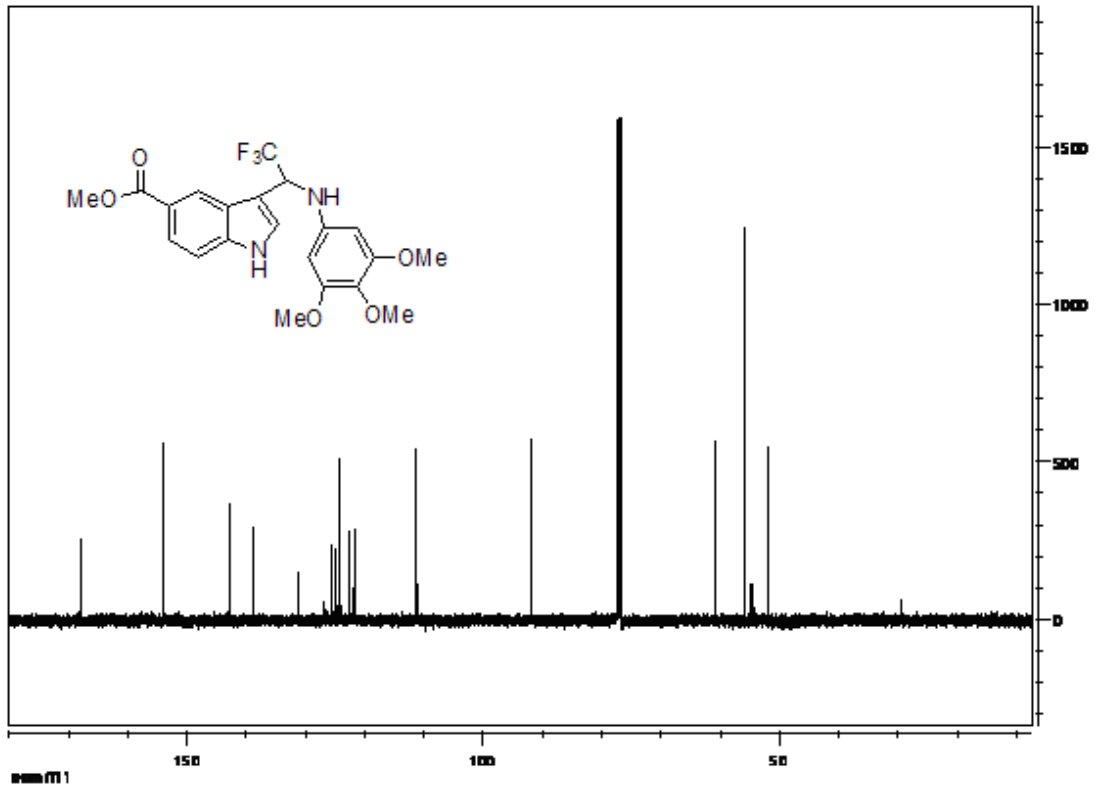


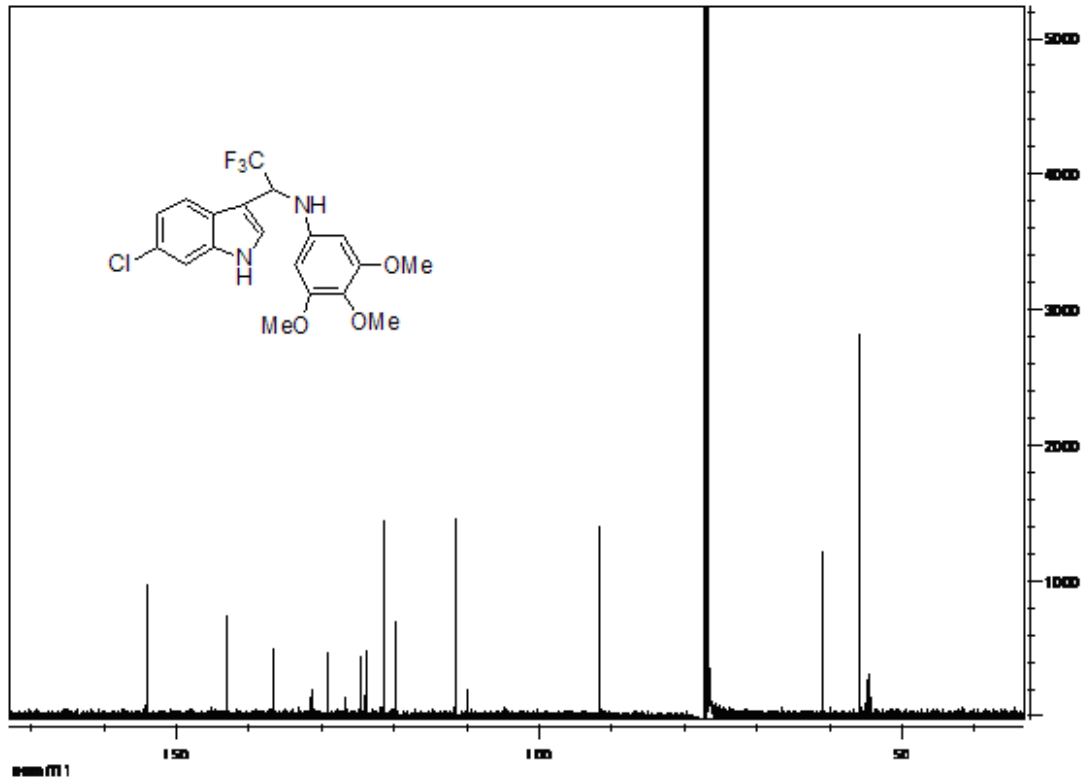
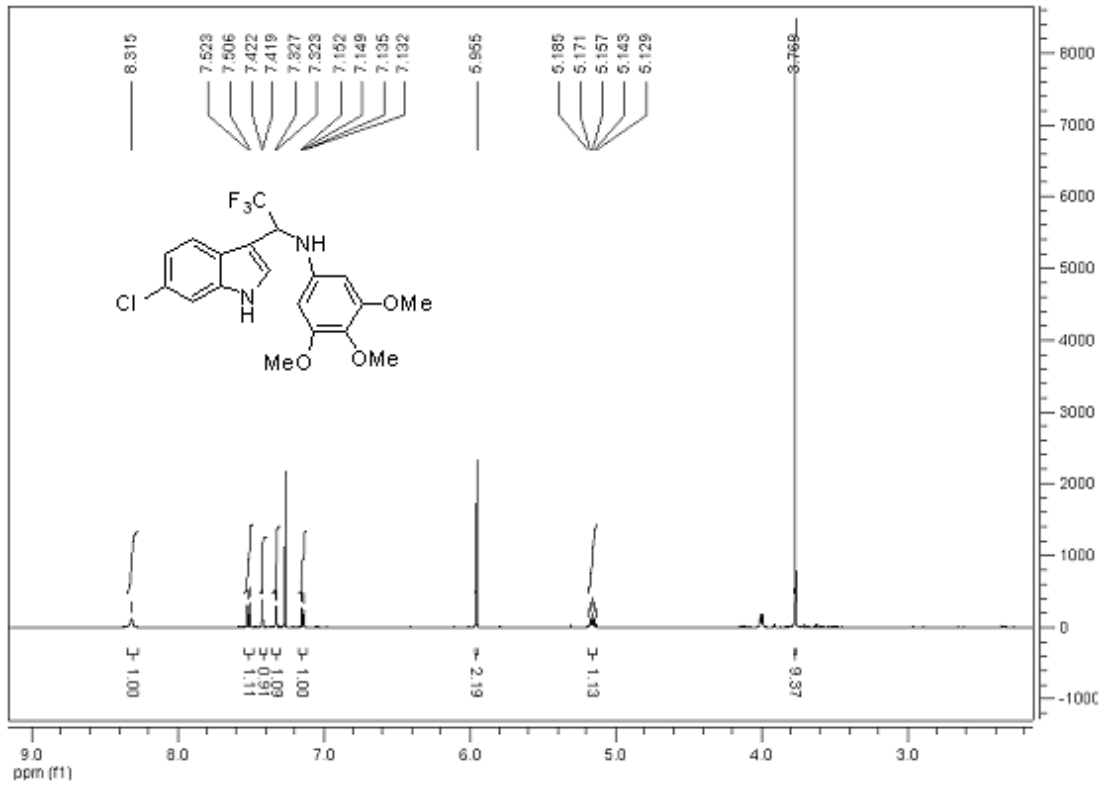


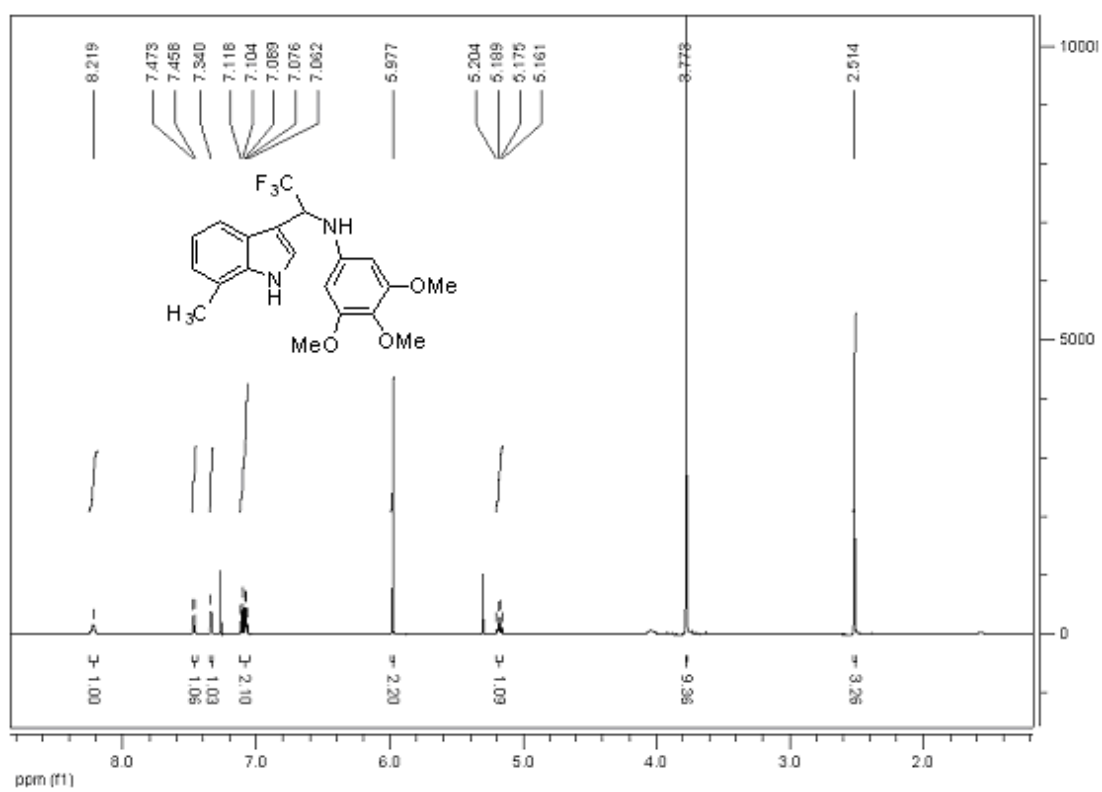
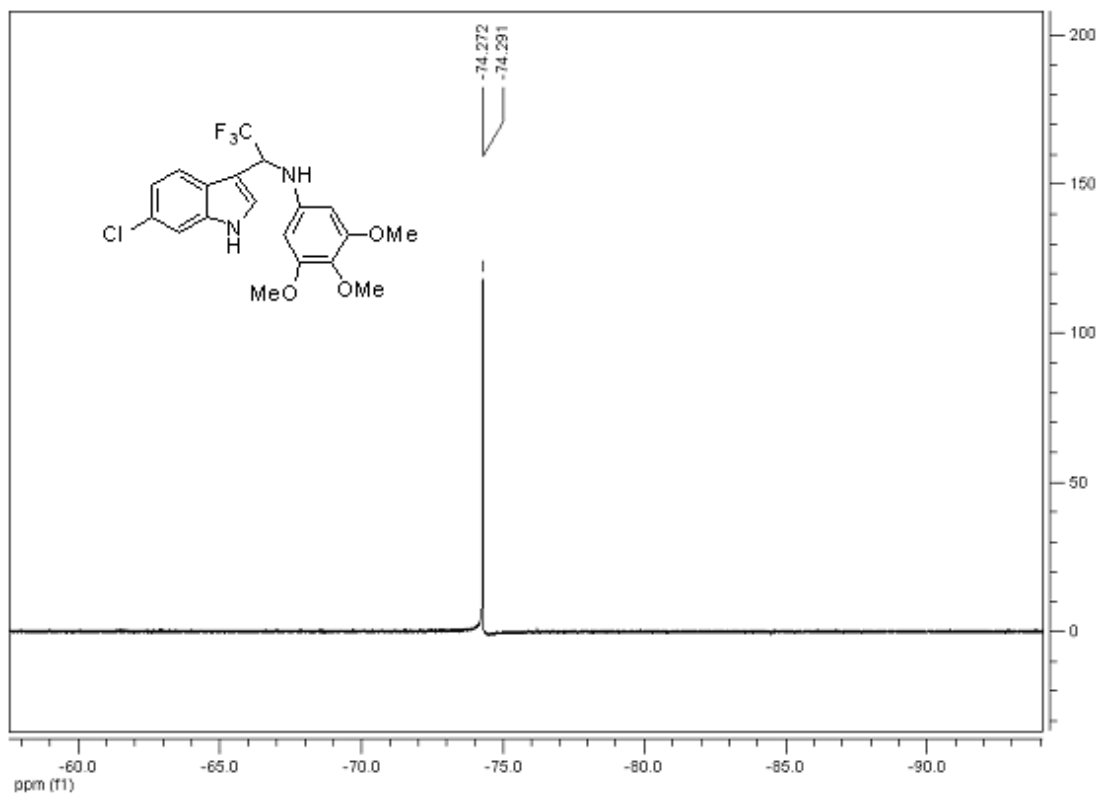


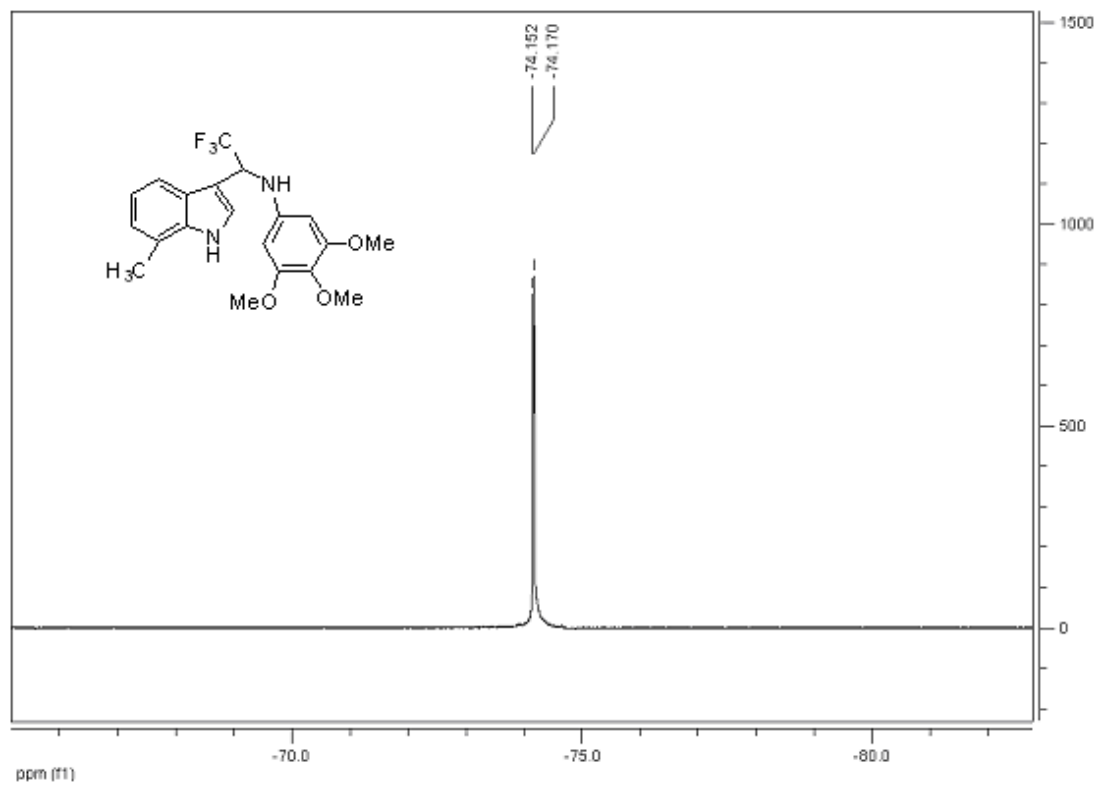
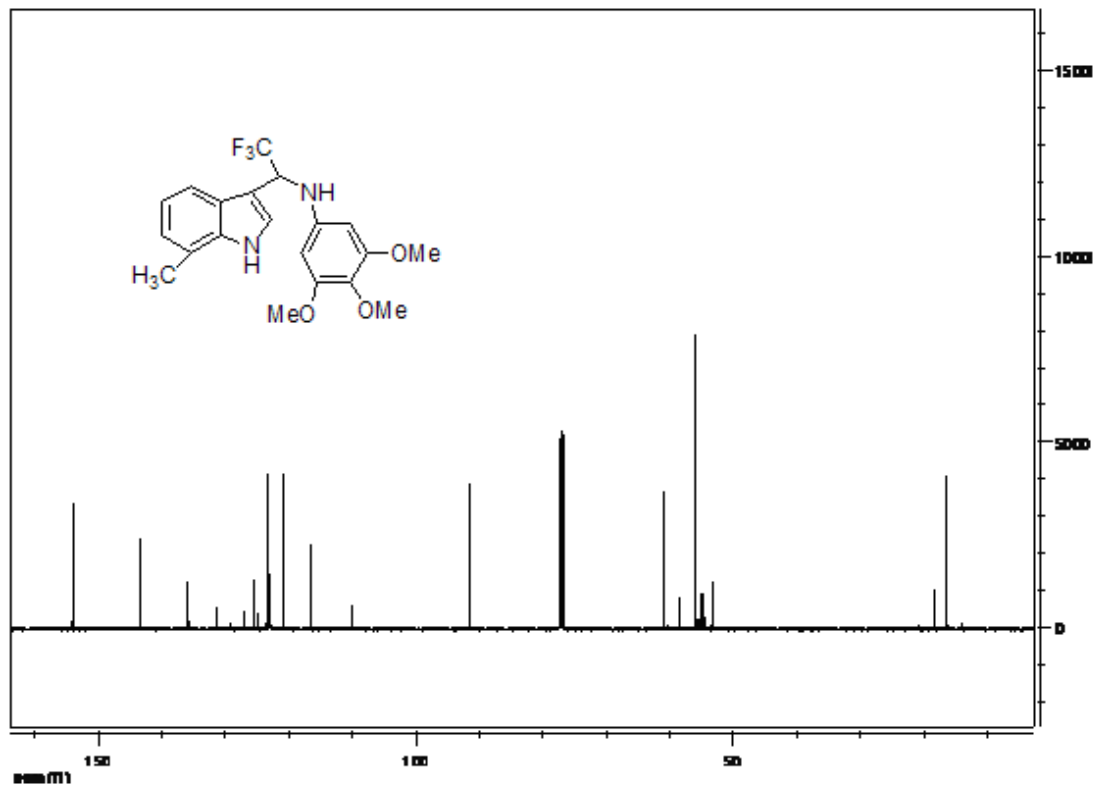


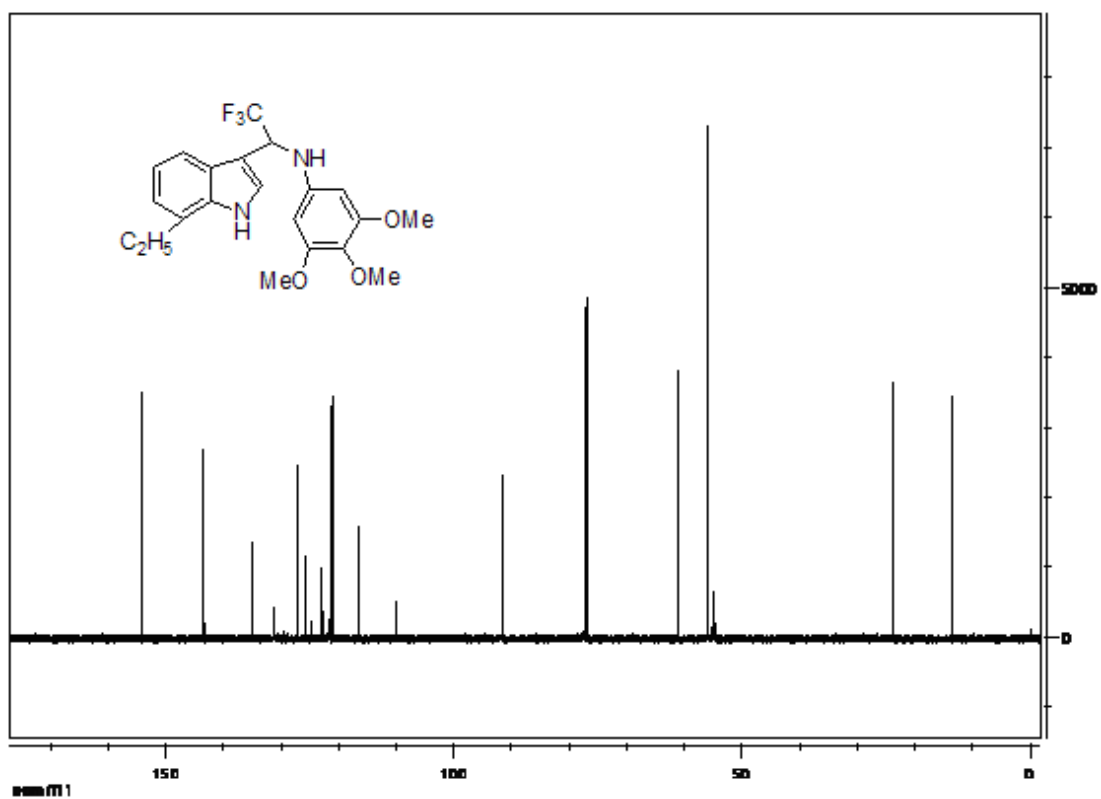
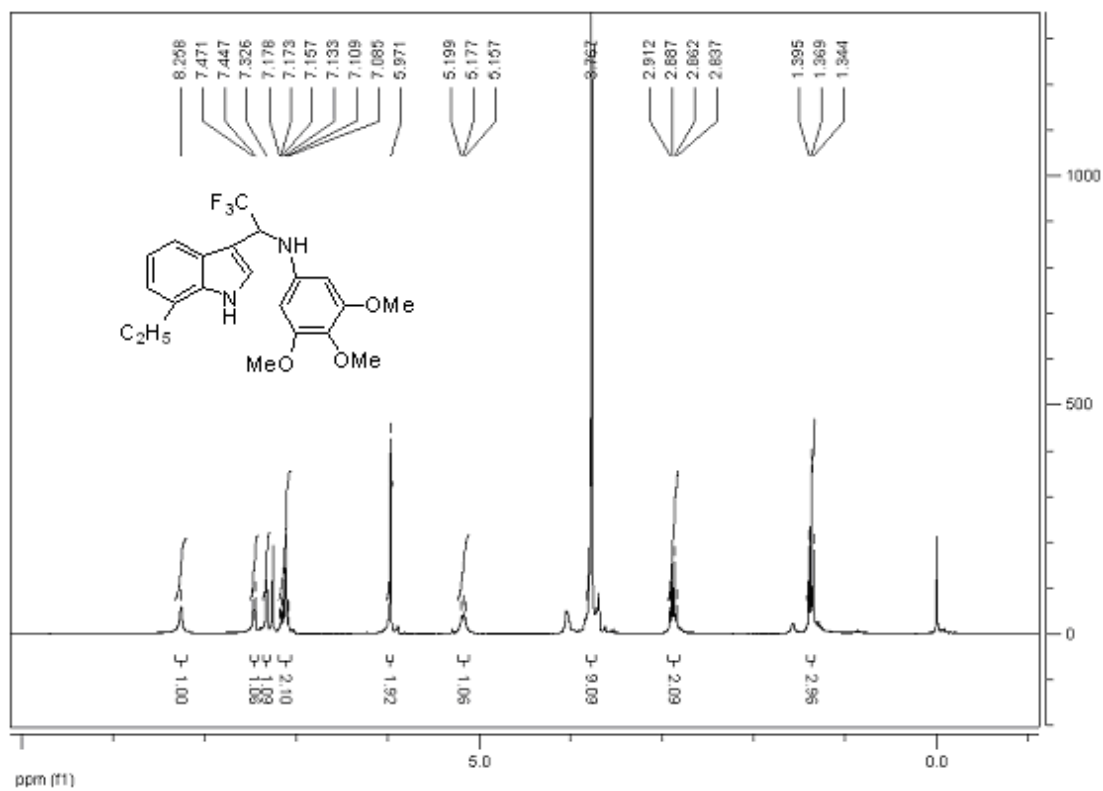


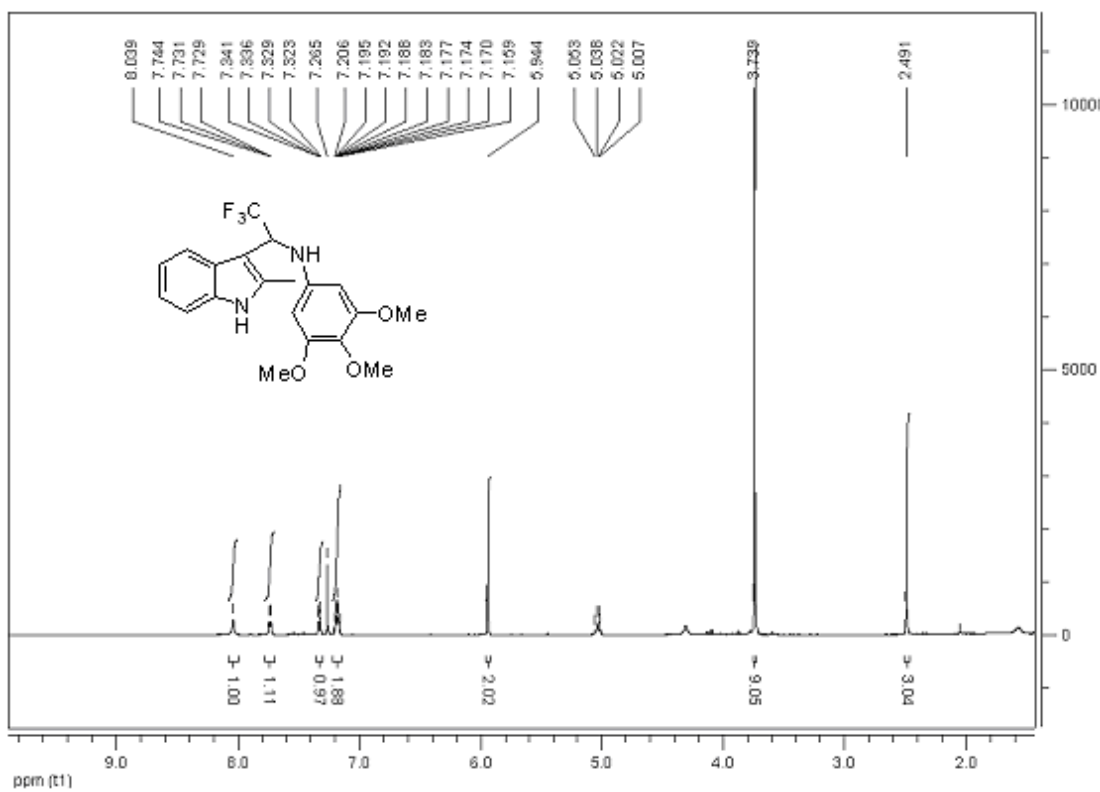
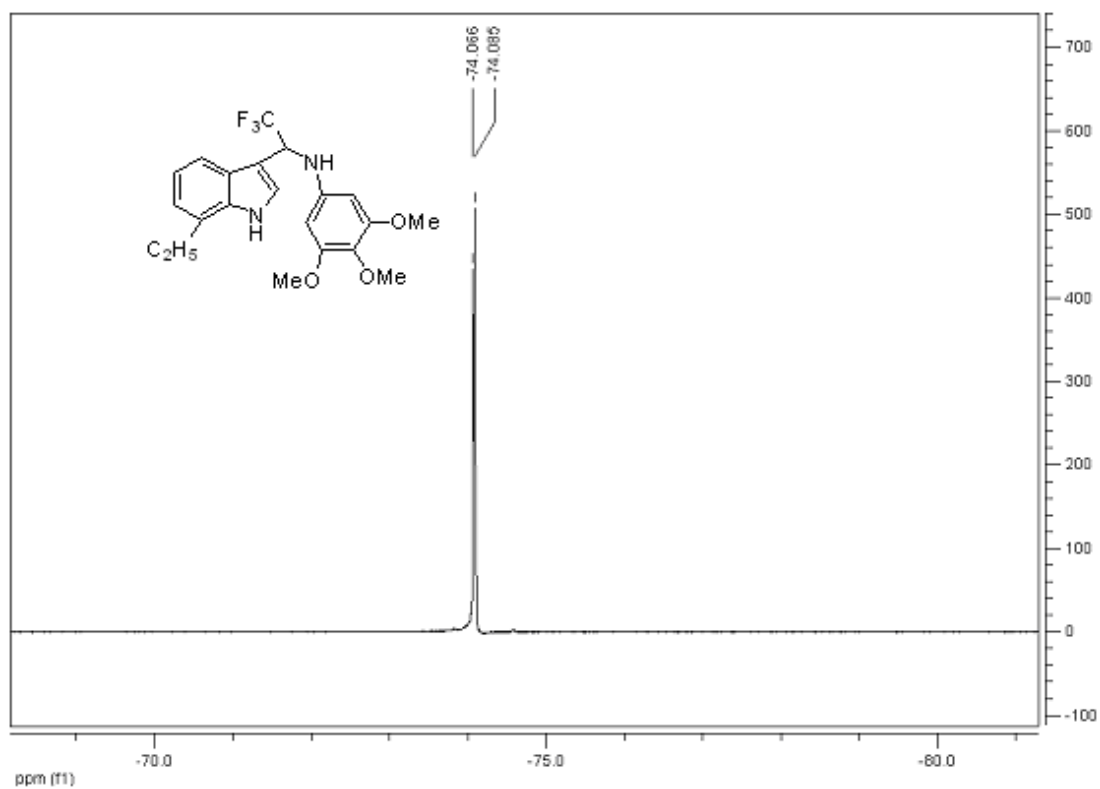


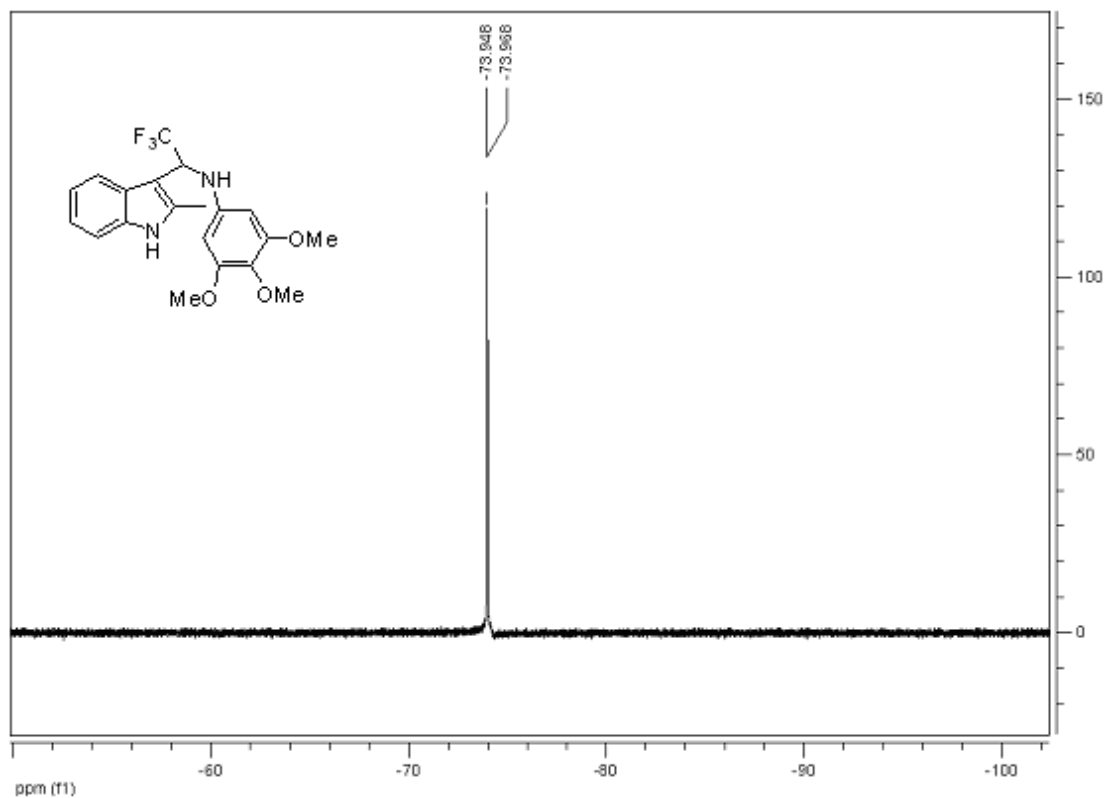
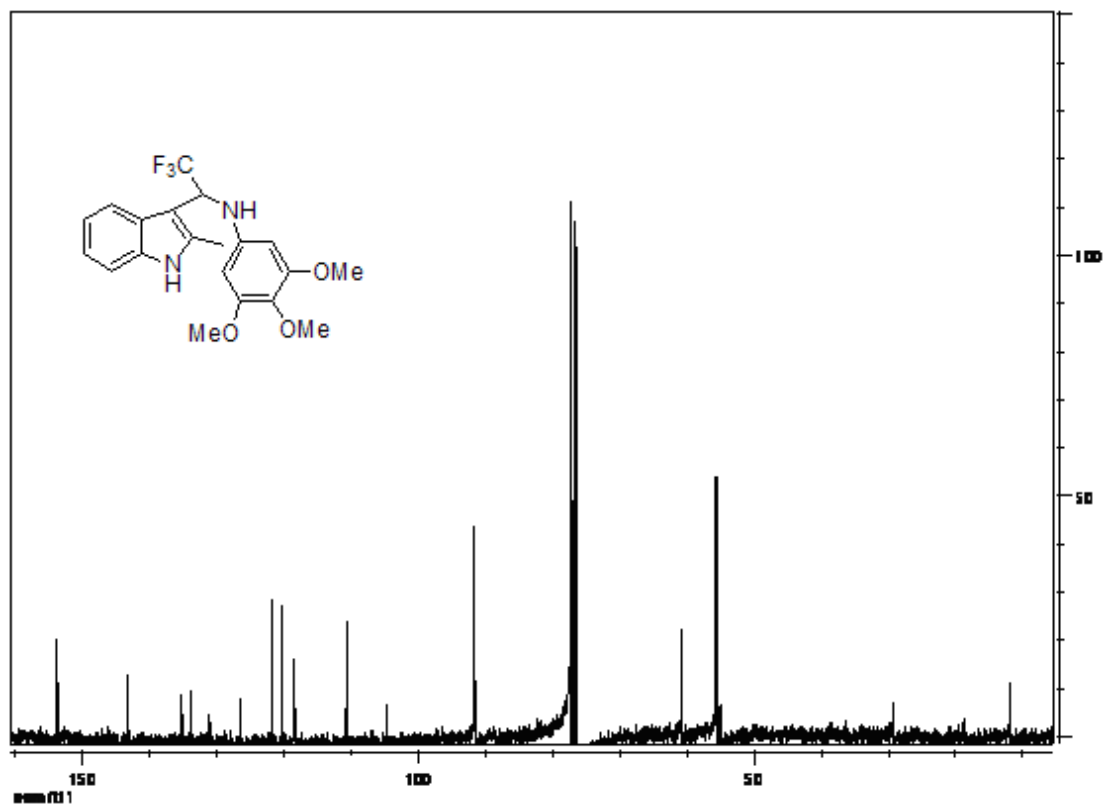


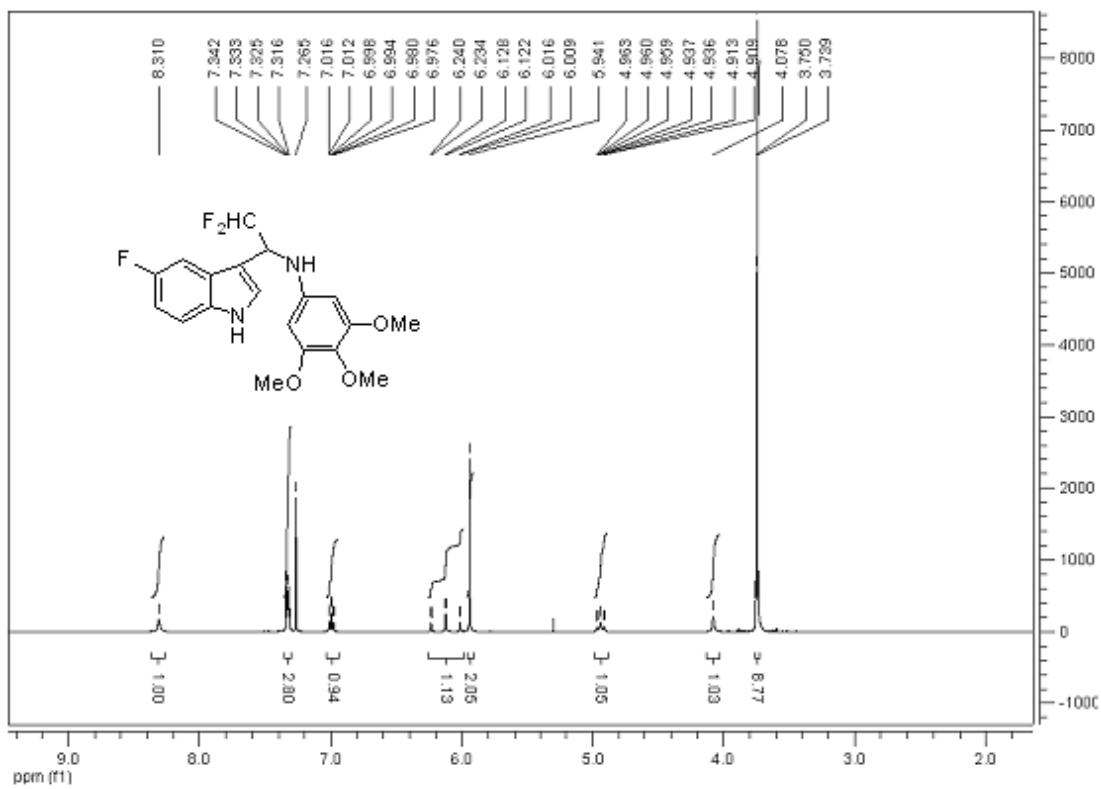
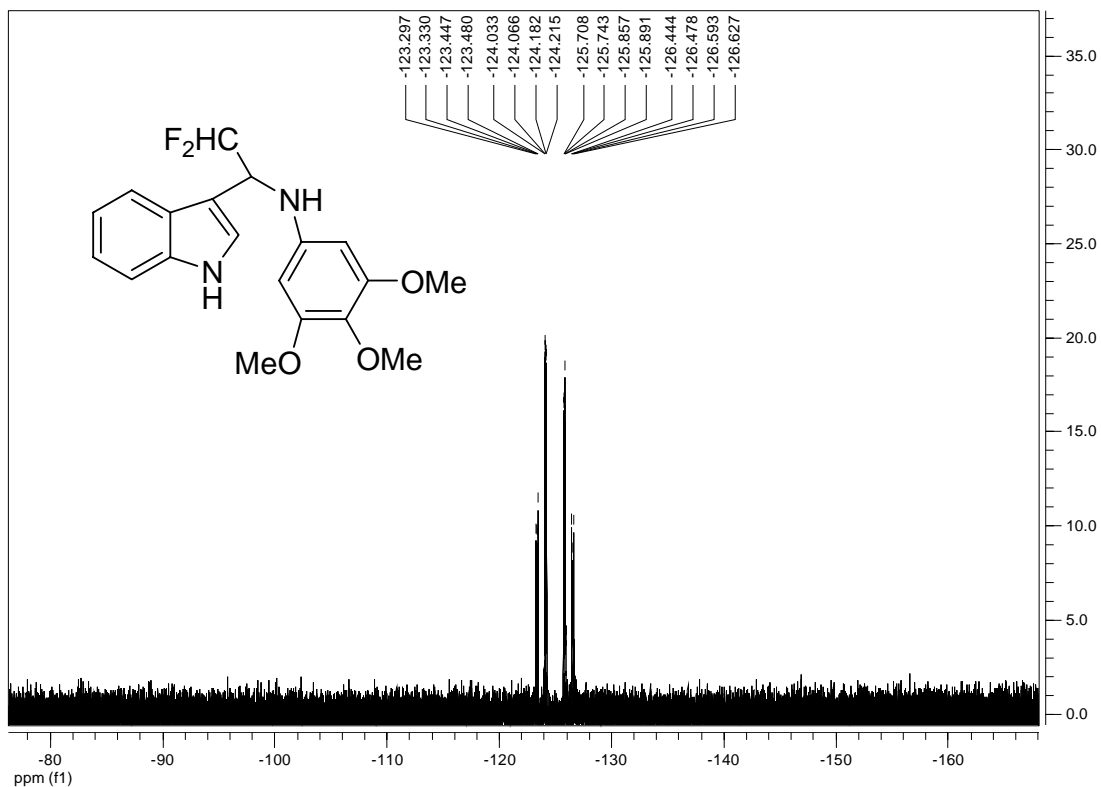


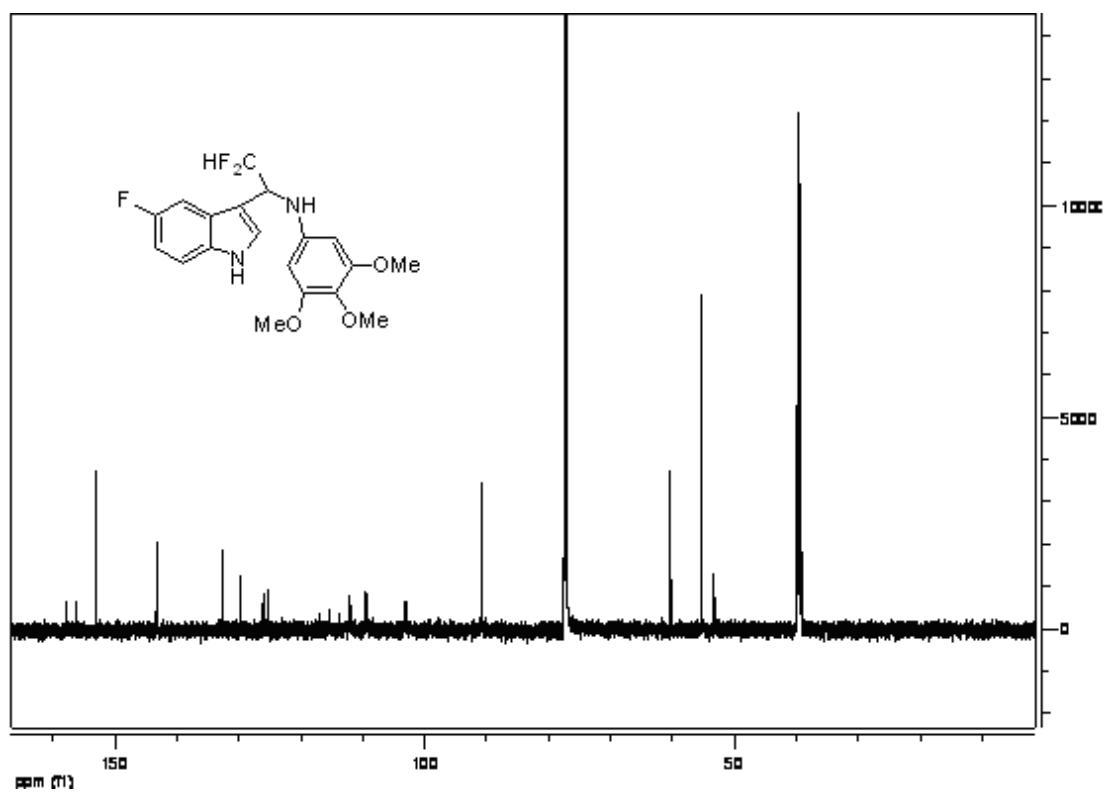
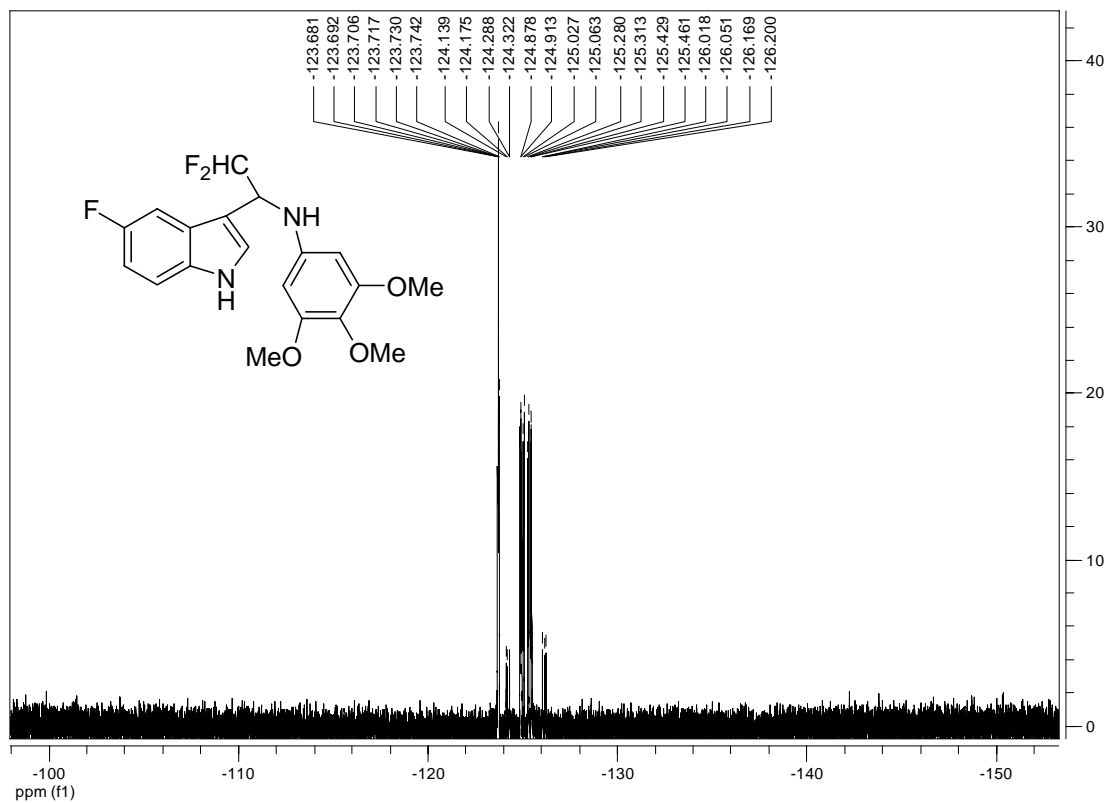




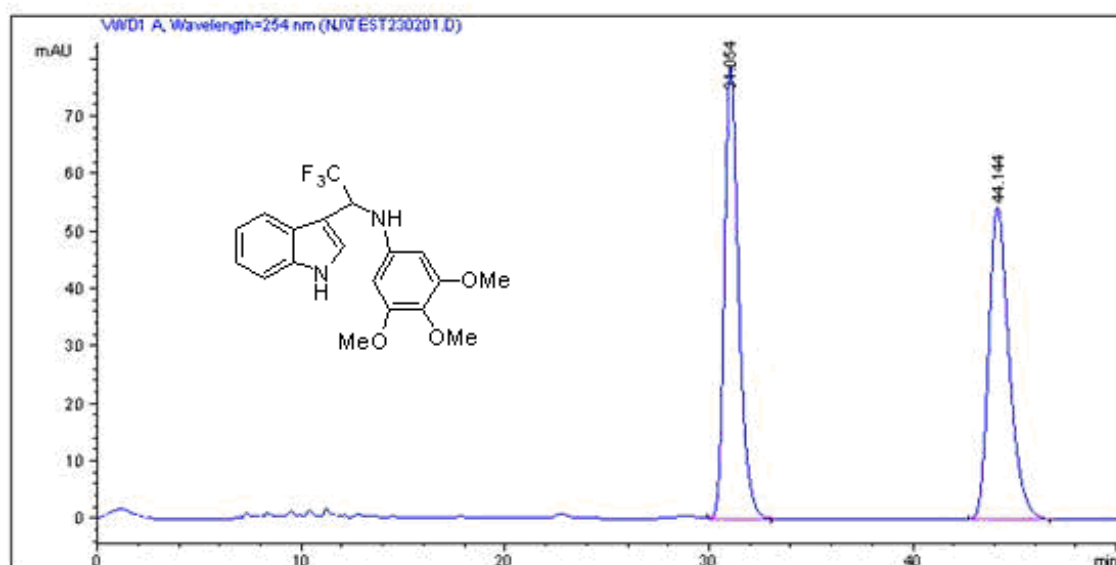








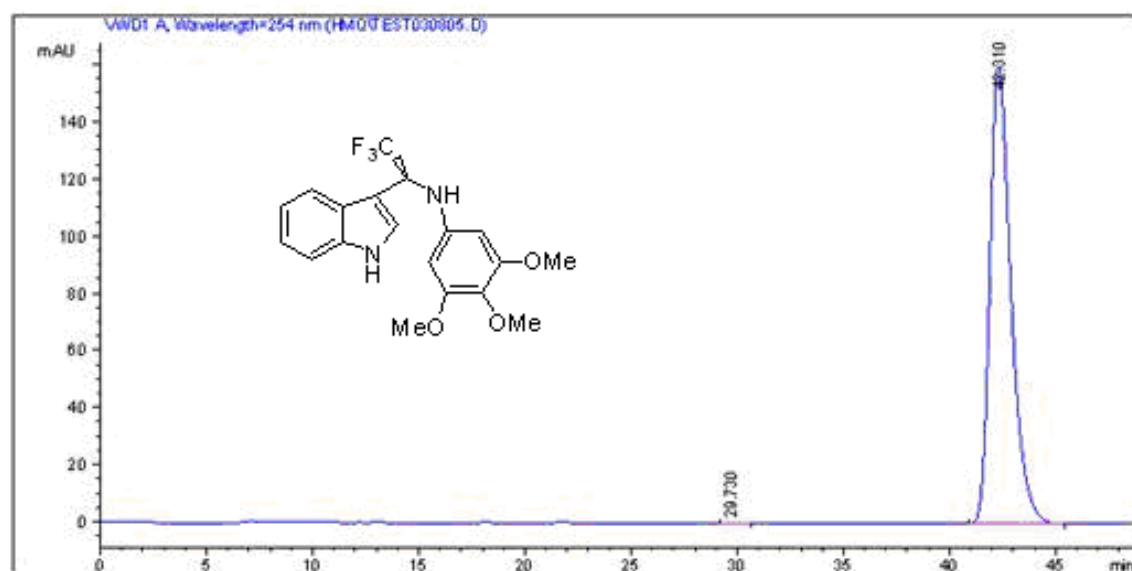
Sample Info : 254nm, i-PrOH:Hexane=20:80, AD-H, 0.5mL/min



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	31.054	VB	0.7271	3935.66650	78.93668	50.2364
2	44.144	BB	1.0145	3799.56030	54.29236	49.7636

Totals : 7635.22681 133.22904

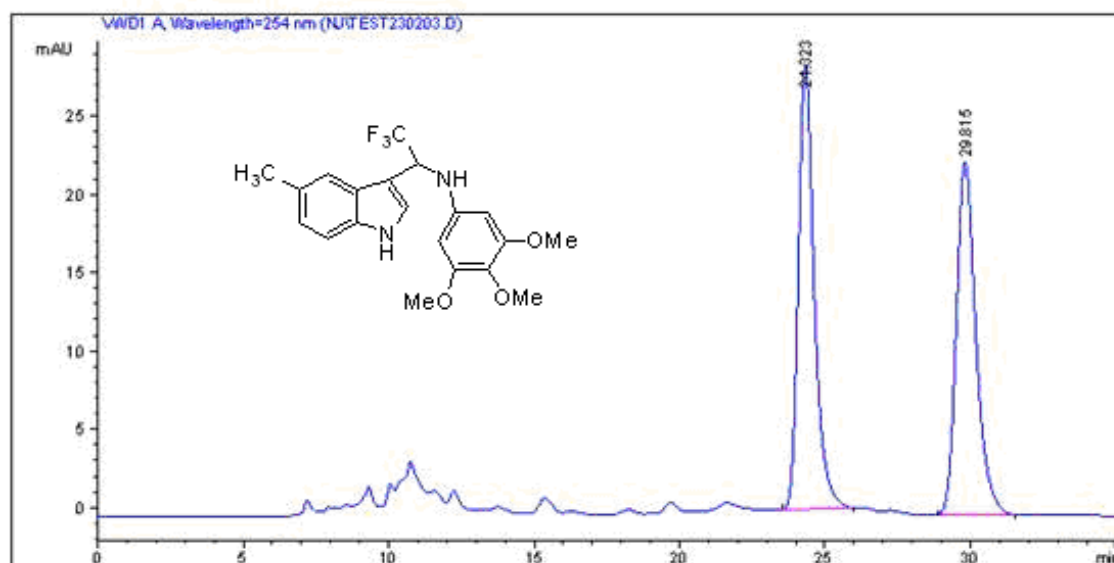


Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	29.730	BB	0.5308	5.31617	1.20133e-1	0.0481
2	42.310	BB	1.0007	1.10510e4	160.61522	99.9519

Totals : 1.10563e4 160.73535

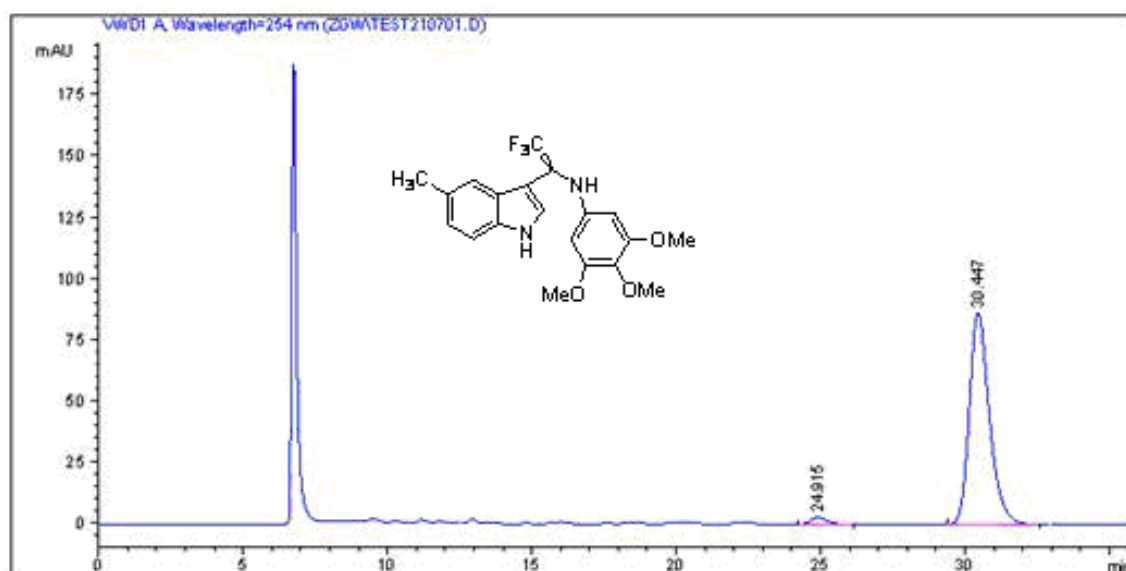
Sample Info : 254nm, i-PrOH:Hexane=20:80, AD-H, 0.5mL/min



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	24.323	BB	0.5641	1078.11609	28.47147	50.5448
2	29.815	BB	0.7029	1054.87463	22.55584	49.4552

Totals : 2132.99072 51.02731

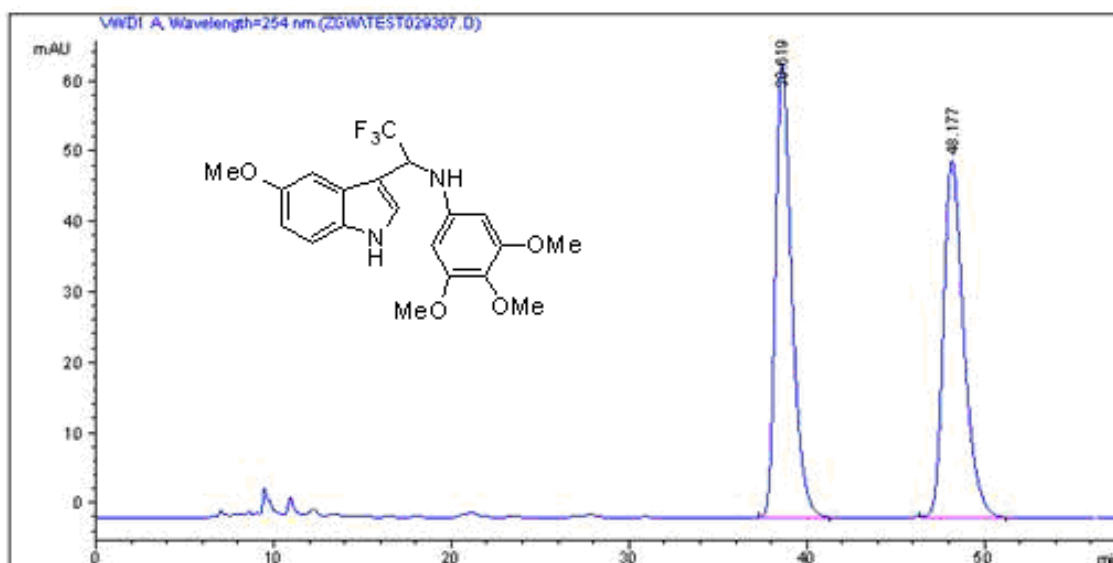


Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	24.915	BB	0.5658	109.40901	2.80468	2.5490
2	30.447	VB	0.7387	4182.77441	86.53175	97.4510

Totals : 4292.18343 89.33643

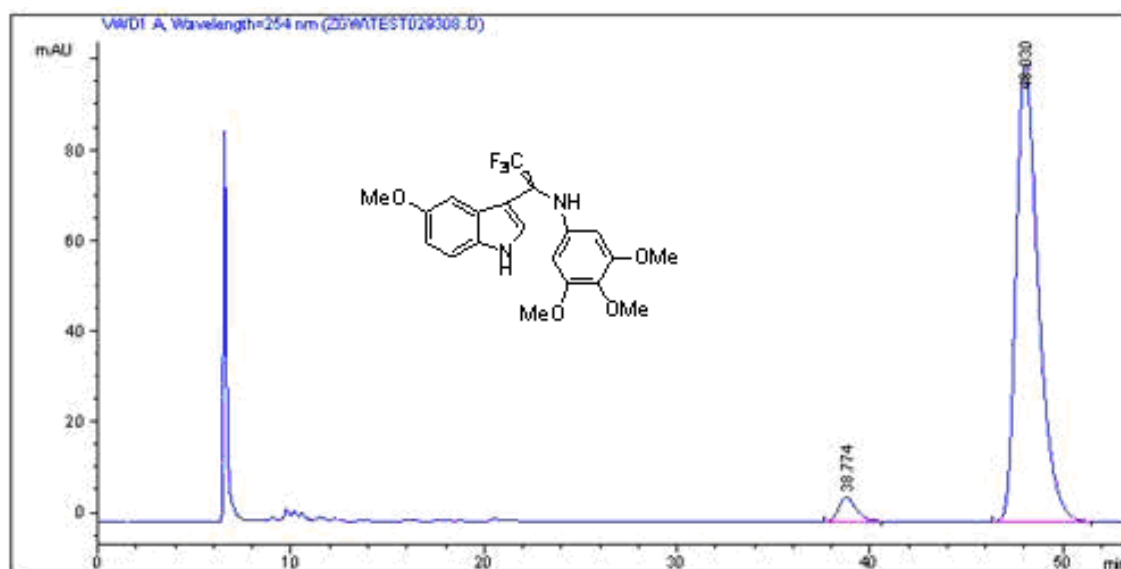
Sample Info : 254nm, i-PrOH:Hexane=20:80, AD-H, 0.5mL/min



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	38.619	BB	0.9417	4123.05664	64.67952	50.5511
2	48.177	BB	1.1656	4033.15405	50.76688	49.4489

Totals : 8156.21069 115.44640

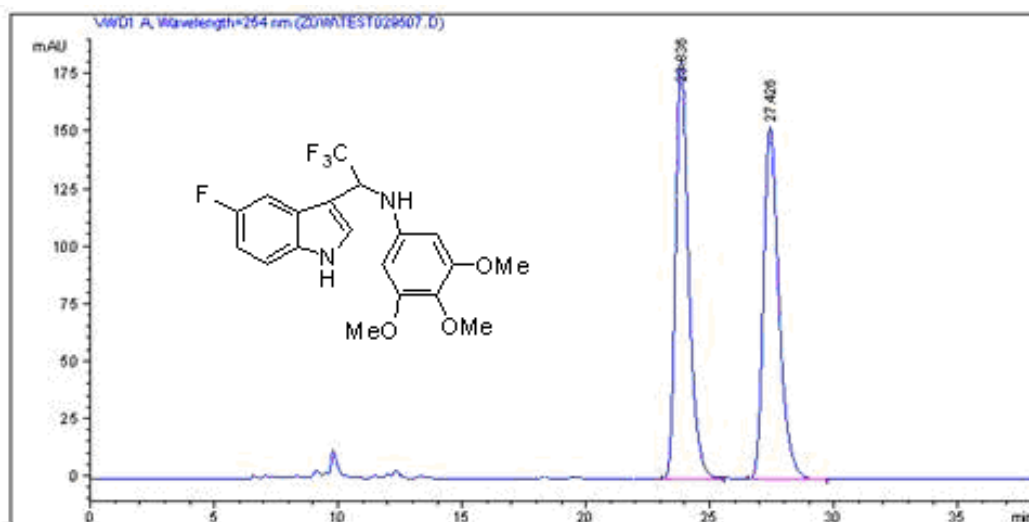


Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	38.774	BB	0.9103	333.63763	5.27098	3.9656
2	48.030	BB	1.1893	8079.70410	101.02324	96.0344

Totals : 8413.34174 106.29422

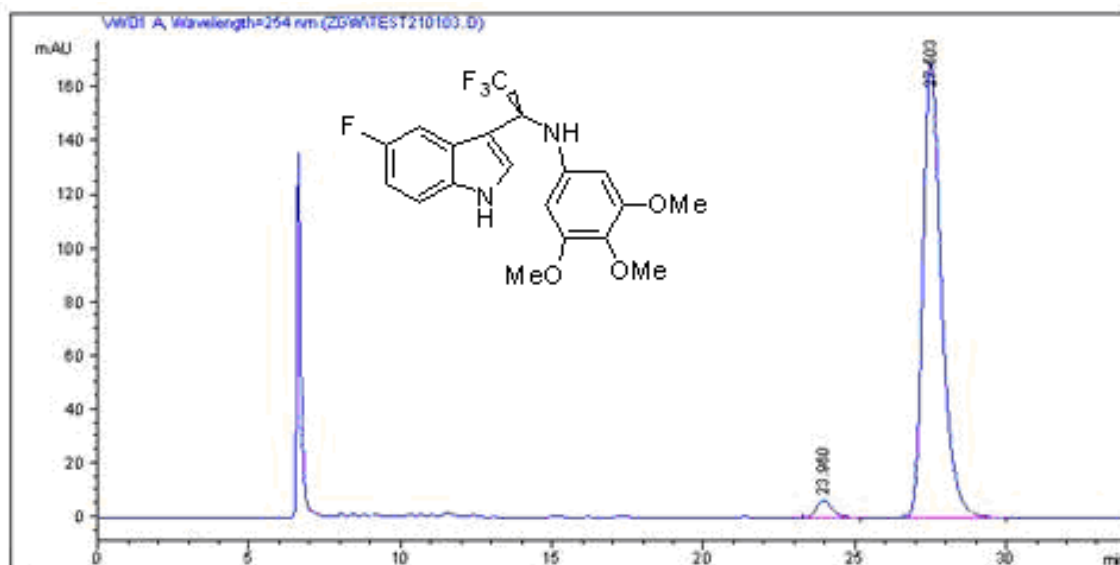
Sample Info : 254nm, i-PrOH:Hexane=20:80, AD-H, 0.5mL/min



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	23.836	BB	0.5653	6749.72852	182.54375	50.4978
2	27.426	BB	0.6582	6616.64209	153.19756	49.5022

Totals : 1.33664e4 335.74130

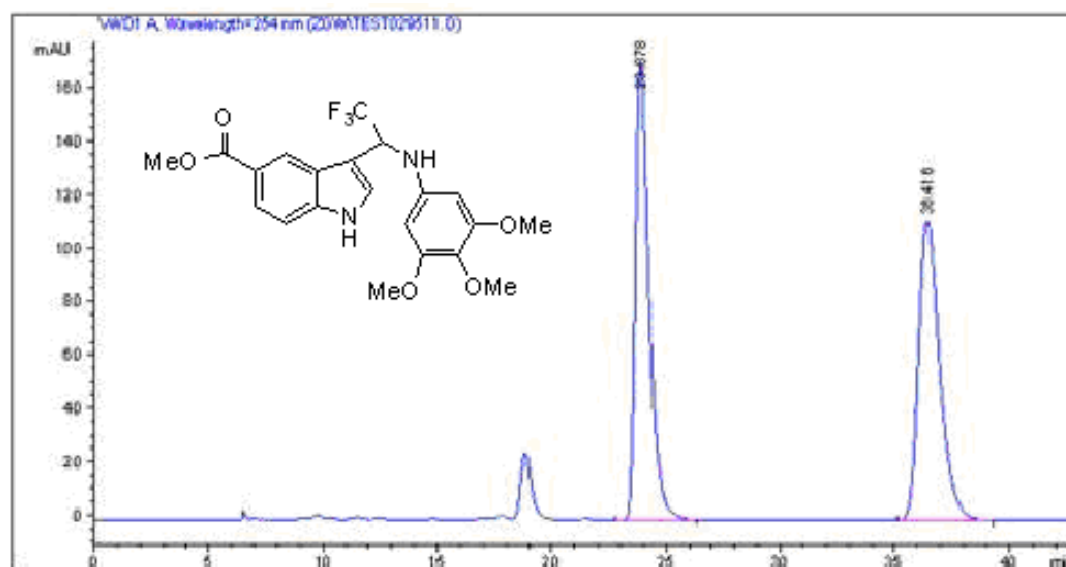


Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	23.960	BB	0.5357	239.24582	6.70483	3.1977
2	27.503	BB	0.6519	7242.51514	169.90284	96.8023

Totals : 7481.76096 176.50767

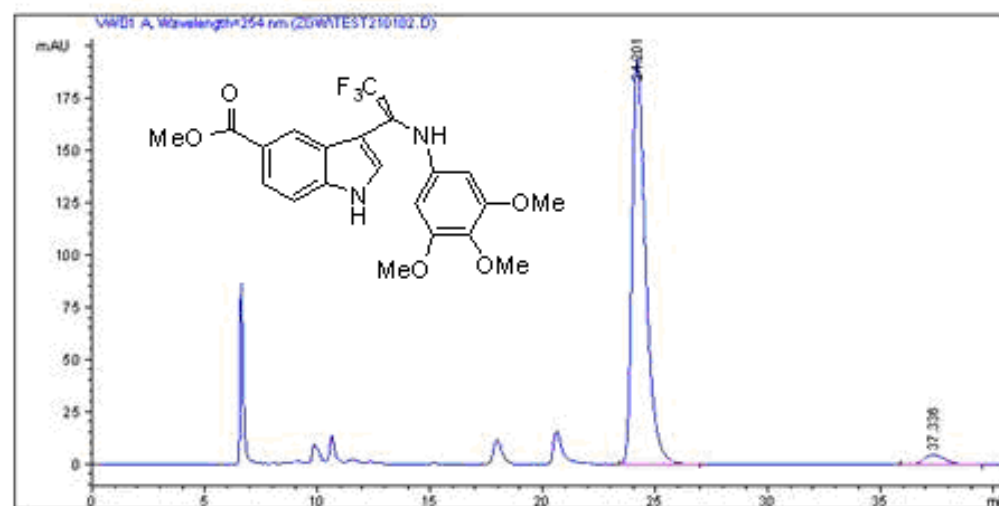
Sample Info : 254nm, i-PrOH:Hexane=20:80, AD-H, 0.5mL/min



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU * s	Height [mAU]	Area %
1	23.878	BB	0.6341	7244.60205	170.96992	50.0322
2	36.416	BB	0.9507	7235.27002	112.58326	49.9678

Totals : 1.44799e4 283.55318

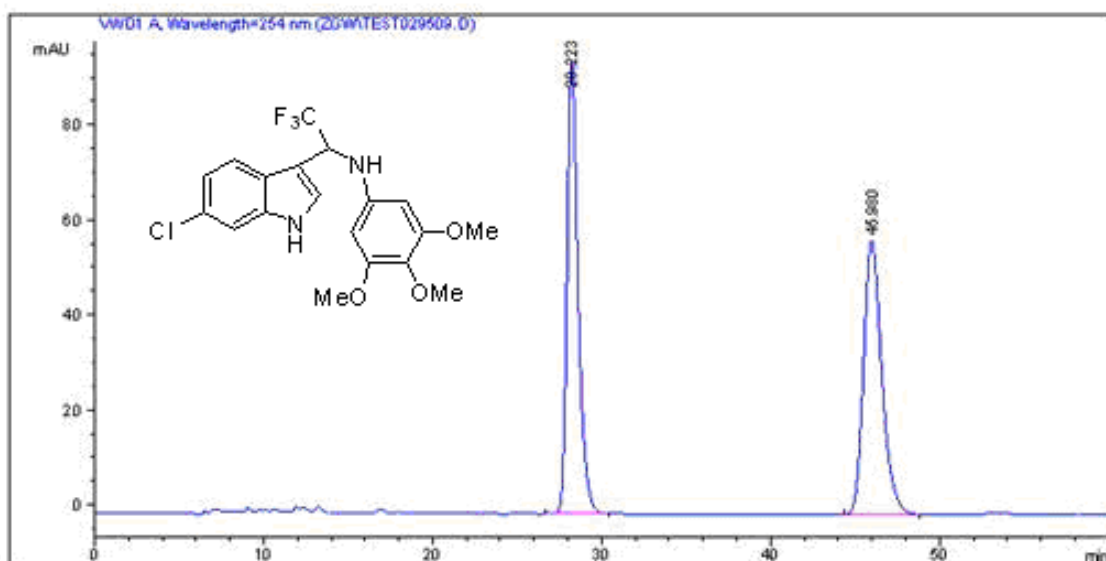


Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU * s	Height [mAU]	Area %
1	24.201	BB	0.6241	8107.94141	194.12651	96.0793
2	37.336	VB	0.9332	330.85590	5.03101	3.9207

Totals : 8438.79730 199.15752

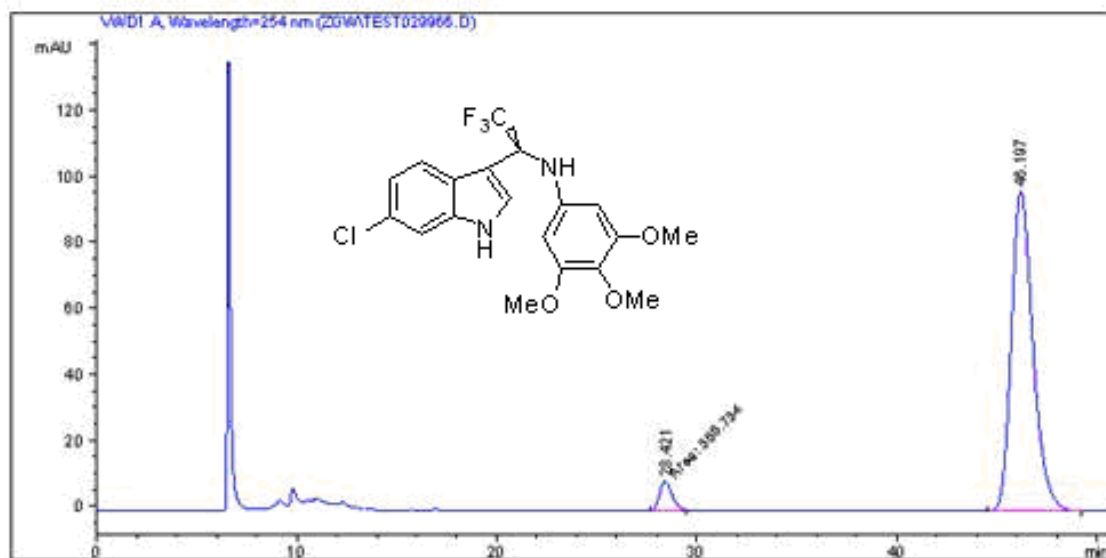
Sample Info : 254nm, i-PrOH:Hexane=20:80, AD-H, 0.5mL/min



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	%s	Height [mAU]	Area %
1	28.223	BB	0.6776	4353.16699		94.94936	50.2117
2	45.980	BB	1.1117	4316.45312		57.69256	49.7883

Totals : 8669.62012 152.64193

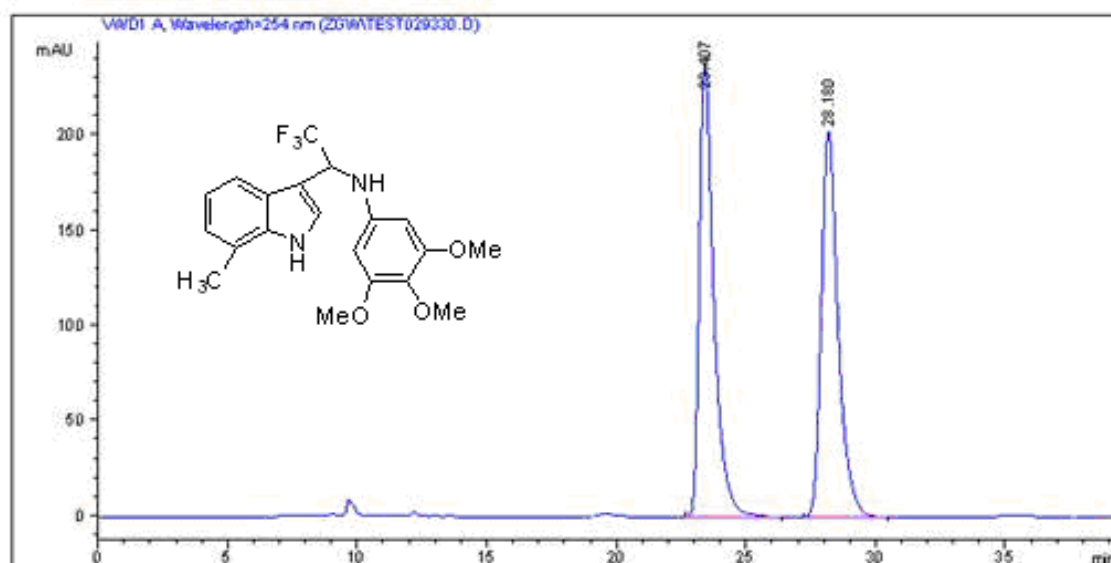


Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	%s	Height [mAU]	Area %
1	28.421	MM	0.7236	388.73447		8.95405	5.0619
2	46.197	BB	1.1109	7290.91504		97.21258	94.9381

Totals : 7679.64951 106.16662

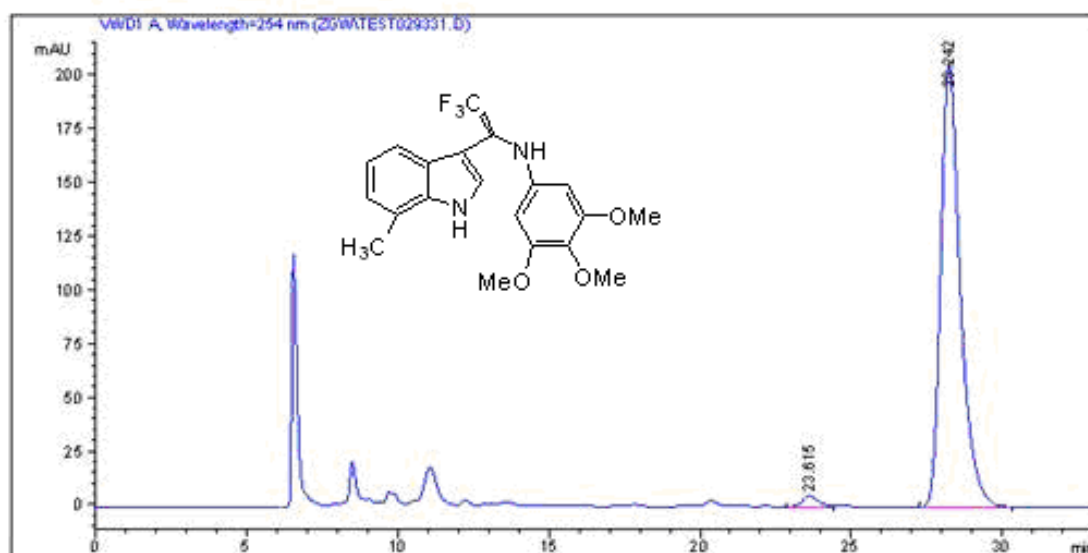
Sample Info : 254nm, i-PrOH:Hexane=20:80, AD-H, 0.5mL/min



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	23.407	BB	0.5749	9315.68652	238.60291	50.4447
2	28.180	BB	0.6796	9151.44531	203.27251	49.5553

Totals : 1.84671e4 441.87541

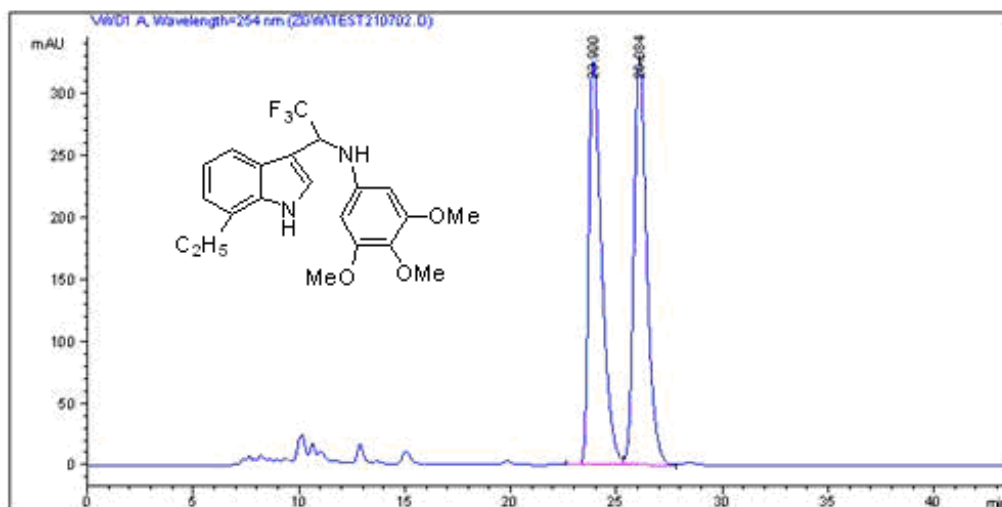


Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	23.615	BV	0.5530	189.95996	5.11081	2.0088
2	28.242	BB	0.6770	9266.19727	206.81906	97.9912

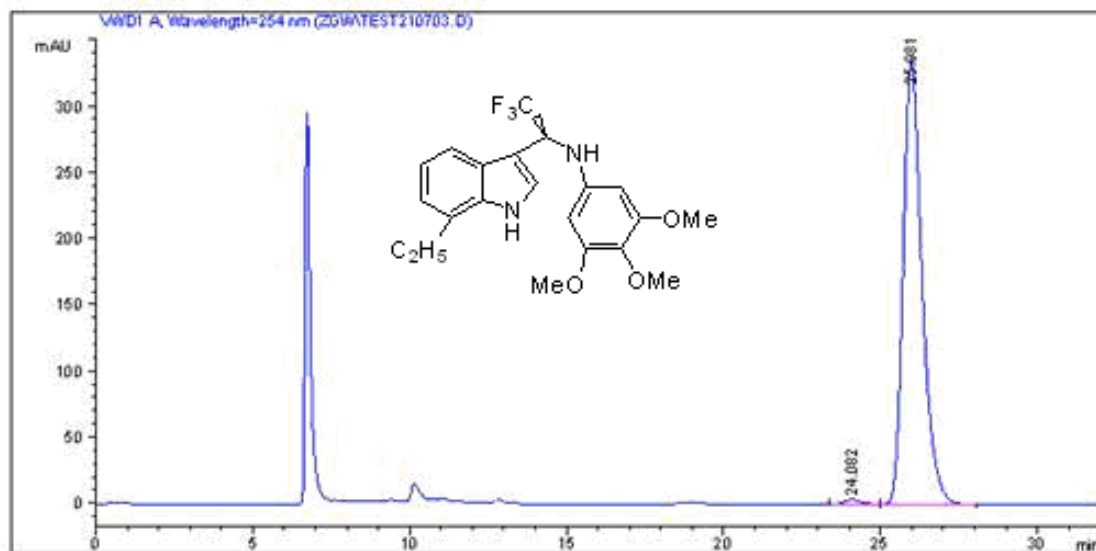
Totals : 9456.15723 211.92987

Sample Info : 254na,i-PrOH:Hexane=20:80,AD-H,0.5uL/min



Signal 1: VWD1 A, Wavelength=254 nm

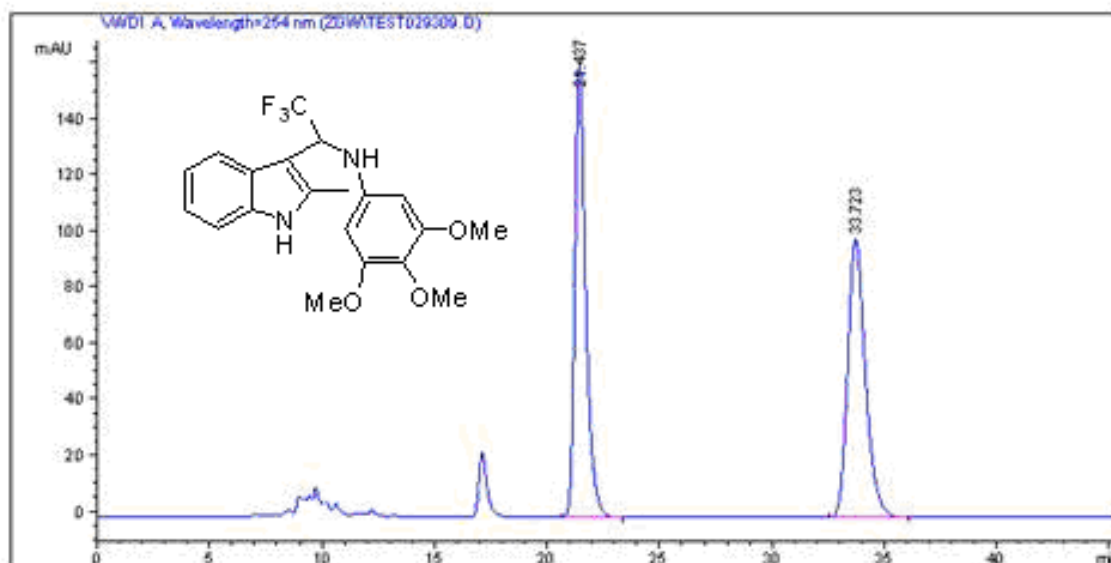
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	23.900	VV	0.6309	1.38785e4	325.83392	50.0352
2	26.084	VV	0.6260	1.38590e4	330.55457	49.9648
Totals :				2.77374e4	656.38849	



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	24.082	BV	0.5793	136.07275	3.49603	0.9656
2	25.981	VB	0.6247	1.39559e4	335.77698	99.0344
Totals :				1.40919e4	339.27301	

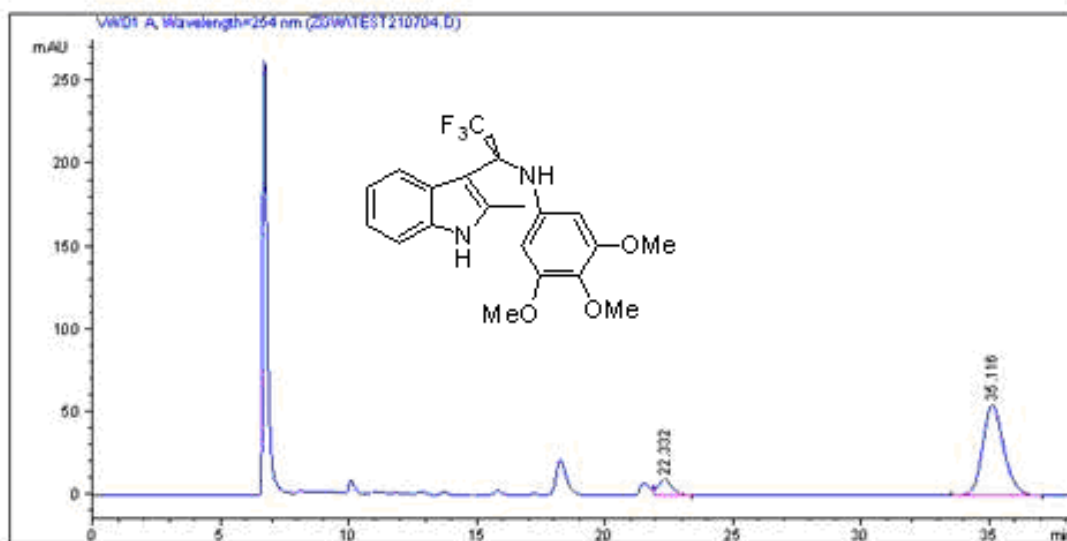
Sample Info : 254nm, i-PrOH:Hexane=20:80, AD-H, 0.5mL/min



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	%s	Height [mAU]	Area %
1	21.437	BB	0.4967	5447.14502		162.17802	50.0607
2	33.723	BB	0.8224	5433.92725		99.21907	49.9393

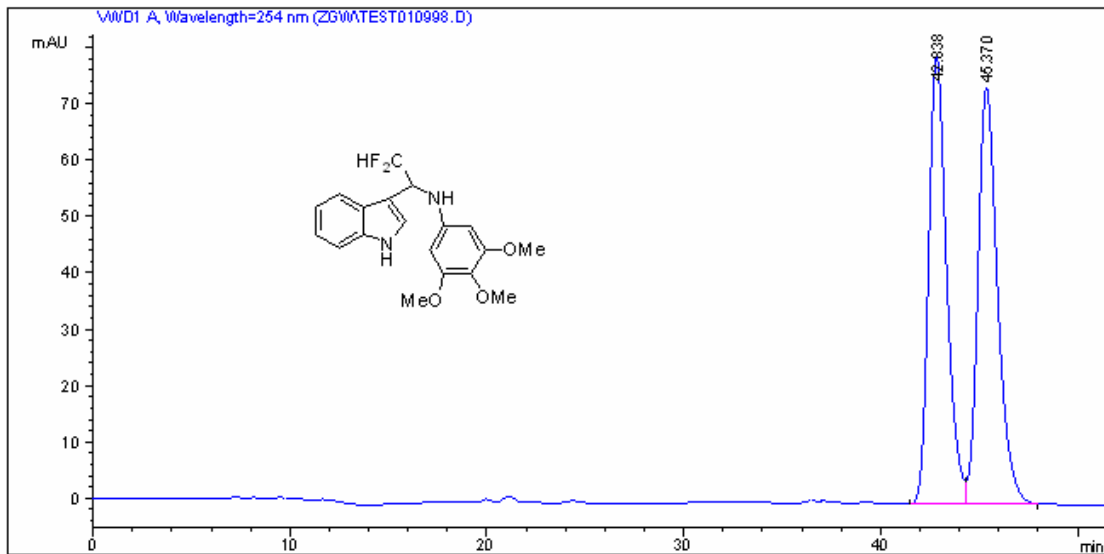
Totals : 1.08811e4 261.39709



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	%s	Height [mAU]	Area %
1	22.332	VB	0.5668	371.15381		9.67797	10.5267
2	35.116	BB	0.8689	3154.68140		54.85662	89.4733

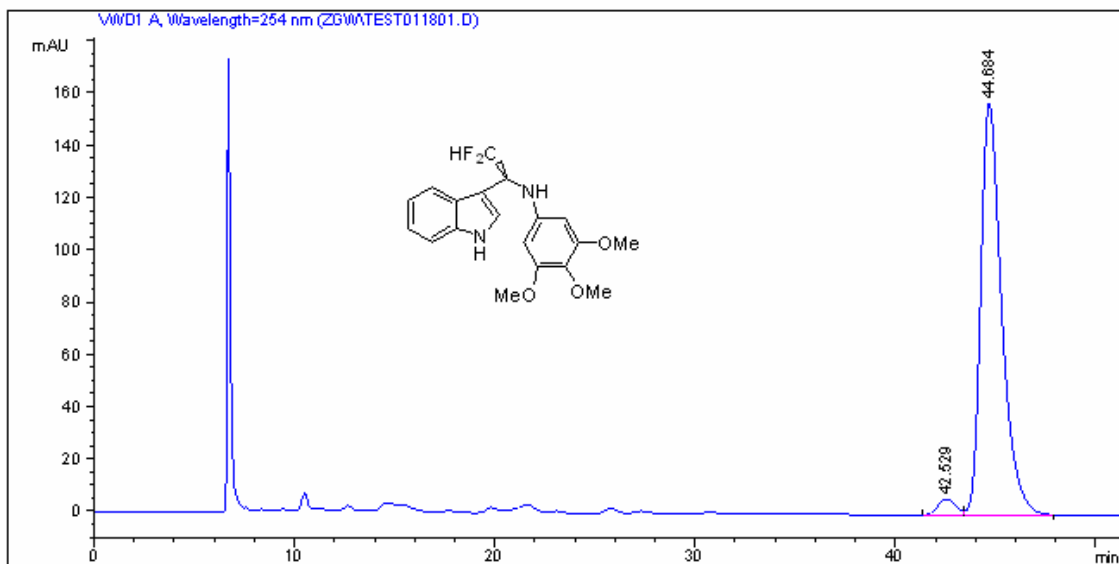
Totals : 3525.83521 64.53459



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	42.838	BV	0.9206	5037.06592	79.56564	49.5995
2	45.370	VB	1.0073	5118.40723	73.91653	50.4005

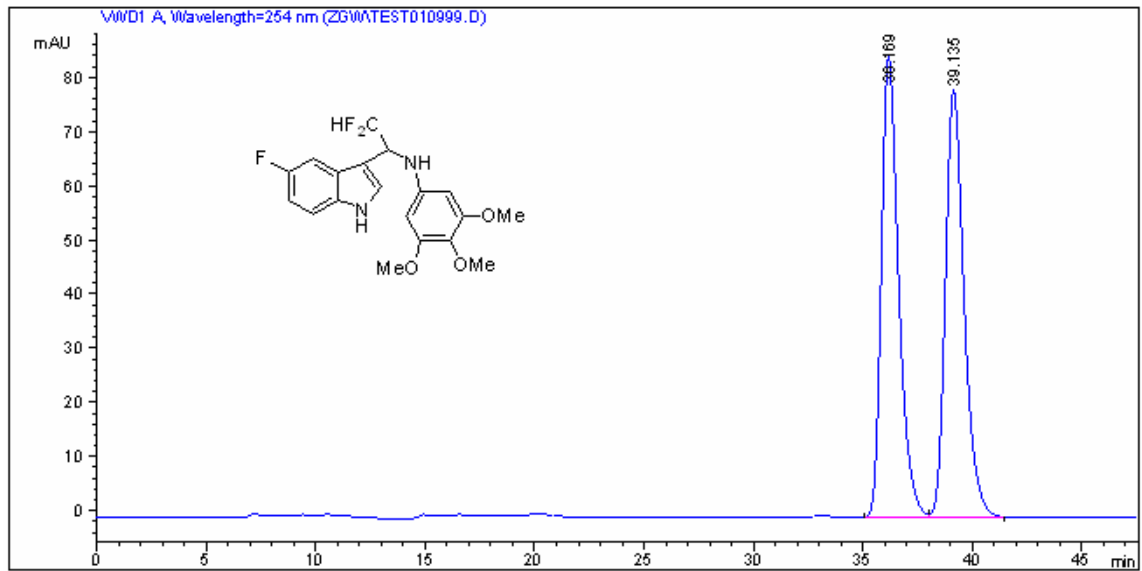
Totals : 1.01555e4 153.48217



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	42.529	BV	0.8602	382.61386	6.13416	3.2354
2	44.684	VB	1.0484	1.14431e4	157.52390	96.7646

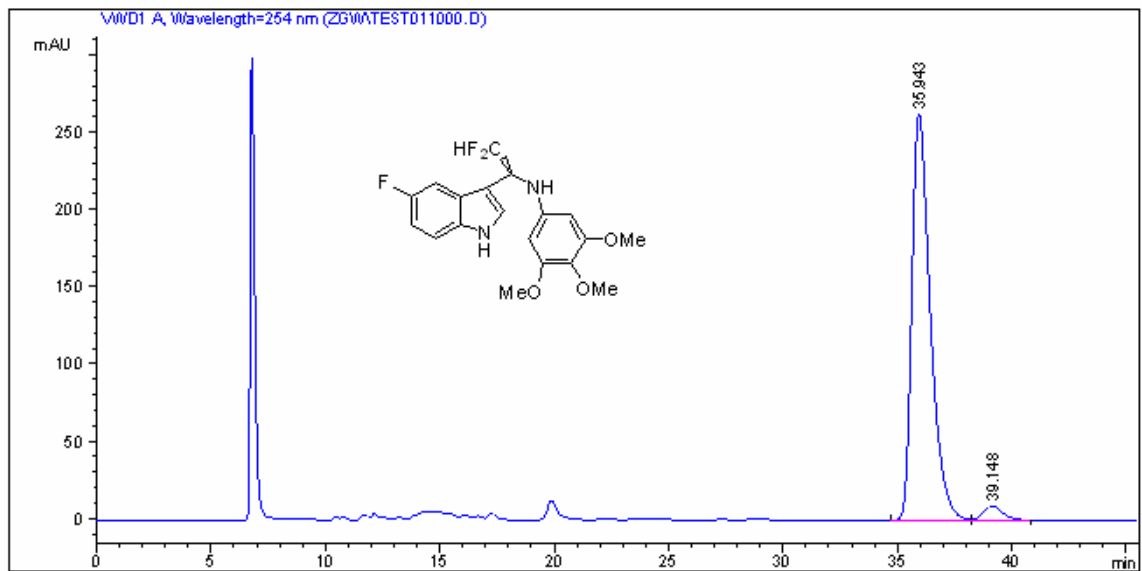
Totals : 1.18257e4 163.65805



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area %	Height [mAU]
1	36.169	BV	0.7997	4650.73975	49.9638	85.03967
2	39.135	VB	0.8559	4657.47900	50.0362	78.76451

Totals : 9308.21875 163.80418



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area %	Height [mAU]
1	35.943	BV	0.8150	1.47289e4	96.4242	263.00061
2	39.148	VB	0.8415	546.21088	3.5758	9.17686

Totals : 1.52751e4 272.17747

CCDC-672342 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

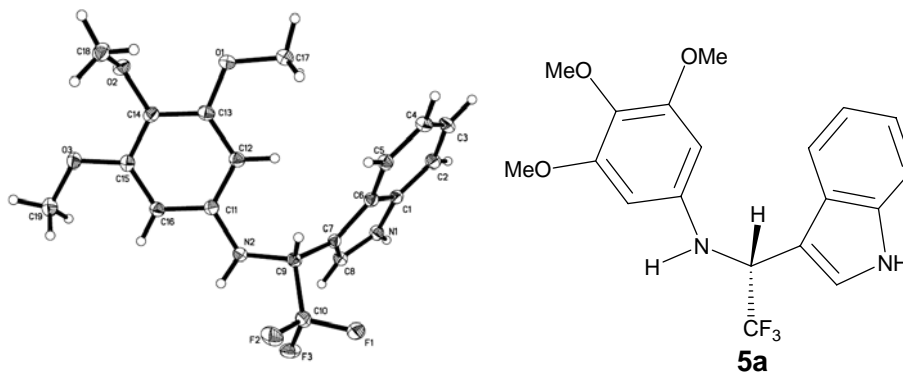


Table 1. Crystal data and structure refinement for shelxl.

Identification code	shelxl
Empirical formula	C ₁₉ H ₁₉ F ₃ N ₂ O ₃
Formula weight	380.36
Temperature	113(2) K
Wavelength	0.71070 Å
Crystal system, space group	Orthorhombic, P 21 21 21
Unit cell dimensions	a = 4.8996(14) Å alpha = 90 deg. b = 16.602(5) Å beta = 90 deg. c = 21.827(6) Å gamma = 90 deg.
Volume	1775.5(9) Å ³
Z, Calculated density	4, 1.423 Mg/m ³
Absorption coefficient	0.118 mm ⁻¹
F(000)	792
Crystal size	0.26 x 0.22 x 0.20 mm
Theta range for data collection	1.54 to 27.88 deg.
Limiting indices	-6 ≤ h ≤ 6, -21 ≤ k ≤ 21, -27 ≤ l ≤ 28
Reflections collected / unique	20217 / 4171 [R(int) = 0.0471]
Completeness to theta = 27.88	98.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9769 and 0.9701
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	4171 / 2 / 257
Goodness-of-fit on F ²	0.943
Final R indices [I > 2σ(I)]	R1 = 0.0427, wR2 = 0.1064
R indices (all data)	R1 = 0.0487, wR2 = 0.1116
Absolute structure parameter	-0.8(6)
Extinction coefficient	0.018(2)
Largest diff. peak and hole	0.203 and -0.211 e.Å ⁻³

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for shelxl. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
C(1)	6670(4)	2082(1)	1982(1)	21(1)
C(2)	5651(4)	1831(1)	1417(1)	26(1)
C(3)	3598(4)	1264(1)	1415(1)	29(1)
C(4)	2525(4)	955(1)	1965(1)	27(1)
C(5)	3497(4)	1209(1)	2527(1)	23(1)
C(6)	5634(4)	1780(1)	2543(1)	20(1)
C(7)	7196(4)	2156(1)	3020(1)	19(1)
C(8)	9045(4)	2651(1)	2741(1)	22(1)
C(9)	6871(4)	2007(1)	3698(1)	20(1)
C(10)	6057(4)	2768(1)	4036(1)	24(1)
C(11)	9753(4)	852(1)	4031(1)	20(1)
C(12)	8409(4)	330(1)	3634(1)	22(1)
C(13)	8894(4)	-498(1)	3687(1)	22(1)
C(14)	10686(4)	-802(1)	4124(1)	22(1)
C(15)	12080(4)	-267(1)	4505(1)	21(1)
C(16)	11628(4)	562(1)	4459(1)	21(1)
C(17)	5883(4)	-798(1)	2851(1)	28(1)
C(18)	9097(4)	-1987(1)	4575(1)	29(1)
C(19)	15415(4)	-95(1)	5295(1)	29(1)
F(1)	3963(2)	3153(1)	3768(1)	33(1)
F(2)	5241(3)	2603(1)	4614(1)	37(1)
F(3)	8088(2)	3300(1)	4084(1)	33(1)
N(1)	8719(3)	2615(1)	2118(1)	22(1)
N(2)	9299(4)	1687(1)	4000(1)	23(1)
O(1)	7679(3)	-1070(1)	3323(1)	26(1)
O(2)	11066(3)	-1625(1)	4171(1)	26(1)
O(3)	13800(3)	-617(1)	4921(1)	26(1)

Table 3. Bond lengths [Å] and angles [deg] for shelxl.

C(1)-N(1)	1.371(2)
C(1)-C(2)	1.395(2)
C(1)-C(6)	1.416(2)
C(2)-C(3)	1.378(3)

C(2)-H(2)	0.9500
C(3)-C(4)	1.407(3)
C(3)-H(3)	0.9500
C(4)-C(5)	1.381(2)
C(4)-H(4)	0.9500
C(5)-C(6)	1.413(3)
C(5)-H(5)	0.9500
C(6)-C(7)	1.436(2)
C(7)-C(8)	1.367(2)
C(7)-C(9)	1.508(2)
C(8)-N(1)	1.371(2)
C(8)-H(8)	0.9500
C(9)-N(2)	1.460(2)
C(9)-C(10)	1.516(2)
C(9)-H(9)	1.0000
C(10)-F(3)	1.335(2)
C(10)-F(1)	1.343(2)
C(10)-F(2)	1.351(2)
C(11)-C(12)	1.391(2)
C(11)-C(16)	1.397(2)
C(11)-N(2)	1.406(2)
C(12)-C(13)	1.400(2)
C(12)-H(12)	0.9500
C(13)-O(1)	1.374(2)
C(13)-C(14)	1.391(3)
C(14)-O(2)	1.382(2)
C(14)-C(15)	1.396(2)
C(15)-O(3)	1.368(2)
C(15)-C(16)	1.397(2)
C(16)-H(16)	0.9500
C(17)-O(1)	1.428(2)
C(17)-H(17A)	0.9800
C(17)-H(17B)	0.9800
C(17)-H(17C)	0.9800
C(18)-O(2)	1.439(2)
C(18)-H(18A)	0.9800
C(18)-H(18B)	0.9800
C(18)-H(18C)	0.9800
C(19)-O(3)	1.430(2)
C(19)-H(19A)	0.9800
C(19)-H(19B)	0.9800
C(19)-H(19C)	0.9800
N(1)-H(1A)	0.901(10)
N(2)-H(2A)	0.885(10)

N(1)-C(1)-C(2)	130.19(16)
N(1)-C(1)-C(6)	107.74(14)
C(2)-C(1)-C(6)	122.06(17)
C(3)-C(2)-C(1)	117.83(16)
C(3)-C(2)-H(2)	121.1
C(1)-C(2)-H(2)	121.1
C(2)-C(3)-C(4)	121.30(16)
C(2)-C(3)-H(3)	119.3
C(4)-C(3)-H(3)	119.3
C(5)-C(4)-C(3)	121.18(17)
C(5)-C(4)-H(4)	119.4
C(3)-C(4)-H(4)	119.4
C(4)-C(5)-C(6)	118.78(16)
C(4)-C(5)-H(5)	120.6
C(6)-C(5)-H(5)	120.6
C(5)-C(6)-C(1)	118.83(15)
C(5)-C(6)-C(7)	134.77(15)
C(1)-C(6)-C(7)	106.39(15)
C(8)-C(7)-C(6)	106.92(14)
C(8)-C(7)-C(9)	127.29(16)
C(6)-C(7)-C(9)	125.77(16)
C(7)-C(8)-N(1)	109.81(16)
C(7)-C(8)-H(8)	125.1
N(1)-C(8)-H(8)	125.1
N(2)-C(9)-C(7)	114.61(15)
N(2)-C(9)-C(10)	107.32(14)
C(7)-C(9)-C(10)	111.64(14)
N(2)-C(9)-H(9)	107.7
C(7)-C(9)-H(9)	107.7
C(10)-C(9)-H(9)	107.7
F(3)-C(10)-F(1)	106.79(14)
F(3)-C(10)-F(2)	106.35(14)
F(1)-C(10)-F(2)	106.06(15)
F(3)-C(10)-C(9)	113.20(15)
F(1)-C(10)-C(9)	112.68(14)
F(2)-C(10)-C(9)	111.28(15)
C(12)-C(11)-C(16)	120.89(16)
C(12)-C(11)-N(2)	120.70(16)
C(16)-C(11)-N(2)	118.39(15)
C(11)-C(12)-C(13)	118.71(16)
C(11)-C(12)-H(12)	120.6
C(13)-C(12)-H(12)	120.6
O(1)-C(13)-C(14)	114.86(15)
O(1)-C(13)-C(12)	123.81(16)

C(14)-C(13)-C(12)	121.32(16)
O(2)-C(14)-C(13)	119.56(16)
O(2)-C(14)-C(15)	121.32(16)
C(13)-C(14)-C(15)	119.12(15)
O(3)-C(15)-C(14)	115.16(15)
O(3)-C(15)-C(16)	124.34(16)
C(14)-C(15)-C(16)	120.47(16)
C(15)-C(16)-C(11)	119.42(16)
C(15)-C(16)-H(16)	120.3
C(11)-C(16)-H(16)	120.3
O(1)-C(17)-H(17A)	109.5
O(1)-C(17)-H(17B)	109.5
H(17A)-C(17)-H(17B)	109.5
O(1)-C(17)-H(17C)	109.5
H(17A)-C(17)-H(17C)	109.5
H(17B)-C(17)-H(17C)	109.5
O(2)-C(18)-H(18A)	109.5
O(2)-C(18)-H(18B)	109.5
H(18A)-C(18)-H(18B)	109.5
O(2)-C(18)-H(18C)	109.5
H(18A)-C(18)-H(18C)	109.5
H(18B)-C(18)-H(18C)	109.5
O(3)-C(19)-H(19A)	109.5
O(3)-C(19)-H(19B)	109.5
H(19A)-C(19)-H(19B)	109.5
O(3)-C(19)-H(19C)	109.5
H(19A)-C(19)-H(19C)	109.5
H(19B)-C(19)-H(19C)	109.5
C(8)-N(1)-C(1)	109.13(14)
C(8)-N(1)-H(1A)	124.0(15)
C(1)-N(1)-H(1A)	126.7(15)
C(11)-N(2)-C(9)	120.61(15)
C(11)-N(2)-H(2A)	114.5(15)
C(9)-N(2)-H(2A)	115.8(16)
C(13)-O(1)-C(17)	117.79(14)
C(14)-O(2)-C(18)	111.53(14)
C(15)-O(3)-C(19)	117.48(13)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for shelxl. The anisotropic displacement factor exponent takes the form: $-2 \pi^2 [h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12}]$

	U11	U22	U33	U23	U13	U12
C(1)	25(1)	18(1)	20(1)	0(1)	-1(1)	2(1)
C(2)	32(1)	27(1)	20(1)	-1(1)	-1(1)	1(1)
C(3)	35(1)	30(1)	21(1)	-5(1)	-6(1)	-2(1)
C(4)	27(1)	24(1)	30(1)	-2(1)	-3(1)	-3(1)
C(5)	23(1)	24(1)	21(1)	0(1)	0(1)	1(1)
C(6)	22(1)	19(1)	18(1)	0(1)	2(1)	2(1)
C(7)	21(1)	19(1)	17(1)	1(1)	0(1)	3(1)
C(8)	24(1)	22(1)	19(1)	-2(1)	-1(1)	0(1)
C(9)	23(1)	19(1)	17(1)	0(1)	0(1)	-1(1)
C(10)	23(1)	25(1)	22(1)	-2(1)	-1(1)	0(1)
C(11)	21(1)	21(1)	18(1)	1(1)	3(1)	-1(1)
C(12)	25(1)	23(1)	19(1)	-1(1)	2(1)	-1(1)
C(13)	23(1)	23(1)	20(1)	-3(1)	4(1)	-2(1)
C(14)	25(1)	20(1)	20(1)	0(1)	5(1)	3(1)
C(15)	19(1)	25(1)	18(1)	1(1)	4(1)	3(1)
C(16)	23(1)	22(1)	18(1)	0(1)	1(1)	-2(1)
C(17)	29(1)	27(1)	28(1)	-3(1)	-4(1)	-1(1)
C(18)	35(1)	25(1)	28(1)	3(1)	2(1)	0(1)
C(19)	27(1)	32(1)	28(1)	4(1)	-3(1)	-1(1)
F(1)	30(1)	33(1)	35(1)	-9(1)	-5(1)	9(1)
F(2)	45(1)	45(1)	20(1)	-6(1)	8(1)	2(1)
F(3)	29(1)	25(1)	44(1)	-13(1)	-1(1)	-3(1)
N(1)	25(1)	22(1)	20(1)	3(1)	0(1)	-2(1)
N(2)	30(1)	20(1)	20(1)	-1(1)	-5(1)	0(1)
O(1)	31(1)	22(1)	25(1)	-5(1)	-4(1)	1(1)
O(2)	31(1)	21(1)	26(1)	0(1)	5(1)	3(1)
O(3)	27(1)	26(1)	24(1)	1(1)	-4(1)	5(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for shelxl.

	x	y	z	U(eq)
H(2)	6349	2045	1045	32
H(3)	2894	1077	1035	34
H(4)	1107	565	1950	32

H(5)	2743	1003	2896	27
H(8)	10360	2972	2948	26
H(9)	5368	1606	3752	24
H(12)	7185	531	3333	27
H(16)	12588	925	4718	25
H(17A)	4332	-515	3035	42
H(17B)	5219	-1261	2616	42
H(17C)	6867	-430	2577	42
H(18A)	9363	-1780	4991	44
H(18B)	9336	-2572	4574	44
H(18C)	7251	-1854	4435	44
H(19A)	16485	265	5033	43
H(19B)	16650	-418	5549	43
H(19C)	14216	225	5559	43
H(1A)	9660(50)	2922(12)	1850(8)	41(7)
H(2A)	9880(50)	1959(12)	4324(7)	41(7)

Table 6. Torsion angles [deg] for shelxl.

N(1)-C(1)-C(2)-C(3)	178.31(18)
C(6)-C(1)-C(2)-C(3)	-0.9(3)
C(1)-C(2)-C(3)-C(4)	1.1(3)
C(2)-C(3)-C(4)-C(5)	-0.2(3)
C(3)-C(4)-C(5)-C(6)	-0.9(3)
C(4)-C(5)-C(6)-C(1)	1.0(2)
C(4)-C(5)-C(6)-C(7)	-177.5(2)
N(1)-C(1)-C(6)-C(5)	-179.51(15)
C(2)-C(1)-C(6)-C(5)	-0.1(3)
N(1)-C(1)-C(6)-C(7)	-0.60(19)
C(2)-C(1)-C(6)-C(7)	178.80(17)
C(5)-C(6)-C(7)-C(8)	178.64(19)
C(1)-C(6)-C(7)-C(8)	-0.02(19)
C(5)-C(6)-C(7)-C(9)	0.1(3)
C(1)-C(6)-C(7)-C(9)	-178.58(16)
C(6)-C(7)-C(8)-N(1)	0.6(2)
C(9)-C(7)-C(8)-N(1)	179.17(16)
C(8)-C(7)-C(9)-N(2)	-59.0(2)
C(6)-C(7)-C(9)-N(2)	119.22(19)
C(8)-C(7)-C(9)-C(10)	63.2(2)
C(6)-C(7)-C(9)-C(10)	-118.51(19)
N(2)-C(9)-C(10)-F(3)	53.74(18)
C(7)-C(9)-C(10)-F(3)	-72.63(19)

N(2)-C(9)-C(10)-F(1)	175.05(14)
C(7)-C(9)-C(10)-F(1)	48.7(2)
N(2)-C(9)-C(10)-F(2)	-65.97(19)
C(7)-C(9)-C(10)-F(2)	167.66(15)
C(16)-C(11)-C(12)-C(13)	2.3(3)
N(2)-C(11)-C(12)-C(13)	-179.09(16)
C(11)-C(12)-C(13)-O(1)	179.79(16)
C(11)-C(12)-C(13)-C(14)	-0.2(3)
O(1)-C(13)-C(14)-O(2)	-1.3(2)
C(12)-C(13)-C(14)-O(2)	178.63(16)
O(1)-C(13)-C(14)-C(15)	178.22(15)
C(12)-C(13)-C(14)-C(15)	-1.8(3)
O(2)-C(14)-C(15)-O(3)	-0.4(2)
C(13)-C(14)-C(15)-O(3)	-179.93(15)
O(2)-C(14)-C(15)-C(16)	-178.74(16)
C(13)-C(14)-C(15)-C(16)	1.7(3)
O(3)-C(15)-C(16)-C(11)	-177.84(15)
C(14)-C(15)-C(16)-C(11)	0.4(3)
C(12)-C(11)-C(16)-C(15)	-2.4(3)
N(2)-C(11)-C(16)-C(15)	178.96(16)
C(7)-C(8)-N(1)-C(1)	-1.0(2)
C(2)-C(1)-N(1)-C(8)	-178.32(18)
C(6)-C(1)-N(1)-C(8)	1.0(2)
C(12)-C(11)-N(2)-C(9)	19.0(2)
C(16)-C(11)-N(2)-C(9)	-162.35(15)
C(7)-C(9)-N(2)-C(11)	-88.48(19)
C(10)-C(9)-N(2)-C(11)	146.92(15)
C(14)-C(13)-O(1)-C(17)	-177.99(16)
C(12)-C(13)-O(1)-C(17)	2.1(2)
C(13)-C(14)-O(2)-C(18)	-87.72(19)
C(15)-C(14)-O(2)-C(18)	92.7(2)
C(14)-C(15)-O(3)-C(19)	175.96(15)
C(16)-C(15)-O(3)-C(19)	-5.8(3)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for shelxl [Å and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
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