



Supporting Information

for

Extension of the 5-alkynyluridine side chain via C–C-bond formation in modified organometallic nucleosides using the Nicholas reaction

Renata Kaczmarek, Dariusz Korczyński, James R. Green and Roman Dembinski

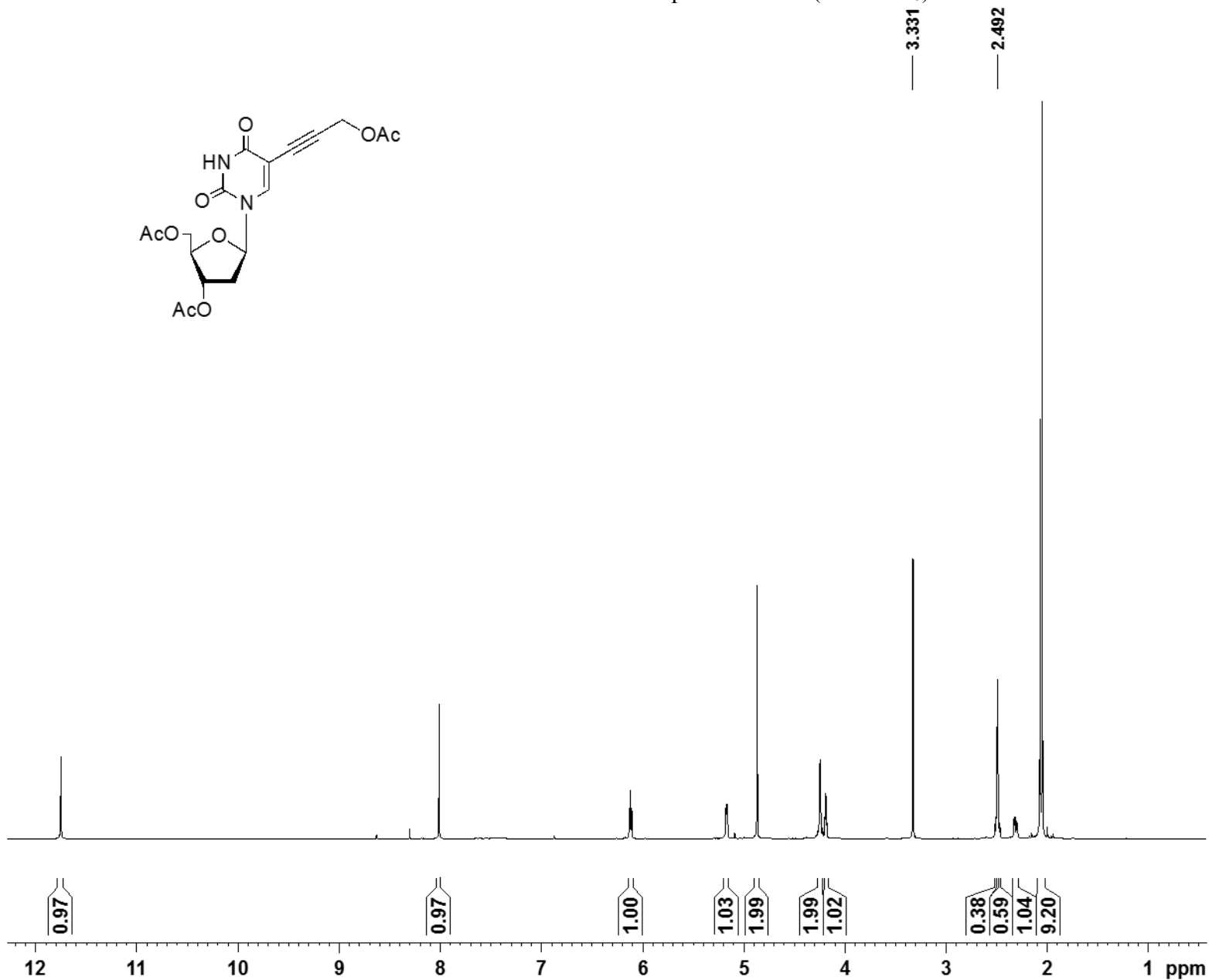
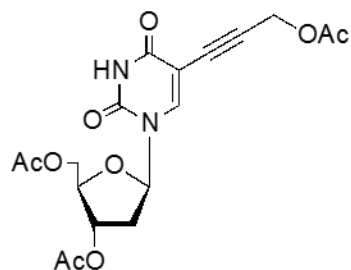
Beilstein J. Org. Chem. **2020**, *16*, 1–8. [doi:10.3762/bjoc.16.1](https://doi.org/10.3762/bjoc.16.1)

^1H and ^{13}C NMR spectra for compounds **2, **3**, **4**, **5**, **6a–c**, and **7a,b,d****

Table of Contents

^1H and ^{13}C NMR spectrum of 2	S2
^1H and ^{13}C NMR spectrum of 3	S4
^1H and ^{13}C NMR spectrum of 4	S6
^1H and ^{13}C NMR spectrum of 5	S8
^1H and ^{13}C NMR spectrum of 6a	S10
^1H and ^{13}C NMR spectrum of 6b	S12
^1H and ^{13}C NMR spectrum of 6c	S14
^1H and ^{13}C NMR spectrum of 7a	S16
^1H and ^{13}C NMR spectrum of 7b	S18
^1H and ^{13}C NMR spectrum of 7d	S20

¹H NMR spectrum for 2 (DMSO-d₆)



CBMIM PAN LODZ

**** NMR LAB ****

AV III 500 MHz

Tel.042 6803 307

Current Data Parameters
DATPATH C:\Bruker\TopSpin3.1\109
NAME kac12310_2017_600
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171023
Time 11.31
INSTRUM AV III 600
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 64
DS 0
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6563926 sec
RG 57
DW 40.533 usec
DE 6.50 usec
TE 294.9 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 600.1339008 MHz
NUC1 1H
P1 11.00 usec
PLW1 25.00000000 W

F2 - Processing parameters
SI 65536
SF 600.1300135 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

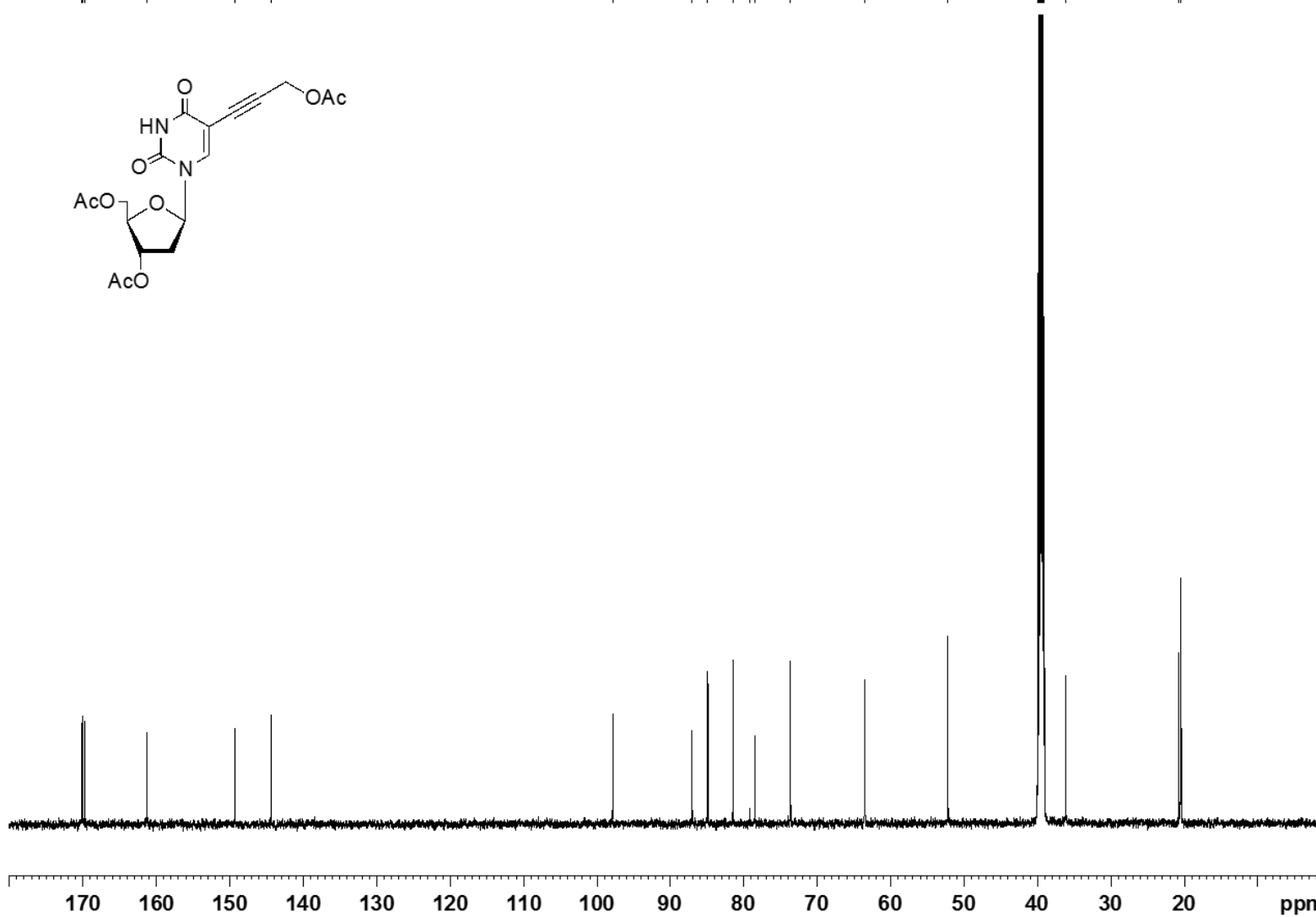
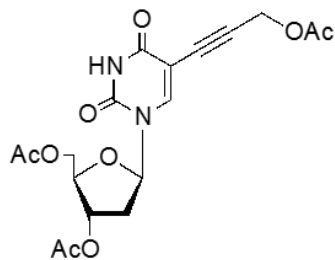
¹³C NMR spectrum for 2 (DMSO-d₆)



170.103
170.032
169.710
161.295
149.335
144.405

97.847
87.048
84.903
81.483
79.171
78.516
73.660

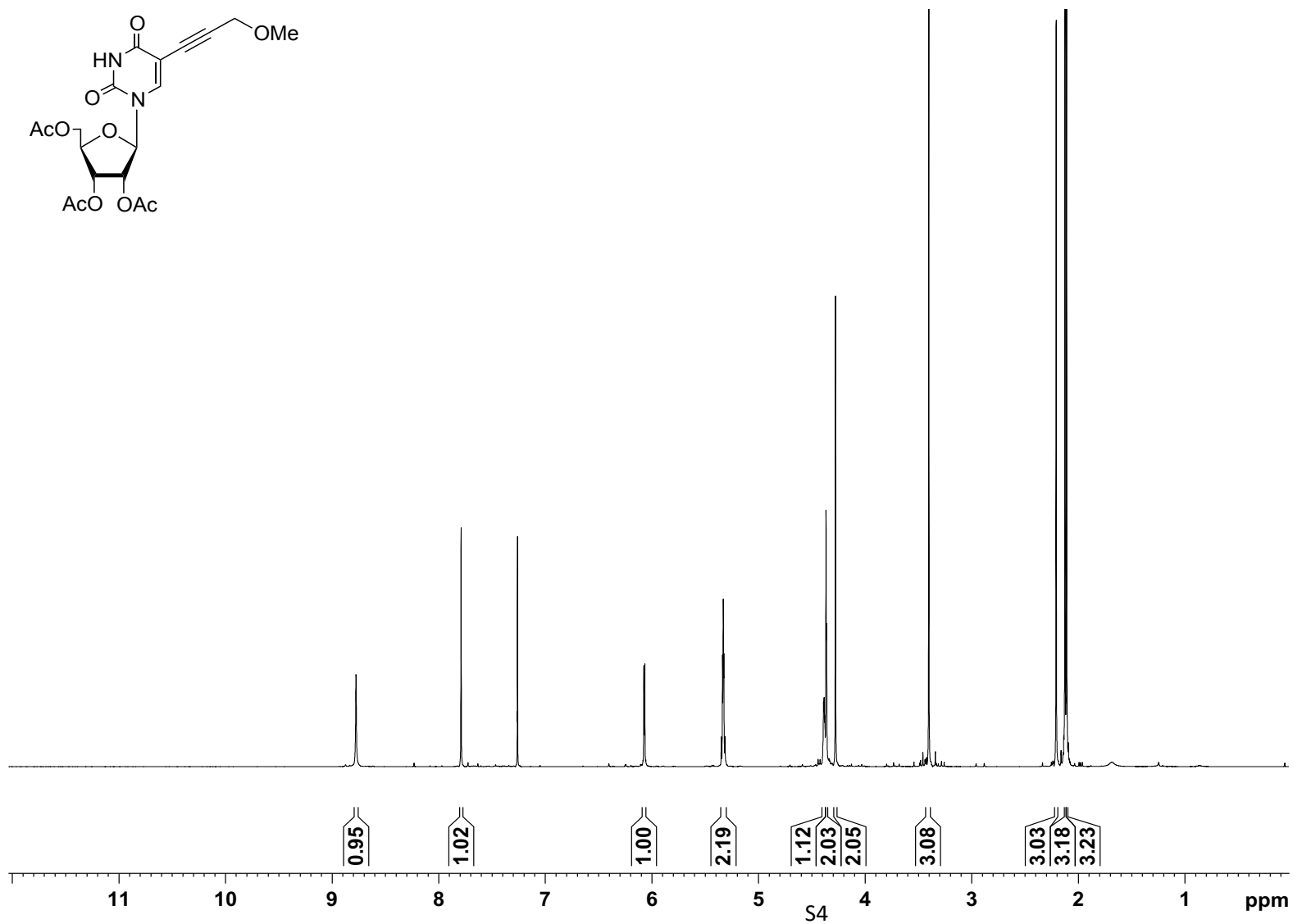
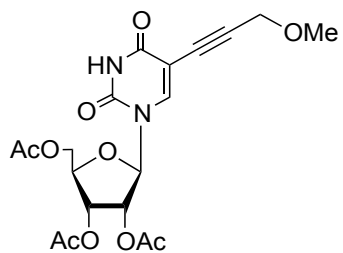
63.529
52.196
40.029
39.910
39.771
39.633
39.494
39.355
39.215
39.076
36.169
20.755
20.542
20.459



CBMIM PAN LODZ
 **** NMR LAB ****
 AV III 500 MHz
 Tel:042 6803 307
 Current Data Parameters
 DATPATH C:\Bruker\TopSpin3.1\109
 NAME kac12310_2017_600
 EXPNO 2
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20171023
 Time 12.04
 INSTRUM AV III 600
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 755
 DS 0
 SWH 42613.637 Hz
 FIDRES 0.650232 Hz
 AQ 0.7689557 sec
 RG 2050
 DW 11.733 usec
 DE 6.50 usec
 TE 295.0 K
 D1 3.00000000 sec
 D11 0.03000000 sec
 TDO 4
 ===== CHANNEL f1 =====
 SFO1 150.9194083 MHz
 NUC1 13C
 P1 12.00 usec
 PLW1 98.00000000 W
 ===== CHANNEL f2 =====
 SFO2 600.1324005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 80.00 usec
 PLW2 25.00000000 W
 PLW12 0.47266001 W
 PLW13 0.30250001 W
 F2 - Processing parameters
 SI 32768
 SF 150.9028801 MHz
 WDW EM
 SSB 0
 LB 2.00 Hz
 GB 0
 PC 1.40

¹H NMR spectrum for 3 (CDCl₃)

7.263



CBMiM PAN LODZ

**** NMR LAB ****

AV III 500 MHz

Tel:042 6803 307

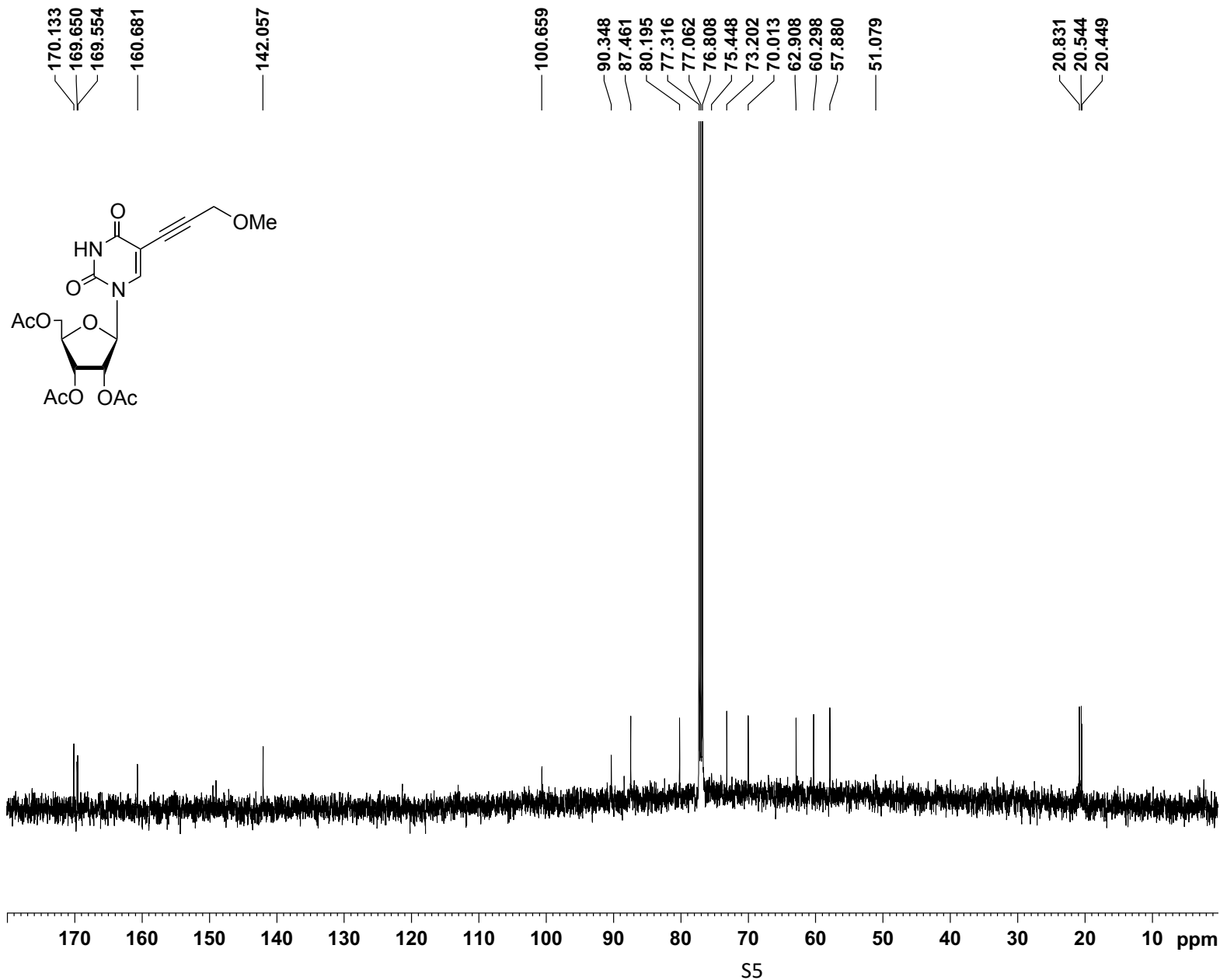
Current Data Parameters
DATPATH C:\Bruker\TopSpin3.1\109
NAME kac10907_2019_500
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190709
Time 10.29
INSTRUM AV_III_500
PROBHD 5 mm TXI 31P Z
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 24
DS 0
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6563926 sec
RG 228
DW 40.533 usec
DE 6.50 usec
TE 295.0 K
D1 1.0000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 500.1330008 MHz
NUC1 1H
P1 9.66 usec
PLW1 9.19999981 W

F2 - Processing parameters
SI 65536
SF 500.1300236 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR spectrum for **3** (CDCl₃)



CBMIM PAN LODZ

**** NMR LAB ****

AV III 500 MHz

Tel:042 6803 307

Current Data Parameters
 DATPATH C:\Bruker\TopSpin3.1\109
 NAME kac10907_2019_500
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20190709
 Time 10.55
 INSTRUM AV III 500
 PROBHD 5 mm TXI 31P Z
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 512
 DS 0
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 2050
 DW 13.867 usec
 DE 6.50 usec
 TE 295.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 125.7728788 MHz
 NUC1 13C
 P1 11.50 usec
 PLW1 226.0000000 W

===== CHANNEL f2 =====
 SFO2 500.1324005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 100.00 usec
 PLW2 9.00000000 W
 PLW12 0.08398400 W
 PLW13 0.08398400 W

F2 - Processing parameters
 SI 32768
 SF 125.7577890 MHz
 WDW EM
 SSB 0
 LB 2.00 Hz
 GB 0
 PC 1.40

¹³C NMR spectrum for 4 (CDCl₃)



CBMiM PAN LODZ

**** NMR LAB ****

AV III 500 MHz

Tel:042 6803 307

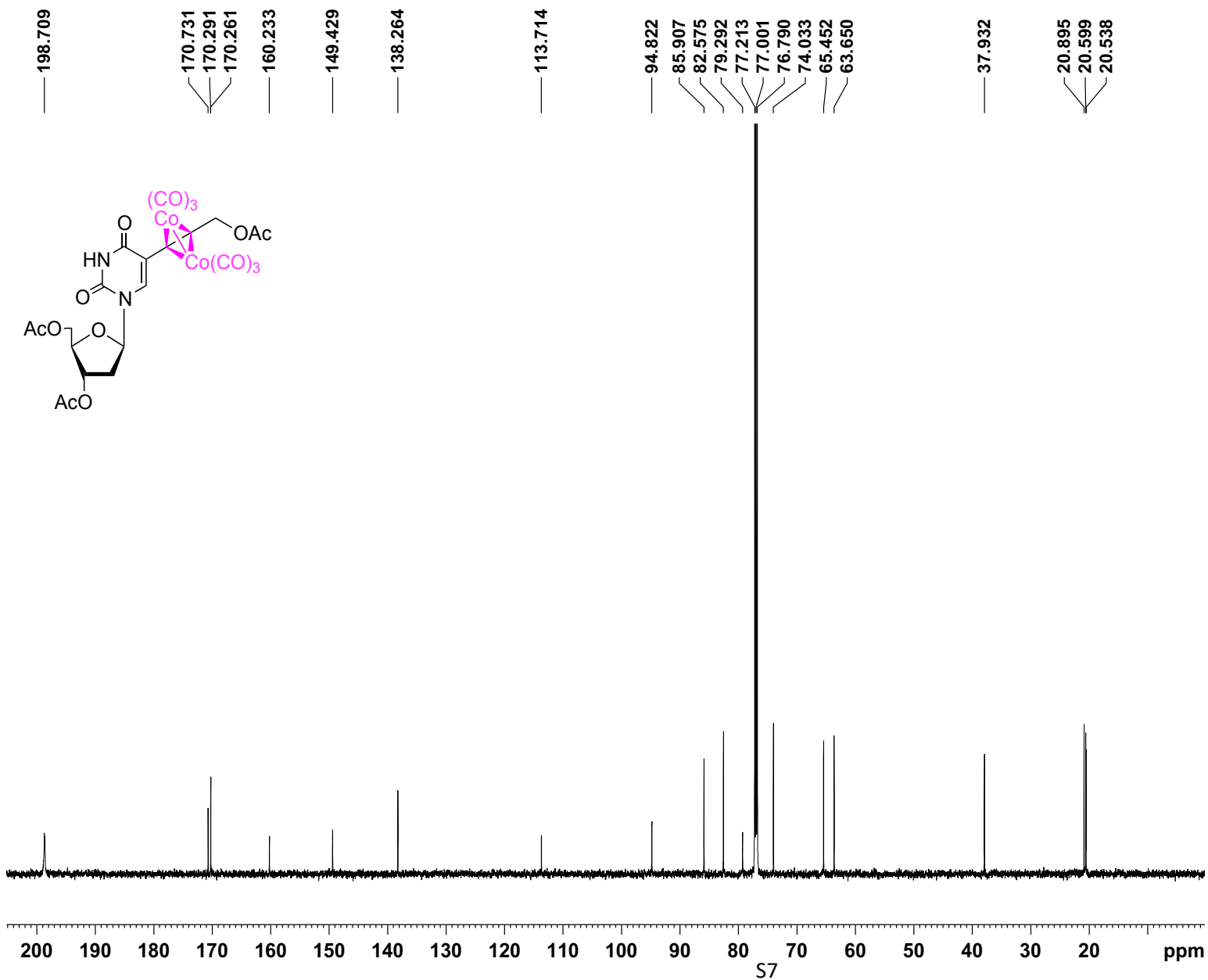
Current Data Parameters
DATPATH C:\Bruker\TopSpin3.1\109
NAME kac12510_2017_600
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171025
Time 11.58
INSTRUM AV III 600
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 633
DS 0
SWH 42613.637 Hz
FIDRES 0.650232 Hz
AQ 0.7689557 sec
RG 2050
DW 11.733 usec
DE 6.50 usec
TE 295.1 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 2

===== CHANNEL f1 =====
SFO1 150.9194083 MHz
NUC1 13C
P1 12.00 usec
PLW1 98.00000000 W

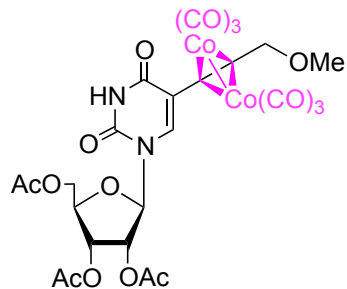
===== CHANNEL f2 =====
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 80.00 usec
PLW2 25.00000000 W
PLW12 0.47266001 W
PLW13 0.30250001 W

F2 - Processing parameters
SI 32768
SF 150.9028155 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40



^1H NMR spectrum for **5** (CDCl_3)

— 7.266



CBMim PAN LODZ

**** NMR LAB ****

AV III 500 MHz

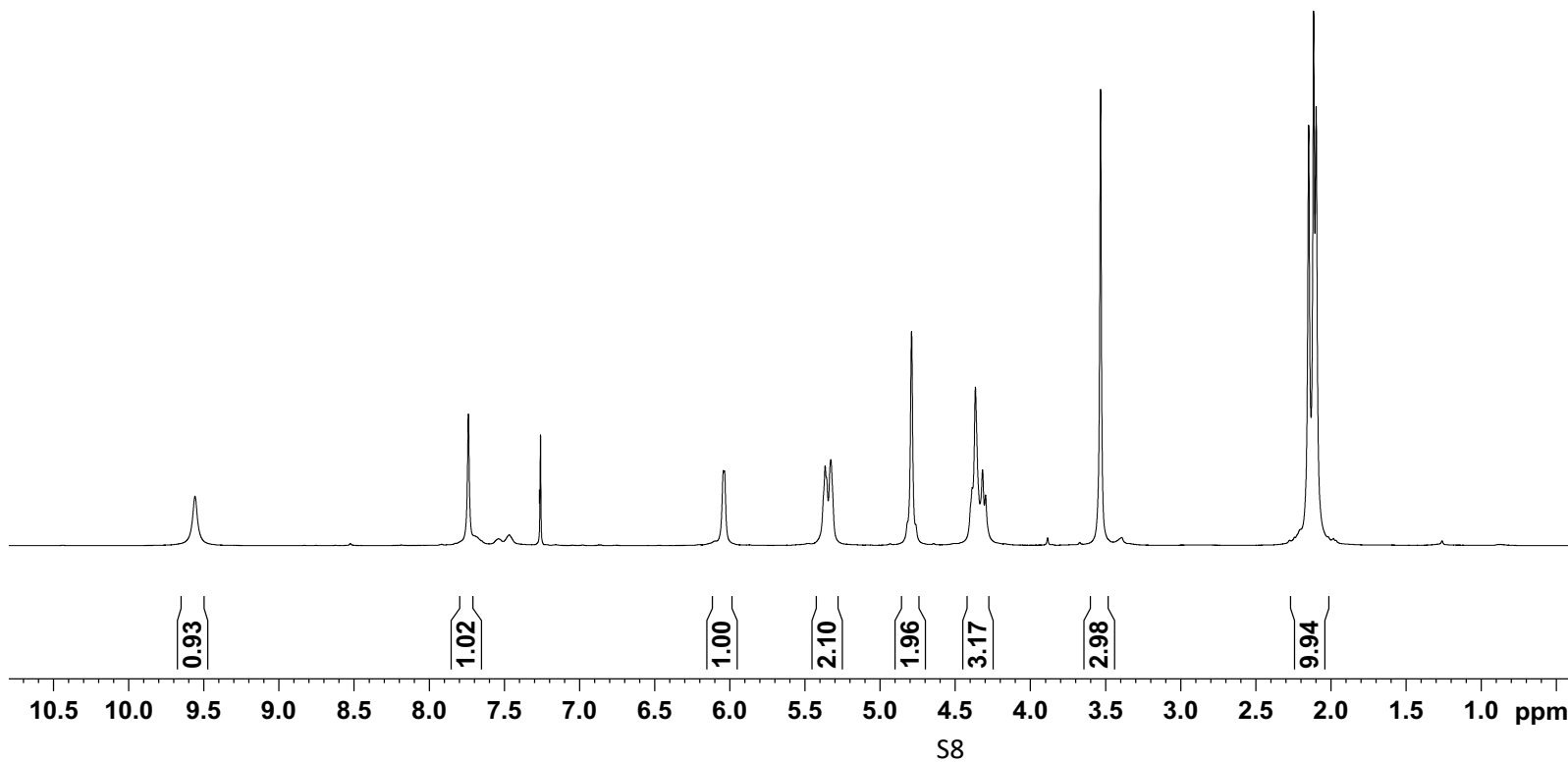
Tel:042 6803 307

Current Data Parameters
DATPATH C:\Bruker\TopSpin3.1\109
NAME kac10907_2019_500
EXPNO 3
PROCNO 1

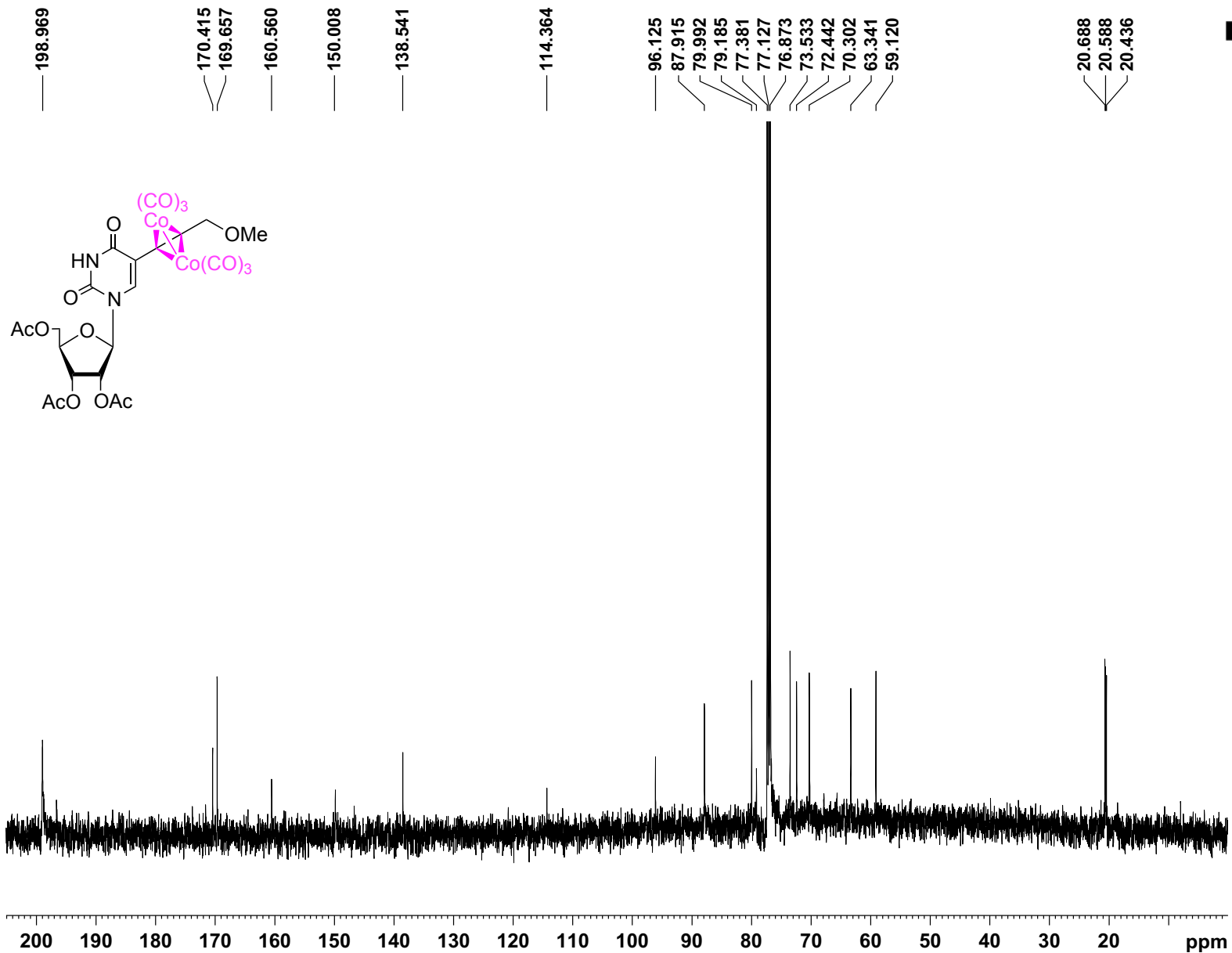
F2 - Acquisition Parameters
Date_ 20190709
Time 10.58
INSTRUM AV III 500
PROBHD 5 mm TXI 31P Z
PULPROG zg30
TD 65536
SOLVENT CDCl_3
NS 24
DS 0
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6563926 sec
RG 114
DW 40.533 usec
DE 6.50 usec
TE 295.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 500.1330008 MHz
NUC1 ^1H
P1 9.66 usec
PLW1 9.19999981 W

F2 - Processing parameters
SI 65536
SF 500.1300236 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



¹³C NMR spectrum for **5** (CDCl₃)



CBMIM PAN LODZ

**** NMR LAB ****

AV III 500 MHz

Tel:042 6803 307

Current Data Parameters
 DATPATH C:\Bruker\TopSpin3.1\109
 NAME kac10907_2019_500
 EXPNO 4
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20190709
 Time 11.07
 INSTRUM AV III 500
 PROBHD 5 mm TXI 31P Z
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 512
 DS 0
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 2050
 DW 13.867 usec
 DE 6.50 usec
 TE 295.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====

SFO1 125.7728788 MHz
 NUC1 13C
 P1 11.50 usec
 PLW1 226.0000000 W

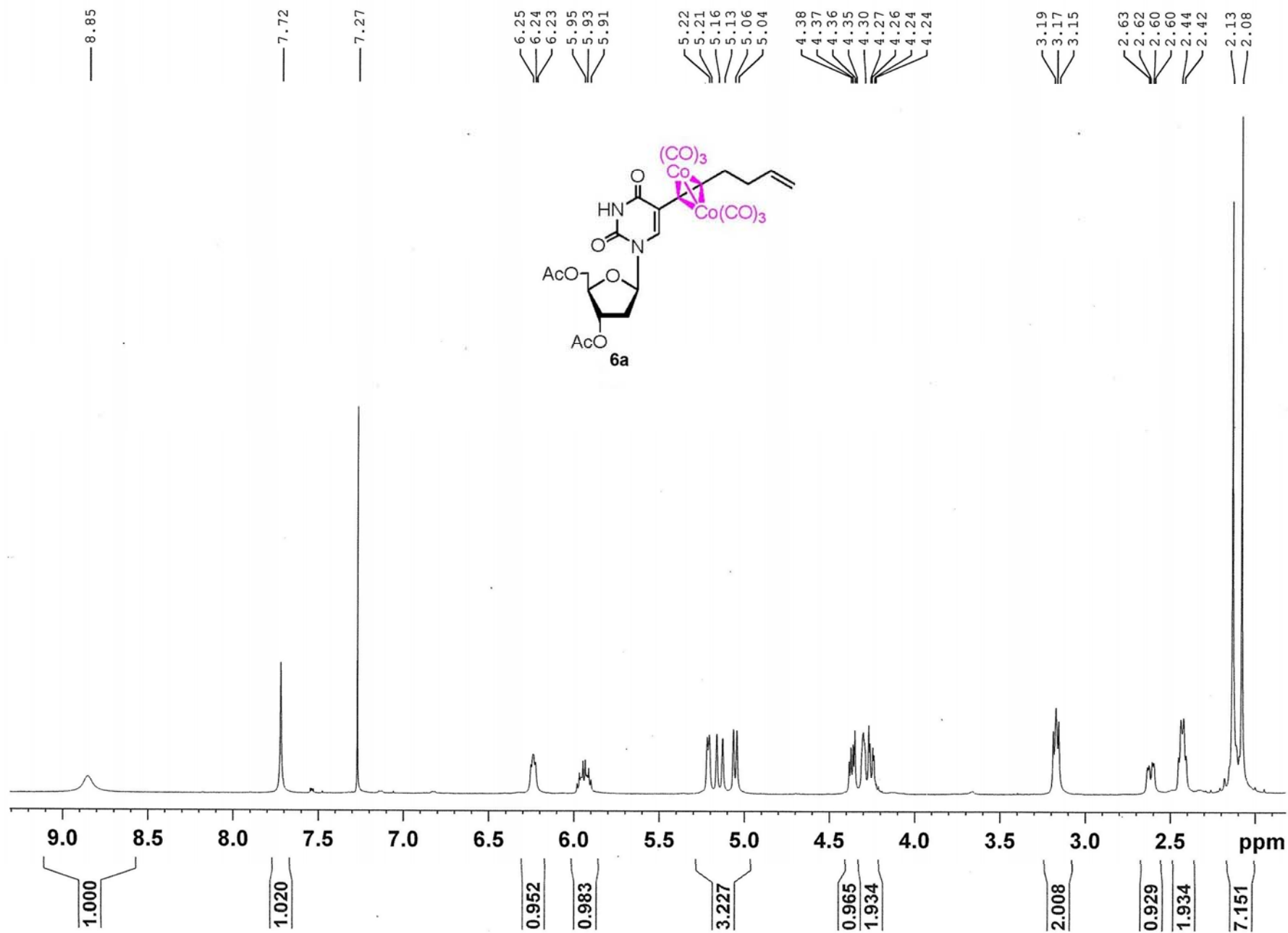
===== CHANNEL f2 =====

SFO2 500.1324005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 100.00 usec
 PLW2 9.00000000 W
 PLW12 0.08398400 W
 PLW13 0.08398400 W

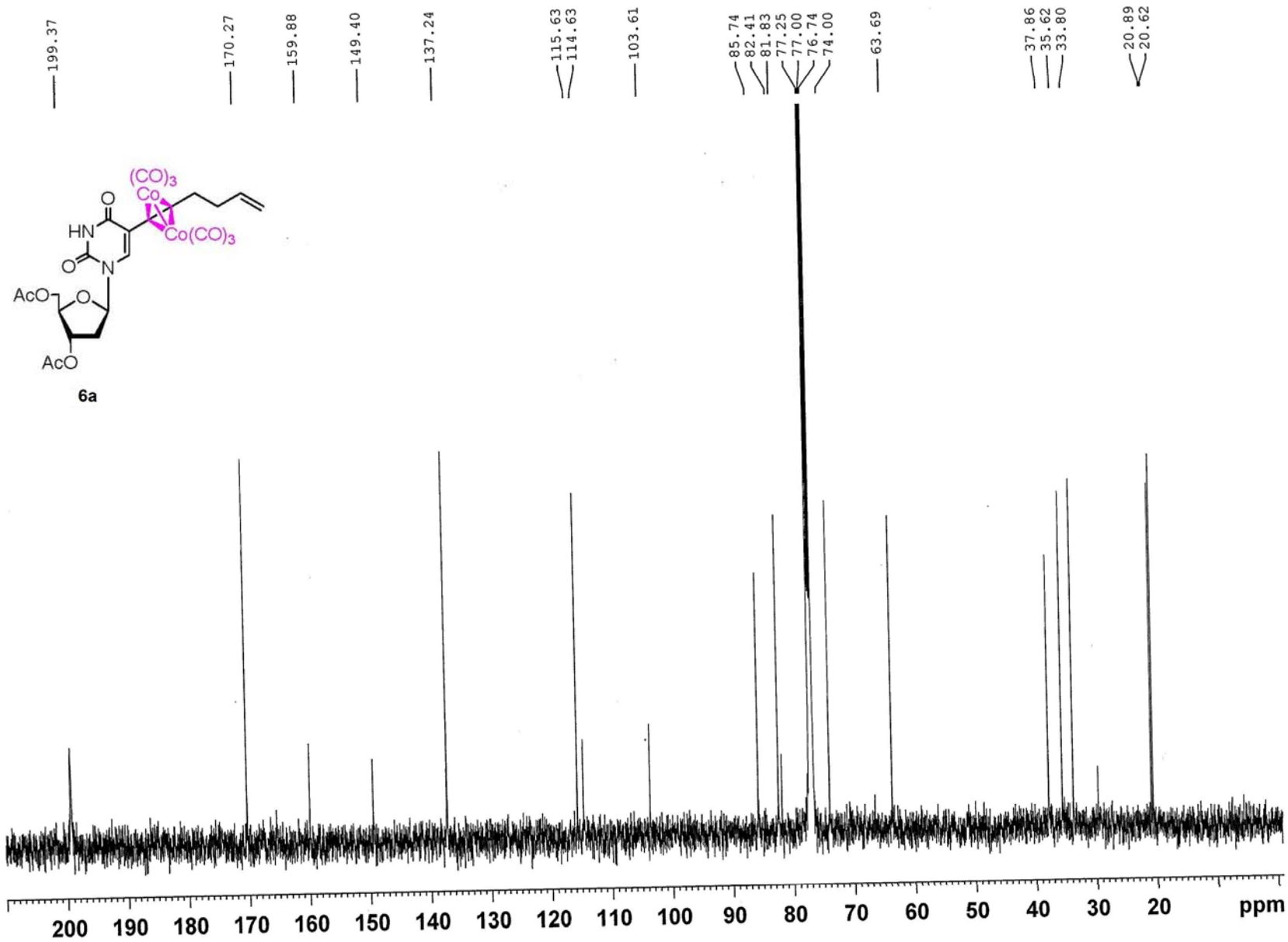
F2 - Processing parameters

SI 32768
 SF 125.7577890 MHz
 WDW EM
 SSB 0
 LB 2.00 Hz
 GB 0
 PC 1.40

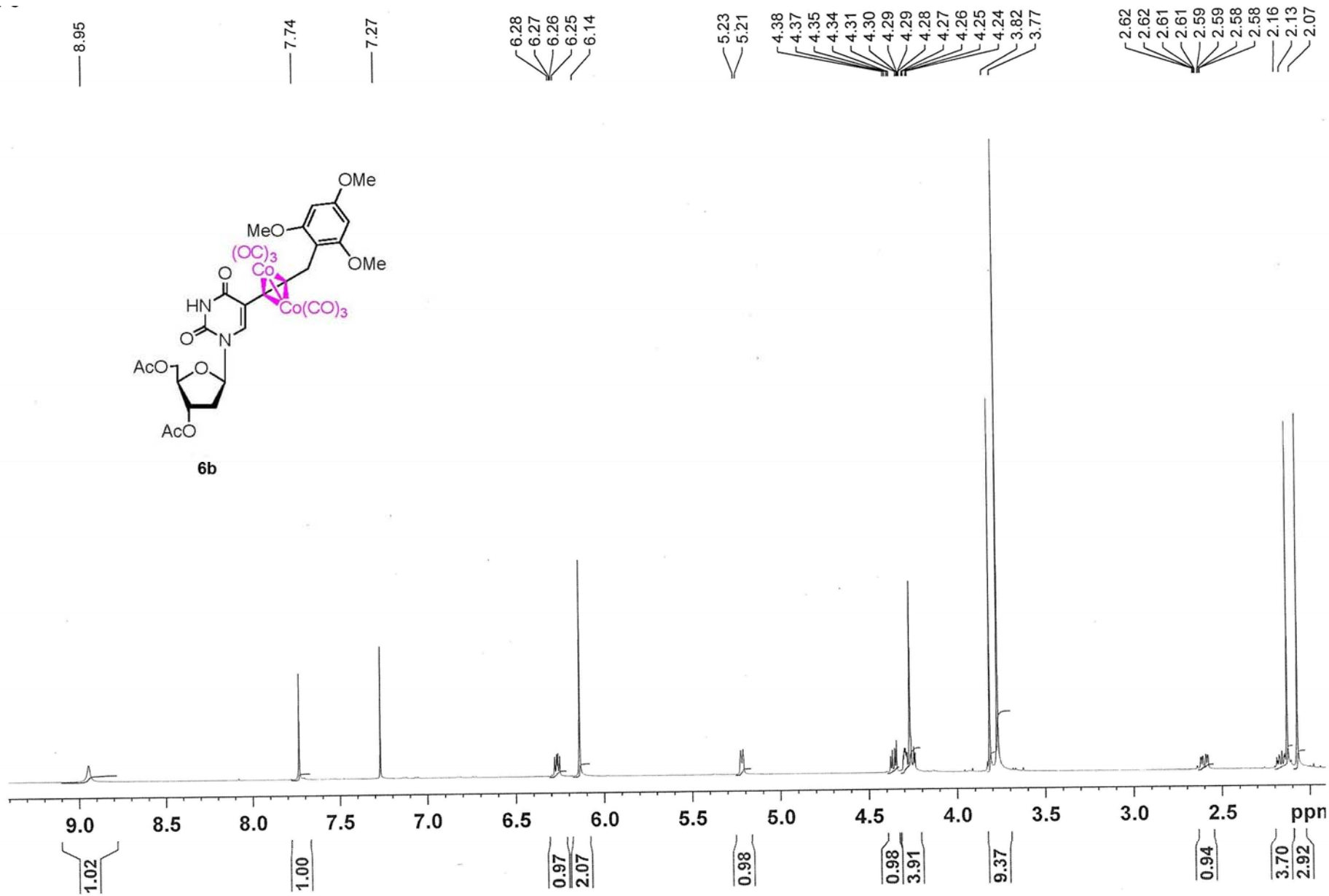
¹H NMR spectrum for **6a** (CDCl₃)



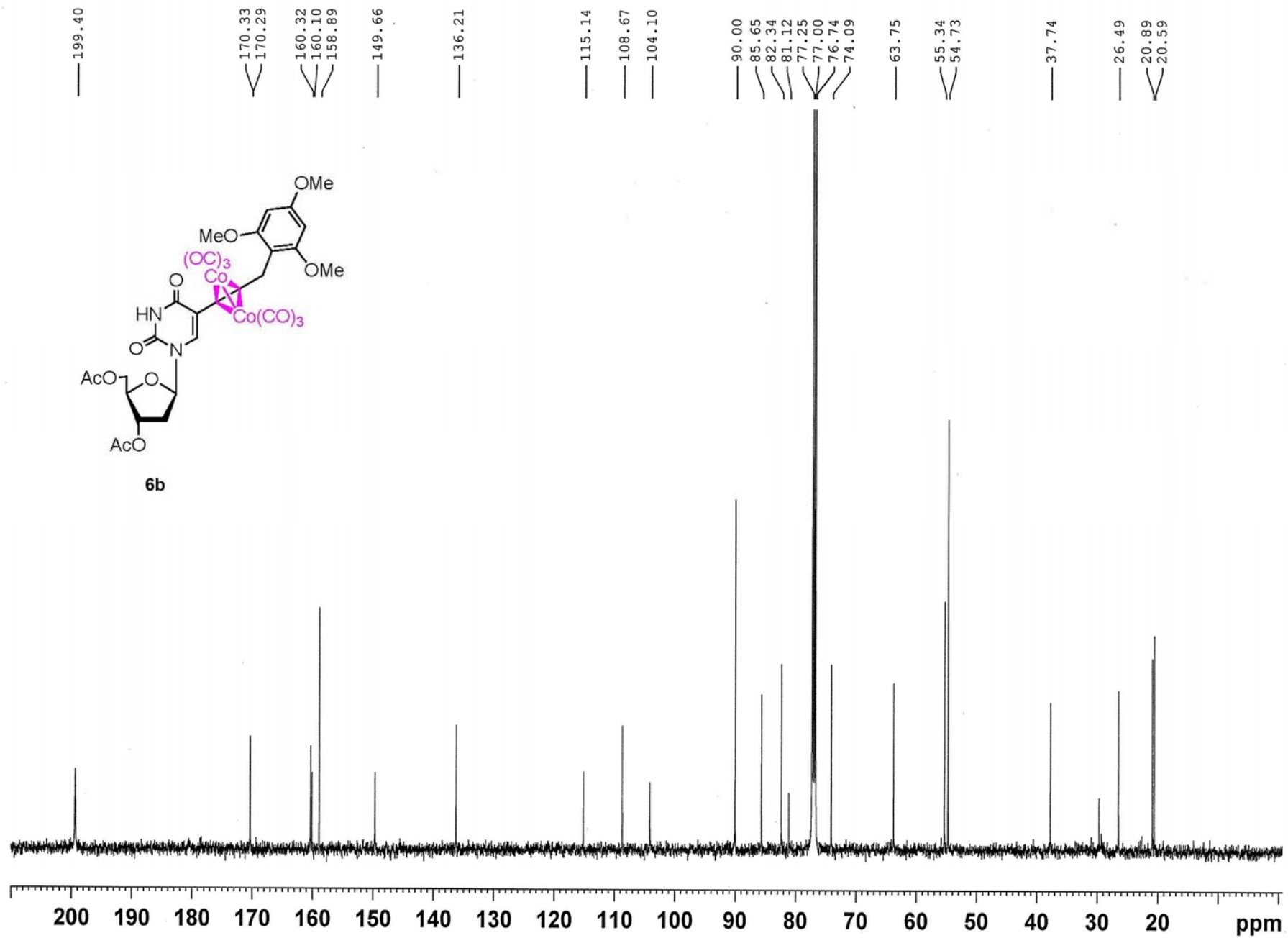
^{13}C NMR spectrum for **6a** (CDCl_3)



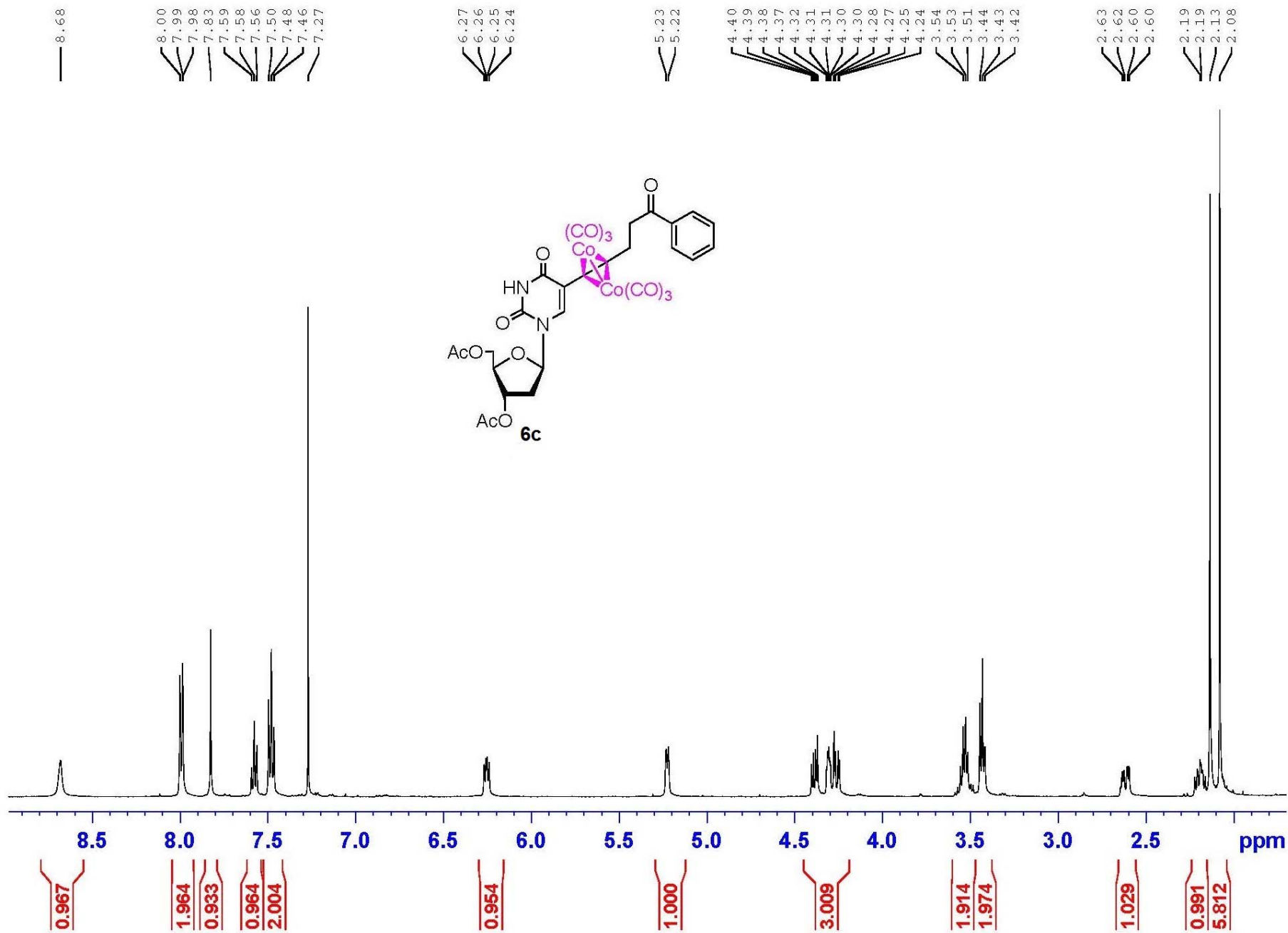
¹H NMR spectrum for **6b** (CDCl₃)



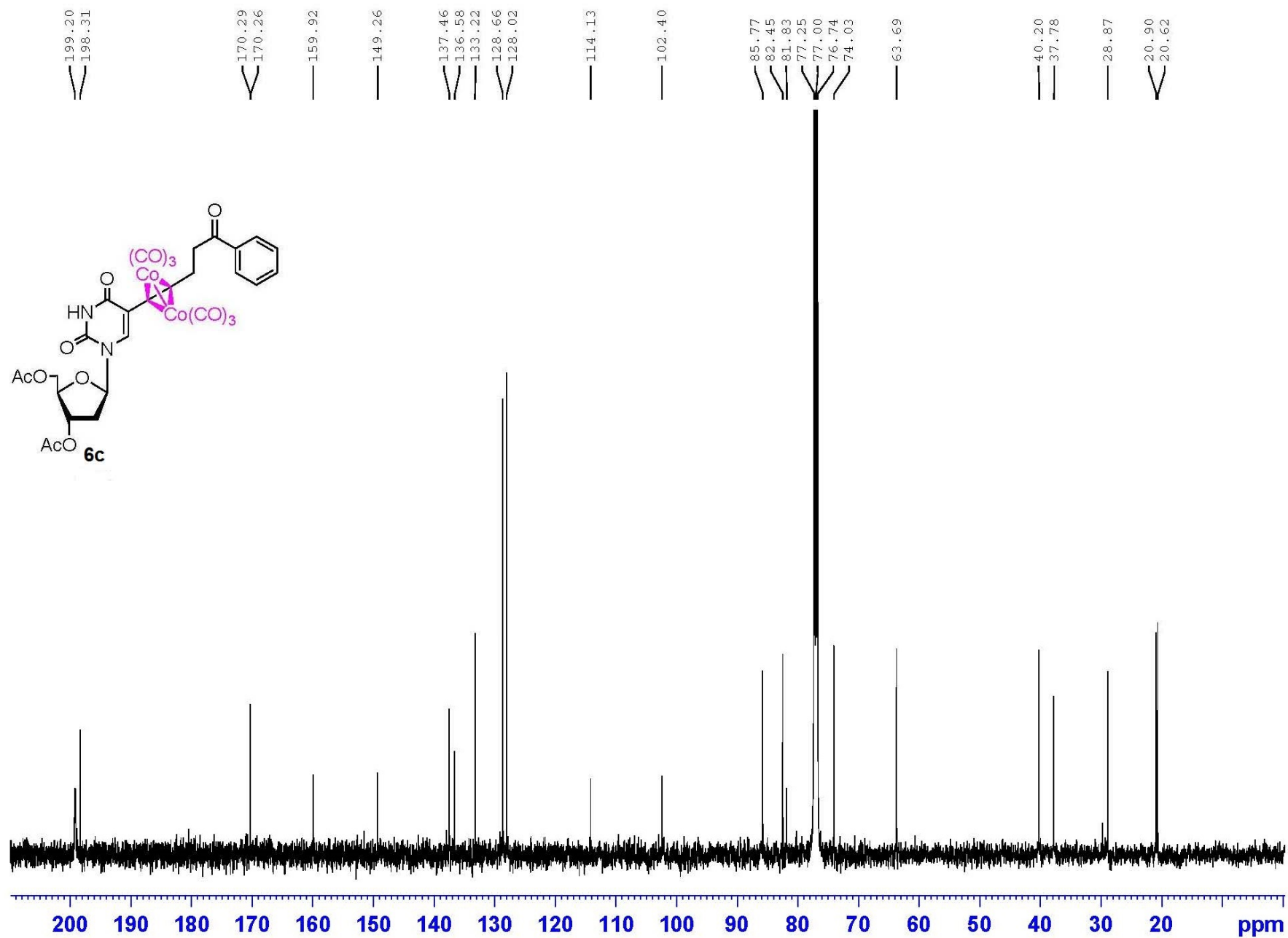
^{13}C NMR spectrum for **6b** (CDCl_3)



¹H NMR spectrum for **6c** (CDCl₃)



^{13}C NMR spectrum for **6c** (CDCl_3)



¹H NMR spectrum for **7a** (CDCl₃)

8.602

7.545

7.270

6.009

5.991

5.950

5.927

5.893

5.367

5.348

5.339

5.328

5.173

5.116

5.074

5.040

4.381

3.191

3.166

3.138

2.465

2.441

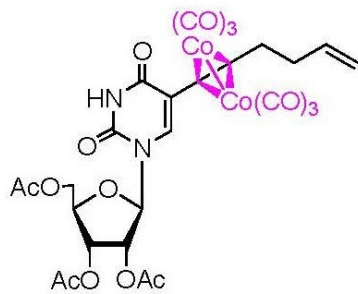
2.415

2.392

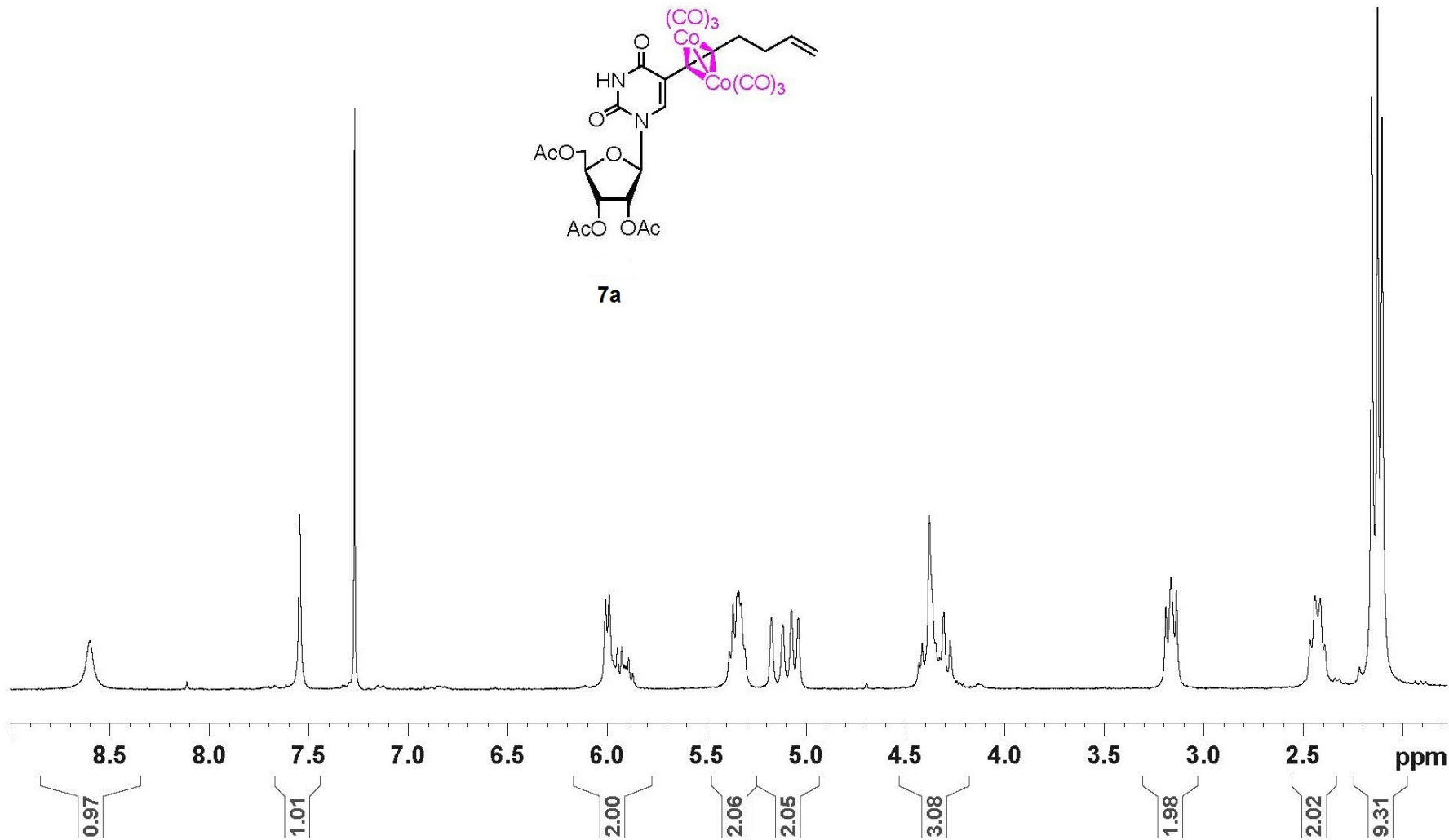
2.155

2.126

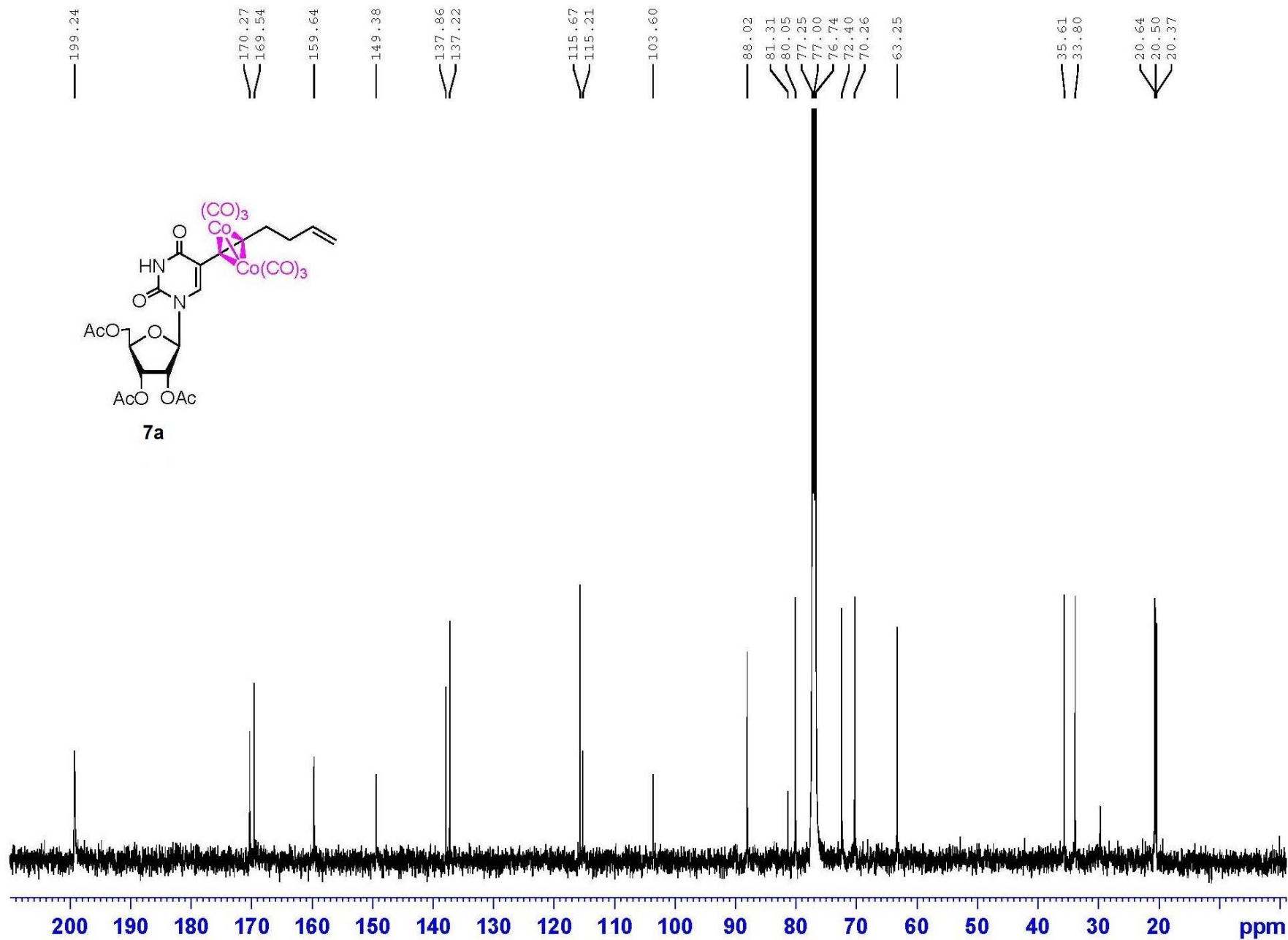
2.103



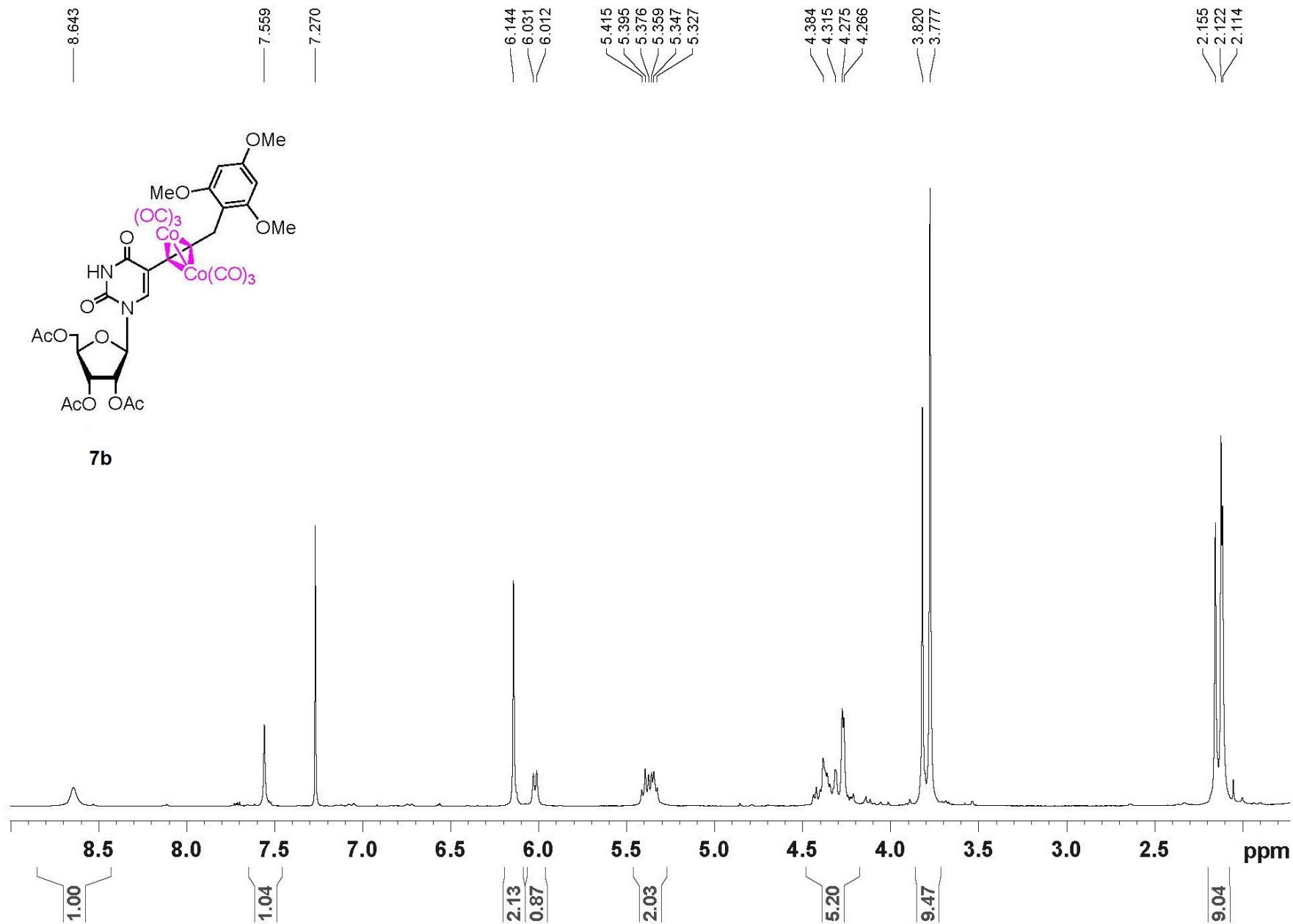
7a



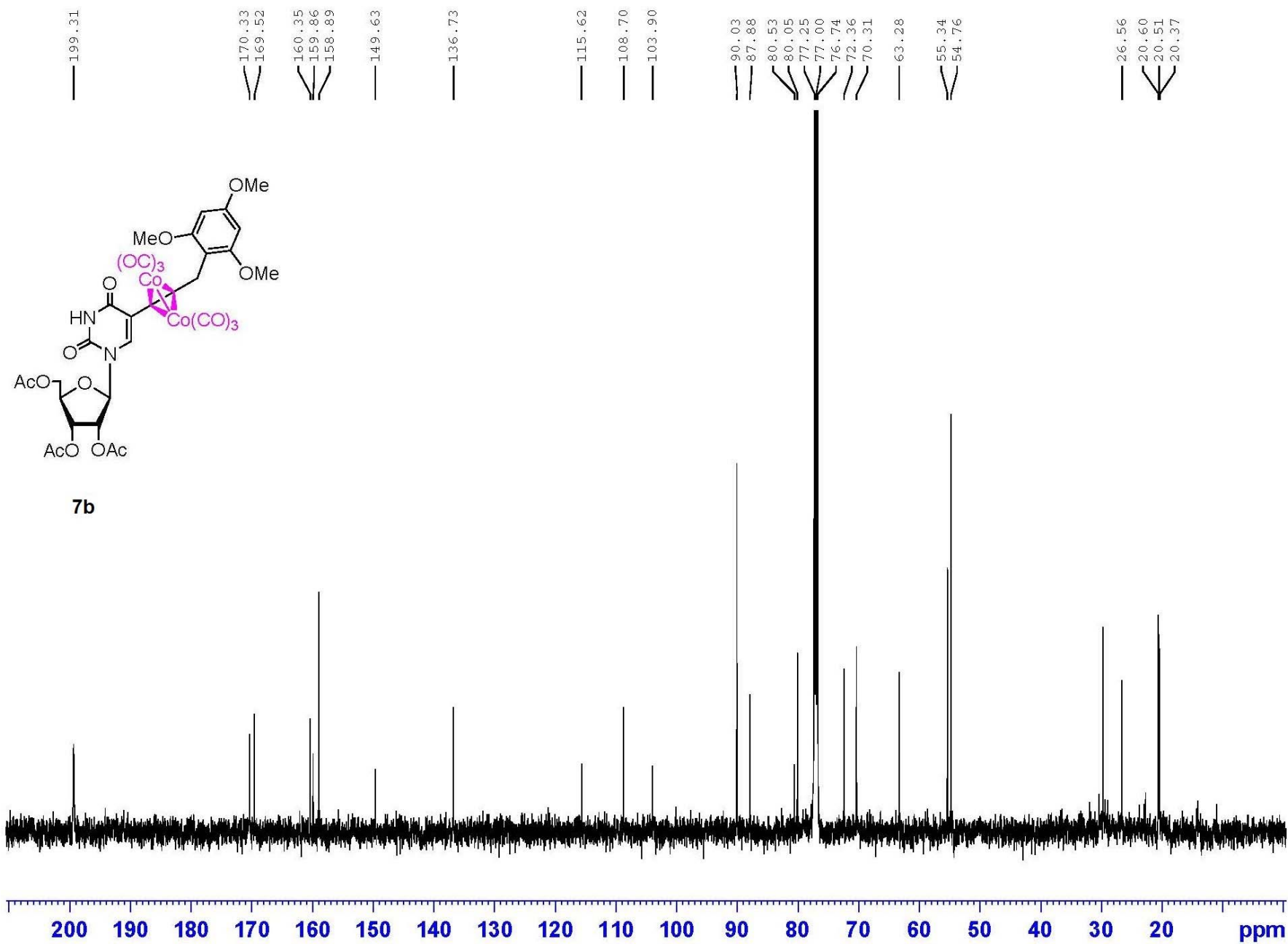
^{13}C NMR spectrum for **7a** (CDCl_3)



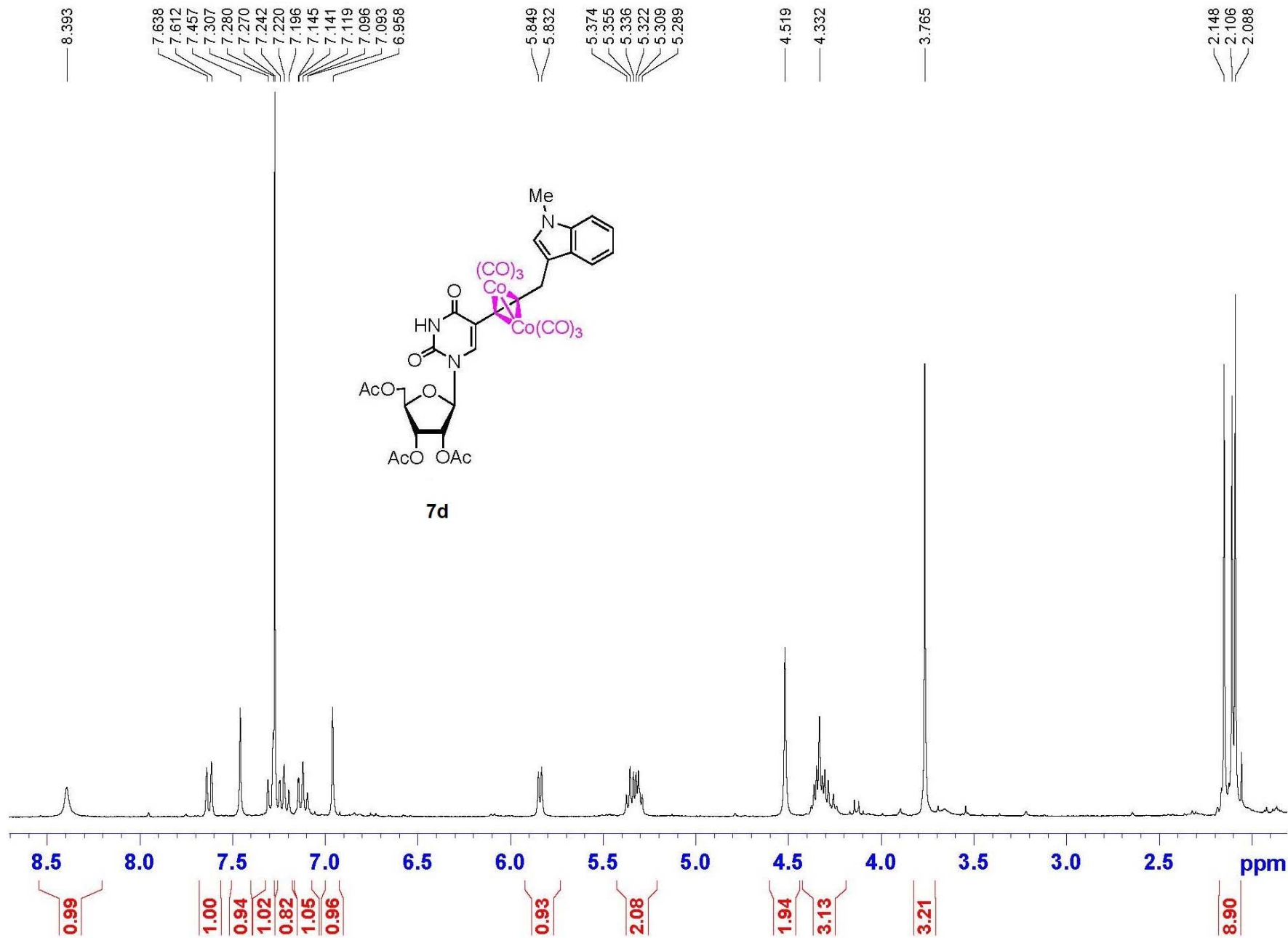
¹H NMR spectrum for **7b** (CDCl₃)



^{13}C NMR spectrum for **7b** (CDCl_3)



¹H NMR spectrum for **7d** (CDCl₃)



^{13}C NMR spectrum for **7d** (CDCl_3)

