Supporting Information

Synthesis of 3-Aryl-2-Aminoquinolines：Palladium-Catalyzed Cascade Reactions of *Gem*-dibromovinylanilines with *tert*-Butyl Isocyanide and Arylboronic Acids

Langxi Hu,[a,‡] Weijun Gui,[a,‡] Zichen Liu,[b] and Baishan Jiang[a,*]

[a]Institute of Chemical Biology, Guangzhou Institutes of Biomedicine and Health, Chinese Academy of Sciences. 190 Kaiyuan Avenue, Guangzhou 510530, China.

Fax: (+86)20-3201-5209; Tel: (+86)20-3201-5209; Email: jiang_baishan@gibh.ac.cn.

[b]Shenzhen University Health Science Center, Shenzhen University.

‡These authors contributed equally.

Table of Contents

1. General Considerations

All reagents were purchased from commercial suppliers and used without further purification. For flash column chromatography, silica gel (200-300 mesh) was applied. Reactions were monitored using thin-layer chromatography (TLC) on commercial silica gel plates (GF 254). Visualization of the developed plates was performed under UV lights (GF 254 nm). ^{1}H and ^{13}C NMR spectra were recorded on a 400 or 500 MHz spectrometer. Chemical shifts (δ) were reported in ppm referenced to an internal tetramethylsilane standard (δ 0.00) or the $CDCl_3$-d1 residual peak (δ 7.26) for ^{1}H NMR. Chemical shifts of ^{13}C NMR were reported relative to $CDCl_3$ (δ 77.0). The following abbreviations were used to describe peak splitting patterns when appropriate: br s = broad singlet, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. Coupling constant, J, was reported in Hertz unit (Hz). High resolution mass spectra (HRMS) were obtained on an ESI-LC-MS/MS spectrometer.

2. Preparation of Substrates 1

2-(2,2-dibromovinyl)aniline **1** and derivatives, they were characterized in our previous work[1].

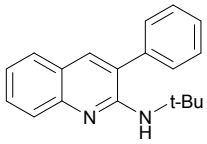
3. General Procedures and Characterization Data

1) Typical procedure for the cascade reaction

A mixture of *gem*-dibromovinylaniline **1** (0.2 mmol), arylboronic acid **2** (0.22 mmol), *t*-butyl isocyanide (0.22 mmol), $Pd(dppf)Cl_2$ (0.01 mmol), and Cs_2CO_3 (0.44 mmol) in 1,4-dioxane (2.0 mL) was stirred at 100°C for 8h in a sealed tube. Upon completion of the reaction, the mixture was concentrated under vacuum and the resulting residue was purified by flash chromatography on silica gel to give the desired product **3**.

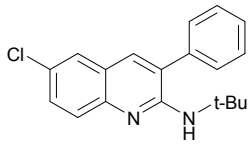
2) Product Characterization

N-(*tert*-butyl)-3-phenylquinolin-2-amine (3a)



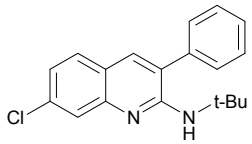
Yellow oil, 43 mg, 78% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (d, J = 8.4 Hz, 1H), 7.52 (s, 1H), 7.39 (m, 7H), 7.11 (t, J = 7.4 Hz, 1H), 4.66 (s, 1H), 1.43 (s, 9H); ^{13}C NMR (101 MHz, DMSO) δ 153.30, 146.55, 137.11, 135.30, 132.99, 132.79, 128.89, 128.65, 128.53, 128.40, 128.35, 128.28, 127.80, 127.12, 125.70, 125.44, 122.46, 121.42, 50.92, 28.51. MS(ESI, m/z): 277.1$[M+H]^+$, HRMS (ESI): Exact mass calcd for $C_{19}H_{21}N_2$ $[M+H]^+$ 277.1699, found 277.1704.

***N*-(tert-butyl)-6-chloro-3-phenylquinolin-2-amine (3b)**



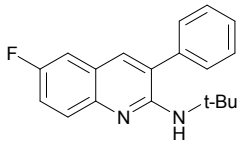
Yellow oil, 54 mg, 87% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 (d, J = 8.8 Hz, 1H), 7.40 (m, 4H), 7.34 (m, 4H), 4.67 (s, 1H), 1.40 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 154.31, 146.18, 137.94, 134.71, 129.51, 129.24, 129.05, 128.29, 128.09, 127.34, 127.01, 125.98, 123.83, 51.92, 29.24. MS(ESI, m/z): 311.1[M+H]$^+$, HRMS (ESI): Exact mass calcd for $C_{19}H_{20}ClN_2$ [M+H]$^+$ 311.1310, found 311.1312.

_N_-(tert-butyl)-7-chloro-3-phenylquinolin-2-amine (3c)



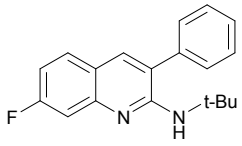
Yellow oil, 52 mg, 83% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (s, 1H), 7.46 (s, 1H), 7.37 (m, 6H), 7.03 (d, J = 8.4 Hz, 1H), 4.71 (s, 1H), 1.40 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 154.75, 148.34, 138.01, 135.30, 134.79, 129.24, 129.06, 128.74, 128.32, 128.23, 127.24, 127.20, 126.57, 125.71, 122.54, 121.57, 51.96, 29.21. MS(ESI, m/z): 311.1[M+H]$^+$, HRMS (ESI): Exact mass calcd for $C_{19}H_{20}ClN_2$ [M+H]$^+$ 311.1310, found 311.1313.

N-(tert-butyl)-6-fluoro-3-phenylquinolin-2-amine (3d)



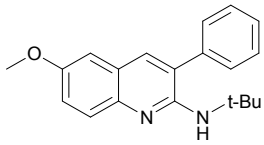
Yellow oil, 50 mg, 85% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 (m, 1H), 7.38 (m, 6H), 7.17 (t, J = 9.8 Hz, 1H), 7.09 (d, J = 8.9 Hz, 1H), 4.60 (s, 1H), 1.40 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.33, 156.94, 153.70, 144.52, 138.05, 135.06, 135.02, 129.20, 129.05, 128.74, 128.42, 128.33, 128.22, 127.32, 127.24, 127.19, 123.23, 123.13, 118.28, 118.04, 110.68, 110.46, 51.80, 29.25. MS(ESI, m/z): 295.2[M+H]$^+$, HRMS (ESI): Exact mass calcd for $C_{19}H_{20}FN_2$ [M+H]$^+$ 295.1605, found 295.1608.

N-(tert-butyl)-7-fluoro-3-phenylquinolin-2-amine (3e)



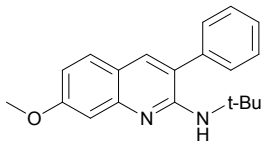
Yellow oil, 51 mg, 86% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (d, J = 7.5 Hz, 1H), 7.46 (s, 1H), 7.36 (dd, J = 16.3, 6.9 Hz, 5H), 7.26 (d, J = 11.1 Hz, 1H), 6.84 (t, J = 8.6 Hz, 1H), 4.70 (s, 1H), 1.40 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.94, 162.49, 154.81, 148.99, 148.86, 141.47, 138.16, 135.41, 129.21, 129.13, 128.93, 128.82, 128.74, 128.13, 127.24, 127.20, 125.58, 120.04, 111.42, 111.18, 110.74, 110.54, 51.92, 29.24. MS(ESI, m/z): 295.2[M+H]$^+$, HRMS (ESI): Exact mass calcd for $C_{19}H_{20}FN_2$ [M+H]$^+$ 295.1605, found 295.1609.

N-(tert-butyl)-6-methoxy-3-phenylquinolin-2-amine (3f)



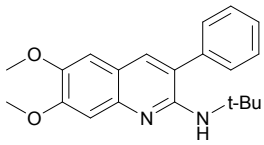
Yellow oil, 48 mg, 79% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (d, *J* = 9.0 Hz, 1H), 7.45 (s, 1H), 7.36 (m, 5H), 7.11 (d, *J* = 9.1 Hz, 1H), 6.85 (s, 1H), 4.50 (s, 1H), 3.77 (s, 3H), 1.40 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 155.06, 153.02, 143.33, 138.66, 135.05, 129.16, 129.09, 128.74, 128.03, 127.95, 127.20, 126.68, 123.45, 120.30, 107.02, 55.69, 51.70, 29.39. MS(ESI, m/z): 307.1[M+H]$^+$, HRMS (ESI): Exact mass calcd for $C_{20}H_{23}N_2O$ [M+H]$^+$ 307.1805, found 307.1810.

N-(tert-butyl)-7-methoxy-3-phenylquinolin-2-amine (3g)



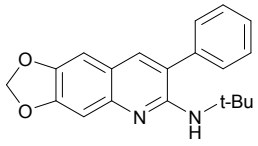
Yellow oil, 50 mg, 81% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (s, 1H), 7.35 (m, 6H), 7.03 (s, 1H), 6.75 (d, *J* = 8.7 Hz, 1H), 4.64 (s, 1H), 3.84 (s, 3H), 1.42 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.03, 154.60, 149.29, 138.70, 135.50, 129.24, 129.10, 128.74, 128.27, 127.79, 127.19, 123.92, 118.00, 113.76, 106.54, 55.38, 51.77, 29.39. MS(ESI, m/z): 307.1[M+H]$^+$, HRMS (ESI): Exact mass calcd for $C_{20}H_{23}N_2O$ [M+H]$^+$ 307.1805, found 307.1811.

N-(tert-butyl)-6,7-dimethoxy-3-phenylquinolin-2-amine (3h)



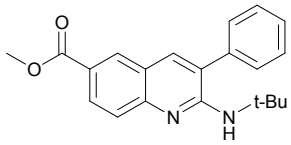
Yellow oil, 53 mg, 79% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (m, 6H), 7.06 (s, 1H), 6.82 (s, 1H), 4.52 (s, 1H), 3.95 (s, 3H), 3.86 (s, 3H), 1.42 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 153.30, 151.85, 146.18, 143.85, 138.59, 134.66, 129.11, 129.03, 127.68, 123.84, 117.02, 106.66, 106.17, 77.25, 77.00, 76.74, 55.97, 55.92, 51.53, 29.30. MS(ESI, m/z): 337.1[M+H]$^+$, HRMS (ESI): Exact mass calcd for $C_{21}H_{25}N_2O_2$ [M+H]$^+$ 337.1911, found 337.1913.

N-(tert-butyl)-7-phenyl-[1,3]dioxolo[4,5-g]quinolin-6-amine (3i)



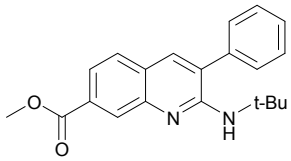
White solid, 54 mg, 85% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 (m, 6H), 7.02 (s, 1H), 6.78 (s, 1H), 5.89 (s, 2H), 4.50 (s, 1H), 1.39 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 153.35, 150.15, 145.27, 144.35, 138.69, 135.15, 129.18, 129.07, 128.73, 127.76, 127.23, 127.18, 123.84, 118.38, 104.52, 103.27, 100.92, 51.66, 29.43. MS(ESI, m/z): 321.1[M+H]$^+$, HRMS (ESI): Exact mass calcd for $C_{20}H_{21}N_2O_2$ [M+H]$^+$ 321.1598, found 321.1602.

methyl 2-(tert-butylamino)-3-phenylquinoline-6-carboxylate (3j)



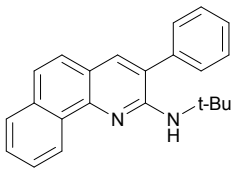
White solid, 55 mg, 82% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.23 (s, 1H), 8.03 (d, *J* = 8.7 Hz, 1H), 7.62 (d, *J* = 8.6 Hz, 1H), 7.55 (s, 1H), 7.39 (m, 5H), 4.86 (s, 1H), 3.85 (s, 3H), 1.42 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.28, 155.54, 150.40, 137.72, 136.45, 130.46, 129.31, 129.12, 129.03, 128.36, 127.14, 126.42, 123.49, 122.24, 52.14, 51.73, 29.17. MS(ESI, m/z): 335.1[M+H]$^+$, HRMS (ESI): Exact mass calcd for $C_{21}H_{23}N_2O_2$ [M+H]$^+$ 335.1754, found 335.1757.

methyl 2-(tert-butylamino)-3-phenylquinoline-7-carboxylate (3k)



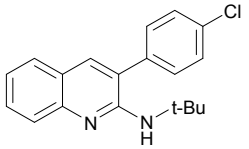
White solid, 52 mg, 78% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.35 (s, 1H), 7.71 (d, *J* = 8.2 Hz, 1H), 7.60 (m, 2H), 7.38 (m, 5H), 4.72 (s, 1H), 3.89 (s, 3H), 1.42 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.54, 154.59, 147.09, 137.83, 135.25, 130.57, 129.25, 128.97, 128.91, 128.72, 128.38, 127.32, 127.17, 126.02, 121.74, 51.98, 51.93, 29.17. MS(ESI, m/z): 335.1[M+H]$^+$, HRMS (ESI): Exact mass calcd for $C_{21}H_{23}N_2O_2$ [M+H]$^+$ 335.1754, found 335.1757.

N-(tert-butyl)-3-phenylbenzo[h]quinolin-2-amine (3l)



White solid, 50 mg, 77% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.07 (d, *J* = 7.9 Hz, 1H), 7.73 (d, *J* = 7.7 Hz, 1H), 7.52 (m, 3H), 7.39 (m, 6H), 7.32 (d, *J* = 6.0 Hz, 1H), 4.77 (s, 1H), 1.52 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 154.02, 145.33, 138.55, 136.29, 134.42, 131.26, 129.21, 129.17, 128.73, 127.95, 127.61, 127.18, 127.12, 125.81, 125.44, 125.12, 124.91, 122.53, 119.48, 51.80, 29.26. MS(ESI, m/z): 327.2[M+H]$^+$, HRMS (ESI): Exact mass calcd for $C_{23}H_{23}N_2$ [M+H]$^+$ 327.1856, found 327.1857.

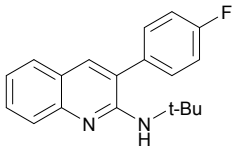
N-(tert-butyl)-3-(4-chlorophenyl)quinolin-2-amine (3m)



Yellow oil, 51 mg, 83% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.64 (d, *J* = 8.3 Hz, 1H), 7.46 (m, 3H), 7.37 (d, *J* = 8.4 Hz, 2H), 7.31 (d, *J* = 8.4 Hz, 2H), 7.10 (t, *J* = 7.4 Hz, 1H), 4.50 (s, 1H), 1.42 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 153.81, 147.81, 136.89, 135.99, 134.28, 130.55, 129.44, 129.22, 127.27, 126.70, 125.10, 123.08, 122.08, 51.95, 29.32. MS(ESI, m/z): 311.1[M+H]$^+$,

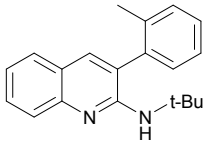
HRMS (ESI): Exact mass calcd for $C_{19}H_{20}ClN_2$ $[M+H]^+$ 311.1310, found 311.1314.

N-(tert-butyl)-3-(4-fluorophenyl)quinolin-2-amine (3n)



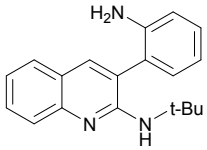
Yellow oil, 54 mg, 92% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (d, *J* = 8.2 Hz, 1H), 7.47 (m, 3H), 7.34(m, 2H), 7.08 (m, 3H), 4.50 (s, 1H), 1.42 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.94, 161.48, 154.06, 147.75, 135.95, 134.30, 130.99, 130.91, 129.10, 127.23, 126.68, 125.31, 123.11, 122.01, 116.27, 116.06, 51.87, 29.30. MS(ESI, m/z): 295.1$[M+H]^+$, HRMS (ESI): Exact mass calcd for $C_{19}H_{20}FN_2$ $[M+H]^+$ 295.1605, found 295.1604.

N-(tert-butyl)-3-(o-tolyl)quinolin-2-amine (3o)



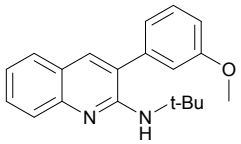
Yellow oil, 50 mg, 87% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (d, *J* = 8.3 Hz, 1H), 7.44 (m, 3H), 7.23 (m, 3H), 7.12(m, 2H), 4.16 (s, 1H), 2.09 (s, 3H), 1.38 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 154.35, 147.88, 137.22, 137.01, 135.47, 130.58, 130.30, 128.83, 128.45, 127.20, 126.72, 126.57, 126.15, 123.07, 121.78, 51.60, 29.24, 19.52. MS(ESI, m/z): 291.2$[M+H]^+$, HRMS (ESI): Exact mass calcd for $C_{20}H_{23}N_2$ $[M+H]^+$ 291.1856, found 291.1855.

3-(2-aminophenyl)-N-(tert-butyl)quinolin-2-amine (3p)



Yellow oil, 48 mg, 83% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.64 (d, *J* = 6.2 Hz, 1H), 7.50 (s, 1H), 7.45 (d, *J* = 7.9 Hz, 1H), 7.41 (t, *J* = 7.7 Hz, 1H), 7.16 (t, *J* = 7.7 Hz, 1H), 7.09 (t, *J* = 7.4 Hz, 1H), 6.74 (d, *J* = 7.5 Hz, 1H), 6.63 (d, *J* = 8.4 Hz, 2H), 4.78 (s, 1H), 3.69 (s, 2H), 1.43 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 154.21, 147.60, 147.31, 139.49, 135.40, 130.01, 128.85, 127.22, 126.71, 126.55, 123.19, 121.79, 119.09, 115.74, 114.71, 51.78, 29.34. MS(ESI, m/z): 292.2$[M+H]^+$, HRMS (ESI): Exact mass calcd for $C_{19}H_{22}N_3$ $[M+H]^+$ 292.1808, found 292.1809.

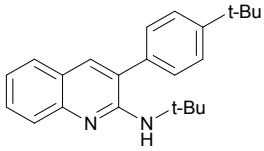
N-(tert-butyl)-3-(3-methoxyphenyl)quinolin-2-amine (3q)



Yellow oil, 53 mg, 86% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.64 (d, *J* = 8.3 Hz, 1H), 7.52 (s, 1H), 7.47 (d, *J* = 7.9 Hz, 1H), 7.42 (m, 1H), 7.31 (t, *J* = 7.9 Hz, 1H), 7.09 (dd, *J* = 10.9, 3.9 Hz, 1H), 6.96 (d, *J* = 7.6 Hz, 1H), 6.90 (m, 1H), 6.87 (dd, *J* = 8.3, 2.1 Hz, 1H), 4.72 (s, 1H), 3.77 (s,

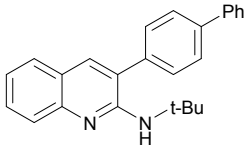
4H), 1.43 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.48, 154.07, 147.73, 139.83, 135.66, 130.18, 129.69, 128.98, 127.26, 126.63, 126.29, 123.16, 121.89, 121.41, 114.96, 113.88, 55.39, 51.84, 29.34. MS(ESI, m/z): 307.1[M+H]$^+$, HRMS (ESI): Exact mass calcd for $C_{20}H_{23}N_2O$ [M+H]$^+$ 307.1805, found 307.1806.

N-(tert-butyl)-3-(4-(tert-butyl)phenyl)quinolin-2-amine (3r)



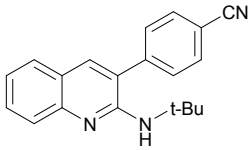
Yellow oil, 52 mg, 79% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.64 (d, *J* = 8.3 Hz, 1H), 7.50 (s, 1H), 7.45 (d, *J* = 7.9 Hz, 1H), 7.41 (dd, *J* = 5.8, 2.2 Hz, 3H), 7.31 (d, *J* = 8.0 Hz, 2H), 7.08 (t, *J* = 7.4 Hz, 1H), 4.74 (s, 1H), 1.43 (s, 9H), 1.30 (d, *J* = 0.4 Hz, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 154.33, 151.25, 147.66, 135.74, 135.40, 128.81, 127.21, 126.61, 126.39, 126.05, 123.34, 121.81, 51.81, 34.73, 31.38, 29.36. MS(ESI, m/z): 333.2[M+H]$^+$, HRMS (ESI): Exact mass calcd for $C_{23}H_{29}N_2$ [M+H]$^+$ 333.2325, found 333.2325.

3-([1,1'-biphenyl]-4-yl)-N-(tert-butyl)quinolin-2-amine (3s)



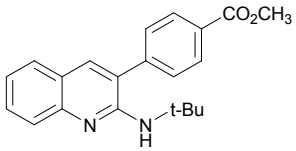
White solid, 49 mg, 70% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 (m, 13H), 7.11 (dd, *J* = 15.3, 8.0 Hz, 1H), 4.72 (s, 1H), 1.44 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 154.11, 147.71, 141.46, 141.07, 140.60, 137.34, 135.87, 129.55, 129.03, 128.93, 128.81, 128.75, 127.84, 127.64, 127.52, 127.28, 127.25, 127.20, 127.11, 126.64, 126.02, 123.24, 121.94, 51.91, 29.36. MS(ESI, m/z): 353.1[M+H]$^+$, HRMS (ESI): Exact mass calcd for $C_{25}H_{25}N_2$ [M+H]$^+$ 353.2012, found 353.2012.

4-(2-(tert-butylamino)quinolin-3-yl)benzonitrile (3t)



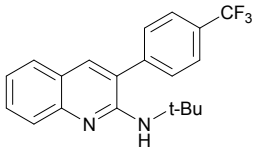
Yellow oil, 43 mg, 71% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (d, *J* = 7.5 Hz, 2H), 7.75 (d, *J* = 8.2 Hz, 1H), 7.60 (m, 5H), 7.23 (t, *J* = 7.3 Hz, 1H), 4.47 (s, 1H), 1.52 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 153.10, 148.01, 143.40, 136.44, 132.92, 129.98, 129.72, 127.42, 126.79, 124.39, 122.86, 122.38, 118.29, 112.30, 52.14, 29.29. MS(ESI, m/z): 302.1[M+H]$^+$, HRMS (ESI): Exact mass calcd for $C_{20}H_{20}N_3$ [M+H]$^+$ 302.1652, found 302.1655.

methyl 4-(2-(tert-butylamino)quinolin-3-yl)benzoate (3u)



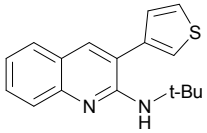
Yellow oil, 51 mg, 76% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 (d, *J* = 7.4 Hz, 2H), 7.75 (d, *J* = 8.3 Hz, 1H), 7.63 (s, 1H), 7.55 (m, 4H), 7.21 (t, *J* = 7.3 Hz, 1H), 4.61 (s, 1H), 3.97 (s, 3H), 1.51 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.62, 153.55, 147.90, 143.22, 136.07, 130.45, 130.11, 129.36, 129.20, 127.36, 126.72, 125.33, 123.01, 122.13, 52.06, 51.99, 29.30. MS(ESI, m/z): 335.1[M+H]$^+$, HRMS (ESI): Exact mass calcd for $C_{21}H_{23}N_2O_2$[M+H]$^+$ 335.1754, found 335.1755.

N-(tert-butyl)-3-(4-(trifluoromethyl)phenyl)quinolin-2-amine (3v)



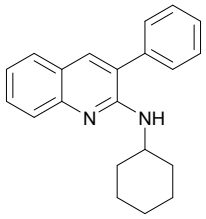
White solid, 50 mg, 72% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (d, *J* = 7.9 Hz, 3H), 7.49 (m, 5H), 7.11 (t, *J* = 7.3 Hz, 1H), 4.46 (s, 1H), 1.42 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 153.48, 147.95, 142.32, 136.32, 129.62, 129.48, 127.37, 126.77, 126.17, 126.14, 124.86, 122.98, 122.22, 52.04, 29.31. MS(ESI, m/z): 345.1[M+H]$^+$, HRMS (ESI): Exact mass calcd for $C_{20}H_{20}F_3N_2$ [M+H]$^+$ 345.1573, found 345.1577.

N-(tert-butyl)-3-(thiophen-3-yl)quinolin-2-amine (3w)



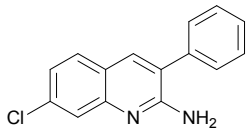
Yellow oil, 43 mg, 76% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (d, *J* = 8.3 Hz, 1H), 7.59 (s, 1H), 7.47 (m, 3H), 7.34 (s, 1H), 7.17 (d, *J* = 4.2 Hz, 1H), 7.09 (t, *J* = 7.3 Hz, 1H), 4.82 (s, 1H), 1.45 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 154.25, 147.66, 138.75, 135.71, 129.02, 128.15, 127.18, 126.84, 126.65, 125.97, 123.65, 123.09, 121.95, 121.31, 119.82, 51.85, 29.33. MS(ESI, m/z): 283.1[M+H]$^+$, HRMS (ESI): Exact mass calcd for $C_{17}H_{19}N_2S$ [M+H]$^+$ 283.1263, found 283.1265.

N-cyclohexyl-3-phenylquinolin-2-amine (3x)



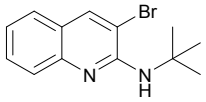
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (d, *J* = 8.3 Hz, 1H), 7.62 (s, 1H), 7.58-7.40 (m, 7H), 7.18 (t, *J* = 7.3 Hz, 1H), 4.68 (d, *J* = 7.5 Hz, 1H), 4.27-4.17 (m, 1H), 2.08 (m, *J* = 12.1 Hz, 2H), 1.47 (m, *J* = 24.1, 11.6 Hz, 3H), 1.28-1.12 (m, 5H). ^{13}C NMR (125 MHz, DMSO) δ 153.62, 147.53, 137.56, 136.56, 129.67, 129.56, 129.16, 128.57, 127.98, 125.88, 125.73, 123.46, 122.01, 49.05, 32.53, 25.81, 24.99. MS(ESI, m/z): 303.2[M+H]$^+$, HRMS (ESI): Exact mass calcd for C21H22N2 [M+H]$^+$ 303.4202, found 303.4202.

7-chloro-3-phenylquinolin-2-amine (4c)



^{1}H NMR (400 MHz, CDCl3) δ 7.74 (s, 1H), 7.68 (d, *J* = 1.7 Hz, 1H), 7.59-7.53 (m, 1H), 7.50 (d, *J* = 4.1 Hz, 4H), 7.45 (dd, *J* = 9.0, 4.7 Hz, 1H), 7.22 (dd, *J* = 8.5, 2.0 Hz, 1H), 5.30 (s, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ155.80, 146.96, 137.03, 136.82, 135.53, 129.24, 128.74, 128.58, 128.48, 125.19, 124.20, 123.65, 122.28. MS(ESI, m/z):254.1[M+H]$^+$. HRMS (ESI): Exact mass calcd for $C_{17}H_{19}N_2S$ [M+H]$^+$ 255.0684, found 255.0688.

3-bromo-N-tert-butylquinolin-2-amine (5)[2]



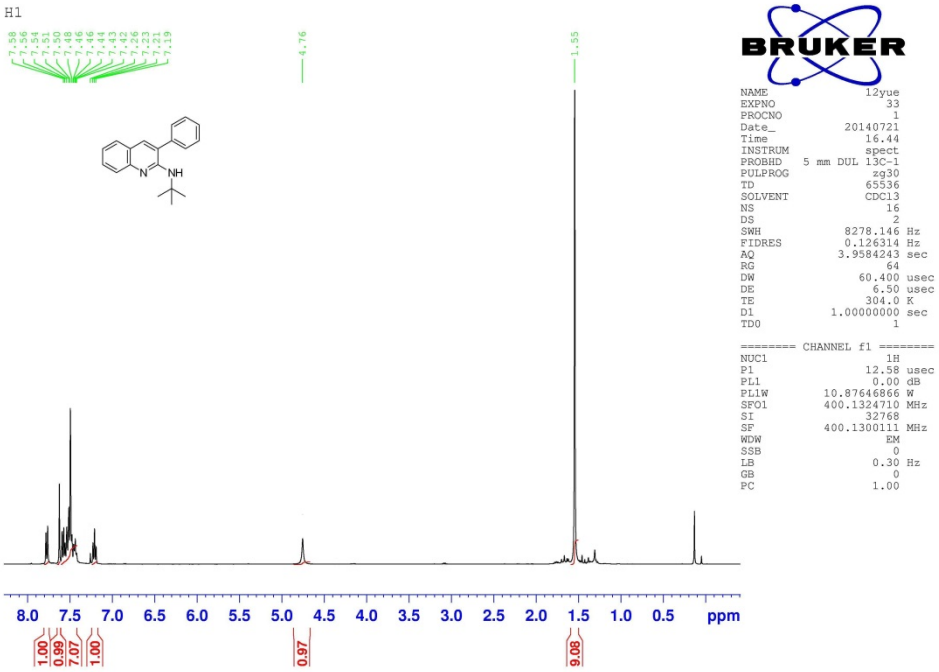
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (s, 1H), 7.61 (d, *J* = 8.3 Hz, 1H), 7.42 (dd, *J* = 19.4, 7.7 Hz, 2H), 7.11 (d, *J* = 7.4 Hz, 1H), 5.22 (s, 1H), 1.50 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 151.70, 146.80, 138.34, 132.25, 132.16, 129.46, 128.54, 128.42, 126.71, 126.38, 123.86, 122.41, 108.96, 52.31, 29.03. MS(ESI, m/z): 279.0[M+H]$^+$ HRMS (ESI): Exact mass calcd for $C_{13}H_{15}BrN_2$ [M+H]$^+$ 279.0491, found 279.0495.

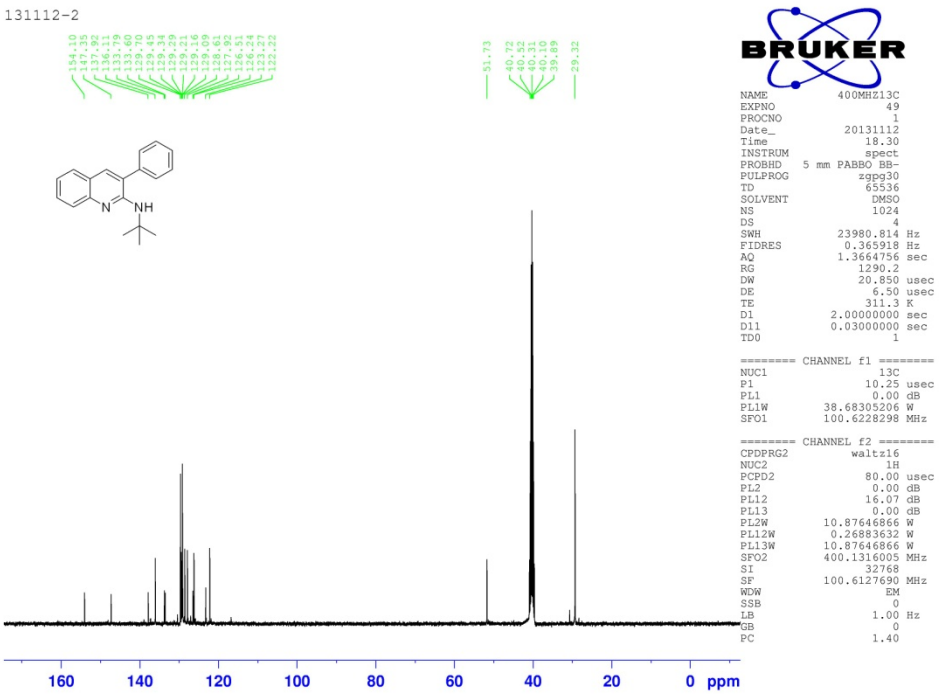
4. References

1. B. Jiang, K. Tao, W. Shen, J. Zhang, *Tetrahedron Lett.* 2010, **51**, 6342-6344.
2. B. Jiang, L. Hu, W. Gui, *RSC Adv.* 2014, **4**, 13850.

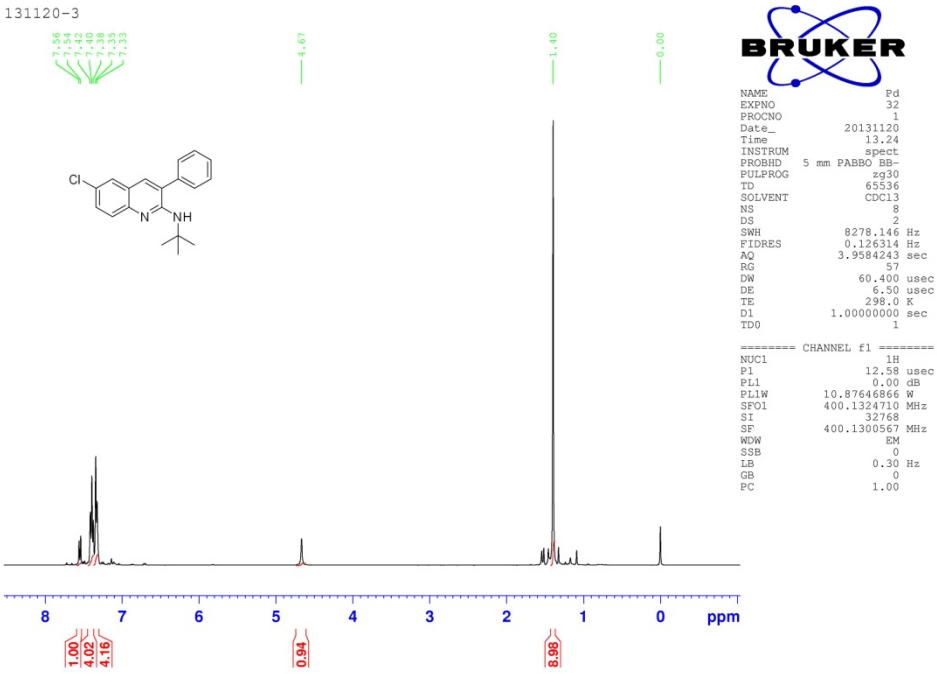
5.Copies of NMR Spectra

3a

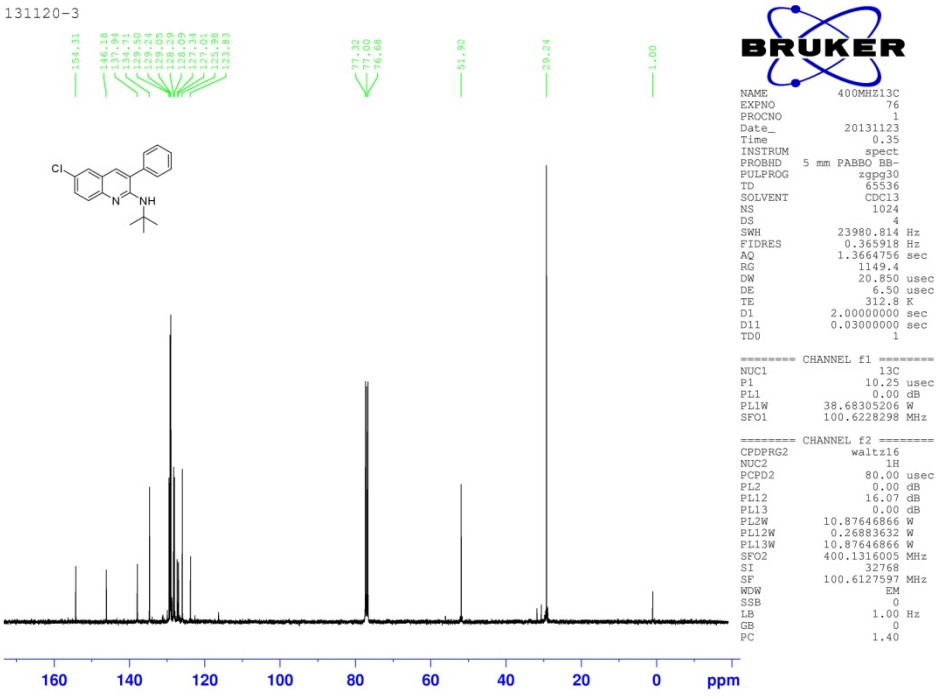




3b

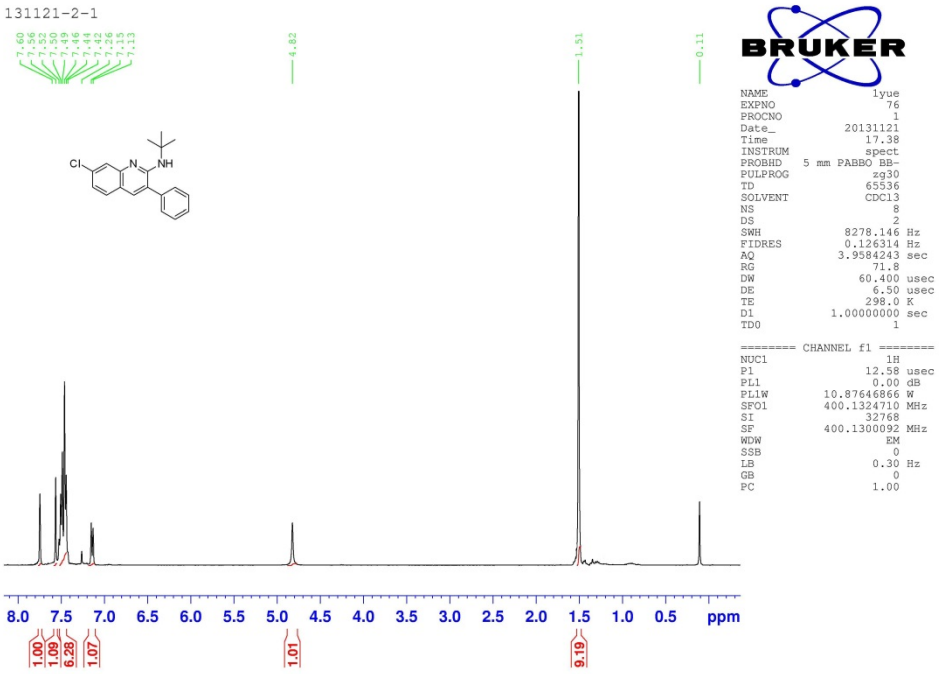


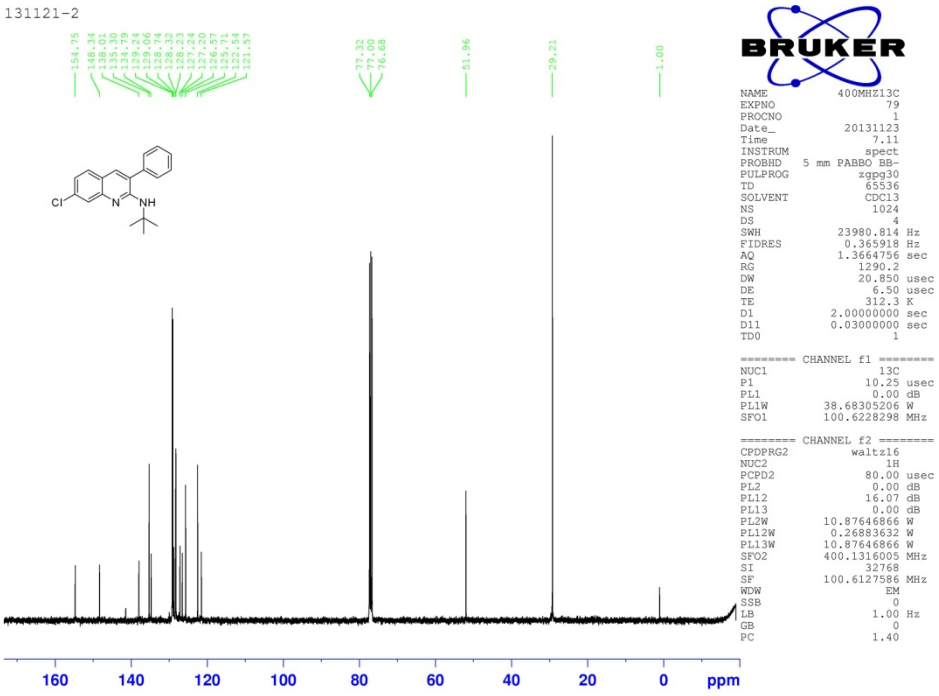

```
NAME                 Pd
EXPNO                32
PROCNO                1
Date_          20131120
Time              13.24
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                    57
DW                60.400 usec
DE                 6.50 usec
TE                298.0 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           400.1300567 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.00
```



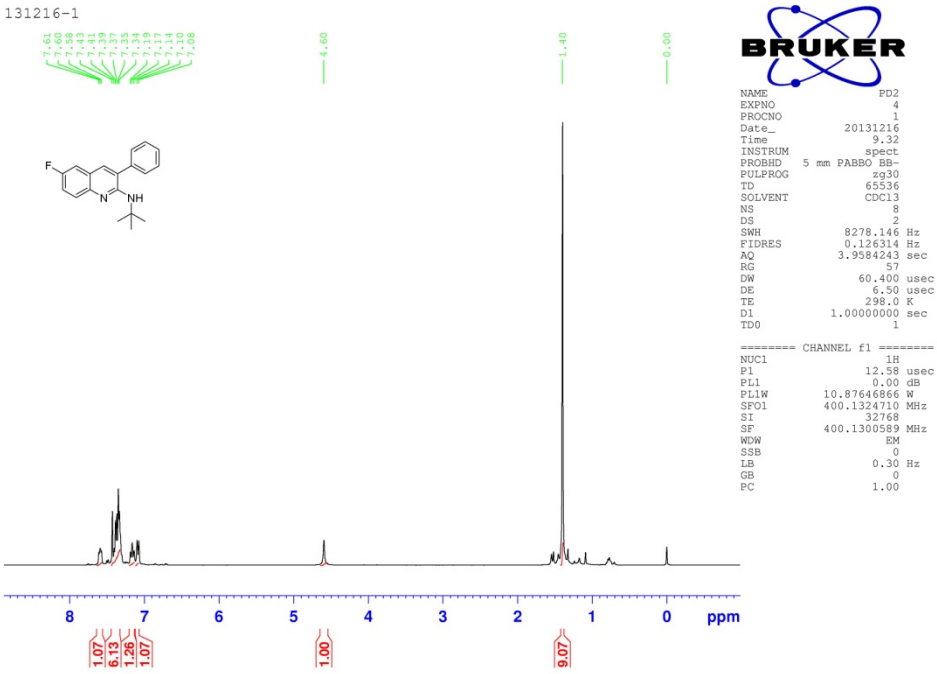

```
NAME           400MHZ13C
EXPNO                 76
PROCNO                 1
Date_          20131123
Time               0.35
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ              1.3664756 sec
RG                1149.4
DW                20.850 usec
DE                 6.50 usec
TE                312.8 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                10.25 usec
PL1                0.00 dB
PL1W         38.68305206 W
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2                0.00 dB
PL12              16.07 dB
PL13               0.00 dB
PL2W         10.87646866 W
PL12W         0.26883632 W
PL13W        10.87646866 W
SFO2         400.1316005 MHz
SI                 32768
SF           100.6127597 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                 1.40
```

3c

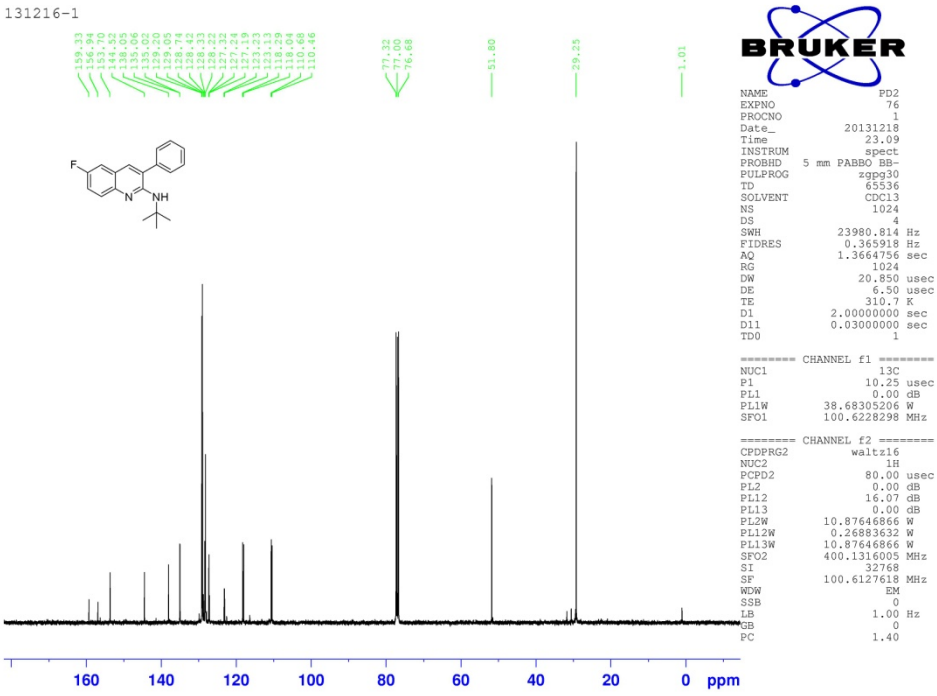

131121-2-1
7.60
7.56
7.53
7.50
7.49
7.46
7.44
7.44
7.26
7.15
7.13
4.82
1.51
0.11
Cl
N
NH
BRUKER
NAME lyue
EXPNO 76
PROCNO 1
Date_ 20131121
Time 17.38
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 71.8
DW 60.400 usec
DE 6.50 usec
TE 298.0 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 400.1300092 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
1.09
6.28
1.07
1.01
9.19


131121-2
154.75
148.34
138.01
136.10
134.79
129.24
129.06
128.74
128.32
128.23
127.24
127.20
126.57
126.31
122.54
121.57
77.32
77.00
76.68
51.96
29.21
1.00
Cl
N
NH
BRUKER
NAME 400MHZ13C
EXPNO 79
PROCNO 1
Date_ 20131123
Time 7.11
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1290.2
DW 20.850 usec
DE 6.50 usec
TE 312.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.25 usec
PL1 0.00 dB
PL1W 38.68305206 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 16.07 dB
PL13 0.00 dB
PL2W 10.87646866 W
PL12W 0.26883632 W
PL13W 10.87646866 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127586 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160 140 120 100 80 60 40 20 0 ppm

3d

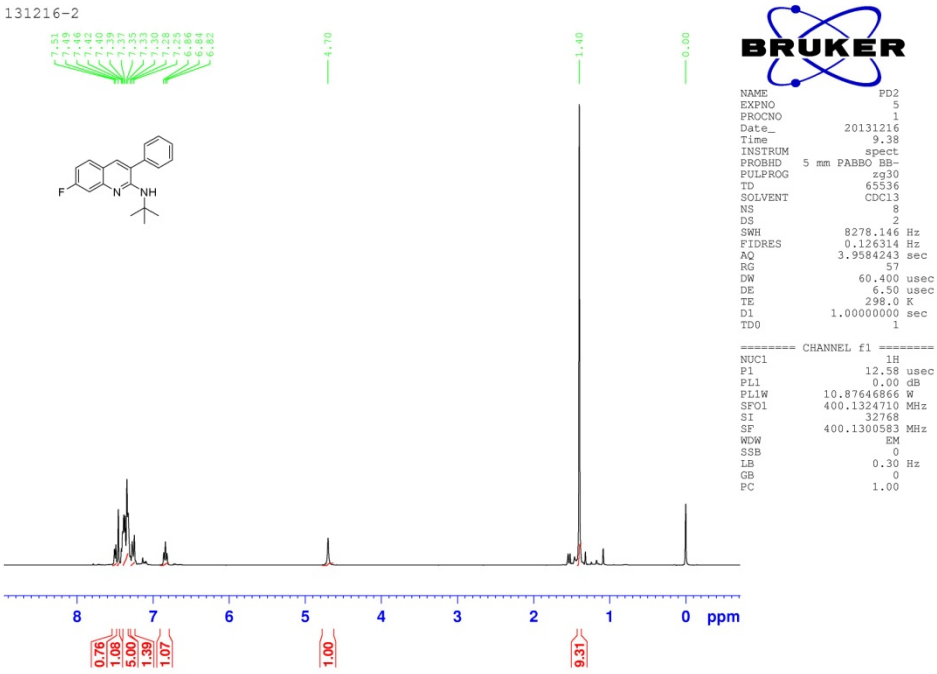


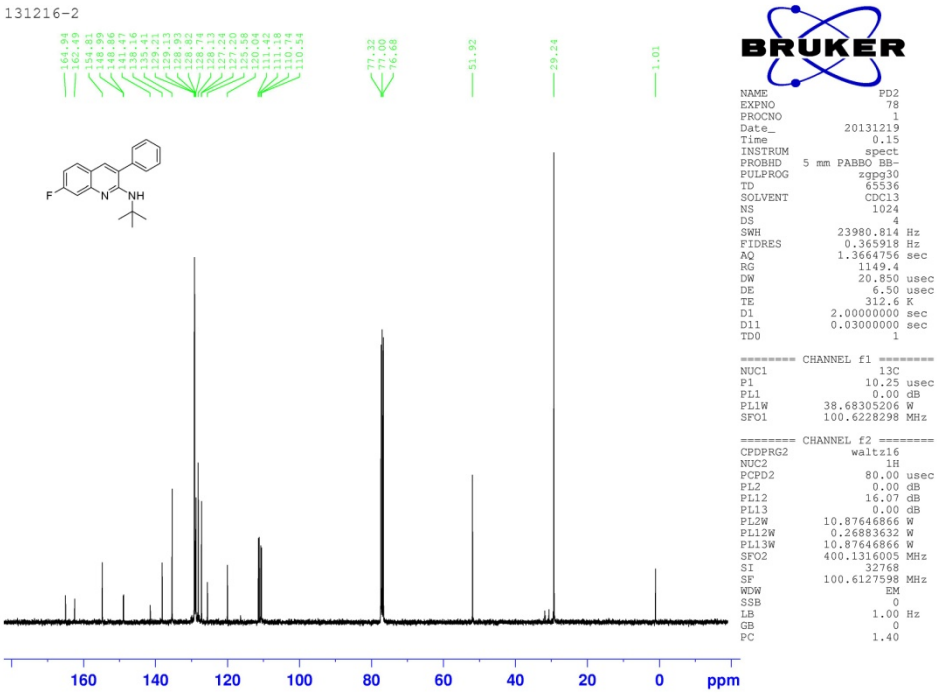

```
NAME                PD2
EXPNO                 4
PROCNO                1
Date_          20131216
Time               9.32
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDC13
NS                    8
DS                    2
SWH            8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                   57
DW               60.400 usec
DE                6.50 usec
TE                298.0 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          400.1300589 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



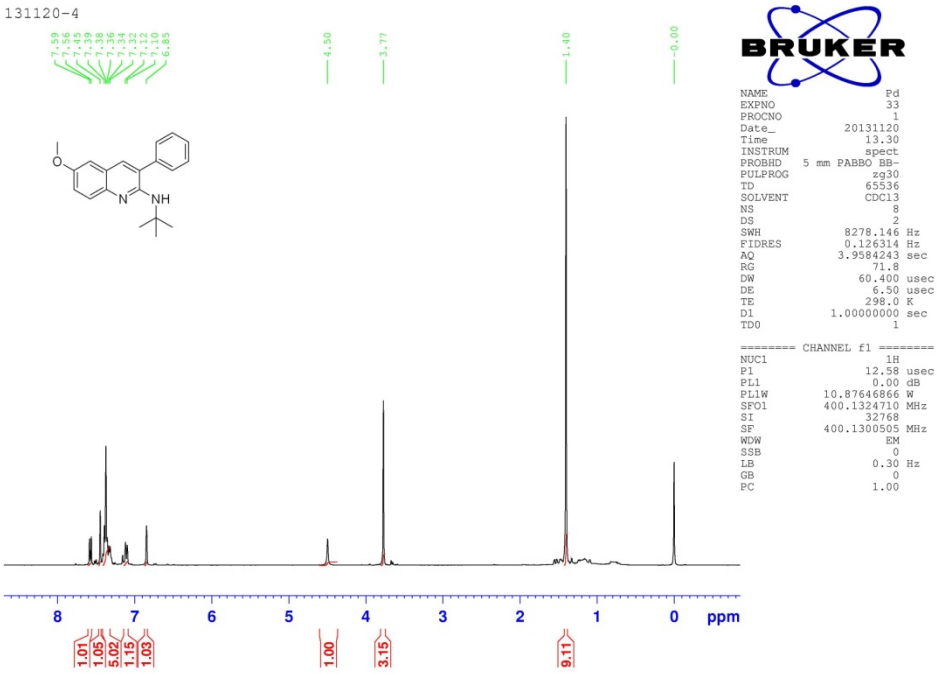

```
NAME                PD2
EXPNO                76
PROCNO                1
Date_          20131218
Time              23.09
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDC13
NS                 1024
DS                    4
SWH           23980.814 Hz
FIDRES        0.365918 Hz
AQ            1.3664756 sec
RG                 1024
DW               20.850 usec
DE                6.50 usec
TE                310.7 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                10.25 usec
PL1                0.00 dB
PL1W        38.68305206 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2                0.00 dB
PL12              16.07 dB
PL13               0.00 dB
PL2W        10.87646866 W
PL12W        0.26883632 W
PL13W       10.87646866 W
SFO2        400.1316005 MHz
SI                32768
SF          100.6127618 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

3e


131216-2
7.51
7.49
7.46
7.42
7.40
7.39
7.37
7.35
7.33
7.30
7.28
7.25
6.86
6.84
6.82
4.70
1.40
0.00
F
N
NH
8
7
6
5
4
3
2
1
0
ppm
0.76
1.08
5.00
1.39
1.07
1.00
9.31
BRUKER
NAME PD2
EXPNO 5
PROCNO 1
Date_ 20131216
Time 9.38
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 57
DW 60.400 usec
DE 6.50 usec
TE 298.0 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 400.1300583 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

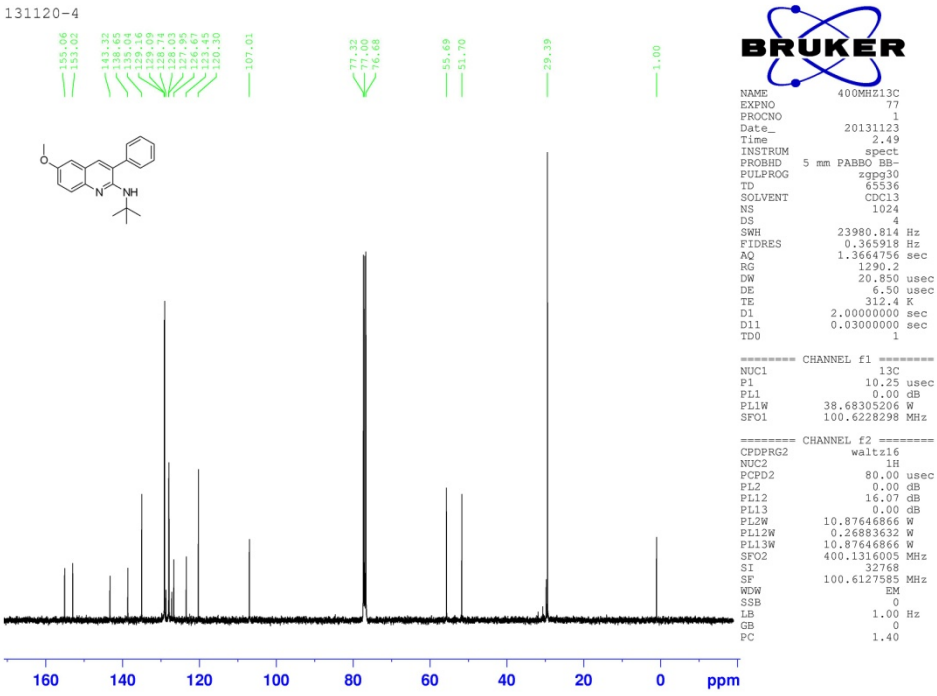

131216-2
164.94
162.49
154.81
148.89
140.86
141.47
138.16
135.41
133.21
129.13
128.93
128.82
128.74
128.33
127.24
127.20
125.58
120.04
119.42
111.18
110.74
110.54
77.32
77.00
76.68
51.92
29.24
1.01
F
N
NH
160
140
120
100
80
60
40
20
0
ppm
BRUKER
NAME PD2
EXPNO 78
PROCNO 1
Date_ 20131219
Time 0.15
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1149.4
DW 20.850 usec
DE 6.50 usec
TE 312.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.25 usec
PL1 0.00 dB
PL1W 38.68305206 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 16.07 dB
PL13 0.00 dB
PL2W 10.87646866 W
PL12W 0.26883632 W
PL13W 10.87646866 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127598 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

3f



BRUKER

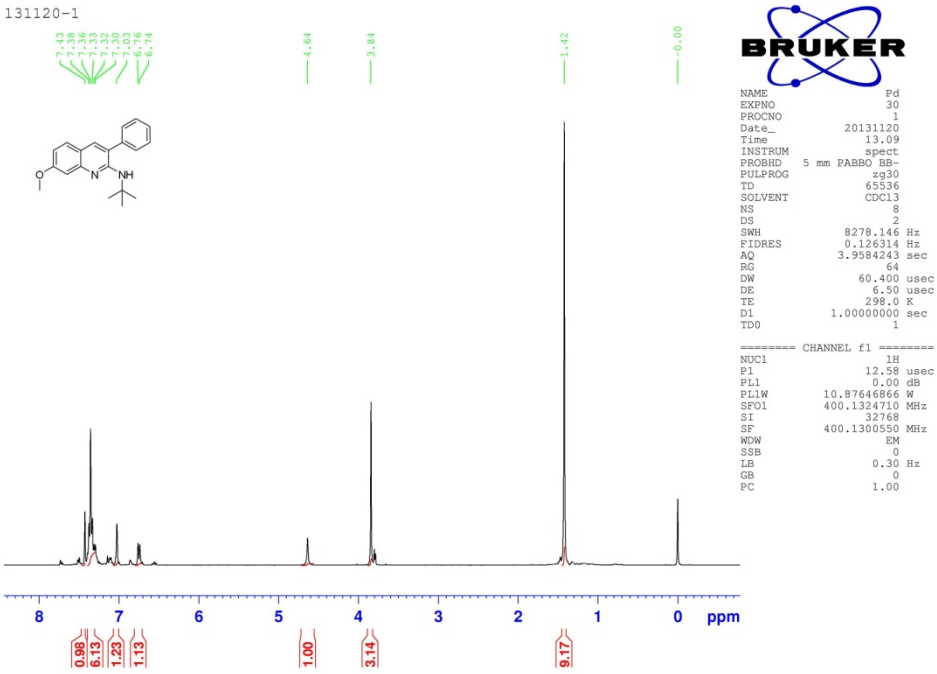
```
NAME                Pd
EXPNO               33
PROCNO               1
Date_         20131120
Time             13.30
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG          zg30
TD              65536
SOLVENT          CDCl3
NS                   8
DS                   2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                71.8
DW              60.400 usec
DE               6.50 usec
TE              298.0 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         400.1300505 MHz
WDW                EM
SSB                 0
LB               0.30 Hz
GB                  0
PC               1.00
```



BRUKER

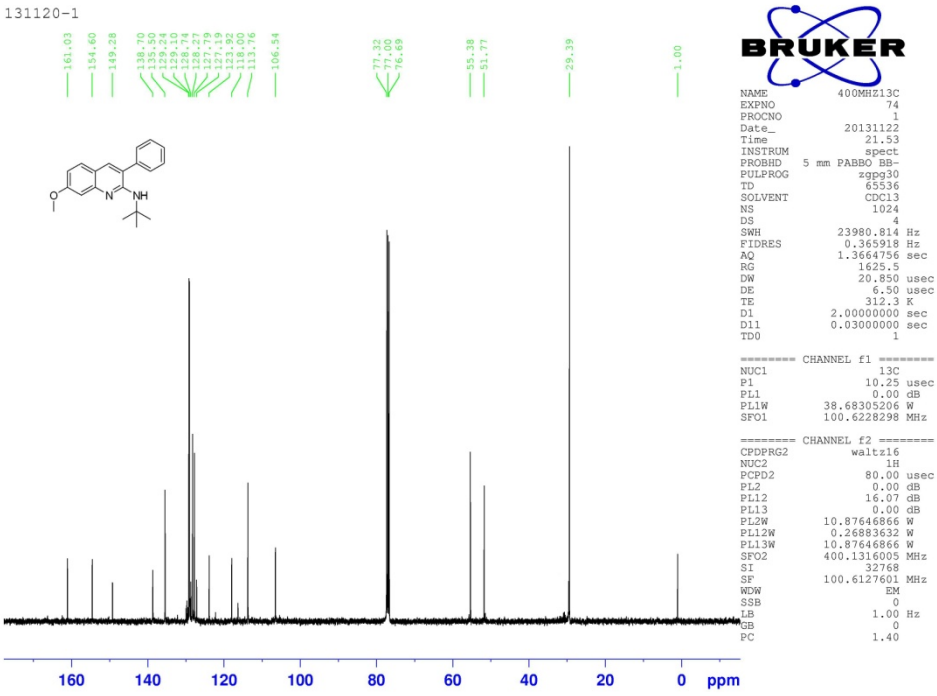
```
NAME          400MHZ13C
EXPNO                77
PROCNO                1
Date_          20131123
Time               2.49
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           23980.814 Hz
FIDRES        0.365918 Hz
AQ            1.3664756 sec
RG               1290.2
DW              20.850 usec
DE               6.50 usec
TE              312.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                13C
P1               10.25 usec
PL1               0.00 dB
PL1W        38.68305206 W
SFO1       100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            80.00 usec
PL2               0.00 dB
PL12             16.07 dB
PL13              0.00 dB
PL2W        10.87646866 W
PL12W        0.26883632 W
PL13W       10.87646866 W
SFO2       400.1316005 MHz
SI              32768
SF         100.6127585 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
```

3g



BRUKER

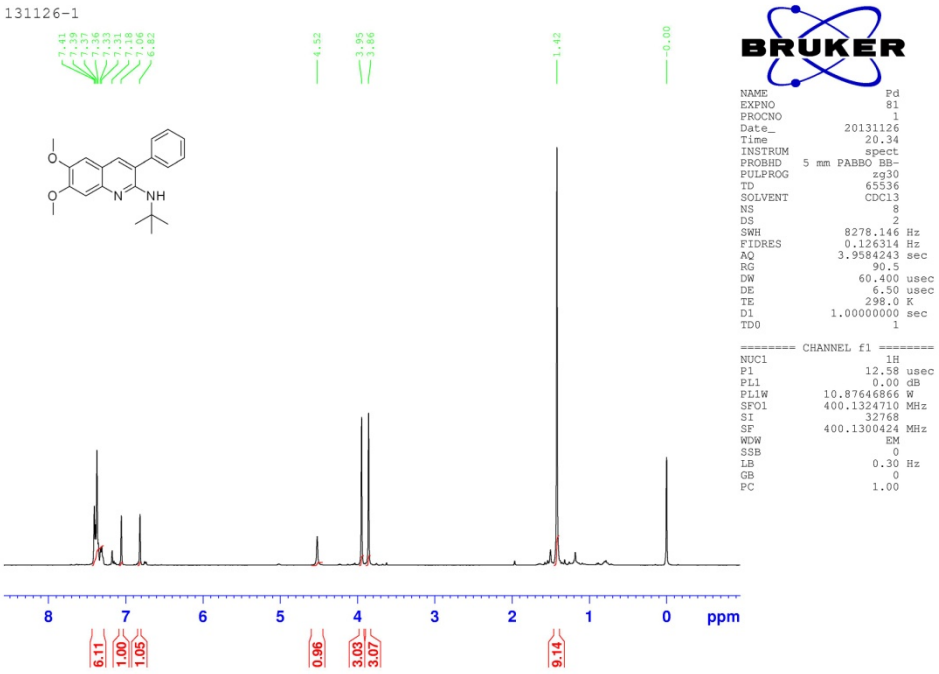
```
NAME                 Pd
EXPNO                30
PROCNO                1
Date_          20131120
Time              13.09
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDC13
NS                    8
DS                    2
SWH            8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                   64
DW               60.400 usec
DE                6.50 usec
TE               298.0 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          400.1300550 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



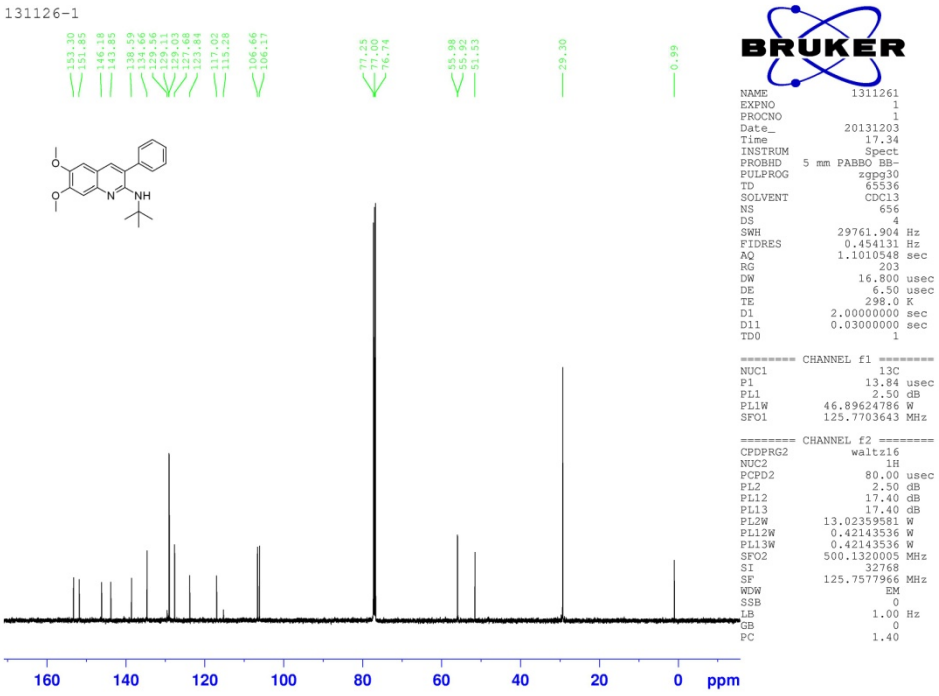
BRUKER

```
NAME           400MHZ13C
EXPNO                 74
PROCNO                 1
Date_           20131122
Time               21.53
INSTRUM            spect
PROBHD    5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDC13
NS                 1024
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664756 sec
RG                1625.5
DW               20.850 usec
DE                6.50 usec
TE               312.3 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.25 usec
PL1                0.00 dB
PL1W        38.68305206 W
SFO1       100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2                0.00 dB
PL12              16.07 dB
PL13               0.00 dB
PL2W        10.87646866 W
PL12W        0.26883632 W
PL13W       10.87646866 W
SFO2       400.1316005 MHz
SI                32768
SF         100.6127601 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

3h

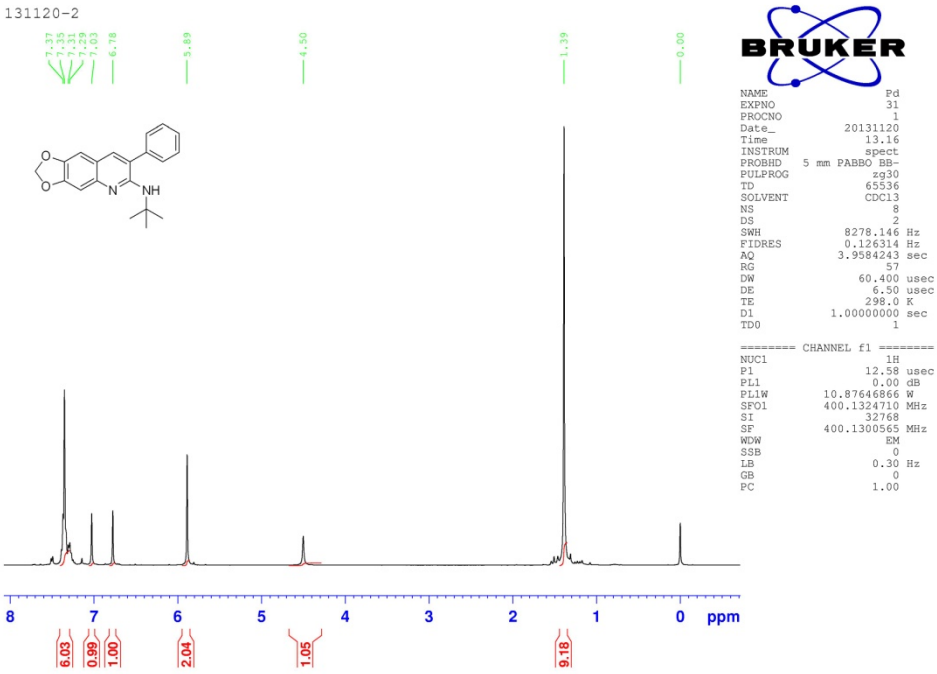


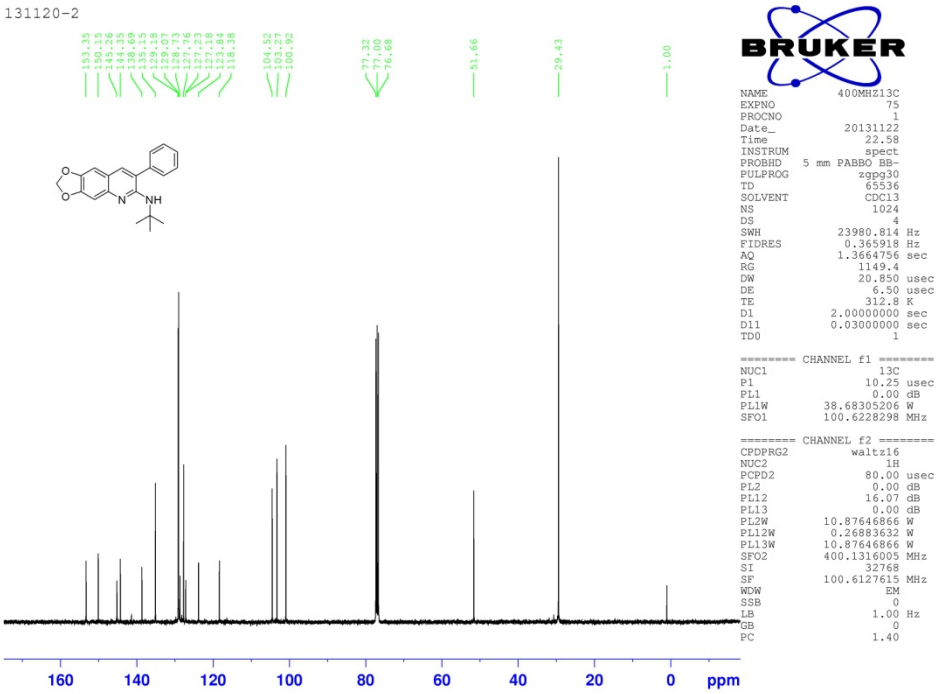

```
NAME                Pd
EXPNO               81
PROCNO              1
Date_           20131126
Time               20.34
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDC13
NS                     8
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                  90.5
DW                60.400 usec
DE                 6.50 usec
TE                298.0 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           400.1300424 MHz
WDW                EM
SSB                0
LB                 0.30 Hz
GB                 0
PC                 1.00
```



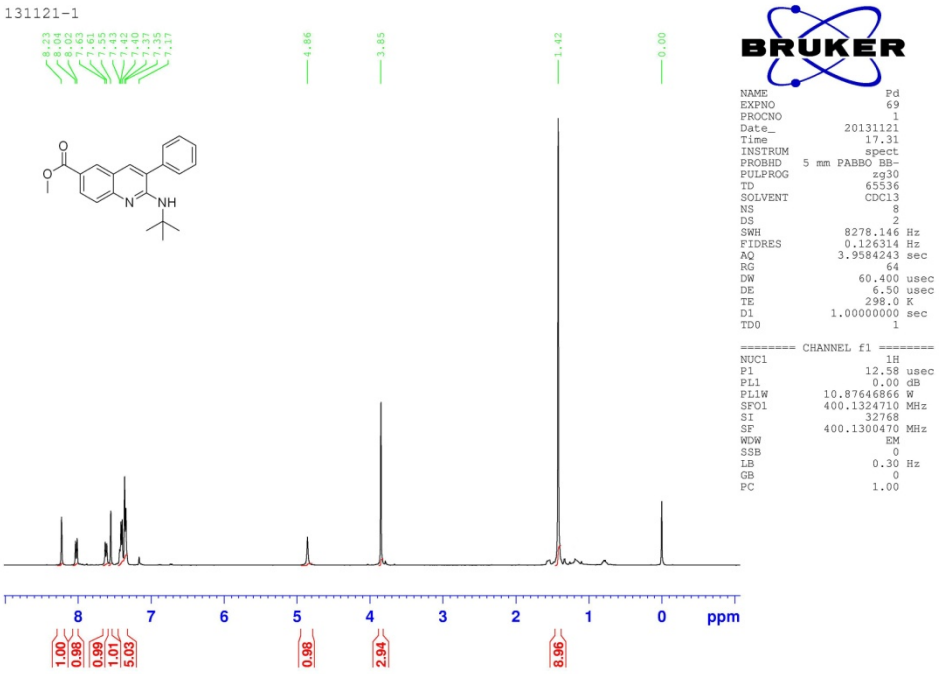

```
NAME             1311261
EXPNO                  1
PROCNO                 1
Date_           20131203
Time               17.34
INSTRUM            Spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDC13
NS                   656
DS                     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           125.7577966 MHz
WDW                EM
SSB                0
LB                 1.00 Hz
GB                 0
PC                 1.40
```

3i


131120-2
7.37
7.35
7.31
7.29
7.03
6.78
5.89
4.50
1.39
0.00
NH
8
7
6
5
4
3
2
1
0
ppm
6.03
0.99
1.00
2.04
1.05
9.18
BRUKER
NAME Pd
EXPNO 31
PROCNO 1
Date_ 20131120
Time 13.16
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 57
DW 60.400 usec
DE 6.50 usec
TE 298.0 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 400.1300565 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

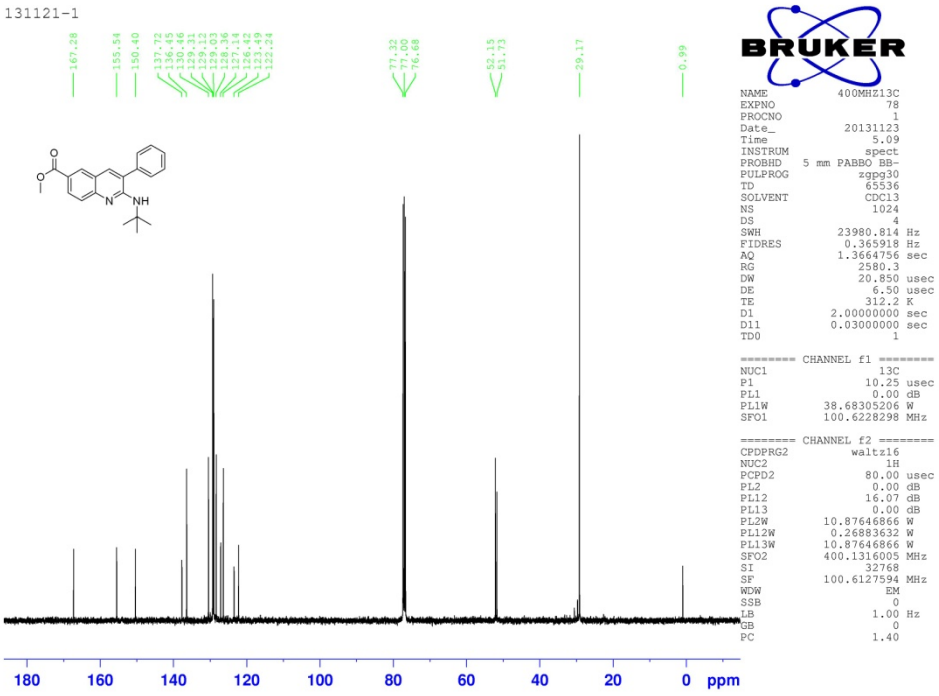

131120-2
153.35
150.15
145.26
144.35
138.69
135.15
129.19
129.07
128.73
127.76
127.73
127.18
123.84
118.38
104.52
103.27
100.92
77.32
77.00
76.68
51.66
29.43
1.00
NH
160
140
120
100
80
60
40
20
0
ppm
BRUKER
NAME 400MHZ13C
EXPNO 75
PROCNO 1
Date_ 20131122
Time 22.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1149.4
DW 20.850 usec
DE 6.50 usec
TE 312.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.25 usec
PL1 0.00 dB
PL1W 38.68305206 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 16.07 dB
PL13 0.00 dB
PL2W 10.87646866 W
PL12W 0.26883632 W
PL13W 10.87646866 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127615 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

3j



BRUKER

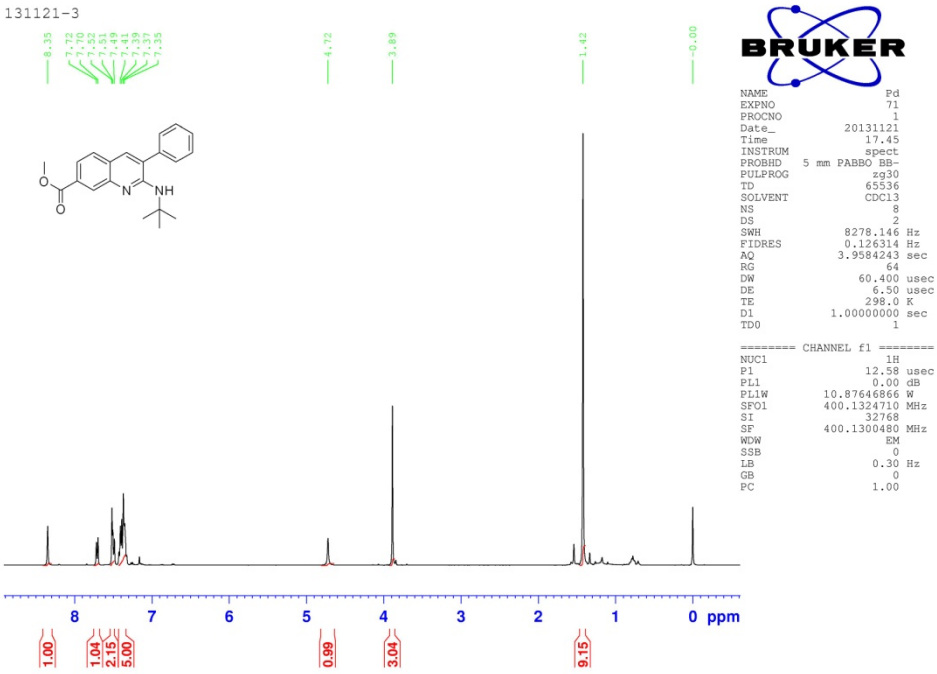
```
NAME                Pd
EXPNO               69
PROCNO               1
Date_         20131121
Time             17.31
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG          zg30
TD              65536
SOLVENT         CDCl3
NS                   8
DS                   2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                  64
DW             60.400 usec
DE              6.50 usec
TE             298.0 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         400.1300470 MHz
WDW                EM
SSB                 0
LB              0.30 Hz
GB                  0
PC              1.00
```

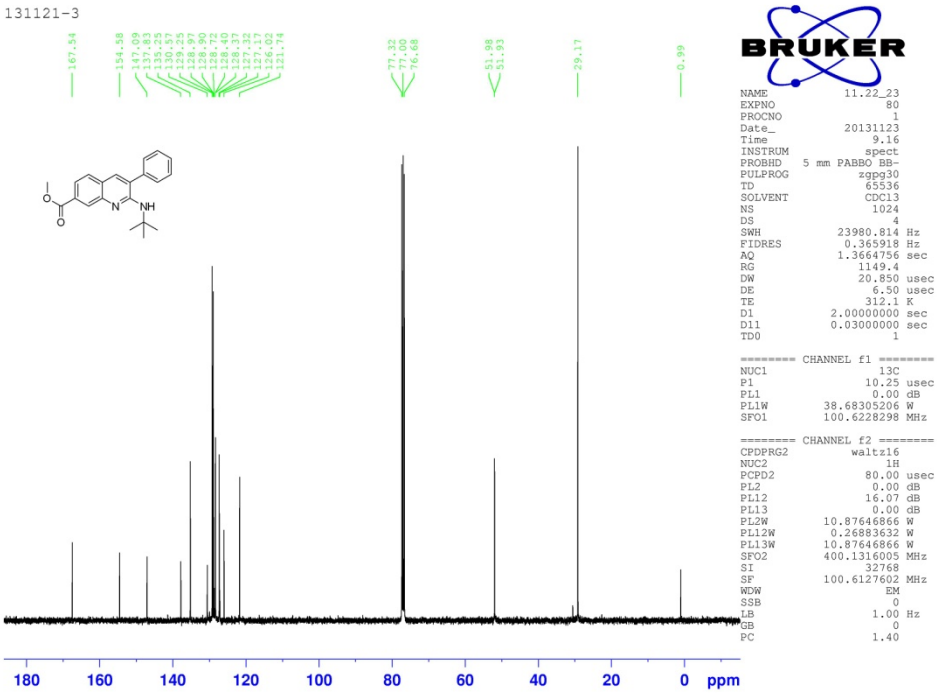


BRUKER

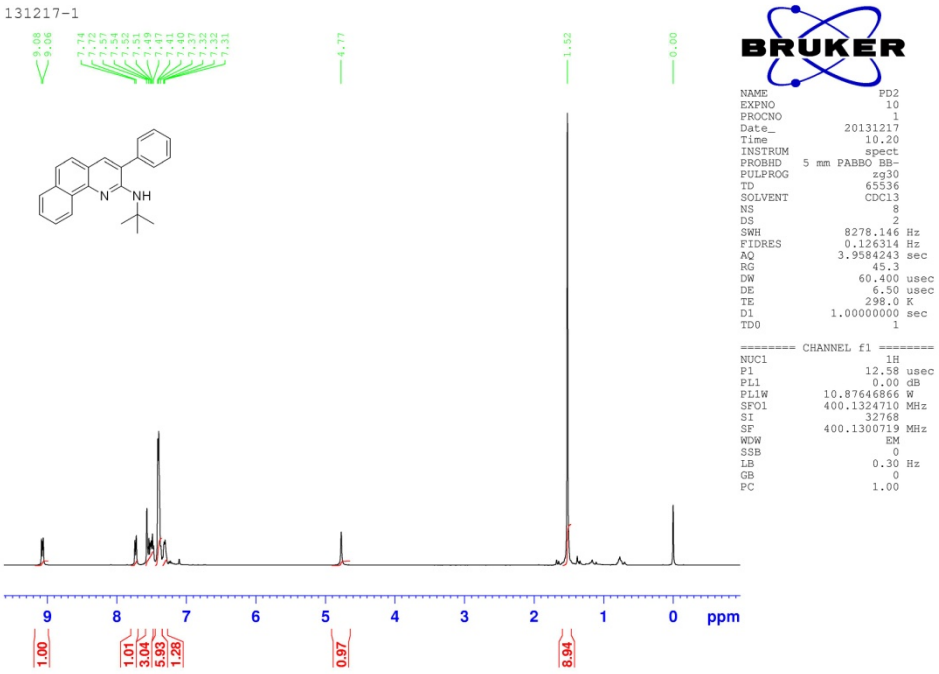
```
NAME          400MHz13C
EXPNO                78
PROCNO                1
Date_          20131123
Time               5.09
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664756 sec
RG                2580.3
DW             20.850 usec
DE              6.50 usec
TE             312.2 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1             10.25 usec
PL1             0.00 dB
PL1W       38.68305206 W
SFO1       100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2          80.00 usec
PL2             0.00 dB
PL12           16.07 dB
PL13            0.00 dB
PL2W       10.87646866 W
PL12W       0.26883632 W
PL13W      10.87646866 W
SFO2       400.1316005 MHz
SI              32768
SF         100.6127594 MHz
WDW                EM
SSB                 0
LB              1.00 Hz
GB                  0
PC              1.40
```

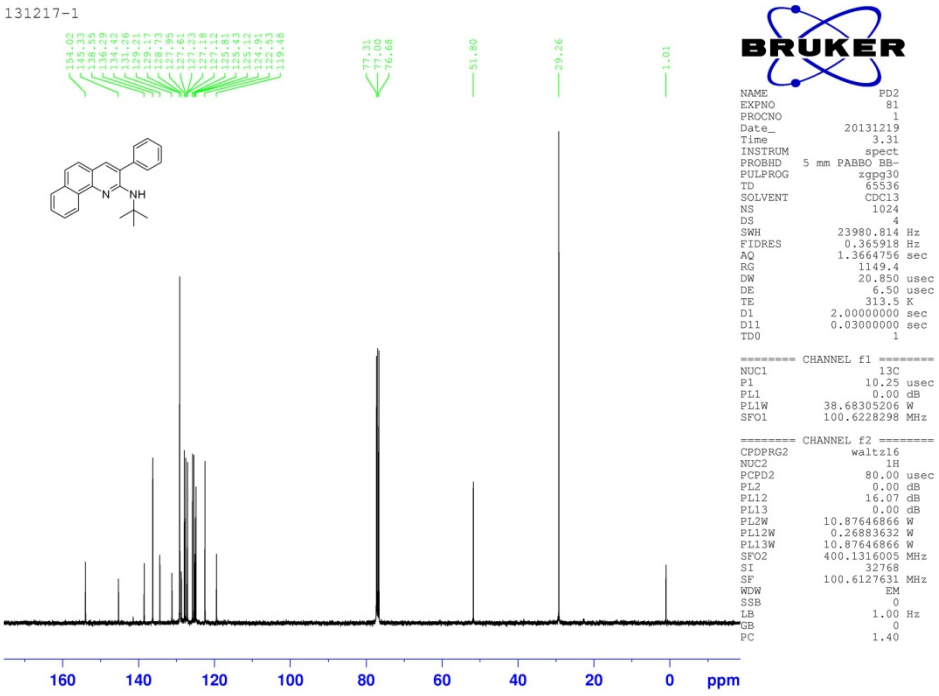
3k


131121-3
8.35
7.72
7.70
7.52
7.51
7.43
7.41
7.39
7.37
7.35
4.72
3.89
1.42
0.00
BRUKER
NAME Pd
EXPNO 71
PROCNO 1
Date_ 20131121
Time 17.45
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 64
DW 60.400 usec
DE 6.50 usec
TE 298.0 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 400.1300480 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8 7 6 5 4 3 2 1 0 ppm
1.00
1.04
2.15
5.00
0.99
3.04
9.15

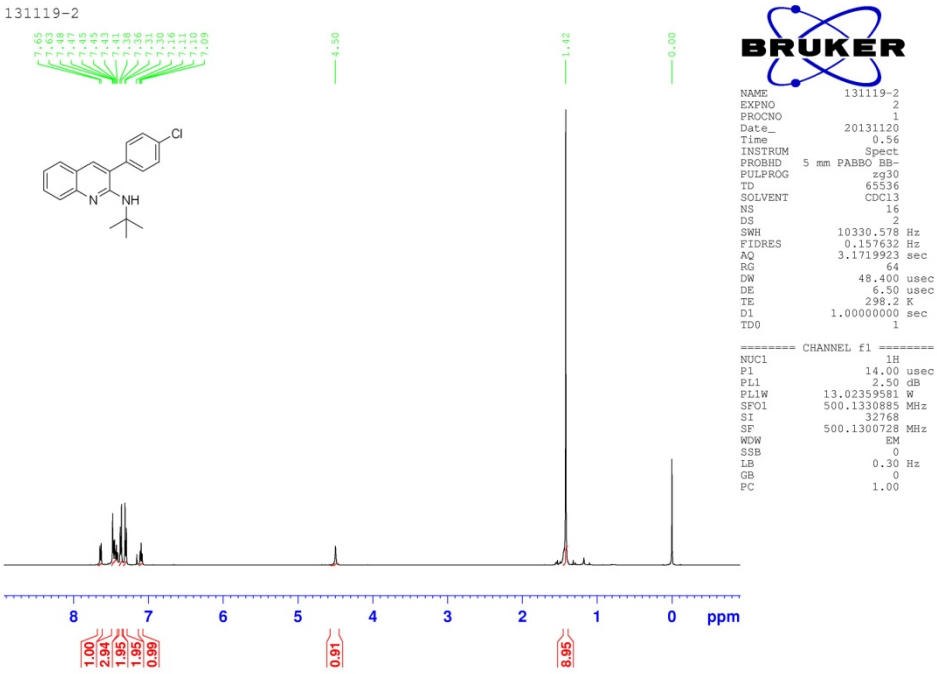

131121-3
167.84
154.58
147.09
137.83
135.25
130.33
129.25
128.97
128.90
128.72
128.40
128.37
127.32
127.17
126.02
121.74
77.32
77.00
76.68
51.98
51.93
29.17
0.99
BRUKER
NAME 11.22_23
EXPNO 80
PROCNO 1
Date_ 20131123
Time 9.16
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1149.4
DW 20.850 usec
DE 6.50 usec
TE 312.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.25 usec
PL1 0.00 dB
PL1W 38.68305206 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 16.07 dB
PL13 0.00 dB
PL2W 10.87646866 W
PL12W 0.26883632 W
PL13W 10.87646866 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127602 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180 160 140 120 100 80 60 40 20 0 ppm

3l

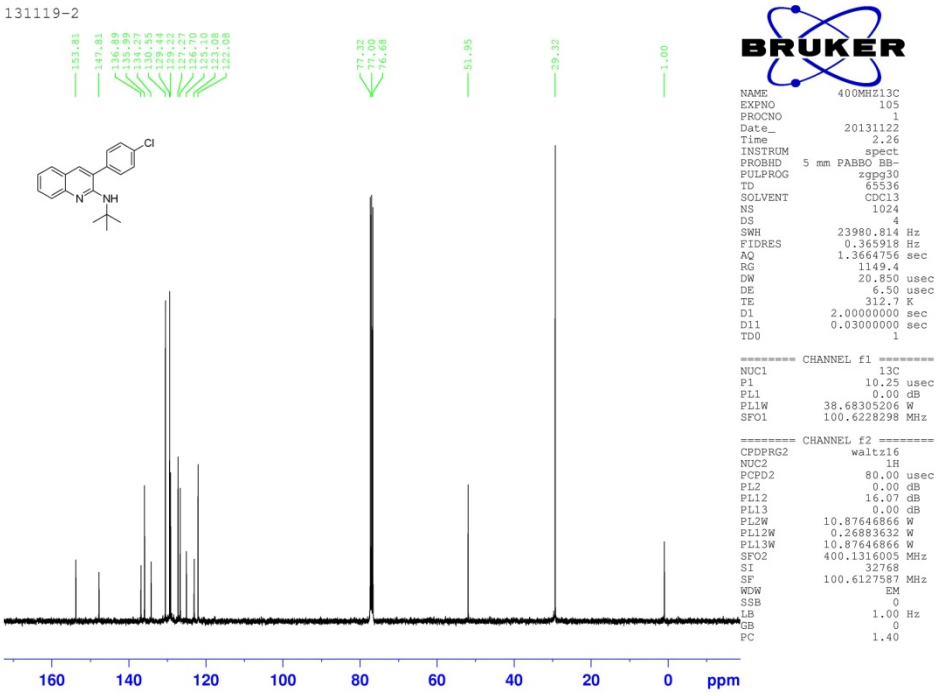

131217-1
9.08
9.06
7.74
7.72
7.57
7.54
7.53
7.51
7.49
7.47
7.41
7.40
7.37
7.32
7.32
7.31
4.77
1.52
0.00
NH
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.01
3.04
5.93
1.28
0.97
8.94
BRUKER
NAME PD2
EXPNO 10
PROCNO 1
Date_ 20131217
Time 10.20
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 45.3
DW 60.400 usec
DE 6.50 usec
TE 298.0 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 400.1300719 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


131217-1
154.02
145.33
138.95
136.29
134.43
131.46
129.21
129.17
128.73
128.36
127.61
127.23
127.18
127.12
127.01
125.43
125.12
124.91
123.33
119.48
77.31
77.00
76.68
51.80
29.26
1.01
NH
160
140
120
100
80
60
40
20
0
ppm
BRUKER
NAME PD2
EXPNO 81
PROCNO 1
Date_ 20131219
Time 3.31
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1149.4
DW 20.850 usec
DE 6.50 usec
TE 313.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.25 usec
PL1 0.00 dB
PL1W 38.68305206 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 16.07 dB
PL13 0.00 dB
PL2W 10.87646866 W
PL12W 0.26883632 W
PL13W 10.87646866 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127631 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

3m

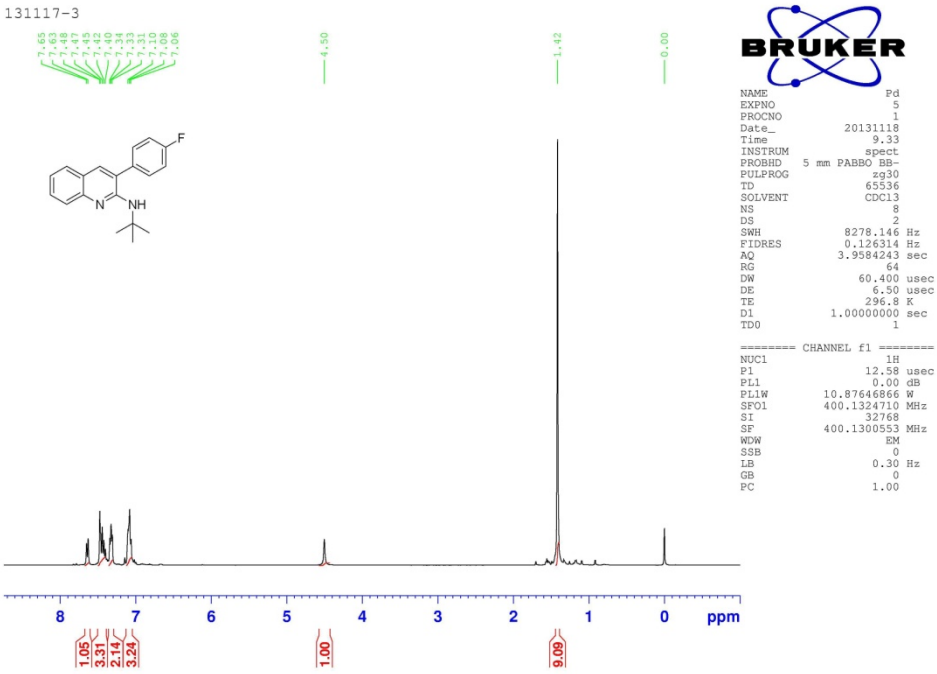



```
NAME                131119-2
EXPNO                      2
PROCNO                     1
Date_               20131120
Time                    0.56
INSTRUM                Spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDC13
NS                       16
DS                        2
SWH                10330.578 Hz
FIDRES             0.157632 Hz
AQ                 3.1719923 sec
RG                       64
DW                   48.400 usec
DE                    6.50 usec
TE                   298.2 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                500.1300728 MHz
WDW                        EM
SSB                         0
LB                     0.30 Hz
GB                          0
PC                     1.00
```

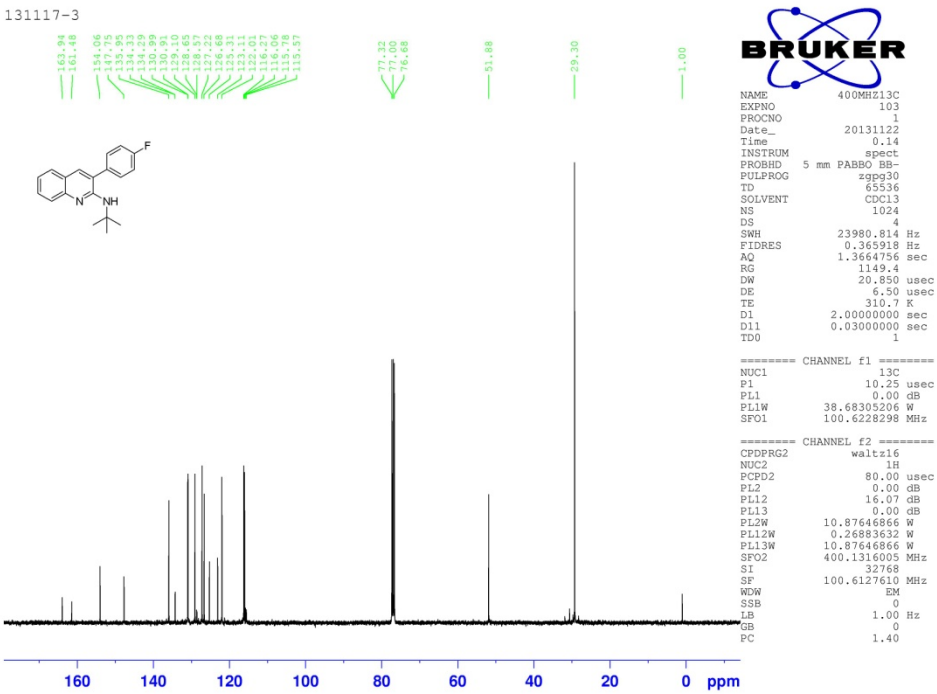



```
NAME              400MHZ13C
EXPNO                   105
PROCNO                    1
Date_              20131122
Time                   2.26
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT               CDC13
NS                     1024
DS                        4
SWH                23980.814 Hz
FIDRES             0.365918 Hz
AQ                 1.3664756 sec
RG                    1149.4
DW                   20.850 usec
DE                    6.50 usec
TE                   312.7 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     13C
P1                     10.25 usec
PL1                     0.00 dB
PL1W              38.68305206 W
SFO1              100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                      1H
PCPD2                  80.00 usec
PL2                     0.00 dB
PL12                   16.07 dB
PL13                    0.00 dB
PL2W              10.87646866 W
PL12W              0.26883632 W
PL13W             10.87646866 W
SFO2              400.1316005 MHz
SI                       32768
SF                100.6127587 MHz
WDW                        EM
SSB                         0
LB                     1.00 Hz
GB                          0
PC                     1.40
```

3n

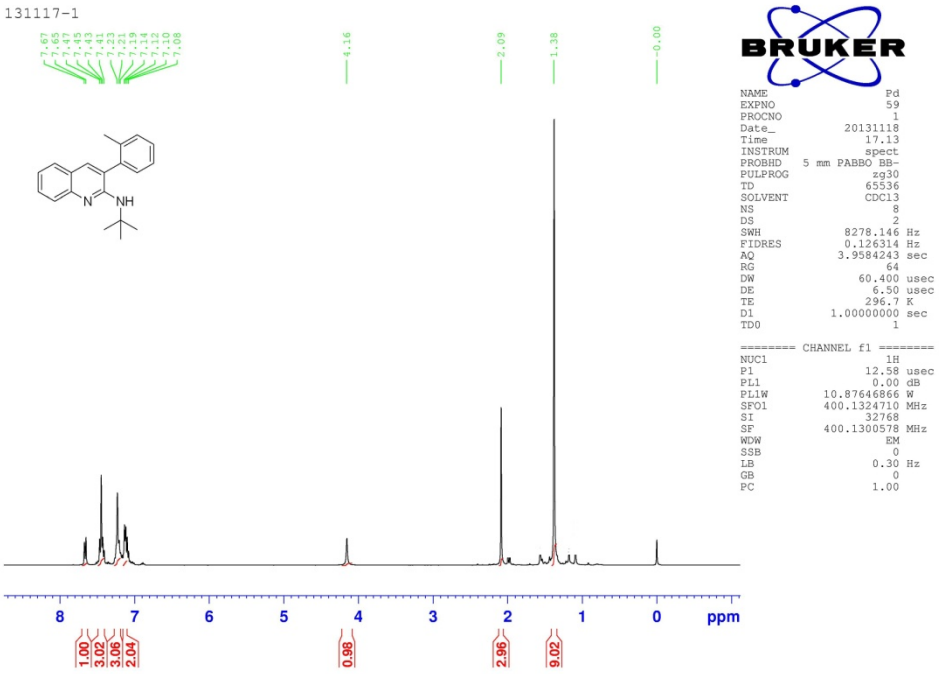


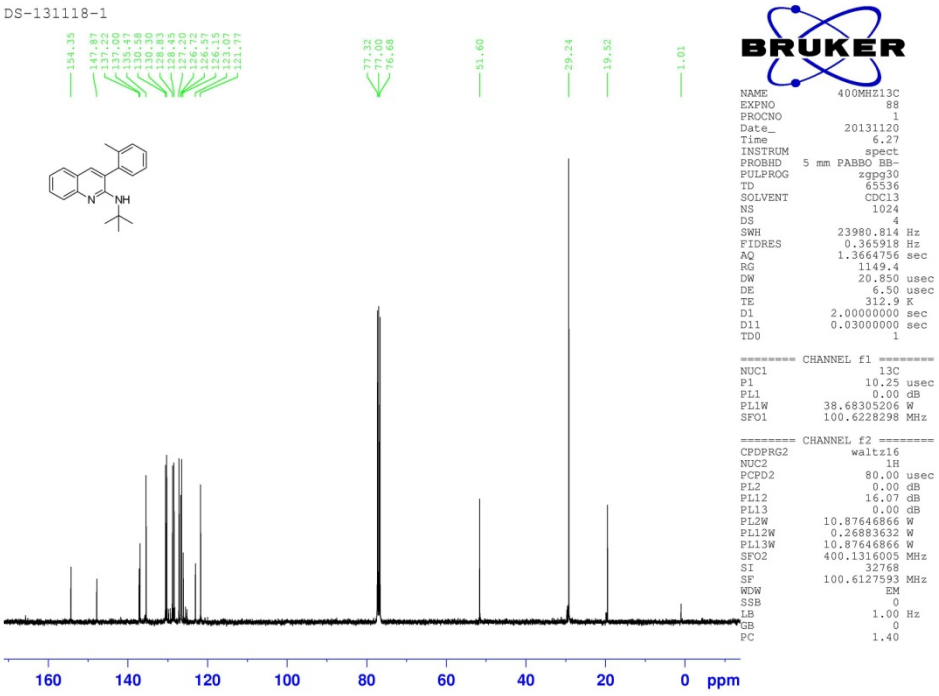

```
NAME                 Pd
EXPNO                 5
PROCNO                1
Date_          20131118
Time               9.33
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                   64
DW               60.400 usec
DE                6.50 usec
TE                296.8 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         400.1300553 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



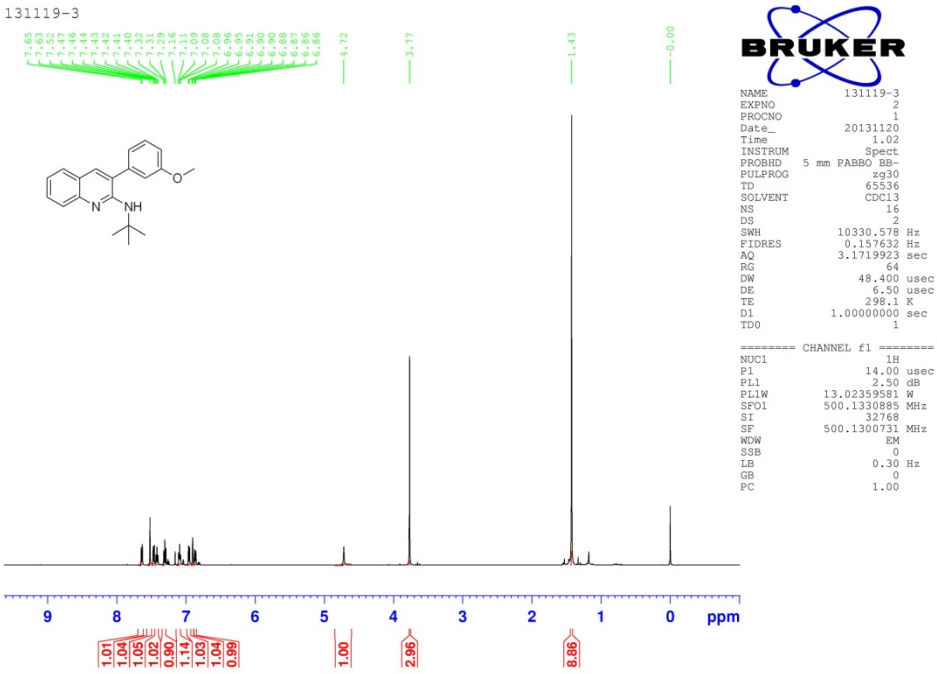

```
NAME           400MHz13C
EXPNO                103
PROCNO                 1
Date_          20131122
Time               0.14
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           23980.814 Hz
FIDRES        0.365918 Hz
AQ            1.3664756 sec
RG               1149.4
DW               20.850 usec
DE                6.50 usec
TE                310.7 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.25 usec
PL1                0.00 dB
PL1W        38.68305206 W
SFO1       100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2                0.00 dB
PL12              16.07 dB
PL13               0.00 dB
PL2W        10.87646866 W
PL12W        0.26883632 W
PL13W       10.87646866 W
SFO2       400.1316005 MHz
SI                32768
SF         100.6127610 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

3o

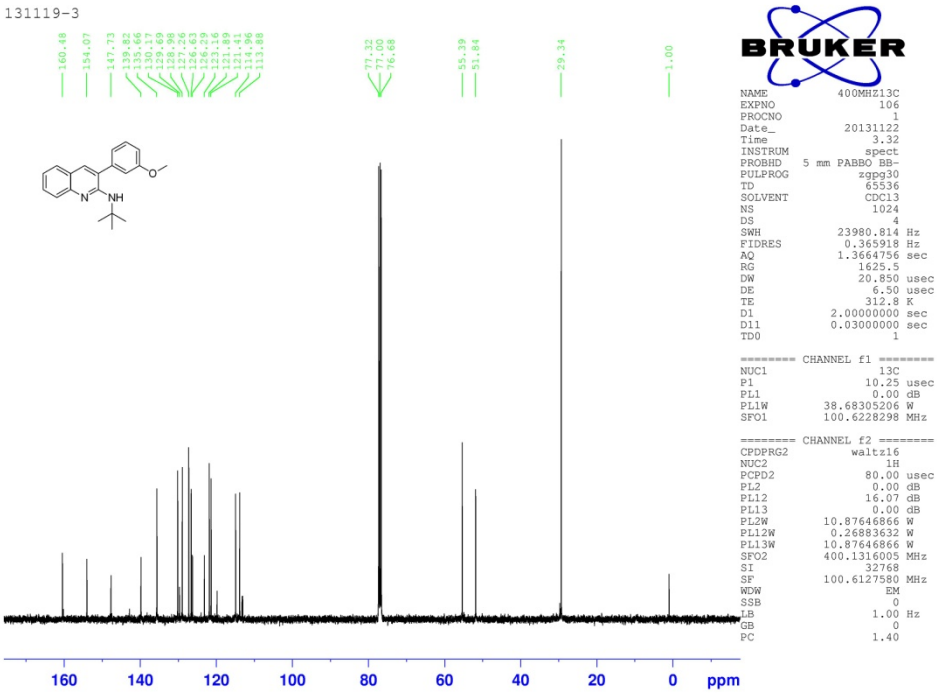

131117-1
7.67
7.65
7.47
7.45
7.43
7.41
7.21
7.19
7.14
7.12
7.10
7.08
4.16
2.09
1.38
0.00
NH
8
7
6
5
4
3
2
1
0
ppm
1.00
3.02
3.06
2.04
0.98
2.96
9.02
BRUKER
NAME Pd
EXPNO 59
PROCNO 1
Date_ 20131118
Time 17.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 64
DW 60.400 usec
DE 6.50 usec
TE 296.7 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 400.1300578 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


DS-131118-1
154.35
147.87
137.22
137.00
133.47
130.38
130.30
128.83
128.45
127.20
126.72
126.57
126.15
123.97
121.77
77.32
77.00
76.68
51.60
29.24
19.52
1.01
NH
160
140
120
100
80
60
40
20
0
ppm
BRUKER
NAME 400MHZ13C
EXPNO 88
PROCNO 1
Date_ 20131120
Time 6.27
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1149.4
DW 20.850 usec
DE 6.50 usec
TE 312.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.25 usec
PL1 0.00 dB
PL1W 38.68305206 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 16.07 dB
PL13 0.00 dB
PL2W 10.87646866 W
PL12W 0.26883632 W
PL13W 10.87646866 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127593 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

3q

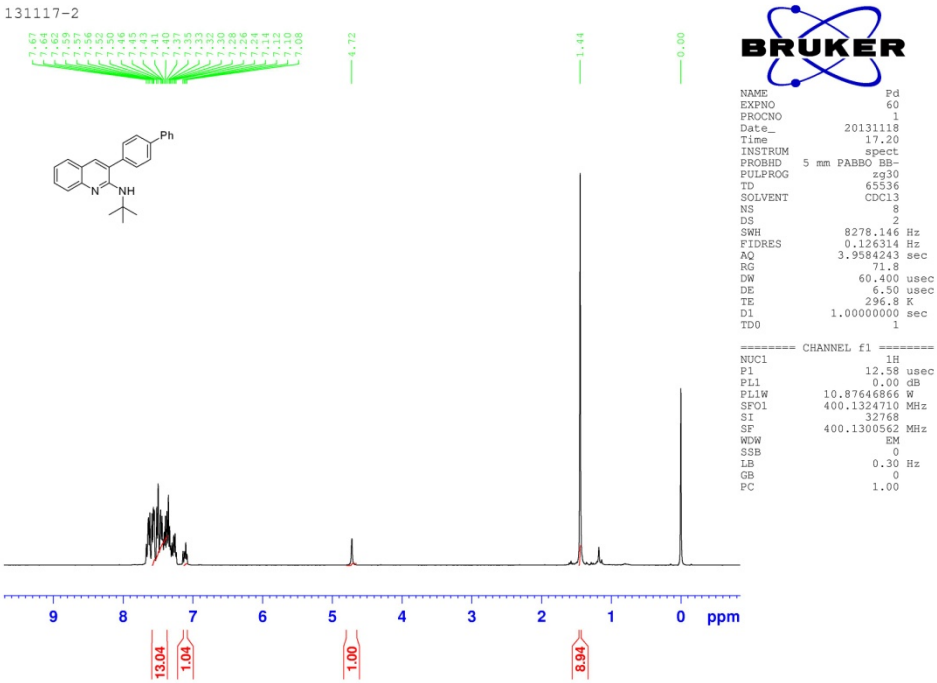


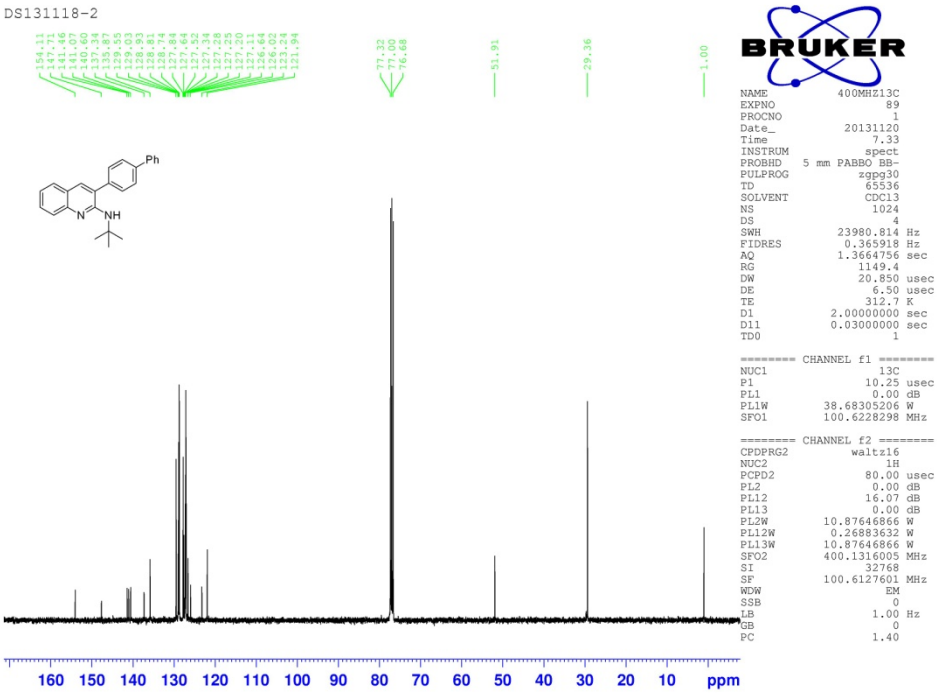

```
NAME                131119-3
EXPNO                      2
PROCNO                     1
Date_               20131120
Time                    1.02
INSTRUM                Spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        16
DS                         2
SWH                10330.578 Hz
FIDRES             0.157632 Hz
AQ                 3.1719923 sec
RG                        64
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                 500.1300731 MHz
WDW                        EM
SSB                         0
LB                      0.30 Hz
GB                          0
PC                      1.00
```



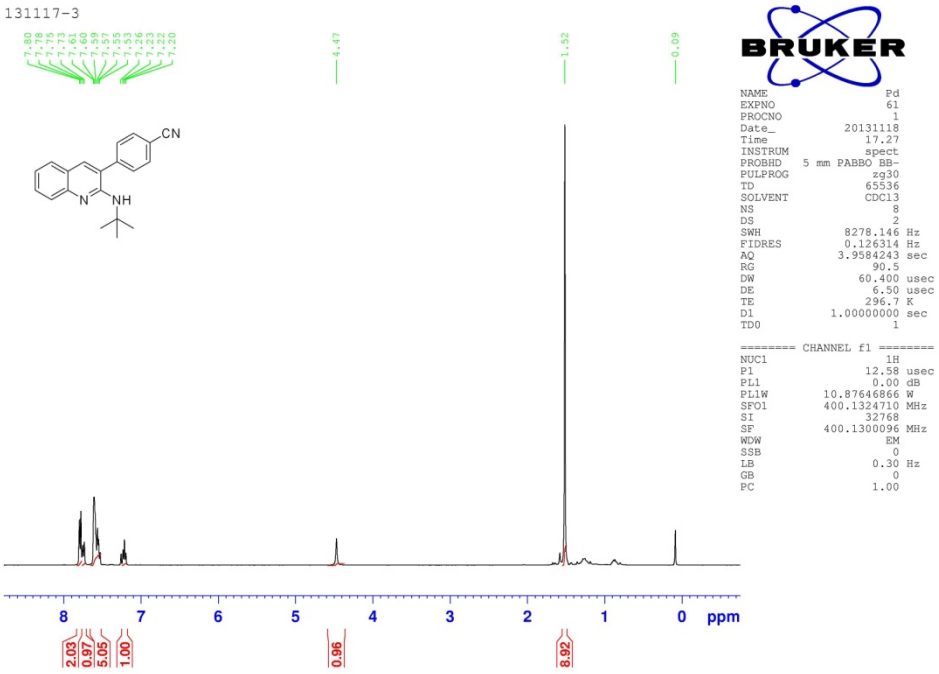

```
NAME              400MHz13C
EXPNO                   106
PROCNO                    1
Date_              20131122
Time                   3.32
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     1024
DS                        4
SWH                23980.814 Hz
FIDRES             0.365918 Hz
AQ                 1.3664756 sec
RG                    1625.5
DW                   20.850 usec
DE                    6.50 usec
TE                    312.8 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     13C
P1                     10.25 usec
PL1                     0.00 dB
PL1W               38.68305206 W
SFO1               100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                  80.00 usec
PL2                     0.00 dB
PL12                   16.07 dB
PL13                    0.00 dB
PL2W               10.87646866 W
PL12W               0.26883632 W
PL13W              10.87646866 W
SFO2               400.1316005 MHz
SI                        32768
SF                 100.6127580 MHz
WDW                        EM
SSB                         0
LB                      1.00 Hz
GB                          0
PC                      1.40
```

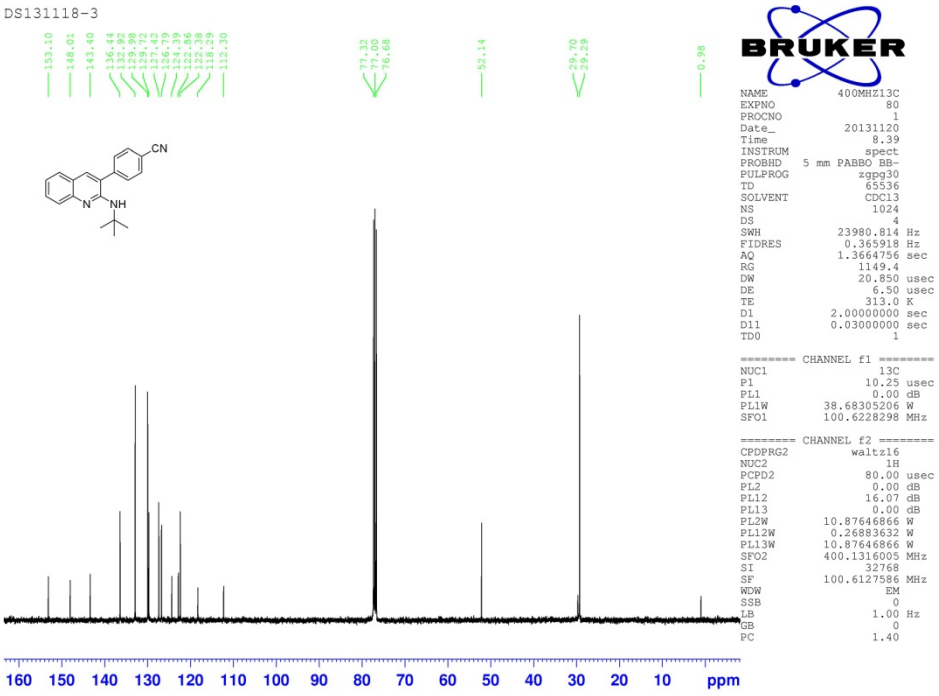
3s


131117-2
7.67
7.64
7.62
7.59
7.57
7.56
7.52
7.50
7.46
7.45
7.43
7.41
7.40
7.37
7.35
7.33
7.32
7.30
7.28
7.26
7.14
7.13
7.12
7.10
7.08
4.72
1.44
0.00
Ph
NH
9
8
7
6
5
4
3
2
1
0
ppm
13.04
1.04
1.00
8.94
BRUKER
NAME Pd
EXPNO 60
PROCNO 1
Date_ 20131118
Time 17.20
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 71.8
DW 60.400 usec
DE 6.50 usec
TE 296.8 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 400.1300562 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

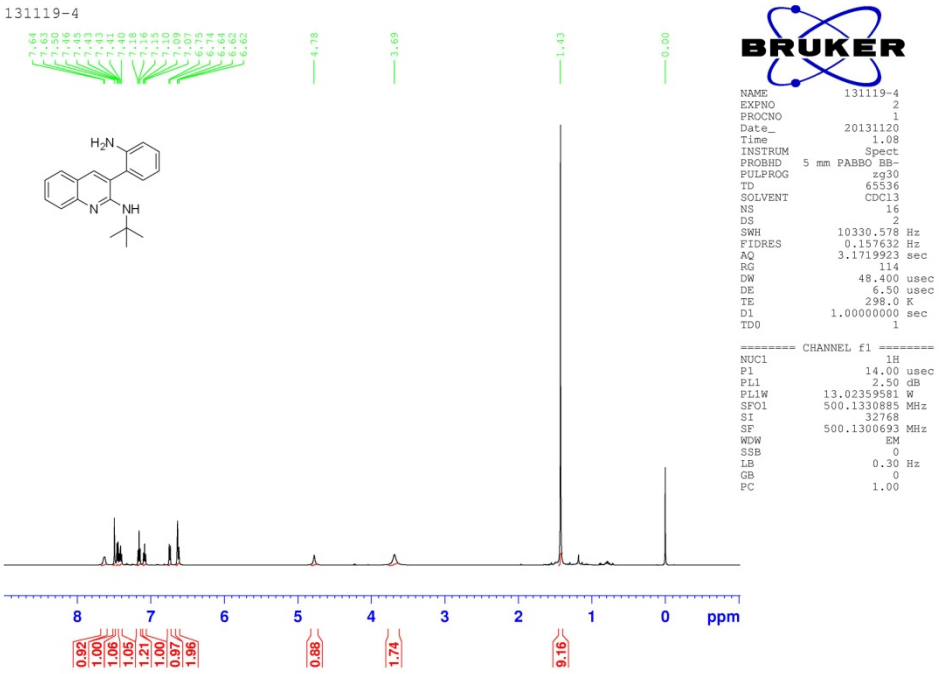

DS131118-2
154.11
147.71
141.46
140.07
140.00
137.34
133.87
129.55
129.03
128.81
128.74
127.84
127.52
127.34
127.28
127.25
127.10
127.11
126.64
126.02
123.34
121.94
77.32
77.00
76.68
51.91
29.36
1.00
Ph
NH
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm
BRUKER
NAME 400MHz13C
EXPNO 89
PROCNO 1
Date_ 20131120
Time 7.33
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1149.4
DW 20.850 usec
DE 6.50 usec
TE 312.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.25 usec
PL1 0.00 dB
PL1W 38.68305206 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 16.07 dB
PL13 0.00 dB
PL2W 10.87646866 W
PL12W 0.26883632 W
PL13W 10.87646866 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127601 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

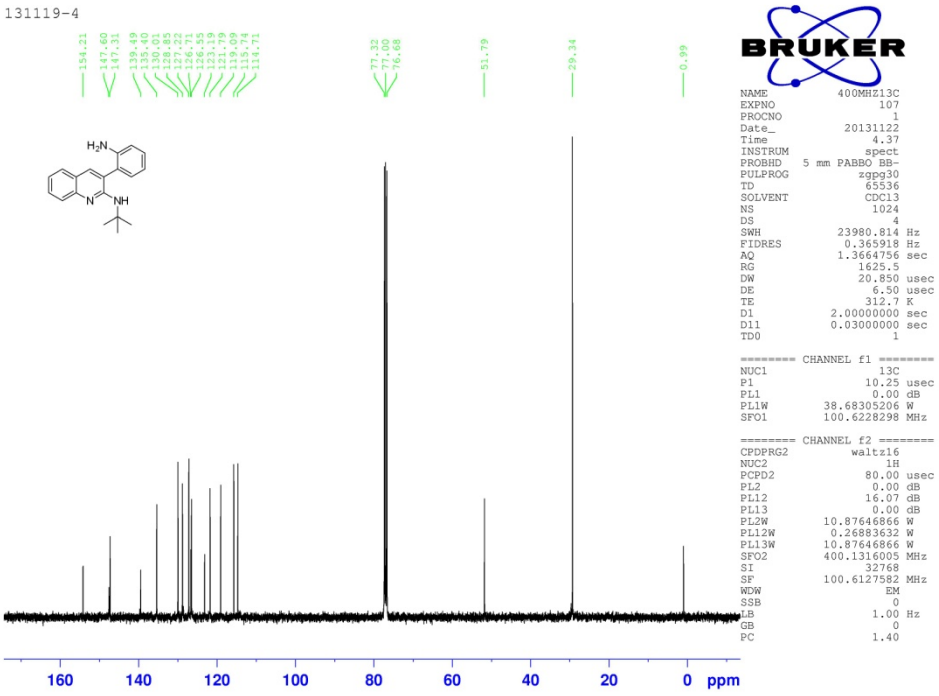
3t


131117-3
7.80
7.78
7.75
7.73
7.61
7.60
7.59
7.57
7.55
7.53
7.26
7.23
7.22
7.20
4.47
1.52
0.09
CN
N
NH
8
7
6
5
4
3
2
1
0
ppm
2.03
0.97
5.05
1.00
0.96
8.92
BRUKER
NAME Pd
EXPNO 61
PROCNO 1
Date_ 20131118
Time 17.27
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 90.5
DW 60.400 usec
DE 6.50 usec
TE 296.7 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 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

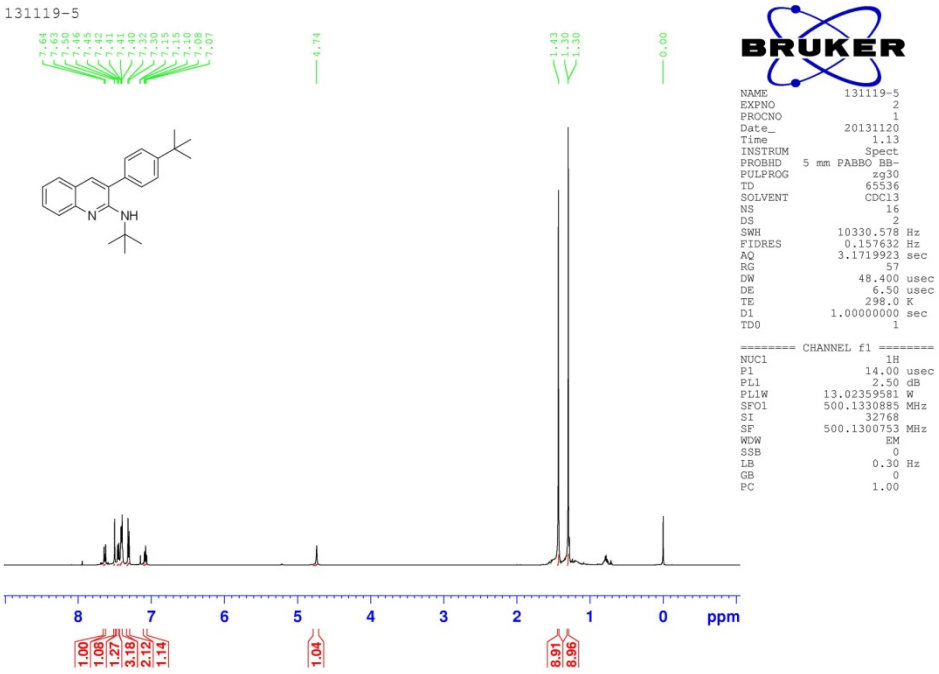

DS131118-3
153.10
148.01
143.40
136.44
132.92
129.98
129.72
127.42
126.79
124.39
122.86
118.28
112.30
77.32
77.00
76.68
52.14
29.70
29.29
0.98
CN
N
NH
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm
BRUKER
NAME 400MHZ13C
EXPNO 80
PROCNO 1
Date_ 20131120
Time 8.39
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1149.4
DW 20.850 usec
DE 6.50 usec
TE 313.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.25 usec
PL1 0.00 dB
PL1W 38.68305206 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 16.07 dB
PL13 0.00 dB
PL2W 10.87646866 W
PL12W 0.26883632 W
PL13W 10.87646866 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127586 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

3p

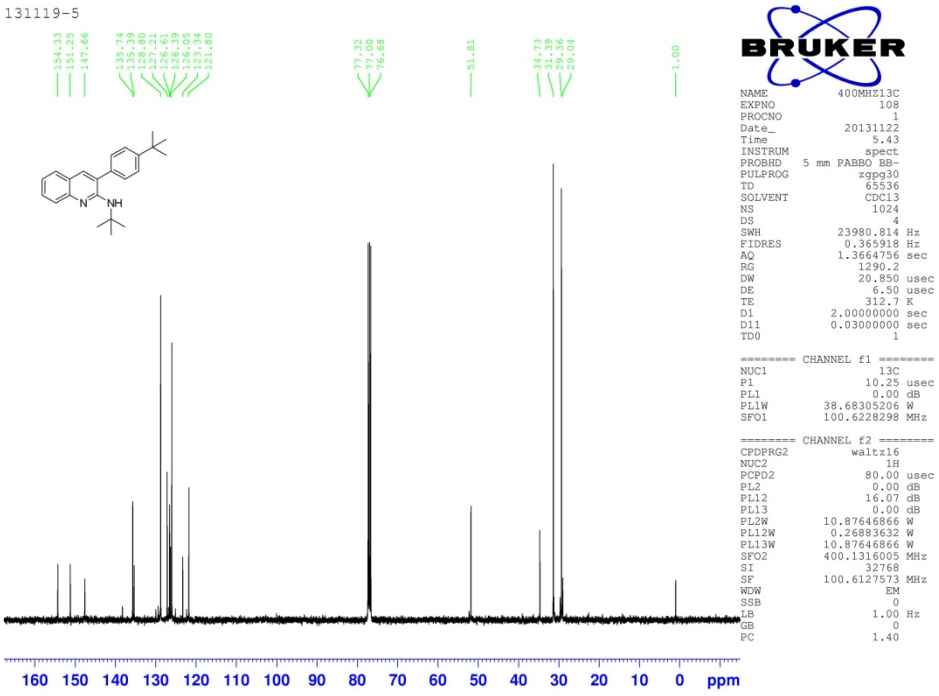

131119-4
7.64
7.63
7.50
7.46
7.45
7.43
7.43
7.41
7.40
7.18
7.16
7.10
7.09
7.07
6.75
6.74
6.64
6.62
6.62
4.78
3.69
1.43
0.00
H2N
N
NH
8
7
6
5
4
3
2
1
0
ppm
0.92
1.00
1.08
1.05
1.21
1.00
0.97
1.96
0.88
1.74
9.16
BRUKER
NAME 131119-4
EXPNO 2
PROCNO 1
Date_ 20131120
Time 1.08
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
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 298.0 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 500.1300693 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


131119-4
154.21
147.60
147.31
139.49
135.40
130.41
128.85
127.22
126.71
126.55
122.19
121.79
119.09
115.74
114.71
77.32
77.00
76.68
51.79
29.34
0.99
H2N
N
NH
160
140
120
100
80
60
40
20
0
ppm
BRUKER
NAME 400MHZ13C
EXPNO 107
PROCNO 1
Date_ 20131122
Time 4.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1625.5
DW 20.850 usec
DE 6.50 usec
TE 312.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.25 usec
PL1 0.00 dB
PL1W 38.68305206 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 16.07 dB
PL13 0.00 dB
PL2W 10.87646866 W
PL12W 0.26883632 W
PL13W 10.87646866 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127582 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

3r

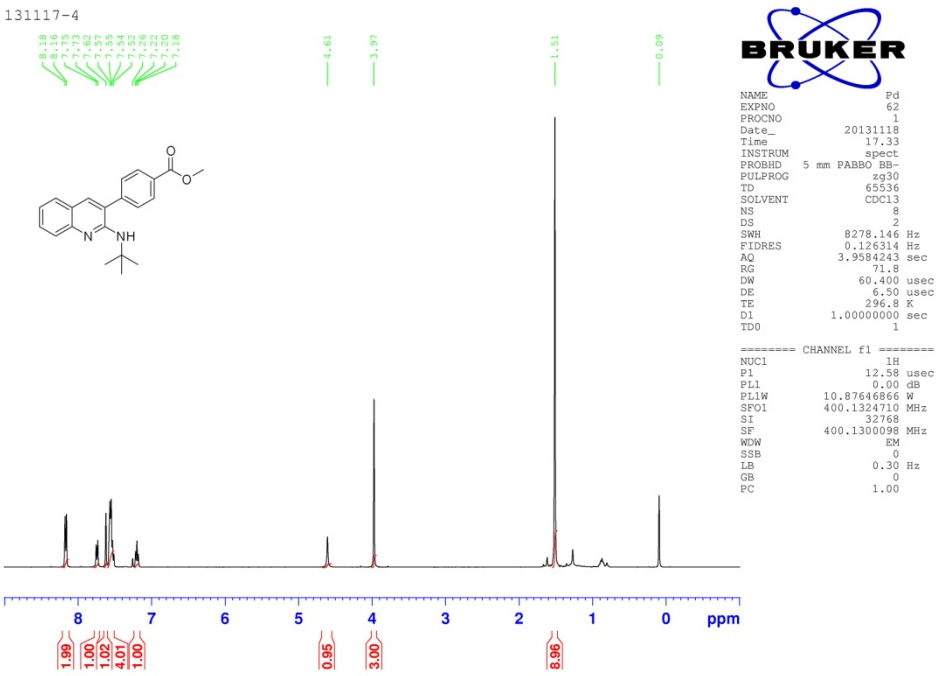



```
BRUKER
NAME                131119-5
EXPNO                      2
PROCNO                     1
Date_               20131120
Time                    1.13
INSTRUM                 Spect
PROBHD    5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                10330.578 Hz
FIDRES             0.157632 Hz
AQ                 3.1719923 sec
RG                        57
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                     32768
SF                500.1300753 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```

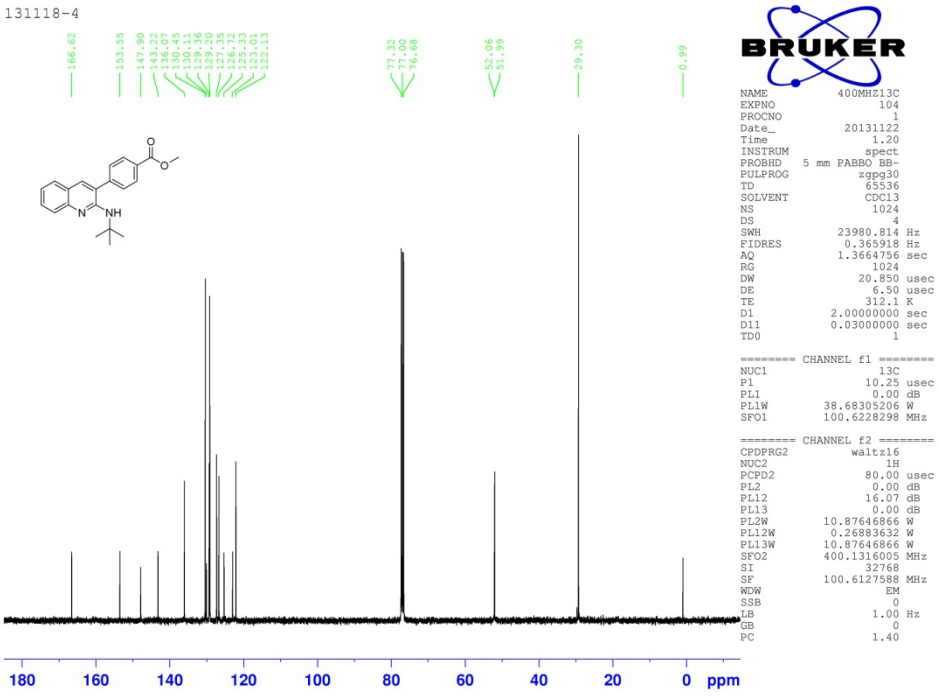



```
BRUKER
NAME              400MHZ13C
EXPNO                   108
PROCNO                    1
Date_               20131122
Time                    5.43
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG              zgpg30
TD                     65536
SOLVENT                CDCl3
NS                      1024
DS                         4
SWH                23980.814 Hz
FIDRES             0.365918 Hz
AQ                 1.3664756 sec
RG                    1290.2
DW                    20.850 usec
DE                     6.50 usec
TE                     312.7 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      13C
P1                     10.25 usec
PL1                     0.00 dB
PL1W              38.68305206 W
SFO1              100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                      1H
PCPD2                  80.00 usec
PL2                     0.00 dB
PL12                   16.07 dB
PL13                    0.00 dB
PL2W              10.87646866 W
PL12W              0.26883632 W
PL13W             10.87646866 W
SFO2              400.1316005 MHz
SI                     32768
SF                100.6127573 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40
```

3u

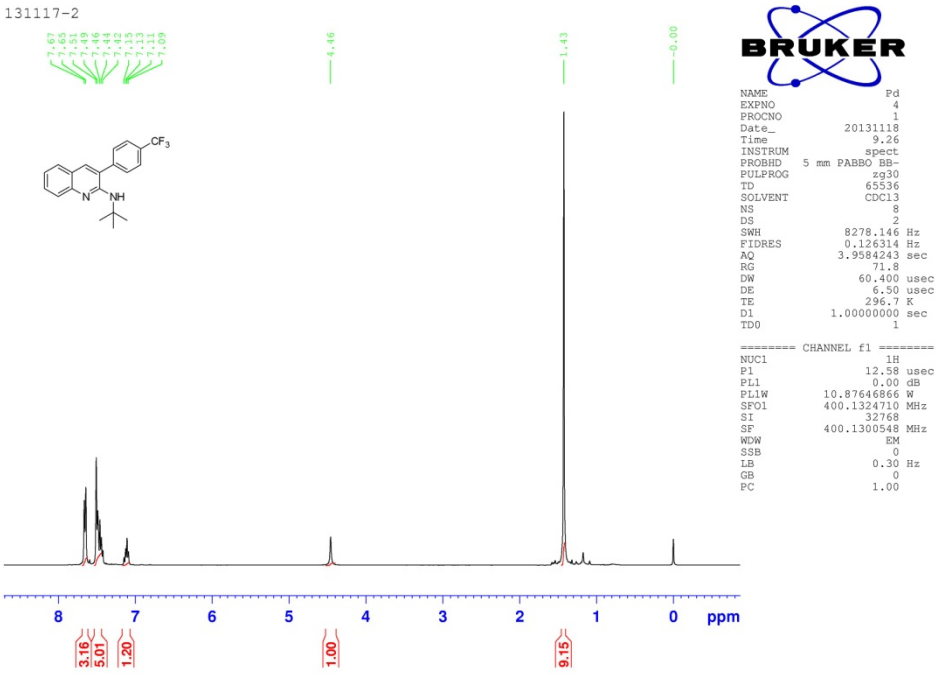


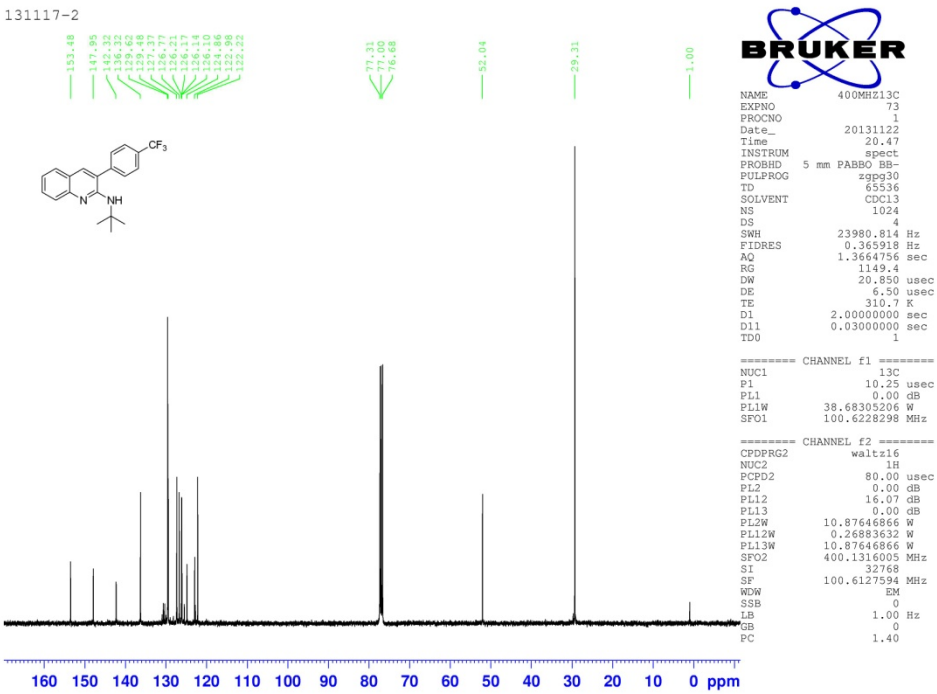

```
NAME                Pd
EXPNO               62
PROCNO              1
Date_               20131118
Time                17.33
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG             zg30
TD                  65536
SOLVENT             CDC13
NS                  8
DS                  2
SWH                 8278.146 Hz
FIDRES              0.126314 Hz
AQ                  3.9584243 sec
RG                  71.8
DW                  60.400 usec
DE                  6.50 usec
TE                  296.8 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                  400.1300098 MHz
WDW                 EM
SSB                 0
LB                  0.30 Hz
GB                  0
PC                  1.00
```



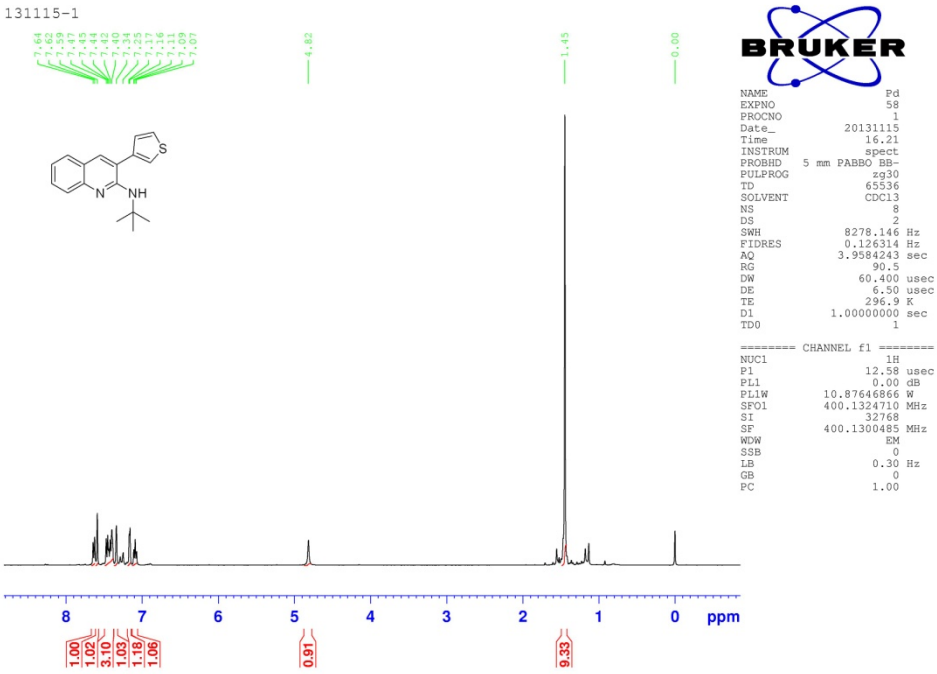

```
NAME                400MHZ13C
EXPNO               104
PROCNO              1
Date_               20131122
Time                1.20
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                  65536
SOLVENT             CDC13
NS                  1024
DS                  4
SWH                 23980.814 Hz
FIDRES              0.365918 Hz
AQ                  1.3664756 sec
RG                  1024
DW                  20.850 usec
DE                  6.50 usec
TE                  312.1 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                 1

======== CHANNEL f1 ========
NUC1                13C
P1                  10.25 usec
PL1                 0.00 dB
PL1W                38.68305206 W
SFO1                100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                1H
PCPD2               80.00 usec
PL2                 0.00 dB
PL12                16.07 dB
PL13                0.00 dB
PL2W                10.87646866 W
PL12W               0.26883632 W
PL13W               10.87646866 W
SFO2                400.1316005 MHz
SI                  32768
SF                  100.6127588 MHz
WDW                 EM
SSB                 0
LB                  1.00 Hz
GB                  0
PC                  1.40
```

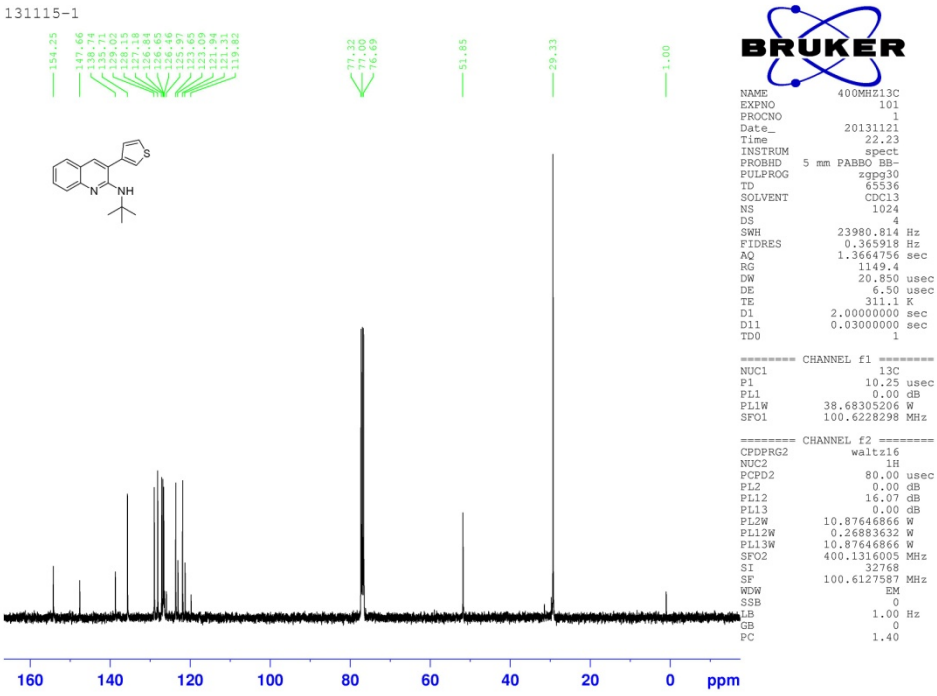
3v


131117-2
7.67
7.65
7.51
7.48
7.46
7.44
7.42
7.13
7.11
7.09
4.46
1.43
0.00
CF3
N
NH
8
7
6
5
4
3
2
1
0
ppm
3.16
5.01
1.20
1.00
9.15
BRUKER
NAME Pd
EXPNO 4
PROCNO 1
Date_ 20131118
Time 9.26
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 71.8
DW 60.400 usec
DE 6.50 usec
TE 296.7 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 400.1300548 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

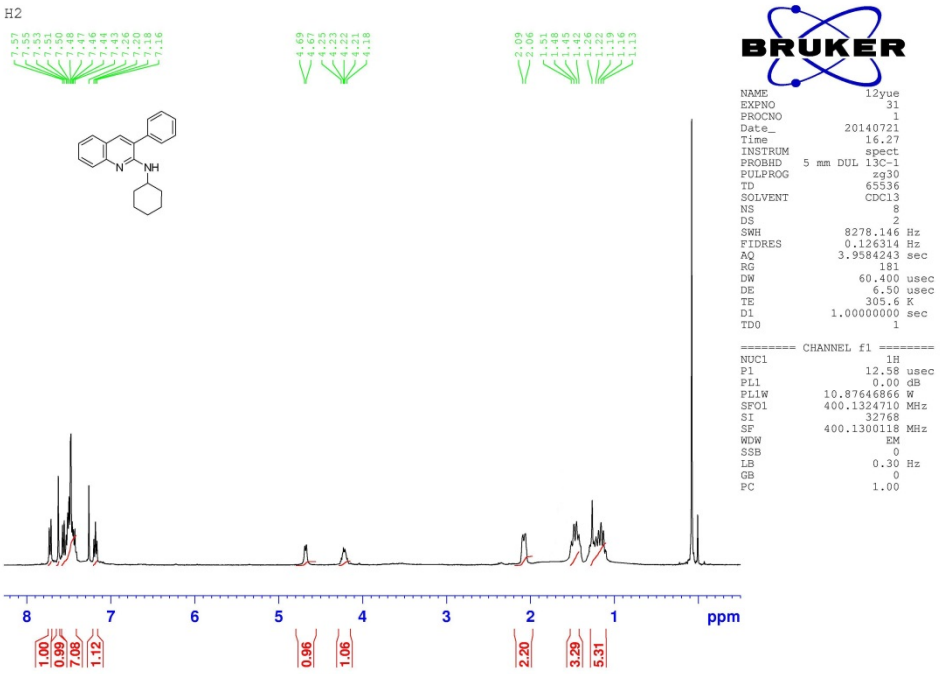

131117-2
153.48
147.95
142.32
136.22
129.68
129.48
127.37
126.77
126.21
126.14
126.10
124.86
122.98
122.22
77.31
77.00
76.68
52.04
29.31
1.00
CF3
N
NH
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm
BRUKER
NAME 400MHZ13C
EXPNO 73
PROCNO 1
Date_ 20131122
Time 20.47
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1149.4
DW 20.850 usec
DE 6.50 usec
TE 310.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.25 usec
PL1 0.00 dB
PL1W 38.68305206 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 16.07 dB
PL13 0.00 dB
PL2W 10.87646866 W
PL12W 0.26883632 W
PL13W 10.87646866 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127594 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

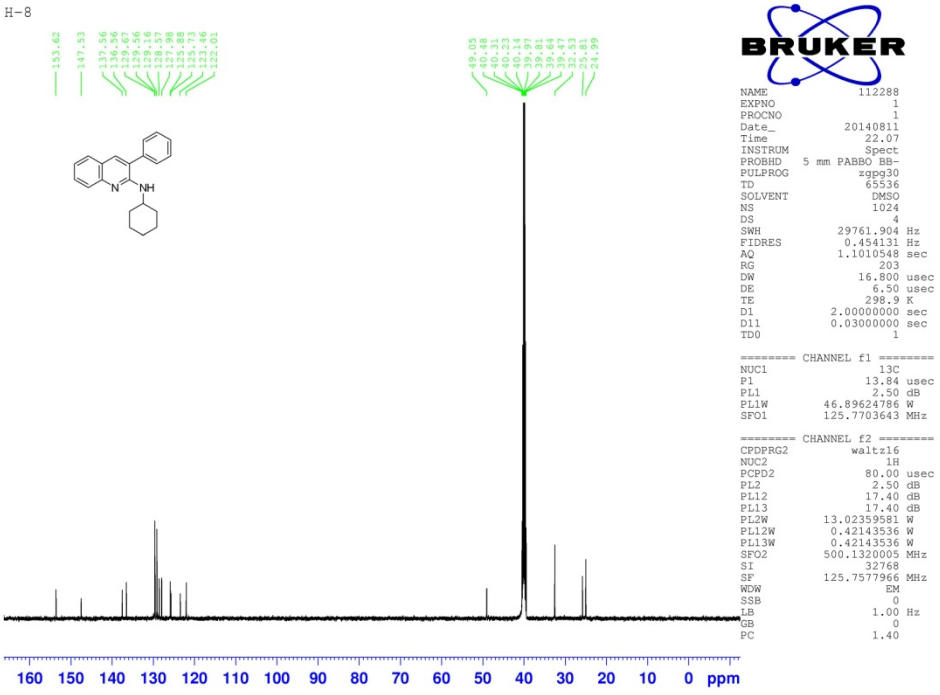
3w


131115-1
7.64
7.62
7.59
7.45
7.43
7.44
7.42
7.40
7.38
7.25
7.17
7.16
7.01
7.00
7.07
4.82
1.45
0.00
S
N
NH
8
7
6
5
4
3
2
1
0
ppm
1.00
1.02
3.10
1.03
1.18
1.06
0.91
9.33
BRUKER
NAME Pd
EXPNO 58
PROCNO 1
Date_ 20131115
Time 16.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 90.5
DW 60.400 usec
DE 6.50 usec
TE 296.9 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 400.1300485 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

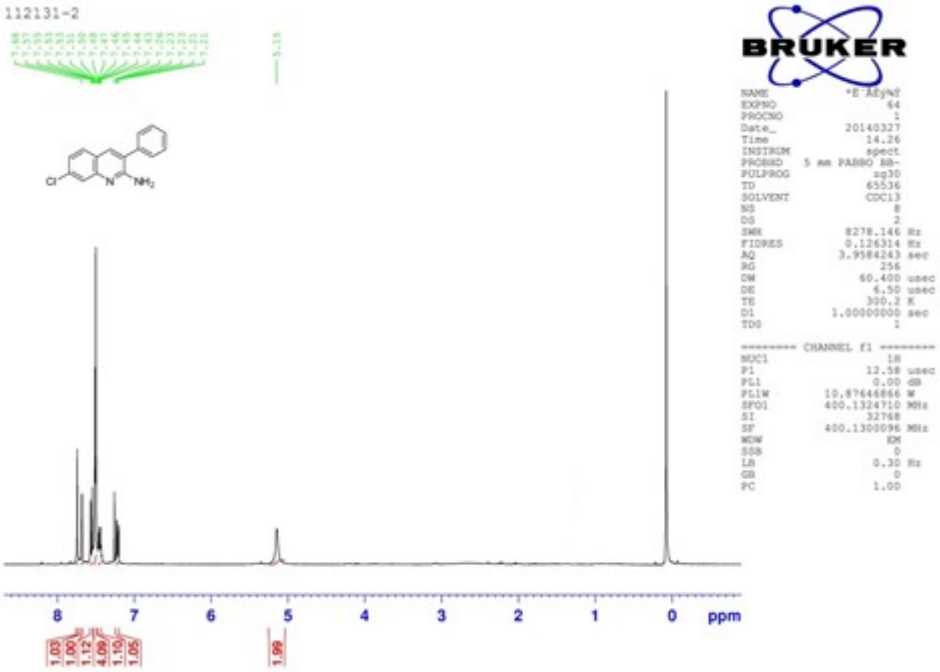

131115-1
154.25
147.66
138.74
135.71
130.02
128.15
127.18
126.84
126.65
126.46
125.97
123.65
123.09
122.94
121.31
119.82
77.32
77.00
76.69
51.85
29.33
1.00
S
N
NH
160
140
120
100
80
60
40
20
0
ppm
BRUKER
NAME 400MHZ13C
EXPNO 101
PROCNO 1
Date_ 20131121
Time 22.23
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1149.4
DW 20.850 usec
DE 6.50 usec
TE 311.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.25 usec
PL1 0.00 dB
PL1W 38.68305206 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 16.07 dB
PL13 0.00 dB
PL2W 10.87646866 W
PL12W 0.26883632 W
PL13W 10.87646866 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127587 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

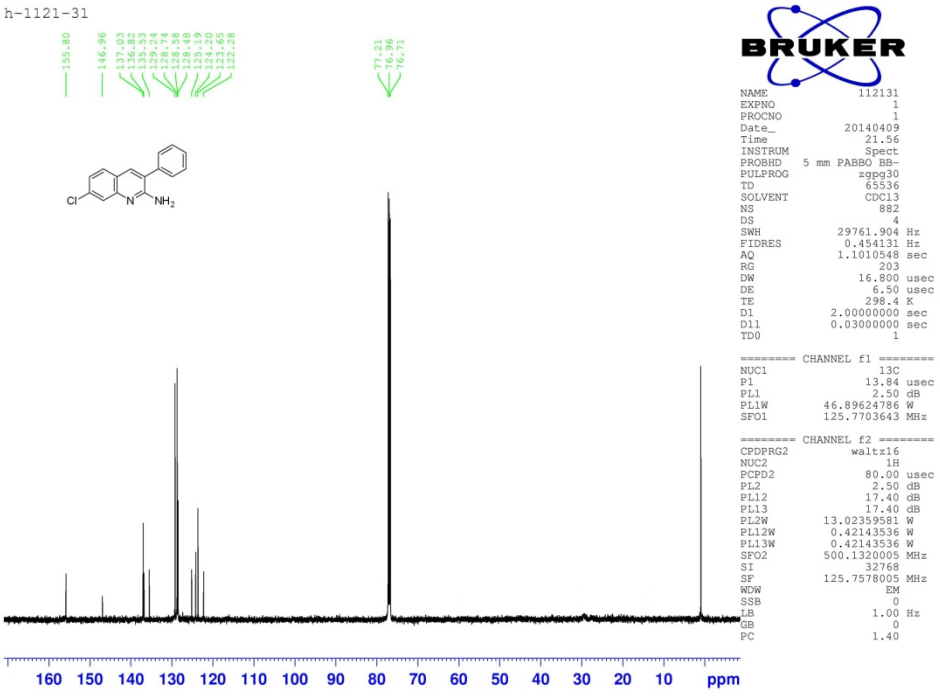
3x


H2
7.57
7.55
7.53
7.51
7.50
7.48
7.47
7.46
7.44
7.43
7.26
7.20
7.18
7.16
4.69
4.67
4.25
4.23
4.22
4.21
4.18
2.09
2.06
1.51
1.48
1.45
1.42
1.26
1.22
1.19
1.16
1.13
NH
8
7
6
5
4
3
2
1
ppm
1.00
0.99
7.08
1.12
0.96
1.06
2.20
3.29
5.31
BRUKER
NAME 12yue
EXPNO 31
PROCNO 1
Date_ 20140721
Time 16.27
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 181
DW 60.400 usec
DE 6.50 usec
TE 305.6 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 400.1300118 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

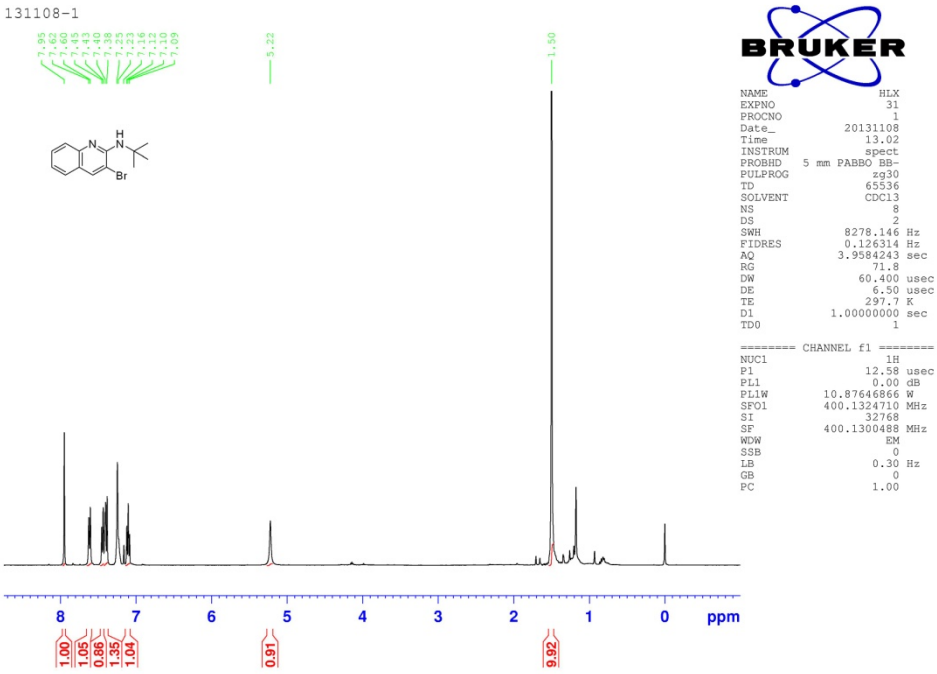

H-8
153.62
147.53
137.56
136.46
129.67
129.56
129.16
128.57
128.38
121.88
125.73
123.46
122.01
49.05
40.48
40.31
40.23
40.14
39.97
39.81
39.64
39.47
32.53
25.81
24.99
NH
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm
BRUKER
NAME 112288
EXPNO 1
PROCNO 1
Date_ 20140811
Time 22.07
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
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 298.9 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 125.7577966 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

4c

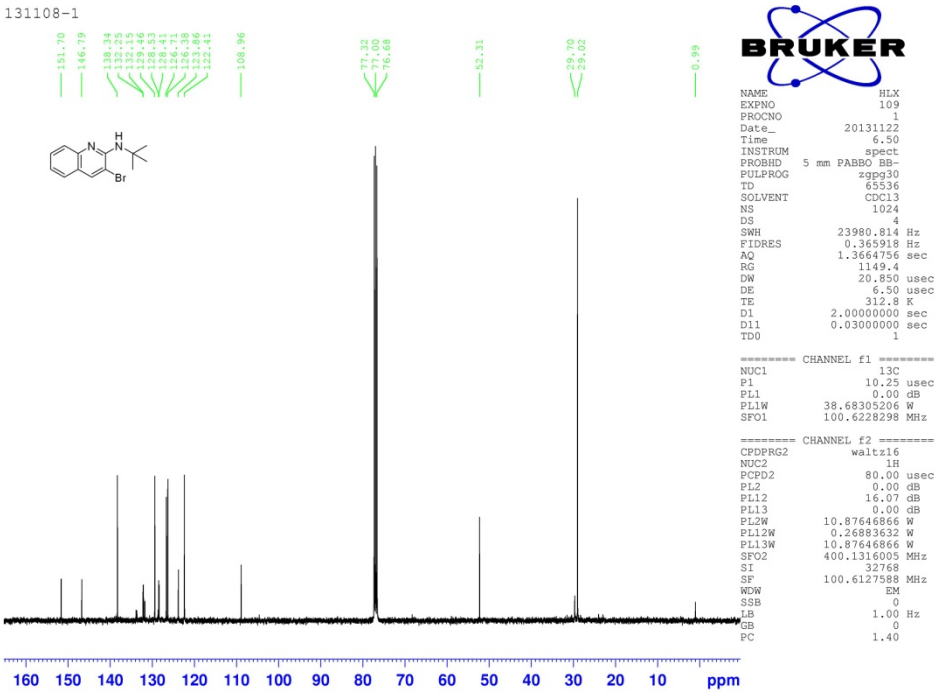

112131-2
BRUKER
NAME *E AEy%S
EXPNO 64
PROCNO 1
Date_ 20140327
Time 14.26
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 256
DW 60.400 usec
DE 6.50 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87644866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8 7 6 5 4 3 2 1 0 ppm
1.03 1.00 1.12 4.09 1.10 1.05
1.99


h-1121-31
155.80 146.96 137.03 136.82 135.53 129.24 128.74 128.58 128.48 128.19 124.20 123.65 122.28
77.21 76.96 76.71
BRUKER
NAME 112131
EXPNO 1
PROCNO 1
Date_ 20140409
Time 21.56
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 882
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 298.4 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 125.7578005 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

5.




```
NAME                  HLX
EXPNO                  31
PROCNO                  1
Date_            20131108
Time                13.02
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDC13
NS                      8
DS                      2
SWH              8278.146 Hz
FIDRES           0.126314 Hz
AQ               3.9584243 sec
RG                   71.8
DW                60.400 usec
DE                 6.50 usec
TE                297.7 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           400.1300488 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                 1.00
```




```
NAME                  HLX
EXPNO                 109
PROCNO                  1
Date_            20131122
Time                 6.50
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CDC13
NS                   1024
DS                      4
SWH             23980.814 Hz
FIDRES           0.365918 Hz
AQ               1.3664756 sec
RG                 1149.4
DW                20.850 usec
DE                 6.50 usec
TE                312.8 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.25 usec
PL1                 0.00 dB
PL1W         38.68305206 W
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                 0.00 dB
PL12               16.07 dB
PL13                0.00 dB
PL2W         10.87646866 W
PL12W         0.26883632 W
PL13W        10.87646866 W
SFO2         400.1316005 MHz
SI                  32768
SF           100.6127588 MHz
WDW                    EM
SSB                     0
LB                 1.00 Hz
GB                      0
PC                 1.40
```