Supporting Information

Highly Regio- and Diastereoselective Synthesis of Novel Tri- and Tetracyclic Perhydroquinoline Architectures Through an Intramolecular [3 + 2] Cycloaddition Reaction

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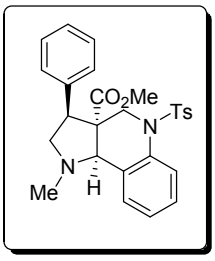
Experimental Section

General Remarks: Melting points were recorded on a Superfit (India) capillary melting point apparatus and were uncorrected. IR spectra were recorded on a Bruker-FT-IR spectrometer using solid samples as KBr plates. For compounds ^{1}H NMR (300 MHz) and ^{13}C NMR (75 MHz) spectra **9a-l**, **12a-h**, **15a-d** and **17a-d** were recorded in deuterochloroform ($CDCl_3$) on a Bruker 300 MHz spectrometer using tetramethylsilane (TMS, $\delta = 0$) as an internal standard at room temperature. Mass spectra were recorded on Agilent 1200 LC/MS-6110 mass spectrometer. The X-ray diffraction measurements were carried out on a Bruker AXS Kappa APEX 2 CCD difractometer. All the compounds were synthesized as racemates.

Typical experimental procedure for the synthesis of Compound 9a: To a stirred solution of methyl (2*E*)-2-{[*N*-(2-formylphenyl)(4-methylbenzene) sulfonamido]methyl}-3-phenylprop-2-enoate (**6a**) (1 mmol, 0.45 g) in acetonitrile (10 mL), *N*-methyl glycine (**7**) (1.1 mmol, 0.10 g) was added and allowed to stir under reflux condition over a period of 6 h. After completion of the reaction as indicated by TLC, the reaction mixture was concentrated and the resulting crude mass was diluted with water (20 mL) and extracted with ethyl acetate (3 x 10 mL). The combined organic layer thus obtained was washed with brine (3 x 10 mL) and dried over anhydrous Na_2SO_4. The organic layer was concentrated and the crude mass was recrystallized using ethyl acetate : hexane mixture (2:8) to provide **9a** as a colorless solid in 94% yield.

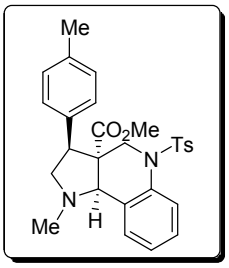
Methyl 1-methyl-5-[(4-methylbenzene)sulfonyl]-3-phenyl-1*H*,2*H*,3*H*,3a*H*,4*H*,5*H*,9b*H*-pyrrolo[3,2-c]quinoline-3a-carboxylate (9a):



Yield: 94%; mp: 226-228°C; IR (KBr): 1754, 1693 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.34 (s, 3H), 2.36 (s, 3H), 2.91 (t, 1H, J = 9.9 Hz), 3.35 (dd, 1H, J = 4.2, 9.9 Hz), 3.51 (d, 1H, J = 12.3 Hz), 3.64 (s, 1H), 3.69 (s, 3H), 3.89 (dd, 1H, J = 4.2, 9.9 Hz), 3.96 (d, 1H, J = 12.6 Hz), 6.96–7.58 (m, 13H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 16.25, 34.64, 42.30, 43.80, 47.17, 48.79, 54.45,

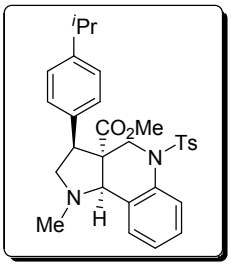
62.98, 114.48, 117.20, 119.19, 121.76, 122.17, 123.05, 123.23, 123.56, 124.29, 126.06, 131.52, 132.04, 132.50, 138.22, 168.30; MS (m/z): 478 (M^++1); Anal. Calcd. for $C_{27}H_{28}N_2O_4S$: C, 68.04; H, 5.92; N, 5.88; Found: C, 68.13; H, 5.99; N, 6.01.

Methyl 1-methyl-5-[(4-methylbenzene)sulfonyl]-3-(4-methylphenyl)-1*H*,2*H*,3*H*,3a*H*,4*H*,5*H*,9b*H*-pyrrolo[3,2-c]quinoline-3a-carboxylate (9b)



Yield: 92%; mp: 229-231°C; IR (KBr): 1740, 1698 cm^{-1}; ^1H NMR: δ 2.20 (s, 3H), 2.30 (s, 3H), 2.35 (s, 3H), 2.89 (t, 1H, J = 9.6 Hz), 3.20 (d, 1H, J = 9.3 Hz), 3.44 (d, 1H, J = 12.6 Hz), 3.63 (s, 4H), 4.02 (t, 2H, J = 12.6 Hz), 6.89 – 7.54 (m, 12H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 19.72, 21.51, 40.30, 45.27, 47.65, 52.42, 62.10, 68.26, 119.43, 122.47, 125.94, 127.06, 127.19, 128.17, 128.45, 129.61, 130.65, 131.13, 136.76, 137.09, 137.52, 143.60, 149.61, 174.16; MS (m/z): 492 (M^++1); Anal. Calcd. for $C_{28}H_{30}N_2O_4S$: C, 68.55; H, 6.16; N, 5.71; Found: C, 68.66; H, 6.24; N, 5.84.

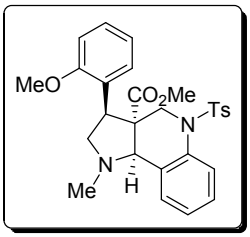
Methyl 1-methyl-5-[(4-methylbenzene)sulfonyl]-3-[4-(propan-2-yl)phenyl]-1*H*,2*H*,3*H*,3a*H*,4*H*,5*H*,9b*H*-pyrrolo[3,2-c]quinoline-3a-carboxylate (9c)



Yield: 91%; mp: 227-229°C; IR (KBr): 1732, 1698 cm^{-1}; ^1H NMR: δ 1.27 (d, 6H, J = 6.9 Hz), 2.31 (s, 3H), 2.36 (s, 3H), 2.81-2.99 (m, 2H), 3.34 (dd, 1H, J = 4.8, 9.9 Hz), 3.53 (d, 1H, J = 12.3

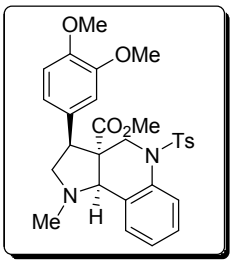
Hz), 3.64 (s, 1H), 3.68 (s, 3H), 3.87 (dd, 1H, *J* = 4.5, 10.2 Hz), 3.98 (d, 1H, *J* = 12.6 Hz), 6.96–7.59 (m, 12H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 21.51, 23.96, 24.00, 33.73, 39.85, 47.40, 48.82, 52.40, 54.12, 59.62, 68.35, 119.81, 122.46, 124.44, 126.54, 127.05, 128.34, 128.72, 129.51, 131.30, 134.85, 136.74, 137.16, 143.47, 147.94, 173.56; MS (m/z): 520 (M^++1); Anal. Calcd for $C_{30}H_{34}N_2O_4S$: C, 69.47; H, 6.61; N, 5.40; Found: C, 69.60; H, 6.73; N, 5.53.

Methyl 1-methyl-5-[(4-methylbenzene)sulfonyl]-3-(2-methoxyphenyl)-1*H*,2*H*,3*H*,3a*H*,4*H*, 5*H*,9b*H*-pyrrolo[3,2-c]quinoline-3a-carboxylate (9d)



Yield: 94%; mp: 223-225°C; IR (KBr): 1761, 1656 cm^{-1}; ^{1}H NMR: δ 2.29 (s, 3H), 2.36 (s, 3H), 2.89 (t, 1H, *J* = 9.9 Hz), 3.39 (dd, 1H, *J* = 4.8, 9.9 Hz), 3.51 (d, 1H, *J* = 12.3 Hz), 3.65 (s, 3H), 3.69 (s, 1H), 3.77 (s, 3H), 4.02 (d, 1H, *J* = 12.3 Hz), 4.37 (dd, 1H, *J* = 4.5, 10.2 Hz), 6.85–7.60 (m, 12H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 21.51, 39.86, 42.58, 46.76, 52.13, 53.21, 55.32, 58.92, 69.29, 110.34, 119.68, 120.37, 122.24, 124.11, 125.56, 127.17, 128.32, 128.42, 128.54, 129.47, 131.37, 136.71, 137.14, 143.37, 157.65, 173.49; MS (m/z): 508 (M^++1); Anal. Calcd. for $C_{28}H_{30}N_2O_5S$: C, 66.38; H, 5.97; N, 5.53; Found: C, 66.45; H, 6.09; N, 5.64.

Methyl 1-methyl-5-[(4-methylbenzene)sulfonyl]-3-(3,4-dimethoxyphenyl)-1*H*,2*H*,3*H*,3a*H*, 4*H*, 5*H*,9b*H*-pyrrolo[3,2-c]quinoline-3a-carboxylate (9e)



Yield: 95%; mp: 227-229°C; IR (KBr): 1777, 1687 cm^{-1}; ^{1}H NMR: δ 2.37 (s, 3H), 2.38 (s, 3H), 2.92 (t, 1H, *J* = 9.9 Hz), 3.32 (dd, 1H, *J* = 4.5, 10.2 Hz), 3.48 (d, 1H, *J* = 12.6 Hz), 3.67 (s, 1H),

3.70 (s, 3H), 3.84 (s, 4H), 3.89 (s, 3H), 4.04 (d, 1H, J = 12.9 Hz), 6.83–7.61 (m, 11H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 21.50, 40.20, 47.57, 49.10, 52.42, 54.90, 55.88, 55.95, 59.79, 67.97, 111.00, 112.11, 120.00, 121.02, 122.69, 125.33, 127.00, 128.20, 129.55, 130.20, 130.90, 136.83, 137.40, 143.49, 148.28, 148.81, 173.80; MS (m/z): 538 (M^++1); Anal. Calcd. for $C_{29}H_{32}N_2O_6S$: C, 64.91; H, 6.01; N, 5.22; Found: C, 64.99; H, 6.12; N, 5.36.

Crystal data for **Methyl 1-methyl-5-[(4-methylbenzene)sulfonyl]-3-(3,4-dimethoxyphenyl)-1*H*,2*H*,3*H*,3a*H*, 4*H*, 5*H*,9b*H*-pyrrolo[3,2-c]quinoline-3a-carboxylate (9e)**

Empirical formula, $C_{29}H_{32}N_2O_6S$; Formula weight, 537; crystal color, colorless; Single crystal X-ray structure of the molecule shown in ORTEP diagram (Figure 1). Detailed X-ray crystallographic data is available from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK (for pyrroloquinoline (**9e**) CCDC # **764210**).

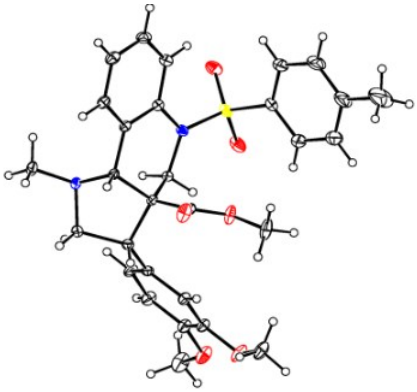
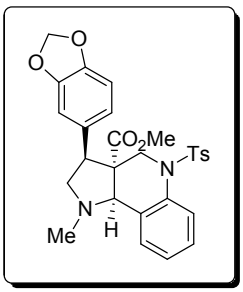


Figure 1. ORTEP diagram of compound 9e

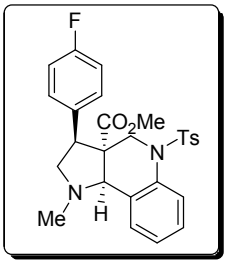
Methyl 3-(2H-1,3-benzodioxol-5-yl)-1-methyl-5-[(4-methylbenzene)sulfonyl]-1*H*,2*H*,3*H*, 3a*H*,4*H*,5*H*,9b*H*-pyrrolo[3,2-c]quinoline-3a-carboxylate (9f)



Yield: 95%; mp: 226-228°C; IR (KBr): 1781, 1595 cm^{-1}; ^{1}H NMR: δ 2.33 (s, 3H), 2.38 (s, 3H), 2.89 (t, 1H, J = 10.2 Hz), 3.25 (dd, 1H, J = 3.9, 9.9 Hz), 3.51 (d, 1H, J = 12.3 Hz), 3.61 (s, 1H),

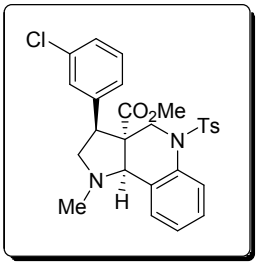
3.69 (s, 3H), 3.81 (dd, 1H, *J* = 3.9, 9.9 Hz), 4.05 (d, 1H, *J* = 12.6 Hz), 5.98 (s, 2H), 6.73–7.62 (m, 11H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 21.52, 39.91, 47.48, 48.85, 52.46, 54.12, 60.01, 68.05, 101.11, 108.15, 109.19, 119.72, 122.01, 122.48, 124.46, 127.01, 128.28, 129.59, 131.27, 131.49, 136.74, 137.35, 143.53, 146.80, 147.74, 173.58; MS (m/z): 522 (M^++1); Anal. Calcd. for $C_{28}H_{28}N_2O_6S$: C, 64.60; H, 5.42; N, 5.38; Found: C, 64.69; H, 5.52; N, 5.49.

Methyl 1-methyl-5-[(4-methylbenzene)sulfonyl]-3-(4-fluorophenyl)-1*H*,2*H*,3*H*,3a*H*,4*H*,5*H*,9b*H*-pyrrolo[3,2-c]quinoline-3a-carboxylate (9g)



Yield: 94%; mp: 230-232°C; IR (KBr): 1784, 1632 cm^{-1}; ^{1}H NMR: δ 2.36 (s, 3H), 2.37 (s, 3H), 2.92 (t, 1H, *J* = 9.9 Hz), 3.29 (dd, 1H, *J* = 3.9, 9.9 Hz), 3.46 (d, 1H, *J* = 12.3 Hz), 3.62 (s, 1H), 3.69 (s, 3H), 3.87 (dd, 1H, *J* = 3.6, 9.9 Hz), 3.94 (d, 1H, *J* = 12.6 Hz), 6.97–7.59 (m, 12H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 21.51, 39.91, 47.68, 48.15, 52.51, 53.82, 60.05, 67.94, 115.33 (d, *J* = 21.1 Hz), 119.61, 122.49, 124.36, 126.93, 128.30, 129.62, 130.36 (d, *J* = 7.9 Hz), 131.30, 133.62, 137.04 (d, *J* = 48.3 Hz), 143.59, 173.51. MS (m/z): 496 (M^++1); Anal. Calcd. for $C_{27}H_{27}FN_2O_4S$: C, 65.57; H, 5.50; N, 5.66; Found: C, 65.68; H, 5.64; N, 5.79.

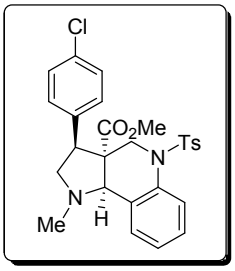
Methyl 1-methyl-5-[(4-methylbenzene)sulfonyl]-3-(3-chlorophenyl)-1*H*,2*H*,3*H*,3a*H*,4*H*,5*H*,9b*H*-pyrrolo[3,2-c]quinoline-3a-carboxylate (9h)



Yield: 90%; mp: 223-225°C; IR (KBr): 1729, 1686 cm^{-1}; ^{1}H NMR: δ 2.36 (s, 6H), 2.91 (t, 1H, *J* = 9.3 Hz), 3.27 (t, 1H, *J* = 9.9 Hz), 3.46 (d, 1H, *J* = 12.3 Hz), 3.62 (s, 1H), 3.69 (s, 3H), 3.85 (d,

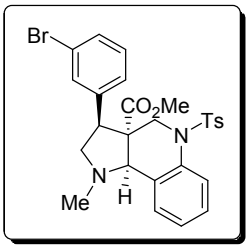
1H, *J* = 6.6 Hz), 3.94 (d, 1H, *J* = 12.3 Hz), 6.91–7.78 (m, 12H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 21.51, 39.90, 47.67, 48.25, 52.53, 53.85, 59.87, 67.90, 119.64, 122.54, 124.35, 126.93, 127.28, 128.31, 128.61, 129.64, 129.78, 130.22, 131.27, 133.19, 136.51, 136.72, 137.36, 143.62, 173.42; MS (m/z): 512 (M^++1); Anal. Calcd. for $C_{27}H_{27}ClN_2O_4S$: C, 63.46; H, 5.33; N, 5.48; Found: C, 63.52; H, 5.40; N, 5.61.

Methyl 1-methyl-5-[(4-methylbenzene)sulfonyl]-3-(4-chlorophenyl)-1*H*,2*H*,3*H*,3a*H*,4*H*,5*H*,9b*H*-pyrrolo[3,2-c]quinoline-3a-carboxylate (9i)



Yield: 92%; mp: 221-223°C; IR (KBr): 1780, 1673 cm^{-1}; ^{1}H NMR: δ 2.37 (s, 3H), 2.38 (s, 3H), 2.91 (t, 1H, *J* = 9.6 Hz), 3.28 (d, 1H, *J* = 7.5 Hz), 3.45 (d, 1H, *J* = 12.3 Hz), 3.62 (s, 1H), 3.70 (s, 3H), 3.85 (d, 1H, *J* = 6.9 Hz), 3.94 (d, 1H, *J* = 12.3 Hz), 7.00–7.60 (m, 12H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 21.51, 39.91, 47.67, 48.25, 52.53, 53.85, 59.90, 67.91, 119.65, 122.52, 124.37, 126.93, 128.30, 128.62, 129.62, 130.20, 131.25, 133.20, 136.51, 136.72, 137.40, 143.59, 173.43; MS (m/z): 512 (M^++1); Anal. Calcd. for $C_{27}H_{27}ClN_2O_4S$: C, 63.46; H, 5.33; N, 5.48; Found: C, 63.53; H, 5.39; N, 5.61.

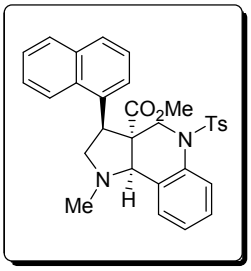
Methyl 1-methyl-5-[(4-methylbenzene)sulfonyl]-3-(3-bromophenyl)-1*H*,2*H*,3*H*,3a*H*,4*H*,5*H*,9b*H*-pyrrolo[3,2-c]quinoline-3a-carboxylate (9j)



Yield: 90%; mp: 232-234°C; IR (KBr): 1794, 1659 cm^{-1}; ^{1}H NMR: δ 2.36 (s, 3H), 2.39 (s, 3H), 2.89 (t, 1H, *J* = 9.9 Hz), 3.26 (dd, 1H, *J* = 3.6, 10.2 Hz), 3.43 (d, 1H, *J* = 12.3 Hz), 3.60 (s, 1H),

3.72 (s, 3H), 3.83 (dd, 1H, J = 3.6, 9.9 Hz), 3.93 (d, 1H, J = 12.3 Hz), 6.99–7.60 (m, 12H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 21.51, 39.89, 47.75, 48.53, 52.56, 53.95, 59.84, 67.86, 119.75, 122.56, 122.59, 124.43, 126.99, 127.78, 128.32, 129.64, 129.99, 130.55, 131.23, 131.74, 136.75, 137.12, 140.53, 143.60, 173.38; MS (m/z): 557 (M^++2); Anal. Calcd. for $C_{27}H_{27}BrN_2O_4S$: C, 58.38; H, 4.90; N, 5.04; Found: C, 58.45; H, 4.99; N, 5.17.

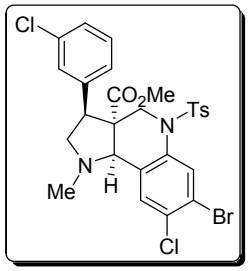
Methyl 1-methyl-5-[(4-methylbenzene)sulfonyl]-3-(naphthalen-1-yl)-1*H*,2*H*,3*H*,3a*H*,4*H*,5*H*,9b*H*-pyrrolo[3,2-c]quinoline-3a-carboxylate (9k)



Yield: 96%; mp: 226-228°C; IR (KBr): 1773, 1686 cm^{-1}; ^{1}H NMR: δ 2.36 (s, 3H), 2.47 (s, 3H), 3.11 (t, 1H, J = 9.9 Hz), 3.53 (d, 2H, J = 12.6 Hz), 3.60 (s, 3H), 3.89 (s, 1H), 3.98 (d, 1H, J = 12.6 Hz), 4.70 (t, 1H, J = 6.3 Hz), 6.99–7.94 (m, 15H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 21.50, 40.46, 44.74, 47.57, 52.44, 55.15, 61.40, 68.47, 119.87, 122.82, 123.01, 124.14, 125.63, 126.11, 126.16, 127.07, 128.20, 129.13, 129.54, 130.72, 132.64, 133.86, 133.97, 136.73, 137.23, 143,50, 174.15; MS (m/z): 528 (M^++1); Anal. Calcd. for $C_{31}H_{30}N_2O_4S$: C, 70.70; H, 5.74; N, 5.32; Found: C, 70.78; H, 5.83; N, 5.45.

Typical experimental procedure for the synthesis of Compound 9l: To a stirred solution of methyl (2*E*)-2-{[*N*-(5-bromo-4-chloro-2-formylphenyl)(4-methylbenzene)sulfonamido]methyl}-3-(3-chlorophenyl)prop-2-enoate (**6l**) (1 mmol, 0.59 g) in acetonitrile (10 mL), *N*-methyl glycine (**7**) (1.1 mmol, 0.10 g) was added and allowed to stir under reflux condition over a period of 6 h. After completion of the reaction as indicated by TLC, the reaction mixture was concentrated and the resulting crude mass was diluted with water (20 mL) and extracted with ethyl acetate (3 x 10 mL). The combined organic layer thus obtained was washed with brine (3 x 10 mL) and dried over anhydrous Na_2SO_4. The organic layer was concentrated and the crude mass was recrystallized using ethyl acetate : hexane mixture (2:8) to provide **9l** as a colorless solid in 86% yield.

Methyl 7-bromo-8-chloro-3-(3-chlorophenyl)-1-methyl-5-[(4-methylbenzene)sulfonyl]-1*H*,2*H*,3*H*,3a*H*,4*H*,5*H*,9b*H*-pyrrolo[3,2-c]quinoline-3a-carboxylate (9l)



Yield: 86%; mp: 239-241°C; IR (KBr): 1786, 1694 cm^{-1}; ^1H NMR: δ 2.33 (s, 3H), 2.39 (s, 3H), 2.89 (t, 1H, J = 9.6 Hz), 3.25-3.36 (m, 2H), 3,52 (s, 1H), 3.72 (s, 3H), 3.81-3.91 (m, 2H), 7.14-7.89 (m, 10H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 21.57, 39.77, 47.49, 48.53, 52.74, 53.96, 59.55, 67.26, 121.33, 124.64, 127.05, 127.22, 127.83, 128.32, 128.86, 129.71, 129.80, 130.59, 131.82, 134.46, 136.21, 139.65, 139.88, 143.91, 144.30, 172.92; MS (m/z): 626 (M^++2); Anal. Calcd. for $C_{27}H_{25}BrCl_2N_2O_4S$: C, 51.94; H, 4.04; N, 4.49; Found: C, 51.99; H, 4.14; N, 4.60.

Typical experimental procedure for the synthesis of pyrroloquinoline (12a)

A mixture of (2*Z*)-2-{[*N*-(2-formylphenyl)(4-methylbenzene)sulfonamido]methyl}-3-phenylprop-2-enenitrile (**10a**) (1 mmol, 0.42 g) and sarcosine (**7a**) (1.1 mmol, 0.18 g) in acetonitrile (10 mL) was stirred under reflux temperature for 6 h. After the completion of the reaction as indicated by TLC, the reaction mixture was concentrated. Then the resulting crude mass was diluted with water (20 mL) and extracted with ethyl acetate (3 x 10 mL). The combined organic layer obtained was washed with brine (3 x 10 mL) and dried over anhydrous Na_2SO_4. The organic layer was concentrated and the crude mass was recrystallized using ethylacetate:hexane mixture (3:7) to provide **12a** as a colorless solid in 94% yield.

Crystal data for 1-Methyl-5-[(4-methylbenzene)sulfonyl]-3-phenyl-1*H*,2*H*,3*H*,3a*H*,4*H*,5*H*, 9b*H*-pyrrolo[3,2-c]quinoline-3a-carbonitrile (12a)

Empirical formula, $C_{26}H_{25}N_3O_2S$; Formula weight, 444; crystal color, colorless; Single crystal X-ray structure of the molecule shown in ORTEP diagram (Figure 2). Detailed X-ray

crystallographic data is available from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK (for pyrroloquinoline (**12a**) CCDC # **833470**).

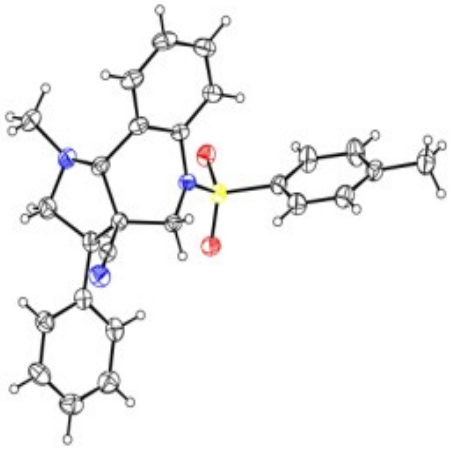
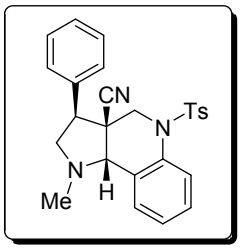


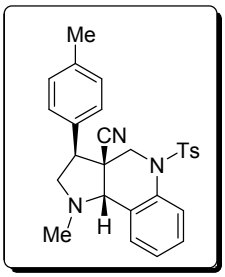
Figure 2. ORTEP diagram of compound 12a

1-Methyl-5-[(4-methylbenzene)sulfonyl]-3-phenyl-1*H*,2*H*,3*H*,3a*H*,4*H*,5*H*, 9b*H*-pyrrolo[3,2-c]quinoline-3a-carbonitrile (12a)



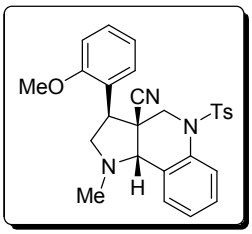
Yield: 94%; mp: 216-218°C; IR (KBr): 2242, 1514 cm^{-1}; ^1H NMR: δ 2.28 (s, 3H), 2.41 (s, 3H), 2.93 (t, 1H, J = 11.1 Hz), 3.25 (dd, 1H, J = 6.3, 9.3 Hz), 3.58-3.61 (m, 2H), 3.85 (dd, 1H, J = 6.3, 11.4 Hz), 4.39 (d, 1H, J = 13.2 Hz), 7.11–7.83 (m, 13H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 21.54, 39.53, 49.41, 52.10, 52.39, 59.97, 70.92, 120.32, 121.12, 124.53, 127.24, 127.93, 128.32, 128.78, 128.95, 129.16, 129.82, 130.47, 135.68, 137.32, 138.91, 144.10; MS (m/z): 445 (M^++1); Anal. Calcd. for $C_{26}H_{25}N_3O_2S$: C, 70.40; H, 5.68; N, 9.47; Found: C, 70.52; H, 5.78; N, 9.59.

1-Methyl-5-[(4-methylbenzene)sulfonyl]-3-(4-methylphenyl-1*H*,2*H*,3*H*,3a*H*,4*H*,5*H*,9b*H*-pyrrolo[3,2-c]quinoline-3a-carbonitrile (12b)



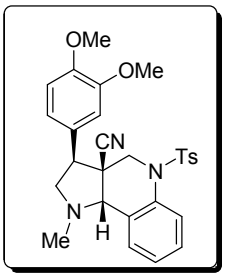
Yield: 92%; mp: 220-222 °C; IR (KBr): 2232, 1544 cm^{-1}; ^1H NMR: δ 2.26 (s, 3H), 2.36 (s, 3H), 2.39 (s, 3H), 2.90 (t, 1H, J = 10.5 Hz), 3.21 (t, 1H, J = 9.0 Hz), 3.56 (s, 1H), 3.60 (d, 1H, J = 13.2 Hz), 3.79 (dd, 1H, J = 6.0, 11.1 Hz), 4.37 (d, 1H, J = 12.9 Hz), 7.12–7.82 (m, 12H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 21.20, 21.56, 39.57, 49.15, 52.04, 52.33, 60.06, 70.83, 120.37, 121.14, 124.54, 127.27, 127.89, 128.96, 129.01, 129.50, 129.84, 130.51, 132.64, 137.28, 138.04, 138.86, 144.13; MS (m/z): 459 (M^++1); Anal. Calcd. for $C_{27}H_{27}N_3O_2S$: C, 70.87; H, 5.95; N, 9.18; Found: C, 70.94; H, 6.09; N, 9.30.

1-Methyl-5-[(4-methylbenzene)sulfonyl]-3-(2-methoxyphenyl-1*H*,2*H*,3*H*,3a*H*,4*H*,5*H*,9b*H*-pyrrolo[3,2-c]quinoline-3a-carbonitrile (12c)



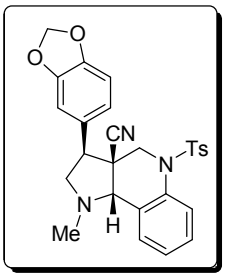
Yield: 96%; mp: 221-223°C; IR (KBr): 2251, 1526 cm^{-1}; ^1H NMR: δ 2.38 (s, 3H), 2.40 (s, 3H), 3.01 (t, 1H, J = 10.2 Hz), 3.29 (dd, 1H, J = 3.3, 9.7 Hz), 3.57 (d, 1H, J = 12.3 Hz), 3.70 (s, 3H), 3.74 (d, 1H, J = 7.5 Hz), 4.15 (d, 1H, J = 12.6 Hz), 4.38-4.42 (m, 1H), 6.96–7.64 (m, 12H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 21.51, 40.03, 45.75, 47.15, 52.52, 53.66, 60.86, 68.26, 119.31, 122.44, 124.35, 126.80, 127.14, 128.33, 128.69, 129.59, 129.76, 129.80, 131.21, 135.21, 135.80, 136.75, 137.31, 143.56; MS (m/z): 475 (M^++1); Anal. Calcd. for $C_{27}H_{27}N_3O_3S$: C, 68.48; H, 5.75; N, 8.87; Found: C, 68.59; H, 5.84; N, 8.99.

1-Methyl-5-[(4-methylbenzene)sulfonyl]-3-(3,4-dimethoxyphenyl-1*H*,2*H*,3*H*,3a*H*,4*H*,5*H*, 9b*H*-pyrrolo[3,2-c]quinoline-3a-carbonitrile (12d)



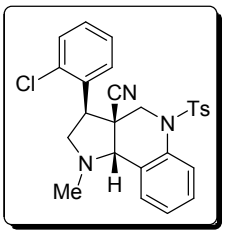
Yield: 95%; mp: 224-226°C; IR (KBr): 2245, 1511 cm^{-1}; ^1H NMR: δ 2.32 (s, 3H), 2.41 (s, 3H), 2.94 (dd, 1H, J = 9.6, 11.7 Hz), 3.25 (dd, 1H, J = 6.0, 9.3 Hz), 3.51 (d, 1H, J = 12.9 Hz), 3.61 (s, 1H), 3.83-3.87 (m, 1H), 3.90 (s, 3H), 3.95 (s, 3H), 4.41 (d, 1H, J = 12.9 Hz), 6.91–7.83 (m, 11H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 21.54, 39.64, 48.87, 52.20, 52.38, 55.89, 56.01, 59.98, 70.82, 111.29, 112.72, 120.54, 120.85, 121.31, 124.52, 127.17, 127.70, 128.19, 128.86, 129.91, 130.33, 137.32, 138.96, 144.17, 148.95, 149.00; MS (m/z): 505 (M^++1); Anal. Calcd. for $C_{28}H_{29}N_3O_4S$: C, 66.78; H, 5.80; N, 8.34; Found: C, 66.89; H, 5.87; N, 8.46.

3-(2*H*-1,3-Benzodioxol-5-yl)-1-methyl-5-[(4-methylbenzene)sulfonyl]1*H*,2*H*,3*H*,3a*H*,4*H*,5*H*, 9b*H*-pyrrolo[3,2-c]quinoline-3a-carbonitrile (12e)



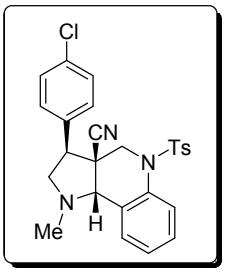
Yield: 92%; mp: 231-233°C; IR (KBr): 2276, 1503 cm^{-1}; ^1H NMR: δ 2.26 (s, 3H), 2.41 (s, 3H), 2.84 (t, 1H, J = 10.2 Hz), 3.21 (t, 1H, J = 7.8 Hz), 3.55-3.59 (m, 2H), 3.76-3.81 (m, 1H), 4.37 (d, 1H, J = 13.2 Hz), 5.98 (s, 2H), 6.84–7.82 (m, 11H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 21.54, 39.49, 49.10, 52.13, 52.35, 60.10, 70.83, 101.21, 108.51, 109.25, 120.30, 121.09, 122.69, 124.52, 127.23, 127.94, 128.93, 129.28, 129.83, 130.46, 137.34, 138.95, 144.11, 147.61, 147.92; MS (m/z): 489 (M^++1); Anal. Calcd. for $C_{27}H_{25}N_3O_4S$: C, 66.51; H, 5.17; N, 8.62; Found: C, 66.66; H, 5.28; N, 8.77.

1-Methyl-5-[(4-methylbenzene)sulfonyl]-3-(2-chlorophenyl-1*H*,2*H*,3*H*,3a*H*,4*H*,5*H*,9b*H*-pyrrolo[3,2-c]quinoline-3a-carbonitrile (12f)



Yield: 91%; mp: 227-229°C; IR (KBr): 2258, 1529 cm^{-1}; ^1H NMR: δ 2.22 (s, 3H), 2.37 (s, 3H), 2.77 (t, 1H, J = 9.6 Hz), 3.23 (t, 1H, J = 9 Hz), 3.47 (s, 1H), 4.13 (t, 1H, J = 9 Hz), 4.36 (dd, 2H, J = 13.2, 26.4 Hz), 7.09–7.81(m, 12H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 21.54, 39.30, 46.03, 49.32, 51.79, 60.40, 69.96, 119.48, 121.41, 123.95, 124.95, 127.14, 127.60, 128.12, 129.22, 129.54, 129.62, 130.11, 131.17, 135.01, 135.08, 137.03, 137.54, 143.94; MS (m/z): 479 (M^++1); Anal. Calcd. for $C_{26}H_{24}ClN_3O_2S$: C, 65.33; H, 5.06; N, 8.79; Found: C, 65.45; H, 5.19; N, 8.92.

1-Methyl-5-[(4-methylbenzene)sulfonyl]-3-(4-chlorophenyl-1*H*,2*H*,3*H*,3a*H*,4*H*,5*H*,9b*H*-pyrrolo[3,2-c]quinoline-3a-carbonitrile (12g)

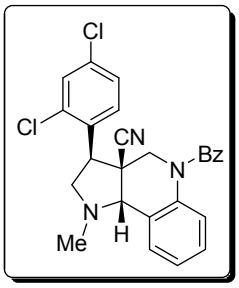


Yield: 93%; mp: 219-221°C; IR (KBr): 2248, 1526 cm^{-1}; ^1H NMR: δ 2.29 (s, 3H), 2.42 (s, 3H), 2.90 (t, 1H, J = 10.2 Hz), 3.24 (t, 1H, J = 8.1 Hz), 3.53 (d, 1H, J = 13.2 Hz), 3.59 (s, 1H), 3.89 (dd, 1H, J = 5.7, 10.8 Hz), 4.37 (d, 1H, J = 12.9 Hz), 7.13–7.82 (m, 12H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 21.55, 39.46, 48.54, 52.16, 52.28, 59.86, 70.86, 120.11, 120.96, 124.58, 127.15, 127.85, 128.98, 129.91, 130.44, 130.55, 134.12, 13.31, 137.33, 138.99, 142.07, 144.22; MS (m/z): 479 (M^++1); Anal. Calcd. for $C_{26}H_{24}ClN_3O_2S$: C, 65.33; H, 5.06; N, 8.79; Found: C, 65.47; H, 5.14; N, 8.92.

Typical experimental procedure for the synthesis of pyrroloquinoline (12h)

A mixture of *N*-[(2*Z*)-2-cyano-2-[(2,4-dichlorophenyl)methylidene]ethyl]-*N*-(2-formylphenyl) benzamide (**10h**) (1 mmol, 0.43 g) and sarcosine (**7a**) (1.1 mmol, 0.10 g) in acetonitrile (10 mL) was stirred under reflux temperature for 6 h. After the completion of the reaction as indicated by TLC, the reaction mixture was concentrated. Then the resulting crude mass was diluted with water (20 mL) and extracted with ethyl acetate (3 x 10 mL). The combined organic layer obtained was washed with brine (3 x 10 mL) and dried over anhydrous Na_2SO_4. The organic layer was concentrated and the crude mass was recrystallized using ethylacetate:hexane mixture (3:7) to provide **12h**as a colorless solid in 84% yield.

5-benzoyl-3-(2,4-dichlorophenyl)-1-methyl-1*H*,2*H*,3*H*,3a*H*,4*H*,5*H*,9b*H*-pyrrolo[3,2-c]quino -line-3a-carbonitrile (12h)



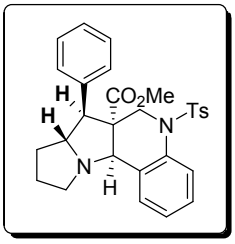
Yield: 84%; mp: 209-211°C; IR (KBr): 2258, 1542 cm^{-1}; ^1H NMR: δ 2.40 (s, 3H), 2.84 (t, 1H, J = 10.2 Hz), 3.33-3.41 (m, 4H), 3.72 (s, 1H), 4.34-4.40 (m, 1H), 5.29 (d, 1H, J = 13.2 Hz), 6.59-7.58 (m, 12H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 39.38, 45.90, 51.30, 52.48, 61.01, 72.67, 122.39, 125.18, 125.64, 127.23, 128.00, 128.11, 128.67, 129.15, 129.98, 130.18, 130.83, 131.22, 132.57, 134.51, 134.80, 135.92, 141.99, 169.63; MS (m/z): 463 (M^++1); Anal. Calcd. for $C_{26}H_{21}Cl_2N_3O$: C, 67.54; H, 4.58; N, 9.09; Found: C, 67.64; H, 4.70; N, 9.20.

Typical experimental procedure for the synthesis of pyrrolizinoquinoline (15a)

A mixture of methyl (2*E*)-2-{[*N*-(2-formylphenyl)(4-methylbenzene)sulfonamido] methyl}-3-phenylprop-2-enoate (**6a**) (1 mmol, 0.45g) and *L*-proline (**7b**) (1.1 mmol, 0.23 g) in acetonitrile (10 mL) was refluxed for 10 h. After the completion of the reaction as indicated by TLC, the reaction mixture was concentrated. Then the resulting crude mass was diluted with water (20 mL) and extracted with ethyl acetate (3 x 10 mL). The combined organic layer

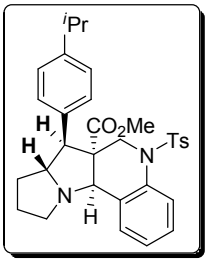
obtained was washed with brine (3 x 10 mL) and dried over anhydrous Na_2SO_4. The organic layer was concentrated and the crude mass was recrystallized using ethylacetate:hexane mixture (1:1) to provide **15a** as a colorless solid in 88% yield.

Methyl 8-[(4-methylbenzene)sulfonyl]-11-phenyl-8,16-diazatetracyclo[8.6.0.0^{2,7}.0^{12,16}] hexadeca-2,4,6-triene-10-carboxylate (15a)



Yield : 88%; mp: 186-188°C; IR (KBr): 1784, 1685 cm^{-1}; ^{1}H NMR: δ 1.56-2.13 (m, 4H), 2.39 (s, 3H), 3.05-3.14 (m, 2H), 3.52 (d, 1H, *J* = 10.5 Hz), 3.62 (s, 3H), 3.78 (d, 1H, *J* = 12.3 Hz), 4.04-4.10 (m, 1H), 4.33 (s, 1H), 4.53 (d, 1H, *J* = 12.6 Hz), 6.97–7.68 (m, 13H); ^{13}C NMR ($CDCl_3$, 75 MHz): *δ* 21.52, 27.18, 31.62, 45.69, 52.26, 54.99, 58.76, 58.94, 66.85, 68.89, 119.36, 123.14, 127.05, 127.55, 127.88, 128.02, 128.52, 128.73, 129.61, 129.76, 135.15, 135.78, 137.68, 143.53, 172.53; MS (m/z): 504 (M^++1); Anal. Calcd. for $C_{29}H_{30}N_2O_4S$: C, 69.30; H, 6.02; N, 5.57; Found: C, 69.41; H, 6.17; N, 5.73.

Methyl 8-[(4-methylbenzene)sulfonyl]-11-[4-(propan-2-yl)phenyl]-8,16-diazatetracyclo [8.6.0.0^{2,7}.0^{12,16}]hexa deca-2,4,6-triene-10-carboxylate (15b)



Yield: 86%; mp: 189-191°C; IR (KBr): 1787, 1639 cm^{-1}; ^{1}H NMR: *δ* 1.26 (s, 3H), 1.29 (s, 3H), 1.57-2.11 (m, 5H), 2.38 (s, 3H), 2.85-3.10 (m, 2H), 3.51 (d, 1H, *J* = 10.2 Hz), 3.63 (s, 3H), 3.77 (d, 1H, *J* = 5.7 Hz), 4.04-4.06 (m, 1H), 4.30 (s, 1H), 4.54 (d, 1H, *J* = 12.6 Hz), 6.96–7.71 (m,

12H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 21.50, 23.88, 27.10, 31.66, 33.68, 45.70, 52.25, 54.88, 58.58, 58.72, 66.89, 68.96, 119.50, 123.11, 126.78, 127.09, 128.02, 128.39, 129.57, 129.73, 132.29, 135.82, 137.71, 143.47, 148.25, 172.62; MS (m/z): 546 (M^++1); Anal. Calcd. for $C_{32}H_{36}N_2O_4S$: C, 70.56; H, 6.66; N, 5.14; Found: C, 70.66; H, 6.75; N, 5.26.

Crystal data for Methyl 8-[(4-methylbenzene)sulfonyl]-11-[4-(propan-2-yl)phenyl]-8,16-diazatetracyclo [8.6.0.0^{2,7}.0^{12,16}]hexa deca-2,4,6-triene-10-carboxylate (15b)

Empirical formula, $C_{32}H_{36}N_2O_4S$; Formula weight, 545; crystal color, colorless; Single crystal X-ray structure of the molecule shown in ORTEP diagram (Figure 3). Detailed X-ray crystallographic data is available from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK (for pyrrolizinoquinoline (**15b**) CCDC # **976506**).

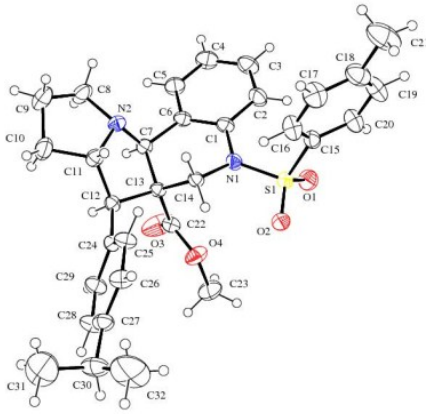
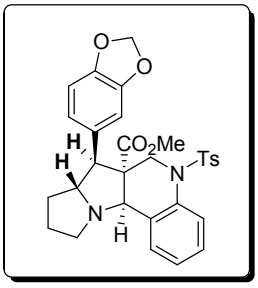


Figure 3. ORTEP diagram of compound 15b

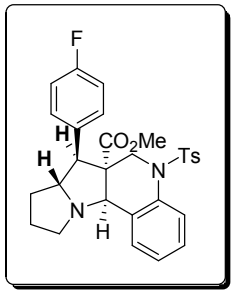
Methyl 11-(2H-1,3-benzodioxol-5-yl)-8-[(4-methylbenzene) sulfonyl]-8,16-diazatetracyclo [8.6.0. 0^{2,7}.0^{12,16}]hexadeca-2,4,6-triene-10-carboxylate (15c)



Yield: 84%; mp: 190-192°C; IR (KBr): 1779, 1648 cm^{-1}; ^{1}H NMR: δ 1.62-2.11 (m, 4H), 2.41 (s, 3H), 3.03-3.13 (m, 2H), 3.36-3.44 (m, 1H), 3.47 (s, 3H), 3.60-3.87 (m, 4H), 6.01–7.71 (m, 13H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 21.62, 26.12, 30.32, 47.75, 52,29, 53.75, 57.47, 58.69, 66.98,

68.78, 74.67, 101.32, 108.56, 109.12, 122.23, 123.86, 124.70, 126.82, 127.04, 129.62, 132.07, 135.18, 135.35, 143.87, 147.02, 147.97, 148.13, 174.22; MS (m/z): 548 (M^++1); Anal. Calcd. for $C_{30}H_{30}N_2O_6S$: C, 65.92; H, 5.53; N, 5.12; Found: C, 66.08; H, 5.62; N, 5.28.

Methyl 11-(4-fluorophenyl)-8-[(4-methylbenzene) sulfonyl]-8,16-diazatetracyclo [8.6.0.0^{2,7}.0^{12,16}]hexa deca-2,4,6-triene-10-carboxylate (15d)

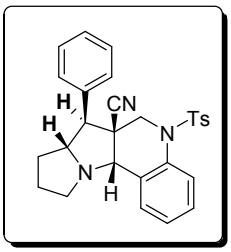


Yield: 87%; mp: 196-198°C; IR (KBr): 1787, 1678 cm^{-1}; ^{1}H NMR: δ 1.59-2.08 (m, 4H), 2.41 (s, 3H), 2.91-3.06 (m, 2H), 3.37-3.42 (m, 1H), 3.49 (s, 3H), 3.56-3.87 (m, 4H), 7.05-7.69 (m, 12H); ^{13}C NMR ($CDCl_3$, 75 MHz) : δ 21.59, 26.23, 30.41, 47.91, 52,32, 53.76, 56.69, 59.55, 66.85, 74.59, 115.81 (d, J = 21.0 Hz), 123.99, 124.15, 124.58, 126.80, 129.57, 130.37 (d, J = 7.5 Hz), 134.82 (d, J = 84.5 Hz), 143.82, 174.07; MS (m/z): 522 (M^++1); Anal. Calcd. for $C_{29}H_{29}FN_2O_4S$: C, 66.90; H, 5.61; N, 5.38; Found: C, 66.99; H, 5.74; N, 5.53.

Typical experimental procedure for the synthesis of pyrrolizinoquinoline (17a)

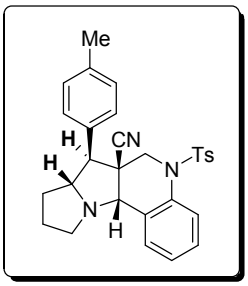
To a stirred solution of (2*Z*)-2-{[*N*-(2-formylphenyl)(4-methylbenzene)sulfonamido] methyl}-3-phenylprop-2-enenitrile (**10a**) (1 mmol, 0.42 g) and *L*-proline (**7b**) (1.1 mmol, 0.17 g) in acetonitrile (10 mL) was refluxed for 10 h. After the completion of the reaction as indicated by TLC, the reaction mixture was concentrated. Then the resulting crude mass was diluted with water (20 mL) and extracted with ethyl acetate (3 x 10 mL). The combined organic layer obtained was washed with brine (3 x 10 mL) and dried over anhydrous Na_2SO_4. The organic layer was concentrated and the crude mass was recrystallized using ethylacetate:hexane mixture (4:6) to provide **17a** as a colorless solid in 83% yield.

8-[(4-Methylbenzene)sulfonyl]-11-phenyl-8,16-diaza tetracyclo[8.6.0.0^{2,7}.0^{12,16}]hexadeca-2,4,6-triene-10-carbonitrile (17a)



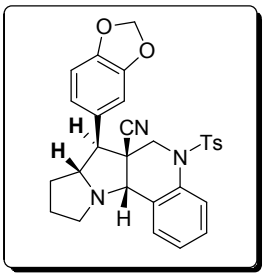
Yield: 83%; mp: 196-198°C; IR (KBr): 2254, 1571 cm^{-1}; ^{1}H NMR: δ 1.60-2.07 (m, 4H), 2.40 (s, 3H), 2.88-2.96 (m, 1H), 3.27-3.32 (m, 1H), 3.47 (d, 1H, J = 7.8 Hz), 3.51 (s, 1H), 3.67 (d, 1H, J = 14.1 Hz), 3.84-3.92 (m, 1H), 4.61 (d, 1H, J = 14.1 Hz), 7.21–7.70 (m, 13H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 21.55, 26.78, 28.60, 47.22, 51.71, 54.40, 56.10, 68.86, 69.21, 122.85, 123.83, 126.16, 127.59, 127.68, 128.22, 128.34, 128.75, 128.90, 129.28, 129.84, 132.11, 135.18, 135.53, 136.62, 144.20; MS (m/z): 471 (M^++1); Anal. Calcd. for $C_{28}H_{27}N_3O_2S$: C, 71.61; H, 5.80; N, 8.95; Found: C, 71.69; H, 5.89; N, 9.09.

8-[(4-Methylbenzene)sulfonyl]-11-(4-methylphenyl)-8,16-diazatetracyclo[8.6.0.0^{2,7}.0^{12,16}] hexadeca-2,4,6-triene-10-carbonitrile (17b)



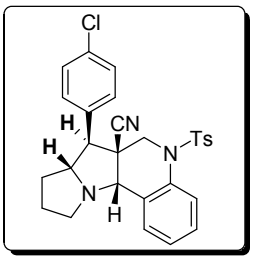
Yield: 84%; mp: 200-202°C; IR (KBr): 2259, 1528 cm^{-1}; ^{1}H NMR: δ 1.58-2.05 (m, 4H), 2.34 (s, 3H), 2.40 (s, 3H), 2.87-2.95 (m, 1H), 3.27-3.32 (m, 1H), 3.43 (d, 1H, J = 7.8 Hz), 3.51 (s, 1H), 3.66 (d, J = 14.1 Hz, 1H), 3.78-3.88 (m, 1H), 4.59 (d, 1H, J = 14.1 Hz), 7.16-7.70 (m, 12H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 21.03, 21.54, 26.81, 28.58, 47.36, 51.68, 54.08, 56.09, 68.89, 69.17, 122.84, 123.74, 126.09, 127.60, 128.24, 128.29, 128.83, 129.44, 129.82, 132.04, 132.10, 135.54, 136.64, 137.42, 144.17; MS (m/z): 485 (M^++1); Anal. Calcd. for $C_{29}H_{29}N_3O_2S$: C, 72.02; H, 6.04; N, 8.69; Found: C, 72.13; H, 6.15; N, 8.84.

11-(2*H*-1,3-Benzodioxol-5-yl)-8-[(4-methylbenzene)sulfonyl]-8,16-diazatetracyclo[8.6.0.0^{2,7}.0^{12,16}] hexa deca-2,4,6-triene-10-carbonitrile (17c)

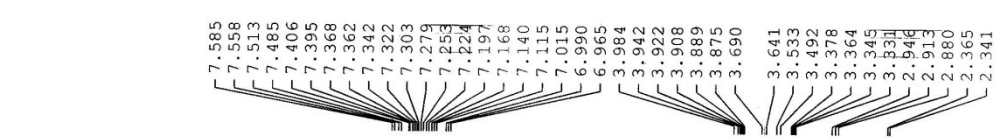


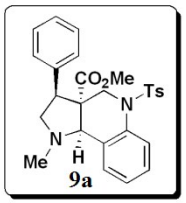
Yield: 82%; mp: 209-211°C; IR (KBr): 2268, 1532 cm^{-1}; ^{1}H NMR: δ 1.63-2.05 (m, 4H), 2.40 (s, 3H), 2.87-2.95 (m, 1H), 3.28-3.80 (m, 5H), 4.53 (d, 1H, J = 14.1 Hz), 5.97 (d, 2H, J = 5.7 Hz), 6.80-7.71 (m, 11H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 21.56, 26.84, 28.45, 47.53, 51.52, 53.91, 55.86, 67.54, 68.90, 69.04, 101.23, 108.39, 109.27, 122.55, 123.43, 126.01, 126.90, 127.00, 127.55, 128.35,128.70, 129.86, 131.71, 135.57, 136.65, 144.23, 147.16, 148.00; MS (m/z): 515 (M^++1); Anal. Calcd. for $C_{29}H_{27}N_3O_4S$: C, 67.82; H, 5.30; N, 8.18; Found: C, 67.93; H, 5.39; N, 8.32.

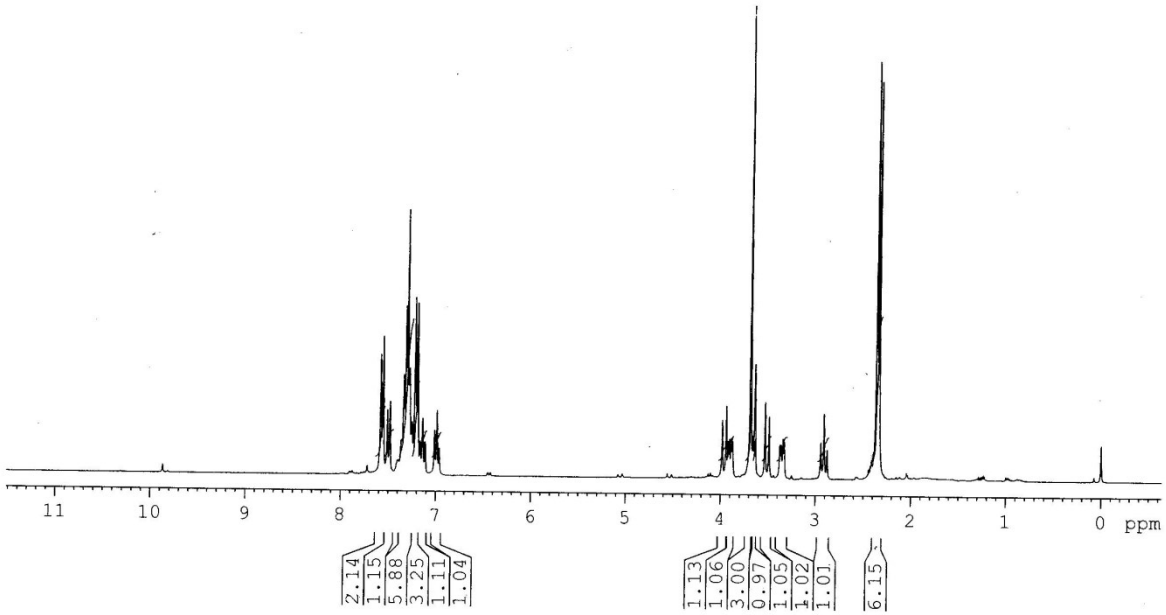
11-(4-Chlorophenyl)-8-[(4-methylbenzene)sulfonyl]-8,16-diazatetracyclo[8.6.0.0^{2,7}.0^{12,16}] hexadeca-2,4,6-triene-10-carbonitrile (17d)



Yield: 86%; mp: 201-203°C; IR (KBr): 2259, 1548 cm^{-1}; ^{1}H NMR: δ 1.73-2.06 (m, 4H), 2.40 (s, 3H), 2.89-2.97 (m, 1H), 3.22-3.31 (m, 1H), 3.51 (d, 1H, J = 7.5 Hz), 3.59 (s, 1H), 3.71 (d, 1H, J = 14.1 Hz), 3.80-3.85 (m, 1H), 4.50 (d, 1H, J = 14.1 Hz), 7.22-7.70 (m, 12H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 21.56, 26.71, 28.53, 47.19, 51.51, 53.33, 55.92, 68.79, 69.16, 122.50, 123.39, 126.09, 127.48, 128.41, 128.94, 129.06, 129.61, 129.92, 130.37, 131.61, 133.74, 135.59, 136.65, 144.32; MS (m/z): 505 (M^++1); Anal. Calcd. for $C_{28}H_{26}ClN_3O_2S$: C, 66.72; H, 5.20; N, 8.34; Found: C, 66.86; H, 5.29; N, 8.47.







BRUKER

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Current Data Parameters
NAME                  DK-V-H-ESt
EXPNO                          1
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20101218
Time                       18.32
INSTRUM                    spect
PROBHD    5 mm DUL 13C-1
PULPROG                    zg30
TD                        65536
SOLVENT                   CDCl3
NS                             7
DS                             2
SWH                    6172.839 Hz
FIDRES                 0.094190 Hz
AQ                     5.3084660 sec
RG                          80.6
DW                        81.000 usec
DE                         6.00 usec
TE                        300.0 K
D1                  1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                          1H
P1                        13.15 usec
PL1                        0.00 dB
SFO1               300.1318534 MHz

F2 - Processing parameters
SI                         32768
SF                 300.1300086 MHz
WDW                           EM
SSB                            0
LB                         0.30 Hz
GB                             0
PC                          1.00
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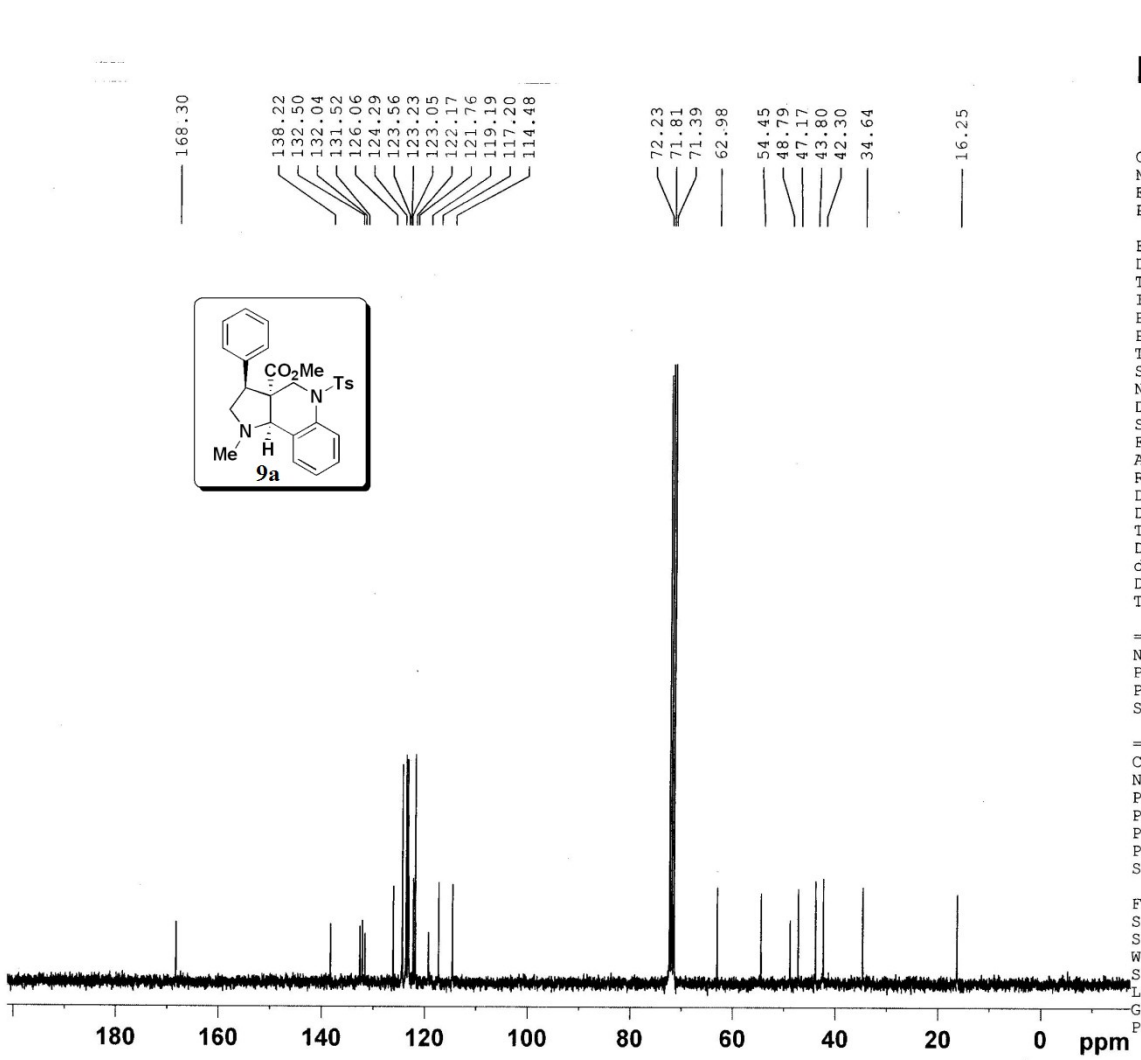


BRUKER

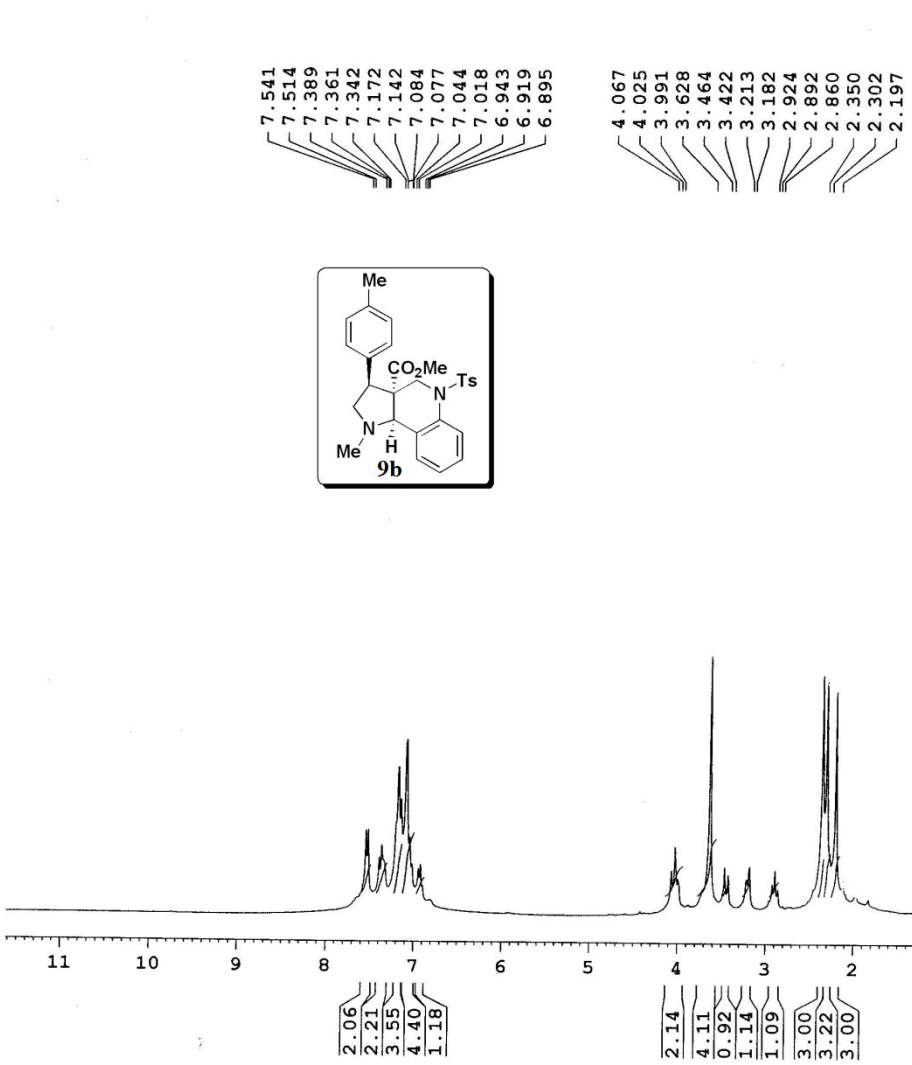
Current Data Parameters
NAME DK-V-HESt-SA
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20101218
Time 17.53
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 433
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 645.1
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 7.40 usec
PL1 -2.00 dB
SFO1 75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.68 dB
PL13 16.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677867 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



BRUKER

Current Data Parameters

NAME	DK-V-H-Est-COUM
EXPNO	3
PROCNO	1

F2 - Acquisition Parameters

Date_	20110102
Time	2.06
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	6172.839 Hz
FIDRES	0.094190 Hz
AQ	5.3084660 sec
RG	101.6
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
TD0	1

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

NUC1	1H
P1	13.15 usec
PL1	0.00 dB
SFO1	300.1318534 MHz

F2 - Processing parameters

SI	32768
SF	300.1300290 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

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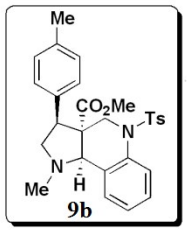
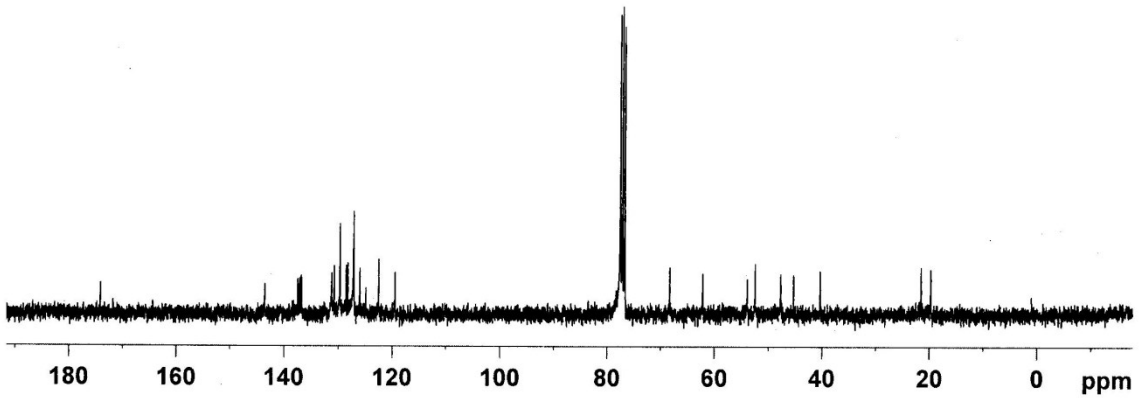


9b



BRUKER

Current Data Parameters
NAME DK-V-4HEst-6ARLO
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_	20110102
Time	1.45
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	319
DS	4
SWH	17985.611 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	362
DW	27.800 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

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

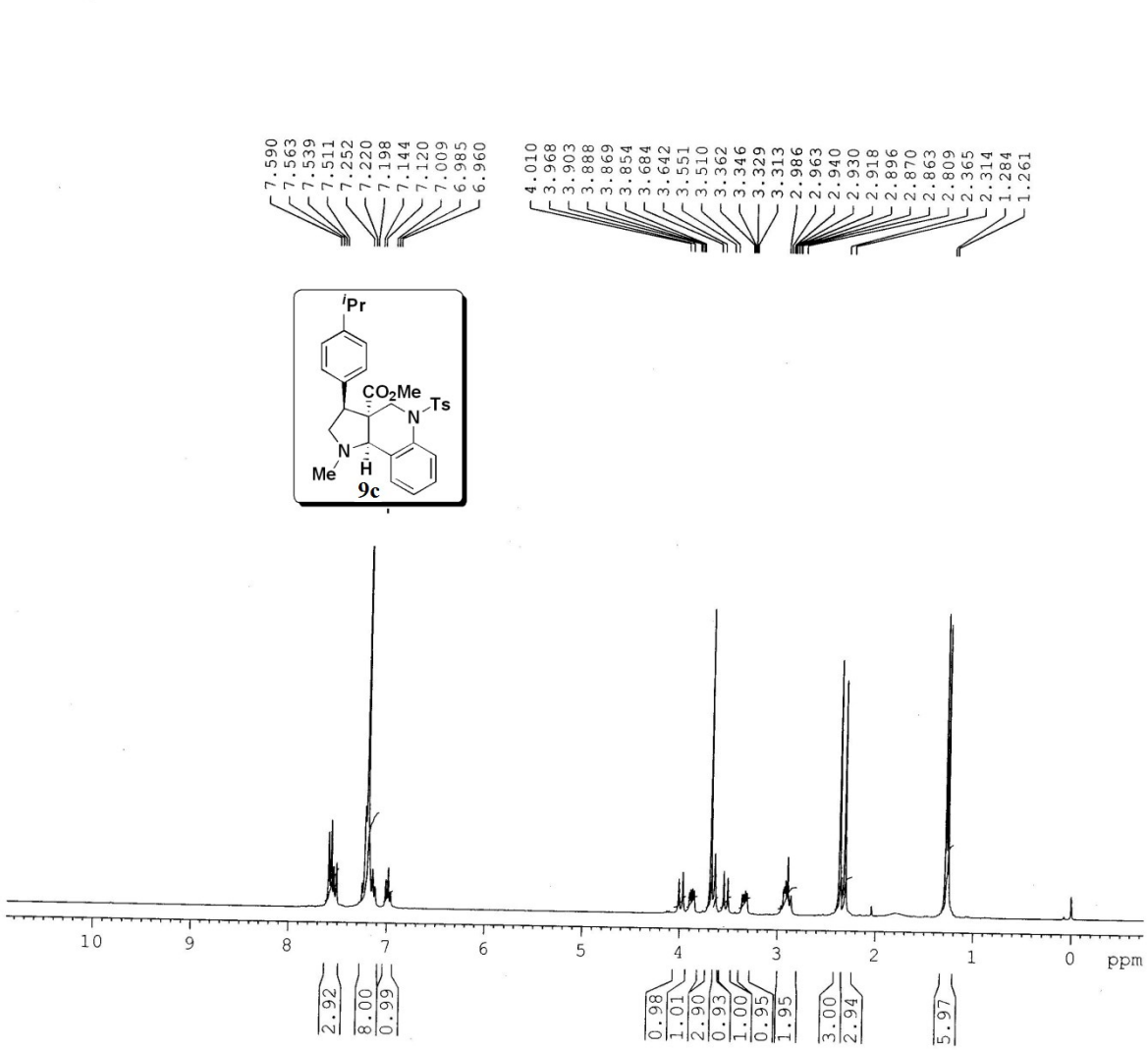
NUC1	13C
P1	7.40 usec
PL1	-2.00 dB
SFO1	75.4752953 MHz

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

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	15.68 dB
PL13	16.00 dB
SFO2	300.1312005 MHz

F2 - Processing parameters

SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



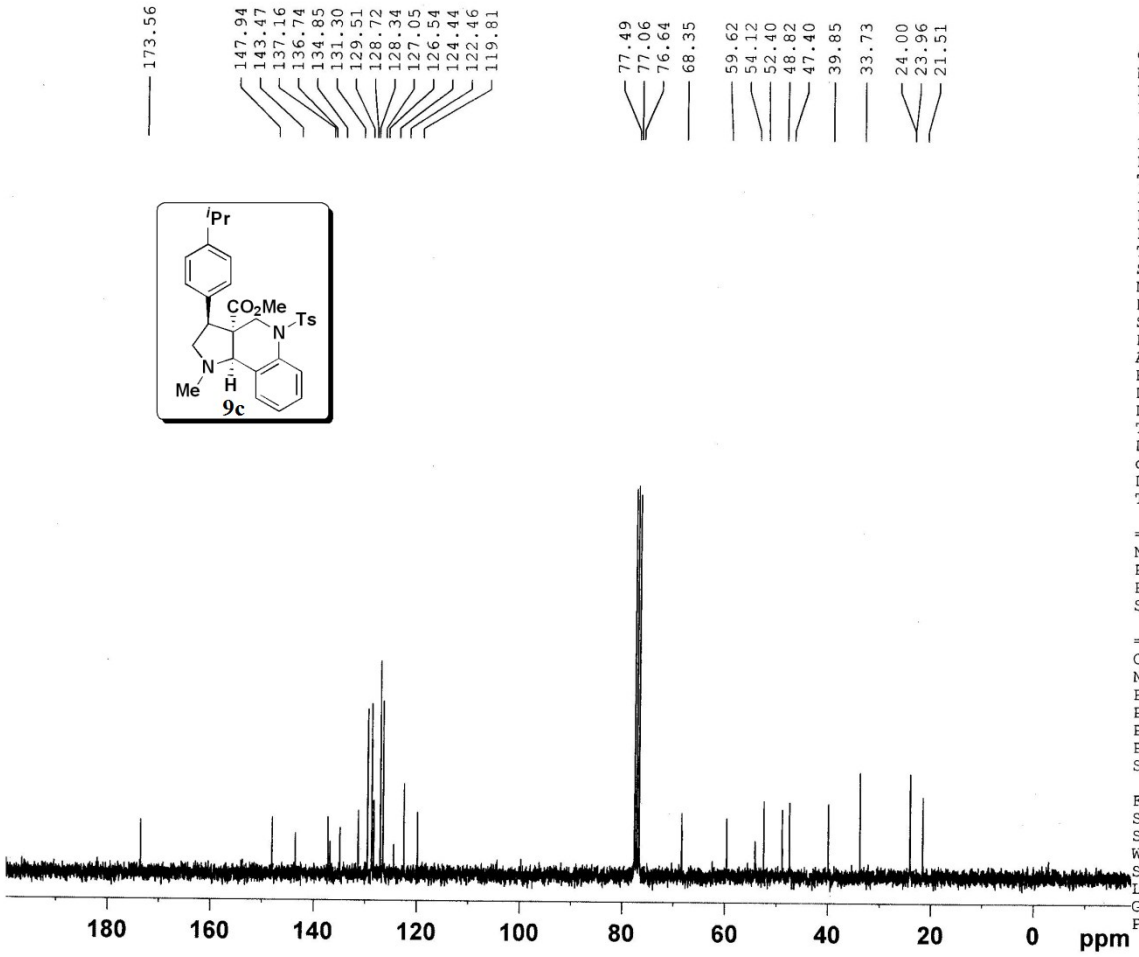
BRUKER

Current Data Parameters
NAME DK-V-4IPr-ESt SA
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20101218
Time 19.04
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 71.8
DW 81.000 use
DE 6.00 use
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.15 use
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300090 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



BRUKER

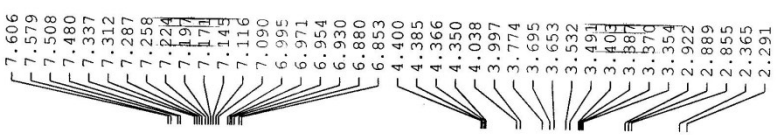
Current Data Parameters
NAME DK-V-4IPr-ESt SA
EXPNO 2
PROCNO 1

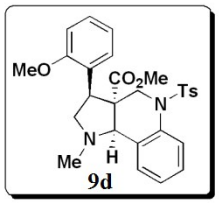
F2 - Acquisition Parameters
Date_ 20101218
Time 19.10
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 83
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 645.1
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 7.40 usec
PL1 -2.00 dB
SFO1 75.4752953 MHz

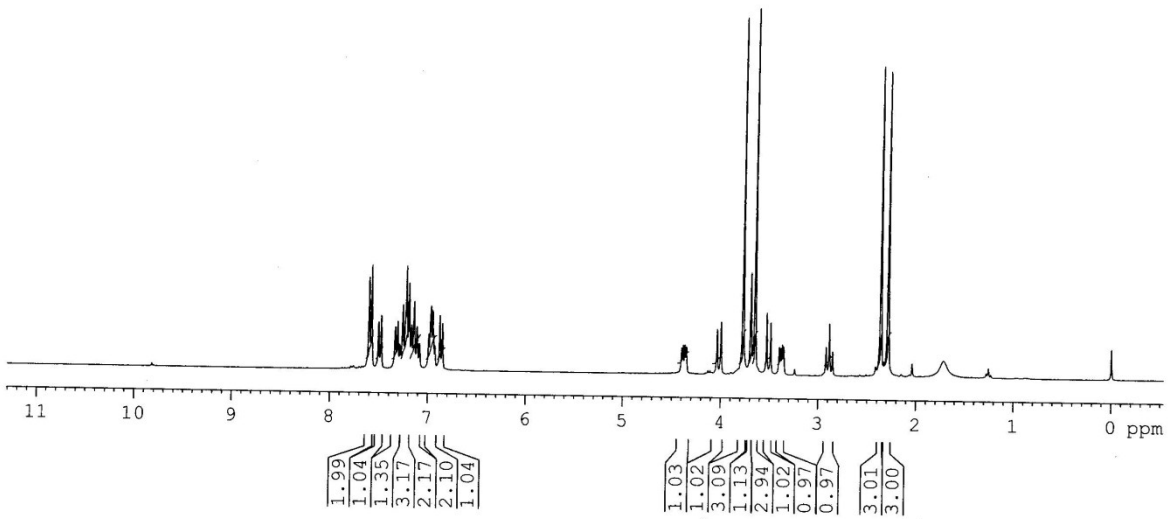
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.68 dB
PL13 16.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





9d



BRUKER

Current Data Parameters
NAME DK-V-2ome-EST-SA
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110102
Time 4.02
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 114
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.15 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300074 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

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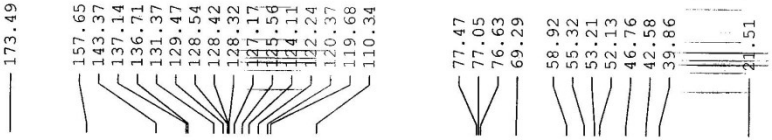
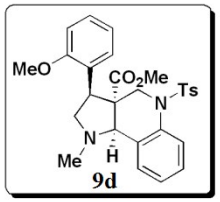
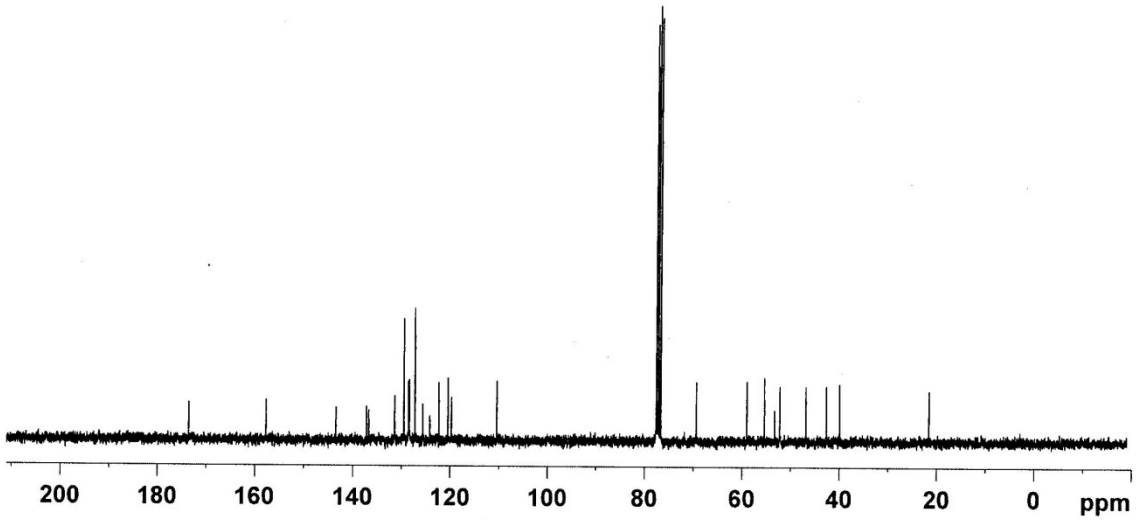






BRUKER

Current Data Parameters
NAME DK-V-2[illegible]-EST-SA
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_	20110102
Time	3.58
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	175
DS	4
SWH	17985.611 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	574.7
DW	27.800 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

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

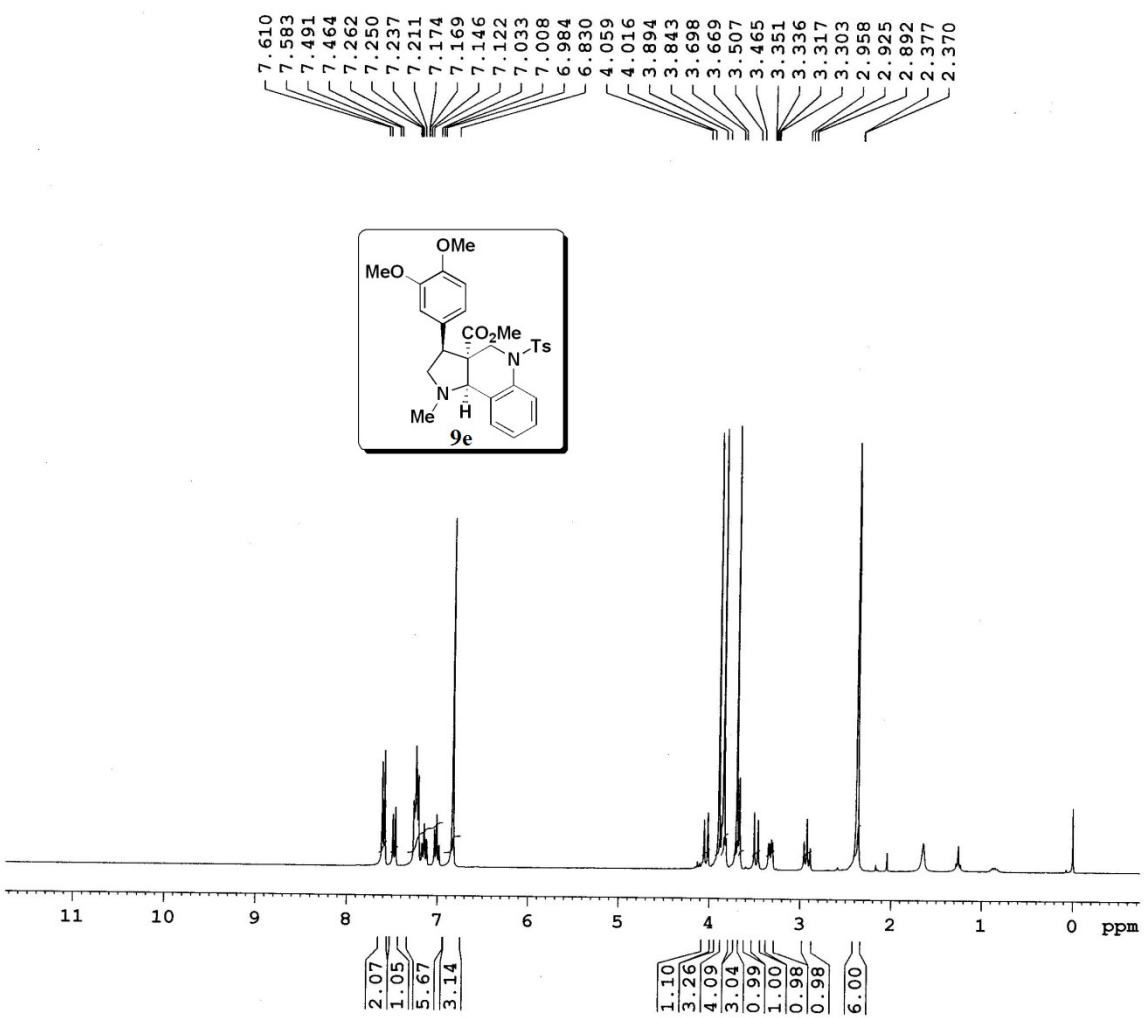
NUC1	13C
P1	7.40 usec
PL1	-2.00 dB
SFO1	75.4752953 MHz

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

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	15.68 dB
PL13	16.00 dB
SFO2	300.1312005 MHz

F2 - Processing parameters

SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



BRUKER

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Current Data Parameters
NAME      DK-V-2-Me ESt SA
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20101225
Time                 23.33
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              6172.839 Hz
FIDRES           0.094190 Hz
AQ               5.3084660 sec
RG                    128
DW                 81.000 usec
DE                  6.00 usec
TE                 300.0 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 13.15 usec
PL1                 0.00 dB
SFO1         300.1318534 MHz

F2 - Processing parameters
SI                  32768
SF           300.1300055 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   1.00
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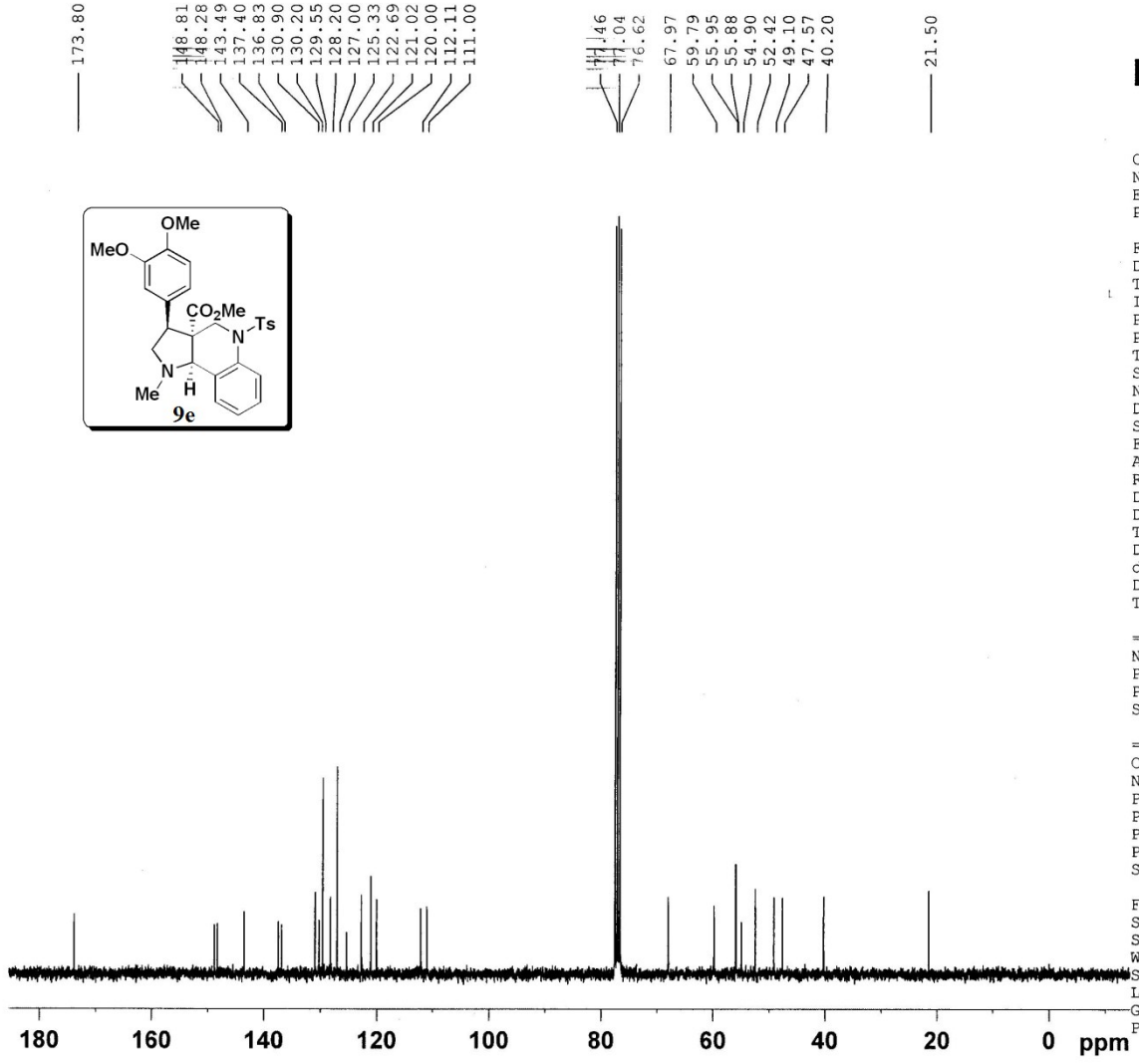


BRUKER

Current Data Parameters
NAME DK-V-16(OMe)2ESt SA
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_	20101225
Time	23.56
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	350
DS	4
SWH	17985.611 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	362
DW	27.800 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

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

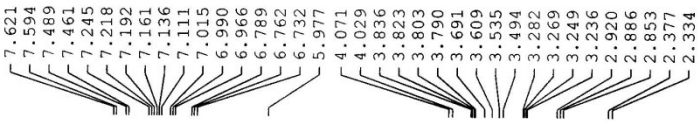
NUC1	13C
P1	7.40 usec
PL1	-2.00 dB
SFO1	75.4752953 MHz

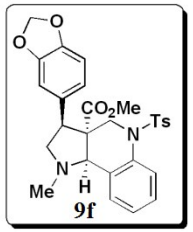
======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	15.68 dB
PL13	16.00 dB
SFO2	300.1312005 MHz

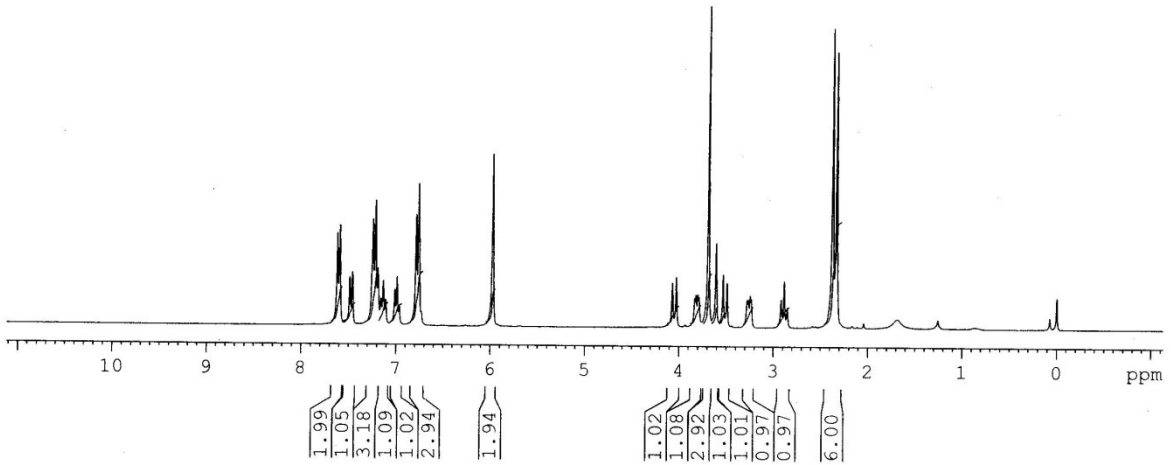
F2 - Processing parameters

SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40





9f



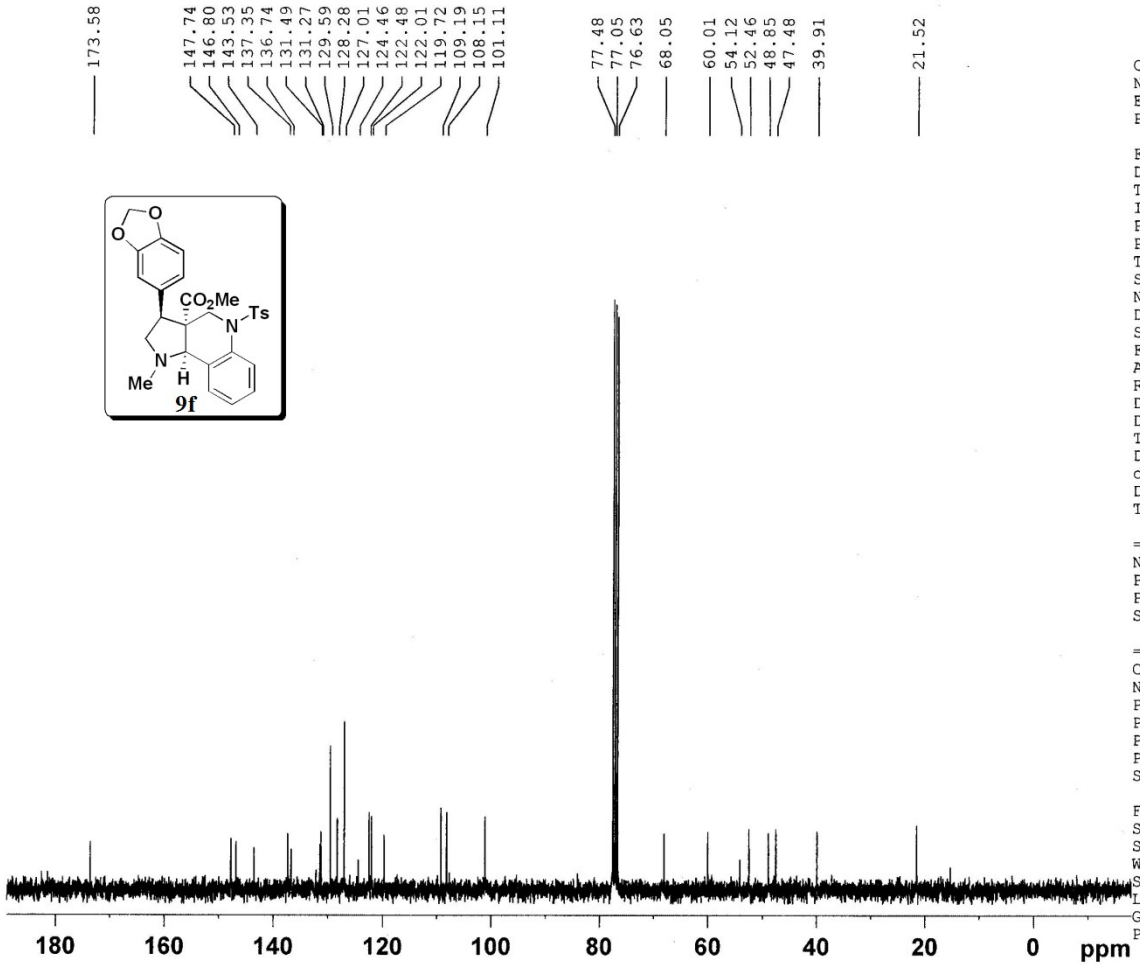
BRUKER

```
Current Data Parameters
NAME          DK-V-ESt PIP
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20101218
Time                 20.05
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              6172.839 Hz
FIDRES           0.094190 Hz
AQ               5.3084660 sec
RG                   101.6
DW                  81.000 usec
DE                   6.00 usec
TE                  300.0 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   1H
P1                  13.15 usec
PL1                  0.00 dB
SFO1          300.1318534 MHz

F2 - Processing parameters
SI                   32768
SF            300.1300067 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



BRUKER

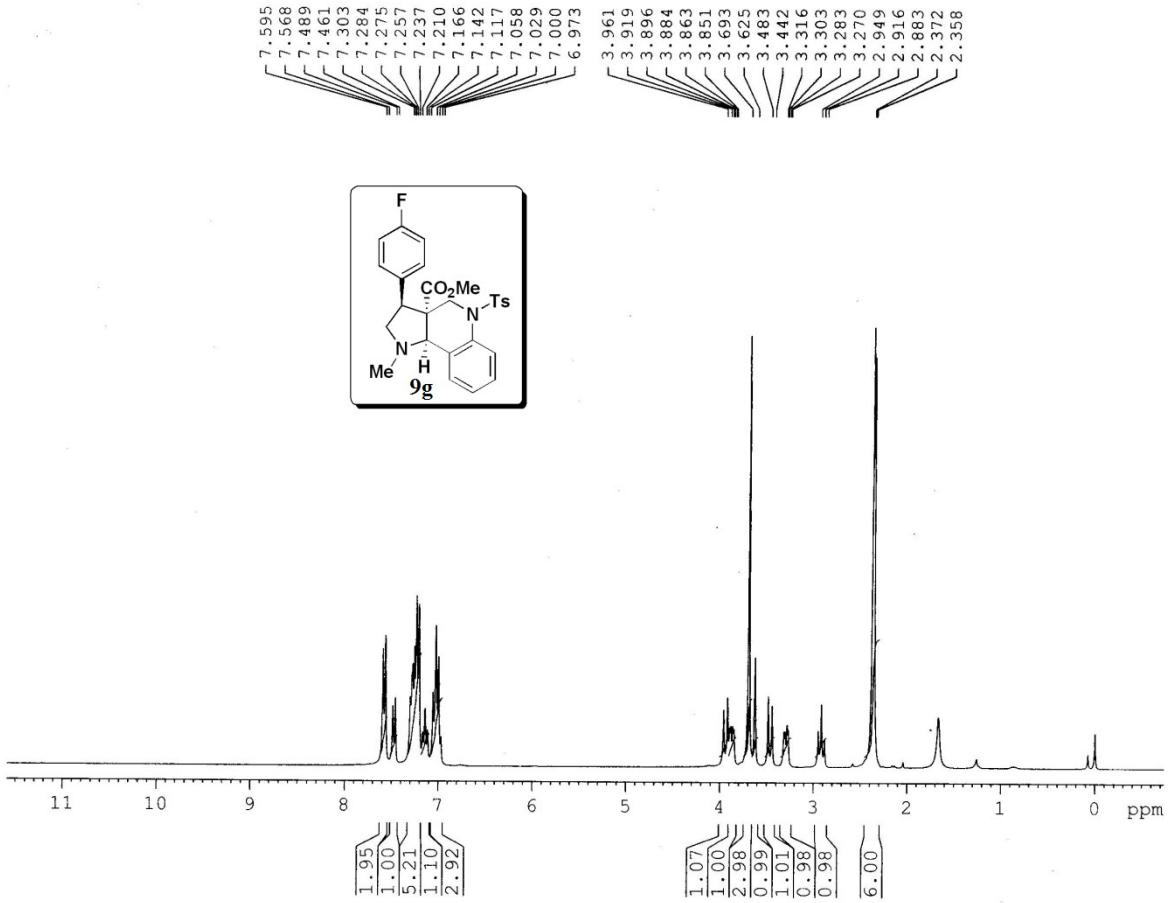
Current Data Parameters
NAME DK-V-ESt PIP
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20101218
Time 20.09
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 66
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 645.1
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 7.40 usec
PL1 -2.00 dB
SFO1 75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.68 dB
PL13 16.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



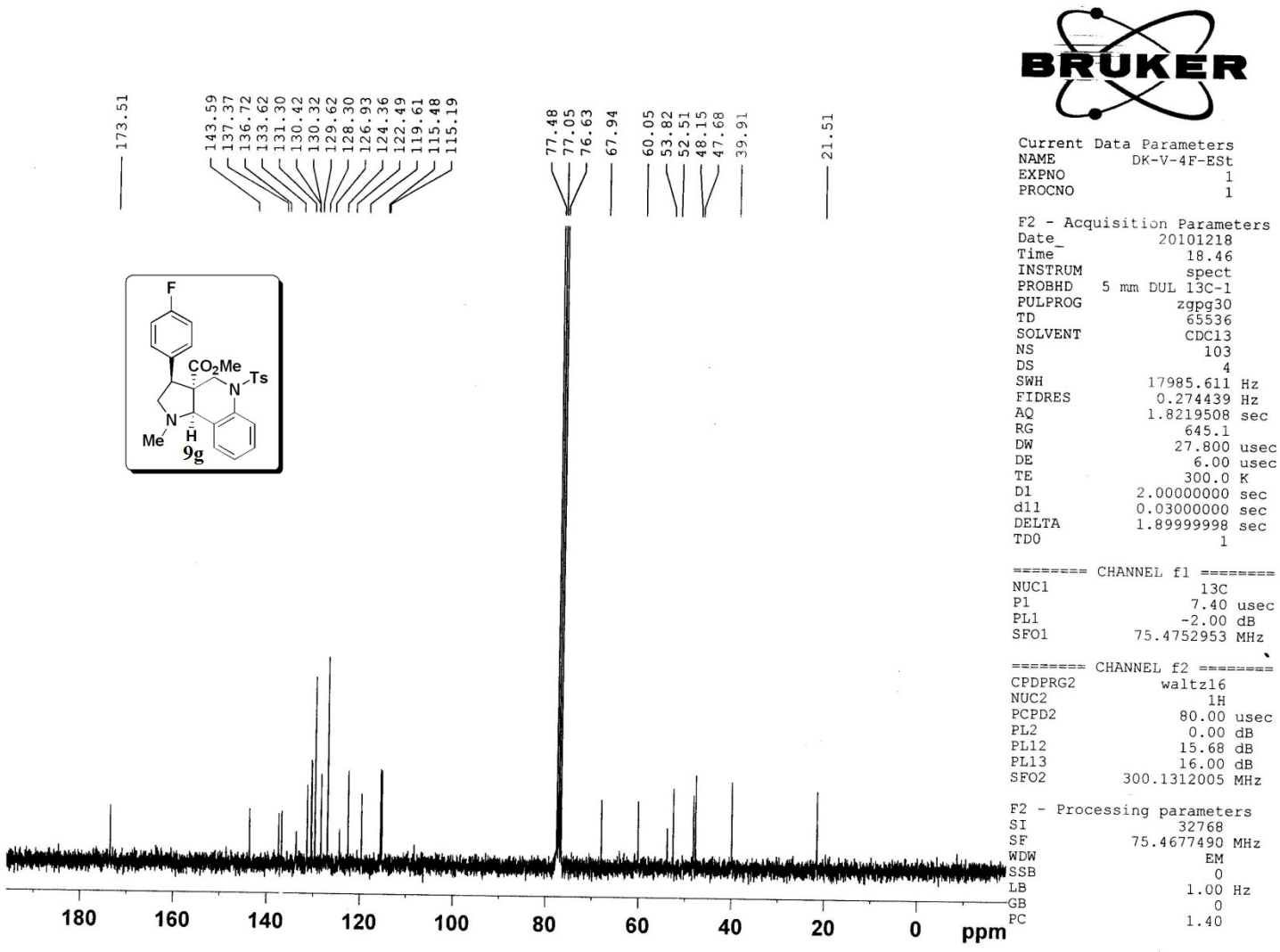
BRUKER

```
Current Data Parameters
NAME          DK-V-4F-ESt
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_          20101218
Time              18.55
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  11
DS                   2
SWH           6172.839 Hz
FIDRES        0.094190 Hz
AQ            5.3084660 sec
RG                90.5
DW              81.000 usec
DE               6.00 usec
TE              300.0 K
D1            1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1              13.15 usec
PL1              0.00 dB
SFO1       300.1318534 MHz

F2 - Processing parameters
SI               32768
SF         300.1300072 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```



BRUKER

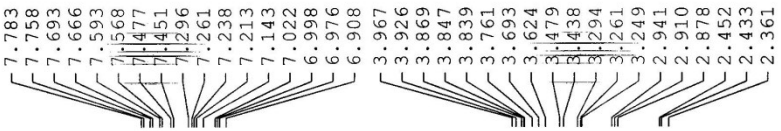
Current Data Parameters
NAME DK-V-4F-ESt
EXPNO 1
PROCNO 1

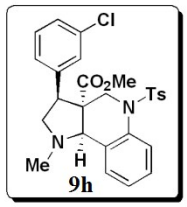
F2 - Acquisition Parameters
Date_ 20101218
Time 18.46
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 103
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 645.1
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 7.40 usec
PL1 -2.00 dB
SFO1 75.4752953 MHz

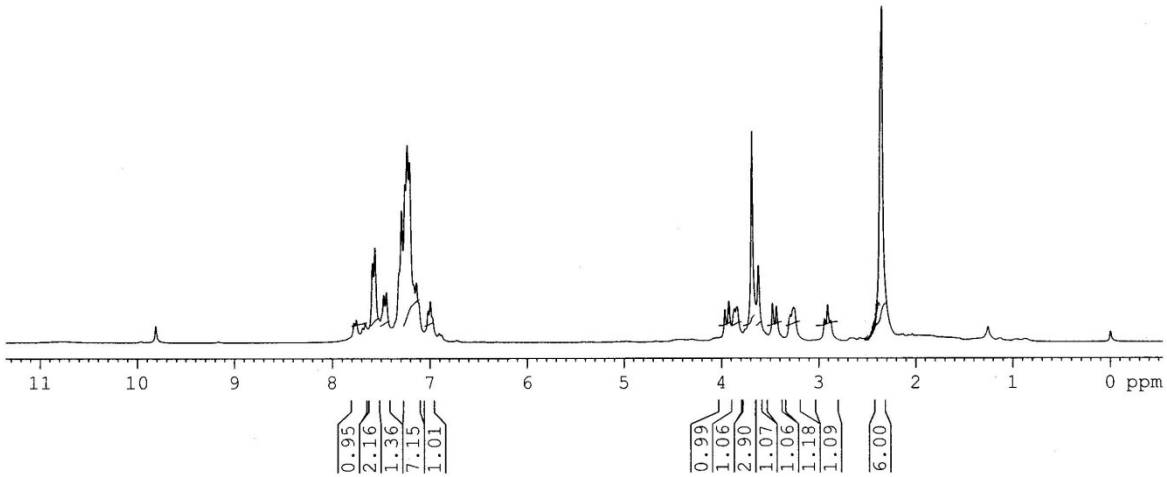
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.68 dB
PL13 16.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





9h

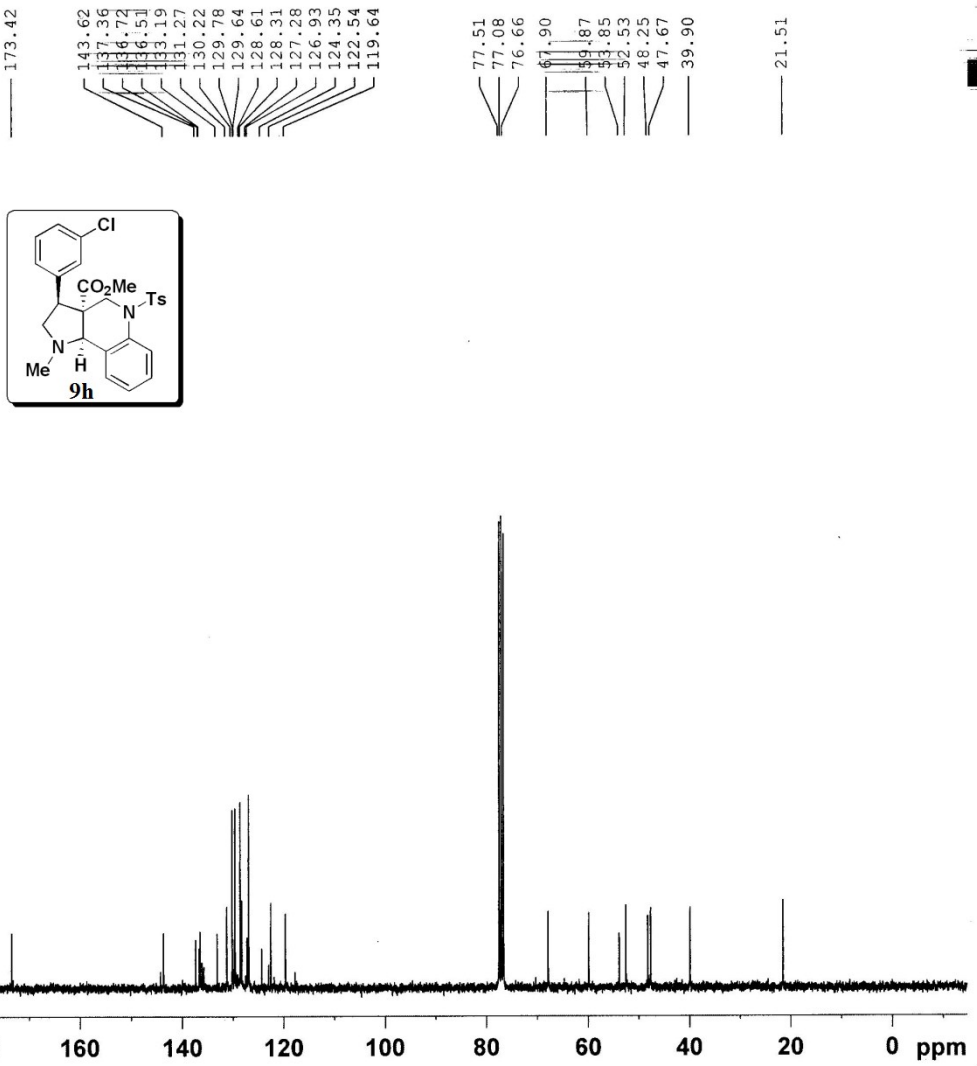



```
Current Data Parameters
NAME      DK-V-3-Cl-Est SA
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20110101
Time               23.35
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6172.839 Hz
FIDRES          0.094190 Hz
AQ              5.3084660 sec
RG                 40.3
DW                81.000 usec
DE                 6.00 usec
TE                300.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                13.15 usec
PL1                0.00 dB
SFO1         300.1318534 MHz

F2 - Processing parameters
SI                 32768
SF          300.1300070 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```



BRUKER

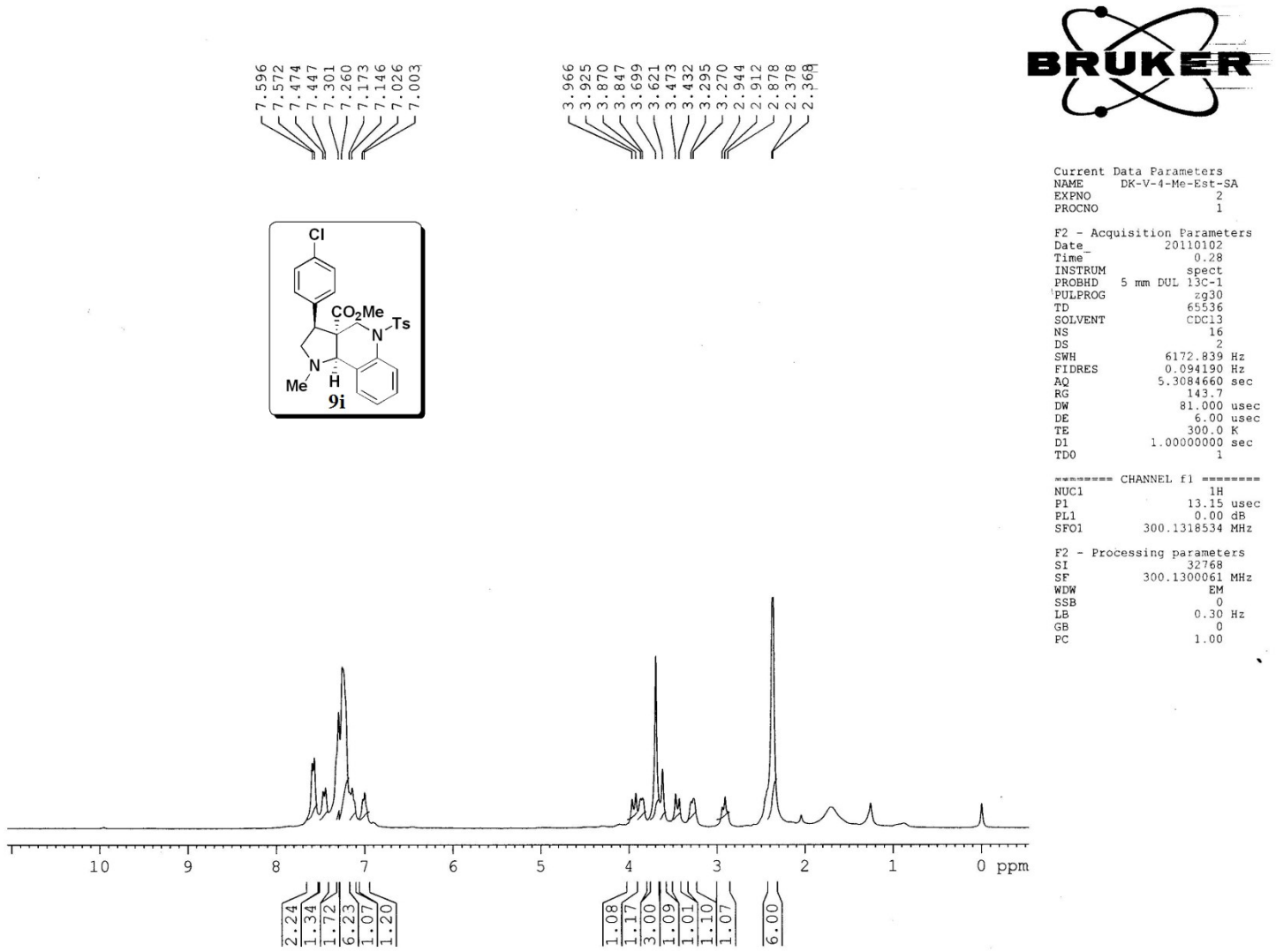
```
Current Data Parameters
NAME      DK-V-3-Cl-Est SA
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_             20110101
Time                 23.49
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                  279
DS                    4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ             1.8219508 sec
RG                645.1
DW                27.800 usec
DE                 6.00 usec
TE                300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  7.40 usec
PL1                -2.00 dB
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              80.00 usec
PL2                 0.00 dB
PL12               15.68 dB
PL13               16.00 dB
SFO2         300.1312005 MHz

F2 - Processing parameters
SI                32768
SF          75.4677490 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



BRUKER

```
Current Data Parameters
NAME      DK-V-4-Me-Est-SA
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_             20110102
Time                 0.28
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH             6172.839 Hz
FIDRES          0.094190 Hz
AQ              5.3084660 sec
RG                  143.7
DW                 81.000 usec
DE                  6.00 usec
TE                 300.0 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 13.15 usec
PL1                 0.00 dB
SFO1          300.1318534 MHz

F2 - Processing parameters
SI                  32768
SF            300.1300061 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   1.00
```

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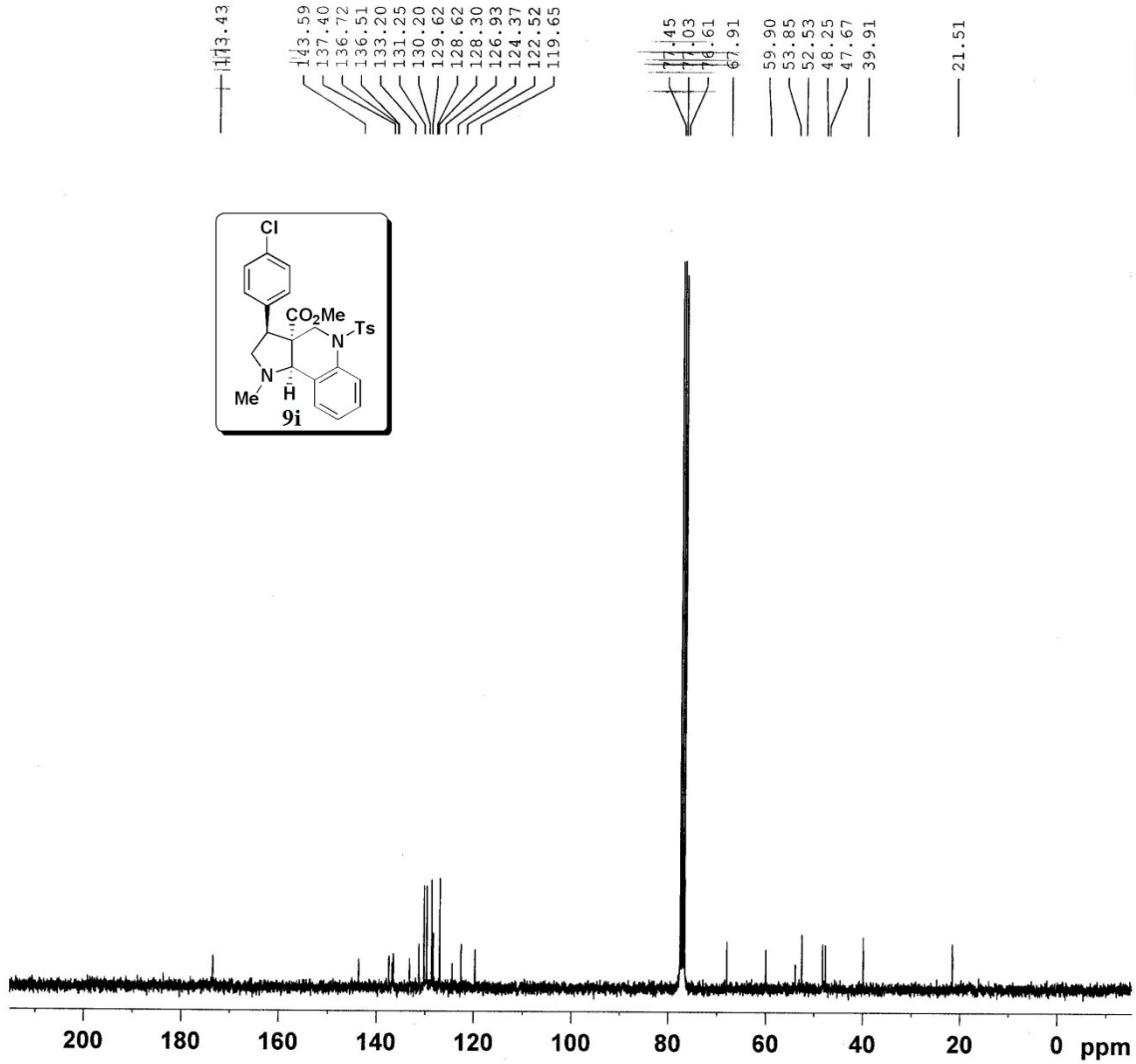


BRUKER

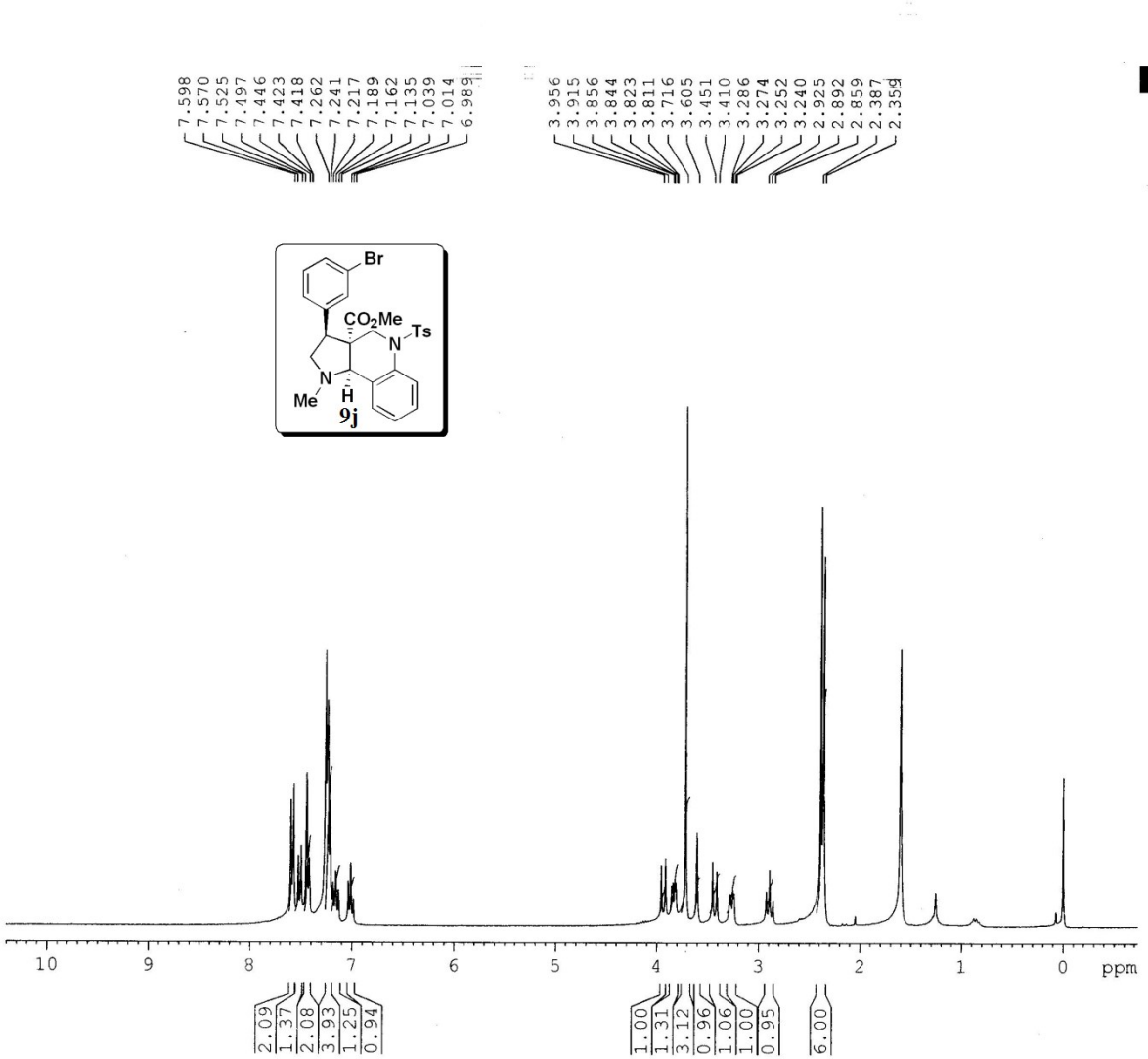
Current Data Parameters
NAME DK-V-4-Me-Est-SA
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110102
Time 0.32
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 414
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 362
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 7.40 usec
PL1 -2.00 dB
SFO1 75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.68 dB
PL13 16.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



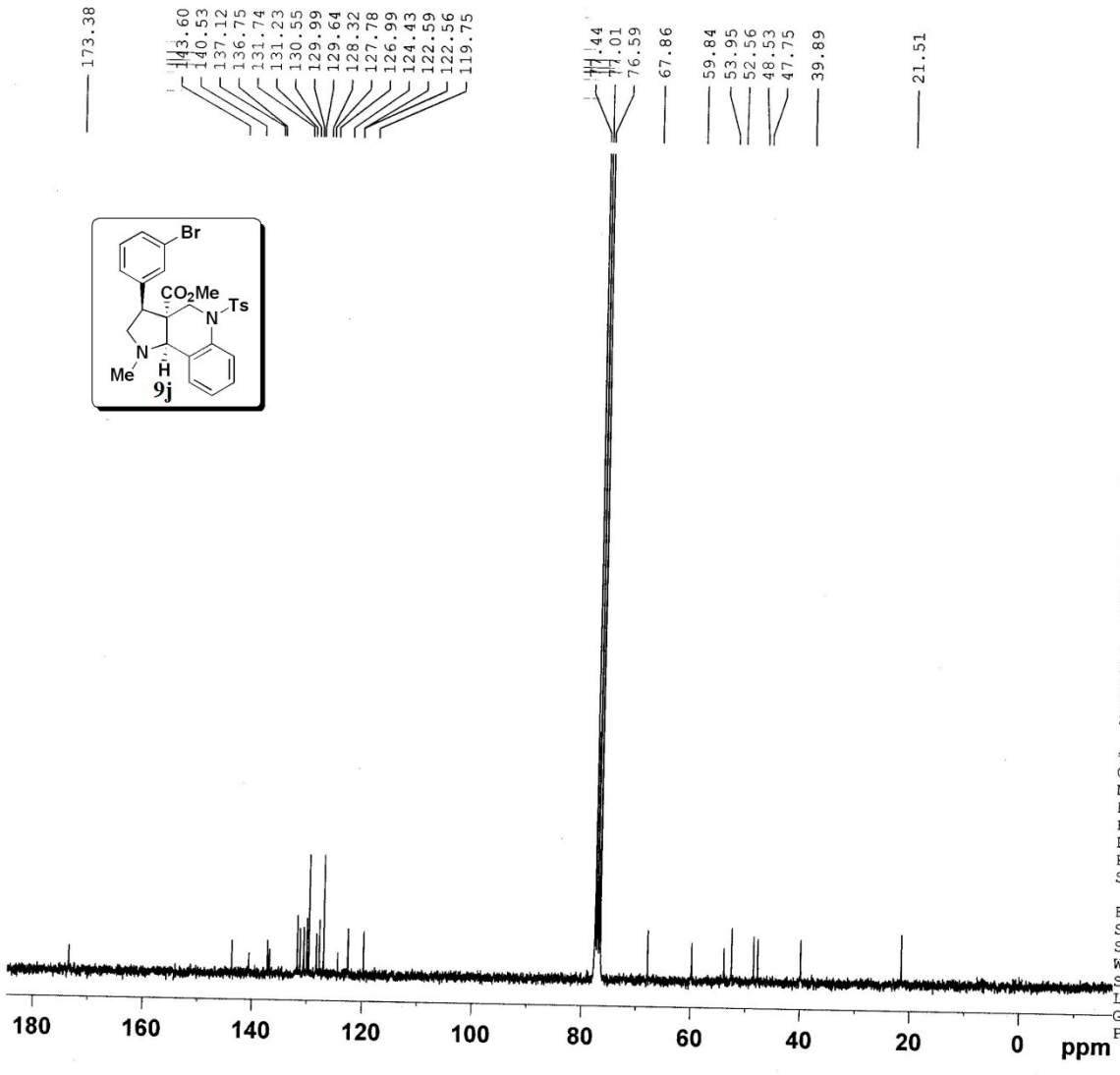
BRUKER

```
Current Data Parameters
NAME       DKV-3-BR-SSA
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20101226
Time              21.48
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            6172.839 Hz
FIDRES        0.094190 Hz
AQ            5.3084660 sec
RG                287.4
DW               81.000 usec
DE                6.00 usec
TE               300.0 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                13.15 usec
PL1                0.00 dB
SFO1        300.1318534 MHz

F2 - Processing parameters
SI                32768
SF          300.1300057 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



BRUKER

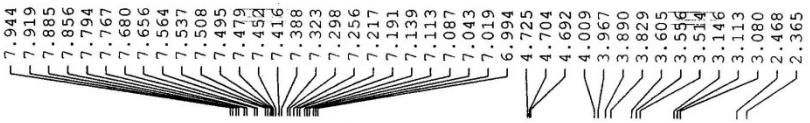
Current Data Parameters
NAME DKV-3-BR-13CNSA
EXPNO 2
PROCNO 1

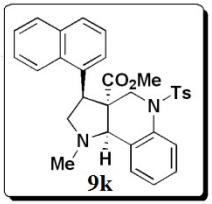
F2 - Acquisition Parameters
Date_ 20101226
Time 21.53
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1787
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 645.1
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 7.40 usec
PL1 -2.00 dB
SFO1 75.4752953 MHz

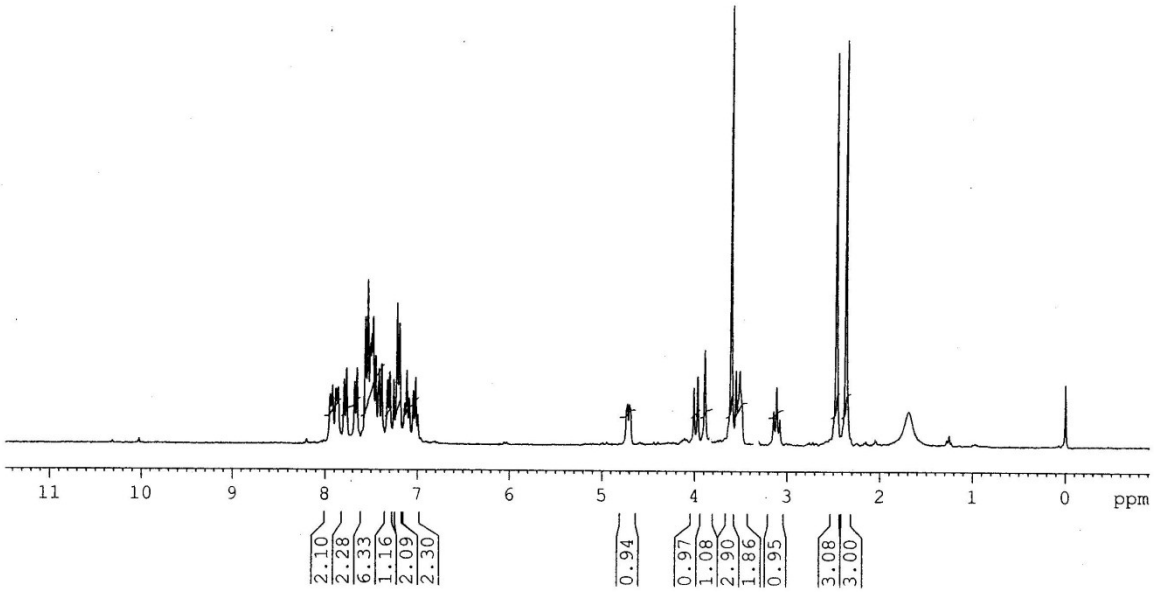
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.68 dB
PL13 16.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





9k



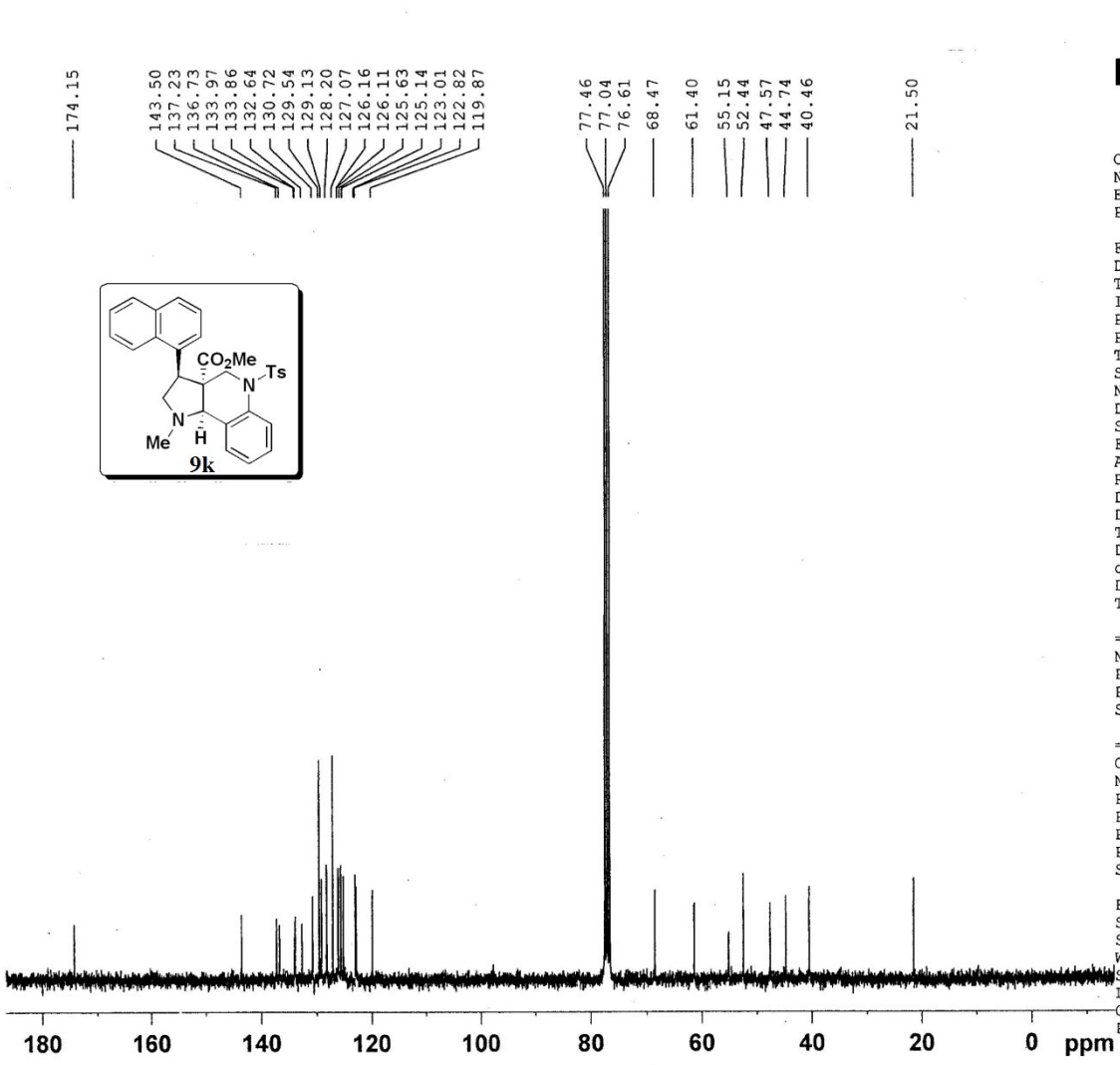
BRUKER

```
Current Data Parameters
NAME          DK-V-Nap-FAST
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20101116
Time                 20.41
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      2
DS                      2
SWH              6172.839 Hz
FIDRES           0.094190 Hz
AQ               5.3084660 sec
RG                  161.3
DW                 81.000 usec
DE                  6.00 usec
TE                 300.0 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 13.15 usec
PL1                 0.00 dB
SFO1          300.1318534 MHz

F2 - Processing parameters
SI                  32768
SF            300.1300077 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



BRUKER

Current Data Parameters

NAME	DK-V-Nap-EST
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20101116
Time	20.50
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	500
DS	4
SWH	17985.611 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	512
DW	27.800 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

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

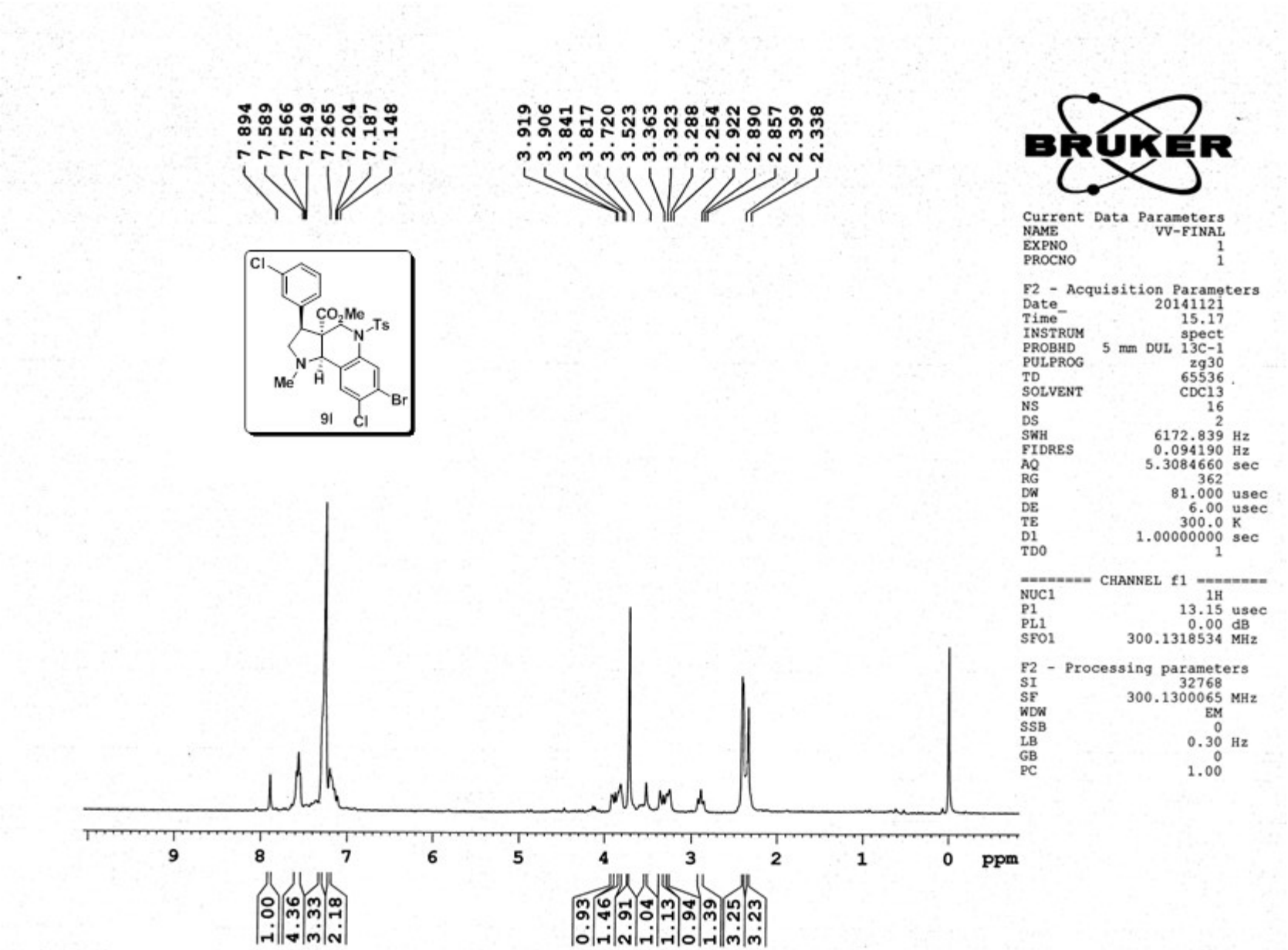
NUC1	13C
P1	7.40 usec
PL1	-2.00 dB
SFO1	75.4752953 MHz

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

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	15.68 dB
PL13	16.00 dB
SFO2	300.1312005 MHz

F2 - Processing parameters

SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



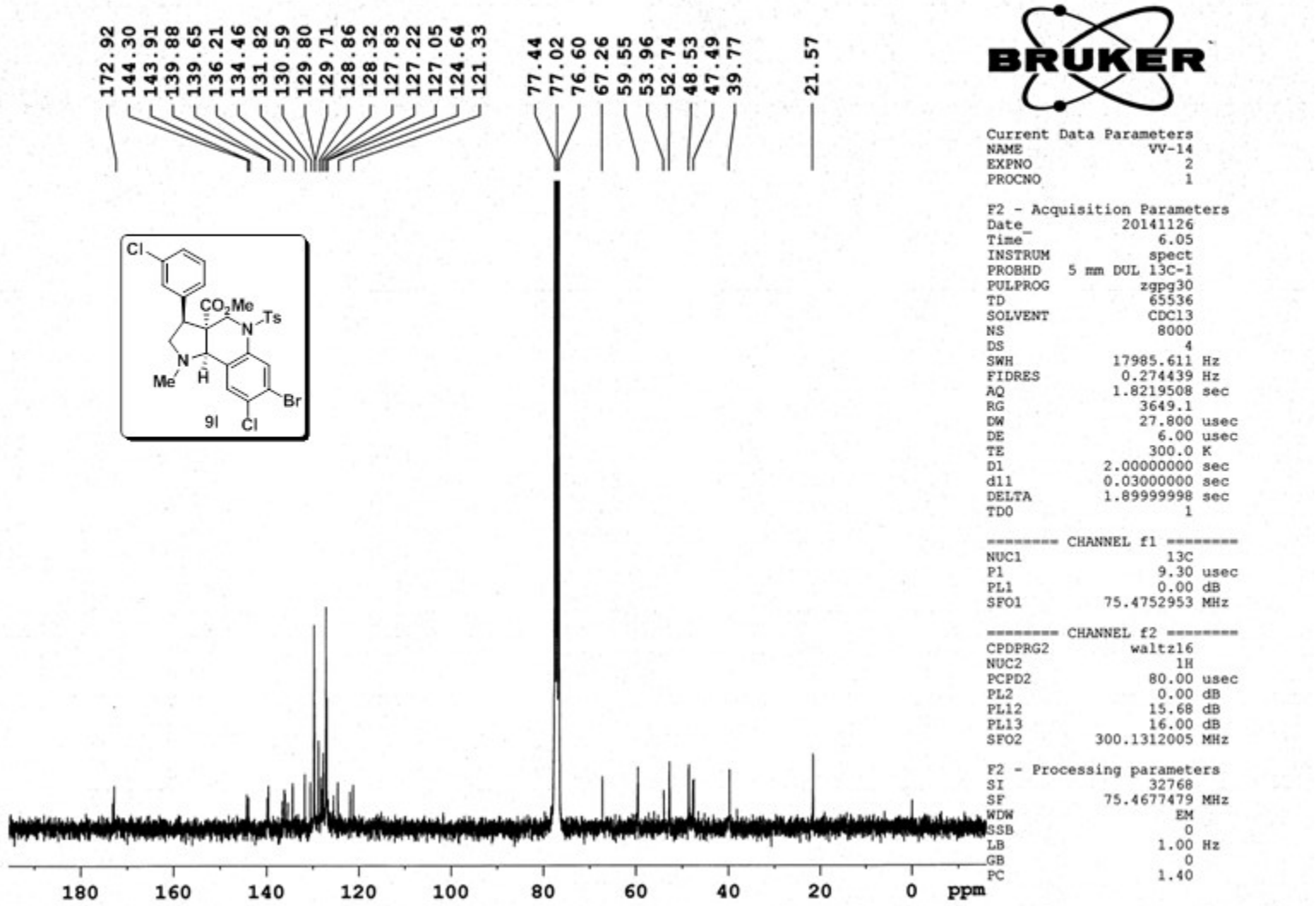
BRUKER

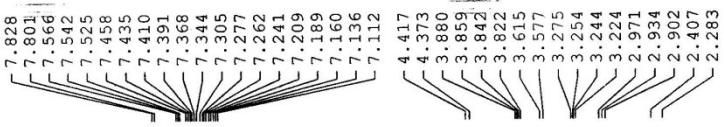
Current Data Parameters
NAME VV-FINAL
EXPNO 1
PROCNO 1

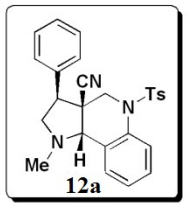
F2 - Acquisition Parameters
Date_ 20141121
Time 15.17
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 362
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.15 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

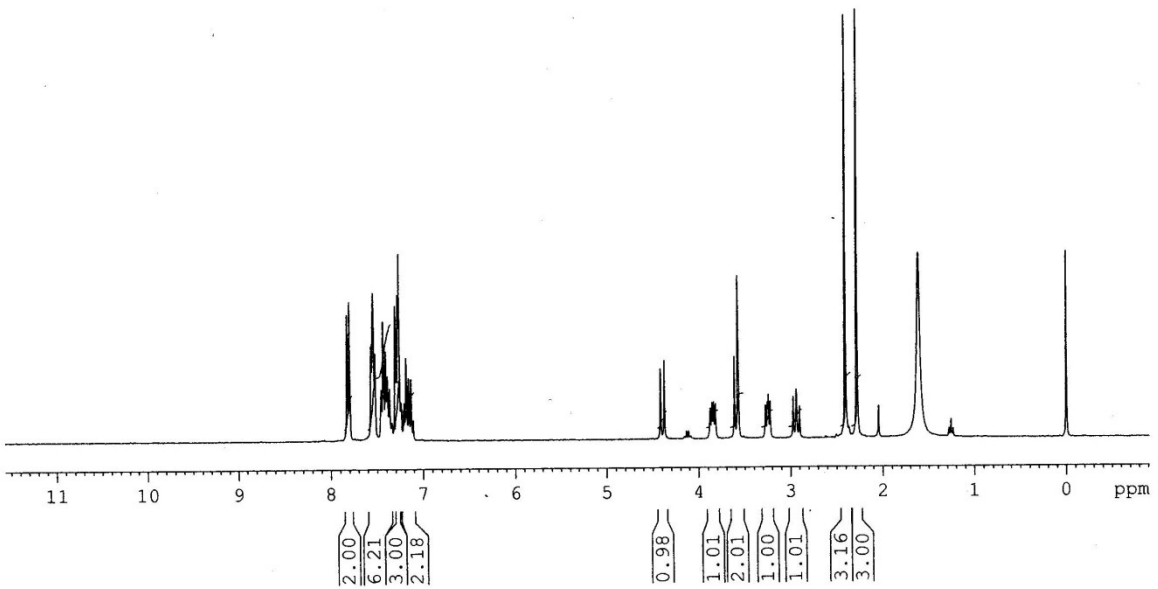
F2 - Processing parameters
SI 32768
SF 300.1300065 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


172.92
144.30
143.91
139.88
139.65
136.21
134.46
131.82
130.59
129.80
129.71
128.86
128.32
127.83
127.22
127.05
124.64
121.33
77.44
77.02
76.60
67.26
59.55
53.96
52.74
48.53
47.49
39.77
21.57
Cl
CO2Me
Ts
N
N
Me
H
Br
Cl
9l
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER
Current Data Parameters
NAME VV-14
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141126
Time 6.05
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 8000
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 3649.1
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 0.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.68 dB
PL13 16.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677479 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





12a



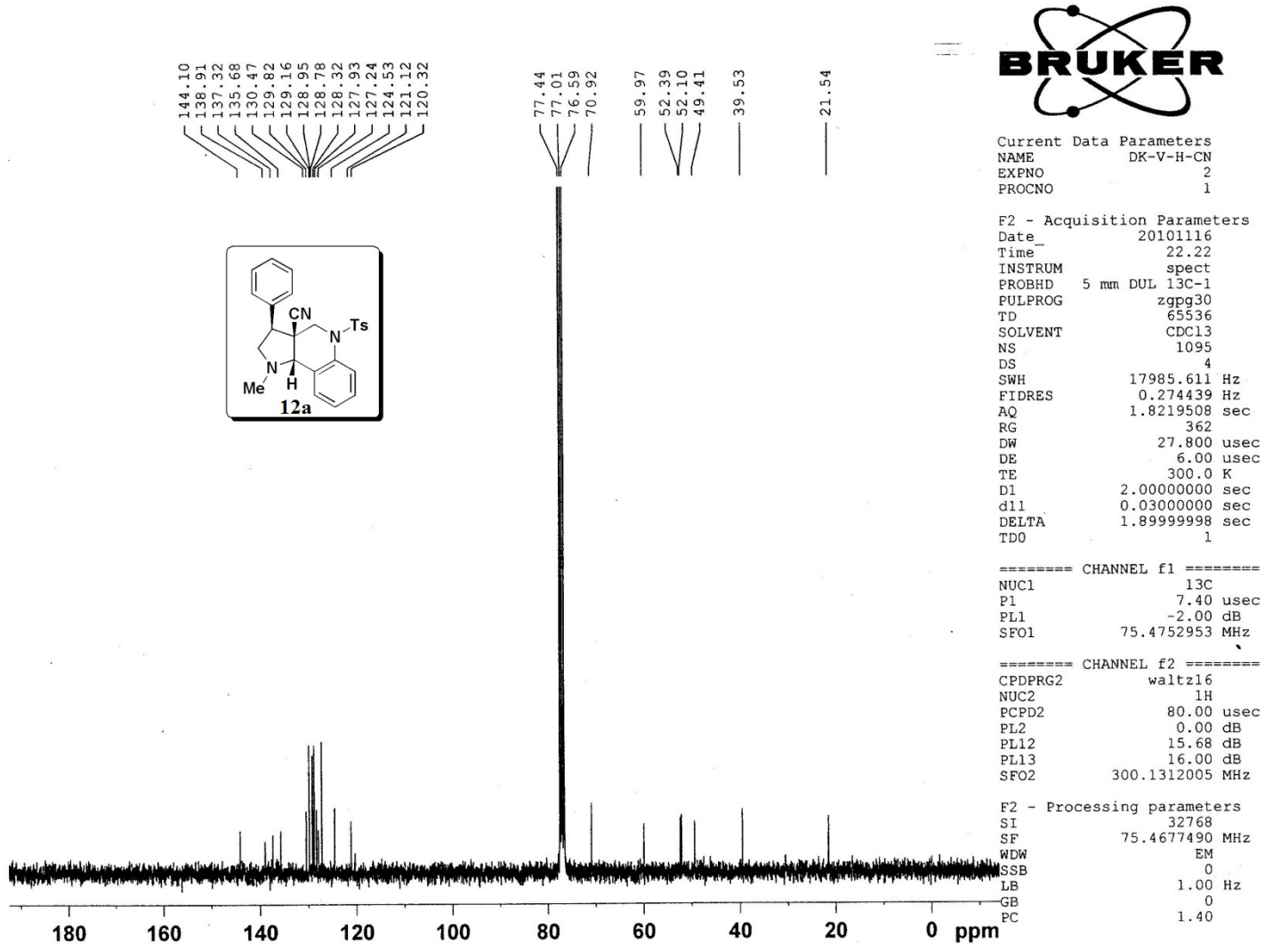
BRUKER

```
Current Data Parameters
NAME                  DK-V-H-CN
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date                   20101116
Time                      21.24
INSTRUM                   spect
PROBHD   5 mm DUL 13C-1
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                 6172.839 Hz
FIDRES              0.094190 Hz
AQ                  5.3084660 sec
RG                       456.1
DW                   81.000 usec
DE                    6.00 usec
TE                     300.0 K
D1                  1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                         1H
P1                    13.15 usec
PL1                    0.00 dB
SFO1             300.1318534 MHz

F2 - Processing parameters
SI                        32768
SF               300.1300059 MHz
WDW                          EM
SSB                           0
LB                     0.30 Hz
GB                            0
PC                         1.00
```



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Current Data Parameters

NAME	DK-V-H-CN
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20101116
Time	22.22
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1095
DS	4
SWH	17985.611 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	362
DW	27.800 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

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

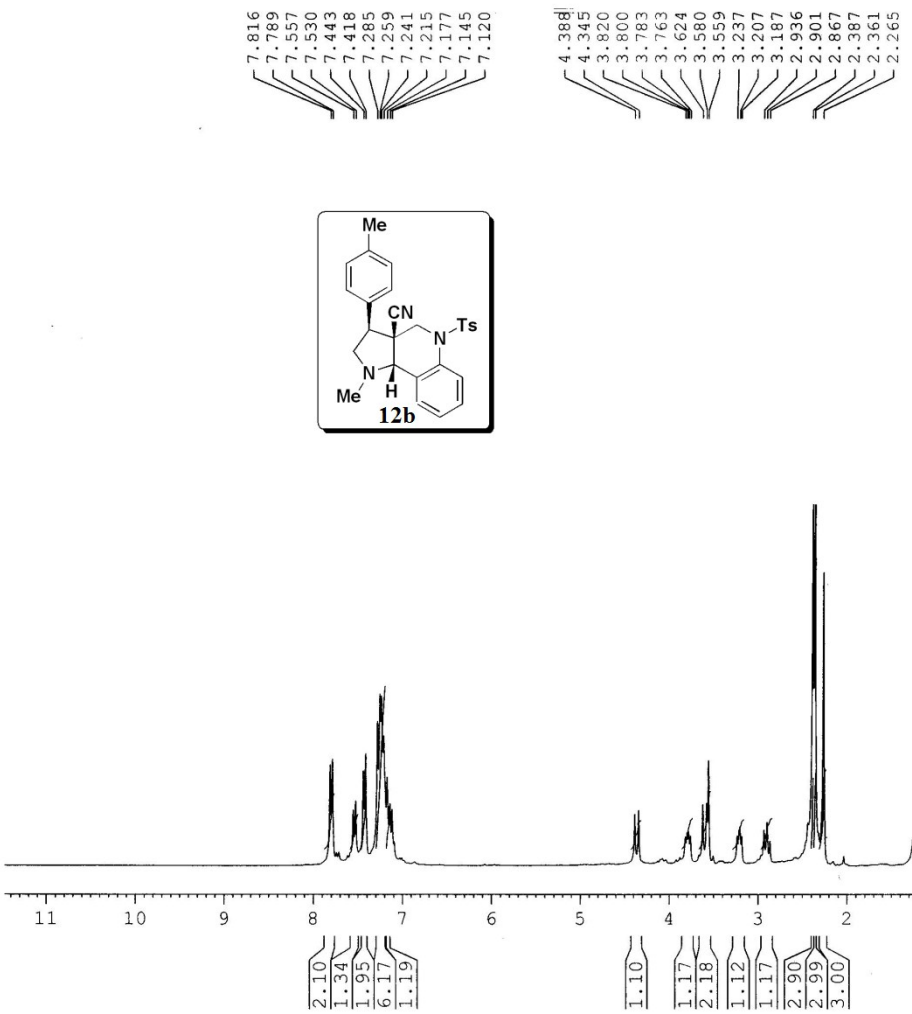
NUC1	13C
P1	7.40 usec
PL1	-2.00 dB
SFO1	75.4752953 MHz

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

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	15.68 dB
PL13	16.00 dB
SFO2	300.1312005 MHz

F2 - Processing parameters

SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



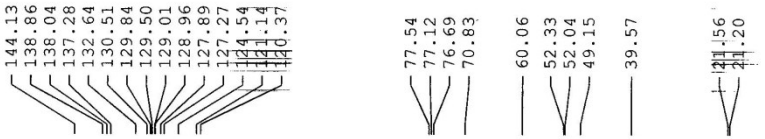
BRUKER

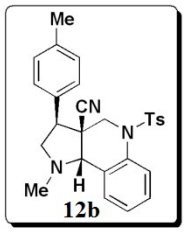
Current Data Parameters
NAME DK-V-4-Me-CN SA
EXPNO 1
PROCNO 1

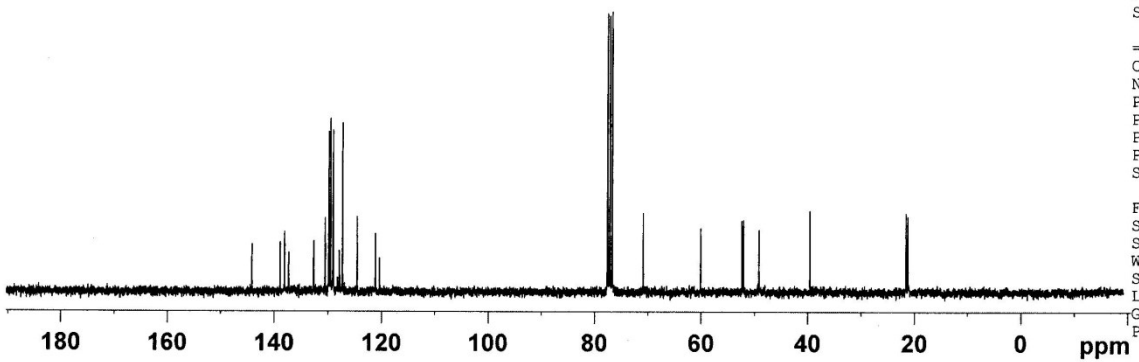
F2 - Acquisition Parameters
Date_ 20101226
Time 0.50
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 7
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 40.3
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.15 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300113 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00







BRUKER

Current Data Parameters
NAME DK-V-4-Me-CN SA
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_	20101226
Time	0.56
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	101
DS	4
SWH	17985.611 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	362
DW	27.800 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

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

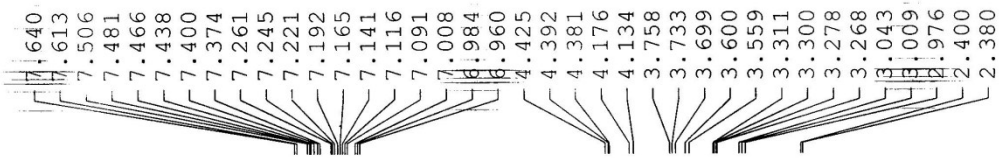
NUC1	13C
P1	7.40 usec
PL1	-2.00 dB
SFO1	75.4752953 MHz

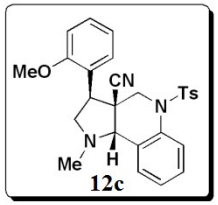
======== CHANNEL f2 ========

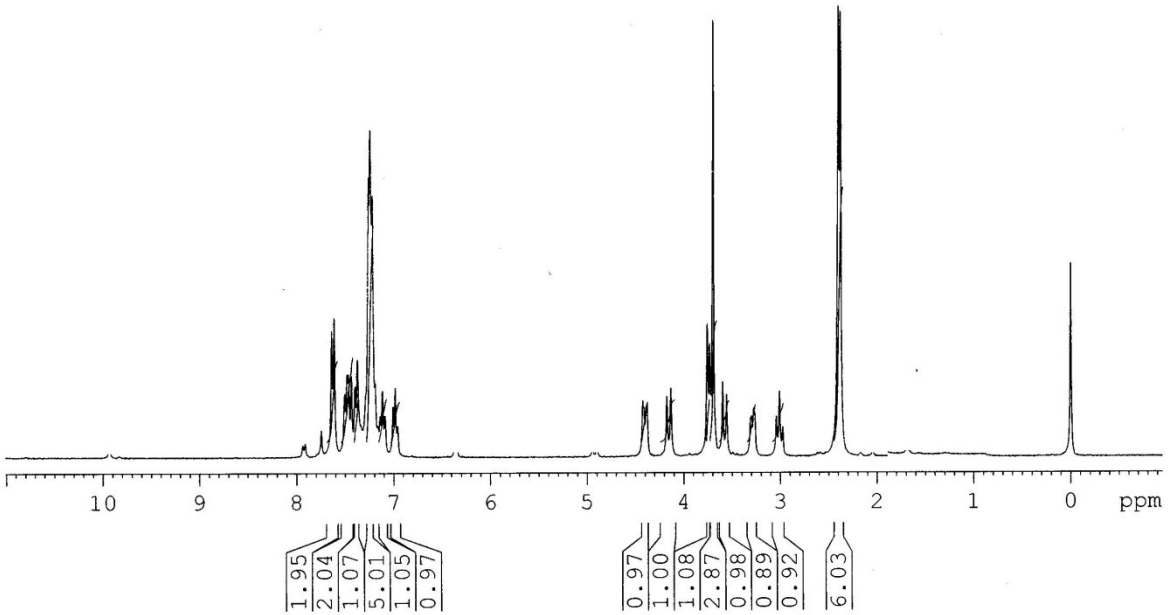
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	15.68 dB
PL13	16.00 dB
SFO2	300.1312005 MHz

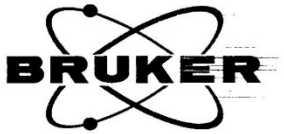
F2 - Processing parameters

SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40







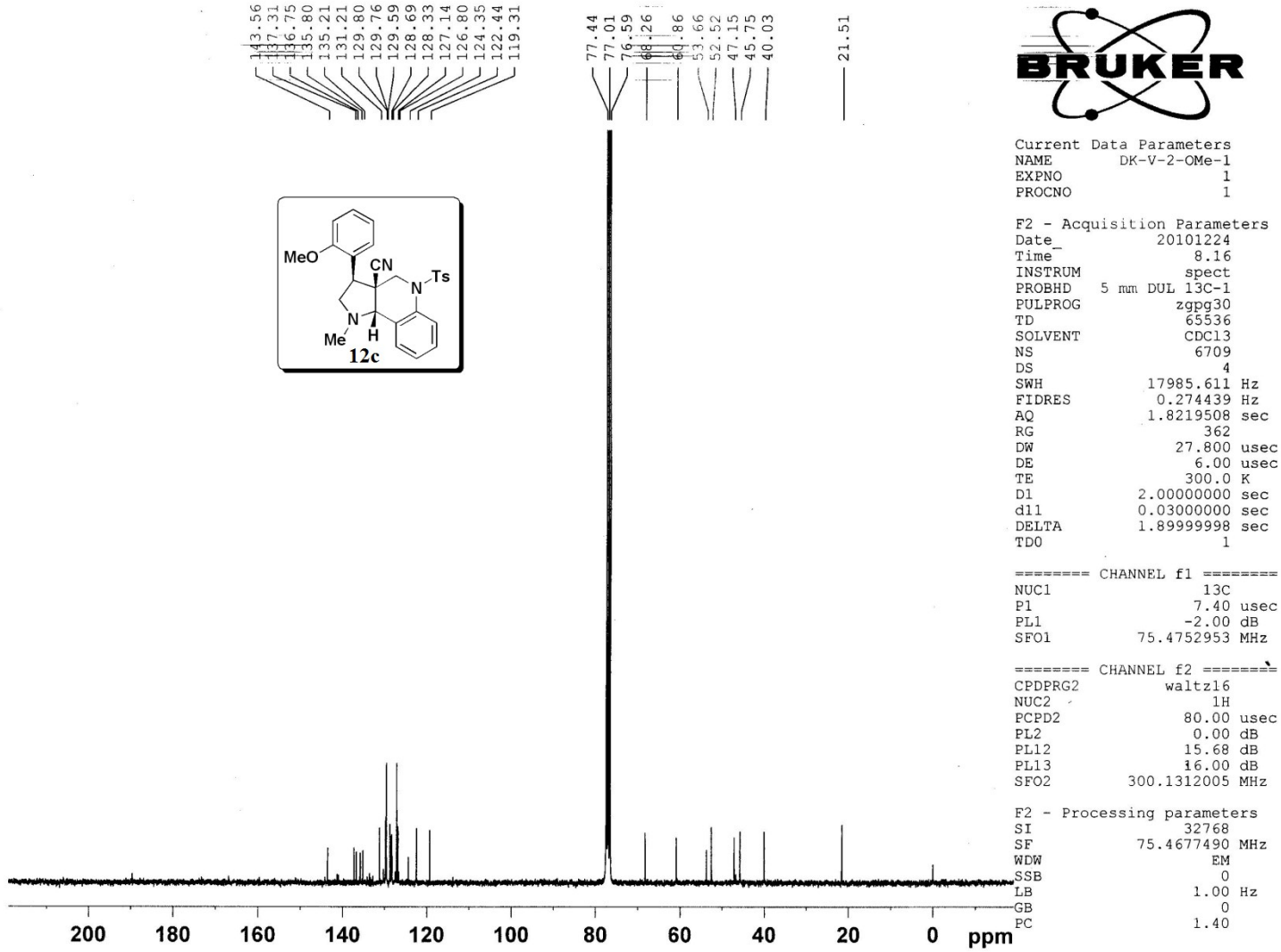



```
Current Data Parameters
NAME      DK-V-2OMe-EST-SA
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20101223
Time                  0.15
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH               6172.839 Hz
FIDRES            0.094190 Hz
AQ                5.3084660 sec
RG                    322.5
DW                 81.000 usec
DE                  6.00 usec
TE                  300.0 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                  13.15 usec
PL1                  0.00 dB
SFO1          300.1318534 MHz

F2 - Processing parameters
SI                   32768
SF            300.1300061 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



BRUKER

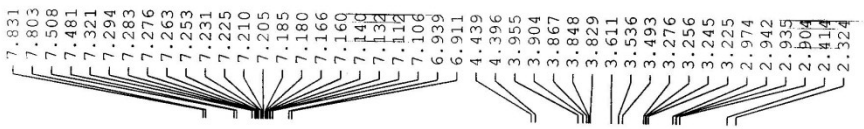
Current Data Parameters
NAME DK-V-2-OMe-1
EXPNO 1
PROCNO 1

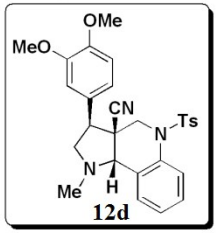
F2 - Acquisition Parameters
Date_ 20101224
Time 8.16
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 6709
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 362
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 7.40 usec
PL1 -2.00 dB
SFO1 75.4752953 MHz

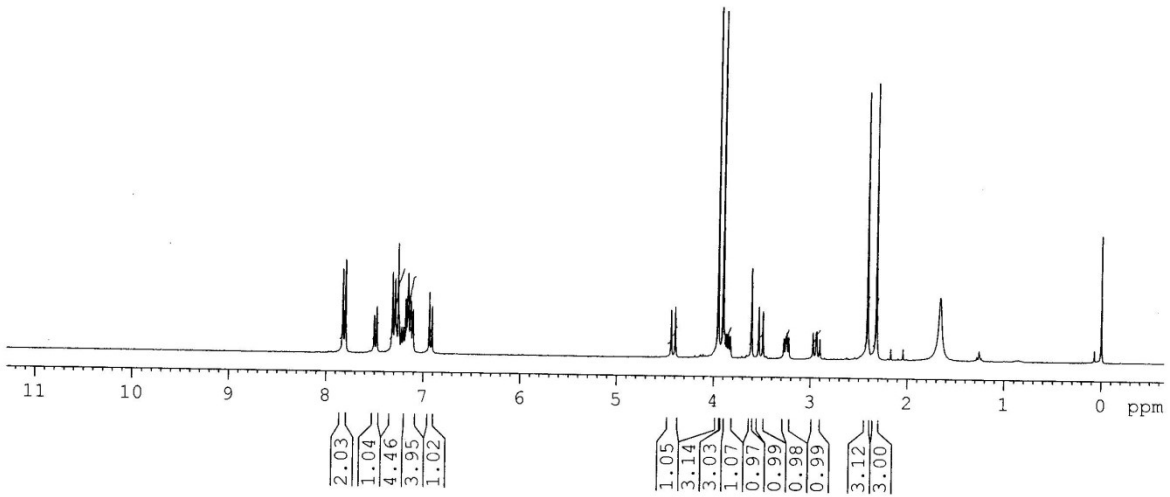
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.68 dB
PL13 16.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





12d



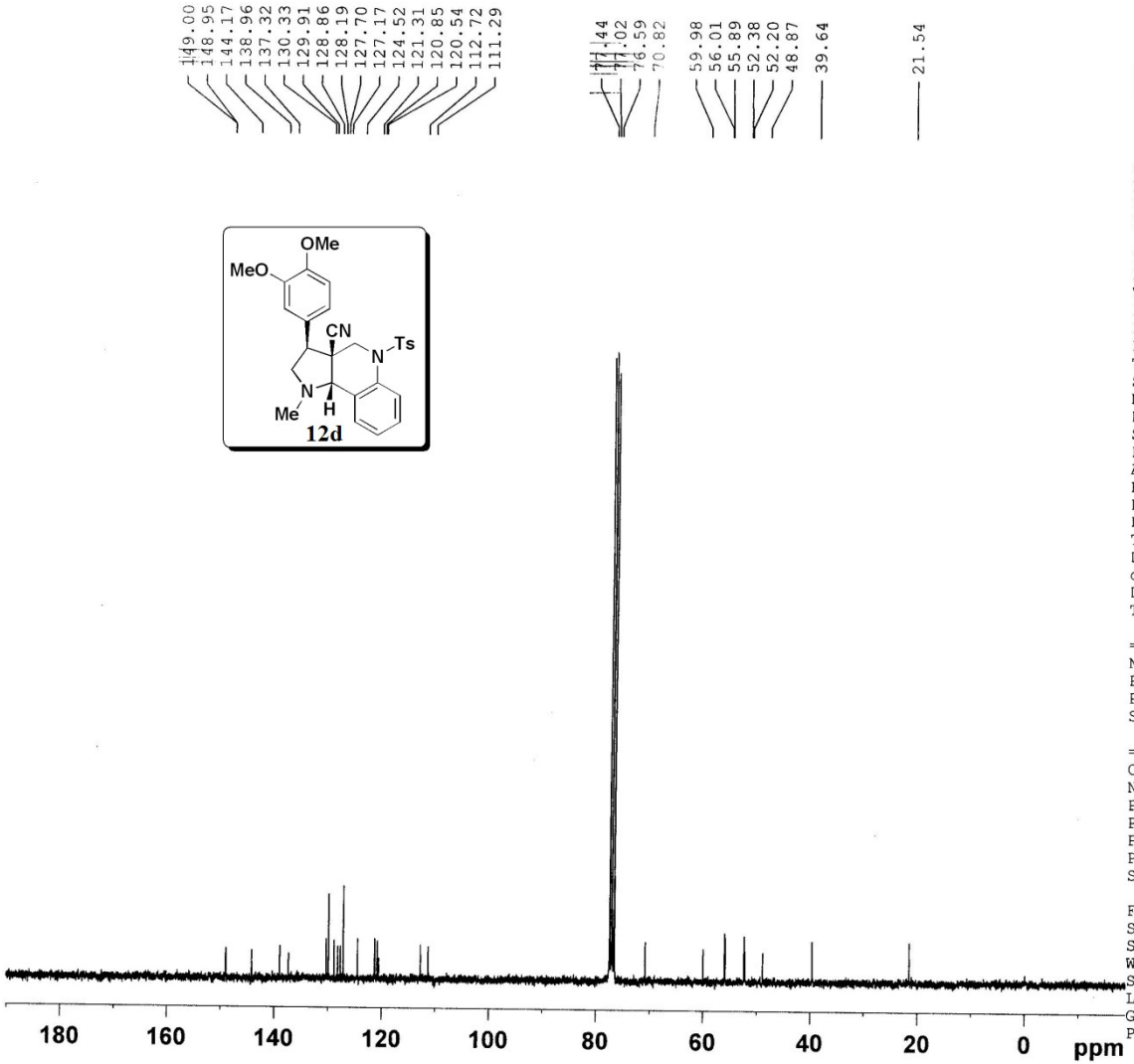
BRUKER

```
Current Data Parameters
NAME      DK-V-OMe2 CN SA
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_         20101225
Time              22.02
INSTRUM           spect
PROBHD  5 mm DUL 13C-1
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                   9
DS                   2
SWH           6172.839 Hz
FIDRES        0.094190 Hz
AQ            5.3084660 sec
RG                 256
DW               81.000 usec
DE                6.00 usec
TE               300.0 K
D1            1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1                13.15 usec
PL1               0.00 dB
SFO1        300.1318534 MHz

F2 - Processing parameters
SI                32768
SF          300.1300055 MHz
WDW                  EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```



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Current Data Parameters
NAME DK-V-OMe2 CN SA
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_	20101225
Time	22.19
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	848
DS	4
SWH	17985.611 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	512
DW	27.800 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

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

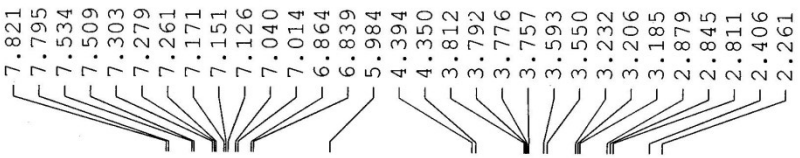
NUC1	13C
P1	7.40 usec
PL1	-2.00 dB
SFO1	75.4752953 MHz

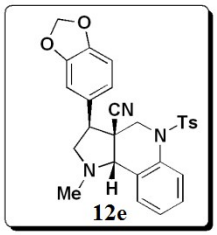
======== CHANNEL f2 ========

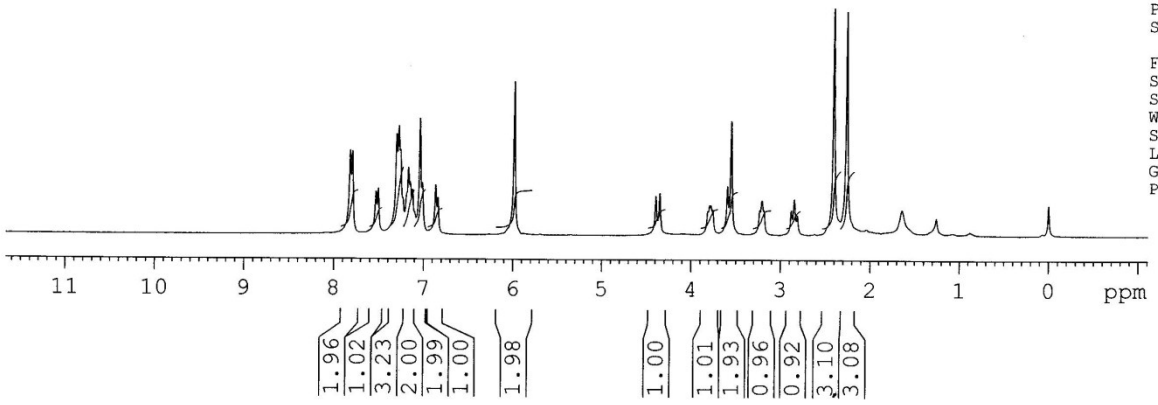
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	15.68 dB
PL13	16.00 dB
SFO2	300.1312005 MHz

F2 - Processing parameters

SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40







Current Data Parameters
NAME DK-V-PIP-CN-SAR
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

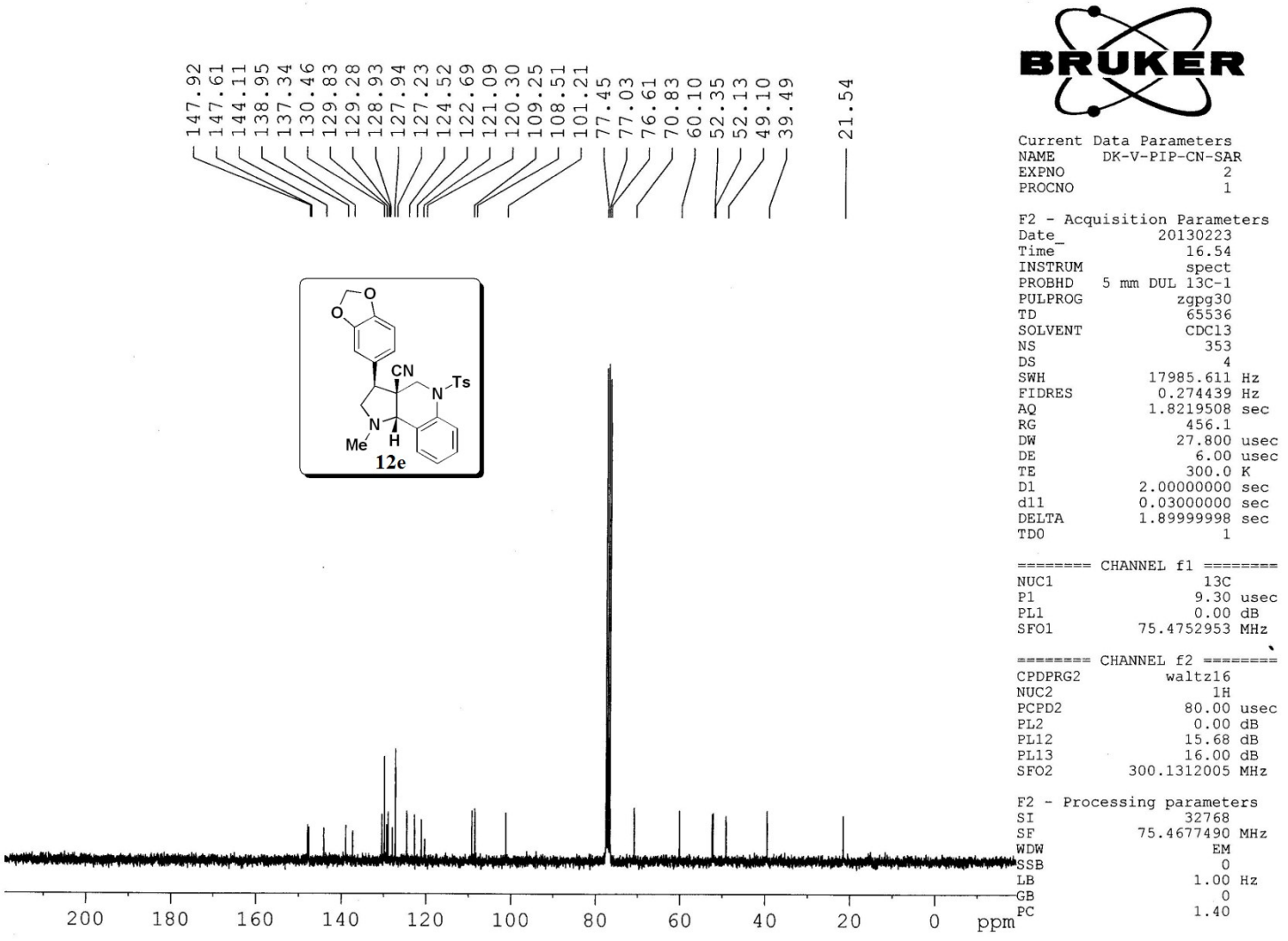
Date_	20130223
Time	16.49
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	6172.839 Hz
FIDRES	0.094190 Hz
AQ	5.3084660 sec
RG	161.3
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
TD0	1

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

NUC1	1H
P1	13.15 usec
PL1	0.00 dB
SFO1	300.1318534 MHz

F2 - Processing parameters

SI	32768
SF	300.1300076 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



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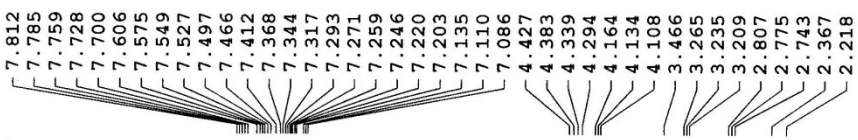
Current Data Parameters
NAME DK-V-PIP-CN-SAR
EXPNO 2
PROCNO 1

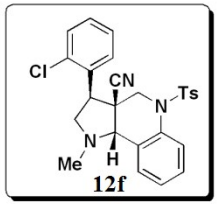
F2 - Acquisition Parameters
Date_ 20130223
Time 16.54
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 353
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 456.1
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 0.00 dB
SFO1 75.4752953 MHz

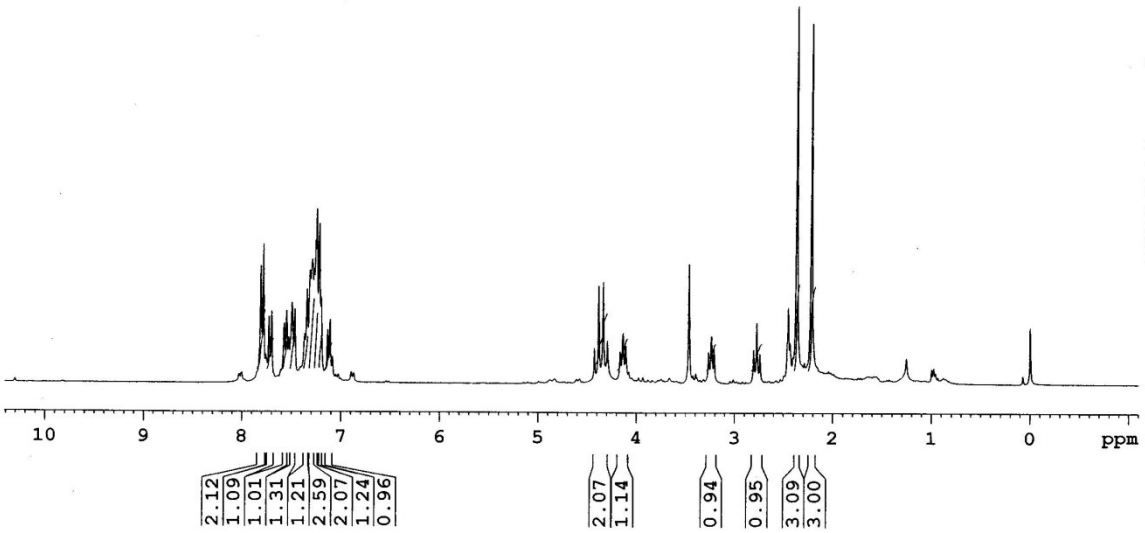
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.68 dB
PL13 16.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





12f



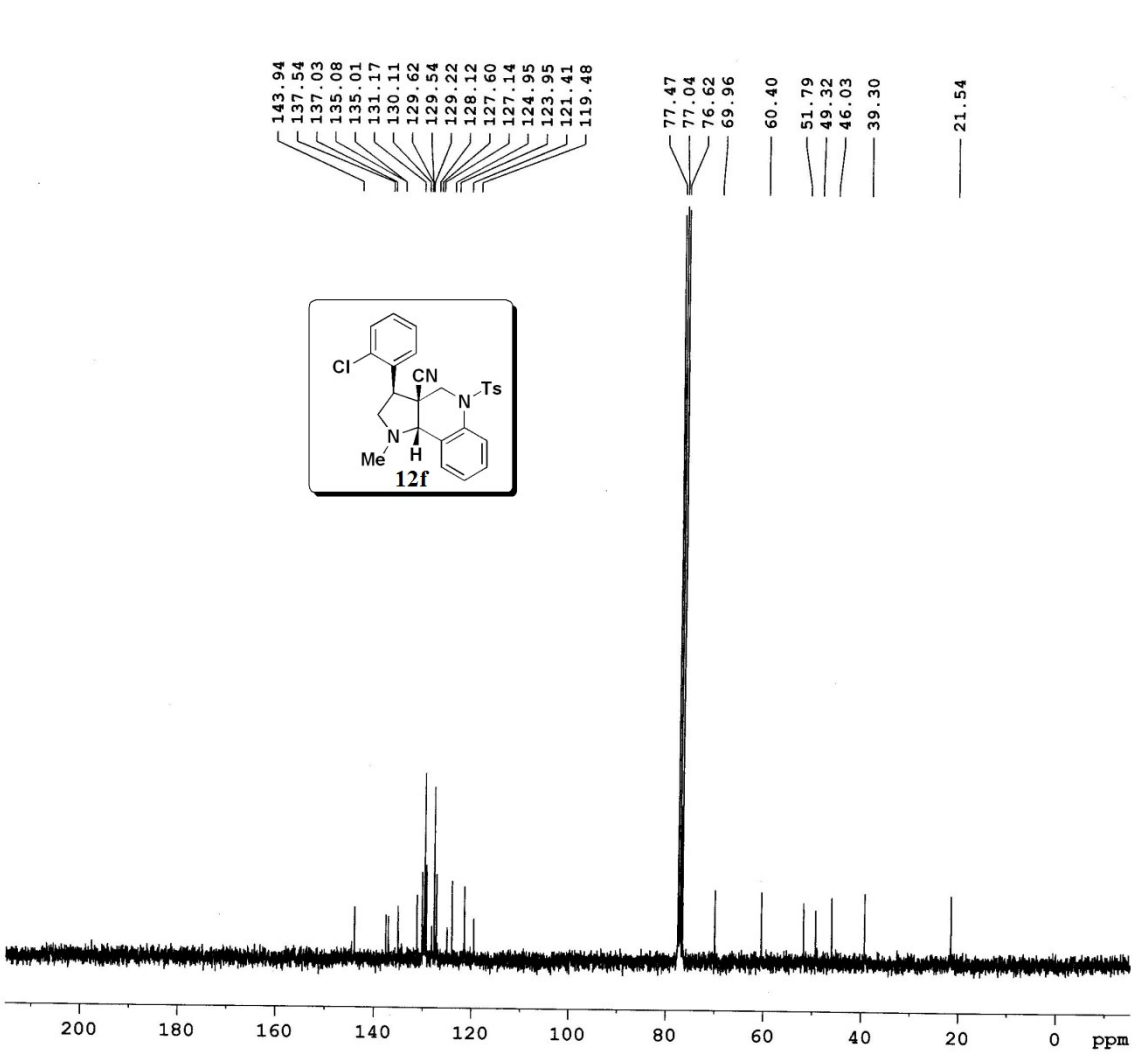
BRUKER

```
Current Data Parameters
NAME      DK-V-2Cl-CN-SA
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20101228
Time                0.23
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    11
DS                     2
SWH            6172.839 Hz
FIDRES         0.094190 Hz
AQ             5.3084660 sec
RG                   128
DW                81.000 usec
DE                 6.00 usec
TE                300.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                13.15 usec
PL1                0.00 dB
SFO1         300.1318534 MHz

F2 - Processing parameters
SI                 32768
SF           300.1300068 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```



BRUKER

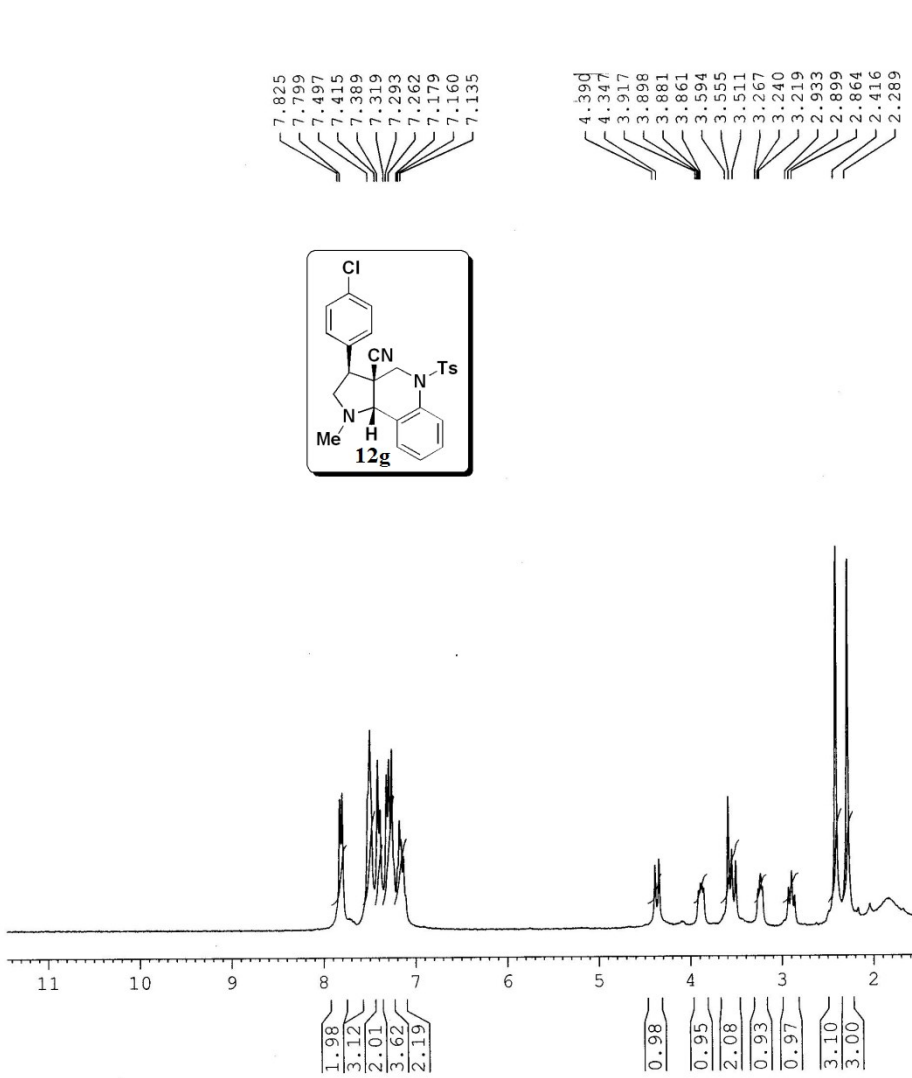
Current Data Parameters
NAME DK-V-2Cl-CN-SA
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20101228
Time 0.31
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 215
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 362
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 7.40 usec
PL1 -2.00 dB
SFO1 75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.68 dB
PL13 16.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



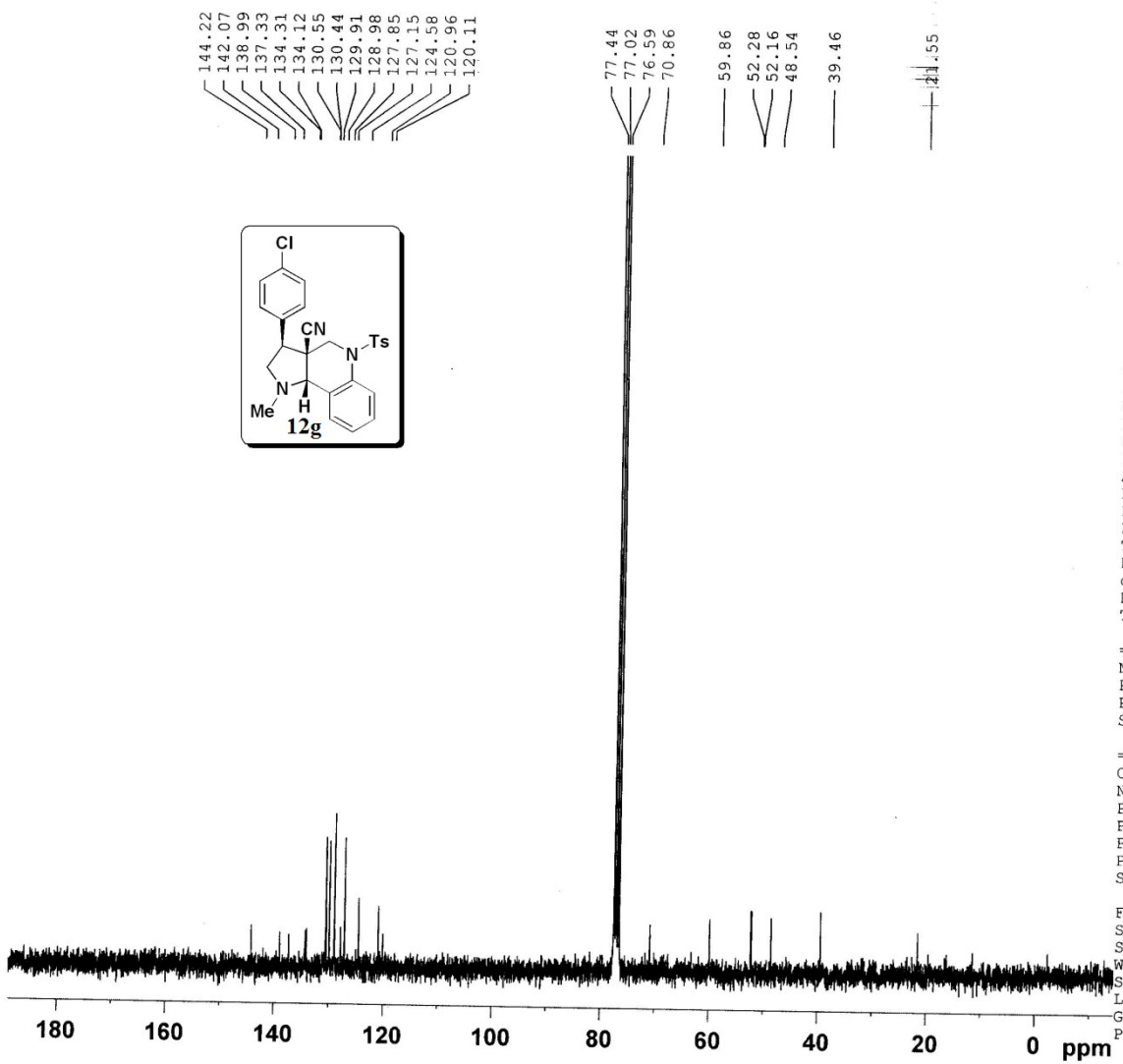
BRUKER

Current Data Parameters
NAME DK-V-4-Cl CN SA
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20101226
Time 0.04
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 287.4
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.15 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300061 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



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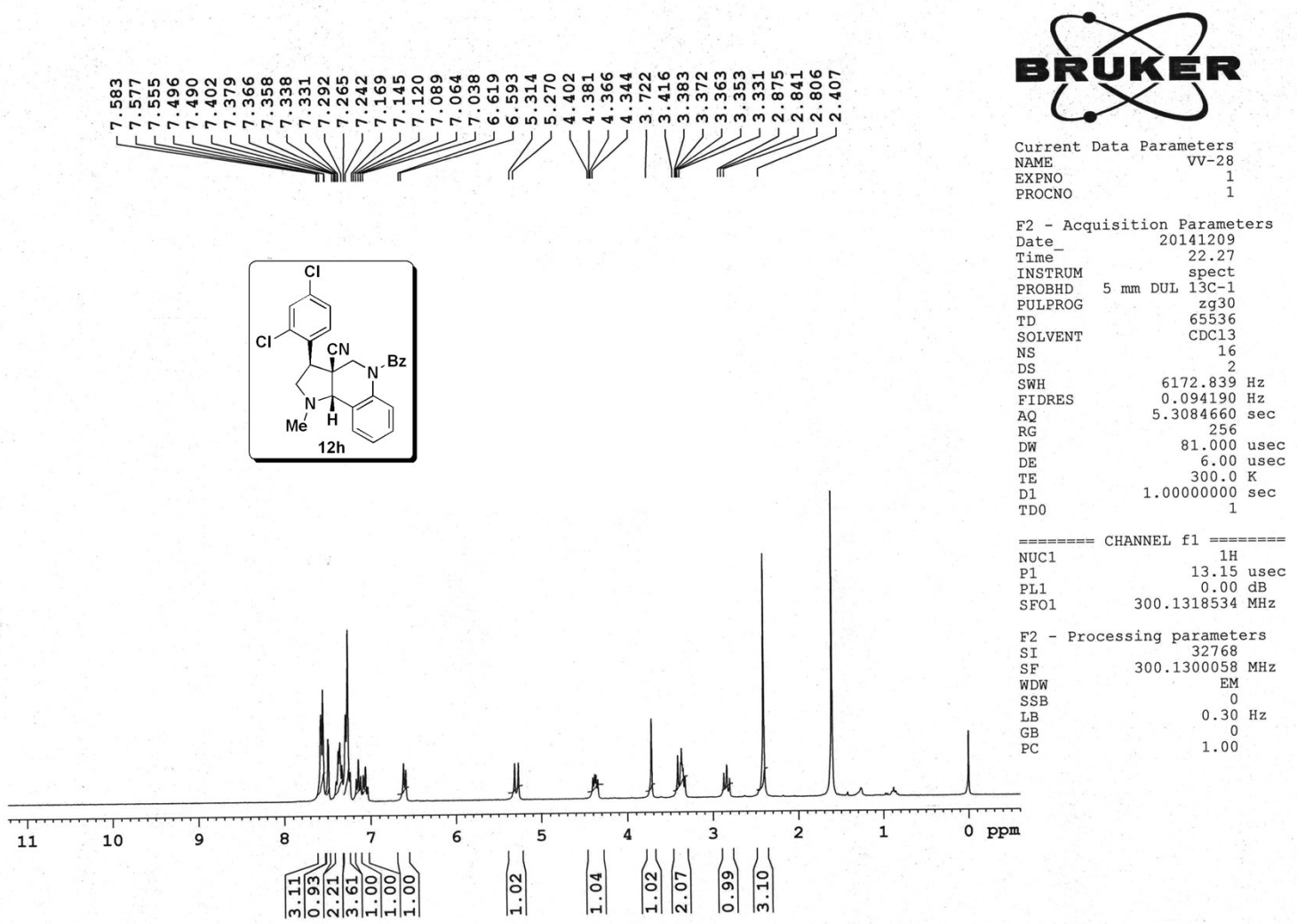
Current Data Parameters
NAME DK-V-4-Cl CN SA
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20101226
Time 0.38
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 500
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 362
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 7.40 usec
PL1 -2.00 dB
SFO1 75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.68 dB
PL13 16.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



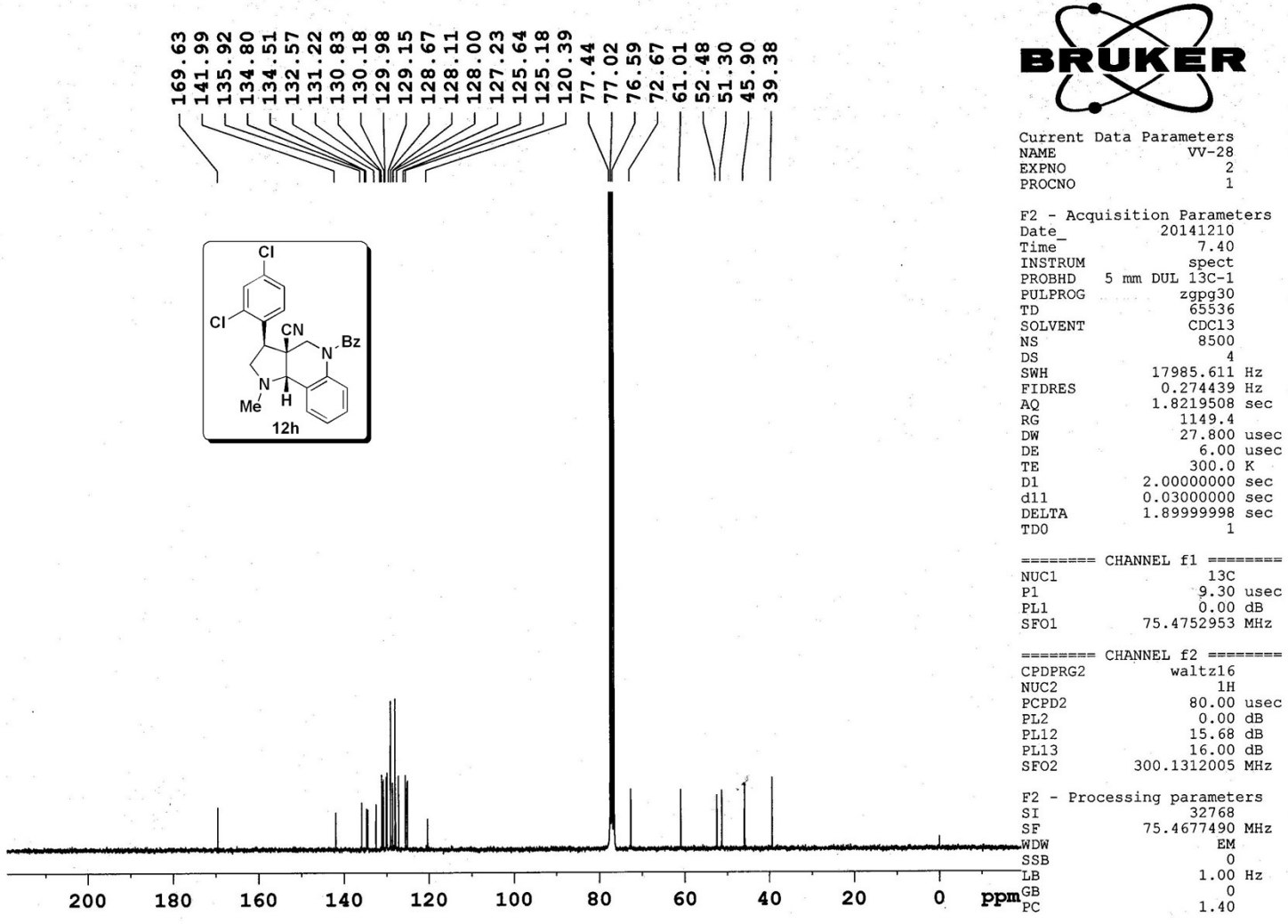
BRUKER

Current Data Parameters
NAME VV-28
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20141209
Time 22.27
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 256
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.15 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300058 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



BRUKER

Current Data Parameters

NAME	VV-28
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20141210
Time	7.40
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	8500
DS	4
SWH	17985.611 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	1149.4
DW	27.800 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

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

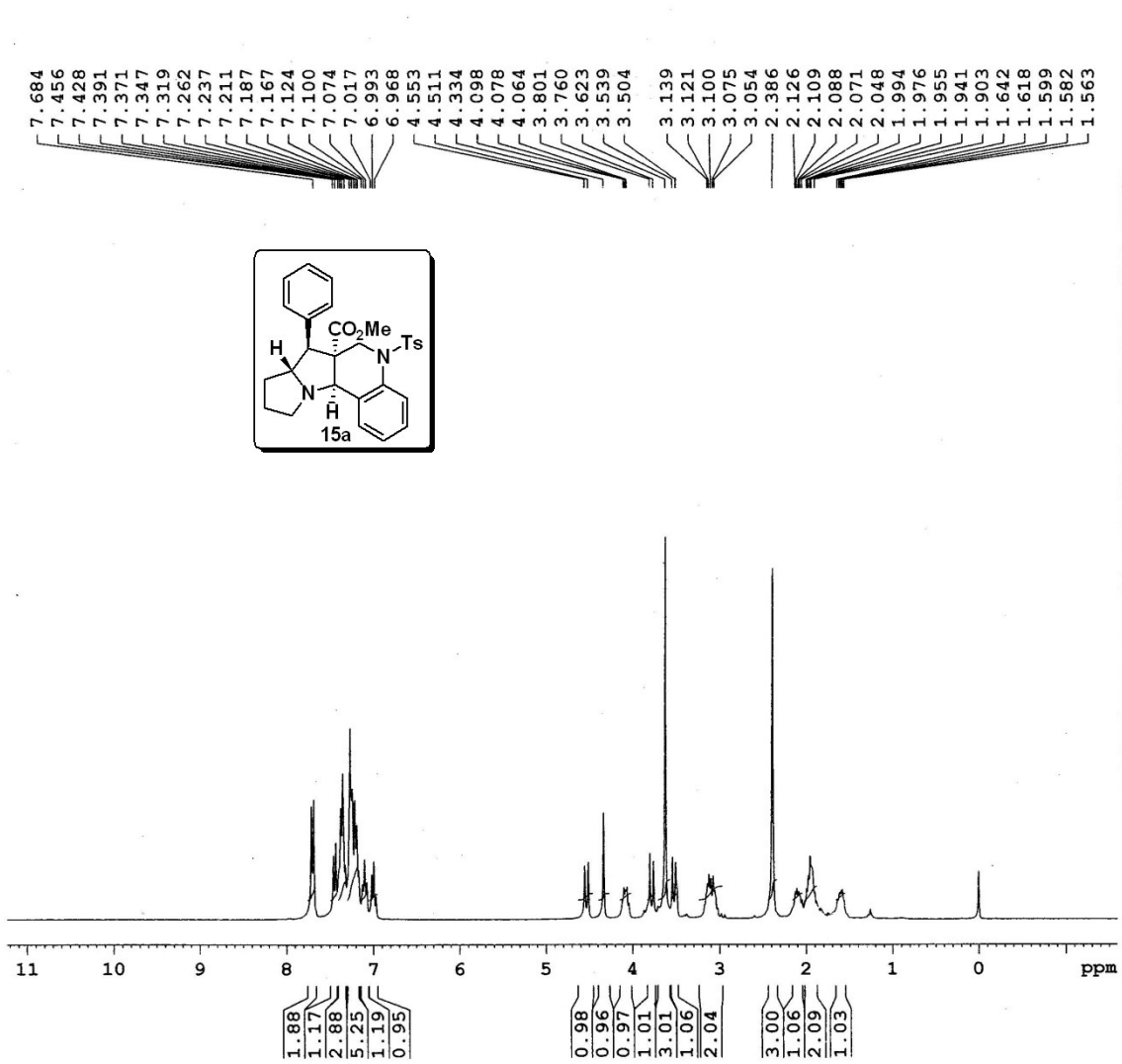
NUC1	13C
P1	9.30 usec
PL1	0.00 dB
SFO1	75.4752953 MHz

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

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	15.68 dB
PL13	16.00 dB
SFO2	300.1312005 MHz

F2 - Processing parameters

SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



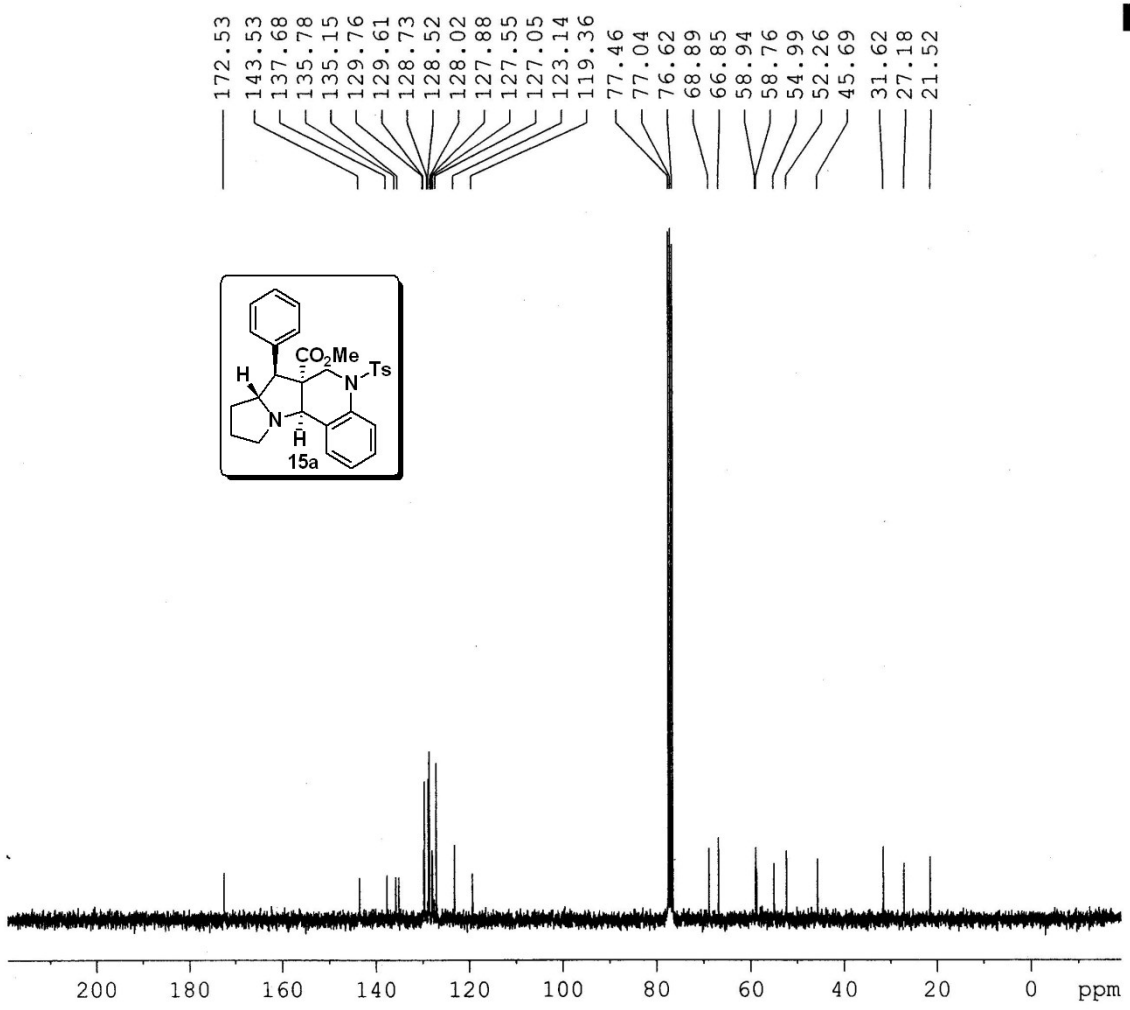
BRUKER

```
Current Data Parameters
NAME          DK-V-Ts-PYZ
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20110923
Time                19.43
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              6172.839 Hz
FIDRES           0.094190 Hz
AQ               5.3084660 sec
RG                 143.7
DW                81.000 usec
DE                 6.00 usec
TE                300.0 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 13.15 usec
PL1                 0.00 dB
SFO1         300.1318534 MHz

F2 - Processing parameters
SI                  32768
SF           300.1300061 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                 1.00
```



BRUKER

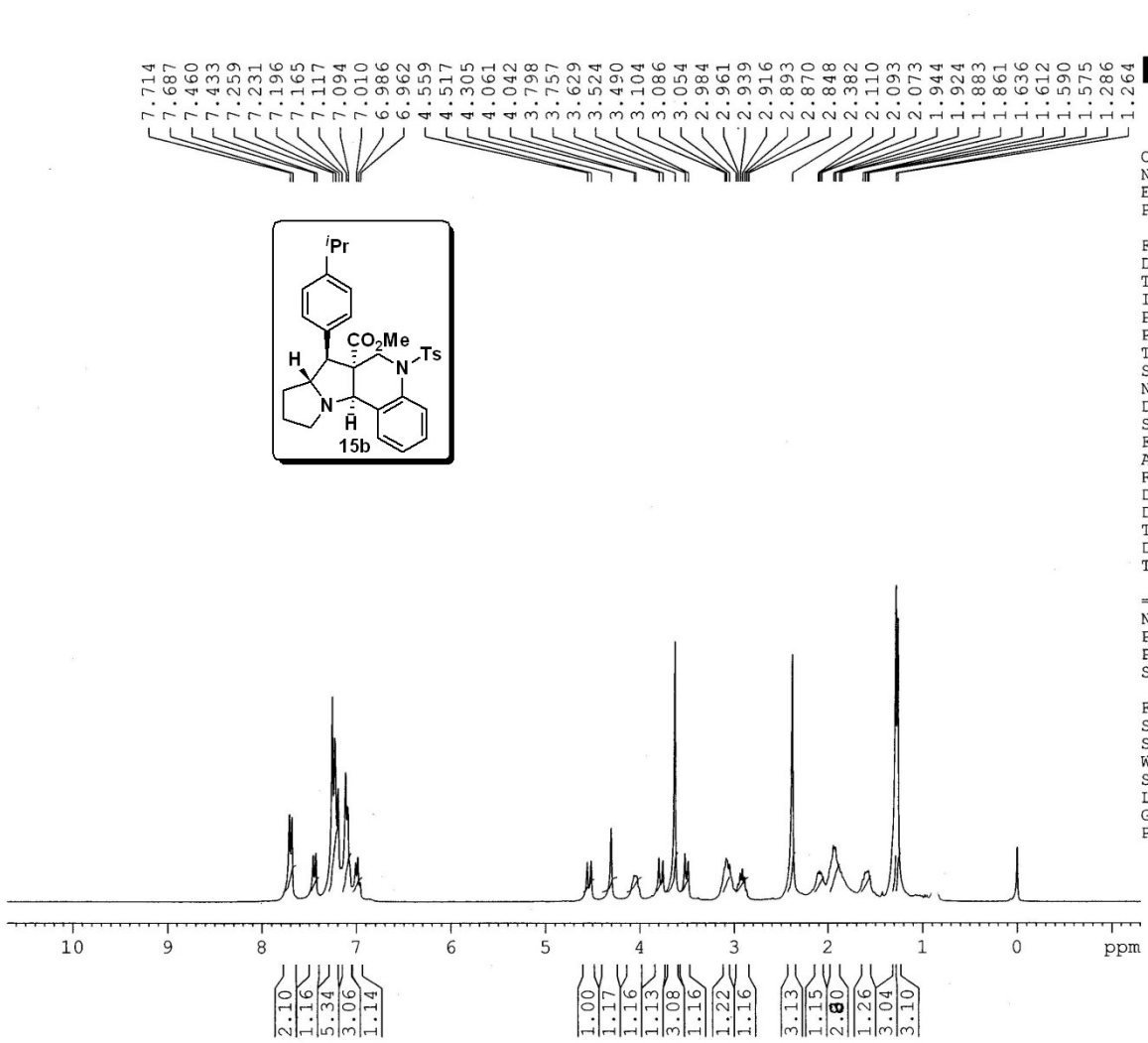
```
Current Data Parameters
NAME          DK-V-Ts-PYZ
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20110923
Time                19.48
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                 65536
SOLVENT             CDCl3
NS                    261
DS                      4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ             1.8219508 sec
RG                 724.1
DW                27.800 usec
DE                 6.00 usec
TE                300.0 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.30 usec
PL1                  0.00 dB
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                 0.00 dB
PL12               15.68 dB
PL13               16.00 dB
SFO2         300.1312005 MHz

F2 - Processing parameters
SI                  32768
SF           75.4677490 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```



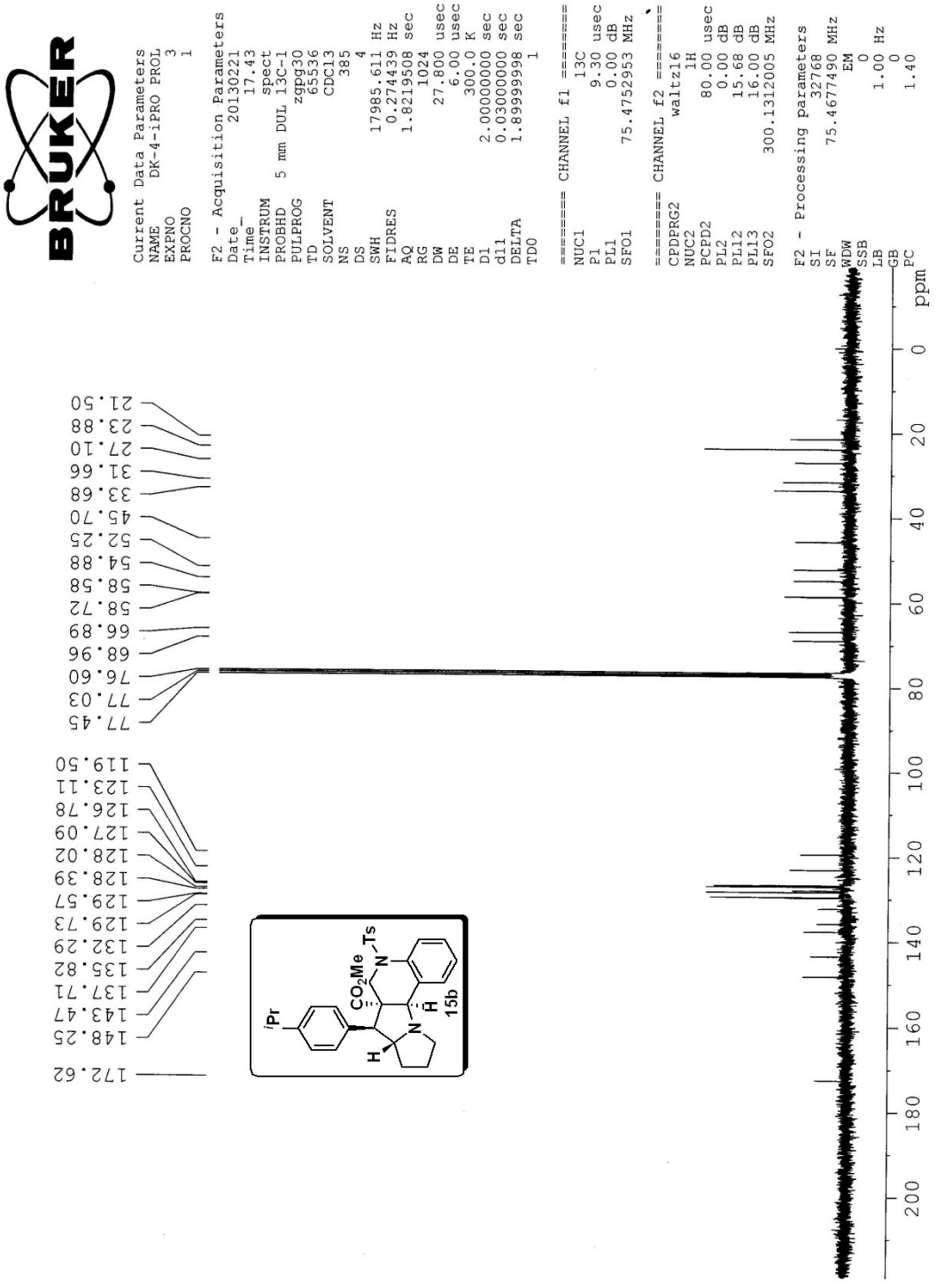
BRUKER

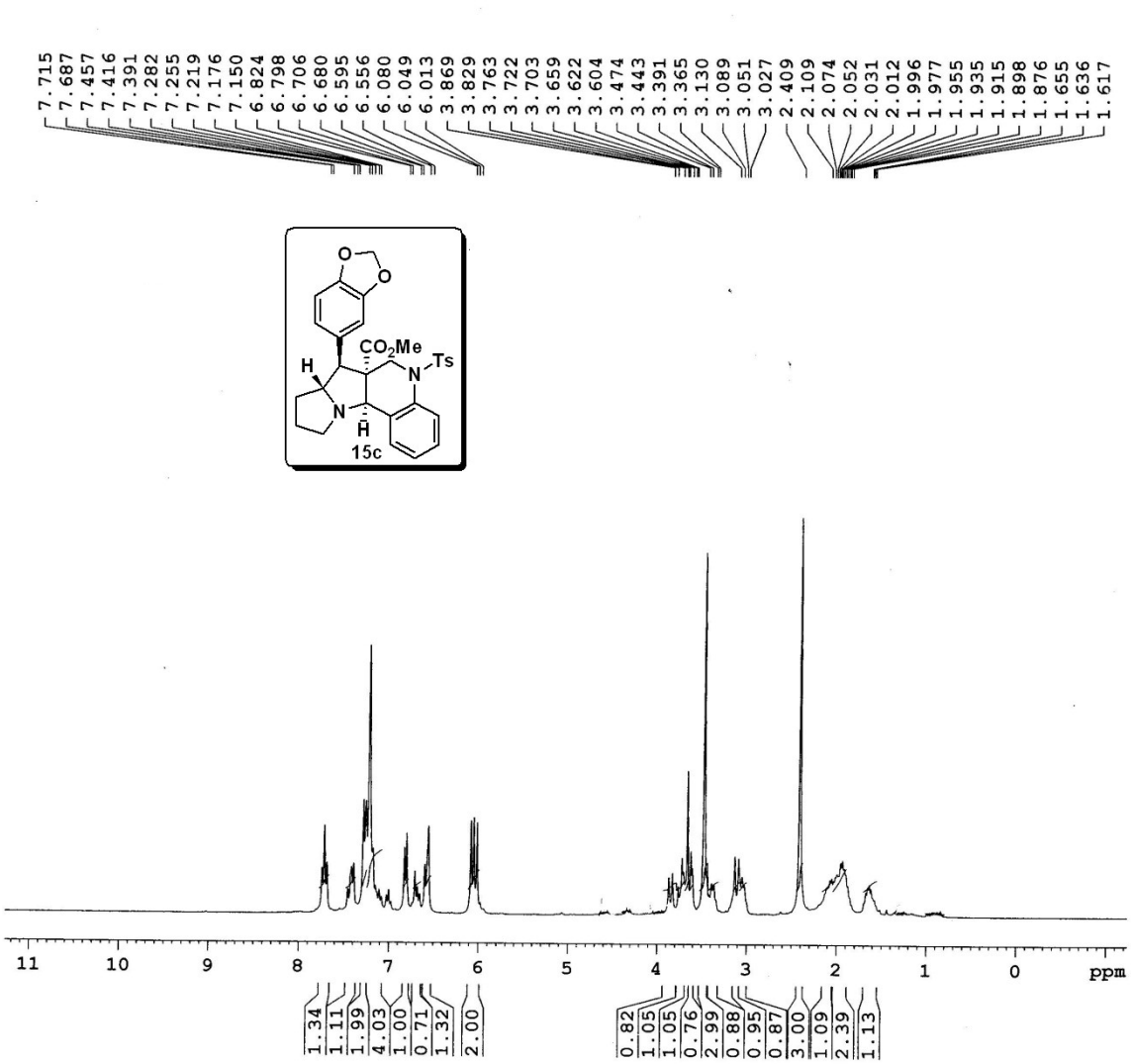
Current Data Parameters
NAME DK-4-iPRO PROL
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130221
Time 17.33
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 128
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.15 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300070 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


BRUKER
Current Data Parameters
NAME DK-4-iPRO PROL
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130221
Time_ 17.43
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 385
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 1024
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 0.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.68 dB
PL13 16.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
172.62
148.25
143.47
137.71
135.82
132.29
129.73
129.57
128.39
128.02
127.09
126.78
123.11
119.50
77.45
77.03
76.60
68.96
66.89
58.72
58.58
54.88
52.25
45.70
33.68
31.66
27.10
23.88
21.50
200
180
160
140
120
100
80
60
40
20
0
ppm
iPr
CO2Me
Ts
N
H
H
15b



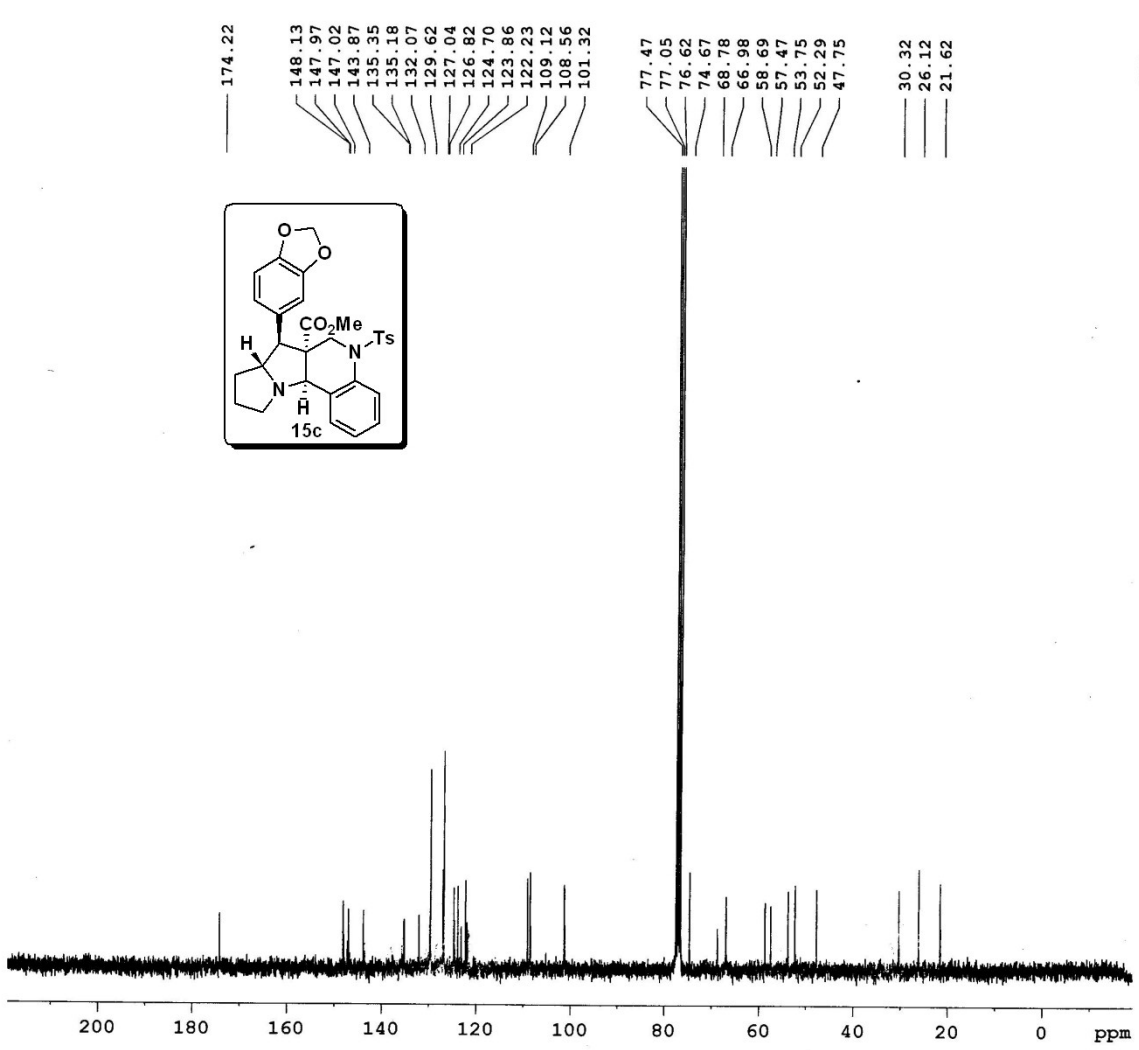
BRUKER

Current Data Parameters
NAME DK-V-PIP-TS-PYZ
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20111024
Time 22.47
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 101.6
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.15 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



BRUKER

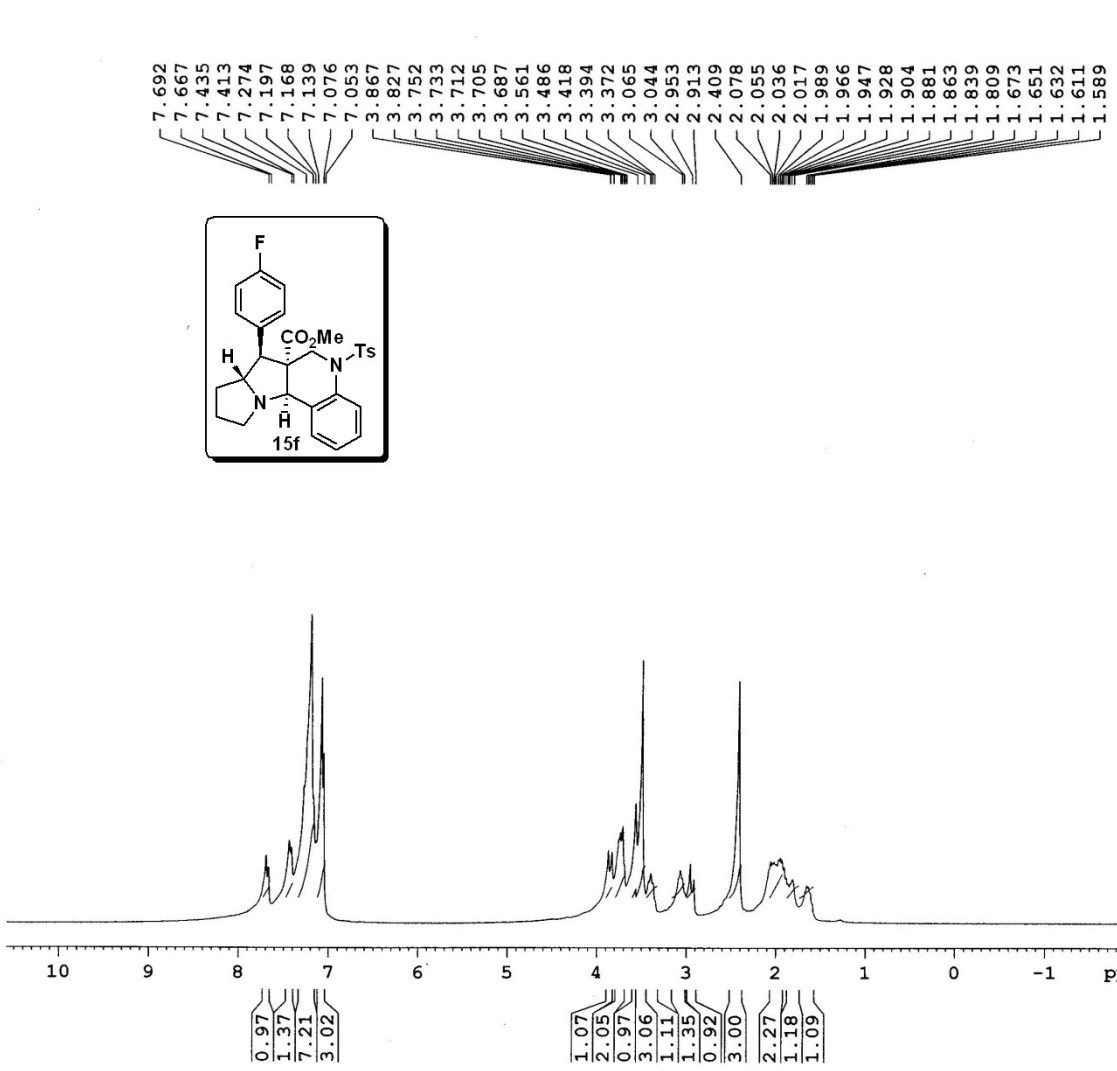
Current Data Parameters
NAME DK-V-PIP-TS-PYZ
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20111024
Time 22.51
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 379
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 2896.3
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 0.00 dB
SFO1 75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.68 dB
PL13 16.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



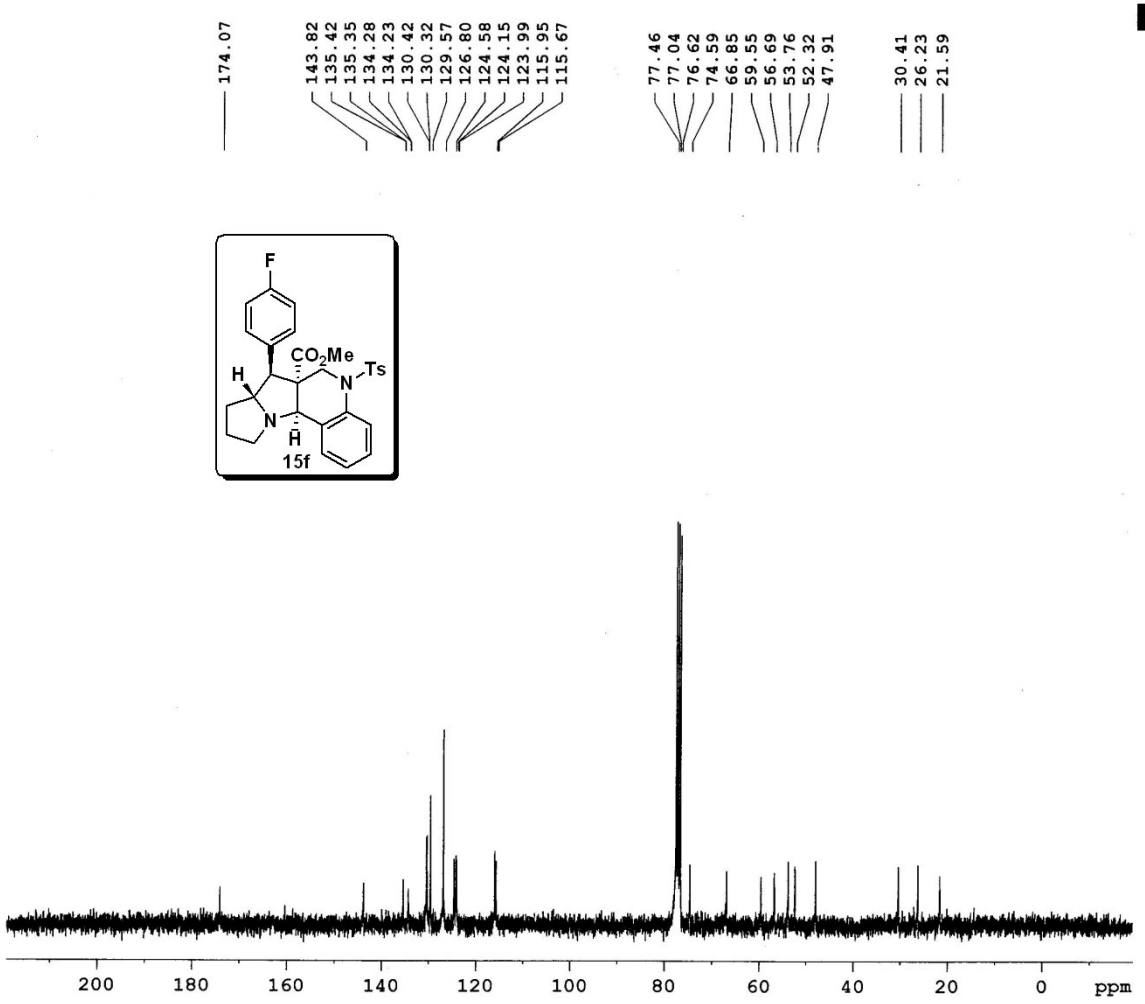
BRUKER

Current Data Parameters
NAME DK-V-4-F-EST-DC-PYZ
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20111017
Time 18.36
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 114
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.15 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



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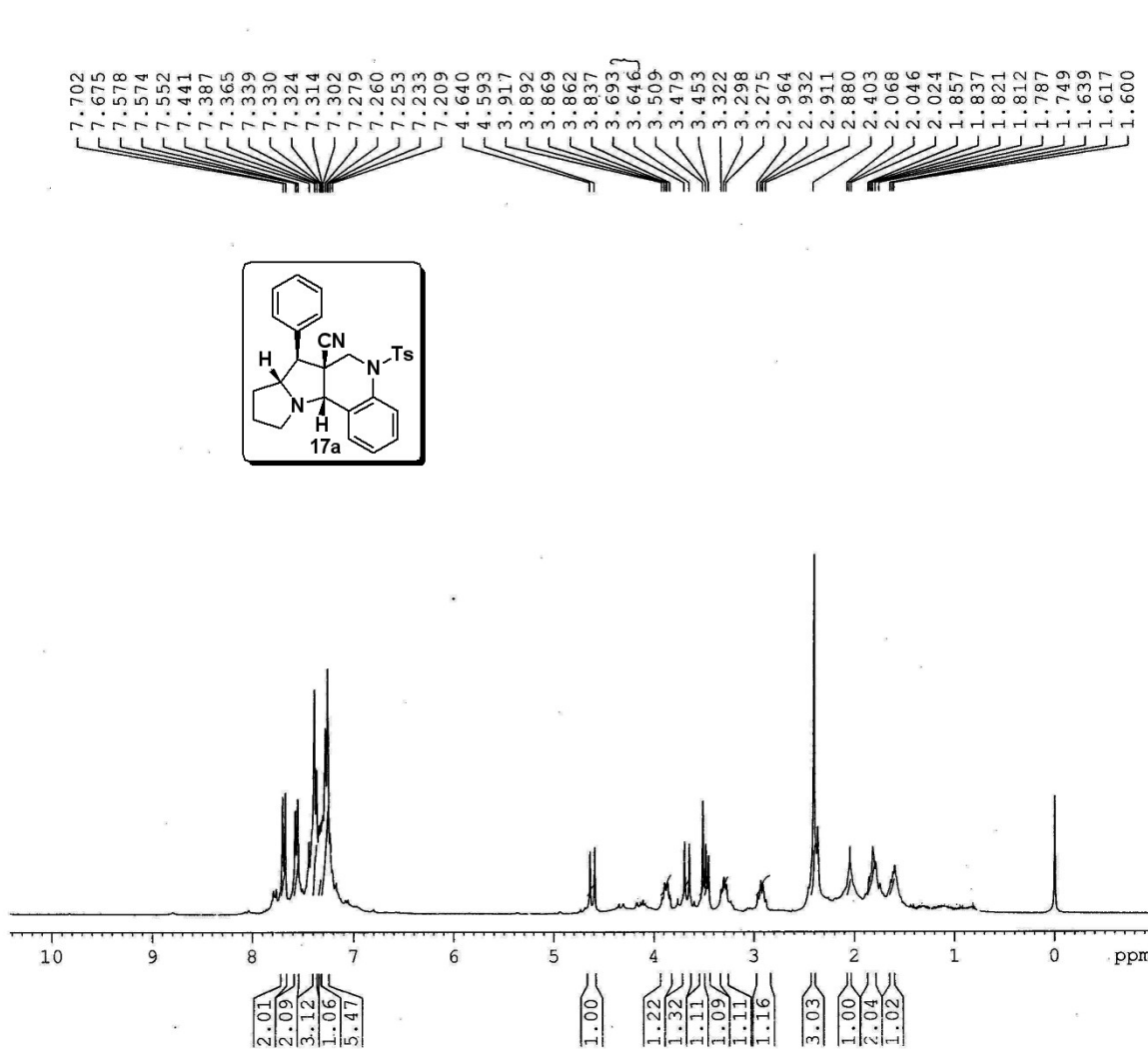
Current Data Parameters
NAME DK-V-4-F-EST-DC-PYZ
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20111017
Time 18.46
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 400
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 724.1
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 0.00 dB
SFO1 75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.68 dB
PL13 16.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



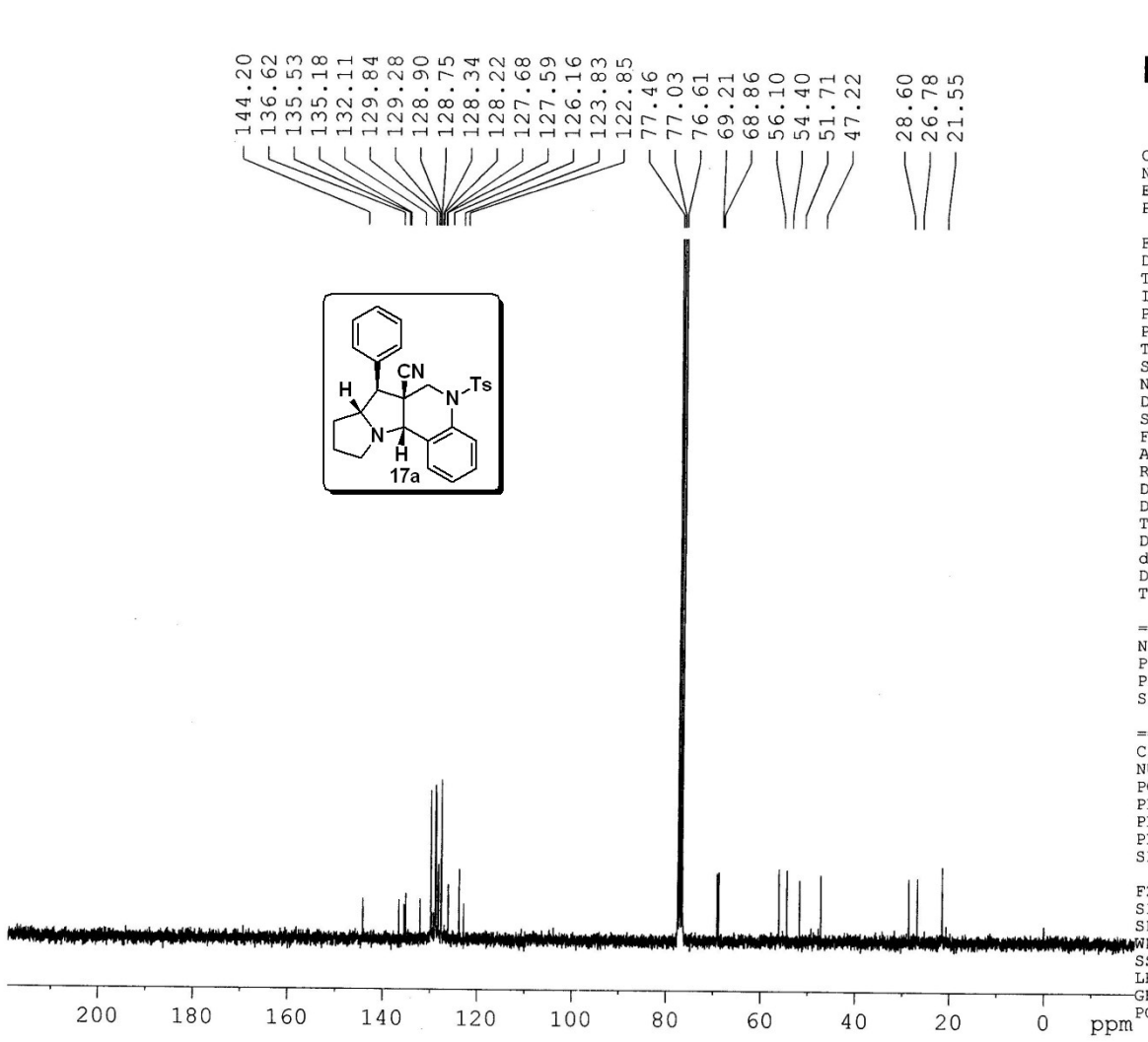
BRUKER

Current Data Parameters
NAME DK-HCN-PYZ
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121218
Time 23.03
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 128
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.15 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300064 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



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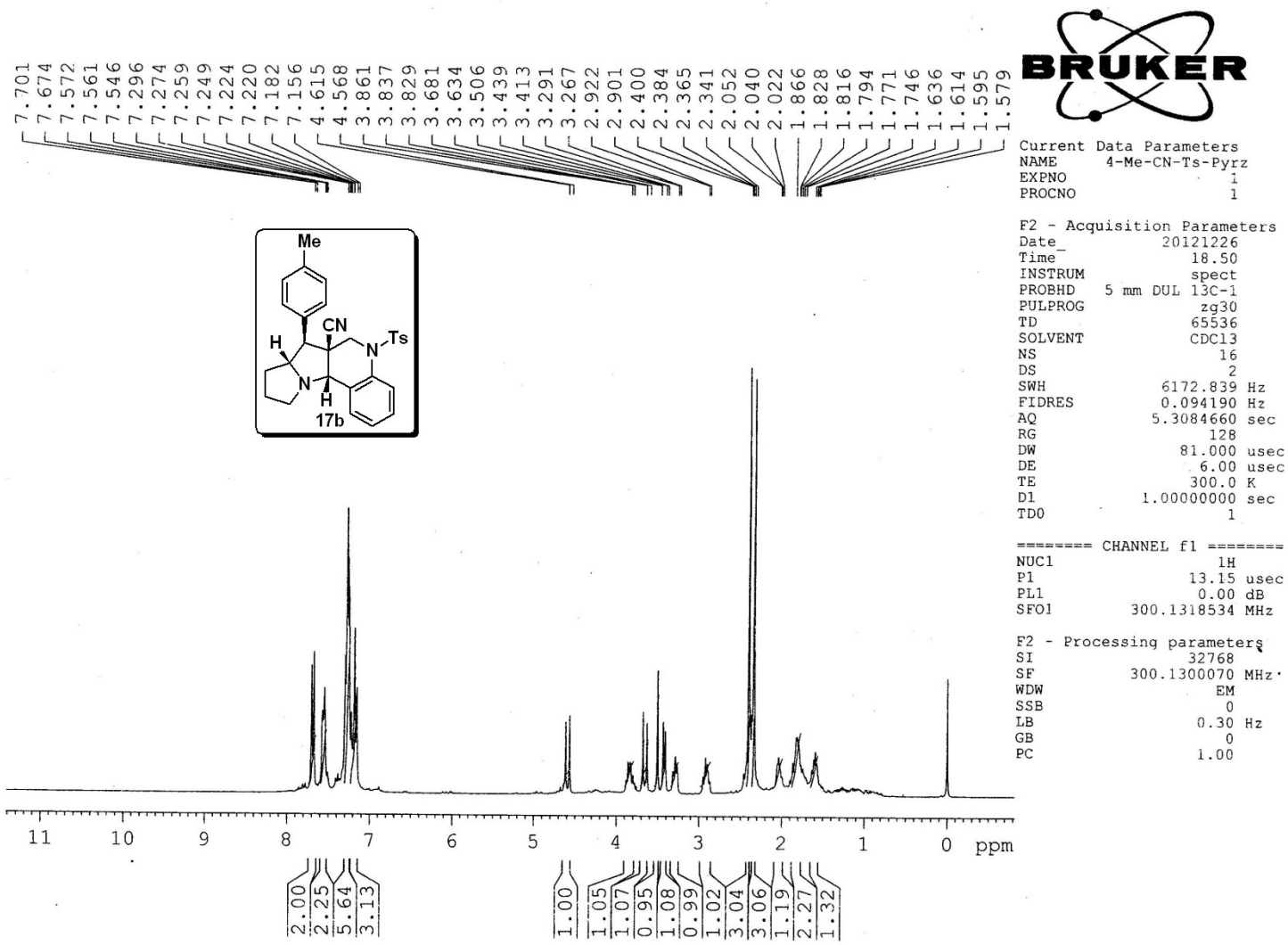
Current Data Parameters
NAME DK-HCN-PYZ
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121218
Time 23.21
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 504
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 574.7
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 0.00 dB
SFO1 75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.68 dB
PL13 16.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



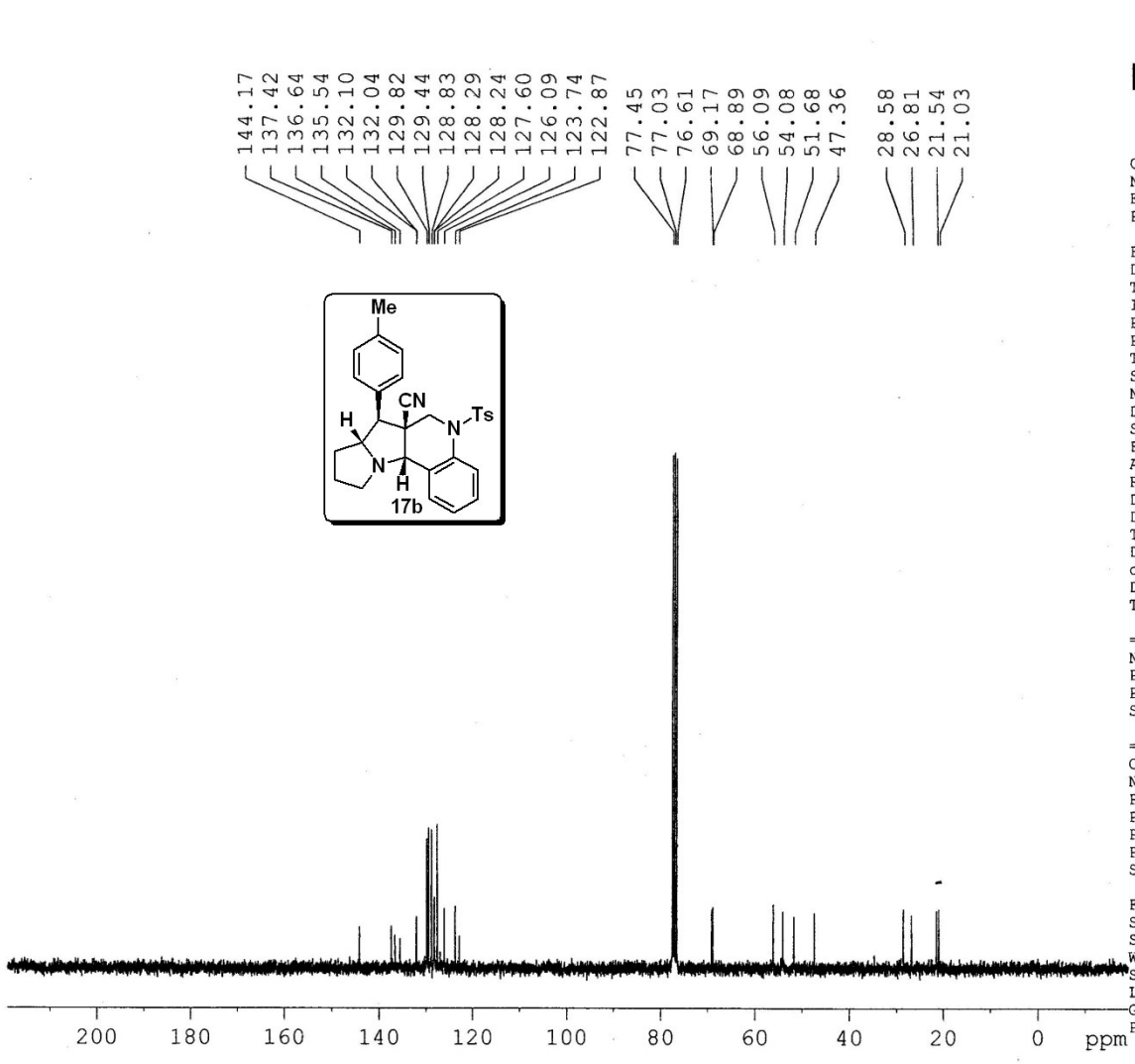
BRUKER

Current Data Parameters
NAME 4-Me-CN-Ts-Pyrz
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121226
Time 18.50
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 128
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.15 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300070 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



BRUKER

Current Data Parameters
NAME 4-Me-CN-Ts-Pyrz
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_	20121226
Time	19.00
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	320
DS	4
SWH	17985.611 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	456.1
DW	27.800 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

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

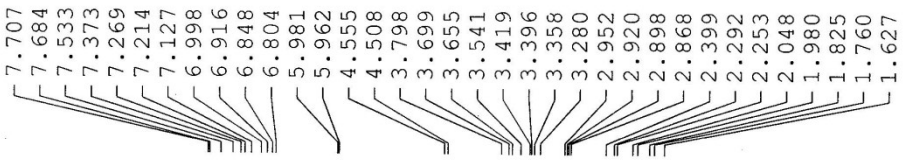
NUC1	13C
P1	9.30 usec
PL1	0.00 dB
SFO1	75.4752953 MHz

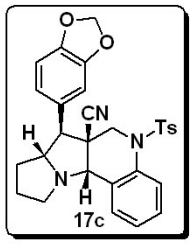
======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	15.68 dB
PL13	16.00 dB
SFO2	300.1312005 MHz

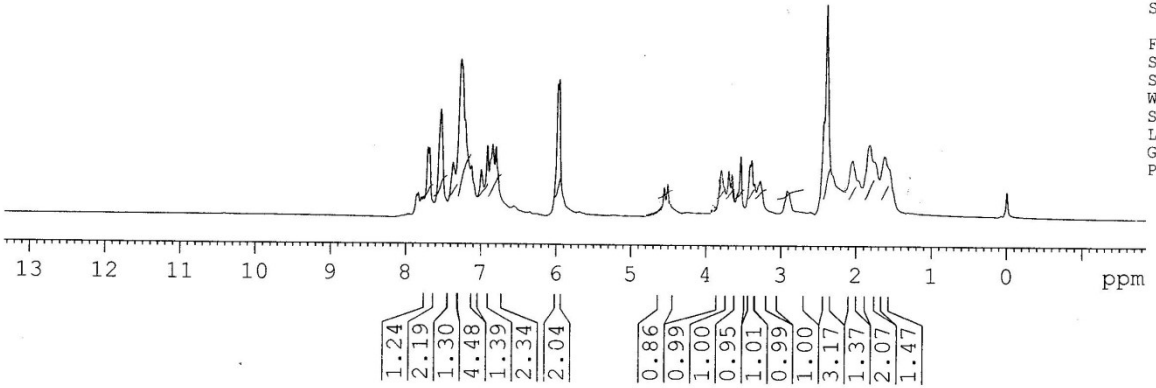
F2 - Processing parameters

SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40





17c



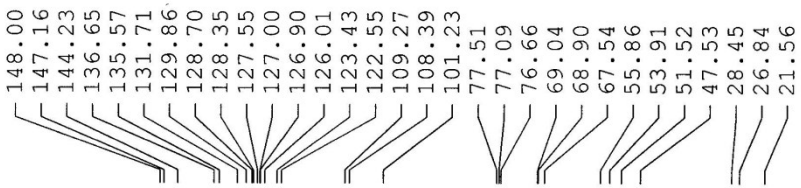
BRUKER

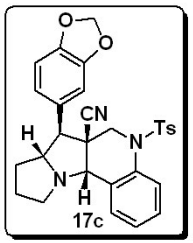
Current Data Parameters
NAME DK-PIP-CN-PYZ
EXPNO 1
PROCNO 1

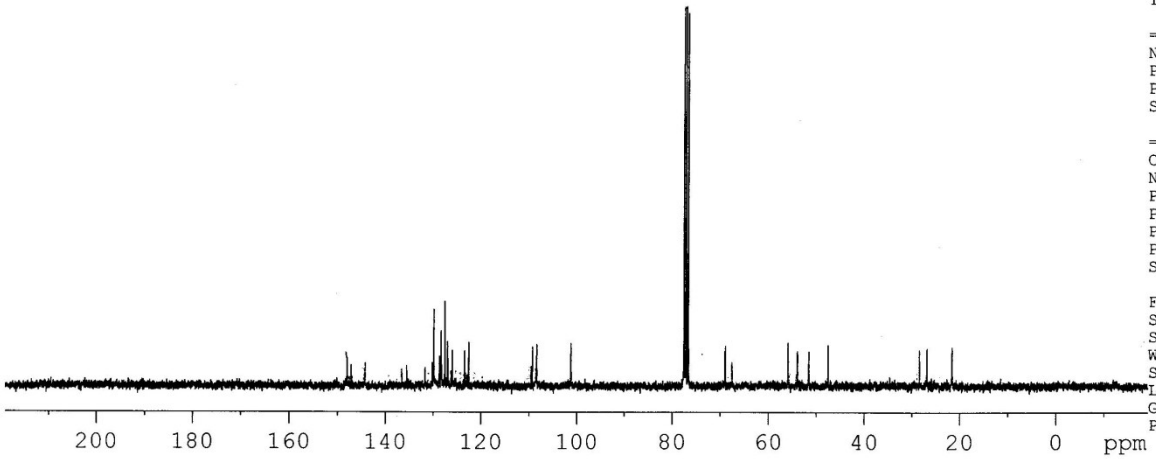
F2 - Acquisition Parameters
Date_ 20121211
Time 20.12
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 50.8
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.15 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300055 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00







BRUKER

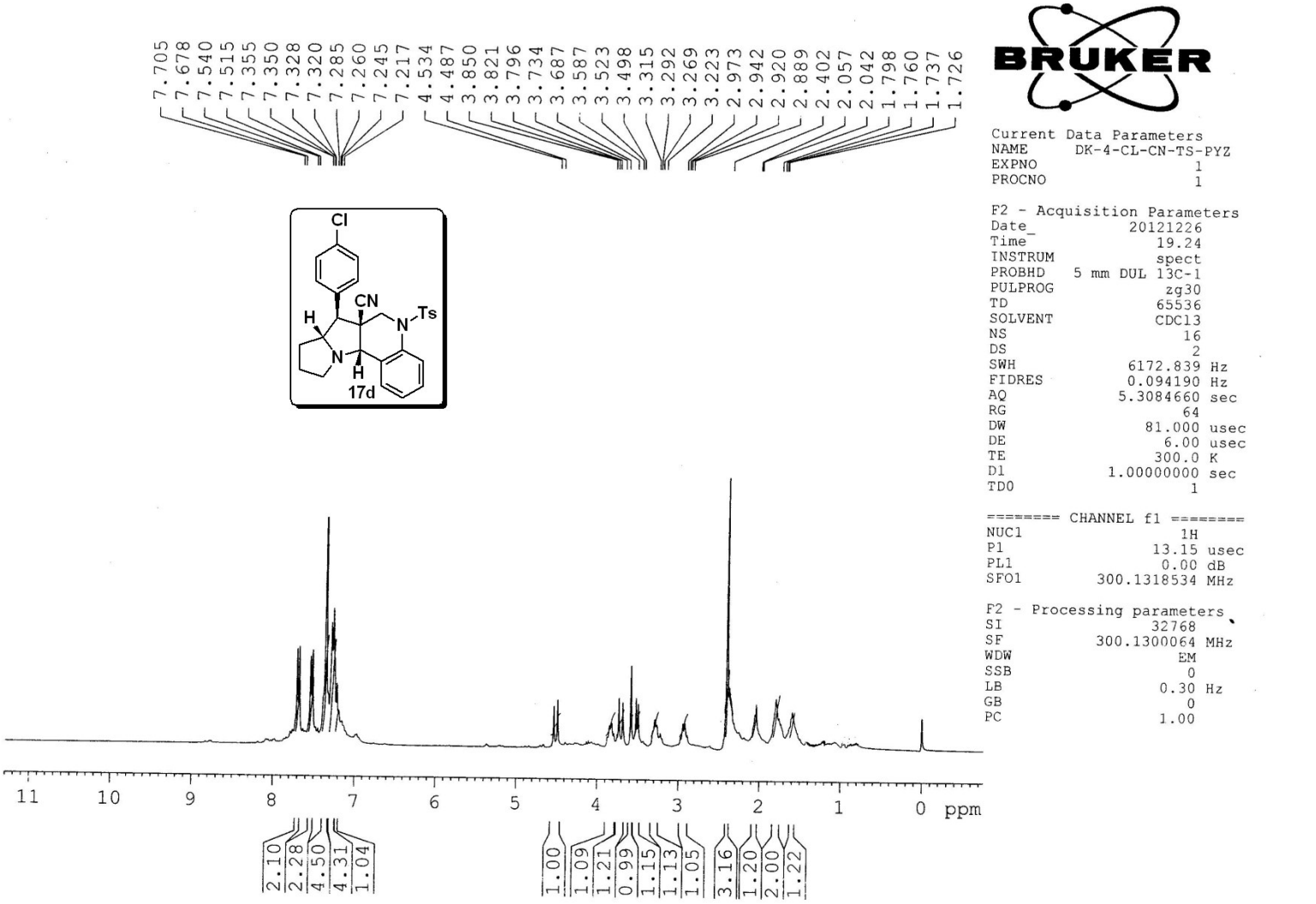
```
Current Data Parameters
NAME       DK-PIP-CN-PYZ
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20121211
Time               20.17
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDC13
NS                  198
DS                    4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ            1.8219508 sec
RG                574.7
DW               27.800 usec
DE                6.00 usec
TE               300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.30 usec
PL1                0.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2                0.00 dB
PL12              15.68 dB
PL13              16.00 dB
SFO2        300.1312005 MHz

F2 - Processing parameters
SI                32768
SF          75.4677490 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



BRUKER

Current Data Parameters

NAME	DK-4-CL-CN-TS-PYZ
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

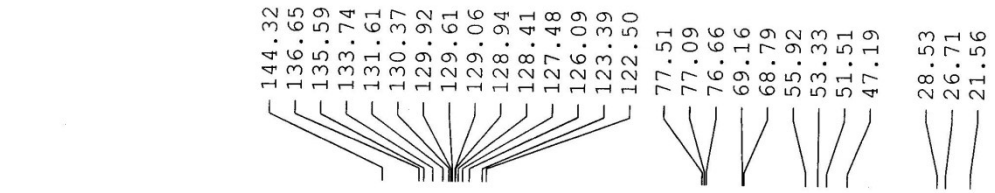
Date_	20121226
Time	19.24
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	6172.839 Hz
FIDRES	0.094190 Hz
AQ	5.3084660 sec
RG	64
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
TD0	1

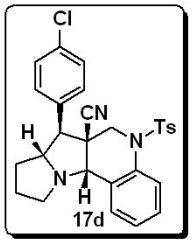
======== CHANNEL f1 ========

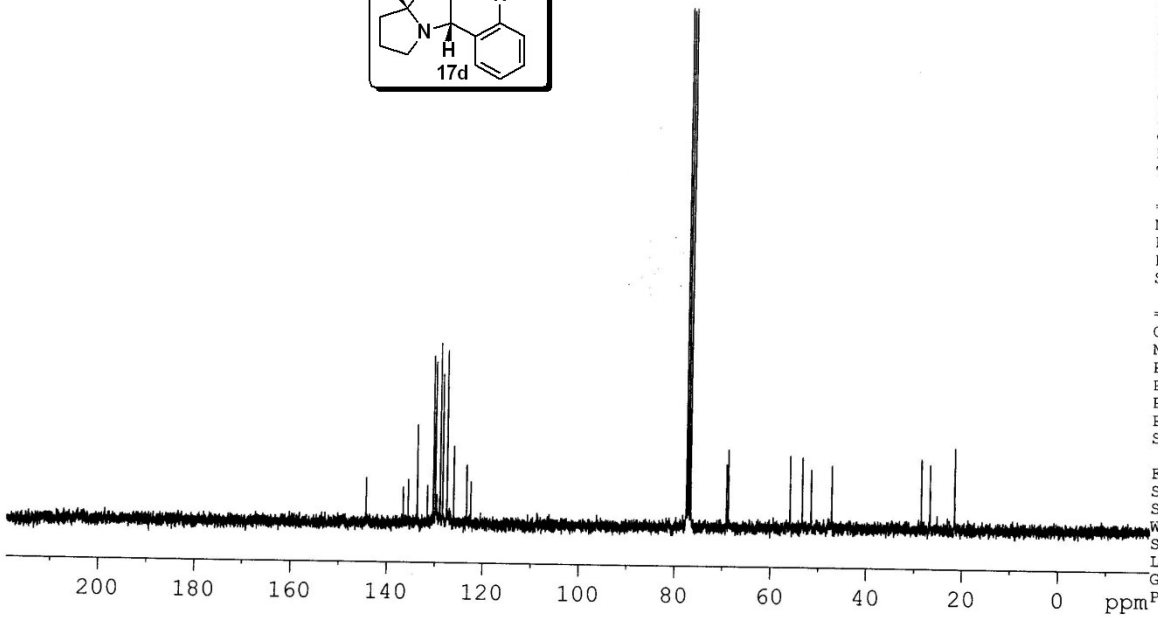
NUC1	1H
P1	13.15 usec
PL1	0.00 dB
SFO1	300.1318534 MHz

F2 - Processing parameters

SI	32768
SF	300.1300064 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00







BRUKER

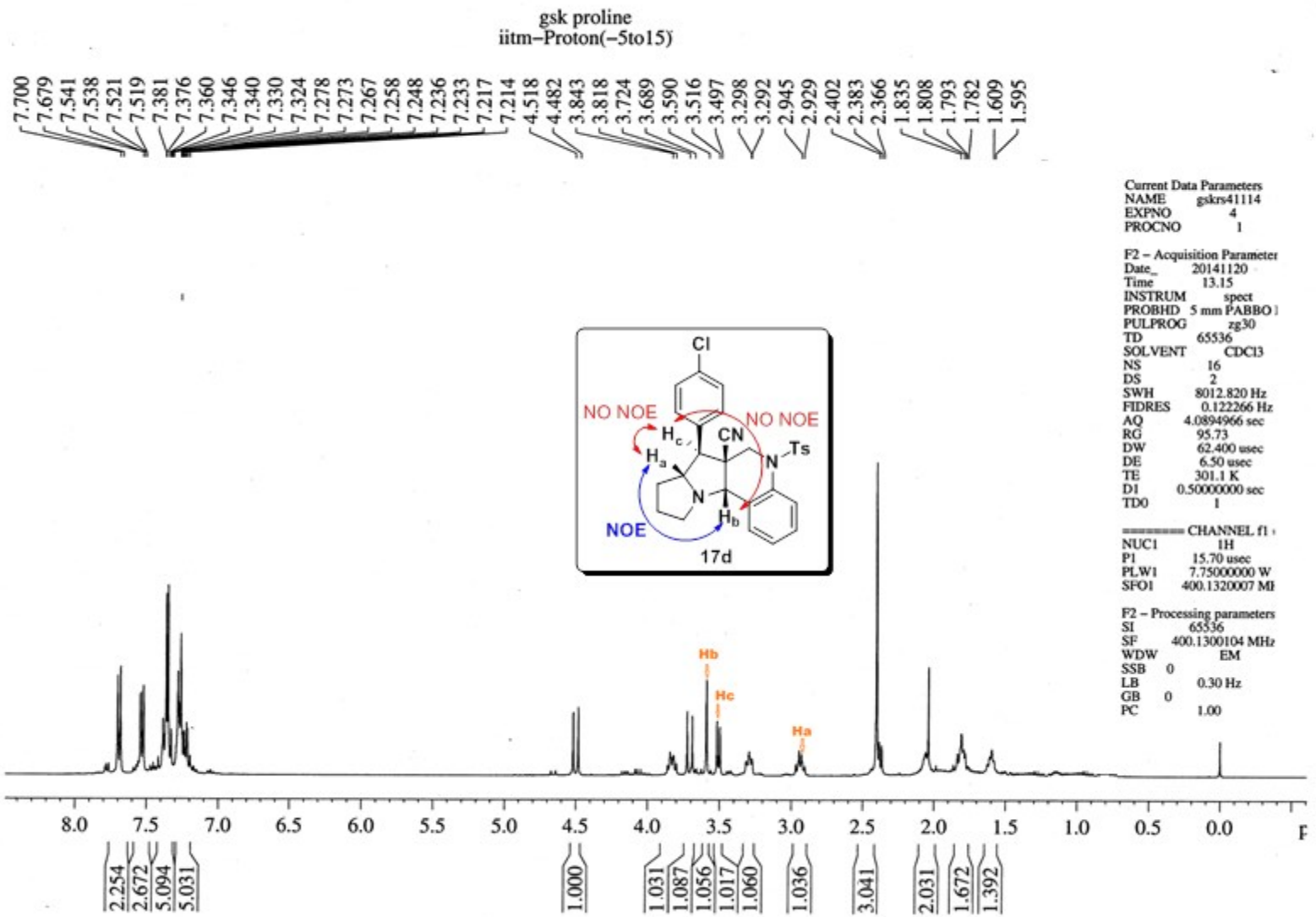
Current Data Parameters
NAME DK-4-CL-CN-TS-PYZ
EXPNO 2
PROCNO 1

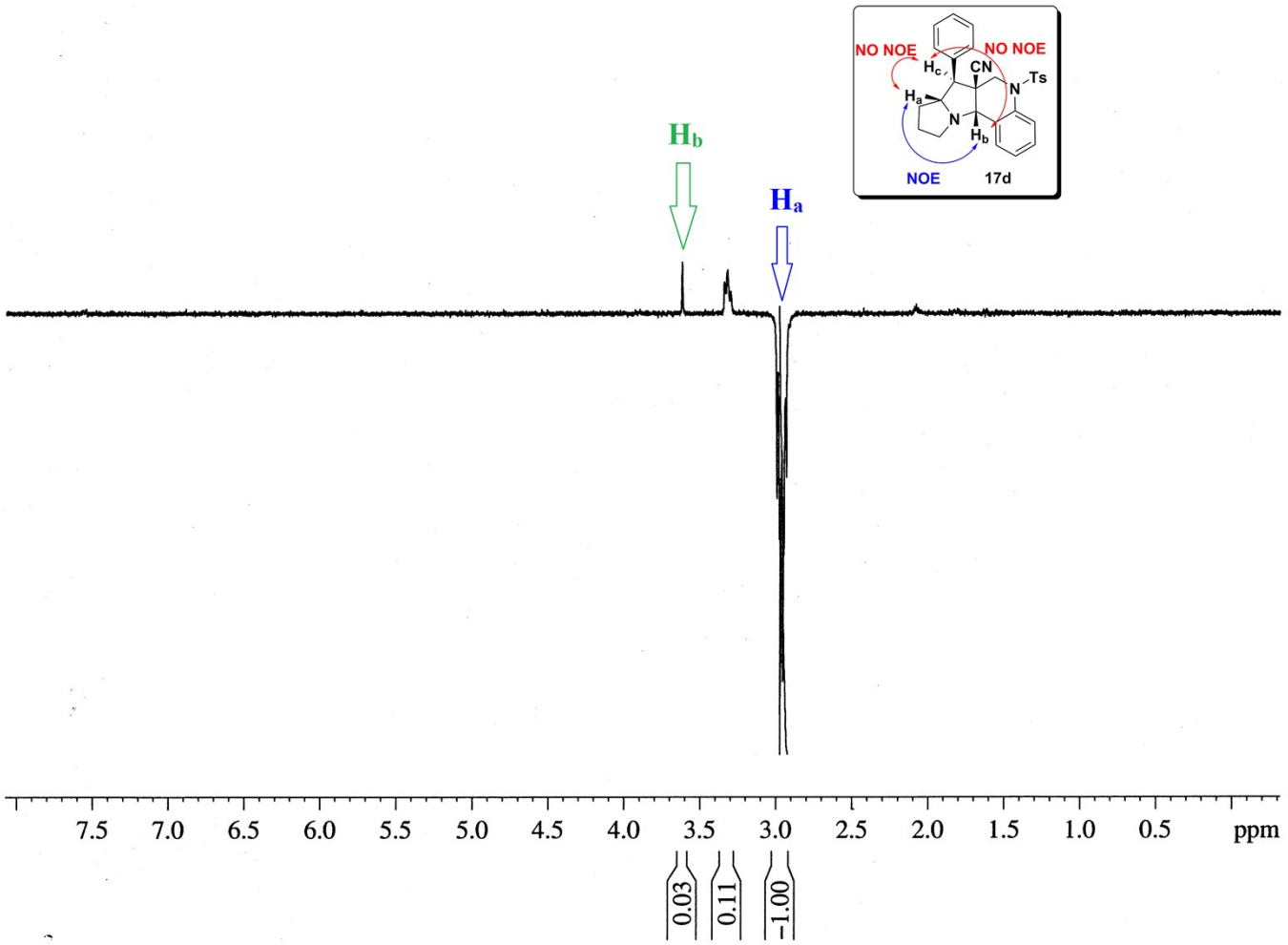
F2 - Acquisition Parameters
Date_ 20121226
Time 19.29
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 143
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 512
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 0.00 dB
SFO1 75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.68 dB
PL13 16.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


gsk proline
iitm-Proton(-5to15)
7.700
7.679
7.541
7.538
7.521
7.519
7.381
7.376
7.360
7.346
7.340
7.330
7.324
7.278
7.273
7.267
7.258
7.248
7.236
7.233
7.217
7.214
4.518
4.482
3.843
3.818
3.724
3.689
3.590
3.516
3.497
3.298
3.292
2.945
2.929
2.402
2.383
2.366
1.835
1.808
1.793
1.782
1.609
1.595
Current Data Parameters
NAME gskrs41114
EXPNO 4
PROCNO 1
F2 - Acquisition Parameter
Date_ 20141120
Time 13.15
INSTRUM spect
PROBHD 5 mm PABBO
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 95.73
DW 62.400 usec
DE 6.50 usec
TE 301.1 K
D1 0.50000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 15.70 usec
PLW1 7.75000000 W
SFO1 400.1320007 MH
F2 - Processing parameters
SI 65536
SF 400.1300104 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Cl
NO NOE
NO NOE
CN
Ts
N
NOE
17d
Hb
Hc
Ha
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
0.0
2.254
2.672
5.094
5.031
1.000
1.031
1.087
1.056
1.017
1.060
1.036
3.041
2.031
1.672
1.392




```
Current Data Parameters
NAME          gskrs41114
EXPNO                  7
PROCNO                 1

F2 - Acquisition Parameters
Date_           20141120
Time               13.32
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          selnogp
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     2
SWH             8012.820 Hz
FIDRES             0.122266 Hz
AQ              4.0894966 sec
RG                  31.9
DW                 62.400 usec
DE                  6.50 usec
TE                 301.1 K
CNST21          2.9359000
D1              2.00000000 sec
D8              0.30000001 sec
D16             0.00020000 sec
D20             0.14880000 sec
TD0                    1
ZGOPTNS   -DCALC_SPOFFS

======== CHANNEL f1 ========
NUC1                   1H
P1                 15.70 usec
P2                 31.40 usec
P12             80000.00 usec
PLW0      0 W
PLW1          7.75000000 W
SFO1        400.1317775 MHz
SPNAM2   Gaus1_180r.1000
SPOAL2               0.500
spoffs2           -602.73 Hz
SPW2           0.00000705 W

====== GRADIENT CHANNEL =====
GPNAM1         SMSQ10.100
GPNAM2         SMSQ10.100
GPZ1                15.00 %
GPZ2                40.00 %
P16               1000.00 usec

F2 - Processing parameters
SI                 32768
SF           400.1300000 MHz
WDW                  EM
SSB       0
LB                  0.10 Hz
GB        0
PC                  1.00
```