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# An oxidative Hosomi-Sakurai strategy toward the synthesis of illioliganones B and C

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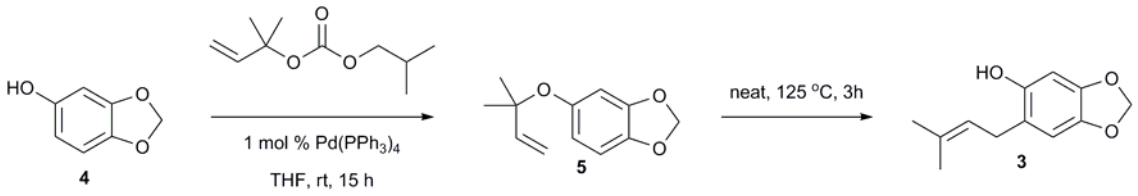
## Contents

**General.** $^{1}$H NMR spectra were recorded at 400 or 500 MHz. $^{13}$C NMR spectra were recorded at 100 or 125 MHz. Chemical shifts are reported in ppm from tetramethylsilane with the solvent resonance as the internal standard ($CDCl_3$: 7.26 ppm). Data are reported as follows: chemical shift, integration, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, br = broad, m = multiplet), and coupling constants (Hz). $^{13}$C NMR were recorded with complete proton decoupling. Chemical shifts are reported in ppm from tetramethylsilane with the solvent as the internal standard ($CDCl_3$: 77.4 ppm). Mass spectrometry (m/z) was performed in ESI mode, with only molecular ions being reported. Infrared (IR) spectra $\nu_{max}$ are reported in cm$^{-1}$. Bands are characterised as broad (br), strong (s), medium (m) and weak (w). All purchased reagents were used as received without further purification. THF was pre-dried with 3A molecular sieves then distilled from sodium benzophenone ketyl. Petroleum ether refers to the fraction boiling at 40-60 $^{o}$C. Isobutyl 2-methylbut-3-en-2-yl carbonate was prepared according to a literature method.[1]

**Synthesis of 6-(3-methylbut-2-enyl)benzo[*d*][1,3]dioxol-5-ol, 3**




Isobutyl 2-methylbut-3-en-2-yl carbonate (3.28 g, 18 mmol, 1.3 equiv) and sesamol (1.87 g, 14 mmol, 1 equiv) were dissolved in THF (50 mL) at room temperature under a nitrogen atmosphere. Tetrakistriphenylphosphine palladium(0) (162 mg, 0.14 mmol, 0.01 equiv) was added in one portion and the mixture was stirred for 15h. The reaction mixture was concentrated, then diluted with 20:1 petroleum ether/ethyl acetate and filtered through celite to remove the palladium salts. The resulting solution was concentrated and then heated neat at 125 $^{o}$C for 3 h. The mixture was purified by flash chromatography on silica gel using 20:1 petroleum ether/ethyl acetate to furnish **3** as a colourless oil (2.6 g, 90%).

IR (neat): 1036 (s), 1164 (s), 1439 (m), 1481 (m), 1503 (m), 3452 (br) cm$^{-1}$.

$^{1}$H NMR (400 MHz, $CDCl_3$): $\delta$ 1.76 (6H, s), 3.24 (2H, d, $J$ = 7.2 Hz), 4.96 (1H, s), 5.22-5.29 (1H, m), 5.86 (2H, s), 6.41 (1H, s), 6.57 (1H, s).

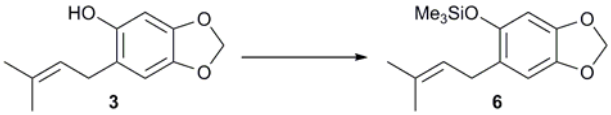
$^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 18.2, 26.2, 30.0, 98.9, 101.3, 109.5, 118.8, 122.2, 135.3, 141.6, 146.7, 149.1.

MS: m/z (M-1) 205.1

HRMS: m/z calc'd for $C_{12}H_{13}O_3$ 205.0870, found 205.0867.

[1] B. Plietker, A. Dieskau, K. Möws and A. Jatsch, *Angew. Chem., Int. Ed.* 2008, **47**, 198.

**Synthesis of trimethyl(6-(3-methylbut-2-enyl)benzo[*d*][1,3]dioxol-5-yloxy)silane, 6**




6-(3-Methylbut-2-enyl)benzo[*d*][1,3]dioxol-5-ol (270 mg, 1.3 mmol, 1 equiv), **3**, was dissolved in THF (2.4 mL) at room temperature under a nitrogen atmosphere. Triethylamine (0.49 mL, 3.5 mmol, 2.4 equiv), followed by chlorotrimethylsilane (0.48 mL, 3.8 mmol, 2.6 equiv), was added slowly to the reaction mixture. After 1 h, the crude mixture was filtered through celite, washed through with petroleum ether, and concentrated. If any triethylaminehydrochloride salt remained, 20:1 petroleum ether/ethyl acetate was added to the sample which was then filtered again and concentrated. The product, **6**, was isolated as a clear pale yellow oil (370 mg, 100%) which was taken through to the next step without further purification.

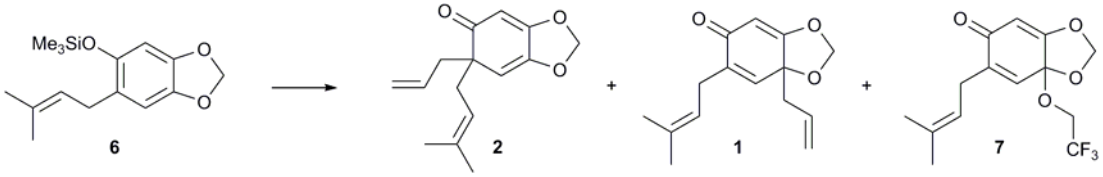
IR (neat): 1157 (s), 1182 (s), 1479 (s), 2962 (w) $cm^{-1}$.

$^{1}H$ NMR (400 MHz, $CDCl_3$): δ 0.26 (9H, s), 1.70 (3H, s), 1.74 (3H, s), 3.19 (2H, d, $J$ = 7.3 Hz), 5.19-5.29 (1H, m), 5.87 (2H, s), 6.38 (1H, s), 6.62 (1H, s).

$^{13}C$ NMR (100 MHz, $CDCl_3$): δ 0.72, 18.1, 26.1, 28.7, 101.2, 101.7, 109.3, 123.3, 124.7, 132.7, 142.0, 145.9, 147.3.

MS: unable to obtain MS data because of lability of O-Si bond.

**Procedure for the oxidative Hosomi-Sakurai reaction**




Trimethyl(6-(3-methylbut-2-enyl)benzo[*d*][1,3]dioxol-5-yloxy)silane, **6** (45 mg, 0.16 mmol, 1 equiv) was dissolved in trifluoroethanol (1 mL) at room temperature and stirred open to air. Sodium bicarbonate (40 mg, 0.48 mmol, 3 equiv) was added, followed by allyltrimethylsilane (0.039 mL, 0.24 mmol, 1.5 equiv). Iodobenzene diacetate (77 mg, 0.24 mmol, 1.5 equiv) was dissolved in trifluoroethanol (0.5 mL) and added over 10 seconds to the reaction mixture. After stirring the dark red solution for about 1 hour, saturated aqueous sodium bicarbonate solution was added and the mixture was extracted with ethyl acetate. This was dried over $MgSO_4$, filtered, concentrated, then purified by flash chromatography on silica gel with 20:1 to 5:1 petroleum ether/ethyl acetate to furnish three compounds as colourless oils (**2**: 15 mg, 38%; **1**: 3 mg, 8%, **7**: 10 mg, 21%).

Data for 6-allyl-6-(3-methylbut-2-enyl)benzo[*d*][1,3]dioxol-5(6*H*)-one, **2**

IR (neat): 1219 (s), 1385 (s), 1626 (s) $cm^{-1}$.

$^{1}$H NMR (400 MHz, $CDCl_3$): δ 1.57 (3H, s), 1.62 (3H, s), 2.14-2.28 (2H, m), 2.51 (1H, dd, *J* = 14, 7.4 Hz), 2.59 (1H, dd, *J* = 14, 7.2 Hz), 4.86-5.04 (3H, m), 5.41 (1H, s), 5.47-5.62 (1H, m), 5.60 (1H, s), 5.80 (1H, s), 5.81 (1H, s).

$^{13}$C NMR (100 MHz, $CDCl_3$): δ 18.4, 26.3, 39.6, 45.1, 54.4, 99.9, 101.6, 109.1, 118.4, 118.7, 133.5, 135.3, 144.3, 164.4, 202.8.

MS: m/z (M+23) 269.1

HRMS: m/z calc'd for $C_{15}H_{18}NaO_3$ 269.1148, found 269.1136.

Data for 7a-allyl-6-(3-methylbut-2-enyl)benzo[*d*][1,3]dioxol-5(7a*H*)-one, **1**

IR (neat): 1175 (s), 1621 (s), 1676 (m), 2913 (w) $cm^{-1}$.

$^{1}$H NMR (400 MHz, $CDCl_3$): δ 1.60 (3H, s), 1.74 (3H, s), 2.38 (1H, dd, *J* = 14, 7.6 Hz), 2.60 (1H, dd, *J* = 14, 7.1 Hz), 2.94 (1H, dd, *J* = 17, 7.3 Hz), 3.03 (1H, dd, *J* = 17, 7.3 Hz), 5.02-5.18 (3H, m), 5.55 (1H, s), 5.56-5.68 (1H, m), 5.58 (1H, s), 5.62 (1H, s), 6.56 (1H, t, *J* = 1.7 Hz).

$^{13}$C NMR (100 MHz, $CDCl_3$): δ 18.0, 26.1, 27.9, 40.9, 81.4, 98.3, 98.9, 120.3, 121.2, 130.5, 133.9, 135.2, 140.7, 173.9, 187.9.

MS: m/z (M+23) 269.1

HRMS: m/z calc'd for $C_{15}H_{18}NaO_3$ 269.1148, found 269.1159.

Data for 6-(3-methylbut-2-enyl)-7a-(2,2,2-trifluoroethoxy)benzo[*d*][1,3]dioxol-5(7a*H*)-one, **7**

IR (neat): 1103 (s), 1123 (s), 1268 (m), 1280 (m), 1635 (m), 1654 (m), 1690 (m) $cm^{-1}$.

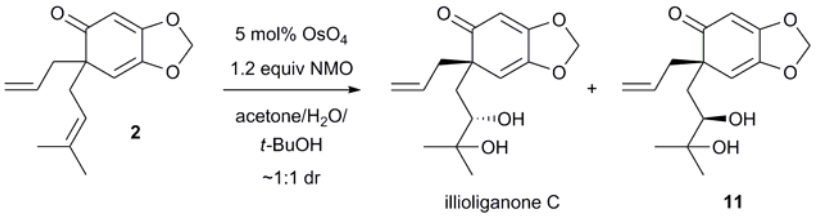
$^{1}$H NMR (400 MHz, $CDCl_3$): δ 1.61 (3H, s), 1.76 (3H, s), 2.99 (1H, dd, *J* = 18, 7.3 Hz), 3.09 (1H, dd, *J* = 18, 7.3 Hz), 3.72-3.95 (2H, m), 5.09-5.17 (1H, m), 5.63 (1H, s), 5.63 (1H, s), 5.67 (1H, s), 6.52 (1H, t, *J* = 1.8 Hz).

$^{13}$C NMR (100 MHz, $CDCl_3$): δ 18.0, 26.1, 28.1, 98.2, 99.4, 99.9, 119.2, 122.2, 125.0, 125.6, 136.3, 144.7, 167.1, 186.8.

MS: m/z (M+23) 327.1

HRMS: m/z calc'd for $C_{14}H_{15}F_3NaO_4$ 327.0815, found 327.0814.

**Synthesis of illioliganone C by Upjohn dihydroxylation**




6-Allyl-6-(3-methylbut-2-enyl)benzo[*d*][1,3]dioxol-5(6*H*)-one, **2**, (160 mg, 0.65 mmol, 1 equiv) was dissolved in acetone (0.3 mL), water (0.72 mL) and *t*-butanol (0.3 mL) at room temperature. *N*-Methylmorpholine *N*-oxide (132 mg, 0.98 mmol, 1.5 equiv) was added to the reaction mixture followed by osmium tetroxide (8 mg, 0.031 mmol, 0.05 equiv). After stirring for 3h, water was added and the mixture extracted with ethyl acetate. The organic layer was separated, dried over $MgSO_4$, filtered and concentrated. Purification by flash chromatography on silica gel using 2:1 petroleum ether/ethyl acetate was undertaken two times and illioliganone C was isolated.

IR (neat): 1221 (s), 1385 (s), 1626 (s) $cm^{-1}$.

$^1$H NMR (500 MHz, acetone-$d^6$): δ 1.06 (6H, s), 1.83 (1H, dd, *J* = 13.7, 1.8 Hz), 2.04-2.10 (1H, m), 2.24 (1H, dd, *J* = 13.0, 7.5 Hz), 2.48 (1H, dd, *J* = 13.1, 7.1 Hz), 3.06 (1H, d, *J* = 6.4 Hz), 3.29 (1H, s), 3.34 (1H, ddd, *J* = 10.7, 6.4, 1.9 Hz), 4.87-5.00 (2H, m), 5.46 (1H, s), 5.52-5.64 (1H, m), 5.54 (1H, s), 5.88 (1H, s), 5.90 (1H, s).

Literature values:[2] (400 MHz, acetone-$d^6$): δ 1.07 (s), 1.82 (dd, *J* = 13.6, 2.0 Hz), 2.10 (d, *J* = 13.6 Hz), 2.24 (dd, *J* = 13.2, 7.2 Hz), 2.48 (dd, *J* = 13.2, 7.2 Hz), 3.05 (d, *J* = 6.0 Hz), 3.27 (s), 3.33 (m), 4.91 (dd, *J* = 10.0, 2.0 Hz), 4.96 (dd, *J* = 16.8, 2.0 Hz), 5.46 (s), 5.54 (s), 5.57 (m), 5.88 (s), 5.89 (s).

$^{13}$C NMR (125 MHz, acetone-$d^6$): δ 25.4, 25.7, 43.3, 46.9, 52.4, 72.6, 76.6, 99.7, 102.4, 108.4, 118.0, 134.3, 145.1, 163.8, 202.0.

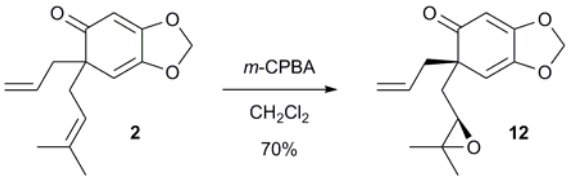
Literature values: (100 MHz, acetone-$d^6$): δ 25.4, 25.7, 43.3, 46.9, 52.3, 72.6, 76.3, 99.6, 102.3, 108.3, 117.9, 134.2, 145.1, 163.8, 202.1.

MS: m/z (M+23) 303.1

HRMS: m/z calc'd for $C_{15}H_{20}NaO_5$ 303.1203, found 303.1203

[2] W.-Z. Tang, S.-G. Ma, S.-S. Yu, J. Qu, Y.-B. Liu and J. Liu, *J. Nat. Prod.*, 2009, **72**, 1017.

**Synthesis of 6-allyl-6-((3,3-dimethyloxiran-2-yl)methyl)benzo[*d*][1,3]dioxol-5(6*H*)-one, 12**




6-Allyl-6-(3-methylbut-2-enyl)benzo[*d*][1,3]dioxol-5(6*H*)-one, **2**, (45 mg, 0.16 mmol, 1 equiv) was dissolved in $CH_2Cl_2$ (2 mL) at room temperature and *m*-chloroperbenzoic acid (70%, 36 mg, 0.21 mmol, 1.3 equiv) was added. After stirring for 5 h, the reaction mixture was quenched with saturated aqueous sodium thiosulfate solution and extracted with $CH_2Cl_2$. The organic layer was dried over $MgSO_4$, filtered and concentrated. Purification by flash chromatography on silica gel using 20:1 petroleum ether/ethyl acetate furnished **12** as a colourless oil (29 mg, 70%).

IR (neat): 1221 (s), 1385 (s), 1626 (s), 1720 (w) $cm^{-1}$.

$^1H$ NMR (400 MHz, $CDCl_3$): δ 1.18 (3H, s), 1.20 (3H, s), 1.56 (1H, dd, *J* = 14, 7.3 Hz), 2.25 (1H, dd, *J* = 13, 7.5 Hz), 2.35 (1H, dd, *J* = 14, 3.8 Hz), 2.42 (1H, dd, *J* = 7.3, 3.8 Hz), 2.56 (1H, dd, *J* = 13, 7.3 Hz), 4.94-5.06 (2H, m), 5.48-5.61 (1H, m), 5.53 (1H, s), 5.67 (1H, s), 5.82 (1H, s), 5.84 (1H, s).

$^{13}C$ NMR (100 MHz, $CDCl_3$): δ 19.2, 25.0, 40.4, 46.4, 53.3, 58.1, 61.3, 99.8, 102.0, 109.0, 119.1, 132.5, 144.3, 165.1, 202.4.

MS: m/z (M+23) 285.1

HRMS: m/z calc'd for $C_{15}H_{18}NaO_4$ 285.1097, found 285.1093.

**Synthesis of 6-allyl-6-(2,3-dihydroxy-3-methylbutyl)benzo[*d*][1,3]dioxol-5(6*H*)-one, 11**


A mixture of 6-allyl-6-((3,3-dimethyloxiran-2-yl)methyl)benzo[*d*][1,3]dioxol-5(6*H*)-one, **12** (49 mg, 0.19 mmol) and water (1.5 mL) was heated at 60 $^oC$ overnight. Upon cooling, the mixture was extracted with ethyl acetate, dried over $MgSO_4$, filtered and concentrated. Purification by flash chromatography on silica gel using 1:1 to 1:2 petroleum ether/ethyl acetate furnished **11** as a colourless oil (42 mg, 79%).

IR (neat): 1221 (s), 1385 (s), 1626 (s) $cm^{-1}$.

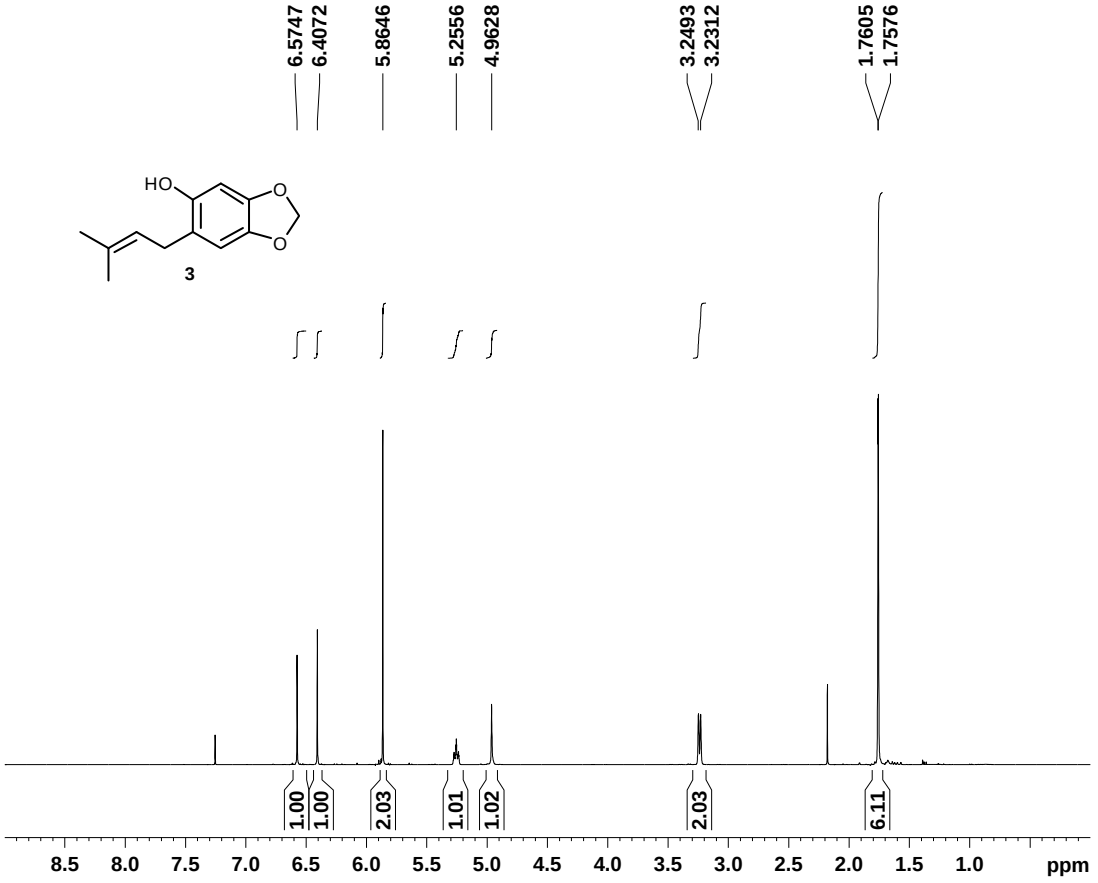
$^{1}$H NMR (400 MHz, acetone-$d^{6}$): δ 1.03 (3H, s), 1.05 (3H, s), 1.55 (1H, dd, *J* = 14, 9.4 Hz), 2.19 (1H, d, *J* = 14 Hz), 2.31 (1H, dd, *J* = 13, 7.4 Hz), 2.50 (1H, dd, *J* = 13, 7.2 Hz), 3.00-3.10 (1H, m), 3.42 (1H, br), 3.63 (1H, d, *J* = 5.0 Hz), 4.88-5.03 (2H, m), 5.52 (1H, s), 5.52-5.64 (1H, m), 5.68 (1H, s), 5.91 (1H, s), 5.94 (1H, s).

$^{13}$C NMR (100 MHz, acetone-$d^{6}$): δ 23.6, 24.8, 42.9, 46.1, 53.0, 72.0, 75.6, 98.4, 101.9, 110.1, 117.2, 133.3, 142.8, 164.9, 201.6.

MS: m/z (M+23) 303.1

HRMS: m/z calc'd for $C_{15}H_{20}NaO_5$ 303.1203, found 303.1203.

# NMR spectra for 6-(3-methylbut-2-enyl)benzo[*d*][1,3]dioxol-5-ol, 3

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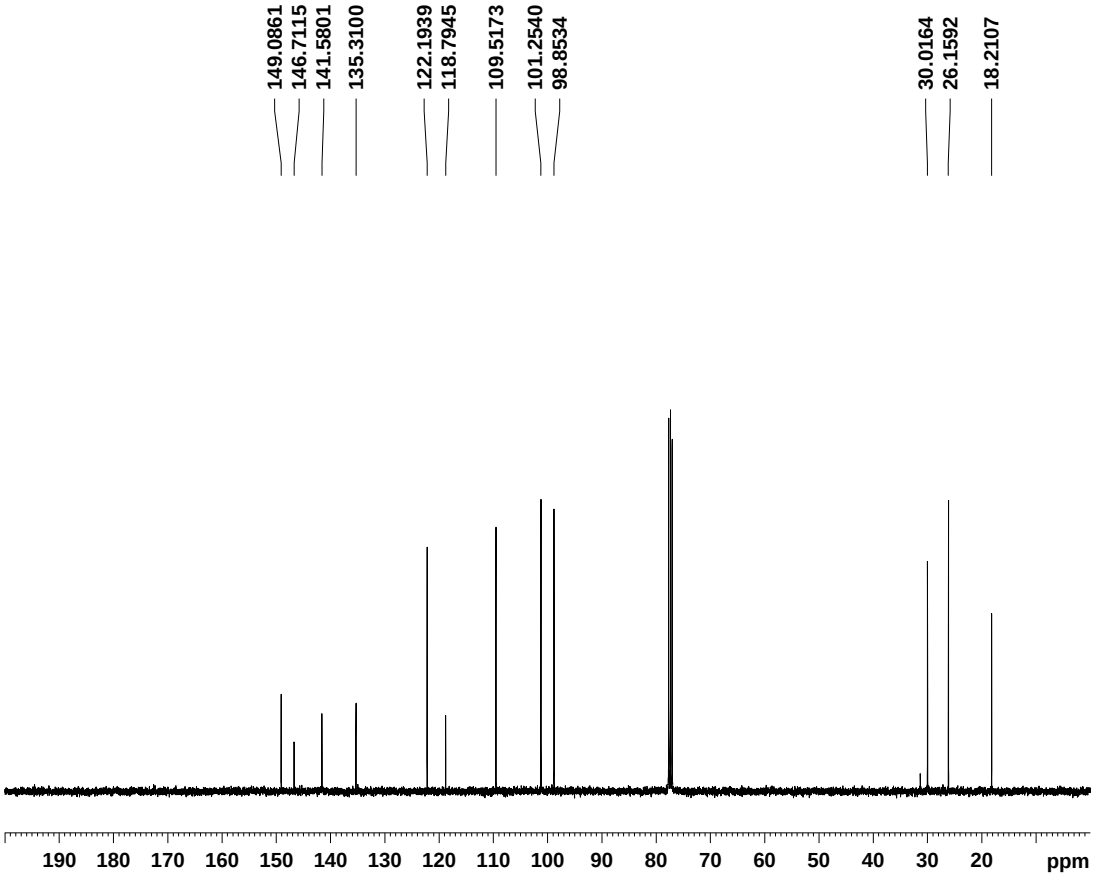


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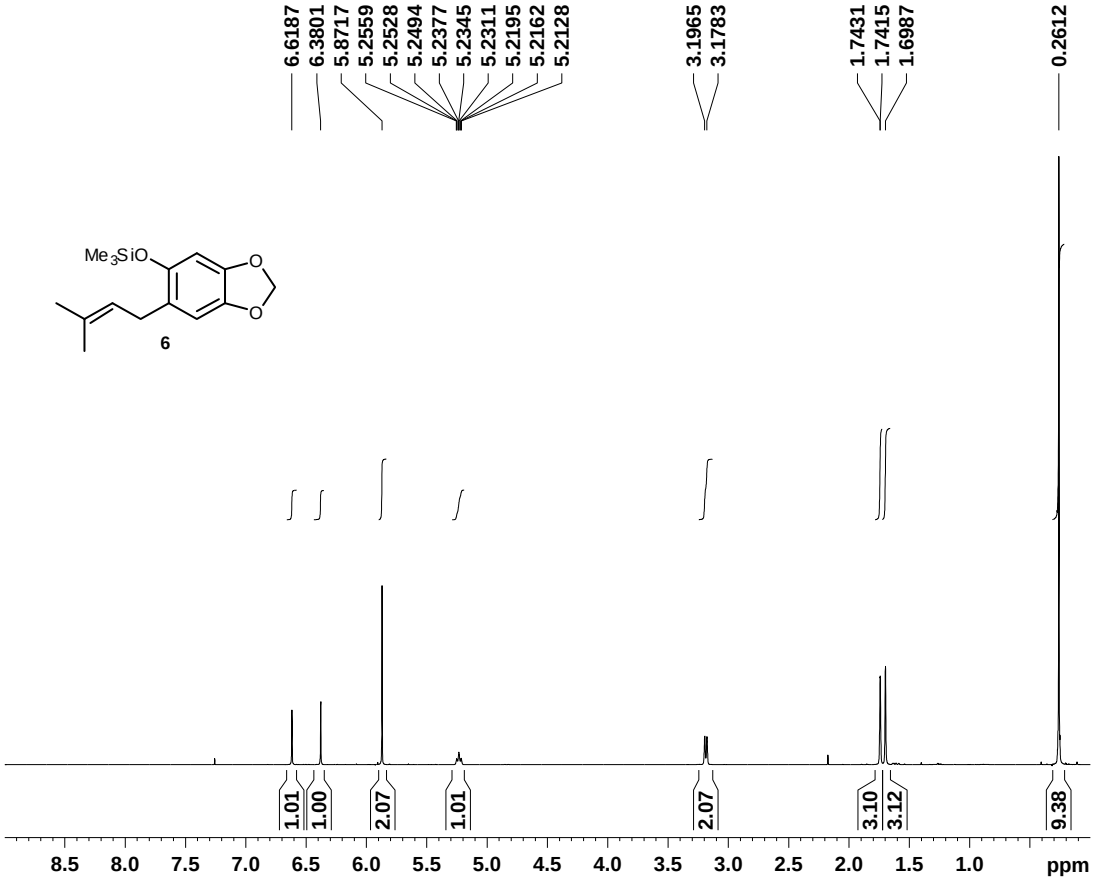
# NMR spectra for trimethyl(6-(3-methylbut-2-enyl)benzo[*d*][1,3]dioxol-5-yloxy)silane, 6

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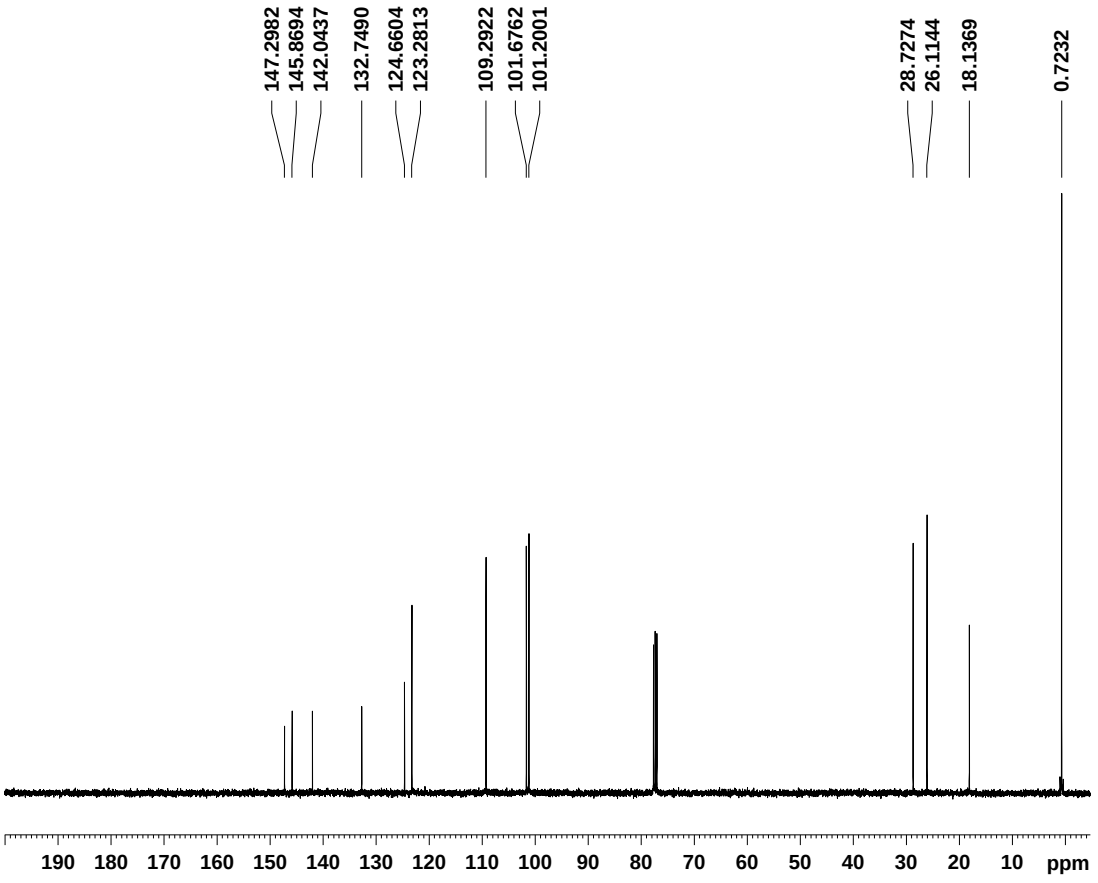


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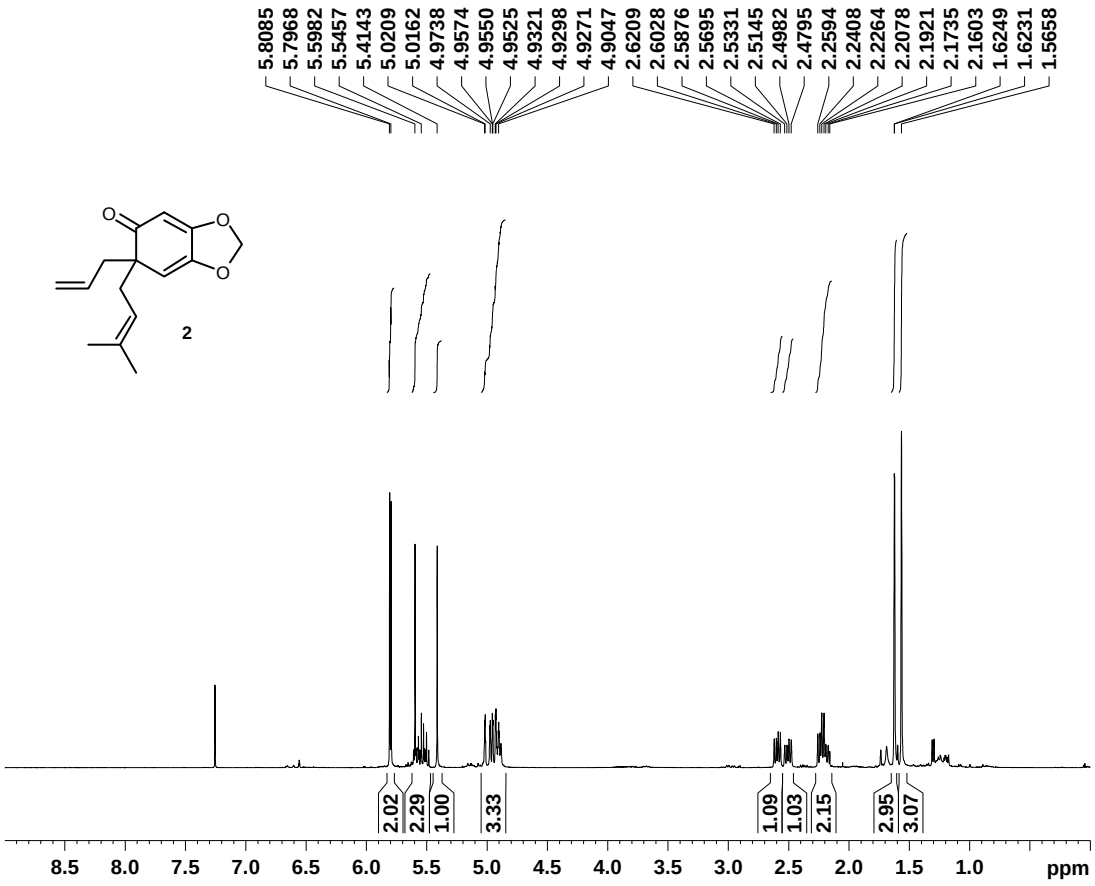
# NMR spectra for 6-allyl-6-(3-methylbut-2-enyl)benzo[*d*][1,3]dioxol-5(6*H*)-one, 2

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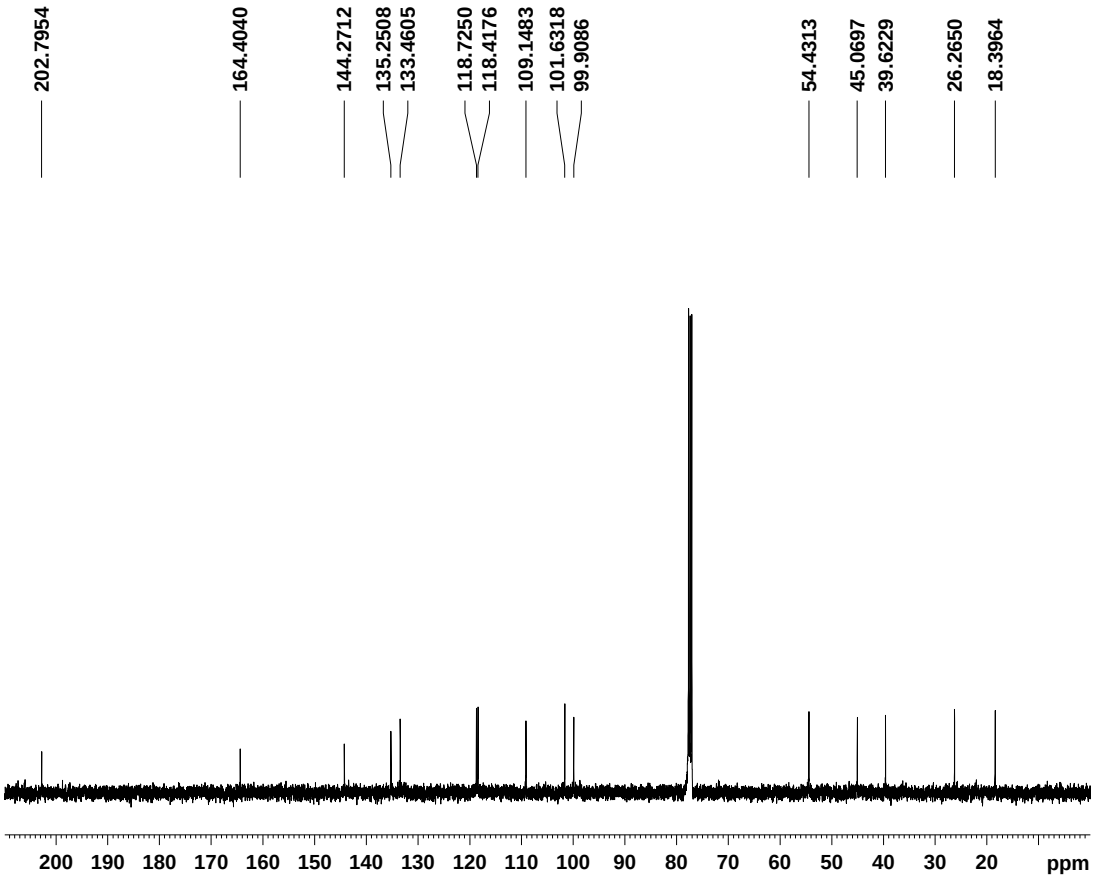


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PROCNO                     1

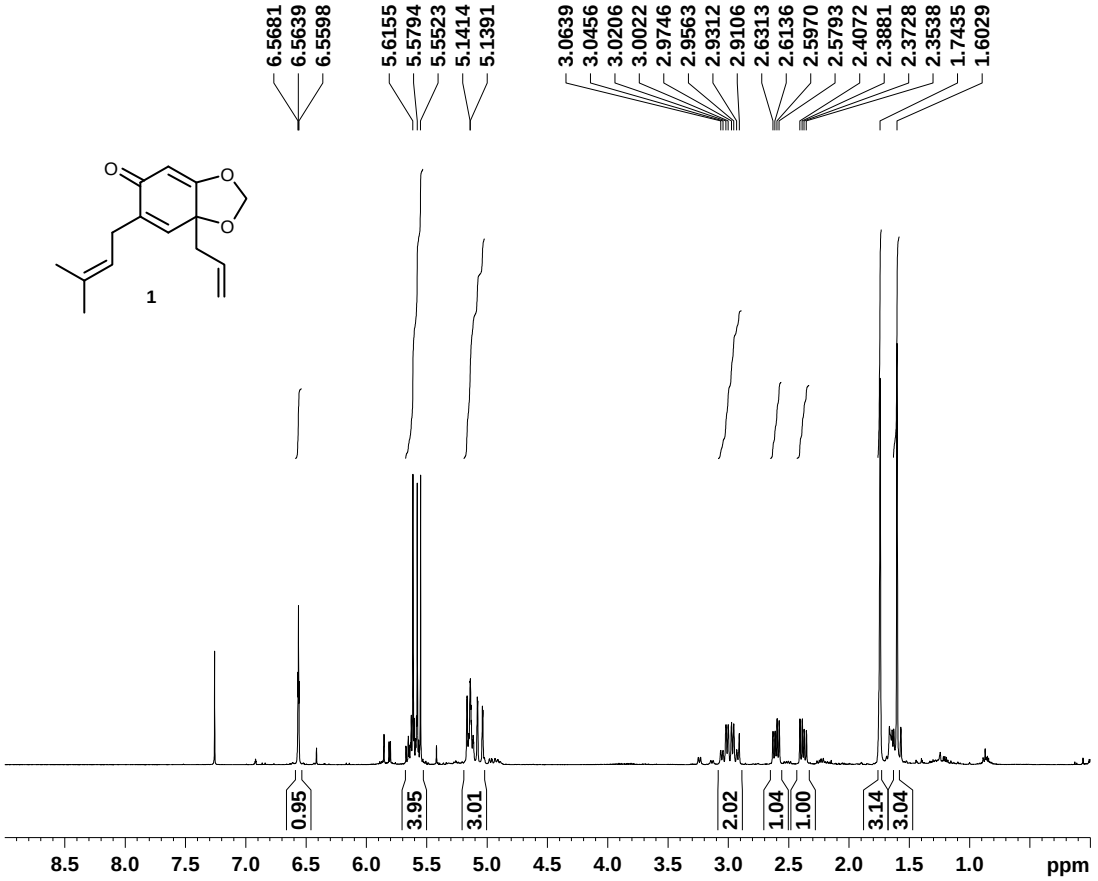
F2 - Acquisition Parameters
Date_              20100520
Time                  12.16
INSTRUM              dpx400
PROBHD    5 mm QNP  1H/1
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     256
DS                       4
SWH               23980.814 Hz
FIDRES            0.365918 Hz
AQ                1.3664756 sec
RG                    6502
DW                  20.850 usec
DE                   6.00 usec
TE                  292.9 K
D1                2.00000000 sec
d11               0.03000000 sec
DELTA             1.89999998 sec
TD0                        8

======== CHANNEL f1 ========
NUC1                     13C
P1                     9.70 usec
PL1                   -3.00 dB
SFO1           100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                  84.00 usec
PL2                    -3.00 dB
PL12                   18.00 dB
PL13                   18.00 dB
SFO2           400.1316005 MHz

F2 - Processing parameters
SI                       32768
SF                100.6127690 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```

# NMR spectra for 7a-allyl-6-(3-methylbut-2-enyl)benzo[*d*][1,3]dioxol-5(7a*H*)-one, 1

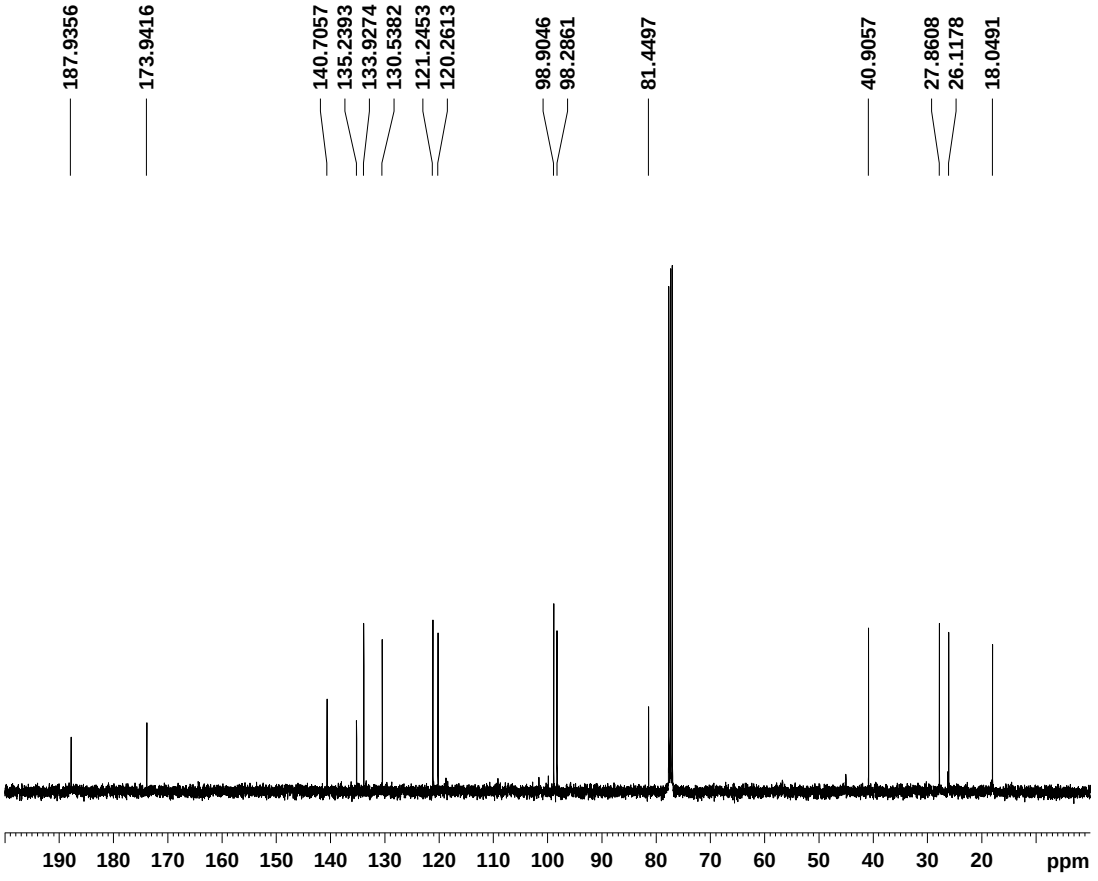



```
Current Data Parameters
NAME                  WM-352
EXPNO                      4
PROCNO                     1

F2 - Acquisition Parameters
Date_              20100607
Time                  15.57
INSTRUM               dpx400
PROBHD    5 mm QNP  1H/1
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8278.146 Hz
FIDRES            0.126314 Hz
AQ                3.9584243 sec
RG                    203.2
DW                   60.400 usec
DE                    6.00 usec
TE                   566.6 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     8.40 usec
PL1                   -3.00 dB
SFO1           400.1324710 MHz

F2 - Processing parameters
SI                        32768
SF               400.1300096 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00
```




```
Current Data Parameters
NAME                  WM-352
EXPNO                      7
PROCNO                     1

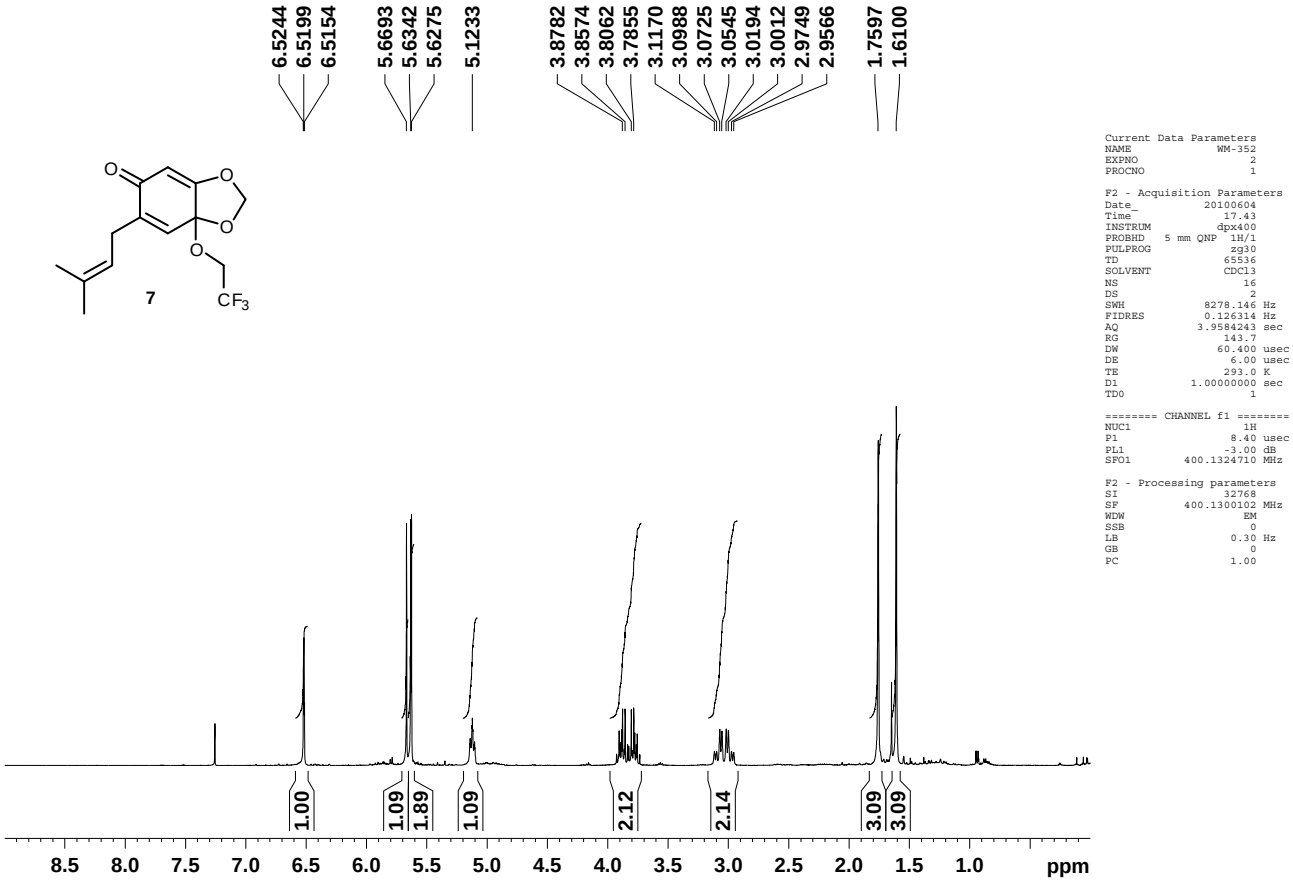
F2 - Acquisition Parameters
Date_              20100608
Time                   9.16
INSTRUM               dpx400
PROBHD    5 mm QNP  1H/1
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      256
DS                        4
SWH               23980.814 Hz
FIDRES            0.365918 Hz
AQ                1.3664756 sec
RG                    16384
DW                   20.850 usec
DE                    6.00 usec
TE                   293.1 K
D1                2.00000000 sec
d11               0.03000000 sec
DELTA             1.89999998 sec
TD0                        8

======== CHANNEL f1 ========
NUC1                     13C
P1                     9.70 usec
PL1                   -3.00 dB
SFO1           100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                  84.00 usec
PL2                   -3.00 dB
PL12                  18.00 dB
PL13                  18.00 dB
SFO2           400.1316005 MHz

F2 - Processing parameters
SI                        32768
SF               100.6127690 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40
```

# NMR spectra for 6-(3-methylbut-2-enyl)-7a-(2,2,2-trifluoroethoxy)benzo[*d*][1,3]dioxol-5(7a*H*)-one, 7

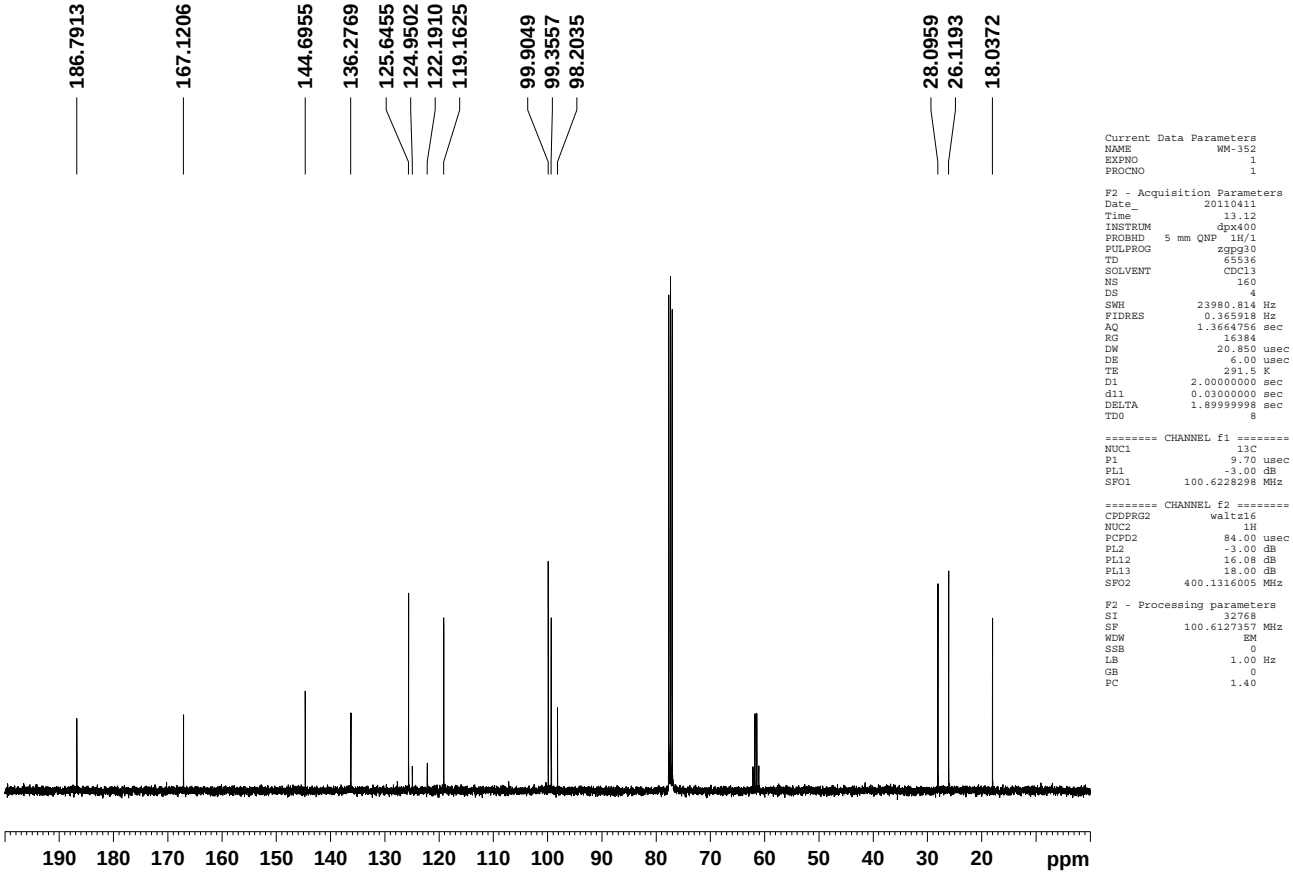



```
Current Data Parameters
NAME                  WM-352
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_              20100604
Time                  17.43
INSTRUM              dpx400
PROBHD    5 mm QNP  1H/1
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH                8278.146 Hz
FIDRES             0.126314 Hz
AQ                 3.9584243 sec
RG                     143.7
DW                    60.400 usec
DE                     6.00 usec
TE                    293.0 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                      8.40 usec
PL1                    -3.00 dB
SFO1              400.1324710 MHz

F2 - Processing parameters
SI                       32768
SF                400.1300102 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00
```




```
Current Data Parameters
NAME                  WM-352
EXPNO                      1
PROCNO                     1

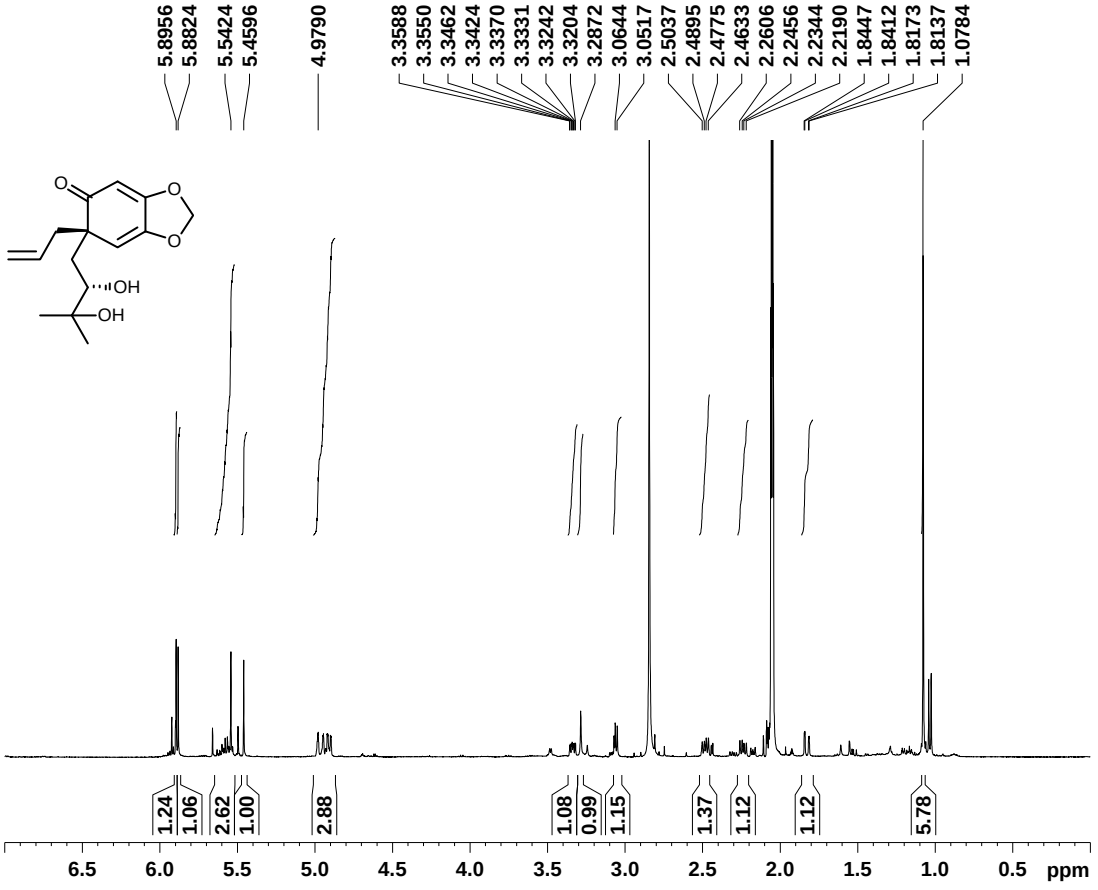
F2 - Acquisition Parameters
Date_              20110411
Time                  13.12
INSTRUM              dpx400
PROBHD    5 mm QNP  1H/1
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                      160
DS                        4
SWH               23980.814 Hz
FIDRES             0.365918 Hz
AQ                 1.3664756 sec
RG                     16384
DW                    20.850 usec
DE                     6.00 usec
TE                    291.5 K
D1                 2.00000000 sec
d11                0.03000000 sec
DELTA              1.89999998 sec
TD0                        8

======== CHANNEL f1 ========
NUC1                     13C
P1                      9.70 usec
PL1                    -3.00 dB
SFO1              100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                  84.00 usec
PL2                    -3.00 dB
PL12                   16.08 dB
PL13                   18.00 dB
SFO2              400.1316005 MHz

F2 - Processing parameters
SI                       32768
SF                100.6127357 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```

# NMR spectra for illioliganone C

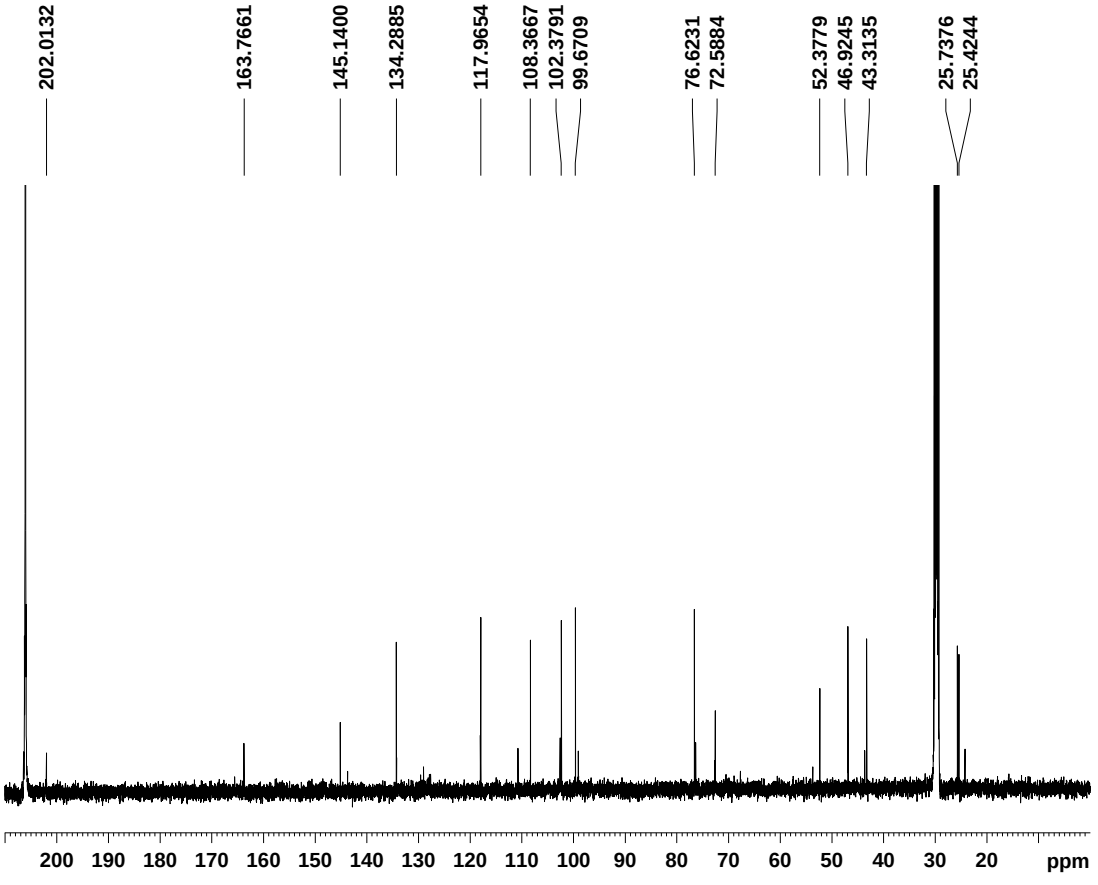



```
Current Data Parameters
NAME          ARM-1136_500
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_              20110421
Time                 10.05
INSTRUM              av500
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT           Acetone
NS                       16
DS                        2
SWH              10000.000 Hz
FIDRES               0.152588 Hz
AQ                3.2768500 sec
RG                       287
DW                  50.000 usec
DE                   6.00 usec
TE                   296.5 K
D1               2.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   9.70 usec
PL1                  0.10 dB
SFO1          500.1330633 MHz

F2 - Processing parameters
SI                   32768
SF            500.1300085 MHz
WDW                     EM
SSB                      0
LB                   0.30 Hz
GB                       0
PC                   1.00
```




```
Current Data Parameters
NAME          ARM-1136_500
EXPNO                    2
PROCNO                   1

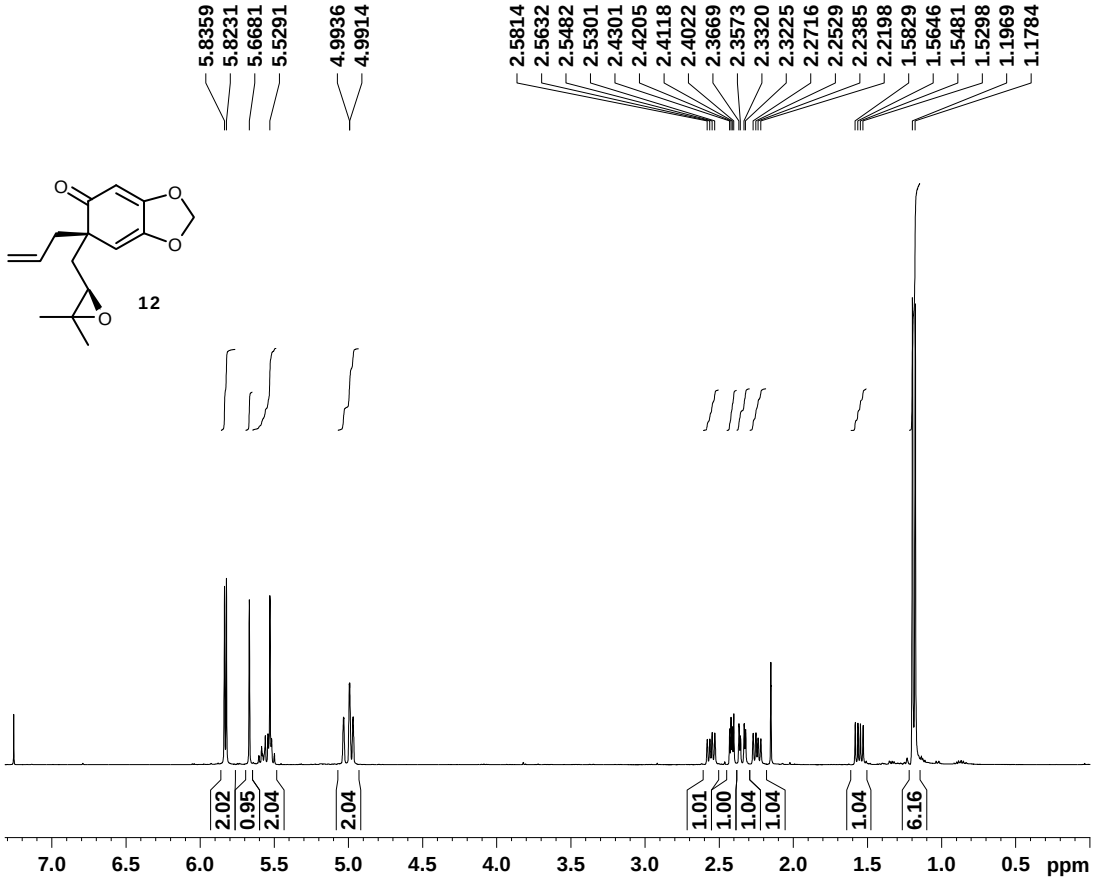
F2 - Acquisition Parameters
Date_              20110421
Time                 10.11
INSTRUM              av500
PROBHD    5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT           Acetone
NS                    2527
DS                       4
SWH              30030.029 Hz
FIDRES               0.458222 Hz
AQ                1.0912244 sec
RG                   11500
DW                  16.650 usec
DE                   6.00 usec
TE                   297.7 K
D1               2.00000000 sec
d11              0.03000000 sec
DELTA            1.89999998 sec
TD0                    256

======== CHANNEL f1 ========
NUC1                   13C
P1                   9.00 usec
PL1                  0.00 dB
SFO1          125.7703637 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  0.00 dB
PL12                18.00 dB
PL13                18.00 dB
SFO2          500.1320005 MHz

F2 - Processing parameters
SI                   32768
SF            125.7576838 MHz
WDW                     EM
SSB                      0
LB                   1.00 Hz
GB                       0
PC                   1.00
```

# NMR spectra for 6-allyl-6-((3,3-dimethyloxiran-2-yl)methyl)benzo[*d*][1,3]dioxol-5(6*H*)-one, 12

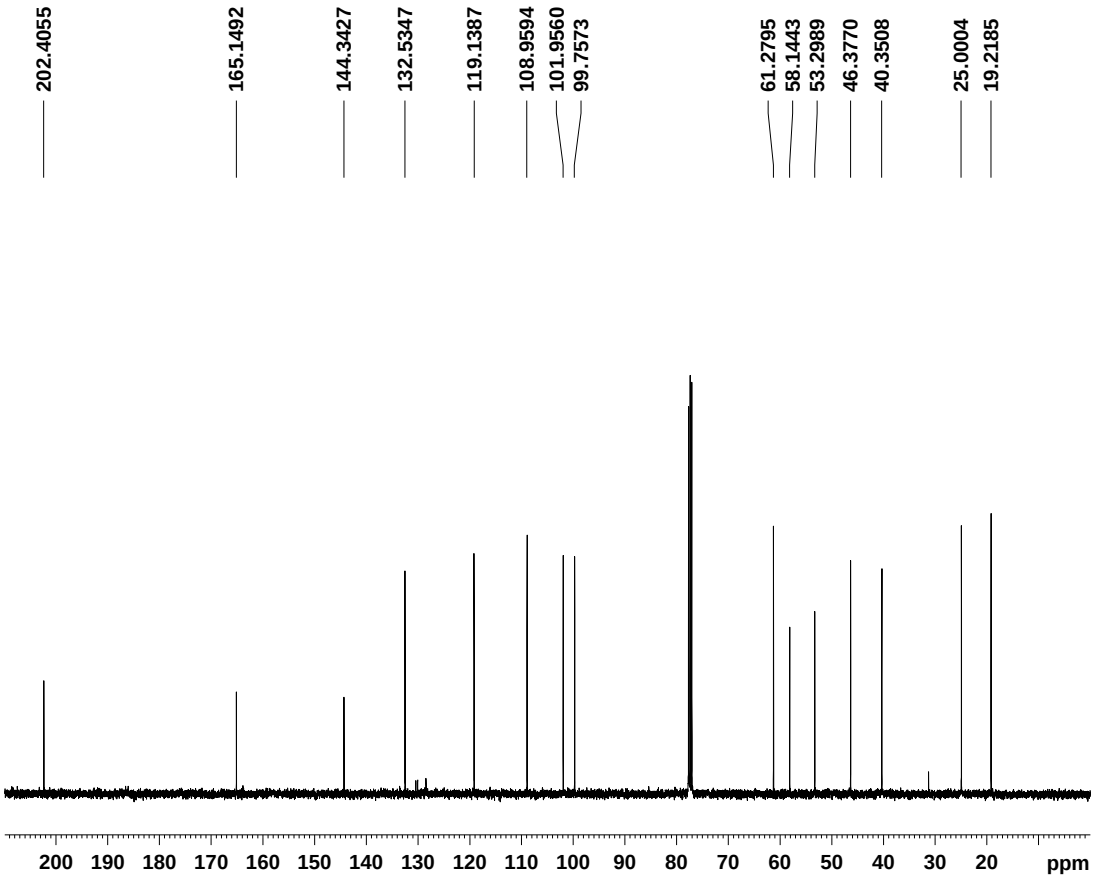



```
Current Data Parameters
NAME                  WM-350
EXPNO                      4
PROCNO                     1

F2 - Acquisition Parameters
Date_              20100602
Time                  10.43
INSTRUM               dpx400
PROBHD     5 mm QNP  1H/1
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8278.146 Hz
FIDRES            0.126314 Hz
AQ                3.9584243 sec
RG                    143.7
DW                   60.400 usec
DE                    6.00 usec
TE                   291.9 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     8.40 usec
PL1                   -3.00 dB
SFO1           400.1324710 MHz

F2 - Processing parameters
SI                        32768
SF               400.1300000 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00
```




```
Current Data Parameters
NAME                  WM-350
EXPNO                      5
PROCNO                     1

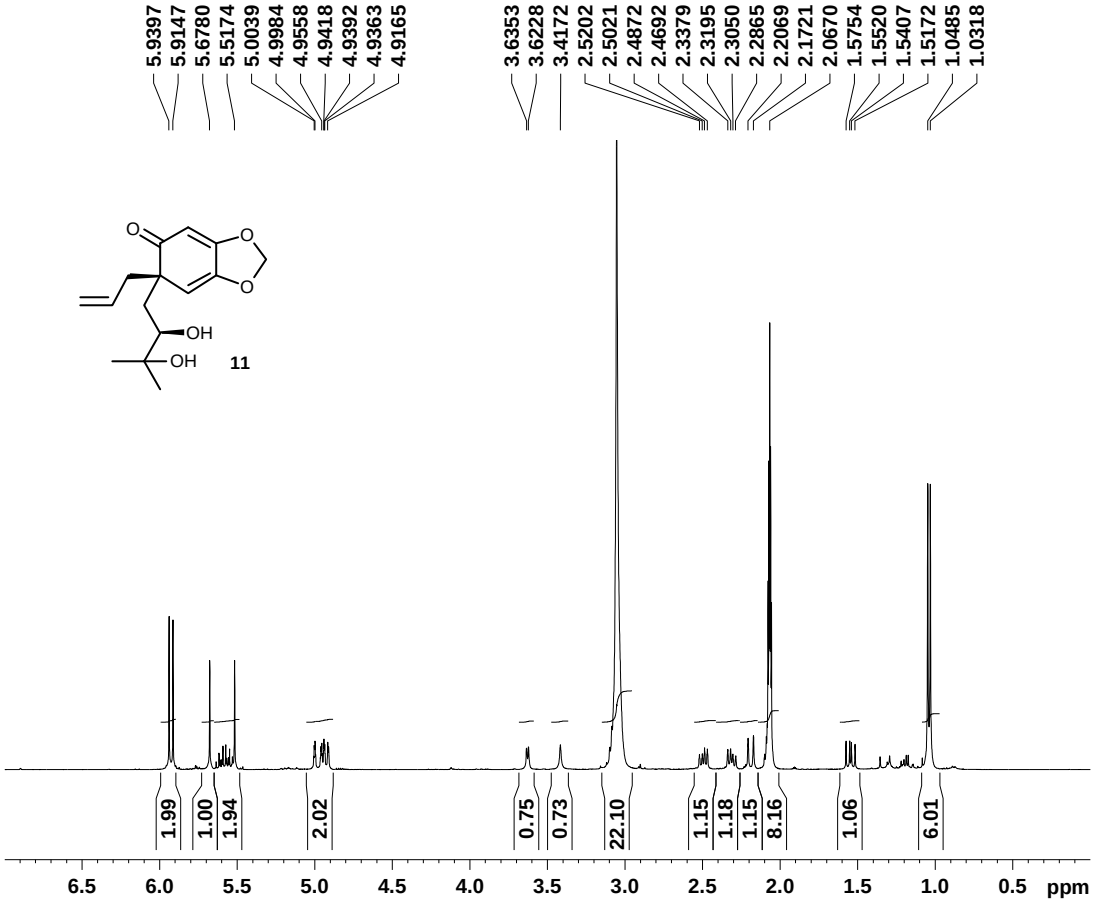
F2 - Acquisition Parameters
Date_              20100602
Time                  10.28
INSTRUM               dpx400
PROBHD     5 mm QNP  1H/1
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      256
DS                        4
SWH               23980.814 Hz
FIDRES            0.365918 Hz
AQ                1.3664756 sec
RG                    16384
DW                   20.850 usec
DE                    6.00 usec
TE                   291.9 K
D1                2.00000000 sec
d11               0.03000000 sec
DELTA             1.89999998 sec
TD0                        8

======== CHANNEL f1 ========
NUC1                     13C
P1                     9.70 usec
PL1                   -3.00 dB
SFO1           100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                  84.00 usec
PL2                   -3.00 dB
PL12                  18.00 dB
PL13                  18.00 dB
SFO2           400.1316005 MHz

F2 - Processing parameters
SI                        32768
SF               100.6127690 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40
```

# NMR spectra for 6-allyl-6-(2,3-dihydroxy-3-methylbutyl)benzo[*d*][1,3]dioxol-5(6*H*)-one, 11

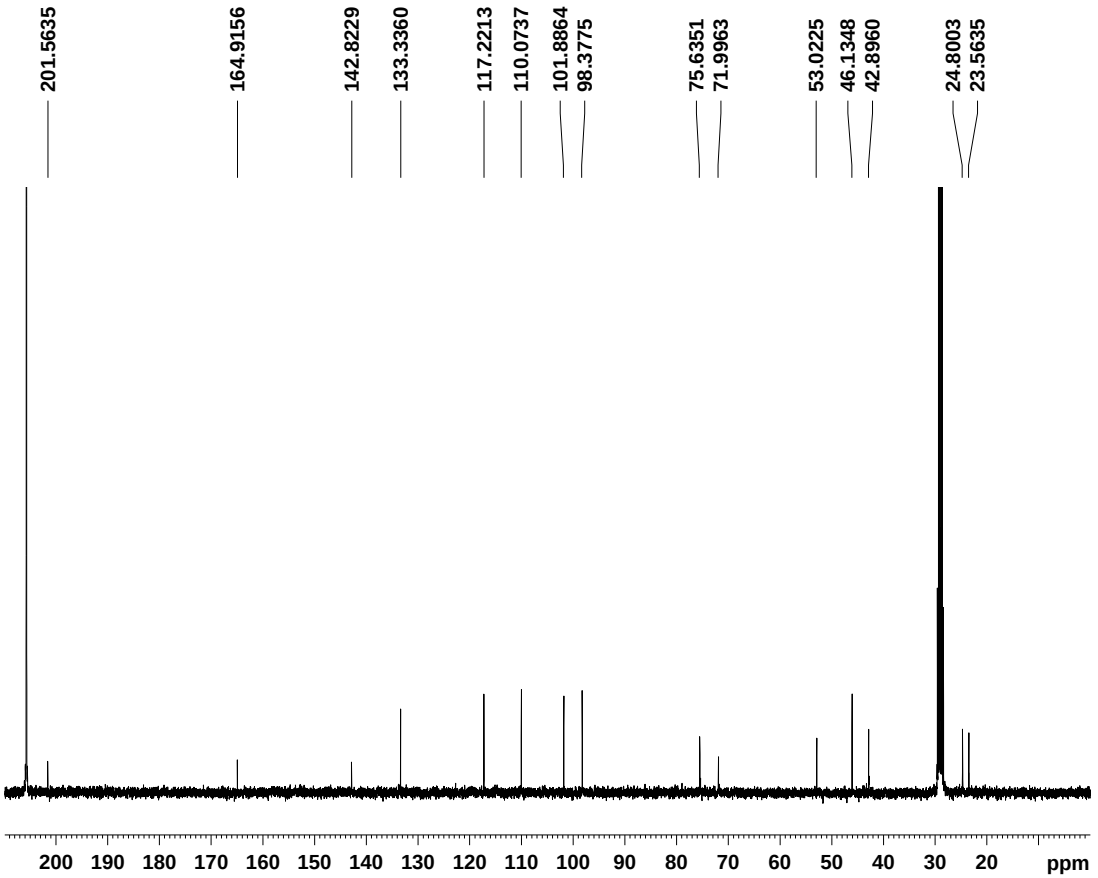



```
Current Data Parameters
NAME                  WM-410
EXPNO                      4
PROCNO                     1

F2 - Acquisition Parameters
Date_              20101014
Time                   9.24
INSTRUM              dpx400
PROBHD    5 mm QNP  1H/1
PULPROG                zg30
TD                    65536
SOLVENT             Aceton
NS                        16
DS                         2
SWH                8278.146 Hz
FIDRES            0.126314 Hz
AQ                3.9584243 sec
RG                     143.7
DW                    60.400 usec
DE                     6.00 usec
TE                    290.9 K
D1              1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     8.40 usec
PL1                   -3.00 dB
SFO1           400.1324710 MHz

F2 - Processing parameters
SI                     32768
SF             400.1300000 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```




```
Current Data Parameters
NAME                  WM-410
EXPNO                      5
PROCNO                     1

F2 - Acquisition Parameters
Date_              20101014
Time                   9.27
INSTRUM              dpx400
PROBHD    5 mm QNP  1H/1
PULPROG              zgpg30
TD                    65536
SOLVENT             Aceton
NS                       256
DS                         4
SWH               23980.814 Hz
FIDRES            0.365918 Hz
AQ                1.3664756 sec
RG                   11585.2
DW                    20.850 usec
DE                     6.00 usec
TE                    291.1 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
TD0                        8

======== CHANNEL f1 ========
NUC1                     13C
P1                     9.70 usec
PL1                   -3.00 dB
SFO1           100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                 84.00 usec
PL2                   -3.00 dB
PL12                  18.00 dB
PL13                  18.00 dB
SFO2           400.1316005 MHz

F2 - Processing parameters
SI                     32768
SF             100.6127690 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40
```