Boronated Enynes as Versatile Sources of Stereodefined and Skeletally Diverse Molecules

Alain Hercouet, Fabienne Berrée, Chia Hui Lin, Loïc Toupet and Bertrand Carboni*

*Sciences Chimiques de Rennes, UMR 6226 et Groupe Matière condensée et Matériaux, UMR 6626
CNRS-Université de Rennes I, Campus de Beaulieu, 35042 Rennes CEDEX, France.*

Supporting Information

I.	General information	S-2
II.	Synthesis of boron-substituted enynes 1	S-2
III.	General procedure for the palladium-catalyzed cycloisomerisation of 1 .	S-6
IV.	Cycloisomerisation/Diels-alder cycloaddition	S-8
V.	General procedure for palladium-catalyzed cycloisomerisation /Diels-Alder cycloaddition/allylboration sequence	S-9
VI.	Metathesis/Diels-Alder/oxidation sequence	S-14
VII.	Platinum-catalyzed cycloisomerisation of 1a	S-15
VIII.	X-ray structure of 5	S-16
IX.	Reproductions of ^1H and ^{13}C spectra	S-17

I- General information

All reactions were carried out with anhydrous solvents. Toluene and tetrahydrofuran were distilled from sodium/benzophenone and methylene chloride from P_2O_5 . 1H NMR spectra (300 MHz) and ^{13}C NMR (75 MHz) were recorded on a Bruker AC 300 spectrometer, in $CDCl_3$ solutions with Me_4Si as internal reference. Chemical shifts are given in ppm and coupling constants J in Hz. Elemental analyses were performed by the Microanalytical Laboratory at the Centre Regional de Mesures Physiques de l'Ouest and high-resolution mass spectra (HRMS) recorded on a Varian MAT 311 spectrometer or a high resolution MS/MS ZABSpec TOF Micromass spectrometer. Analytical thin layer chromatography was performed on Merck Silica Gel 60 F254 plates.

II- Synthesis of boron-substituted enynes **1**

To a solution of (*E*)-2-(3-chloro-1-propenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (0.789 g, 3.9 mmol) in CH_3CN (10 mL) was added K_2CO_3 (1.08 g, 7.8 mmol) and propargylamine (660 mg, 820 μL , 12 mmol). The reaction mixture was stirred at room temperature during three days. Diethylether (10 mL) was then added and the precipitate was removed by filtration through celite. The filtrate was concentrated *in vacuo*. Excess of amine was eliminated by distillation (bp 50-60°C, 0.1 mm Hg).

1H -NMR (300 MHz, $CDCl_3$) δ 6.62 (dt, $J = 18.1, 5.3$ Hz, 1H), 5.61 (dt, $J = 18.1, 1.5$ Hz, 1H), 3.35-3.42 (m, 4H), 2.20 (t, $J = 2.3$ Hz, 1H), 1.25 (s, 12H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 150.1, 121.2 (br), 82.6, 81.4, 71.0, 41.3, 36.9, 24.7.

To a solution of this crude aminoboronate in dry CH_2Cl_2 (20 mL) was added dropwise at 0°C Net_3 (557 μL , 4 mmol) and a solution of *p*-toluenesulfonyl chloride (0.76 g, 4 mmol) in dry CH_2Cl_2 (5 mL). The reaction mixture was stirred during 12 h at room temperature and washed with water (2x10 mL). The organic phase was dried over $MgSO_4$, filtered and concentrated *in vacuo*. The product was purified by recrystallisation from ethanol.

1a (700 mg, 48%), mp 86°C (ethanol). 1H -NMR (300 MHz, $CDCl_3$) δ 7.75 (d, $J = 8.1$ Hz, 2H), 7.30 (d, $J = 8.3$ Hz, 2H), 6.47 (dt, $J = 17.9, 5.7$ Hz, 1H), 5.70 (dt, $J = 17.9, 1.5$ Hz, 1H), 4.10 (d, $J = 2.4$ Hz, 2H), 3.91 (dd, $J = 5.7, 1.3$ Hz, 2H), 2.43 (s, 3H), 2.00 (t, $J = 1.3$ Hz, 1H), 1.27 (s, 12H). ^{13}C -NMR (75

MHz, CDCl₃) δ 145.8, 143.5, 136.1, 129.5, 127.7, 123.3 (br), 83.4, 76.4, 73.8, 49.9, 36.1, 24.7, 21.5.

MS (EI) calcd. For C₁₈H₂₃NO₄¹¹BS [M-CH₃]⁺: 360.1440, found: 360.1452.

To a solution of (*E*)-2-(3-chloro-1-propenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (2 g, 10 mmol) in CH₃CN (20 mL) was added Net₃ (7 mL, 50 mmol) and but-3-yn-2-amine, hydrochloride (2.10 g, 20 mmol). The reaction mixture was stirred at room temperature during three days. Diethylether (20 mL) was then added and the resulting precipitate was removed by filtration through celite. The filtrate was concentrated *in vacuo*. Excess of amines was eliminated by distillation (bp 50-60°C, 0.1 mm Hg).

¹H-NMR (200 MHz, CDCl₃) δ 6.45 (dt, *J* = 17.8, 7.0 Hz, 1H), 6.08 (d, *J* = 17.7 Hz, 1H), 3.31-3.59 (m, 3H), 2.28 (t, *J* = 2.1 Hz, 1H), 1.44 (d, *J* = 7.4 Hz, 3H), 1.28 (s, 12H).

To a solution of this crude aminoboronate in dry CH₂Cl₂ (30 mL) was added dropwise at 0°C Net₃ (1.67 mL, 12 mmol) and a solution of *p*-toluenesulfonyl chloride (2.27 g, 12 mmol) in dry CH₂Cl₂ (20 mL). The reaction mixture was stirred during 12 h at room temperature and washed with water (2x10 mL). The organic phase was dried over MgSO₄, filtered and concentrated *in vacuo*. The product was purified by silica gel chromatography (cyclohexane/Ethyl acetate: 6/1).

1b (2.1 g, 54%). White solid, mp 132-133°C (diisopropylether). ¹H-NMR (300 MHz, CDCl₃) δ 7.73 (d, *J* = 7.9 Hz, 2H), 7.29 (d, *J* = 7.8 Hz, 2H), 6.65 (dt, *J* = 17.9, 5.3 Hz, 1H), 5.68 (d, *J* = 18.1 Hz, 1H), 4.88 (q, *J* = 6.9 Hz, 1H), 4.00 (dd, *J* = 18.3, 3.6 Hz, 1H), 3.79 (dd, *J* = 17.4, 6.1 Hz, 1H), 2.42 (s, 3H), 2.11 (s, 1H), 1.43 (d, *J* = 6.6 Hz, 3H), 1.27 (s, 12H); ¹³C-NMR (75 MHz, CDCl₃) δ 149.9, 143.4, 136.2, 129.4, 127.7, 83.3, 81.2, 73.3, 48.5, 46.3, 24.8, 22.7, 21.5. MS (EI) calcd. For C₁₉H₂₅NO₄¹¹BS [M-CH₃]⁺: 374.15974, found: 374.1589.

The reaction mixture from (*E*)-2-(3-chloro-1-propenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1.01 g, 5 mmol), *N*-benzylprop-2-yn-1-amine (800 mg, 5.5 mmol), and K₂CO₃ (1.97 g, 15 mmol) in CH₃CN

(10 mL) was stirred during three days at room temperature. The solid was removed by filtration through celite and washed with ether. The filtrate was concentrated *in vacuo*. The residue was purified through silica gel chromatography (cyclohexane/Ethyl acetate: 4/1).

1c (720 mg, 46%). ¹H-NMR (300 MHz, CDCl₃) δ 7.39-7.23 (m, 5H), 6.66 (dt, *J* = 18.0, 5.5 Hz, 1H), 5.75 (d, *J* = 18.0 Hz, 1H), 3.67 (s, 2H), 3.32 (s, 2H), 3.27 (d, *J* = 5.6 Hz, 2H), 2.26 (s, 1H), 1.28 (s, 12H). ¹³C-NMR (75 MHz, CDCl₃) δ 150.3, 138.4, 128.8, 128.1, 127.0, 121.2 (br), 83.0, 78.3, 73.2, 57.35, 57.3, 41.4, 24.6. MS (EI) calcd. For C₁₉H₂₆NO₂¹¹B [M]⁺ : 311.2056, found: 311.2052.

(2^E)-*N*-benzyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)prop-2-en-1-amine was first prepared as previously described in the synthesis of **1a** using benzylamine instead of propargylamine.

¹H-NMR (200 MHz, CDCl₃) δ 7.45-7.15 (m, 5H), 6.64 (dt, *J* = 18.0, 5.3 Hz, 1H), 5.65 (dt, *J* = 18.1, 1.5 Hz, 1H), 3.78 (s, 2H), 3.35 (dd, *J* = 5.3, 1.5 Hz, 2H), 1.27 (s, 12H).

To a solution of this crude boronate (740 mg, 2.7 mmol) in dry CH₂Cl₂ (10 mL) was first added at 0°C a solution of propionic acid (180 μL, 2.9 mmol) in dry CH₂Cl₂ (5 mL) and then DCC (634 mg, 3 mmol) and DMAP (37 mg, 0.3 mmol). The reaction mixture was stirred overnight at room temperature. The resulting solid was removed by filtration through celite and the filtrate was concentrated *in vacuo*. The residue was purified by silica gel chromatography (cyclohexane/Ethyl acetate: 1/4 to 1/1).

1d (450 mg, 51%) as a mixture of two rotamers (1/1). ¹H-NMR (300 MHz, CDCl₃) δ 7.24-7.38 (m, 5H), 6.51 (dt, *J* = 18.0, 5.0 Hz, 1H) and 6.47 (dt, *J* = 18.1, 5.2 Hz, 1H), 5.62 (d, *J* = 18.0 Hz, 1H) and 5.52 (d, *J* = 18.1 Hz, 1H), 4.78 (s, 2H) and 4.60 (s, 2H), 4.17 (dd, *J* = 5.2, 1.3 Hz, 2H) and 4.01 (dd, *J* = 5.0, 1.3 Hz, 2H), 3.14 (s, 1H) and 3.10 (s, 1H), 1.31 (s, 12H) and 1.29 (s, 12H). ¹³C-NMR (75 MHz, CDCl₃) δ 153.5 and 153.1, 145.9 and 145.4, 135.9 and 135.6, 128.6 and 128.5, 128.2 and 127.8, 127.5 and 127.4, 122.3 (br), 83.4 and 83.2, 79.2 and 79.1, 75.5 and 75.4, 51.7 and 51.2, 46.8 and 46.7, 24.6. MS (EI) calcd. For C₁₉H₂₄NO₃¹¹B [M]⁺ : 325.1849, found: 325.1876.

To a suspension of NaH (120 mg, 3 mmol) in dry THF (5 mL) at 0°C was added diethyl propargylmalonate (489 mg, 3 mmol) in THF (5 mL).¹ After stirring for 30 min, (*E*)-2-(3-chloro-1-propenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (607 mg, 3 mmol) was added and the reaction was stirred additional 15 h. After addition of a saturated ammonium chloride solution (5 mL), the mixture was extracted with diethylether, and dried over MgSO₄. The solvent was removed *in vacuo* and the crude product was by distillation (bp 100-105°C/0.1 mm Hg).

1e (830 mg, 76%). ¹H-NMR (300 MHz, CDCl₃) δ 6.34 (dt, *J* = 17.8, 6.3 Hz, 1H), 5.61 (d, *J* = 17.8 Hz, 1H), 4.21-4.10 (m, 4H), 2.85 (d, *J* = 7.3 Hz, 2H), 2.75 (d, *J* = 2.5 Hz, 2H), 1.97 (t, *J* = 2.5 Hz, 1H), 1.20 (t, *J* = 7.3 Hz, 6H), 1.20 (s, 12H). ¹³C-NMR (75 MHz, CDCl₃) δ 169.6, 146.3, 83.2, 78.8, 71.5, 61.7, 56.5, 38.4, 24.8, 22.7, 14.0. MS (EI) calcd. For C₁₈H₂₆O₄¹¹B [M-CH₃]⁺: 349.1822, found : 349.1797.

A 50 mL round bottom flask equipped with a septum inlet was charged with borane/dimethylsulfide complex (10M, 0.5 mL, 5 mmol) and dry THF (2 mL) under an argon atmosphere. α-Pinene (1.67 mL, 10 mmol) was then added dropwise at 0°C and the mixture was stirred for 10 min followed by 2h at room temperature. To the resulting suspension cooled to -40°C was added dropwise a solution of the diyne (1.88 g, 20 mmol) in THF (5 mL). The mixture was stirred at 0°C for 1 h and further stirred at room temperature for additional 4h. It was cooled back to 0°C again prior to the addition of freshly distilled acetaldehyde (4 mL). The solution was warmed up and stirred for 15 h before removal of volatiles *in vacuo*. Diethylether (20 mL) were added, followed by pinacol (5.3 g, 4.5 mmol). After 5h at room temperature, the solvent was evaporated and the residue dissolved in pentane, washed twice with water and dried with MgSO₄. The crude product was purified by distillation (bp 75-80°C/1 mm Hg).

1f (0.65 g, 65%). ¹H-NMR (300 MHz, CDCl₃) δ 6.63 (dt, *J* = 18.2, 4.9 Hz, 1H), 5.73 (dt, *J* = 18.2, 1.5 Hz, 1H), 4.21-4.12 (m, 4H), 2.43 (t, *J* = 2.3 Hz, 1H), 1.26 (s, 12H). ¹³C-NMR (75 MHz, CDCl₃) δ

¹ Brummond, K. M.; Chen, H.; Sill, P.; You, L. *J. Am. Chem. Soc.*, **2002**, *124*, 15186.

148.0, 83.3, 79.5, 74.5, 71.1, 57.4, 24.7. MS (EI) calcd. For $C_{11}H_{16}O_3^{11}B$ $[M-CH_3]^+$: 207.1192, found: 207.1197.

The same sequence as for **1a** was carried out starting from the corresponding (+)-pinanediol derivative instead of (*E*)-2-(3-chloro-1-propenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane pinacol. The residue was purified by silica gel chromatography (cyclohexane/Ethyl acetate: 1/5 to 1/1).

1g (510 mg, 31%). 1H -NMR (300 MHz, $CDCl_3$) δ 7.75 (d, J = 8.3 Hz, 2H), 7.31 (d, J = 8.2 Hz, 2H), 6.48 (dt, J = 18.0, 5.7 Hz, 1H), 5.72 (d, J = 18.0 Hz, 1H), 4.32 (dd, J = 8.8, 1.6 Hz, 1H), 4.11 (d, J = 2.3 Hz, 2H), 3.92 (dd, J = 5.7, 0.8 Hz, 2H), 2.44 (s, 3H), 2.39-2.20 (m, 2H), 2.07 (t, J = 5.7 Hz, 1H), 2.02 (t, J = 2.4 Hz, 1H), 1.95-1.84 (m, 2H), 1.41 (s, 3H), 1.31 (s, 3H), 1.12 (d, J = 10.6 Hz, 1H), 0.86 (s, 3H). ^{13}C -NMR (75 MHz, $CDCl_3$) δ 145.7, 143.6, 136.0, 129.5, 127.7, 122.7 (br), 85.9, 77.8, 76.4, 73.9, 51.3, 50.0, 39.4, 38.1, 36.1, 35.4, 28.6, 27.1, 26.4, 24.0, 21.5. MS (EI) calcd. For $C_{16}H_{23}NO_2^{11}B$ $[M-Ts]^+$: 272.1822, found: 272.1828. $[\alpha]_D^{20}$ = + 9.6° (c 1, CH_2Cl_2).

III- General procedure for the palladium-catalyzed cycloisomerisation of **1**.

In a Schlenk flask, tris(dibenzylideneacetone)dipalladium(0)-chloroform adduct (5.2 mg, 0.05 mmol, 2.5% mol), tri-*o*-tolylphosphine (3.0 mg, 0.05 mmol, 5% mol), acetic acid (0.6 μ L, 0.01 mmol, 5% mol) and toluene (1 mL) were stirred for 15 min at room temperature. Enyne **1** (0.2 mmol) was added and the mixture was kept 2h at room temperature under an argon atmosphere, except for **1c** and **1b**, respectively 18h at room temperature and 2h at 50°C. Attempts to purify compounds **2** by chromatography on silica gel resulted in partial decomposition that explained that the NMR data of dienes **2** were extracted from the NMR spectra of the crude mixtures.

^1H -NMR (300 MHz, CDCl_3) δ 7.74 (d, J = 8.1 Hz, 2H), 7.33 (d, J = 8.1 Hz, 2H), 5.75 (t, J = 2.3 Hz, 1H), 5.52 (t, J = 2.2 Hz, 1H), 5.02 (s, 1H), 4.23 (d, J = 2.3 Hz, 2H), 3.97 (t, J = 2.2 Hz, 2H), 2.43 (s, 3H), 1.28 (s, 12H). ^{13}C -NMR (75 MHz, CDCl_3) δ 154.2, 143.7, 142.5, 132.8, 129.7, 127.9, 125.3, 107.3, 83.5, 53.4, 52.4, 24.8, 21.5.

^1H -NMR (300 MHz, CDCl_3) δ 7.70 (d, J = 8.1 Hz, 2H), 7.36 (d, J = 8.1 Hz, 2H), 5.65 (t, J = 2.4 Hz, 1H), 5.45 (t, J = 1.9 Hz, 1H), 5.02 (s, 1H), 4.36-4.25 (m, 3H), 2.43 (s, 3H), 1.45 (d, J = 6.4 Hz, 3H), 1.28 (s, 12H). ^{13}C -NMR (75 MHz, CDCl_3) δ 154.2, 148.9, 143.4, 137.8, 129.6, 127.5, 107.4, 83.6, 59.8, 52.6, 24.7, 23.1, 21.5.

^1H -NMR (300 MHz, CDCl_3) δ 7.38-7.20 (m, 5H), 5.73 (s, 1H), 5.52 (s, 1H), 4.98 (s, 1H), 3.72 (s, 2H), 3.35 (s, 2H), 3.28 (d, J = 4.2 Hz, 2H), 1.28 (s, 12H).

The dimerisation product was the only isolated compound. 35% from **1d**. ^1H -NMR (300 MHz, CDCl_3) δ 7.49-7.28 (m, 10H), 5.19 (t, J = 2.2 Hz, 1H), 5.05 (d, J = 15.1 Hz, 1H), 4.81 (d, J = 15.0 Hz, 1H), 4.38 (d, J = 15.0 Hz, 1H), 4.28 (d, J = 15.1 Hz, 1H), 4.27 (dd, J = 18.9, 3.2 Hz, 1H), 4.14 (dd, J = 16.5, 2.5 Hz, 1H), 4.05 (dd, J = 16.5, 2.5 Hz, 1H), 3.70 (d, J = 19.0 Hz, 1H), 2.83-2.80 (m, 1H), 2.58-2.47 (m, 1H), 2.33-2.24 (m, 1H), 1.90 (dt, J = 11.7, 5.2 Hz, 1H), 1.72-1.65 (m, 1H), 1.19 (s, 6H), 1.17 (s, 6H), 1.12 (s, 6H), 1.02 (s, 6H).

$^1\text{H-NMR}$ (300 MHz, CDCl_3) δ 5.80 (t, $J = 2.3$ Hz, 1H), 5.53 (t, $J = 2.3$ Hz, 1H), 5.04 (t, $J = 1.8$ Hz, 1H), 4.21 (q, $J = 7.1$ Hz, 4H), 3.34 (m, 2H), 3.05 (m, 2H), 1.29 (s, 12H), 1.25 (t, $J = 7.1$ Hz, 6H).

$^1\text{H-NMR}$ (300 MHz, CDCl_3) δ 5.80 (t, $J = 2.3$ Hz, 1H), 5.57 (t, $J = 2.3$ Hz, 1H), 5.05 (s, 1H), 4.69 (d, $J = 2.3$ Hz, 2H), 4.47 (t, $J = 2.3$ Hz, 2H), 1.27 (s, 12H).

IV- Cycloisomerisation/Diels-alder cycloaddition

A Schlenk flask with a magnetic stirrer and a rubber septum was charged with tris(dibenzylideneacetone)dipalladium (0) chloroform adduct (5.2 mg, 0.05 mmol, 2.5% mol), tri-*o*-tolylphosphine (3.0 mg, 0.01 mmol, 5% mol), acetic acid (0.6 μL , 10^{-2} mmol, 5% mol) and toluene (1 mL). After stirring 15 min at room temperature, enyne **1** (0.2 mmol) was added and the mixture was kept 2h at room temperature under an argon atmosphere. N-phenylmaleimide (0.3 mmol, 1.5 eq) or maleic anhydride was then added and the mixture was stirred for 15 h at room temperature. After removal of volatiles, the residue was purified by washing with ethanol.

4a (85 mg, 77%). $^1\text{H-NMR}$ (300 MHz, CDCl_3) δ 7.75 (d, $J = 8.2$ Hz, 2H), 7.48-7.22 (m, 7H), 4.30-4.10 (m, 4H), 3.36 (dd, $J = 9.8, 7.6$ Hz, 1H), 3.21 (dt, $J = 9.8, 9.3$ Hz, 1H), 2.63-2.32 (m, 3H), 2.42 (s, 3H), 1.10 (s, 12H). $^{13}\text{C-NMR}$ (75 MHz, CDCl_3) δ 178.5, 178.4, 143.4, 134.1, 132.0, 131.1, 129.8, 129.0, 128.9, 128.5, 127.5, 126.3, 84.3, 56.6, 56.5, 40.8, 38.7, 24.6, 24.2, 22.2, 21.5. MS (LSIMS) calcd. For $\text{C}_{29}\text{H}_{34}\text{N}_2\text{O}_6$ ^{11}BS $[\text{M}+\text{H}]^+$: 549.2230, found : 549.2236.

4b (78 mg, 83%). $^1\text{H-NMR}$ (300 MHz, CD_3COCD_3) δ 7.72 (d, $J = 8.2$ Hz, 2H), 7.34 (d, $J = 8.2$ Hz, 2H), 4.18-4.08 (m, 4H), 3.42-3.26 (m, 2H), 2.55-2.33 (m, 3H), 2.43 (s, 3H), 1.14 (s, 6H), 1.12 (s, 6H). $^{13}\text{C-NMR}$ (75 MHz, CD_3COCD_3) δ 174.4, 174.2, 143.6, 134.1, 129.8, 128.7, 127.5, 84.3, 56.6, 56.3, 40.8, 38.4, 24.0, 23.7, 21.0, 20.5. MS (EI) calcd. For $\text{C}_{22}\text{H}_{28}\text{NO}_6$ ^{11}BS $[\text{M-CO}]^+$: 445.1730, found : 445.1702.

4c. $^1\text{H-NMR}$ (300 MHz, CDCl_3) δ 7.75 (d, $J = 8.2$ Hz, 2H), 7.50-7.27 (m, 7H), 4.43-4.11 (m, 3H), 3.32 (dd, $J = 9.8, 7.2$ Hz, 1H), 3.16 (dt, $J = 9.7, 7.6$ Hz, 1H), 2.46-2.41 (m, 2H), 2.45 (s, 3H), 2.29 (d, $J = 6.7$ Hz, 1H), 1.36 (d, $J = 6.3$ Hz, 3H), 1.21 (s, 12H).

V- General procedure for palladium-catalyzed cycloisomerisation /Diels-Alder cycloaddition/allylboration sequence

In a Schlenk flask tris(dibenzylideneacetone)dipalladium(0)-chloroform adduct (5.2 mg, 0.05 mmol, 2% mol) tri-*o*-tolylphosphine (3 mg, 0.01 mmol, acetic acid (0.6 μL , 10^{-2} mmol, 5% mol) and toluene (1 mL) were stirred for 15 min. Enyne **1** (0.2 mmol) was added and the mixture was reacted 2h at room temperature under an argon atmosphere (18h for **1c** and 2h at 50°C for **1b**). Then dienophile (0.3 mmol, 1.5 eq) and aldehyde (0.4 mmol, 2 eq) were successively added and the mixture was stirred for 15 h at 60°C. For **4b**, toluene was first evaporated before performing the allylboration in THF. After removal of volatiles, THF (2 mL) and saturated Na_2CO_3 (2 mL) were added and the mixture was stirred for 15 h. Ethyl acetate (10 mL) and water (2 mL) were added and the aqueous phase was extracted with ethyl acetate (2x10 mL). The combined organic phases were washed with H_2O (5 mL)

and brine (5 mL), dried over MgSO₄ and evaporated under *vacuo*. The residue was purified by column chromatography on silica gel (cyclohexane/ethyl acetate: 1/4 to 1/1).

5 (75 mg, 71%). ¹H-NMR (300 MHz, CDCl₃) δ 7.66 (d, *J* = 8.2 Hz, 2H), 7.46-7.28 (m, 12H), 6.03 (d, *J* = 6.1 Hz, 1H), 4.70 (d, *J* = 3.2 Hz, 1H), 4.05 (dt, *J* = 14.1, 2.1 Hz, 1H), 3.79-3.66 (m, 3H), 3.25 (t, *J* = 9.3 Hz, 1H), 2.93 (d, *J* = 14.7 Hz, 1H), 2.46 (s, 3H), 2.39 (d, *J* = 9.7 Hz, 1H), 2.19 (d, *J* = 3.2 Hz, 1H), 1.27 (dd, *J* = 15.0, 9.5 Hz, 1H). ¹³C-NMR (75 MHz, DMSO-D₆) δ 183.8, 181.5, 148.9, 145.5, 144.9, 138.0, 137.6, 135.1, 133.9, 133.3, 132.9, 132.8, 132.7, 132.6, 132.3, 122.2, 78.1, 61.7, 55.5, 53.2, 42.3, 29.8, 26.2. MS (LSIMS) calcd. For C₃₀H₂₉N₂O₅S [M+H]⁺: 529.1797, found: 529.1776.

6 (82 mg, 74%). ¹H-NMR (300 MHz, CDCl₃) δ 7.68 (d, *J* = 8.2 Hz, 2H), 7.50-7.23 (m, 9H), 6.88 (d, *J* = 8.2 Hz, 2H), 6.04 (d, *J* = 6.0 Hz, 1H), 4.70 (d, *J* = 3.2 Hz, 1H), 4.09 (dt, *J* = 13.9, 1.9 Hz, 1H), 3.82 (s, 3H), 3.78-3.70 (m, 2H), 3.64 (d, *J* = 9.6 Hz, 1H), 3.25 (t, *J* = 9.3 Hz, 1H), 2.91 (d, *J* = 14.9 Hz, 1H), 2.46 (s, 3H), 2.38 (d, *J* = 9.7 Hz, 1H), 2.02 (d, *J* = 3 Hz, 1H), 1.28 (dd, *J* = 15.2, 9.6 Hz, 1H). ¹³C-NMR (75 MHz, DMSO-D₆) δ 179.0, 176.8, 159.0, 144.2, 140.3, 133.6, 133.2, 132.9, 130.4, 129.1, 128.5, 128.0, 127.6, 117.3, 113.5, 72.9, 57.0, 55.5, 50.8, 48.6, 37.5, 25.2, 21.5. MS (ESI) calcd. For C₃₁H₃₀N₂O₆NaS [M+Na]⁺: 581.1722, found: 581.1731.

7 (89 mg, 78%). ¹H-NMR (300 MHz, CDCl₃) δ 8.18 (d, *J* = 8.7 Hz, 2H), 7.63 (d, *J* = 8.7 Hz, 2H), 7.60 (d, *J* = 8.7 Hz, 2H), 7.50-7.25 (m, 7H), 6.1 (d, *J* = 7.3 Hz, 1H), 4.79 (d, *J* = 3.6 Hz, 1H), 3.98 (d, *J* = 13.8 Hz, 1H), 3.85-3.73 (m, 2H), 3.65 (d, *J* = 9.9 Hz, 1H), 3.30 (t, *J* = 9.1 Hz, 1H), 2.86 (d, *J* = 15.1

Hz, 1H), 2.50-2.38 (m, 5H), 1.37 (dd, $J = 15.0, 9.2$ Hz, 1H). ^{13}C -NMR (75 MHz, DMSO- D_6) δ 179.1, 176.7, 149.7, 147.2, 144.3, 139.7, 133.1, 132.6, 130.4, 129.2, 129.0, 128.6, 128.0, 127.5, 127.4, 123.2, 118.2, 72.9, 56.4, 50.4, 50.7, 48.7, 25.6, 21.5. MS (ESI) calcd. For $\text{C}_{30}\text{H}_{27}\text{N}_3\text{O}_7\text{NaS}$ $[\text{M}+\text{Na}]^+$: 596.1467, found: 596.1457.

8 (65 mg, 69%). ^1H -NMR (300 MHz, CDCl_3) δ 7.72 (d, $J = 8.1$ Hz, 2H), 7.50-7.18 (m, 7H), 5.98 (d, $J = 5.6$ Hz, 1H), 4.07 (d, $J = 14$ Hz, 1H), 3.89 (d, $J = 14$ Hz, 1H), 3.72 (dd, $J = 8.1, 6.9$ Hz, 1H), 3.64 (d, $J = 10.0$ Hz, 1H), 3.18 (t, $J = 8.8$ Hz, 1H), 2.95 (d, $J = 8.5$ Hz, 1H), 2.84 (d, $J = 14.3$ Hz, 1H), 2.76 (d, $J = 10.0$ Hz, 1H), 2.45 (s, 3H), 1.85 (d, $J = 3.4$ Hz, 1H), 1.60-1.20 (m, 3H), 0.86 (t, $J = 7.2$ Hz, 3H). ^{13}C -NMR (75 MHz, DMSO- D_6) δ 178.4, 176.7, 144.0, 140.6, 133.4, 133.3, 130.4, 129.0, 128.4, 127.8, 127.6, 117.0, 72.3, 58.0, 51.0, 48.7, 37.4, 31.2, 25.6, 24.2, 21.5, 12.4. MS (ESI) calcd. For $\text{C}_{26}\text{H}_{28}\text{N}_2\text{O}_5\text{NaS}$ $[\text{M}+\text{Na}]^+$: 503.1616, found: 503.1617.

9 was purified as its methyl ester. The residue was dissolved in THF (5 mL). After addition of MeOH (1 mL) and trimethylsilyldiazomethane (2M in diethylether, 500 μL , 4 eq), the reaction mixture was stirred 15h at room temperature. After evaporation, the residue was purified by column chromatography on silica gel (cyclohexane/ethyl acetate: 2/1).

9' (66 mg, 71 %), mp: 145-148°C (diisopropylether). ^1H -NMR (300 MHz, CDCl_3) δ 7.70 (d, $J = 8.2$ Hz, 2H), 7.47-7.28 (m, 7H), 5.88 (t, $J = 2.05$ Hz, 1H), 5.50 (s, 1H), 4.43 (dt, $J = 14.3, 2.6$ Hz, 1H), 3.77 (dd, $J = 14.3, 1.8$ Hz, 1H), 3.72 (s, 3H), 3.58 (d, $J = 4.05$ Hz, 1H), 3.53-3.49 (m, 2H), 3.36 (d, $J = 10.0$ Hz, 1H), 3.31 (dd, $J = 13.5, 4.3$ Hz, 1H), 2.48 (s, 3H), 1.38 (d, $J = 13.5$ Hz, 1H). ^{13}C -NMR (75 MHz, CDCl_3) δ 171.8, 170.75, 144.25, 140.5, 135.7, 132.3, 130.0, 128.9, 128.6, 127.8, 127.2, 118.2, 84.5, 55.0, 52.6, 49.6, 45.7, 43.9, 38.2, 21.95, 21.6. MS (EI) calcd. For $\text{C}_{25}\text{H}_{25}\text{NO}_6\text{S}$ $[\text{M}+\text{H}]^+$: 467.14026, found: 467.1377.

10 (46 mg, 47 %), mixture of 2 isomers (60/40). Major isomer: $^1\text{H-NMR}$ (300 MHz, CDCl_3) δ 7.64 (d, $J = 8.2$ Hz, 2H), 7.38-7.26 (m, 7H), 5.77 (d, $J = 2.0$ Hz, 1H), 4.87 (s, 1H), 4.00 (dd, $J = 5.5, 2.1$ Hz, 1H), 3.85 (d, $J = 9.8$ Hz, 1H), 3.79-3.48 (m, 1H), 3.75 (s, 3H), 3.71 (s, 3H), 2.91-2.88 (m, 1H), 2.78 (dt, $J = 8.6, 2.0$ Hz, 1H), 2.64 (d, $J = 9.7$ Hz, 1H), 2.48 (d, $J = 4.2$ Hz, 1H), 2.45 (s, 3H), 2.15 (s, br, 1H), 1.40 (dd, $J = 14.1, 6.6$ Hz, 1H); $^{13}\text{C-NMR}$ (75 MHz, CDCl_3) δ 174.8, 172.8, 143.5, 139.7, 138.7, 133.5, 129.65, 128.1, 127.3 (2C), 119.9, 74.6, 55.5, 52.4, 52.2, 50.6, 49.4, 43.8, 38.5, 31.3, 21.4 (2C). Minor isomer (characteristic signals): $^1\text{H-NMR}$ (300 MHz, CDCl_3) δ 7.72 (d, $J = 8.3$ Hz, 2H), 5.83 (d, $J = 1.7$ Hz, 1H), 4.71 (s, 1H), 3.73 (s, 3H), 3.68 (s, 3H), 2.56 (d, $J = 8.9$ Hz, 1H), 2.39 (d, $J = 3.8$ Hz, 1H); $^{13}\text{C-NMR}$ (75 MHz, CDCl_3) δ 175.2, 173.3, 143.6, 139.9, 139.4, 133.4, 129.7, 128.0, 127.9, 127.1, 121.1, 73.5, 56.4, 52.4, 52.25, 50.4, 49.9, 42.5, 40.6, 27.5, 22.7. MS (EI) calcd. For $\text{C}_{25}\text{H}_{26}\text{NO}_6\text{S}$ $[\text{M-OCH}_3]^+$: 468.1481, found : 468.1484.

After purification by chromatography on silica gel, three compounds were isolated: **11**, the addition product to 4-phenyl triazolidinedione and another unidentified by-product.

11 (25 mg, 23%). $^1\text{H-NMR}$ (300 MHz, $\text{CDCl}_3/\text{DMSO-D}_6$) δ 7.33 (d, $J = 8.2$ Hz, 2H), 7.28-7.16 (m, 6H), 7.10 (d, $J = 8.1$ Hz, 2H), 6.99-6.85 (m, 4H), 6.69 (s, 1H), 4.59 (d, $J = 11.7$ Hz, 1H), 4.54 (s, 1H), 3.68 (d, $J = 10.4$ Hz, 1H), 3.48 (d, $J = 12.7$ Hz, 1H), 2.78 (d, $J = 4.2$ Hz, 1H), 2.74 (d, $J = 2.9$ Hz, 1H), 2.67 (s, br, 1H), 2.55 (dd, $J = 12.2, 1.8$ Hz, 1H), 2.25 (s, 3H). $^{13}\text{C-NMR}$ (75 MHz, $\text{CDCl}_3/\text{DMSO-D}_6$) δ 148.7, 144.5, 143.0, 139.6, 132.4, 130.35, 129.1, 128.4, 127.6, 127.1, 126.9, 126.8, 126.3, 125.0, 117.3, 111.9, 72.7, 49.9, 49.6, 49.0, 45.5, 20.9. MS (EI) calcd. For $\text{C}_{21}\text{H}_{20}\text{N}_4\text{O}_4\text{S}$ $[\text{M-PhCHO}]^+$: 424.12053, found: 424.1191.

(10 mg, 8%). $^1\text{H-NMR}$ (300 MHz, CDCl_3) δ 7.60 (d, $J = 7.9$ Hz, 2H), 7.33-7.03 (m, 12H), 6.84 (s, 1H), 4.78 (d, $J = 13.1$ Hz, 1H), 4.35 (dd, $J = 15.35, 14.1$ Hz, 2H), 3.79 (d, $J = 13.7$ Hz, 1H), 2.84 (d, $J = 10.3$ Hz, 1H), 2.57 (d, $J = 11.4$ Hz, 1H), 2.19 (s, 3H). $^{13}\text{C-NMR}$ (75 MHz, CD_3OD) δ 156.5, 152.8, 152.45, 147.4, 145.4, 135.3, 135.1, 132.5, 130.9, 130.1, 129.6, 129.5, 128.8, 127.7, 127.5, 127.0, 116.0, 115.8, 63.3, 56.6, 50.6, 48.0, 21.7. MS (ESI) calcd. For $\text{C}_{29}\text{H}_{25}\text{N}_7\text{O}_6\text{NaS}$ $[\text{M}+\text{Na}]^+$: 622.1485, found: 622.1464.

Unidentified product (21 mg). $^1\text{H-NMR}$ (300 MHz, $\text{CDCl}_3/\text{DMSO-D}_6$) δ 7.60 (d, $J = 8.1$ Hz, 2H), 7.35-7.23 (m, 7H), 5.66 (s, 1H), 4.25-3.99 (m, 5H), 3.78 (d, $J = 15.3$ Hz, 1H), 2.46 (s, 1H), 2.32 (s, 3H).

13 (51 mg, 52%). Mp 178-180°C (diisopropylether). $^1\text{H-NMR}$ (300 MHz, CDCl_3) δ 7.46-7.26 (m, 15H), 5.99 (d, $J = 6.2$ Hz, 1H), 4.93 (s, 1H), 3.80 (ddt, $J = 9.5, 6.3, 1.75$ Hz, 1H), 3.69 (d, $J = 14.1$ Hz, 1H), 3.62 (d, $J = 12.9$ Hz, 1H), 3.57 (d, $J = 12.8$ Hz, 1H), 3.30 (t, $J = 9.4$ Hz, 1H), 3.09 (d, $J = 14.1$ Hz, 1H), 2.90 (s, 1H), 2.89 (d, $J = 24.8$ Hz, 1H), 2.06 (s, br, 1H), 1.99 (d, $J = 9.3$ Hz, 1H), 1.41 (dd, $J = 14.9, 9.9$ Hz, 1H). $^{13}\text{C-NMR}$ (75 MHz, CDCl_3) δ 179.5, 177.1, 145.6, 140.6, 138.7, 132.35, 129.1, 128.8, 128.4, 128.2, 128.0, 127.8, 127.7, 127.2, 126.4, 114.0, 75.7, 63.7, 59.9, 56.7, 48.95, 40.5, 38.24, 25.4. MS (EI) calcd. For $\text{C}_{30}\text{H}_{28}\text{N}_2\text{O}_3$ $[\text{M}+\text{H}]^+$: 464.20999, found: 464.2116.

14 (47 mg, 42%). $^1\text{H-NMR}$ (300 MHz, CDCl_3) δ 8.21 (d, $J = 8.7$ Hz, 2H), 7.59 (d, $J = 8.7$ Hz, 2H), 7.52-7.33 (m, 3H), 7.29-7.22 (m, 2H), 6.04 (dd, $J = 6.1, 1.9$ Hz, 1H), 4.54 (d, $J = 4$ Hz, 1H), 4.23-4.10 (m, 4H), 3.90-3.83 (m, 1H), 3.32 (t, $J = 8.6$ Hz, 1H), 3.05 (d, $J = 14.8$ Hz, 1H), 2.80 (d, $J = 15.4$ Hz,

1H), 2.58-2.52 (m, 1H), 2.50 (d, $J = 8.6$ Hz, 1H), 2.45 (d, $J = 15.4$ Hz, 1H), 1.71 (dd, $J = 14.8, 8.6$ Hz, 1H), 1.43 (s, 1H), 1.27-1.20 (m, 6H). ^{13}C -NMR (75 MHz, CDCl_3) δ 179.6, 176.3, 171.7, 171.2, 147.5, 147.2, 145.4, 131.9, 129.3, 128.9, 128.7, 126.2, 125.4, 123.2, 116.9, 61.9, 56.7, 48.7, 40.8, 40.5, 37.8, 29.7, 29.5, 14.0. MS (ESI) calcd. For $\text{C}_{30}\text{H}_{30}\text{N}_2\text{O}_9\text{Na}$ $[\text{M}+\text{Na}]^+$: 585.1849, found: 585.1851.

15 (39 mg, 52%). ^1H -NMR (300 MHz, CDCl_3) δ 7.50-7.22 (m, 10H), 6.07 (dt, $J = 6.1, 1.8$ Hz, 1H), 4.60 (s, 1H), 4.35 (s, 2H), 4.15 (d, $J = 9.1$ Hz, 1H), 3.93-3.83 (m, 1H), 3.36 (t, $J = 9.3$ Hz, 1H), 3.14 (d, $J = 10.1$ Hz, 1H), 2.96 (d, $J = 14.8$ Hz, 1H), 2.2 (m, 1H), 1.39 (dd, $J = 14.8, 9.3$ Hz, 1H). ^{13}C -NMR (75 MHz, CDCl_3) δ 179.2, 176.6, 144.7, 140.0, 132.2, 129.2, 128.5, 128.3, 128.1, 127.8, 126.4, 114.0, 76.8, 74.8, 69.8, 49.4, 40.5, 38.3, 24.2. MS (EI) calcd. For $\text{C}_{16}\text{H}_{15}\text{NO}_3$ $[\text{M}-\text{PhCHO}]^+$: 269.1051, found: 269.1067.

VI- Metathesis/Diels-Alder/oxidation sequence

To a solution of the enyne **1a** (75 mg, 0.2 mmol) in dry CH_2Cl_2 (5 mL) was added Grubbs catalyst (2nd generation) (8.5 mg, 0.01 mmol, 5 mol%). The solution was stirred for 15 h at room temperature under an argon atmosphere. The solvent was evaporated and the crude product was used in the next step without further purification. **16** was the only compound detected in the crude ^1H NMR. Due to its low stability on silica gel, no attempts were done to purify this diene.

16. ^1H -NMR (300 MHz, CDCl_3) δ 7.70 (d, $J = 8.1$ Hz, 2H), 7.31 (d, $J = 8.1$ Hz, 2H), 6.98 (d, $J = 18.4$ Hz, 1H), 5.72 (s, 1H), 5.33 (d, $J = 18.4$ Hz, 1H), 4.19 (s, 4H), 2.42 (s, 3H), 1.26 (s, 12H); ^{13}C -NMR (75 MHz, CDCl_3) δ 143.6, 141.6, 138.6, 133.9, 129.8, 127.4, 126.4, 119.5 (br), 83.5, 55.4, 53.4, 24.8, 21.5. MS (EI) calcd. for $\text{C}_{19}\text{H}_{26}\text{NO}_4$ ^{11}BS $[\text{M}+\text{H}]^+$: 375.1675, found: 375.1684.

The reaction was carried out in the same pot. At the end of the metathesis reaction, the solvent was evaporated and the residue was dissolved in dry toluene. N-phenyl maleimide (52 mg, 0.3 mmol, 1.5

eq) was added. The solution was stirred for 15 h at 80°C. After removing of volatiles, the residue was purified by silica gel chromatography (cyclohexane/ethyl acetate: 6/4) to afford **17**.

17 (77 mg, 70%, two steps). ¹H-NMR (300 MHz, CDCl₃) δ 7.70 (d, *J* = 8.2 Hz, 2H), 7.25-7.16 (m, 5H), 6.96 (d, *J* = 8.2 Hz, 2H), 5.98-6.05 (m, 1H), 3.90 (d, *J* = 13.8 Hz, 1H), 3.71 (dd *J* = 10.2, 8.4 Hz, 1H), 3.63-3.48 (m, 3H), 3.39 (t, *J* = 9.1 Hz, 1H), 2.88-2.78 (m, 1H), 2.43 (s, 3H), 1.80-1.72 (m, 1H), 1.28 (s, 12H). ¹³C-NMR (75 MHz, CDCl₃) δ 178.1, 175.7, 143.8, 138.7, 131.8, 131.6, 129.6, 128.9, 128.5, 128.2, 126.3, 121.3, 84.2, 50.8, 49.6, 43.8, 42.1, 37.5, 24.9, 24.8, 21.6. MS (EI) calcd. for C₂₆H₂₇N₂O₅¹¹BS [M-CH₃-COCH₃]⁺: 490.1733, found: 490.1714.

An aqueous solution of NaOAc (3M, 0.5 mL, 1.5 mmol) was added dropwise to a precooled solution (0°C) of the crude **17** (from 0.1 mmol of **1a**) in THF (5mL) and the temperature maintained below 5°C. Hydrogen peroxide (0.5 mL, 5.5 mmol) was added and the mixture was stirred at 0°C for 2h. Water (5 mL) was added, and the aqueous layer was extracted with ether (2x15 mL). The ether layers were washed with aqueous saturated solution of NaCl, dried over MgSO₄. After the solvent was removed, the residue was purified by silica gel chromatography (cyclohexane-ethyl acetate: 6/4).

18 (15 mg, 35%, three steps). ¹H-NMR (300 MHz, CDCl₃) δ 7.72 (d, *J* = 8.3 Hz, 2H), 7.45-7.28 (m, 5H), 7.04 (d, *J* = 8.3 Hz, 2H), 5.94 (s, 1H), 4.41 (m, 2H), 3.86 (d, *J* = 19.9 Hz, 1H), 3.75-3.61 (m, 3H), 3.51 (dd, *J* = 8.7, 5.3 Hz, 1H), 3.38 (t, *J* = 8.7 Hz, 1H), 2.81 (m, 1H), 2.06 (s, 3H). ¹³C-NMR (75 MHz, CDCl₃) δ 178.6, 174.7, 144.0, 137.1, 130.8, 129.7, 129.2, 129.0, 128.2, 126.2, 126.0, 67.8, 50.6, 49.0, 44.9, 39.7, 36.9, 24.9, 21.6. MS (EI) calcd. for C₁₆H₁₅N₂O₅ [M-CH₃C₆H₅SO₂]⁺: 283.1082, found: 283.1076.

VII- Platinum-catalyzed cycloisomerisation of **1a**

To a solution of boronate **1a** (150 mg, 0.54 mmol) in dry toluene (2 mL) was added platinum (II) chloride (5 mg, 0.02 mmol). The reaction was stirred one hour at 70°C. The volatiles were evaporated under *vacuo*. The residue was purified by silica gel chromatography (cyclohexane/ethyl acetate: 1/2).

19 (126 mg, 62%). $^1\text{H-NMR}$ (300 MHz, CDCl_3) δ 7.64 (d, $J = 8.1$ Hz, 2H), 7.31 (d, $J = 8.0$ Hz, 2H), 6.32 (d, $J = 8.0$ Hz, 1H), 5.35 (dd, $J = 5.35, 7.85$ Hz, 1H), 3.86 (d, $J = 11.6$ Hz, 1H), 3.10 (dd, $J = 2.7, 11.5$ Hz, 1H), 2.42 (s, 3H), 1.70 (t, $J = 6.1$ Hz, 1H), 1.33-1.24 (m, 2H), 1.19 (s, 12H). $^{13}\text{C-NMR}$ (75 MHz, CDCl_3) δ 143.7, 134.8, 129.8, 127.0, 121.3, 110.9, 83.5, 41.4, 24.8, 24.6, 23.4, 21.5, 13.3. MS (EI) calcd. for $\text{C}_{19}\text{H}_{26}\text{NO}_4$ ^{11}BS $[\text{M}+\text{H}]^+$: 375.16756, found: 375.1684.

VIII- X-ray structure of **5**

The crystal structure has been deposited at the CambridgeCrystallographic Data Centre and allocated the deposition number CCDC 635610

Summary of Data CCDC 635610

Formula: $\text{C}_{30}\text{H}_{28}\text{N}_2\text{O}_5\text{S}_1$

Unit cell parameters: a 9.0954(3) b 13.7732(4) c 13.8767(4) α 61.2960(10) β 78.0230(10) γ 77.9740(10) space group P-1

Current Data Parameters
NAME 137
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 2008106
Time 12.05
INSTRUM spect
PROBHD 5 mm BBO HX-1H
PULPROG zgpg30
TO 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 4456.403 Hz
FIDRES 0.137210 Hz
AQ 3.6438515 sec
RG 256
DA 111.250 sec
DE 5.00 usec
TE 300.2 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 8.50 usec
PL1 0.00 dB
SFO1 300.132508 MHz

F2 - Processing parameters
SI 16384
SF 300.130500 MHz
WDW EM
SSB 0
LA 0.25 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 28.50 cm
CT 20.00 cm
F1P 11.000 MHz
F2 320.42 MHz
F3 1.000 MHz
F4 -320.42 MHz
FREQ 0.330333 MHz
NUC1 1H
NUC2 13C

NAME	1a
SMILES	CC(C)(C)OC1(C)OC(C)(C)OC1/C=C/CNCC#C
EXP. COND.	200 MHz, CDCl ₃
EXP. TEMP.	25 °C
EXP. TIME	1.00 min
EXP. DATE	2023-03-10
EXP. USER	XXX
EXP. INSTR.	Agilent 1H NMR
EXP. PULP.	zgpg30
EXP. ACQ.	128
EXP. RES.	0.10 Hz
EXP. FWHM	0.10 Hz
EXP. S/N	100
EXP. T1	1.00 s
EXP. T2	1.00 s
EXP. T3	1.00 s
EXP. T4	1.00 s
EXP. T5	1.00 s
EXP. T6	1.00 s
EXP. T7	1.00 s
EXP. T8	1.00 s
EXP. T9	1.00 s
EXP. T10	1.00 s
EXP. T11	1.00 s
EXP. T12	1.00 s
EXP. T13	1.00 s
EXP. T14	1.00 s
EXP. T15	1.00 s
EXP. T16	1.00 s
EXP. T17	1.00 s
EXP. T18	1.00 s
EXP. T19	1.00 s
EXP. T20	1.00 s
EXP. T21	1.00 s
EXP. T22	1.00 s
EXP. T23	1.00 s
EXP. T24	1.00 s
EXP. T25	1.00 s
EXP. T26	1.00 s
EXP. T27	1.00 s
EXP. T28	1.00 s
EXP. T29	1.00 s
EXP. T30	1.00 s
EXP. T31	1.00 s
EXP. T32	1.00 s
EXP. T33	1.00 s
EXP. T34	1.00 s
EXP. T35	1.00 s
EXP. T36	1.00 s
EXP. T37	1.00 s
EXP. T38	1.00 s
EXP. T39	1.00 s
EXP. T40	1.00 s
EXP. T41	1.00 s
EXP. T42	1.00 s
EXP. T43	1.00 s
EXP. T44	1.00 s
EXP. T45	1.00 s
EXP. T46	1.00 s
EXP. T47	1.00 s
EXP. T48	1.00 s
EXP. T49	1.00 s
EXP. T50	1.00 s
EXP. T51	1.00 s
EXP. T52	1.00 s
EXP. T53	1.00 s
EXP. T54	1.00 s
EXP. T55	1.00 s
EXP. T56	1.00 s
EXP. T57	1.00 s
EXP. T58	1.00 s
EXP. T59	1.00 s
EXP. T60	1.00 s
EXP. T61	1.00 s
EXP. T62	1.00 s
EXP. T63	1.00 s
EXP. T64	1.00 s
EXP. T65	1.00 s
EXP. T66	1.00 s
EXP. T67	1.00 s
EXP. T68	1.00 s
EXP. T69	1.00 s
EXP. T70	1.00 s
EXP. T71	1.00 s
EXP. T72	1.00 s
EXP. T73	1.00 s
EXP. T74	1.00 s
EXP. T75	1.00 s
EXP. T76	1.00 s
EXP. T77	1.00 s
EXP. T78	1.00 s
EXP. T79	1.00 s
EXP. T80	1.00 s
EXP. T81	1.00 s
EXP. T82	1.00 s
EXP. T83	1.00 s
EXP. T84	1.00 s
EXP. T85	1.00 s
EXP. T86	1.00 s
EXP. T87	1.00 s
EXP. T88	1.00 s
EXP. T89	1.00 s
EXP. T90	1.00 s
EXP. T91	1.00 s
EXP. T92	1.00 s
EXP. T93	1.00 s
EXP. T94	1.00 s
EXP. T95	1.00 s
EXP. T96	1.00 s
EXP. T97	1.00 s
EXP. T98	1.00 s
EXP. T99	1.00 s
EXP. T100	1.00 s

```
Current Data Parameters
NAME          ch193
EXPNO        2
PROCNO       1
```

F2 - Acquisition Parameters
Date: 20050909

Time	10:23
Location	9000-1

Part No. 5 mm B80 B3-1H
PUFPO: 291030

TO SOLVENT	32760	DMC13
TO SOLVENT	32760	DMC13

११७

SN1	17985.611
F10NES	0.540077

AD	0.9110024
MS	11265.2

27.820
5.00

16	350.0
01	2.00000000

0.1	0.03500400
0.2	0.00202300

***** CHANNEL 14 *****

1991	1.35
1992	5.20

75.4756983

***** CHANNEL 12 *****

011110
M22
14

2000

00:02
20:00
00:00

0
1
2
3
4
5
6
7
8
9

12760
00248

100

	0	0.55	0.97
--	---	------	------

1.03
PC30 new plot parameters
25.50

CR	15.00
EXP	219,200

F1	16243.65
F20	-20.000

F2
POWDER
-1509.35
R 13435

520.0024) 1

205.50	cm
20.00	cm
215.20	ppm
16240.66	Hz
-22.000	ppm
-159.35	Hz
6.52515	ppm/Hz
240.00040	Hz/cm

Current Data Parameters
Name: 330
Date: 20160711
Time: 10:30
INSTRUM: spect
PROBHD: 5 mm BBO 90-1H
PULPROG: zgpg30
F4: 499.930
SOLVENT: CDCl3
NS: 512
DS: 2
SWH: 17966.811 Hz
FIDRES: 0.54687 Hz
AQ: 0.9110264 sec
RG: 2310.5
GB: 27.850 kHz
DE: 6.50 kHz
TE: 300.2 K
D1: 2.0000000 sec
S11: 0.0300000 sec
S12: 0.0000000 sec
===== CHANNEL f1 =====
NUC1: 13C
P1: 6.20 usec
PL1: -6.00 dB
SFO1: 75.4703263 MHz
===== CHANNEL f2 =====
PROBHD: msi125
NUC2: 1H
P2: 50.00 usec
PL2: -6.00 dB
SFO2: 299.9994705 MHz
===== Processing parameters =====
SI: 32768
SF: 75.467450 MHz
WDW: EM
SSB: 0
LB: 0.55 Hz
GB: 0
PC: 1.40
===== 2D NMR Data Parameters =====
CZ: 75.50 Hz
CY: 20.00 Hz
F1P: 215.800 MHz
F2P: 100.620 MHz
F2: -100.620 MHz
PCPD: 6.0000000 sec
NUCZ: 510.0000000 Hz

Current Data Parameters		F2 - Acquisition Parameters	
AME	151.1	Date	20010119
EXPNO	1	Time	15.27
PROCNO	1	INSTRUM	SPCKIT
		PROBHD	5 mm BBO BB-H1
		PULPROG	zgpg30
		TD	65536
		SOLVENT	D2O
		NS	256
		DS	2
		SWH	11700.811 Hz
		FIDRES	0.040077 Hz
		RES	0.0151004 Hz
		PC	16.664
		GC	6.000 sec
		DE	6.000 sec
		TE	300.2 K
		DELTA	2.0000000 sec
		DELTA2	6.0000000 sec
		DELTA3	0.00012000 sec
		DELTA4	0.00012000 sec

===== CHANNEL f1 =====	
NUC1	13C
MASS1	13.004353
RG1	-6.500000
RG2	-6.500000
RG3	75.0752800 MHz
RG4	
RG5	

===== CHANNEL f2 =====	
CPDPRG2	zgpg30
NUC2	1H
MASS2	1.00614619
RG2C2	80.000000
RG2C3	-6.000000
RG2C4	20.000000
RG2C5	20.000000
RG2C6	20.000000
RG2C7	300.1352000 MHz
RG2C8	
RG2C9	
RG2C10	
RG2C11	
RG2C12	
RG2C13	
RG2C14	
RG2C15	
RG2C16	
RG2C17	
RG2C18	
RG2C19	
RG2C20	
RG2C21	
RG2C22	
RG2C23	
RG2C24	
RG2C25	
RG2C26	
RG2C27	
RG2C28	
RG2C29	
RG2C30	
RG2C31	
RG2C32	
RG2C33	
RG2C34	
RG2C35	
RG2C36	
RG2C37	
RG2C38	
RG2C39	
RG2C40	
RG2C41	
RG2C42	
RG2C43	
RG2C44	
RG2C45	
RG2C46	
RG2C47	
RG2C48	
RG2C49	
RG2C50	
RG2C51	
RG2C52	
RG2C53	
RG2C54	
RG2C55	
RG2C56	
RG2C57	
RG2C58	
RG2C59	
RG2C60	
RG2C61	
RG2C62	
RG2C63	
RG2C64	
RG2C65	
RG2C66	
RG2C67	
RG2C68	
RG2C69	
RG2C70	
RG2C71	
RG2C72	
RG2C73	
RG2C74	
RG2C75	
RG2C76	
RG2C77	
RG2C78	
RG2C79	
RG2C80	
RG2C81	
RG2C82	
RG2C83	
RG2C84	
RG2C85	
RG2C86	
RG2C87	
RG2C88	
RG2C89	
RG2C90	
RG2C91	
RG2C92	
RG2C93	
RG2C94	
RG2C95	
RG2C96	
RG2C97	
RG2C98	
RG2C99	
RG2C100	

10 MHz offset parameters	
CX	25.00 MHz
CY	20.00 MHz
CZ	20.00 MHz
F1	255.135 MHz
F2	500.135 MHz
F3	500.135 MHz
F4	500.135 MHz
F5	500.135 MHz
F6	500.135 MHz
F7	500.135 MHz
F8	500.135 MHz
F9	500.135 MHz
F10	500.135 MHz
F11	500.135 MHz
F12	500.135 MHz
F13	500.135 MHz
F14	500.135 MHz
F15	500.135 MHz
F16	500.135 MHz

S-32

Current Data Parameters
 Name: 7
 ExpNo: 1
 Process: 1

F2 - Acquisition Parameters
 Date_: 20070215
 Time: 11:04
 INSTRUM: spect
 PROCNO: 1
 F1: 400.136 MHz
 F2: 100.628 MHz
 SOLVENT: CDCl3
 NS: 150
 DS: 2
 SWH: 6172.839 Hz
 FIDRES: 0.18760 Hz
 AQ: 1.207346 sec
 RG: 655.5
 DA: 16.1024 sec
 DE: 6.00 dB
 TE: 300.2 K
 D1: 1.00000000 sec

===== CHANNEL f1 =====
 NUC1: 13C
 P1: 0.50 sec
 PL1: 0.00 dB
 SFO1: 100.628344 MHz

F2 - Processing parameters
 SI: 32768
 SF: 100.628344 MHz
 WF: 32768
 SSF: 0
 SSB: 0
 LB: 0.30 Hz
 GB: 0
 PC: 1.00

10 MHz 0.101 parameters
 C4: 35.51 Hz
 C5: 20.00 Hz
 F1P: 41.000 000
 F1: 3361.43 Hz
 F2P: -1.000 000
 F2: -320.13 Hz
 PPM0N: 0.33803 ppm/Hz
 HZ0N: 101.45345 Hz/Hz

```

Current Data Parameters
NAME          20 31
EPRM         1
PSSCM        1

F2 - Acquisition Parameters
Date_        20/02/14
Time         16 09
Unit         1001
INSTRUM      1MS100
PROBHD      5 mm BBO 80-1H
PULPROG      zgpg30
TD          32768
SOLVENT      CDCl3
NS           6
DS           2
SWH          6372.695 Hz
FIDRES       0.376760 Hz
AQ           1.2671540 sec
RG           382
OR           61.050 usec
DE           6.05 usec
TE           300.2 K
D1           1.00000000 sec

***** CHANNEL f1 *****
NUC1         1H
P1           8.50 usec
PL1          0.50 dB
SFO1        300.1156131 Mhz

F2 - Processing Parameters
SI           1024
SF          300.1300600 Mhz
WDW          EM
SSB          0
GB           0
LB           0.30 Hz
GB           0
PC           1.05

10 MHz p201 parameters
CT          20.00 Hz
F1P         11.000 014
F2P         11.000 014
F3P         11.000 014
F4P         11.000 014
F5P         11.000 014
F6P         11.000 014
F7P         11.000 014
F8P         11.000 014
F9P         11.000 014
F10P        11.000 014
F11P        11.000 014
F12P        11.000 014
F13P        11.000 014
F14P        11.000 014
F15P        11.000 014
F16P        11.000 014
F17P        11.000 014
F18P        11.000 014
F19P        11.000 014
F20P        11.000 014
F21P        11.000 014
F22P        11.000 014
F23P        11.000 014
F24P        11.000 014
F25P        11.000 014
F26P        11.000 014
F27P        11.000 014
F28P        11.000 014
F29P        11.000 014
F30P        11.000 014
F31P        11.000 014
F32P        11.000 014
F33P        11.000 014
F34P        11.000 014
F35P        11.000 014
F36P        11.000 014
F37P        11.000 014
F38P        11.000 014
F39P        11.000 014
F40P        11.000 014
F41P        11.000 014
F42P        11.000 014
F43P        11.000 014
F44P        11.000 014
F45P        11.000 014
F46P        11.000 014
F47P        11.000 014
F48P        11.000 014
F49P        11.000 014
F50P        11.000 014
F51P        11.000 014
F52P        11.000 014
F53P        11.000 014
F54P        11.000 014
F55P        11.000 014
F56P        11.000 014
F57P        11.000 014
F58P        11.000 014
F59P        11.000 014
F60P        11.000 014
F61P        11.000 014
F62P        11.000 014
F63P        11.000 014
F64P        11.000 014
F65P        11.000 014
F66P        11.000 014
F67P        11.000 014
F68P        11.000 014
F69P        11.000 014
F70P        11.000 014
F71P        11.000 014
F72P        11.000 014
F73P        11.000 014
F74P        11.000 014
F75P        11.000 014
F76P        11.000 014
F77P        11.000 014
F78P        11.000 014
F79P        11.000 014
F80P        11.000 014
F81P        11.000 014
F82P        11.000 014
F83P        11.000 014
F84P        11.000 014
F85P        11.000 014
F86P        11.000 014
F87P        11.000 014
F88P        11.000 014
F89P        11.000 014
F90P        11.000 014
F91P        11.000 014
F92P        11.000 014
F93P        11.000 014
F94P        11.000 014
F95P        11.000 014
F96P        11.000 014
F97P        11.000 014
F98P        11.000 014
F99P        11.000 014
F100P       11.000 014

```

Current Data Parameters
 NAME: 75431-2
 EXPNO: 1
 PROCNO: 1
 F2 - Acquisition Parameters
 Date_: 20070215
 Time: 18.30
 INSTRUM: spect
 PULPROG: zgpg30
 PALETTE: 5
 TO: 10.000
 SOLVENT: DMSO
 NS: 128
 DS: 2
 SWH: 6172.834 Hz
 FIDRES: 0.197500 Hz
 AQ: 1.587500 sec
 RG: 724.1
 DQ: 61.000 usec
 DE: 391.0 Hz
 TE: 300.2 K
 DT: 1.0000000 sec
 ***** CHANNEL f1 *****
 NUC1: 1H
 P1: 8.50 usec
 PL1: 0.00 dB
 SC1: 300.131814 MHz
 F2 - Processing parameters
 SI: 32768
 SF: 300.131814 MHz
 WDW: EM
 SSF: 0
 LB: 0.30 Hz
 GB: 0
 PC: 1.00
 10 MHz D101 parameters
 CX: 30.50 cm
 CY: 15.50 cm
 F1P: 51.000 MHz
 F2P: 320.145 MHz
 F2R: -1.000 MHz
 P1PDM: 0.33000 sec/Hz
 N2CH: 101.42041 Hz/Hz

Current Data Parameters
 Name 1b12
 ExpNo 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20070220
 Time 3.55
 INSTRUM spect
 NUC1 13C
 PULPROG zgpg30
 TO 30768
 SOLVENT C0C13
 NS 1200
 DS 2
 FWH 12985.811 Hz
 FIDRES 0.548377 Hz
 AQ 0.9110024 sec
 RG 8192
 DE 27.800 uV
 TE 300.2 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 D12 0.0002000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 6.20 uV
 PL1 -6.00 dB
 SFO1 75.4752653 MHz

===== CHANNEL f2 =====
 PROCNO2 1
 NUC2 1H
 P2 0.00 uV
 PL2 -6.00 dB
 SFO2 400.1434700 MHz

F2 - Processing parameters
 SI 32768
 SF 75.46815776 MHz
 NQW 0
 EQ 0
 LB 0.55 Hz
 GB 0
 PC 1.40

1D 1H NMR 400 MHz
 C1 30.50 CH
 C7 220.00 CH
 F10 215.815 CH
 F1 15.538.45 Hz
 F2 -13.707.600 Hz
 F2 -1.487.24 Hz
 FWHM 0.71329 CH/CH
 GCW 506.63360 Hz/Hz

1H NMR (400 MHz, CDCl3) δ 7.245 (d, 1H), 7.238 (d, 1H), 7.230 (d, 1H), 7.228 (d, 1H), 7.224 (d, 1H), 7.220 (d, 1H), 7.213 (d, 1H), 7.206 (d, 1H), 7.205 (d, 1H), 7.202 (d, 1H), 7.200 (d, 1H), 7.193 (d, 1H), 7.187 (d, 1H), 7.184 (d, 1H), 7.182 (d, 1H), 6.014 (d, 1H), 5.995 (d, 1H), 5.976 (d, 1H), 5.183 (d, 1H), 4.367 (d, 1H), 4.350 (d, 1H), 4.333 (d, 1H), 4.316 (d, 1H), 4.299 (d, 1H), 4.282 (d, 1H), 4.265 (d, 1H), 4.248 (d, 1H), 4.231 (d, 1H), 4.214 (d, 1H), 4.197 (d, 1H), 4.180 (d, 1H), 4.163 (d, 1H), 4.146 (d, 1H), 4.129 (d, 1H), 4.112 (d, 1H), 4.095 (d, 1H), 4.078 (d, 1H), 4.061 (d, 1H), 4.044 (d, 1H), 4.027 (d, 1H), 4.010 (d, 1H), 3.993 (d, 1H), 3.976 (d, 1H), 3.959 (d, 1H), 3.942 (d, 1H), 3.925 (d, 1H), 3.908 (d, 1H), 3.891 (d, 1H), 3.874 (d, 1H), 3.857 (d, 1H), 3.840 (d, 1H), 3.823 (d, 1H), 3.806 (d, 1H), 3.789 (d, 1H), 3.772 (d, 1H), 3.755 (d, 1H), 3.738 (d, 1H), 3.721 (d, 1H), 3.704 (d, 1H), 3.687 (d, 1H), 3.670 (d, 1H), 3.653 (d, 1H), 3.636 (d, 1H), 3.619 (d, 1H), 3.602 (d, 1H), 3.585 (d, 1H), 3.568 (d, 1H), 3.551 (d, 1H), 3.534 (d, 1H), 3.517 (d, 1H), 3.500 (d, 1H), 3.483 (d, 1H), 3.466 (d, 1H), 3.449 (d, 1H), 3.432 (d, 1H), 3.415 (d, 1H), 3.398 (d, 1H), 3.381 (d, 1H), 3.364 (d, 1H), 3.347 (d, 1H), 3.330 (d, 1H), 3.313 (d, 1H), 3.296 (d, 1H), 3.279 (d, 1H), 3.262 (d, 1H), 3.245 (d, 1H), 3.228 (d, 1H), 3.211 (d, 1H), 3.194 (d, 1H), 3.177 (d, 1H), 3.160 (d, 1H), 3.143 (d, 1H), 3.126 (d, 1H), 3.109 (d, 1H), 3.092 (d, 1H), 3.075 (d, 1H), 3.058 (d, 1H), 3.041 (d, 1H), 3.024 (d, 1H), 3.007 (d, 1H), 2.990 (d, 1H), 2.973 (d, 1H), 2.956 (d, 1H), 2.939 (d, 1H), 2.922 (d, 1H), 2.905 (d, 1H), 2.888 (d, 1H), 2.871 (d, 1H), 2.854 (d, 1H), 2.837 (d, 1H), 2.820 (d, 1H), 2.803 (d, 1H), 2.786 (d, 1H), 2.769 (d, 1H), 2.752 (d, 1H), 2.735 (d, 1H), 2.718 (d, 1H), 2.701 (d, 1H), 2.684 (d, 1H), 2.667 (d, 1H), 2.650 (d, 1H), 2.633 (d, 1H), 2.616 (d, 1H), 2.599 (d, 1H), 2.582 (d, 1H), 2.565 (d, 1H), 2.548 (d, 1H), 2.531 (d, 1H), 2.514 (d, 1H), 2.497 (d, 1H), 2.480 (d, 1H), 2.463 (d, 1H), 2.446 (d, 1H), 2.429 (d, 1H), 2.412 (d, 1H), 2.395 (d, 1H), 2.378 (d, 1H), 2.361 (d, 1H), 2.344 (d, 1H), 2.327 (d, 1H), 2.310 (d, 1H), 2.293 (d, 1H), 2.276 (d, 1H), 2.259 (d, 1H), 2.242 (d, 1H), 2.225 (d, 1H), 2.208 (d, 1H), 2.191 (d, 1H), 2.174 (d, 1H), 2.157 (d, 1H), 2.140 (d, 1H), 2.123 (d, 1H), 2.106 (d, 1H), 2.089 (d, 1H), 2.072 (d, 1H), 2.055 (d, 1H), 2.038 (d, 1H), 2.021 (d, 1H), 2.004 (d, 1H), 1.987 (d, 1H), 1.970 (d, 1H), 1.953 (d, 1H), 1.936 (d, 1H), 1.919 (d, 1H), 1.902 (d, 1H), 1.885 (d, 1H), 1.868 (d, 1H), 1.851 (d, 1H), 1.834 (d, 1H), 1.817 (d, 1H), 1.800 (d, 1H), 1.783 (d, 1H), 1.766 (d, 1H), 1.749 (d, 1H), 1.732 (d, 1H), 1.715 (d, 1H), 1.698 (d, 1H), 1.681 (d, 1H), 1.664 (d, 1H), 1.647 (d, 1H), 1.630 (d, 1H), 1.613 (d, 1H), 1.596 (d, 1H), 1.579 (d, 1H), 1.562 (d, 1H), 1.545 (d, 1H), 1.528 (d, 1H), 1.511 (d, 1H), 1.494 (d, 1H), 1.477 (d, 1H), 1.460 (d, 1H), 1.443 (d, 1H), 1.426 (d, 1H), 1.409 (d, 1H), 1.392 (d, 1H), 1.375 (d, 1H), 1.358 (d, 1H), 1.341 (d, 1H), 1.324 (d, 1H), 1.307 (d, 1H), 1.290 (d, 1H), 1.273 (d, 1H), 1.256 (d, 1H), 1.239 (d, 1H), 1.222 (d, 1H), 1.205 (d, 1H), 1.188 (d, 1H), 1.171 (d, 1H), 1.154 (d, 1H), 1.137 (d, 1H), 1.120 (d, 1H), 1.103 (d, 1H), 1.086 (d, 1H), 1.069 (d, 1H), 1.052 (d, 1H), 1.035 (d, 1H), 1.018 (d, 1H), 1.001 (d, 1H), 0.984 (d, 1H), 0.967 (d, 1H), 0.950 (d, 1H), 0.933 (d, 1H), 0.916 (d, 1H), 0.899 (d, 1H), 0.882 (d, 1H), 0.865 (d, 1H), 0.848 (d, 1H), 0.831 (d, 1H), 0.814 (d, 1H), 0.797 (d, 1H), 0.780 (d, 1H), 0.763 (d, 1H), 0.746 (d, 1H), 0.729 (d, 1H), 0.712 (d, 1H), 0.695 (d, 1H), 0.678 (d, 1H), 0.661 (d, 1H), 0.644 (d, 1H), 0.627 (d, 1H), 0.610 (d, 1H), 0.593 (d, 1H), 0.576 (d, 1H), 0.559 (d, 1H), 0.542 (d, 1H), 0.525 (d, 1H), 0.508 (d, 1H), 0.491 (d, 1H), 0.474 (d, 1H), 0.457 (d, 1H), 0.440 (d, 1H), 0.423 (d, 1H), 0.406 (d, 1H), 0.389 (d, 1H), 0.372 (d, 1H), 0.355 (d, 1H), 0.338 (d, 1H), 0.321 (d, 1H), 0.304 (d, 1H), 0.287 (d, 1H), 0.270 (d, 1H), 0.253 (d, 1H), 0.236 (d, 1H), 0.219 (d, 1H), 0.202 (d, 1H), 0.185 (d, 1H), 0.168 (d, 1H), 0.151 (d, 1H), 0.134 (d, 1H), 0.117 (d, 1H), 0.100 (d, 1H), 0.083 (d, 1H), 0.066 (d, 1H), 0.049 (d, 1H), 0.032 (d, 1H), 0.015 (d, 1H), 0.000 (d, 1H).

Current Data Parameters
NAME 3p-cl3cpd256
EXPNO 1
PROCNO 1
P2 - Acquisition Parameters
Date_ 20061107
Time 23.04
INSTRUM spect
PROBHD 5 mm BBO-BB-1H
PULPROG zgpg30
TD 32768
FIDRES 0.32768
SOLVENT CDCl3
NS 6000
DS 2
SWH 37983.611 Hz
F2 - 127.68 MHz
AQ 0.345877 Hz
RG 0.3110204 sec
B0 500.136050 MHz
P1 13.00 usec
PL1 0.00 dB
PC 77.00 usec
PL2 0.00 dB
TE 300.0 K
DT 1.0000000 sec
d11 0.0300000 sec
d12 0.0002000 sec
===== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
PC1 75.4752553 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -6.00 dB
PUL2 20.00 dB
PL3 20.00 dB
PC3 300.1312005 MHz
F3 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
GB 0.55 Hz
MC 1.40

Current Data Parameters

NAME	14
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

DATE_	20100125
TIME	15.32
INSTRUM	SXCI
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	65536
SF	500.13
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

F2 - Processing parameters

SI	32768
SD	32768
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR FID parameters

CH	20.55 cm
CH	15.05 cm
F2	11.005 ppm
F1	3301.43 Hz
F2	-1.005 ppm
F1	-3301.43 Hz
PRGCM	0.338613 sec/cm
RGCM	101.45041 sec/cm

```

Current Data Parameters
NAME          0023-1
PNO          1
EXPNO        1

===== CHANNEL 1: =====
F2 - Acquisition Parameters
Date_         20160508
Time          16:49
Observer      50680
INSTRUM       spect
PROBHD        1H1
PULPROG       zgpg30
TD            65536
SFO           400.136
AQ            0.0123
RG            326
DS            2
SWH            17880.41 Hz
FIDRES        0.158873 Hz
AQRES         0.0170004 sec
RGRES         10.8384
SC            10.8384
PC            27.860
PC2           6.00
PC3           320.0 Hz
PC4            2.00
PC5            2.00
PC6            2.00
PC7            2.00
PC8            2.00
PC9            2.00
PC10           2.00
PC11           2.00
PC12           2.00
PC13           2.00
PC14           2.00
PC15           2.00
PC16           2.00
PC17           2.00
PC18           2.00
PC19           2.00
PC20           2.00
PC21           2.00
PC22           2.00
PC23           2.00
PC24           2.00
PC25           2.00
PC26           2.00
PC27           2.00
PC28           2.00
PC29           2.00
PC30           2.00
PC31           2.00
PC32           2.00
PC33           2.00
PC34           2.00
PC35           2.00
PC36           2.00
PC37           2.00
PC38           2.00
PC39           2.00
PC40           2.00
PC41           2.00
PC42           2.00
PC43           2.00
PC44           2.00
PC45           2.00
PC46           2.00
PC47           2.00
PC48           2.00
PC49           2.00
PC50           2.00
PC51           2.00
PC52           2.00
PC53           2.00
PC54           2.00
PC55           2.00
PC56           2.00
PC57           2.00
PC58           2.00
PC59           2.00
PC60           2.00
PC61           2.00
PC62           2.00
PC63           2.00
PC64           2.00
PC65           2.00
PC66           2.00
PC67           2.00
PC68           2.00
PC69           2.00
PC70           2.00
PC71           2.00
PC72           2.00
PC73           2.00
PC74           2.00
PC75           2.00
PC76           2.00
PC77           2.00
PC78           2.00
PC79           2.00
PC80           2.00
PC81           2.00
PC82           2.00
PC83           2.00
PC84           2.00
PC85           2.00
PC86           2.00
PC87           2.00
PC88           2.00
PC89           2.00
PC90           2.00
PC91           2.00
PC92           2.00
PC93           2.00
PC94           2.00
PC95           2.00
PC96           2.00
PC97           2.00
PC98           2.00
PC99           2.00
PC100          2.00
PC101          2.00
PC102          2.00
PC103          2.00
PC104          2.00
PC105          2.00
PC106          2.00
PC107          2.00
PC108          2.00
PC109          2.00
PC110          2.00
PC111          2.00
PC112          2.00
PC113          2.00
PC114          2.00
PC115          2.00
PC116          2.00
PC117          2.00
PC118          2.00
PC119          2.00
PC120          2.00
PC121          2.00
PC122          2.00
PC123          2.00
PC124          2.00
PC125          2.00
PC126          2.00
PC127          2.00
PC128          2.00
PC129          2.00
PC130          2.00
PC131          2.00
PC132          2.00
PC133          2.00
PC134          2.00
PC135          2.00
PC136          2.00
PC137          2.00
PC138          2.00
PC139          2.00
PC140          2.00
PC141          2.00
PC142          2.00
PC143          2.00
PC144          2.00
PC145          2.00
PC146          2.00
PC147          2.00
PC148          2.00
PC149          2.00
PC150          2.00
PC151          2.00
PC152          2.00
PC153          2.00
PC154          2.00
PC155          2.00
PC156          2.00
PC157          2.00
PC158          2.00
PC159          2.00
PC160          2.00
PC161          2.00
PC162          2.00
PC163          2.00
PC164          2.00
PC165          2.00
PC166          2.00
PC167          2.00
PC168          2.00
PC169          2.00
PC170          2.00
PC171          2.00
PC172          2.00
PC173          2.00
PC174          2.00
PC175          2.00
PC176          2.00
PC177          2.00
PC178          2.00
PC179          2.00
PC180          2.00
PC181          2.00
PC182          2.00
PC183          2.00
PC184          2.00
PC185          2.00
PC186          2.00
PC187          2.00
PC188          2.00
PC189          2.00
PC190          2.00
PC191          2.00
PC192          2.00
PC193          2.00
PC194          2.00
PC195          2.00
PC196          2.00
PC197          2.00
PC198          2.00
PC199          2.00
PC200          2.00
PC201          2.00
PC202          2.00
PC203          2.00
PC204          2.00
PC205          2.00
PC206          2.00
PC207          2.00
PC208          2.00
PC209          2.00
PC210          2.00
PC211          2.00
PC212          2.00
PC213          2.00
PC214          2.00
PC215          2.00
PC216          2.00
PC217          2.00
PC218          2.00
PC219          2.00
PC220          2.00
PC221          2.00
PC222          2.00
PC223          2.00
PC224          2.00
PC225          2.00
PC226          2.00
PC227          2.00
PC228          2.00
PC229          2.00
PC230          2.00
PC231          2.00
PC232          2.00
PC233          2.00
PC234          2.00
PC235          2.00
PC236          2.00
PC237          2.00
PC238          2.00
PC239          2.00
PC240          2.00
PC241          2.00
PC242          2.00
PC243          2.00
PC244          2.00
PC245          2.00
PC246          2.00
PC247          2.00
PC248          2.00
PC249          2.00
PC250          2.00
PC251          2.00
PC252          2.00
PC253          2.00
PC254          2.00
PC255          2.00
PC256          2.00
PC257          2.00
PC258          2.00
PC259          2.00
PC260          2.00
PC261          2.00
PC262          2.00
PC263          2.00
PC264          2.00
PC265          2.00
PC266          2.00
PC267          2.00
PC268          2.00
PC269          2.00
PC270          2.00
PC271          2.00
PC272          2.00
PC273          2.00
PC274          2.00
PC275          2.00
PC276          2.00
PC277          2.00
PC278          2.00
PC279          2.00
PC280          2.00
PC281          2.00
PC282          2.00
PC283          2.00
PC284          2.00
PC285          2.00
PC286          2.00
PC287          2.00
PC288          2.00
PC289          2.00
PC290          2.00
PC291          2.00
PC292          2.00
PC293          2.00
PC294          2.00
PC295          2.00
PC296          2.00
PC297          2.00
PC298          2.00
PC299          2.00
PC300          2.00
PC301          2.00
PC302          2.00
PC303          2.00
PC304          2.00
PC305          2.00
PC306          2.00
PC307          2.00
PC308          2.00
PC309          2.00
PC310          2.00
PC311          2.00
PC312          2.00
PC313          2.00
PC314          2.00
PC315          2.00
PC316          2.00
PC317          2.00
PC318          2.00
PC319          2.00
PC320          2.00
PC321          2.00
PC322          2.00
PC323          2.00
PC324          2.00
PC325          2.00
PC326          2.00
PC327          2.00
PC328          2.00
PC329          2.00
PC330          2.00
PC331          2.00
PC332          2.00
PC333          2.00
PC334          2.00
PC335          2.00
PC336          2.00
PC337          2.00
PC338          2.00
PC339          2.00
PC340          2.00
PC341          2.00
PC342          2.00
PC343          2.00
PC344          2.00
PC345          2.00
PC346          2.00
PC347          2.00
PC348          2.00
PC349          2.00
PC350          2.00
PC351          2.00
PC352          2.00
PC353          2.00
PC354          2.00
PC355          2.00
PC356          2.00
PC357          2.00
PC358          2.00
PC359          2.00
PC360          2.00
PC361          2.00
PC362          2.00
PC363          2.00

```

```

Current Data Parameters
NAME          an-23-1
EXPNO         1
PROCNO        1

F2 - Acquisition Parameters
Date_         20101213
Time          19.30
INSTRUM       spect
PROBHD        5 mm BBO BBH
PULPROG       zgpg30
TD            65536
SOLVENT       DMSO
NS            2
DS            4
SWH           17000.00 Hz
FIDRES        0.54047 Hz
AQ            0.9110240 sec
RG            1538.4
DE            27.000 uSec
TE            300.2 K
DW            8.00 nSec
DECT          0.000000 sec
O1            0.0000000 sec
O2            0.0000000 sec

***** CHANNEL f1 *****
NUC1           13C
P1             6.20 uSec
PL1            -6.00 dB
SFO1           75.47555 MHz

***** CHANNEL f2 *****
DISPENS       1H
NUC2           1H
P2             80.00 uSec
PL2            -6.00 dB
SFO2           200.00 MHz
RG2            20.00 dB
SFO3           200.00 MHz
SFO4           300.134200 MHz

F3 - Processing parameters
SI            32768
SF            75.467000 MHz
WDW           EM
SSB            0
LB             0.05 Hz
GB             0.00 Hz
PC            1.40

3D Map Data Parameters
CX            35.50 cm
CY            25.00 cm
CZ            25.00 cm
F1P           215.200 MHz
F1           16500.00 Hz
F2P           -190.00 MHz
F2           -190.23 Hz
F3P           -100.23 Hz
F3           0.0000000 MHz
SFO           300.134200 MHz
SFO2          200.000000 MHz

```