Electronic Supplementary Information

REVEALING THE DIPOLAROPHILIC CHARACTER OF PHTHALIC ANHYDRIDES: 1,3-DIPOLAR CYCLOADDITIONS WITH AN AZOMETHINE YLIDE

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1. General experimental

Unless otherwise noted, all reactions were performed under an argon atmosphere in oven-dried glassware with magnetic stirring. Dry dichloromethane, methanol and tetrahydrofuran were obtained from a J. C. Meyer Solvent Dispensing System (two packed columns of neutral alumina). Petroleum ether (or petroleum spirit) refers to the fraction of liquid hydrocarbons with boiling point 40-60 °C. Trifluoroacetic acid was freshly distilled under nitrogen prior to use. All starting materials, reagents and solvents were of analytical grade, obtained from commercial sources and used as supplied unless otherwise noted. Commercial 4-methylphthalic anhydride **5e** had partially hydrolysed to the corresponding diacid, and was dehydrated by refluxing in acetic anhydride (4.0 equivalents) for approximately 4 hours, filtered and dried *in vacuo*. 3,6-Dimethylphthalic anhydride **5b** was prepared *via* a Diels-Alder reaction between 2,5-dimethylfuran and maleic anhydride;[1] 3,6-dimethoxyphthalic anhydride **5c** was prepared from 2,3-dicyanohydroquinone;[2] 3-methoxyphthalic anhydride **5f** was prepared from 2,3-dimethylphenol.[3] 4Å molecular sieves (1.6 mm pellets, from Sigma-Aldrich) were activated by several 20-30 s microwave cycles interspersed with shaking to remove any absorbed moisture, followed by placement of the microwaved sieves under high vacuum to ensure complete drying before use. Reaction products were purified by dry column vacuum chromatography (DCVC)[4] or flash column chromatography[5] using silica gel (15-40 μm and 40−63 μm, respectively). Analytical thin-layer chromatography (TLC) was performed on Merck precoated silica gel 60 F_{254} (5-40 µm thickness) plates, and were visualized using a 254 nm ultraviolet lamp and/or by dipping in ethanolic phosphomolybdic acid followed by heating. ^{1}H, ^{13}C and ^{19}F NMR spectra were recorded on a Bruker AV400 instrument in $CDCl_3$ solutions at 25 °C, unless noted otherwise. Hydrogen, proton-decoupled carbon-13 and fluorine-19 spectra were recorded at 400, 100 and 376 MHz, respectively. Chemical shifts (δ) are reported in parts per million (ppm). For ^{1}H NMR spectra, the peak due to residual solvent (δ 7.24 for $CDCl_3$) was used as the internal reference. The reference for proton-decoupled ^{13}C NMR spectra, when recorded in $CDCl_3$, was the central peak (δ 77.23) of the $CDCl_3$ triplet. Data are reported as

follows: chemical shift (δ), multiplicity, coupling constant *(J)* in Hz and integrated intensity. Abbreviations to denote the multiplicity of a particular signal are: s (singlet), bs (broad singlet), d (doublet), bd (broad doublet), t (triplet), q (quartet), dd (double doublet), dt (double triplet) and m (multiplet). Electrospray Ionisation (ESI) mass spectra were acquired with a VG Platform mass spectrometer using a cone voltage of 50 V and the source was maintained at 80 °C. The solvent system used was acetonitrile with a flow rate of 0.04 mL min^{-1}. Positive and negative ion atmospheric pressure on chemical ionization (APCI) mass spectra were acquired with a VG Platform mass spectrometer using a cone voltage of 50 V, and the source was maintained at 100 °C. Nitrogen was used as the nebuliser and sheath gas, and the probe temperature was 400 °C. The solvent system used was acetonitrile with a flow rate of 0.3 mL min^{-1}. High-resolution mass spectrometric analyses (HRMS) were performed on a Thermo Scientific Q Exactive mass spectrometer fitted with a HESI-II ion source, an APCI ion source or an ASAP ion source[6] according to the sample.

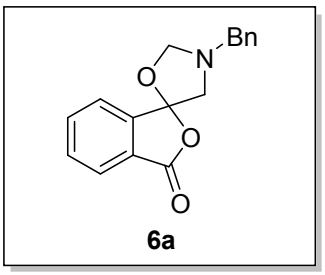
2. Spiro(isobenzofuran-1,5'-oxazolidin)-3-ones (6)

Initially, phthalic anhydride **5a** was reacted with azomethine ylide precursor **2** (1.80 mole equivalent) and lithium fluoride (1.25 mole equivalent) at 35 °C in acetonitrile, with sonication in the presence of 4Å molecular sieves over 3 h, to give 3'-benzyl-3H-spiro(isobenzofuran-1,5'-oxazolidin)-3-one **6a**. Repeating the cycloaddition reaction using TFA (0.05 mole equivalent) in CH_2Cl_2 (in place of LiF/sonication) also gave cycloadduct **6a**. The latter conditions involved milder reaction conditions, easier operations and work-up. These advantages, led us to complete the rest of the studies using the latter conditions

The spiro(isobenzofuran-1,5'-oxazolidin)-3-ones **6** were then prepared from phthalic anhydrides **5** according to the reaction conditions described in Table 1 of the manuscript.

3'-benzyl-3H-spiro(isobenzofuran-1,5'-oxazolidin)-3-one **6a**

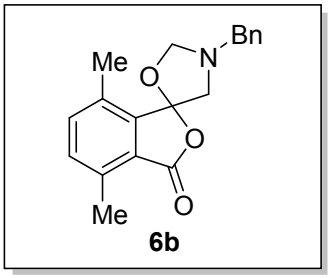
General procedure 1. To a solution of phthalic anhydride (**5a**) (150 mg, 1.01 mmol) in CH_2Cl_2 (7.5 mL) over 4Å molecular sieves at 0 °C under an atmosphere of nitrogen gas, was added *N*-(methoxymethyl)-*N*-trimethylsilylmethyl)benzylamine (**2**) (0.29 mL, 1.11 mmol). To the resulting mixture was added a solution of trifluoroacetic acid (1 M in CH_2Cl_2, 50 μL, 0.05 mmol) dropwise with stirring. The resulting mixture was allowed to warm to room temperature, and allowed to stir until TLC analysis indicated consumption of starting material (18 h). The reaction mixture was concentrated *in vacuo*, and the residue was filtered through a plug of Florisil™ using EtOAc as eluent to furnish the *title compound* **6a** (255 mg, 90% crude yield) as a viscous pale yellow oil.



6a

R_f 0.41 (1:1 EtOAc/heptane). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (d, *J* = 7.4 Hz, 1H), 7.71 (t, *J* = 7.3 Hz, 1H), 7.58 (t, *J* = 7.3 Hz, 1H), 7.50 (d, *J* = 7.4 Hz, 1H), 7.41 (d, *J* = 7.0 Hz, 2H), 7.34 (t, *J* = 7.1 Hz, 2H), 7.28 (m, 1H), 4.92 (d, *J* = 5.6 Hz, 1H), 4.85 (d, *J* = 5.6 Hz, 1H), 4.13 (m, 2H), 3.60 (d, *J* = 13.2 Hz, 1H), 3.45 (d, *J* = 13.2 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.1 (C=O), 146.5 (C_q), 138.5 (C_q), 134.8 (CH), 130.9 (CH), 129.1 (CH), 128.8 (CH), 127.8 (CH), 127.6 (C_q), 125.7 (CH), 122.7 (CH), 110.6 (C_q), 90.4 (CH_2), 63.0 (CH_2), 59.3 (CH_2). HRMS (ASAP) *m/z* [M + H]$^+$ calcd for $C_{17}H_{16}NO_3$ 282.1125, found 282.1125.

3'-benzyl-4,7-dimethyl-3H-spiro(isobenzofuran-1,5'-oxazolidin)-3-one **6b**



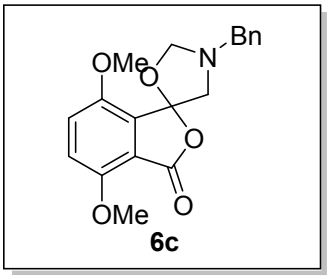
6b

The *title compound* **6b** was prepared from 3,6-dimethylphthalic anhydride **5b** (128 mg, 0.73 mmol), *N*-(methoxymethyl)-*N*-trimethylsilylmethyl)benzylamine (**2a**) (0.37 mL, 1.45 mmol) and TFA (73 μL, 0.07 mmol) using **General Procedure 1** to afford the *title compound* **6b** (299 mg, ~100% crude yield) as a pale yellow oil.

R_f 0.35 (1:4 EtOAc/heptane). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (m, 2H), 7.38-7.27 (m, 4H), 7.23 (d, 7.6 Hz, 1H), 4.96 (d, *J* = 5.4 Hz, 1H), 4.91 (d, *J* = 5.4 Hz, 1H), 4.21 (d, *J* = 13.2 Hz, 1H), 4.16 (d, *J* = 13.2 Hz, 1H), 3.60 (d, *J* = 12.8 Hz, 1H),

3.40 (d, *J* = 12.8 Hz, 1H), 2.65 (s, 3H), 2.40 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.3, 143.6, 138.8, 137.2, 136.4, 132.8, 131.3, 129.0, 128.8, 127.7, 125.2, 110.3, 90.1, 61.0, 59.3, 17.7, 17.4. HRMS (ASAP) *m/z* [M + H]$^+$ calcd for $C_{19}H_{20}NO_3$ 310.1438, found 310.1436.

3'-benzyl-4,7-dimethoxy-3H-spiro(isobenzofuran-1,5'-oxazolidin)-3-one **6c**

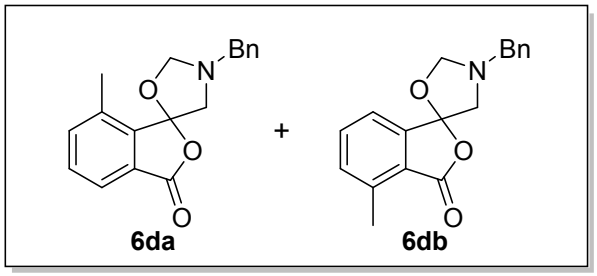


6c

The *title compound* **6c** was prepared from 3,6-dimethoxyphthalic anhydride **5c** (694 mg, 3.33 mmol) using **General Procedure 1** to afford the *title compound* **6c** (1.03 g, 90% crude yield) as an amorphous yellow oil.

R_f 0.24 (1:1 EtOAc/heptane). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42-7.26 (m, 5H), 7.08 (d, 8.8 Hz, 1H), 6.93 (d, 8.8 Hz, 1H), 4.90 (d, *J* = 5.7 Hz, 1H), 4.82 (d, *J* = 5.7 Hz, 1H), 4.17 (d, *J* = 13.1 Hz, 1H), 4.10 (d, *J* = 13.1 Hz, 1H), 3.92 (s, 3H), 3.84 (s, 3H), 3.76 (d, *J* = 13.0 Hz, 1H), 3.40 (d, *J* = 13.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.0, 152.2, 148.3, 139.1, 135.0, 129.2, 129.0, 128.7, 127.6, 118.5, 114.2, 108.9, 90.0, 60.6, 59.3, 56.6, 56.3. HRMS (ASAP) *m/z* [M + H]$^+$ calcd for $C_{19}H_{20}NO_5$ 342.1336, found 342.1338.

3'-benzyl-7-methyl-3H-spiro(isobenzofuran-1,5'-oxazolidin)-3-one **6da** and *3'-benzyl-4-methyl-3H-spiro(isobenzofuran-1,5'-oxazolidin)-3-one* **6db** *(mixture of regioisomers)*

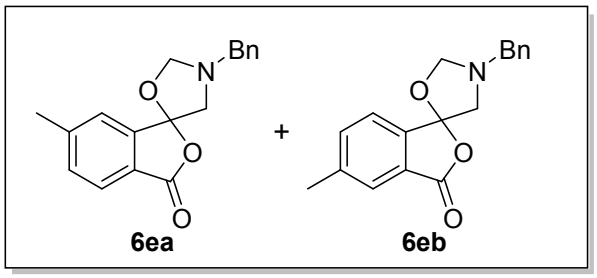


6da **6db**

The *title compounds* **6da** and **6db** were prepared from 3-methylphthalic anhydride **5d** (658 mg, 4.06 mmol) using **General Procedure 1** to afford the *title compounds* **6da** and **6db** in a ratio of 48:52, respectively (as determined by ^{1}H NMR) (1.23 g, >100% crude yield) as a viscous brown oil.

R_f 0.65 and 0.49 (1:1 EtOAc/heptane). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (d, *J* = 5.5 Hz, 1H), 7.55 (t, *J* = 7.5 Hz, 1H), 7.48-7.40 (m, 6H), 7.35-7.27 (m, 8H), 4.95 (d, *J* = 5.4 Hz, 1H), 4.89 (d, *J* = 5.9 Hz, 2H), 4.82 (d, *J* = 5.4 Hz, 1H), 4.17 (d, *J* = 13.2 Hz, 1H), 4.15 (d, *J* = 13.2 Hz, 1H), 4.12 (m, 2H), 3.60 (d, *J* = 12.6 Hz, 1H), 3.57 (d, *J* = 12.6 Hz, 1H), 3.41 (d, *J* = 13.1 Hz, 2H), 2.67 (s, 3H, **6db**), 2.43 (s, 3H, **6da**). ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.3, 168.2, 147.0, 143.3, 139.8, 138.7, 138.6, 136.6, 134.5, 134.3, 132.6, 131.1, 129.1, 129.0, 128.8, 128.0, 127.8, 127.7, 124.9, 123.2, 120.0, 111.4, 109.5, 90.3, 90.1, 63.1, 61.0, 59.3, 59.2, 18.0, 17.7. HRMS (ASAP) *m/z* [M + H]$^+$ calcd for $C_{18}H_{18}NO_3$ 296.1281, found 296.1281.

3'-benzyl-6-methyl-3H-spiro(isobenzofuran-1,5'-oxazolidin)-3-one **6ea** *and 3'-benzyl-5-methyl-3H-spiro(isobenzofuran-1,5'-oxazolidin)-3-one* **6eb** *(mixture of regioisomers)*



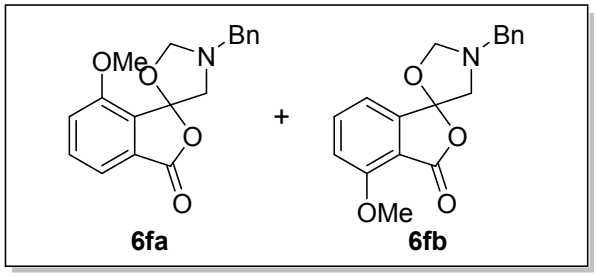
6ea **6eb**

The *title compounds* **6ea** and **6eb** were prepared from 4-methylphthalic anhydride **5e** (350 mg, 2.16 mmol), *N*-(methoxymethyl)-*N*-trimethylsilylmethyl)benzylamine (**2a**) (0.83 mL, 3.24 mmol) and TFA (162 μL, 0.16 mmol) using **General Procedure 1** to afford the *title compounds* **6ea** and **6eb** in a ratio of 63:37, respectively (as determined by ^{1}H NMR) (675 mg, ~100% crude yield) as a yellow oil.

R_f 0.42 (**6ea**) and 0.35 (**6eb**) (1:1 EtOAc/heptane). ^{1}H NMR (400 MHz, $CDCl_3$) (mixture of regioisomers) δ 7.73 (d, 7.8 Hz, 1H), 7.65 (s, 1H), 7.52 (d, *J* = 7.8 Hz, 1H), 7.42-7.23 (m, 13H), 4.90 (d, *J* = 5.9 Hz, 1H), 4.89 (d, *J* = 5.9 Hz, 1H), 4.83 (d, *J* = 5.9 Hz, 1H), 4.82 (d, *J* = 5.9 Hz, 1H), 4.12 (s, 2H), 4.11 (s, 2H), 3.59 (d, *J* = 13.2 Hz, 1H), 3.58 (d, *J* = 13.3 Hz, 1H), 3.43 (d, *J* = 13.2 Hz, 1H), 3.41 (d, *J* = 13.3 Hz, 1H), 2.49 (s, 3H, **6ea**), 2.45 (s, 3H, **6eb**). ^{13}C NMR (100 MHz, $CDCl_3$) (mixture of regioisomers) δ 168.2 ($C{=}O_{min}$), 168.1 ($C{=}O_{maj}$), 146.9,

146.2, 143.8, 141.5, 138.6, 135.9, 132.0, 129.1, 128.8, 127.8, 125.8, 125.7, 125.5, 125.0, 123.0, 110.6 ($C_{q,min}$), 110.3 ($C_{q,maj}$), 90.3 ($CH_{2,maj}$), 90.2 ($CH_{2,min}$), 63.0 ($CH_{2,maj}$), 62.9 ($CH_{2,min}$), 59.3 ($CH_{2,maj}$ + $CH_{2,min}$), 22.3 ($CH_{3,maj}$), 21.6 ($CH_{3,min}$). HRMS (APCI) *m/z* $[M + H]^+$ calcd for $C_{18}H_{18}NO_3$ 296.1281, found 296.1282.

3'-benzyl-7-methoxy-3H-spiro(isobenzofuran-1,5'-oxazolidin)-3-one **6fa** *and 3'-benzyl-4-methoxy-3H-spiro(isobenzofuran-1,5'-oxazolidin)-3-one* **6fb** *(mixture of regioisomers)*

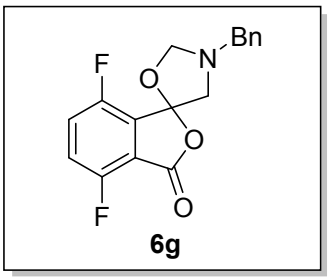


6fa **6fb**

The *title compounds* **6fa** and **6fb** were prepared from 3-methoxyphthalic anhydride **5f** (360 mg, 2.02 mmol) using **General Procedure 1** to afford the *title compounds* **6fa** and **6fb** in a ratio of 30:70, respectively (as determined by ^{1}H NMR) (376 mg, 60% crude yield) as a clear pale yellow oil.

R_f 0.39 (**6fa**) and 0.28 (**6fb**) (1:1 EtOAc/heptane). ^{1}H NMR (400 MHz, $CDCl_3$) (mixture of regioisomers) δ 7.64 (dd, *J* = 7.6 and 7.6 Hz, 1H), 7.52 (t, *J* = 7.7 Hz, 1H), 7.46 (m, 1H), 7.40 (m, 4H), 7.35-7.26 (m, 6H), 7.12 (d, *J* = 7.9 Hz, 1H), 7.03 (d, *J* = 7.5 Hz, 1H), 6.98 (d, *J* = 8.3 Hz, 1H), 4.92 (dd, *J* = 6.0 and 1.1 Hz, 1H), 4.88 (d, *J* = 6.0 Hz, 1H), 4.87 (d, *J* = 6.0 Hz, 1H), 4.85 (d, *J* = 6.0 Hz, 1H), 4.17 (m, 2H), 4.10 (s, 2H), 3.98 (s, 3H, **6fb**), 3.91 (s, 3H, **6fa**), 3.76 (d, *J* = 13.2 Hz, 1H), 3.57 (d, *J* = 13.3 Hz, 1H), 3.42 (dd, *J* = 13.2 and 1.1 Hz, 1H), 3.41 (d, *J* = 13.3 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) (mixture of regioisomers) δ 168.1 ($C{=}O_{min}$), 166.1 ($C{=}O_{maj}$), 158.4, 154.9, 149.3, 139.0, 138.6, 137.1, 133.1, 132.9, 129.5, 129.2, 129.1, 128.8, 128.7, 127.7, 127.6, 117.3 (CH_{min}), 116.2 (CH_{min}), 114.6 ($C_{q,min}$), 114.3 (CH_{maj}), 112.7 (CH_{maj}), 110.3 ($C_{q,min}$), 109.2 ($C_{q,maj}$), 90.2 ($CH_{2,maj}$), 90.0 ($CH_{2,min}$), 63.2 ($CH_{2,maj}$), 60.5 ($CH_{2,min}$), 59.4 ($CH_{2,min}$), 59.2 ($CH_{2,maj}$), 56.4 ($CH_{3,maj}$), 56.0 ($CH_{3,min}$). HRMS (ASAP) *m/z* $[M + H]^+$ calcd for $C_{18}H_{18}NO_4$ 312.1230, found 312.1230.

3'-benzyl-4,7-difluoro-3H-spiro(isobenzofuran-1,5'-oxazolidin)-3-one **6g**

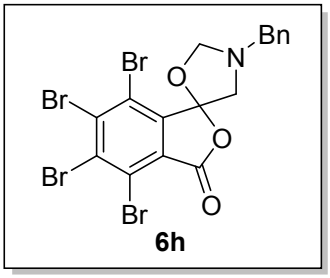


6g

The *title compound* **6g** was prepared from 3,6-difluorophthalic anhydride **5g** (185 mg, 1.0 mmol) using **General Procedure 1** to afford the *title compound* **6g** (449 mg, ~100% crude yield) as a viscous yellow oil.

R_f 0.09 (1:9 MeOH/$CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (m, 2H), 7.37-7.26 (m, 4H), 7.24-7.17 (m, 1H), 4.95 (d, *J* = 6.1 Hz, 1H), 4.85 (d, *J* = 6.1 Hz, 1H), 4.13 (m, 2H), 3.63 (d, *J* = 13.4 Hz, 1H), 3.56 (d, *J* = 13.4 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.9, 155.3 (dd, $^1J_{C\text{-}F}$ = 260.5 Hz and $^4J_{C\text{-}F}$ = 2.9 Hz), 152.9 (dd, $^1J_{C\text{-}F}$ = 249.6 Hz and $^4J_{C\text{-}F}$ = 3.1 Hz), 138.4, 133.7 (d, $^2J_{C\text{-}F}$ = 17.4 Hz), 129.2, 128.8, 127.8, 124.1 (dd, $^2J_{C\text{-}F}$ = 22.6 Hz and $^3J_{C\text{-}F}$ = 7.7 Hz), 120.3 (dd, $^2J_{C\text{-}F}$ = 21.9Hz and $^3J_{C\text{-}F}$ = 6.8 Hz), 116.8 (dd, $^2J_{C\text{-}F}$ = 15.8 Hz and $^3J_{C\text{-}F}$ = 3.0 Hz), 108.5 (d, $J_{C\text{-}F}$ = 2.5 Hz), 90.5, 61.5, 59.1. ^{19}F NMR (376 MHz, $CDCl_3$) δ -119.4 (d, $^5J_{F\text{-}F}$ = 23.9 Hz, 1F), -124.1 (d, $^5J_{F\text{-}F}$ = 23.9 Hz, 1F). HRMS (ESI) *m/z* $[M + H]^+$ calcd for $C_{17}H_{14}NO_3F_2$ 318.0936, found 318.0937.

3'-benzyl-4,5,6,7-tetrabromo-3H-spiro(isobenzofuran-1,5'-oxazolidin)-3-one **6h**



6h

The *title compound* **6h** was prepared from 3,4,5,6-tetrabromophthalic anhydride **5h** (1.81 g, 3.90 mmol) using **General Procedure 1** to afford the *title compound* **6g** (1.56 g, 67% crude yield) as a yellow amorphous solid.

R_f 0.68 (1:1 EtOAc/heptane). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42-7.39 (m, 2H), 7.36-7.31 (m, 2H), 7.28 (m, 1H), 5.06 (s, 2H), 4.20 (d, *J* = 13.2 Hz, 1H), 4.13 (d, *J* = 13.2 Hz, 1H), 3.95 (d, *J* = 13.2 Hz, 1H), 3.37 (d, *J* = 13.2 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.8, 145.7, 138.4, 137.7, 134.3, 129.2, 128.8, 127.9,

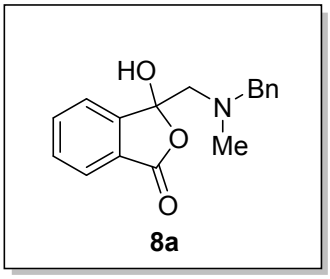
127.8, 123.7, 120.2, 108.8, 92.1, 60.2, 59.7. HRMS (ASAP) *m/z* $[M + H]^+$ calcd for $C_{17}H_{12}Br_4NO_3$ 593.7545, found 593.7548.

3. Isobenzofuran-1(3*H*)-ones (8)

The isobenzofuran-1(3*H*)-ones **8** were prepared from spiro(isobenzofuran-1,5'-oxazolidin)-3-ones **6** according to the reaction conditions described in Table 2 of the manuscript.

3-((benzyl(methyl)amino)methyl)-3-hydroxyisobenzofuran-1(3H)-one **8a**

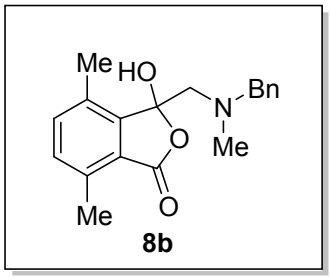
General procedure 2. To a solution of 3'-benzyl-3*H*-spiro(isobenzofuran-1,5'-oxazolidin)-3-one **6a** (260 mg, 0.92 mmol) in methanol (6.0 mL) under an atmosphere of nitrogen gas, sodium borohydride (52 mg, 1.39 mmol) was added. The resulting solution was stirred until TLC indicated consumption of starting material (18 h). The reaction was quenched with the addition of aqueous saturated NH_4Cl (15 mL) and extracted with CH_2Cl_2 (3x15 mL). The combined organic layers were dried over $MgSO_4$ and concentrated *in vacuo*. The residue was purified by dry column vacuum chromatography (i.d. 2-4 cm; 10-25 mL fractions; 0-15% MeOH in $CHCl_3$; 1% increments) to furnish the *title compound* **8a** (196 mg, 76% yield) as a yellow amorphous solid.



8a

R_f 0.43 (8:92 MeOH/$CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (d, *J* = 7.6 Hz, 1H), 7.63 (t, *J* = 7.2 Hz, 1H), 7.54 (t, *J* = 7.4 Hz, 1H), 7.38-7.29 (m, 5H), 7.25 (d, *J* = 7.4 Hz, 1H), 3.82 (s, 2H), 2.93 (s, 2H), 2.55 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.5, 147.7, 137.4, 134.5, 130.9, 129.6, 128.9, 128.2, 128.1, 125.7, 122.9, 107.6, 63.3, 62.8, 43.5. HRMS (ASAP) *m/z* $[M + H]^+$ calcd for $C_{17}H_{18}NO_3$ 284.1281, found 282.1283.

3-((benzyl(methyl)amino)methyl)-3-hydroxy-4,7-dimethylisobenzofuran-1(3H)-one **8b**

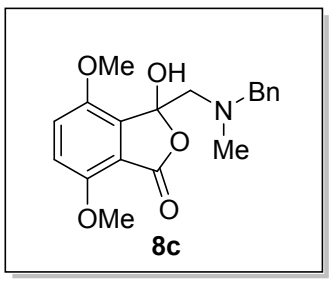


8b

The *title compound* **8b** was prepared from 3'-benzyl-4,7-dimethyl-3*H*-spiro(isobenzofuran-1,5'-oxazolidin)-3-one **6b** (283 mg, 0.91 mmol) using **General Procedure 2** to afford the *title compound* **8b** (132 mg, 69% yield) as a white crystalline solid.

***R*$_f$** 0.4 (1:9 MeOH/$CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37-7.27 (m, 5H), 7.16 (d, *J* = 7.8 Hz, 1H), 7.13 (d, *J* = 7.8 Hz, 1H), 6.80 (bs, 1H), 4.00 (d, *J* = 12.6 Hz, 1H), 3.54 (d, *J* = 12.6 Hz, 1H), 3.18 (d, *J* = 13.5 Hz, 1H), 2.64 (s, 3H), 2.58 (s, 3H), 2.52 (d, *J* = 13.5 Hz, 1H), 1.84 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.7, 145.5, 137.8, 136.8, 136.2, 132.6, 131.5, 129.8, 128.9, 128.1, 125.6, 103.9, 63.1, 61.1, 44.4, 17.4, 16.2. HRMS (ESI) *m/z* $[M + H]^+$ calcd for $C_{19}H_{22}NO_3$ 312.1594, found 312.1594; *m/z* $[M - H]^-$ calcd for $C_{19}H_{20}NO_5$ 310.1449, found 310.1450.

3-((benzyl(methyl)amino)methyl)-3-hydroxy-4,7-dimethoxyisobenzofuran-1(3H)-one **8c**



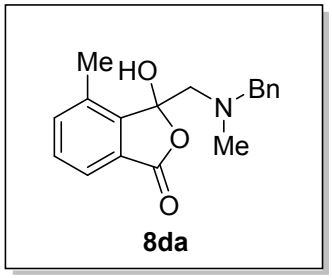
8c

The *title compound* **8c** was prepared from 3'-benzyl-4,7-dimethoxy-3*H*-spiro(isobenzofuran-1,5'-oxazolidin)-3-one **6c** (450 mg, 1.32 mmol) using **General Procedure 2** to afford the *title compound* **8c** (146 mg, 39%) as a viscous yellow oil.

R_f 0.4 (5:95 MeOH/$CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36-7.26 (m, 5H), 7.00 (d, *J* = 8.9 Hz, 1H), 6.89 (d, *J* = 8.9 Hz, 1H), 3.89 (s, 3H), 3.78 (bs, 2H), 3.62 (s, 3H), 3.21 (bs, 1H), 2.82 (bs, 1H), 2.53 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.2, 152.1, 148.5, 137.7, 136.1, 129.6, 128.7, 127.7, 118.7, 116.6, 114.2,

103.3, 63.0, 60.8, 56.5, 56.3, 43.8. HRMS (ESI) *m/z* $[M + H]^+$ calcd for $C_{19}H_{22}NO_5$ 344.1492, found 344.1493; *m/z* $[M - H]^-$ calcd for $C_{19}H_{20}NO_3$ 342.1347, found 342.1350.

3-((benzyl(methyl)amino)methyl)-3-hydroxy-4-methylisobenzofuran-1(3H)-one **8da**

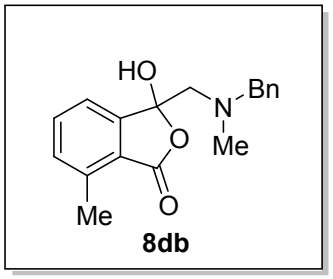


8da

The *title compound* **8da** was prepared from the regioisomeric mixture of 3'-benzyl-7-methyl-3*H*-spiro(isobenzofuran-1,5'-oxazolidin)-3-one **6da** and 3'-benzyl-4-methyl-3*H*-spiro(isobenzofuran-1,5'-oxazolidin)-3-one **6db** (299 mg, 1.01 mmol) using **General Procedure 2** to afford the *title compound* **8da** (81 mg, 31% yield) as a white amorphous solid.

R_f 0.69 (1:9 MeOH/$CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (d, *J* = 7.2 Hz, 1H), 7.41-7.27 (m, 7H), 6.77 (bs, 1H), 3.97 (bs, 1H), 3.59 (bs, 1H), 3.17 (bs, 1H), 2.65 (s, 3H), 2.60 (bs, 1H), 1.88 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.6, 145.1, 137.7, 136.4, 134.5, 130.8, 129.8, 129.0, 128.4, 128.1, 123.0, 105.3, 63.1, 60.9, 44.4, 16.5. HRMS (ASAP) *m/z* $[M + H]^+$ calcd for $C_{18}H_{20}NO_3$ 298.1438, found 298.1432.

3-((benzyl(methyl)amino)methyl)-3-hydroxy-7-methylisobenzofuran-1(3H)-one **8db**

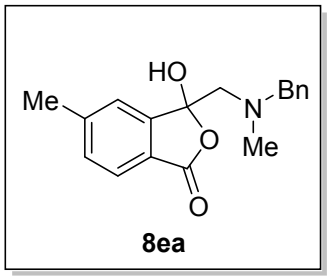


8db

The *title compound* **8db** was prepared from the regioisomeric mixture of 3'-benzyl-7-methyl-3*H*-spiro(isobenzofuran-1,5'-oxazolidin)-3-one **6da** and 3'-benzyl-4-methyl-3*H*-spiro(isobenzofuran-1,5'-oxazolidin)-3-one **6db** (299 mg, 1.01 mmol) using **General Procedure 2** to afford the *title compound* **8db** (79 mg, 31% yield) as a viscous colourless oil.

R_f 0.62 (1:9 MeOH/$CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 (t, *J* = 7.6 Hz, 1H), 7.38-7.27 (m, 6H), 7.05 (d, *J* = 7.6 Hz, 1H), 3.79 (bs, 2H), 2.87 (bs, 2H), 2.65 (s, 3H), 2.53 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.6, 148.5, 139.7, 137.7, 134.2, 132.5, 129.5, 128.8, 127.9, 125.4, 120.1, 103.7, 63.6, 63.0, 43.6, 17.7. HRMS (ASAP) *m/z* $[M + H]^+$ calcd for $C_{18}H_{20}NO_3$ 298.1438, found 298.1437.

3-((benzyl(methyl)amino)methyl)-3-hydroxy-5-methylisobenzofuran-1(3H)-one **8ea**

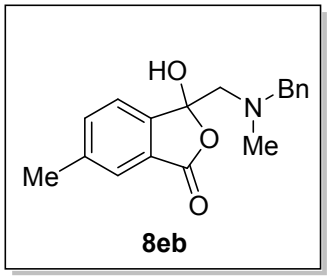


8ea

The *title compound* **8ea** was prepared from the regioisomeric mixture of 3'-benzyl-6-methyl-3H-spiro(isobenzofuran-1,5'-oxazolidin)-3-one **6ea** *and* 3'-benzyl-5-methyl-3H-spiro(isobenzofuran-1,5'-oxazolidin)-3-one **6eb** (596 mg, 1.01 mmol) using **General Procedure 2** to afford the *title compound* **8ea** (143 mg, 29%yield) as a pale yellow amorphous solid.

R_f 0.40 (1:9 MeOH/$CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (d, *J* = 7.8 Hz, 1H), 7.37-7.28 (m, 6H), 7.04 (s, 1H), 4.99 (bs, 1H), 3.82 (s, 2H), 2.96 (s, 2H), 2.52 (s, 3H), 2.42 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.6, 148.2, 145.7, 137.6, 131.9, 129.5, 128.8, 127.9, 125.7, 125.5, 123.2, 108.0, 63.6, 62.9, 43.4, 22.2. HRMS (ASAP) *m/z* $[M + H]^+$ calcd for $C_{18}H_{20}NO_3$ 298.1438, found 298.1444.

3-((benzyl(methyl)amino)methyl)-3-hydroxy-6-methylisobenzofuran-1(3H)-one **8eb**

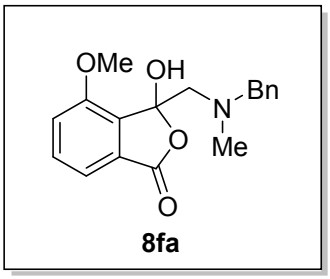


8eb

The *title compound* **8eb** was prepared from the regioisomeric mixture of 3'-benzyl-6-methyl-3H-spiro(isobenzofuran-1,5'-oxazolidin)-3-one **6ea** *and* 3'-benzyl-5-methyl-3H-spiro(isobenzofuran-1,5'-oxazolidin)-3-one **6eb** (596 mg, 1.01 mmol) using **General Procedure 2** to afford the *title compound* **8eb** (69 mg, 14% yield) as a viscous yellow oil.

R_f 0.32 (1:9 MeOH/$CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (s, 1H), 7.40 (d, *J* = 7.8 Hz, 1H), 7.36-7.28 (m, 5H), 7.14 (d, *J* = 7.8 Hz, 1H), 5.52 (bs, 1H), 3.82 (s, 2H), 2.98 (s, 2H), 2.52 (s, 3H), 2.42 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.8, 144.9, 141.3, 137.6, 135.3, 129.5, 129.2, 128.7, 128.0, 126.0, 122.7, (C_q not observed), 63.5, 62.8, 43.4, 21.5. HRMS (ASAP) *m/z* $[M + H]^+$ calcd for $C_{18}H_{20}NO_3$ 298.1438, found 298.1432.

3-((benzyl(methyl)amino)methyl)-3-hydroxy-4-methoxyisobenzofuran-1(3H)-one **8fa**

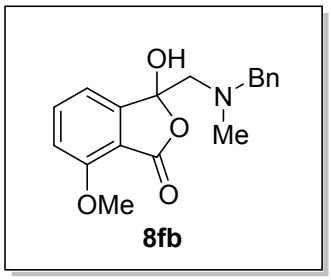


8fa

The *title compound* **8fa** was prepared from the regioisomeric mixture of 3'-benzyl-7-methoxy-3H-spiro(isobenzofuran-1,5'-oxazolidin)-3-one **6fa** and 3'-benzyl-4-methoxy-3H-spiro(isobenzofuran-1,5'-oxazolidin)-3-one **6fb** (314 mg, 1.01 mmol) using **General Procedure 2** to afford the *title compound* **8fa** (79 mg, 26%yield) as a yellow oil.

R_f 0.20 (5:95 MeOH/$CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (bs, 2H), 7.33-7.25 (m, 5H), 7.05 (bs, 1H), 5.58 (bs, 1H), 3.80 (s, 2H), 3.69 (s, 3H), 3.14 (bs, 2H), 2.53 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.8, 155.2, 137.9, 133.9, 132.5, 129.7, 128.7, 128.6, 127.7, 117.7, 115.9, (C_q not observed), 62.9, 61.3, 55.9, 43.7. HRMS (ASAP) *m/z* $[M + H]^+$ calcd for $C_{18}H_{20}NO_4$ 314.1387, found 314.1385.

3-((benzyl(methyl)amino)methyl)-3-hydroxy-7-methoxyisobenzofuran-1(3H)-one **8fb**

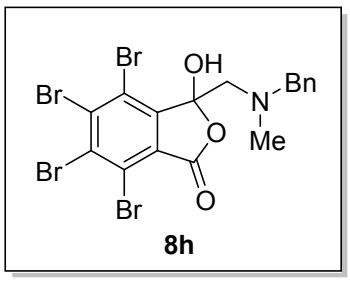


8fb

The *title compound* **8fb** was prepared from the regioisomeric mixture of 3'-benzyl-7-methoxy-3H-spiro(isobenzofuran-1,5'-oxazolidin)-3-one **6fa** and 3'-benzyl-4-methoxy-3H-spiro(isobenzofuran-1,5'-oxazolidin)-3-one **6fb** (314 mg, 1.01 mmol) using **General Procedure 2** to afford the *title compound* **8fb** (100 mg, 33%yield) as a viscous light yellow oil.

R_f 0.47 (5:95 MeOH/$CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 (dd, *J* = 8.3 and 7.5 Hz, 1H), 7.38-7.27 (m, 5H), 6.95 (d, *J* = 8.3 Hz, 1H), 6.78 (d, *J* = 7.5 Hz, 1H), 3.97 (s, 3H), 3.71 (bs, 2H), 2.87 (bs, 2H), 2.52 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.4, 158.4, 150.8, 137.7, 136.7, 129.5, 128.8, 127.9, 115.2, 114.4, 112.6, 103.4, 63.5, 62.9, 56.3, 43.6. HRMS (ASAP) *m/z* $[M + H]^+$ calcd for $C_{18}H_{20}NO_4$ 314.1387, found 314.1388.

3-((benzyl(methyl)amino)methyl)-4,5,6,7-tetrabromo-3-hydroxyisobenzofuran-1(3H)-one **8h**

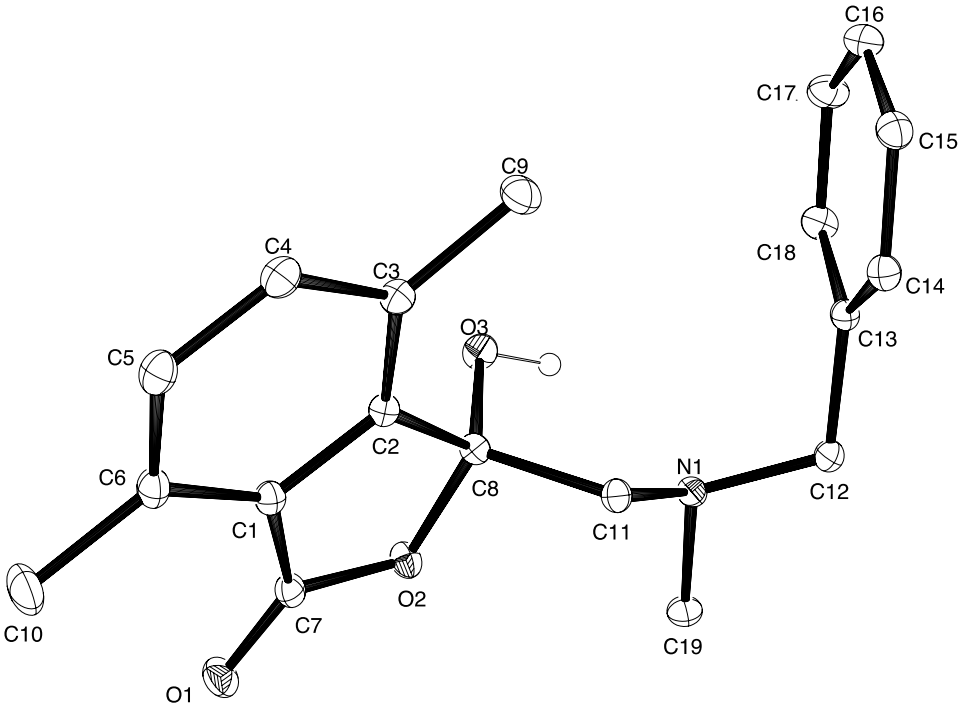


8h

The *title compound* **8h** was prepared from *3'-benzyl-4,5,6,7-tetrabromo-3H-spiro(isobenzofuran-1,5'-oxazolidin)-3-one* **6h** (601 mg, 1.01 mmol) using **General Procedure 2** to afford the *title compound* **8h** (107 mg, 21% yield) as an amorphous yellow solid.

R_f 0.80 (8:92 MeOH/$CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38-7.28 (m, 5H), 3.99 (bd, *J* = 12.0 Hz, 1H), 3.63-3.59 (m, 2H), 2.61 (s, 3H), 2.56 (bd, *J* = 12.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.1, 147.3, 137.5, 137.3, 134.1, 130.0, 129.0, 128.2, 128.1, 123.3, 120.8, 103.4, 62.9, 59.5, 44.0. HRMS (ASAP) m/z $[M + H]^+$ calcd for $C_{17}H_{14}{}^{79}Br_4NO_3$ 595.7702, found 595.7704.

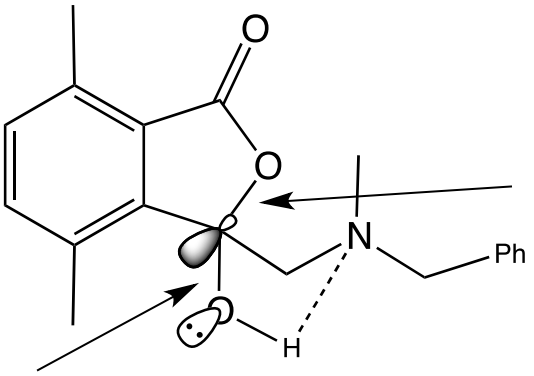
4. X-ray crystallography of 8b and CCDC searches



Crystallography. Intensity data were collected on an Oxford SuperNova CCD diffractometer using Mo-Kα radiation (graphite crystal monochromators) λ = 0.71073 Å, the temperature during data collection was maintained at 130.0(1). The structure was solved by direct methods (SHELXT) and difference Fourier synthesis (SHELX-2014).[7] Thermal ellipsoid plots were generated using the program ORTEP-3[8] integrated within the WINGX[9] suite of programs.

Crystal data for **8b**. $C_{19}H_{21}NO_3$, M = 311.37, T = 130.0(2) K, λ = 0.71073 Å, Monoclinic, space group $P2_1/c$ a = 7.2941(2), b = 13.2249(3), c = 17.2264(4) Å, β= 96.919(2)° V = 1649.62(7) Å^3, Z = 4, D_c = 1.254 Mg M^{-3} μ(Mo-Kα) = 0.085 mm^{-1}, *F(000)* = 664, crystal size 0.49 x 0.38 x 0.29 mm. θ_{max} = 36.5°, 45689 reflections measured, 7831 independent reflections (R_{int} = 0.034) the final R = 0.0461 [I > 2σ(I), 6084 data] and wR(F^2) = 0.10369 (all data) GOOF = 1.073.

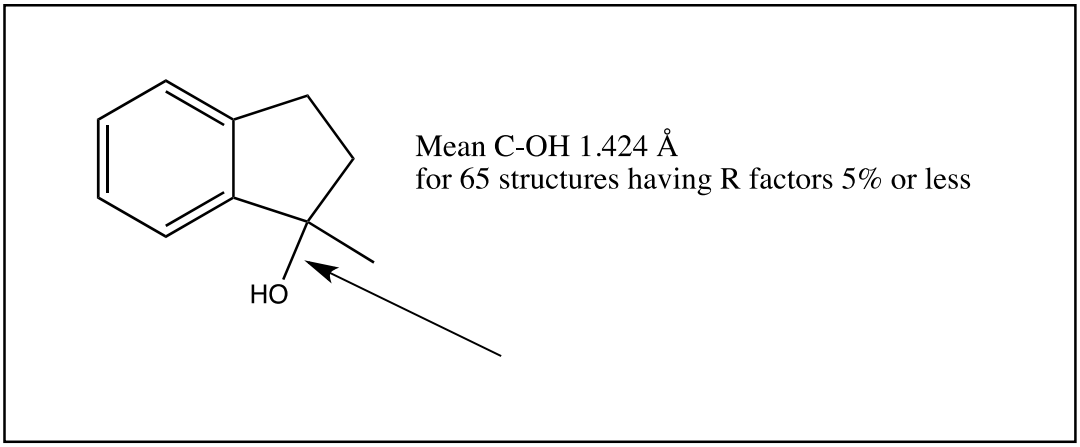
Anomeric Effect:

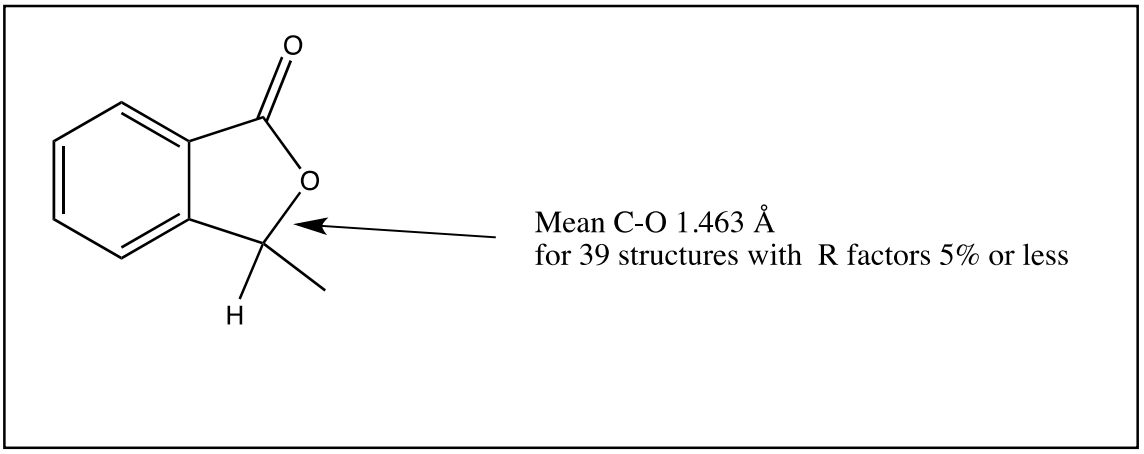


1.477(1) Å

1.383(1) Å

CCDC search of the following fragments:





5. References

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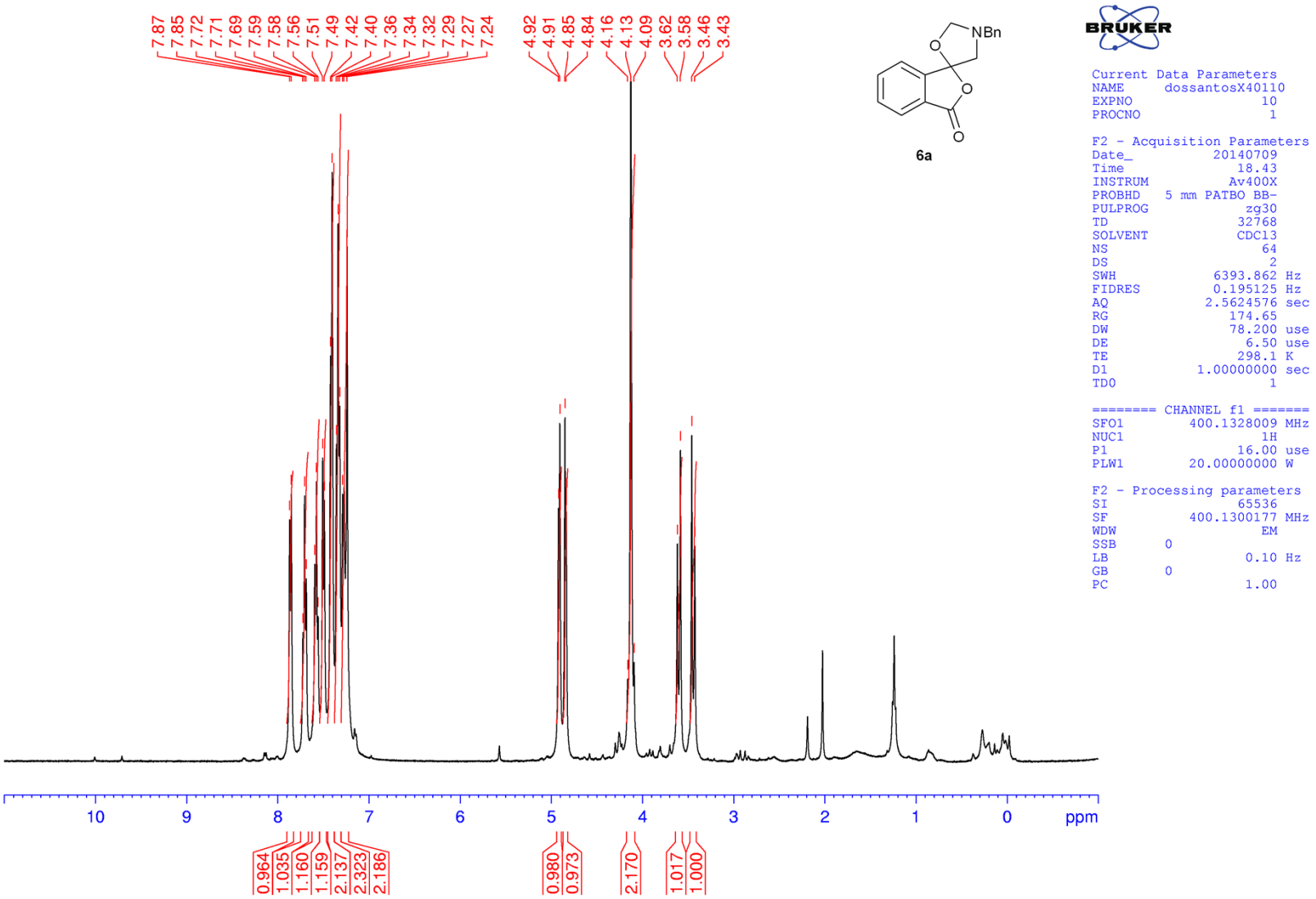
6. ^{1}H, ^{13}C and ^{19}F NMR spectra for new compounds (only resonance signals associated with each compound have been integrated and/or assigned chemical shift values)



BRUKER

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PROCNO                  1

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PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    64
DS                     2
SWH             6393.862 Hz
FIDRES          0.195125 Hz
AQ              2.5624576 sec
RG                174.65
DW                78.200 use
DE                 6.50 use
TE                298.1 K
D1           1.00000000 sec
TD0                    1

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NUC1                   1H
P1                 16.00 use
PLW1         20.00000000 W

F2 - Processing parameters
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SF           400.1300177 MHz
WDW                   EM
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LB                 0.10 Hz
GB        0
PC                 1.00
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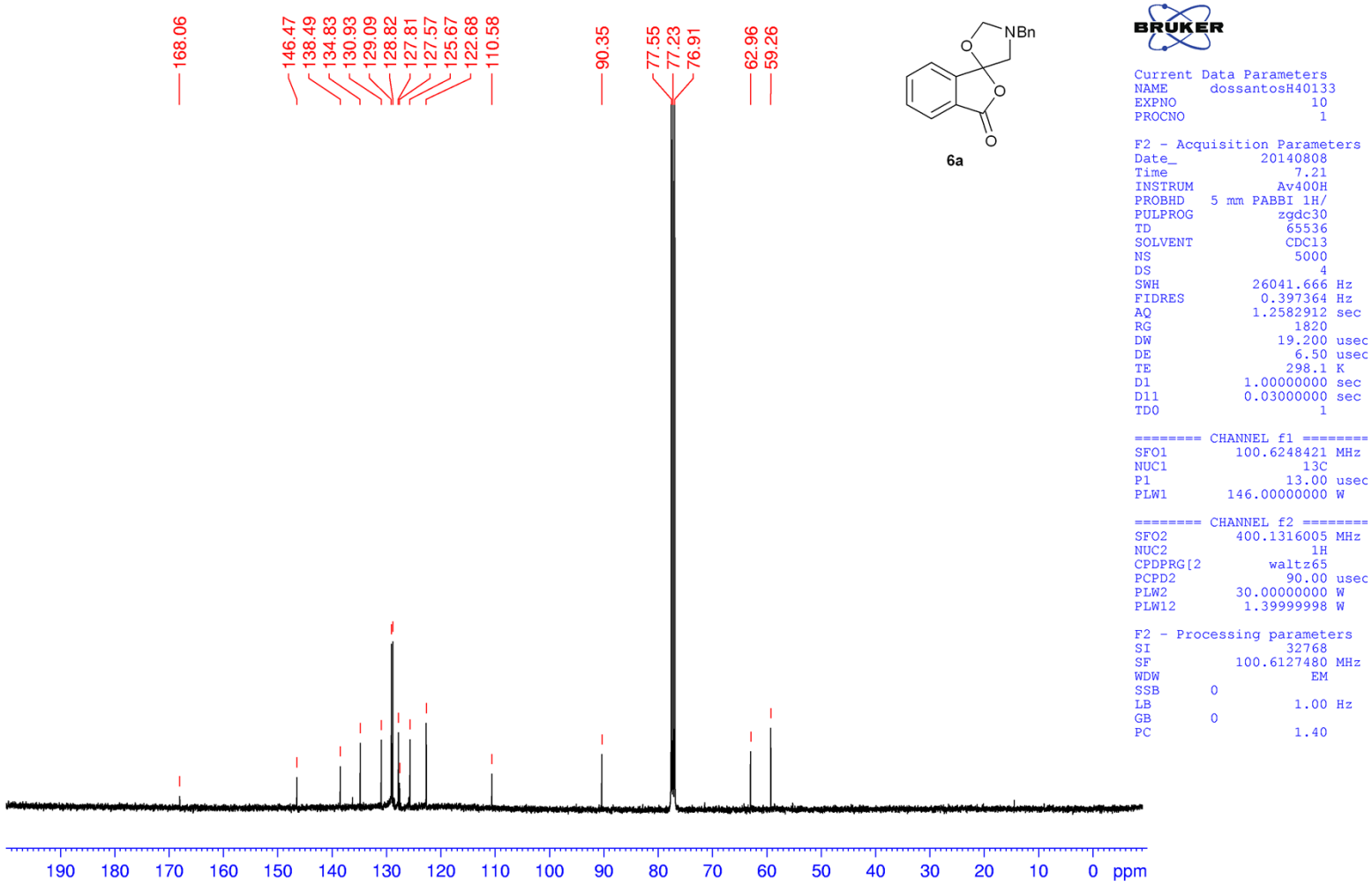
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of cycloadduct **6a**.



BRUKER

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DS                    4
SWH           26041.666 Hz
FIDRES         0.397364 Hz
AQ            1.2582912 sec
RG                 1820
DW               19.200 usec
DE                6.50 usec
TE                298.1 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

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NUC1                 13C
P1                 13.00 usec
PLW1       146.00000000 W

======== CHANNEL f2 ========
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NUC2                 1H
CPDPRG[2         waltz65
PCPD2              90.00 usec
PLW2         30.00000000 W
PLW12         1.39999998 W

F2 - Processing parameters
SI                 32768
SF           100.6127480 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
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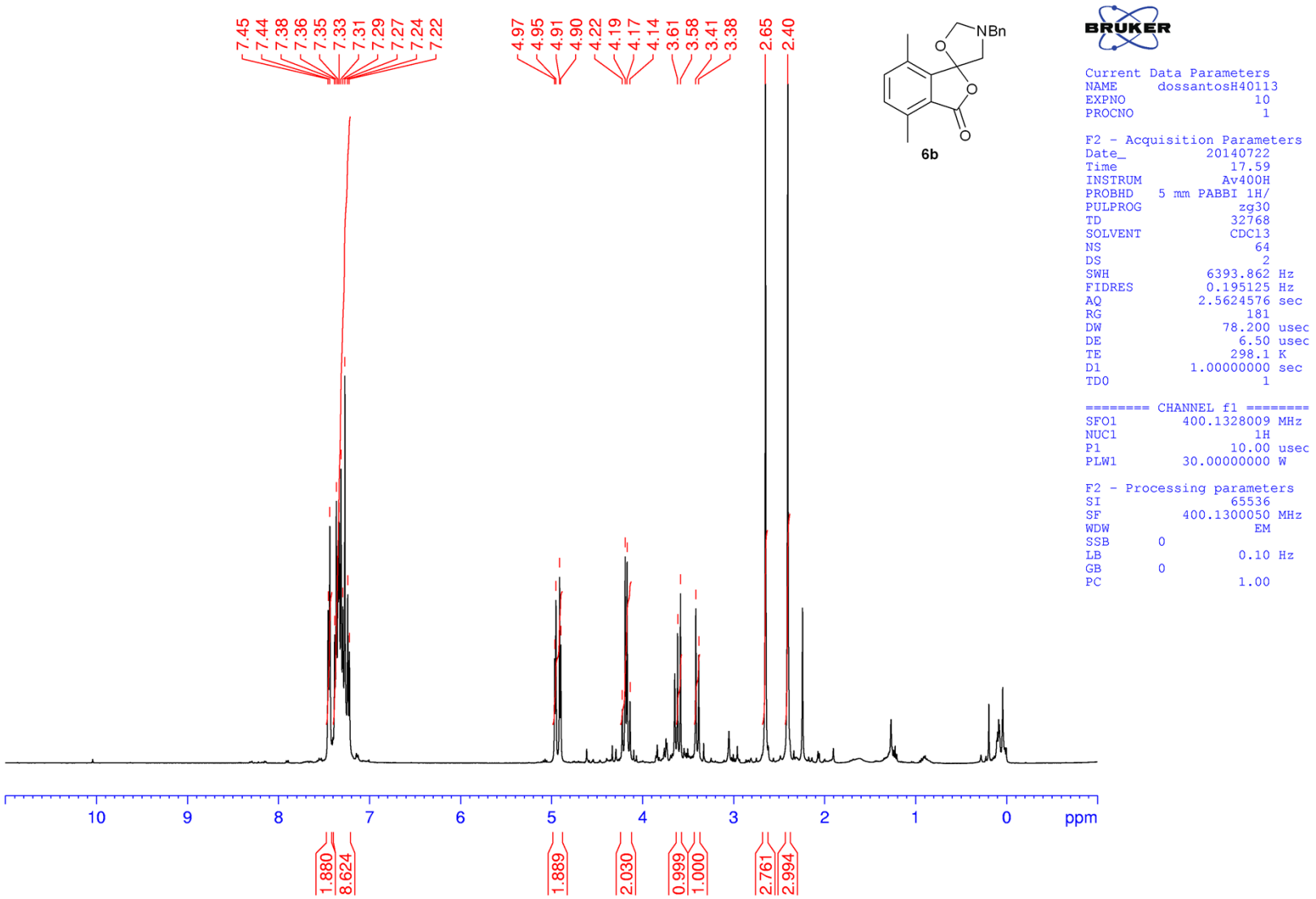
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of cycloadduct **6a**.

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PROCNO                  1

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TD                32768
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NS                   64
DS                    2
SWH             6393.862 Hz
FIDRES          0.195125 Hz
AQ             2.5624576 sec
RG                  181
DW                78.200 usec
DE                 6.50 usec
TE                298.1 K
D1           1.00000000 sec
TD0                    1

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NUC1                   1H
P1                 10.00 usec
PLW1         30.00000000 W

F2 - Processing parameters
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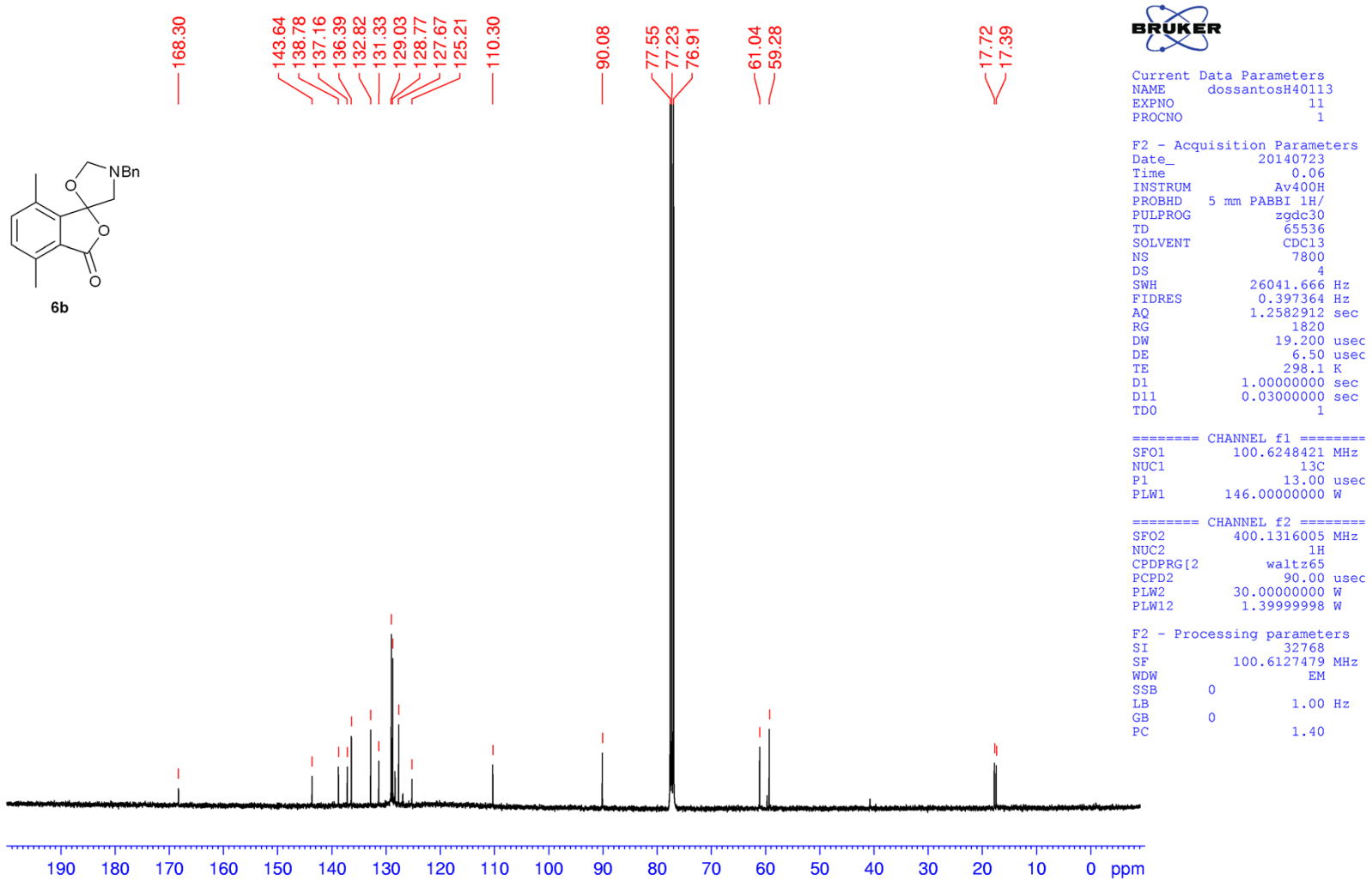
WDW                   EM
SSB       0
LB                 0.10 Hz
GB        0
PC                 1.00
```

^{1}H NMR (400 MHz, $CDCl_3$) spectrum of cycloadduct **6b**.



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```
Current Data Parameters
NAME      dossantosH40113
EXPNO                  11
PROCNO                  1

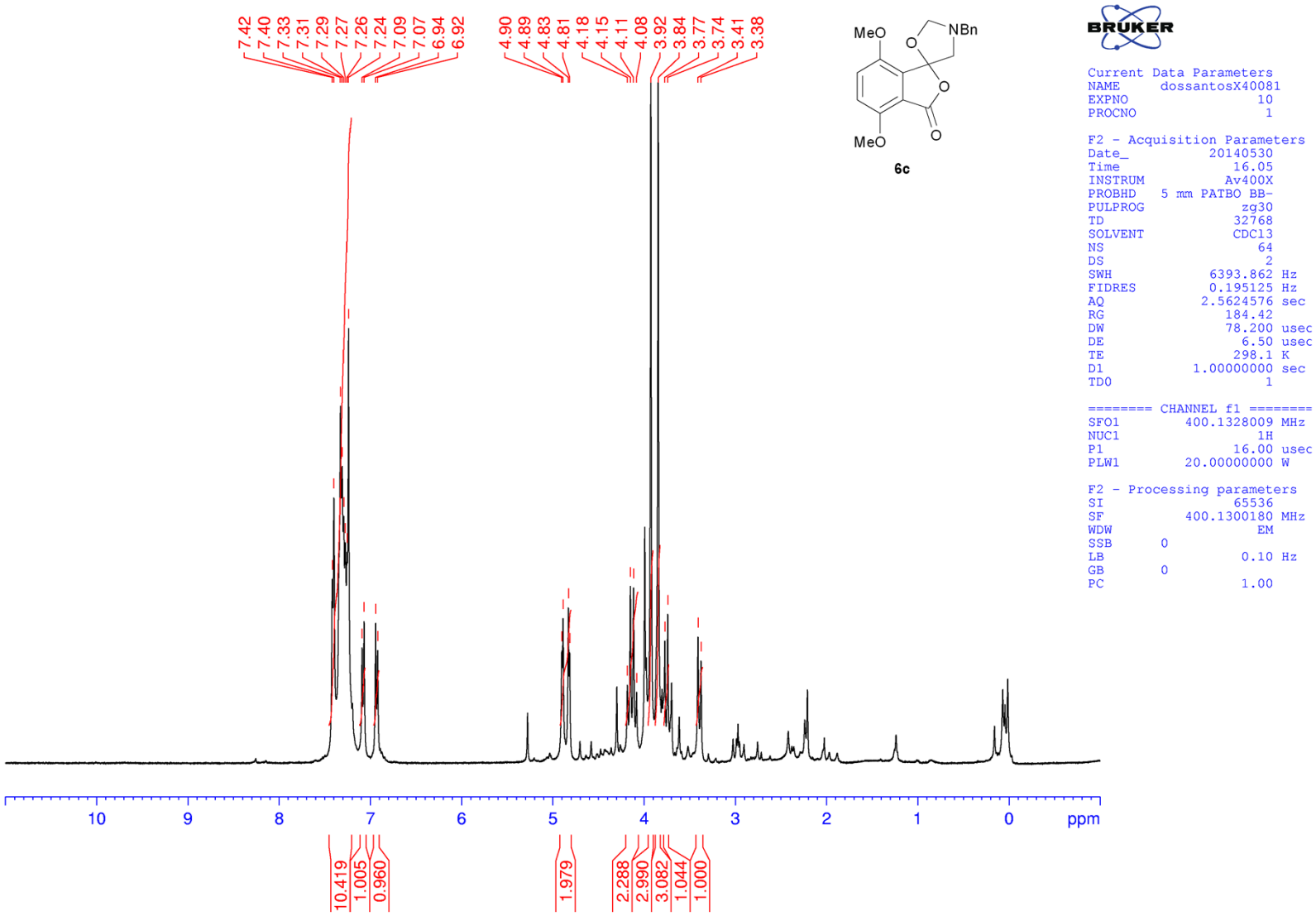
F2 - Acquisition Parameters
Date_            20140723
Time                 0.06
INSTRUM           Av400H
PROBHD   5 mm PABBI 1H/
PULPROG          zgdc30
TD                65536
SOLVENT           CDCl3
NS                 7800
DS                    4
SWH           26041.666 Hz
FIDRES         0.397364 Hz
AQ            1.2582912 sec
RG                 1820
DW               19.200 usec
DE                6.50 usec
TE               298.1 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6248421 MHz
NUC1                13C
P1                13.00 usec
PLW1       146.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz65
PCPD2             90.00 usec
PLW2         30.00000000 W
PLW12         1.39999998 W

F2 - Processing parameters
SI                 32768
SF           100.6127479 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

^{13}C NMR (100 MHz, $CDCl_3$) spectrum of cycloadduct **6b**.



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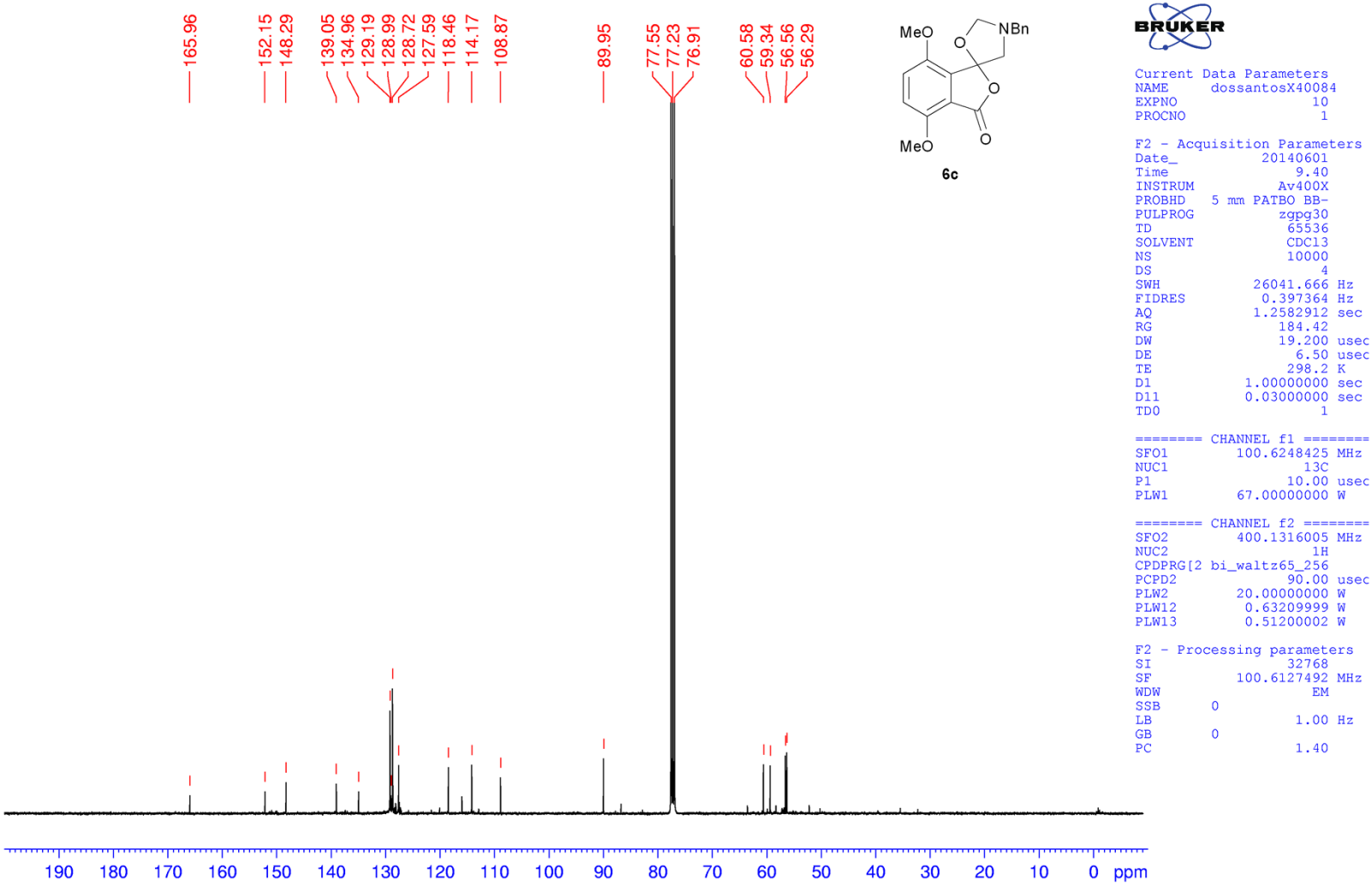
```
Current Data Parameters
NAME      dossantosX40081
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20140530
Time               16.05
INSTRUM           Av400X
PROBHD   5 mm PATBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    64
DS                     2
SWH             6393.862 Hz
FIDRES          0.195125 Hz
AQ             2.5624576 sec
RG                184.42
DW                78.200 usec
DE                 6.50 usec
TE                298.1 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1328009 MHz
NUC1                   1H
P1                 16.00 usec
PLW1         20.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300180 MHz
WDW                   EM
SSB       0
LB                 0.10 Hz
GB        0
PC                 1.00
```

^{1}H NMR (400 MHz, $CDCl_3$) spectrum of cycloadduct **6c**.




```
Current Data Parameters
NAME      dossantosX40084
EXPNO                  10
PROCNO                  1

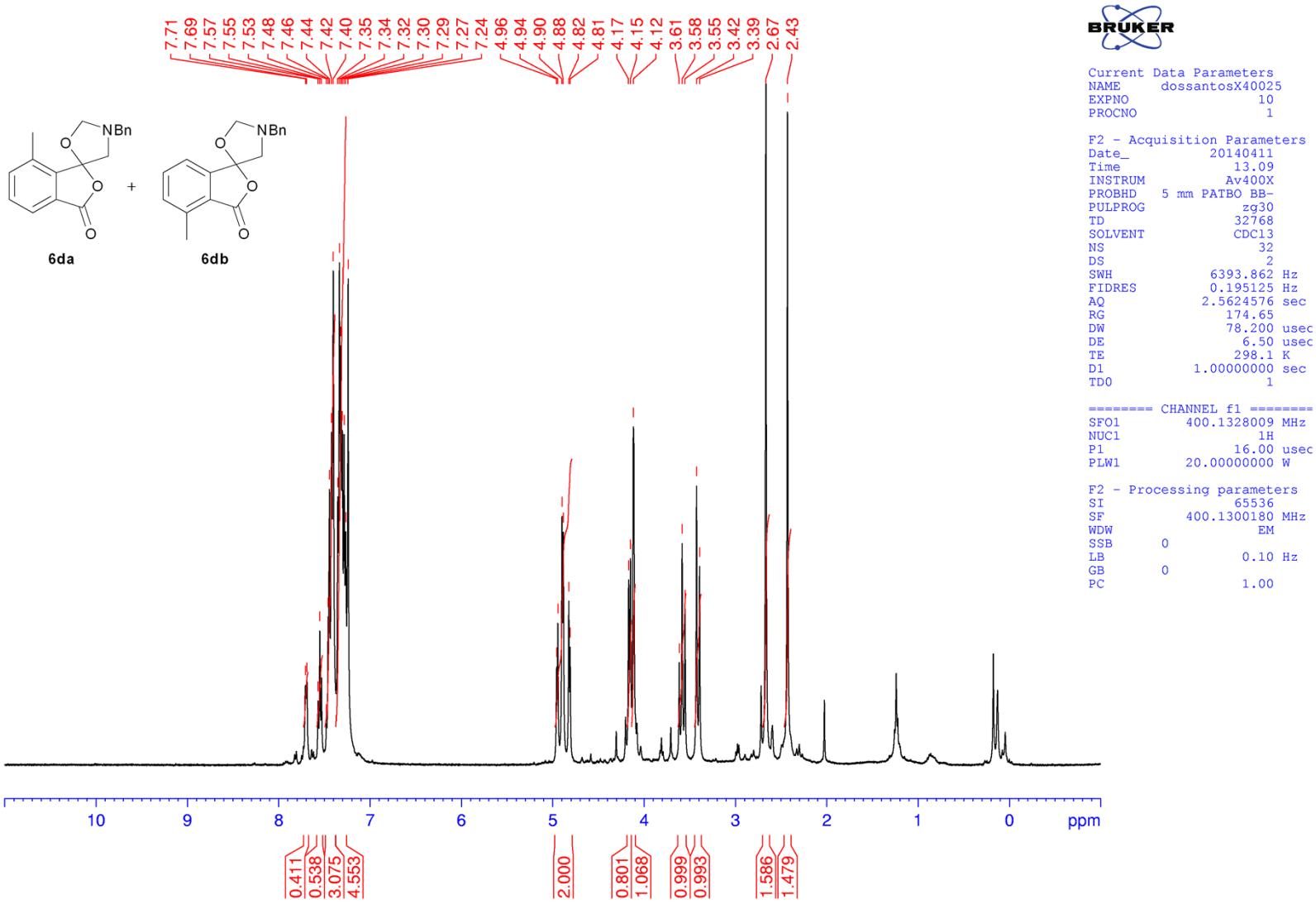
F2 - Acquisition Parameters
Date_           20140601
Time                9.40
INSTRUM          Av400X
PROBHD   5 mm PATBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                10000
DS                    4
SWH           26041.666 Hz
FIDRES         0.397364 Hz
AQ            1.2582912 sec
RG               184.42
DW               19.200 usec
DE                6.50 usec
TE               298.2 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6248425 MHz
NUC1               13C
P1               10.00 usec
PLW1        67.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2 bi_waltz65_256
PCPD2            90.00 usec
PLW2        20.00000000 W
PLW12        0.63209999 W
PLW13        0.51200002 W

F2 - Processing parameters
SI                32768
SF          100.6127492 MHz
WDW                EM
SSB       0
LB               1.00 Hz
GB        0
PC               1.40
```

^{13}C NMR (100 MHz, $CDCl_3$) spectrum of cycloadduct **6c**.



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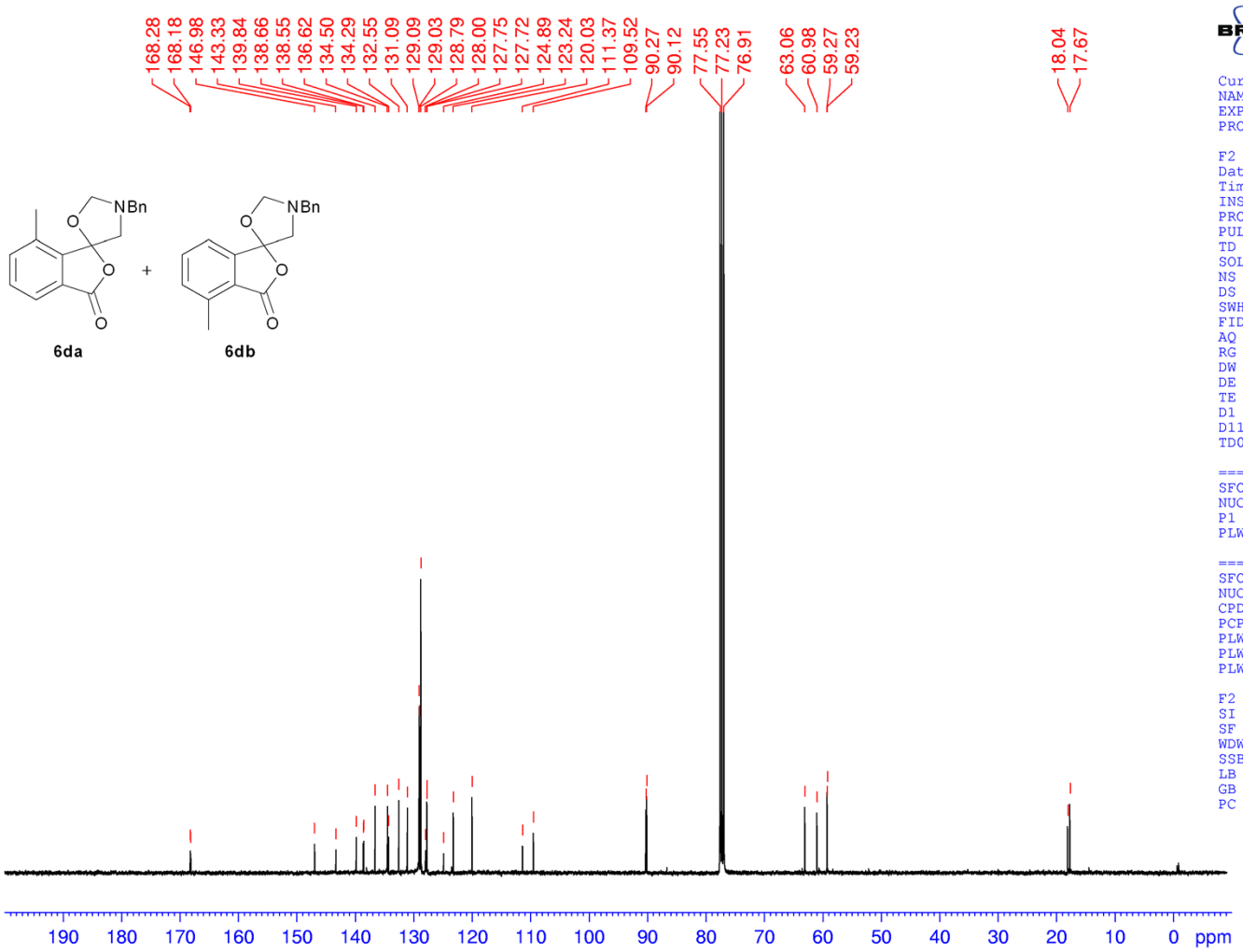
Current Data Parameters
NAME dossantosX40025
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140411
Time 13.09
INSTRUM Av400X
PROBHD 5 mm PATBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5624576 sec
RG 174.65
DW 78.200 usec
DE 6.50 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1328009 MHz
NUC1 1H
P1 16.00 usec
PLW1 20.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1300180 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

^{1}H NMR (400 MHz, $CDCl_3$) spectrum of cycloadducts **6da** and **6db**.




```
Current Data Parameters
NAME      dossantosX40030
EXPNO                  10
PROCNO                  1

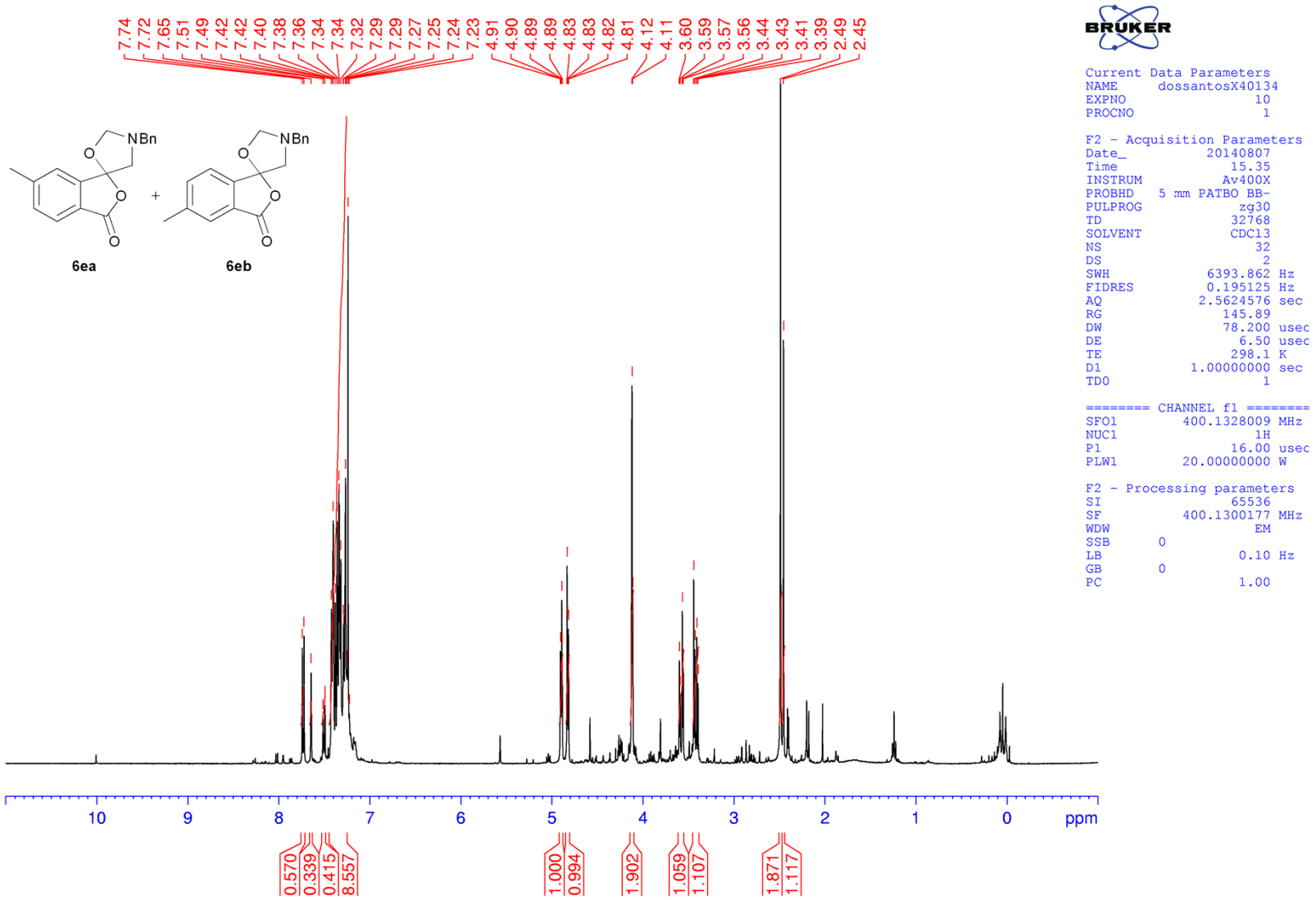
F2 - Acquisition Parameters
Date_            20140412
Time                8.04
INSTRUM           Av400X
PROBHD   5 mm PATBO BB-
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3600
DS                    4
SWH           26041.666 Hz
FIDRES         0.397364 Hz
AQ            1.2582912 sec
RG               184.42
DW               19.200 usec
DE                6.50 usec
TE                298.1 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6248425 MHz
NUC1                13C
P1                10.00 usec
PLW1         67.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2 bi_waltz65_256
PCPD2             90.00 usec
PLW2         20.00000000 W
PLW12         0.63209999 W
PLW13         0.51200002 W

F2 - Processing parameters
SI                 32768
SF           100.6127492 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{13}C NMR (100 MHz, $CDCl_3$) spectrum of cycloadducts **6da** and **6db**.



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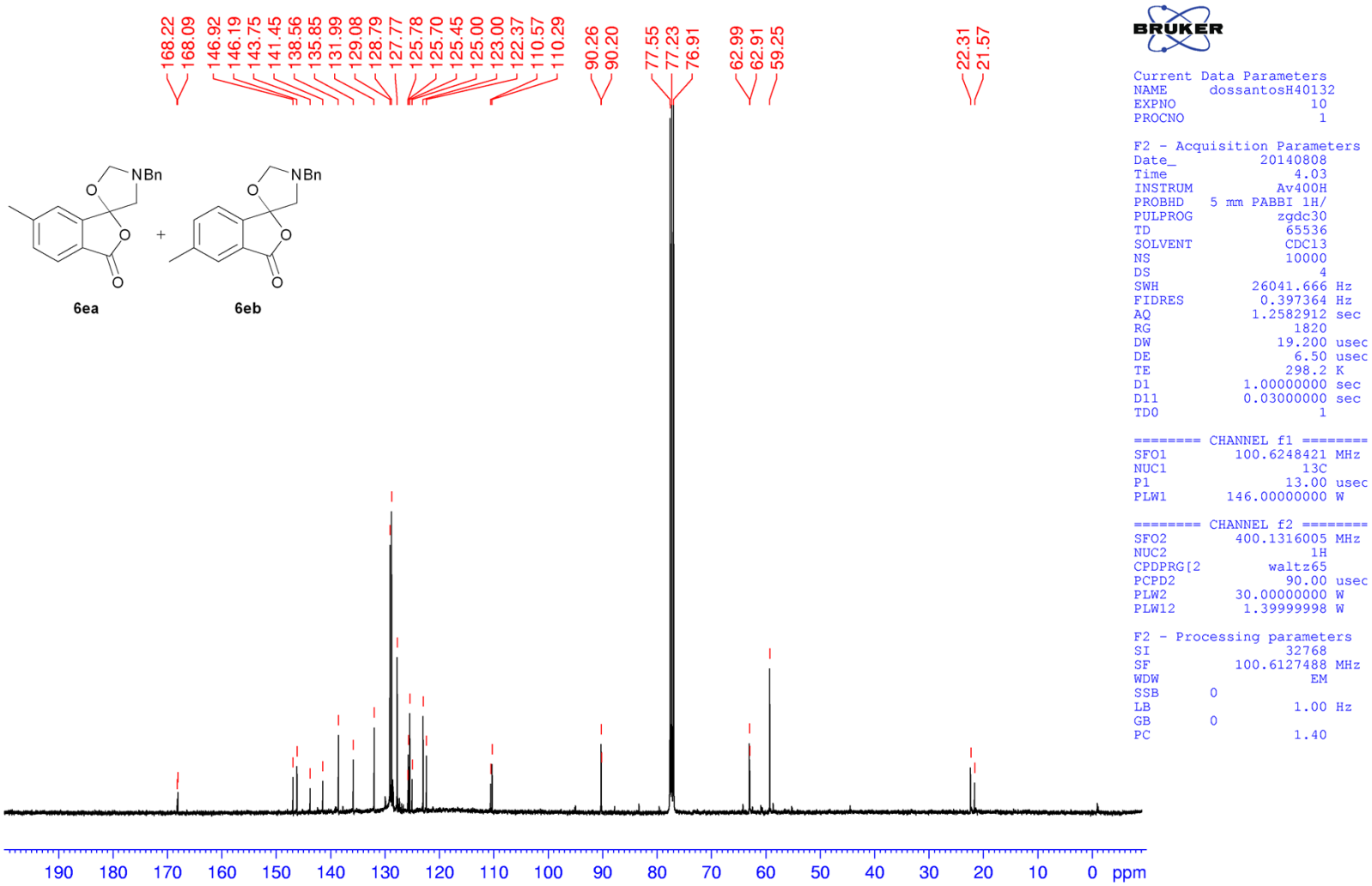
```
Current Data Parameters
NAME      dossantosX40134
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_           20140807
Time               15.35
INSTRUM          Av400X
PROBHD   5 mm PATBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     2
SWH             6393.862 Hz
FIDRES          0.195125 Hz
AQ             2.5624576 sec
RG                145.89
DW                78.200 usec
DE                 6.50 usec
TE                298.1 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1328009 MHz
NUC1                   1H
P1                 16.00 usec
PLW1         20.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300177 MHz
WDW                   EM
SSB       0
LB                 0.10 Hz
GB        0
PC                 1.00
```

^{1}H NMR (400 MHz, $CDCl_3$) spectrum of cycloadducts **6ea** and **6eb**.



BRUKER

```
Current Data Parameters
NAME      dossantosH40132
EXPNO                  10
PROCNO                  1

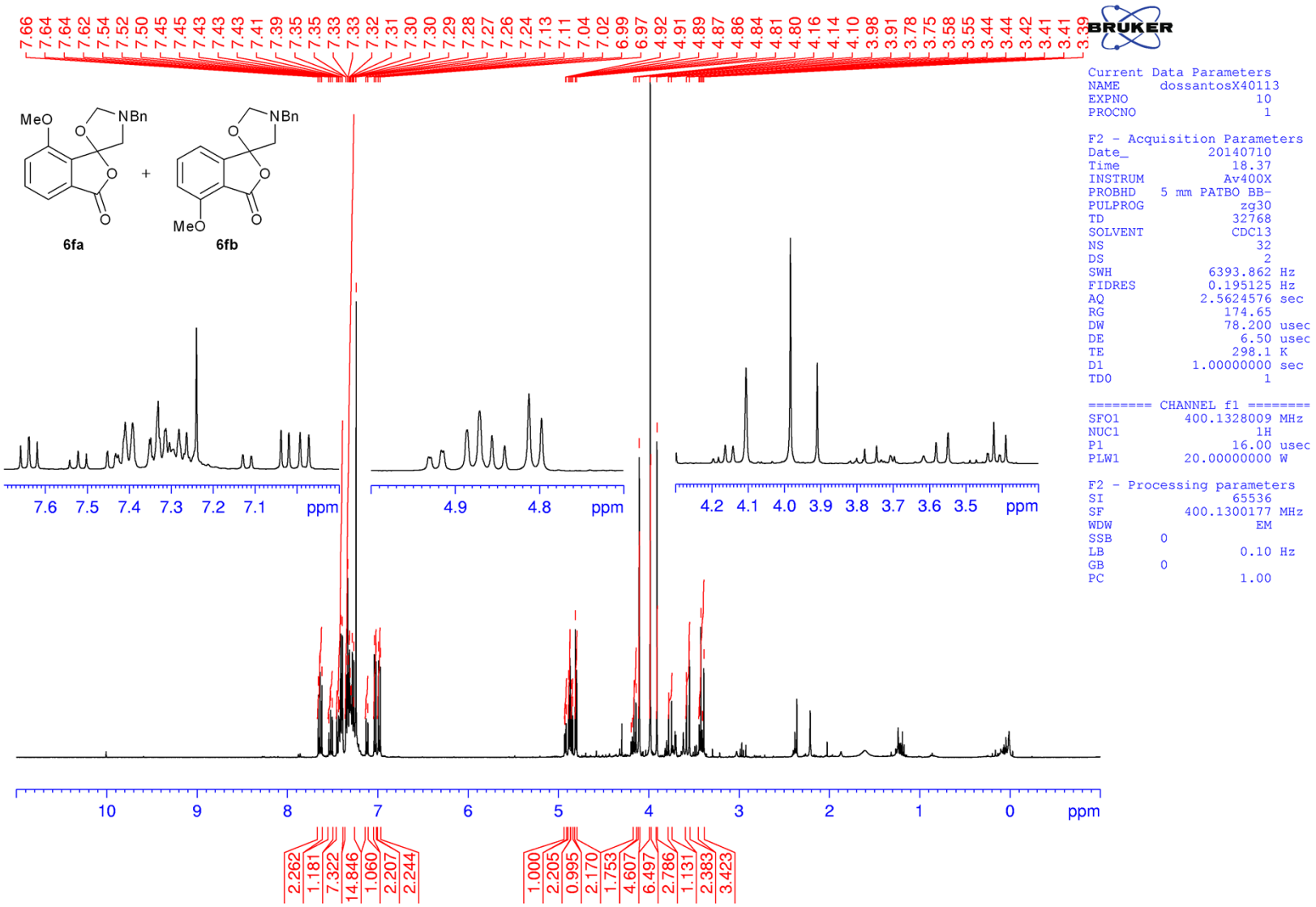
F2 - Acquisition Parameters
Date_            20140808
Time                4.03
INSTRUM            Av400H
PROBHD   5 mm PABBI 1H/
PULPROG          zgdc30
TD                65536
SOLVENT           CDCl3
NS                10000
DS                    4
SWH           26041.666 Hz
FIDRES         0.397364 Hz
AQ            1.2582912 sec
RG                 1820
DW               19.200 usec
DE                6.50 usec
TE                298.2 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6248421 MHz
NUC1                13C
P1                13.00 usec
PLW1       146.00000000 W

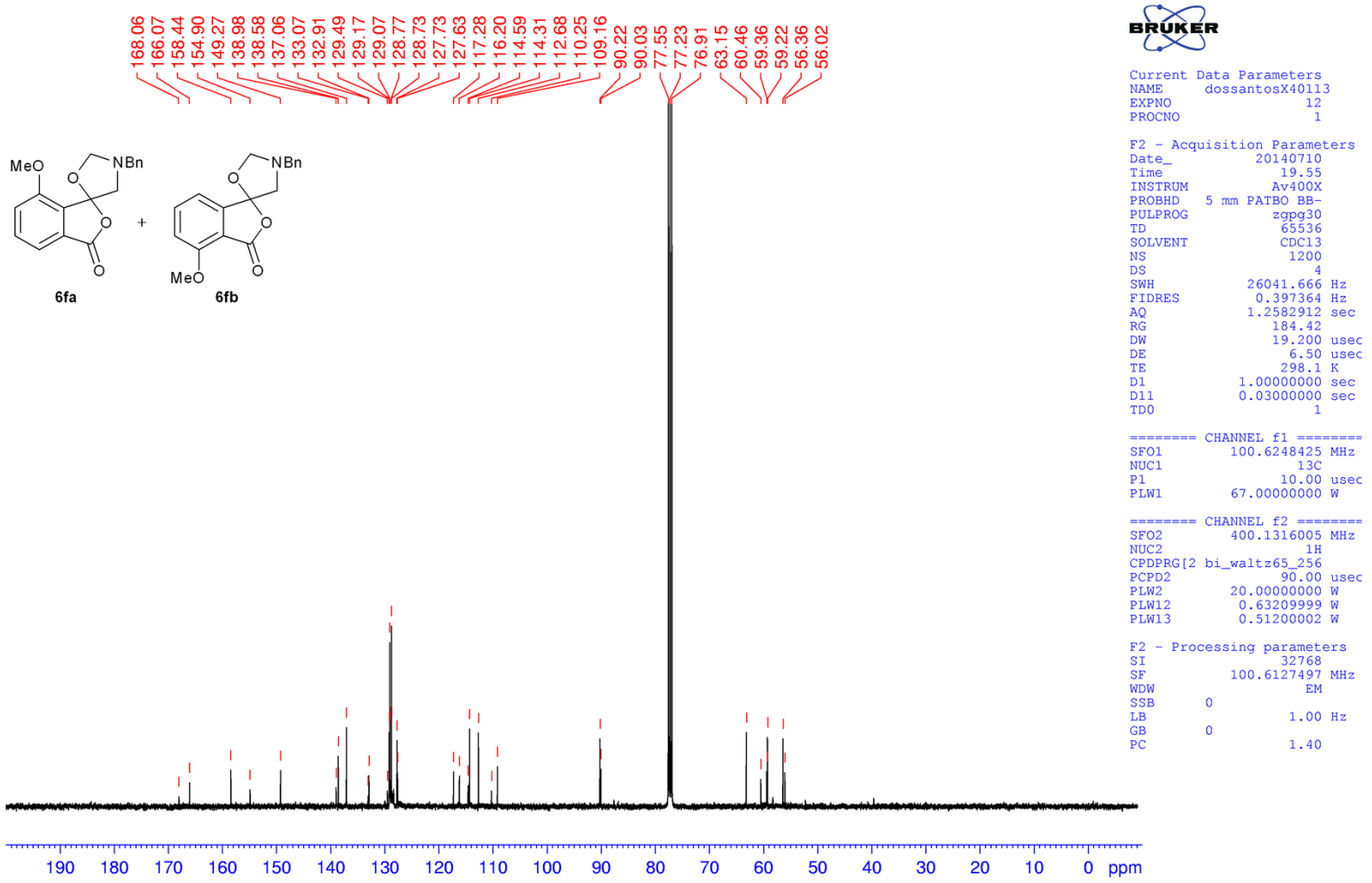
======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz65
PCPD2             90.00 usec
PLW2        30.00000000 W
PLW12        1.39999998 W

F2 - Processing parameters
SI                 32768
SF          100.6127488 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{13}C NMR (100 MHz, $CDCl_3$) spectrum of cycloadducts **6ea** and **6eb**.



^{1}H NMR (400 MHz, $CDCl_3$) spectrum of cycloadducts **6fa** and **6fb**.




```
Current Data Parameters
NAME      dossantosX40113
EXPNO                  12
PROCNO                  1

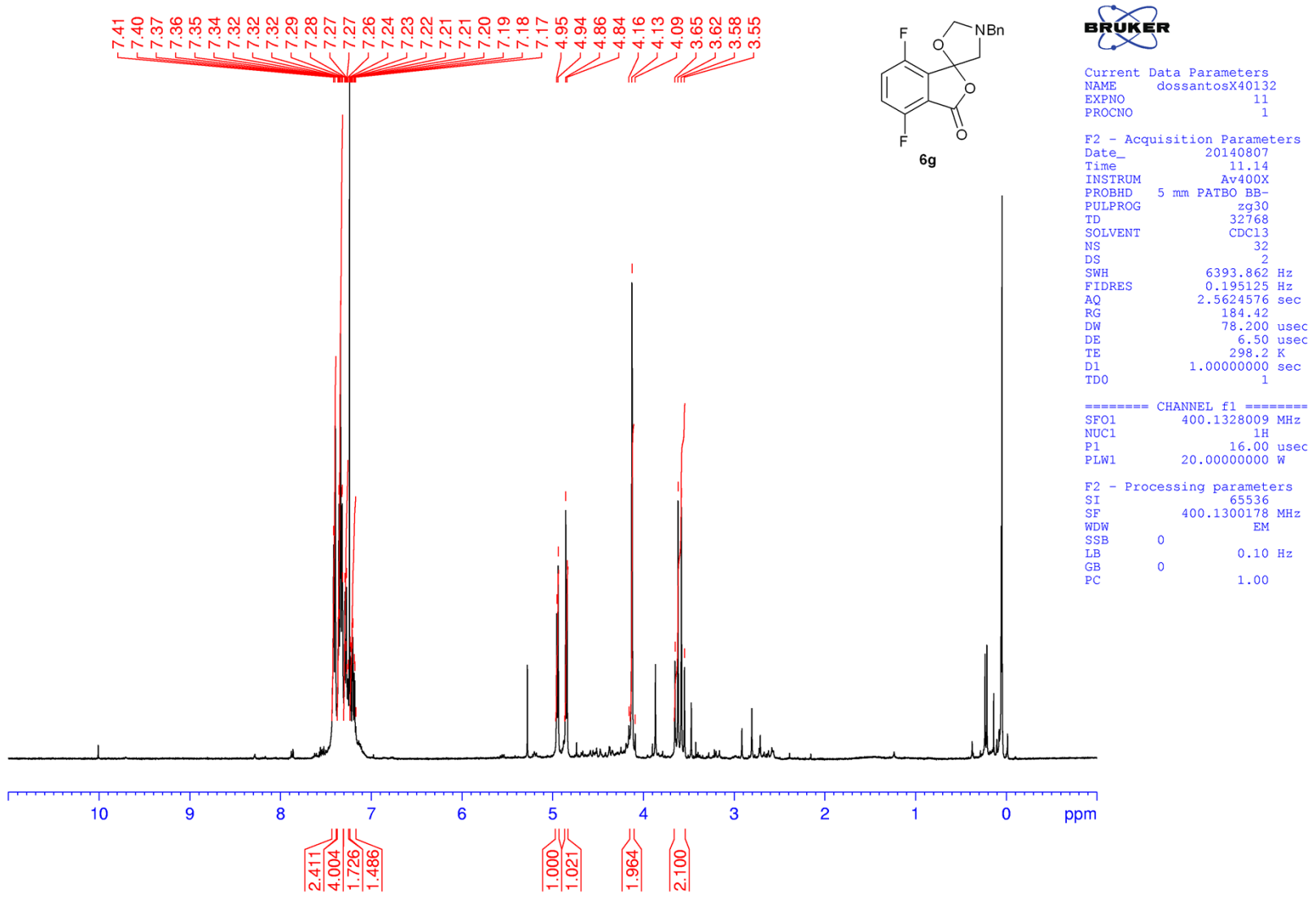
F2 - Acquisition Parameters
Date_            20140710
Time                19.55
INSTRUM           Av400X
PROBHD   5 mm PATBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1200
DS                     4
SWH          26041.666 Hz
FIDRES        0.397364 Hz
AQ            1.2582912 sec
RG               184.42
DW               19.200 usec
DE                6.50 usec
TE                298.1 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        100.6248425 MHz
NUC1               13C
P1               10.00 usec
PLW1        67.00000000 W

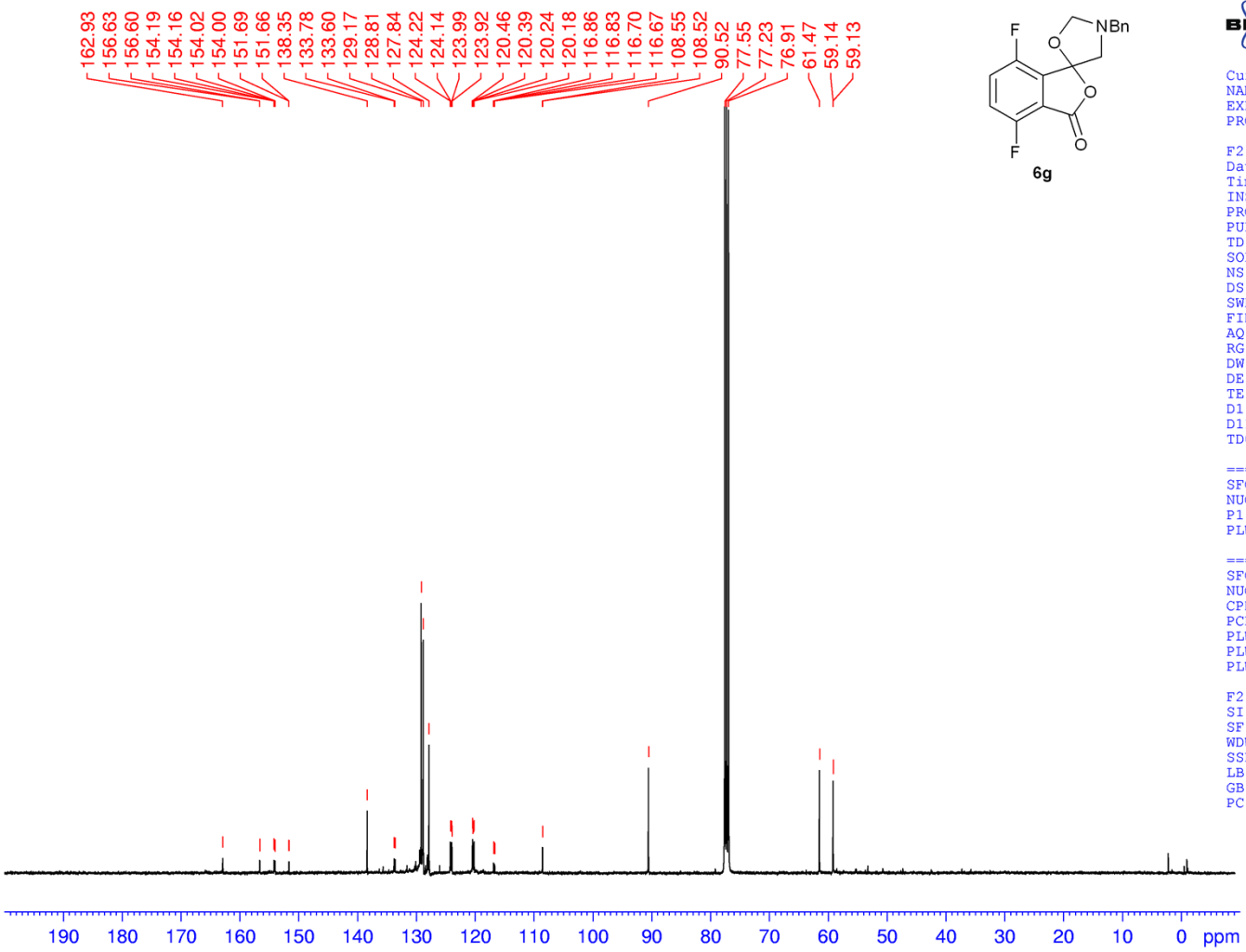
======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2 bi_waltz65_256
PCPD2            90.00 usec
PLW2        20.00000000 W
PLW12        0.63209999 W
PLW13        0.51200002 W

F2 - Processing parameters
SI                 32768
SF          100.6127497 MHz
WDW                EM
SSB       0
LB               1.00 Hz
GB        0
PC                 1.40
```

^{13}C NMR (100 MHz, $CDCl_3$) spectrum of cycloadducts **6fa** and **6fb**.



^{1}H NMR (400 MHz, $CDCl_3$) spectrum of cycloadduct **6g**.



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```
Current Data Parameters
NAME      dossantosX40136
EXPNO                  10
PROCNO                  1

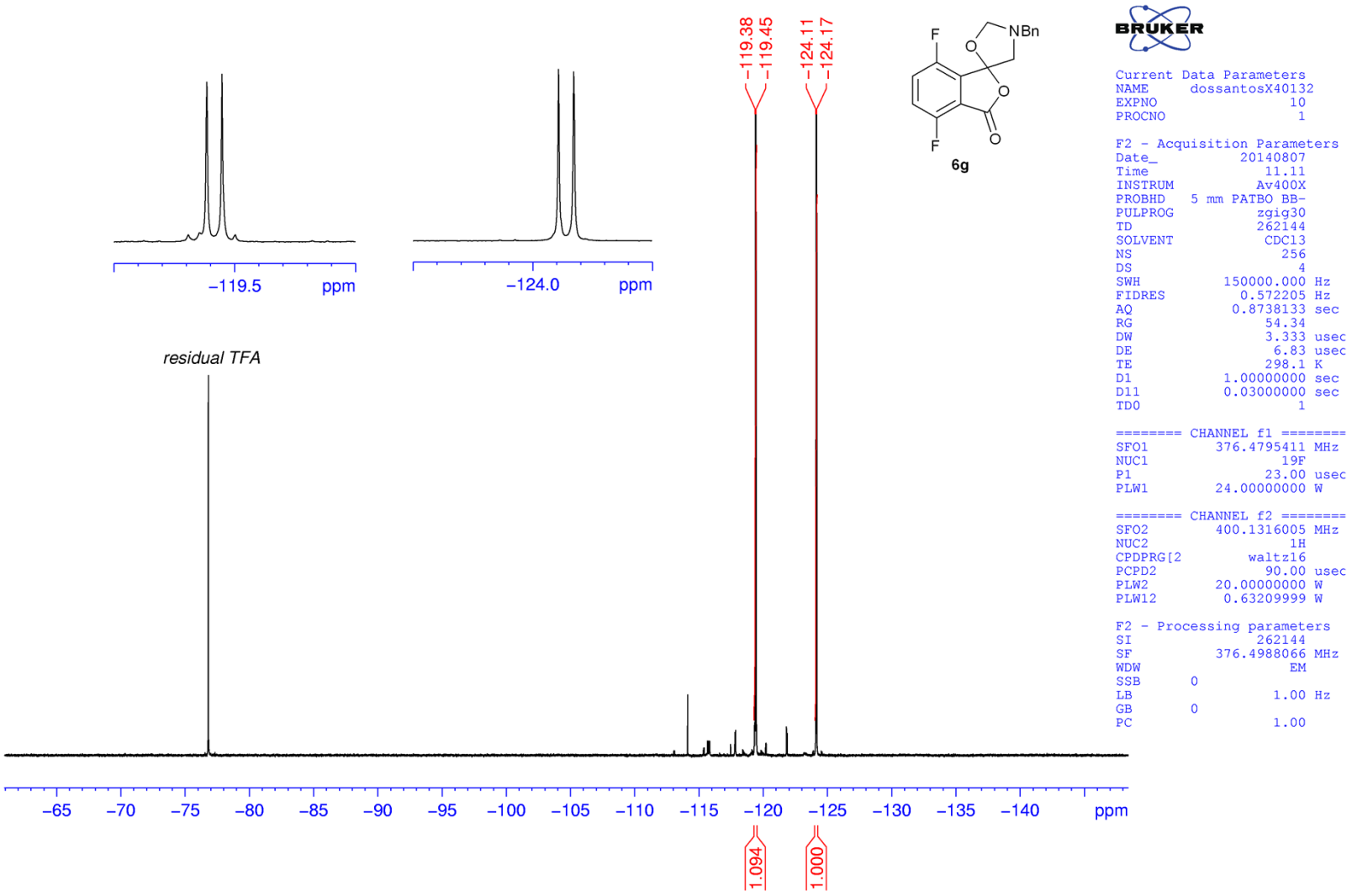
F2 - Acquisition Parameters
Date_            20140808
Time                7.16
INSTRUM           Av400X
PROBHD   5 mm PATBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                 14000
DS                     4
SWH            26041.666 Hz
FIDRES          0.397364 Hz
AQ             1.2582912 sec
RG                184.42
DW                19.200 usec
DE                 6.50 usec
TE                298.1 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6248425 MHz
NUC1                 13C
P1                 10.00 usec
PLW1         67.00000000 W

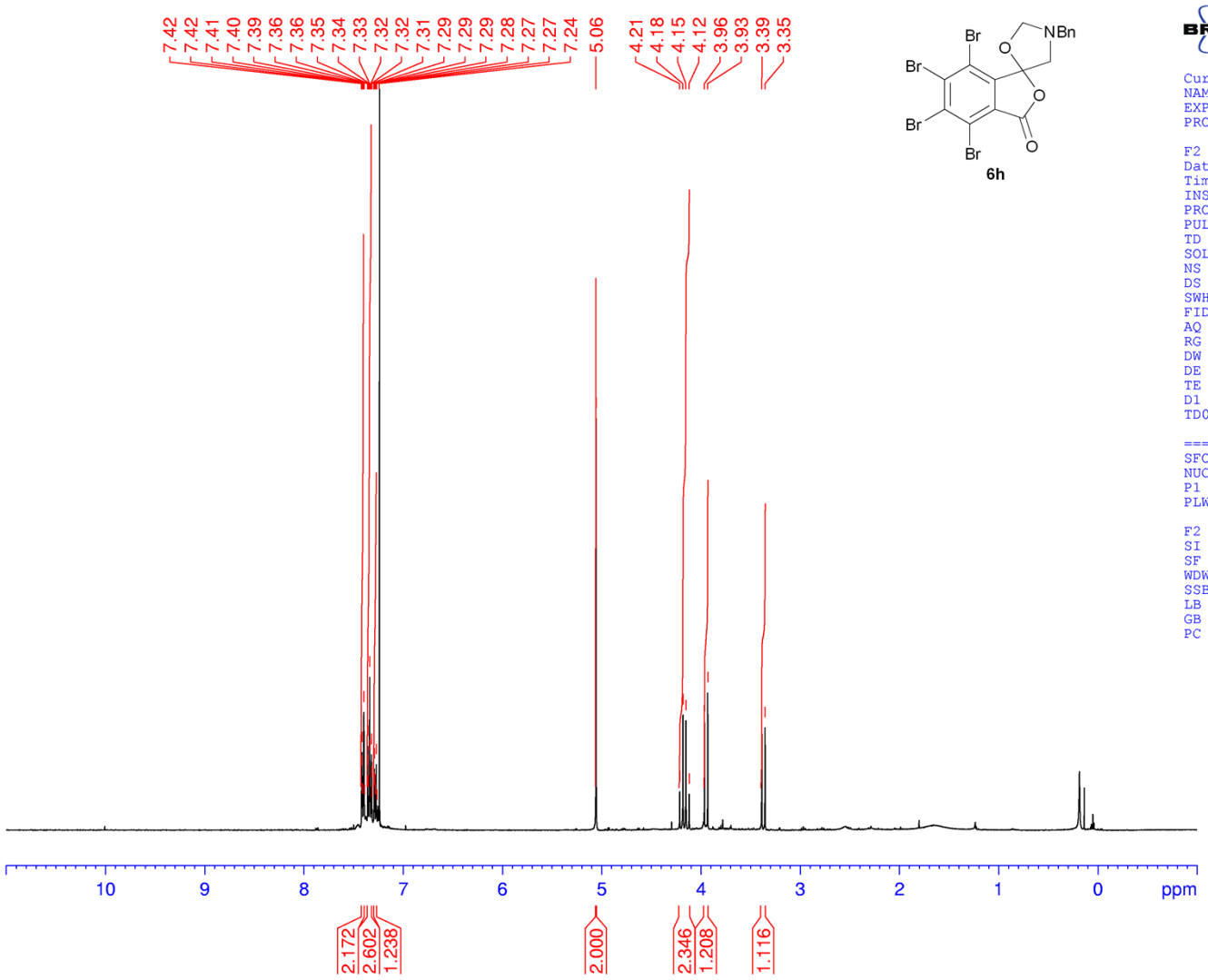
======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2 bi_waltz65_256
PCPD2              90.00 usec
PLW2         20.00000000 W
PLW12         0.63209999 W
PLW13         0.51200002 W

F2 - Processing parameters
SI                 32768
SF           100.6127493 MHz
WDW                 EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{13}C NMR (100 MHz, $CDCl_3$) spectrum of cycloadduct **6g**.



^{19}F NMR (376 MHz, $CDCl_3$) spectrum of cycloadduct **6g**.



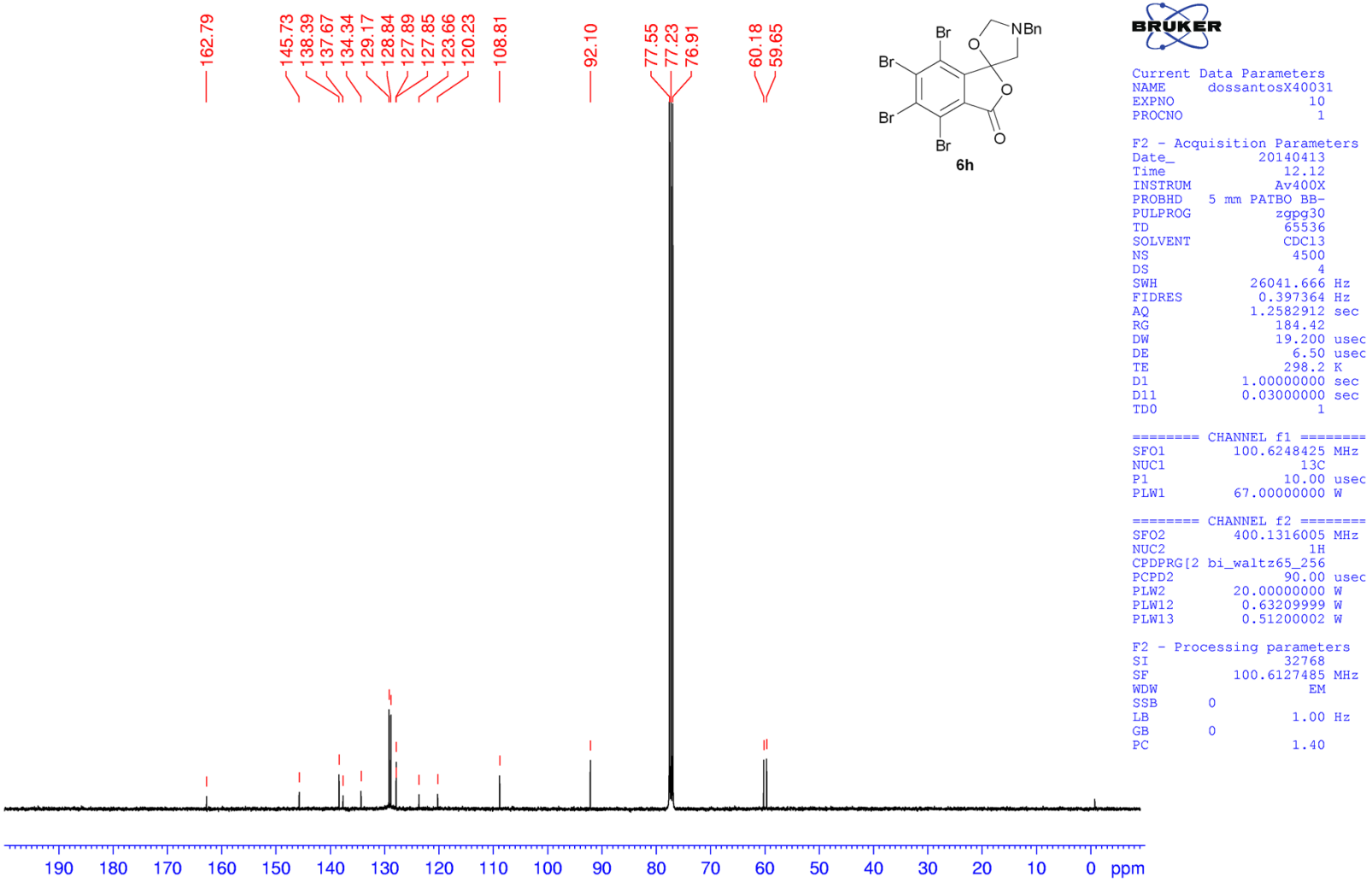

```
Current Data Parameters
NAME      dossantosX40031
EXPNO                  11
PROCNO                  1

F2 - Acquisition Parameters
Date_            20140413
Time                12.15
INSTRUM           Av400X
PROBHD    5 mm PATBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     2
SWH             6393.862 Hz
FIDRES          0.195125 Hz
AQ             2.5624576 sec
RG                184.42
DW                78.200 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec
TD0                    1

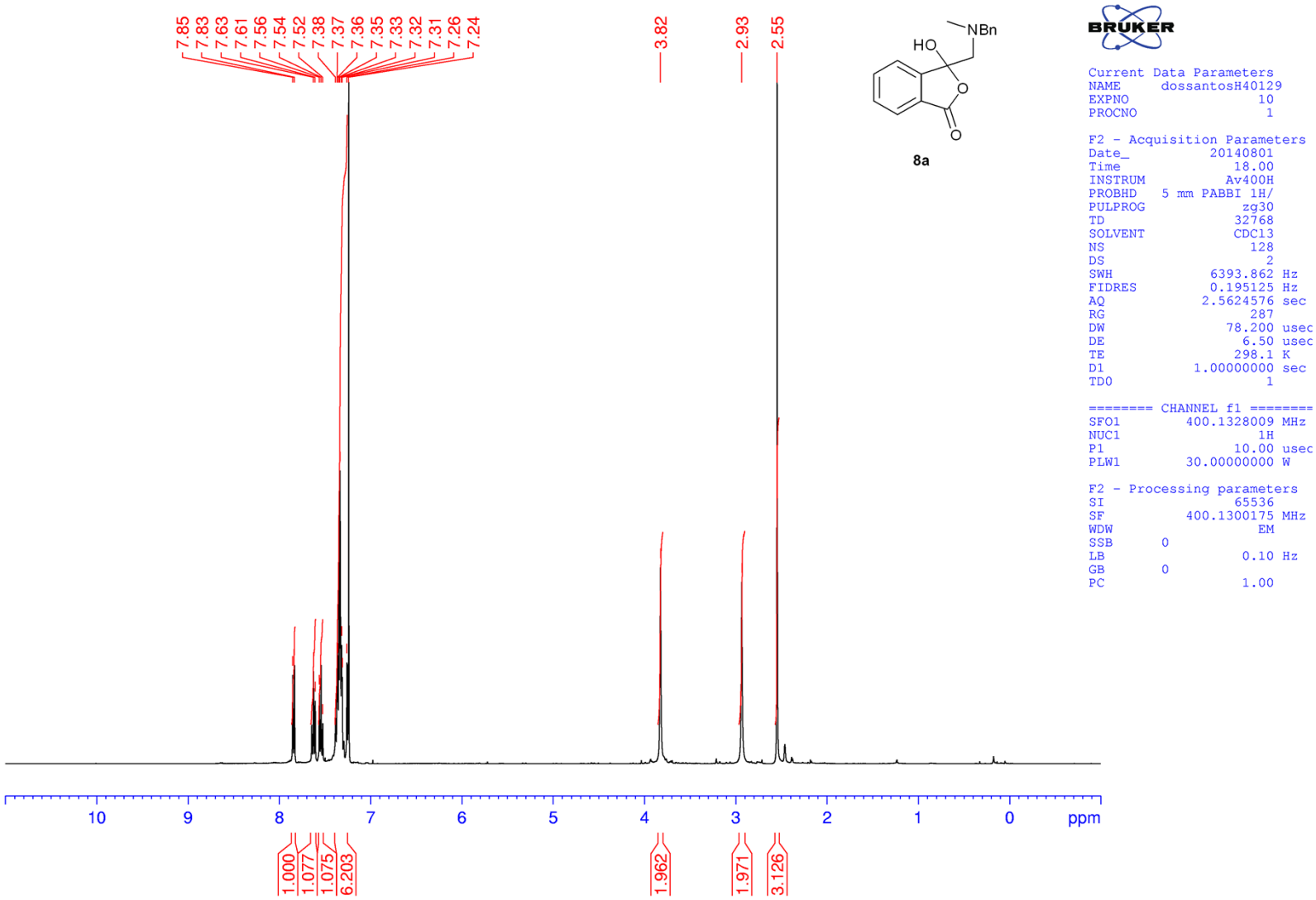
======== CHANNEL f1 ========
SFO1         400.1328009 MHz
NUC1                   1H
P1                 16.00 usec
PLW1        20.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300179 MHz
WDW                   EM
SSB       0
LB                 0.10 Hz
GB        0
PC                 1.00
```

^{1}H NMR (400 MHz, $CDCl_3$) spectrum of cycloadduct **6h**.



^{13}C NMR (100 MHz, $CDCl_3$) spectrum of cycloadduct **6h**.



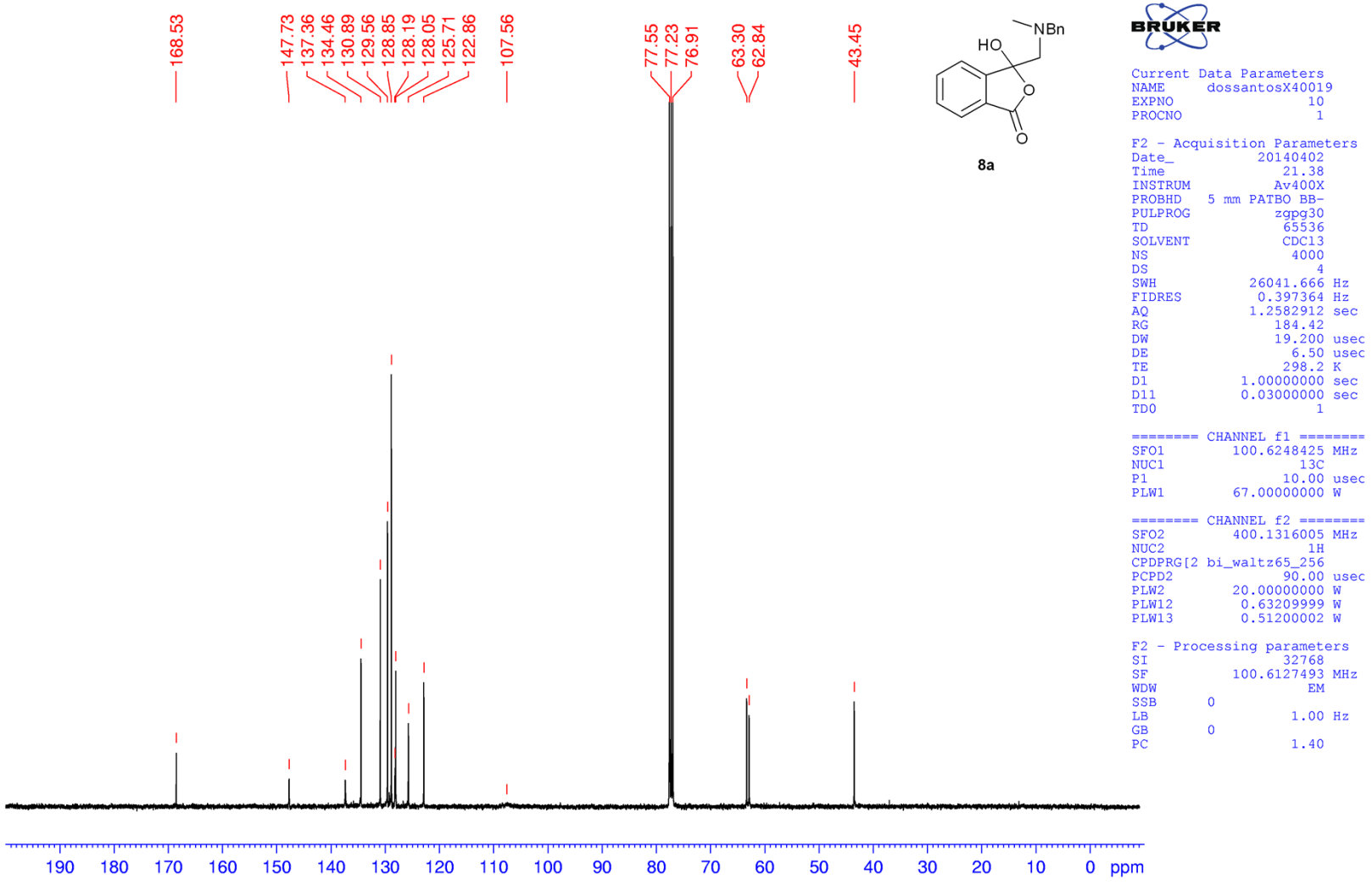

```
Current Data Parameters
NAME      dossantosH40129
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20140801
Time               18.00
INSTRUM          Av400H
PROBHD   5 mm PABBI 1H/
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                  128
DS                    2
SWH            6393.862 Hz
FIDRES         0.195125 Hz
AQ            2.5624576 sec
RG                  287
DW               78.200 usec
DE                6.50 usec
TE               298.1 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1328009 MHz
NUC1                1H
P1                10.00 usec
PLW1         30.00000000 W

F2 - Processing parameters
SI                65536
SF           400.1300175 MHz
WDW                 EM
SSB       0
LB                0.10 Hz
GB        0
PC                1.00
```

^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **8a**.



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```
Current Data Parameters
NAME      dossantosX40019
EXPNO                  10
PROCNO                  1

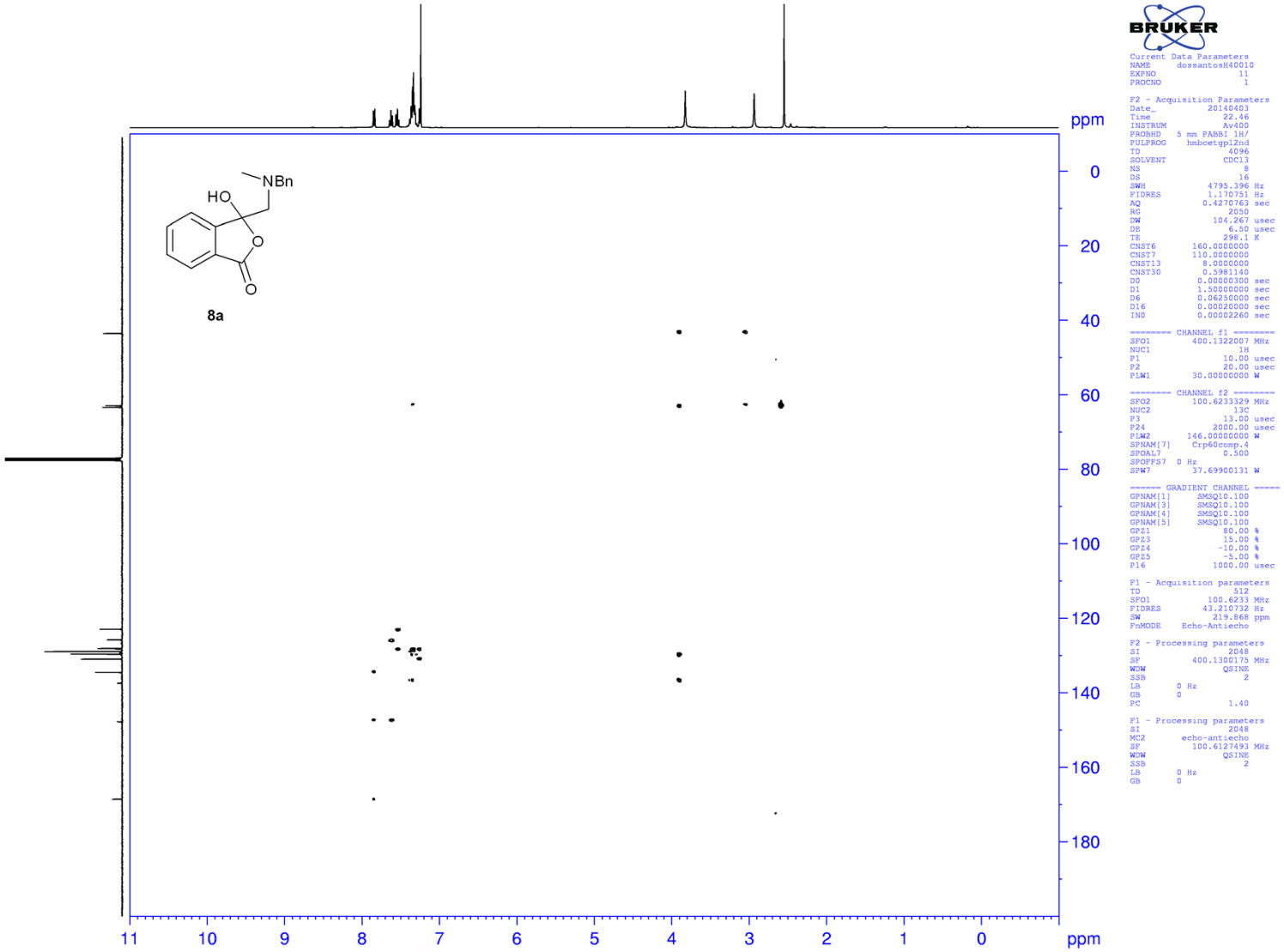
F2 - Acquisition Parameters
Date_           20140402
Time              21.38
INSTRUM          Av400X
PROBHD   5 mm PATBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 4000
DS                    4
SWH           26041.666 Hz
FIDRES         0.397364 Hz
AQ             1.2582912 sec
RG               184.42
DW               19.200 usec
DE                6.50 usec
TE                298.2 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6248425 MHz
NUC1               13C
P1               10.00 usec
PLW1        67.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2 bi_waltz65_256
PCPD2            90.00 usec
PLW2        20.00000000 W
PLW12        0.63209999 W
PLW13        0.51200002 W

F2 - Processing parameters
SI                32768
SF          100.6127493 MHz
WDW                EM
SSB       0
LB               1.00 Hz
GB        0
PC               1.40
```

^{13}C NMR (100 MHz, $CDCl_3$) spectrum of compound **8a**.



BRUKER

```
Current Data Parameters
NAME        dossantosH40010
EXPNO                    11
PROCNO                    1

F2 - Acquisition Parameters
Date_              20140403
Time                  22.46
INSTRUM                Av400
PROBHD   5 mm PABBI 1H/
PULPROG     hmbcetgpl2nd
TD                     4096
SOLVENT               CDCl3
NS                        8
DS                       16
SWH                4795.396 Hz
FIDRES             1.170751 Hz
AQ                 0.4270763 sec
RG                      2050
DW                  104.267 usec
DE                    6.50 usec
TE                   298.1 K
CNST6            160.0000000
CNST7            110.0000000
CNST13             8.0000000
CNST30             0.5981140
D0                 0.00000300 sec
D1                 1.50000000 sec
D6                 0.06250000 sec
D16                0.00020000 sec
IN0                0.00002260 sec

======== CHANNEL f1 ========
SFO1             400.1322007 MHz
NUC1                       1H
P1                      10.00 usec
P2                      20.00 usec
PLW1             30.00000000 W

======== CHANNEL f2 ========
SFO2             100.6233329 MHz
NUC2                       13C
P3                      13.00 usec
P24                   2000.00 usec
PLW2            146.00000000 W
SPNAM[7]     Crp60comp.4
SPOAL7                   0.500
SPOFFS7  0 Hz
SPW7             37.69900131 W

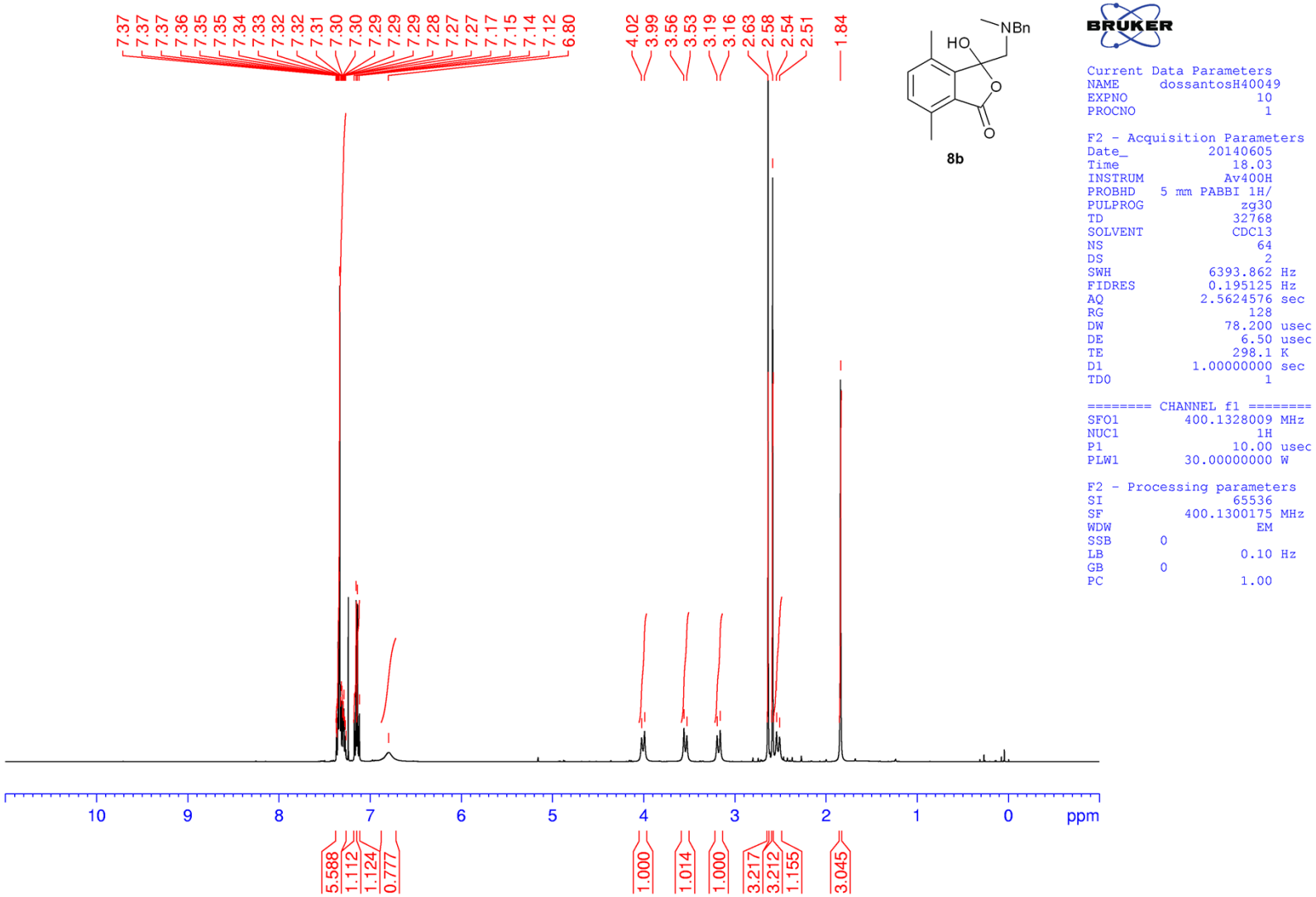
====== GRADIENT CHANNEL =====
GPNAM[1]       SMSQ10.100
GPNAM[3]       SMSQ10.100
GPNAM[4]       SMSQ10.100
GPNAM[5]       SMSQ10.100
GPZ1                 80.00 %
GPZ3                 15.00 %
GPZ4                -10.00 %
GPZ5                 -5.00 %
P16                1000.00 usec

F1 - Acquisition parameters
TD                       512
SFO1               100.6233 MHz
FIDRES           43.210732 Hz
SW                 219.868 ppm
FnMODE     Echo-Antiecho

F2 - Processing parameters
SI                      2048
SF               400.1300175 MHz
WDW                     QSINE
SSB                        2
LB         0 Hz
GB         0
PC                      1.40

F1 - Processing parameters
SI                      2048
MC2          echo-antiecho
SF               100.6127493 MHz
WDW                     QSINE
SSB                        2
LB         0 Hz
GB         0
```

HMBC contour plot of **8a**.



BRUKER

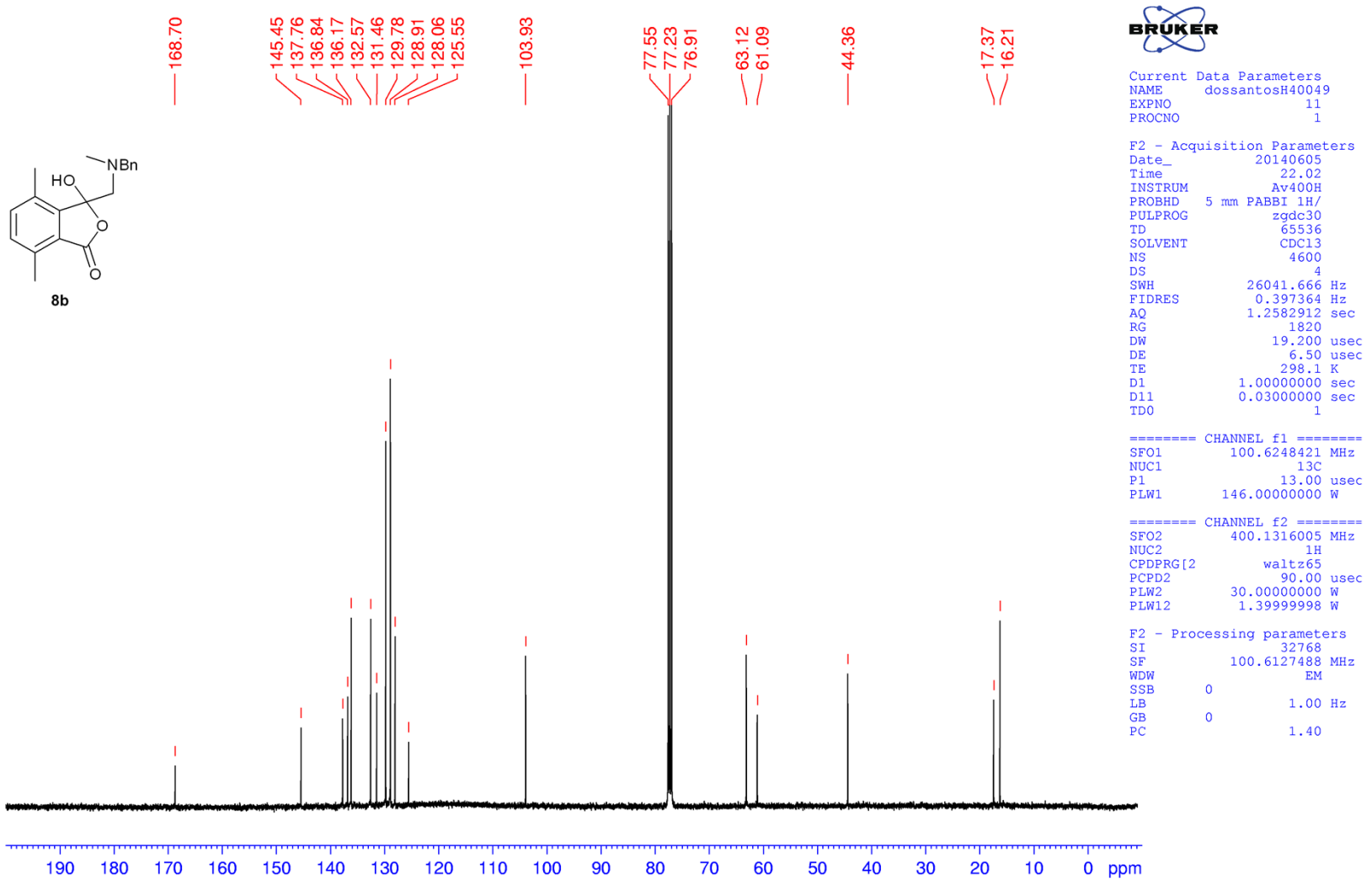
```
Current Data Parameters
NAME      dossantosH40049
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20140605
Time               18.03
INSTRUM          Av400H
PROBHD   5 mm PABBI 1H/
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   64
DS                    2
SWH             6393.862 Hz
FIDRES          0.195125 Hz
AQ              2.5624576 sec
RG                  128
DW                78.200 usec
DE                 6.50 usec
TE                298.1 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1328009 MHz
NUC1               1H
P1                10.00 usec
PLW1         30.00000000 W

F2 - Processing parameters
SI                65536
SF           400.1300175 MHz
WDW                EM
SSB       0
LB                0.10 Hz
GB        0
PC                1.00
```

^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **8b**.



BRUKER

```
Current Data Parameters
NAME      dossantosH40049
EXPNO                  11
PROCNO                  1

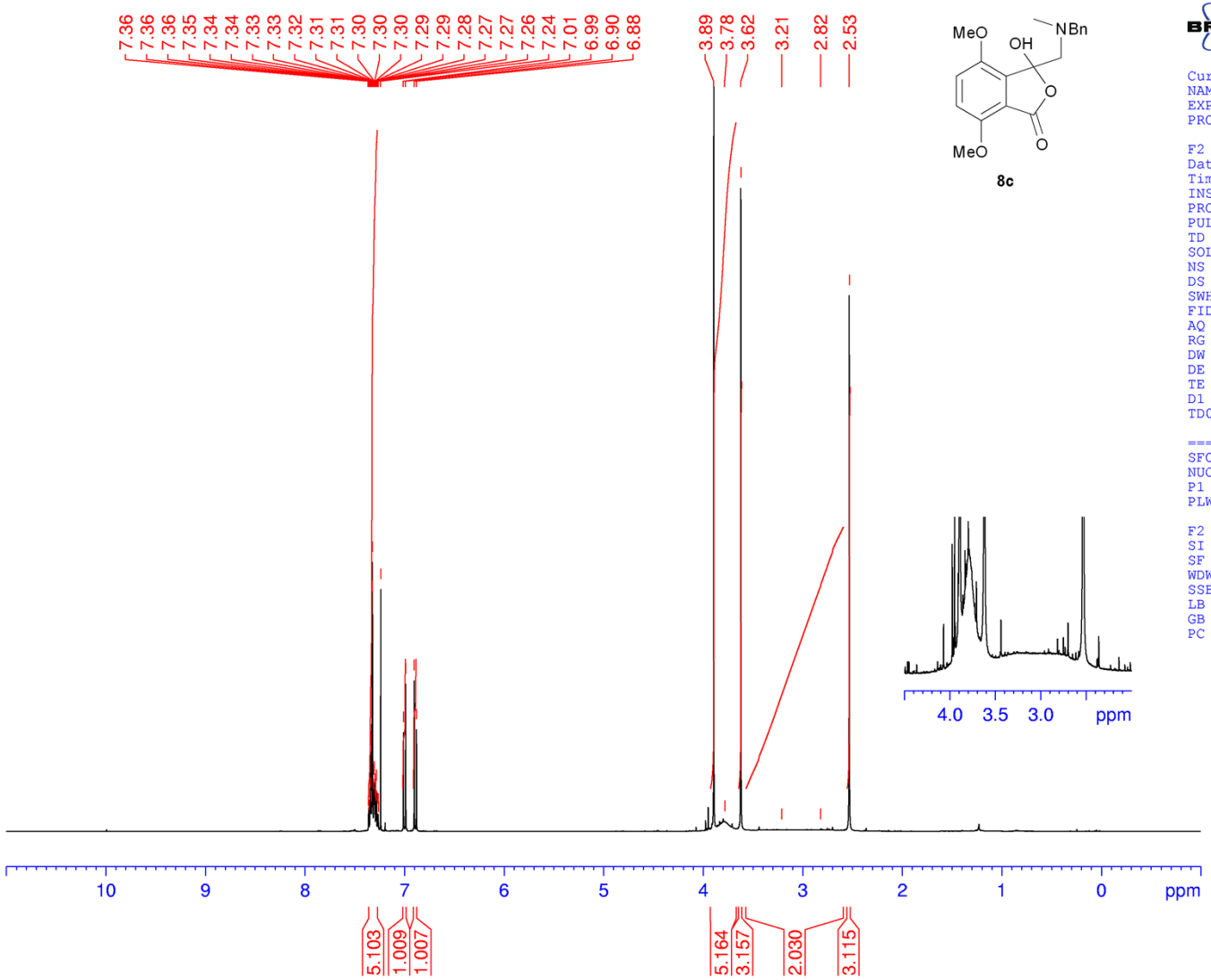
F2 - Acquisition Parameters
Date_           20140605
Time               22.02
INSTRUM          Av400H
PROBHD   5 mm PABBI 1H/
PULPROG          zgdc30
TD                65536
SOLVENT           CDCl3
NS                 4600
DS                    4
SWH           26041.666 Hz
FIDRES        0.397364 Hz
AQ            1.2582912 sec
RG                 1820
DW               19.200 usec
DE                6.50 usec
TE                298.1 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6248421 MHz
NUC1                  13C
P1                13.00 usec
PLW1       146.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                  1H
CPDPRG[2         waltz65
PCPD2             90.00 usec
PLW2         30.00000000 W
PLW12         1.39999998 W

F2 - Processing parameters
SI                 32768
SF           100.6127488 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

^{13}C NMR (100 MHz, $CDCl_3$) spectrum of compound **8b**.



8c

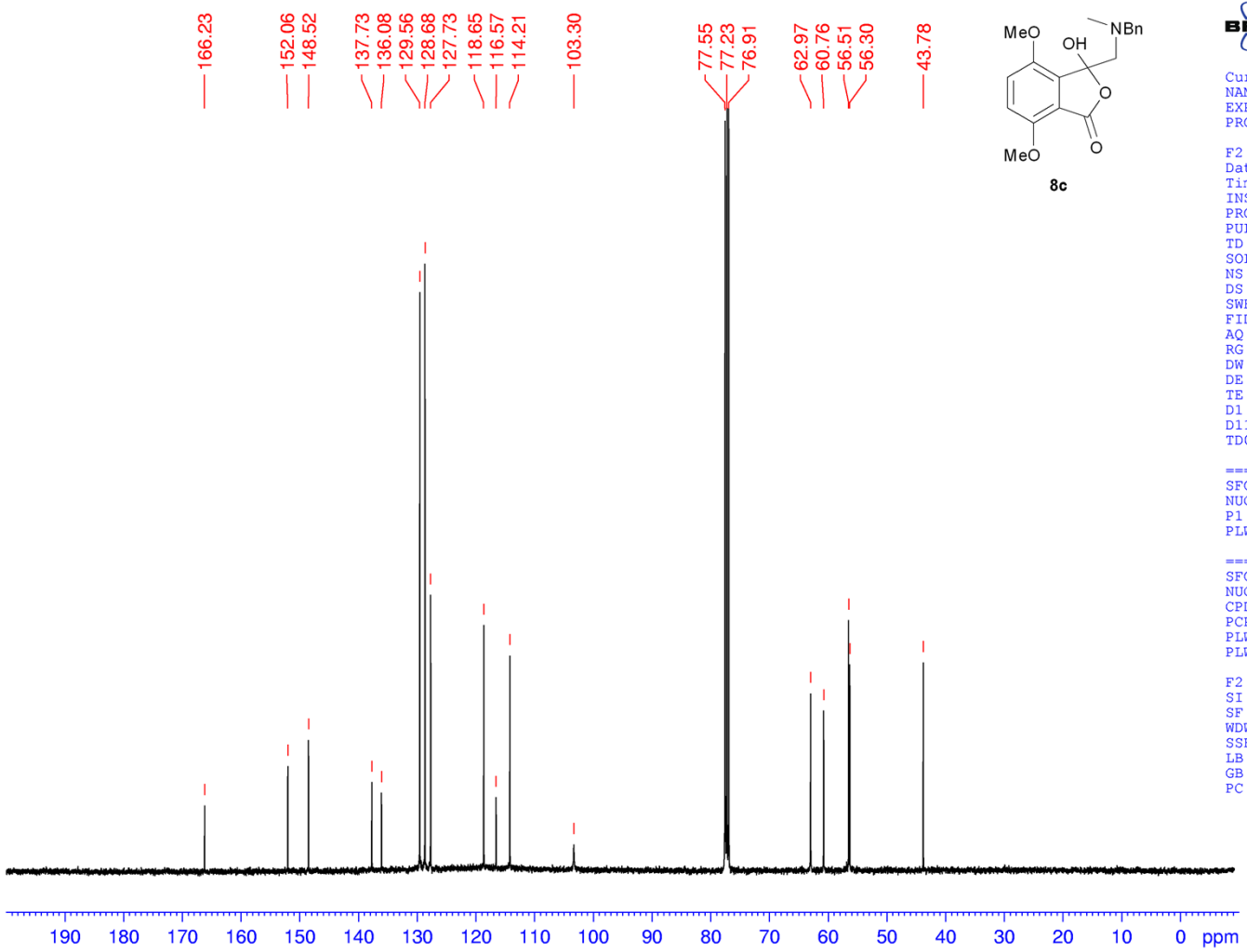

```
Current Data Parameters
NAME      4x019DosSantos
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140619
Time                8.05
INSTRUM          Av400X
PROBHD   5 mm PATBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     2
SWH             6393.862 Hz
FIDRES          0.195125 Hz
AQ             2.5624576 sec
RG                119.37
DW                78.200 usec
DE                 6.50 usec
TE                298.1 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1328009 MHz
NUC1                   1H
P1                 16.00 usec
PLW1         20.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300177 MHz
WDW                   EM
SSB       0
LB                 0.10 Hz
GB        0
PC                 1.00
```

^{1}H NMR (400 MHz, $CDCl_3$,) spectrum of compound **8c**.



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```
Current Data Parameters
NAME      dossantosH40062
EXPNO                  11
PROCNO                  1

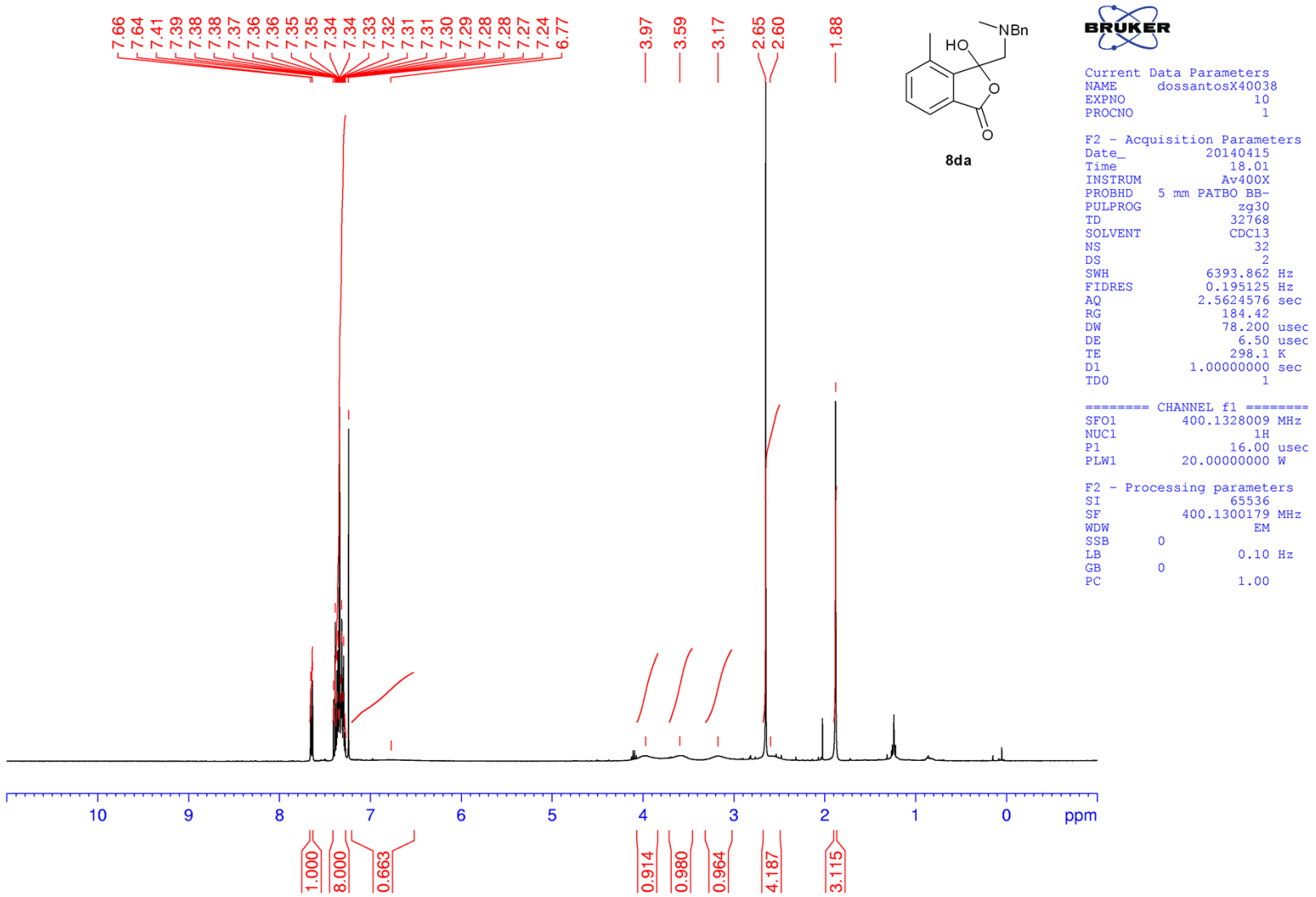
F2 - Acquisition Parameters
Date_            20140611
Time                22.02
INSTRUM           Av400H
PROBHD   5 mm PABBI 1H/
PULPROG          zgdc30
TD                 65536
SOLVENT            CDCl3
NS                  4600
DS                     4
SWH            26041.666 Hz
FIDRES          0.397364 Hz
AQ             1.2582912 sec
RG                  1820
DW                19.200 usec
DE                 6.50 usec
TE                298.1 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6248421 MHz
NUC1                  13C
P1                  13.00 usec
PLW1        146.00000000 W

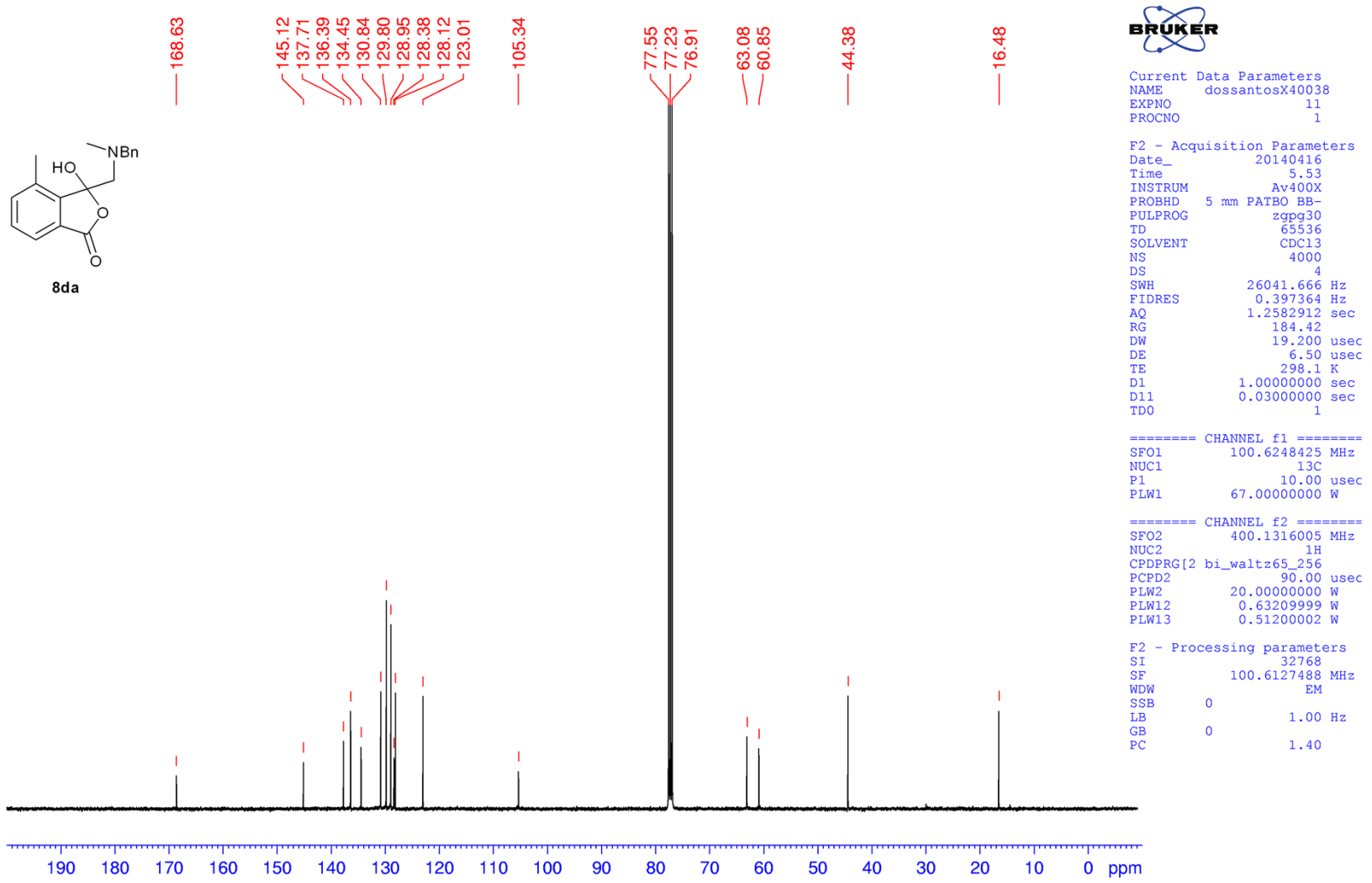
======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz65
PCPD2               90.00 usec
PLW2         30.00000000 W
PLW12         1.39999998 W

F2 - Processing parameters
SI                 32768
SF           100.6127512 MHz
WDW                   EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

^{13}C NMR (100 MHz, $CDCl_3$) spectrum of compound **8c**.



^{1}H NMR (400 MHz, $CDCl_3$,) spectrum of compound **8da**.



BRUKER

```
Current Data Parameters
NAME      dossantosX40038
EXPNO                  11
PROCNO                  1

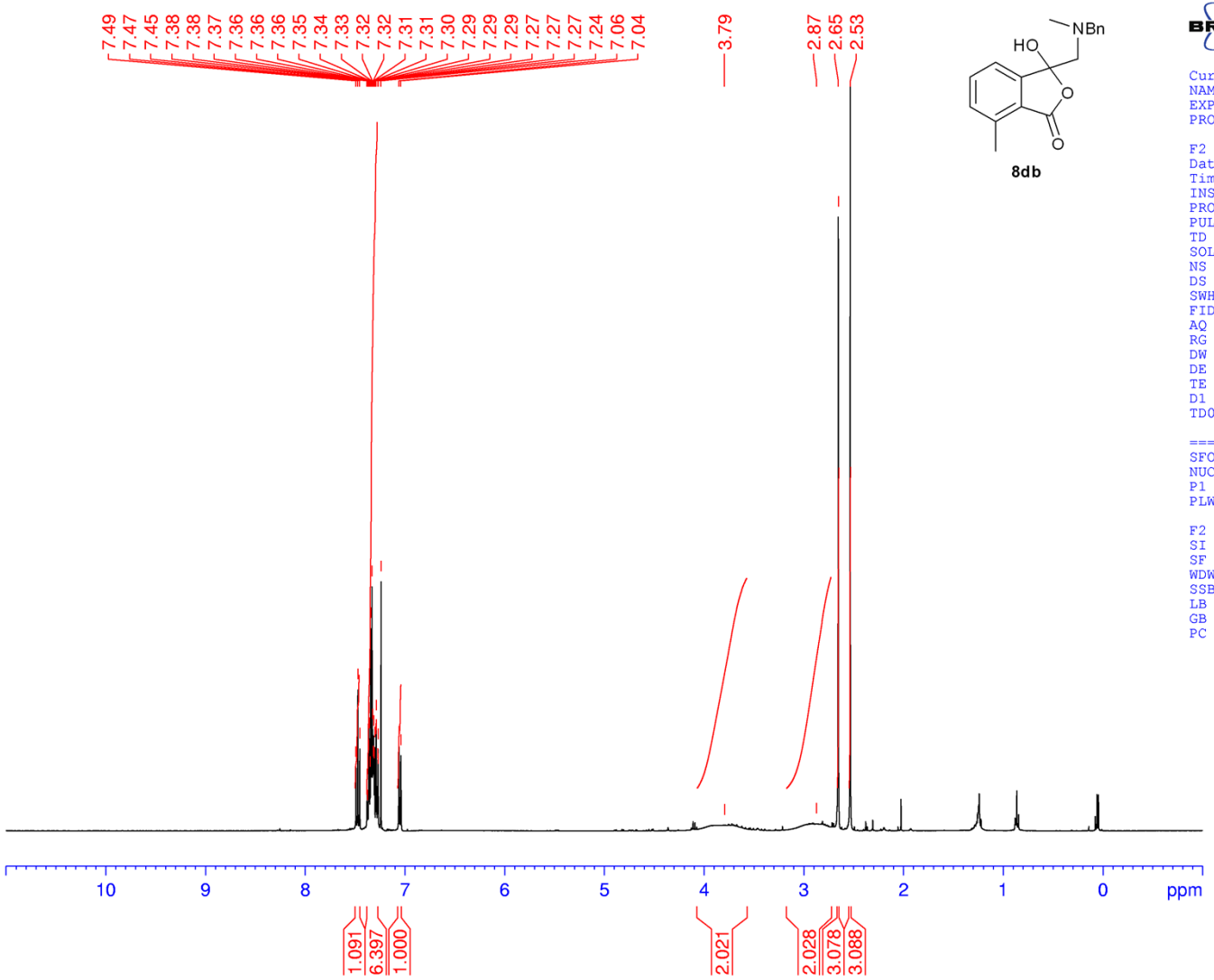
F2 - Acquisition Parameters
Date_           20140416
Time                5.53
INSTRUM          Av400X
PROBHD   5 mm PATBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 4000
DS                    4
SWH           26041.666 Hz
FIDRES        0.397364 Hz
AQ            1.2582912 sec
RG               184.42
DW               19.200 usec
DE                6.50 usec
TE                298.1 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6248425 MHz
NUC1                  13C
P1                10.00 usec
PLW1         67.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                  1H
CPDPRG[2 bi_waltz65_256
PCPD2             90.00 usec
PLW2         20.00000000 W
PLW12         0.63209999 W
PLW13         0.51200002 W

F2 - Processing parameters
SI                 32768
SF           100.6127488 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

^{13}C NMR (100 MHz, $CDCl_3$) spectrum of compound **8da**.



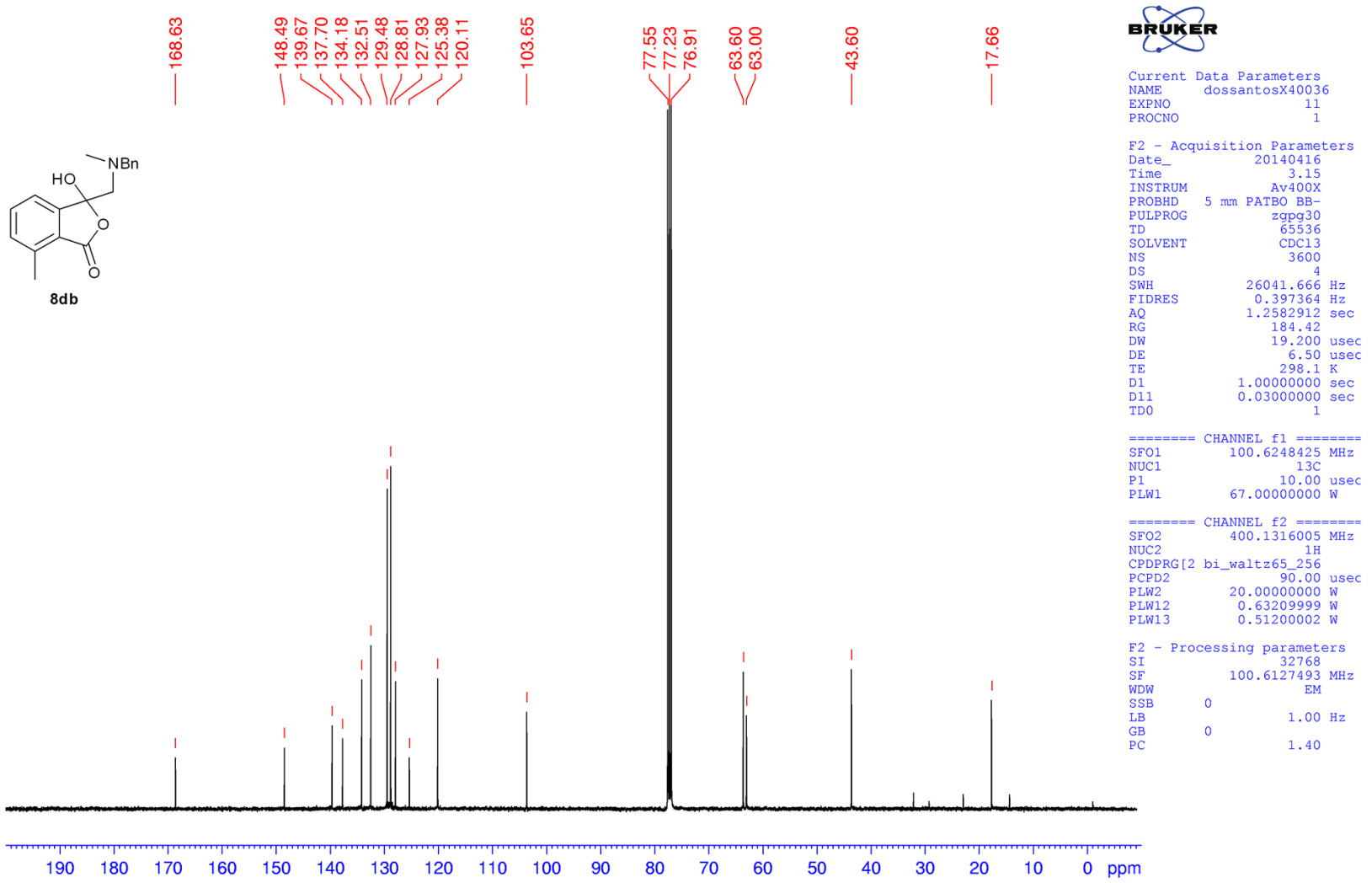

```
Current Data Parameters
NAME      dossantosX40036
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20140415
Time                17.41
INSTRUM           Av400X
PROBHD   5 mm PATBO BB-
PULPROG              zg30
TD                  32768
SOLVENT              CDCl3
NS                      32
DS                       2
SWH              6393.862 Hz
FIDRES           0.195125 Hz
AQ             2.5624576 sec
RG                 174.65
DW                 78.200 usec
DE                   6.50 usec
TE                  298.2 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         400.1328009 MHz
NUC1                   1H
P1                  16.00 usec
PLW1         20.00000000 W

F2 - Processing parameters
SI                   65536
SF           400.1300179 MHz
WDW                    EM
SSB       0
LB                  0.10 Hz
GB        0
PC                  1.00
```

^{1}H NMR (400 MHz, $CDCl_3$,) spectrum of compound **8db**.



BRUKER

```
Current Data Parameters
NAME      dossantosX40036
EXPNO                  11
PROCNO                  1

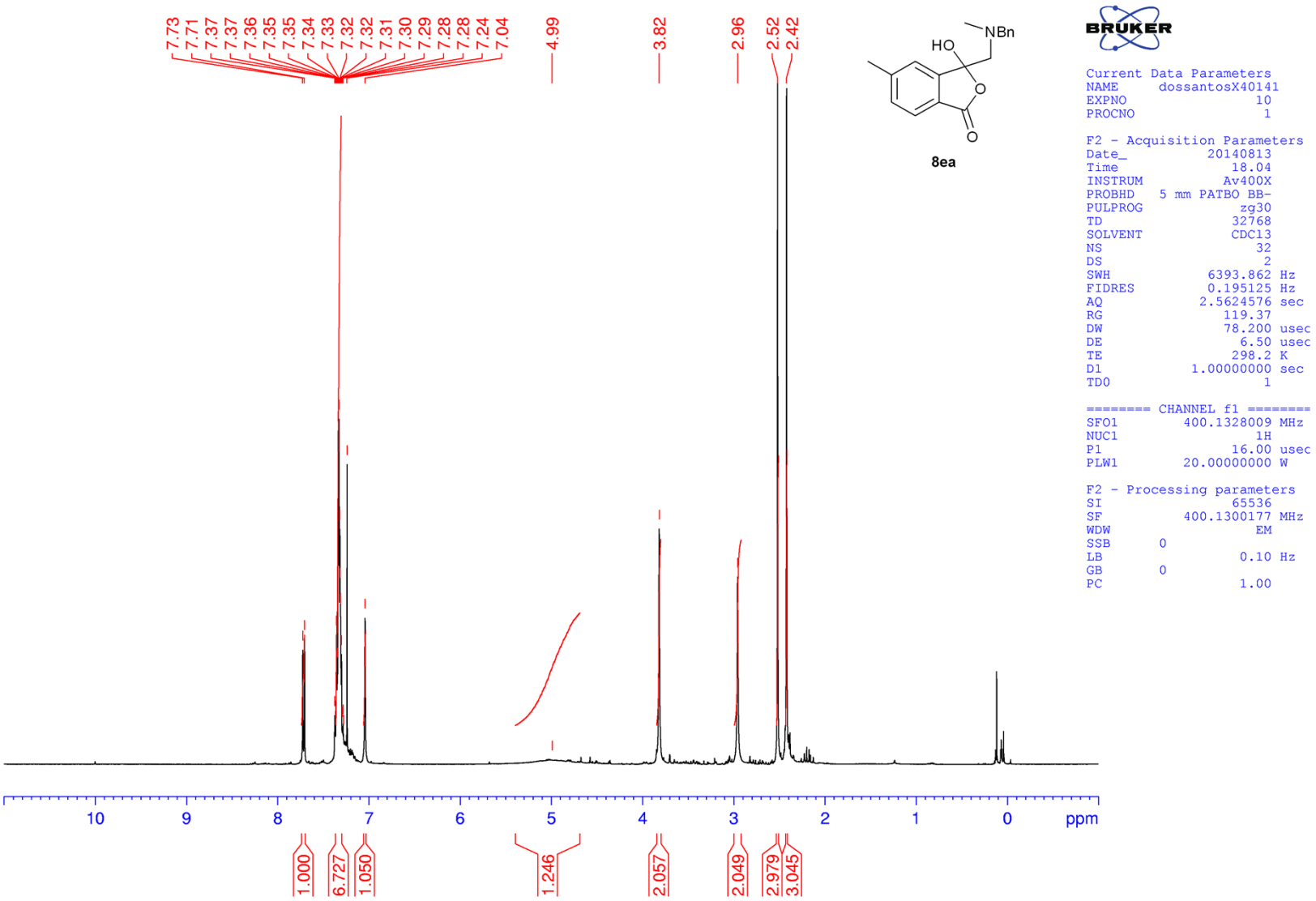
F2 - Acquisition Parameters
Date_           20140416
Time                3.15
INSTRUM          Av400X
PROBHD   5 mm PATBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3600
DS                    4
SWH           26041.666 Hz
FIDRES         0.397364 Hz
AQ            1.2582912 sec
RG               184.42
DW               19.200 usec
DE                6.50 usec
TE                298.1 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6248425 MHz
NUC1                  13C
P1                  10.00 usec
PLW1         67.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                  1H
CPDPRG[2 bi_waltz65_256
PCPD2               90.00 usec
PLW2         20.00000000 W
PLW12         0.63209999 W
PLW13         0.51200002 W

F2 - Processing parameters
SI                 32768
SF           100.6127493 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{13}C NMR (100 MHz, $CDCl_3$) spectrum of compound **8db**.



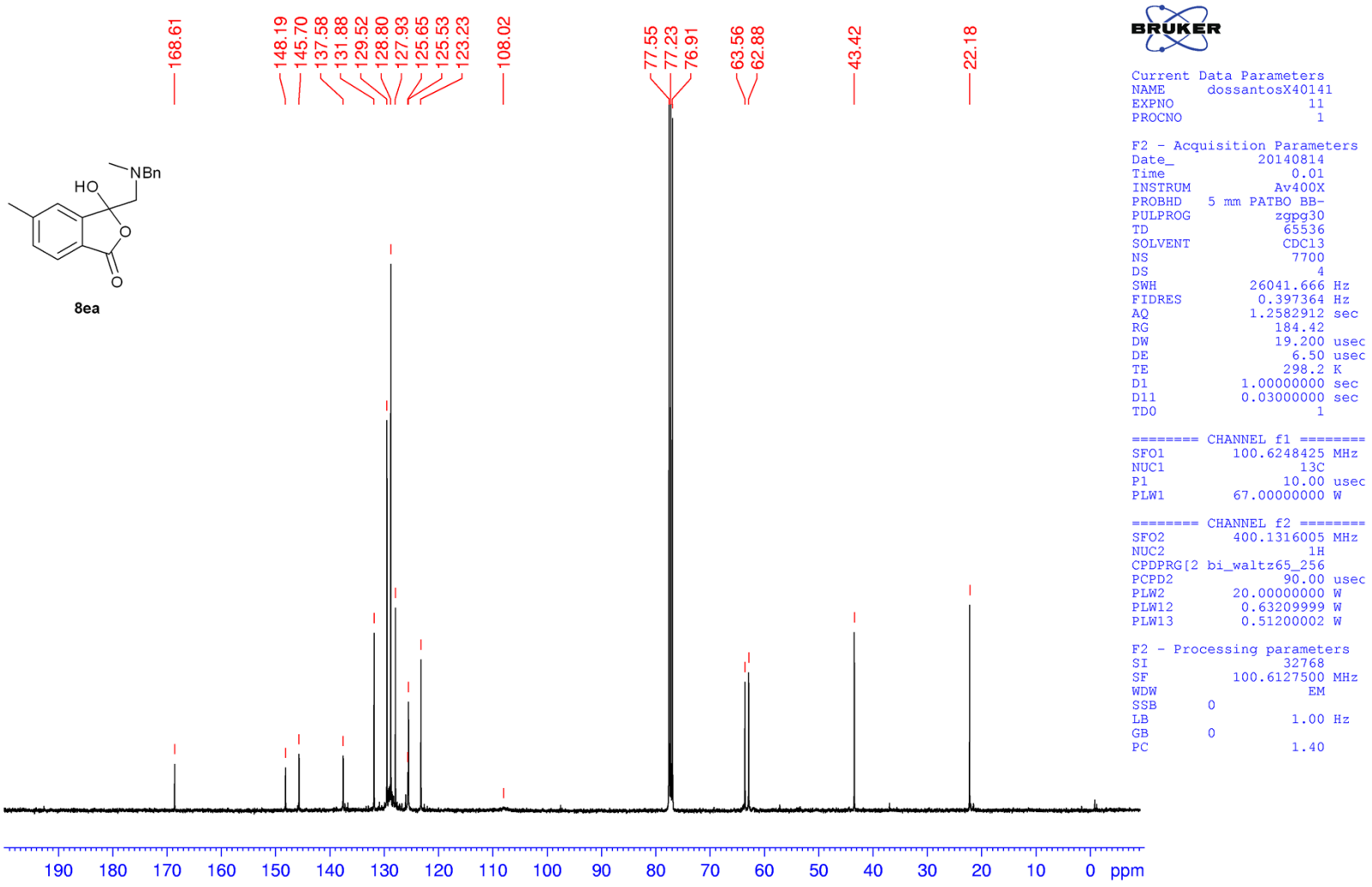

```
Current Data Parameters
NAME      dossantosX40141
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_           20140813
Time               18.04
INSTRUM          Av400X
PROBHD   5 mm PATBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     2
SWH             6393.862 Hz
FIDRES          0.195125 Hz
AQ              2.5624576 sec
RG                 119.37
DW                 78.200 usec
DE                  6.50 usec
TE                 298.2 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1328009 MHz
NUC1                   1H
P1                 16.00 usec
PLW1         20.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300177 MHz
WDW                   EM
SSB       0
LB                 0.10 Hz
GB        0
PC                 1.00
```

^{1}H NMR (400 MHz, $CDCl_3$,) spectrum of compound **8ea**.




```
Current Data Parameters
NAME      dossantosX40141
EXPNO                  11
PROCNO                  1

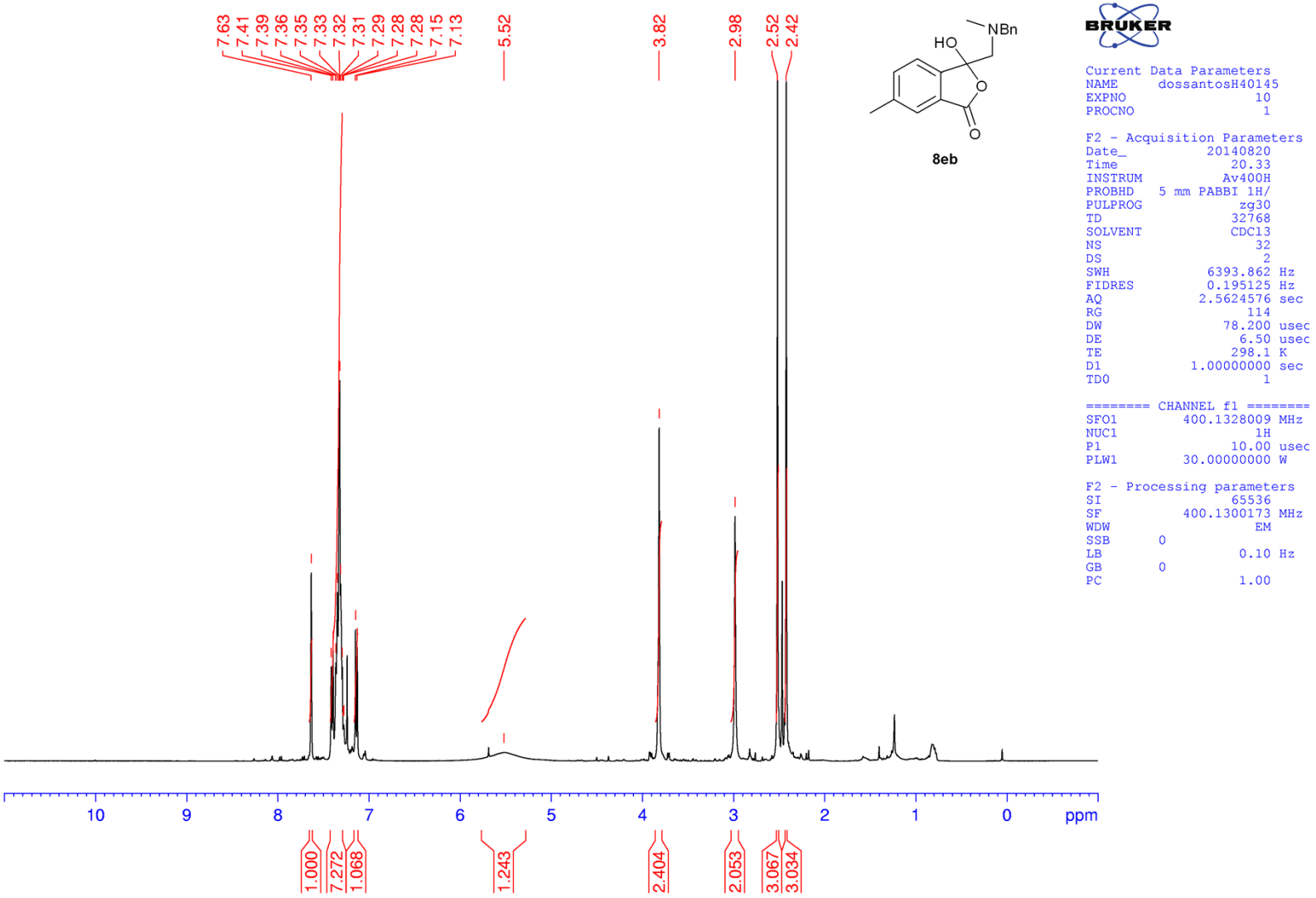
F2 - Acquisition Parameters
Date_            20140814
Time                 0.01
INSTRUM           Av400X
PROBHD   5 mm PATBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 7700
DS                    4
SWH           26041.666 Hz
FIDRES         0.397364 Hz
AQ            1.2582912 sec
RG               184.42
DW               19.200 usec
DE                6.50 usec
TE               298.2 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6248425 MHz
NUC1                  13C
P1                  10.00 usec
PLW1         67.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                  1H
CPDPRG[2 bi_waltz65_256
PCPD2                90.00 usec
PLW2         20.00000000 W
PLW12         0.63209999 W
PLW13         0.51200002 W

F2 - Processing parameters
SI                   32768
SF           100.6127500 MHz
WDW                     EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

^{13}C NMR (100 MHz, $CDCl_3$) spectrum of compound **8ea**.



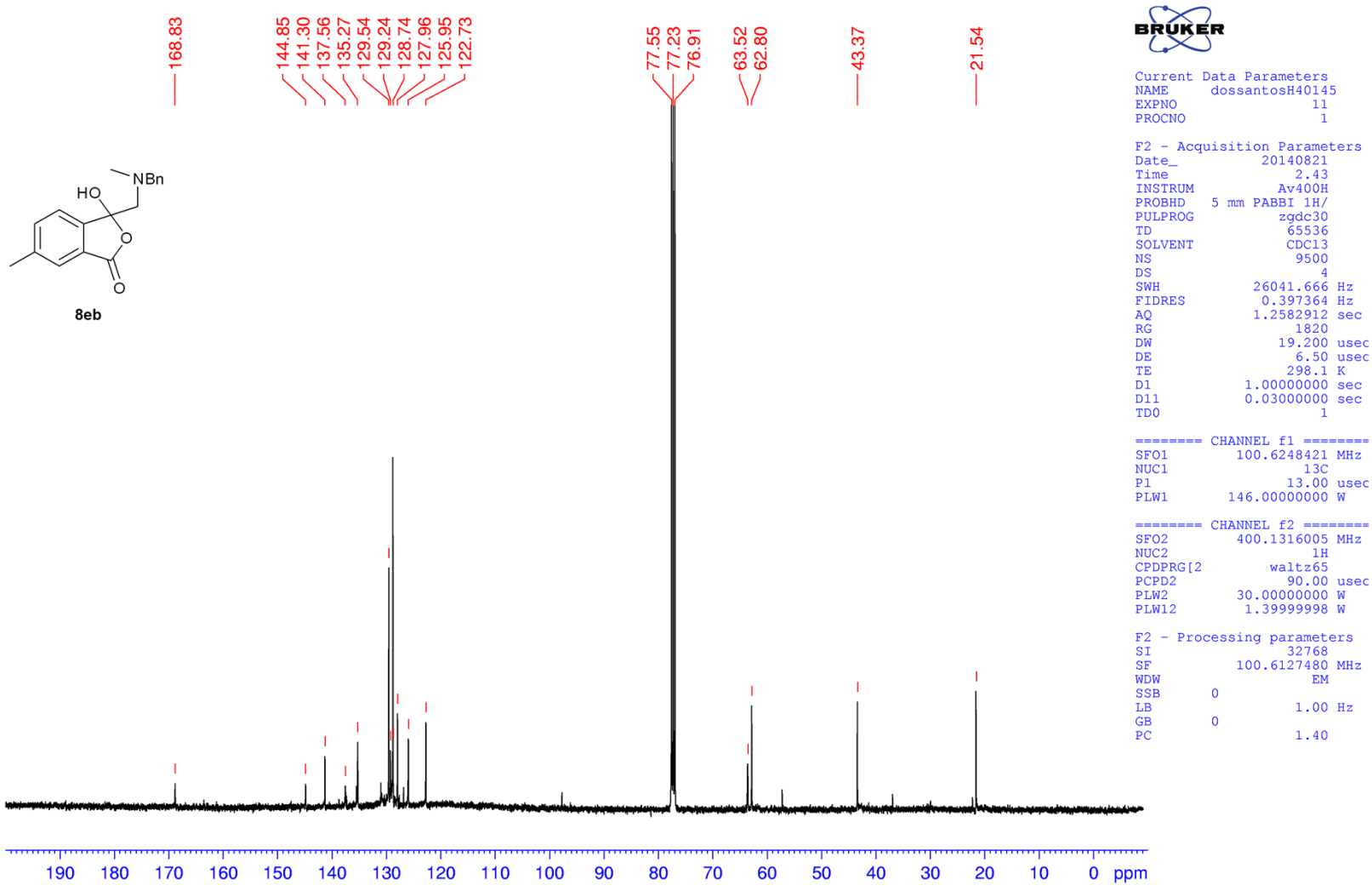

```
Current Data Parameters
NAME      dossantosH40145
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20140820
Time                20.33
INSTRUM           Av400H
PROBHD   5 mm PABBI 1H/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     2
SWH             6393.862 Hz
FIDRES          0.195125 Hz
AQ             2.5624576 sec
RG                   114
DW                78.200 usec
DE                 6.50 usec
TE                298.1 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1328009 MHz
NUC1                   1H
P1                 10.00 usec
PLW1         30.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300173 MHz
WDW                   EM
SSB       0
LB                 0.10 Hz
GB        0
PC                 1.00
```

^{1}H NMR (400 MHz, $CDCl_3$,) spectrum of compound **8eb**.




```
Current Data Parameters
NAME      dossantosH40145
EXPNO                  11
PROCNO                  1

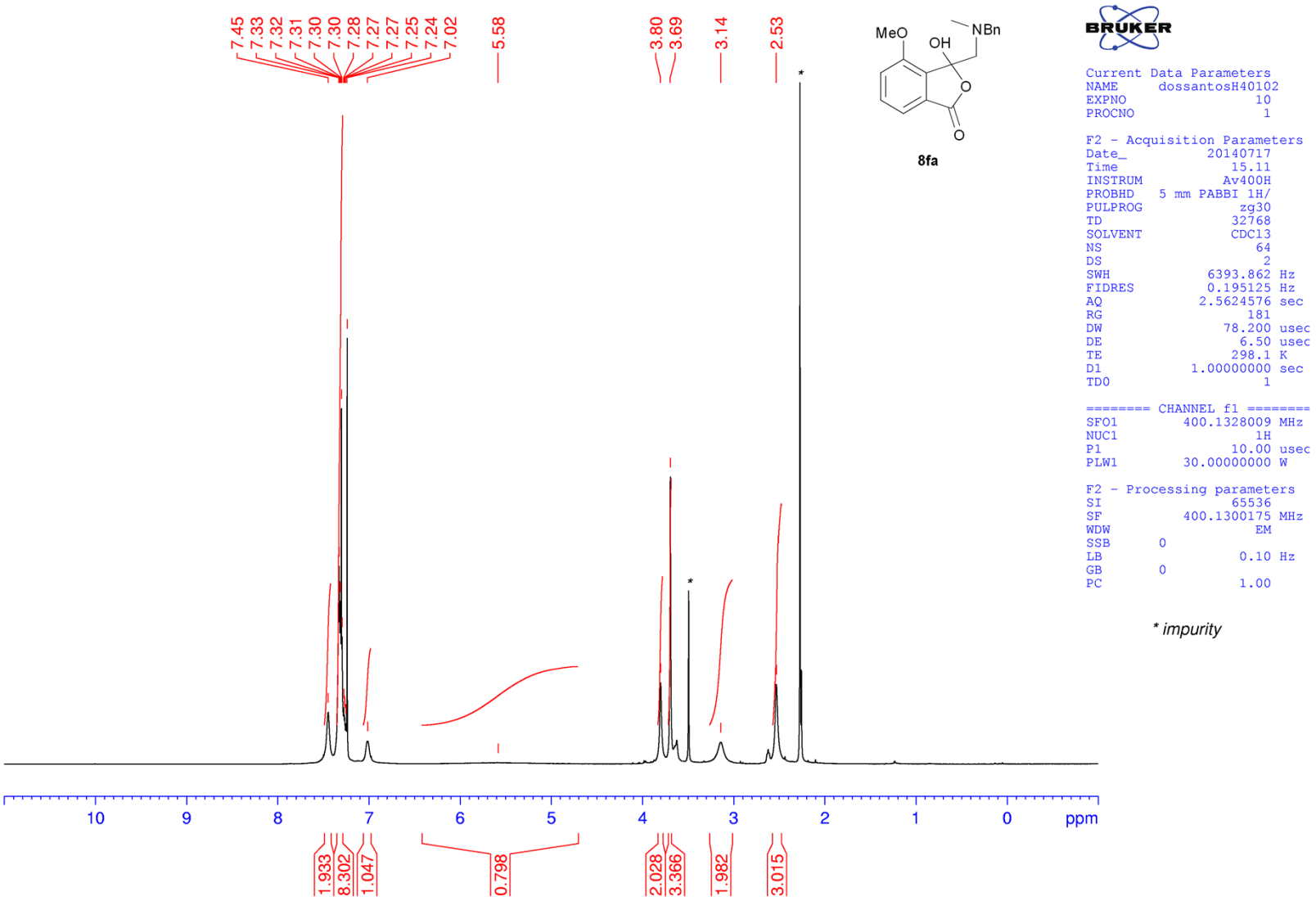
F2 - Acquisition Parameters
Date_           20140821
Time                2.43
INSTRUM          Av400H
PROBHD   5 mm PABBI 1H/
PULPROG          zgdc30
TD                65536
SOLVENT           CDCl3
NS                 9500
DS                    4
SWH           26041.666 Hz
FIDRES         0.397364 Hz
AQ             1.2582912 sec
RG                 1820
DW               19.200 usec
DE                6.50 usec
TE                298.1 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6248421 MHz
NUC1                13C
P1                13.00 usec
PLW1       146.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                 1H
CPDPRG[2         waltz65
PCPD2             90.00 usec
PLW2         30.00000000 W
PLW12         1.39999998 W

F2 - Processing parameters
SI                 32768
SF          100.6127480 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{13}C NMR (100 MHz, $CDCl_3$) spectrum of compound **8eb**.




```
Current Data Parameters
NAME      dossantosH40102
EXPNO                  10
PROCNO                  1

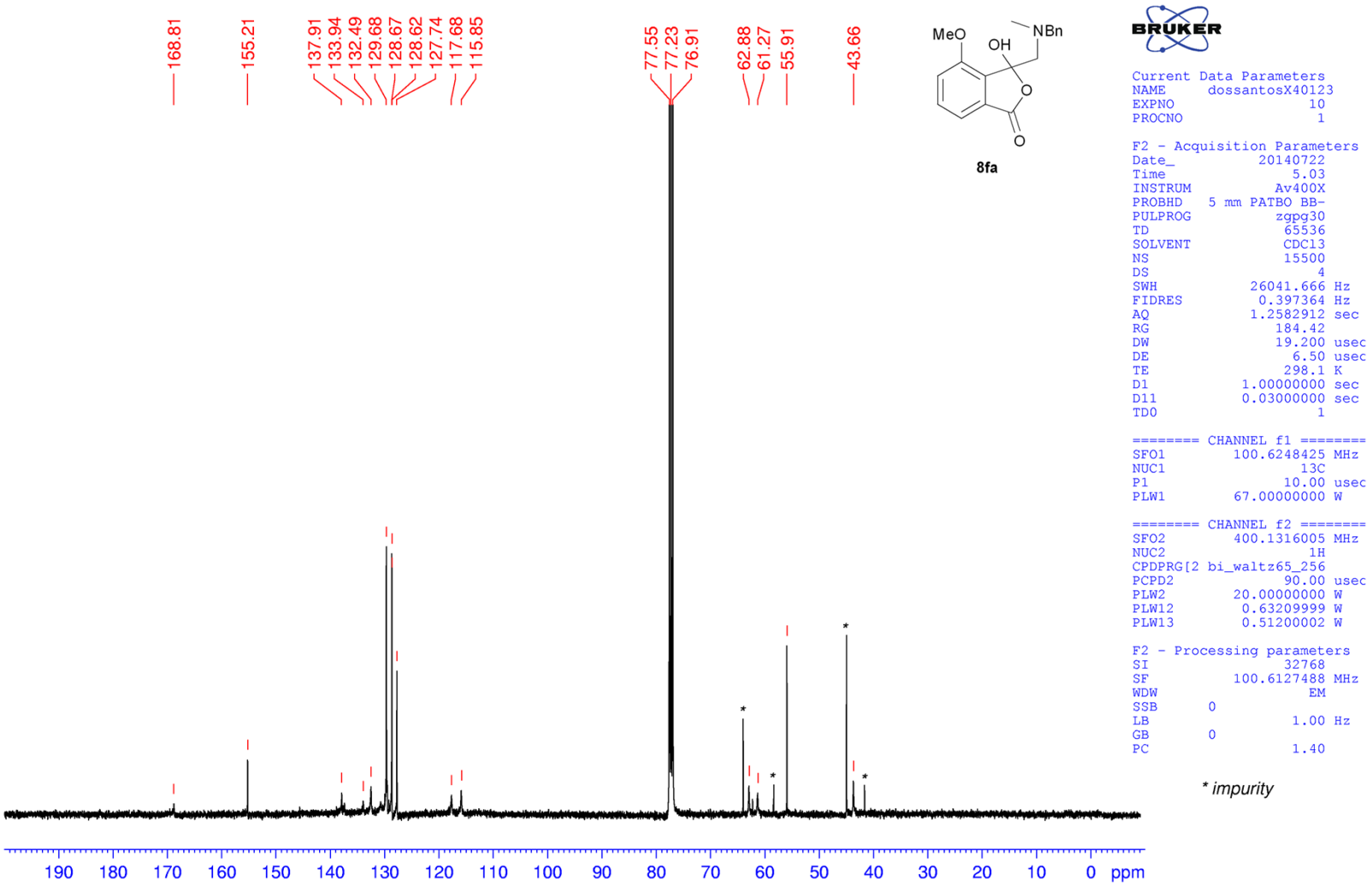
F2 - Acquisition Parameters
Date_           20140717
Time               15.11
INSTRUM          Av400H
PROBHD   5 mm PABBI 1H/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    64
DS                     2
SWH             6393.862 Hz
FIDRES          0.195125 Hz
AQ             2.5624576 sec
RG                   181
DW                78.200 usec
DE                 6.50 usec
TE                298.1 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1328009 MHz
NUC1                   1H
P1                 10.00 usec
PLW1         30.00000000 W

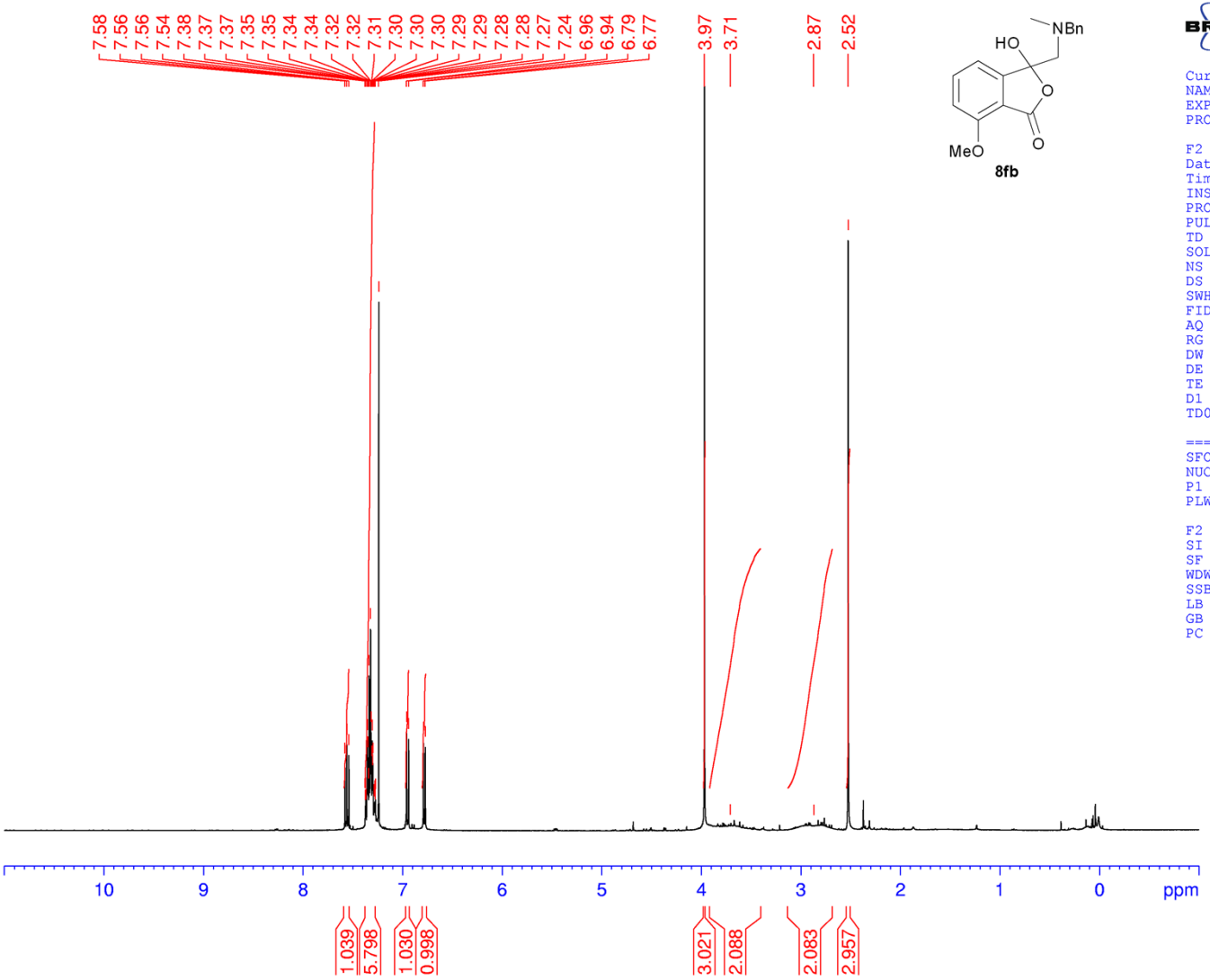
F2 - Processing parameters
SI                 65536
SF           400.1300175 MHz
WDW                   EM
SSB       0
LB                 0.10 Hz
GB        0
PC                 1.00
```

** impurity*

^{1}H NMR (400 MHz, $CDCl_3$,) spectrum of compound **8fa**.



^{13}C NMR (100 MHz, $CDCl_3$) spectrum of compound **8fa**.



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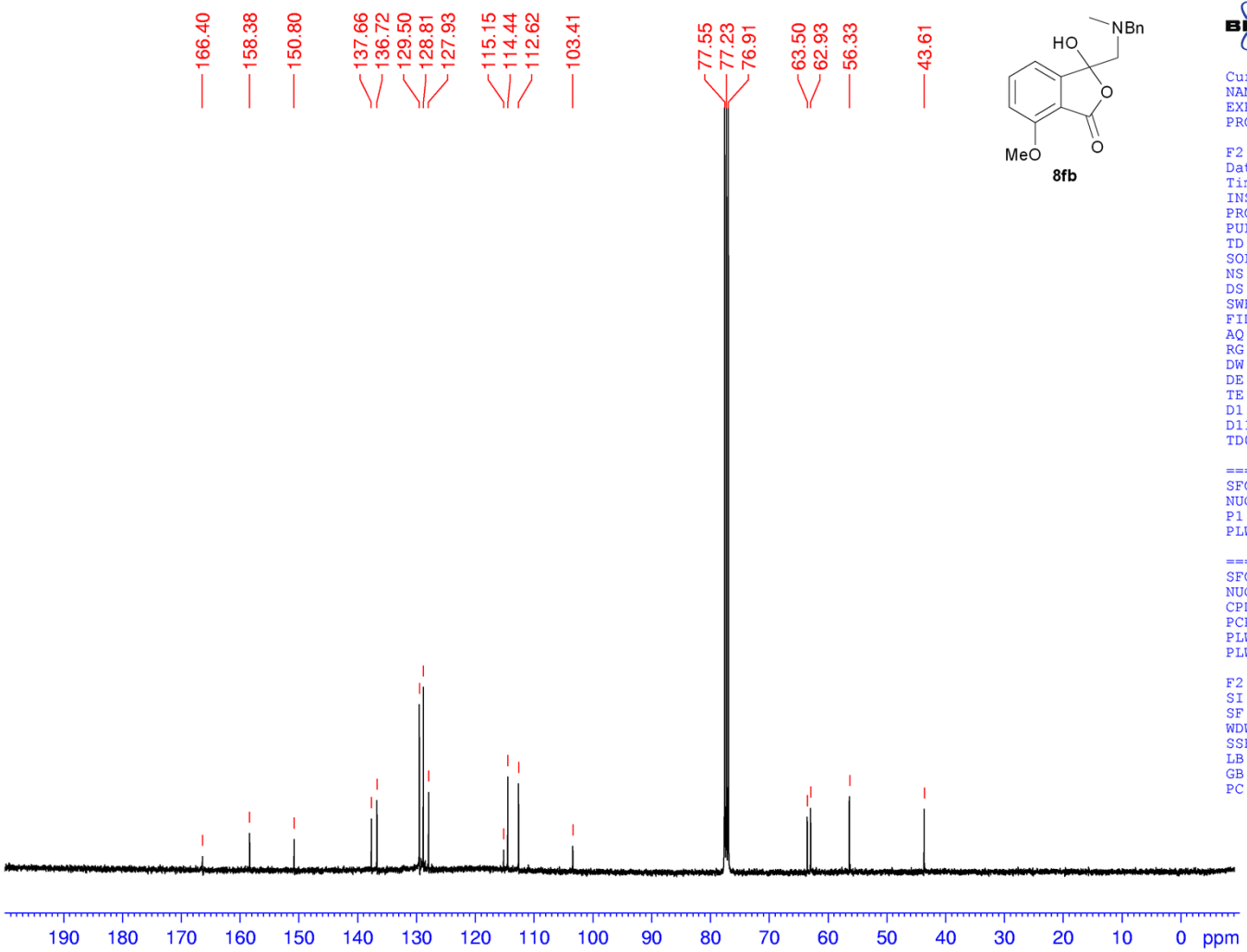
```
Current Data Parameters
NAME      dossantosX40118
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20140715
Time               13.21
INSTRUM          Av400X
PROBHD   5 mm PATBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     2
SWH             6393.862 Hz
FIDRES          0.195125 Hz
AQ             2.5624576 sec
RG                184.42
DW                78.200 usec
DE                 6.50 usec
TE                298.1 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1328009 MHz
NUC1                   1H
P1                 16.00 usec
PLW1         20.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300177 MHz
WDW                   EM
SSB       0
LB                 0.10 Hz
GB        0
PC                 1.00
```

^{1}H NMR (400 MHz, $CDCl_3$,) spectrum of compound **8fb**.




```
Current Data Parameters
NAME      dossantosH40100
EXPNO                  11
PROCNO                  1

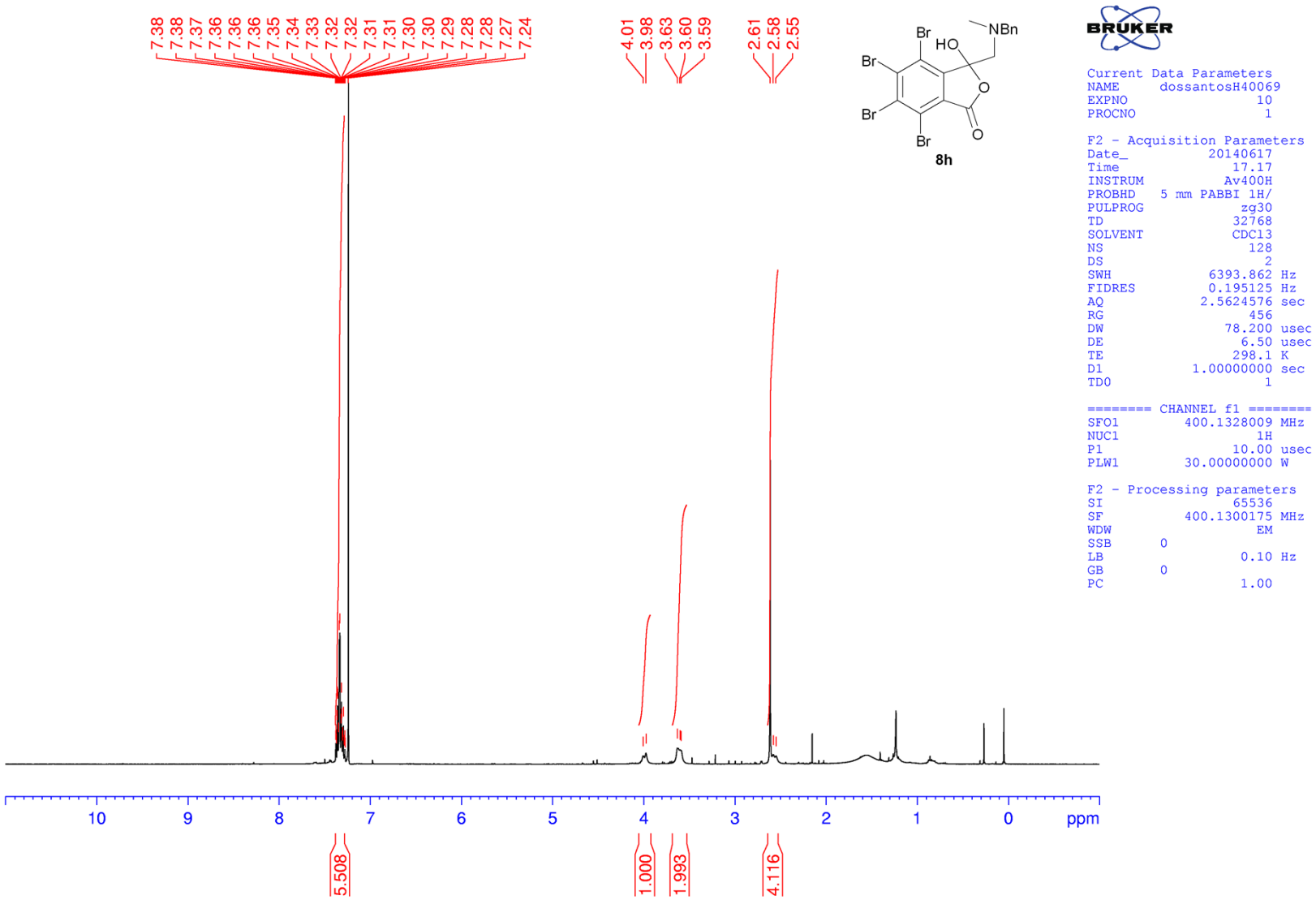
F2 - Acquisition Parameters
Date_           20140715
Time               22.49
INSTRUM          Av400H
PROBHD   5 mm PABBI 1H/
PULPROG          zgdc30
TD                65536
SOLVENT           CDCl3
NS                 5800
DS                    4
SWH           26041.666 Hz
FIDRES         0.397364 Hz
AQ            1.2582912 sec
RG                 1820
DW               19.200 usec
DE                6.50 usec
TE               298.1 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6248421 MHz
NUC1               13C
P1               13.00 usec
PLW1       146.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2         waltz65
PCPD2             90.00 usec
PLW2         30.00000000 W
PLW12         1.39999998 W

F2 - Processing parameters
SI                32768
SF           100.6127480 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

^{13}C NMR (100 MHz, $CDCl_3$) spectrum of compound **8fb**.



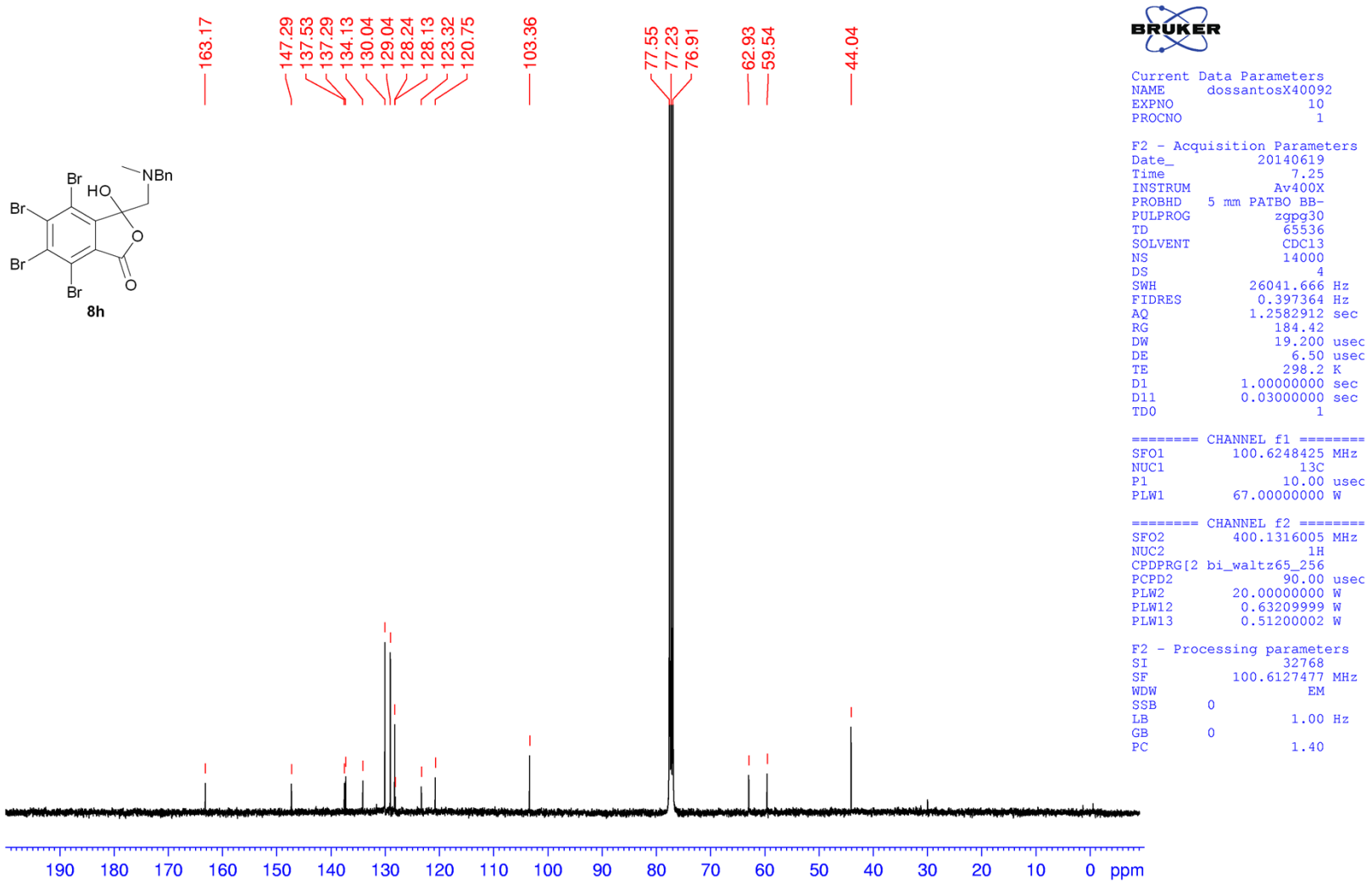

```
Current Data Parameters
NAME      dossantosH40069
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_           20140617
Time              17.17
INSTRUM          Av400H
PROBHD   5 mm PABBI 1H/
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                 128
DS                   2
SWH           6393.862 Hz
FIDRES        0.195125 Hz
AQ            2.5624576 sec
RG                 456
DW               78.200 usec
DE                6.50 usec
TE                298.1 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        400.1328009 MHz
NUC1                1H
P1                10.00 usec
PLW1        30.00000000 W

F2 - Processing parameters
SI                 65536
SF          400.1300175 MHz
WDW                EM
SSB      0
LB               0.10 Hz
GB       0
PC               1.00
```

^{1}H NMR (400 MHz, $CDCl_3$,) spectrum of compound **8h**.




```
Current Data Parameters
NAME      dossantosX40092
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_           20140619
Time                7.25
INSTRUM          Av400X
PROBHD   5 mm PATBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                14000
DS                    4
SWH           26041.666 Hz
FIDRES         0.397364 Hz
AQ             1.2582912 sec
RG               184.42
DW               19.200 usec
DE                6.50 usec
TE               298.2 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6248425 MHz
NUC1               13C
P1               10.00 usec
PLW1        67.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2 bi_waltz65_256
PCPD2            90.00 usec
PLW2        20.00000000 W
PLW12        0.63209999 W
PLW13        0.51200002 W

F2 - Processing parameters
SI                32768
SF          100.6127477 MHz
WDW                EM
SSB       0
LB               1.00 Hz
GB        0
PC               1.40
```

^{13}C NMR (100 MHz, $CDCl_3$) spectrum of compound **8h**.