

Ülesanne 3

TMR-katseklaasis segati kokku väike kogus erinevaid puhtaid orgaanilisi lahusteid ja lisati 0,5 mL deutereeritud kloroformi (99,8% deuteriumrikastusega CDCl_3).

Mõõdeti ^1H (400,1 MHz) ja $^{13}\text{C}\{^1\text{H}\}$ (100,6 MHz) TMR spektrid. Need spektrid ja suurendused nendest on toodud järgnevatel lehtedel.

Raportisse kirjutada:

1. Kasutage viidetes 1 või 2 toodud tabelleid või muid saadaolevaid tabelleid ning leidke, mis orgaanilisi lahusteid kokku segati. Nimekiri esitage tabeli kujul:

Ühendi nimetus	Ühendile kuuluvad ^1H TMR signaalide keemilised nihked	Ühendile kuuluvad $^{13}\text{C}\{^1\text{H}\}$ TMR signaalide keemilised nihked
Trikloro(^2H)metaan	–	77,0 ppm
<i>jne.</i>		

NB! Arvestage, et tabelis olevad puhaste ainete keemiliste nihete väärtused on veidi erinevad reaalselt solventide segus mõõdetud väärtustest. Samuti pange tähele, et spektris peavad olema olema kõikide ühe molekuli fragmentide signaalid ning ka signaalide multipletsused peavad klappima. Mõni signaal võib jääda määramata, aga enamik on kindlasti tuvastatavad.

2. Millele vastab $^{13}\text{C}\{^1\text{H}\}$ TMR spektris väike singlett keemilise nihkega 77,19 ppm?
Joonistage selle aine struktuurivalem koos isotooptähistega:

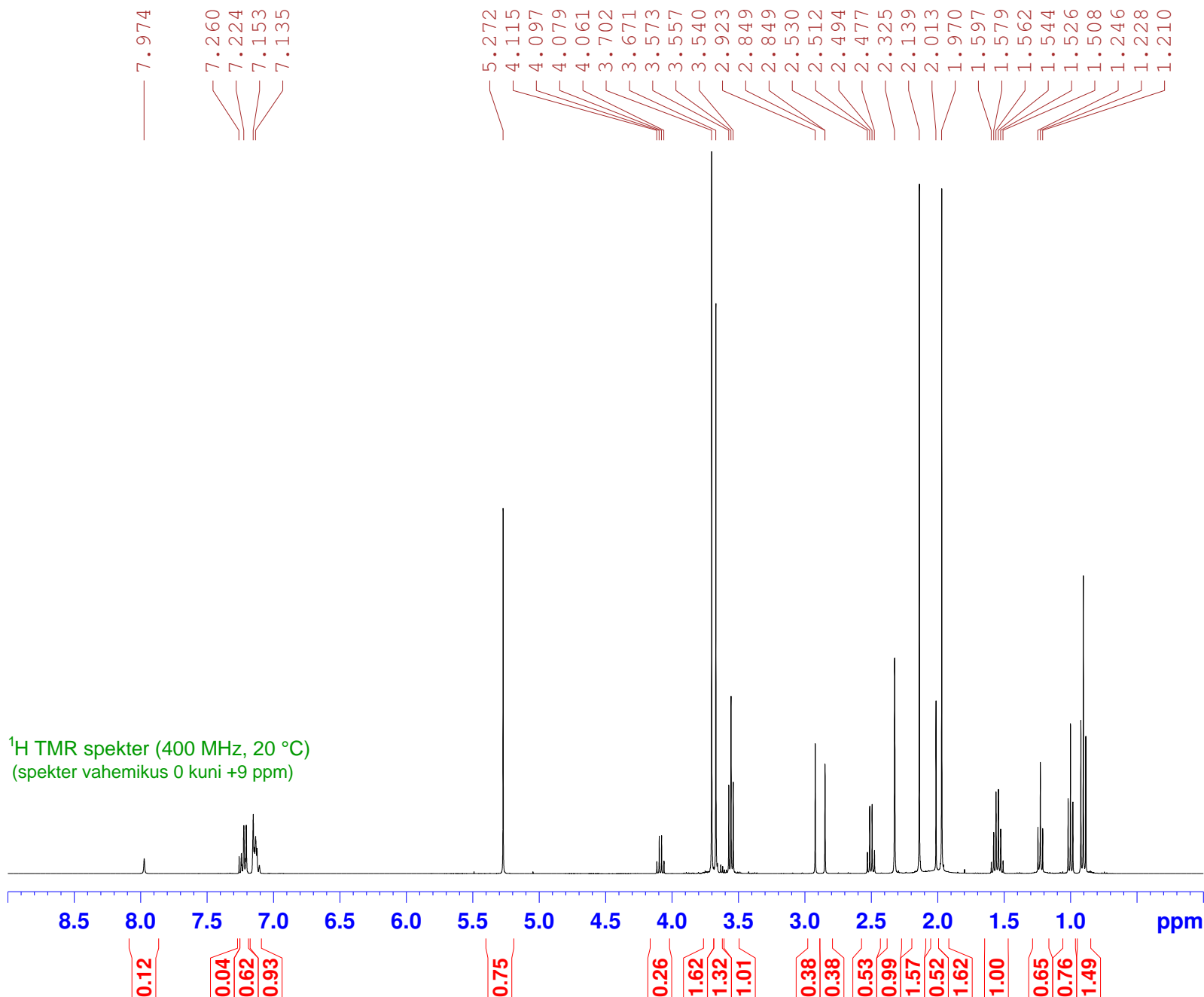


Viited:

1. Fulmer, G.R.; Miller, A.J.M.; Sherden, N.H.; Gottlieb, H.E.; Nudelman, A.; Stoltz, B.M.; Bercaw, J.E.; Goldberg, K.I. NMR Chemical Shifts of Trace Impurities: Common Laboratory Solvents, Organics, and Gases in Deuterated Solvents Relevant to the Organometallic Chemist. *Organometallics* **2010**, *29*, 2176-2179.
DOI: 10.1021/om100106e
2. Gottlieb, H. E.; Kotlyar, V.; Nudelman, A. NMR Chemical Shifts of Common Laboratory Solvents as Trace Impurities. *J. Org. Chem.* **1997**, *62*, 7512-7515.
DOI: 10.1021/jo971176v

Ülesanne on kättesaadav PDF-formaadis aadressilt: <http://kodu.ut.ee/~laurit/AK2/>

Raportit soovin saada oma e-posti aadressile lauri.toom@ut.ee ainult PDF-formaadis. Raporti 1. versiooni esitamise tähtaeg on 15. mai 2015 kell 17:00. Minupoolse tagasiside põhjal korrektselt parandatud lõpp-versiooni esitamise tähtaeg on 29. mai 2015 kell 17:00.



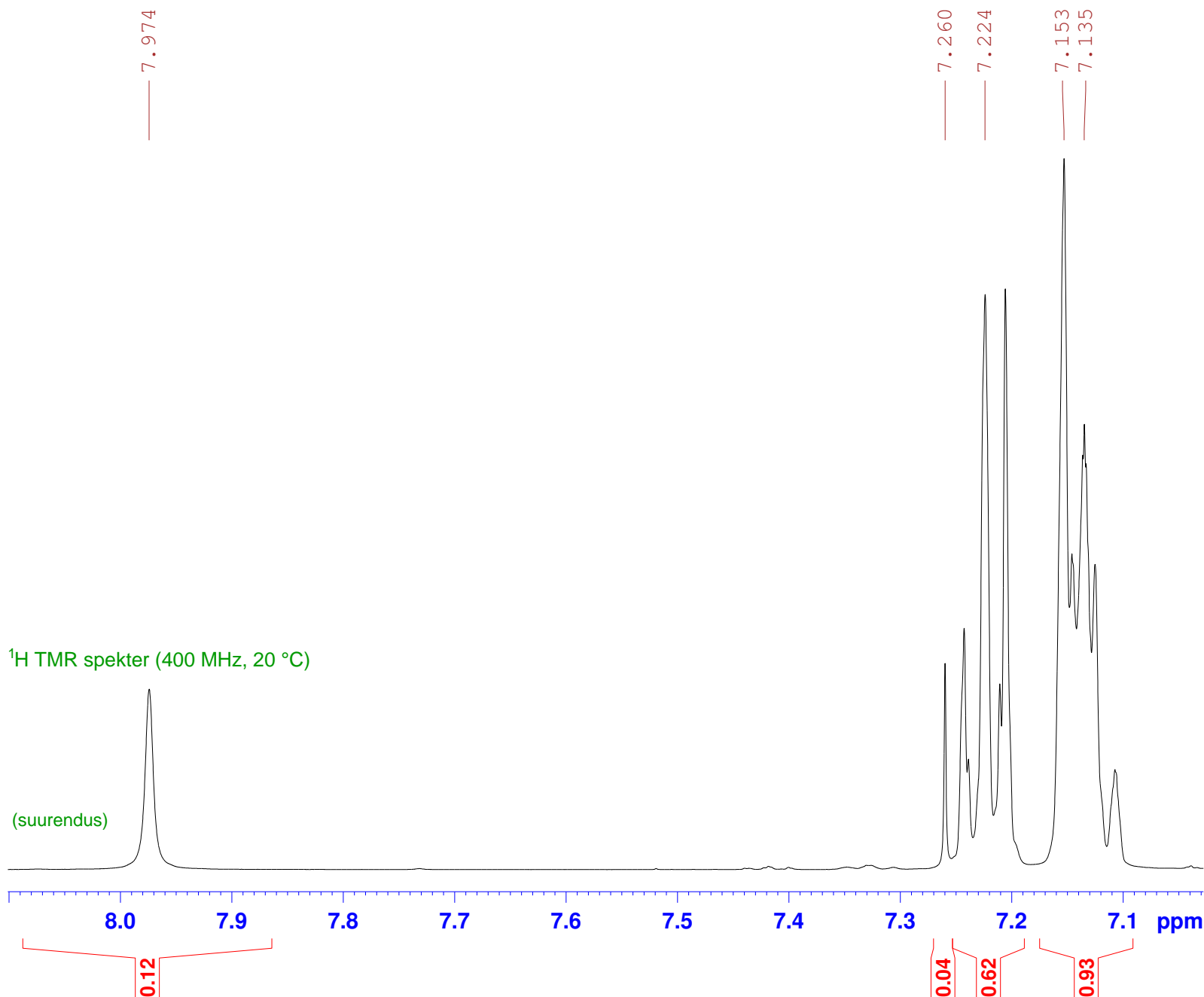
```

Current Data Parameters
NAME           Solvents
EXPNO          103
PROCNO         1

F2 - Acquisition Parameters
Date_          20100603
Time           15.53
INSTRUM        spect
PROBHD         5 mm BBO BB-1H
PULPROG        zg30
TD             88064
SOLVENT        CDC13
NS             256
DS             4
SWH            7211.539 Hz
FIDRES         0.081890 Hz
AQ             6.1058207 sec
RG             71.8
DW             69.333 usec
DE             6.00 usec
TE             293.2 K
D1             3.0000000 sec
TD0            1

===== CHANNEL f1 =====
NUC1           1H
P1             14.35 usec
PL1            0.00 dB
SFO1           400.1324008 MHz

F2 - Processing parameters
SI             131072
SF             400.1300176 MHz
WDW            EM
SSB            0
LB             0.30 Hz
GB             0
PC             1.00
    
```

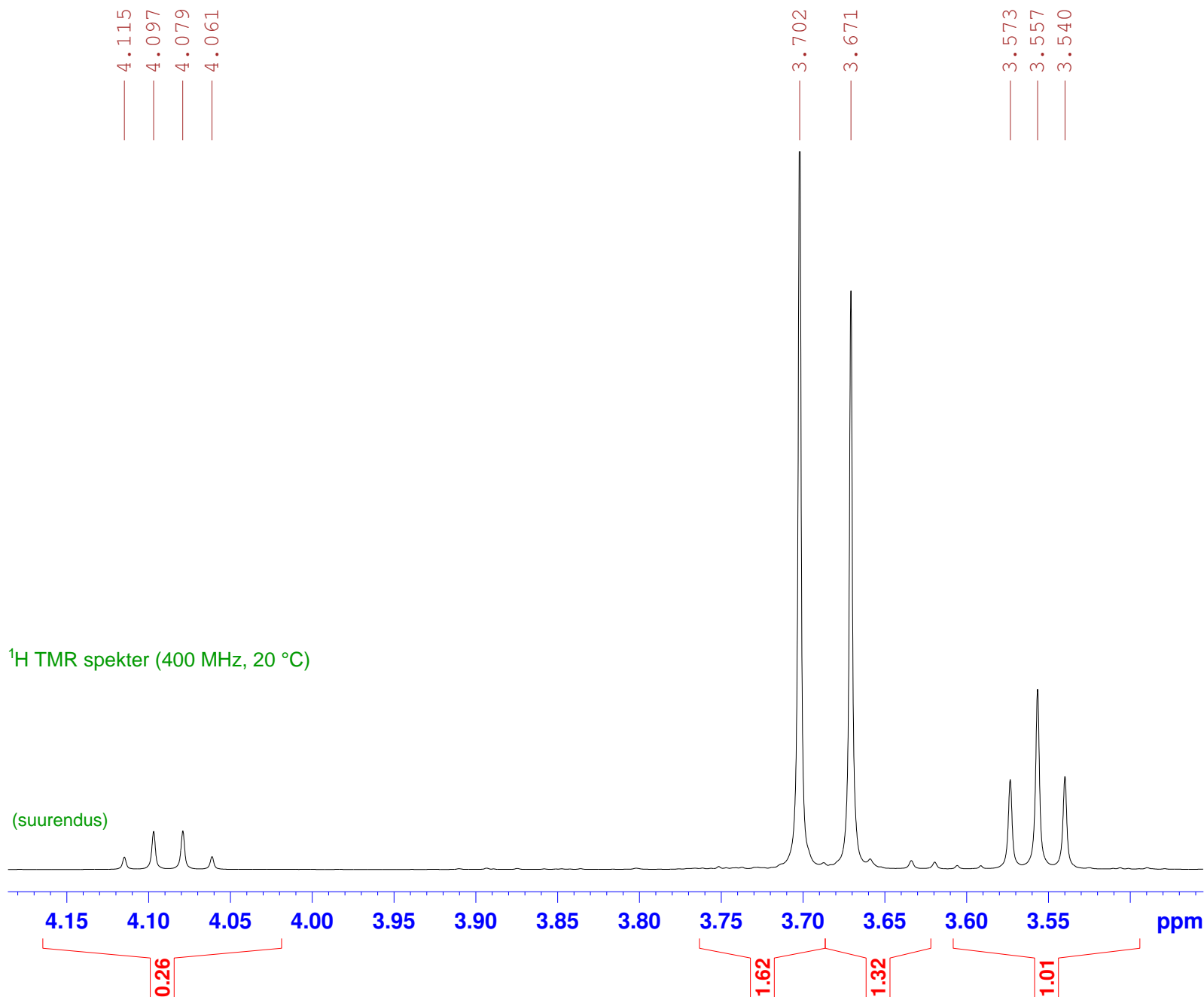


Current Data Parameters
NAME Solvents
EXPNO 103
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100603
Time 15.53
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 88064
SOLVENT CDC13
NS 256
DS 4
SWH 7211.539 Hz
FIDRES 0.081890 Hz
AQ 6.1058207 sec
RG 71.8
DW 69.333 usec
DE 6.00 usec
TE 293.2 K
D1 3.0000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.35 usec
PL1 0.00 dB
SFO1 400.1324008 MHz

F2 - Processing parameters
SI 131072
SF 400.1300176 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

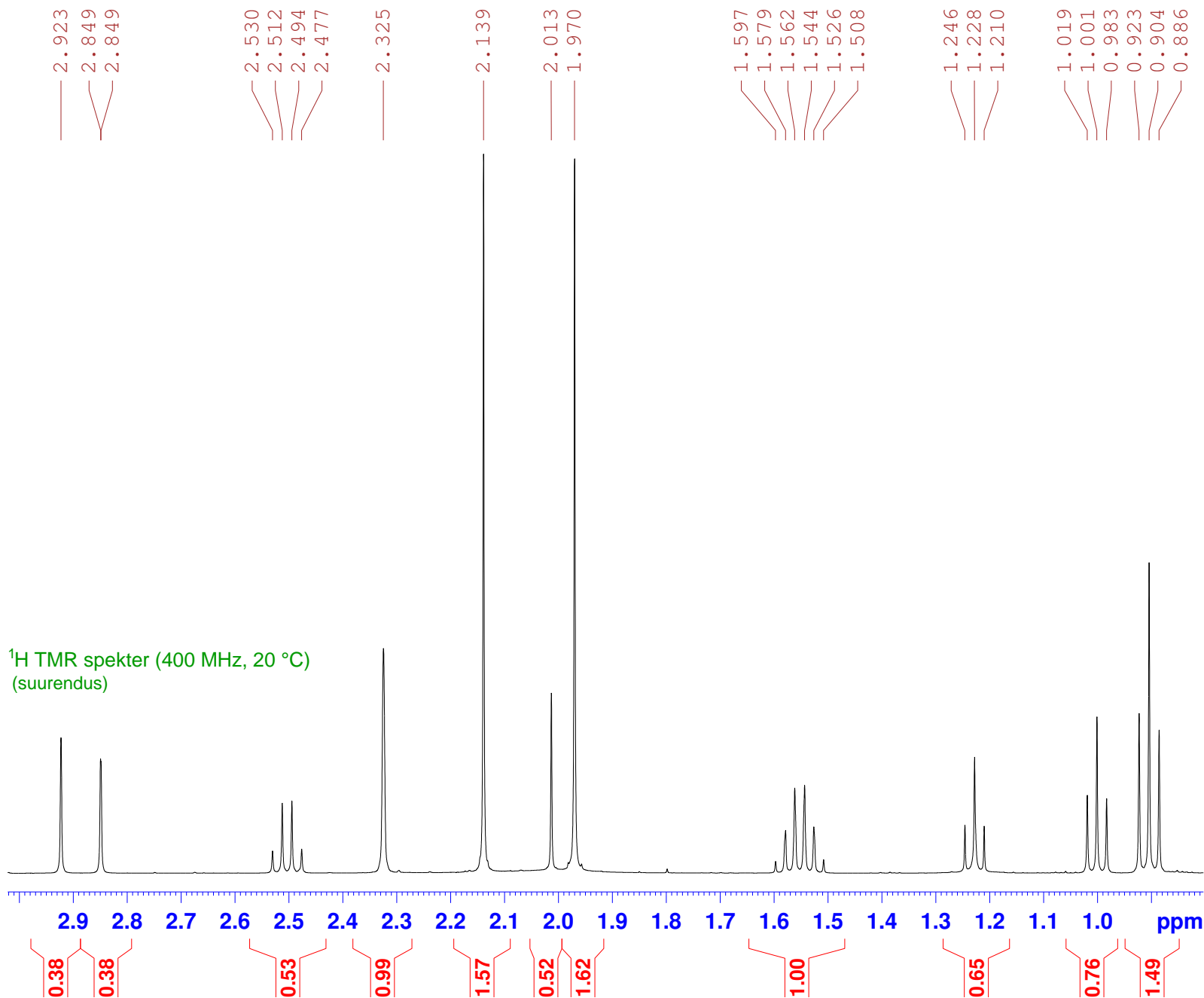


Current Data Parameters
NAME Solvents
EXPNO 103
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100603
Time 15.53
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 88064
SOLVENT CDC13
NS 256
DS 4
SWH 7211.539 Hz
FIDRES 0.081890 Hz
AQ 6.1058207 sec
RG 71.8
DW 69.333 usec
DE 6.00 usec
TE 293.2 K
D1 3.0000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.35 usec
PL1 0.00 dB
SFO1 400.1324008 MHz

F2 - Processing parameters
SI 131072
SF 400.1300176 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

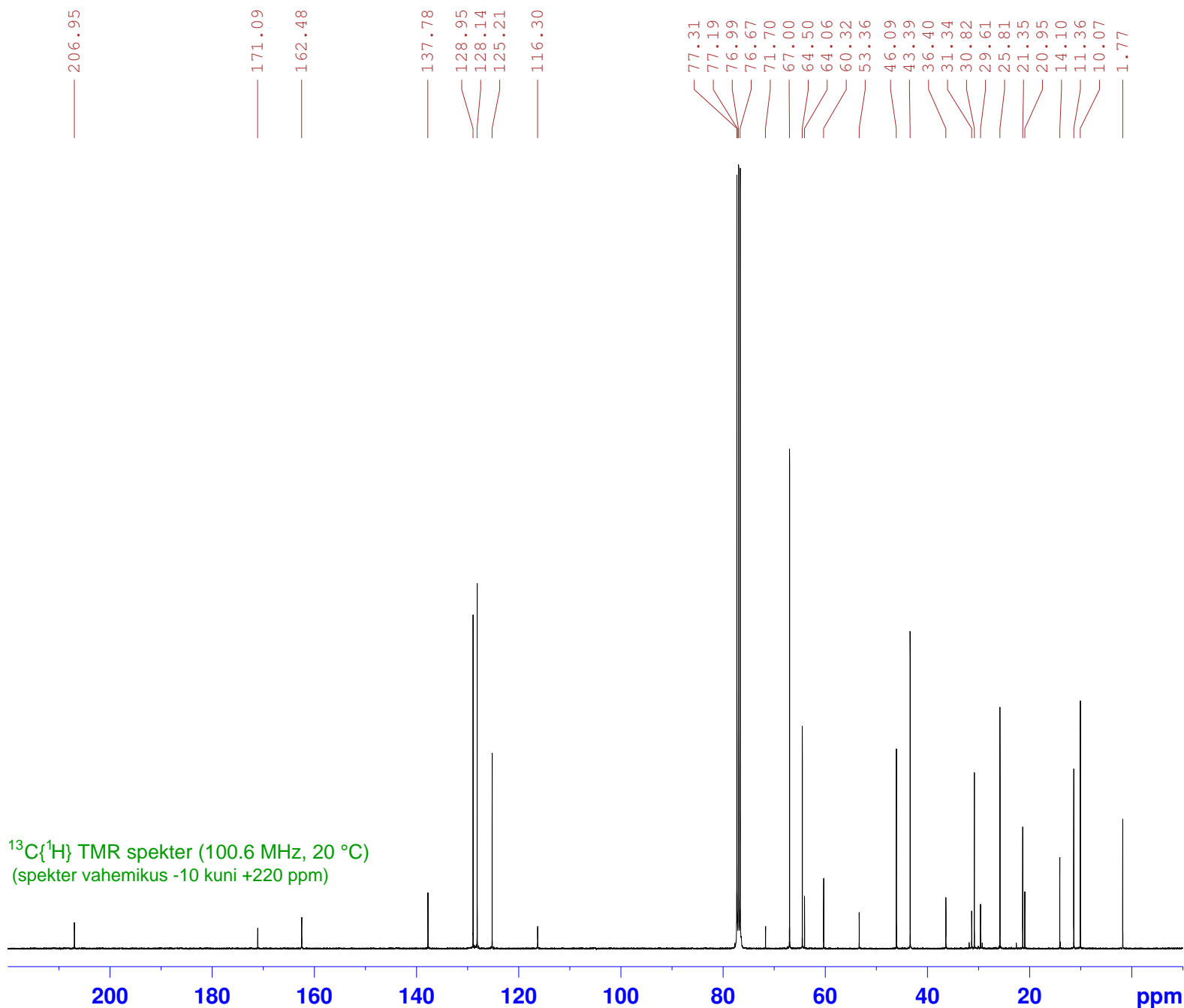


Current Data Parameters
NAME Solvents
EXPNO 103
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100603
Time 15.53
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 88064
SOLVENT CDC13
NS 256
DS 4
SWH 7211.539 Hz
FIDRES 0.081890 Hz
AQ 6.1058207 sec
RG 71.8
DW 69.333 usec
DE 6.00 usec
TE 293.2 K
D1 3.0000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 1H
P1 14.35 usec
PL1 0.00 dB
SFO1 400.1324008 MHz

F2 - Processing parameters
SI 131072
SF 400.1300176 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



$^{13}\text{C}\{^1\text{H}\}$ TMR spekter (100.6 MHz, 20 °C)
(spekter vahemikus -10 kuni +220 ppm)

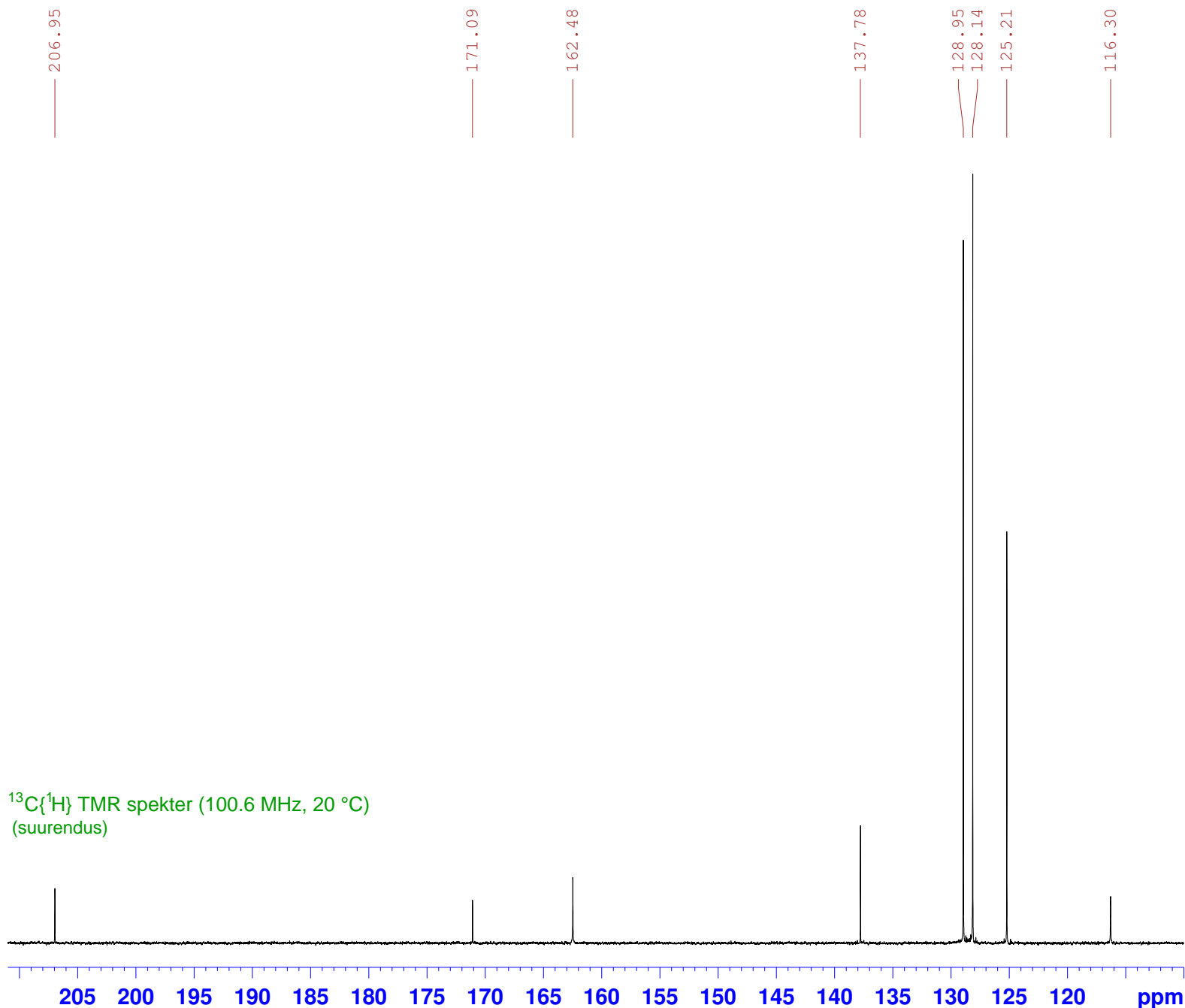
Current Data Parameters
NAME Solvents
EXPNO 106
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100702
Time 20.07
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 73728
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 32800
DW 20.800 usec
DE 6.00 usec
TE 293.5 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 9.50 usec
PL1 -2.00 dB
SFO1 100.6233333 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL12 13.76 dB
PL13 14.00 dB
PL2 0.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 65536
SF 100.6127789 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



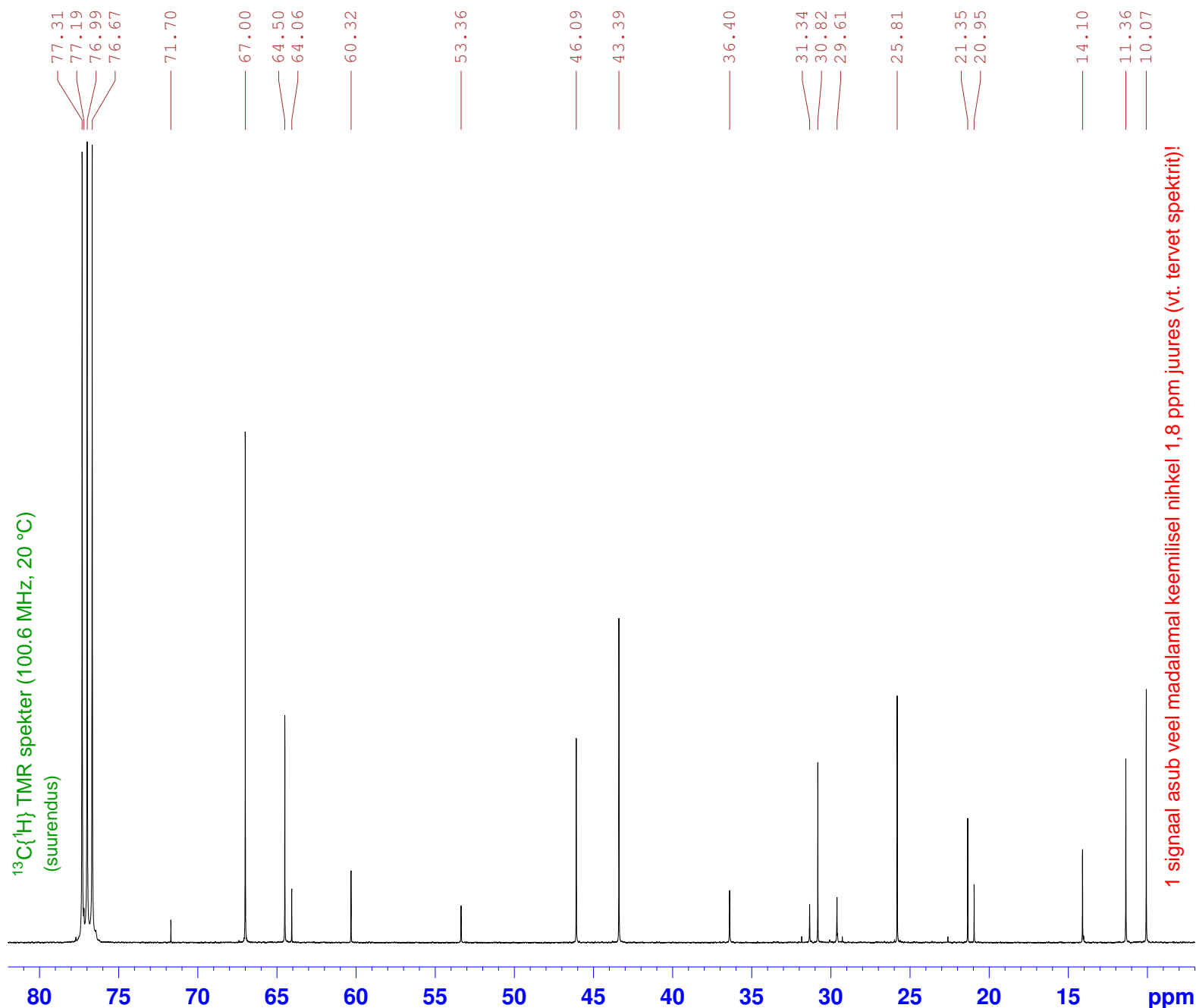
Current Data Parameters
NAME Solvents
EXPNO 106
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100702
Time 20.07
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 73728
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 32800
DW 20.800 usec
DE 6.00 usec
TE 293.5 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 9.50 usec
PL1 -2.00 dB
SFO1 100.6233333 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL12 13.76 dB
PL13 14.00 dB
PL2 0.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 65536
SF 100.6127789 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



1 signaal asub veel madalamal keemilisel nihkel 1,8 ppm juures (vt. tervet spektrit)!

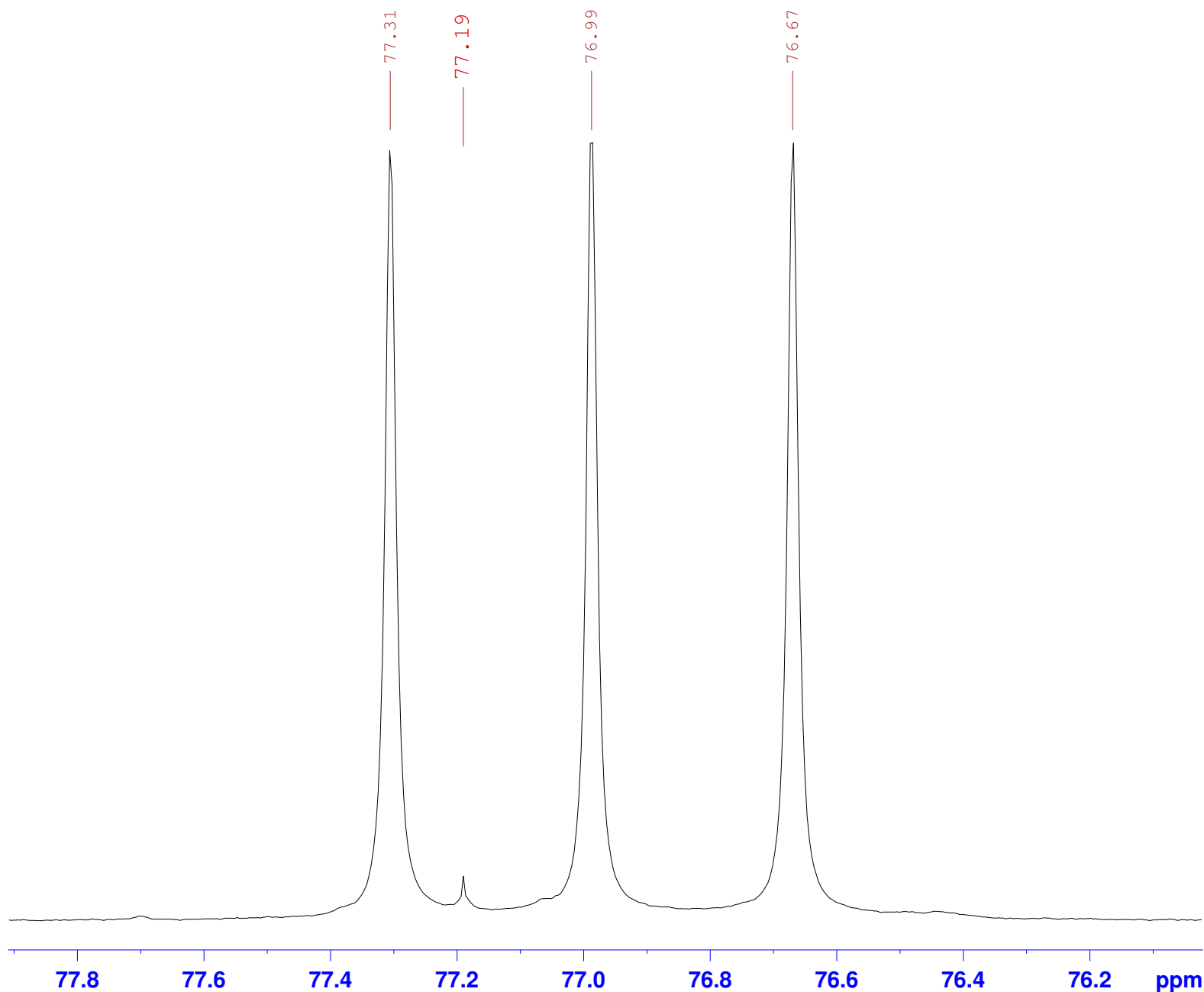
Current Data Parameters
NAME Solvents
EXPNO 106
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100702
Time 20.07
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 73728
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 32800
DW 20.800 usec
DE 6.00 usec
TE 293.5 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 9.50 usec
PL1 -2.00 dB
SFO1 100.6233333 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL12 13.76 dB
PL13 14.00 dB
PL2 0.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 65536
SF 100.6127789 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



Current Data Parameters
NAME Solvents
EXPNO 106
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100702
Time 20.07
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 73728
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 32800
DW 20.800 usec
DE 6.00 usec
TE 293.5 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 9.50 usec
PL1 -2.00 dB
SFO1 100.6233333 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL12 13.76 dB
PL13 14.00 dB
PL2 0.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 65536
SF 100.6127789 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40