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# Hydrolysis of Difluorocyclopropenes: the Role of the Cyclopropenyl Cation and the Effects of Substituents

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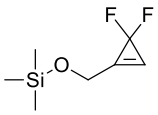
# Table of contents

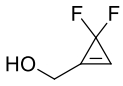
# Characterization of the synthesized compounds



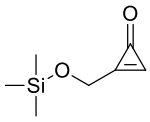
***3,3-Difluorocycloprop-1-en-1-ylmethyl TMS-ether (1a)***

$^1$H NMR (400 MHz, Chloroform-*d*) δ 7.37 (s, 1H), 4.62 (s, 2H), 0.14 (s, 9H).

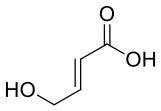
**Conditions 4:** MeOH-$H_2O$(10:1) $SiO_2$, 18 h – **1a´**(60%)*, **2a**(25%)**, **3a**(15%)***.



**1a' ***



**2a****

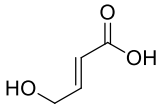


**3a*****

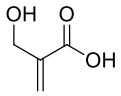
***(3,3-difluorocycloprop-1-en-1-yl)methanol (1a´), 2-(((trimethylsilyl)oxy)methyl)cycloprop-2-en-1-one (2a) and (E)-4-hydroxybut-2-enoic acid (3a)***

$^1$H NMR (400 MHz, Chloroform-*d*) δ 12.49 (s, 1H$^{***}$), 8.53 (s, 1H$^{**}$), 7.29 (s, 2H$^{*}$), 6.91 – 6.80 (m, 1H$^{***}$), 5.87 (d, *J* = 8.0 Hz, 1H$^{***}$), 4.76 (s, 2H$^{**}$), 4.42 (s, 5H$^{*}$), 4.18 (s, 1H$^{***}$), 3.38 (s, 1H$^{***}$).

**Conditions 5:** MeOH-$H_2O$(10:1) $SiO_2$, 72 h - **3a**(95%)*, **4a**(5%)**.



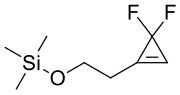
**3a**



**4a**

***(E)-4-hydroxybut-2-enoic acid (3a) and 2-(hydroxymethyl)acrylic acid (4a)***

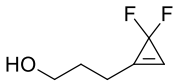
$^1$H NMR (400 MHz, Chloroform-*d*) δ 11.36 (s, 1H$^{*+**}$), 6.87 (d, *J* = 8.0 Hz, 1H$^{*}$), 6.22 (s, 1H$^{**}$), 5.87 (d, *J* = 8.0 Hz, 1H$^{*}$), 5.82 (s, 1H$^{**}$), 4.18 (s, 2H$^{*}$), 3.80 (s, 1H$^{**}$), 3.22 (s, 1H$^{*+**}$).



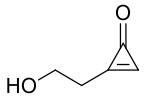
***3,3-Difluorocycloprop-1-en-1-ylethyl TMS-ether (1b)***

$^1$H NMR (500 MHz, Chloroform-*d*) δ 7.27 (s, 1H), 3.85 – 3.75 (m, 2H), 2.83 – 2.65 (m, 2H), 0.11 (s, 9H).

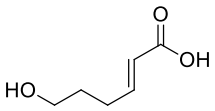
**Conditions 4:** MeOH-$H_2O$(10:1) $SiO_2$, 18 h –**1b'**(40%)*, **2b**(45%)**, **3b**(12%)**, **4b**(3%)****



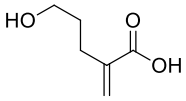
1b' *



2b**



3b***

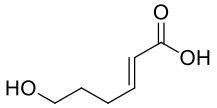


4b****

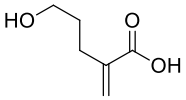
***2-(3,3-difluorocycloprop-1-en-1-yl)ethan-1-ol (1b´), 2-(2-hydroxyethyl)cycloprop-2-en-1-one (2b), (E)-6-hydroxyhex-2-enoic acid (3b) and 5-hydroxy-2-methylenepentanoic acid (4b)***

$^1$H NMR (400 MHz, DMSO-$d_6$) δ 12.05 (s, 1H$^{*+**+***+****}$), 8.52 (s, 2H$^{*}$), 7.35 (s, 2H$^{**}$), 6.89 (d, *J* = 15.5 Hz, 1H$^{***}$), 5.90 (s, 1H$^{****}$), 5.82 – 5.71 (m, 2H$^{***+****}$), 3.94 (t, *J* = 5.6 Hz, 2H$^{*}$), 3.80 (t, *J* = 5.6 Hz, 2H$^{**}$), 3.57 (t, *J* = 5.3 Hz, 2H$^{****}$), 3.47 (t, *J* = 6.2 Hz, 2H$^{***}$), 2.79 (t, *J* = 5.6 Hz, 2H$^{*}$), 2.75 – 2.62 (m, 3H$^{**+****}$), 2.28 (t, *J* = 6.2 Hz, 2H$^{***}$).

**Conditions 5:** MeOH-$H_2O$(10:1) $SiO_2$, 72 h – **3b**(72%)*, **4b**(28%)**.



3b*

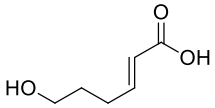
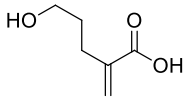


4b**

***(E)-6-hydroxyhex-2-enoic acid (3b) and 5-hydroxy-2-methylenepentanoic acid (4b)***

$^1$H NMR (400 MHz, DMSO-$d_6$) δ 12.05 (s, 1H$^{*+**}$), 6.89 (d, *J* = 15.6 Hz, 1H$^{*}$), 5.90 (s, 1H$^{**}$), 5.79 (s, 1H$^{**}$), 5.76 (d, *J* = 15.6 Hz, 1H$^{*}$), 3.57 (t, *J* = 5.4 Hz, 2H$^{**}$), 3.47 (t, *J* = 6.2 Hz, 2H$^{*}$), 2.67 (t, *J* = 5.3 Hz, 2H$^{**}$), 2.28 (t, *J* = 6.2 Hz, 2H$^{*}$).

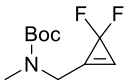
**Conditions 6:** MeOH-$H_2O$(5:1) $SiO_2$, 60C, 18 h – **3b**(72%)*, **4b**(28%)**.

3b* 4b**

***(E)-6-hydroxyhex-2-enoic acid (3b) and 5-hydroxy-2-methylenepentanoic acid (4b)***

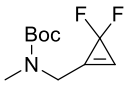
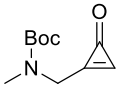
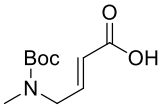
$^1$H NMR (400 MHz, Chloroform-*d*) δ 12.77 (s, 1H$^{*+**}$), 6.89 (d, *J* = 15.6 Hz, 1H$^{*}$), 5.90 (s, 1H$^{**}$), 5.79 (s, 1H$^{**}$), 5.76 (d, *J* = 15.6 Hz, 1H$^{*}$), 3.57 (t, *J* = 5.4 Hz, 2H$^{**}$), 3.47 (t, *J* = 6.2 Hz, 2H$^{*}$), 2.67 (t, *J* = 5.4 Hz, 2H$^{**}$), 2.28 (t, *J* = 6.2 Hz, 2H$^{*}$).



***N-Boc-N-methyl-N-((3,3-difluorocycloprop-1-en-1-yl)methyl)amine (1c)***

$^1$H NMR (400 MHz, Chloroform-*d*) δ 7.33 (s, 1H), 4.27 (s, 2H), 2.87 (s, 3H), 1.40 (s, 9H).

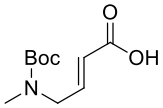
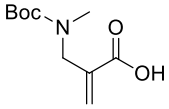
**Conditions 4:** MeOH-$H_2O$(10:1) $SiO_2$, 18 h – **1c**(55%)*, **2c**(35%)**, **3c**(20%)***.

**1c*** **2c**** **3c*****

$^1$H NMR (400 MHz, Chloroform-*d*) δ 10.46 (s, 1H$^{*+***+****}$), 7.88 (s, 1H$^{**}$), 7.21 (s, 1H$^{*}$), 6.89 (d, *J* = 12.1 Hz, 1H$^{***}$), 5.80 (d, *J* = 12.0 Hz, 1H$^{***}$), 3.97 – 3.78 (m, 2H$^{*+***}$), 3.58 (s, 2H$^{**}$), 3.08 – 2.92 (m, 3H$^{*+***}$), 2.81 (s, 3H$^{***}$), 1.53 – 1.29 (m, 9H$^{*+**+***}$).

**Conditions 5:** MeOH-$H_2O$(10:1) $SiO_2$, 72 h – **3c**(84%)*, **4c**(16%)**.

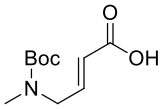
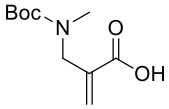



**3c*** **4c****

***(E)-4-((tert-butoxycarbonyl)(methyl)amino)but-2-enoic acid (3c) and 2-(((tert-butoxycarbonyl)(methyl)-amino)methyl)acrylic acid (4c)***

$^1$H NMR (400 MHz, Chloroform-*d*) δ 10.87 (s, 1H$^{*+**}$), 6.89 (d, *J* = 12.1 Hz, 1H$^{*}$), 6.25 (s, 1H$^{**}$), 5.80 (d, *J* = 12.0 Hz, 1H$^{*}$), 5.52 (s, 1H$^{**}$), 4.08 – 3.88 (m, 2H$^{*+***}$), 2.90 – 2.73 (m, 3H$^{*+***}$), 1.50 – 1.30 (m, 9H$^{*+***}$).

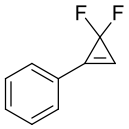
**Conditions 6:** MeOH-$H_2O$(5:1) $SiO_2$, 60C, 18 h – **3c**(85%)*, **4c**(15%)**.

**3c*** **4c****

***(E)-4-((tert-butoxycarbonyl)(methyl)amino)but-2-enoic acid (3c) and 2-(((tert-butoxycarbonyl)(methyl)-amino)methyl)acrylic acid (4c)***

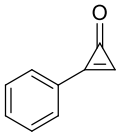
$^1$H NMR (400 MHz, Chloroform-*d*) δ 10.85 (s, 1H$^{*+**}$), 6.89 (d, *J* = 12.1 Hz, 1H$^{*}$), 6.25 (s, 1H$^{**}$), 5.80 (d, *J* = 12.0 Hz, 1H$^{*}$), 5.52 (s, 1H$^{**}$), 4.10 – 3.83 (m, 2H$^{*+***}$), 2.91 – 2.71 (m, 3H$^{*+***}$), 1.51 – 1.32 (m, 9H$^{*+***}$).

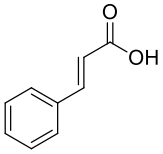


***(3,3-difluoro-cycloprop-1-en-1-yl)benzene (1d)***

$^1$H NMR (400 MHz, Chloroform-*d*) δ 7.68 – 7.66 (m, 2H), 7.53 – 7.49 (m, 2H), 7.47 – 7.45 (m, 2H).

**Conditions 2:** DCM/MeOH(1:1) $SiO_2$, 18h - **2d** (40%)*, **3d** (60%)**

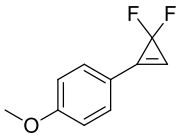




**2d*** **3d****

***2-phenylcycloprop-2-en-1-one (2d) and cinnamic acid (3d)***

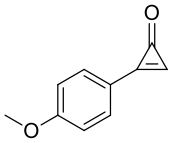
$^1$H NMR (400 MHz, Chloroform-*d*) δ 8.50 (s, 1H)**, 7.89 – 7.83 (m, 2H)*, 7.80 (d, *J* = 16.1 Hz, 1H)**, 7.60 (s, 1H)*, 7.55 (m, 4H)*+**, 7.41 (br. s, 4H)*+**, 6.46 (d, *J* = 16.0 Hz, 1H)**.

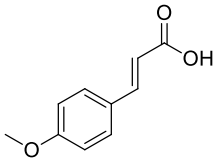


***1-(3,3-difluoro-cycloprop-1-en-1-yl)-4-methoxybenzene (1e)***

$^1$H NMR (400 MHz, Chloroform-*d*) δ 7.58 (d, *J* = 8.6 Hz, 2H), 7.29 – 7.22 (m, 1H), 6.96 (d, *J* = 8.6 Hz, 2H), 3.84 (s, 3H).

**Conditions 1:** DCM/ $SiO_2$/ Air, 18h – **2e** (80%), **3e** (20%)*



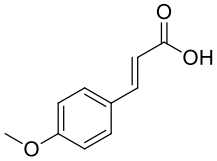


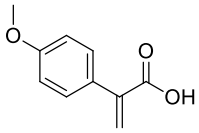
**2e** **3e***

***2-(4-methoxyphenyl)cycloprop-2-en-1-one (2e) and (E)-3-(4-methoxyphenyl)acrylic acid (3e)***

$^1$H NMR (400 MHz, Chloroform-*d*) δ 10.43 (s, 1H)*, 8.27 (s, 1H), 7.81 (d, *J* = 7.5 Hz, 2H), 7.74 (d, *J* = 16.0 Hz, 1H)*, 7.51 (d, *J* = 7.7 Hz, 2H)*, 7.03 (d, *J* = 7.5 Hz, 2H)*, 6.92 (d, *J* = 7.7 Hz, 2H)*, 6.32 (d, *J* = 15.9 Hz, 1H)*, 3.89 (s, 3H), 3.85 (s, 3H)*.

**Conditions 2:** DCM/MeOH(1:1) $SiO_2$, 18h - **3e** (82%), **4e** (16%)**

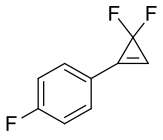




**3e** **4e**

***(E)-3-(4-methoxyphenyl)acrylic acid (3e) and 2-(4-methoxyphenyl)acrylic acid (4e)****

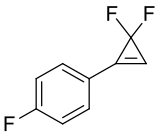
$^1$H NMR (400 MHz, Chloroform-*d*) δ 10.38 (s, 1H), 8.58 (s, 1H)*, 7.74 (d, *J* = 16.0 Hz, 1H), 7.51 (d, *J* = 7.7 Hz, 2H), 7.39 (d, *J* = 7.7 Hz, 2H)*, 6.92 (d, *J* = 7.8 Hz, 2H+2H*), 6.45 (s, 1H)*, 6.32 (d, *J* = 16.0 Hz, 1H), 5.95 (s, 1H)*, 3.85 (s, 3H), 3.82 (s, 3H)*.

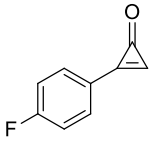


***1-(3,3-difluorocycloprop-1-en-1-yl)-4-fluorobenzene (1f)***

$^1$H NMR (500 MHz, Chloroform-*d*) δ 7.67 – 7.65 (m, 2H), 7.44 (s, 1H), 7.18 (t, 2H).

**Conditions 2:** DCM/MeOH(1:1) $SiO_2$, 18h – **1f**(75%), **2f**(25%)**

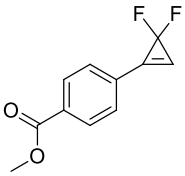



**1f** **2f***

***1-(3,3-difluorocycloprop-1-en-1-yl)-4-fluorobenzene (1f) and 2-(4-fluorophenyl)cycloprop-2-en-1-one (2f)***

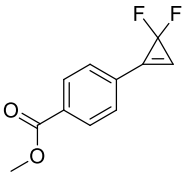
$^1$H NMR (400 MHz, Chloroform-*d*) δ 8.47 (s, 1H)*, 7.88 (d, *J* = 7.8 Hz, 1H)*, 7.25 – 7.20 (m, 5H), 7.04 (d, *J* = 8.2 Hz, 2H), 6.97 (s, 1H).

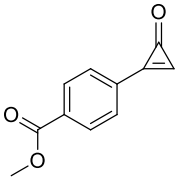



***methyl 4-(3,3-difluorocycloprop-1-en-1-yl)benzoate (1g)***

$^1$H NMR (400 MHz, Chloroform-*d*) δ 8.10 (d, *J* = 8.1 Hz, 2H), 7.67 (d, *J* = 8.1 Hz, 2H), 7.60 (s, 1H), 3.92 (s, 3H).

**Conditions 2:** DCM/MeOH(1:1) $SiO_2$, 18h – **1g**(80%), **2g**(20%)*****




**1g** **2g***

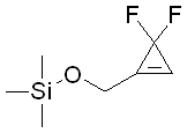
***methyl 4-(3,3-difluorocycloprop-1-en-1-yl)benzoate (1g) and methyl 4-(3-oxocycloprop-1-en-1-yl)benzoate (2g)***

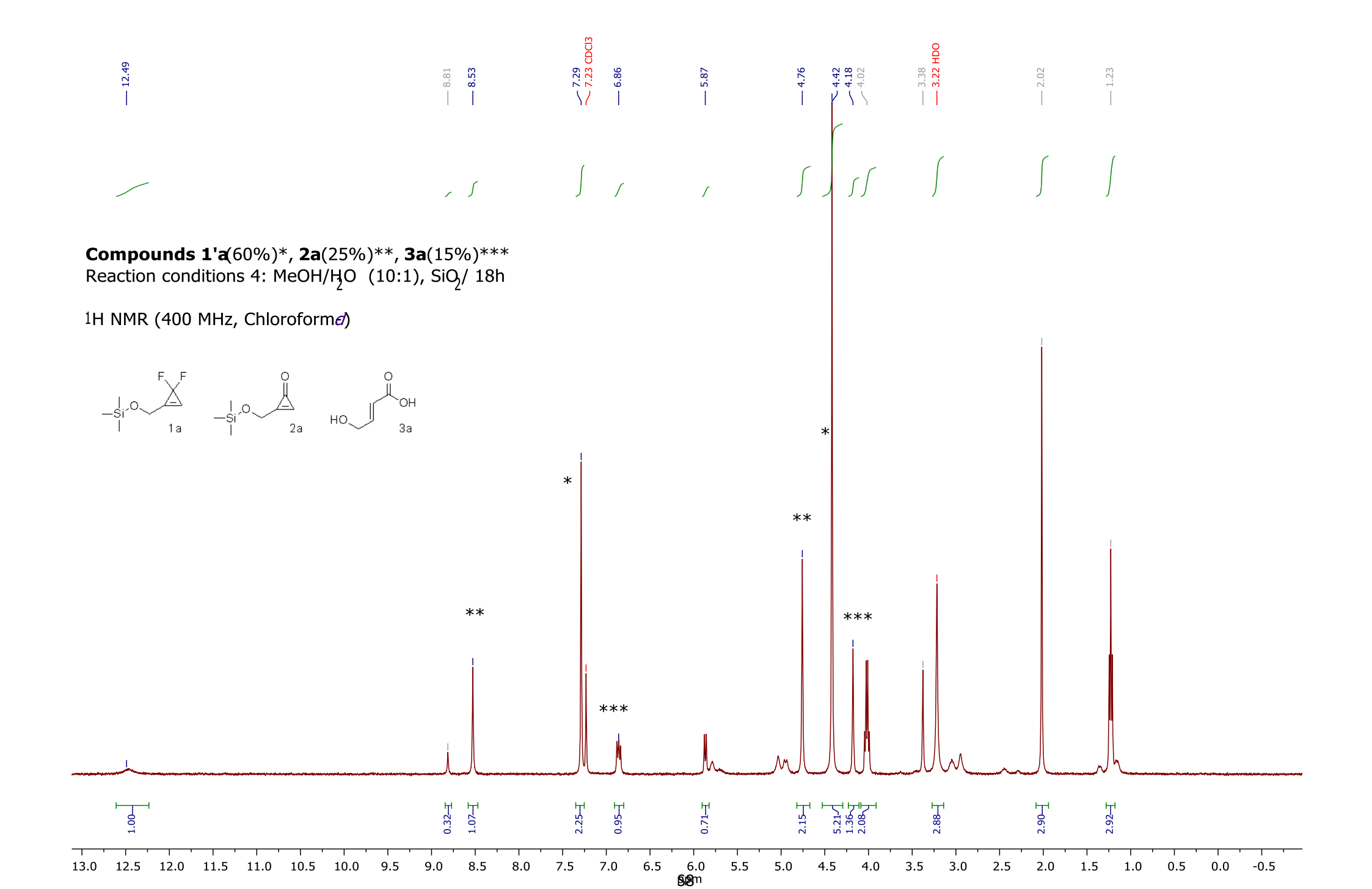
$^1$H NMR (400 MHz, Chloroform-*d*) δ 8.25 (s, 1H)*, 8.08 (d, *J* = 11.2 Hz, 2H), 7.70 (d, *J* = 3.9 Hz, 2H)*, 7.69 – 7.63 (m, 4H (2H+2H*)), 7.60 (s, 1H), 3.95 (s, 3H)*, 3.90 (s, 3H).

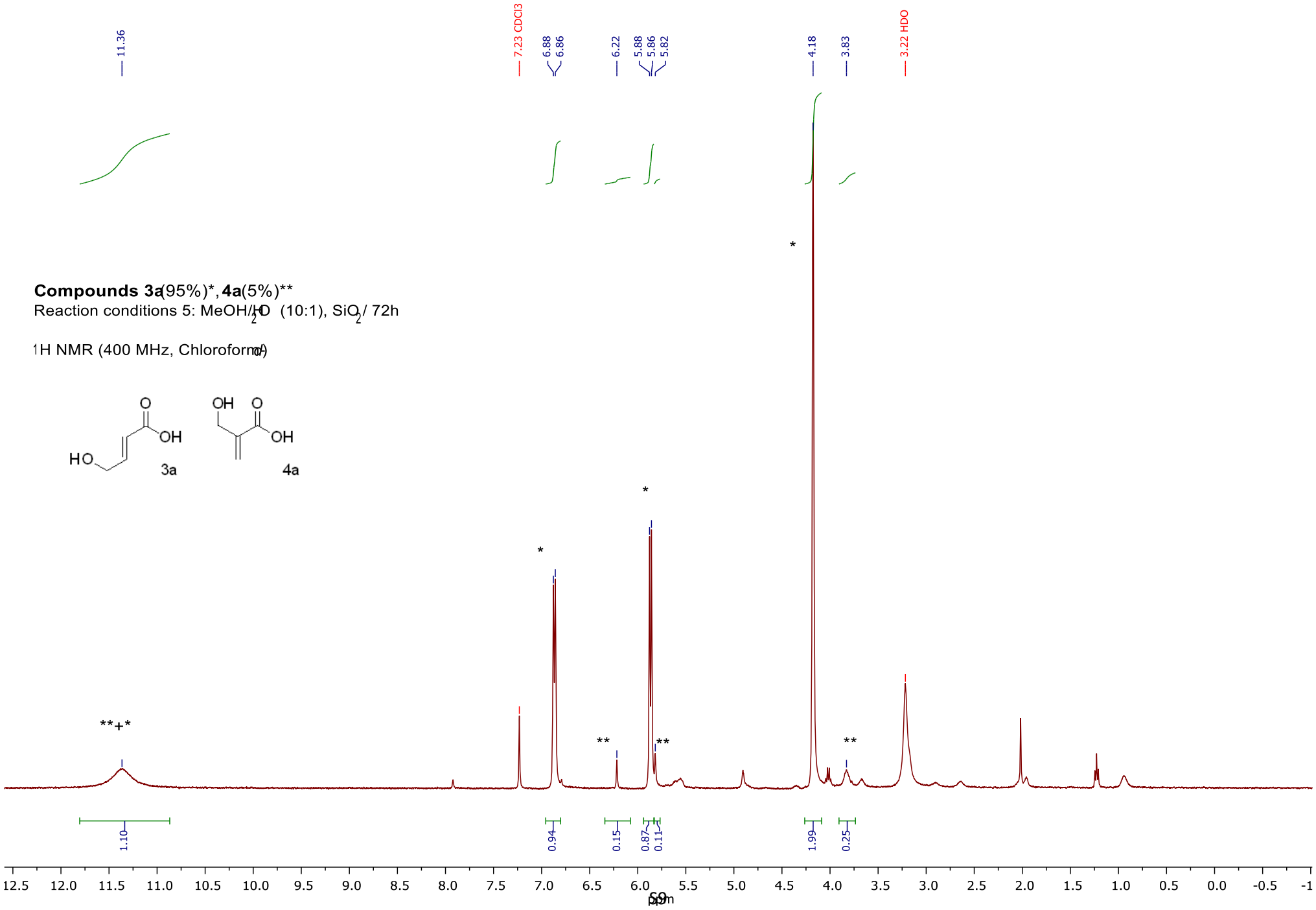
# $^{1}$H NMR Spectra

**Compound 1a**

$^{1}$H NMR (400 MHz, Chloroform-*d*)

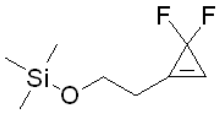


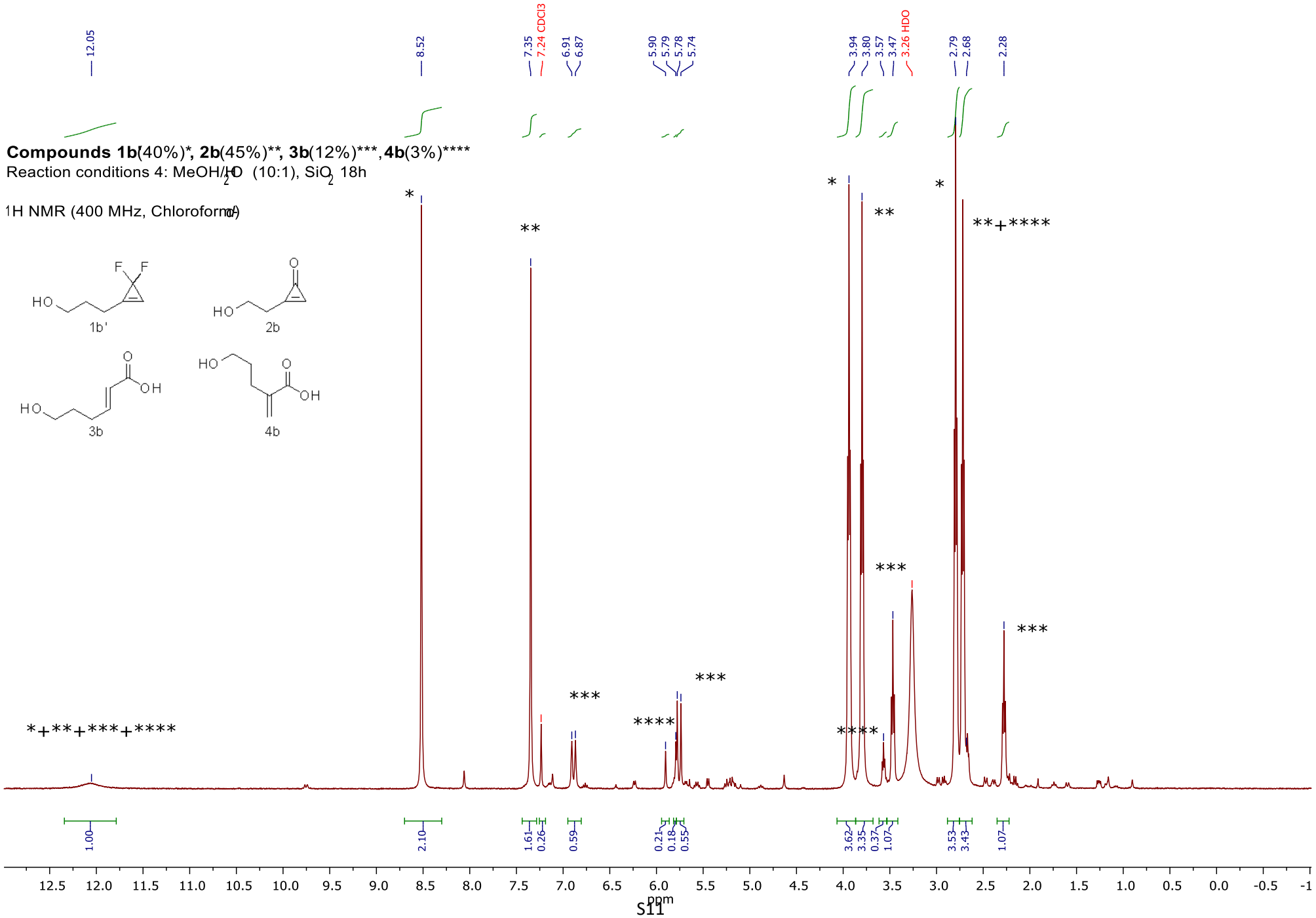

12.49
8.81
8.53
7.29
7.23 CDCl3
6.86
5.87
4.76
4.42
4.18
4.02
3.38
3.22 HDO
2.02
1.23
**Compounds 1'a**(60%)*, **2a**(25%)**, **3a**(15%)***
Reaction conditions 4: MeOH/H2O (10:1), SiO2/ 18h
1H NMR (400 MHz, Chloroform-d)
1a
2a
3a
1.00
0.32
1.07
2.25
0.95
0.71
2.15
5.21
1.36
2.08
2.88
2.90
2.92
f2 (ppm)

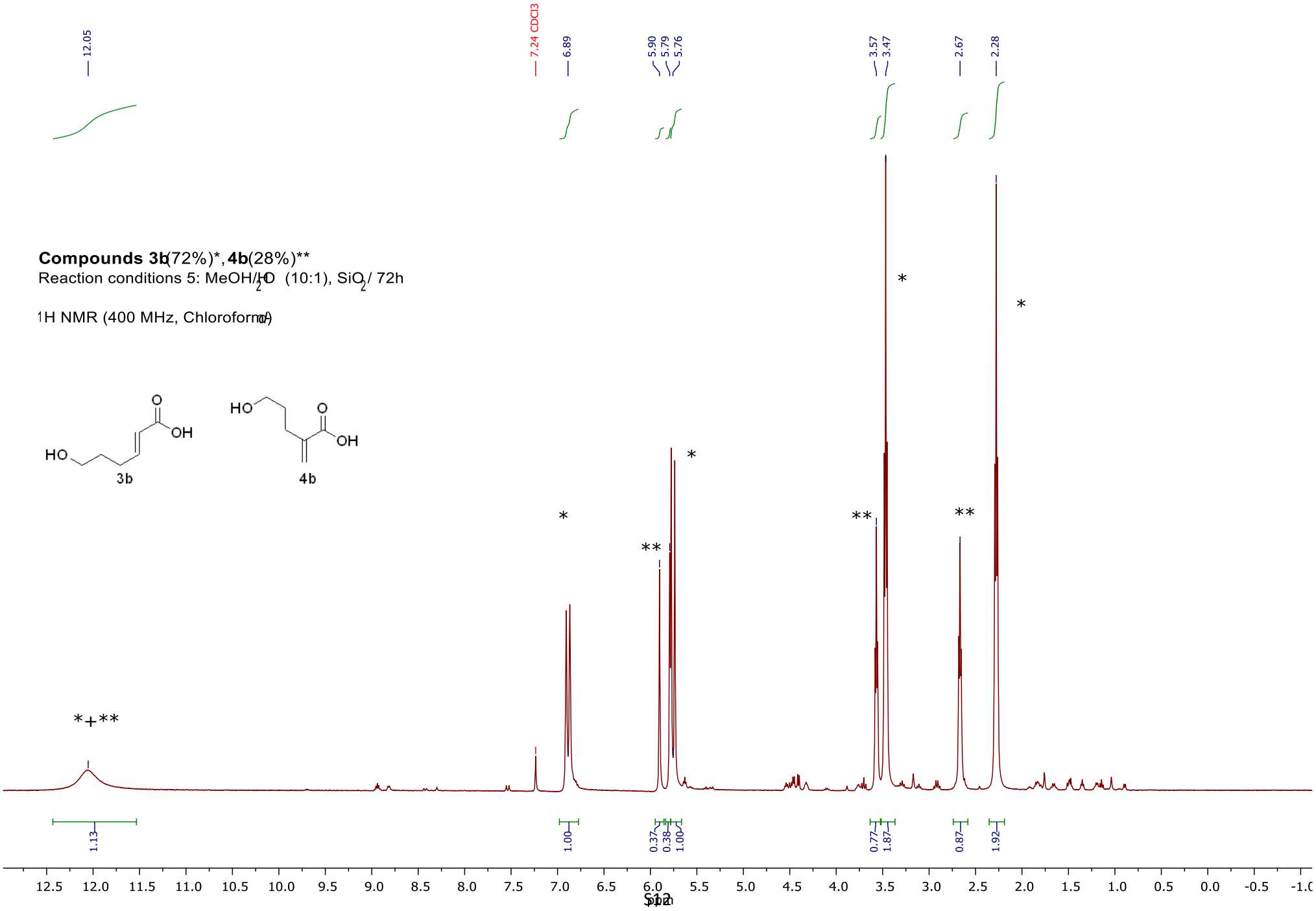

11.36
7.23 CDCl3
6.88
6.86
6.22
5.88
5.86
5.82
4.18
3.83
3.22 HDO
**Compounds 3a**(95%)*, **4a**(5%)**
Reaction conditions 5: MeOH/H2O (10:1), SiO2/ 72h
1H NMR (400 MHz, Chloroform-d)
HO
OH
O
3a
OH
OH
O
4a
**+*
**
**
**
*
*
*
1.10
0.94
0.15
0.87
0.11
1.99
0.25
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
ppm

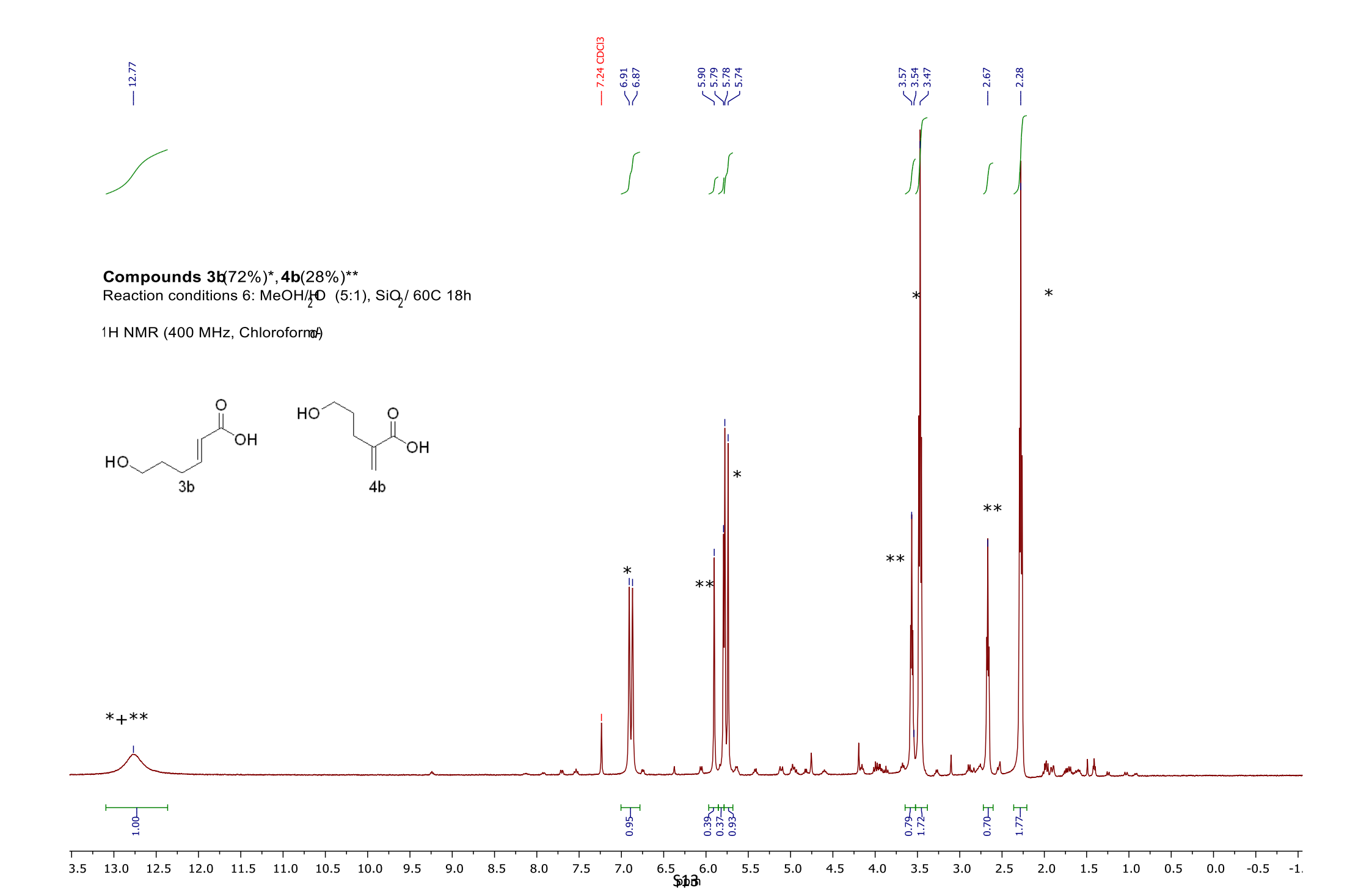
**Compound 1b**

$^1$H NMR (500 MHz, Chloroform-*d*)



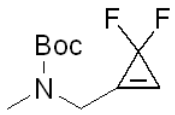

12.05
8.52
7.35
7.24 CDCl3
6.91
6.87
5.90
5.79
5.78
5.74
3.94
3.80
3.57
3.47
3.26 HDO
2.79
2.68
2.28
Compounds 1b(40%)*, 2b(45%)**, 3b(12%)***, 4b(3%)****
Reaction conditions 4: MeOH/H2O (10:1), SiO2, 18h
1H NMR (400 MHz, Chloroform-d)
1b'
2b
3b
4b
1.00
2.10
1.61
0.26
0.59
0.21
0.18
0.55
3.62
3.35
0.37
1.07
3.53
3.43
1.07
ppm


12.05
7.24 CDCl3
6.89
5.90
5.79
5.76
3.57
3.47
2.67
2.28
Compounds 3b(72%)*, 4b(28%)**
Reaction conditions 5: MeOH/H2O (10:1), SiO2/ 72h
1H NMR (400 MHz, Chloroform-d)
HO
O
OH
3b
HO
O
OH
4b
*+**
*
**
*
**
**
*
1.13
1.00
0.37
0.38
1.00
0.77
1.87
0.87
1.92
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
ppm


12.77
7.24 CDCl3
6.91
6.87
5.90
5.79
5.78
5.74
3.57
3.54
3.47
2.67
2.28
Compounds 3b(72%)*, 4b(28%)**
Reaction conditions 6: MeOH/H2O (5:1), SiO2/ 60C 18h
1H NMR (400 MHz, Chloroform-d)
HO
O
OH
3b
HO
O
OH
4b
*+**
*
**
*
**
**
*
1.00
0.95
0.39
0.37
0.93
0.79
1.72
0.70
1.77
3.5
13.0
12.5
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.
ppm

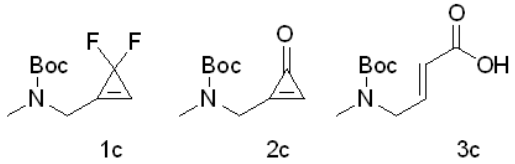
**Compound 1c**

$^1$H NMR (400 MHz, Chloroform-*d*)



**Compounds 1c**(55%)*, **2c**(35%)**, **3c**(20%)***
Reaction conditions 4: MeOH/H2O (10:1), SiO2 18h

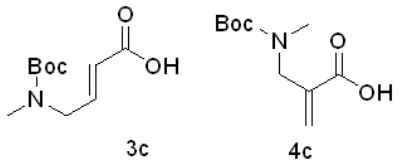
1H NMR (400 MHz, Chloroform-d)




1c 2c 3c

**Compounds 3c**(84%)*, **4c**(16%)**
Reaction conditions 5: MeOH/$H_2O$ (10:1), $SiO_2$ 72h

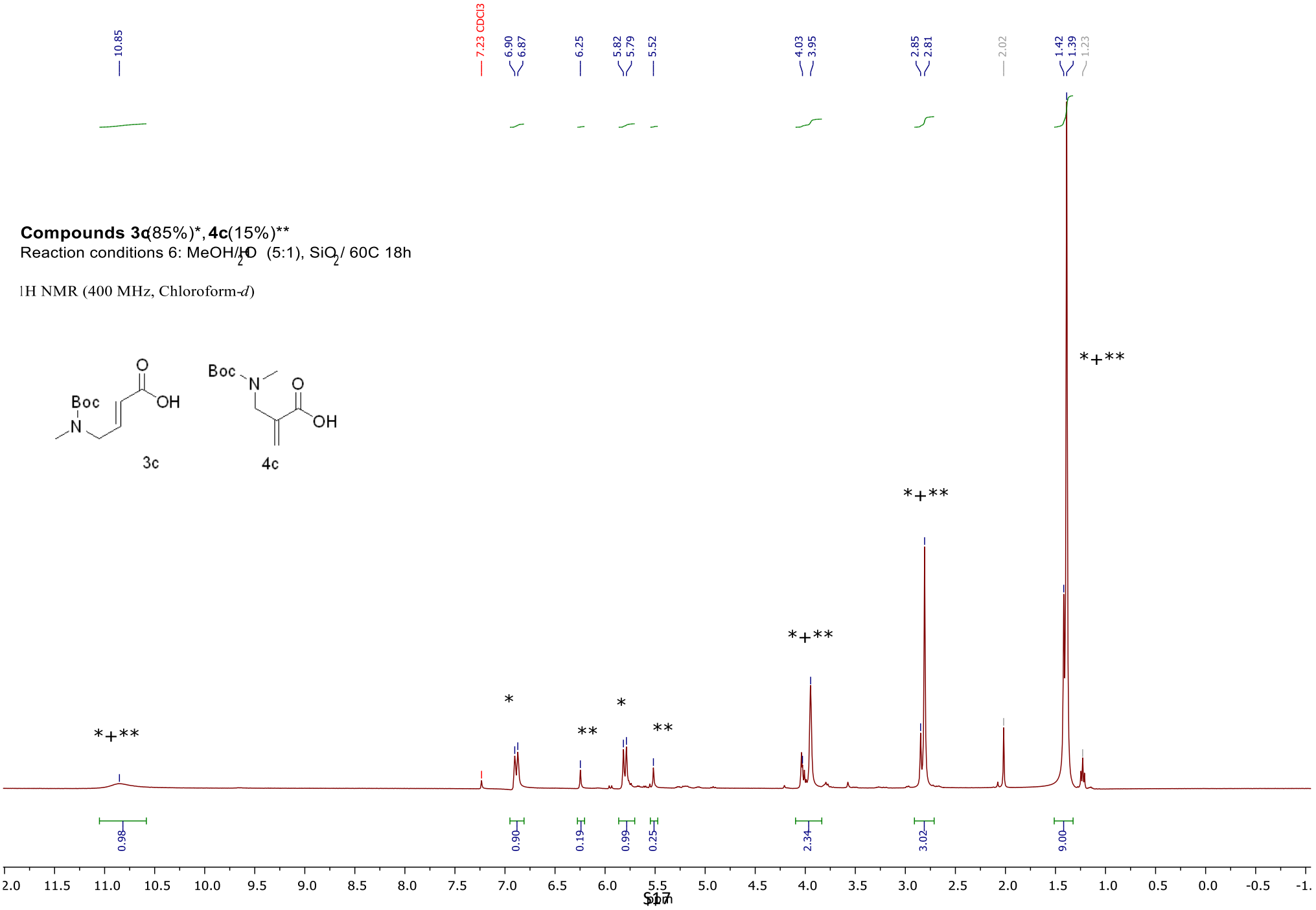
1H NMR (400 MHz, Chloroform-*d*)




3c

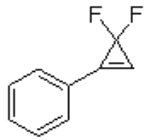


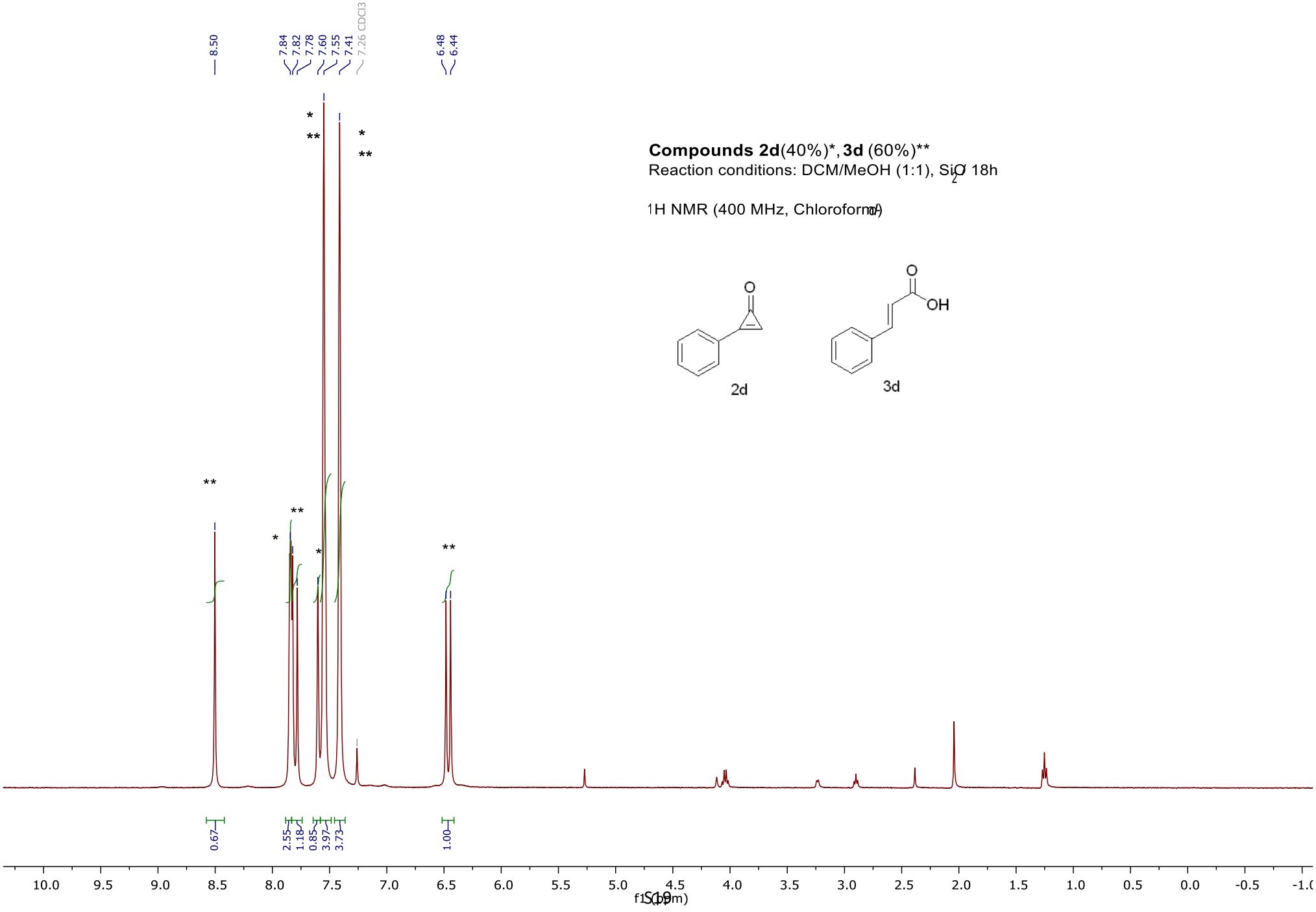

4c


10.85
7.23 CDCl3
6.90
6.87
6.25
5.82
5.79
5.52
4.03
3.95
2.85
2.81
2.02
1.42
1.39
1.23
Compounds 3c(85%)*, 4c(15%)**
Reaction conditions 6: MeOH/H2O (5:1), SiO2/ 60C 18h
1H NMR (400 MHz, Chloroform-d)
Boc
N
O
OH
3c
Boc
N
O
OH
4c
*+**
*
**
*
**
*+**
*+**
*+**
0.98
0.90
0.19
0.99
0.25
2.34
3.02
9.00
2.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.
ppm

**Compound 1d**

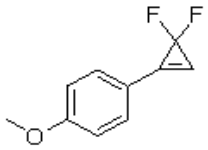
$^1$H NMR (400 MHz, Chloroform-*d*)

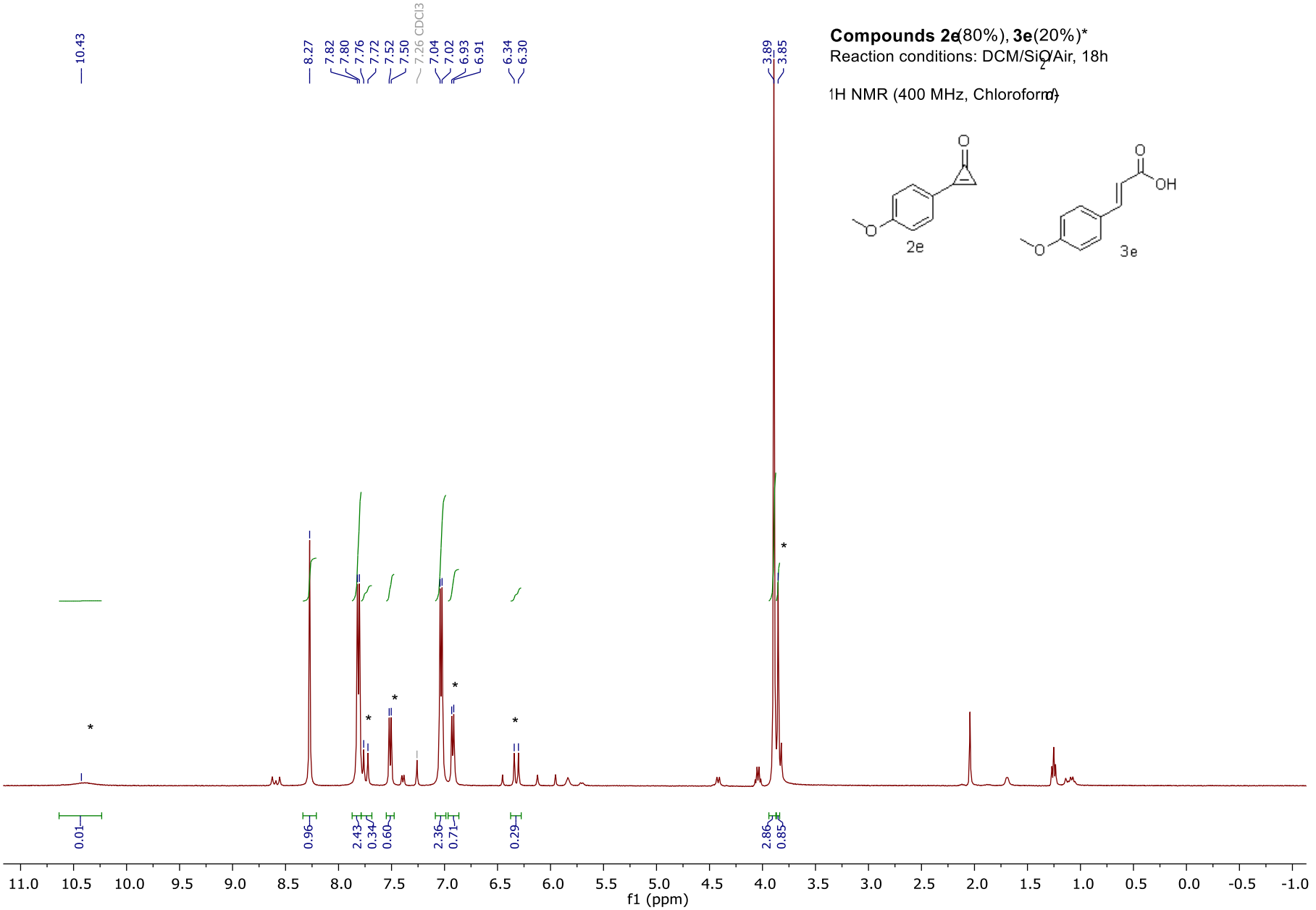


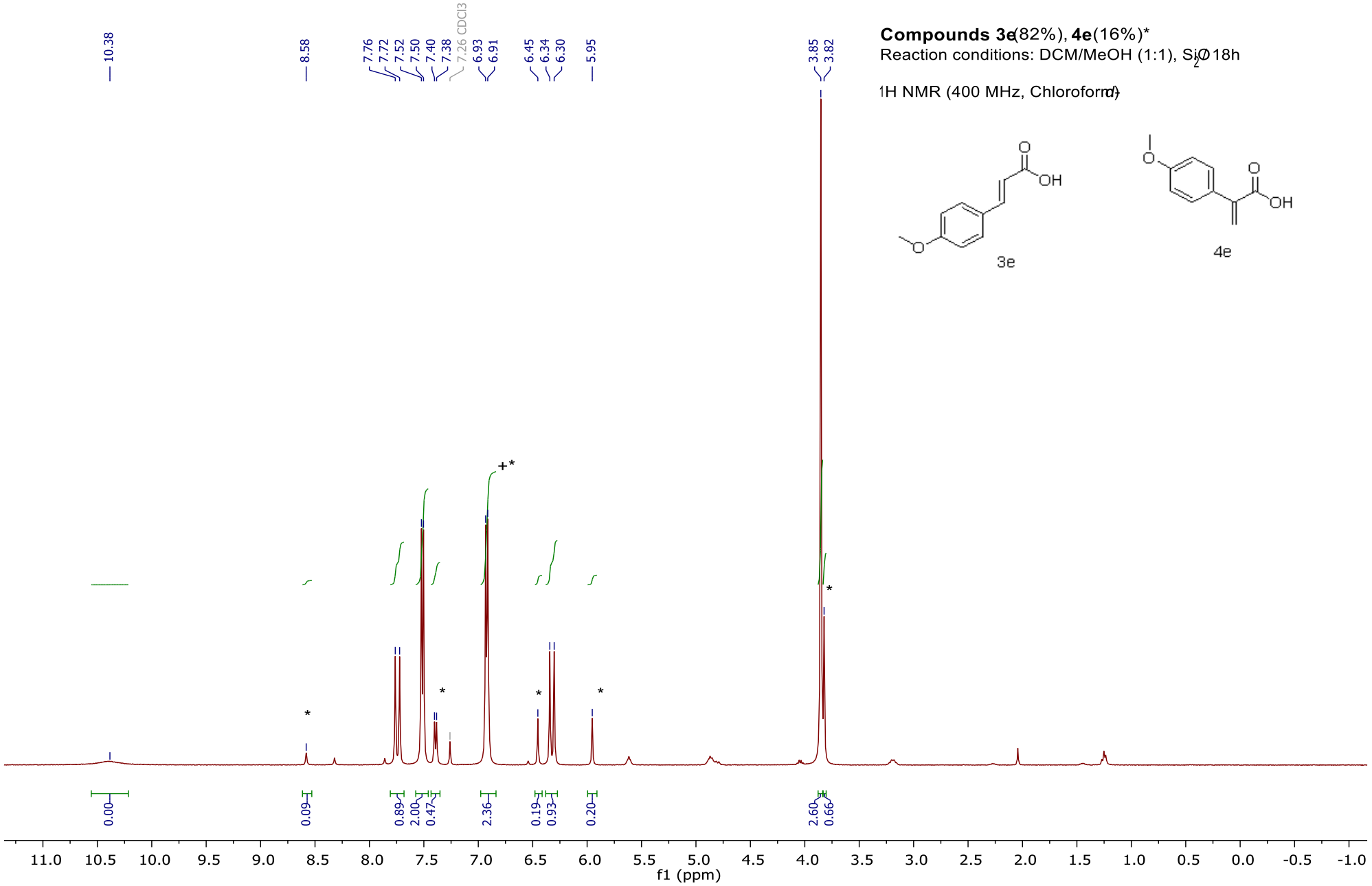

8.50
7.84
7.82
7.78
7.60
7.55
7.41
7.26 CDCl3
6.48
6.44
Compounds 2d(40%)*, 3d (60%)**
Reaction conditions: DCM/MeOH (1:1), SiO2 18h
1H NMR (400 MHz, Chloroform-d)
O
O
OH
2d
3d
0.67
2.55
1.18
0.85
3.97
3.73
1.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

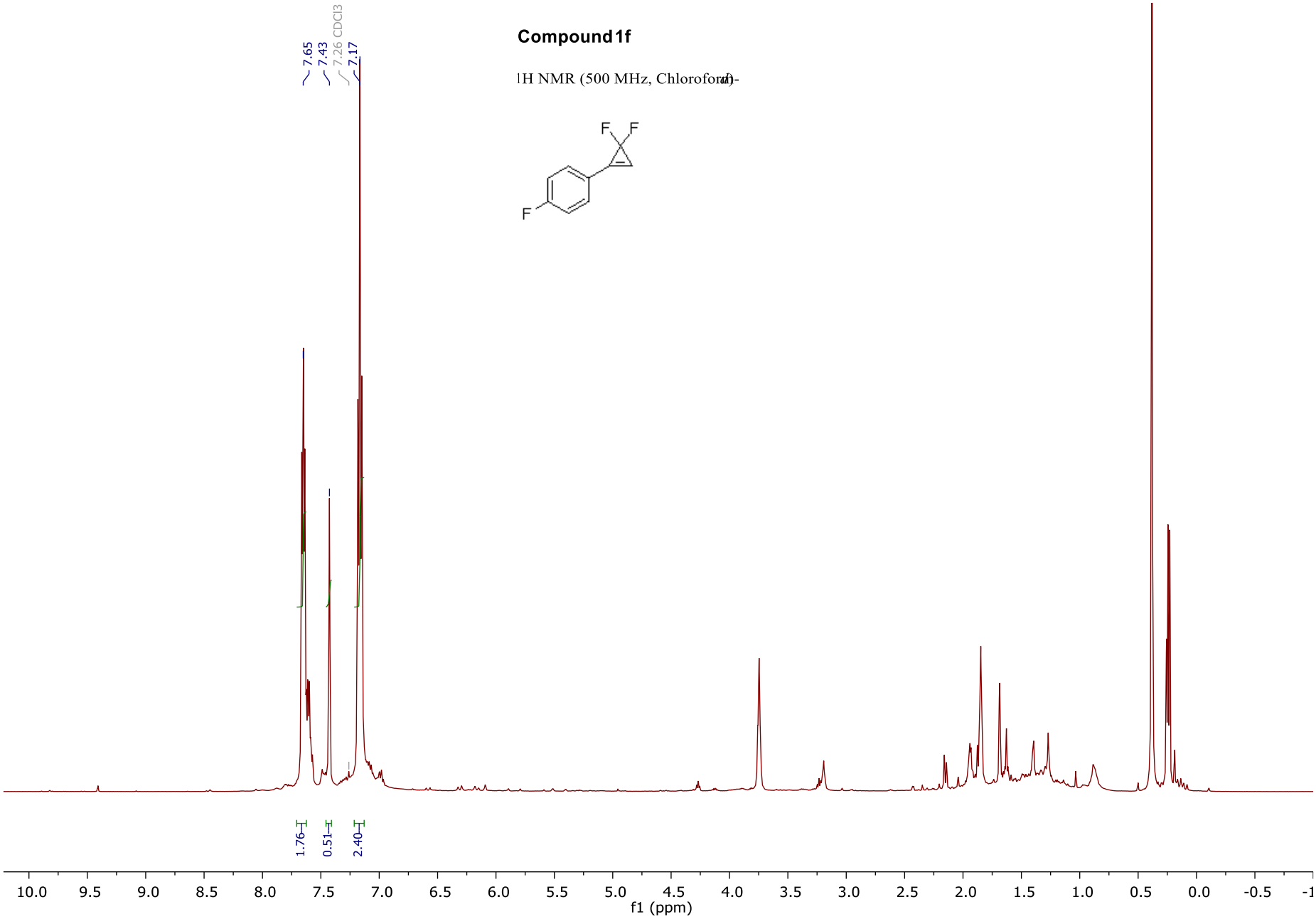
**Compound 1e**

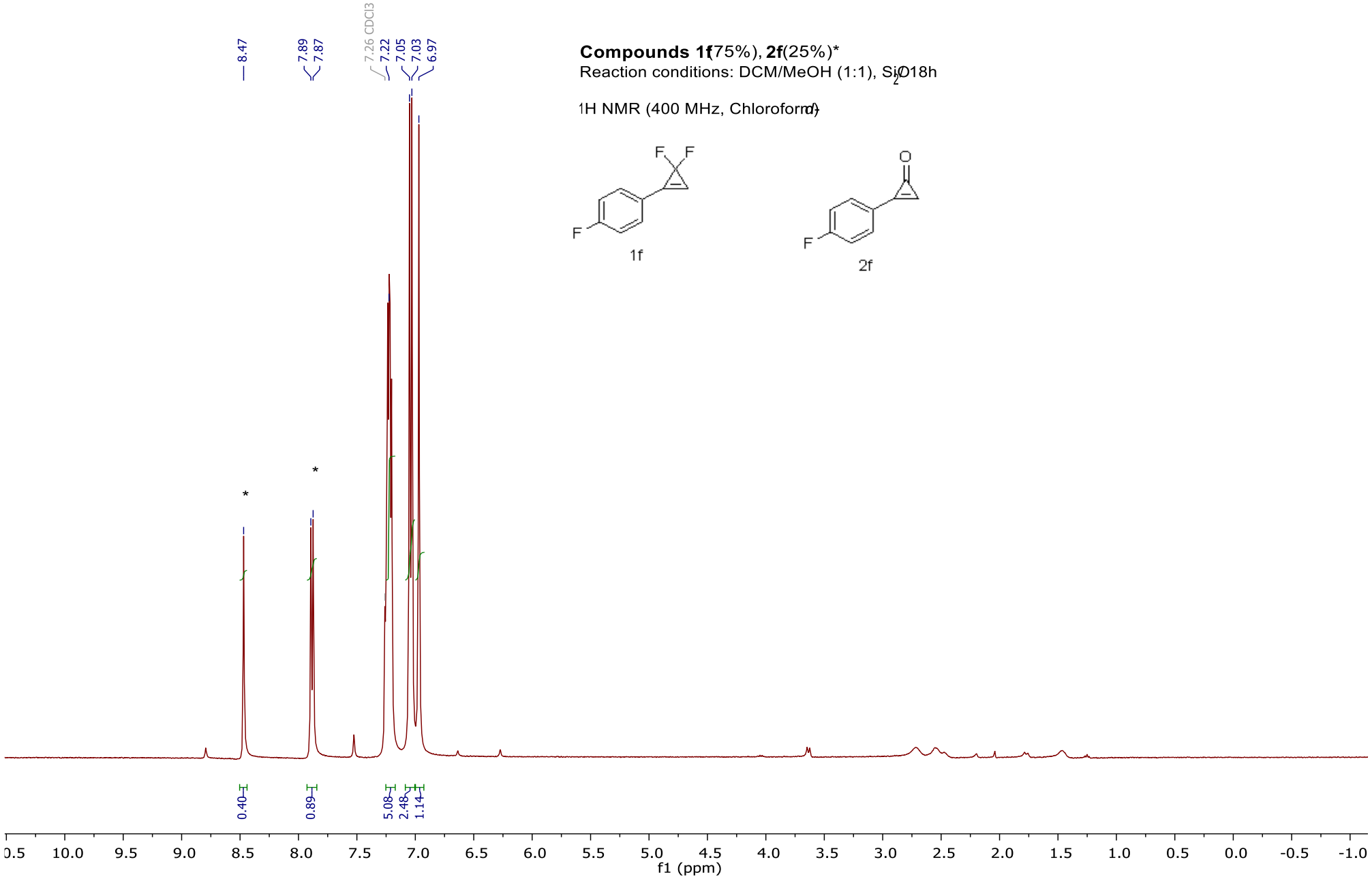
$^1$H NMR (400 MHz, Chloroform-*d*)




Compounds 2e(80%), 3e(20%)*
Reaction conditions: DCM/SiO2/Air, 18h
1H NMR (400 MHz, Chloroform-d)
2e
3e
OH
10.43
8.27
7.82
7.80
7.76
7.72
7.52
7.50
7.26 CDCl3
7.04
7.02
6.93
6.91
6.34
6.30
3.89
3.85
0.01
0.96
2.43
0.34
0.60
2.36
0.71
0.29
2.86
0.85
f1 (ppm)


Compounds 3e (82%), 4e (16%)*
Reaction conditions: DCM/MeOH (1:1), SiO2, 18h
1H NMR (400 MHz, Chloroform-d)
3e
4e
10.38
8.58
7.76
7.72
7.52
7.50
7.40
7.38
7.26 CDCl3
6.93
6.91
6.45
6.34
6.30
5.95
3.85
3.82
0.00
0.09
0.89
2.00
0.47
2.36
0.19
0.93
0.20
2.60
0.66
f1 (ppm)


Compound 1f
1H NMR (500 MHz, Chloroform-d)
7.65
7.43
7.26 CDCl3
7.17
F
F
F
1.76
0.51
2.40
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1
f1 (ppm)




**Compounds 1f**(75%), **2f**(25%)*
Reaction conditions: DCM/MeOH (1:1), $SiO_2$, 18h

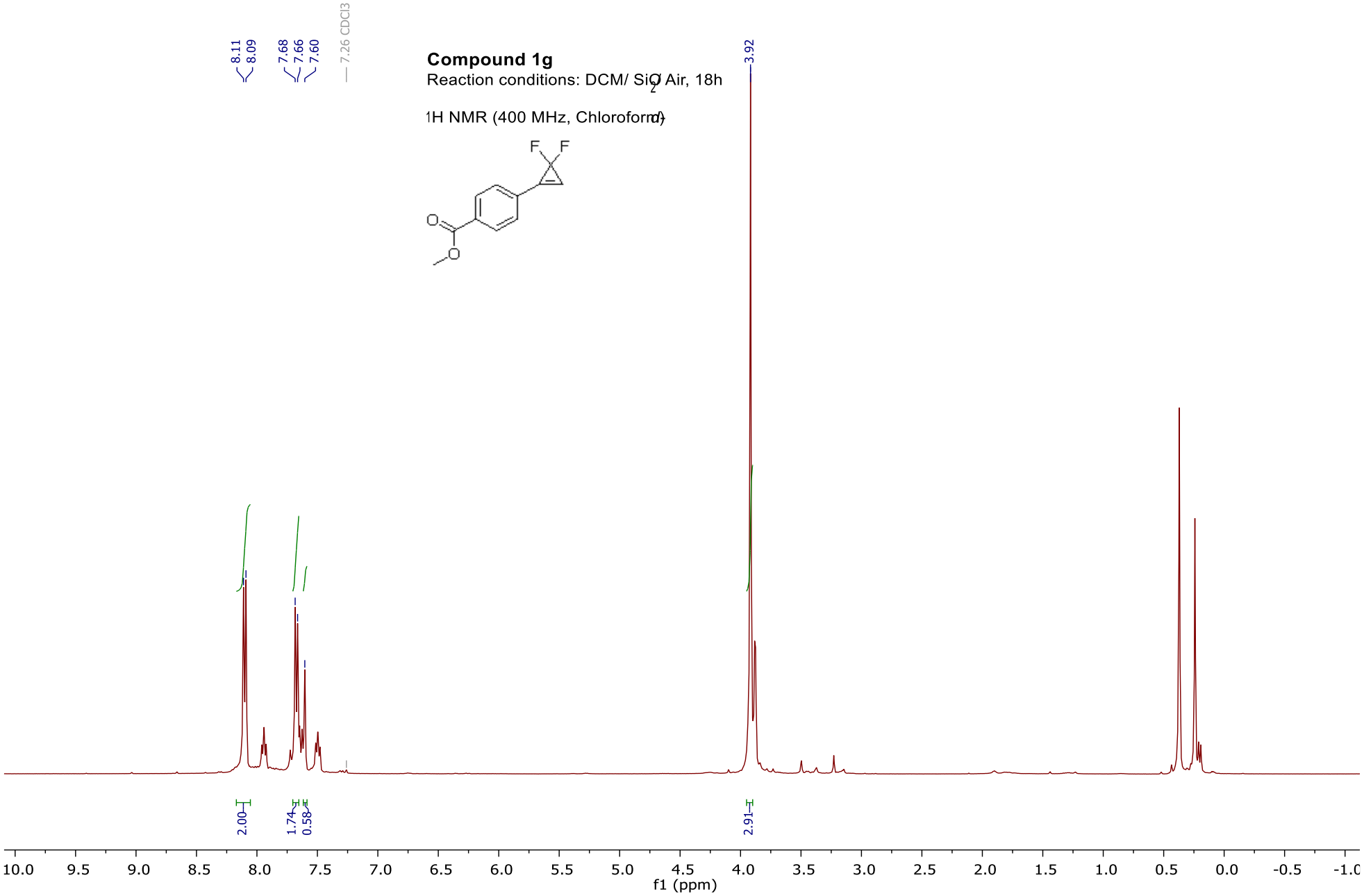
1H NMR (400 MHz, Chloroform-*d*)

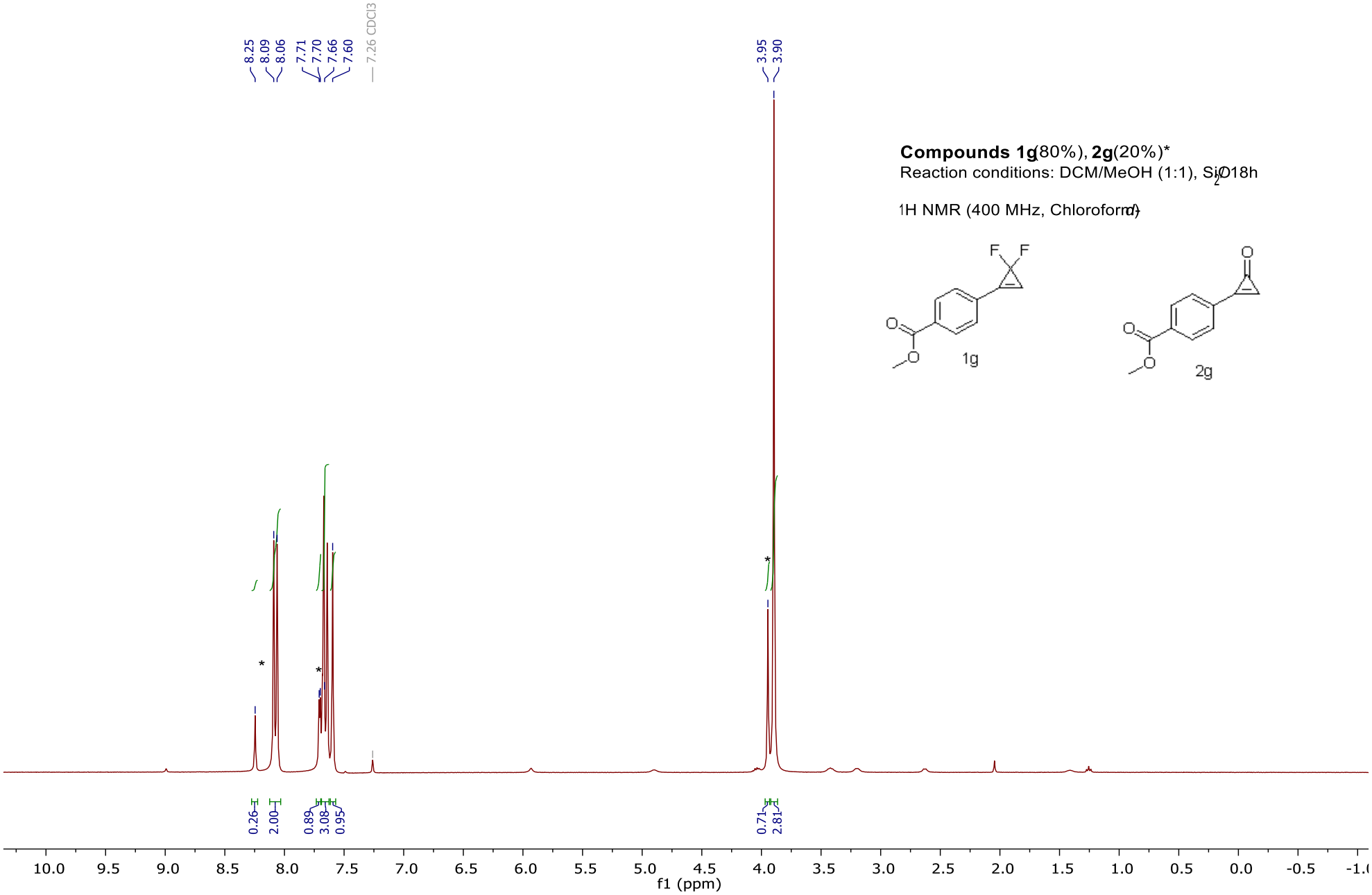



1f




2f


Compound 1g
Reaction conditions: DCM/ SiO2 Air, 18h
1H NMR (400 MHz, Chloroform-d)
8.11
8.09
7.68
7.66
7.60
7.26 CDCl3
3.92
F
F
O
O
2.00
1.74
0.58
2.91
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


8.25
8.09
8.06
7.71
7.70
7.66
7.60
7.26 CDCl3
3.95
3.90
Compounds 1g(80%), 2g(20%)*
Reaction conditions: DCM/MeOH (1:1), SiO2, 18h
1H NMR (400 MHz, Chloroform-d)
F F
O
O
1g
O
O
2g
0.26
2.00
0.89
3.08
0.95
0.71
2.81
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)