SUPPORTING INFROMATION

Access to 1-Amino-3,4-dihydroisoquinolines via Palladium-Catalyzed C-H Bond Aminoimidoylation Reaction from Functionalized Isocyanides

Zhuang Xiong,*[a] Panyuan Cai,[b] Yingshuang Mei,[a] and Jian Wang*[b]

[a]School of Biotechnology and Health Sciences, Wuyi University, Jiangmen, Guangdong 529020, P. R China

[b]Key Laboratory of Functional Molecular Solids, Ministry of Education, Anhui Laboratory of Molecule-Based Materials, College of Chemistry and Materials Science, Anhui Normal University, Wuhu, Anhui 241000, P. R. China

Contents

I. General Information 2

II. General Procedure 2

III. Characterization Data 3

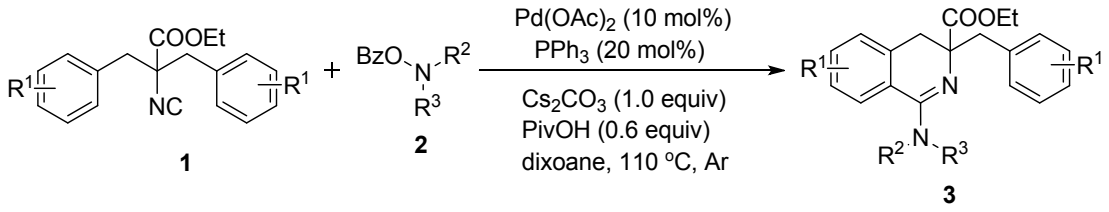
IV. References 19

V. Copies of ^{1}H and ^{13}C NMR Spectra 20

I. General Information

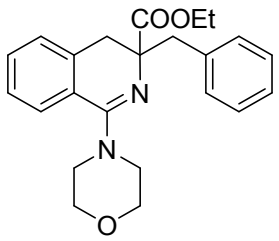
^{1}H NMR (400 MHz) and ^{13}C NMR (125 MHz) were registered on 400 MHz and 500 MHz spectrometers. Chemical shifts were reported in units (ppm) by assigning TMS resonance in the ^{1}H spectrum as 0.00 ppm, $CDCl_3$ resonance in the ^{13}C spectrum as 77.0 ppm. All coupling constants (*J* values) were reported in Hertz (Hz). NMR analysis was carried out at 298 K unless noted otherwise. HRMS was obtained on an ESI-LC-MS/MS spectrometer.

II. General Procedure



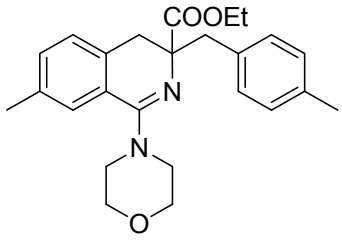
General procedure : An oven-dried 25 mL schlenk tube charged with $Pd(OAc)_2$ (0.01 mmol, 2.24 mg), PPh_3 (0.02 mmol, 5.24 mg), Cs_2CO_3 (0.1 mmol, 32.6 mg) and **2** (0.15 mmol) was refilled with Ar for 3 times. Then a solution of pivalic acid (0.06 mmol, 7 uL) in 0.5 mL of dioxane was added by syringe and the tube was placed in an 110 °C oil-bath. A solution of **1** (0.1 mmol) in 1.0 mL of dioxane was added dropwise within 1 h by a syringe pump to the reaction mixture. After reacting for another 1-2 h, the reaction was completed. The crude reaction mixture was extracted with EA (20 mL × 3) and washed with brine (20 mL). The organic phase was concentrated in *vacuo* and the residue was purified by silica gel flash column chromatography to afford the corresponding amino substituted isoquinolines **3**.

III. Characterization Data



ethyl 3-benzyl-1-morpholino-3,4-dihydroisoquinoline-3-carboxylate (3a)

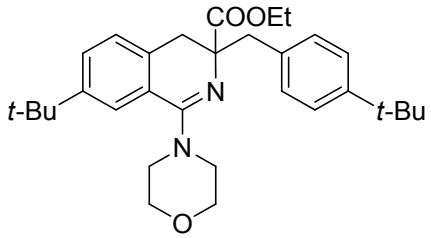
Prepared from ethyl 2-benzyl-2-isocyano-3-phenylpropanoate (29.3 mg, 0.1 mmol, 1.0 equiv) and morpholino benzoate (31.05 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 1 : 4) furnished the product **3a** as a light yellow oil (30 mg, 0.080 mmol, 80% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.40 (d, J = 7.4 Hz, 1H), 7.33-7.29 (m, 1H), 7.26-7.19 (m, 7H), 3.94-3.88 (m, 2H), 3.88-3.78 (m, 4H), 3.38-3.24 (m, 4H), 3.13-3.02 (m, 3H), 2.73 (d, J = 15.2 Hz, 1H), 0.96 (t, J = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ174.1, 161.8, 138.4, 137.4, 130.8, 130.4, 128.8, 127.8, 126.8, 126.6, 126.2, 125.1, 66.9, 65.9, 60.7, 49.3, 44.5, 35.2, 14.1; HRMS: calcd for $C_{23}H_{26}N_2O_3$ ($M+H^+$) 379.2016; found 379.2014.



ethyl 7-methyl-3-(4-methylbenzyl)-1-morpholino-3,4-dihydroisoquinoline-3-carboxylate (3b)

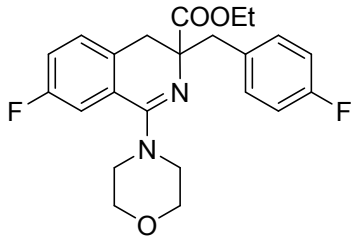
Prepared from ethyl 2-isocyano-2-(4-methylbenzyl)-3-(p-tolyl)propanoate (32.1 mg, 0.1 mmol, 1.0 equiv) and morpholino benzoate (31.05 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 1 : 4) furnished the product **3b** as a colorless oil (29 mg, 0.072 mmol, 72% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.19 (s, 1H), 7.14-7.07 (m, 4H), 7.05 (d, J = 7.8 Hz, 2H), 3.96-3.90 (m, 2H), 3.89-3.78 (m, 4H), 3.38-3.32 (m, 2H), 3.29-3.23 (m, 2H), 3.07-2.95 (m, 3H), 2.68 (d, J = 15.2 Hz, 1H), 2.34 (s, 3H), 2.30 (s, 3H), 0.99 (t, J = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.3, 161.9, 136.2, 135.9, 135.3,

134.3, 131.0, 130.6, 128.6, 128.5, 126.7, 124.9, 66.9, 66.1, 60.7, 49.3, 43.9, 34.7, 21.5, 21.2, 14.1; HRMS: calcd for $C_{25}H_{30}N_2O_3$ ($M+H^+$) 407.2329; found 407.2316.



ethyl 7-(tert-butyl)-3-(4-(tert-butyl)benzyl)-1-morpholino-3,4-dihydroisoquinoline-3-carboxylate (3c)

Prepared from dimethyl ethyl 2-(4-(tert-butyl)benzyl)-3-(4-(tert-butyl)phenyl)-2-isocyanopropanoate (40.6 mg, 0.1 mmol, 1.0 equiv) and morpholino benzoate (31.05 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 1 : 8) furnished the product **3c** as colorless oil (35 mg, 0.071 mmol, 71% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.44 (s, 1H), 7.35 (d, *J* = 7.8 Hz, 1H), 7.24 (d, *J* = 6.9 Hz, 2H), 3.97-3.88 (m, 2H), 3.87-3.79 (m, 4H), 3.39-3.33 (m, 2H), 3.31-3.25 (m, 2H), 3.08-3.00 (m, 2H), 2.97 (d, *J* = 13.2 Hz, 1H), 2.72 (d, *J* = 15.3 Hz, 1H), 1.31 (s, 9H), 1.29 (s, 9H), 0.93 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.3, 161.9, 149.5, 149.2, 135.3, 134.2, 130.3, 128.3, 127.2, 124.6, 124.4, 123.1, 66.8, 66.1, 60.5, 49.4, 43.7, 34.6, 34.4, 34.3, 31.4, 31.3, 13.9; HRMS: calcd for $C_{31}H_{42}N_2O_3$ ($M+H^+$) 491.3268; found 491.3256.

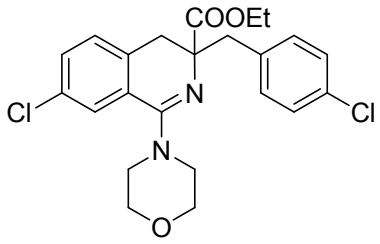


ethyl 7-fluoro-3-(4-fluorobenzyl)-1-morpholino-3,4-dihydroisoquinoline-3-carboxylate

(3d)

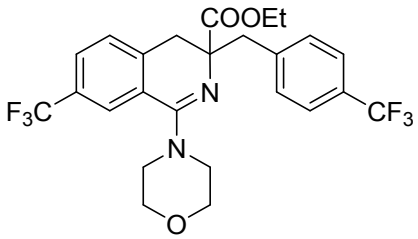
Prepared from ethyl 2-(4-fluorobenzyl)-3-(4-fluorophenyl)-2-isocyanopropanoate (32.9 mg, 0.1 mmol, 1.0 equiv) and morpholino benzoate (31.05 mg, 0.15 mmol, 1.5

equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 1 : 8) furnished the product **3d** as a colorless oil (26 mg, 0.063 mmol, 63% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.25-7.22 (m, 2H), 7.18-7.15 (m, 1H), 7.10-7.08 (m, 1H), 7.05-7.00 (m, 1H), 6.95-6.91 (m, 2H), 3.91-3.85 (m, 4H), 3.81-3.77 (m, 2H), 3.36-3.32 (m, 2H), 3.24-3.20 (m, 2H), 3.07-3.04 (m, 3H), 2.66 (d, *J* = 15.1 Hz, 1H), 0.95 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ173.6, 161.9 (d, *J* = 242.5 Hz), 161.5 (d, *J* = 243.8 Hz), 161.1 (d, *J* = 2.5 Hz), 133.5 (d, *J* = 2.5 Hz), 132.8 (d, *J* =3.8Hz), 132.2 (d, *J* = 7.5Hz), 129.9 (d, *J* = 7.5 Hz), 126.5 (d, *J* = 7.5 Hz), 117.2 (d, *J* =21.3 Hz), 114.5 (d, *J* = 21.3 Hz), 113.1 (d, *J* = 22.5 Hz), 66.7, 66.1, 60.8, 49.2, 43.9, 34.9, 13.9; HRMS: calcd for $C_{23}H_{24}F_2N_2O_3$(M+H$^+$) 415.1828; found 415.1823.



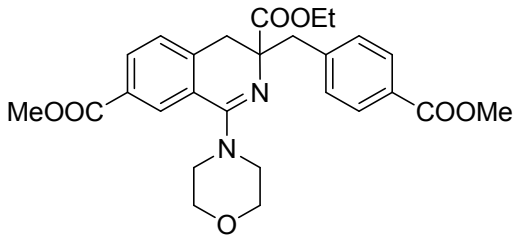
ethyl 7-chloro-3-(4-chlorobenzyl)-1-morpholino-3,4-dihydroisoquinoline-3-carboxylate (3e)

Prepared from ethyl 2-(4-chlorobenzyl)-3-(4-chlorophenyl)-2-isocyanopropanoate (36.2 mg, 0.1 mmol, 1.0 equiv) and morpholino benzoate (31.05 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 1 : 8) furnished the product **3h** as colorless oil (32 mg, 0.070 mmol, 70% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35(d, *J* = 2.1Hz, 1H), 7.29(dd, *J* = 8.1, 2.1Hz, 1H), 7.21-7.16 (m, 4H), 7.14(d, *J* = 8.1Hz, 1H), 3.93-3.85(m, 4H), 3.82-3.77(m, 2H), 3.37-3.30(m, 2H), 3.24-3.18(m, 2H), 3.11-3.03 (m, 3H), 2.65(d, *J* = 15.1 Hz, 1H), 0.95 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 173.5, 160.9, 136.4, 135.7, 132.7, 132.6, 132.2, 130.4, 129.9, 127.9, 126.5, 126.3, 66.8, 65.9, 60.9, 49.3, 44.2, 35.2, 14.1; HRMS: calcd for $C_{23}H_{24}Cl_2N_2O_3$ (M+H$^+$) 447.1237; found 447.1240.



ethyl 1-morpholino-7-(trifluoromethyl)-3-(4-(trifluoromethyl)benzyl)-3,4-dihydroisoquinoline-3-carboxylate (3f)

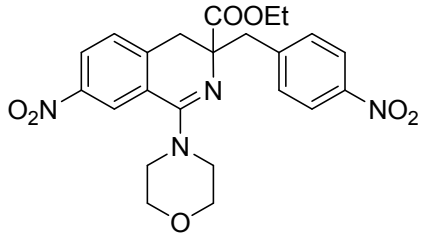
Prepared from ethyl 2-isocyano-2-(4-(trifluoromethyl)benzyl)-3-(4-(trifluoromethyl)phenyl)propanoate (42.9 mg, 0.1 mmol, 1.0 equiv) and morpholino benzoate (31.05 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 1 : 8) furnished the product **3f** as a light yellow oil (48 mg, 0.093 mmol, 93% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.62 (s, 1H), 7.58 (d, *J* = 7.9 Hz, 1H), 7.51 (d, *J* = 8.1 Hz, 2H), 7.41 (d, *J* = 8.1 Hz, 2H), 7.33 (d, *J* = 7.9 Hz, 1H), 3.91-3.86 (m, 3H), 3.86-3.78 (m, 3H), 3.38-3.33 (m, 2H), 3.25-3.15 (m, 5H), 2.76 (d, *J* = 15.3 Hz, 1H), 0.89 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ^{13}C NMR (125 MHz, $CDCl_3$): δ 172.1, 160.9, 141.8, 141.0, 131.1, 134.2, 132.9, 131.1, 129.5 (q, *J* = 32.5 Hz), 128.7 (q, *J* = 31.3 Hz), 126.9 (q, *J* = 2.5 Hz), 124.7 (q, *J* = 3.8 Hz), 124.4 (q, *J* = 270.8 Hz), 123.8 (q, *J* = 276.3 Hz), 66.6, 65.6, 61.0, 49.2, 44.7, 35.8, 13.8; HRMS: calcd for $C_{25}H_{24}F_6N_2O_3$ ($M+H^+$) 515.1764; found 515.1756.



3-ethyl 7-methyl 3-(4-(methoxycarbonyl)benzyl)-1-morpholino-3,4-dihydroisoquinoline-3,7-dicarboxylate (3g)

Prepared from dimethyl 4,4'-(2-(ethoxycarbonyl)-2-isocyanopropane-1,3-diyl)dibenzoate (40.9 mg, 0.1 mmol, 1.0 equiv) and morpholino benzoate (31.05 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 1 : 2) furnished the product **3g** as colorless oil (33 mg, 0.067 mmol, 67% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.04 (s, 1H), 7.99 (d, *J* = 7.8 Hz, 1H), 7.92 (d, *J* = 8.3 Hz, 2H), 7.34 (d, *J* = 8.3 Hz, 2H), 7.28 (d, *J* = 7.8 Hz,

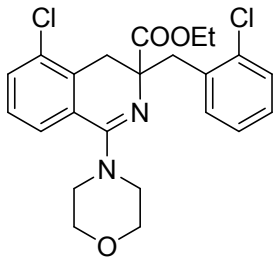
1H), 3.92 (s, 3H), 3.89-3.85 (m, 6H), 3.83-3.78 (m, 3H), 3.40-3.34 (m, 2H), 3.27-3.21 (m, 2H), 3.17-3.13 (m, 3H), 2.75 (d, J = 15.4 Hz, 1H), 0.92 (t, J = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 173.4, 167.3, 166.6, 161.2, 143.3, 142.6, 131.4, 130.9, 129.1, 128.9, 128.6, 127.4, 125.2, 66.8, 65.7, 61.0, 52.4, 52.1, 49.2, 44.9, 36.0, 14.1; HRMS: calcd for $C_{27}H_{30}N_2O_7$ ($M+H^+$) 495.2126; found 495.2115.



ethyl 1-morpholino-7-nitro-3-(4-nitrobenzyl)-3,4-dihydroisoquinoline-3-carboxylate

(3h)

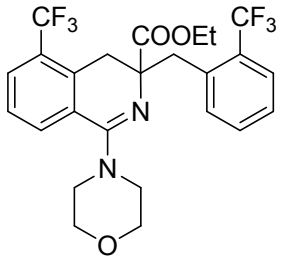
Prepared from ethyl 2-isocyano-2-(4-nitrobenzyl)-3-(4-nitrophenyl)propanoate (38.3 mg, 0.1 mmol, 1.0 equiv) and morpholino benzoate (31.05 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 1 : 4) furnished the product **3e** as a light yellow oil (26 mg, 0.056 mmol, 56% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.23 (s, 1H), 8.20 (d, J = 8.2 Hz, 1H), 8.13 (d, J = 8.6 Hz, 2H), 7.50 (d, J = 8.6 Hz, 2H), 7.40 (d, J = 8.2 Hz, 1H), 3.95-3.88 (m, 2H), 3.88-3.79 (m, 4H), 3.41-3.34 (m, 3H), 3.29-3.19 (m, 4H), 2.80 (d, J = 15.4 Hz, 1H), 0.90 (t, J = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 172.4, 160.5, 147.2, 147.1, 144.8, 144.6, 131.7, 129.5, 125.9, 125.1, 122.9, 121.1, 66.5, 65.6, 61.3, 49.1, 44.9, 36.5, 13.9; HRMS: calcd for $C_{23}H_{24}N_4O_7$ ($M+H^+$) 469.1718; found 469.1719.



ethyl 5-chloro-3-(2-chlorobenzyl)-1-morpholino-3,4-dihydroisoquinoline-3-carboxylate (3i)

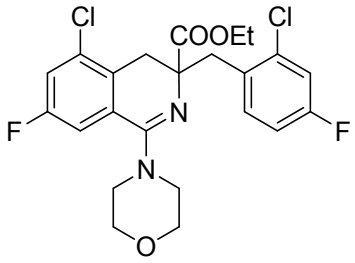
Prepared from ethyl 2-(2-chlorobenzyl)-3-(2-chlorophenyl)-2-isocyanopropanoate (36.2 mg, 0.1 mmol, 1.0 equiv) and morpholino benzoate (31.05 mg, 0.15 mmol, 1.5

equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 1 : 4) furnished the product **3i** as a colorless oil (26 mg, 0.058 mmol, 58% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.53 (d, *J* = 7.1 Hz, 1H), 7.36 (d, *J* = 7.9 Hz, 1H), 7.33-7.28 (m, 2H), 7.19-7.10 (m, 3H), 4.02-3.90 (m, 2H), 3.87-3.82 (m, 2H), 3.79-3.73 (m, 2H), 3.51 (d, *J* = 15.9 Hz, 1H), 3.44 (d, *J* = 13.7 Hz, 1H), 3.38 (d, *J* = 13.7 Hz, 1H), 3.35-3.30 (m, 2H), 3.23-3.17 (m, 2H), 2.67 (d, *J* = 15.9 Hz, 1H), 0.99 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 173.6, 161.6, 136.3, 135.4, 135.2, 133.9, 133.2, 131.1, 129.3, 127.9, 127.3, 126.8, 126.1, 124.6, 66.9, 65.7, 61.0, 49.3, 41.1, 32.1, 13.9; HRMS: calcd for $C_{23}H_{24}Cl_2N_2O_3$(M+H$^+$) 447.1237; found 447.1230.



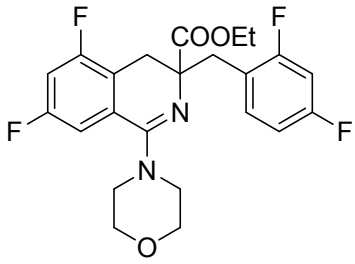
ethyl 1-morpholino-5-(trifluoromethyl)-3-(2-(trifluoromethyl)benzyl)-3,4-dihydroisoquinoline-3-carboxylate (3j)

Prepared from ethyl 2-isocyano-2-(2-(trifluoromethyl)benzyl)-3-(2-(trifluoromethyl)phenyl)propanoate (42.9 mg, 0.1 mmol, 1.0 equiv) and morpholino benzoate (31.05 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 1 : 4) furnished the product **3j** as a colorless oil (42 mg, 0.082 mmol, 82% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.03 (d, *J* = 7.9 Hz, 1H), 7.64-7.60 (m, 3H), 7.44 (t, *J* = 7.6 Hz, 1H), 7.36 (t, *J* = 7.8 Hz, 1H), 7.30 (t, *J* = 7.7 Hz, 1H), 3.91-3.84 (m, 3H), 3.83-3.76 (m, 3H), 3.57 (d, *J* = 14.7 Hz, 1H), 3.51 (d, *J* = 16.0 Hz, 1H), 3.46 (d, *J* = 14.9 Hz, 1H), 3.46 (d, *J* = 14.9 Hz, 1H), 3.37-3.32 (m, 2H), 3.23-3.19 (m, 2H), 2.62 (d, *J* = 16.0 Hz, 1H), 0.89 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ172.9, 161.5, 136.7 (q, *J* = 1.3Hz), 136.3 (q, *J* = 1.3Hz), 132.7, 130.9, 129.55, 129.51 (q, *J* =28.8Hz), 129.0 (q, *J* = 30.0Hz), 127.4 (q, *J* =5.0Hz), 126.5, 126.4, 126.0, 125.7 (q, *J* = 5.0Hz), 124.5 (q, *J* = 272.6Hz), 123.8 (q, *J* = 272.6Hz), 66.7, 64.8, 61.0, 49.1, 40.1, 32.8, 13.5; HRMS: calcd for $C_{25}H_{24}F_6N_2O_3$(M+H$^+$) 515.1764; found 515.1765.



ethyl 5-chloro-3-(2-chloro-4-fluorobenzyl)-7-fluoro-1-morpholino-3,4-dihydroisoquinoline-3-carboxylate (3k)

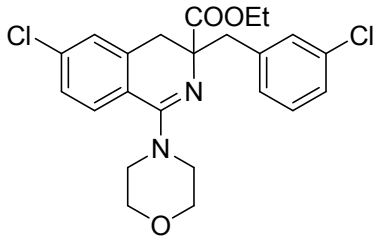
Prepared from ethyl 2-(2-chloro-4-fluorobenzyl)-3-(2-chloro-4-fluorophenyl)-2-isocyanopropanoate (39.8 mg, 0.1 mmol, 1.0 equiv) and morpholino benzoate (31.05 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 1 : 4) furnished the product **3k** as a colorless oil (34 mg, 0.070 mmol, 70% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.52 (dd, *J* = 8.4, 6.6 Hz, 1H), 7.14 (dd, *J* = 8.4, 2.2 Hz, 1H), 7.07 (dd, *J* = 8.6, 2.3 Hz, 1H), 7.02 (dd, *J* = 8.5, 2.1 Hz, 1H), 6.89 (dt, *J* = 8.3, 2.3 Hz, 1H), 4.01-3.89 (m, 2H), 3.87-3.83 (m, 2H), 3.79-3.74 (m, 2H), 3.48 (d, *J* = 15.8 Hz, 1H), 3.39-3.36 (m, 2H), 3.32-3.28 (m, 2H), 3.19-3.15 (m, 2H), 2.57 (d, *J* = 15.6 Hz, 1H), 1.01 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 173.0, 160.8 (d, *J* = 1.3Hz), 161.2 (d, *J* = 247.5Hz), 160.7 (d, *J* =247.5Hz), 135.6(d, *J* = 10.0Hz), 134.4 (d, *J* = 8.8Hz), 133.8 (d, *J* = 8.8Hz), 131.7 (d, *J* = 3.8Hz), 130.8 (d, *J* = 3.8Hz), 127.6 (d, *J* = 6.3Hz), 118.4 (d, *J* = 25.0Hz), 116.3 (d, *J* = 25.0Hz), 113.3 (d, *J* = 21.3Hz), 111.9 (d, *J* = 22.5Hz), 66.6, 65.6, 61.1, 49.2, 40.2, 31.6, 13.9; HRMS: calcd for $C_{23}H_{22}Cl_2F_2N_2O_3$(M+H$^+$) 483.1048; found 483.1045.



ethyl 3-(2,4-difluorobenzyl)-5,7-difluoro-1-morpholino-3,4-dihydroisoquinoline-3-carboxylate (3l)

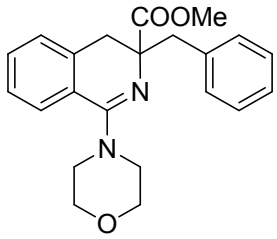
Prepared from ethyl 2-(2,4-difluorobenzyl)-3-(2,4-difluorophenyl)-2-isocyanopropanoate (36.5 mg, 0.1 mmol, 1.0 equiv) and morpholino benzoate (31.05 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column

chromatography purification (EtOAc : petroleum ether 1 : 8) furnished the product **3l** as a colorless oil (22 mg, 0.049 mmol, 49% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.34 (q, *J* = 8.4 Hz, 1H), 6.91 (d, *J* = 8.4 Hz, 1H), 6.83 (dt, *J* = 8.4, 2.3 Hz 1H), 6.79-6.71 (m, 2H), 4.01-3.92 (m, 2H), 3.87-3.82 (m, 2H), 3.79-3.75 (m, 2H), 3.33-3.26 (m, 3H), 3.21-3.17 (m, 4H), 2.53 (d, *J* = 15.6 Hz, 1H), 1.03 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ173.2, 162.1 (dd, *J* = 247.5, 12.5 Hz), 161.6 (dd, *J* = 246.2, 11.3 Hz), 161.4 (dd, *J* = 242.5, 12.5 Hz), 160.4, 160.2 (dd, *J* = 247.5, 11.3 Hz), 133.7 (dd, *J* = 10.0, 6.3 Hz), 127.4 (dd, *J* = 7.5, 7.5 Hz), 120.8 (dd, *J* = 18.8, 3.8 Hz), 119.8 (dd, *J* = 16.3, 3.8 Hz), 110.7 (dd, *J* = 20.0, 2.5 Hz), 109.3 (dd, *J* = 23.8, 3.8 Hz), 105.7 (dd, *J* = 25.6, 25.6 Hz), 103.4 (dd, *J* = 26.3, 26.3 Hz), 66.7, 65.2, 61.2, 49.2, 36.8, 27.2, 14.0; HRMS: calcd for $C_{23}H_{22}F_4N_2O_3$(M+H$^+$) 451.1639; found 451.1634.



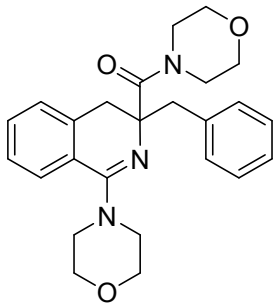
ethyl 6-chloro-3-(3-chlorobenzyl)-1-morpholino-3,4-dihydroisoquinoline-3-carboxylate(3m)

Prepared from ethyl 2-(3-chlorobenzyl)-3-(3-chlorophenyl)-2-isocyanopropanoate (36.2 mg, 0.1 mmol, 1.0 equiv) and morpholino benzoate (31.05 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 1 : 4) furnished the product **3m** as a colorless oil (28 mg, 0.063 mmol, 63% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.45 (s, 1H), 7.35-7.33 (m, 1H), 7.30-7.26 (m, 4H), 7.20 (d, *J* = 7.3 Hz, 1H), 3.99-3.94 (m, 3H), 3.85-3.81 (m, 4H), 3.36-3.22 (m, 3H), 3.13 (d, *J* = 14.1 Hz, 2H), 3.05-2.75 (m, 1H), 2.68 (d, *J* = 14.7 Hz, 1H), 0.95 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 173.6, 159.3, 141.5, 139.3, 133.5, 131.4, 131.2, 130.6, 130.1, 129.0, 128.9, 127.2, 126.8, 124.6, 66.7, 66.0, 60.8, 44.8, 38.5, 29.8, 13.9; HRMS: calcd for $C_{23}H_{24}Cl_2N_2O_3$(M+H$^+$) 447.1237; found 447.1234.



methyl 3-benzyl-1-morpholino-3,4-dihydroisoquinoline-3-carboxylate (3n)

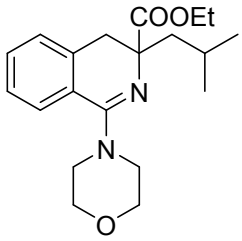
Prepared from methyl 2-benzyl-2-isocyano-3-phenylpropanoate (27.9 mg, 0.1 mmol, 1.0 equiv) and morpholino benzoate (31.05 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 1 : 4) furnished the product **3n** as a colorless oil (32 mg, 0.088 mmol, 88% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ7.41(d, *J* = 7.74 Hz, 1H), 7.35-7.31(m, 1H), 7.27-7.22 (m, 4H), 7.21-7.17(m, 3H), 3.87-3.79(m, 4H), 3.49(s, 3H), 3.35-3.22(m, 4H), 3.10-2.96(m, 3H), 2.75 (d, *J* = 15.3 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ174.7, 161.8, 138.3, 137.2, 130.7, 130.5, 128.8, 127.9, 126.8, 126.6, 126.3, 124.9, 66.9, 66.2, 52.1, 49.3, 44.3, 34.8; HRMS: calcd for $C_{22}H_{24}N_2O_3$(M+H$^+$) 365.1860; found 365.1862.



(3-benzyl-1-morpholino-3,4-dihydroisoquinolin-3-yl)(morpholino)methanone (3o)

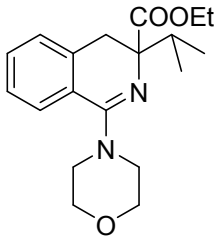
Prepared from 2-benzyl-2-isocyano-1-morpholino-3-phenylpropan-1-one (33.4 mg, 0.1 mmol, 1.0 equiv) and morpholino benzoate (31.05 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 1 : 2) furnished the product **3o** as a colorless oil (36 mg, 0.086 mmol, 86% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.36-7.32 (m, 2H), 7.29-7.20 (m, 5H), 7.19-7.16 (m, 2H), 4.25-4.23 (brs, 1H), 3.88-3.76 (m, 4H), 3.59 (brs, 2H), 3.38 (brs, 2H), 3.33-3.27 (m, 5H), 3.18-3.13 (m, 3H), 3.04 (d, *J* = 13.2 Hz, 1H), 2.97(d, *J* = 13.2 Hz, 1H), 2.68 (d, *J* = 15.0 Hz, 1H), 0.96 (t, *J* = 7.08 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ172.1, 160.6, 140.3, 137.3, 130.8, 129.3, 128.0, 126.8, 126.4, 125.8, 124.4, 67.9, 66.8, 49.2, 44.5, 36.6; HRMS: calcd for $C_{25}H_{29}N_3O_3$(M+H$^+$) 420.2282; found

420.2285.



ethyl 3-isobutyl-1-morpholino-3,4-dihydroisoquinoline-3-carboxylate (3p)

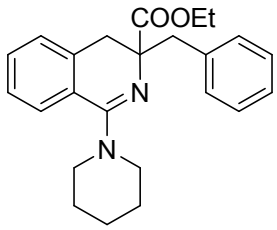
Prepared from ethyl 2-benzyl-2-isocyano-4-methylpentanoate (26 mg, 0.1 mmol, 1.0 equiv) and morpholino benzoate (31.05 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 8 : 1) furnished the product **3p** as colorless oil (23 mg, 67% yield). (new compound). ^{1}H NMR (400 MHz, CDCl3) δ 7.43-7.41 (m, 1H), 7.34-7.25 (m, 2H), 7.20 (d, *J* = 6.8 Hz, 1H), 4.01-3.94 (m, 2H), 3.91-3.80 (m, 4H), 3.38-3.25 (m, 4H), 3.06 (d, *J* = 15.2 Hz, 1H), 2.76 (d, *J* = 15.2 Hz, 1H), 1.95-1.86 (m, 1H), 1.81-1.77 (m, 1H), 1.69-1.64 (m, 1H), 1.04 (t, *J* = 7.2 Hz, 3H), 0.99 (d, *J* = 6.4 Hz, 3H), 0.88 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, CDCl3) δ 175.0, 161.4, 138.0, 130.1, 128.4, 126.7, 126.0, 125.0, 66.8, 64.6, 60.5, 49.1, 47.4, 37.2, 24.9, 24.2, 23.8, 14.0. HRMS (ESI) calcd for $C_{20}H_{28}N_2O_3$ $[M+H]^+$: 345.2173, Found: 345.2175.



ethyl 3-isopropyl-1-morpholino-3,4-dihydroisoquinoline-3-carboxylate (3q)

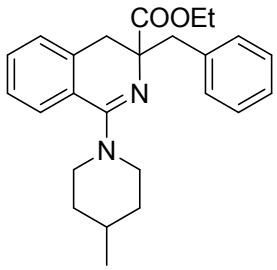
Prepared from ethyl 2-benzyl-2-isocyano-3-methylbutanoate (25 mg, 0.1 mmol, 1.0 equiv) and morpholino benzoate (31.05 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 8 : 1) furnished the product **3q** as colorless oil (16 mg, 50% yield). (new compound). ^{1}H NMR (400 MHz, CDCl3) δ 7.41-7.39 (m, 1H), 7.32-7.21 (m, 3H), 3.93-3.86 (m, 4H), 3.81-3.76 (m, 2H), 3.47-3.41 (m, 2H), 3.26-3.21 (m, 2H), 2.99 (d, *J* = 14.8 Hz, 1H), 2.82 (d, *J* = 14.8 Hz, 1H), 2.24-2.17 (m, 1H), 1.04 (dd, *J* = 6.8, 3.6 Hz, 6H), 0.94 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, CDCl3) δ 174.4, 161.8, 138.6, 130.0, 128.4,

126.5, 125.9, 67.8, 66.8, 60.2, 49.1, 35.7, 33.7, 17.9, 17.6, 14.0. HRMS (ESI) calcd for $C_{19}H_{26}N_2O_3$ $[M+H]^+$: 331.2016, Found: 331.2017.



ethyl 3-benzyl-1-(piperidin-1-yl)-3,4-dihydroisoquinoline-3-carboxylate (4a)

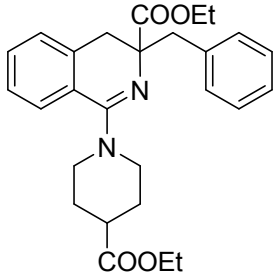
Prepared from ethyl 2-benzyl-2-isocyano-3-phenylpropanoate (29.3 mg, 0.1 mmol, 1.0 equiv) and piperidin-1-yl benzoate (30.75 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 1 : 2) furnished the product **4a** as a light yellow oil (35 mg, 0.092 mmol, 92% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.41 (d, *J* = 7.43 Hz, 1H), 7.31-7.28 (m, 1H), 7.26-7.21 (m, 5H), 7.20-7.17 (m, 2H), 3.30-3.21 (m, 4H), 3.11 (d, *J* = 13.2 Hz, 1H), 3.05 (d, *J* = 15.1 Hz, 1H), 2.98 (d, *J* = 13.2 Hz, 1H), 2.73 (d, *J* = 15.1 Hz, 1H), 1.72-1.65 (m, 6H), 0.96 (t, *J* = 7.15 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.4, 162.4, 138.3, 137.6, 130.8, 129.9, 128.6, 127.8, 126.6, 126.5, 126.4, 125.9, 65.9, 60.6, 49.7, 44.5, 35.2, 26.0, 25.2, 14.1; HRMS: calcd for $C_{24}H_{28}N_2O_2$ ($M+H^+$) 377.2224; found 377.2223.



ethyl 3-benzyl-1-(4-methylpiperidin-1-yl)-3,4-dihydroisoquinoline-3-carboxylate (4b)

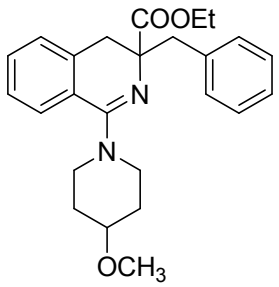
Prepared from ethyl 2-benzyl-2-isocyano-3-phenylpropanoate (29.3 mg, 0.1 mmol, 1.0 equiv) and 4-methylpiperidin-1-yl benzoate (32.85 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 1 : 4) furnished the product **4b** as a colorless oil (26 mg, 0.067 mmol,

67% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.41 (d, *J* = 7.45 Hz, 1H), 7.30-7.28 (m, 1H), 7.26-7.23 (m, 5H), 7.19-7.17 (m, 2H),3.94-3.80 (m, 4H), 3.11(d, *J* = 13.2 Hz, 1H), 3.05 (d, *J* = 15.0 Hz, 1H), 2.98 (d, *J* = 13.2 Hz, 1H), 2.80-2.70 (m, 2H), 2.66-2.61 (m, 1H), 1.72-1.58 (m, 3H), 1.45-1.37 (m, 1H), 1.35-1.23 (m, 1H), 1.00 (d, *J* = 6.5 Hz, 3H), 0.96 (t, *J* = 7.10 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ174.3, 162.1, 138.1, 137.5, 130.7, 129.8, 128.4, 127.7, 126.5, 126.3, 125.8, 65.9, 60.5, 49.1, 48.7, 44.3, 35.0, 34.3, 34.1, 31.5, 22.1, 13.9; HRMS: calcd for $C_{25}H_{30}N_2O_2$ ($M+H^+$) 391.2380; found 391.2371.



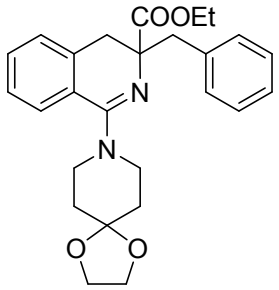
ethyl 3-benzyl-1-(4-(ethoxycarbonyl)piperidin-1-yl)-3,4-dihydroisoquinoline-3-carboxylate (4c)

Prepared from ethyl 2-benzyl-2-isocyano-3-phenylpropanoate (29.3 mg, 0.1 mmol, 1.0 equiv) and ethyl 1-(benzoyloxy)piperidine-4-carboxylate (41.55 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 1 : 4) furnished the product **4c** as a colorless oil (31 mg, 0.069 mmol, 69% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.40 (d, *J* = 7.25 Hz, 1H), 7.31-7.29 (m, 1H), 7.26-7.22 (m, 5H), 7.20-7.18 (m, 2H), 4.18(q, *J* = 7.10 Hz, 2H), 3.95-3.80 (m, 4H), 3.12-2.97 (m, 3H), 2.86-2.72 (m, 3H), 2.51 (brs, 1H), 2.02-1.92 (m, 3H), 1.88-1.80(m, 1H), 1.29 (t, *J* = 7.25 Hz, 1H), 0.95 (t, *J* = 7.10 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 175.2, 174.3, 162.1, 138.3, 137.4, 130.8, 130.2, 128.7, 127.9, 126.8, 126.5, 126.3, 125.6, 65.9, 60.7, 60.6, 48.4, 48.0, 44.4, 41.9, 35.1, 28.3, 28.2, 14.4, 14.0; HRMS: calcd for $C_{27}H_{32}N_2O_4$ ($M+H^+$) 449.2435; found 449.2431.



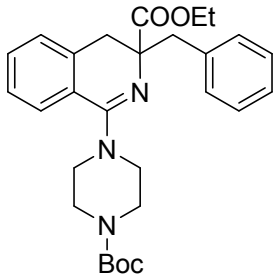
ethyl 3-benzyl-1-(4-methoxypiperidin-1-yl)-3,4-dihydroisoquinoline-3-carboxylate (4d)

Prepared from ethyl 2-benzyl-2-isocyano-3-phenylpropanoate (29.3 mg, 0.1 mmol, 1.0 equiv) and 4-methoxypiperidin-1-yl benzoate (35.25 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 1 : 2) furnished the product **4d** as a colorless oil (27 mg, 0.067 mmol, 67% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.40 (d, *J* = 7.40 Hz, 1H), 7.31-7.29 (m, 1H), 7.26-7.23 (m, 5H), 7.20-7.18 (m, 2H), 3.93-3.86 (m, 2H), 3.71(brs, 2H), 3.39 (s, 4H), 3.12-2.97 (m, 4H), 2.92-2.87 (m, 1H), 2.73(d, *J* = 15.1 Hz, 1H), 2.04-1.97 (m, 2H), 1.75-1.63 (m, 3H), 0.95 (t, *J* = 7.10 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.3, 161.8, 138.3, 137.5, 130.8, 130.2, 128.7, 127.8, 126.7, 126.5, 126.3, 125.7, 66.0, 60.6, 55.7, 46.4, 46.2, 44.5, 35.3, 30.9, 30.8, 14.0; HRMS: calcd for $C_{25}H_{30}N_2O_3$ ($M+H^+$) 407.2329; found 407.2331.



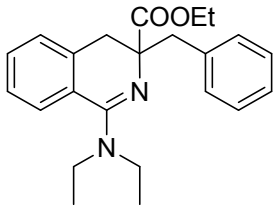
ethyl 3-benzyl-1-(1,4-dioxa-8-azaspiro[4.5]decan-8-yl)-3,4-dihydroisoquinoline-3-carboxylate (4e)

Prepared from ethyl 2-benzyl-2-isocyano-3-phenylpropanoate (29.3 mg, 0.1 mmol, 1.0 equiv) and 1, 4-dioxa-8-azaspiro[4.5]decan-8-yl benzoate (39.45 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 1 : 2) furnished the product **4e** as a colorless oil (42 mg, 0.096 mmol, 96% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ7.39 (d, *J* = 7.55 Hz, 1H), 7.31-7.28 (m, 1H), 7.26-7.21 (m, 5H), 7.19-7.17 (m, 2H), 3.99 (s, 4H), 3.92-3.85(m, 2H), 3.49-3.38 (m, 4H), 3.10(d, *J* = 13.1 Hz, 1H), 3.06(d, *J* = 15.1 Hz, 1H), 3.01(d, *J* = 13.1 Hz, 1H), 2.71(d, *J* = 15.1 Hz, 1H),1.90-1.75 (m, 4H), 0.93 (t, *J* = 7.14 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.2, 161.4, 138.3, 137.5, 130.8, 130.1, 128.6, 127.8, 126.7, 126.5, 126.2, 125.7, 107.9, 66.1, 64.5, 60.6, 46.5, 44.7, 35.4, 34.8, 14.0; HRMS: calcd for $C_{26}H_{30}N_2O_4$ ($M+H^+$) 435.2278; found 435.2273.



ethyl 3-benzyl-1-(4-(tert-butoxycarbonyl)piperazin-1-yl)-3,4-dihydroisoquinoline-3-carboxylate (4f)

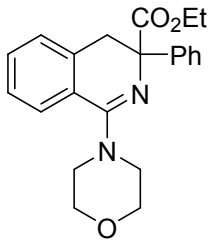
Prepared from ethyl 2-benzyl-2-isocyano-3-phenylpropanoate (29.3 mg, 0.1 mmol, 1.0 equiv) and tert-butyl 4-(benzoyloxy)piperazine-1-carboxylate (45.9 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 1 : 4) furnished the product **4f** as a colorless oil (47 mg, 0.099 mmol, 99% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.39 (d, *J* = 7.6 Hz, 1H), 7.30-7.33 (m, 1H), 7.25-7.27 (m, 1H), 7.23-7.24 (m, 4H), 7.16-7.21 (m, 2H), 3.86-3.98 (m, 2H), 3.56-3.61 (m, 2H), 3.49-3.54 (m, 2H), 3.28-3.33 (m, 2H), 3.21-3.26 (m, 2H), 3.05-3.11 (m, 2H), 3.02 (d, *J* = 13.1 Hz, 1H), 2.73 (d, *J* = 15.1 Hz, 4H), 1.49 (s, 9H), 0.95 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.1, 161.7, 155.1, 138.3, 137.3, 130.8, 130.4, 128.7, 127.8, 126.8, 126.5, 126.1, 125.2, 79.9, 65.9, 60.7, 48.5, 44.4, 35.2, 28.6, 14.0; HRMS: calcd for $C_{28}H_{35}N_3O_4$ ($M+H^+$) 478.2700; found 478.2700.



ethyl 3-benzyl-1-(diethylamino)-3,4-dihydroisoquinoline-3-carboxylate (4g)

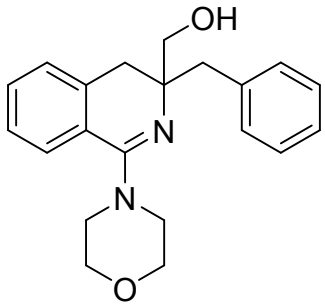
Prepared from ethyl 2-benzyl-2-isocyano-3-phenylpropanoate (29.3 mg, 0.1 mmol, 1.0 equiv) and *O*-benzoyl-*N*,*N*-diethylhydroxylamine (28.95 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 1 : 4) furnished the product **4g** as a colorless oil (25 mg, 0.069 mmol, 69% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.40 (d, *J* = 7.6 Hz, 1H), 7.32 (d, *J* = 7.3 Hz, 2H), 7.28-7.21 (m, 4H), 7.17 (t, *J* = 6.7 Hz, 2H), 3.81 (q, *J* = 7.1 Hz, 2H), 3.46-3.38 (m, 2H), 3.25-3.18 (m, 2H), 3.13 (d, *J* = 13.1 Hz, 1H), 3.09-3.04 (m, 2H), 2.67 (d, *J* = 14.9 Hz, 1H), 1.14 (t, *J* = 7.0 Hz, 6H), 0.88 (t, *J* = 7.1 Hz, 3H);

^{13}C NMR (125 MHz, $CDCl_3$): δ 174.6, 160.8, 138.3, 137.8, 130.9, 129.7, 128.4, 127.7, 126.8, 126.5, 126.4, 126.0, 66.5, 60.4, 45.3, 43.6, 35.9, 13.9, 13.1; HRMS: calcd for $C_{23}H_{28}N_2O_2$ ($M+H^+$) 365.2224; found 365.2219.



ethyl 1-morpholino-3-phenyl-3,4-dihydroisoquinoline-3-carboxylate (6)

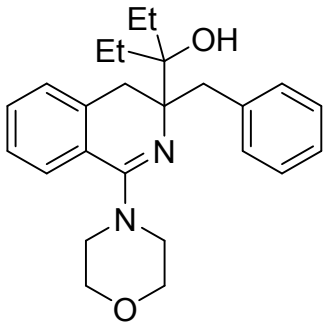
Prepared from ethyl 2-isocyano-2,3-diphenylpropanoate (27.9 mg, 0.1 mmol, 1.0 equiv) and morpholino benzoate (31.05 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 8 : 1) furnished the product **6** as colorless oil (14 mg, 37% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78-7.76 (m, 2H), 7.46-7.45 (m, 1H), 7.38-7.26 (m, 6H), 3.97-3.91 (m, 4H), 3.89-3.84 (m, 2H), 3.68-3.65 (m, 1H), 3.55-3.50 (m, 2H), 3.42-3.37 (m, 2H), 3.06-3.02 (m, 1H), 0.96 (t, J = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, CDCl3) δ 173.2, 162.1, 142.6, 138.6, 130.5, 128.7, 128.2, 127.2, 126.7, 126.6, 126.1, 125.0, 66.8, 61.0, 49.2, 37.6, 13.8. HRMS calcd for $C_{22}H_{24}N_2O_3$ $[M+H]^+$: 365.1860, Found: 365.1866.



(3-benzyl-1-morpholino-3,4-dihydroisoquinolin-3-yl)methanol (8)

To an oven-dried flask containing **3a** (37.8 mg) was added 1.0 mL of anhydrous THF under argon. The mixture was cooled to 0 ^{o}C in an ice-water bath. To the solution was added a solution of $LiAlH_4$ (50 uL, 0.12 mmol, 2.4 M in THF) dropwise. The resulting mixture was warmed to room temperature and stirred for 1 h. Then the reaction was quenched with aqueous NH_4Cl solution and extracted with DCM for three times. Column chromatography purification (EtOAc : petroleum ether 1 : 1) furnished the product **8** as colorless oil (23.5 mg, 70% yield). ^{1}H NMR (400 MHz,

$CDCl_3$) δ 7.53-7.46 (m, 2H), 7.38–7.16 (m, 6H), 7.02-7.01 (m, 2H), 3.96-3.93 (m, 2H), 3.85–3.80 (m, 2H), 3.73 (s, 1H), 3.41–3.17 (m, 6H), 2.77 (d, *J* = 13.2 Hz, 1H), 2.54-2,46 (m, 2H), 2.13 (d, *J* = 12.8 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.3, 138.4, 137.5, 130.8, 130.4, 129.4, 129. 3, 128.0, 126.7, 126.2, 66.8, 60.0, 49.4, 37.7, 32.0. HRMS calcd for $C_{21}H_{24}N_2O_2$ $[M+H]^+$: 337.1911, Found: 337.1915.



3-(3-benzyl-1-morpholino-3,4-dihydroisoquinolin-3-yl)pentan-3-ol (9)

To an oven-dried flask containing **3a** (37.8 mg) was added 1.0 mL of anhydrous THF under argon. The mixture was cooled to 0 °C in an ice-water bath. To the solution was added a solution of EtMgBr (300 uL, 0.3 mmol, 1.0 M in THF) dropwise. The resulting mixture was stirred at 0 °C for 3 h. Then the reaction was quenched with aqueous NH_4Cl solution and extracted with DCM for three times. Column chromatography purification (EtOAc : petroleum ether 1 : 1) furnished the product **9** as colorless oil (17.6 mg, 45% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21-7.17 (m, 1H), 7.09-7.07 (m, 1H), 6.96-9.92 (m, 1H), 6.89-6.82 (m, 4H), 6.65-6.63 (m, 2H), 3.91-3.85 (m, 2H), 3.77-3.72 (m, 2H), 3.35-3.30 (m, 2H), 3.18-3.03 (m, 4H), 2.85-2.81 (m, 1H), 2.67-2.66 (m, 1H), 2.04-1.95 (m, 1H), 1.87-1.78 (m, 1H), 1.66-1.60 (m, 4H), 1.11 (t, *J* = 7.6 Hz, 3H), 1.01 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.1, 138.5, 130.4, 130.0, 128.5, 126.7, 125.9, 125.7, 124.9, 77.8, 66.8, 65.1, 49.4, 41.0, 33.4, 27.9, 26.5, 9.8, 9.3. HRMS calcd for $C_{25}H_{32}N_2O_2$ $[M+H]^+$: 393.2537, Found: 393.2535.

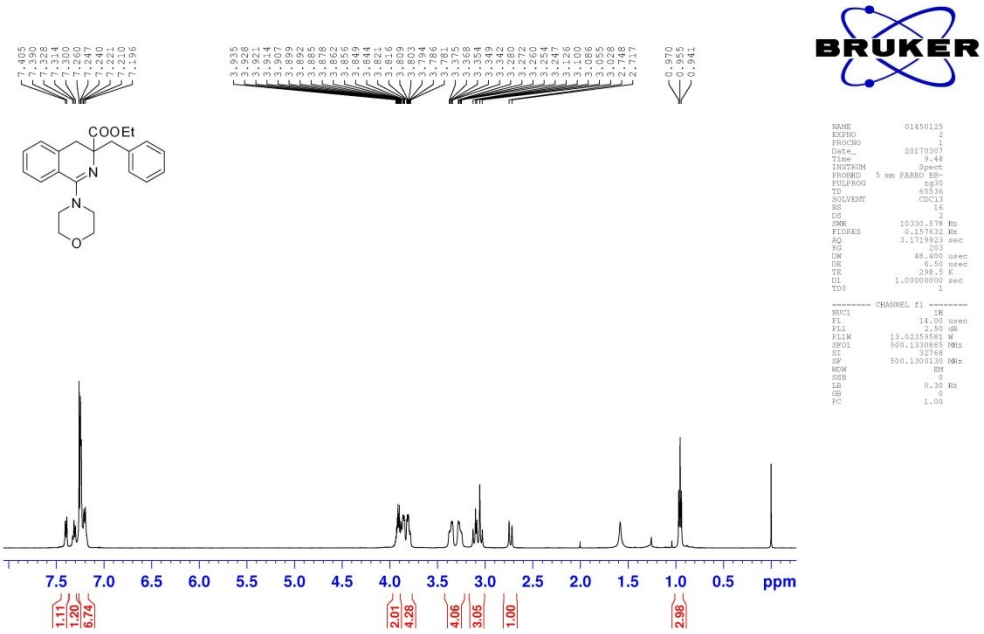
IV. References

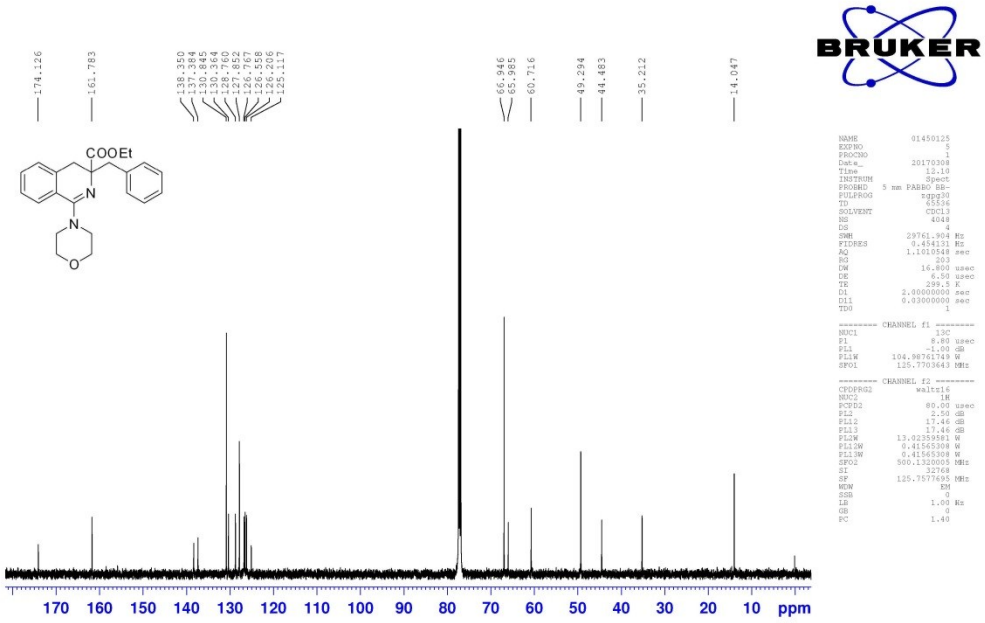
The isocyanide materials were synthesized according to the literature: (a) Ito, Y.; Higuchi N.; Murakami, M. *Tetrahedron Lett*. 1988, **29**, 5151-5154; (b) Zhou, F.; Guo, J.; Liu, J.; Ding, K.; Yu S.; Cai, Q. *J. Am. Chem. Soc*. 2012, **134**, 14326-14329. (2) Yang, W.; Long, Y.; Zhang, S.; Zeng Y.; Cai, Q.

Org. Lett., 2013, **15**, 3598-3601.

V. Copies of ^{1}H and ^{13}C NMR Spectra

3a

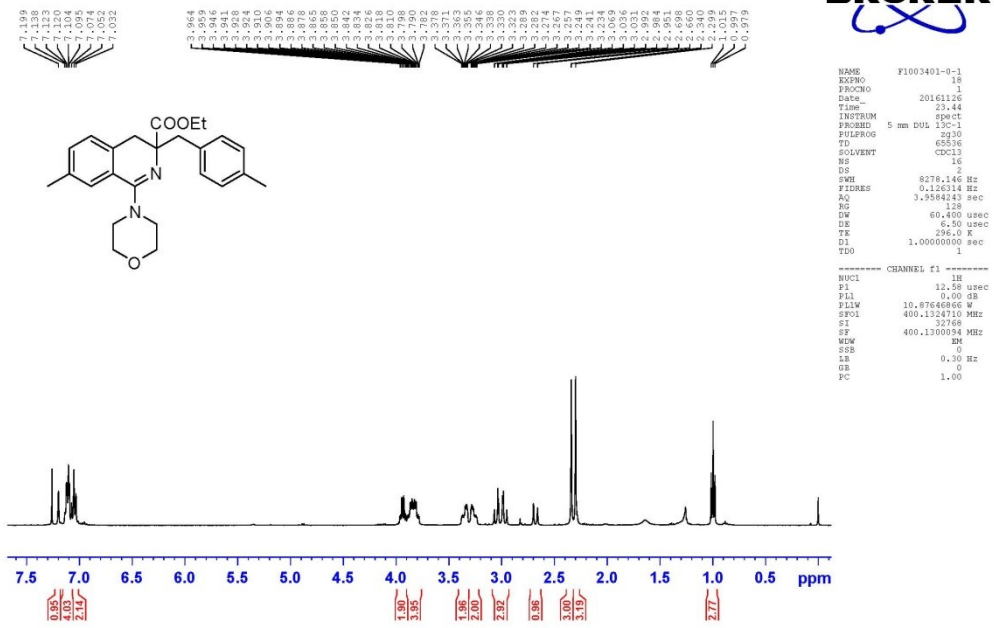




BRUKER

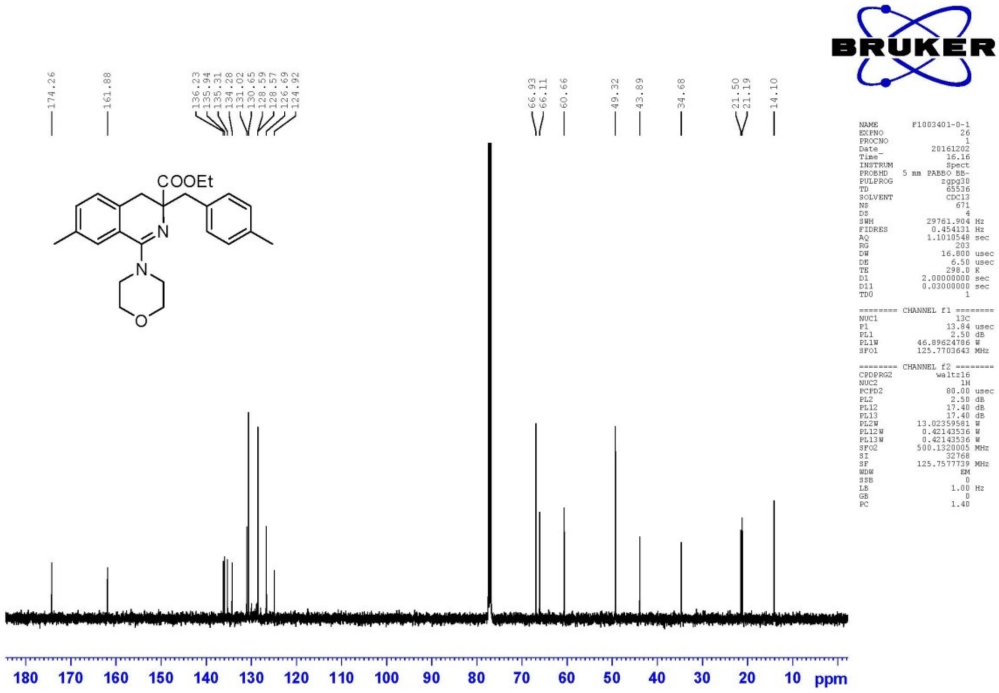
```
NAME            01450125
EXPNO                5
PROCNO               1
Date_         20170308
Time              12.10
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                4048
DS                   4
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010548 sec
RG                 203
DW               16.800 usec
DE                6.50 usec
TE               299.5 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                13C
P1                 8.80 usec
PL1               -1.00 dB
PL1W       104.98761749 W
SFO1       125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2             80.00 usec
PL2                2.50 dB
PL12              17.46 dB
PL13              17.46 dB
PL2W       13.02359581 W
PL12W       0.41565308 W
PL13W       0.41565308 W
SFO2       500.1320005 MHz
SI                32768
SF         125.7577695 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC                1.40
```

3b

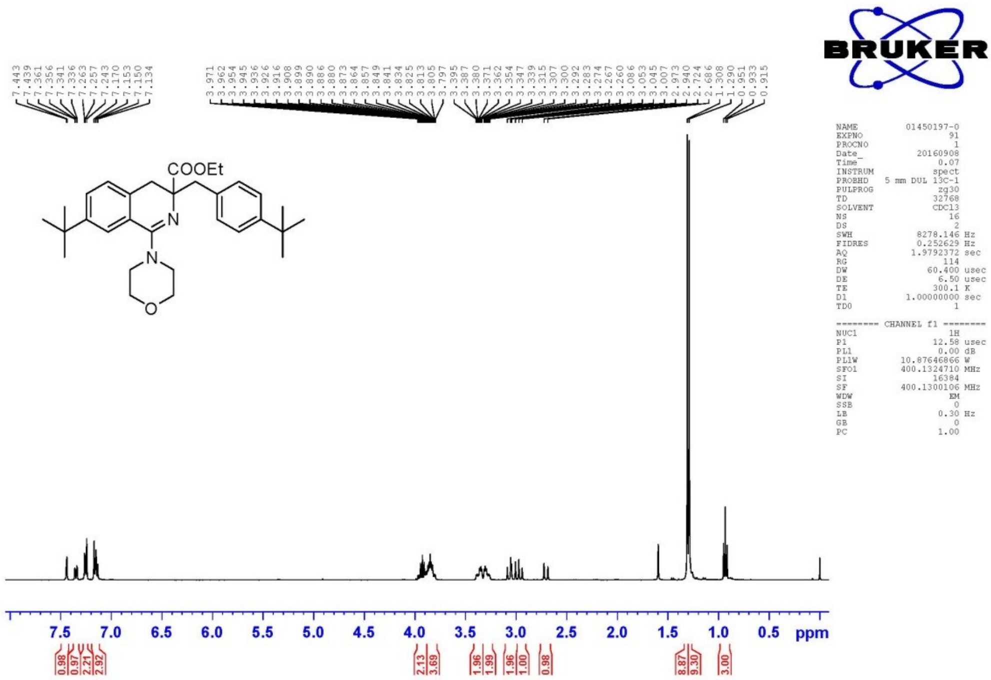


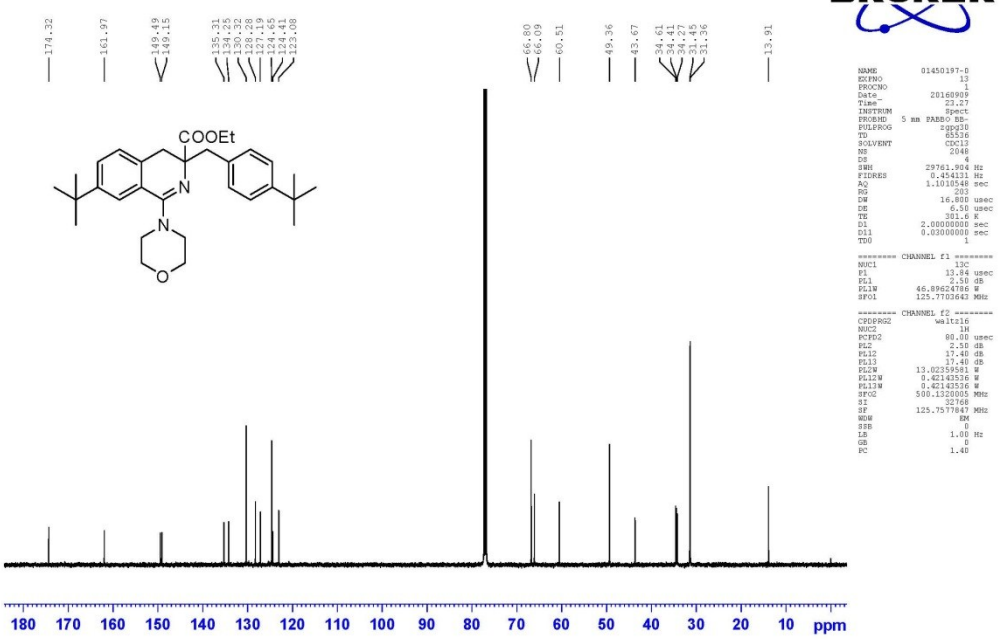
BRUKER

```
NAME         F1003401-0-1
EXPNO                  18
PROCNO                  1
Date_           20161126
Time               23.44
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG           zg30
TD                65536
SOLVENT           CDCl3
NS                  16
DS                   2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                 128
DW               60.400 usec
DE                6.50 usec
TE               296.0 K
D1            1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1                12.58 usec
PL1                0.00 dB
PL1W       10.87646866 W
SFO1       400.1324710 MHz
SI                32768
SF         400.1300094 MHz
WDW                EM
SSB                 0
LB               0.30 Hz
GB                  0
PC                1.00
```


174.26
161.88
136.23
135.94
135.31
134.28
131.02
130.65
128.59
128.57
126.69
124.92
66.93
66.11
60.66
49.32
43.89
34.68
21.50
21.19
14.10
COOEt
N
O
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

3c


COOEt
N
O
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.98
0.97
2.21
2.92
2.13
3.69
1.96
1.99
1.96
1.00
0.98
8.87
9.30
3.00

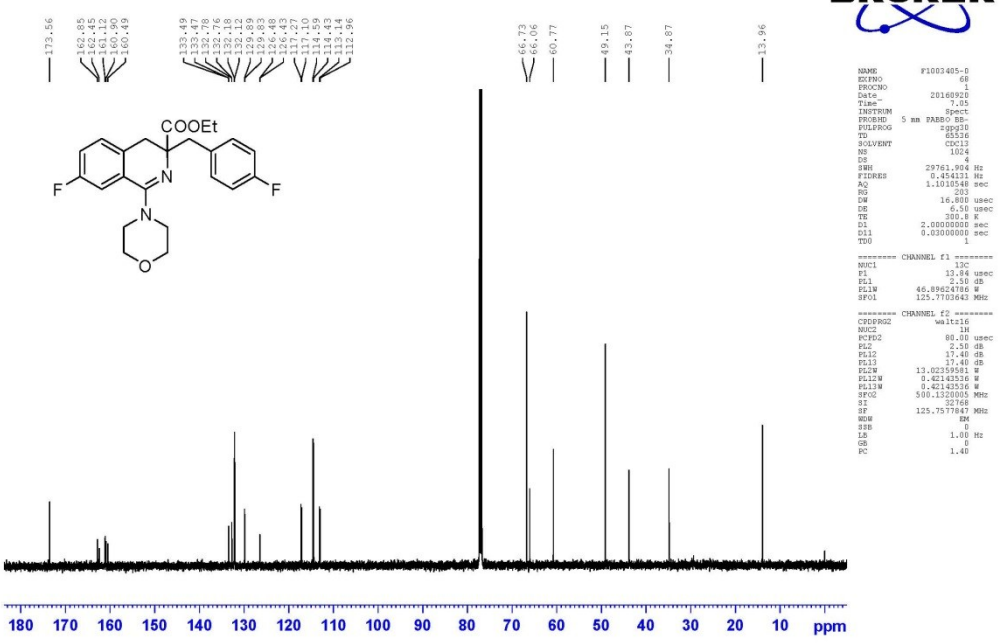


NAME 01450197-D
EXPNO 12
PROCNO 1
Date_ 20160909
Time 23.27
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 301.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 13.84 usec
PL1 2.50 dB
PL1W 46.89624786 W
SFO1 125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577847 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

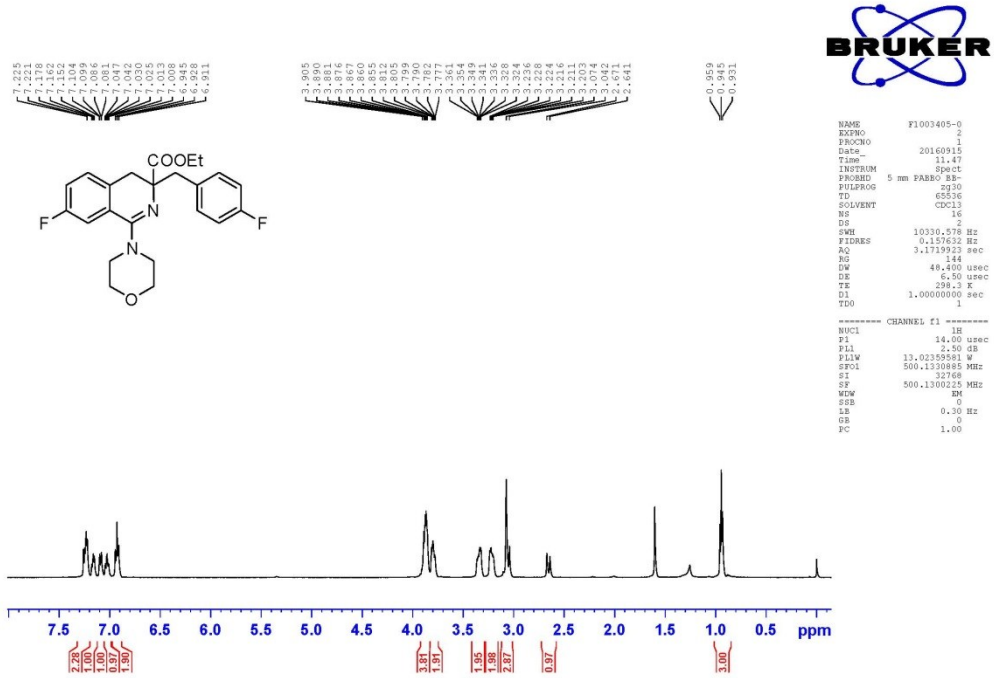
3d



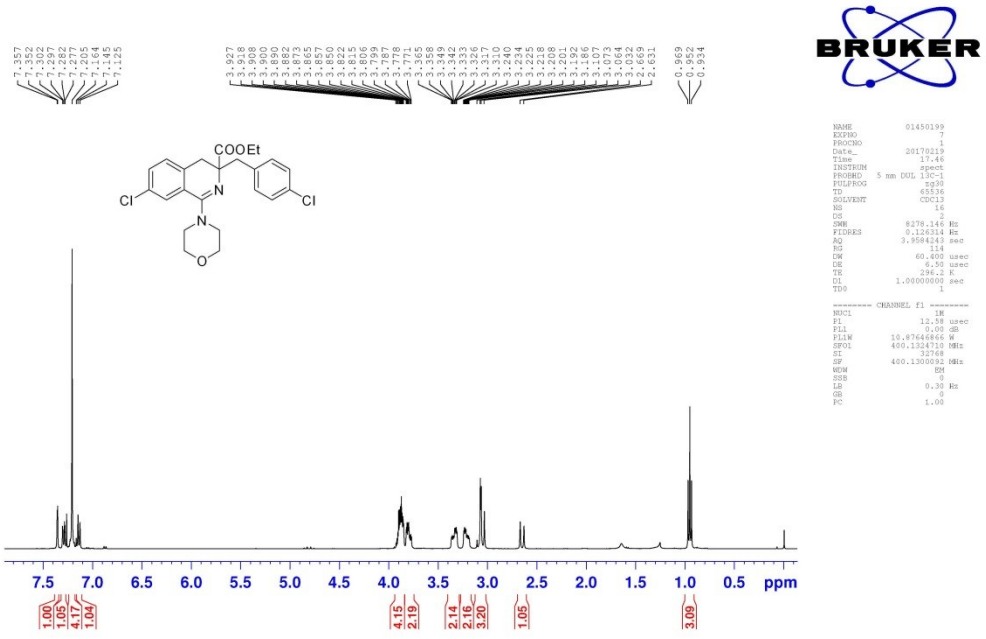
NAME F1003405-D
EXPNO 68
PROCNO 1
Date_ 20160920
Time 7.05
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 300.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

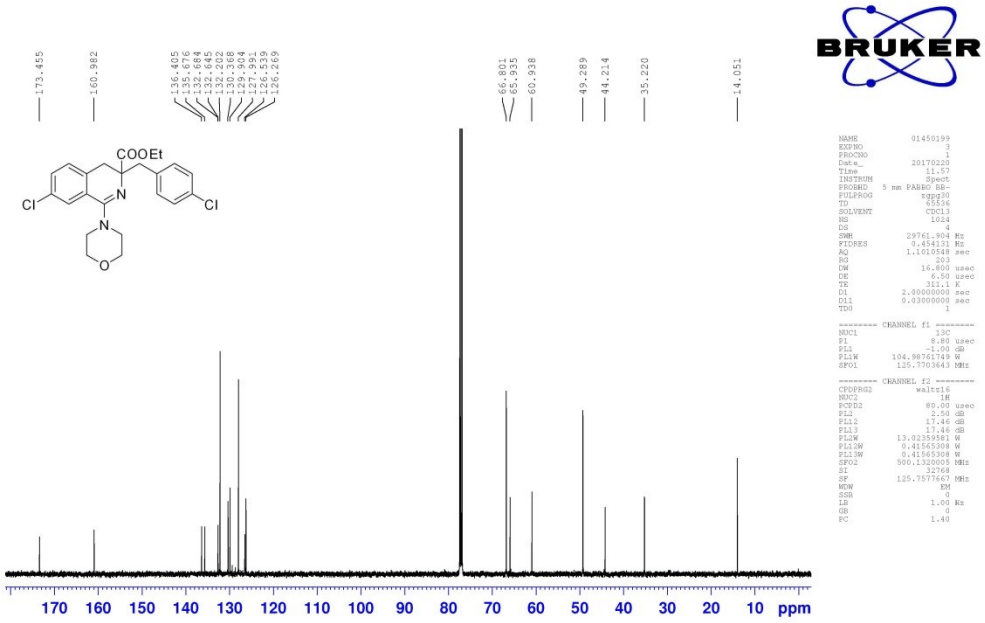
======== CHANNEL f1 ========
NUC1 13C
P1 13.84 usec
PL1 2.50 dB
PL1W 46.89624786 W
SFO1 125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577847 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

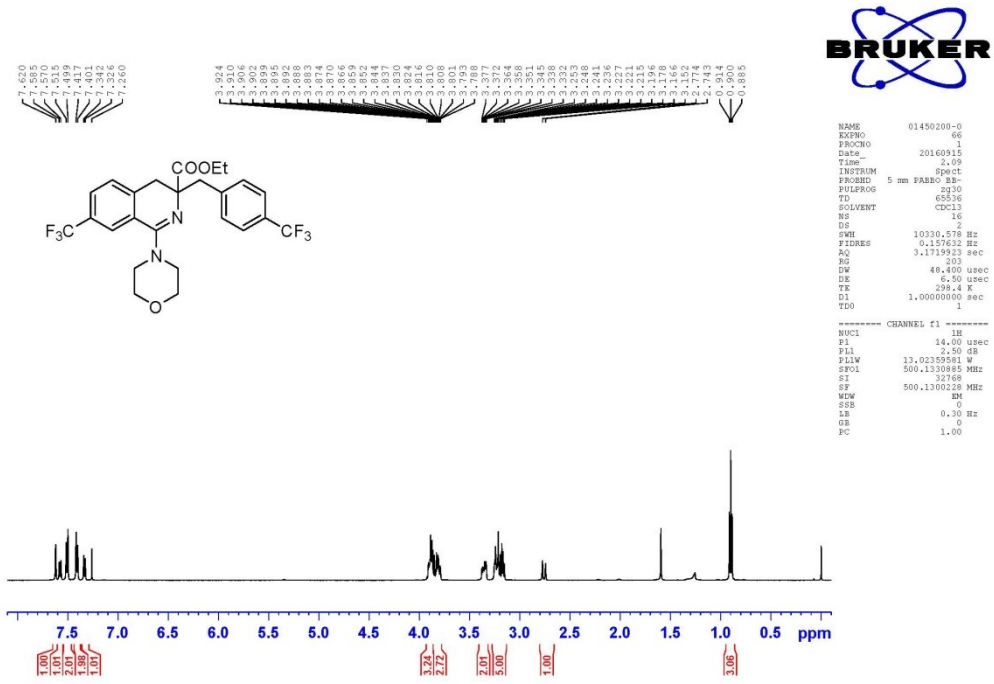

7.225
7.221
7.178
7.162
7.152
7.104
7.099
7.086
7.081
7.047
7.042
7.030
7.025
7.013
7.008
6.945
6.928
6.911
3.905
3.880
3.811
3.876
3.867
3.860
3.815
3.812
3.805
3.799
3.790
3.782
3.777
3.361
3.354
3.349
3.341
3.336
3.328
3.324
3.236
3.226
3.224
3.216
3.211
3.203
3.074
3.042
2.671
2.641
0.959
0.945
0.931
COOEt
F
F
N
N
O
NAME F1003405-0
EXPNO 2
PROCNO 1
Date_ 20160915
Time 11.47
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 144
DW 48.400 usec
DE 6.50 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 2.50 dB
PL1W 13.02359581 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300225 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.28
1.00
1.00
0.97
1.90
3.81
1.91
1.95
1.98
2.87
0.97
3.00

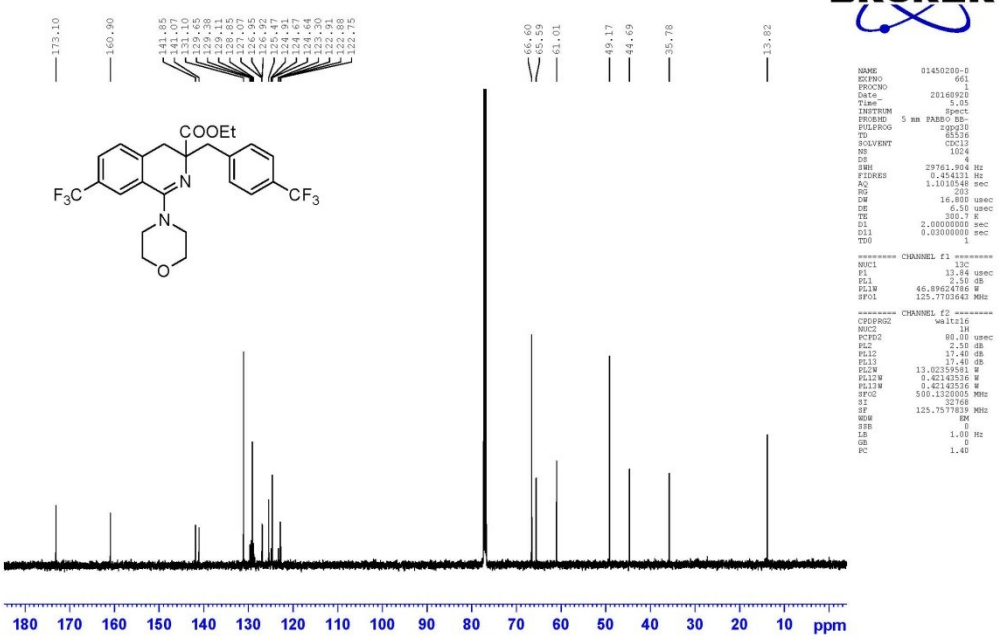
3e


7.357
7.352
7.302
7.297
7.282
7.277
7.205
7.164
7.145
7.125
3.927
3.918
3.908
3.900
3.890
3.882
3.873
3.865
3.857
3.850
3.822
3.815
3.805
3.799
3.787
3.778
3.771
3.765
3.358
3.349
3.342
3.333
3.326
3.317
3.310
3.240
3.234
3.225
3.218
3.208
3.201
3.192
3.186
3.107
3.073
3.064
3.032
2.669
2.631
0.969
0.952
0.934
COOEt
Cl
Cl
N
N
O
NAME 01450199
EXPNO 7
PROCNO 1
Date_ 20170219
Time 17.46
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 114
DW 60.400 usec
DE 6.50 usec
TE 296.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
1.05
4.17
1.04
4.15
2.19
2.14
2.16
3.20
1.05
3.09


173.455
160.982
136.405
135.676
132.684
132.655
132.202
130.368
129.904
127.991
127.539
126.269
66.801
65.935
60.938
49.289
44.214
35.220
14.051
COOEt
Cl
Cl
N
N
O
BRUKER
NAME 01450199
EXPNO 3
PROCNO 1
Date_ 20170220
Time 11.57
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 313.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.80 usec
PL1 -1.00 dB
PL1W 104.98761749 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 13.02359581 W
PL12W 0.41565308 W
PL13W 0.41565308 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577467 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

3f


7.620
7.585
7.570
7.515
7.499
7.487
7.401
7.342
7.326
7.260
3.924
3.910
3.906
3.902
3.899
3.895
3.892
3.888
3.883
3.874
3.870
3.866
3.859
3.852
3.844
3.837
3.830
3.824
3.816
3.810
3.808
3.801
3.793
3.788
3.377
3.372
3.364
3.358
3.351
3.345
3.338
3.332
3.253
3.248
3.241
3.236
3.227
3.221
3.215
3.196
3.178
3.166
3.152
2.774
2.773
0.914
0.900
0.885
COOEt
F3C
CF3
N
N
O
BRUKER
NAME 01450200-0
EXPNO 66
PROCNO 1
Date_ 20160915
Time 2.09
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 203
DW 48.400 usec
DE 6.50 usec
TE 298.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 2.50 dB
PL1W 13.02359581 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300228 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
1.01
2.01
1.98
1.01
3.24
2.72
2.01
5.00
1.00
3.06

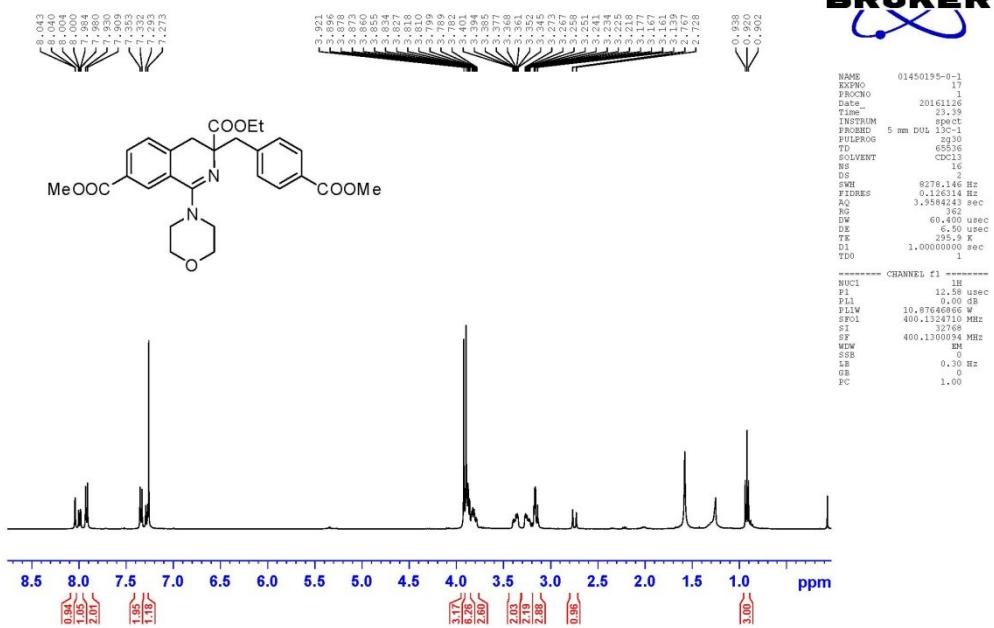


NAME 01450200-0
EXPNO 661
PROCNO 1
Date_ 20160926
Time_ 5.05
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 300.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 13.84 usec
PL1 2.50 dB
PL1W 46.89624786 W
SFO1 125.7703643 MHz

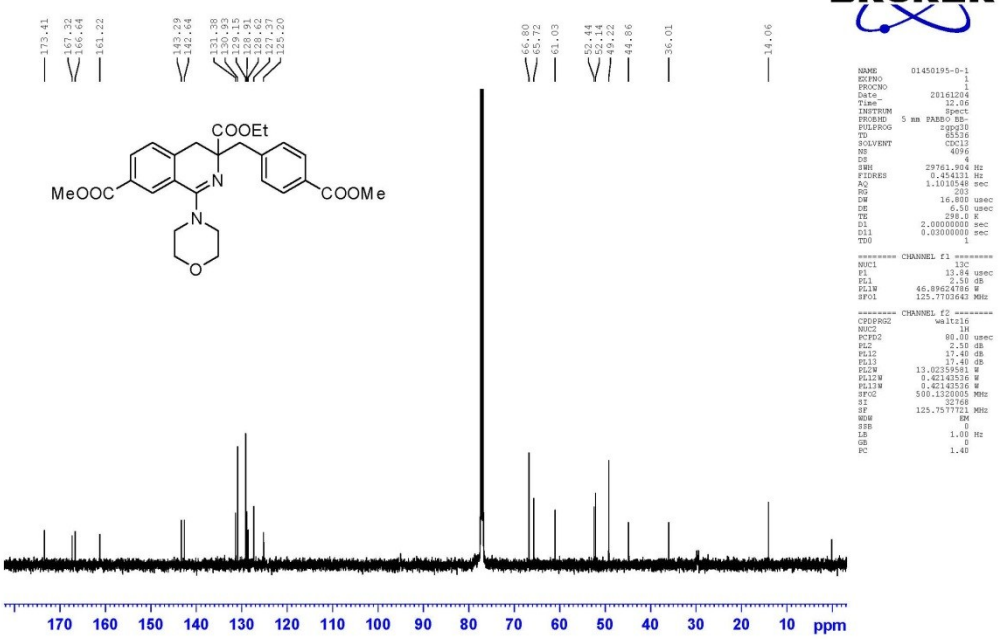
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577839 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

3g



NAME 01450195-0-1
EXPNO 17
PROCNO 1
Date_ 20161126
Time_ 23.39
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.50 usec
TE 295.9 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

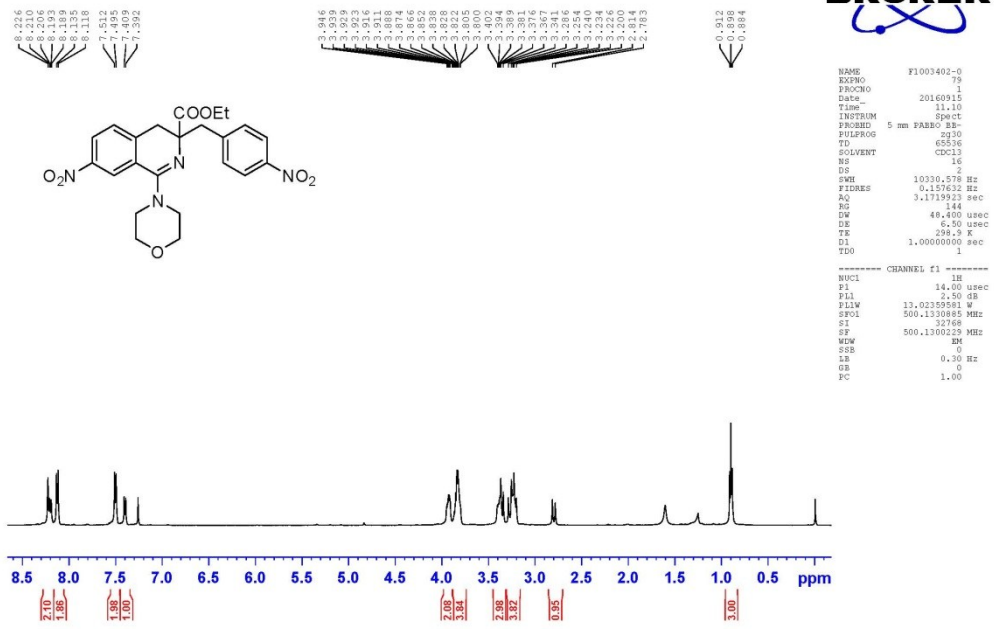


NAME 01450195-0-1
EXPNO 1
PROCNO 1
Date_ 20161204
Time_ 12.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4096
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 13.84 usec
PL1 2.50 dB
PL1W 46.89624786 W
SFO1 125.7703643 MHz

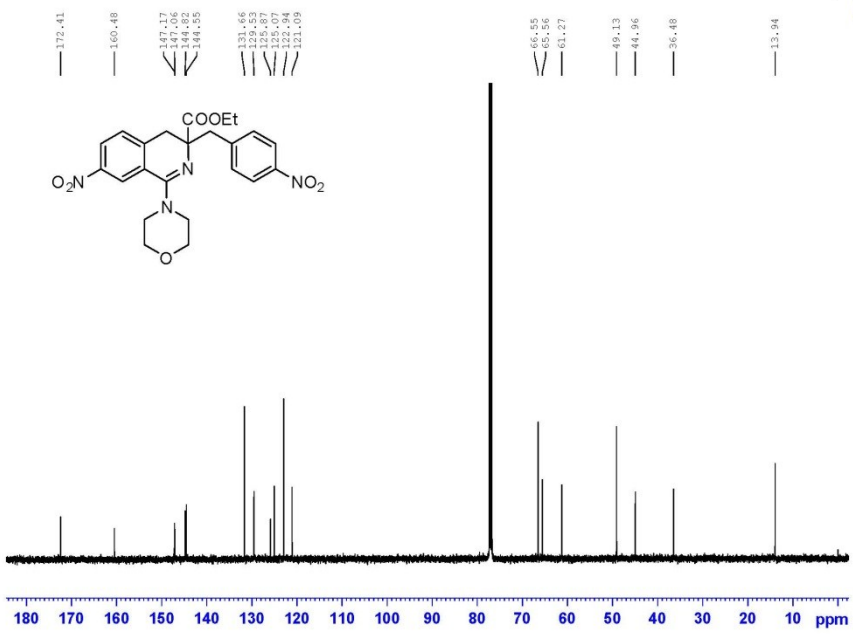
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577721 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

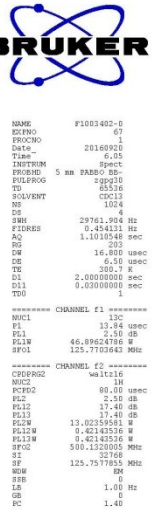
3h



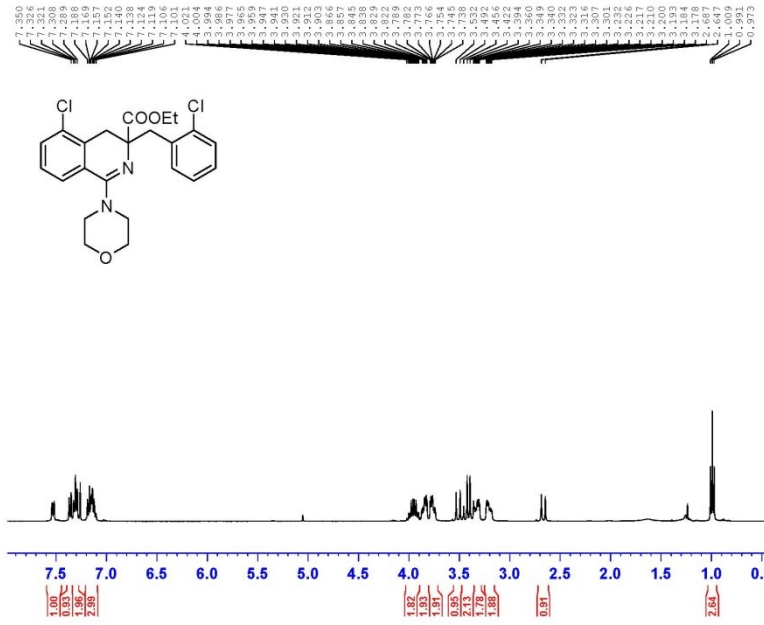
NAME F1003402-0
EXPNO 79
PROCNO 1
Date_ 20160915
Time_ 11.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 144
DW 48.400 usec
DE 6.50 usec
TE 298.9 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 2.50 dB
PL1W 13.02359581 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300229 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

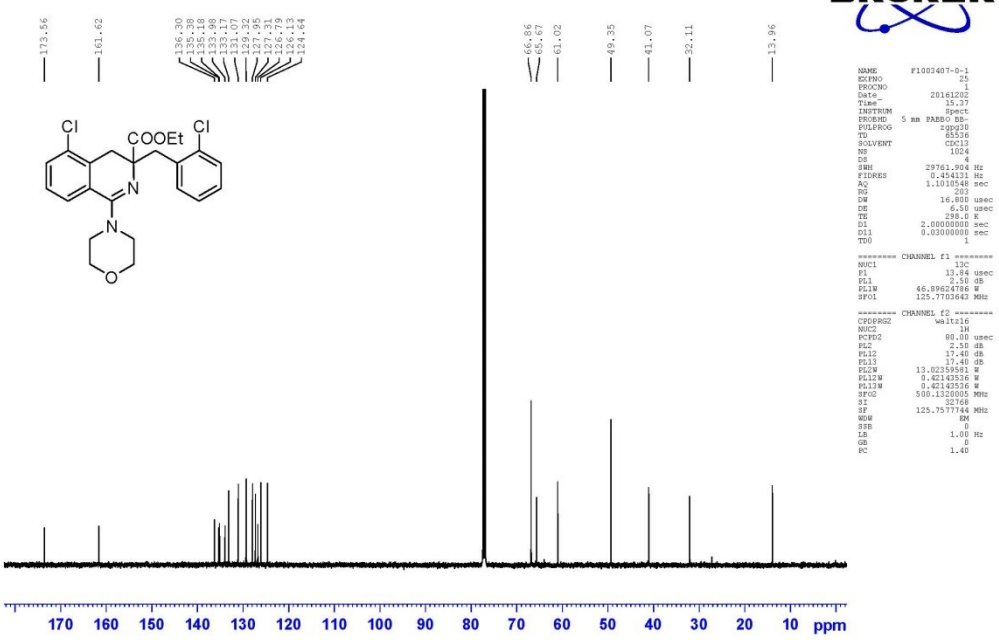

172.41
160.48
147.17
147.06
144.82
144.55
131.66
129.53
125.87
125.07
122.94
121.09
66.55
65.56
61.27
49.13
44.96
36.48
13.94
COOEt
O2N
NO2
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


NAME F1003402-0
EXPNO 67
PROCNO 1
Date_ 20160926
Time 6.05
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 300.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 13.84 usec
PL1 2.50 dB
PL1W 46.89624786 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577855 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

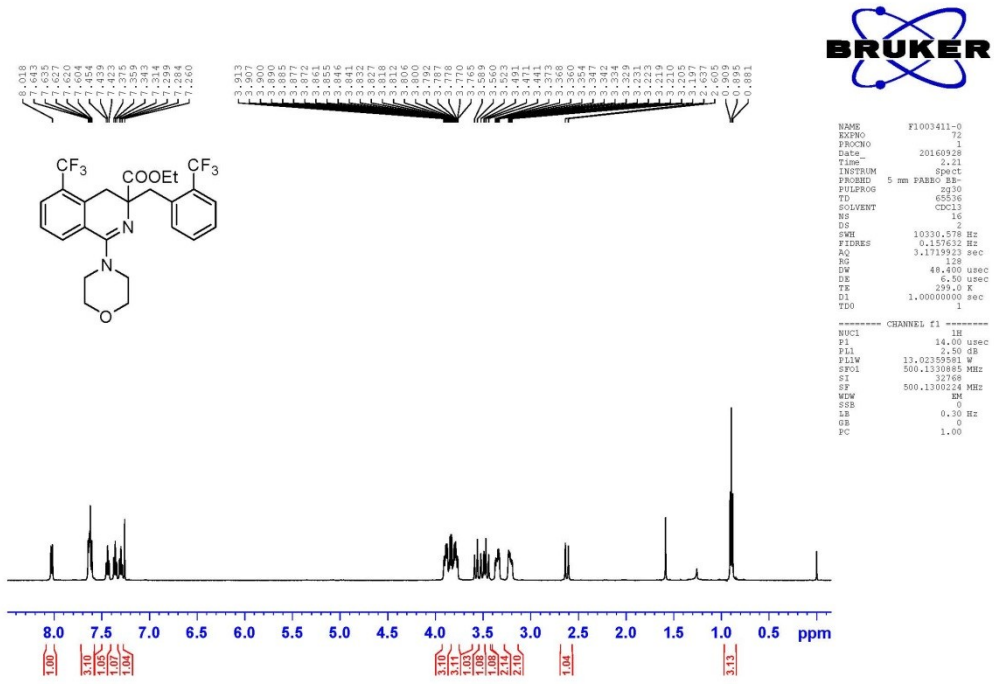
3i

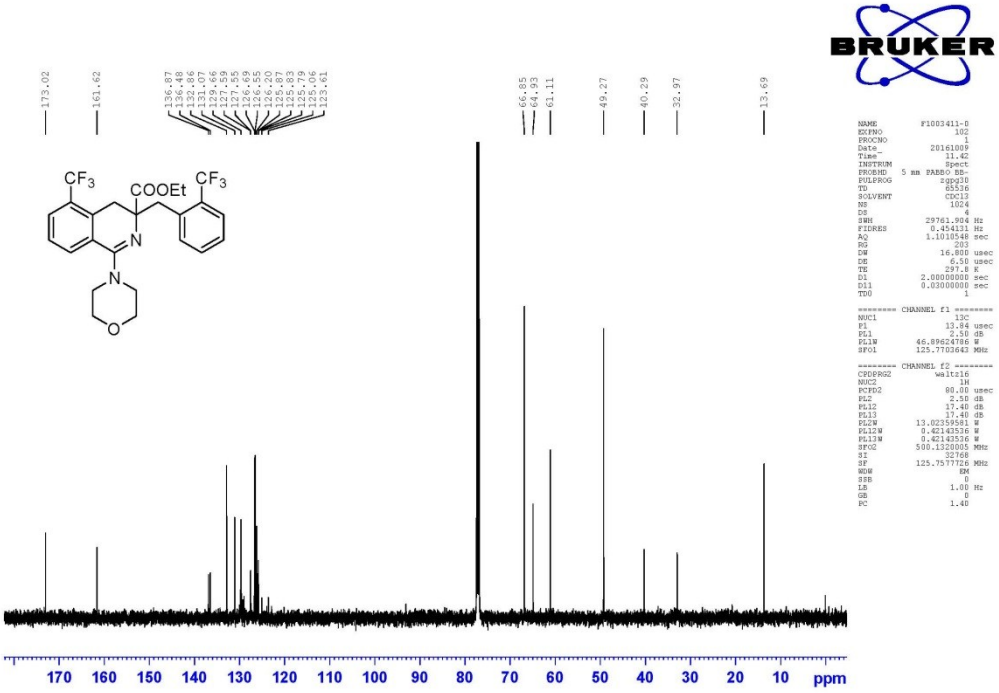

7.350
7.326
7.321
7.308
7.288
7.188
7.169
7.157
7.152
7.140
7.138
7.124
7.119
7.106
7.101
4.021
4.004
3.994
3.977
3.976
3.965
3.959
3.947
3.931
3.930
3.921
3.912
3.903
3.866
3.857
3.845
3.838
3.829
3.822
3.789
3.782
3.773
3.766
3.754
3.745
3.738
3.532
3.502
3.466
3.422
3.394
3.360
3.348
3.340
3.332
3.323
3.316
3.307
3.301
3.232
3.226
3.217
3.210
3.200
3.193
3.184
3.178
3.167
2.647
1.009
0.991
0.973
Cl
COOEt
Cl
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
0.93
1.86
2.89
1.82
1.93
1.91
0.85
2.13
1.78
1.88
0.91
2.84


NAME F1003407-0-1
EXPNO 19
PROCNO 1
Date_ 20161126
Time 23.50
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 128
DW 60.400 usec
DE 6.50 usec
TE 296.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

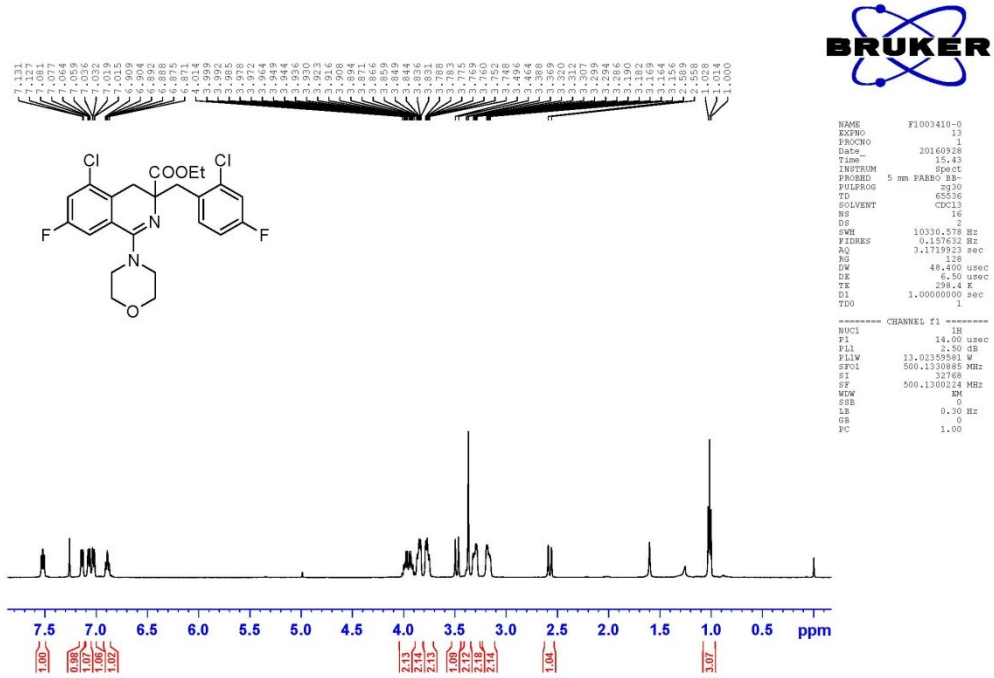

BRUKER
173.56
161.62
136.30
135.38
135.18
133.98
133.17
131.07
129.32
127.95
127.31
126.79
126.13
124.64
66.86
65.67
61.02
49.35
41.07
32.11
13.96
Cl
COOEt
Cl
N
N
O
NAME F1003407-0-1
EXPNO 25
PROCNO 1
Date_ 20161202
Time_ 15.37
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 13.84 usec
PL1 2.50 dB
PL1W 46.89624786 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577744 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

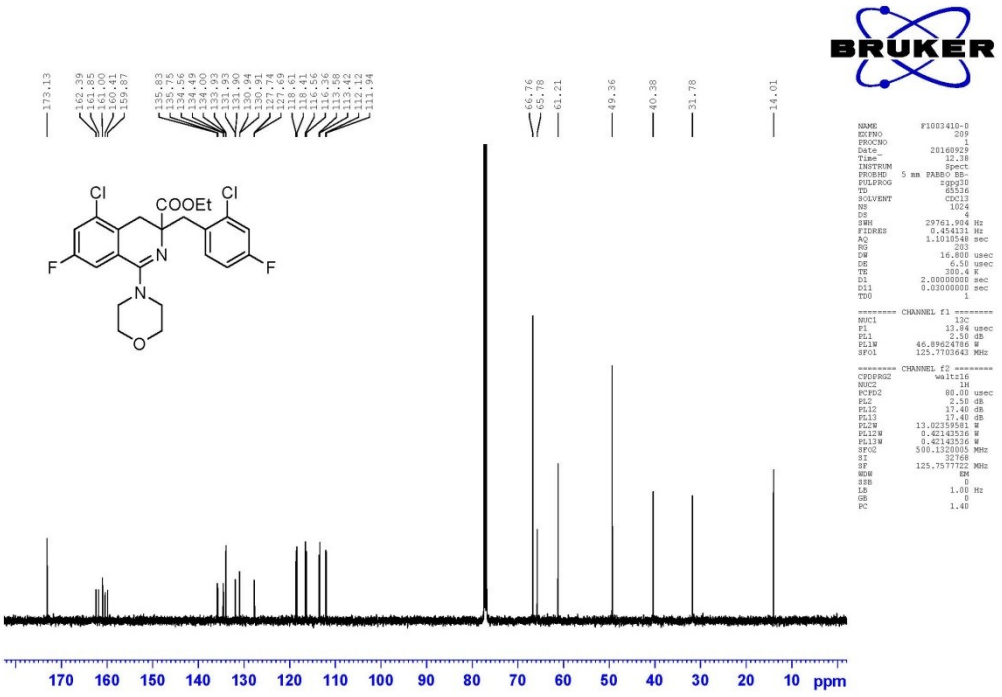
3j


BRUKER
8.018
7.643
7.635
7.627
7.620
7.464
7.454
7.439
7.423
7.375
7.358
7.343
7.314
7.299
7.284
7.260
3.913
3.907
3.900
3.885
3.877
3.872
3.861
3.855
3.846
3.841
3.832
3.827
3.818
3.812
3.806
3.800
3.792
3.787
3.778
3.770
3.765
3.559
3.560
3.523
3.491
3.441
3.373
3.368
3.360
3.354
3.347
3.342
3.334
3.329
3.221
3.219
3.210
3.205
3.197
2.637
2.605
0.909
0.885
0.881
CF3
COOEt
CF3
N
N
O
NAME F1003411-0
EXPNO 72
PROCNO 1
Date_ 20160928
Time_ 2.21
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 128
DW 48.400 usec
DE 6.50 usec
TE 299.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 2.50 dB
PL1W 13.02359581 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300224 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
3.10
1.05
1.07
1.04
3.10
3.11
1.03
1.08
2.14
2.10
1.04
3.13

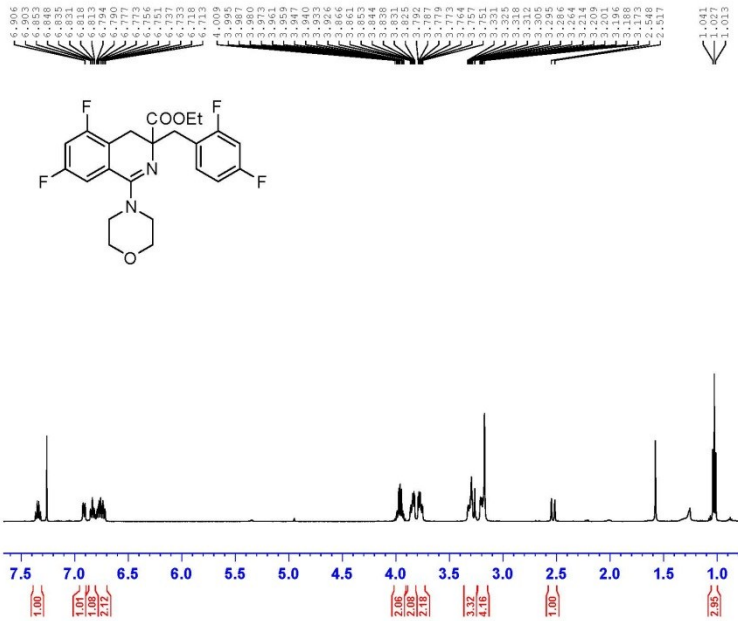

173.02
161.62
136.87
136.48
132.86
131.07
129.56
129.08
127.55
126.69
126.55
126.20
125.87
125.83
125.79
125.06
123.61
66.85
64.93
61.11
49.27
40.29
32.97
13.69
CF3
COOEt
CF3
N
N
O
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

3k

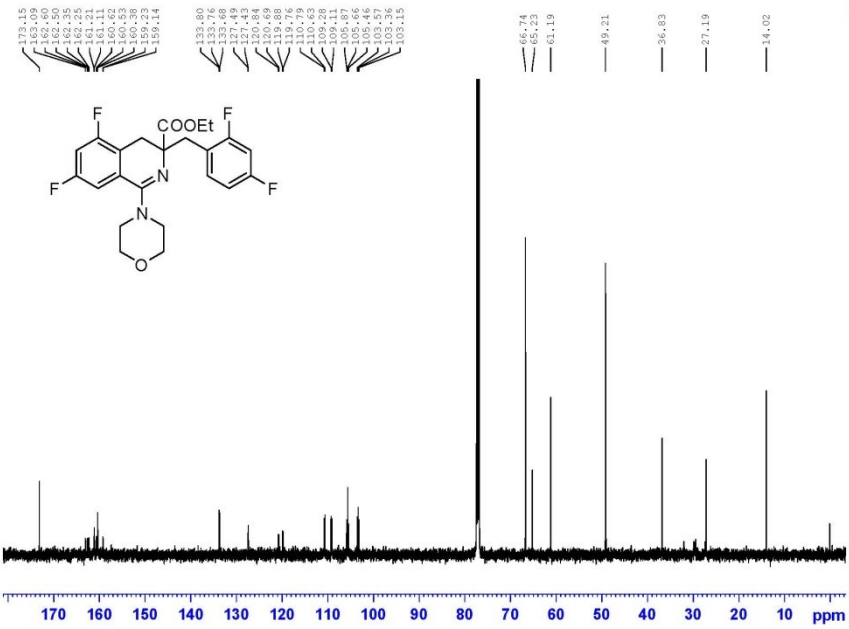

BRUKER
Cl
COOEt
Cl
F
N
F
N
O
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

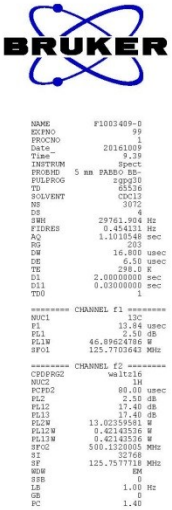

BRUKER
Cl
COOEt
Cl
F
N
F
N
O
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

3l

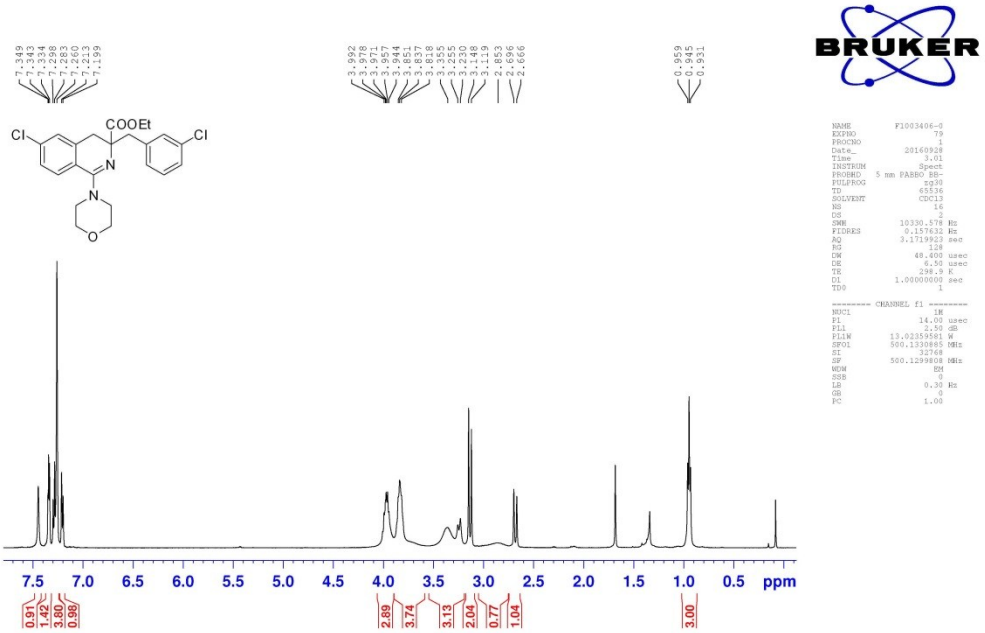

6.906
6.903
6.853
6.848
6.835
6.831
6.818
6.813
6.804
6.790
6.777
6.773
6.756
6.751
6.737
6.733
6.718
6.713
4.009
3.995
3.987
3.980
3.973
3.961
3.949
3.947
3.940
3.933
3.926
3.866
3.861
3.853
3.844
3.838
3.831
3.825
3.792
3.787
3.779
3.773
3.764
3.757
3.751
3.331
3.325
3.318
3.312
3.305
3.295
3.286
3.264
3.214
3.209
3.201
3.196
3.188
3.173
2.548
2.537
1.041
1.027
1.013
F
COOEt
F
F
N
F
N
O
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.00
1.01
1.08
2.12
2.06
2.08
2.05
2.19
3.32
4.16
1.00
2.35

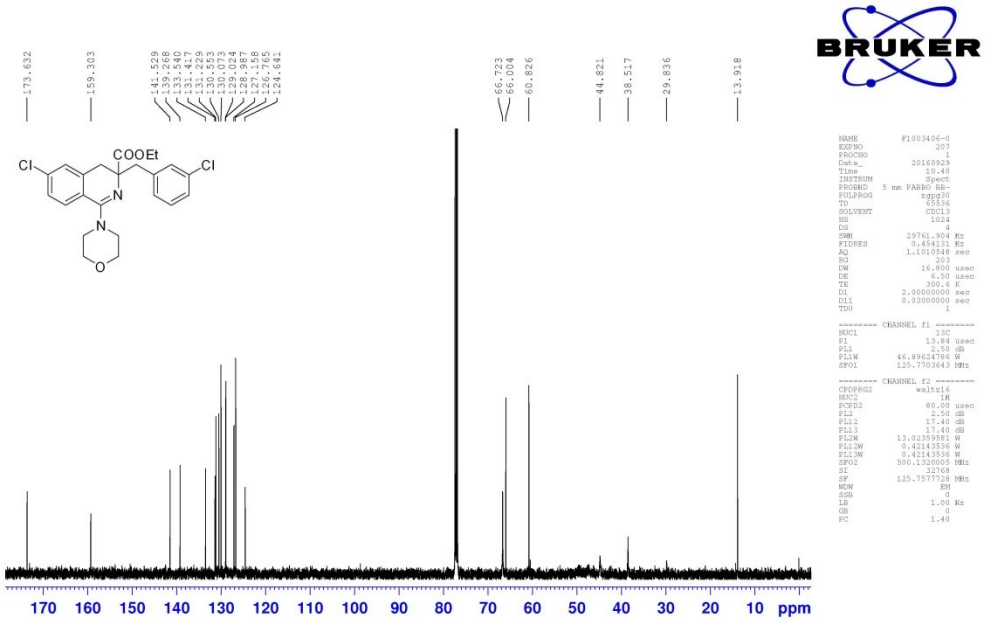

NAME F1003409-0
EXPNO 71
PROCNO 1
Date_ 20160928
Time 2.14
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 203
DW 48.400 usec
DE 6.50 usec
TE 298.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 2.50 dB
PL1W 13.02359581 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300227 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


173.15
163.09
162.60
162.50
162.35
162.25
161.21
161.11
160.62
160.53
160.38
159.23
159.14
133.80
133.76
133.68
127.49
127.43
120.84
120.68
119.88
119.76
110.79
110.63
109.28
109.11
105.87
105.66
105.46
105.37
103.36
103.15
66.74
65.23
61.19
49.21
36.83
27.19
14.02
F
COOEt
F
F
N
F
N
O
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

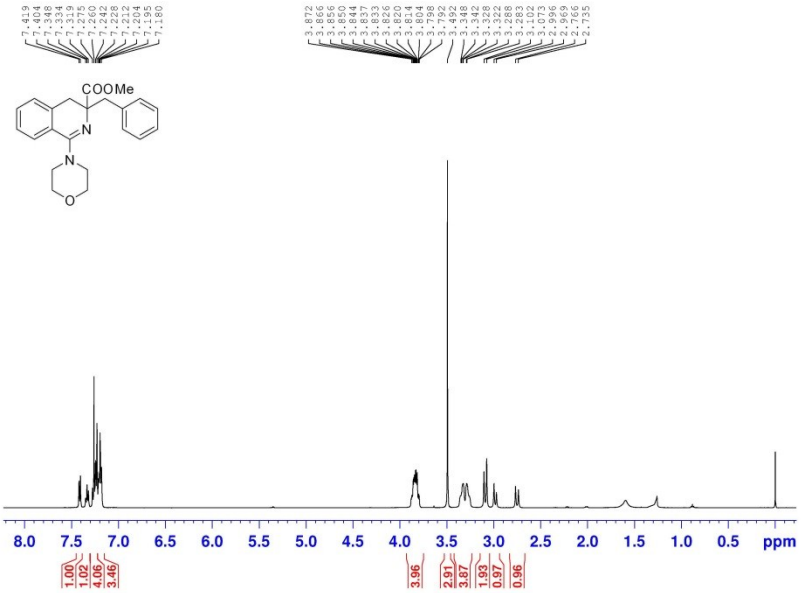

NAME F1003409-0
EXPNO 99
PROCNO 1
Date_ 20161009
Time 9.39
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3072
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 13.84 usec
PL1 2.50 dB
PL1W 46.89624786 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577718 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

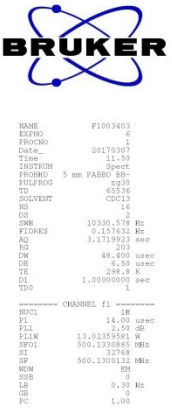
3m

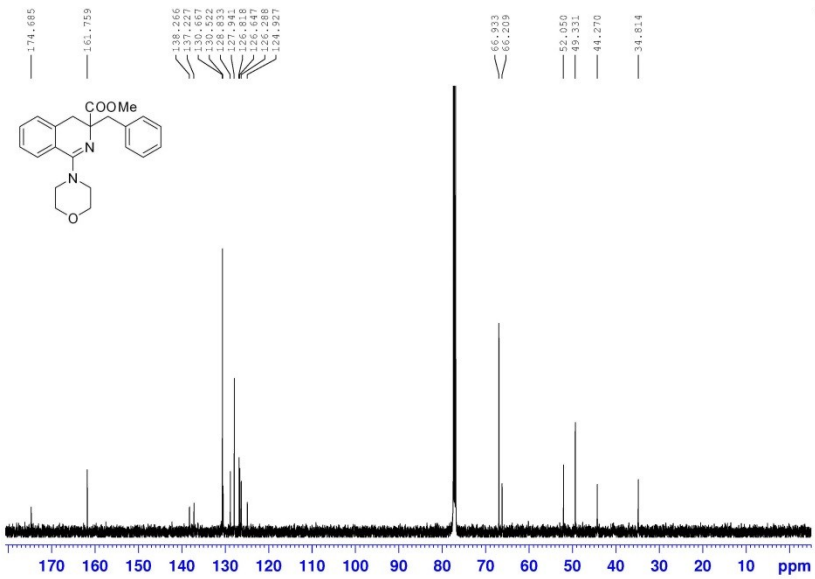

7.349
7.343
7.334
7.286
7.283
7.260
7.213
7.199
3.992
3.978
3.971
3.957
3.944
3.851
3.837
3.818
3.355
3.255
3.230
3.148
3.119
2.853
2.696
2.666
0.959
0.945
0.931
COOEt
Cl
Cl
N
O
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.91
1.42
3.80
0.98
2.89
3.74
3.13
2.04
0.77
1.04
3.00


173.632
159.303
141.529
139.268
133.540
131.417
131.229
130.553
130.073
129.024
128.987
127.158
126.765
124.641
66.723
66.004
60.826
44.821
38.517
29.836
13.918
COOEt
Cl
Cl
N
O
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

3n

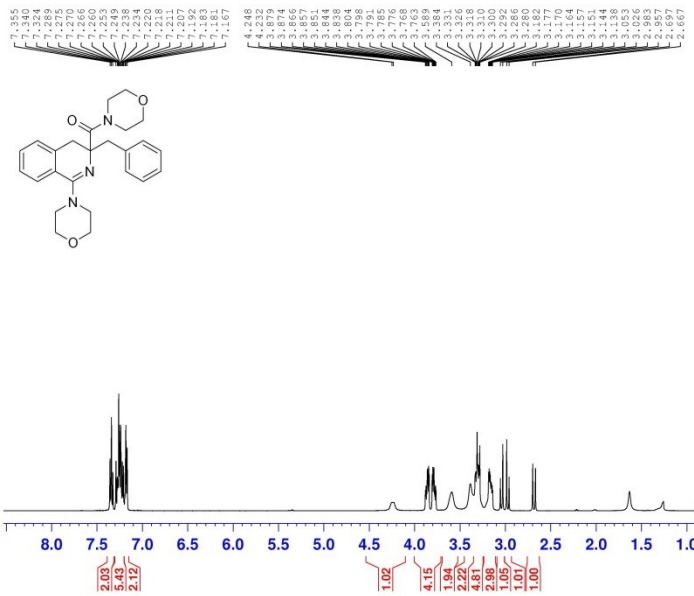

7.419
7.404
7.348
7.334
7.319
7.275
7.260
7.242
7.228
7.224
7.204
7.195
7.180
3.872
3.866
3.856
3.850
3.844
3.837
3.833
3.826
3.820
3.814
3.804
3.798
3.792
3.492
3.348
3.342
3.332
3.322
3.288
3.263
3.222
3.073
2.996
2.969
2.766
2.735
COOMe
N
N
O
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
1.02
4.06
3.46
3.96
2.91
3.87
1.93
0.97
0.96


NAME F1003403
EXPNO 6
PROCNO 1
Date_ 20170307
Time 11.50
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157631 Hz
AQ 3.1719923 sec
RG 203
DW 48.400 usec
DE 6.50 usec
TE 298.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 2.50 dB
PL1W 13.02359581 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300132 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

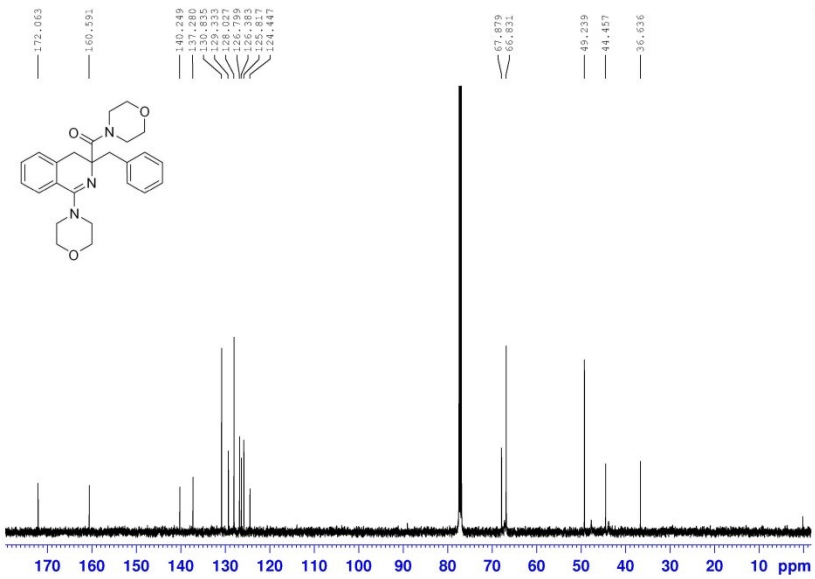

174.685
161.759
138.266
137.227
130.667
129.272
128.833
127.941
126.818
126.647
126.080
124.927
66.933
66.209
52.050
49.331
44.270
34.814
COOMe
N
N
O
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


NAME F1003403
EXPNO 5
PROCNO 1
Date_ 20170307
Time 11.47
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.80 usec
PL1 -1.00 dB
PL1W 104.98761749 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 13.02359581 W
PL12W 0.41565308 W
PL13W 0.41565308 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577698 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

3o

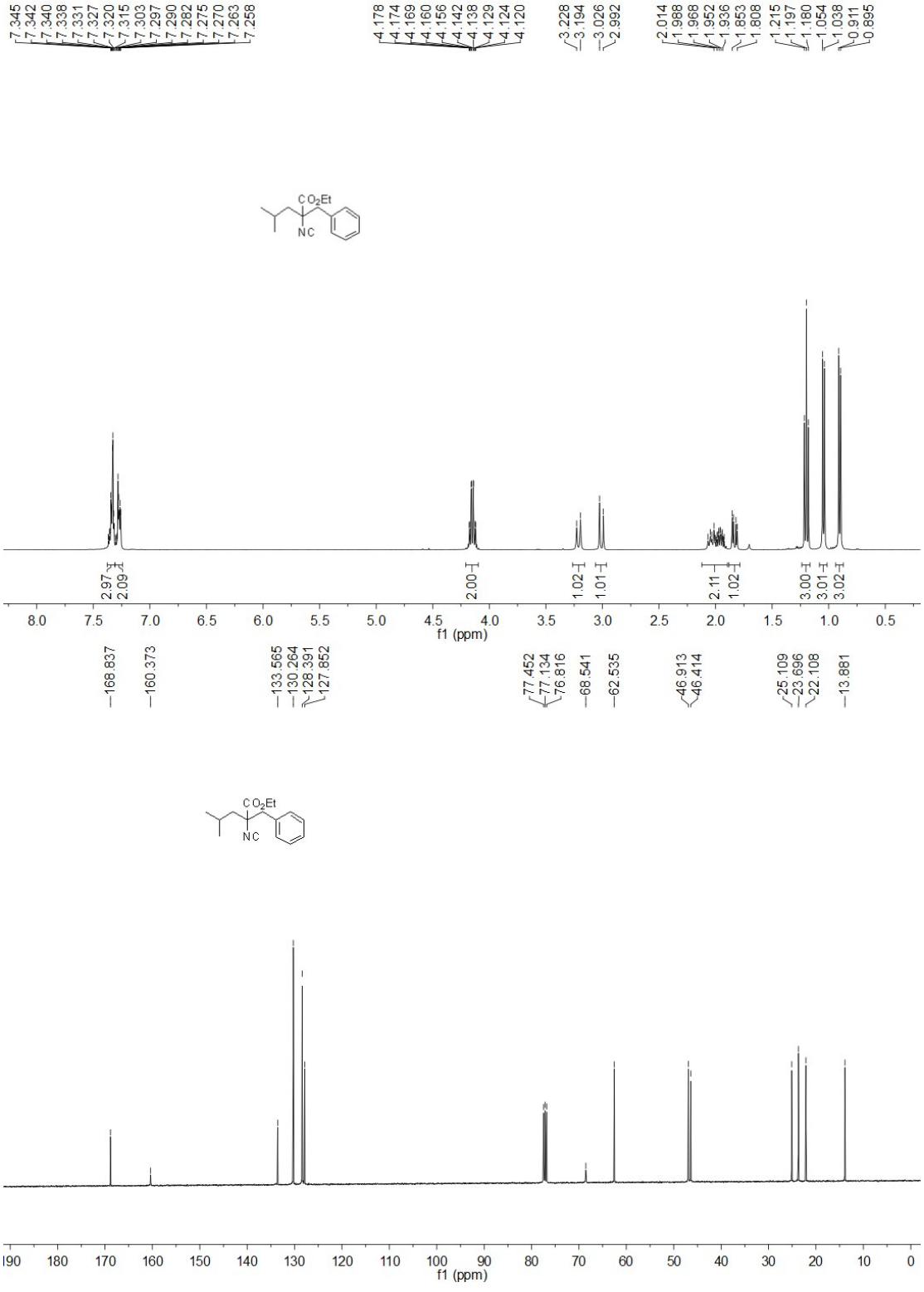



```
NAME                F1003404
EXPNO                      8
PROCNO                     1
Date_               20170307
Time                   12.52
INSTRUM                Spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH               10330.578 Hz
FIDRES             0.157632 Hz
AQ                 3.1719923 sec
RG                       144
DW                  48.400 usec
DE                   6.50 usec
TE                  298.5 K
D1                 1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                   14.00 usec
PL1                   2.50 dB
PL1W           13.02359581 W
SFO1          500.1330885 MHz
SI                    32768
SF            500.1300131 MHz
WDW                      EM
SSB                       0
LB                 0.30 Hz
GB                        0
PC                     1.00
```

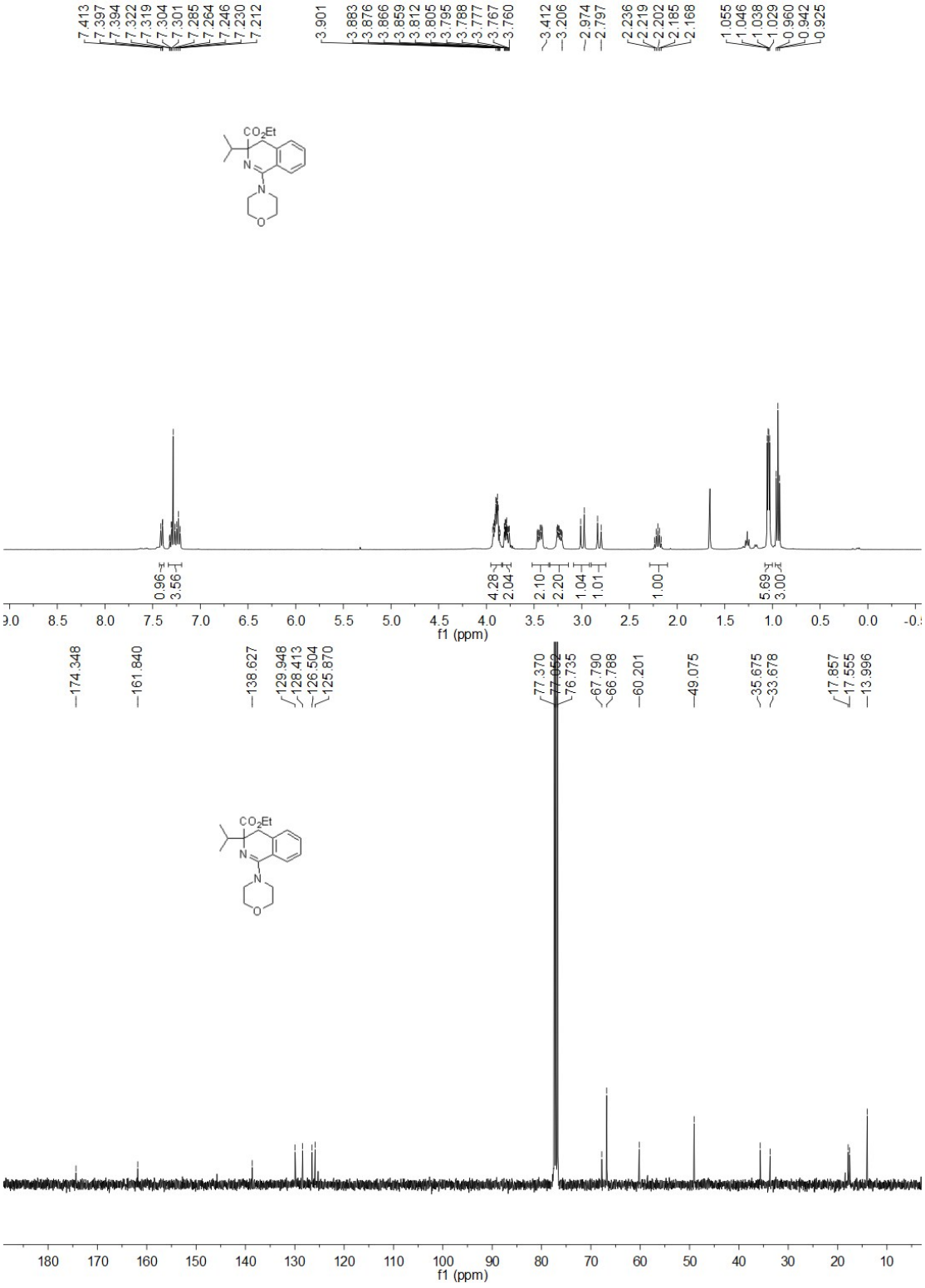



```
NAME                F1003404
EXPNO                      7
PROCNO                     1
Date_               20170307
Time                   12.48
INSTRUM                Spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     1024
DS                        4
SWH               29761.904 Hz
FIDRES             0.454131 Hz
AQ                 1.1010548 sec
RG                       203
DW                  16.800 usec
DE                   6.50 usec
TE                  299.9 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     13C
P1                    8.80 usec
PL1                  -1.00 dB
PL1W          104.98761749 W
SFO1          125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                80.00 usec
PL2                   2.50 dB
PL12                 17.46 dB
PL13                 17.46 dB
PL2W           13.02359581 W
PL12W           0.41565308 W
PL13W           0.41565308 W
SFO2          500.1320005 MHz
SI                    32768
SF            125.7577703 MHz
WDW                      EM
SSB                       0
LB                 1.00 Hz
GB                        0
PC                     1.40
```

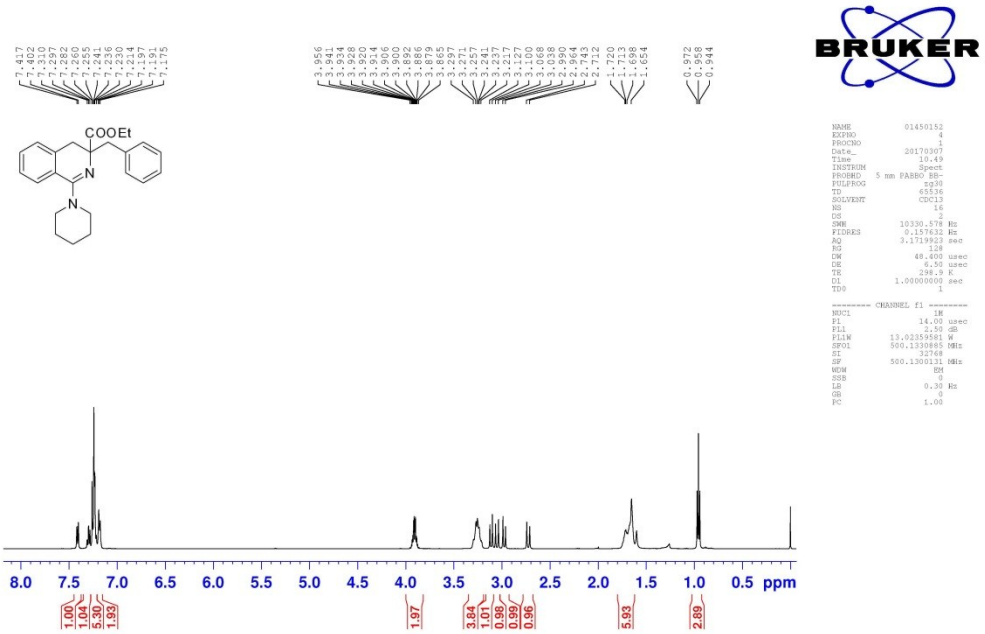
3p

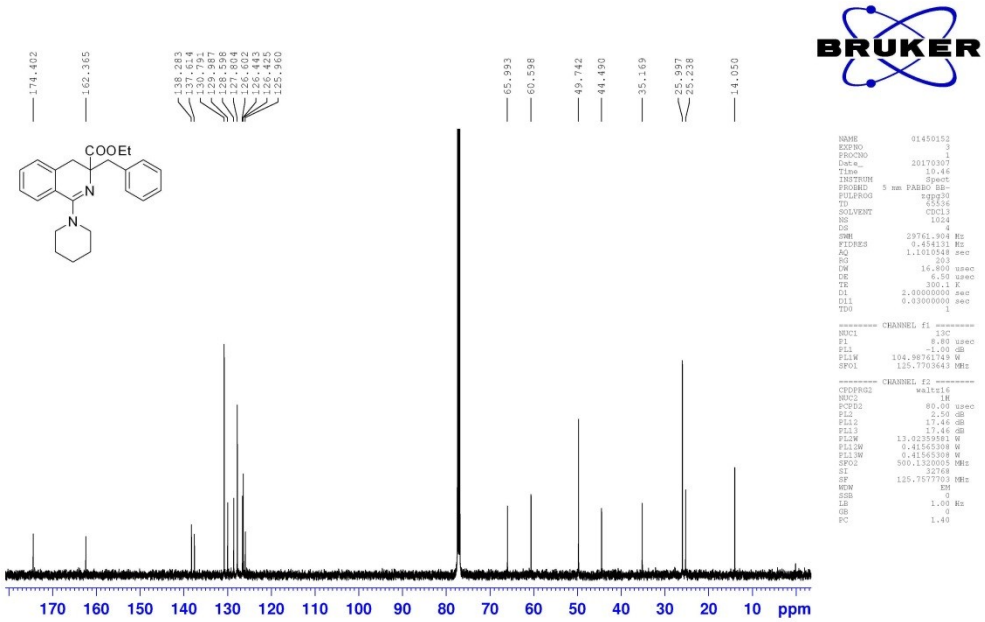

7.345
7.342
7.340
7.338
7.331
7.327
7.320
7.315
7.303
7.297
7.290
7.282
7.275
7.270
7.263
7.258
4.178
4.174
4.169
4.160
4.156
4.142
4.138
4.129
4.124
4.120
3.228
3.194
3.026
2.992
2.014
1.988
1.968
1.952
1.936
1.853
1.808
1.215
1.197
1.180
1.054
1.038
0.911
0.895
CO2Et
NC
2.97
2.09
2.00
1.02
1.01
2.11
1.02
3.00
3.01
3.02
f1 (ppm)
168.837
160.373
133.565
130.264
128.391
127.852
77.452
77.134
76.816
68.541
62.535
46.913
46.414
25.109
23.696
22.108
13.881
CO2Et
NC
f1 (ppm)

3q

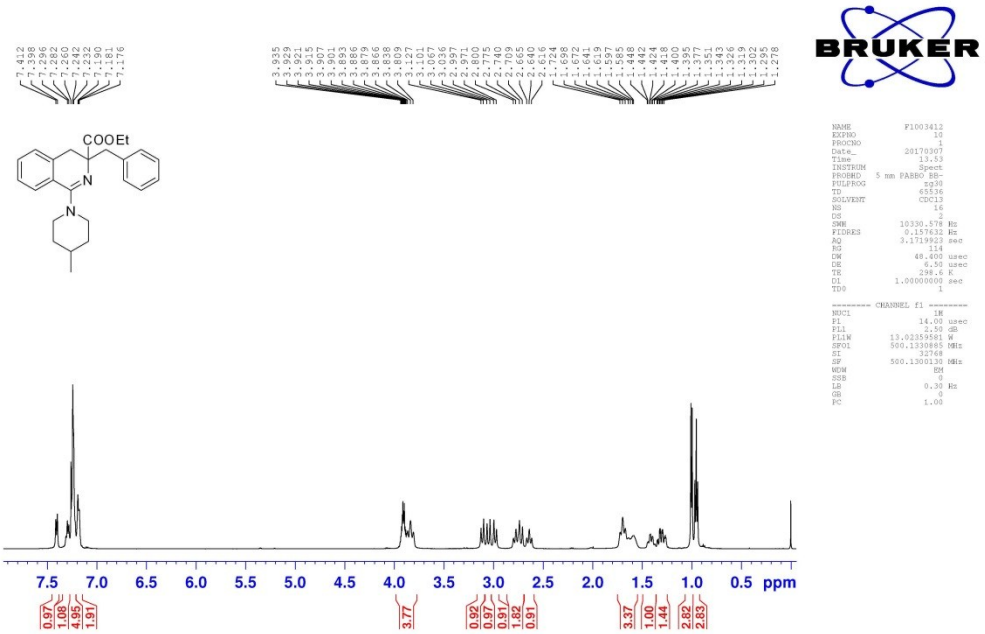

7.413
7.397
7.394
7.322
7.319
7.304
7.301
7.285
7.264
7.246
7.230
7.212
3.901
3.883
3.876
3.866
3.859
3.812
3.805
3.795
3.788
3.777
3.767
3.760
3.412
3.206
2.974
2.797
2.236
2.219
2.202
2.185
2.168
1.055
1.046
1.038
1.029
0.960
0.942
0.925
CO2Et
N
O
0.96
3.56
4.28
2.04
2.10
2.20
1.04
1.01
1.00
5.69
3.00
f1 (ppm)
174.348
161.840
138.627
129.948
128.413
126.504
125.870
77.370
77.052
76.735
67.790
66.788
60.201
49.075
35.675
33.678
17.857
17.555
13.996
f1 (ppm)

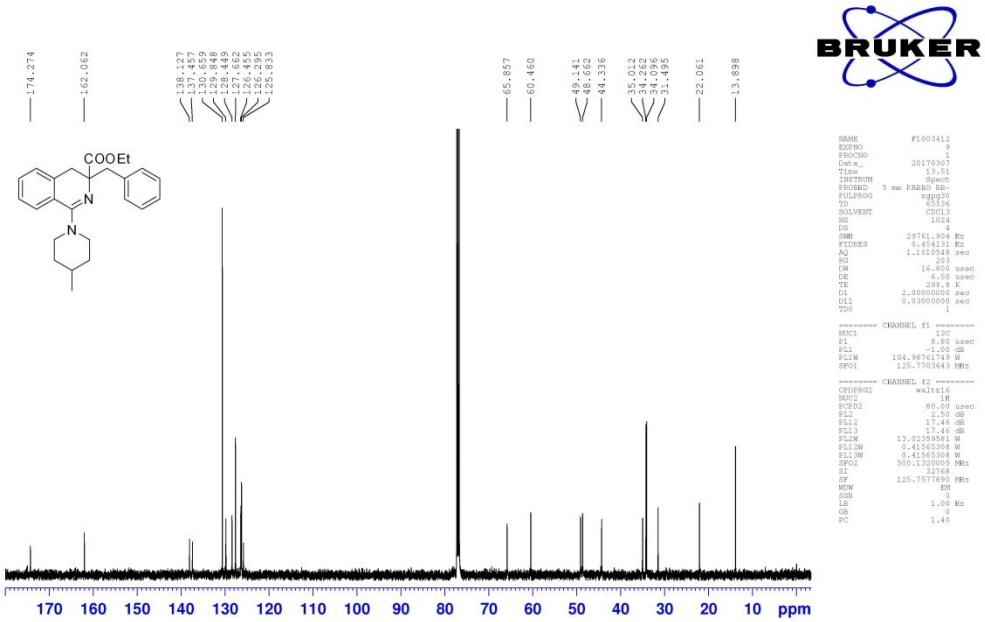
4a


7.417
7.402
7.390
7.367
7.282
7.260
7.255
7.241
7.236
7.230
7.214
7.197
7.191
7.175
3.956
3.941
3.934
3.928
3.920
3.914
3.906
3.900
3.892
3.886
3.879
3.865
3.257
3.247
3.237
3.241
3.237
3.217
3.117
3.110
3.068
3.038
2.990
2.954
2.943
2.712
1.720
1.713
1.698
1.654
0.972
0.968
0.944
COOEt
N
N
BRUKER
NAME 01450152
EXPNO 4
PROCNO 1
Date_ 20170307
Time 10.49
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157631 Hz
AQ 3.1719923 sec
RG 128
DW 48.400 usec
DE 6.50 usec
TE 298.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 2.50 dB
PL1W 13.02359581 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300131 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
1.04
5.49
1.89
1.97
3.84
1.01
0.98
0.99
0.96
5.93
2.89

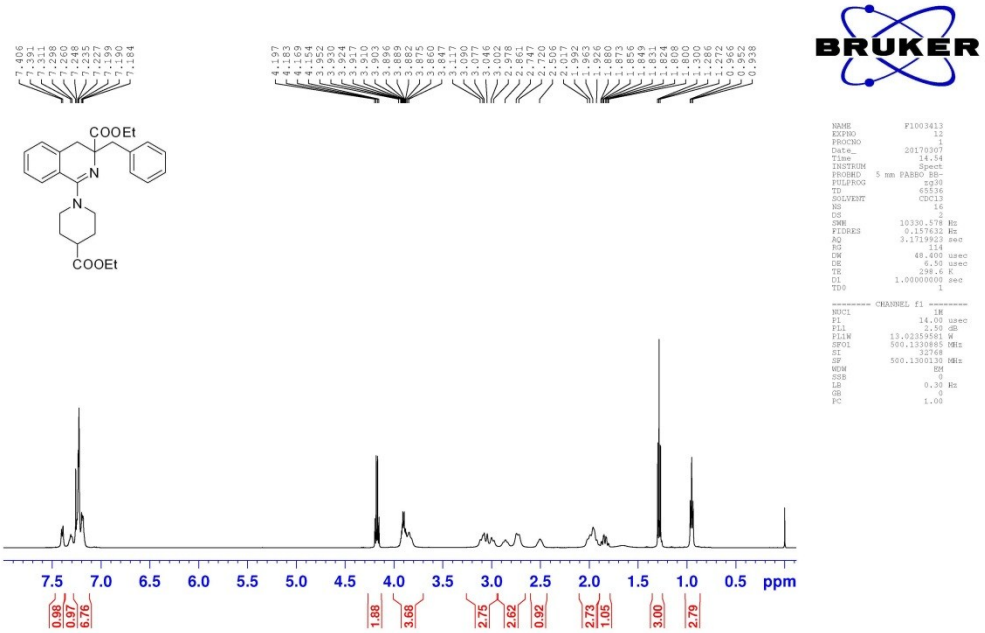

174.402
162.365
138.283
137.614
130.791
129.987
128.598
127.804
126.602
126.443
126.425
125.960
65.993
60.598
49.742
44.490
35.169
25.997
25.238
14.050
COOEt
N
N
BRUKER
NAME 01450152
EXPNO 3
PROCNO 1
Date_ 20170307
Time 10.46
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.80 usec
PL1 -1.00 dB
PL1W 104.98761749 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 13.02359581 W
PL12W 0.41565308 W
PL13W 0.41565308 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577703 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

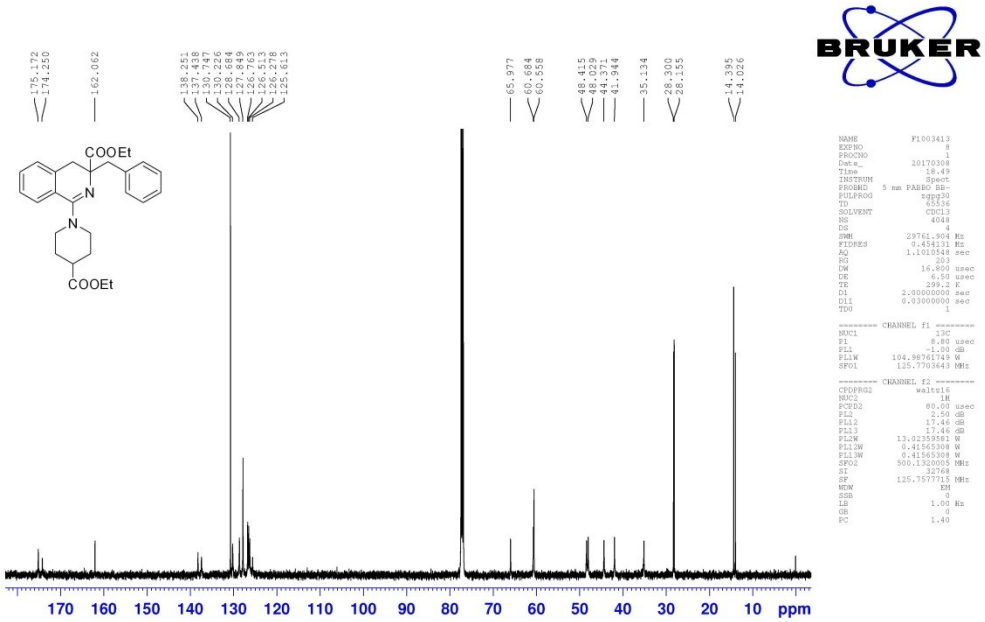
4b


BRUKER
COOEt
N
N
7.412
7.398
7.296
7.282
7.260
7.242
7.232
7.190
7.181
7.176
3.935
3.929
3.921
3.915
3.907
3.901
3.893
3.886
3.879
3.866
3.838
3.809
3.127
3.101
3.083
3.036
2.997
2.971
2.870
2.835
2.740
2.709
2.665
2.640
2.616
1.724
1.698
1.672
1.641
1.619
1.597
1.565
1.448
1.442
1.424
1.418
1.400
1.395
1.377
1.351
1.343
1.326
1.309
1.302
1.295
1.278
NAME F1003412
EXPNO 10
PROCNO 1
Date_ 20170307
Time 13.53
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 114
DW 48.400 usec
DE 6.50 usec
TE 298.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 2.50 dB
PL1W 13.02359581 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300130 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.97
1.08
4.95
1.91
3.77
0.92
0.97
0.91
1.82
0.91
3.37
1.00
1.44
2.02
2.03

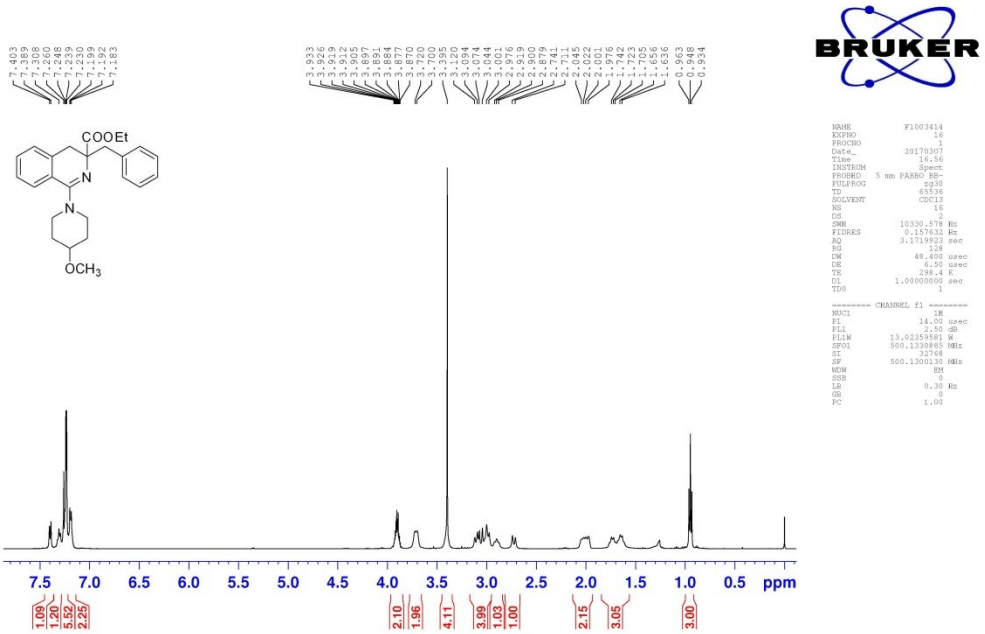

BRUKER
COOEt
N
N
174.274
162.062
138.127
137.457
130.659
129.848
128.449
127.462
126.455
126.295
125.833
65.857
60.460
49.141
48.602
44.336
35.012
34.582
34.096
31.495
22.061
13.898
NAME F1003412
EXPNO 9
PROCNO 1
Date_ 20170307
Time 13.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 298.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.80 usec
PL1 -1.00 dB
PL1W 104.98761749 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 13.02359581 W
PL12W 0.41565308 W
PL13W 0.41565308 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

4c

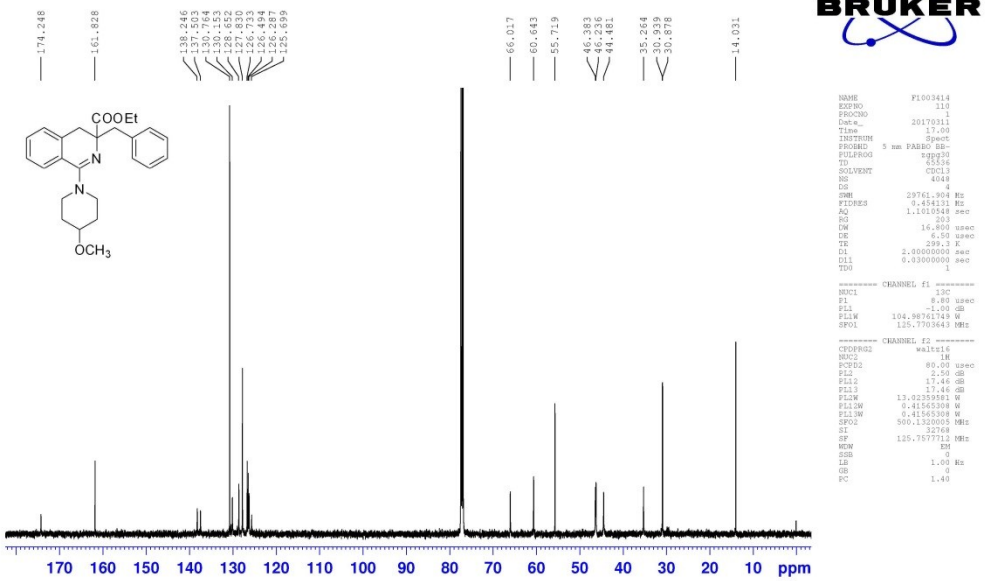

7.406
7.391
7.311
7.268
7.260
7.248
7.235
7.227
7.219
7.190
7.184
4.197
4.183
4.169
4.154
4.142
3.930
3.924
3.917
3.910
3.903
3.896
3.889
3.882
3.875
3.870
3.847
3.117
3.099
3.077
3.046
3.002
2.978
2.861
2.827
2.720
2.506
2.017
1.992
1.953
1.926
1.880
1.873
1.856
1.839
1.831
1.824
1.808
1.800
1.300
1.286
1.272
0.966
0.952
0.938
COOEt
N
N
COOEt
BRUKER
NAME F1003413
EXPNO 12
PROCNO 1
Date_ 20170307
Time 14.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157631 Hz
AQ 3.1719923 sec
RG 114
DW 48.400 usec
DE 6.50 usec
TE 298.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 2.50 dB
PL1W 13.02359581 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300130 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.98
0.97
6.76
1.88
3.68
2.75
2.62
0.92
2.73
1.05
3.00
2.79


175.172
174.250
162.062
138.251
137.418
130.747
130.226
128.684
127.949
126.853
126.513
126.278
125.613
65.977
60.684
60.558
48.415
48.029
44.371
41.944
35.134
28.300
26.155
14.395
14.026
COOEt
N
N
COOEt
BRUKER
NAME F1003413
EXPNO 8
PROCNO 1
Date_ 20170308
Time 18.49
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4048
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 299.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.80 usec
PL1 -1.00 dB
PL1W 104.98761749 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 13.02359581 W
PL12W 0.41565308 W
PL13W 0.41565308 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577715 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

4d

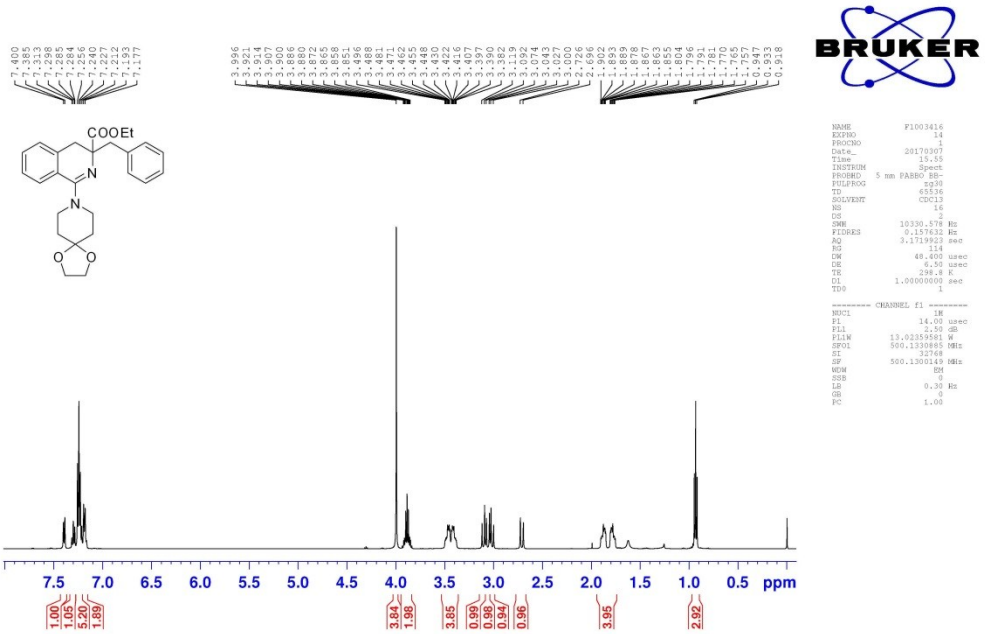

7.403
7.369
7.368
7.260
7.248
7.239
7.230
7.199
7.192
7.183
3.933
3.926
3.919
3.912
3.905
3.897
3.891
3.884
3.877
3.870
3.720
3.700
3.395
3.120
3.054
3.044
3.001
2.976
2.919
2.899
2.879
2.741
2.711
2.045
2.002
2.001
1.976
1.742
1.723
1.715
1.656
1.636
0.963
0.948
0.934
COOEt
N
N
OCH3
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.09
1.20
5.52
2.25
2.10
1.96
4.11
3.99
1.03
1.00
2.15
3.05
3.00

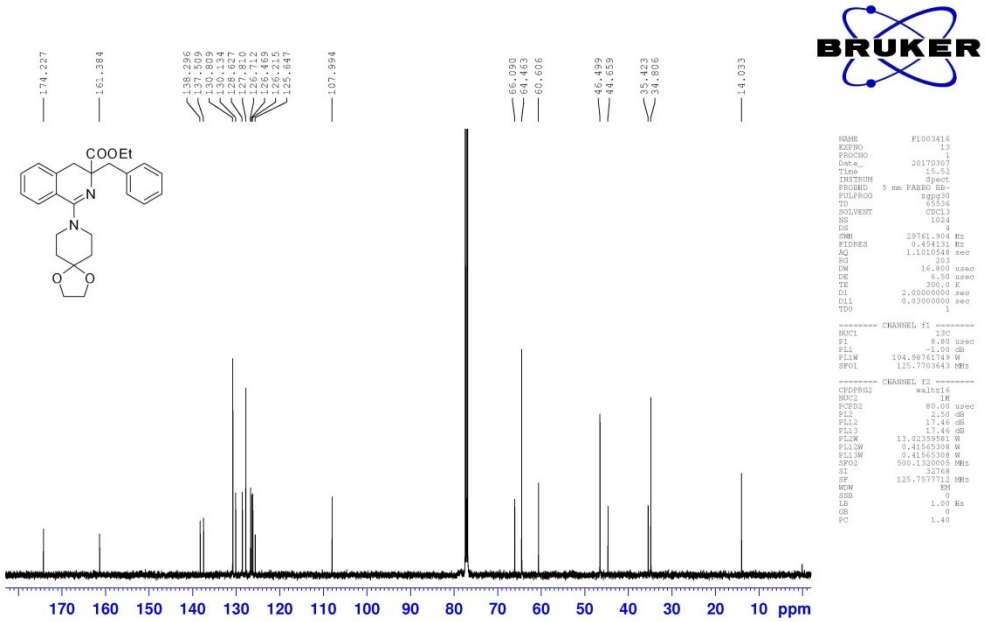
BRUKER
NAME F1003414
EXPNO 16
PROCNO 1
Date_ 20170307
Time 16.56
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 128
DW 48.400 usec
DE 6.50 usec
TE 298.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 2.50 dB
PL1W 13.02359581 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300130 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


174.248
161.828
138.646
137.693
130.764
130.153
128.652
127.930
126.953
126.494
126.287
125.699
66.017
60.643
55.719
46.383
45.236
44.481
35.264
30.899
30.878
14.031
COOEt
N
N
OCH3
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

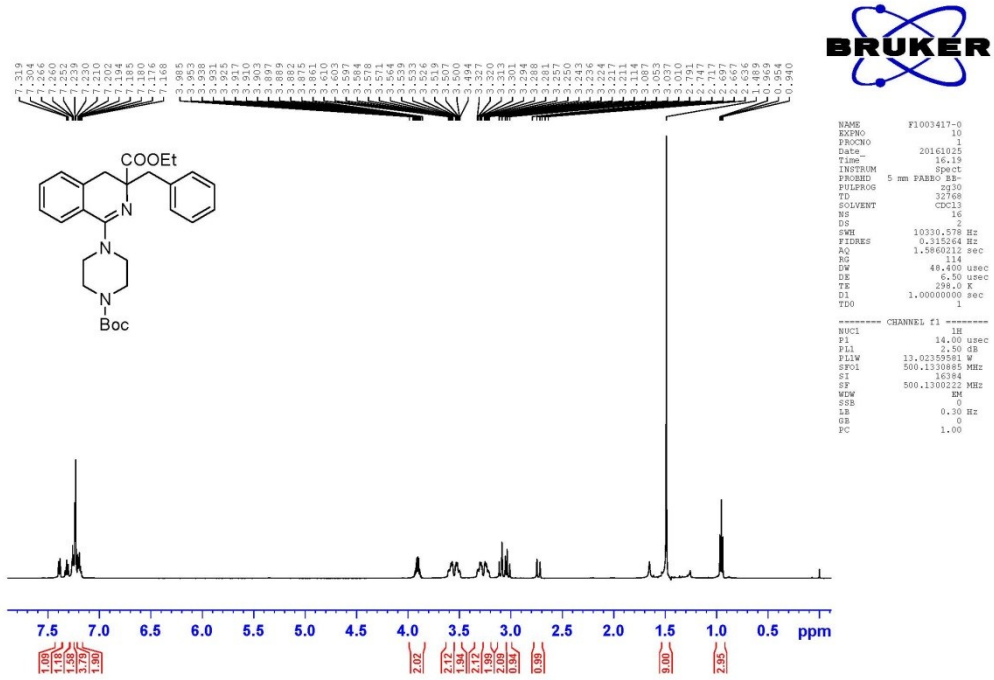

BRUKER
NAME F1003414
EXPNO 110
PROCNO 1
Date_ 20170311
Time 17.00
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4048
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 299.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.80 usec
PL1 -1.00 dB
PL1W 104.98761749 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 13.02359581 W
PL12W 0.41565309 W
PL13W 0.41565309 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577712 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

4e

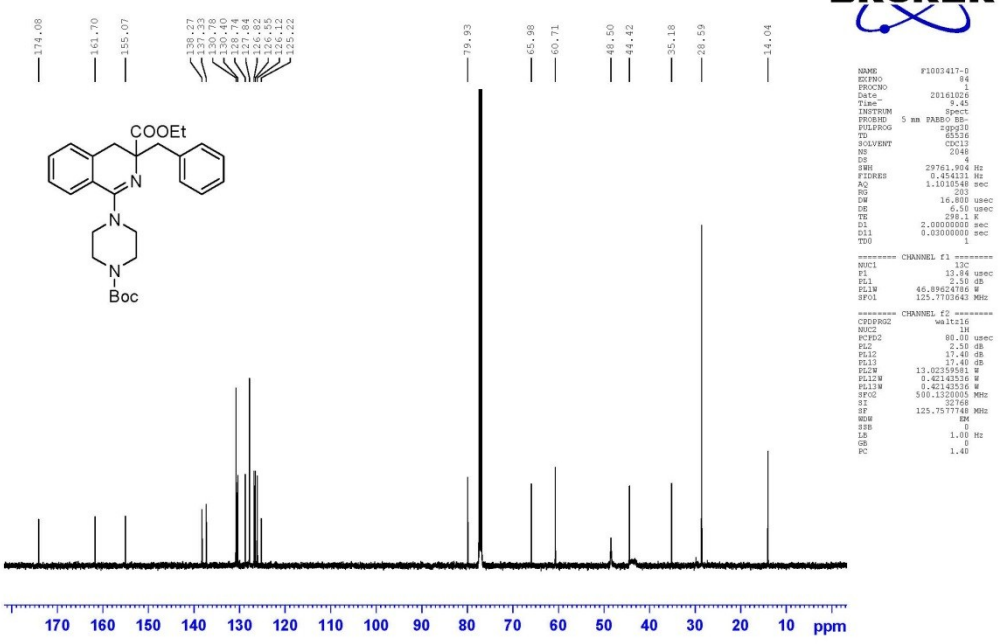

7.400
7.385
7.313
7.298
7.285
7.264
7.246
7.240
7.227
7.212
7.193
7.177
3.996
3.921
3.914
3.907
3.880
3.880
3.872
3.865
3.858
3.851
3.496
3.488
3.481
3.471
3.462
3.455
3.448
3.430
3.422
3.416
3.407
3.397
3.390
3.382
3.119
3.092
3.074
3.057
3.037
3.000
2.726
2.696
2.092
1.893
1.889
1.878
1.867
1.853
1.855
1.804
1.796
1.791
1.781
1.770
1.765
1.757
0.947
0.933
0.918
COOEt
N
N
O
O
BRUKER
NAME F1003416
EXPNO 14
PROCNO 1
Date_ 20170307
Time 15.55
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157631 Hz
AQ 3.1719923 sec
RG 114
DW 48.400 usec
DE 6.50 usec
TE 298.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 2.50 dB
PL1W 13.02359581 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300149 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.00
1.05
5.20
1.69
3.84
1.98
3.85
0.99
0.98
0.94
0.96
3.95
2.92


174.227
161.384
138.296
137.609
130.034
128.627
127.810
126.712
126.569
126.215
125.647
107.994
66.090
64.463
60.606
46.499
44.659
35.433
34.806
14.033
COOEt
N
N
O
O
BRUKER
NAME F1003416
EXPNO 13
PROCNO 1
Date_ 20170307
Time 15.52
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.80 usec
PL1 -1.00 dB
PL1W 104.98761749 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 13.02359581 W
PL12W 0.41565308 W
PL13W 0.41565308 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577712 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

4f

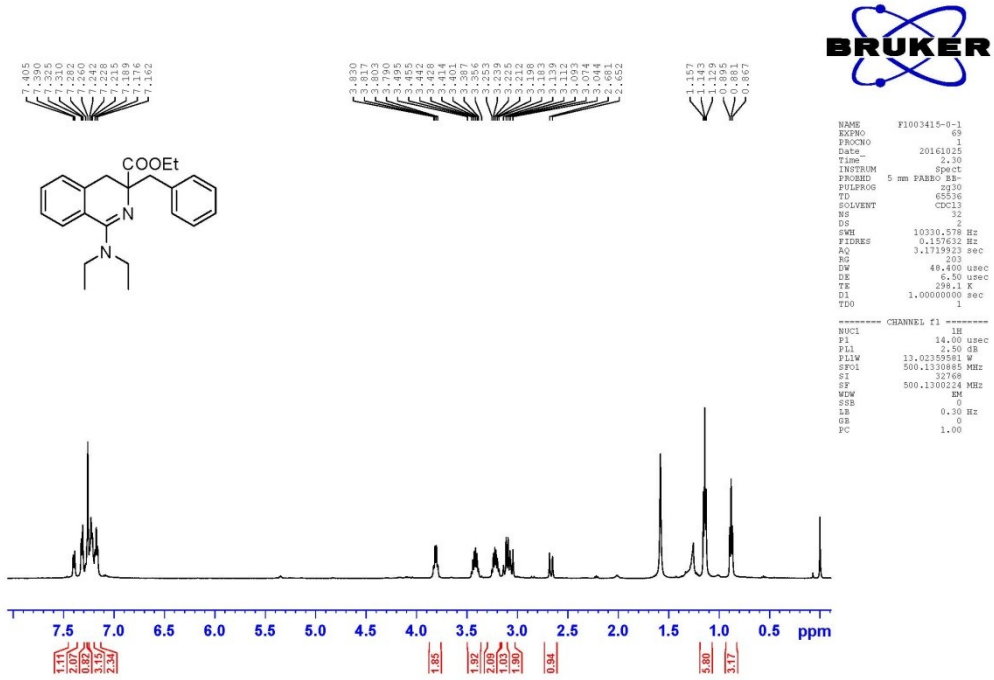


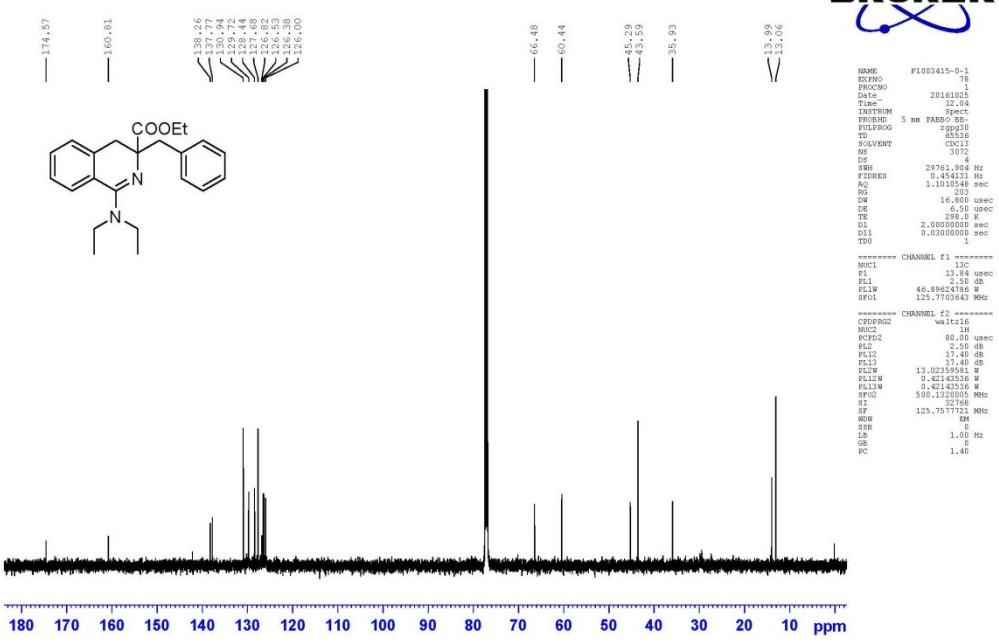

```
NAME                F1003417-0
EXPNO                       10
PROCNO                       1
Date_             20161025
Time_                 16.19
INSTRUM                 Spect
PROBHD     5 mm PABBO BB-
PULPROG                 zg30
TD                      32768
SOLVENT                 CDCl3
NS                         16
DS                          2
SWH                 10330.578 Hz
FIDRES              0.315264 Hz
AQ                  1.5860212 sec
RG                        114
DW                   48.400 usec
DE                    6.50 usec
TE                   298.0 K
D1                  1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                   14.00 usec
PL1                   2.50 dB
PL1W            13.02359581 W
SFO1           500.1330885 MHz
SI                      16384
SF             500.1300222 MHz
WDW                        EM
SSB                         0
LB                   0.30 Hz
GB                          0
PC                       1.00
```



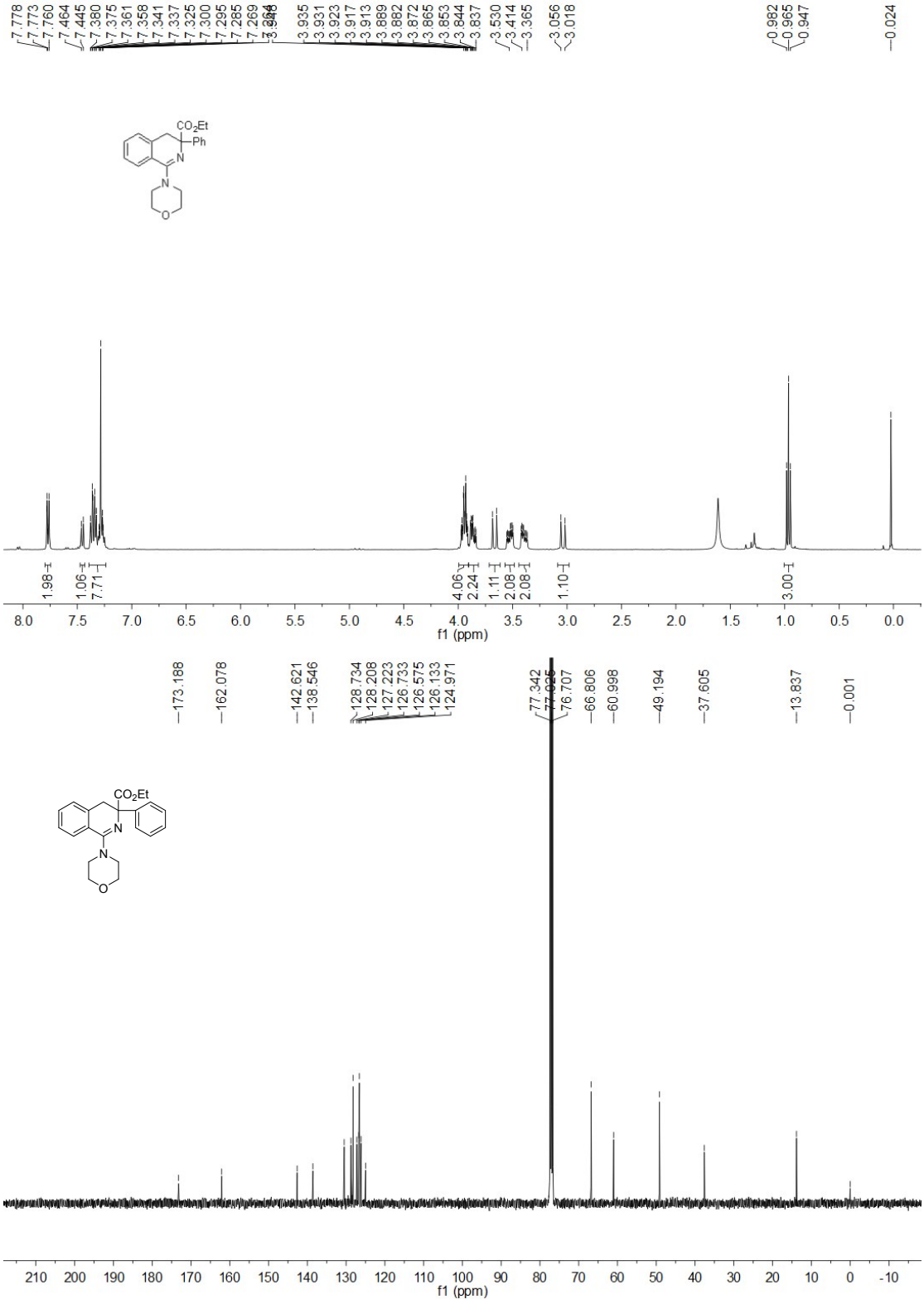

```
NAME                F1003417-0
EXPNO                       94
PROCNO                       1
Date_             20161026
Time_                  9.45
INSTRUM                 Spect
PROBHD     5 mm PABBO BB-
PULPROG                 zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       2048
DS                          4
SWH                 29761.904 Hz
FIDRES              0.454131 Hz
AQ                  1.1010548 sec
RG                        203
DW                   16.800 usec
DE                    6.50 usec
TE                   298.1 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       13C
P1                   13.84 usec
PL1                   2.50 dB
PL1W            46.89624786 W
SFO1           125.7703642 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                       1H
PCPD2                80.00 usec
PL2                   2.50 dB
PL12                 17.40 dB
PL13                 17.40 dB
PL2W            13.02359581 W
PL12W           0.42143536 W
PL13W           0.42143536 W
SFO2           500.1320005 MHz
SI                      32768
SF             125.7577748 MHz
WDW                        EM
SSB                         0
LB                   1.00 Hz
GB                          0
PC                       1.40
```

4g

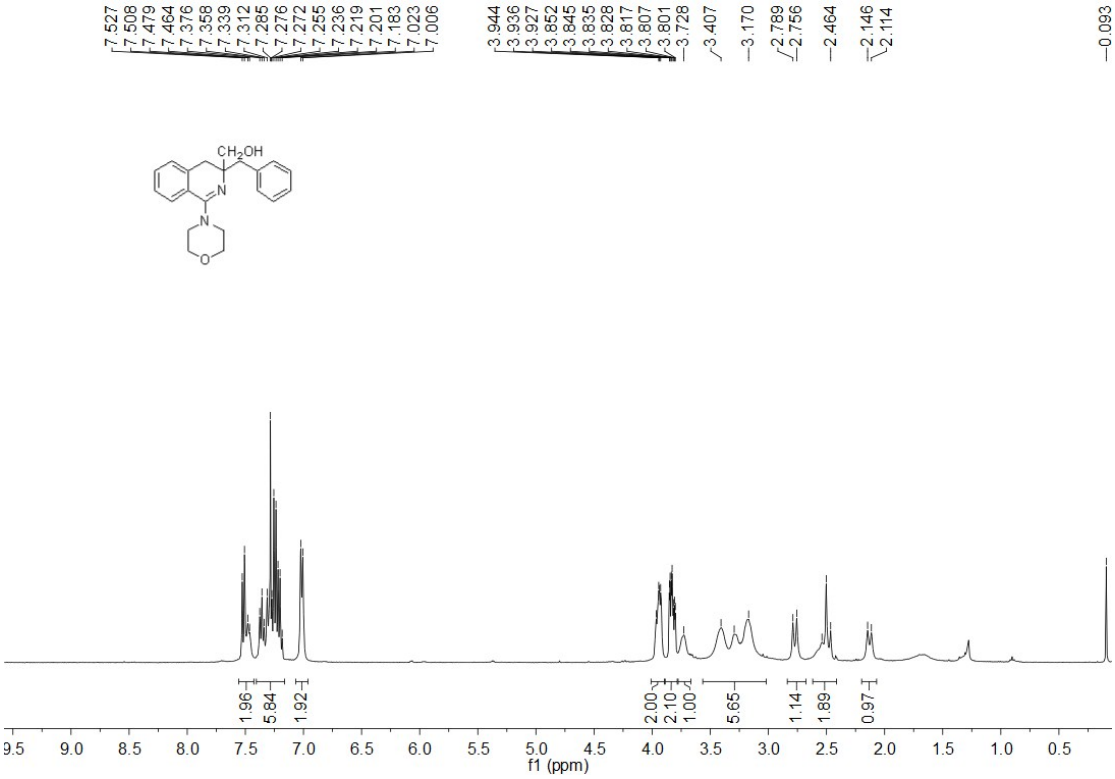

7.405
7.390
7.310
7.282
7.260
7.238
7.215
7.189
7.176
7.162
3.830
3.817
3.803
3.790
3.495
3.455
3.442
3.428
3.414
3.401
3.387
3.356
3.253
3.239
3.225
3.212
3.198
3.183
3.169
3.112
3.093
3.074
3.041
3.027
2.652
1.157
1.143
1.129
0.885
0.871
0.867
COOEt
N
N
NAME F1003415-0-1
EXPNO 69
PROCNO 1
Date_ 20161025
Time_ 2.30
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 203
DW 48.400 usec
DE 6.50 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 2.50 dB
PL1W 13.02359581 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300224 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.11 2.07 0.82 3.15 2.34 1.85 1.92 2.09 1.03 1.90 0.94 5.80 3.17

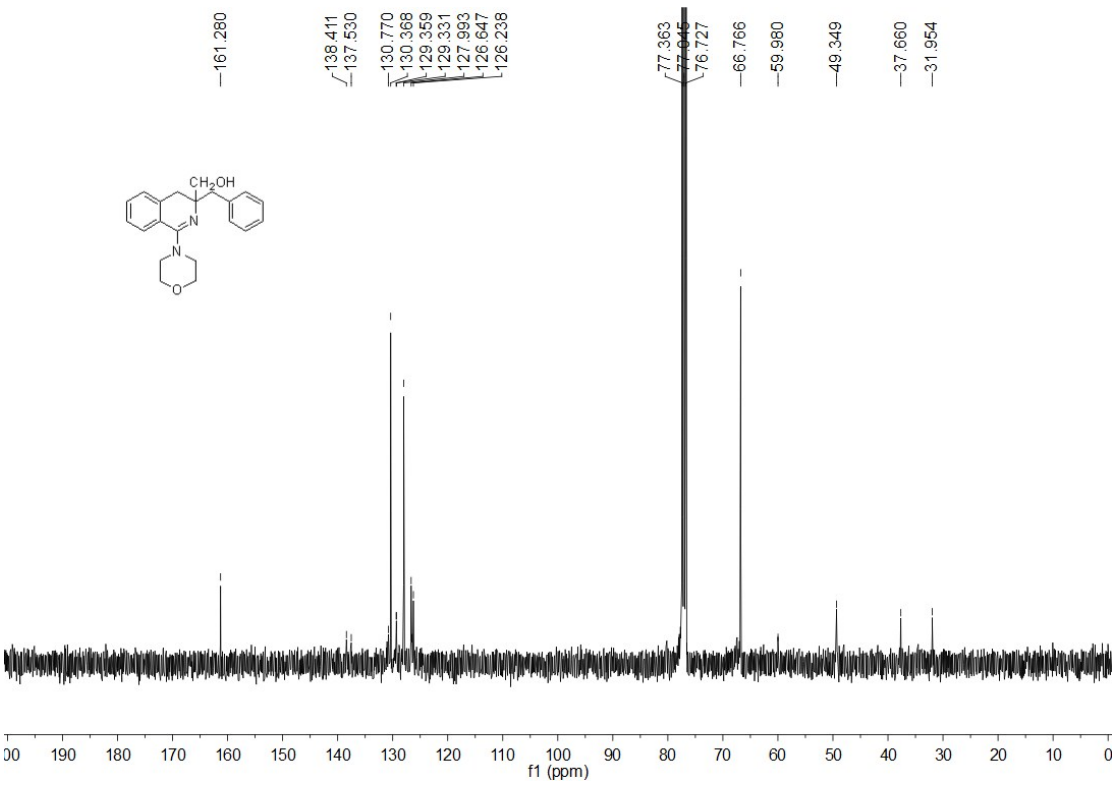

174.57
160.81
138.26
137.77
130.94
129.72
128.44
127.68
126.82
126.53
126.38
126.00
66.48
60.44
45.29
43.59
35.93
13.99
13.06
COOEt
N
N
NAME F1003415-0-1
EXPNO 78
PROCNO 1
Date_ 20161025
Time_ 12.04
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3072
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 13.84 usec
PL1 2.50 dB
PL1W 46.89624786 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577721 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

6

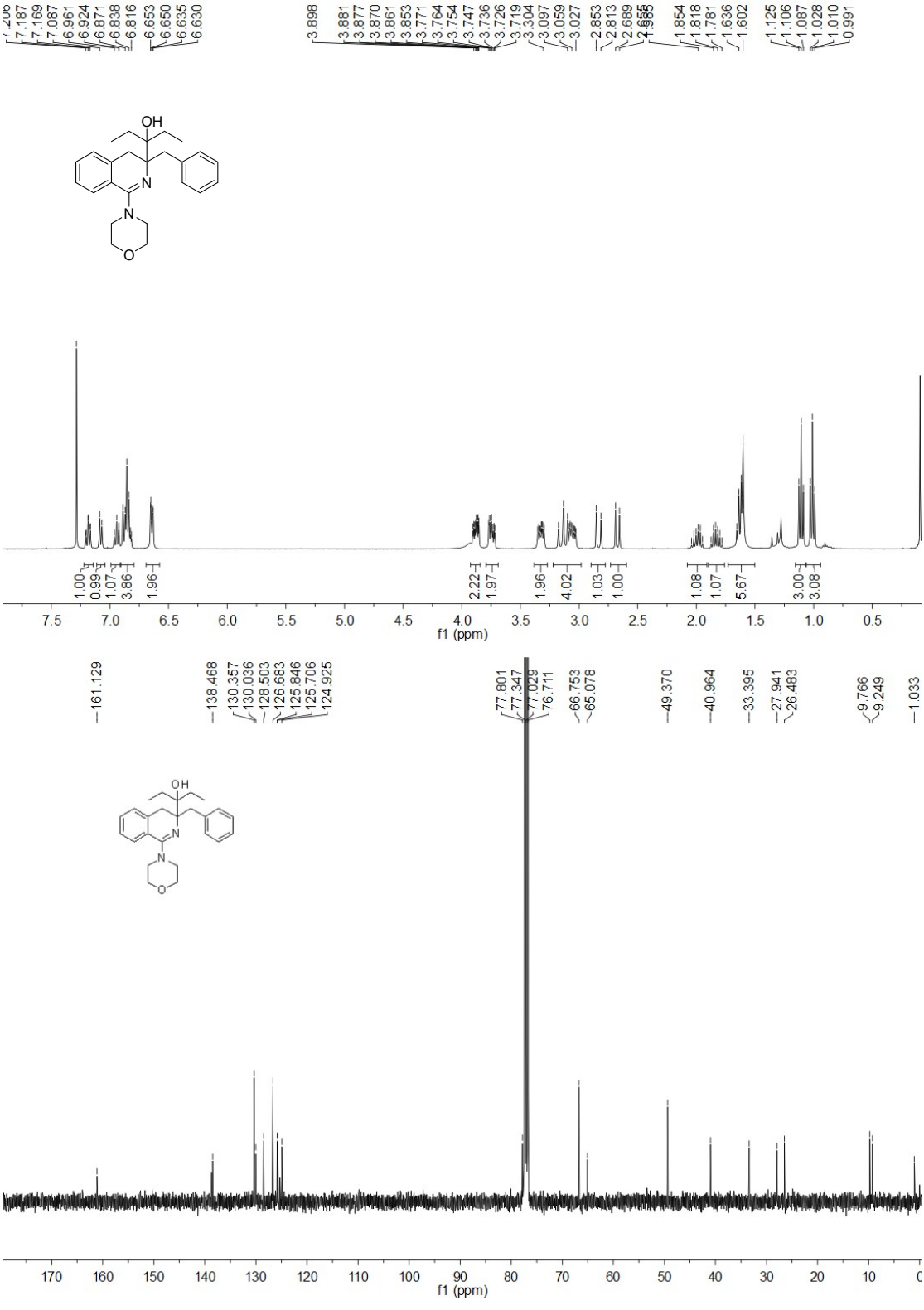

7.778
7.773
7.760
7.464
7.445
7.380
7.375
7.361
7.358
7.341
7.337
7.325
7.300
7.295
7.285
7.269
3.935
3.931
3.923
3.917
3.913
3.889
3.882
3.872
3.865
3.853
3.844
3.837
3.530
3.414
3.365
3.056
3.018
0.982
0.965
0.947
0.024
CO2Et
Ph
N
O
1.98
1.06
7.71
4.06
2.24
1.11
2.08
2.08
1.10
3.00
f1 (ppm)
173.188
162.078
142.621
138.546
128.734
128.208
127.223
126.733
126.575
126.133
124.971
77.342
77.025
76.707
66.806
60.998
49.194
37.605
13.837
0.001
CO2Et
N
O
f1 (ppm)

8


7.527
7.508
7.479
7.464
7.376
7.358
7.339
7.312
7.285
7.276
7.272
7.255
7.236
7.219
7.201
7.183
7.023
7.006
3.944
3.936
3.927
3.852
3.845
3.835
3.828
3.817
3.807
3.801
3.728
3.407
3.170
2.789
2.756
2.464
2.146
2.114
0.093
CH2OH
N
N
O
1.96
5.84
1.92
2.00
2.10
1.00
5.65
1.14
1.89
0.97
f1 (ppm)


161.280
138.411
137.530
130.770
130.368
129.359
129.331
127.993
126.647
126.238
77.363
77.045
76.727
66.766
59.980
49.349
37.660
31.954
CH2OH
N
N
O
f1 (ppm)

9


7.187
7.169
7.087
6.961
6.924
6.871
6.838
6.816
6.653
6.650
6.635
6.630
3.898
3.881
3.877
3.870
3.861
3.853
3.771
3.764
3.754
3.747
3.736
3.726
3.719
3.304
3.097
3.059
3.027
2.853
2.813
2.689
1.854
1.818
1.781
1.636
1.602
1.125
1.106
1.087
1.028
1.010
0.991
OH
N
N
O
1.00
0.99
1.07
3.86
1.96
2.22
1.97
1.96
4.02
1.03
1.00
1.08
1.07
5.67
3.00
3.08
f1 (ppm)
161.129
138.468
130.357
130.036
128.503
126.683
125.846
125.706
124.925
77.801
77.347
77.029
76.711
66.753
65.078
49.370
40.964
33.395
27.941
26.483
9.766
9.249
1.033
f1 (ppm)