

Building the housane: diastereoselective synthesis and characterization of bicyclo[2.1.0]pentane carboxylic acids

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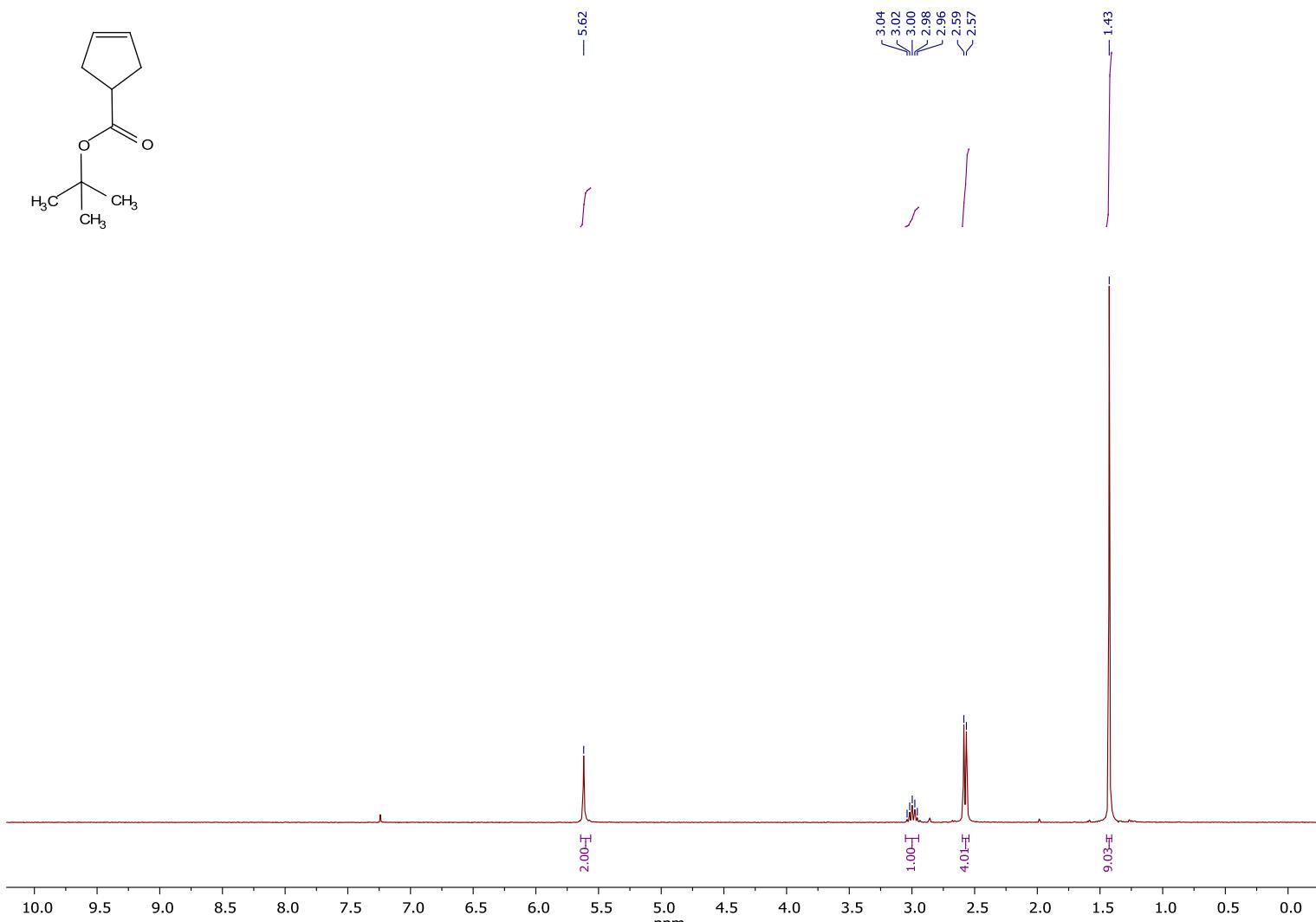
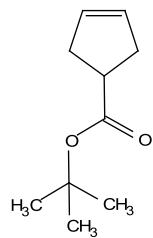
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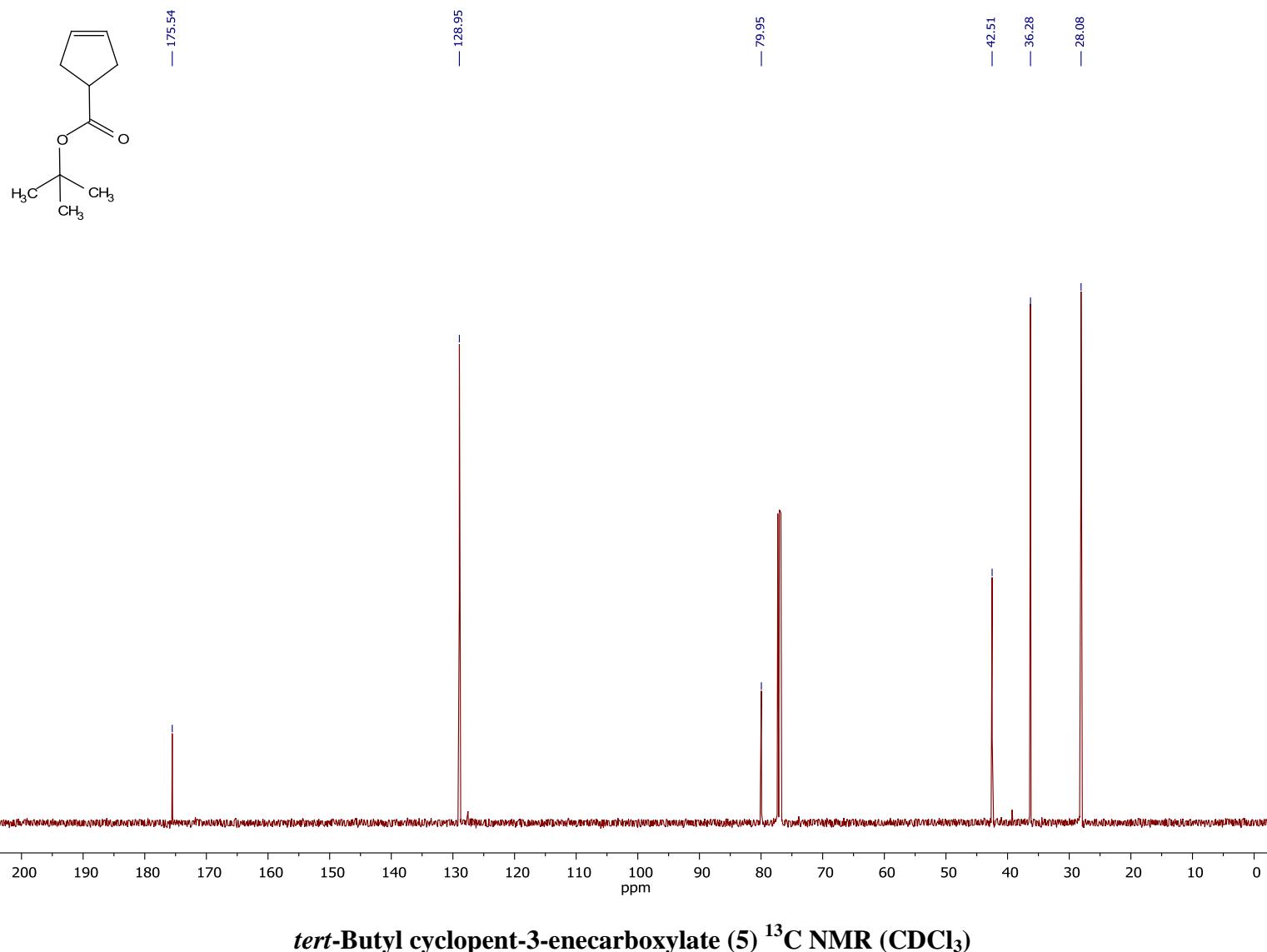
(1 <i>R</i> ^{*,3<i>R</i>^{*,4<i>R</i>[*]})-3-Methylbicyclo[2.1.0]pentane-1-carboxylic acid (17a) ¹H NMR.....}	S108
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(1 <i>R</i> ^{*,3<i>R</i>^{*,4<i>S</i>[*]})-3-Azidobicyclo[2.1.0]pentane-1-carboxylic acid (17f) ¹³C NMR}	S121
(1 <i>R</i> ^{*,3<i>R</i>^{*,4<i>S</i>[*]})-3-Hydroxybicyclo[2.1.0]pentane-1-carboxylic acid (17g) ¹H NMR}	S122
(1 <i>R</i> ^{*,3<i>R</i>^{*,4<i>S</i>[*]})-3-Hydroxybicyclo[2.1.0]pentane-1-carboxylic acid (17g) ¹³C NMR}	S123
(1 <i>R</i> ^{*,3<i>R</i>^{*,4<i>S</i>[*]})-<i>tert</i>-Butyl 3-((4-nitrobenzoyl)oxy)bicyclo[2.1.0]pentane-1-carboxylate (18g) ¹H NMR}	S124
(1 <i>R</i> ^{*,3<i>R</i>^{*,4<i>S</i>[*]})-<i>tert</i>-Butyl 3-((4-nitrobenzoyl)oxy)bicyclo[2.1.0]pentane-1-carboxylate (18g) ¹³C NMR}	S125
(1 <i>R</i> ^{*,3<i>S</i>^{*,4<i>S</i>[*]})-<i>tert</i>-Butyl 3-aminobicyclo[2.1.0]pentane-1-carboxylate (19) ¹H NMR}	S126
(1 <i>R</i> ^{*,3<i>S</i>^{*,4<i>S</i>[*]})-<i>tert</i>-Butyl 3-aminobicyclo[2.1.0]pentane-1-carboxylate (19) ¹³C NMR}	S127
(1 <i>R</i> ^{*,3<i>R</i>^{*,4<i>S</i>[*]})-<i>tert</i>-Butyl 3-aminobicyclo[2.1.0]pentane-1-carboxylate (20) ¹H NMR}	S128
(1 <i>R</i> ^{*,3<i>R</i>^{*,4<i>S</i>[*]})-<i>tert</i>-Butyl 3-aminobicyclo[2.1.0]pentane-1-carboxylate (20) ¹H NMR}	S129

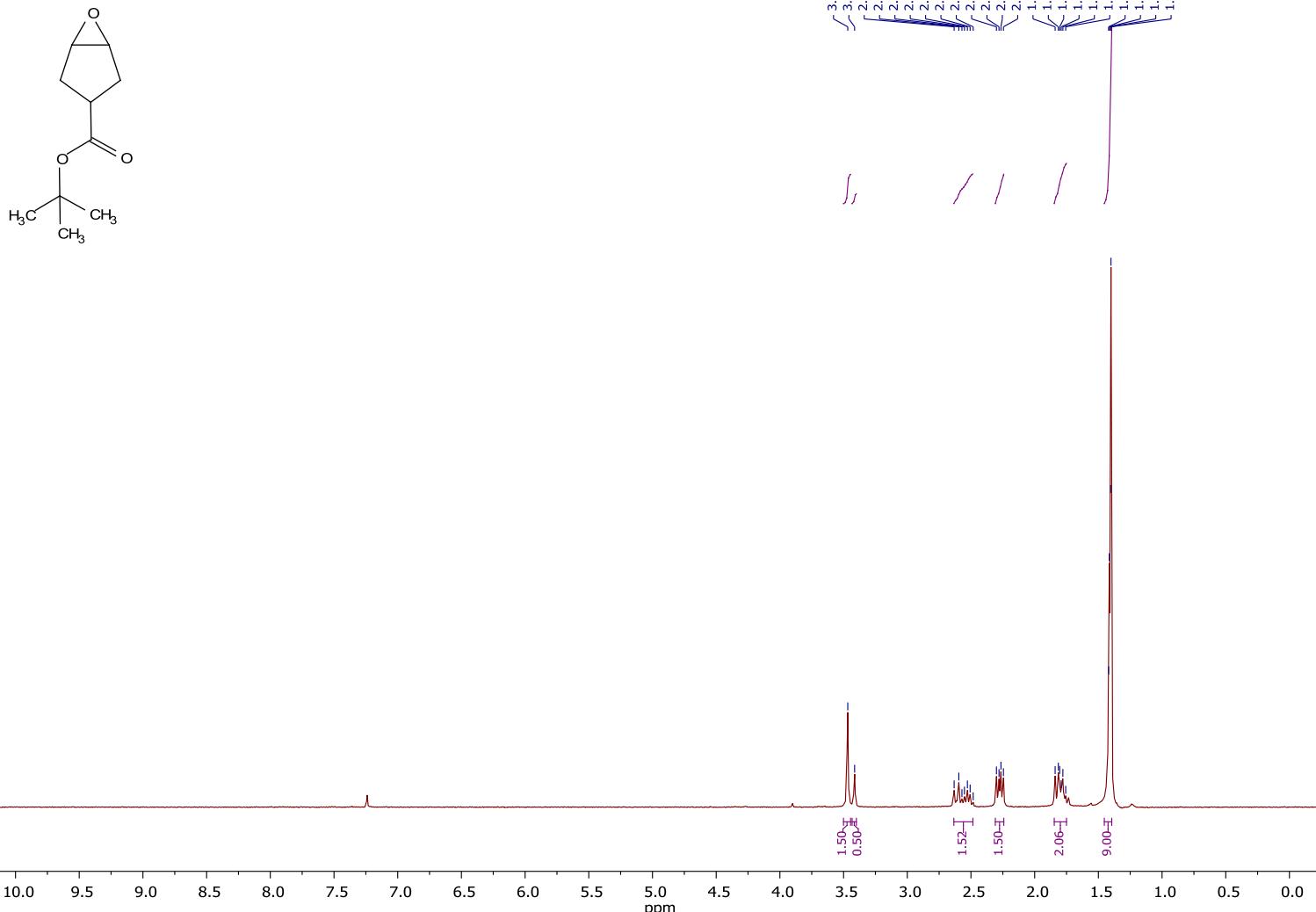
(1 <i>S</i> ^{*,2<i>S</i>^{*,4<i>R</i>[*]})-4-Carboxybicyclo[2.1.0]pentan-2-aminium 2,2,2-trifluoroacetate (21) ¹H NMR}	S130
(1 <i>S</i> ^{*,2<i>S</i>^{*,4<i>R</i>[*]})-4-Carboxybicyclo[2.1.0]pentan-2-aminium 2,2,2-trifluoroacetate (21) ¹³C NMR}	S131
(1 <i>S</i> ^{*,2<i>S</i>^{*,4<i>R</i>[*]})-4-Carboxybicyclo[2.1.0]pentan-2-aminium 2,2,2-trifluoroacetate (21) ¹⁹F NMR.....}	S132
(1 <i>S</i> ^{*,2<i>S</i>^{*,4<i>R</i>[*]})-4-Carboxybicyclo[2.1.0]pentan-2-aminium 2,2,2-trifluoroacetate (21) (COSY)}	S133
(1 <i>S</i> ^{*,2<i>S</i>^{*,4<i>R</i>[*]})-4-Carboxybicyclo[2.1.0]pentan-2-aminium 2,2,2-trifluoroacetate (21) (HSQC).....}	S134
(1 <i>S</i> ^{*,2<i>S</i>^{*,4<i>R</i>[*]})-4-Carboxybicyclo[2.1.0]pentan-2-aminium 2,2,2-trifluoroacetate (21) (H-H NOE).....}	S135
(1 <i>S</i> ^{*,2<i>R</i>^{*,4<i>R</i>[*]})-4-Carboxybicyclo[2.1.0]pentan-2-aminium 2,2,2-trifluoroacetate (22) ¹H NMR}	S136
(1 <i>S</i> ^{*,2<i>R</i>^{*,4<i>R</i>[*]})-4-Carboxybicyclo[2.1.0]pentan-2-aminium 2,2,2-trifluoroacetate (22) ¹³C NMR}	S137
(1 <i>S</i> ^{*,2<i>R</i>^{*,4<i>R</i>[*]})-4-Carboxybicyclo[2.1.0]pentan-2-aminium 2,2,2-trifluoroacetate (22) ¹⁹F NMR}	S138
(1 <i>R</i> ^{*,3<i>S</i>^{*,4<i>S</i>[*]})-3-((<i>tert</i>-Butoxycarbonyl)amino)bicyclo[2.1.0]pentane-1-carboxylic acid (23) ¹H NMR}	S139
(1 <i>R</i> ^{*,3<i>S</i>^{*,4<i>S</i>[*]})-3-((<i>tert</i>-Butoxycarbonyl)amino)bicyclo[2.1.0]pentane-1-carboxylic acid (23) ¹³C NMR.....}	S140
(1 <i>R</i> ^{*,3<i>R</i>^{*,4<i>S</i>[*]})-3-((<i>tert</i>-Butoxycarbonyl)amino)bicyclo[2.1.0]pentane-1-carboxylic acid (24) ¹H NMR}	S141
(1 <i>R</i> ^{*,3<i>R</i>^{*,4<i>S</i>[*]})-3-((<i>tert</i>-Butoxycarbonyl)amino)bicyclo[2.1.0]pentane-1-carboxylic acid (24) ¹³C NMR}	S142
(1 <i>R</i> ^{*,3<i>S</i>^{*,4<i>S</i>[*]})-3-(((9<i>H</i>-Fluoren-9-yl)methoxy)carbonyl)amino)bicyclo[2.1.0]pentane-1-carboxylic acid (25) ¹H NMR}	S143
(1 <i>R</i> ^{*,3<i>S</i>^{*,4<i>S</i>[*]})-3-(((9<i>H</i>-Fluoren-9-yl)methoxy)carbonyl)amino)bicyclo[2.1.0]pentane-1-carboxylic acid (25) ¹³C NMR.....}	S144
(1 <i>R</i> ^{*,3<i>R</i>^{*,4<i>S</i>[*]})-3-(((9<i>H</i>-fluoren-9-yl)methoxy)carbonyl)amino)bicyclo[2.1.0]pentane-1-carboxylic acid (26) ¹H NMR.....}	S145
(1 <i>R</i> ^{*,3<i>R</i>^{*,4<i>S</i>[*]})-3-(((9<i>H</i>-fluoren-9-yl)methoxy)carbonyl)amino)bicyclo[2.1.0]pentane-1-carboxylic acid (26) ¹³C NMR.....}	S146
<i>tert</i> -Butyl 3-oxocyclopentanecarboxylate (28) ¹ H NMR.....	S147
<i>tert</i> -Butyl 3-oxocyclopentanecarboxylate (28) ¹³ C NMR.....	S148
<i>tert</i> -Butyl 3-hydroxycyclopentanecarboxylate (29) ¹ H NMR.....	S149
<i>tert</i> -Butyl 3-hydroxycyclopentanecarboxylate (29) ¹³ C NMR.....	S150
<i>tert</i> -Butyl 3-bromocyclopentanecarboxylate (30) ¹ H NMR	S151

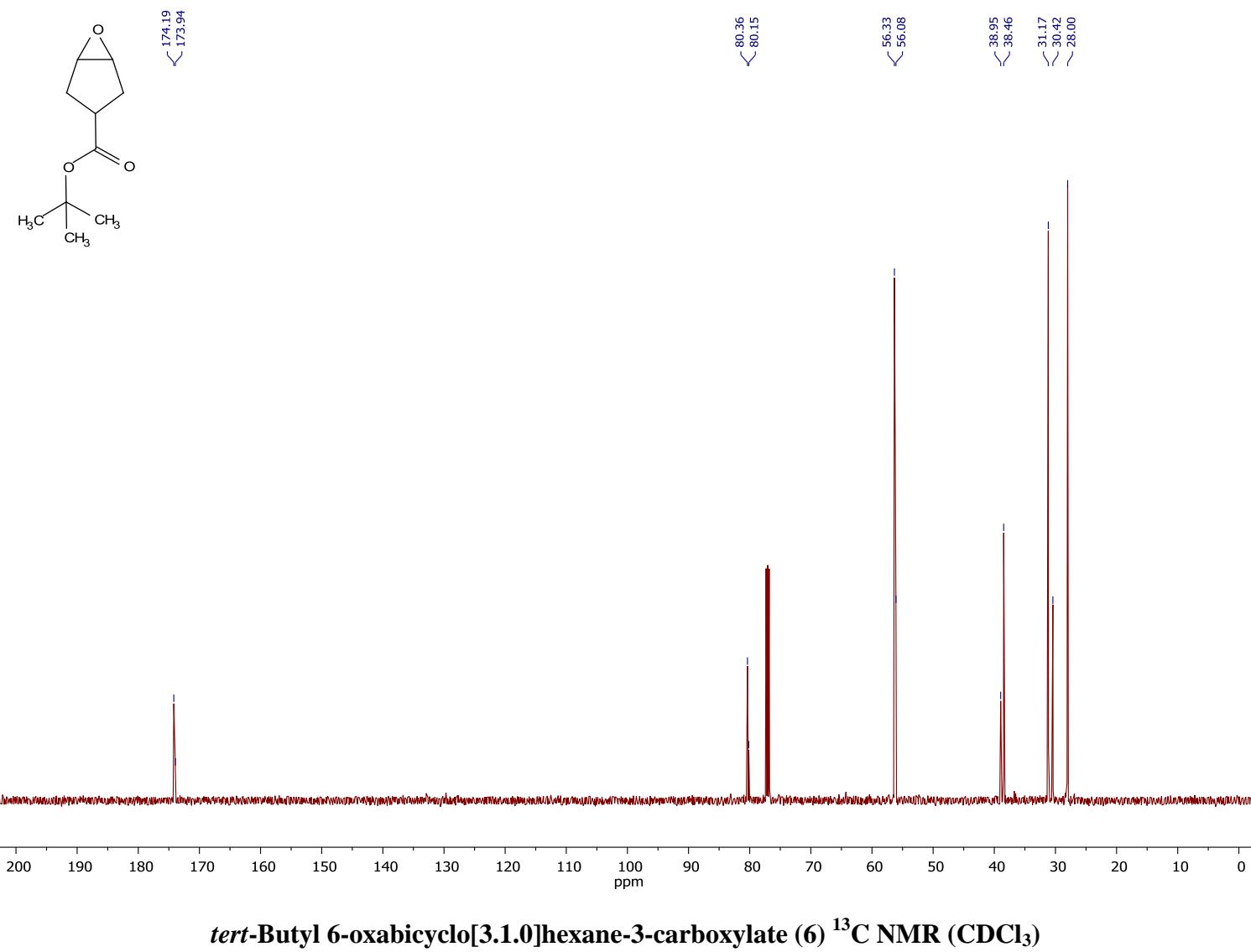
<i>tert</i> -Butyl 3-bromocyclopentanecarboxylate (30) ^{13}C NMR	S152
<i>tert</i> -Butyl bicyclo[2.1.0]pentane-1-carboxylate (31) ^1H NMR	S153
<i>tert</i> -Butyl bicyclo[2.1.0]pentane-1-carboxylate (31) ^1H NMR	S154
<i>cis</i> -4-Carboxybicyclo[2.1.0]pentan-2-aminium chloride ((<i>1S,2S,4R</i>)-23 or (<i>1R,2R,4S</i>)-23) ^1H NMR.....	S155
<i>cis</i> -4-Carboxybicyclo[2.1.0]pentan-2-aminium chloride ((<i>1S,2S,4R</i>)-23 or (<i>1R,2R,4S</i>)-23) ^{13}C NMR.....	S156
<i>trans</i> -4-Carboxybicyclo[2.1.0]pentan-2-aminium chloride ((<i>1S,2R,4R</i>)-24 or (<i>1R,2S,4S</i>)-24) ^1H NMR	S157
<i>trans</i> -4-Carboxybicyclo[2.1.0]pentan-2-aminium chloride ((<i>1S,2R,4R</i>)-24 or (<i>1R,2S,4S</i>)-24) ^{13}C NMR	S158
(<i>1R*,3S*,4R*</i>)-3-phenylbicyclo[2.1.0]pentane-1-carboxylic acid 10b (conformer A) ORTEP diagrams.....	S159
(<i>1R*,3S*,4R*</i>)-3-phenylbicyclo[2.1.0]pentane-1-carboxylic acid 10b (conformer B) ORTEP diagrams.....	S160
(<i>1R*,3R*,4R*</i>)-3-phenylbicyclo[2.1.0]pentane-1-carboxylic acid 17b ORTEP diagrams.....	S161
(<i>1S,2S,4R</i>)-3-aminobicyclo[2.1.0]pentane-1-carboxylic acid hydrochloride (<i>1S,2S,4R</i>)-21 ORTEP diagrams	S162
(<i>1R,2R,4S</i>)-3-aminobicyclo[2.1.0]pentane-1-carboxylic acid hydrochloride (<i>1R,2R,4S</i>)-21 ORTEP diagrams.....	S163
(<i>1S,2R,4R</i>)-3-aminobicyclo[2.1.0]pentane-1-carboxylic acid hydrochloride (<i>1S,2R,4R</i>)-21 ORTEP diagrams.....	S164
(<i>1R,2S,4S</i>)-3-aminobicyclo[2.1.0]pentane-1-carboxylic acid hydrochloride (<i>1R,2S,4S</i>)-21 ORTEP diagrams	S165
X-Ray diffraction studies of 10b, 17b, (<i>1S,2S,4R</i>)-21, (<i>1R,2R,4S</i>)-21, (<i>1S,2R,4R</i>)-22, (<i>1R,2S,4S</i>)-22.	S166
Table S1. The crystallographic data and experimental parameters for compounds 10b, 17b, (<i>1S,2S,4R</i>)-21, (<i>1R,2R,4S</i>)-21, (<i>1S,2R,4R</i>)-22, (<i>1R,2S,4S</i>)-22	S166

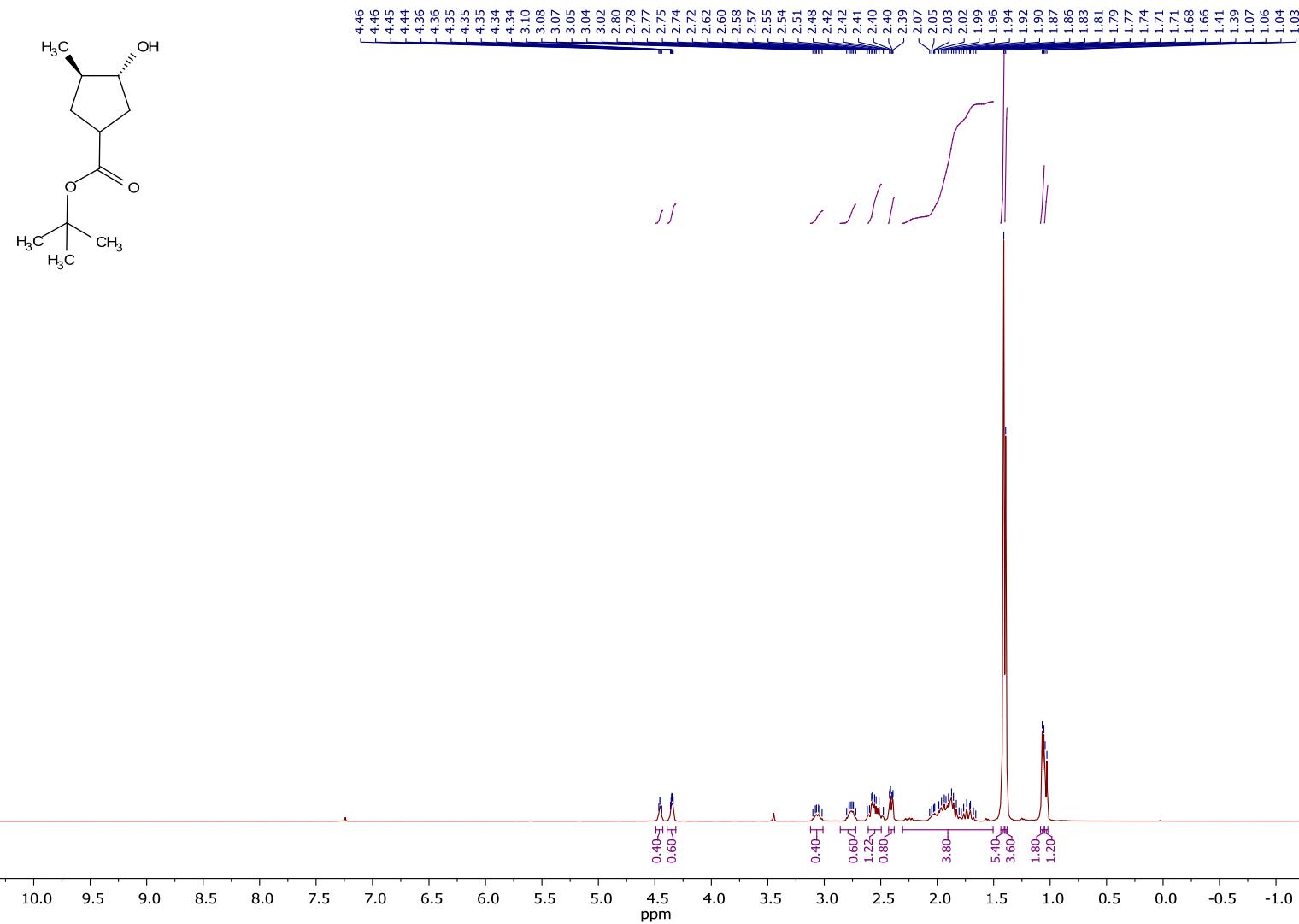


tert-Butyl cyclopent-3-enecarboxylate (**5**) ¹H NMR (CDCl₃)

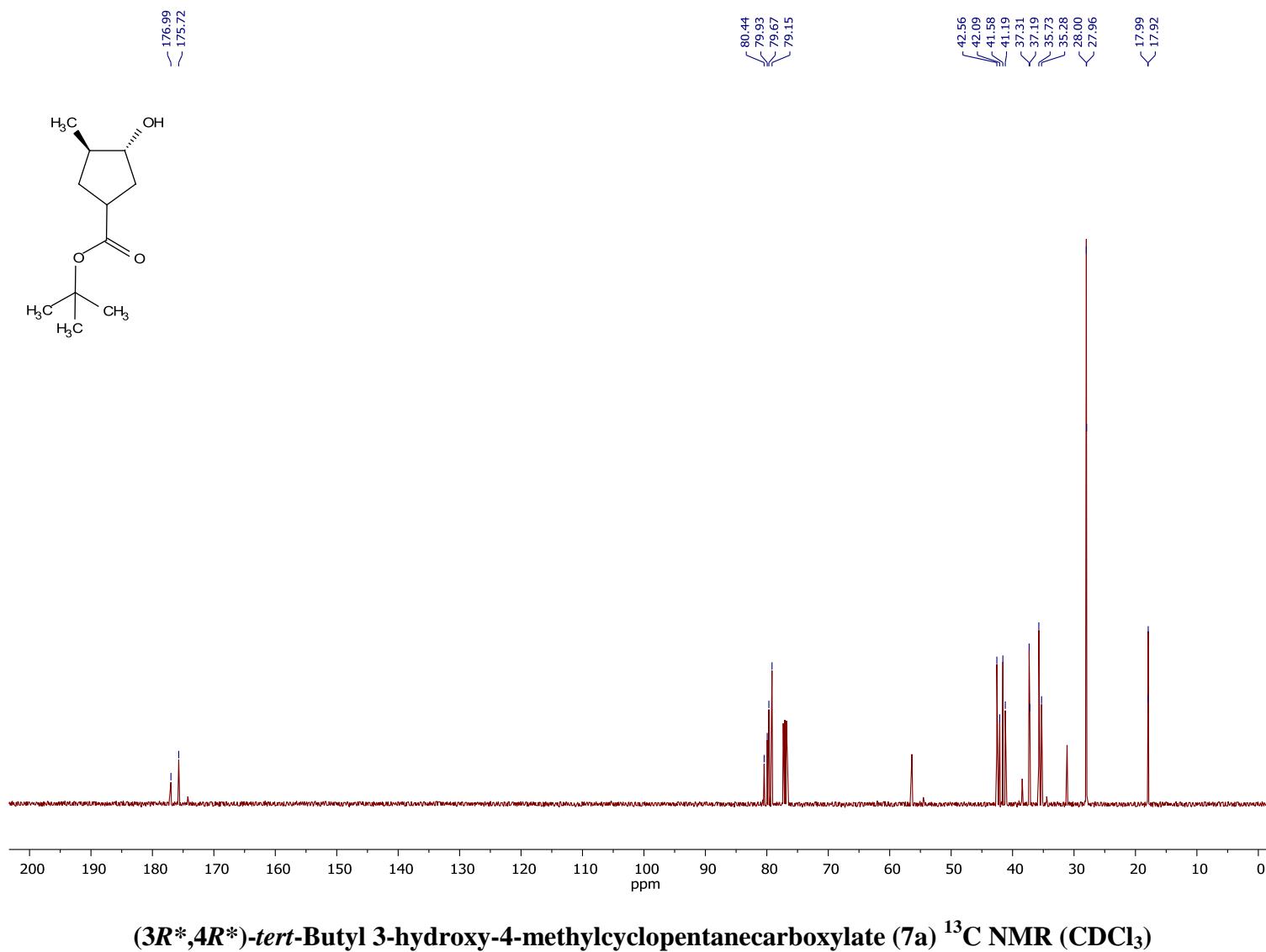


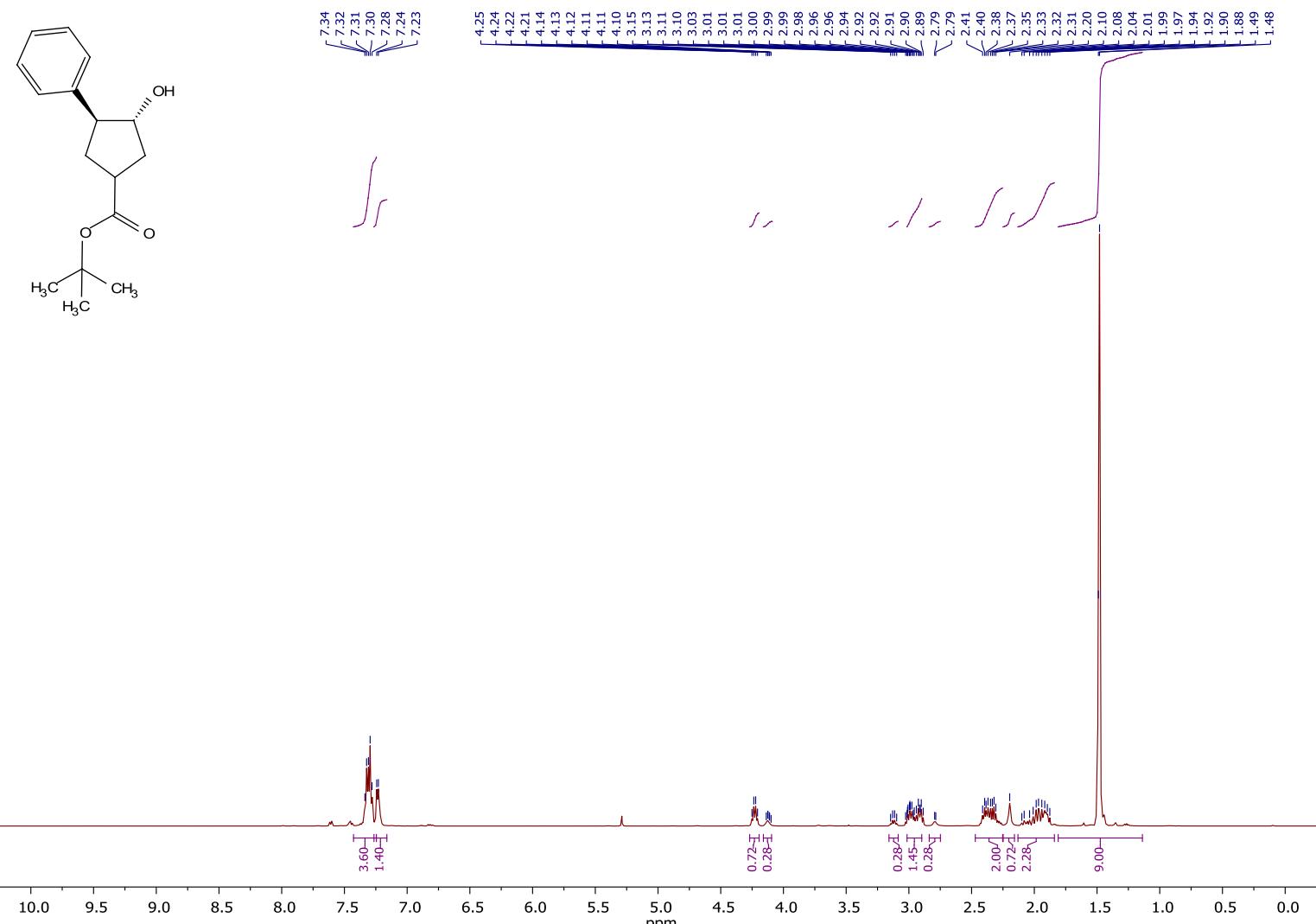




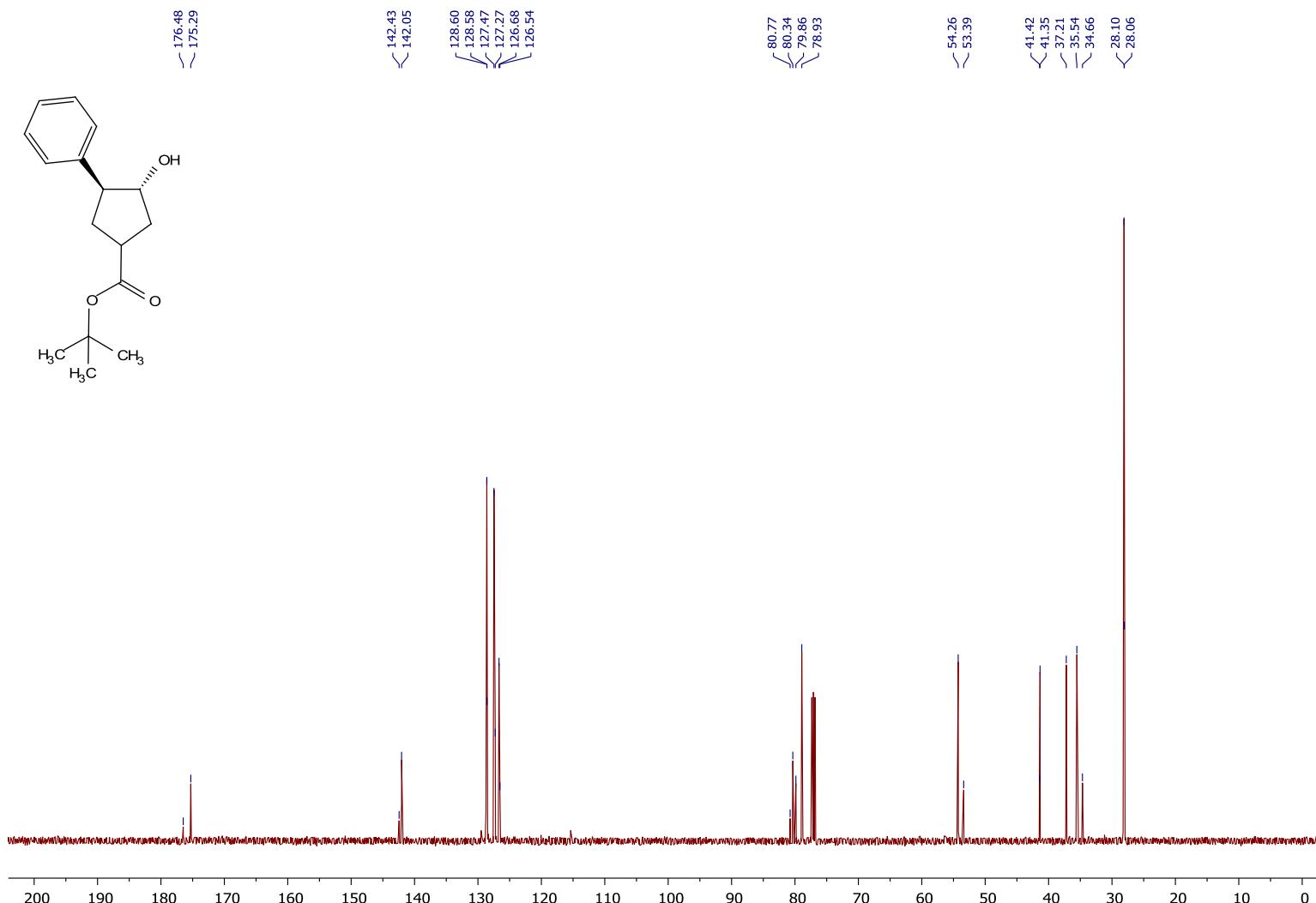


(3*R*^{*,4*R*^{*})-*tert*-Butyl 3-hydroxy-4-methylcyclopentanecarboxylate (7a)} ¹H NMR (CDCl₃)

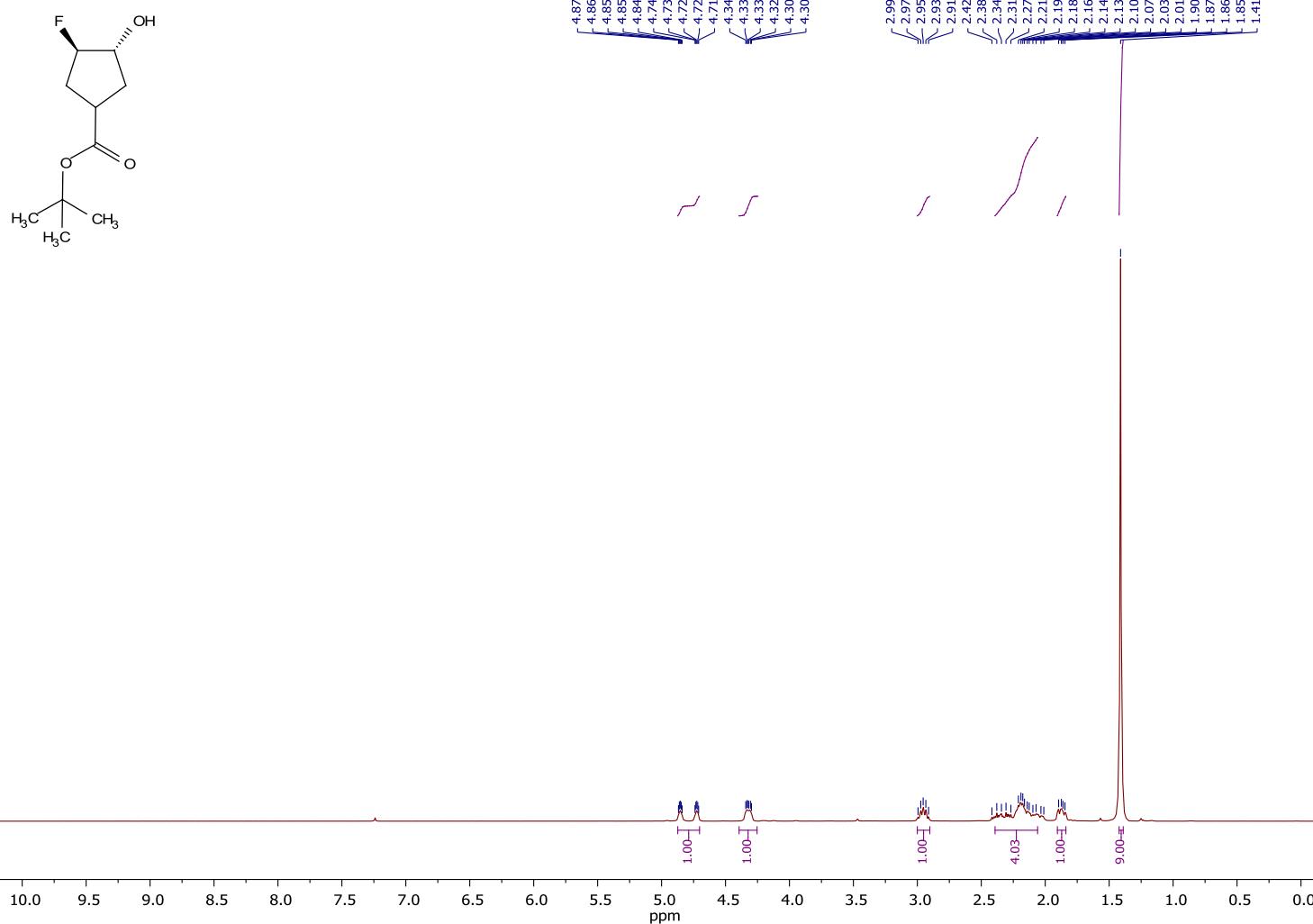




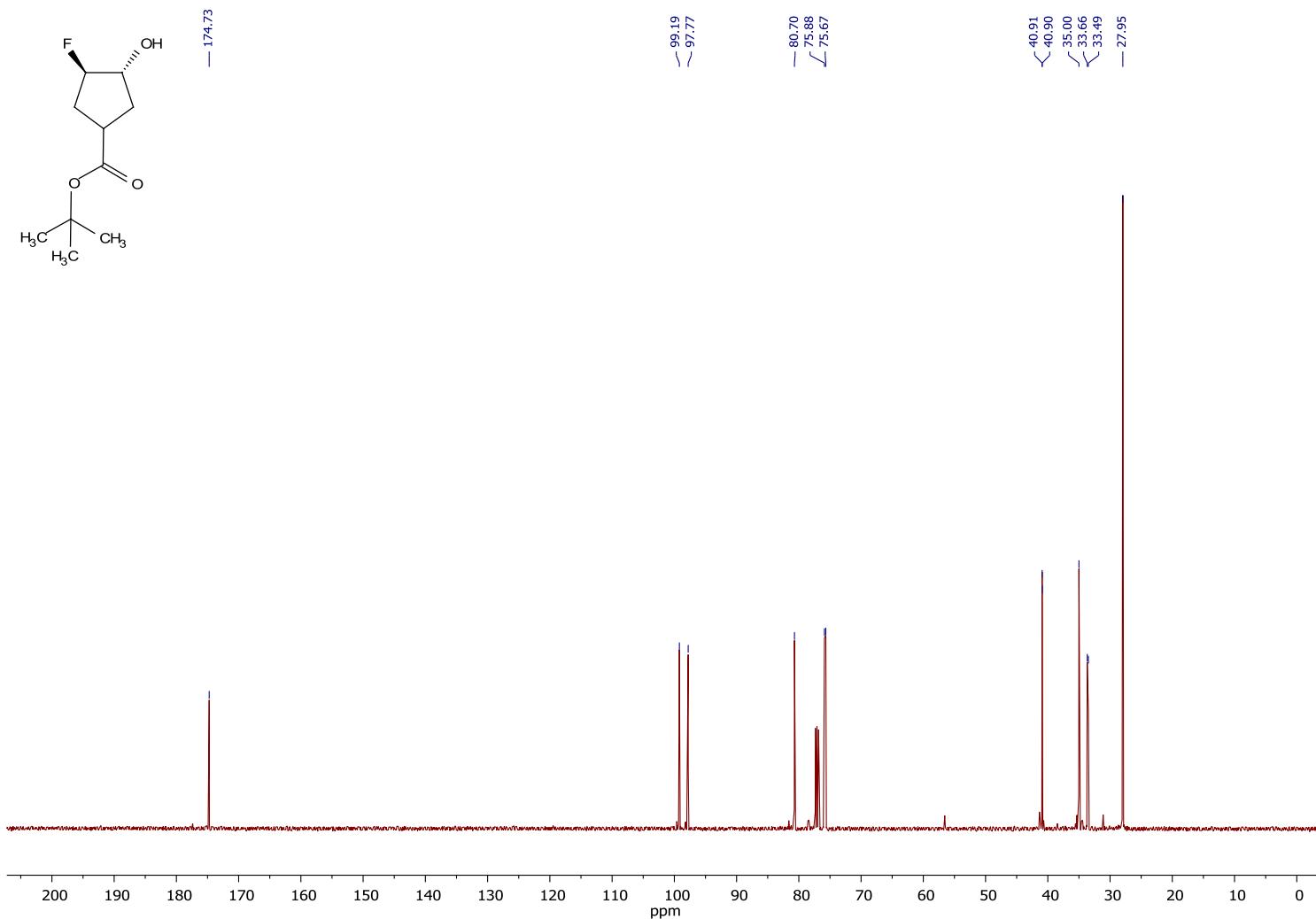
(3*R,4*S**)-tert-Butyl 3-hydroxy-4-phenylcyclopentanecarboxylate (7b) ^1H NMR (CDCl_3)**

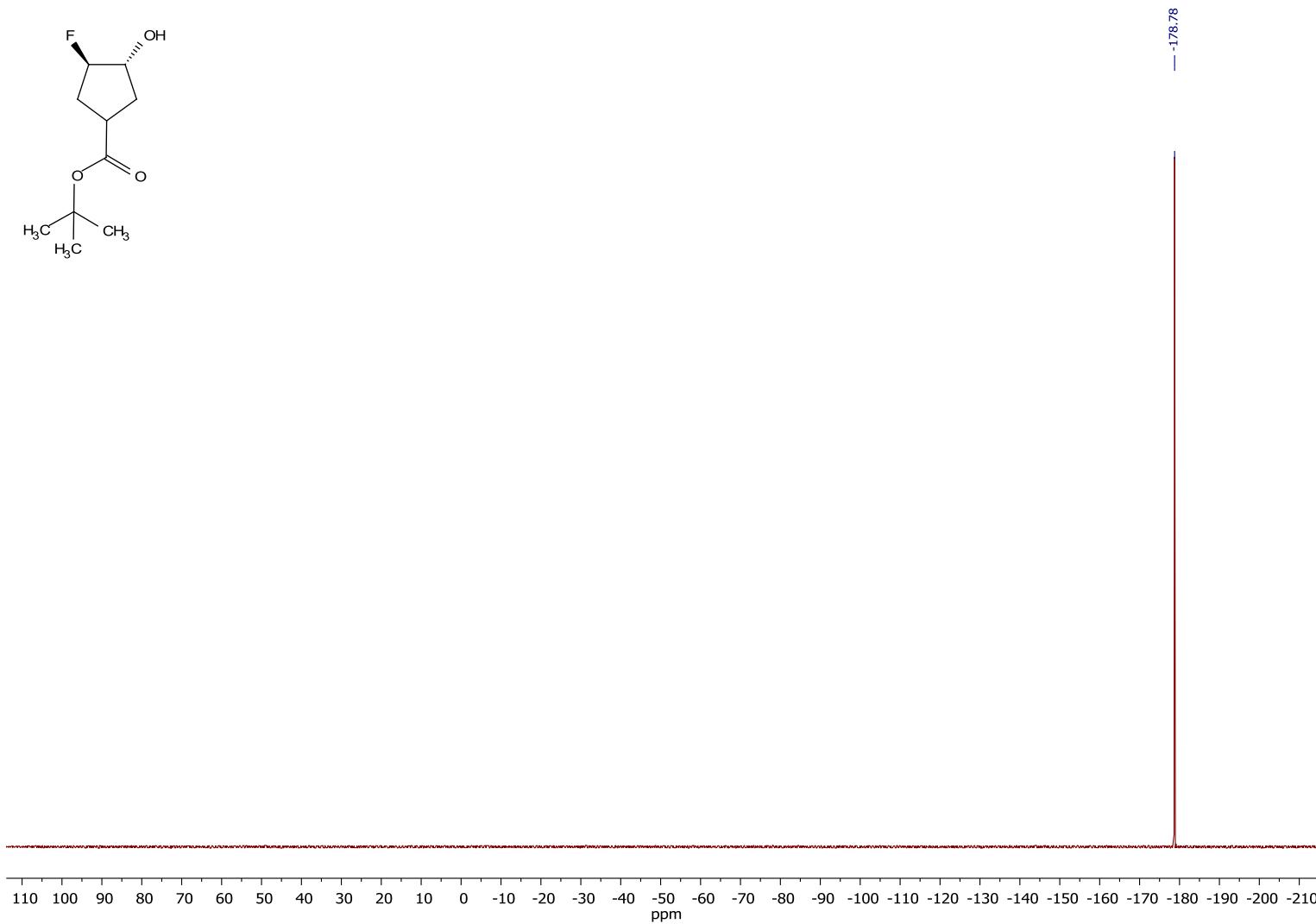
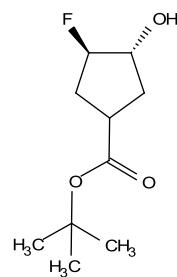


(3*R*^{*,4*S*^{*})-*tert*-Butyl 3-hydroxy-4-phenylcyclopentanecarboxylate (7b) ^{13}C NMR (CDCl_3)}

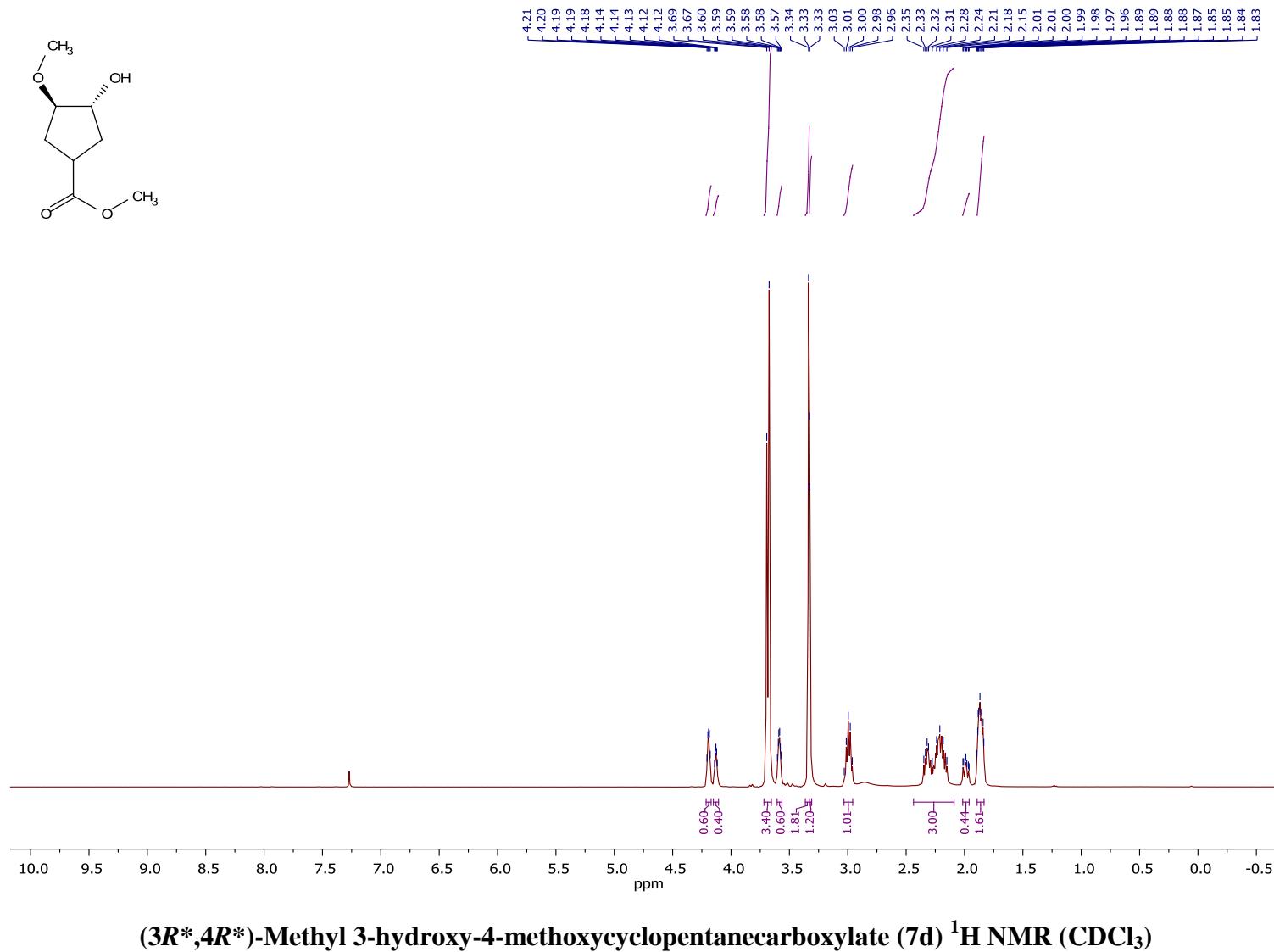
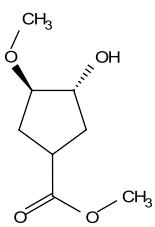


(3*R*^{*},4*R*^{*})-*tert*-Butyl 3-fluoro-4-hydroxycyclopentanecarboxylate (7c) ^1H NMR (CDCl_3)

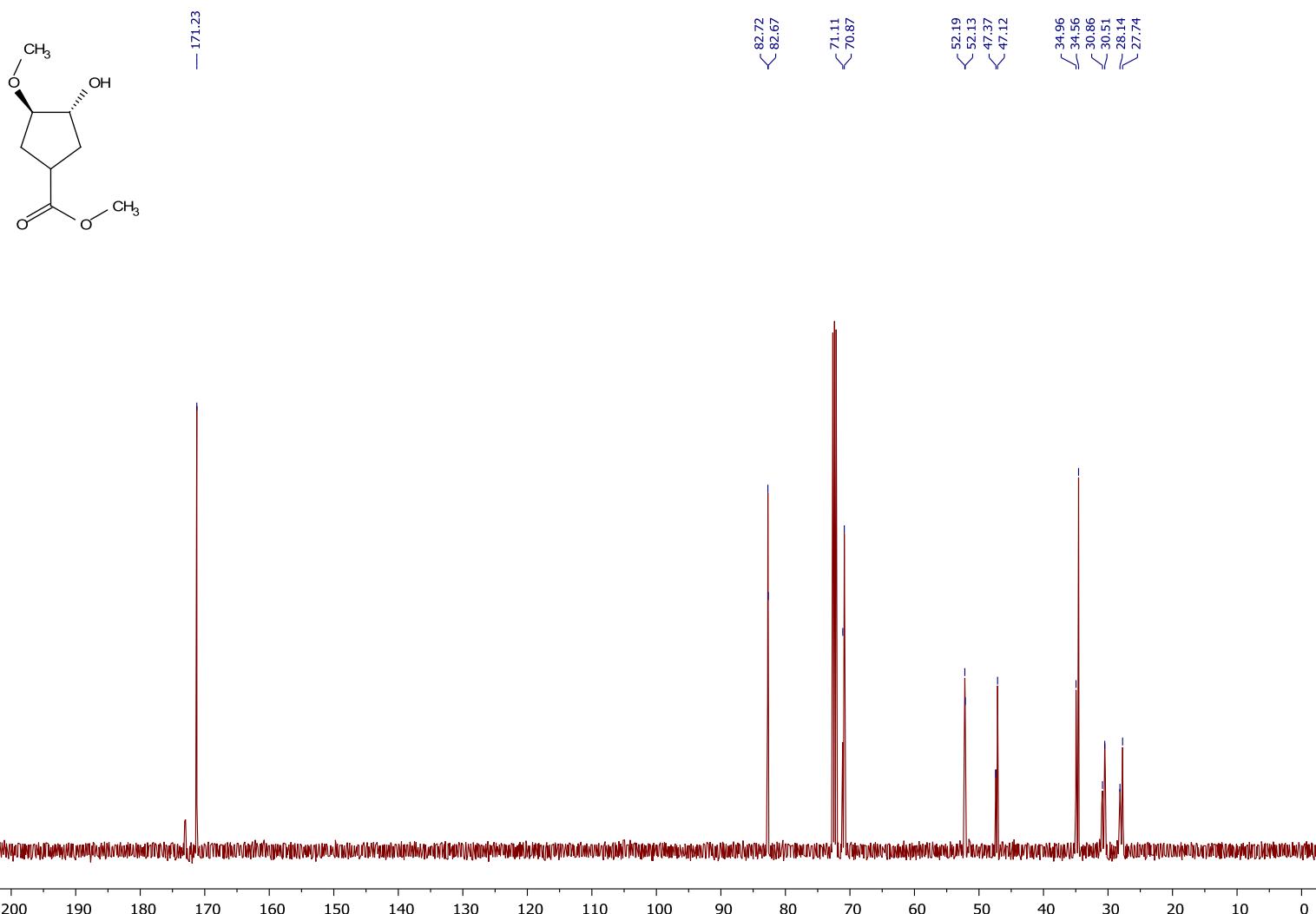




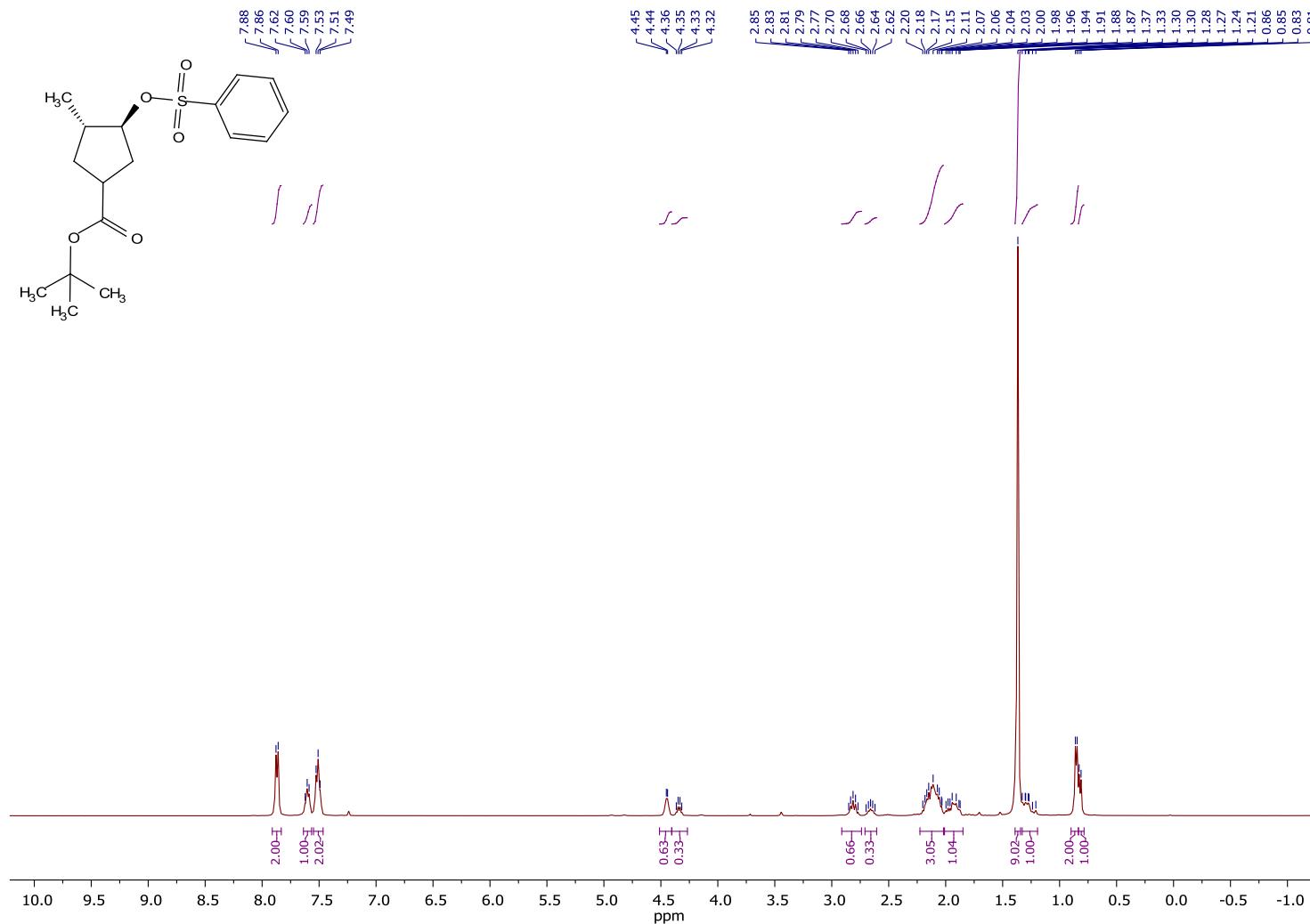
(3*R*^{*},4*R*^{*})-*tert*-Butyl 3-fluoro-4-hydroxycyclopentanecarboxylate (7c) ^{19}F NMR (CDCl_3)

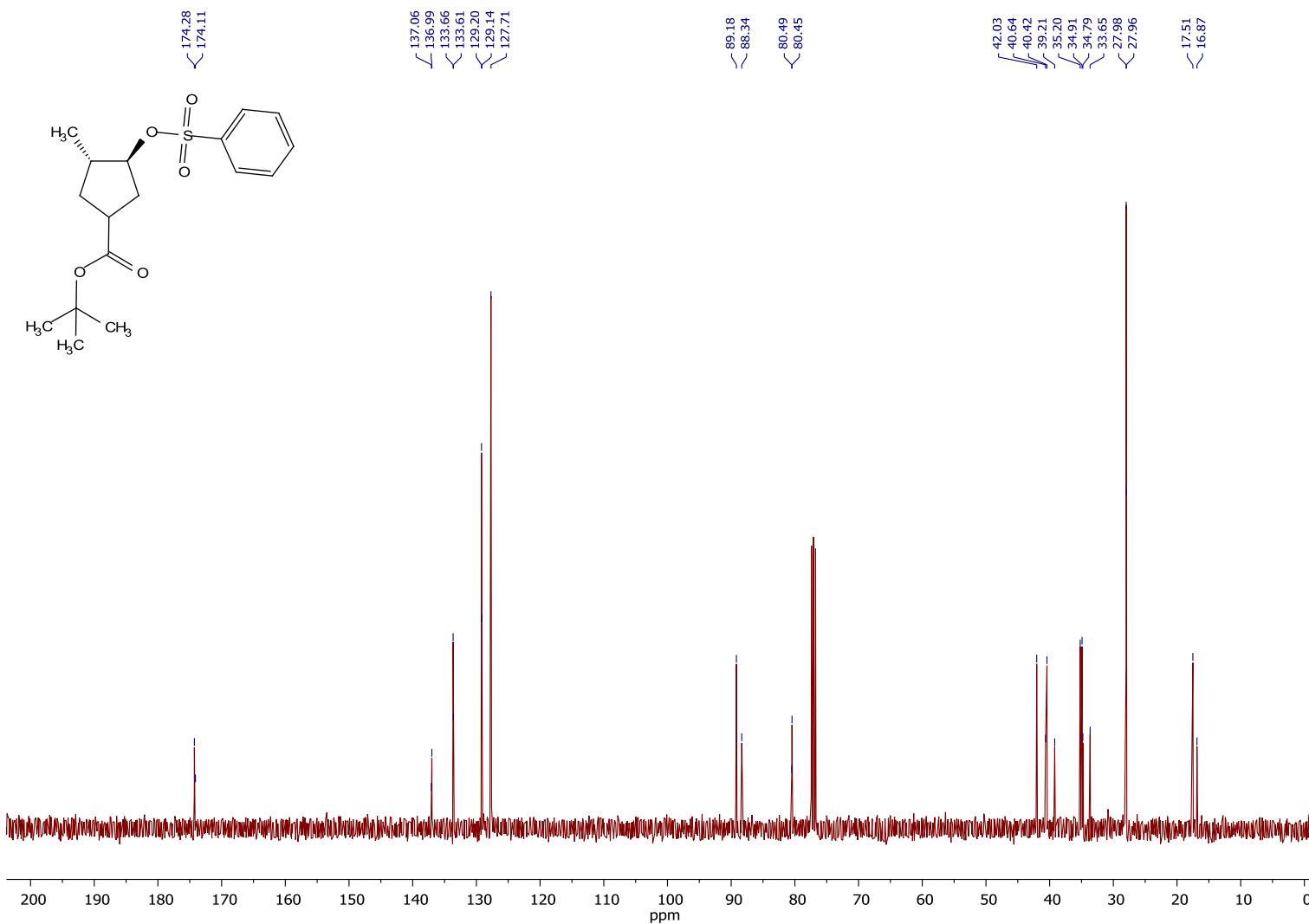


(3*R*^{*},4*R*^{*})-Methyl 3-hydroxy-4-methoxycyclopentanecarboxylate (7d) ¹H NMR (CDCl₃)

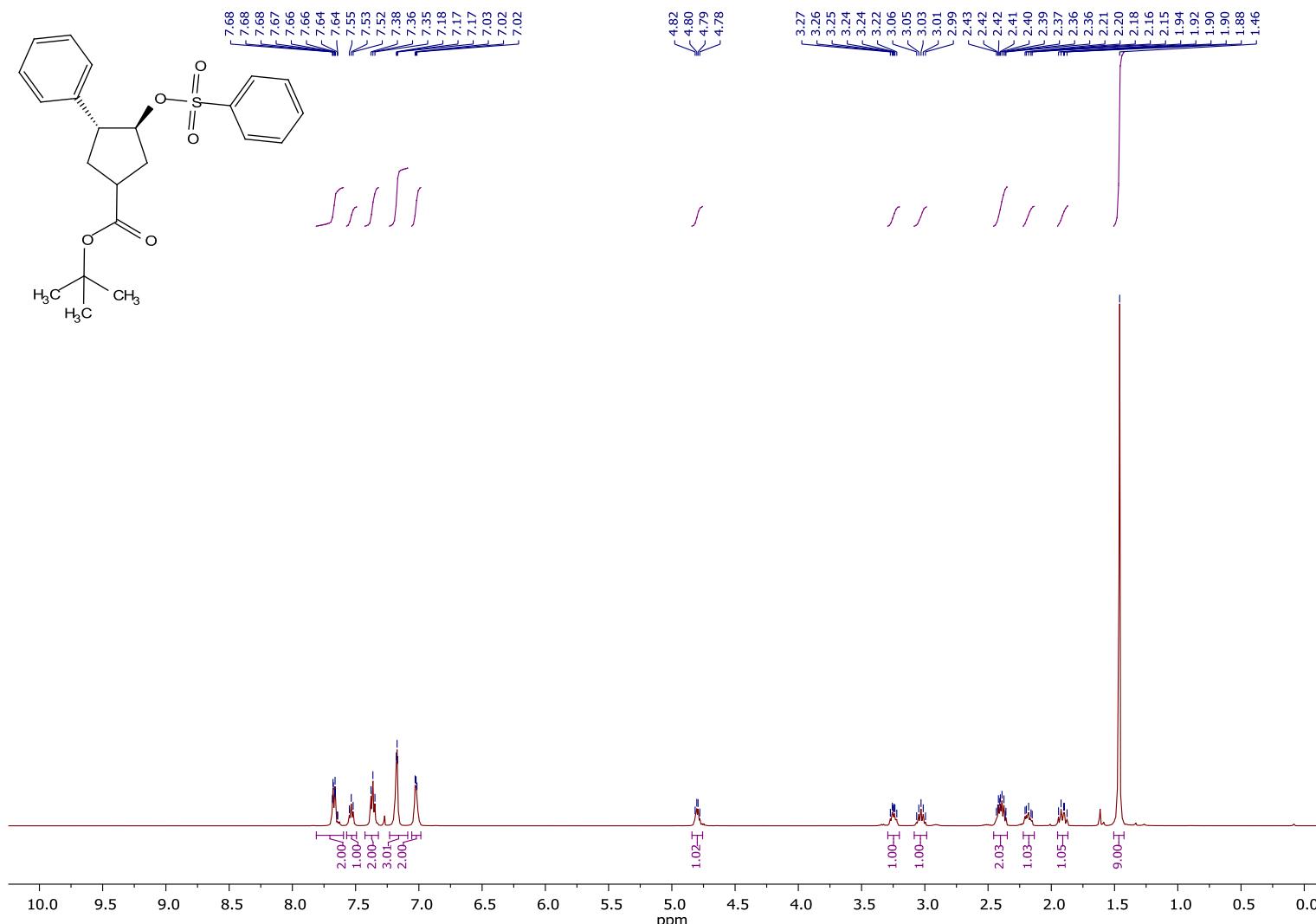


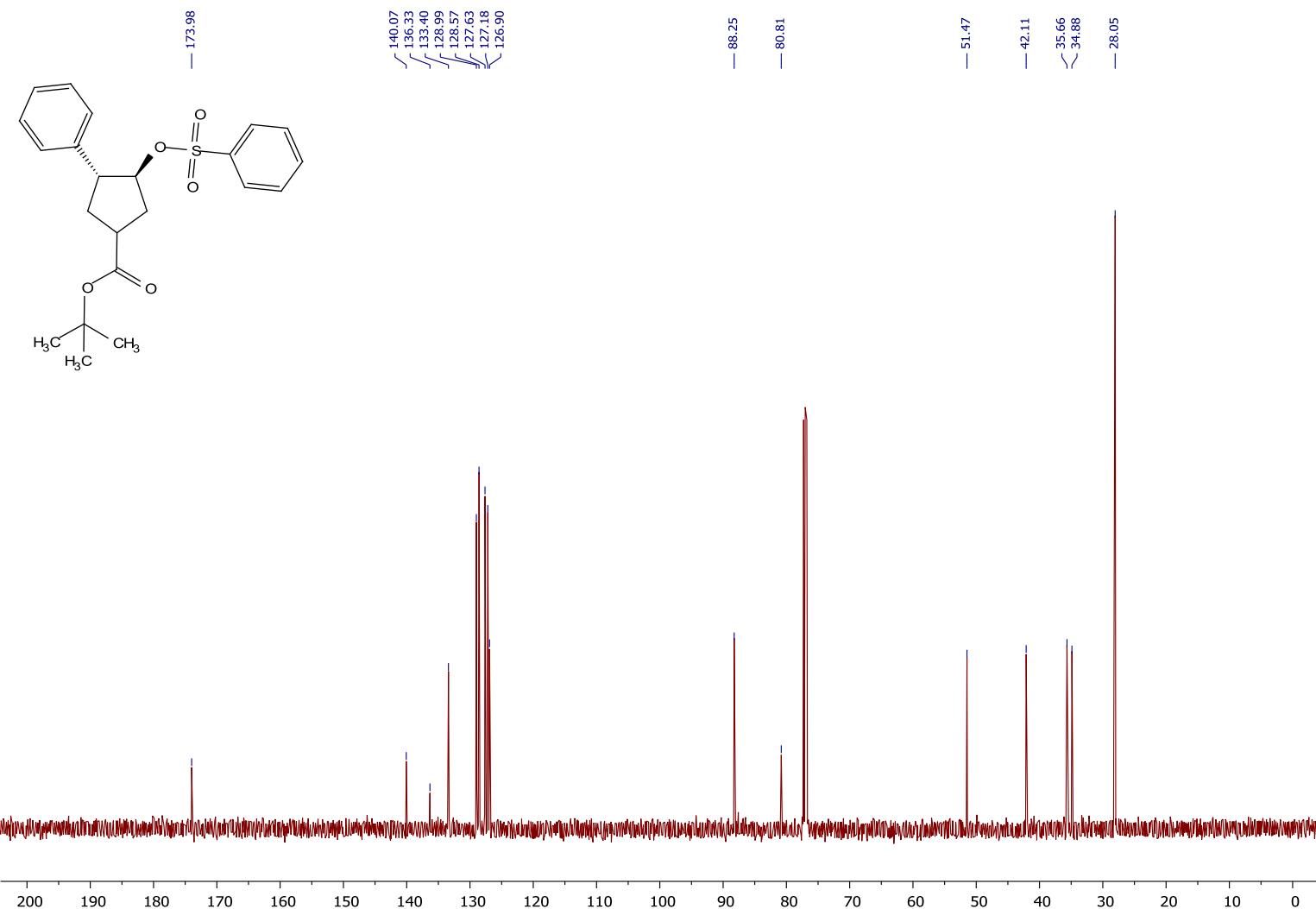
(3*R*^{*,4*R*^{*})-Methyl 3-hydroxy-4-methoxycyclopentanecarboxylate (7d) ^{13}C NMR (CDCl_3)}

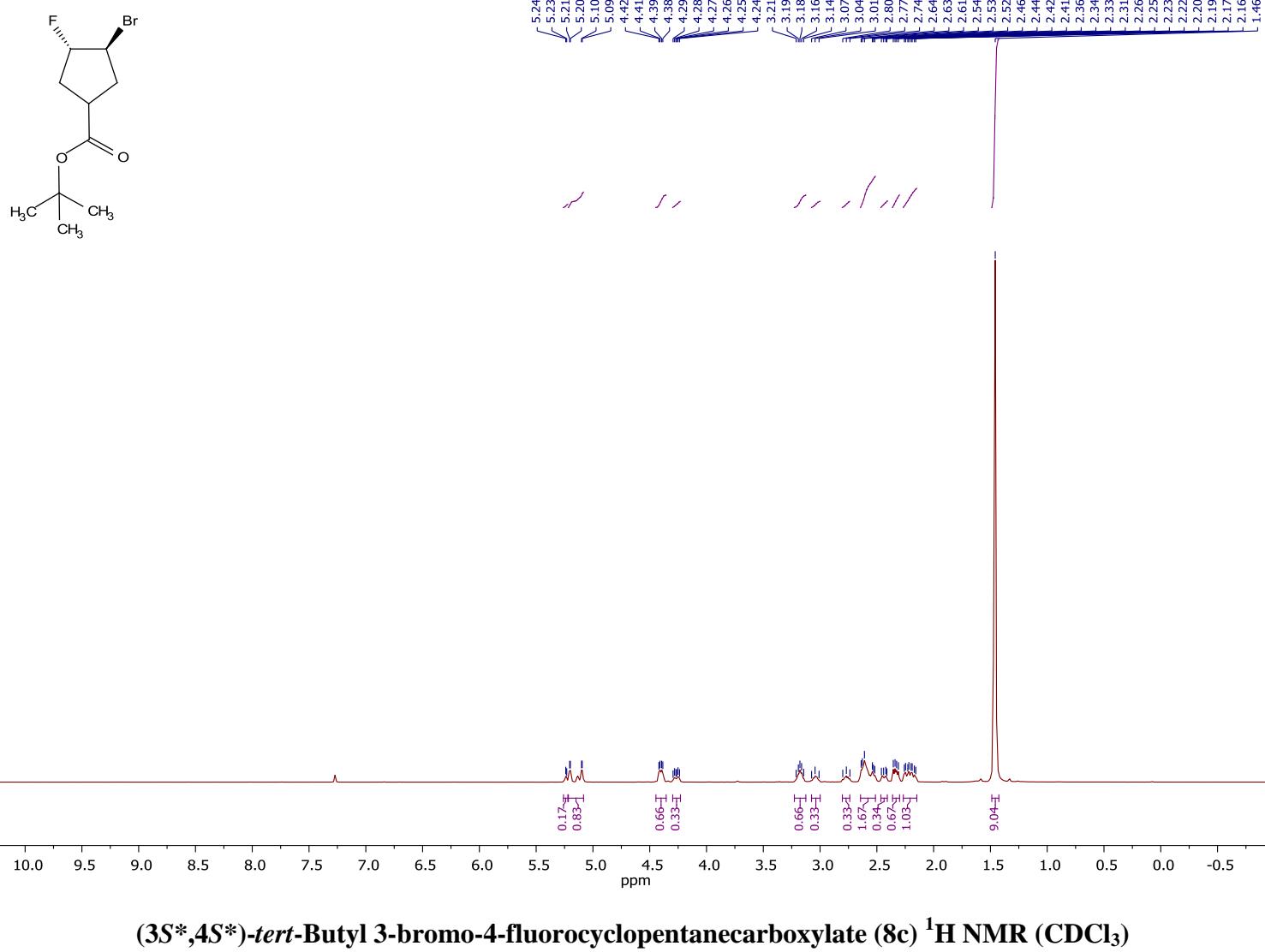




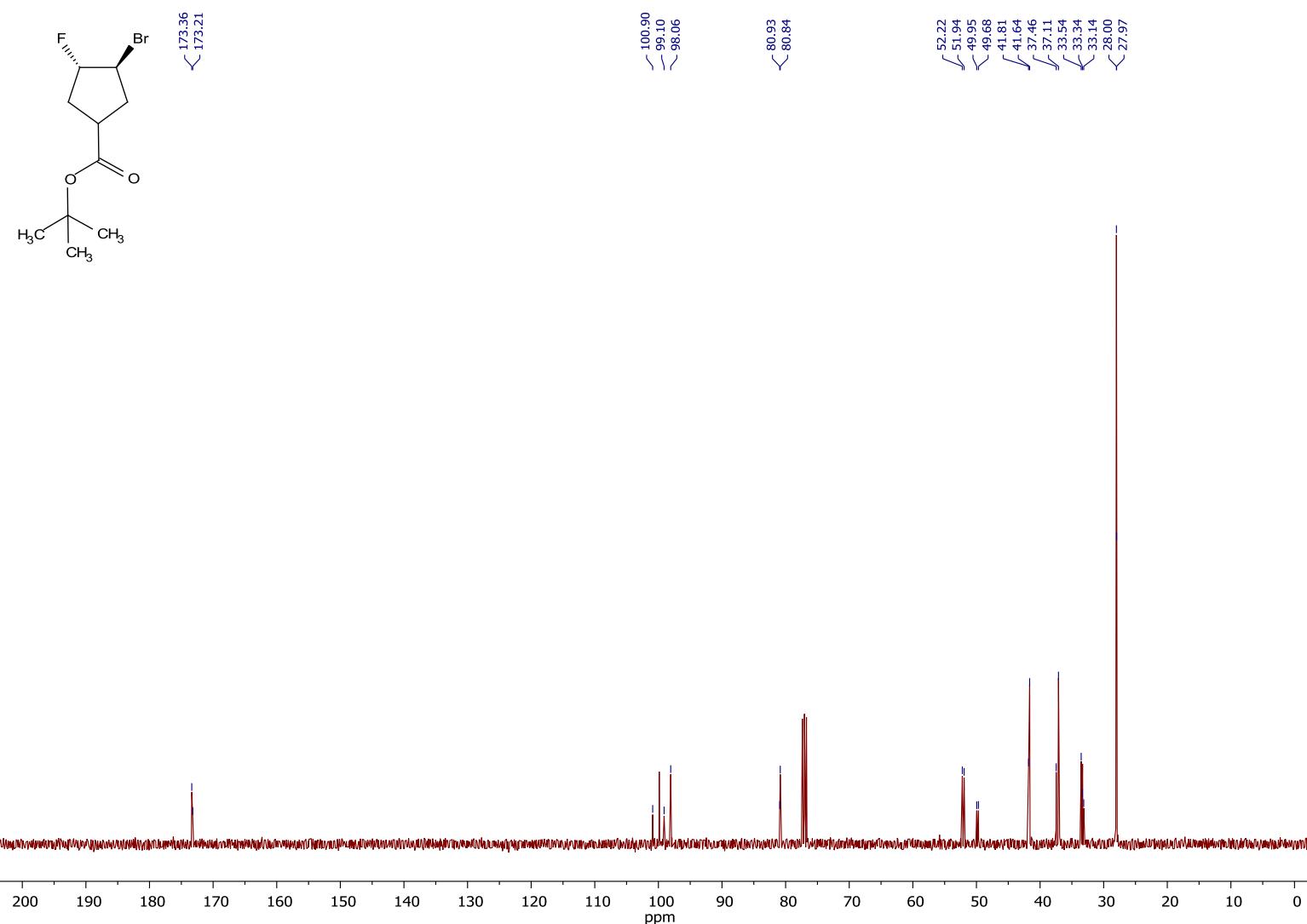
$(3S^*,4S^*)$ -*tert*-Butyl 3-methyl-4-((phenylsulfonyl)oxy)cyclopentanecarboxylate (**8a**) ^{13}C NMR (CDCl_3)

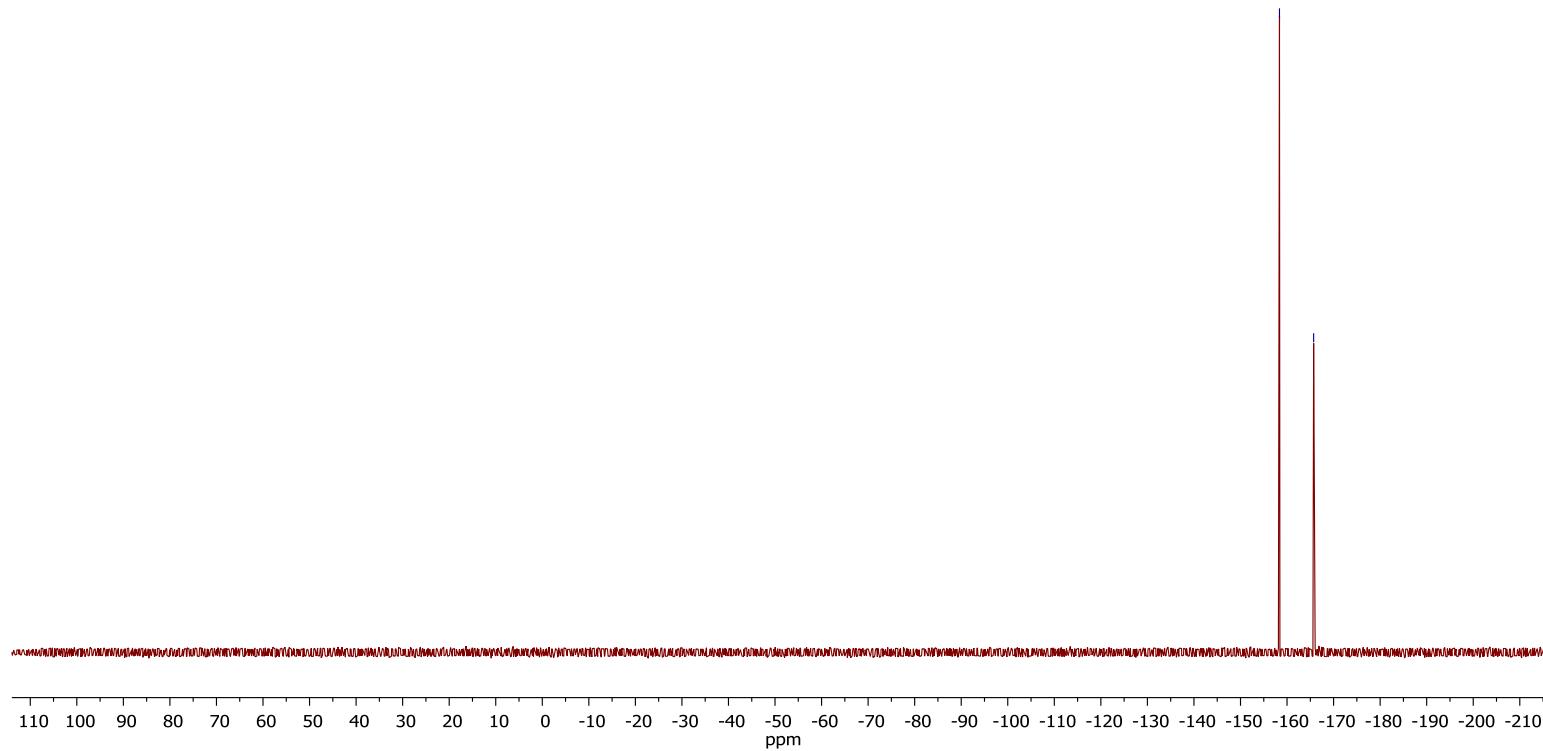
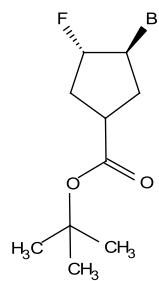




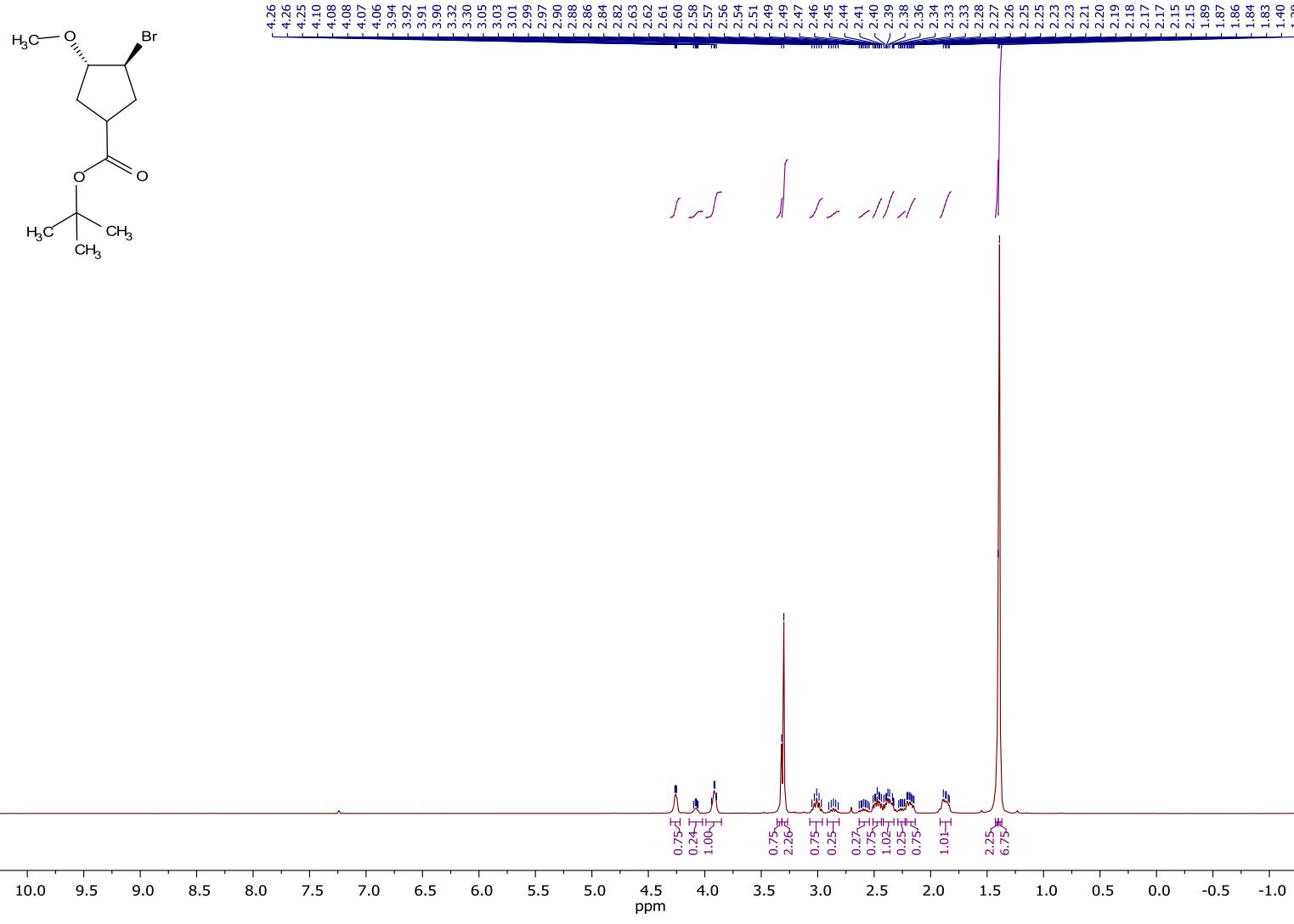


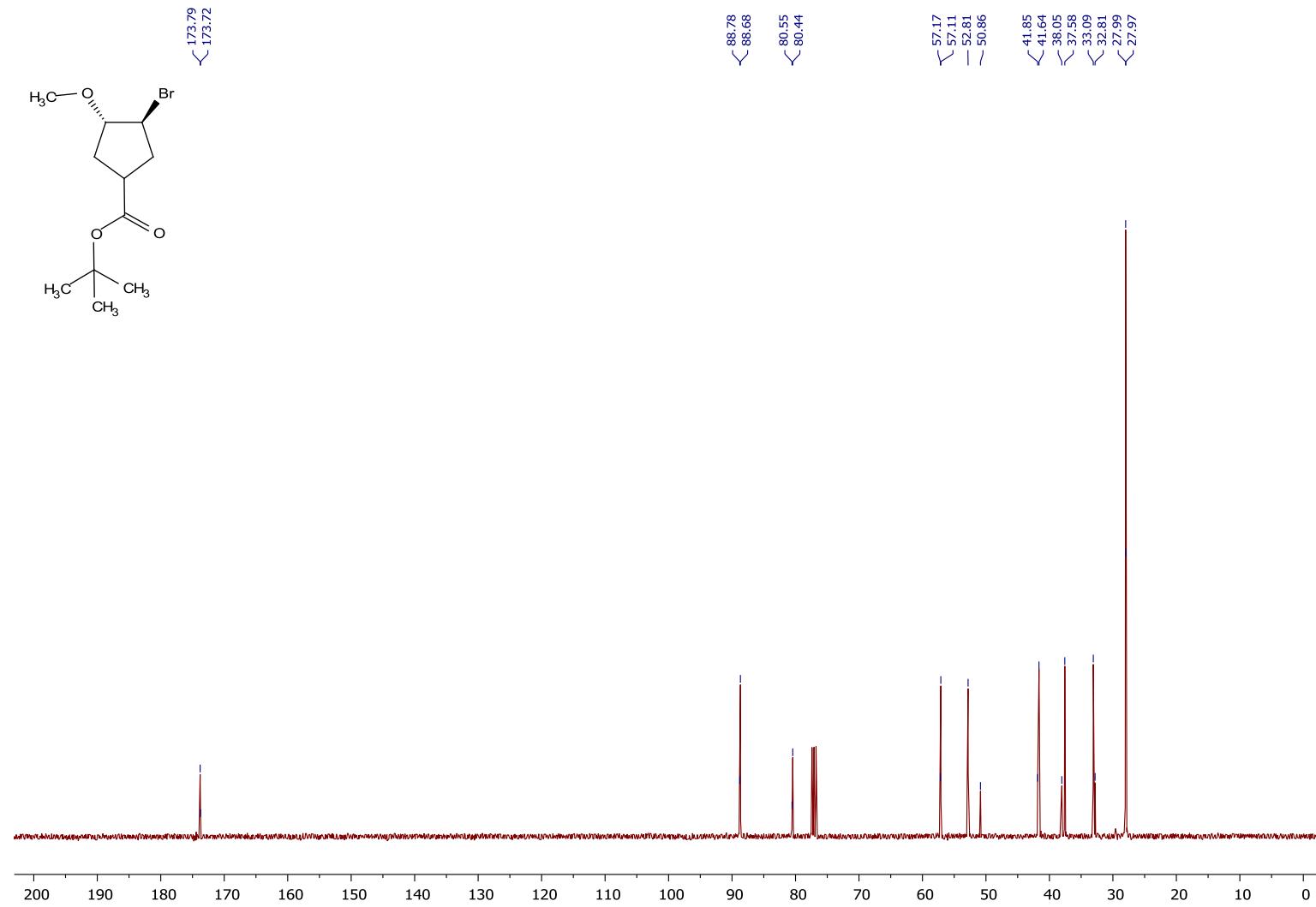
(3*S**,4*S**)-*tert*-Butyl 3-bromo-4-fluorocyclopentanecarboxylate (8c) ^1H NMR (CDCl₃)

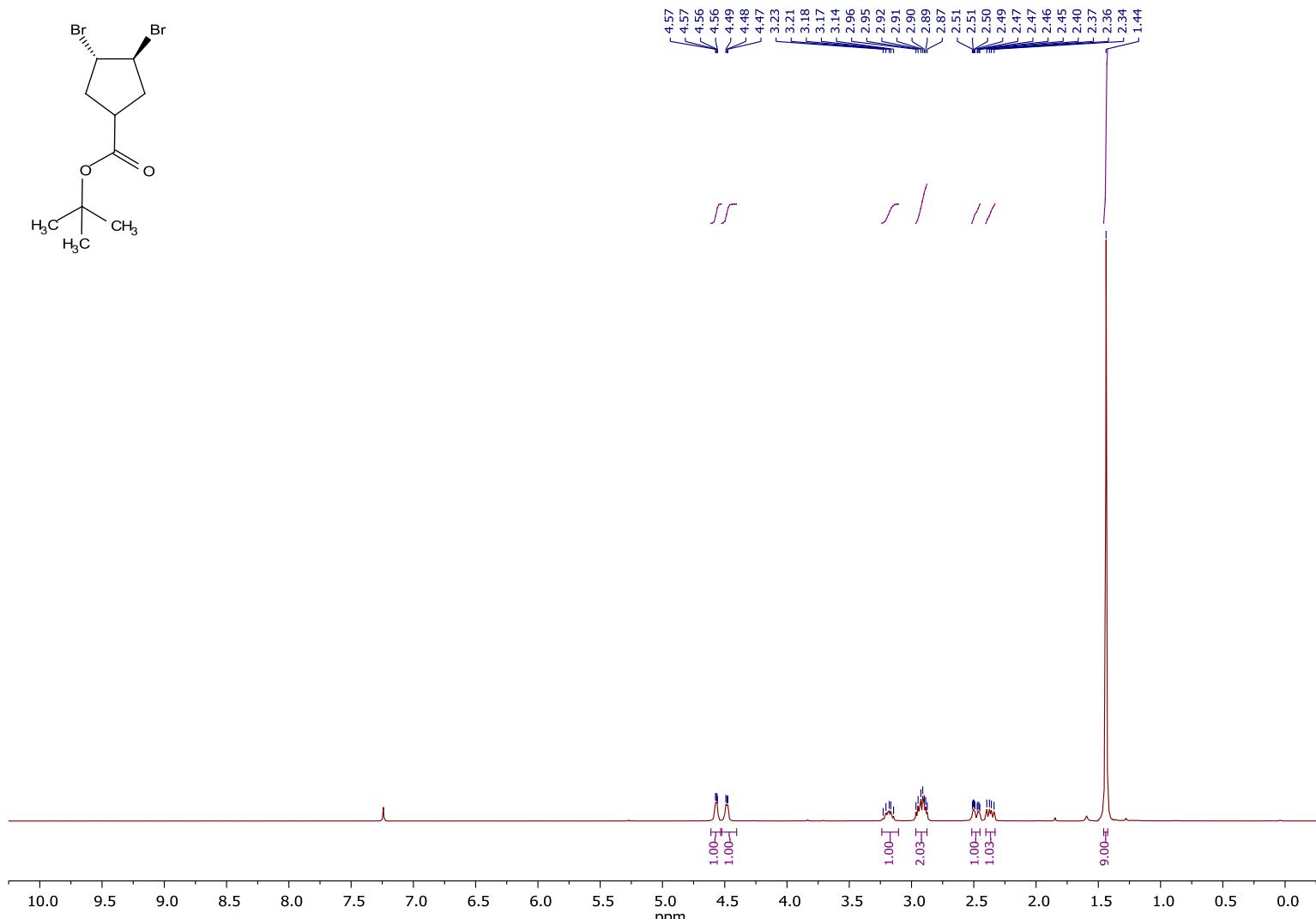
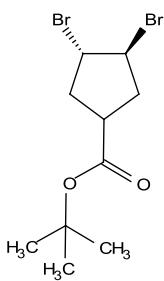




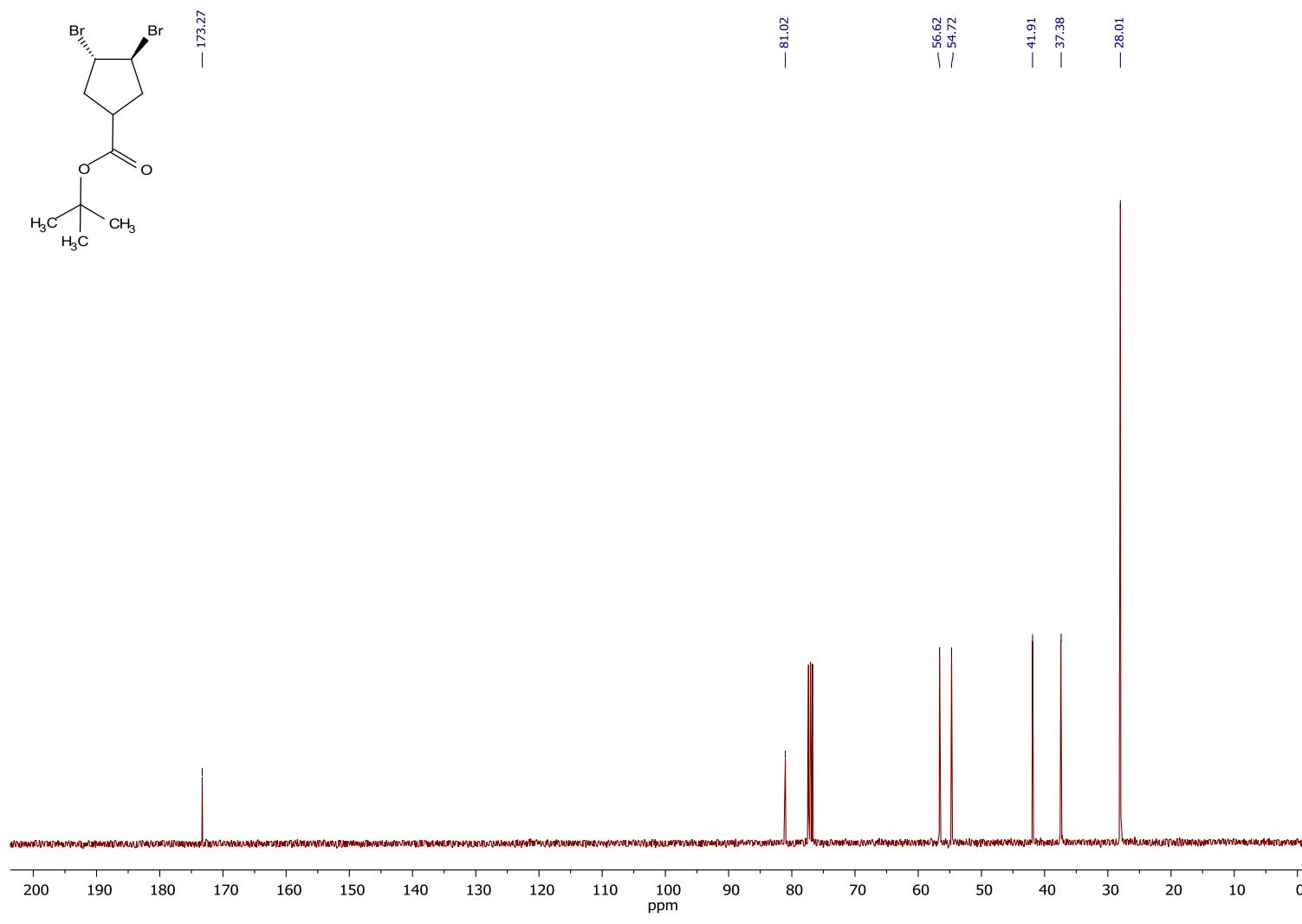
(3*S,4*S**)-tert-Butyl 3-bromo-4-fluorocyclopentanecarboxylate (8c)** ¹⁹F NMR (CDCl_3)



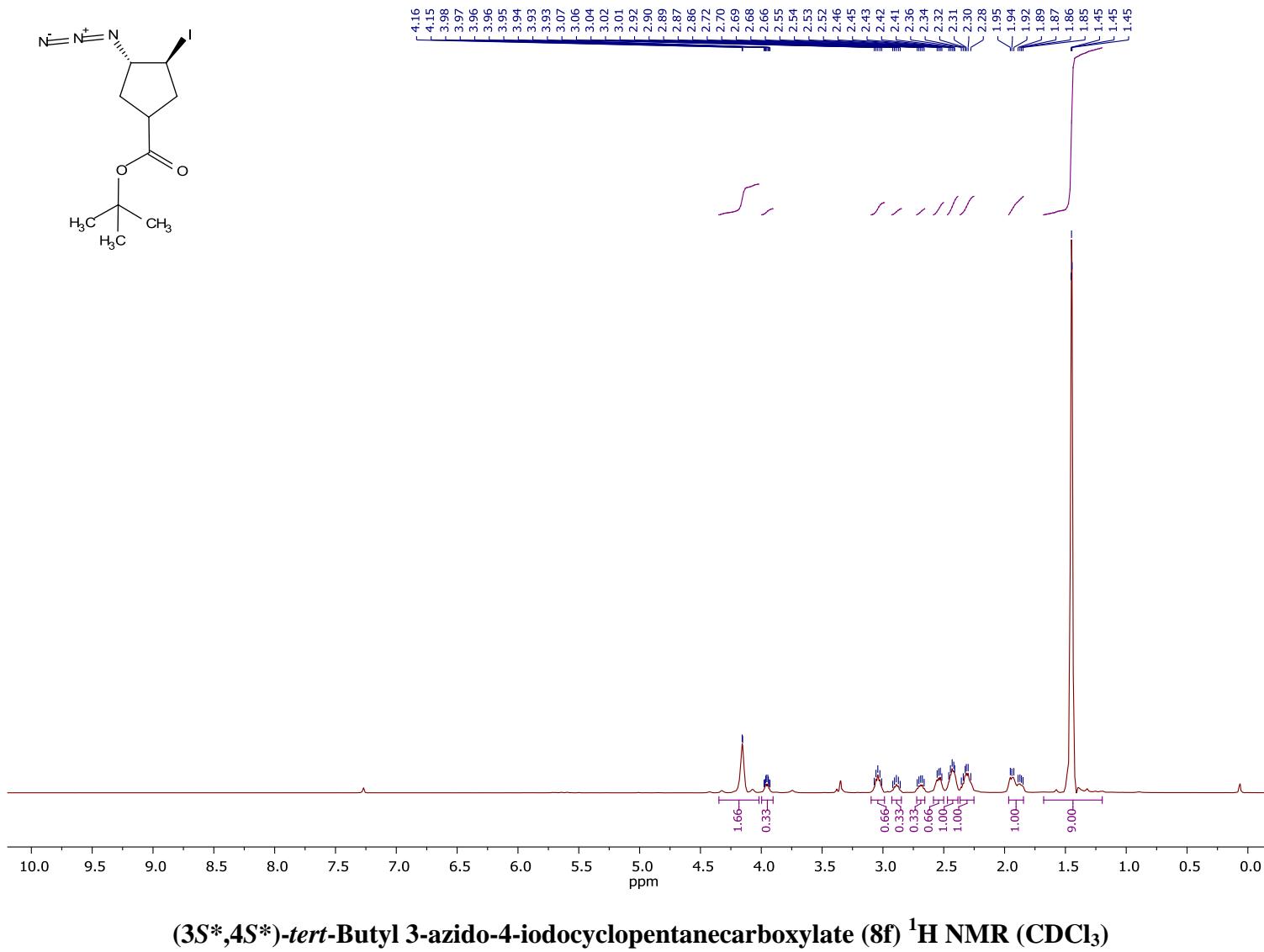
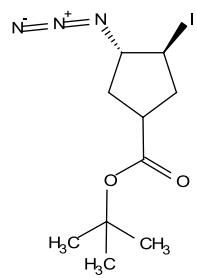




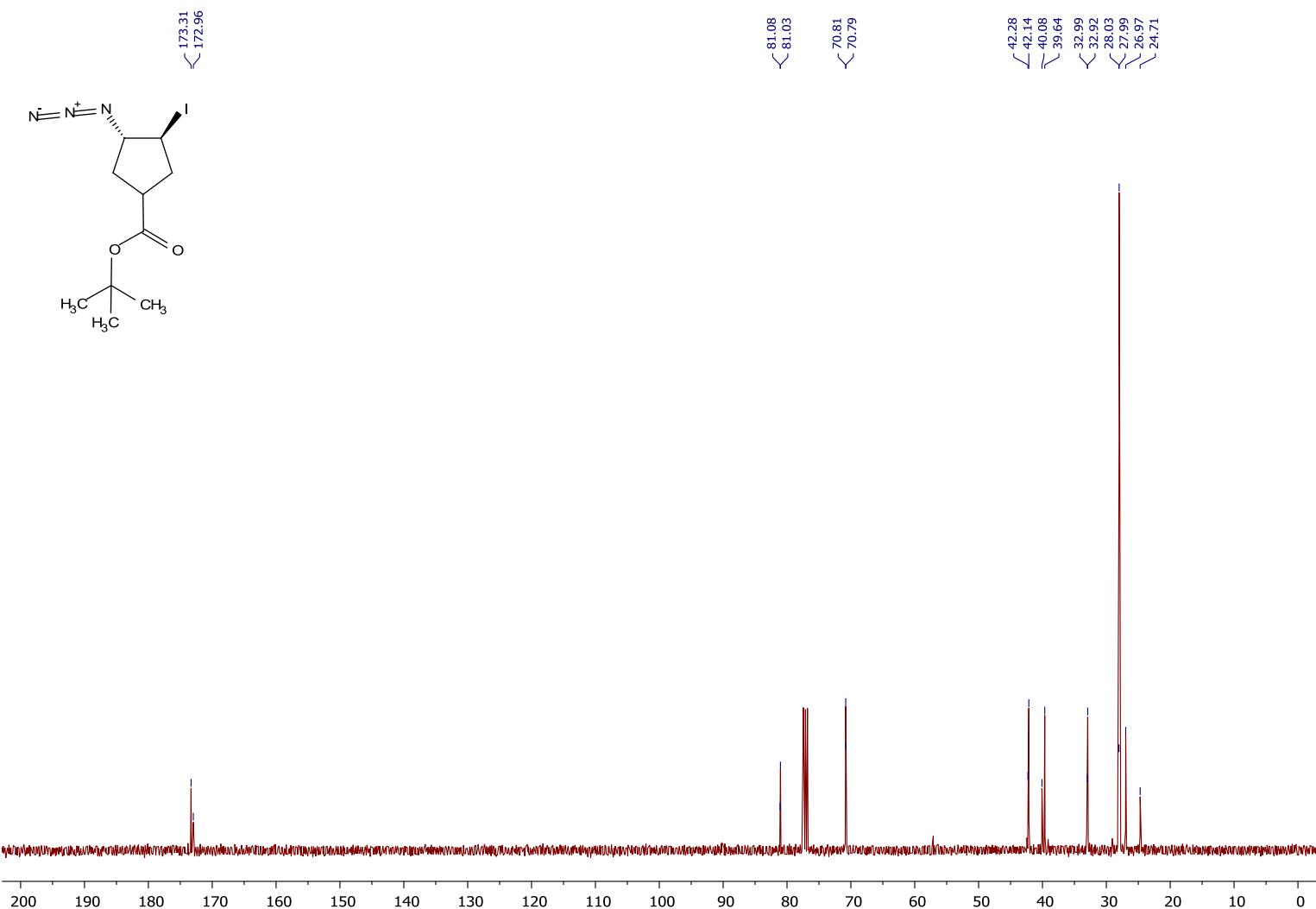
(3S*,4S*)-tert-Butyl 3,4-dibromocyclopentanecarboxylate (8e) ^1H NMR (CDCl_3)

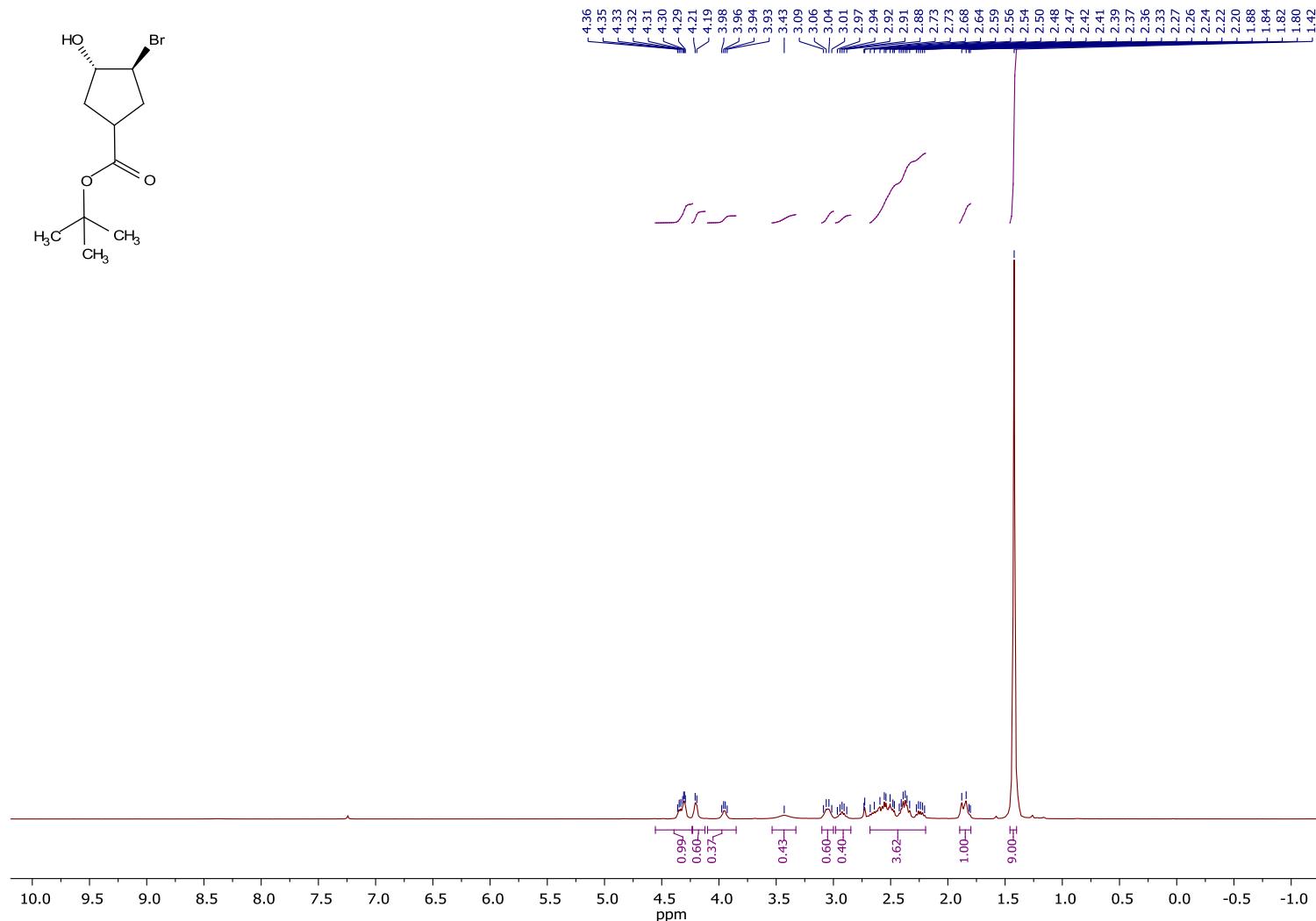
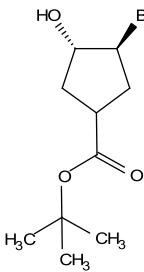


(*3S*,4S**)-*tert*-Butyl 3,4-dibromocyclopentanecarboxylate (8e) ^{13}C NMR (CDCl_3)

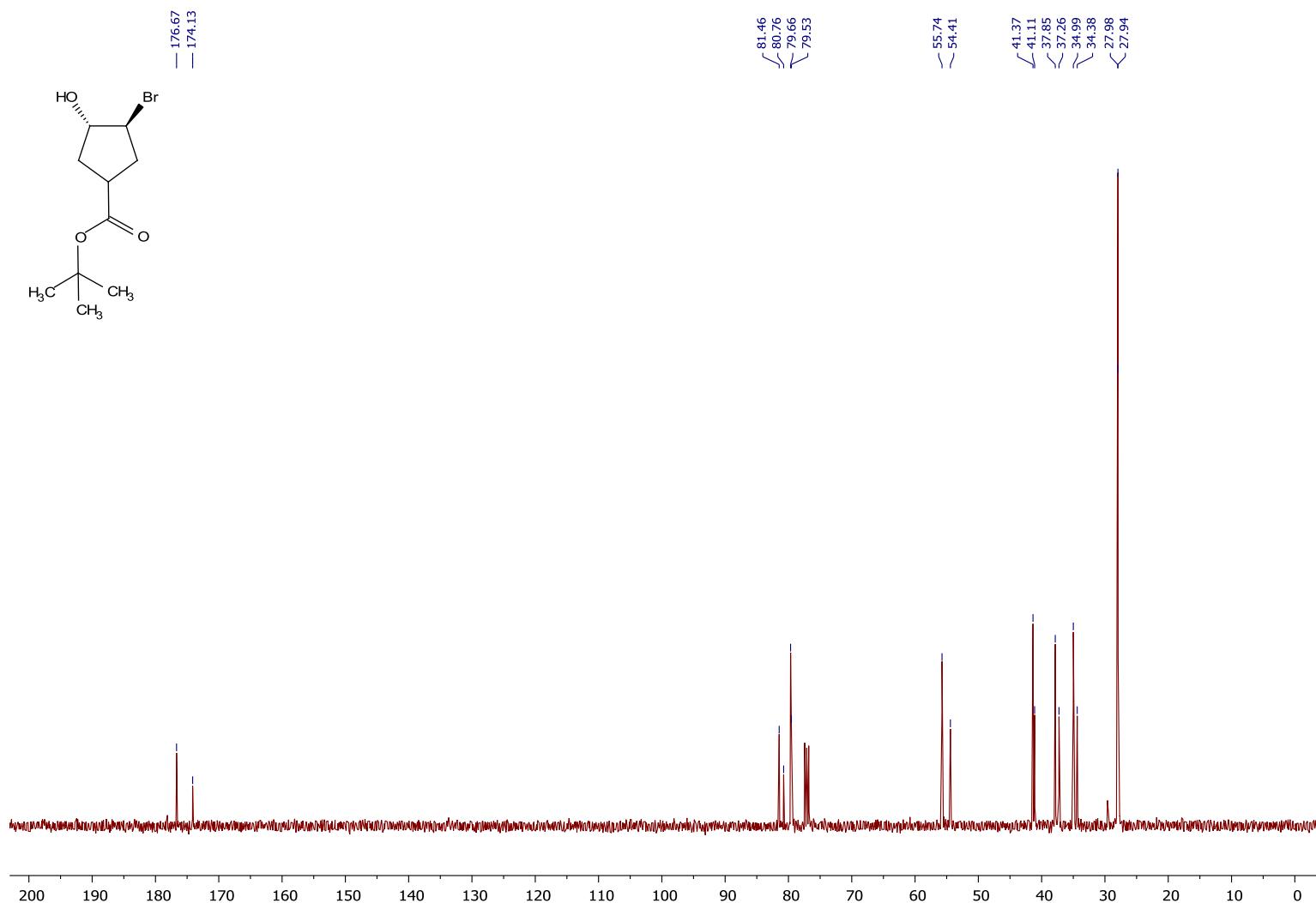


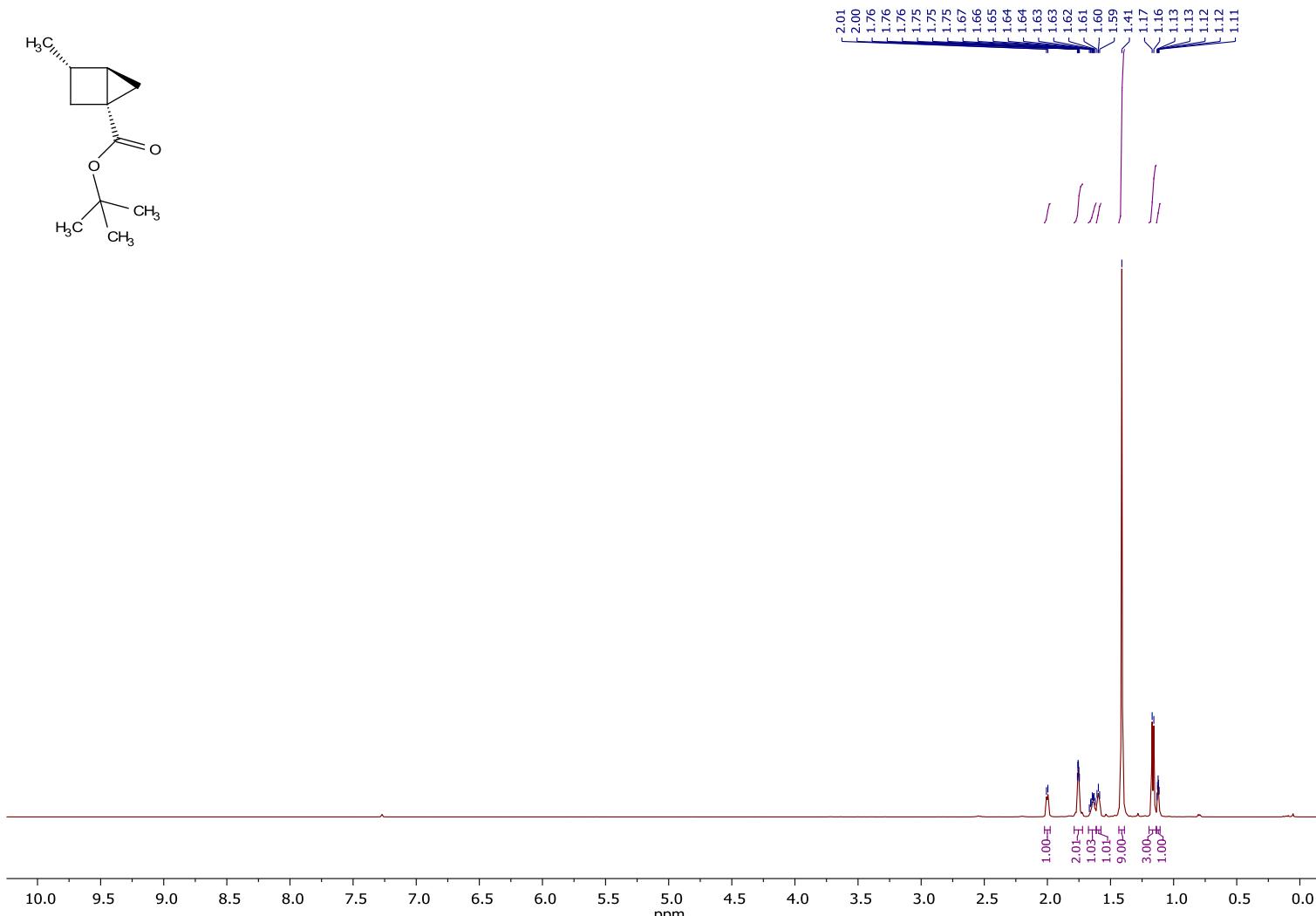
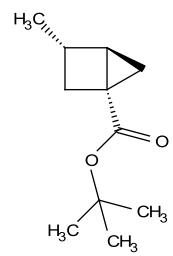
(3*S,4*S**)-tert-Butyl 3-azido-4-iodocyclopentanecarboxylate (8f) ^1H NMR (CDCl_3)**



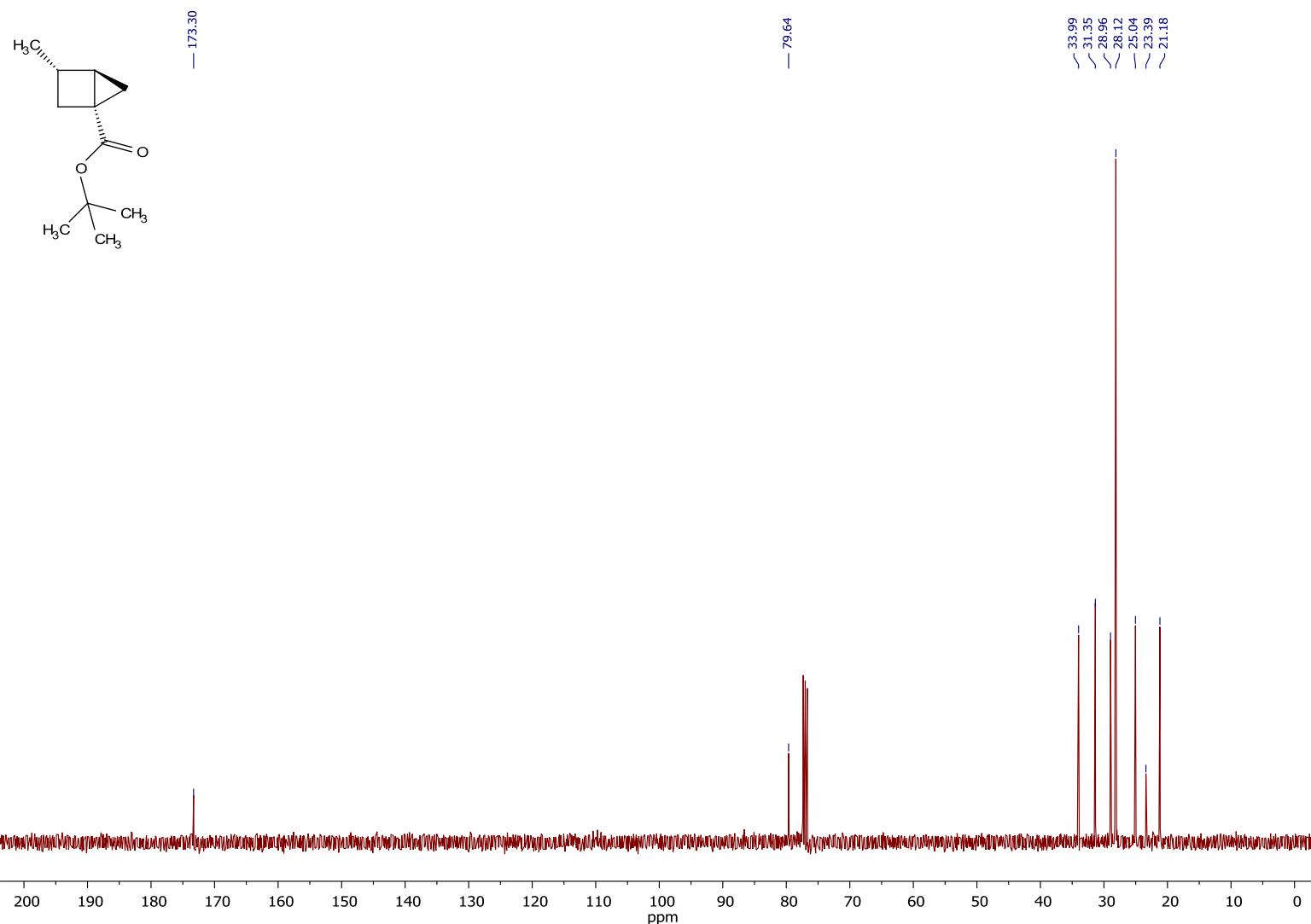


(3*S,4*S**)-*tert*-Butyl 3-bromo-4-hydroxycyclopentanecarboxylate (8g)** ^1H NMR (CDCl_3)

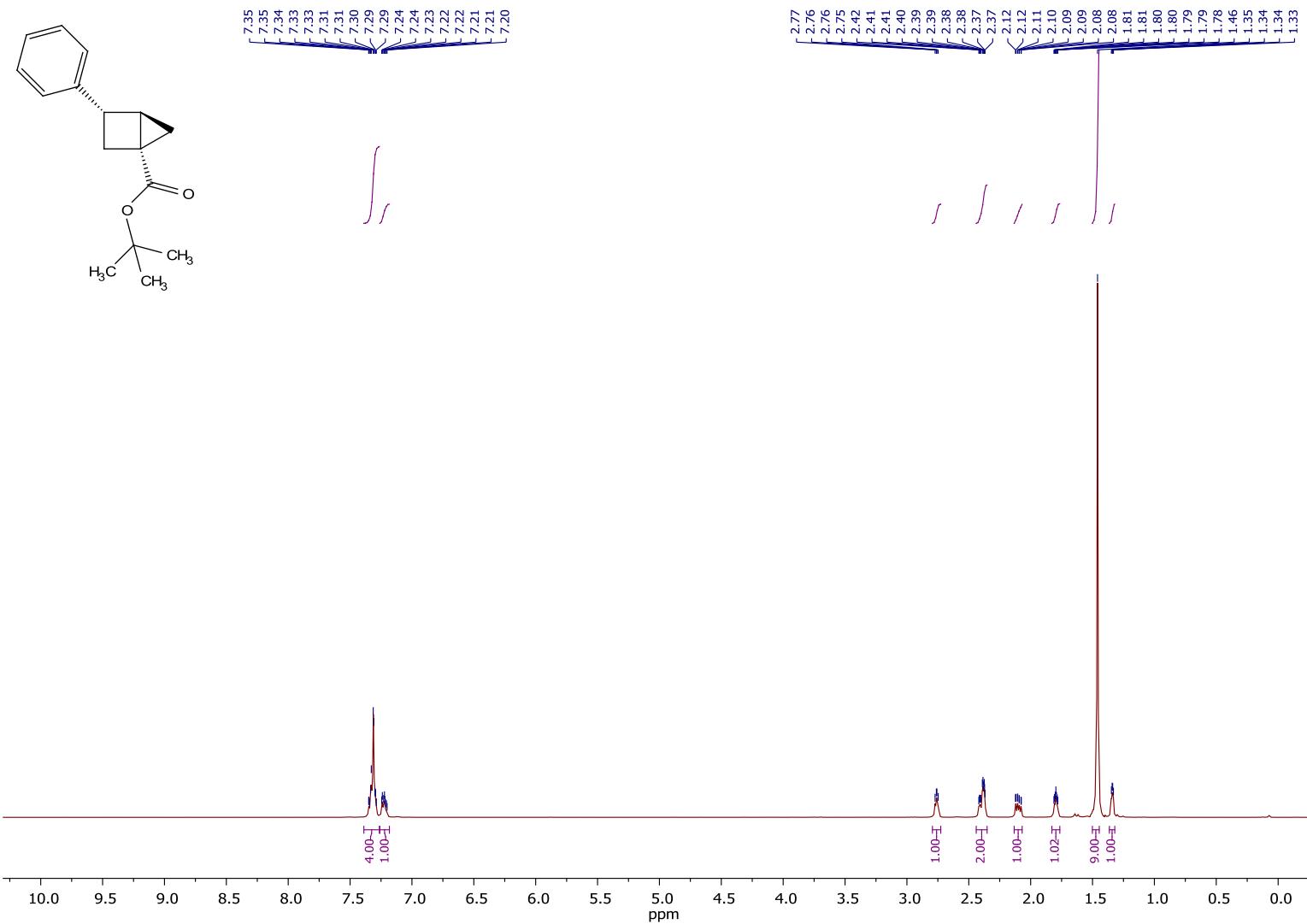


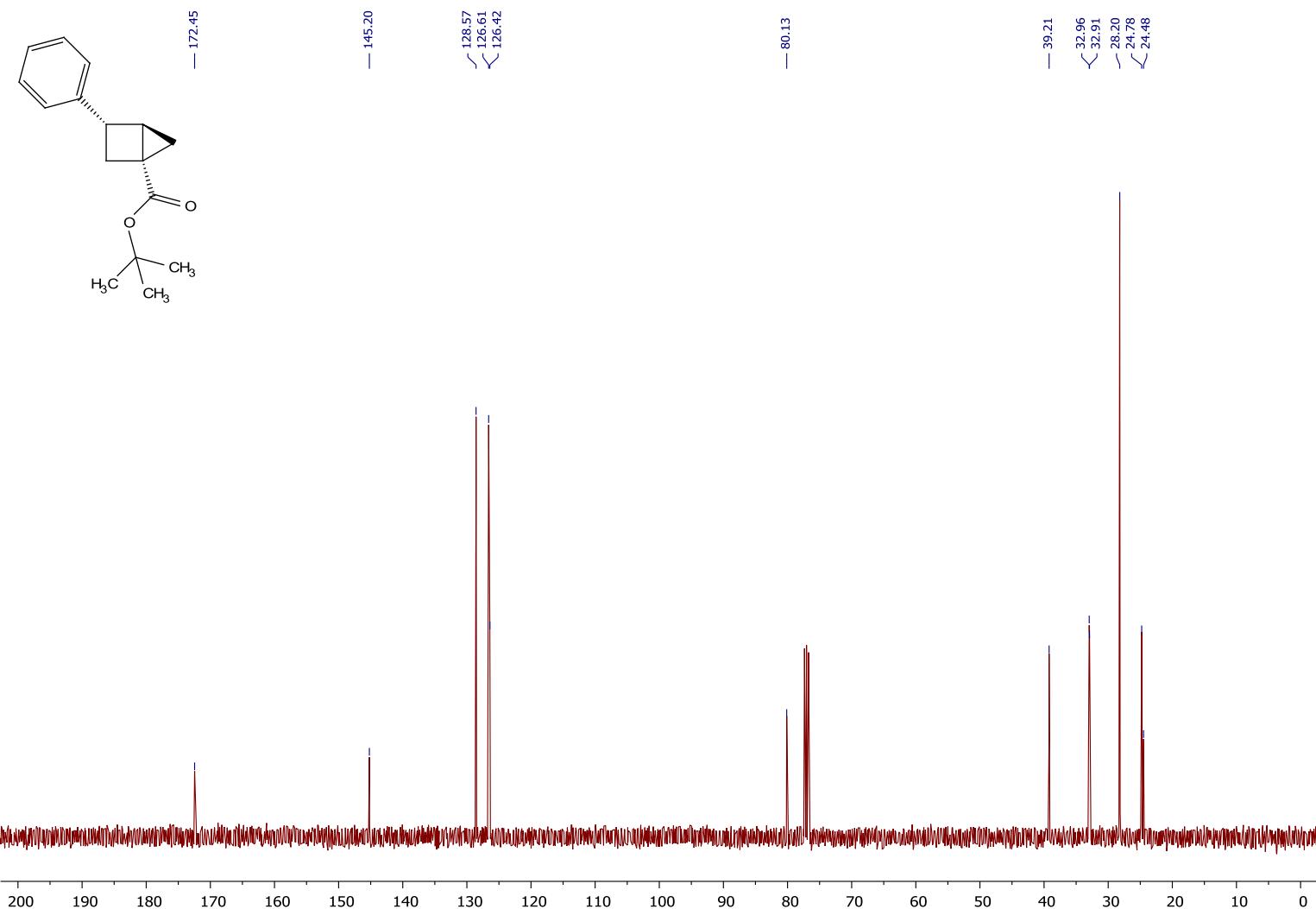


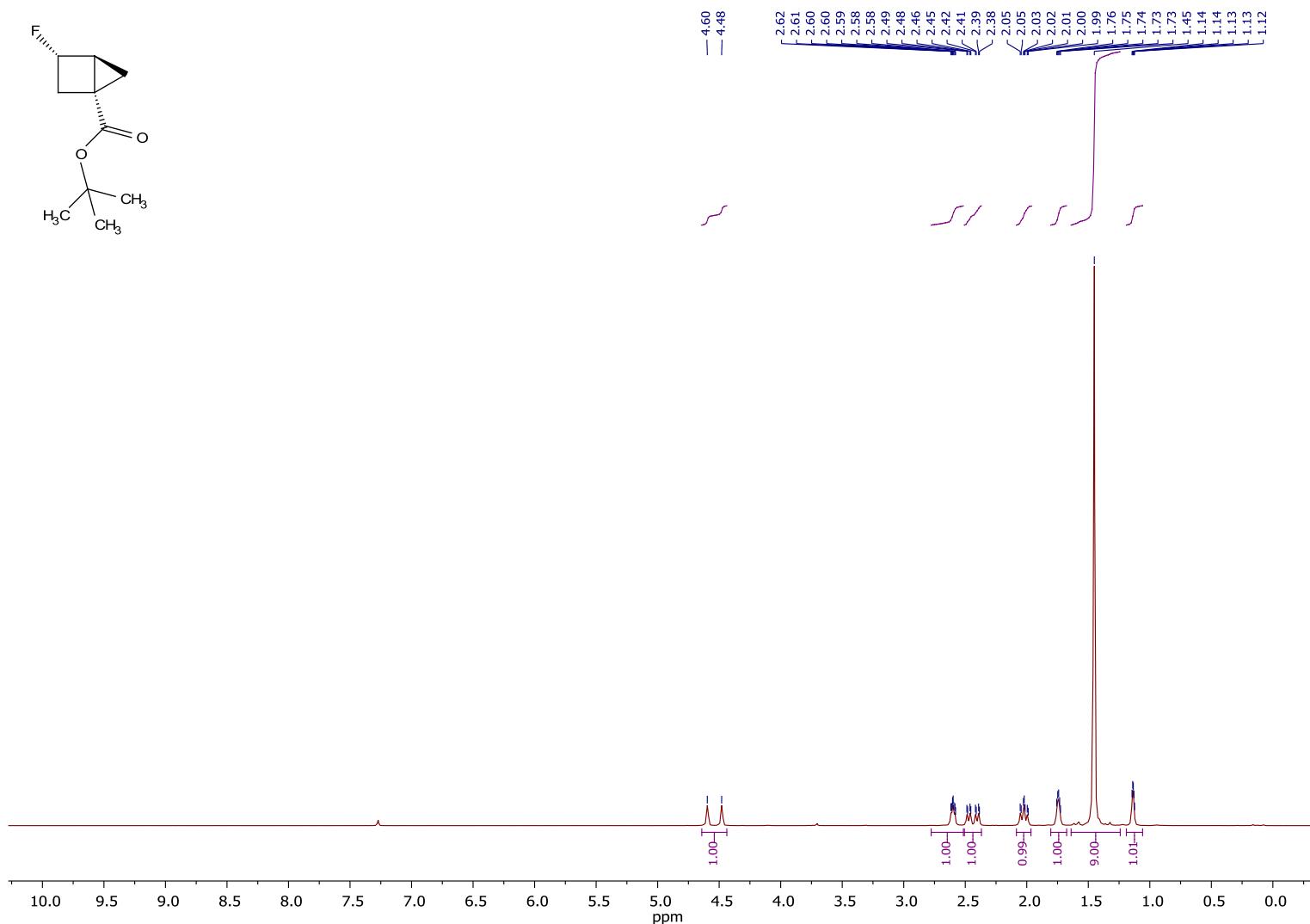
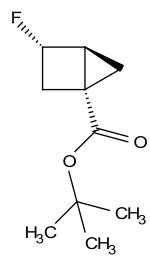
(1*R*^{*},3*S*^{*},4*R*^{*})-*tert*-Butyl 3-methylbicyclo[2.1.0]pentane-1-carboxylate (9a) ¹H NMR (CDCl_3)



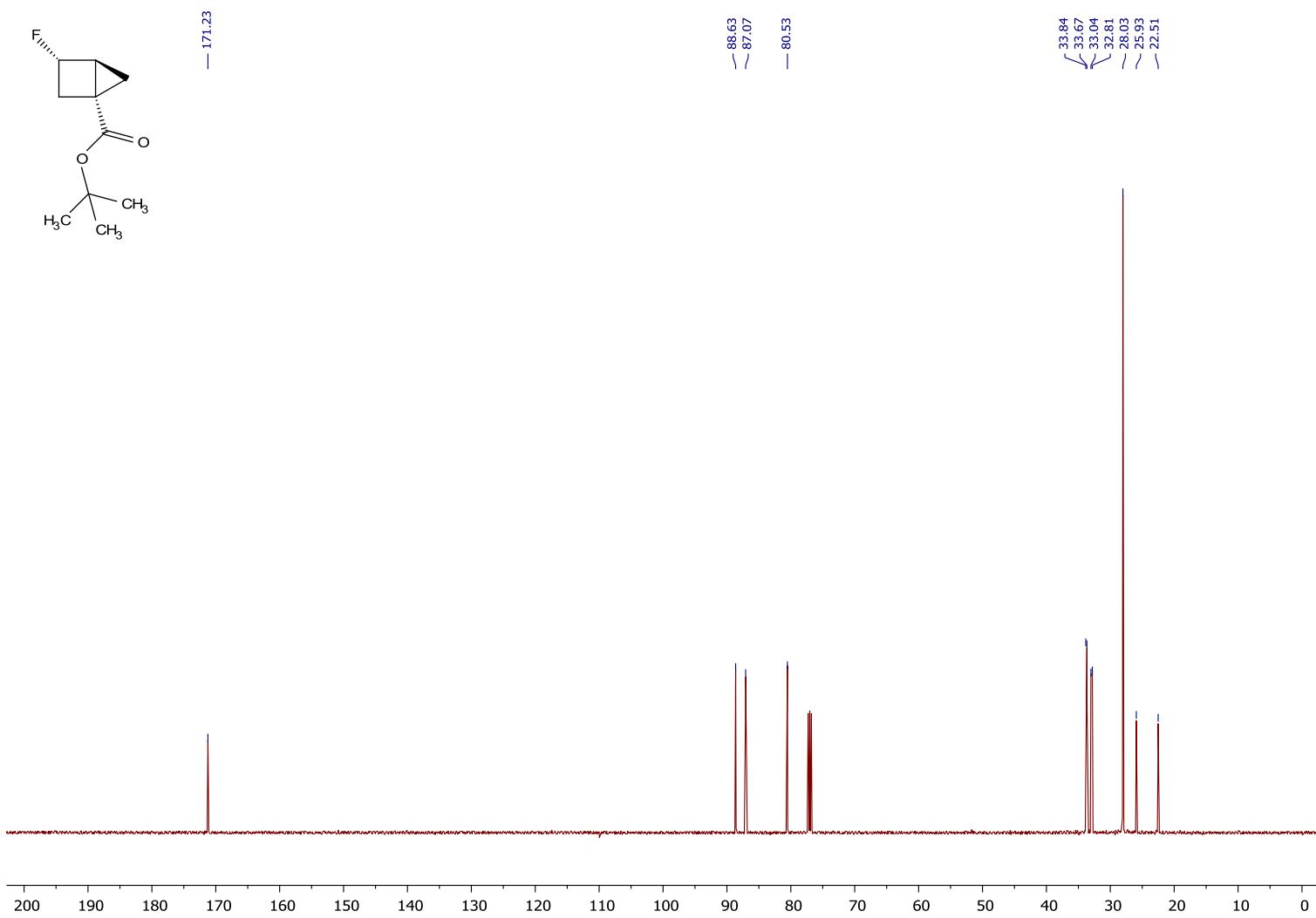
(1*R*^{*,3*S*^{*,4*R*^{*}}}-tert-Butyl 3-methylbicyclo[2.1.0]pentane-1-carboxylate (**9a**) ^{13}C NMR (CDCl_3)



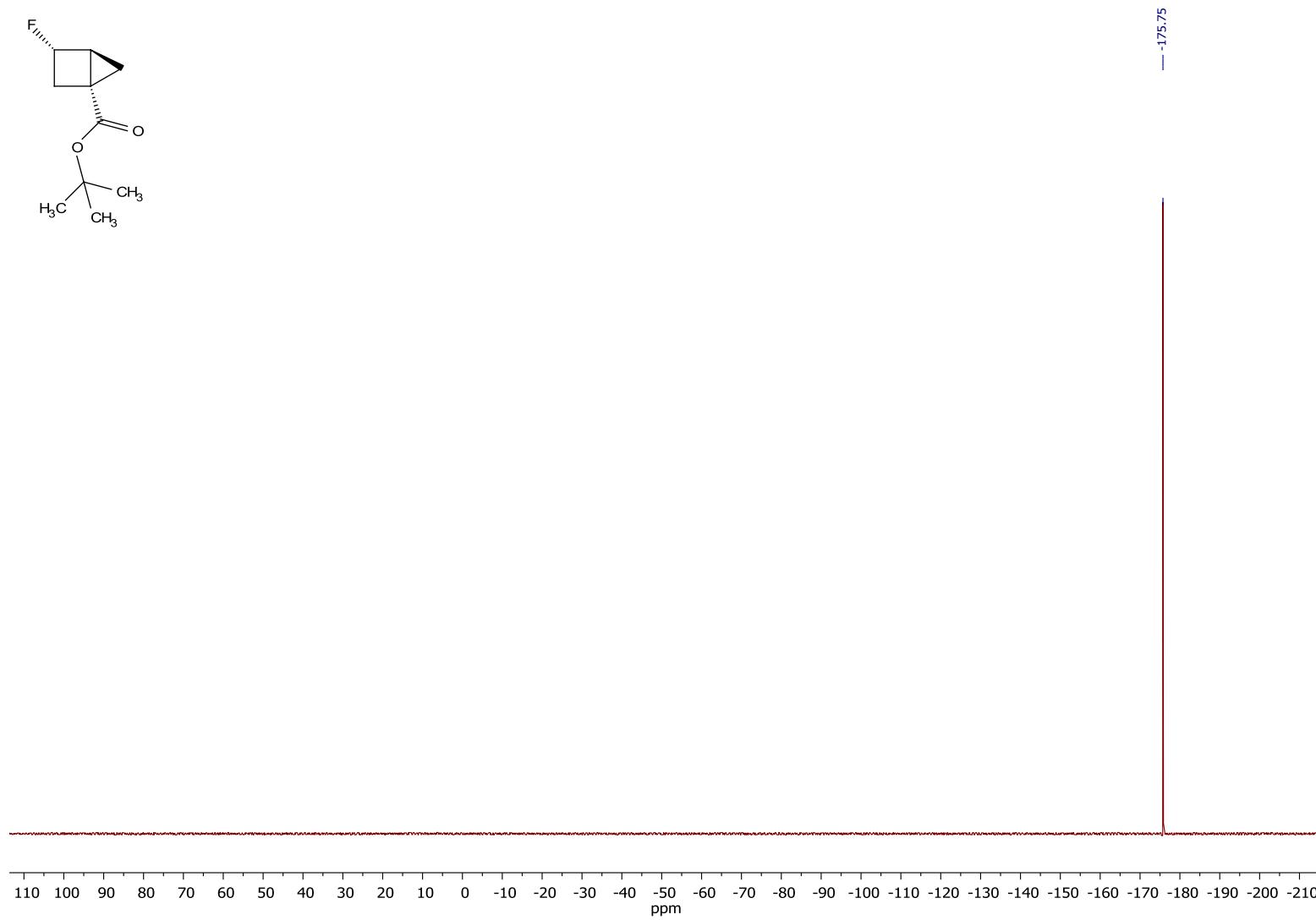
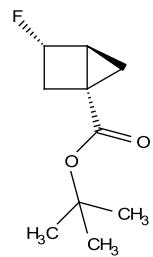




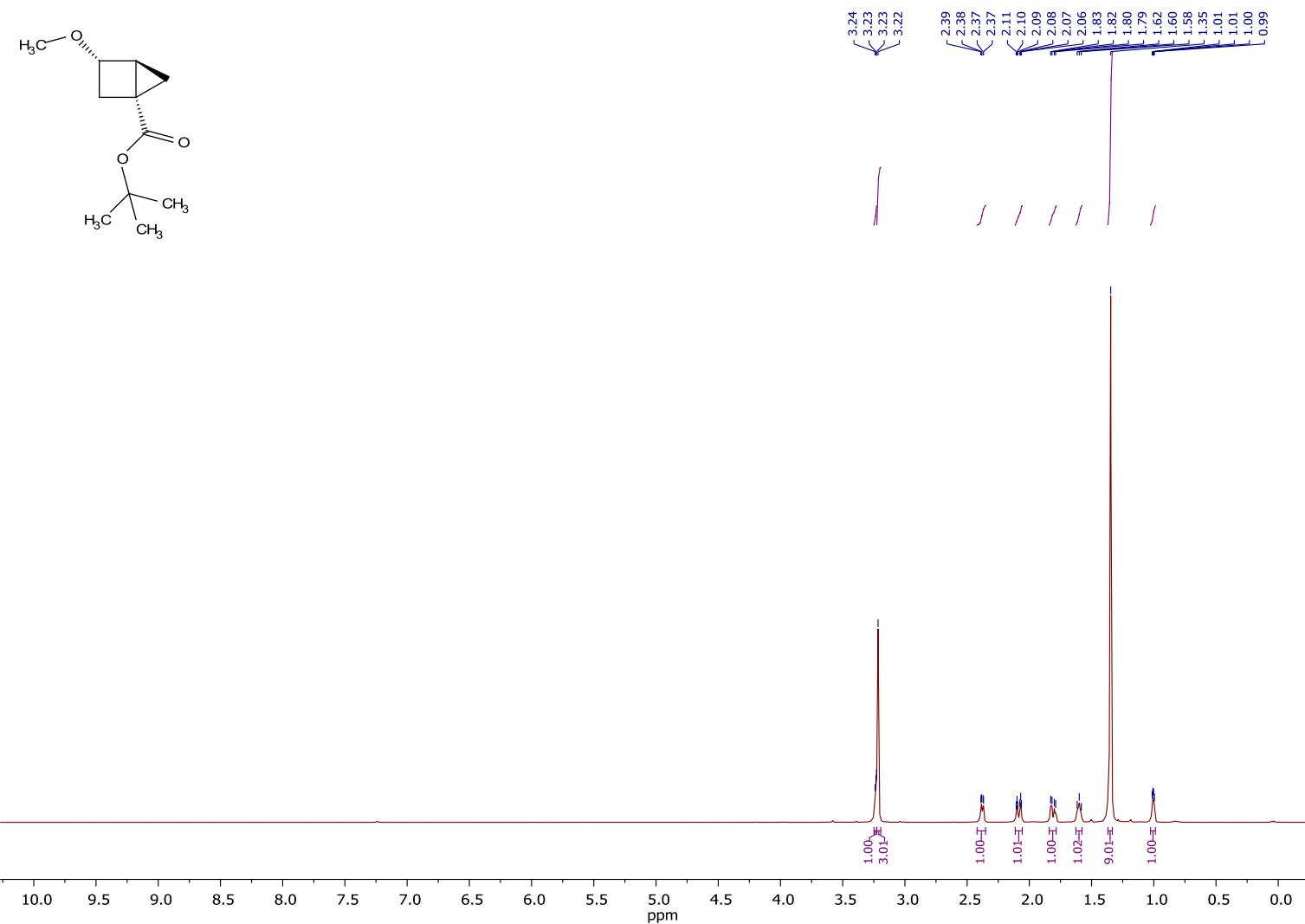
(1*R*^{*},3*S*^{*},4*S*^{*})-tert-Butyl 3-fluorobicyclo[2.1.0]pentane-1-carboxylate (9c) ^1H NMR (CDCl_3)



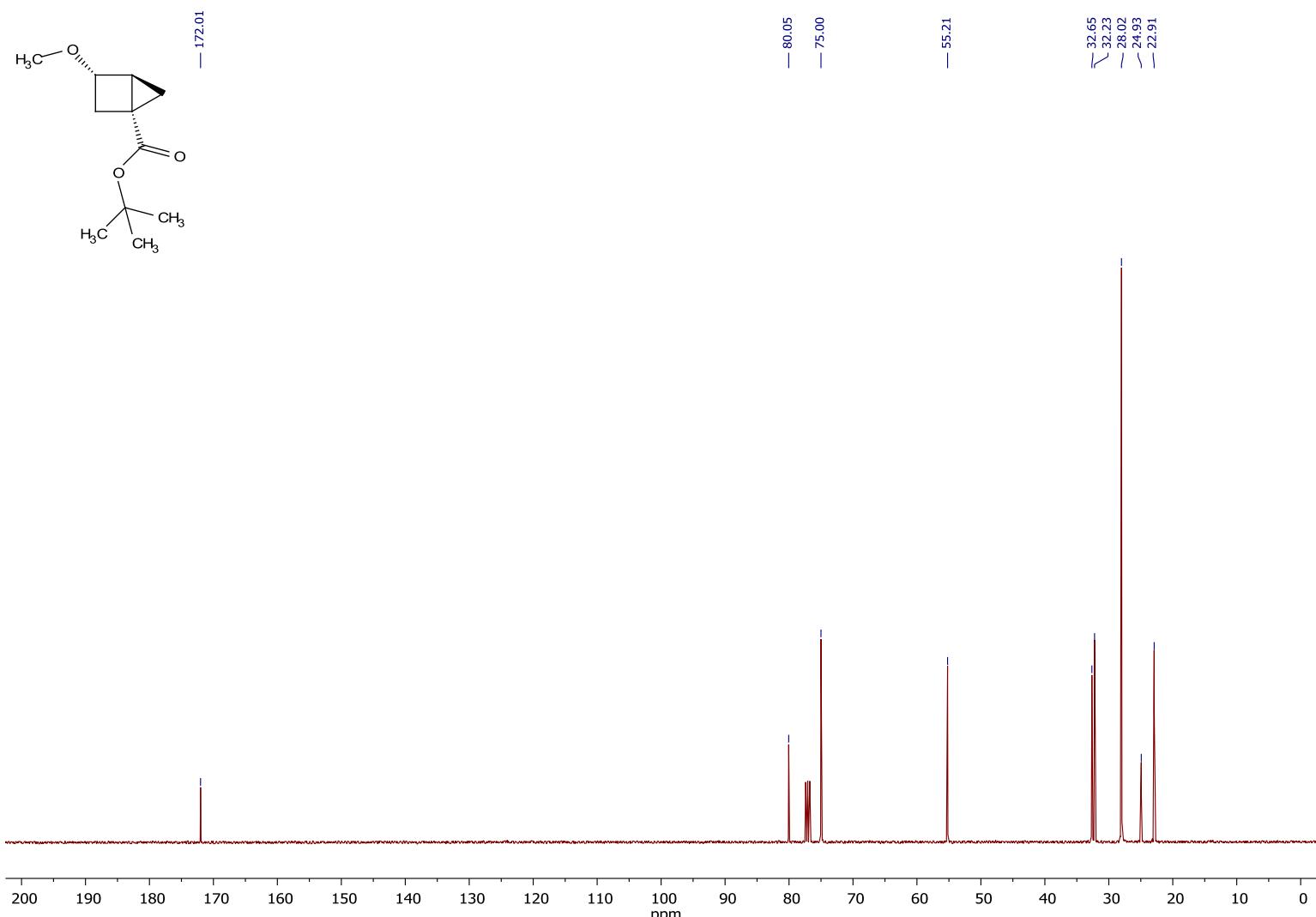
(1*R*^{*,3*S*^{*,4*S*^{*}}}-tert-Butyl 3-fluorobicyclo[2.1.0]pentane-1-carboxylate (9c) ^{13}C NMR (CDCl₃)



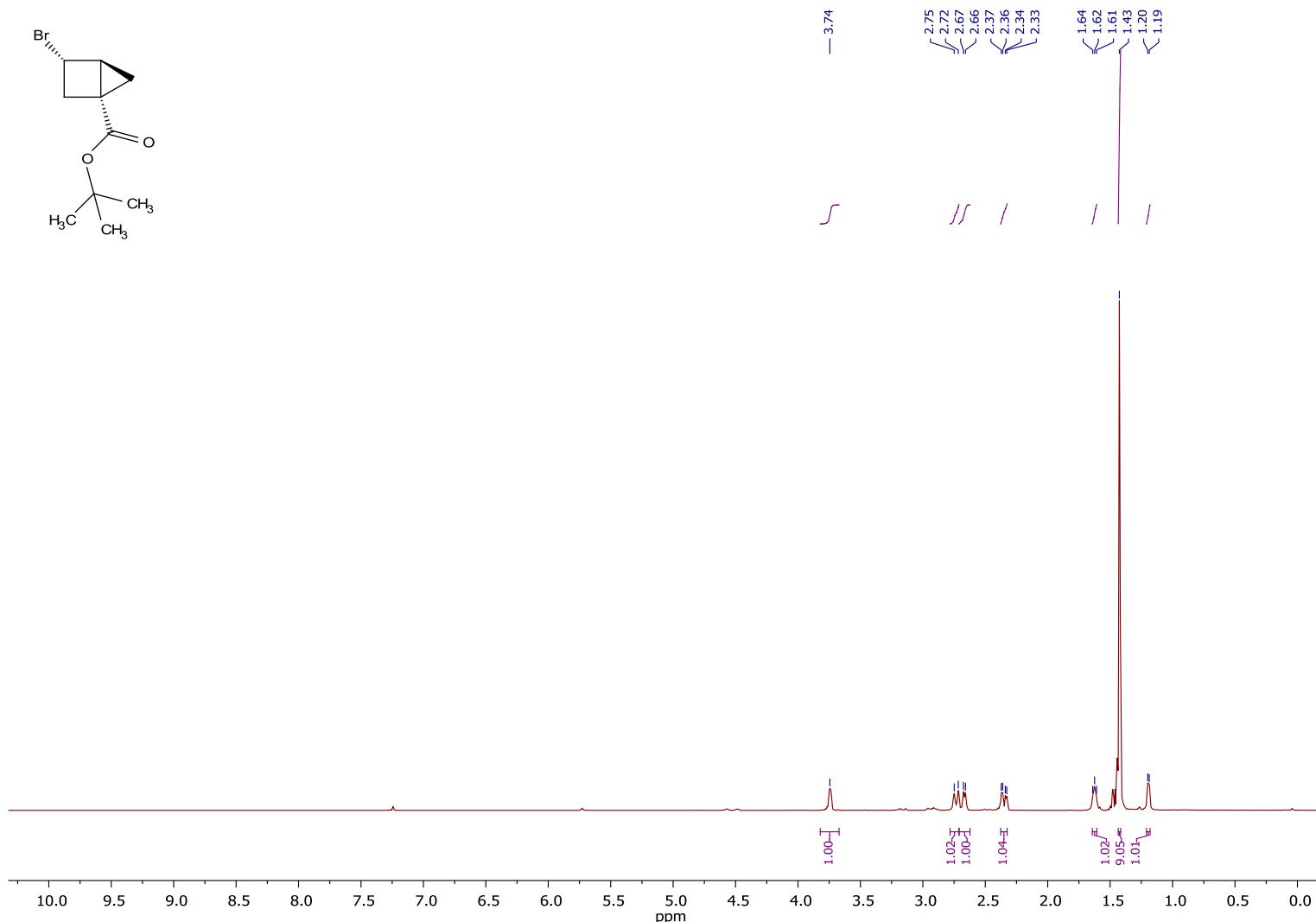
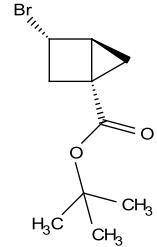
(1*R*^{*},3*S*^{*},4*S*^{*})-*tert*-Butyl 3-fluorobicyclo[2.1.0]pentane-1-carboxylate (9c) ^{19}F NMR (CDCl_3)



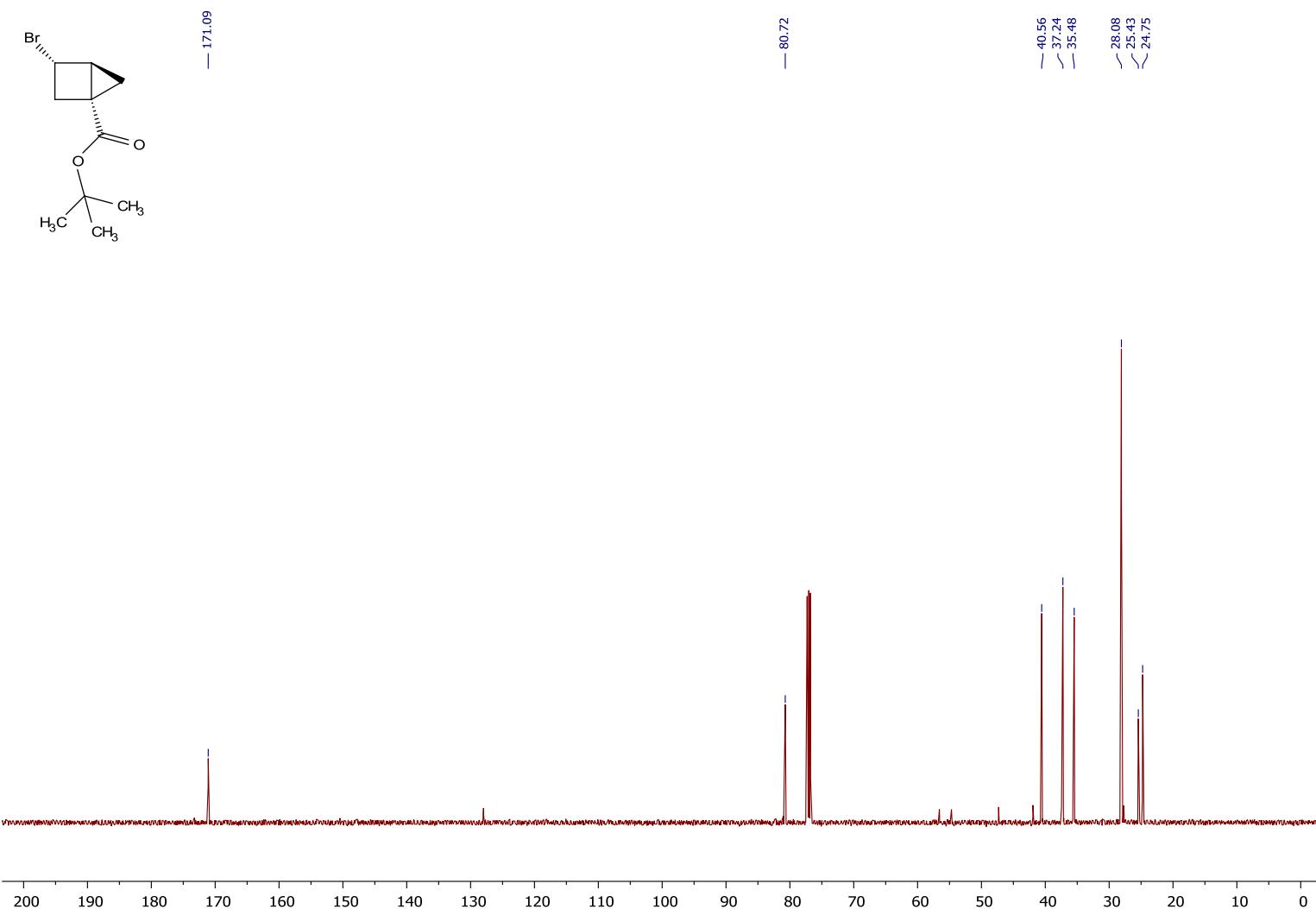
(1*R,3*S**,4*S**)-*tert*-Butyl 3-methoxybicyclo[2.1.0]pentane-1-carboxylate (9d) ^1H NMR (CDCl_3)**



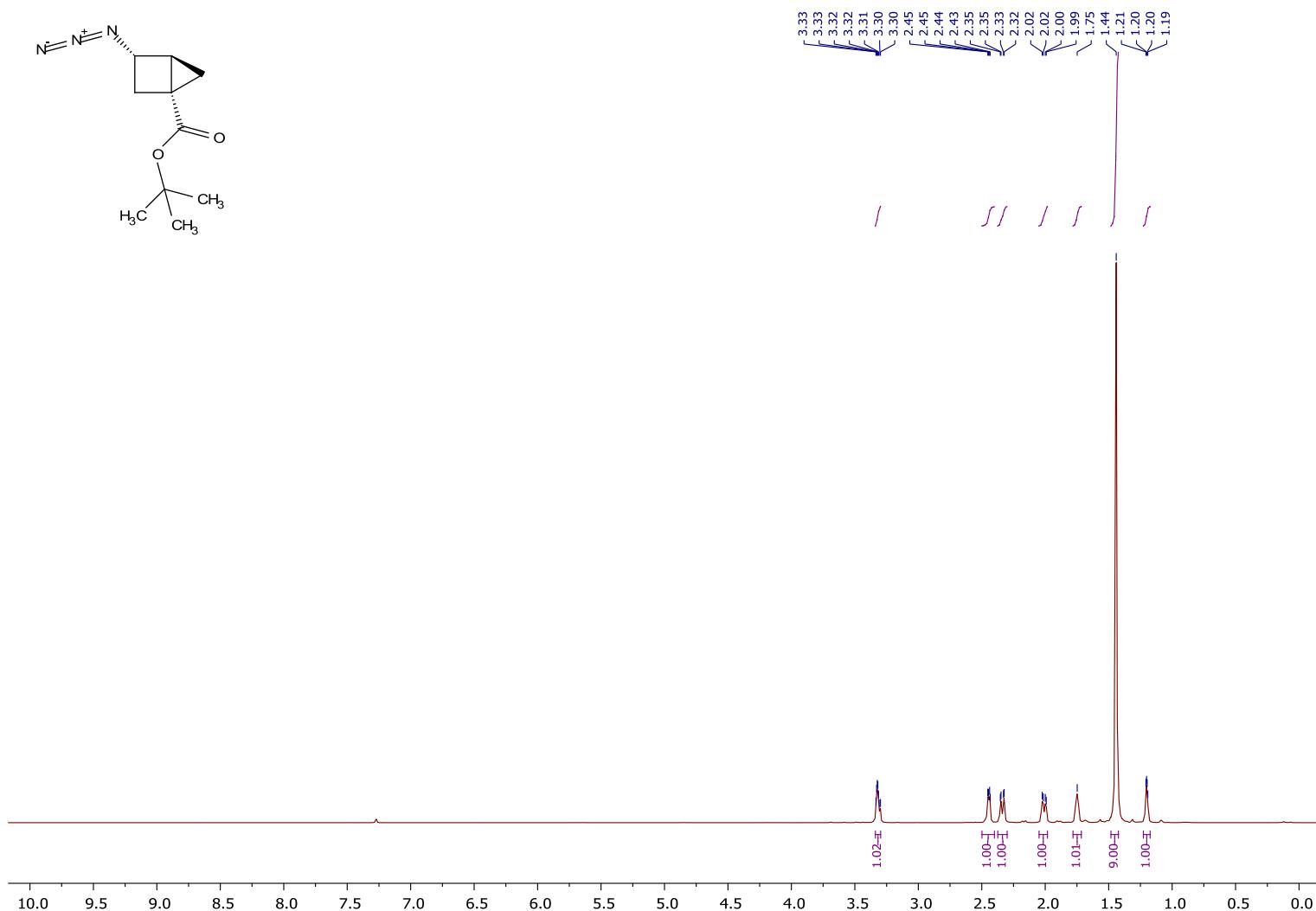
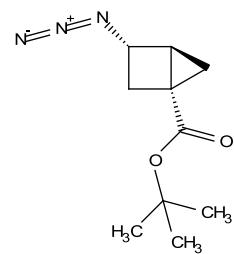
(*1R*,3S*,4S**)-*tert*-Butyl 3-methoxybicyclo[2.1.0]pentane-1-carboxylate (**9d**) ^{13}C NMR (CDCl_3)



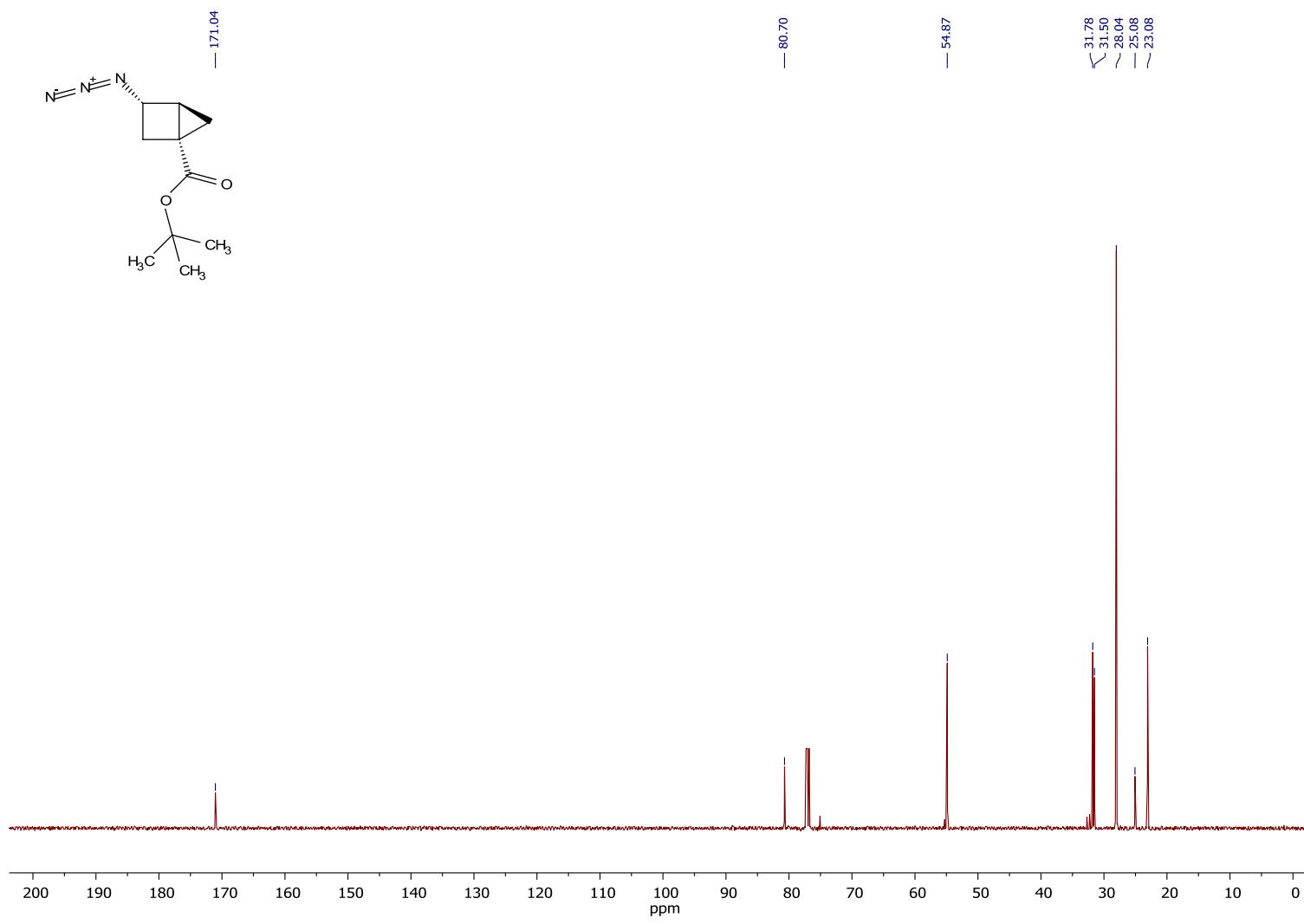
(1*R*^{*},3*S*^{*},4*S*^{*})-*tert*-Butyl 3-bromobicyclo[2.1.0]pentane-1-carboxylate (9e) ¹H NMR (CDCl₃)



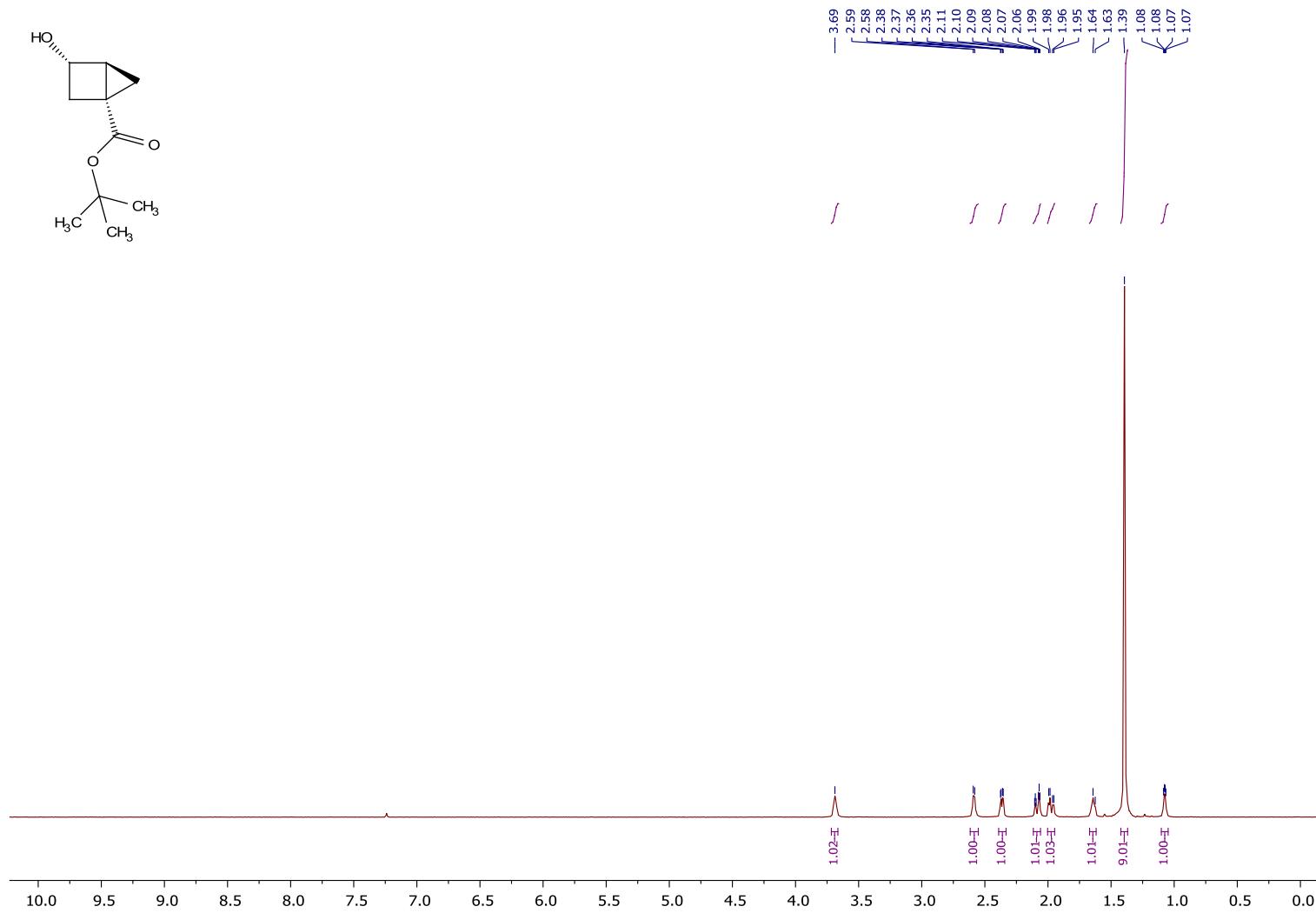
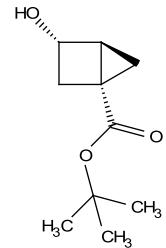
(1*R,3*S**,4*S**)-tert-Butyl 3-bromobicyclo[2.1.0]pentane-1-carboxylate (9e) ^{13}C NMR (CDCl_3)**



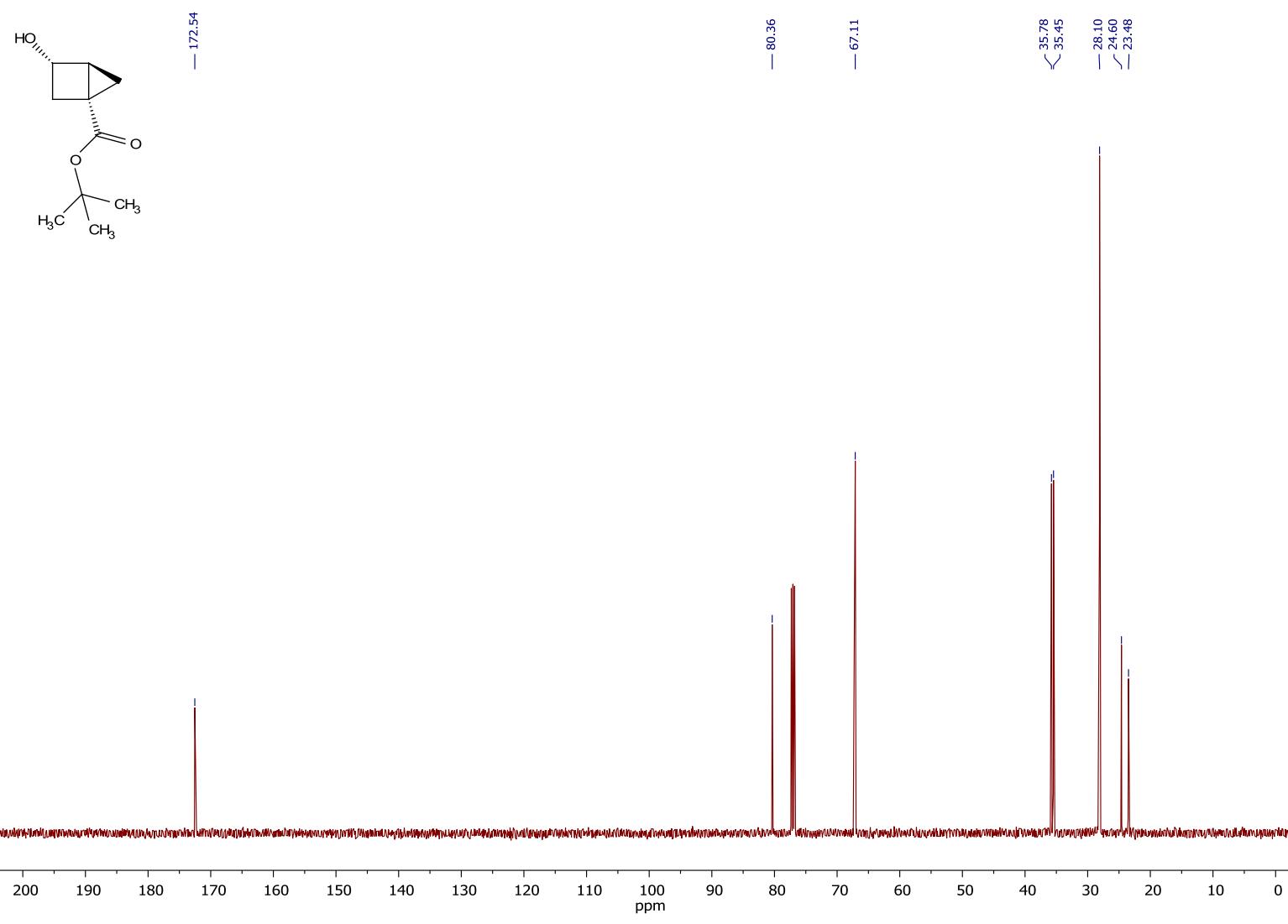
(1R*,3S*,4S*)-tert-Butyl 3-azidobicyclo[2.1.0]pentane-1-carboxylate (9f) ¹H NMR (CDCl₃)



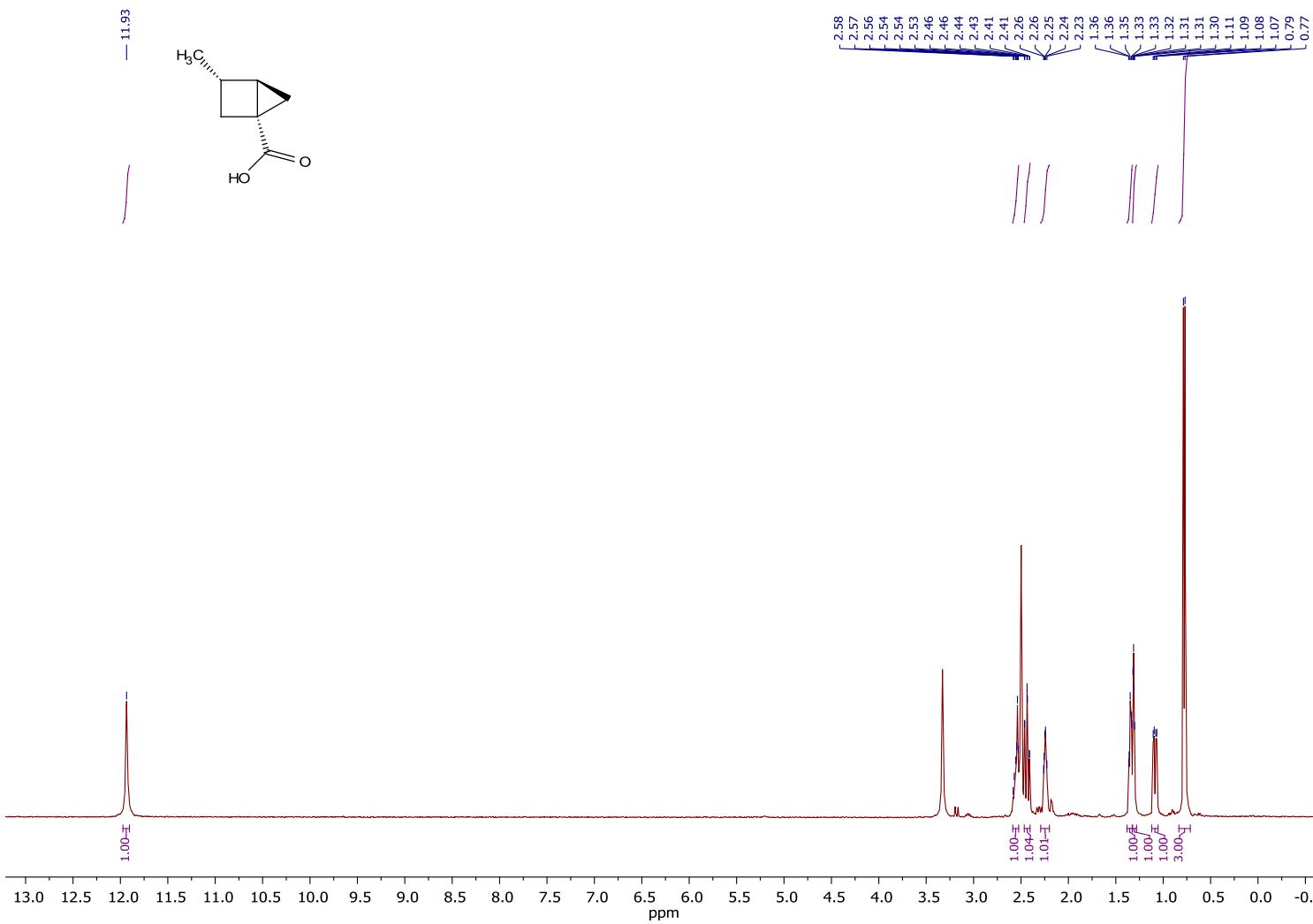
(1*R*^{*,3*S*^{*,4*S*^{*}})-*tert*-Butyl 3-azidobicyclo[2.1.0]pentane-1-carboxylate (9f) ^{13}C NMR (CDCl_3)}



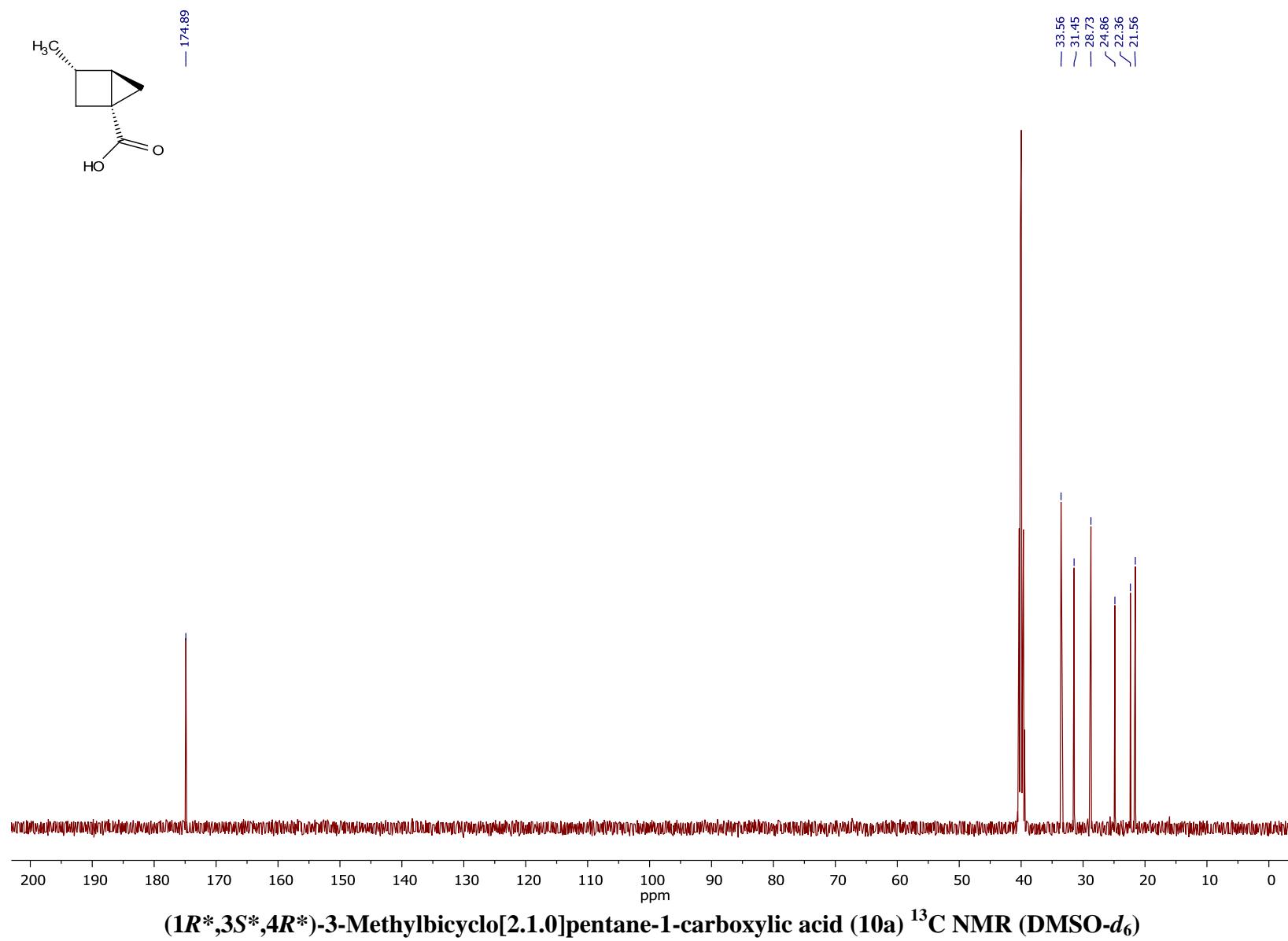
(1*R*^{*},3*S*^{*},4*S*^{*})-*tert*-Butyl 3-hydroxybicyclo[2.1.0]pentane-1-carboxylate (9g) ¹H NMR (CDCl_3)



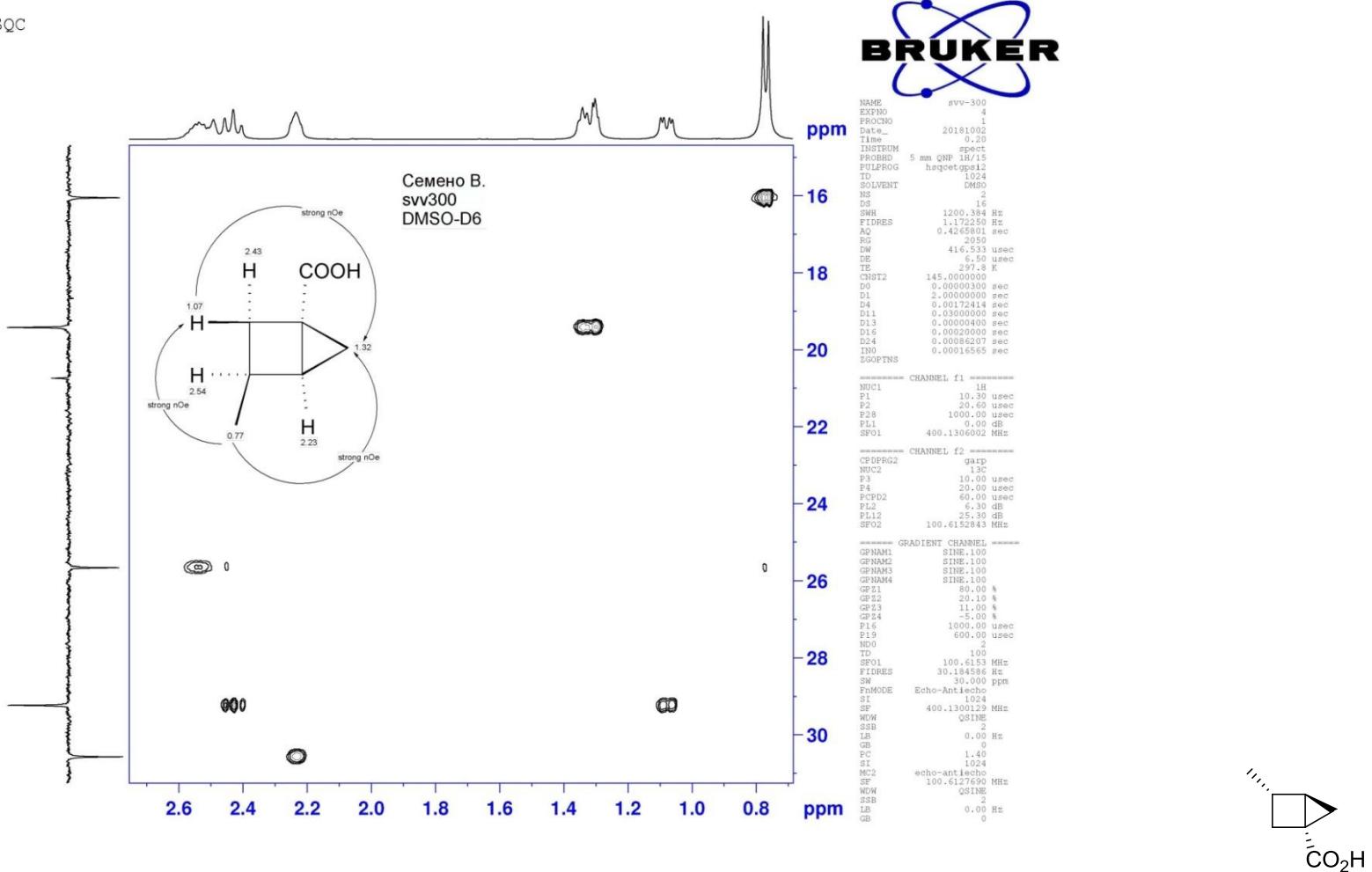
(*1R*,3S*,4S**)-*tert*-Butyl 3-hydroxybicyclo[2.1.0]pentane-1-carboxylate (**9g**) ^{13}C NMR (CDCl_3)



(1*R*^{*},3*S*^{*},4*R*^{*})-3-Methylbicyclo[2.1.0]pentane-1-carboxylic acid (10a) ^{1}H NMR (DMSO- d_6)

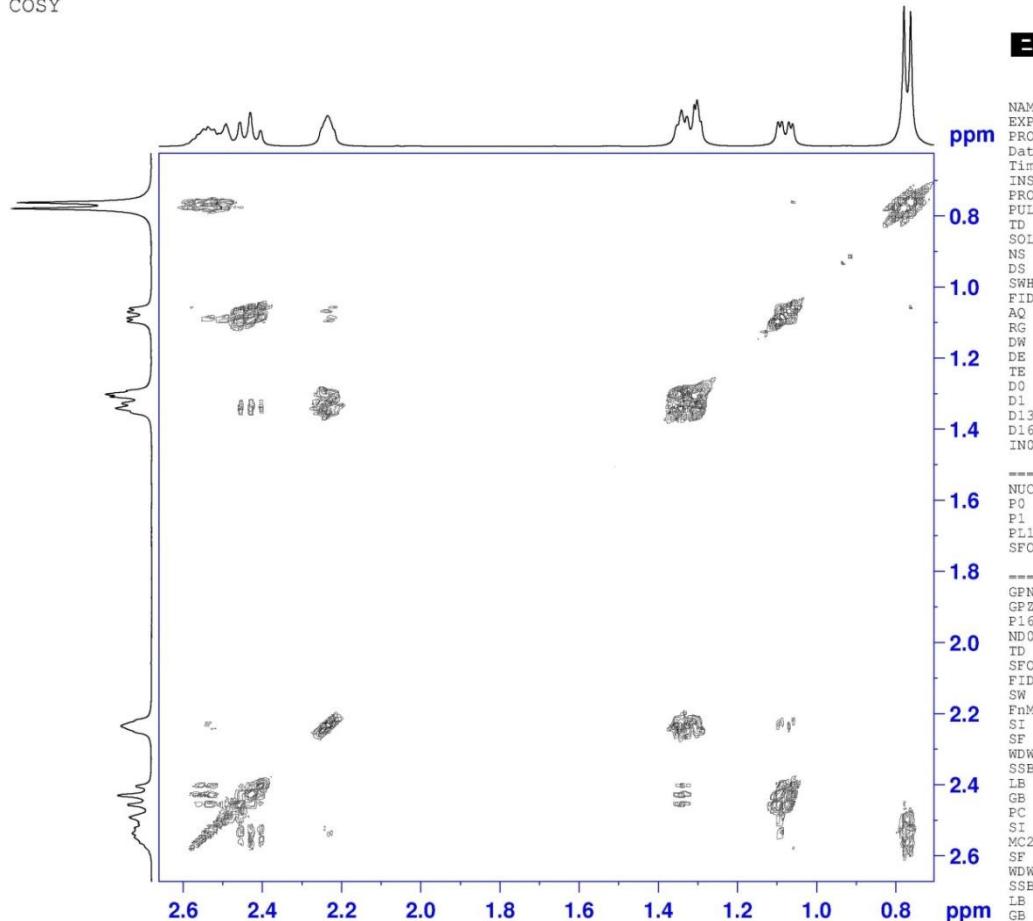


HSQC



(1*R**,3*S**,4*R**)-3-Methylbicyclo[2.1.0]pentane-1-carboxylic acid (10a) (HSQC) (DMSO-*d*₆)

COSY



```

NAME          svv-300
EXPNO         3
PROCNO        1
Date_        20181002
Time       0.16
INSTRUM      spect
PROBHD      5 mm QNP 1H/15
PULPROG     cosyppgf
TD           2048
SOLVENT      DMSO
NS            1
DS            8
SWH          1200.384 Hz
FIDRES      0.586125 Hz
AQ           0.8531103 sec
RG            512
DW           416.533 usec
DE           6.50 usec
TE           297.8 K
D0           0.00000300 sec
D1           1.48689198 sec
D13          0.00000400 sec
D16          0.00020000 sec
INO          0.00083305 sec

```

```

----- CHANNEL f1 -----
NUC1          1H
P0           5.15 usec
P1           10.30 usec
PL1          0.00 dB
SFO1        400.1306002 MHz

```

```

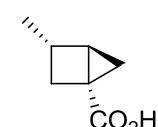
----- GRADIENT CHANNEL -----
GPNAME1      SINE.100
GPZ1         10.00 %
P16          1000.00 usec
ND0           1
TD           100
SFO1        400.1306 MHz
FIDRES      12.003918 Hz
SW           3.000 ppm

```

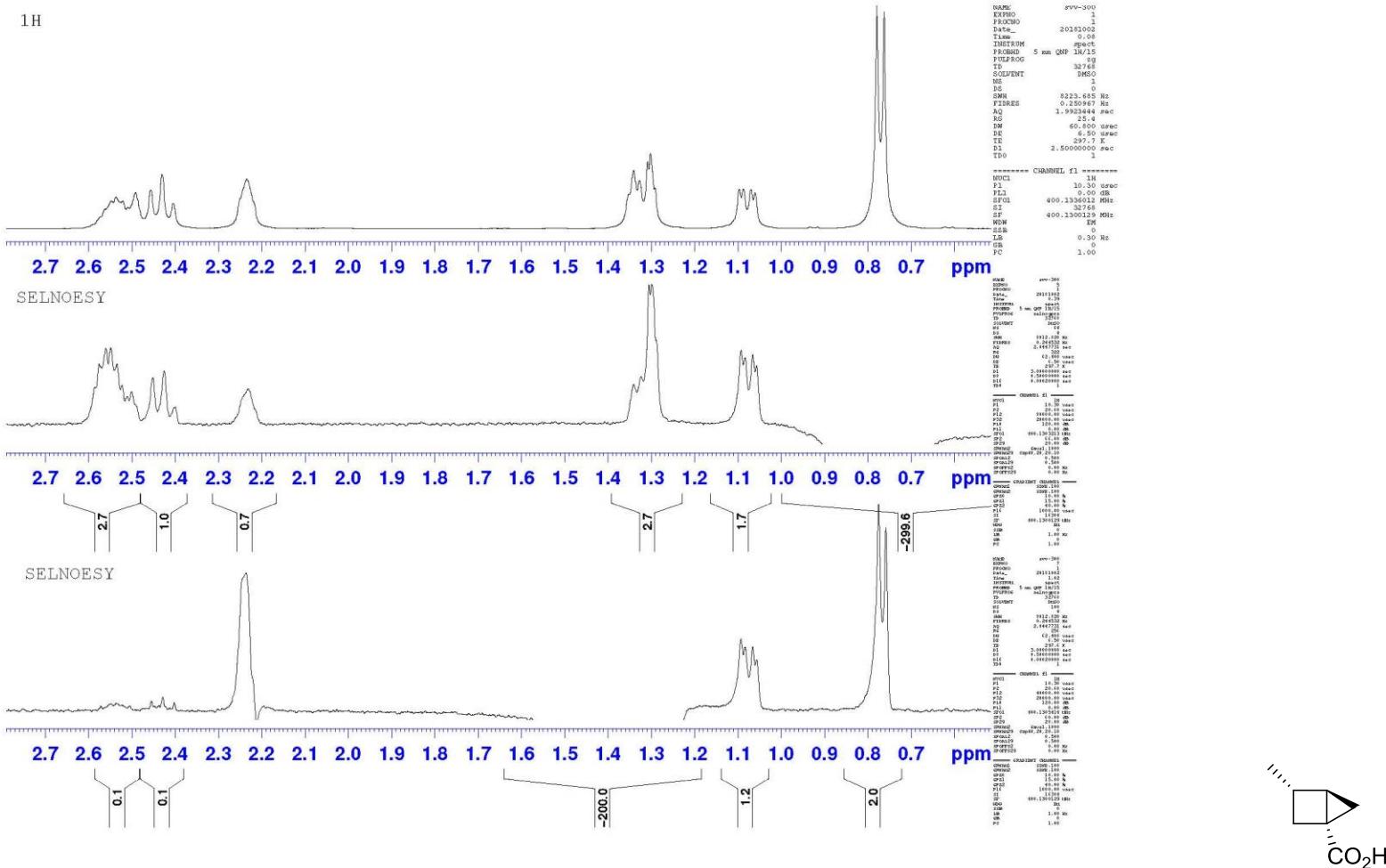
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FnMODE      QF
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SF          400.1300129 MHz
WDW          SINE
SSB           0
LB           0.00 Hz
GB           0
PC           1.40
SI            2048
MC2          QF
SF          400.1300129 MHz
WDW          SINE
SSB           0
LB           0.00 Hz
GB           0

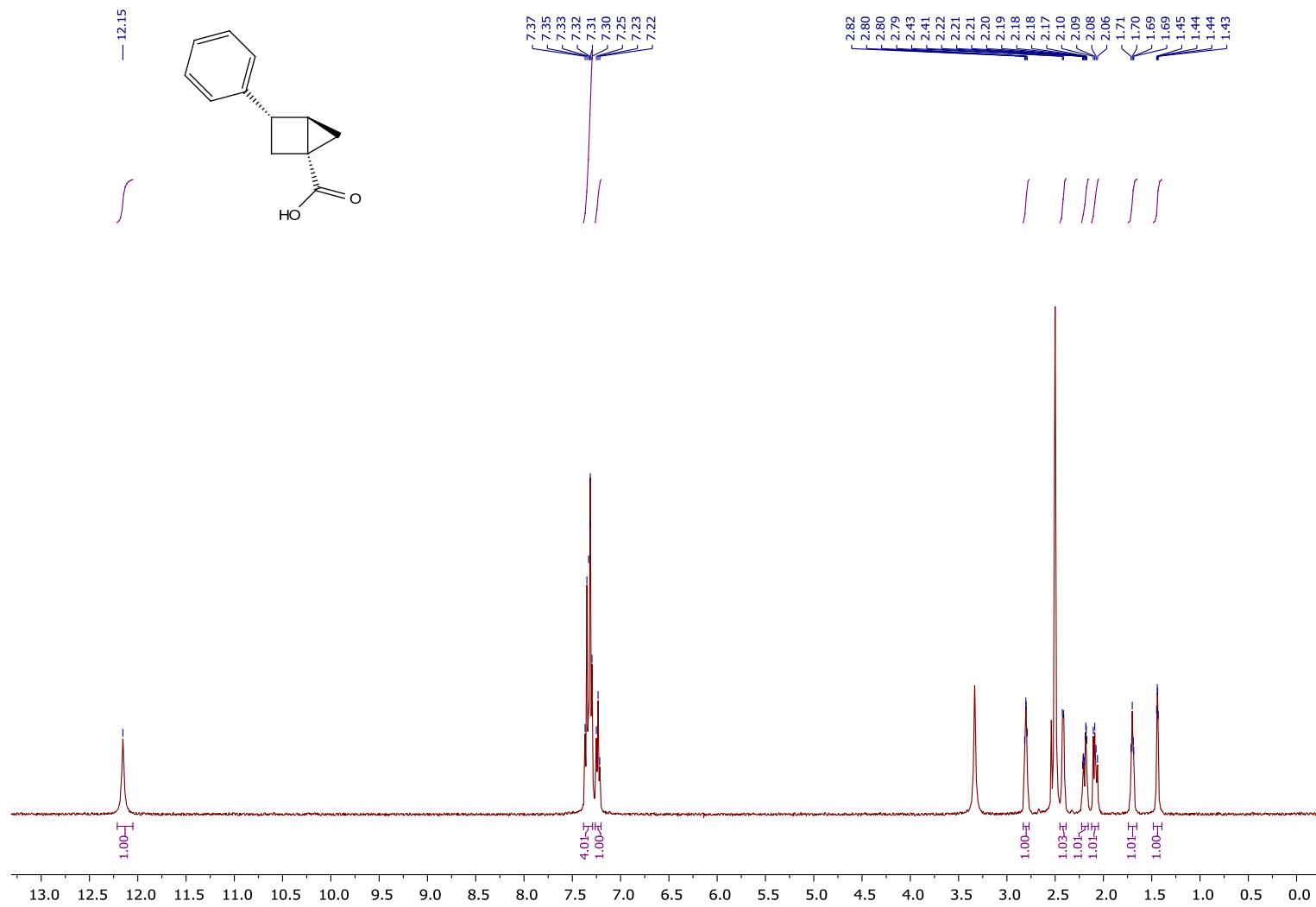
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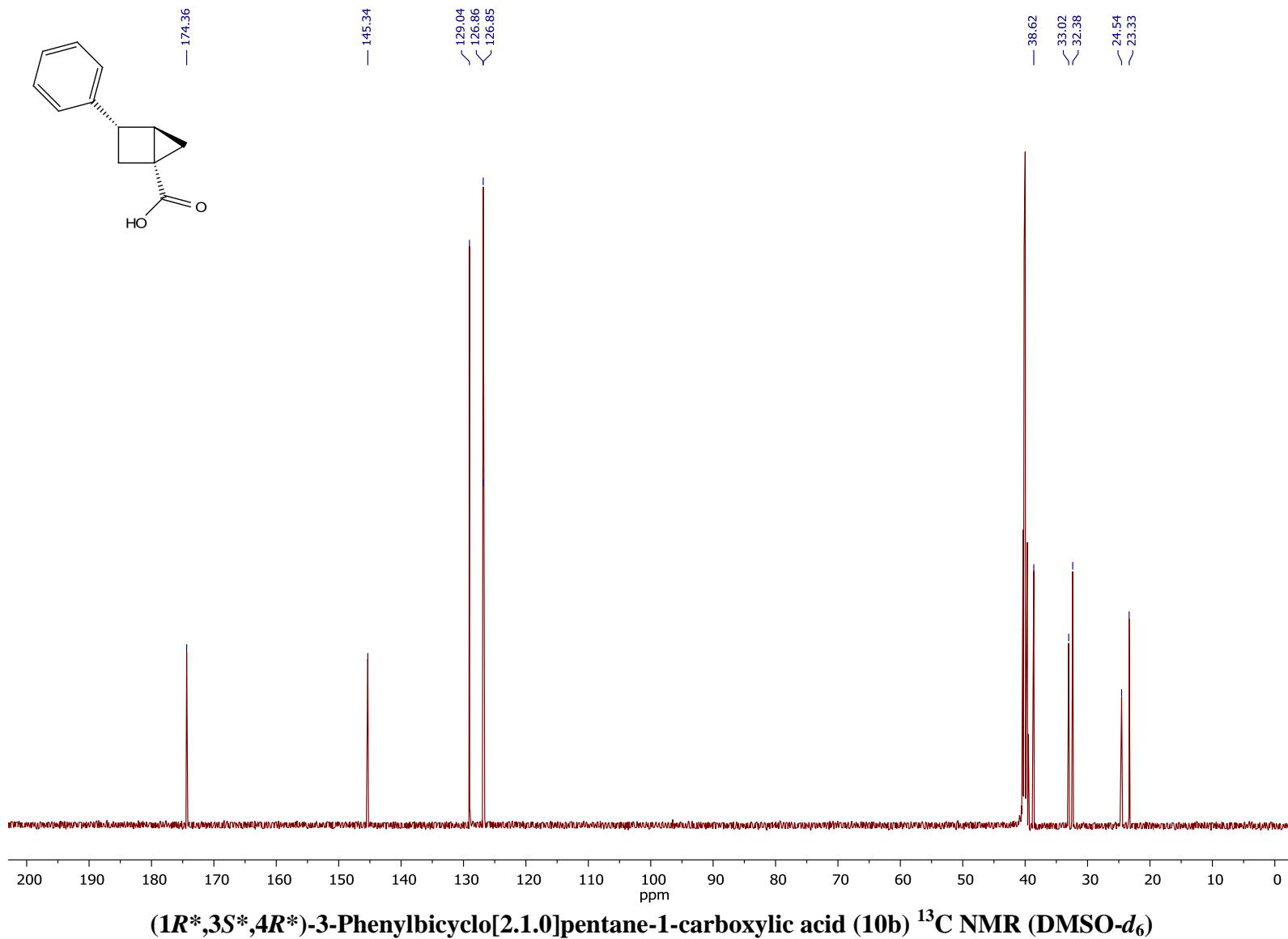


(1*R*^{*},3*S*^{*},4*R*^{*})-3-Methylbicyclo[2.1.0]pentane-1-carboxylic acid (**10a**) (COSY) (DMSO-*d*₆)

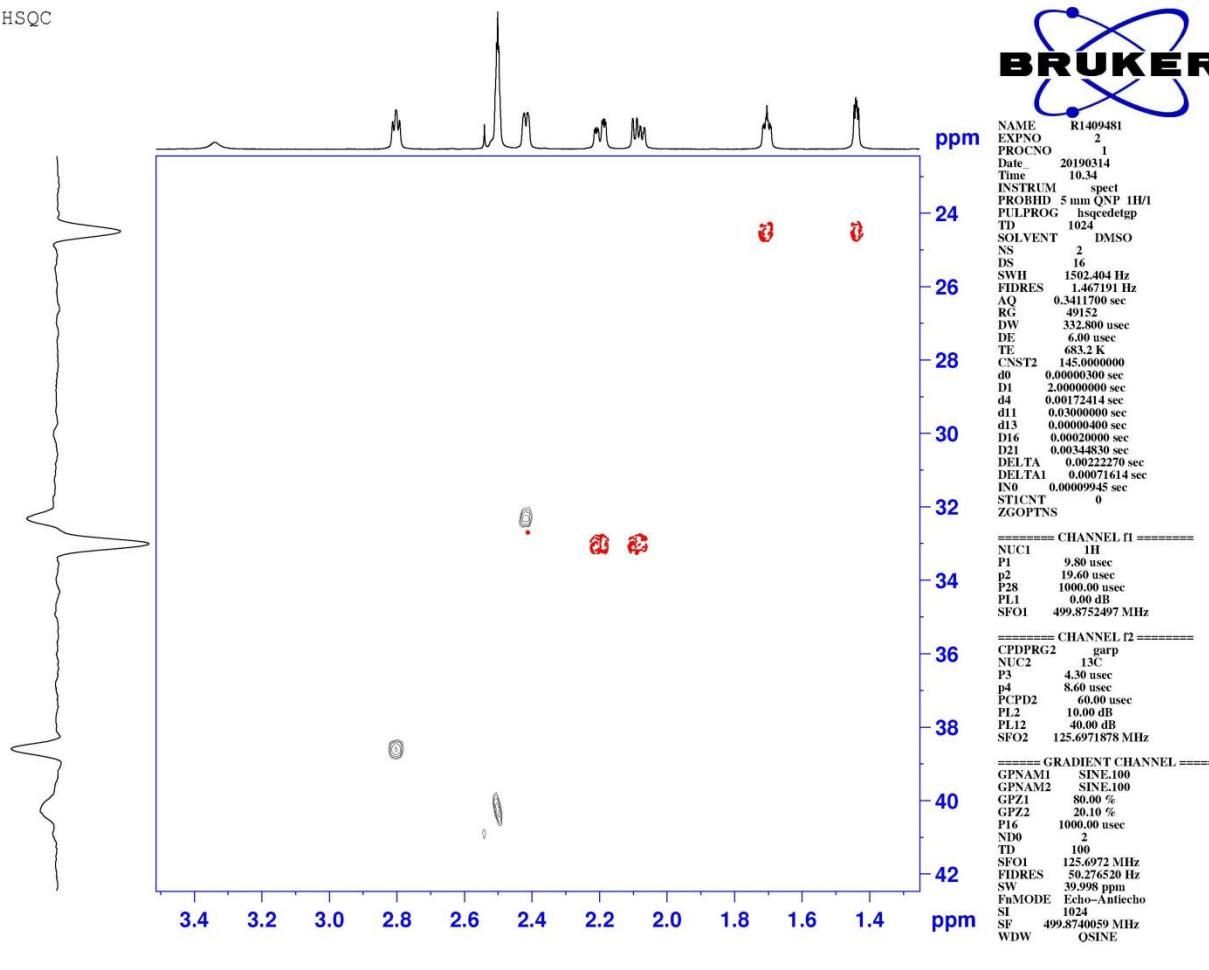
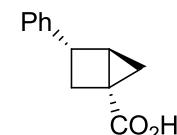


(1*R*^{*},3*S*^{*},4*R*^{*})-3-Methylbicyclo[2.1.0]pentane-1-carboxylic acid (10a) (H–H NOE) (DMSO-*d*₆)

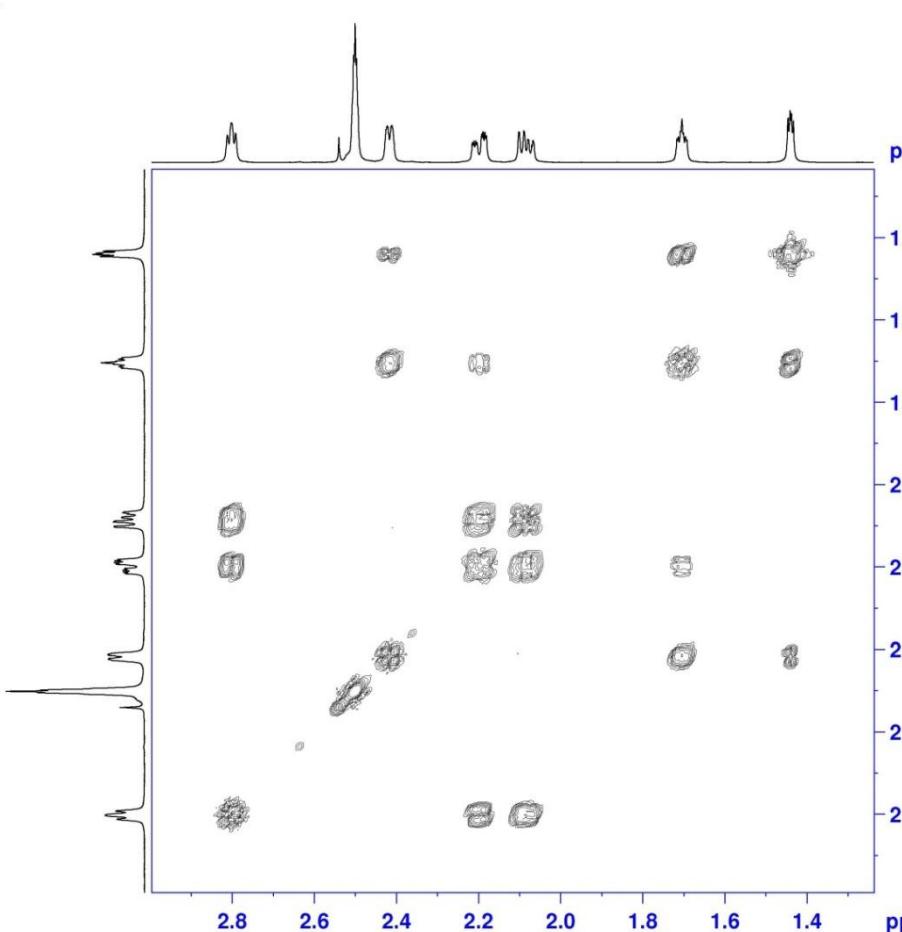




HSQC

(1*R*^{*,3*S*^{*,4*R*^{*}})-3-Phenylbicyclo[2.1.0]pentane-1-carboxylic acid (10b) (HSQC) (DMSO-*d*₆)}

COSY



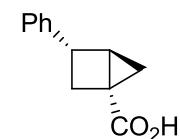
NAME R1409481
 EXPNO 3
 PROCN0 1
 Date_ 20190314
 Time 10.42
 INSTRUM spect
 PROBHD 5 mm QNP 1H/1
 PULPROG cosyppqf
 TD 1024
 SOLVENT DMSO
 NS 1
 DS 16
 SWH 1502.404 Hz
 FIDRES 1.467191 Hz
 AQ 0.3411700 sec
 RG 161.3
 DW 332.800 usec
 DE 6.00 usec
 TE 683.2 K
 d0 0.00000300 sec
 D1 2.0000000 sec
 d13 0.00000400 sec
 D16 0.00050000 sec
 INO 0.00066685 sec

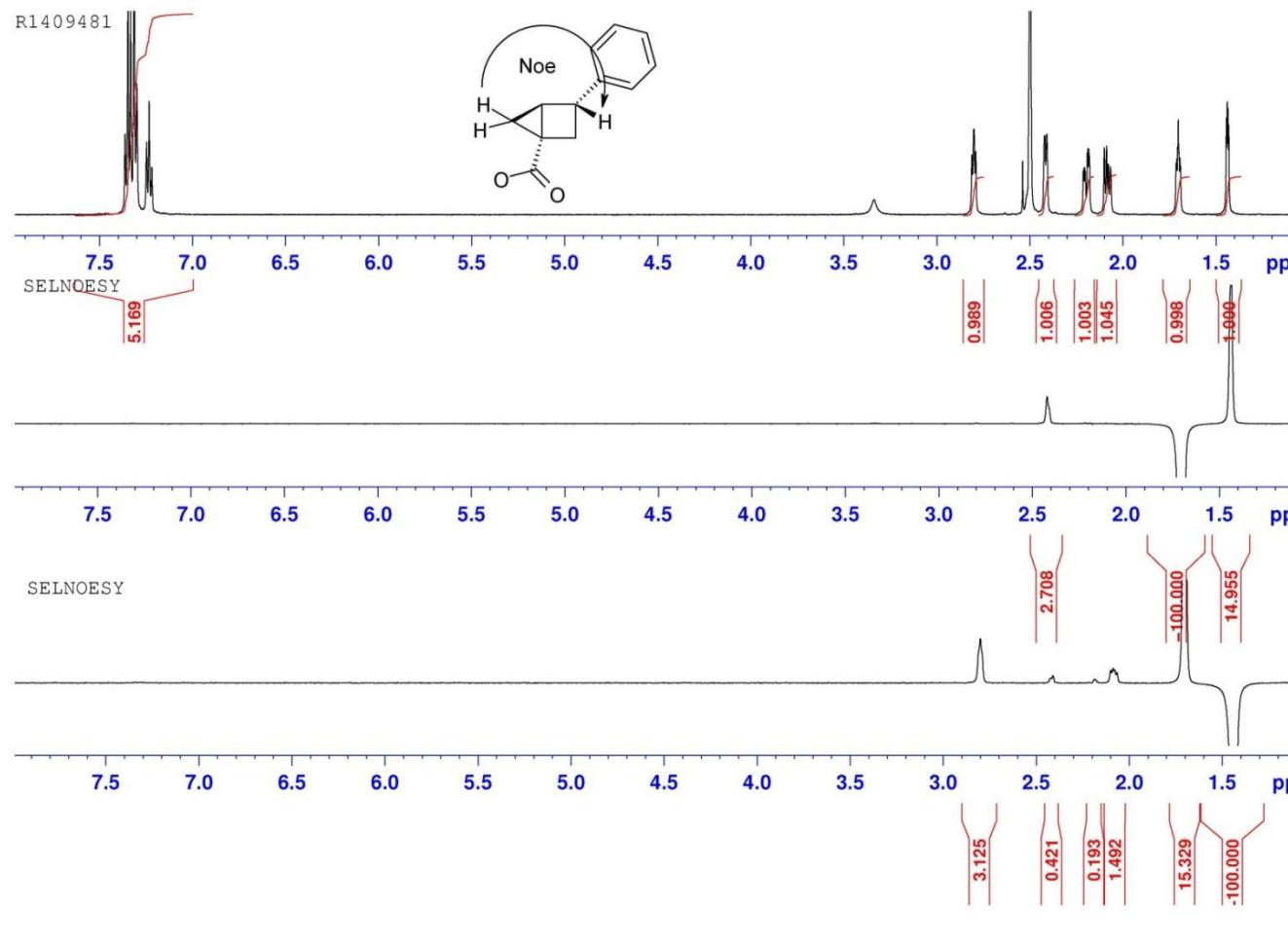
===== CHANNEL f1 =====

NUC1 1H
 P0 9.50 usec
 P1 9.50 usec
 PL1 0.00 dB
 SFO1 499.8752497 MHz

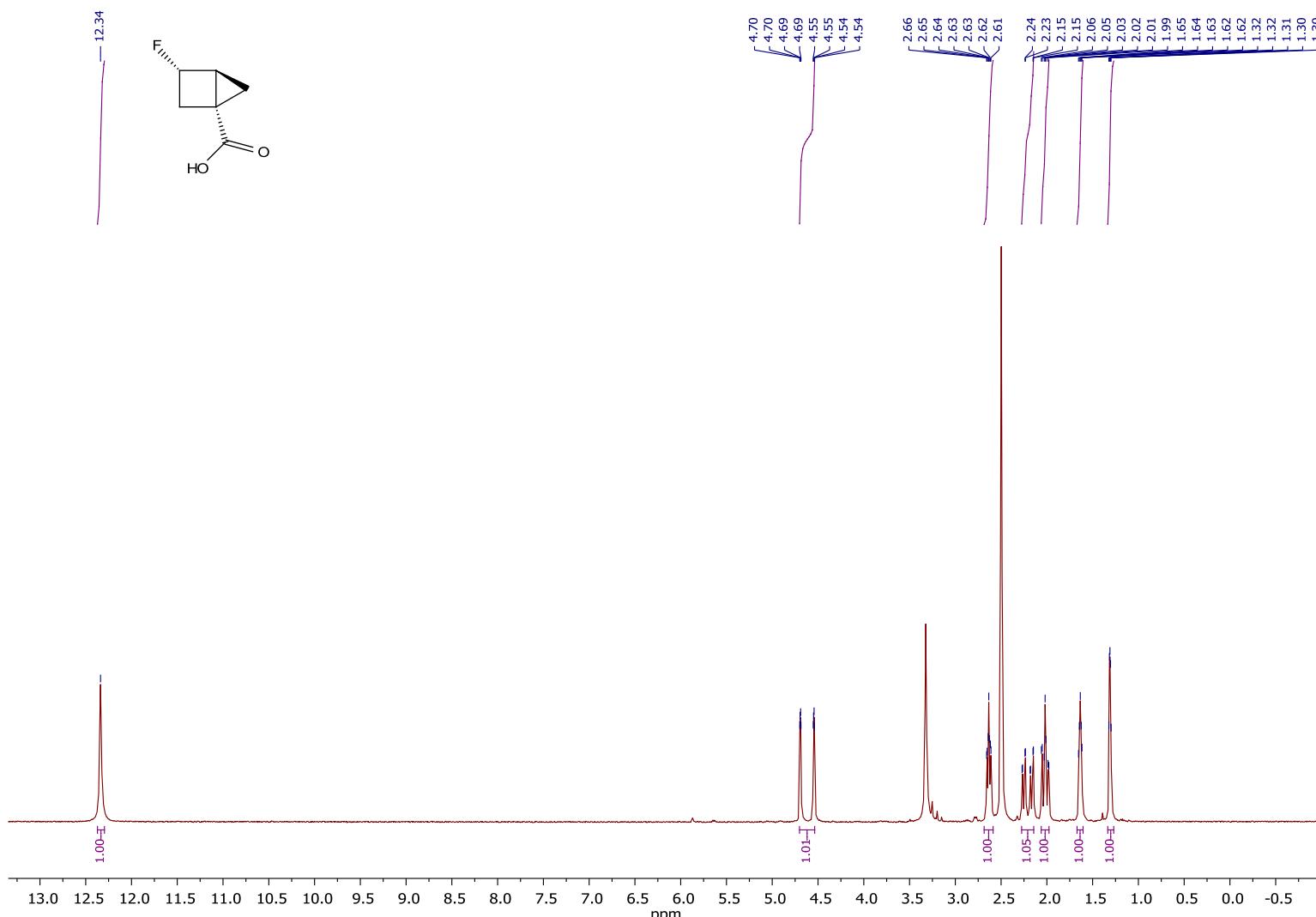
===== GRADIENT CHANNEL =====

GPNAM1 SINE.100
 GPNAM2 SINE.100
 GPZ1 10.00 %
 GPZ2 10.00 %
 P1.6 1000.00 usec
 ND0 1
 TD 128
 SFO1 499.8752 MHz
 FIDRES 11.715528 Hz
 SW 3.000 ppm
 F1MODE QF
 SI 1024
 SF 499.8740069 MHz
 WDW SINE
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.40
 SI 1024
 MC2 QF
 SF 499.8740069 MHz
 WDW SINE
 SSB 0
 LB 0.00 Hz
 GB 0

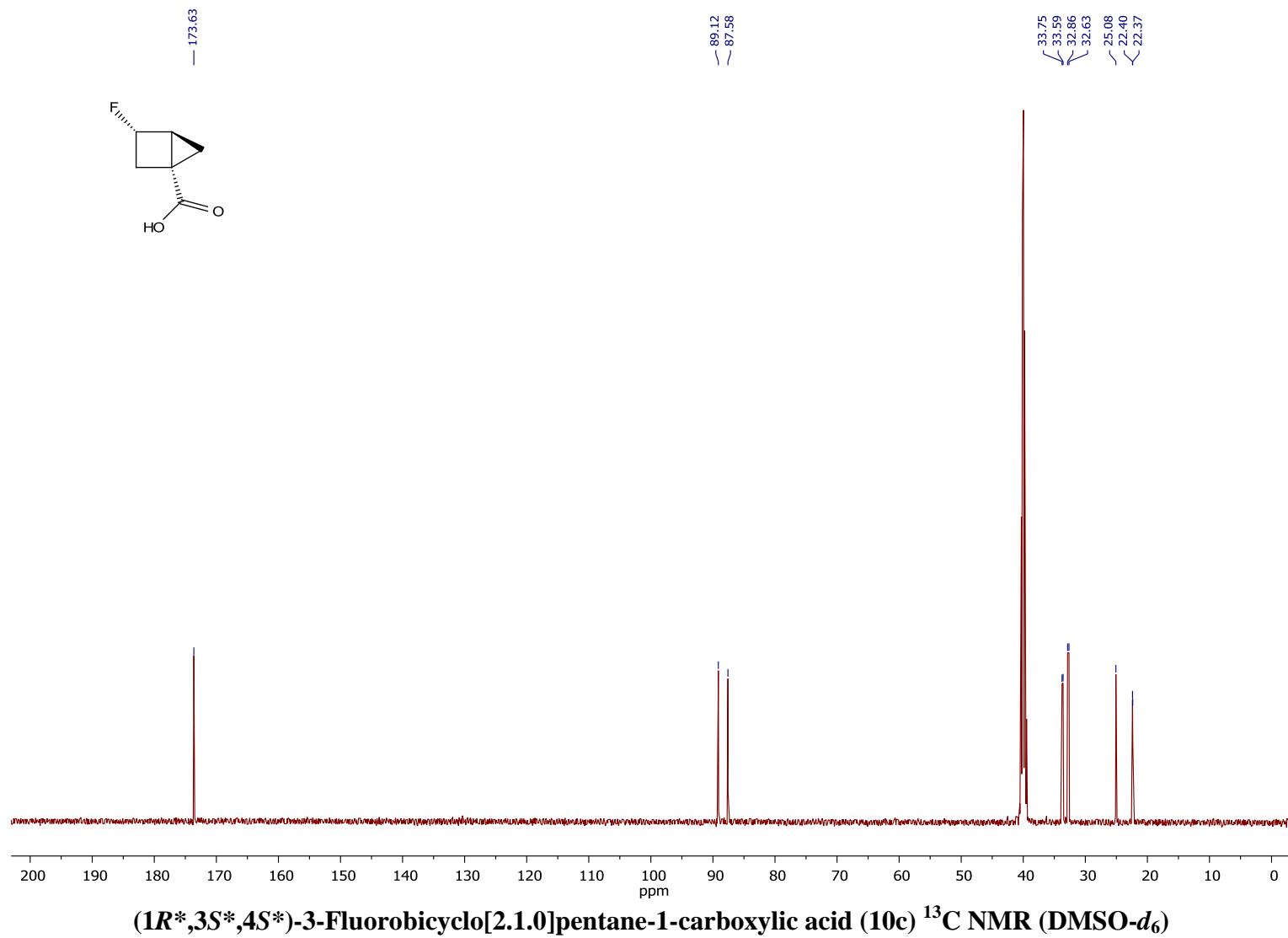
(1*R*^{*},3*S*^{*},4*R*^{*})-3-Phenylbicyclo[2.1.0]pentane-1-carboxylic acid (**10b**) (COSY) (DMSO-*d*₆)

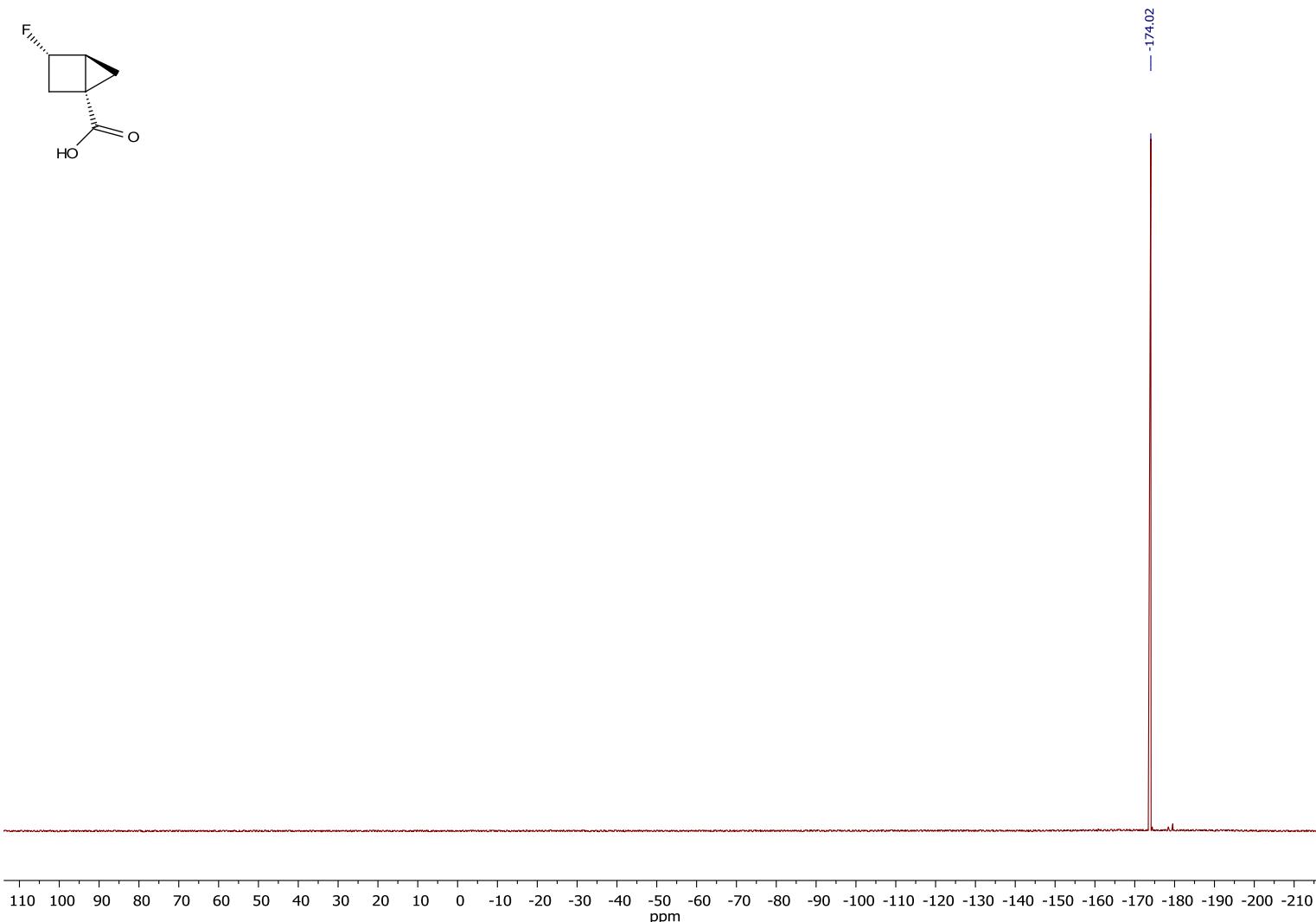
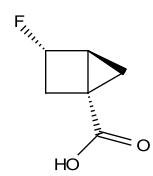


(1*R*^{*},3*S*^{*},4*R*^{*})-3-Phenylbicyclo[2.1.0]pentane-1-carboxylic acid (**10b**) (H–H NOE) (DMSO-*d*₆)



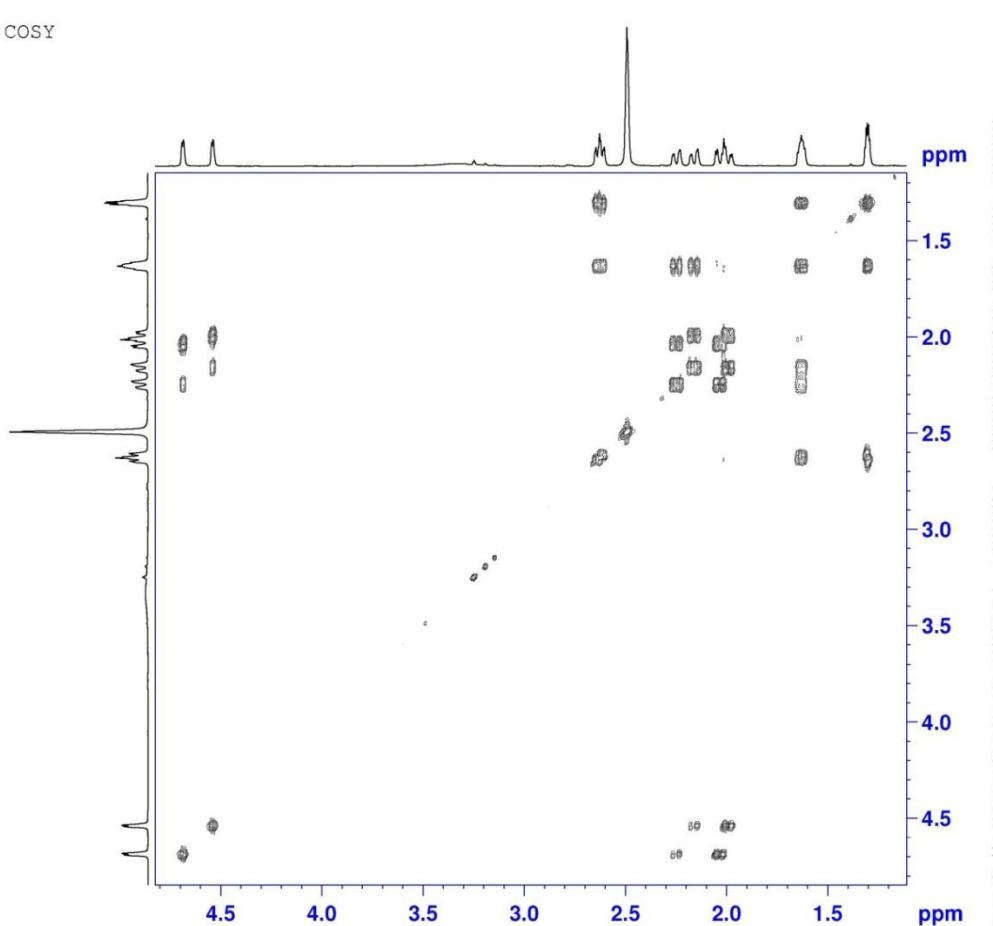
(1*R,3*S**,4*S**)-3-Fluorobicyclo[2.1.0]pentane-1-carboxylic acid (10c) ^1H NMR (DMSO- d_6)**





(1*R*^{*},3*S*^{*},4*S*^{*})-3-Fluorobicyclo[2.1.0]pentane-1-carboxylic acid (10c) ¹⁹F NMR (DMSO-*d*₆)

COSY



NAME R1229402
 EXPNO 6
 PROCNO 1
 Date_ 20180705
 Time 5.37
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG cosygppf
 TD 2048
 SOLVENT DMSO
 NS 1
 DS 16
 SWH 2000.000 Hz
 FIDRES 0.976563 Hz
 AQ 0.5120500 sec
 RG 181
 DW 250.000 usec
 DE 6.50 usec
 TE 300.0 K
 D0 0.00000300 sec
 D1 2.0000000 sec
 D13 0.00000400 sec
 D16 0.00020000 sec
 IN0 0.00049985 sec

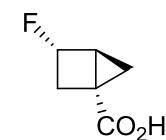
===== CHANNEL f1 =====

NUC1	1H
P0	10.00 usec
P1	10.00 usec
PL1	-1.80 dB
SFO1	400.1314005 MHz

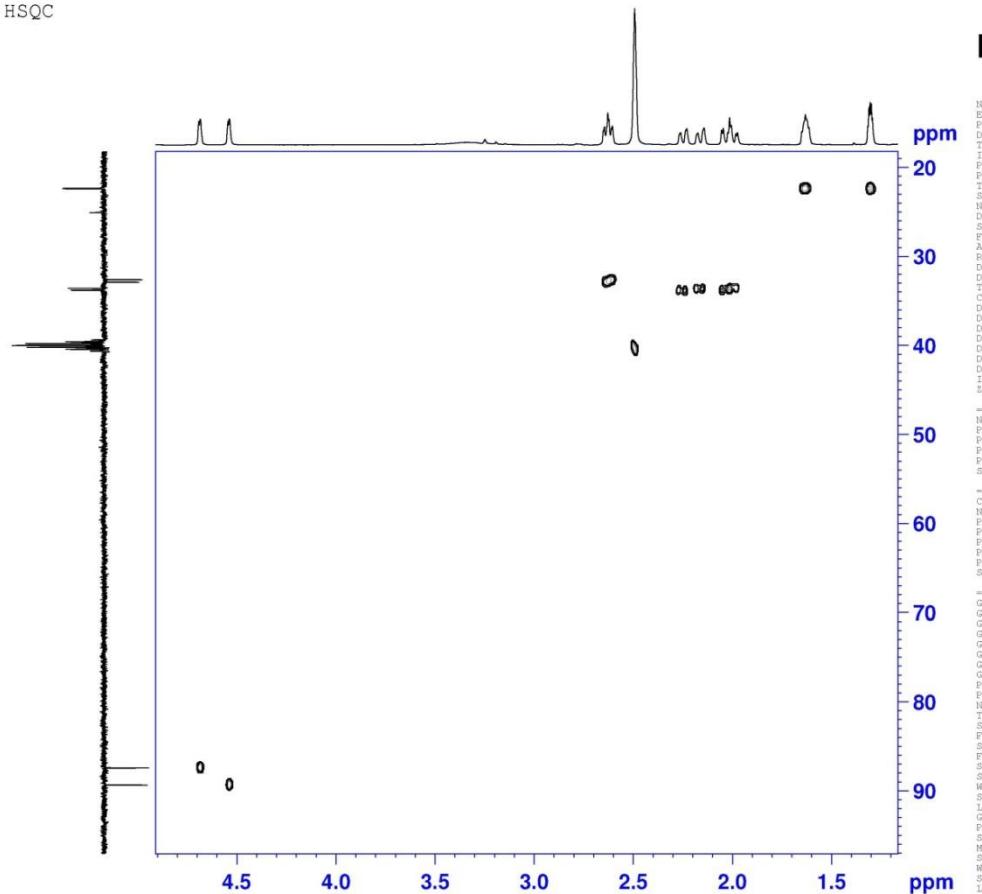
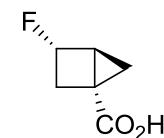
===== GRADIENT CHANNEL =====

GPNAM1	SINE.100
GPZ1	10.00 %
P16	1000.00 usec
NDO	1
TD	256
SFO1	400.1314 MHz
FIDRES	7.815066 Hz
SW	5.000 ppm

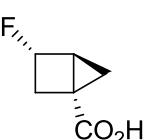
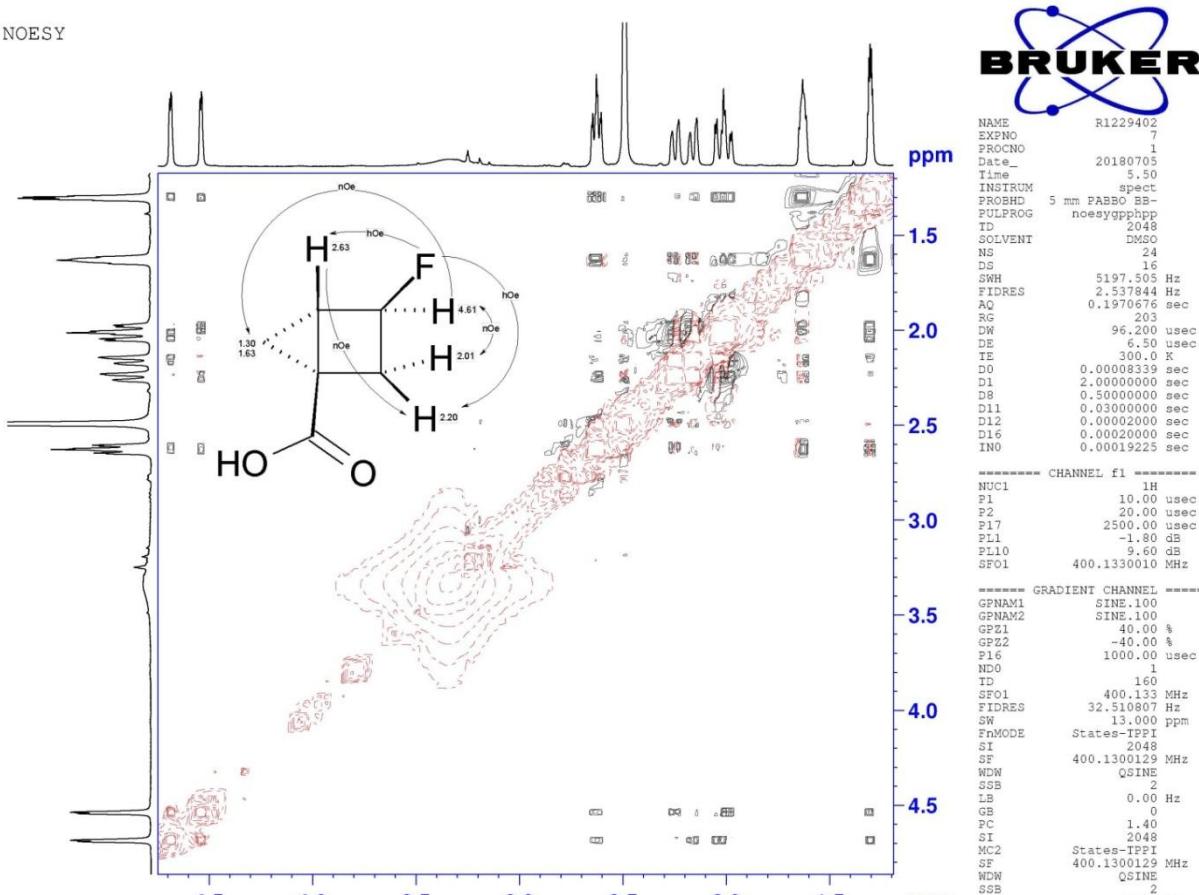
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SSB	0
LB	0.00 Hz
GB	0
PC	1.40
SI	2048
MC2	QF
SF	400.1300129 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0

(1*R*^{*},3*S*^{*},4*S*^{*})-3-Fluorobicyclo[2.1.0]pentane-1-carboxylic acid (**10c**) (COSY) (DMSO-*d*₆)

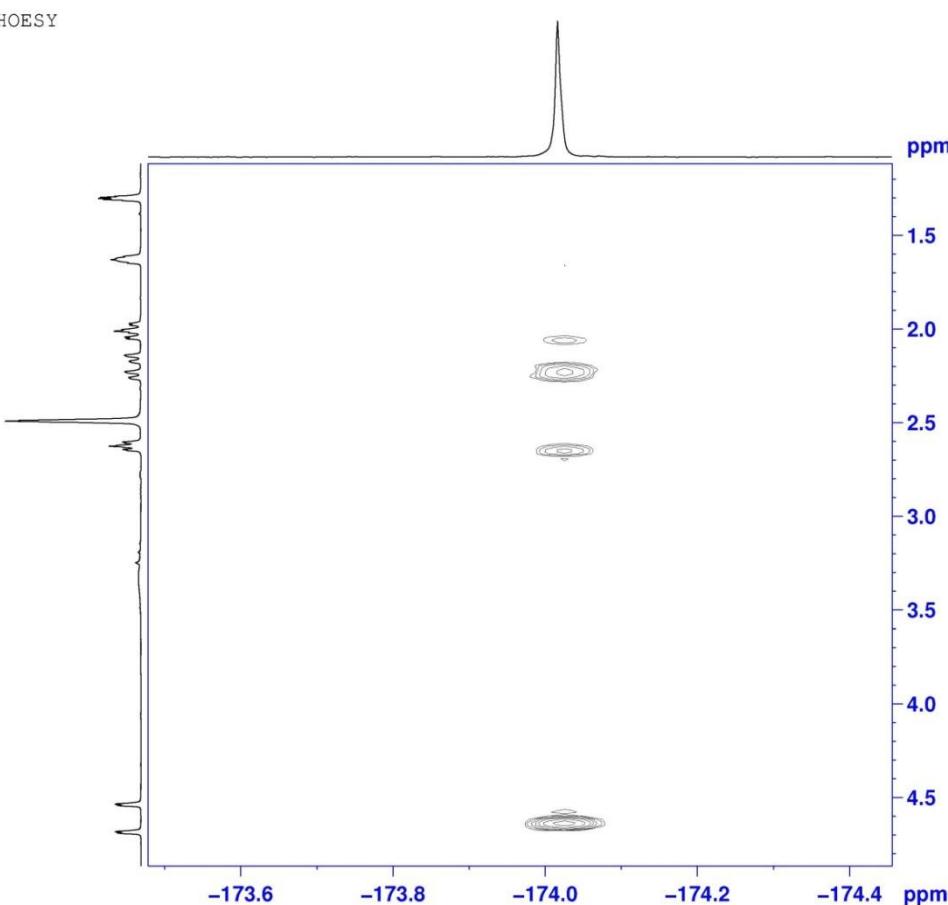
HSQC

(1*R*^{*,}3*S*^{*,}4*S*^{*})-3-Fluorobicyclo[2.1.0]pentane-1-carboxylic acid (10c) (HSQC) (DMSO-*d*₆)

NOESY

(1*R*^{*},3*S*^{*},4*S*^{*})-3-Fluorobicyclo[2.1.0]pentane-1-carboxylic acid (**10c**) (NOESY) (DMSO-*d*₆)

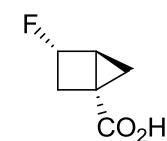
HOESY

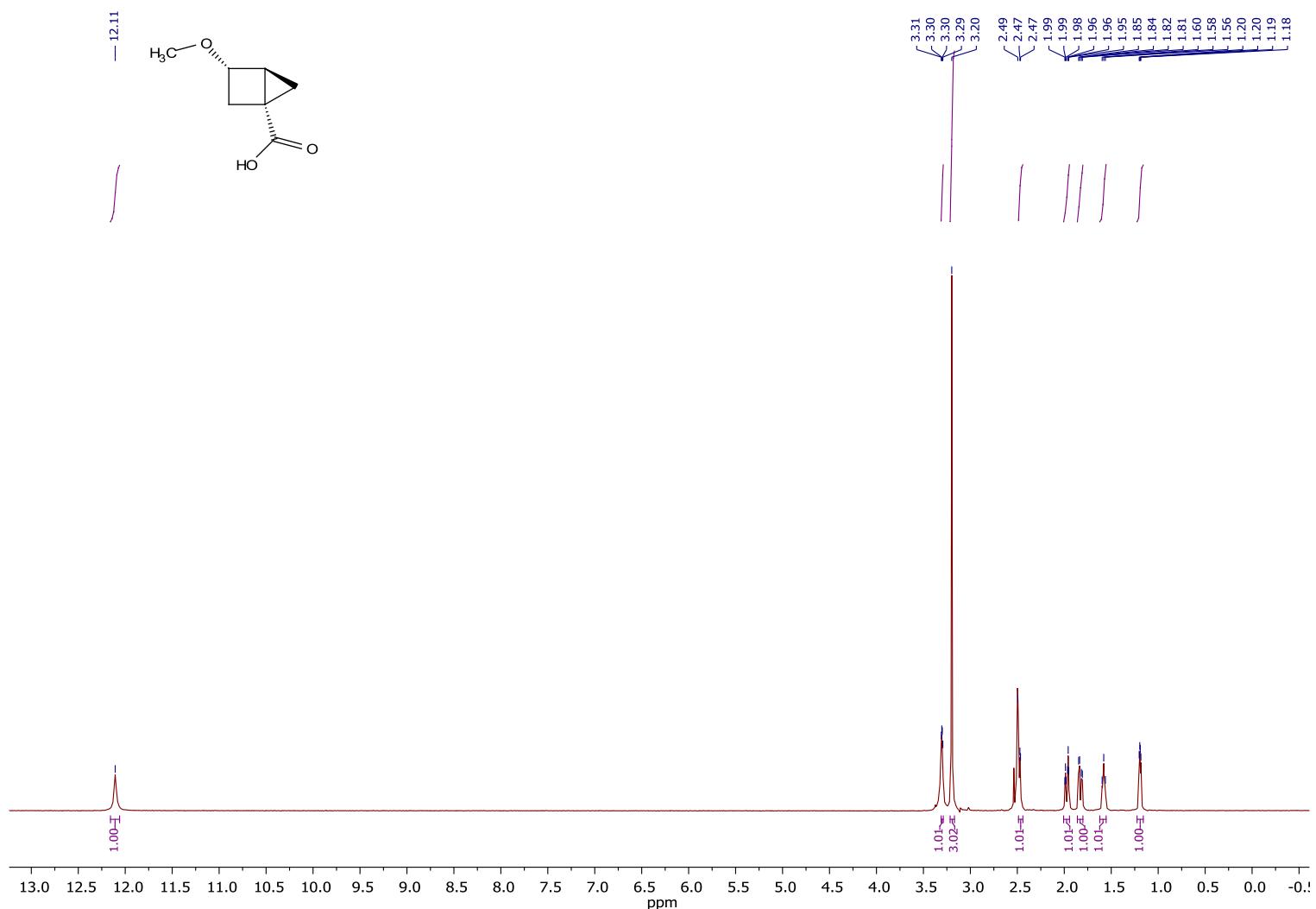


NAME R1229402
 EXPNO 5
 PROGNO 1
 Date 20180705
 Time 2.53
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG hoesypn
 TD 1024
 SOLVENT DMSO
 NS 32
 DS 16
 SWH 7530.121 Hz
 FIDRES 7.353633 Hz
 AC 0.06800 sec
 RG 645
 DW 66.400 usec
 DE 6.50 usec
 TE 300.0 K
 D0 0.00000300 sec
 D1 2.00000000 sec
 D8 1.00000000 sec
 D12 0.00002000 sec
 IN0 0.00024990 sec

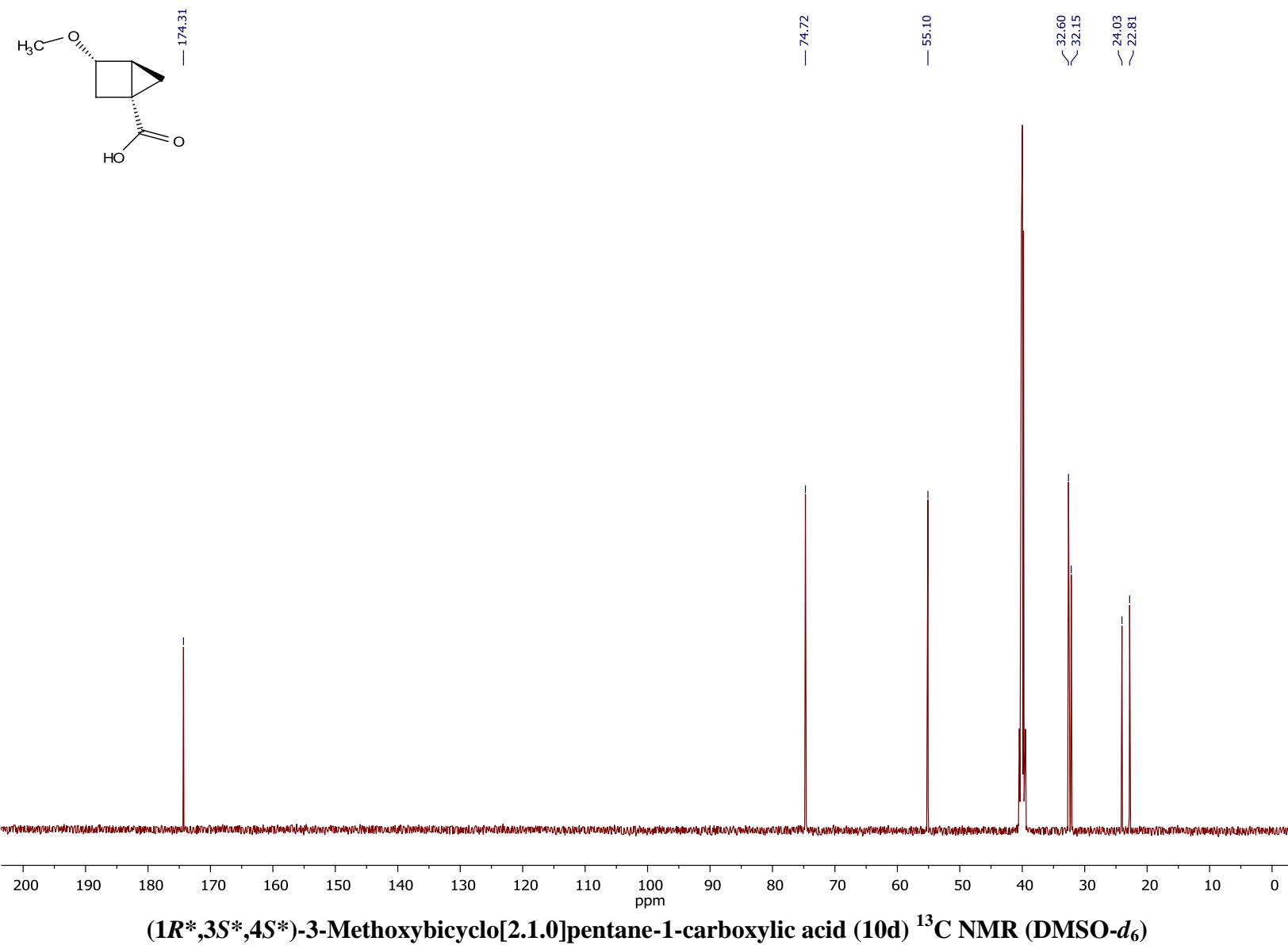
----- CHANNEL f1 -----
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 P1 12.30 usec
 P2 24.60 usec
 PL1 -2.00 dB
 SFO1 376.4330355 MHz

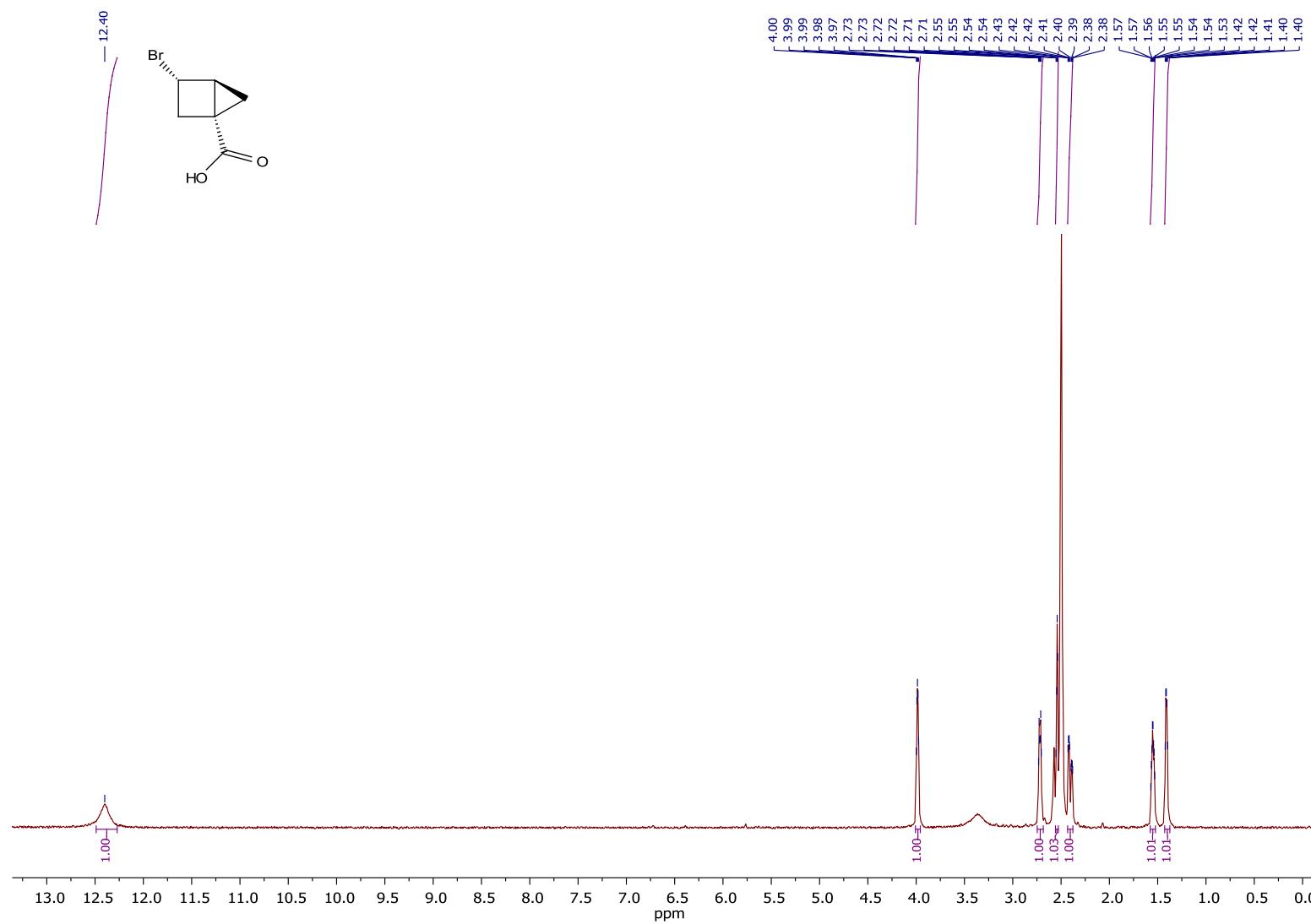
----- CHANNEL f2 -----
 CPDPRG2 waltz16
 NUC2 1H
 P3 10.00 usec
 P4 20.00 usec
 PCPD2 90.00 usec
 PL2 -1.80 dB
 PL12 19.10 dB
 SFO2 400.1314005 MHz
 ND0 2
 TD 100
 SFO1 400.1314 MHz
 FIDRES 20.006571 Hz
 SW 5.000 ppm
 FnMODE States-TPPI
 SI 1024
 SF 376.4985527 MHz
 WDW QSINE
 SSB 2
 LB 0.00 Hz
 GB 0
 RC 1.40
 SI 512
 MC2 States-TPPI
 SF 400.1300000 MHz
 WDW QSINE
 SSB 2
 LB 0.00 Hz
 GB 0

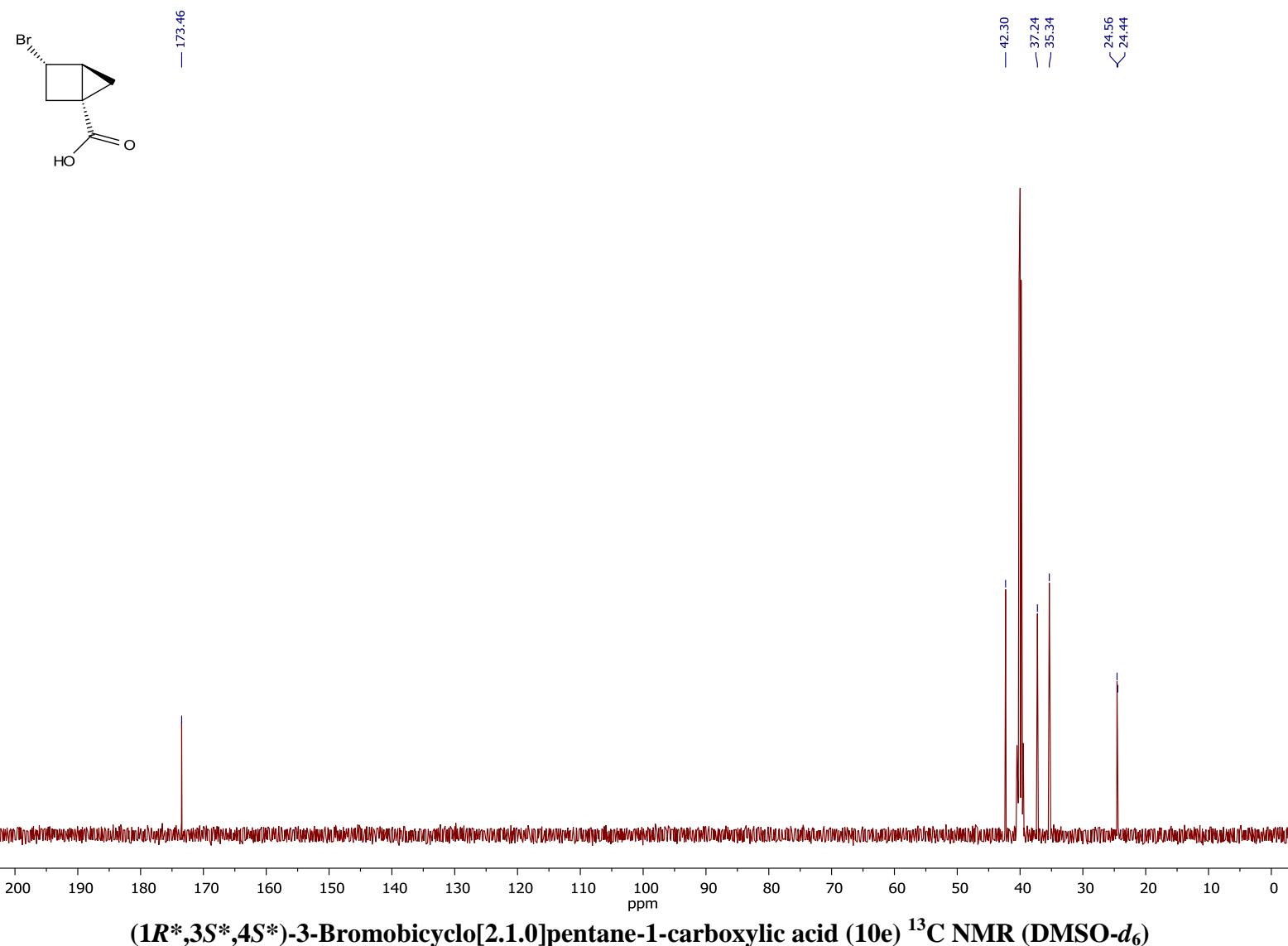
(1*R*^{*},3*S*^{*},4*S*^{*})-3-Fluorobicyclo[2.1.0]pentane-1-carboxylic acid (10c) (HOESY) (DMSO-*d*₆)

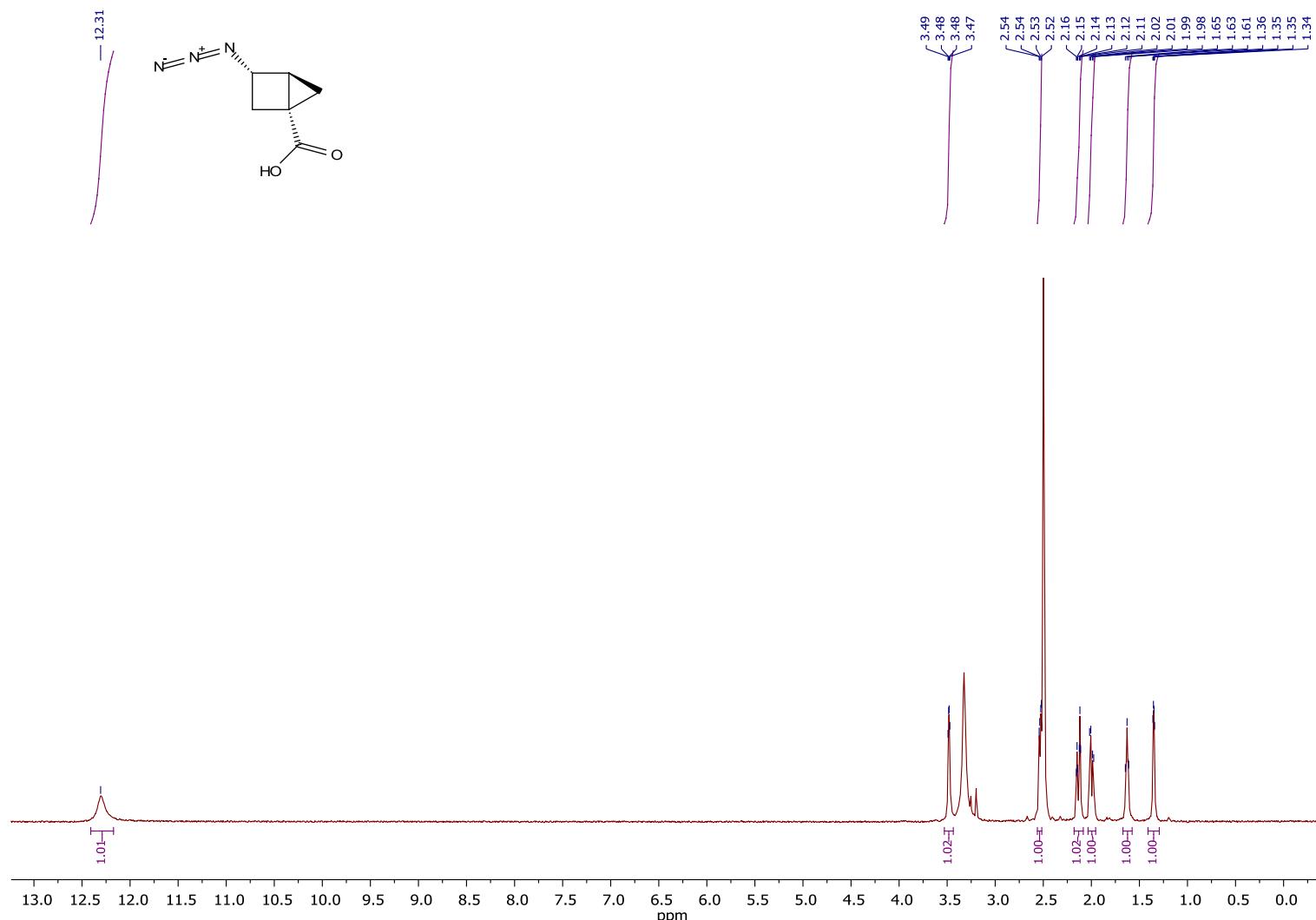


(1*R*^{*},3*S*^{*},4*S*^{*})-3-Methoxybicyclo[2.1.0]pentane-1-carboxylic acid (10d) ^1H NMR (DMSO-*d*₆)

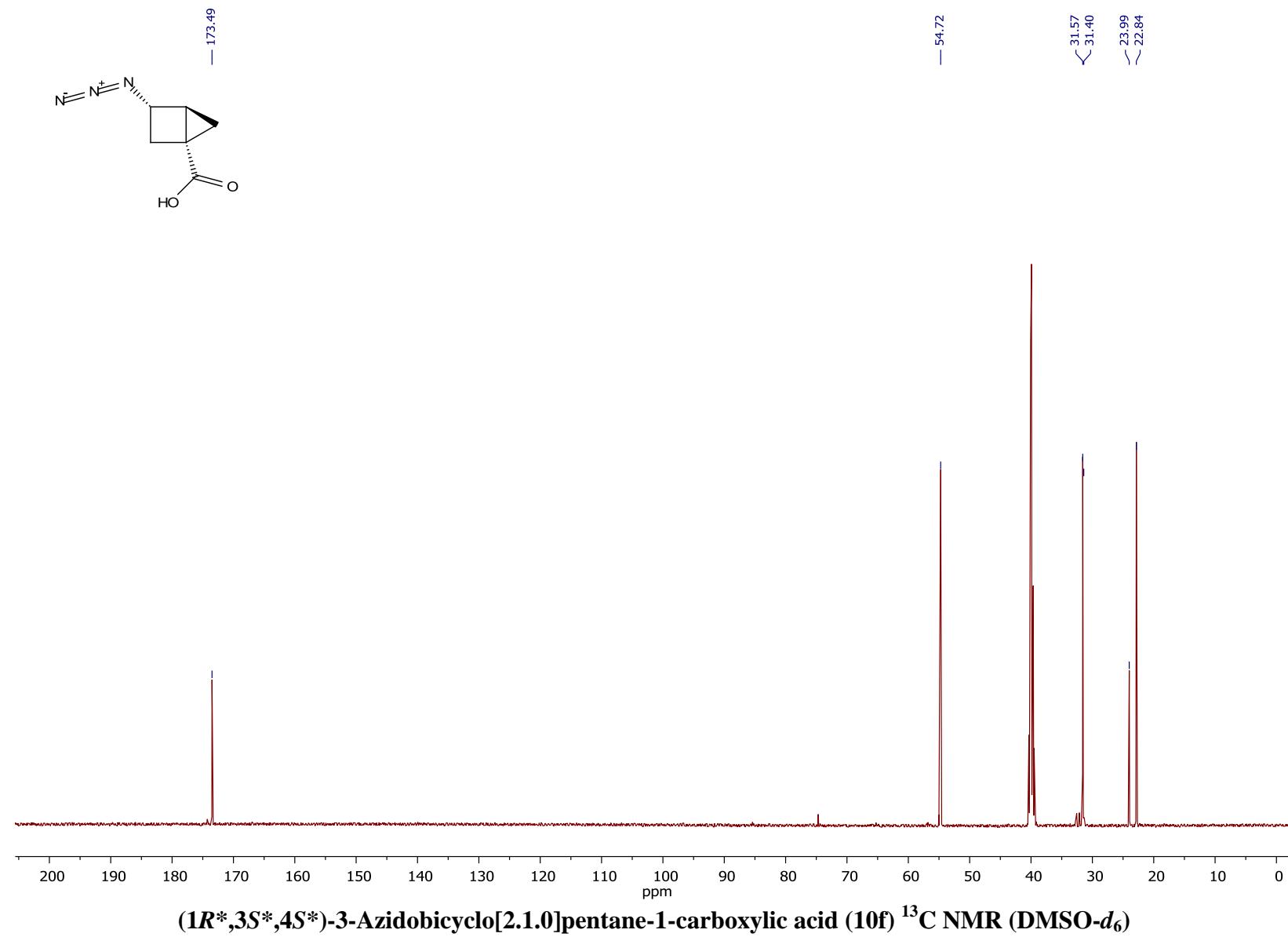


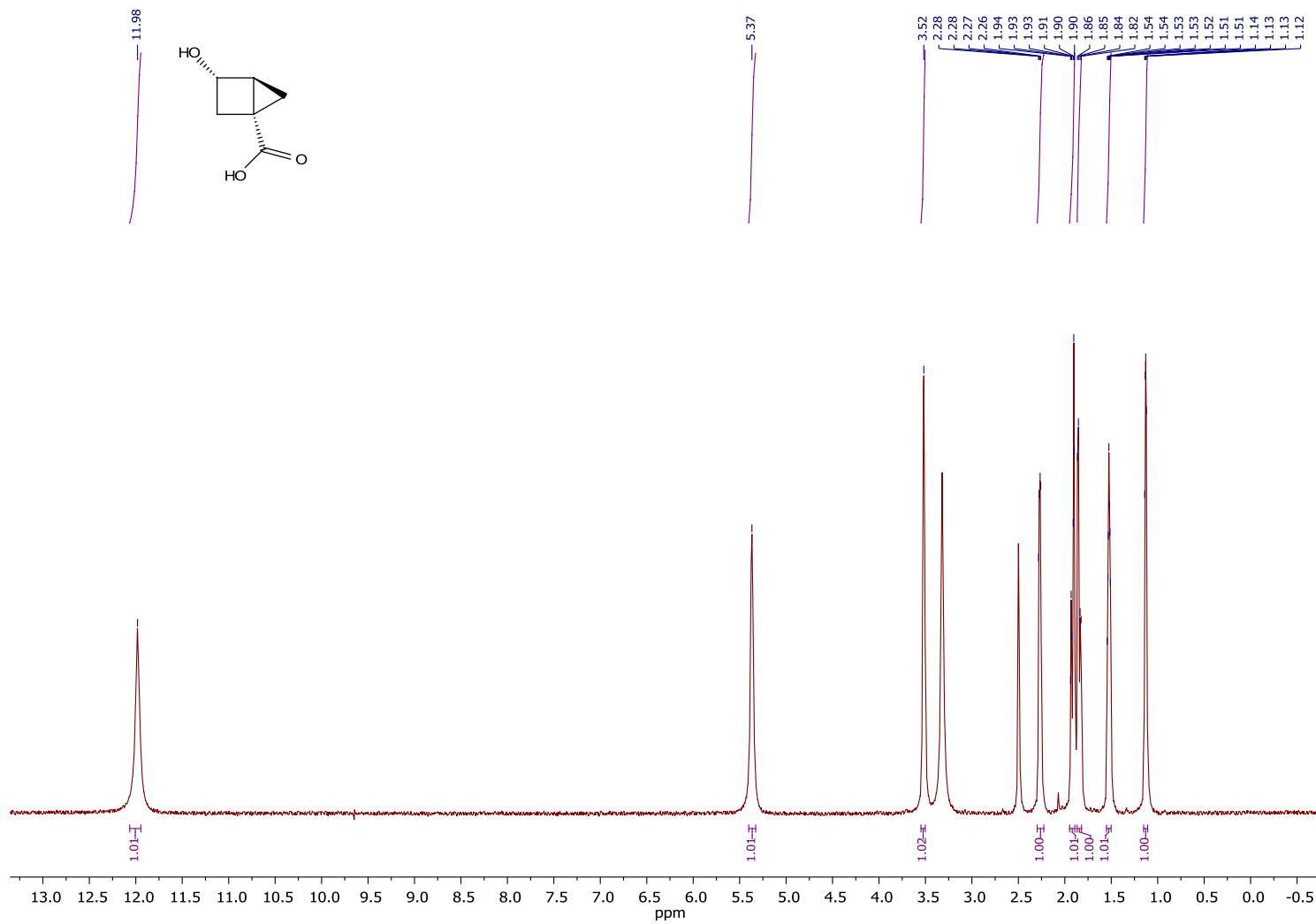




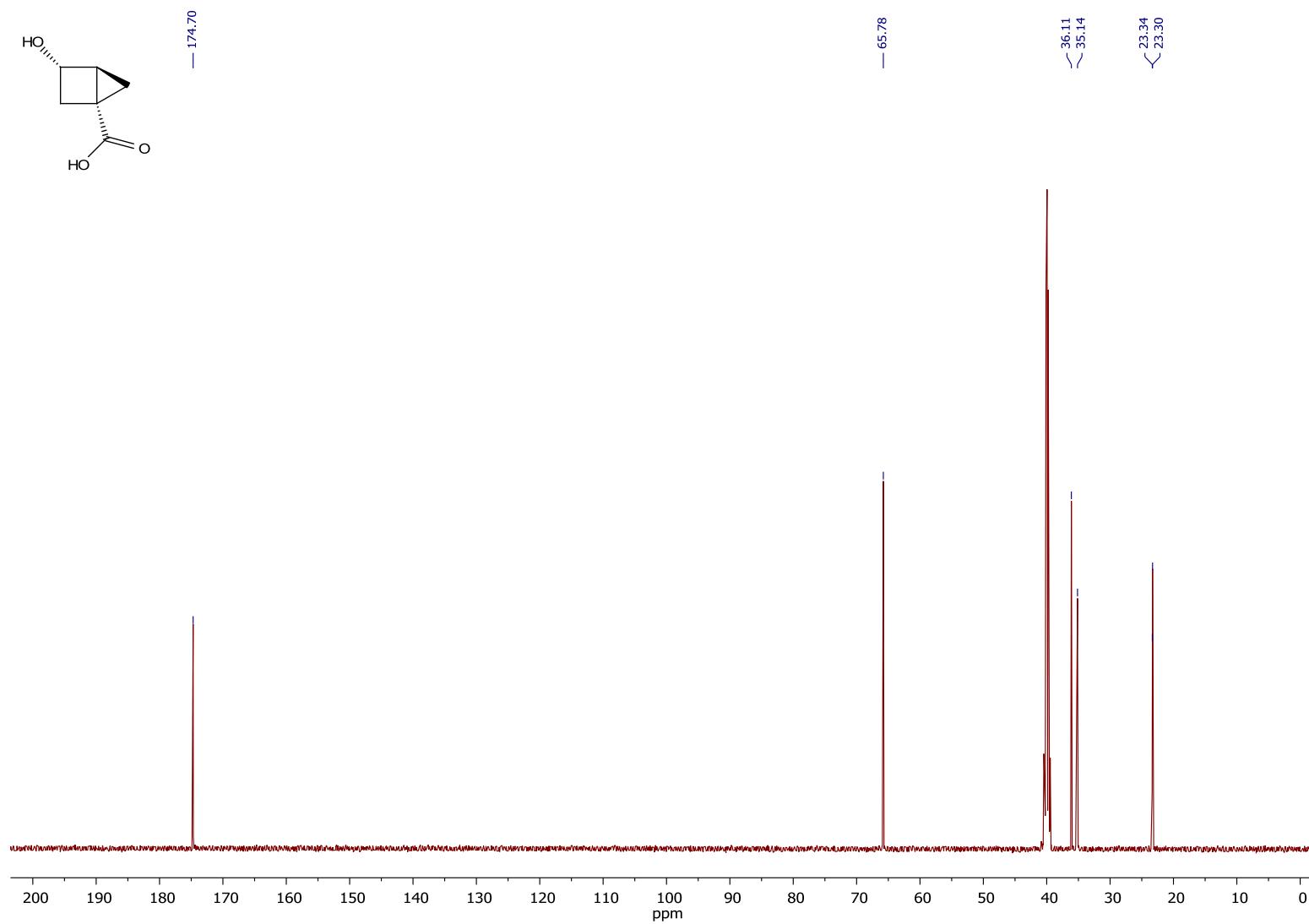


(1R*,3S*,4S*)-3-Azidobicyclo[2.1.0]pentane-1-carboxylic acid (10f) ^1H NMR (DMSO- d_6)



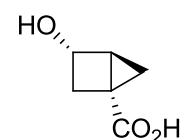
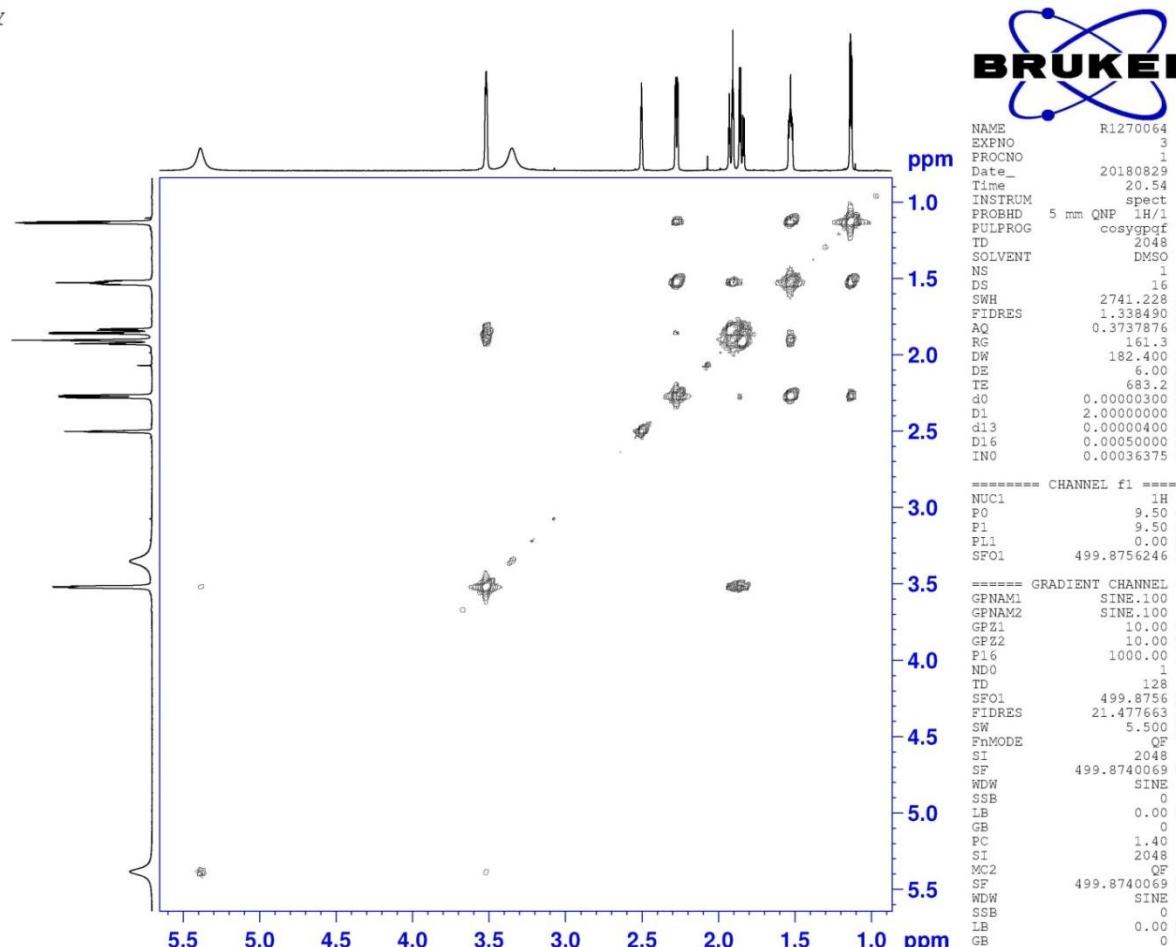


(1*R*^{*,3*S*^{*,4*S*^{*}})-3-Hydroxybicyclo[2.1.0]pentane-1-carboxylic acid (10g) ¹H NMR (DMSO-*d*₆)}

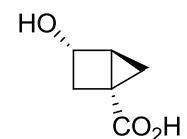
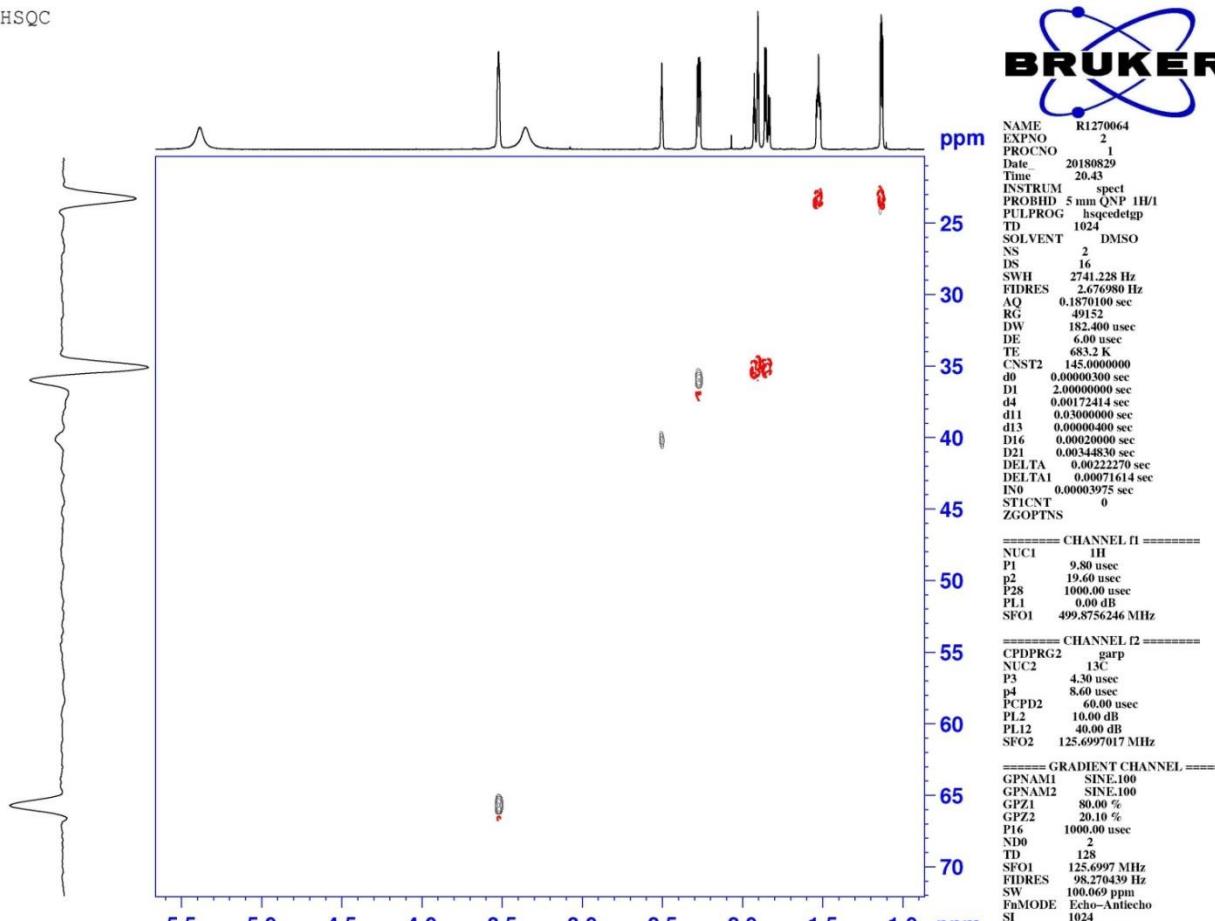


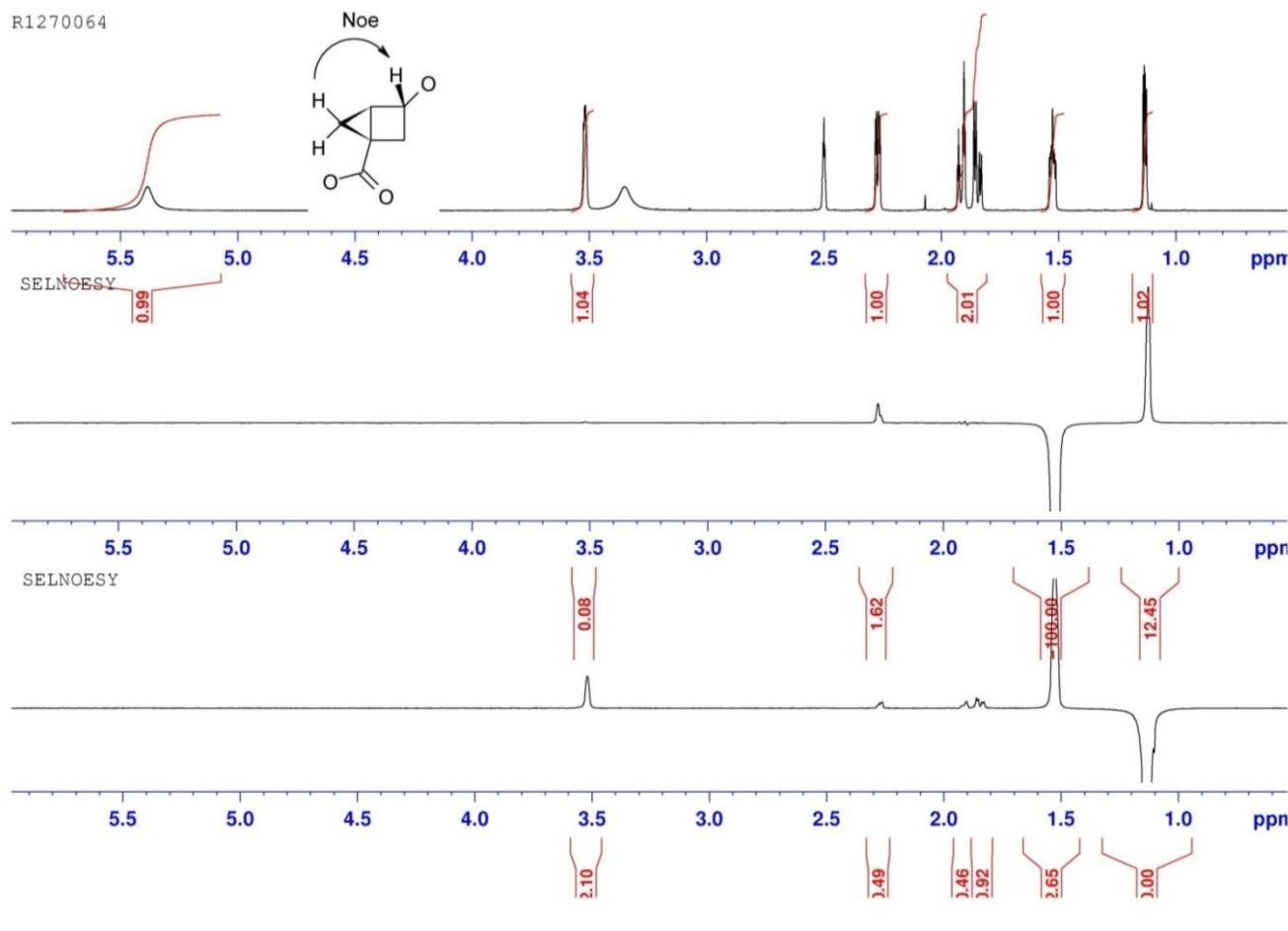
(1*R*^{*,3*S*^{*,4*S*^{*}}}-3-Hydroxybicyclo[2.1.0]pentane-1-carboxylic acid (10g) ^{13}C NMR (DMSO- d_6)

COSY

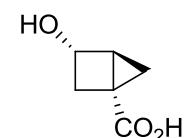
(1*R**,3*S**,4*S**)-3-Hydroxybicyclo[2.1.0]pentane-1-carboxylic acid (**10g**) (COSY) (DMSO-*d*₆)

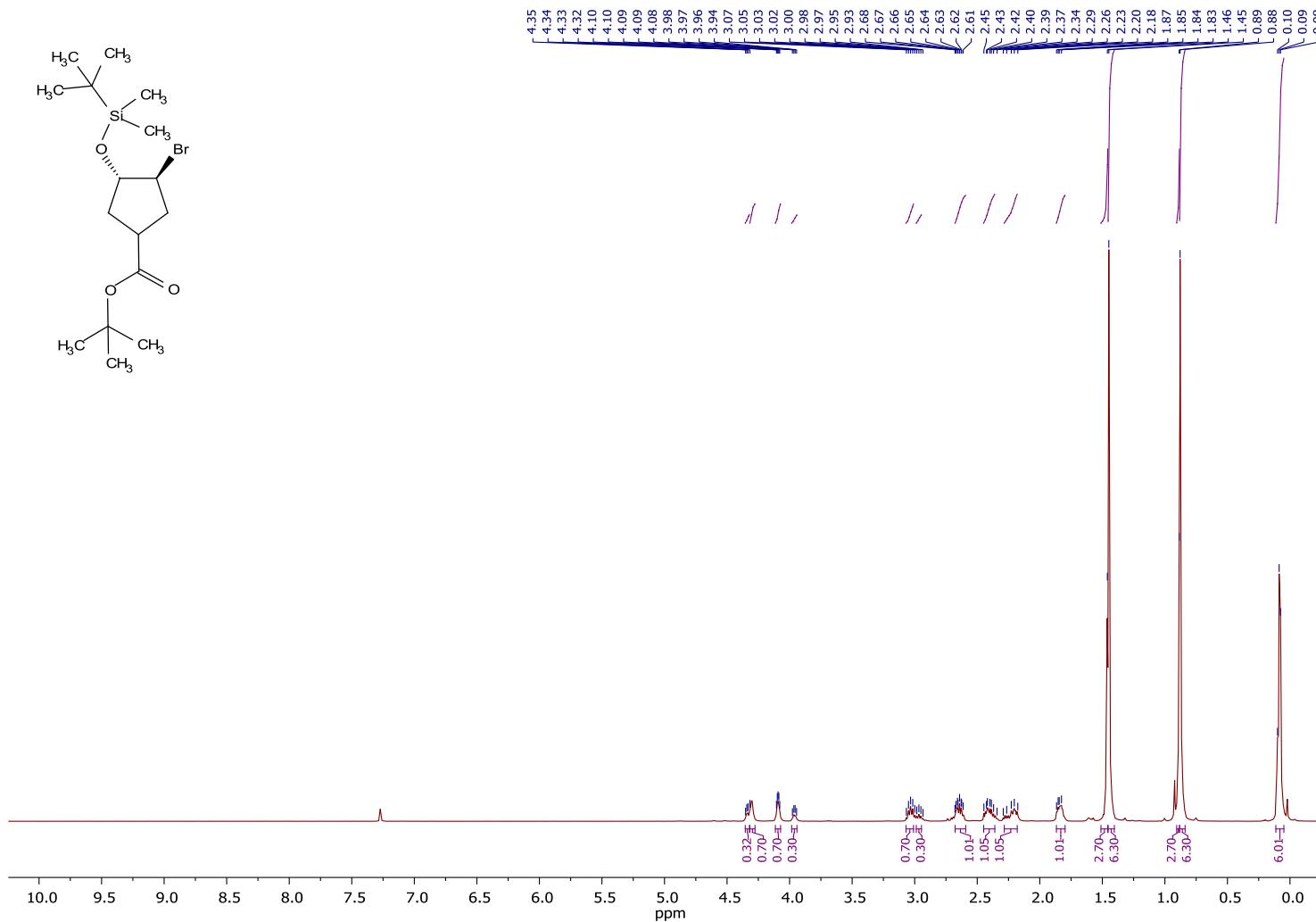
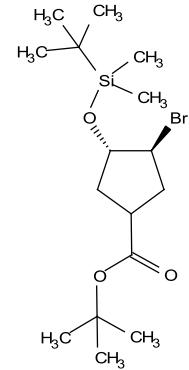
HSQC

(1*R*^{*},3*S*^{*},4*S*^{*})-3-Hydroxybicyclo[2.1.0]pentane-1-carboxylic acid (10g) (HSQC) (DMSO-*d*₆)

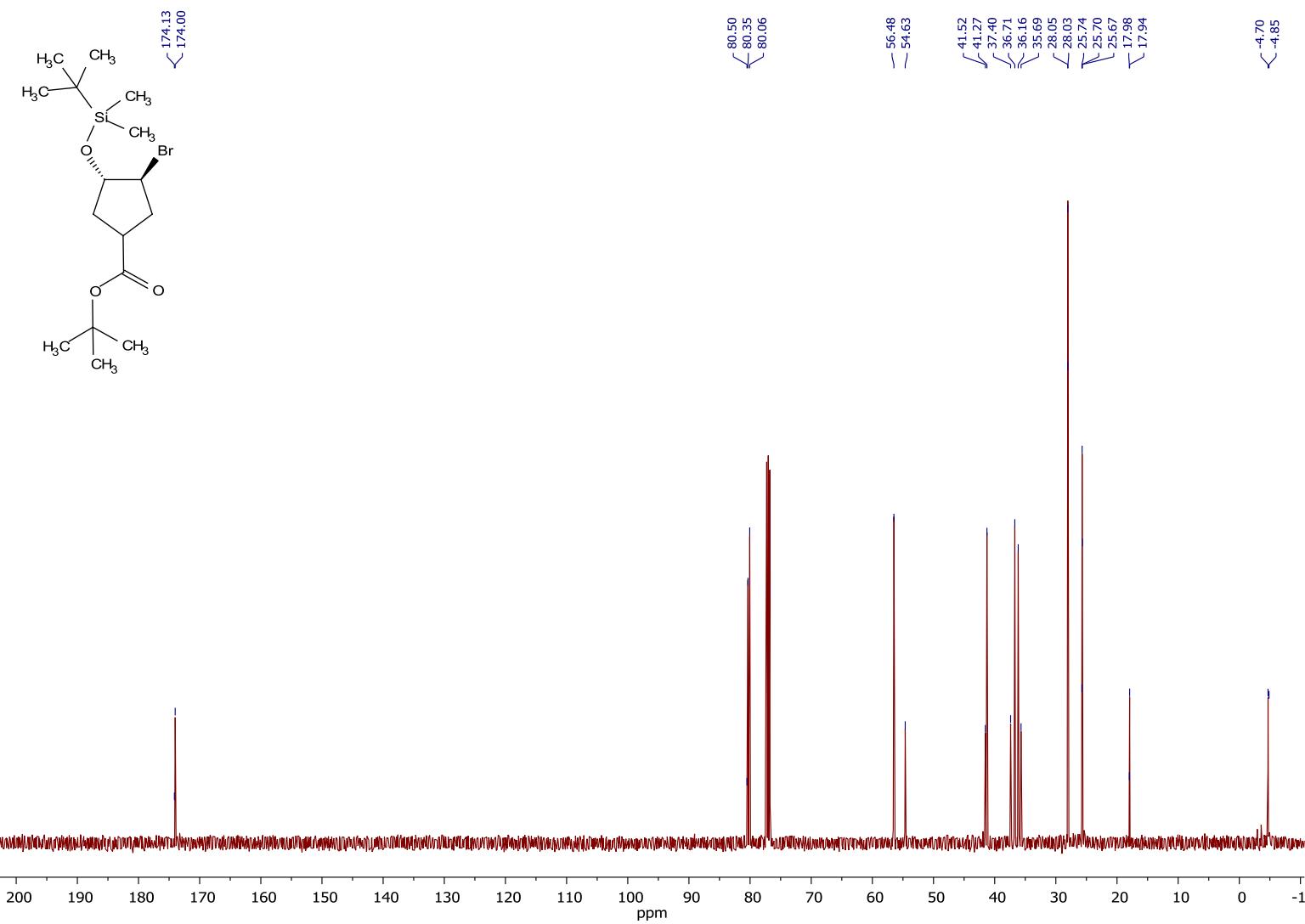


($1R^*,3S^*,4S^*$)-3-Hydroxybicyclo[2.1.0]pentane-1-carboxylic acid (**10g**) ($\text{H}-\text{H}$ NOE) ($\text{DMSO}-d_6$)

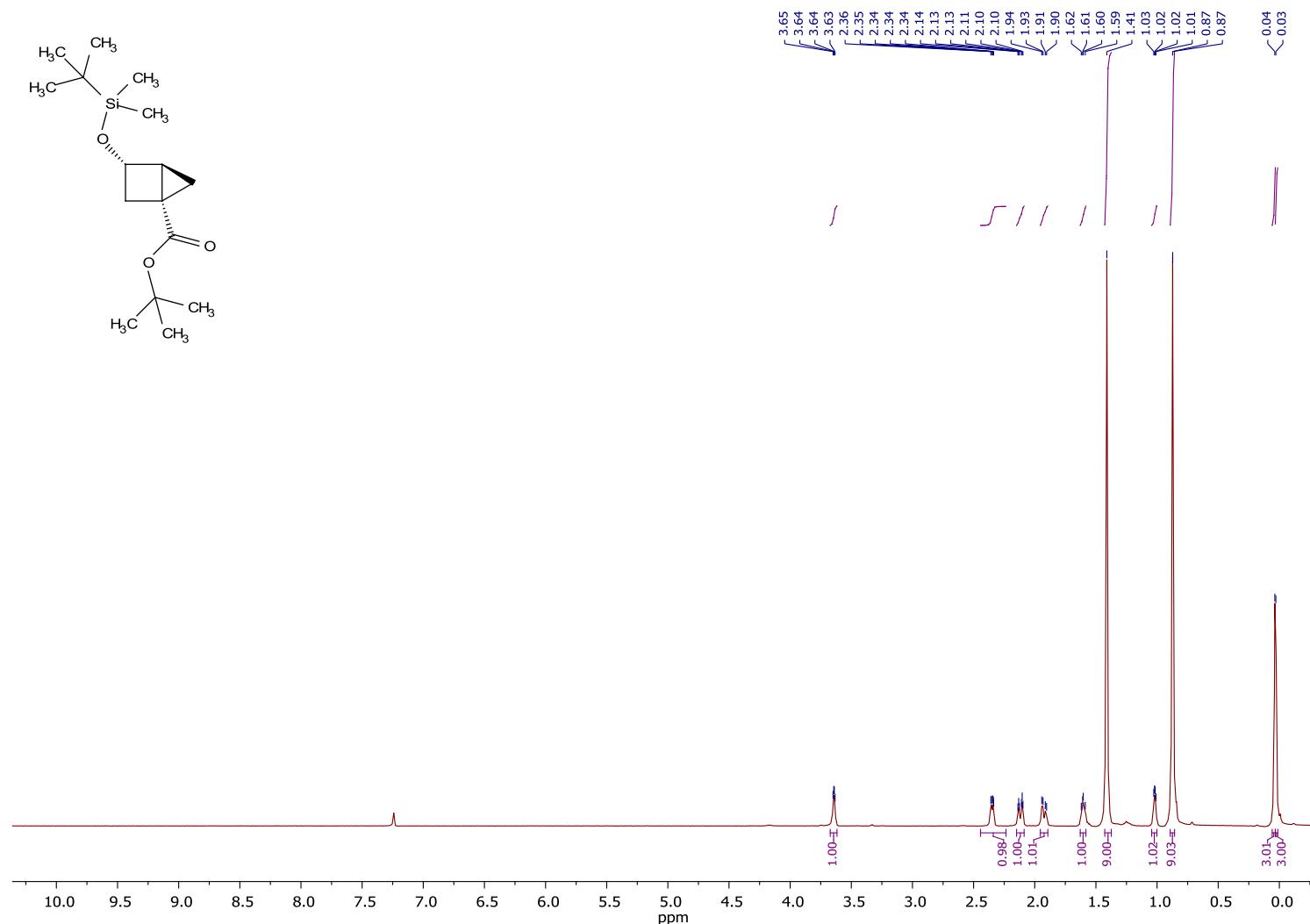
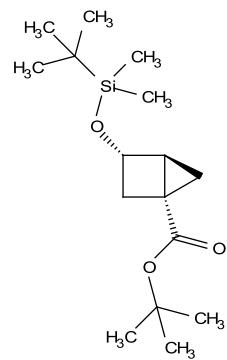




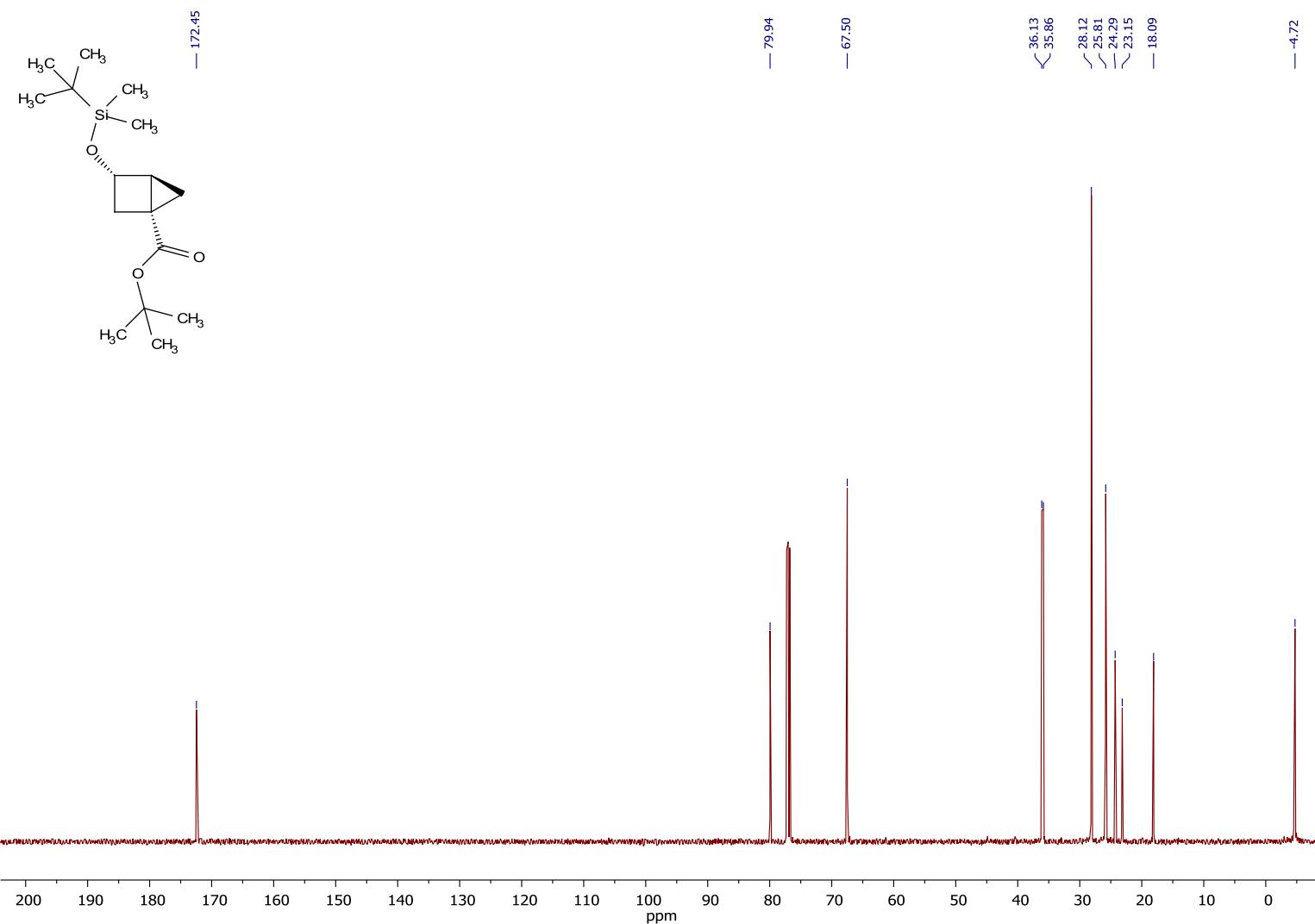
(3S*,4S*)-tert-Butyl 3-bromo-4-((tert-butyldimethylsilyl)oxy)cyclopentanecarboxylate (11g) ¹H NMR (CDCl_3)



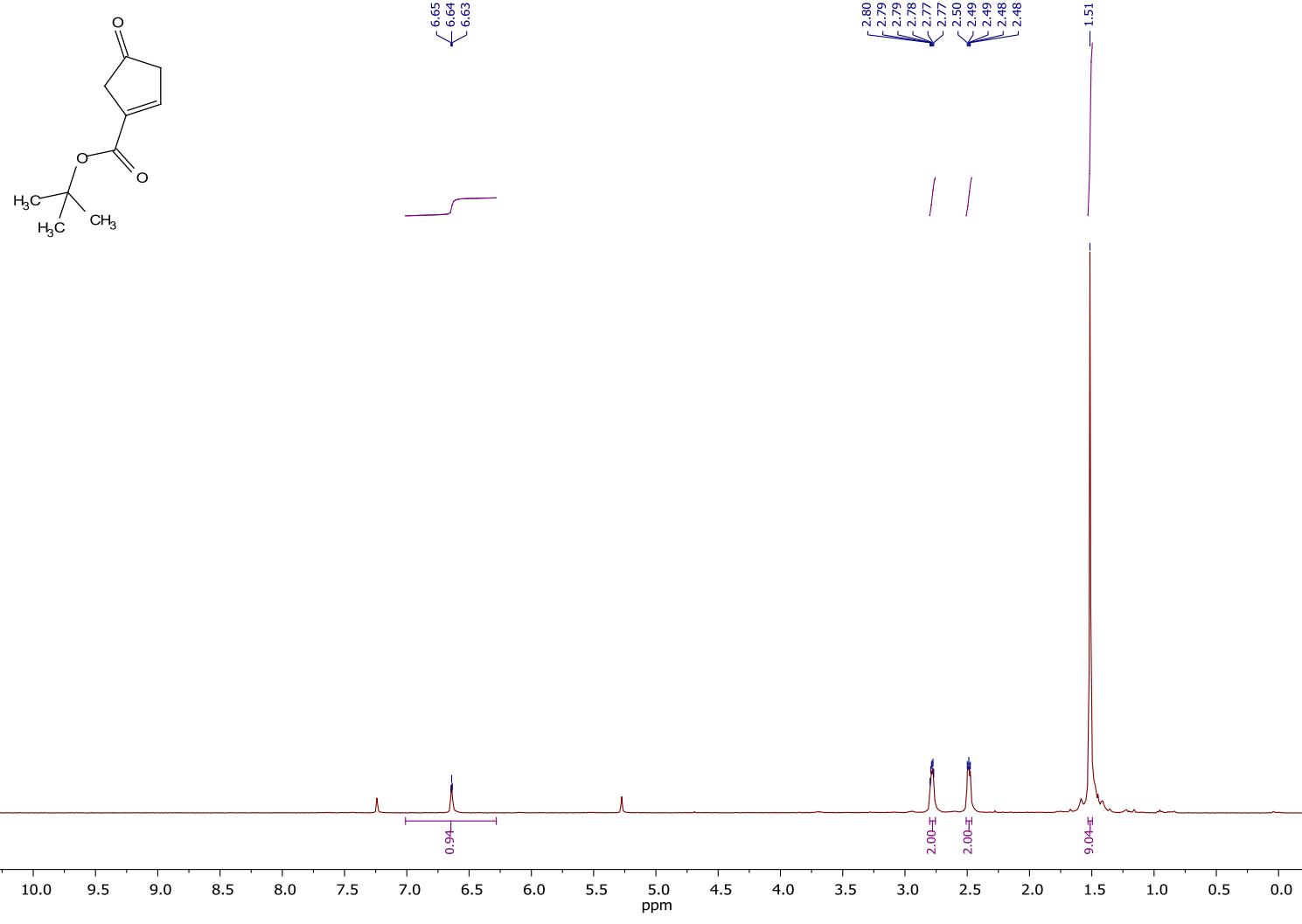
(3*S,4*S**)-*tert*-Butyl 3-bromo-4-((*tert*-butyldimethylsilyl)oxy)cyclopentanecarboxylate (11g)** ^{13}C NMR (CDCl_3)

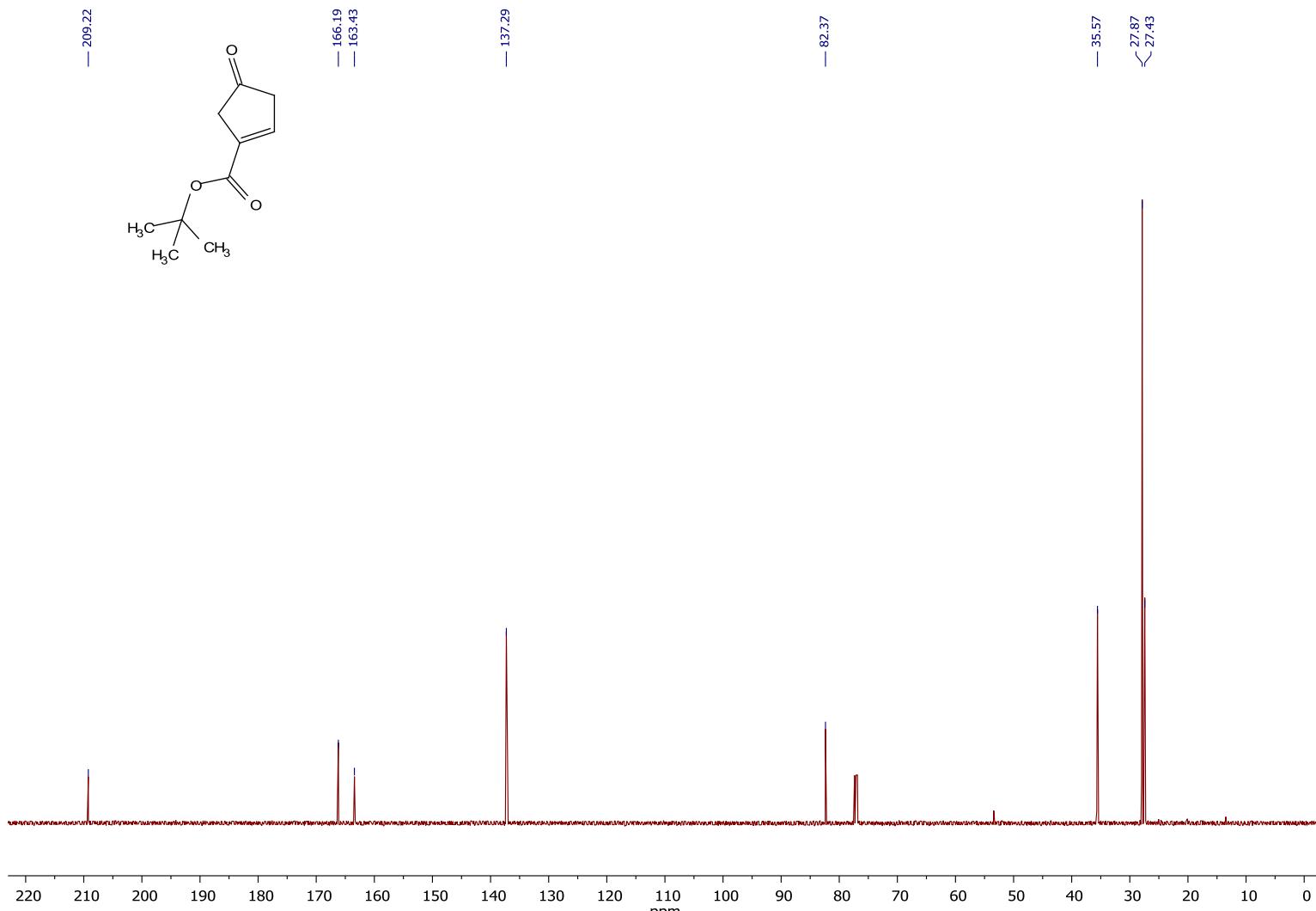


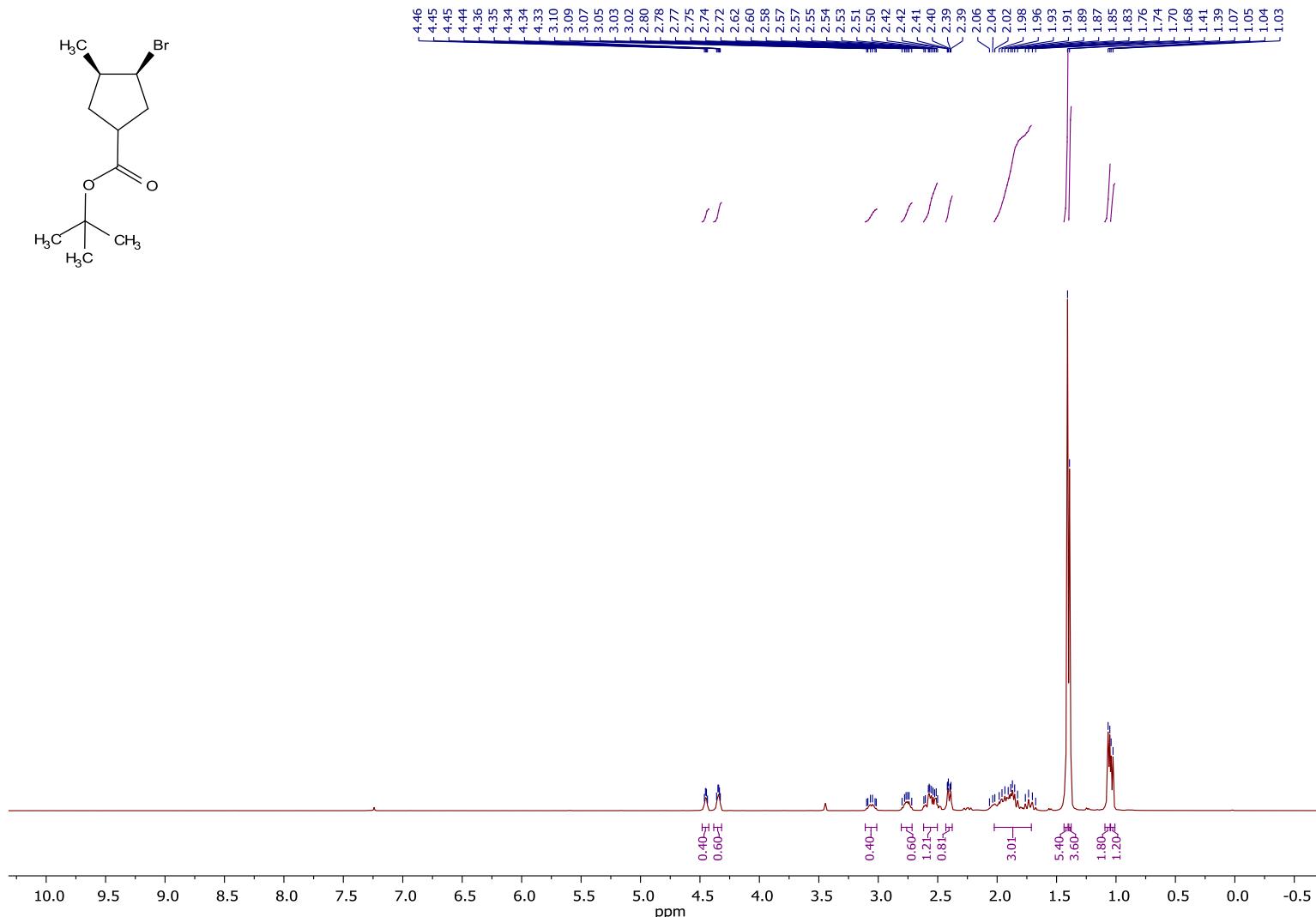
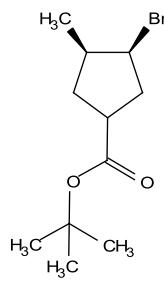
(1*R*^{*},3*S*^{*},4*S*^{*})-*tert*-Butyl 3-((*tert*-butyldimethylsilyl)oxy)bicyclo[2.1.0]pentane-1-carboxylate (12g) ¹H NMR (CDCl_3)



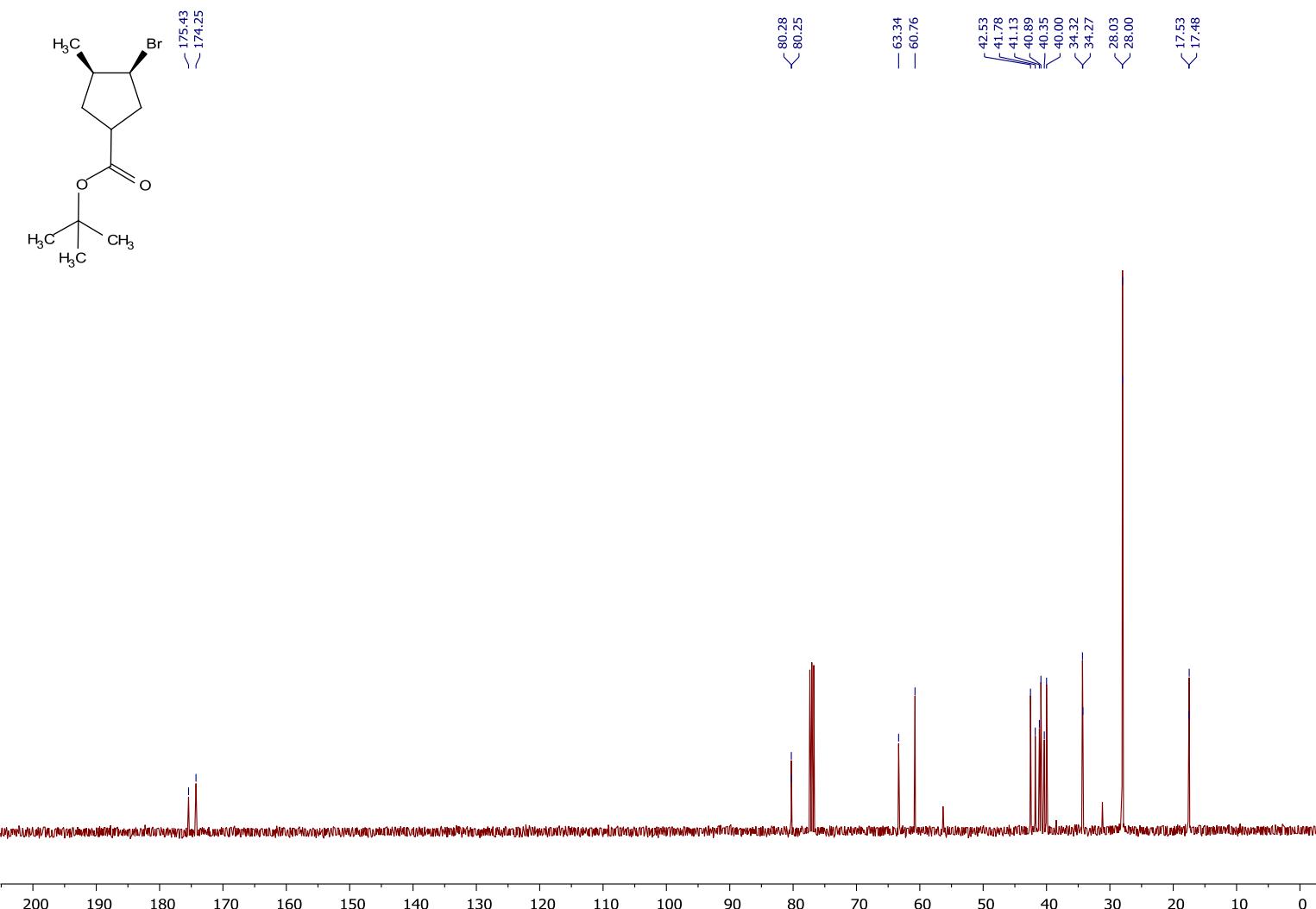
(1*R*^{*,3*S*^{*,4*S*^{*}}})-*tert*-Butyl 3-((*tert*-butyldimethylsilyl)oxy)bicyclo[2.1.0]pentane-1-carboxylate (12g) ^{13}C NMR (CDCl_3)

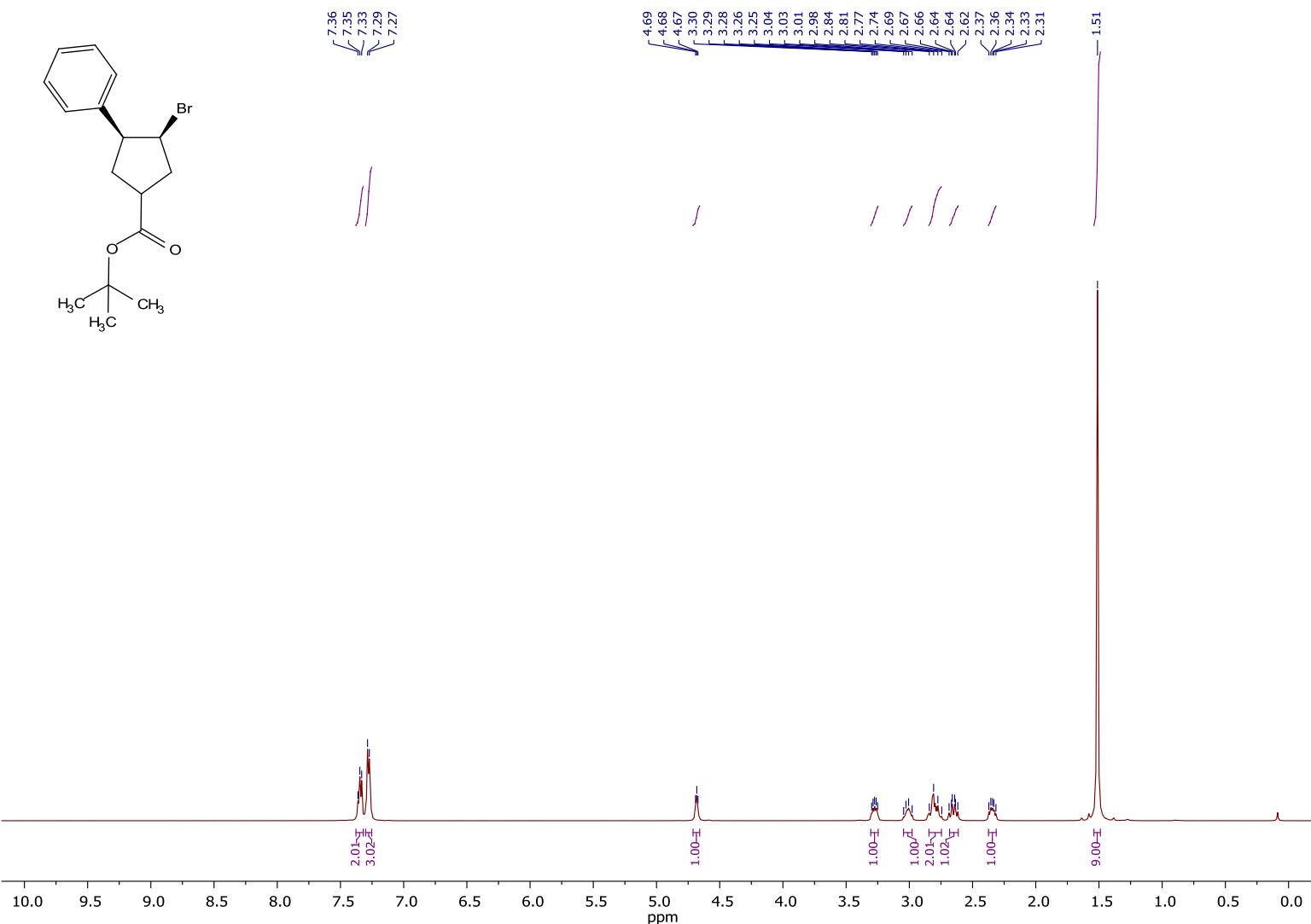




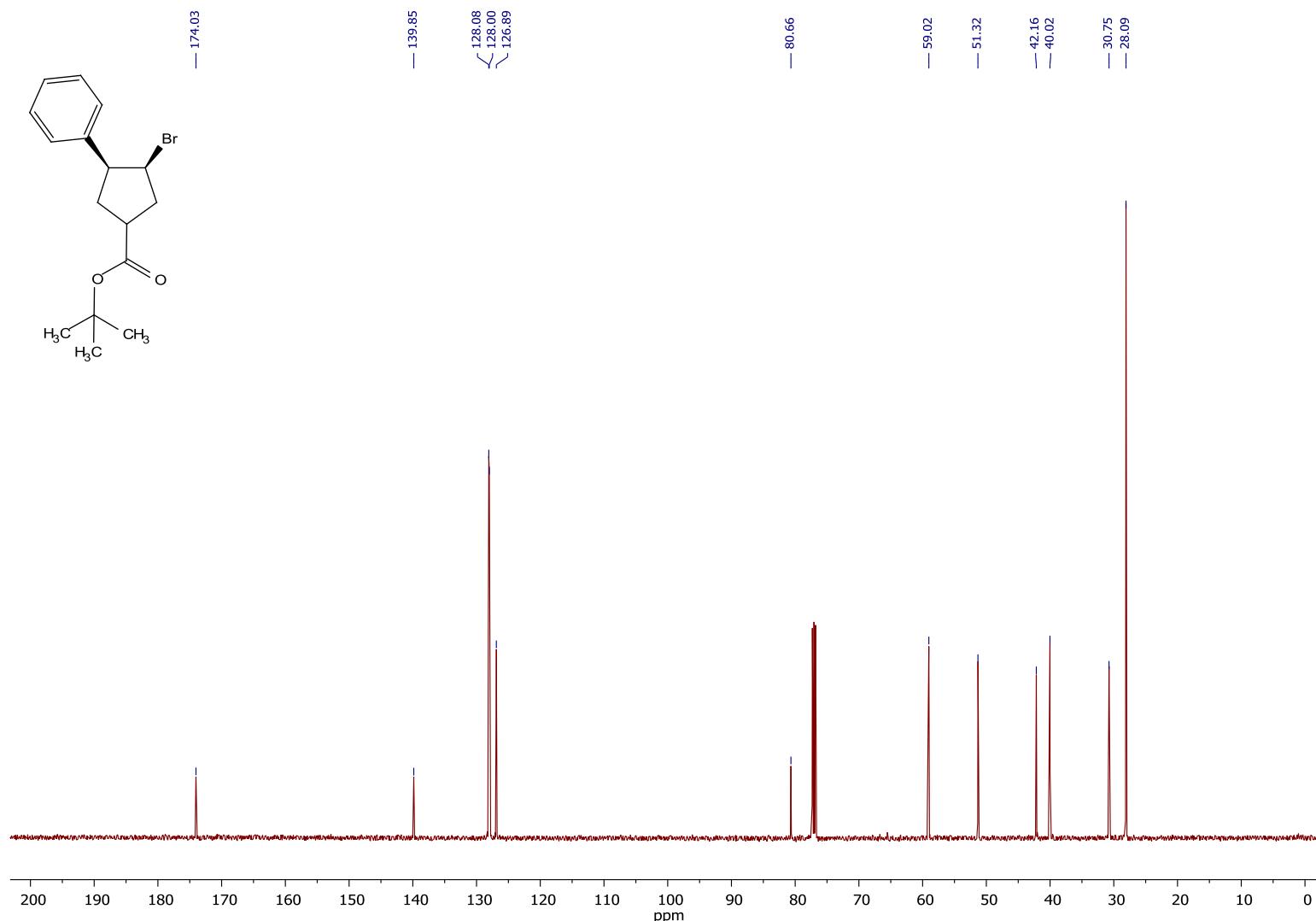


(3*S,4*R**)-tert-Butyl 3-bromo-4-methylcyclopentanecarboxylate (15a)** ¹H NMR (CDCl₃)

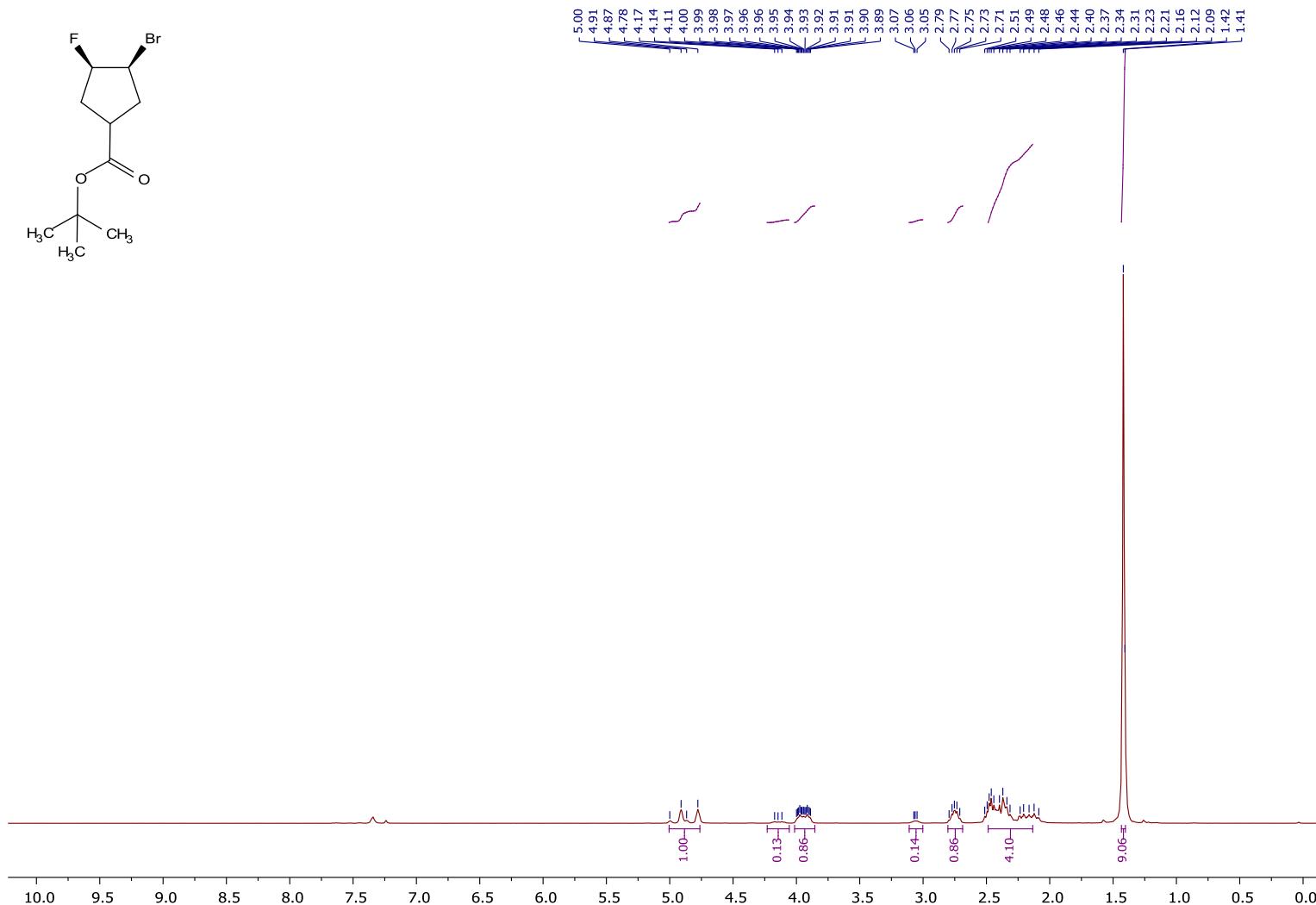
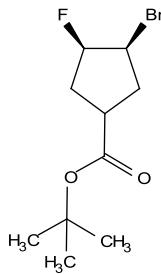




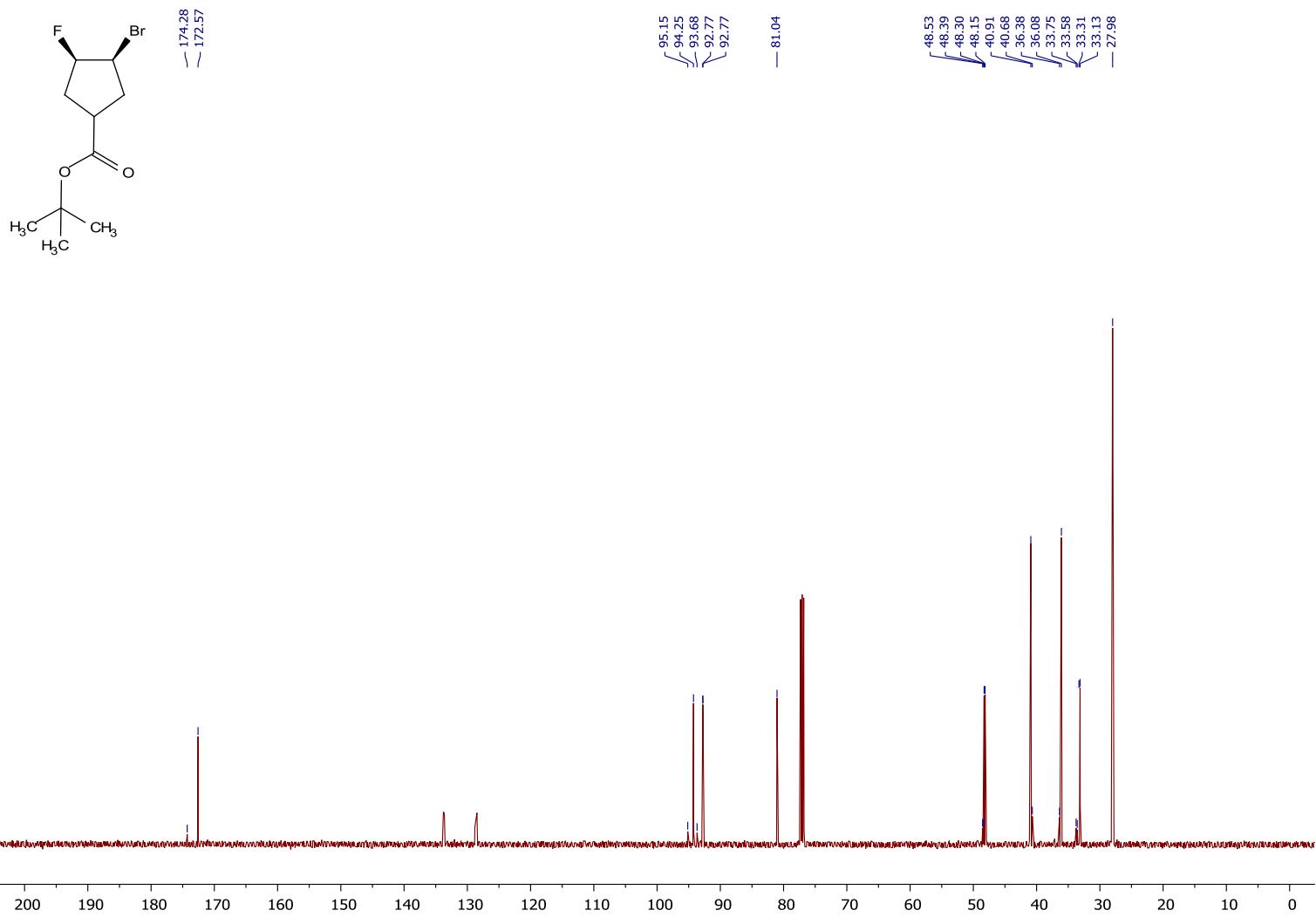
(3*S,4*S**)-tert-Butyl 3-bromo-4-phenylcyclopentanecarboxylate (15b) ^1H NMR (CDCl_3)**



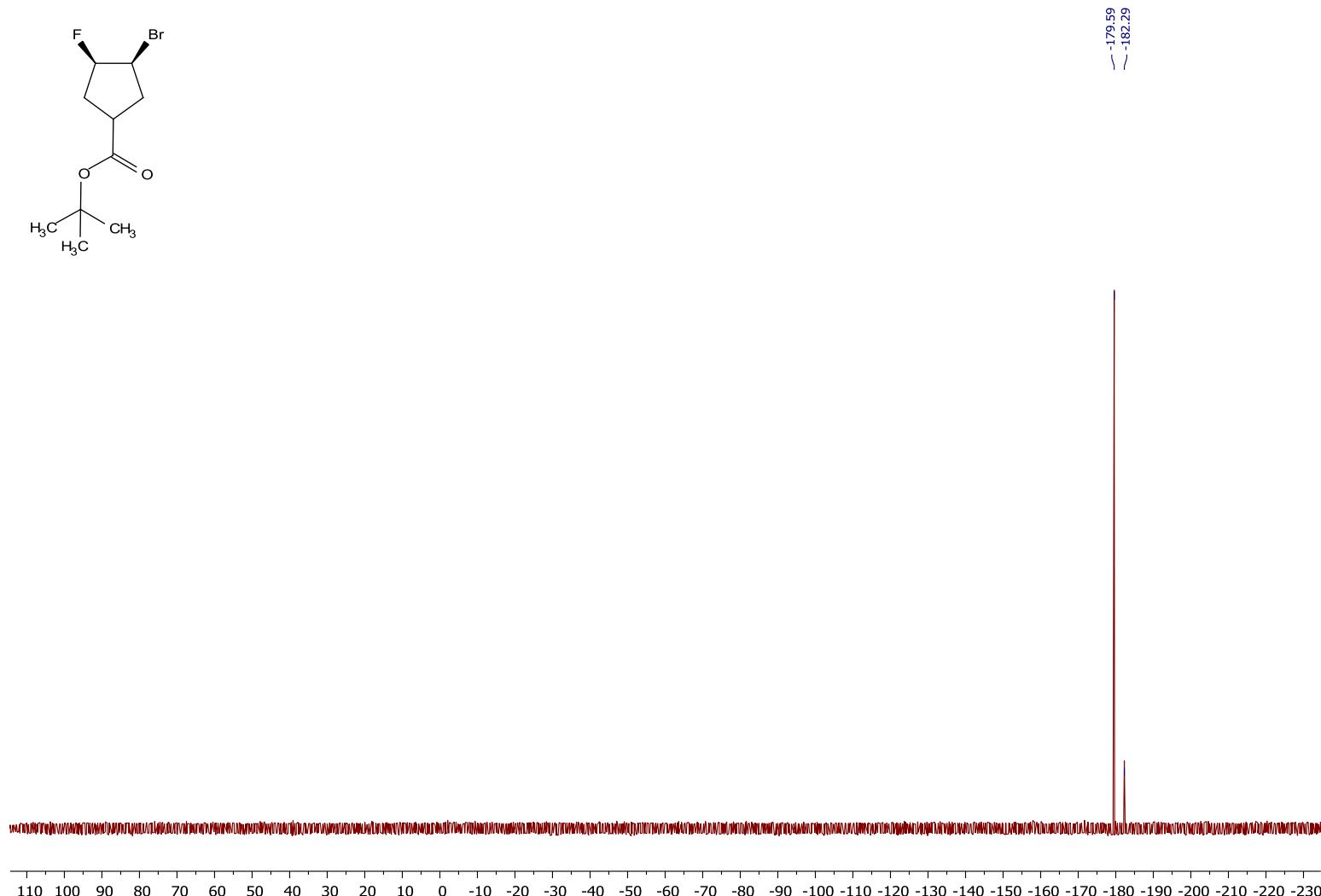
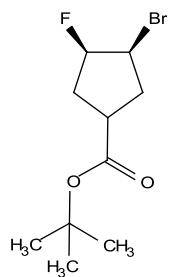
(3S*,4S*)-tert-Butyl 3-bromo-4-phenylcyclopentanecarboxylate (15b) 13C NMR (CDCl₃)



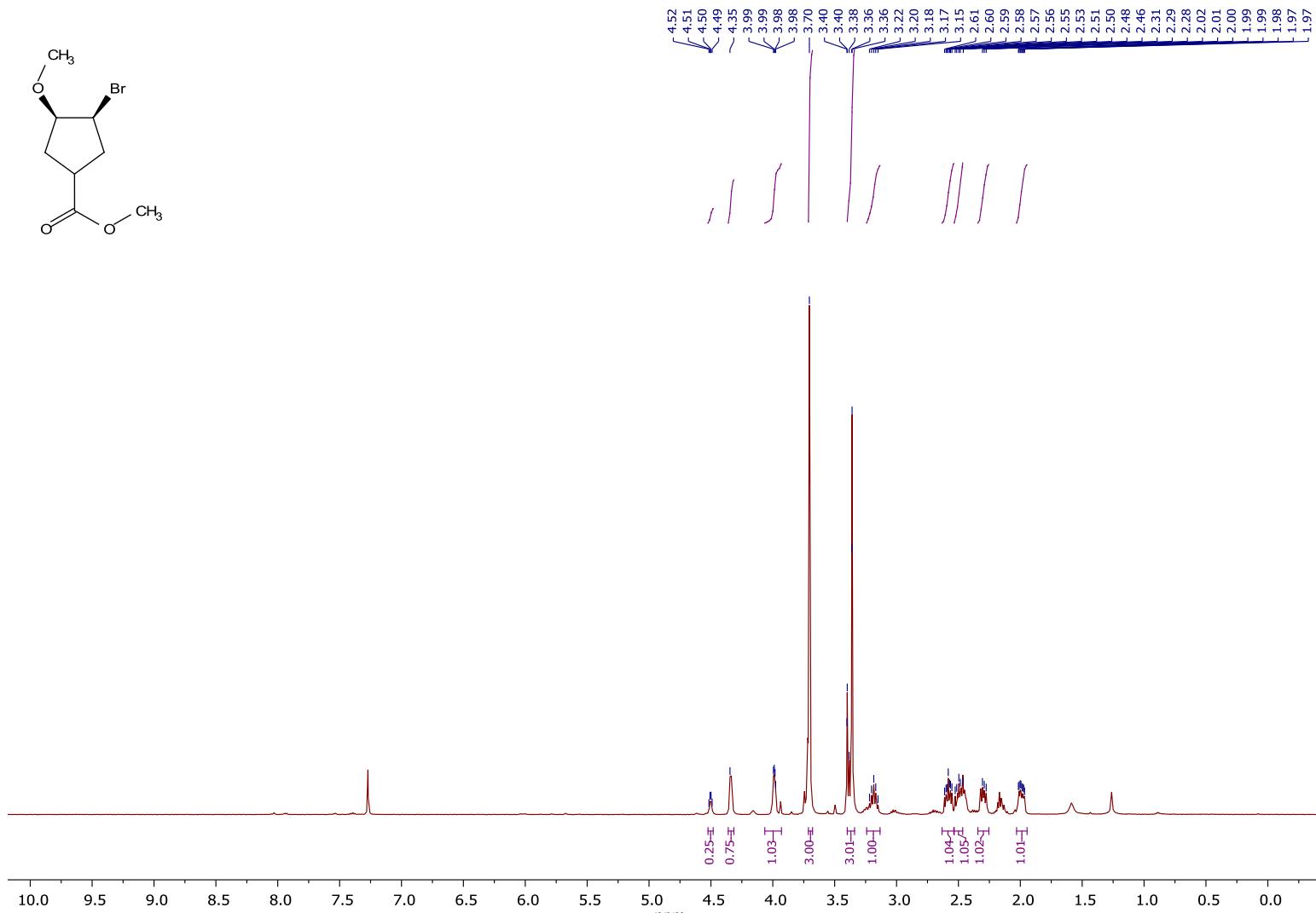
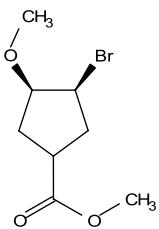
(3*S,4*R**)-*tert*-Butyl 3-bromo-4-fluorocyclopentanecarboxylate (15c)** ¹H NMR (CDCl_3)



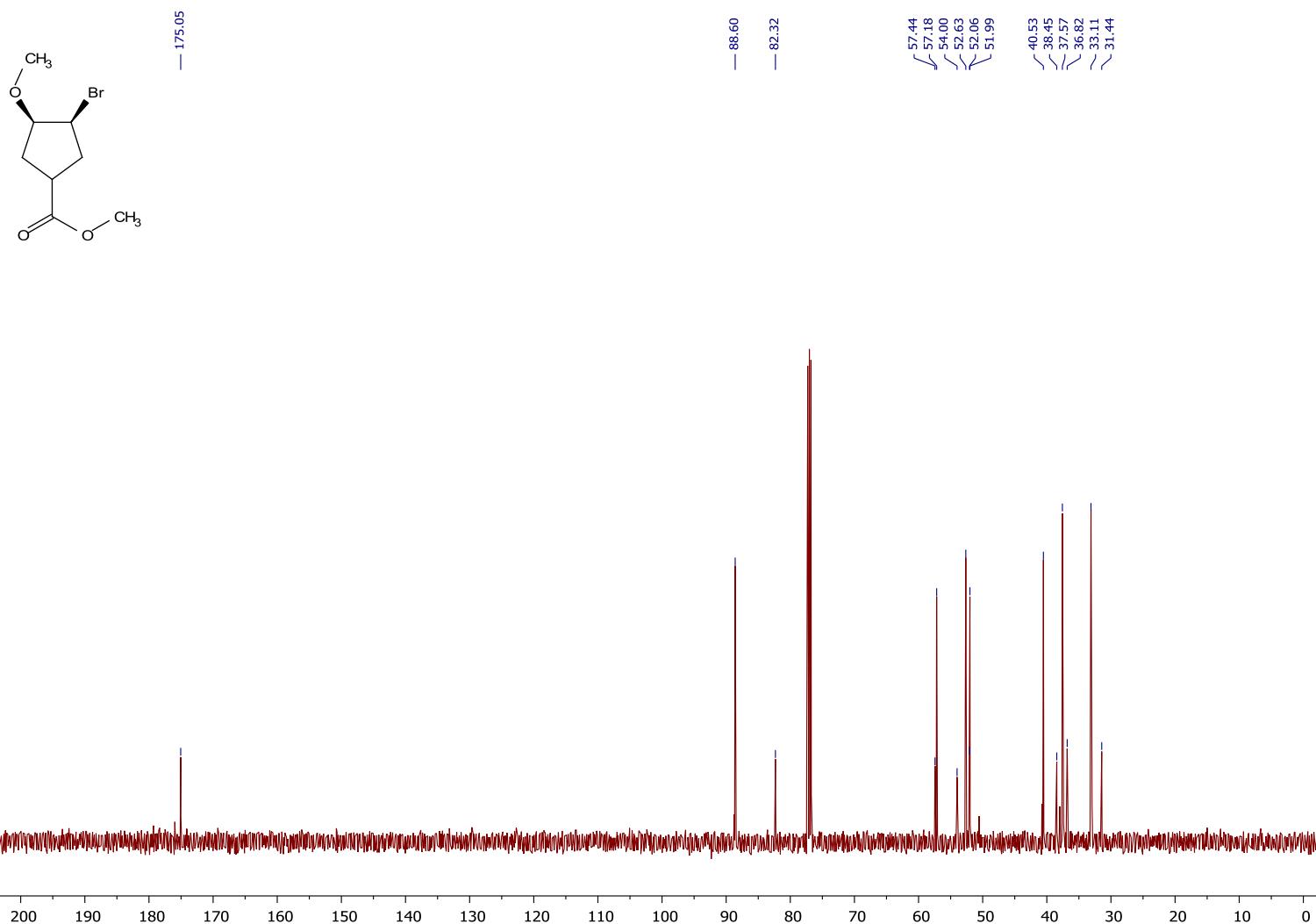
(3*S,4*R**)-*tert*-Butyl 3-bromo-4-fluorocyclopentanecarboxylate (15c)** ^{13}C NMR (CDCl_3)



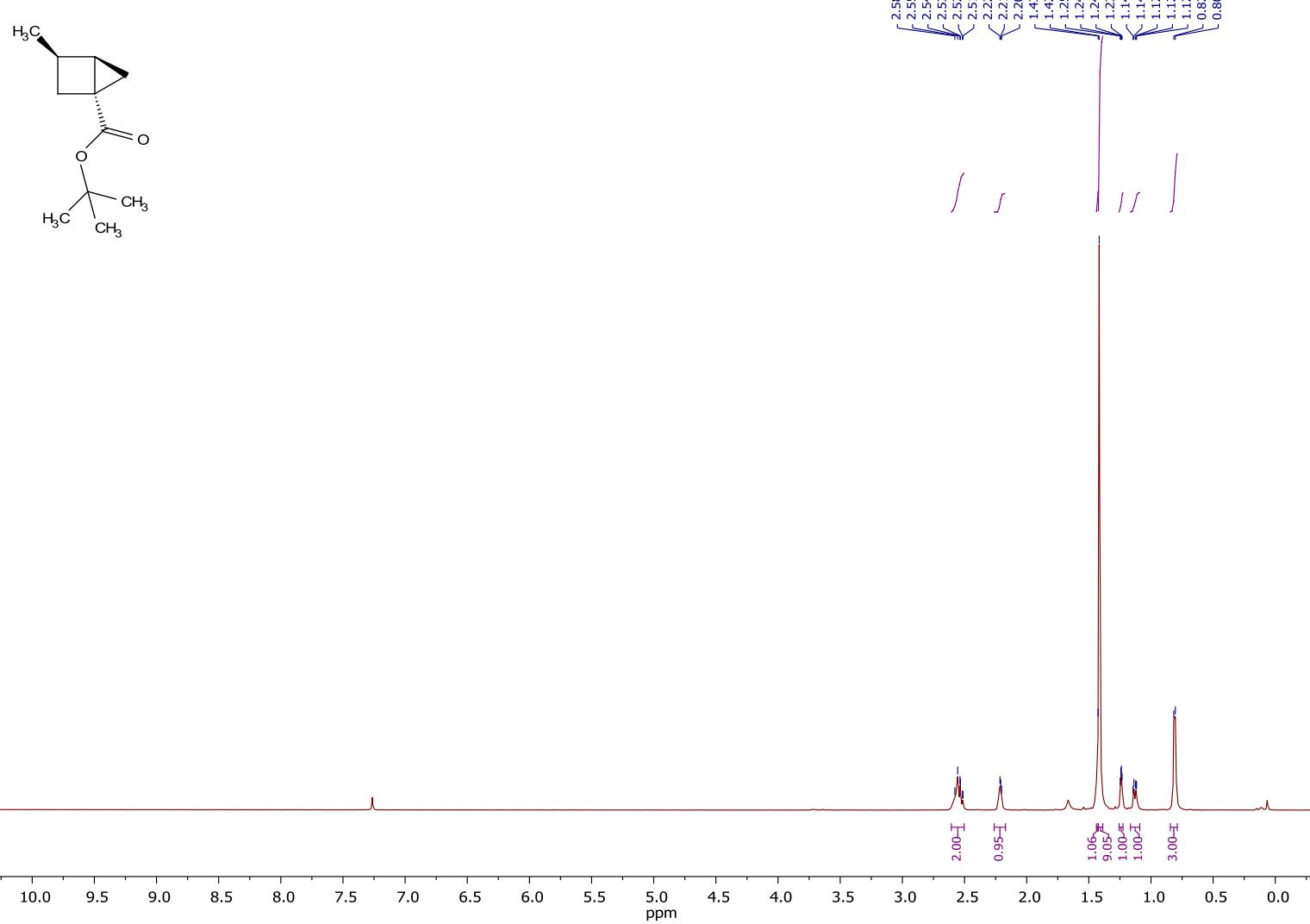
(3*S,4*R**)-*tert*-Butyl 3-bromo-4-fluorocyclopentanecarboxylate (15c)** ^{19}F NMR (CDCl_3)



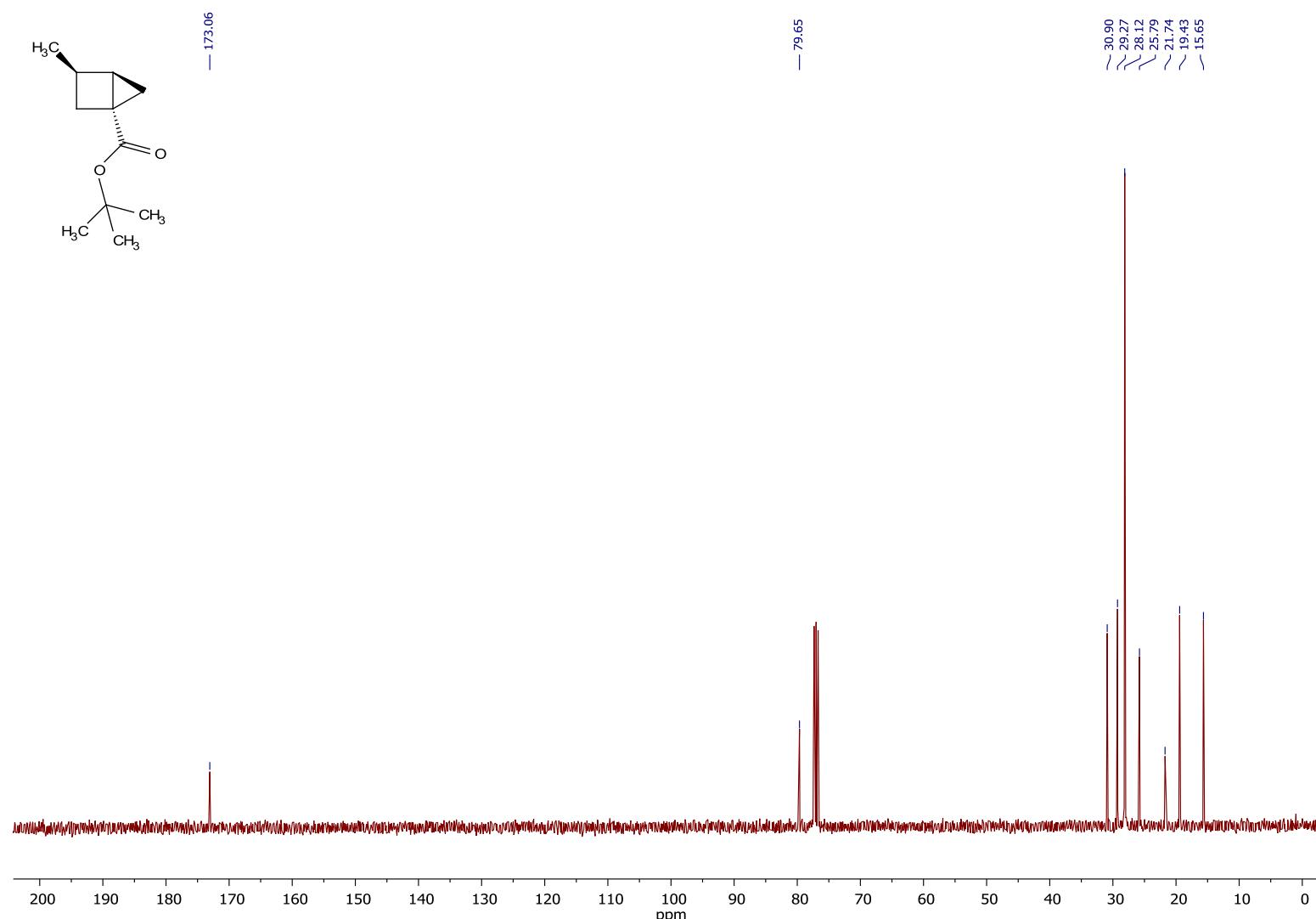
(3*S*^{*},4*R*^{*})-methyl 3-bromo-4-methoxycyclopentanecarboxylate (15d) ¹H NMR (CDCl₃)

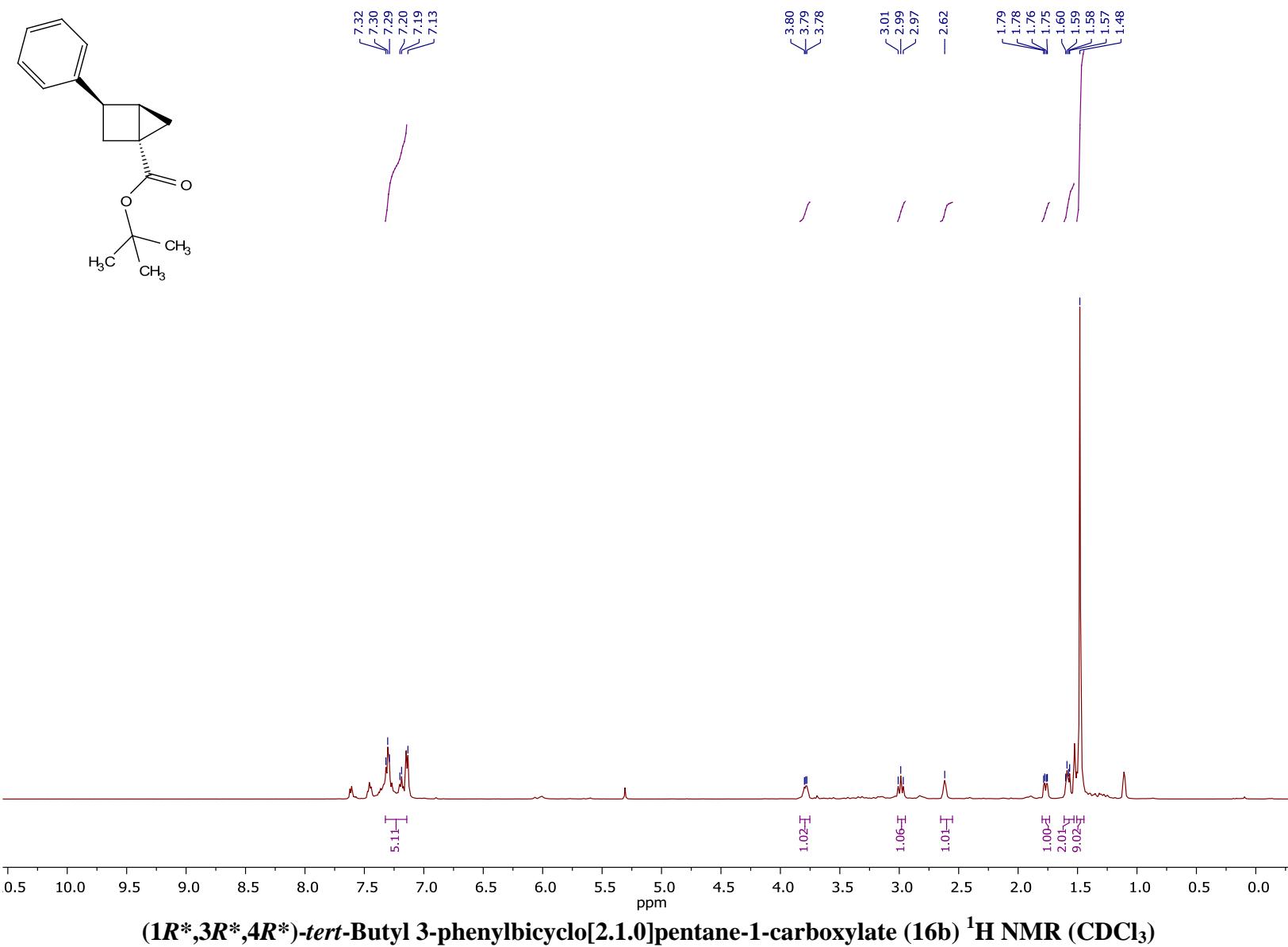


(3*S,4*R**)-methyl 3-bromo-4-methoxycyclopentanecarboxylate (15d) ^{13}C NMR (CDCl_3)**

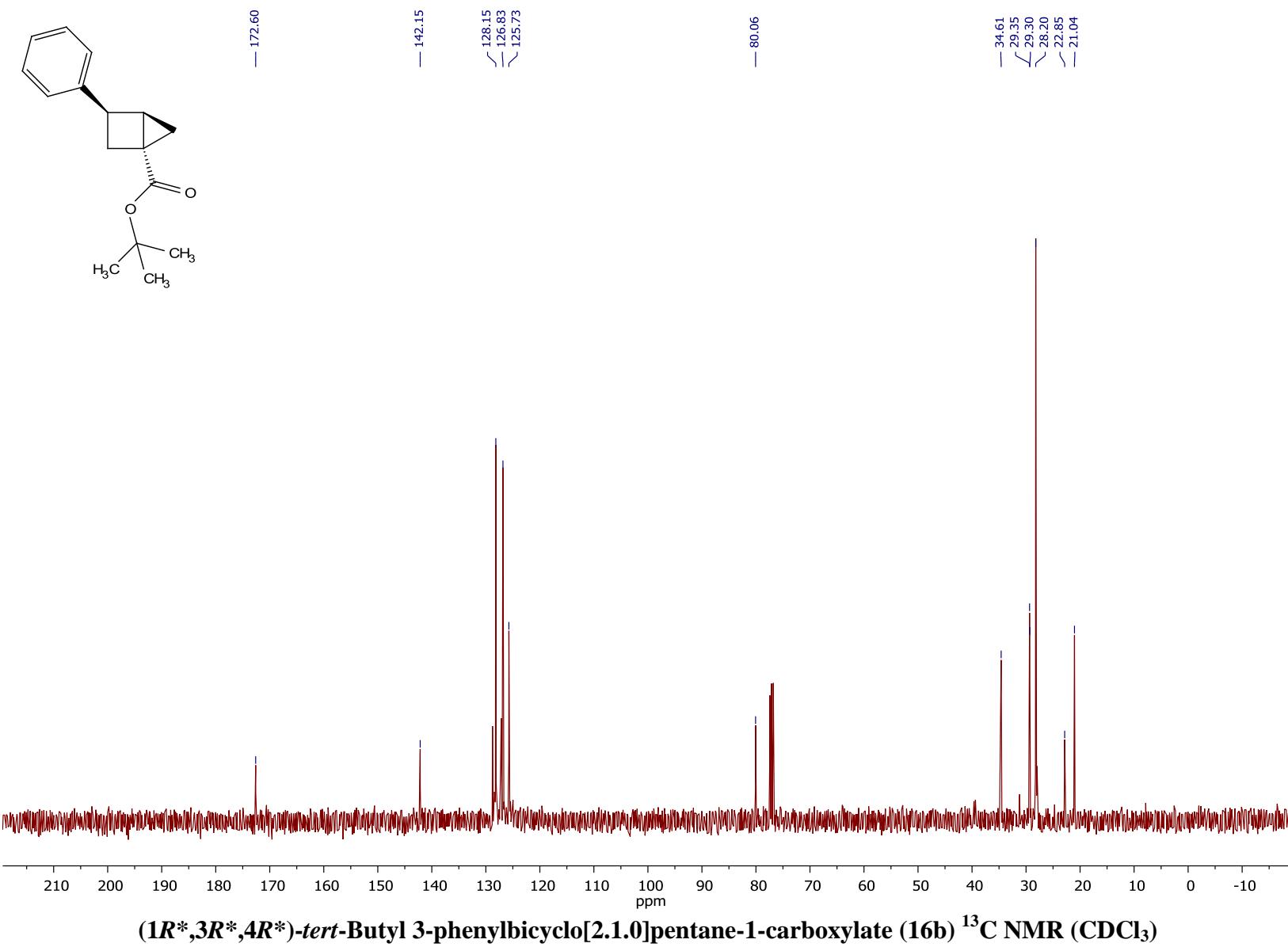


(1*R*^{*,3*R*^{*,4*R*^{*}})-*tert*-Butyl 3-methylbicyclo[2.1.0]pentane-1-carboxylate (16a)} ^1H NMR (CDCl₃)

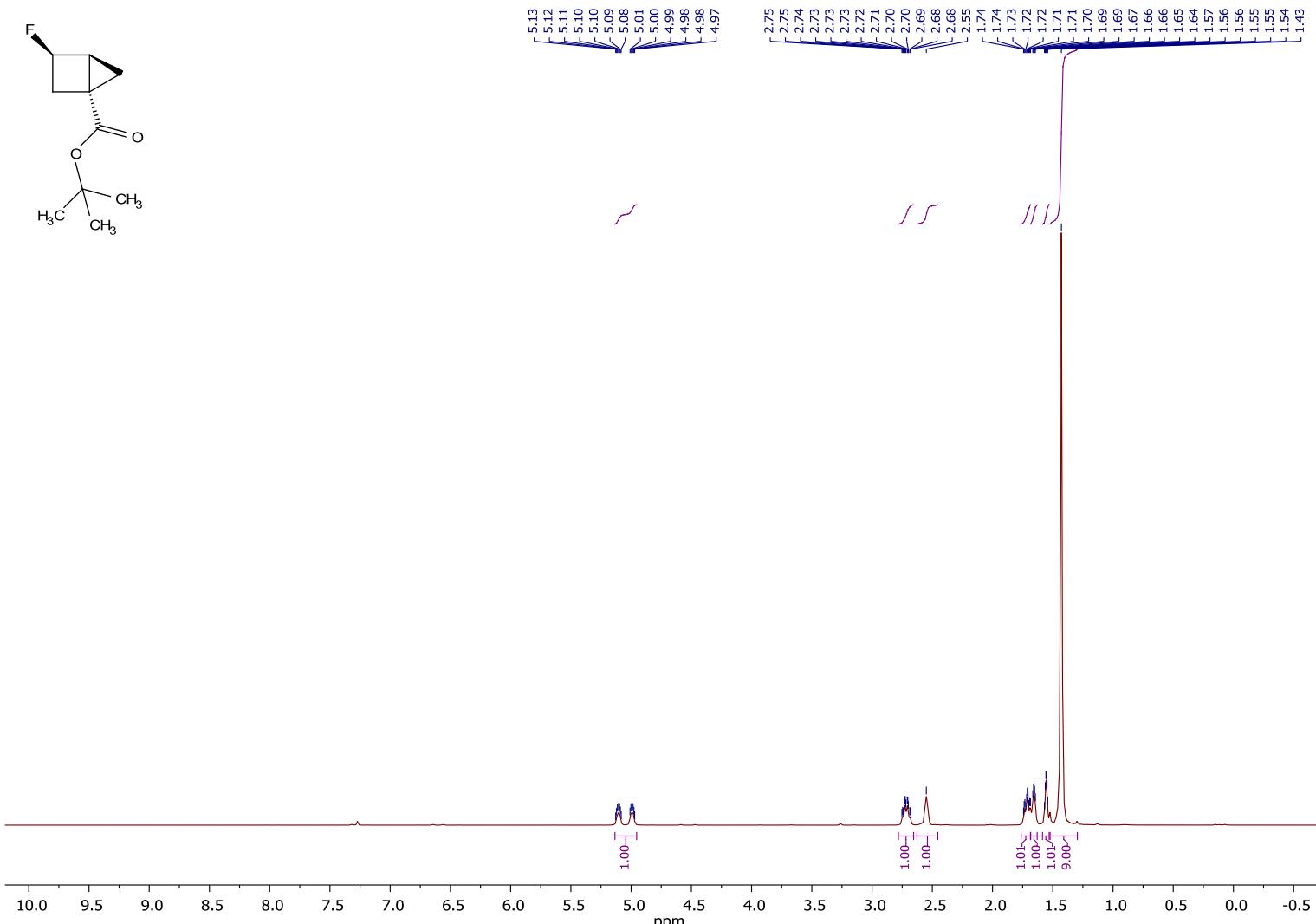
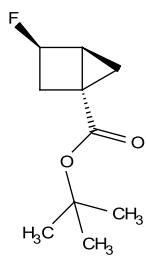




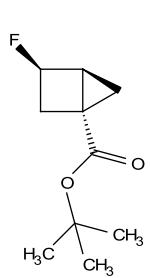
(1*R*^{*,3*R*^{*,4*R*^{*}})-*tert*-Butyl 3-phenylbicyclo[2.1.0]pentane-1-carboxylate (16b) ¹H NMR (CDCl₃)}



(*1R*,3R*,4R**)-*tert*-Butyl 3-phenylbicyclo[2.1.0]pentane-1-carboxylate (**16b**) ^{13}C NMR (CDCl₃)



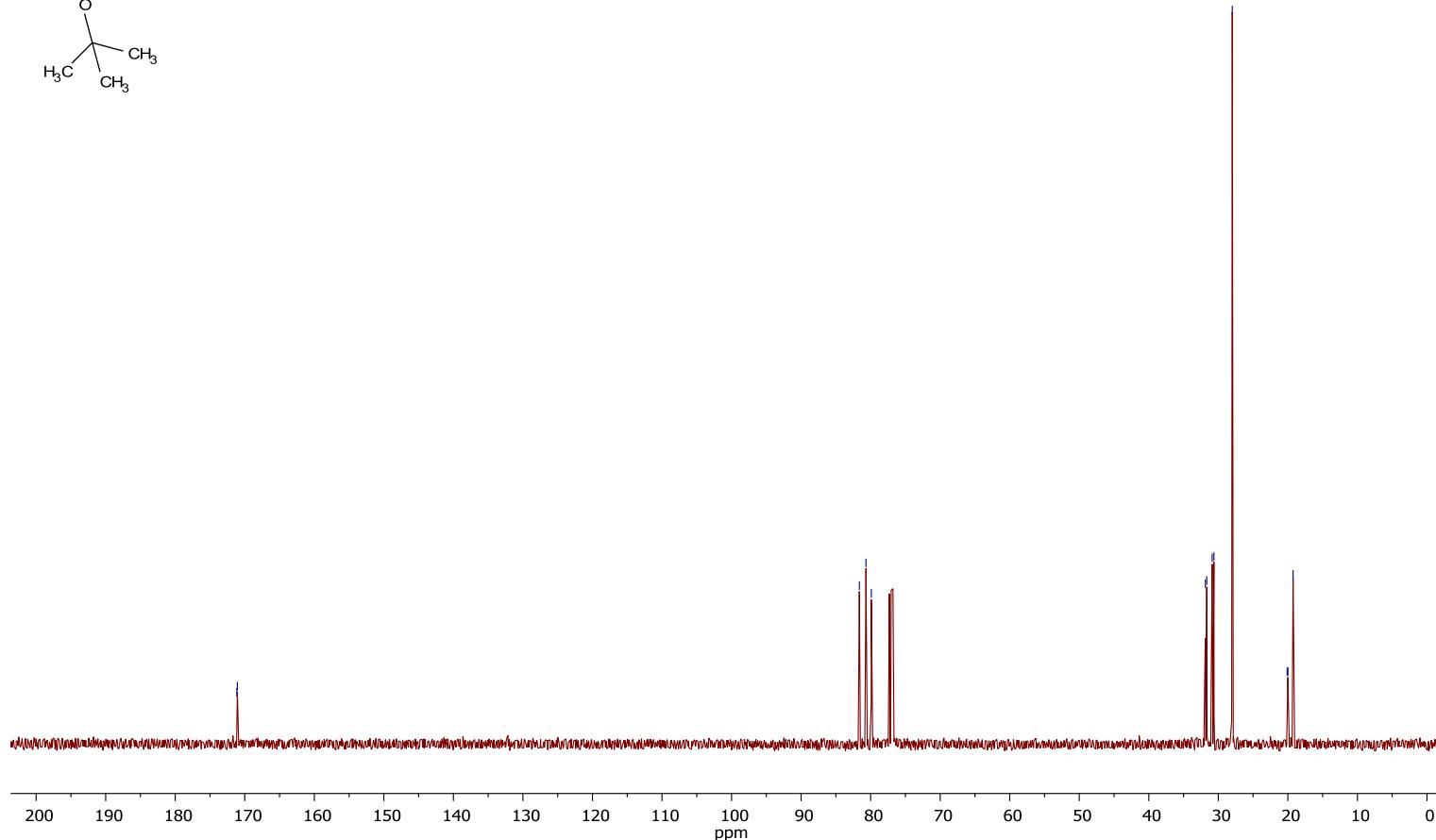
(1R*,3R*,4S*)-tert-Butyl 3-fluorobicyclo[2.1.0]pentane-1-carboxylate (16c) ¹H NMR (CDCl_3)



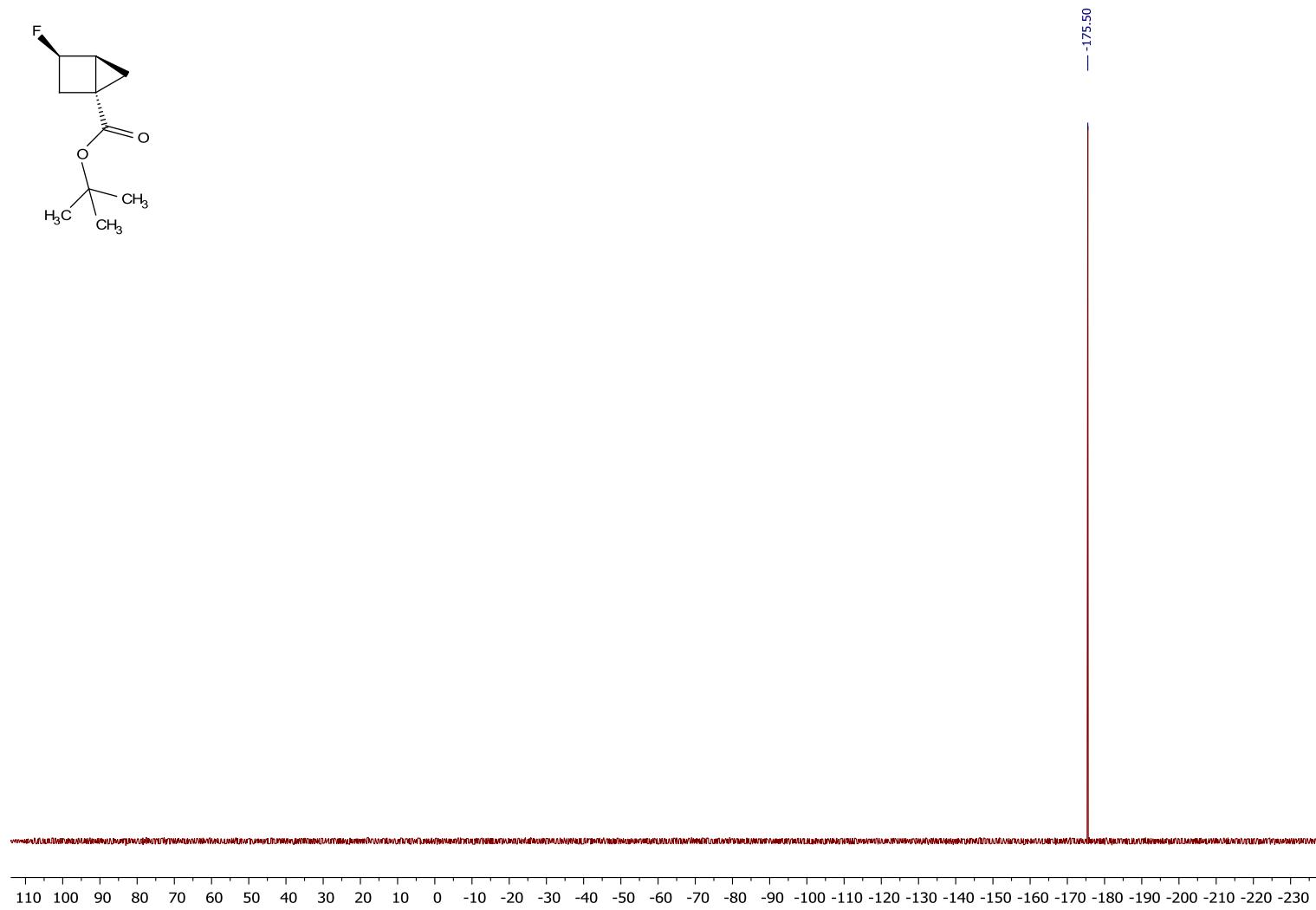
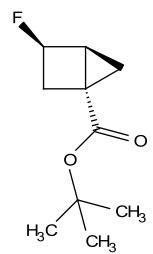
171.14
171.06

81.62
80.67
79.90

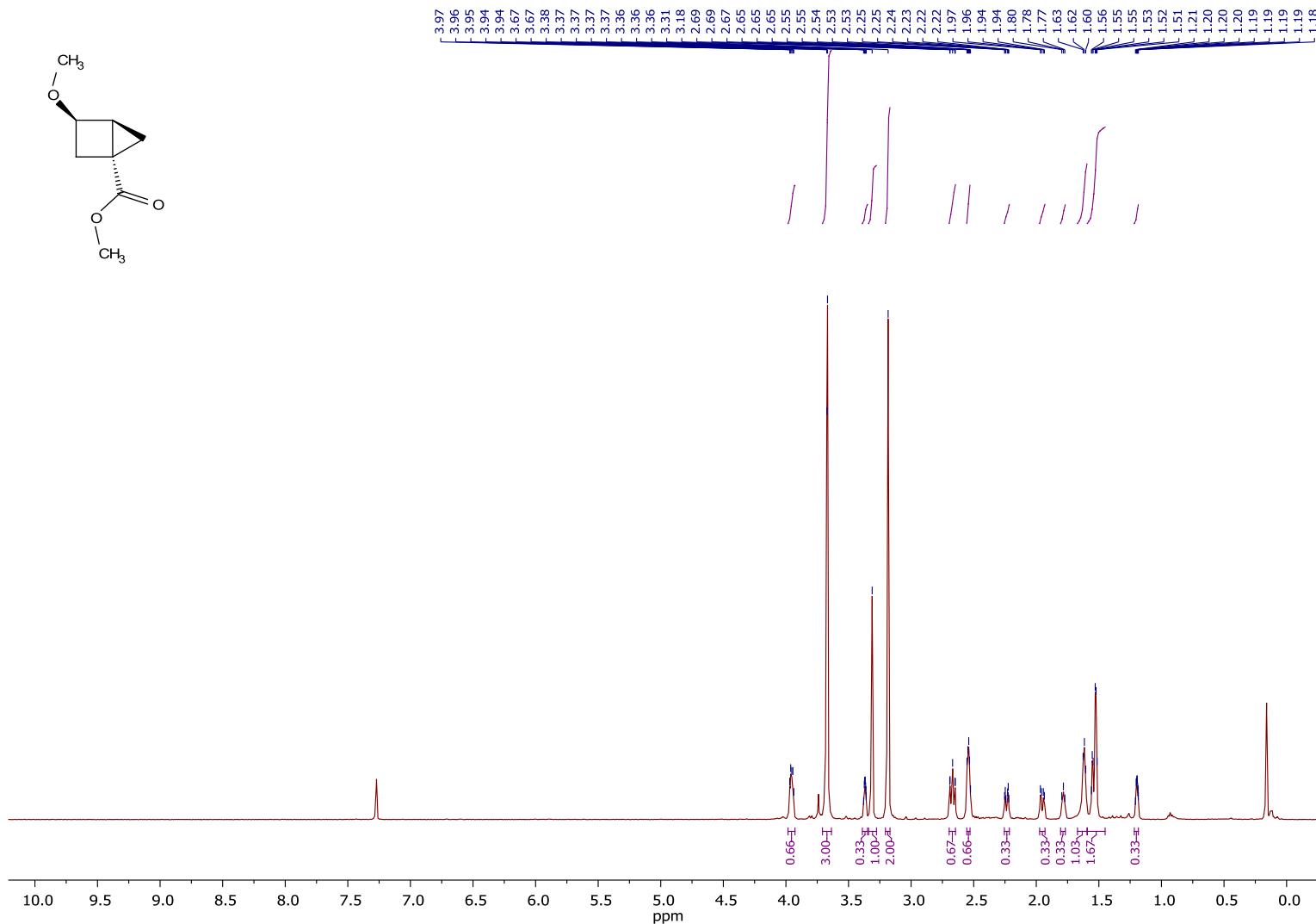
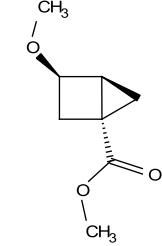
31.87
31.67
30.89
30.64
27.99
20.10
20.00
19.25



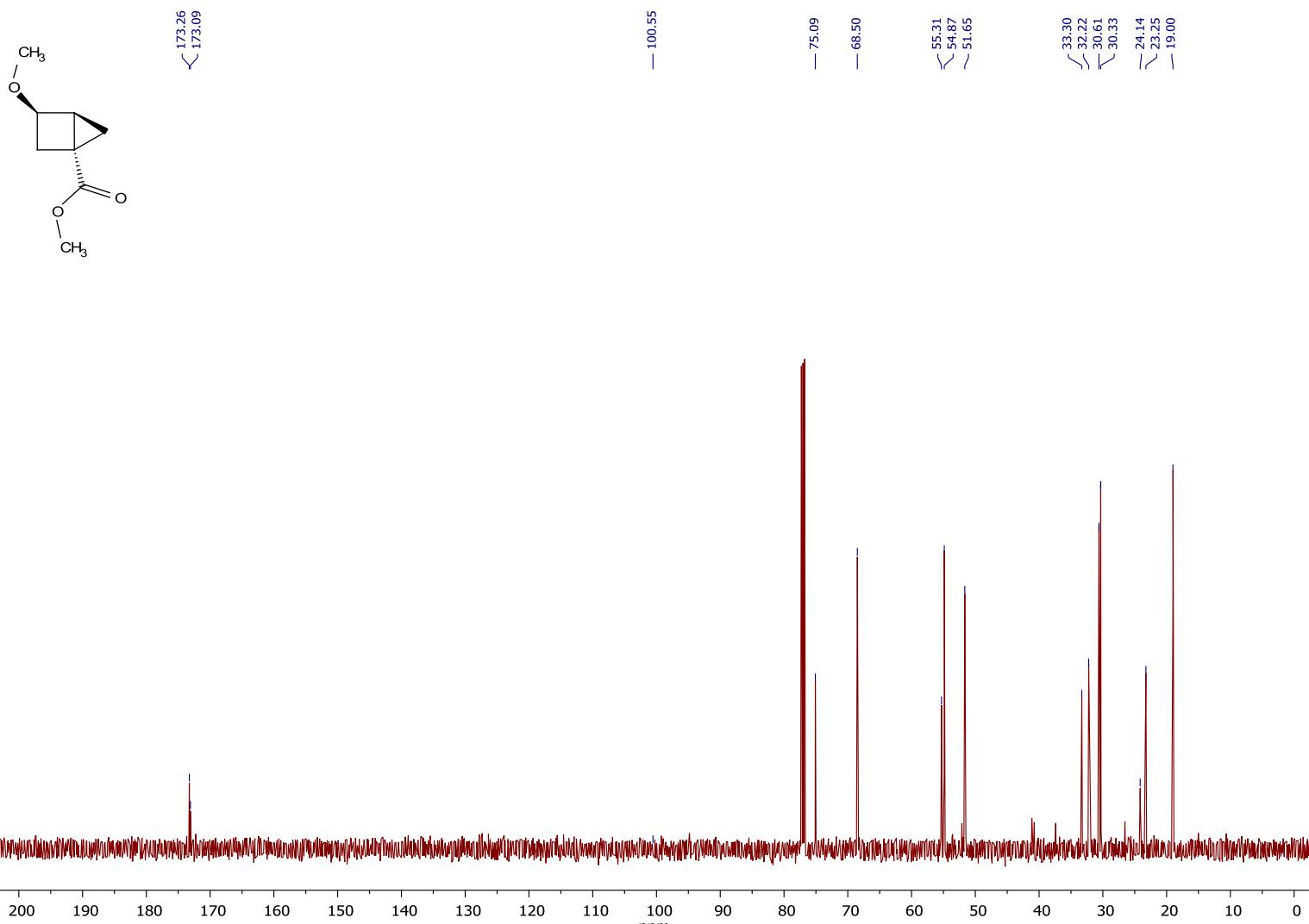
(1*R*^{*,3*R*^{*,4*S*^{*}}-tert-Butyl 3-fluorobicyclo[2.1.0]pentane-1-carboxylate (16c) ¹³C NMR (CDCl₃)}



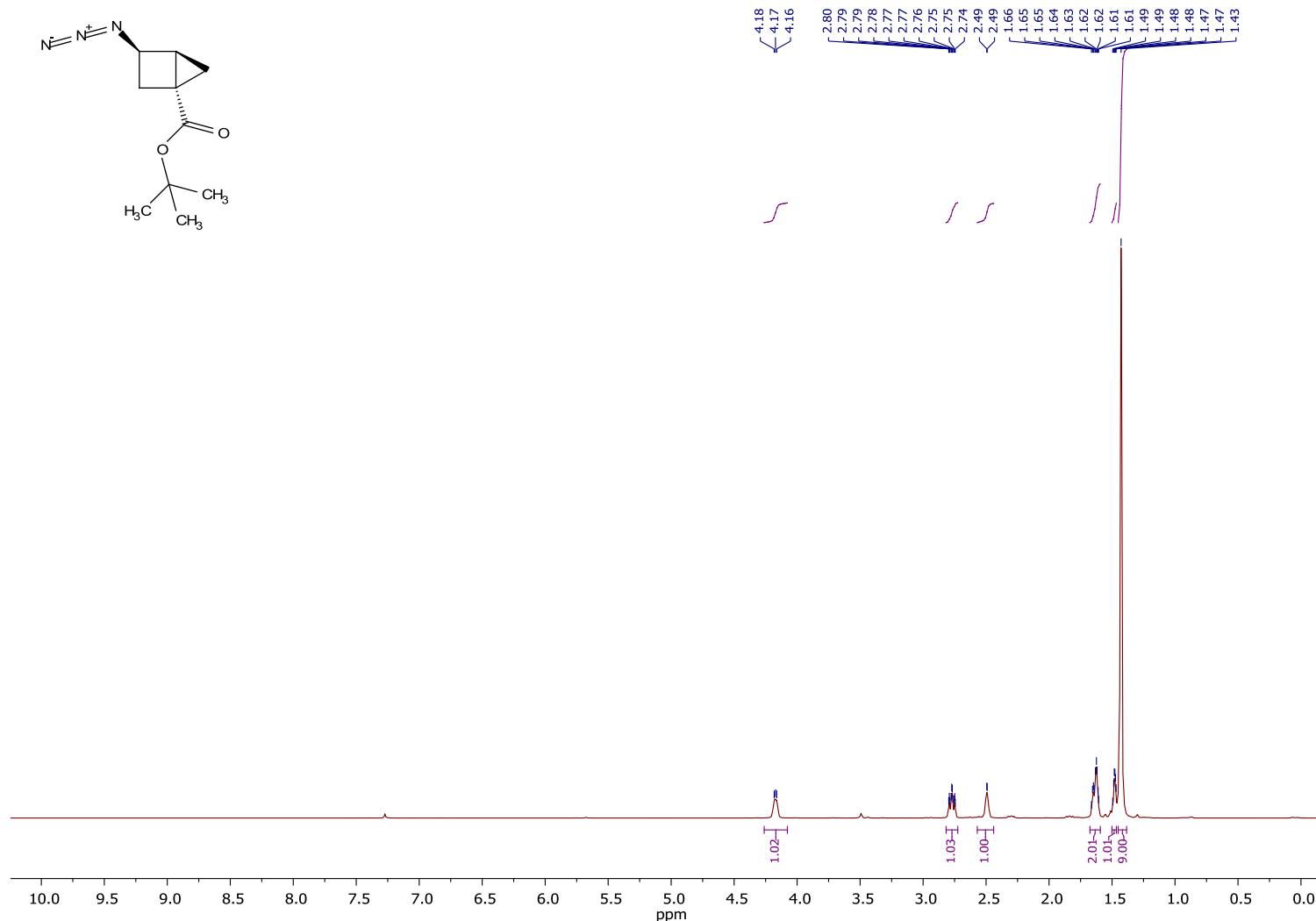
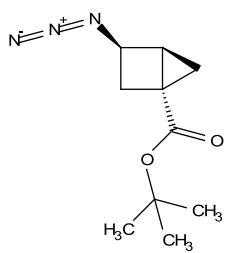
(1*R*^{*,3*R*^{*,4*S*^{*}}})-*tert*-Butyl 3-fluorobicyclo[2.1.0]pentane-1-carboxylate (16c) ^{19}F NMR (CDCl_3)



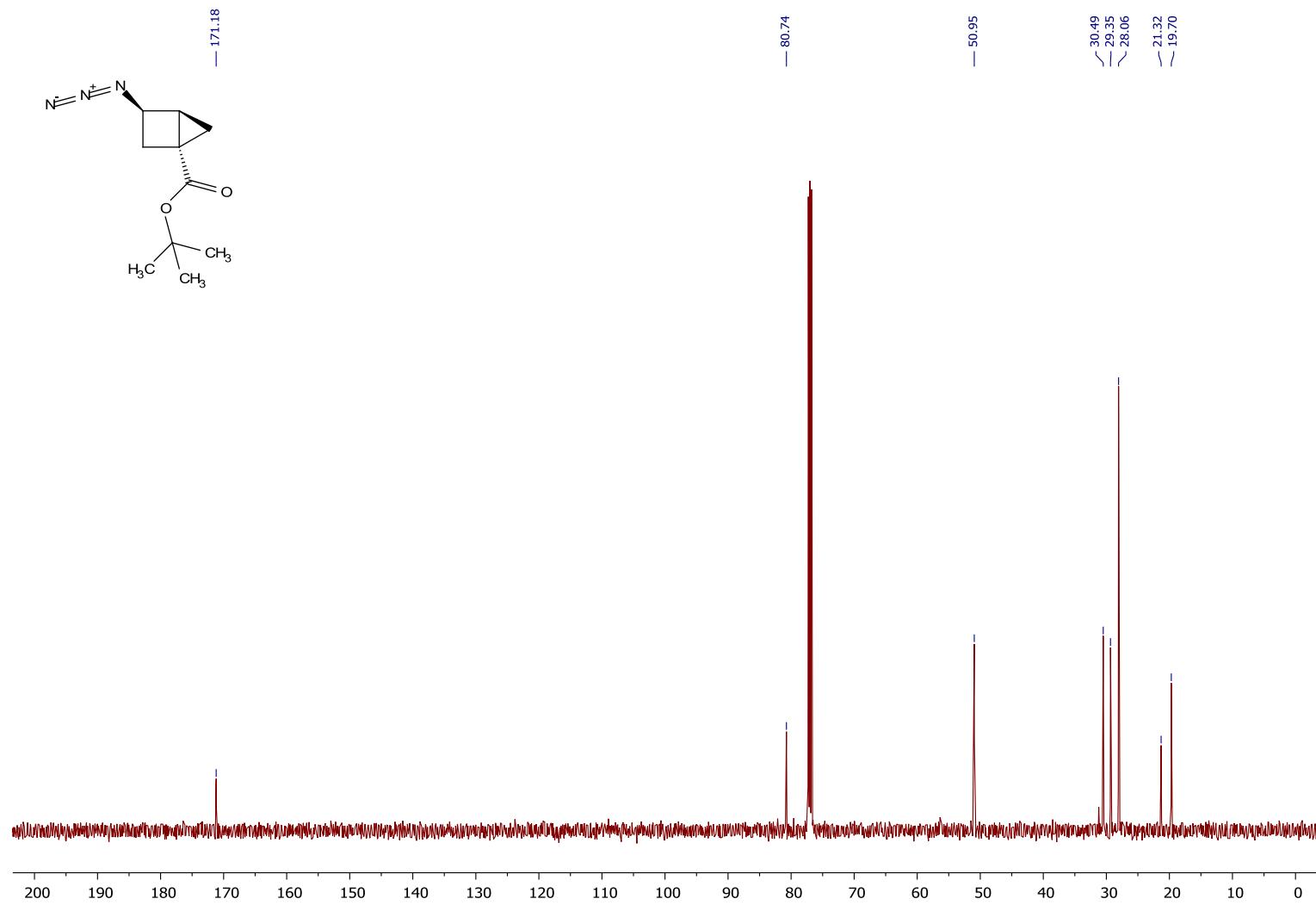
(1*R*^{*},3*R*^{*},4*S*^{*})-Methyl 3-methoxybicyclo[2.1.0]pentane-1-carboxylate (16d) ¹H NMR (CDCl_3)



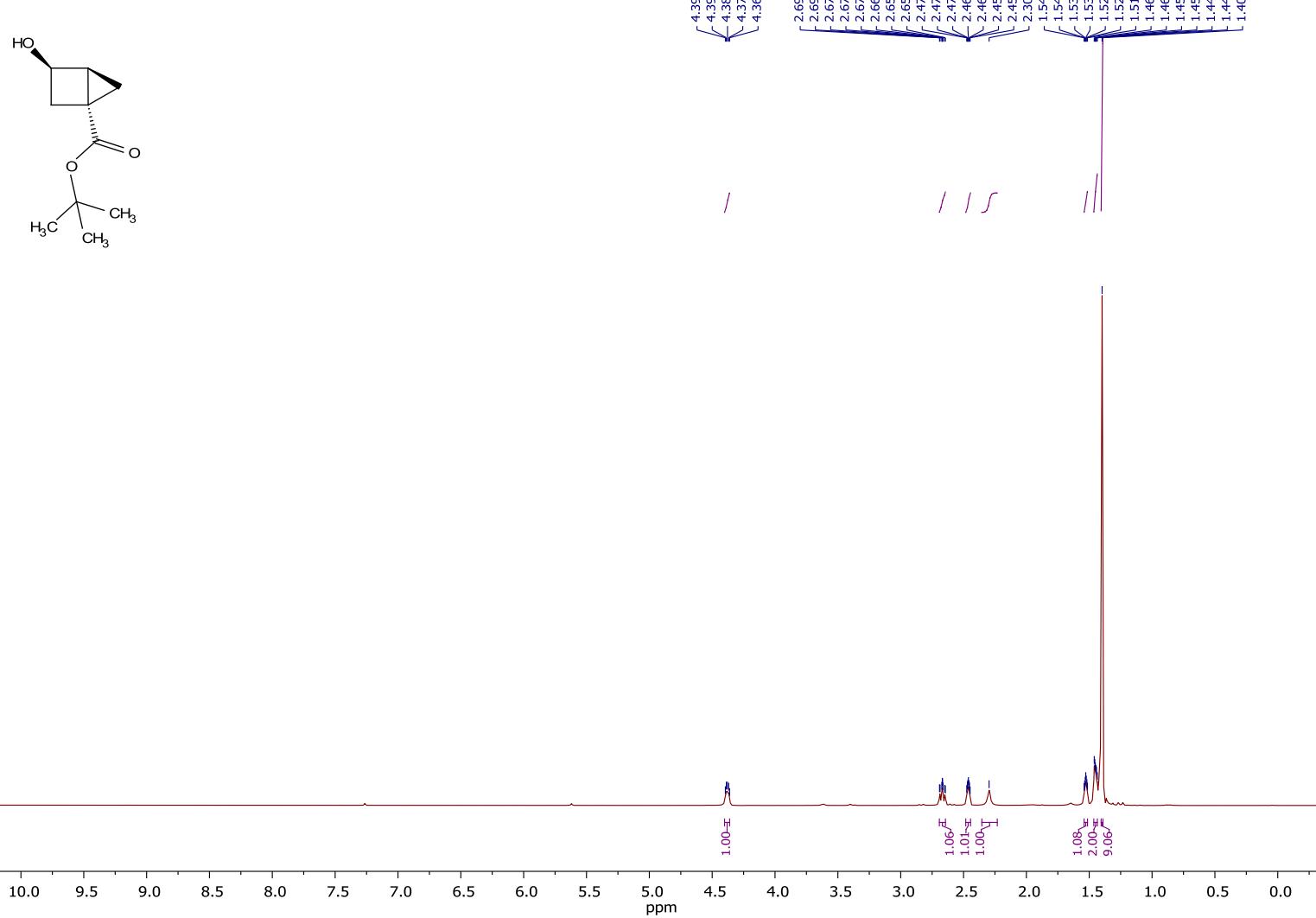
(1*R,3*R**,4*S**)-Methyl 3-methoxybicyclo[2.1.0]pentane-1-carboxylate (16d) ^{13}C NMR (CDCl_3)**



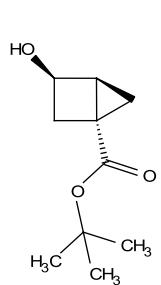
(1*R,3*R**,4*S**)-tert-Butyl 3-azidobicyclo[2.1.0]pentane-1-carboxylate (16f)** ¹H NMR (CDCl₃)



$(1R^*, 3R^*, 4S^*)$ -*tert*-Butyl 3-azidobicyclo[2.1.0]pentane-1-carboxylate (**16f**) ^{13}C NMR (CDCl_3)



(1R*,3R*,4S*)-tert-Butyl 3-hydroxybicyclo[2.1.0]pentane-1-carboxylate (16g) ¹H NMR (CDCl₃)



— 172.40

— 80.39

— 60.94

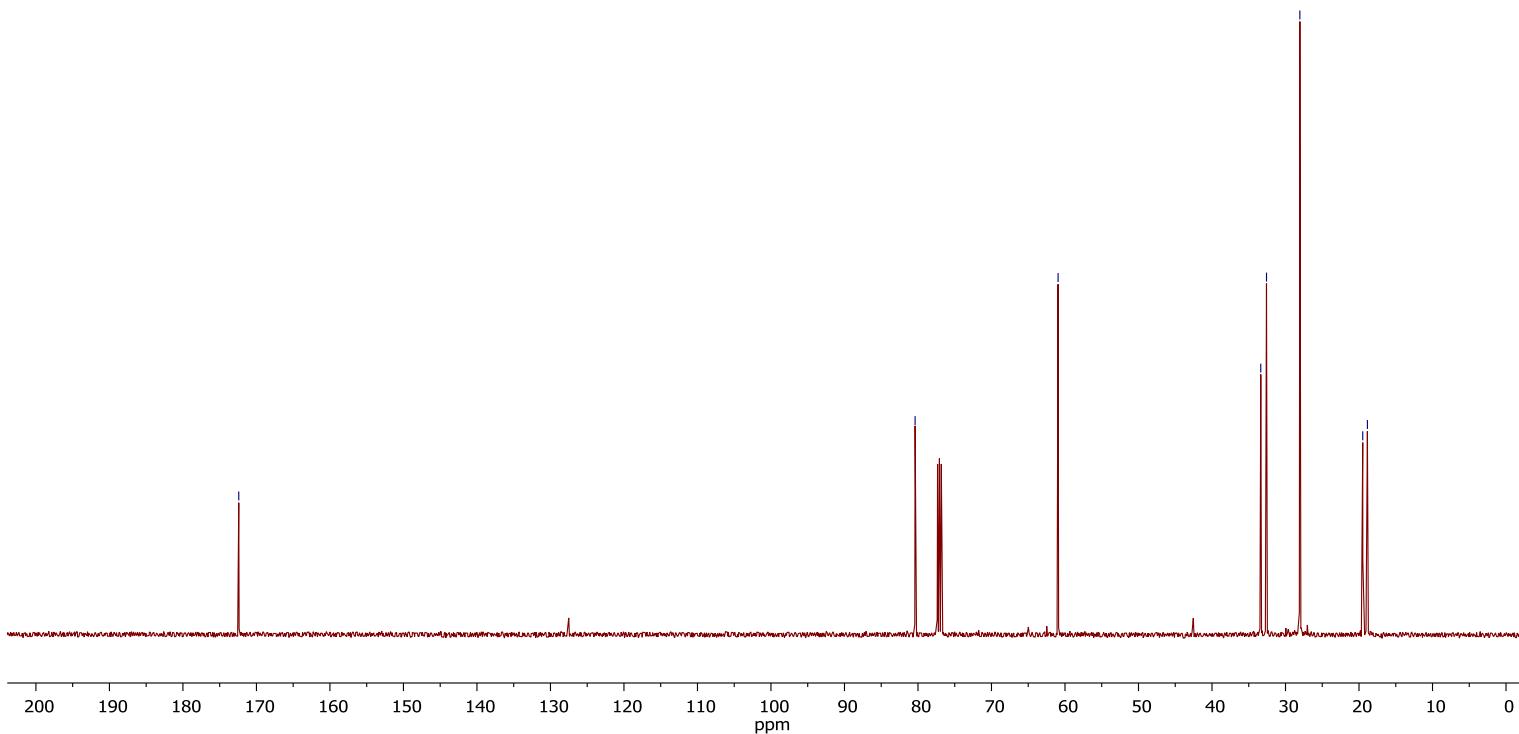
— 33.37

— 32.58

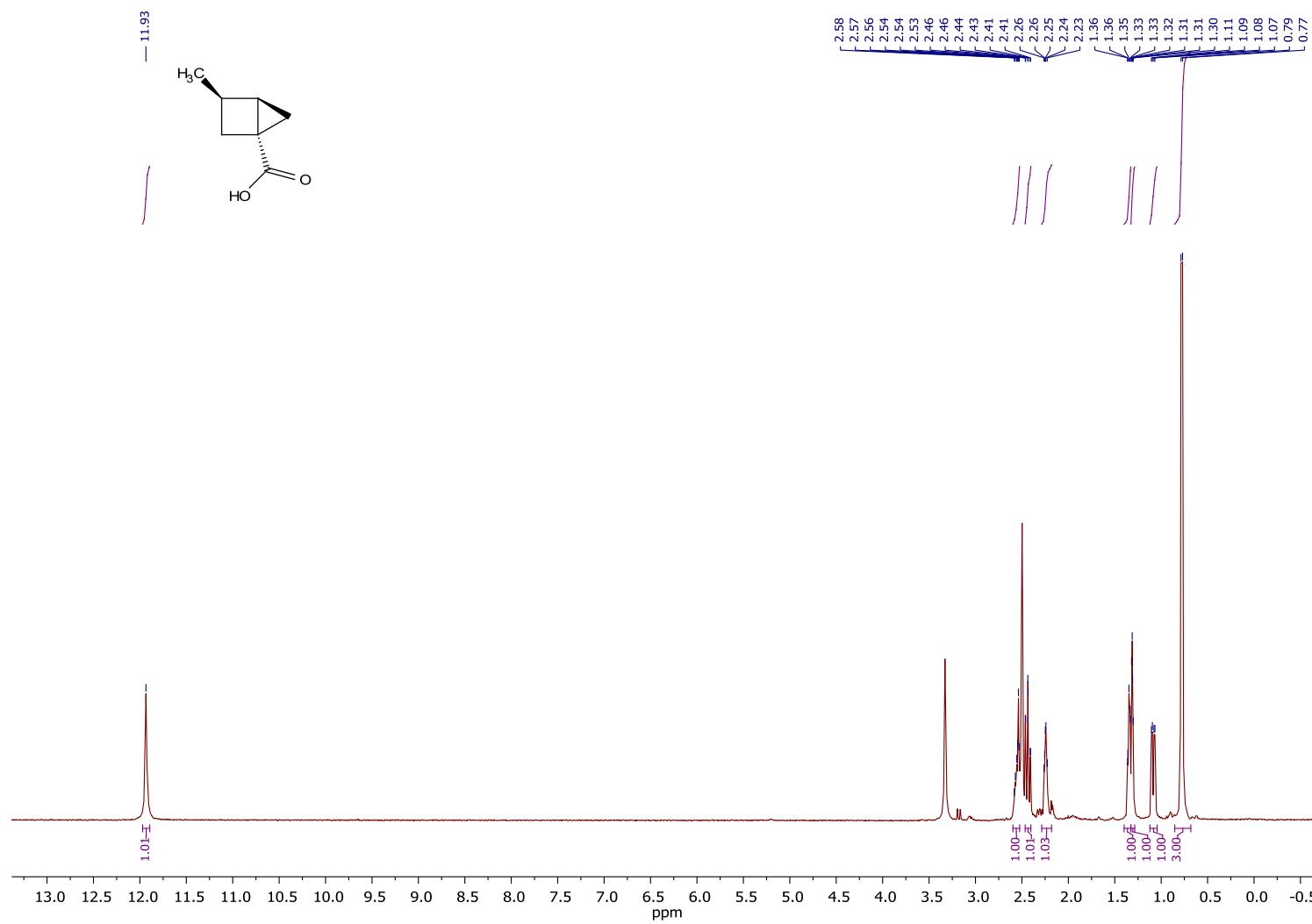
— 28.04

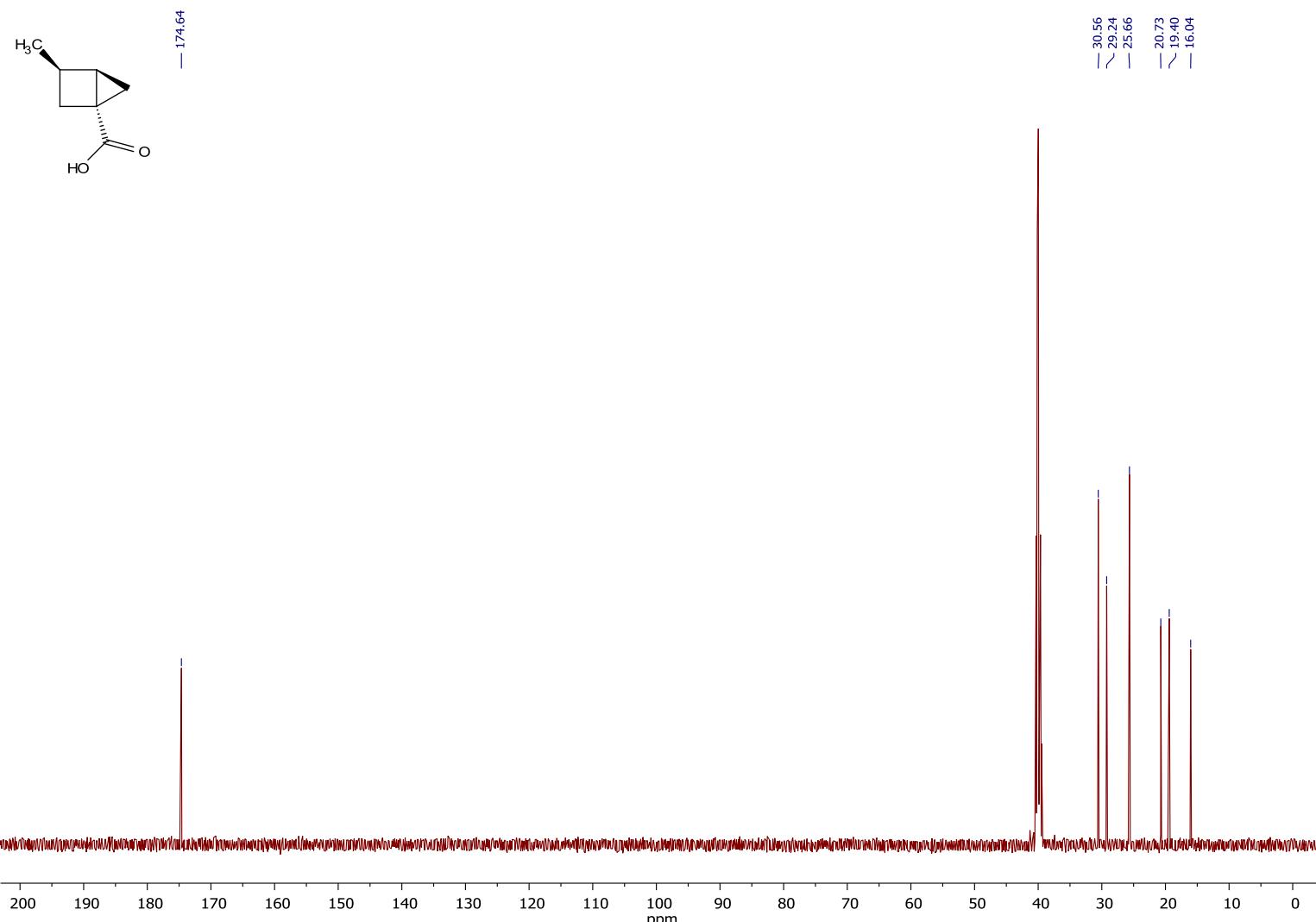
— 19.49

— 18.86

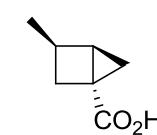
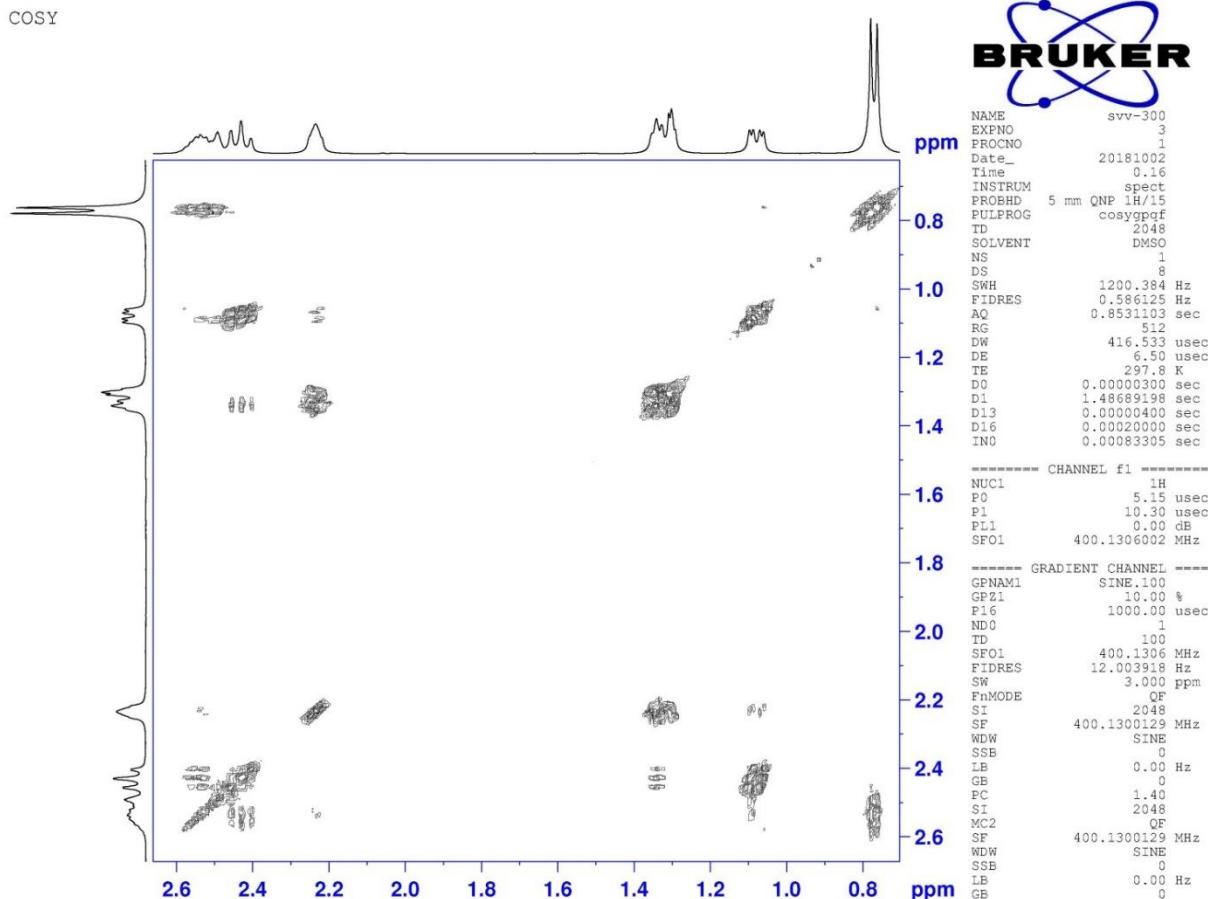


(1R*,3R*,4S*)-tert-Butyl 3-hydroxybicyclo[2.1.0]pentane-1-carboxylate (16g) ^{13}C NMR (CDCl_3)

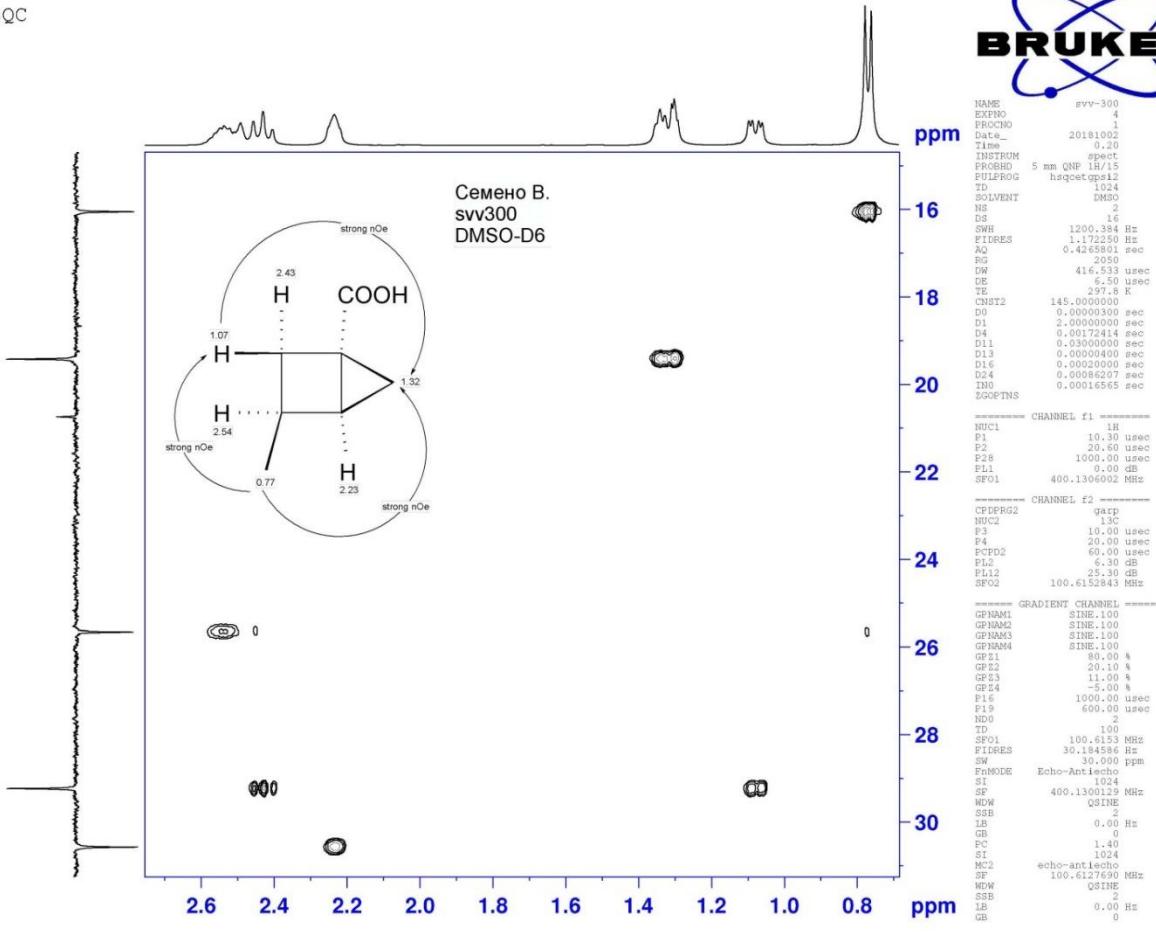


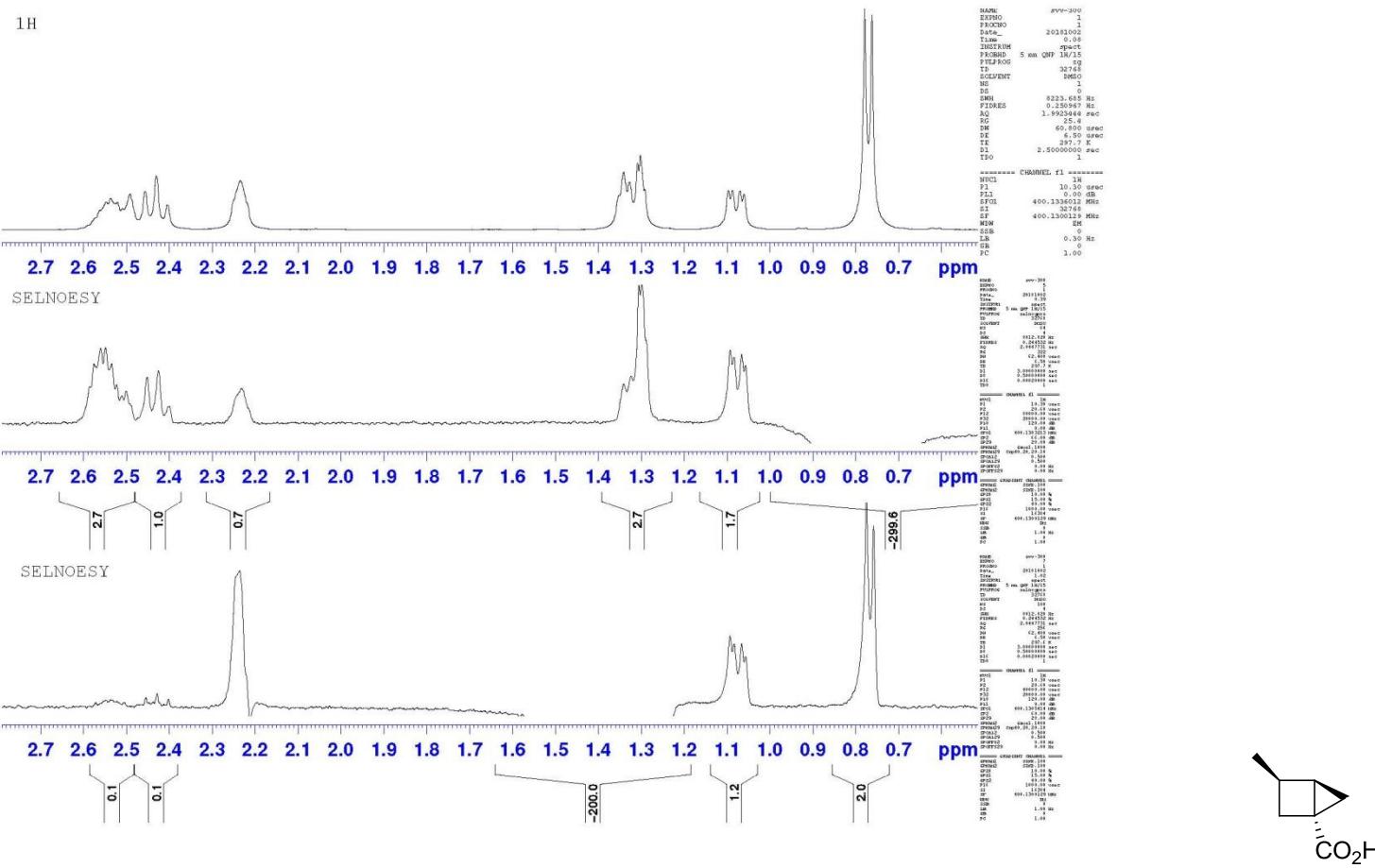


COSY

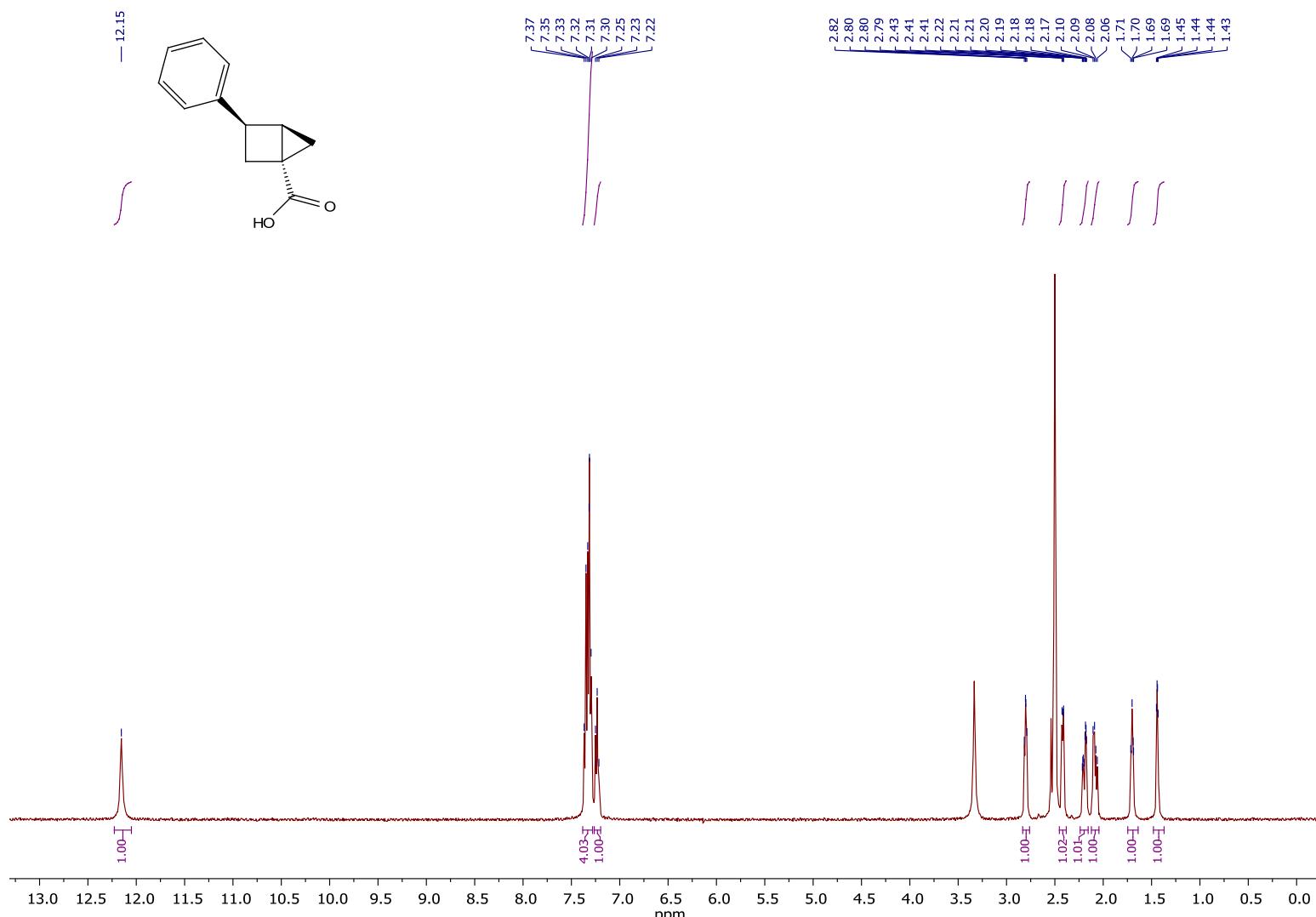
(1*R*^{*},3*R*^{*},4*R*^{*})-3-Methylbicyclo[2.1.0]pentane-1-carboxylic acid (17a) (COSY) (DMSO-*d*₆)

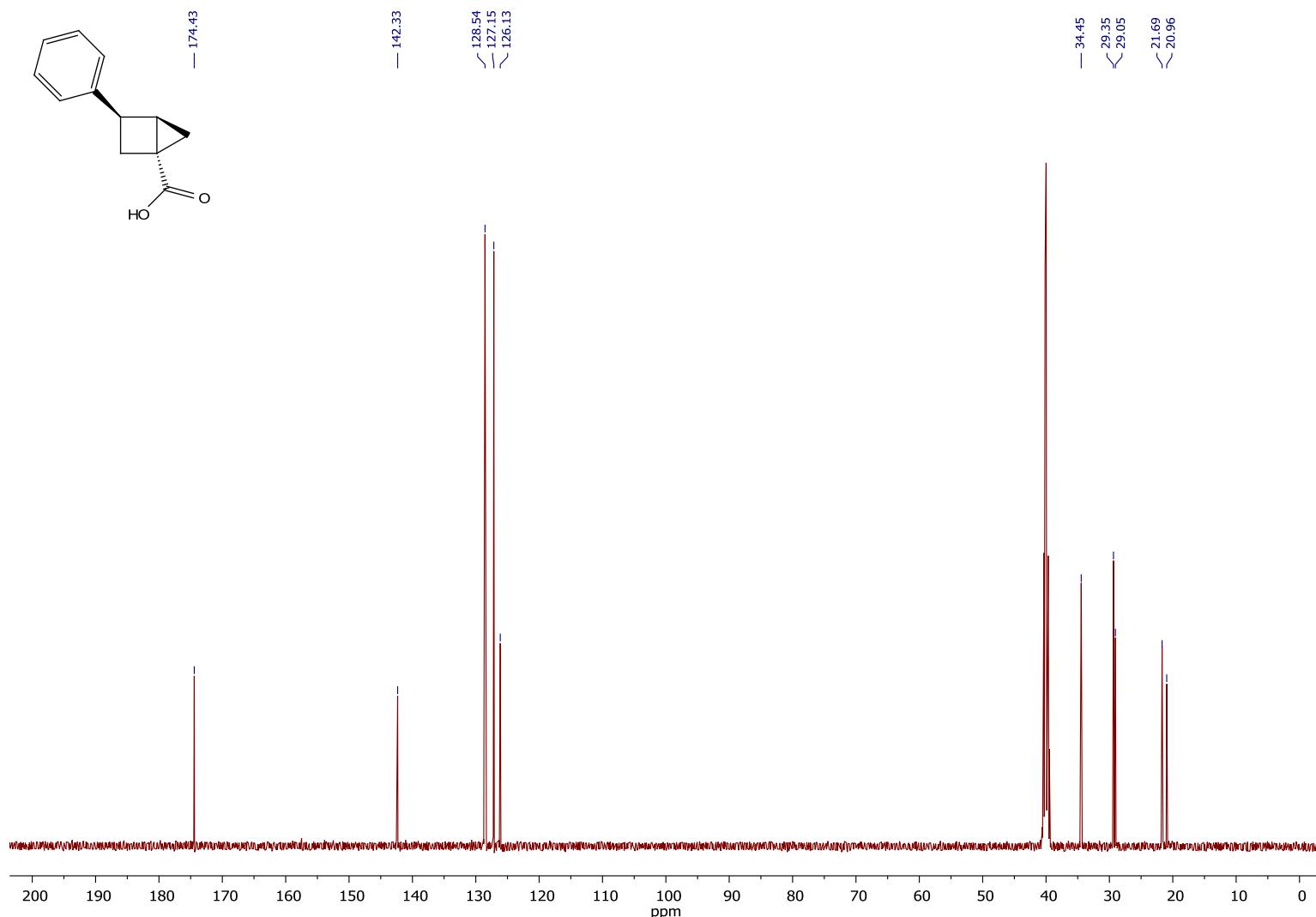
HSQC

(1*R*^{*},3*R*^{*},4*R*^{*})-3-Methylbicyclo[2.1.0]pentane-1-carboxylic acid (17a) (HSQC) (DMSO-*d*₆)



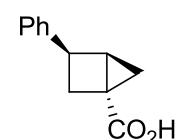
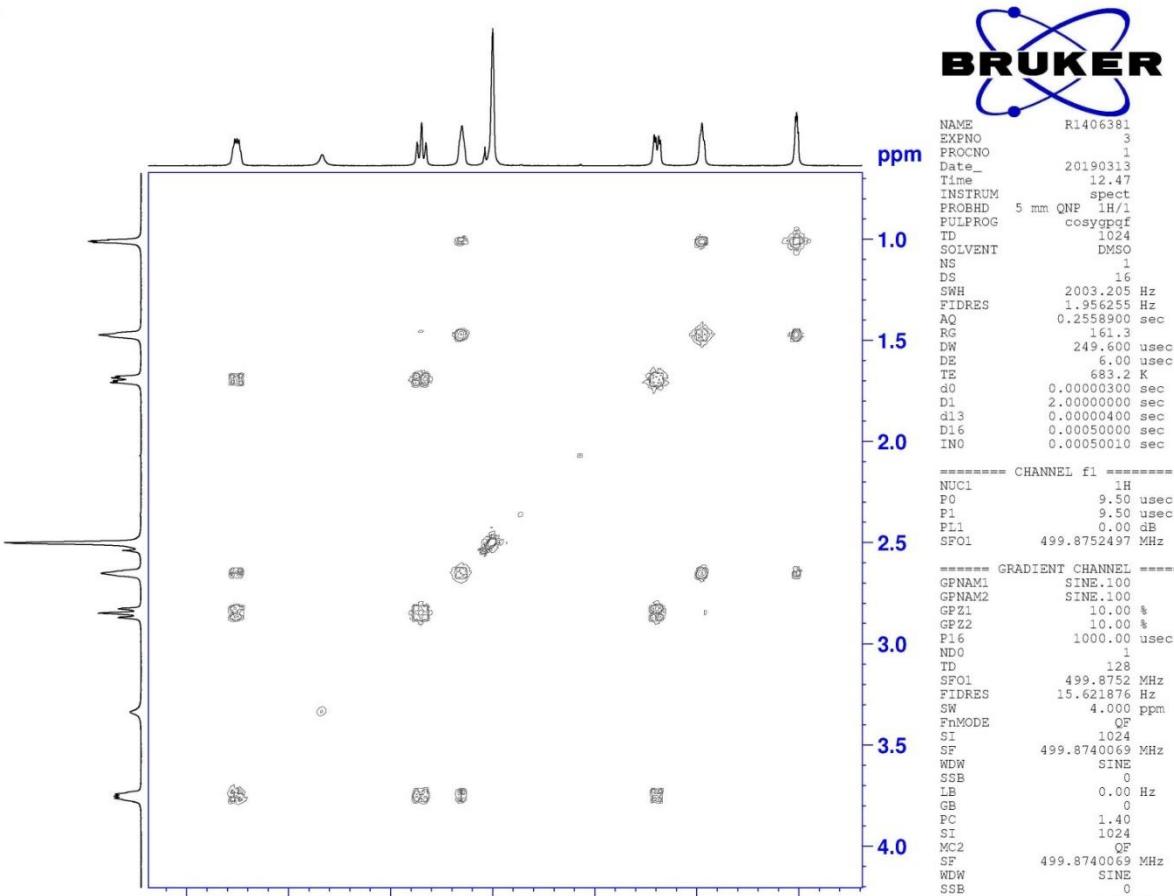
(1*R**,3*R**,4*R**)-3-Methylbicyclo[2.1.0]pentane-1-carboxylic acid (17a) (H–H NOE) (DMSO-*d*₆)



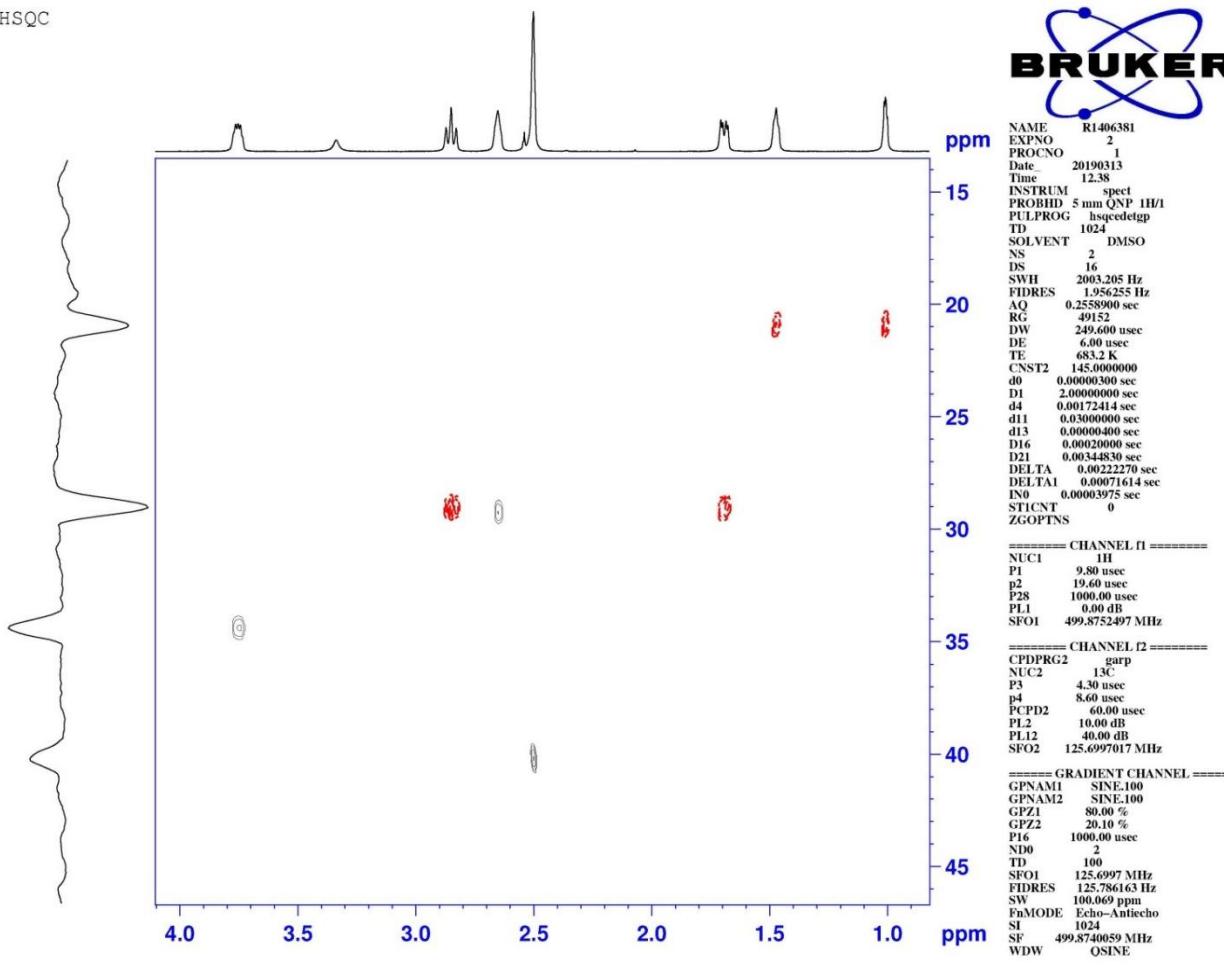
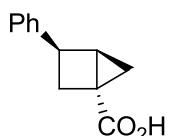


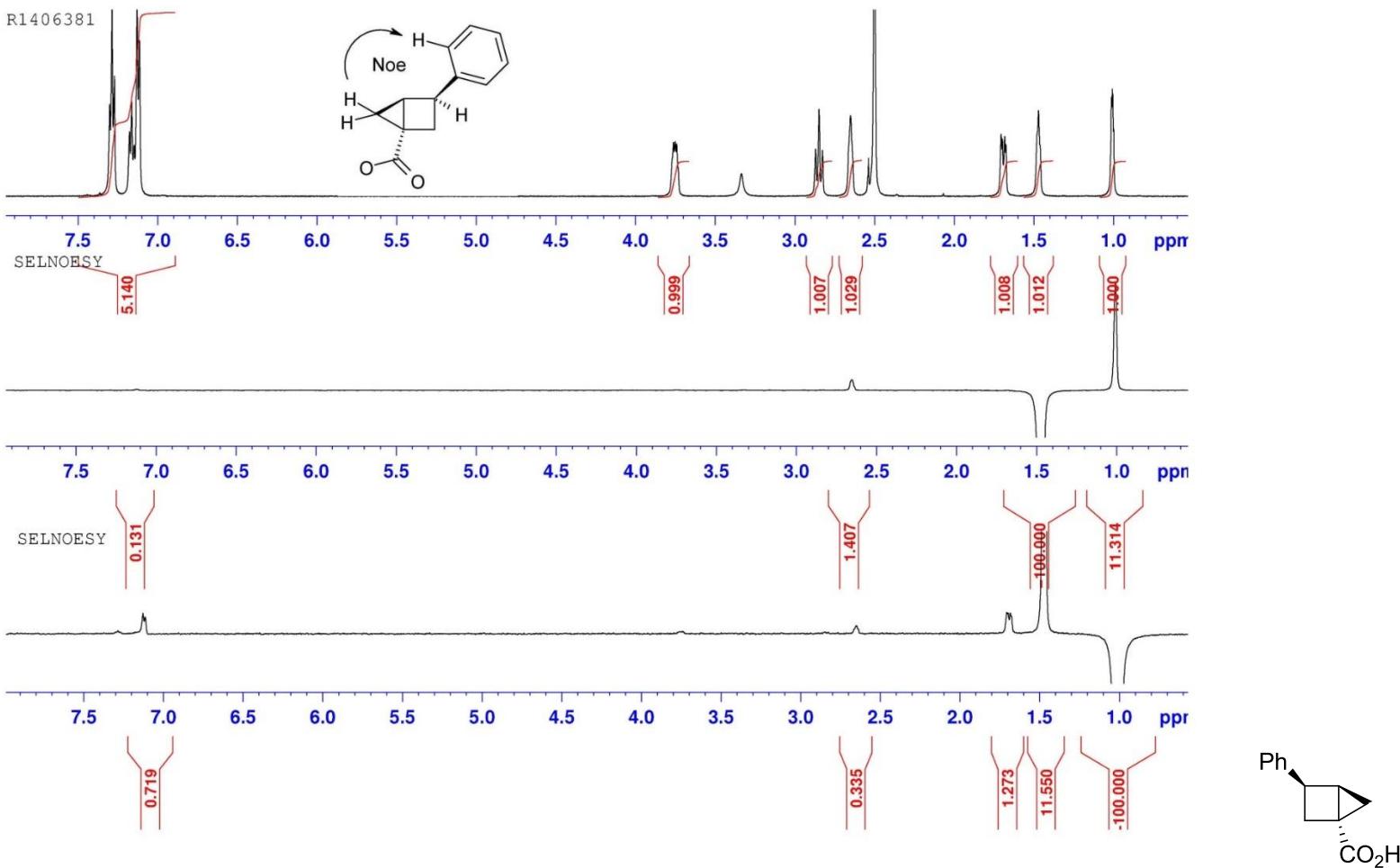
(1*R*^{*},3*R*^{*},4*R*^{*})-3-Phenylbicyclo[2.1.0]pentane-1-carboxylic acid (17b) ^{13}C NMR (DMSO-*d*₆)

COSY

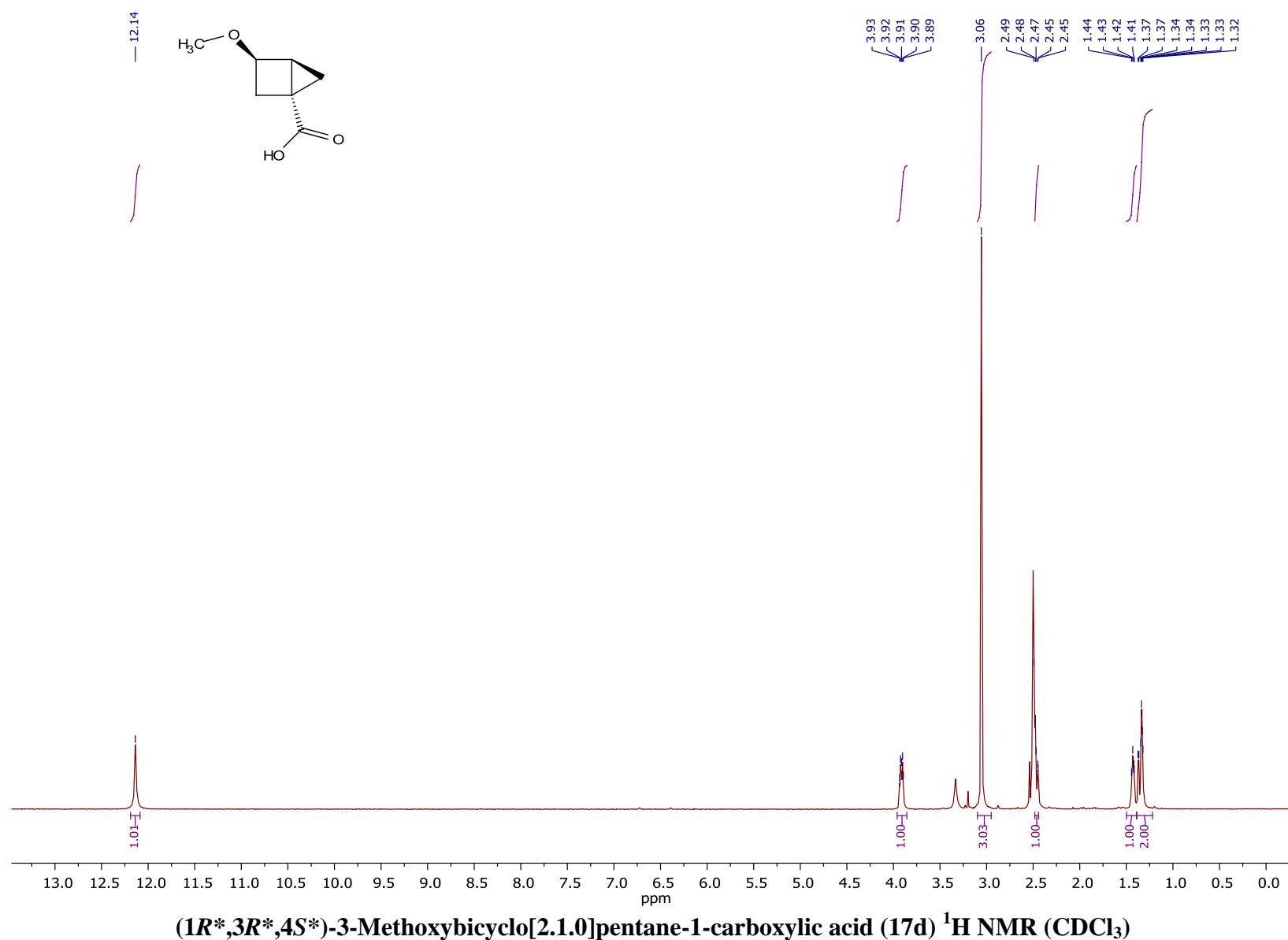
(1*R*^{*},3*R*^{*},4*R*^{*})-3-Phenylbicyclo[2.1.0]pentane-1-carboxylic acid (17b) (COSY) (DMSO-*d*₆)

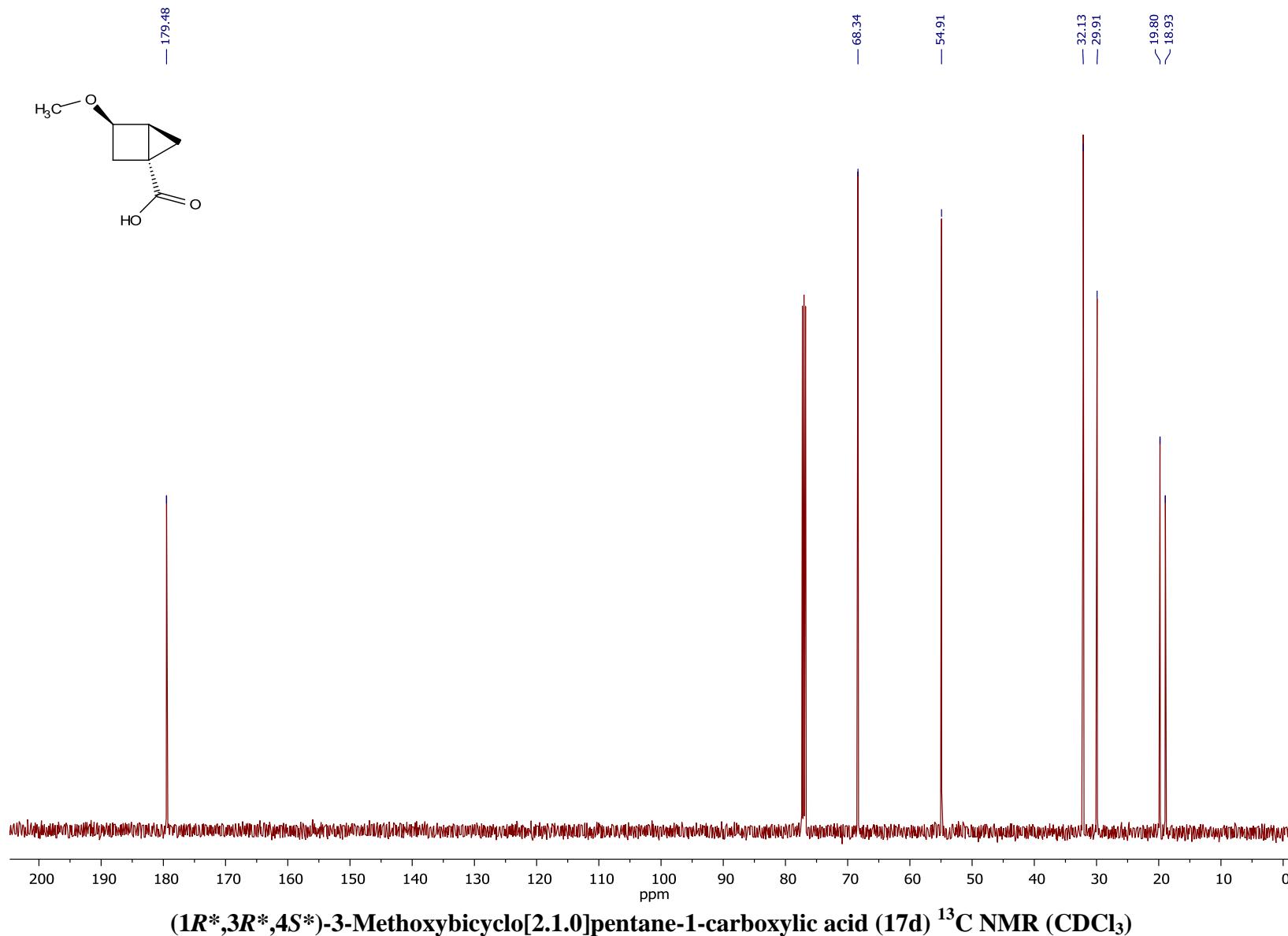
HSQC

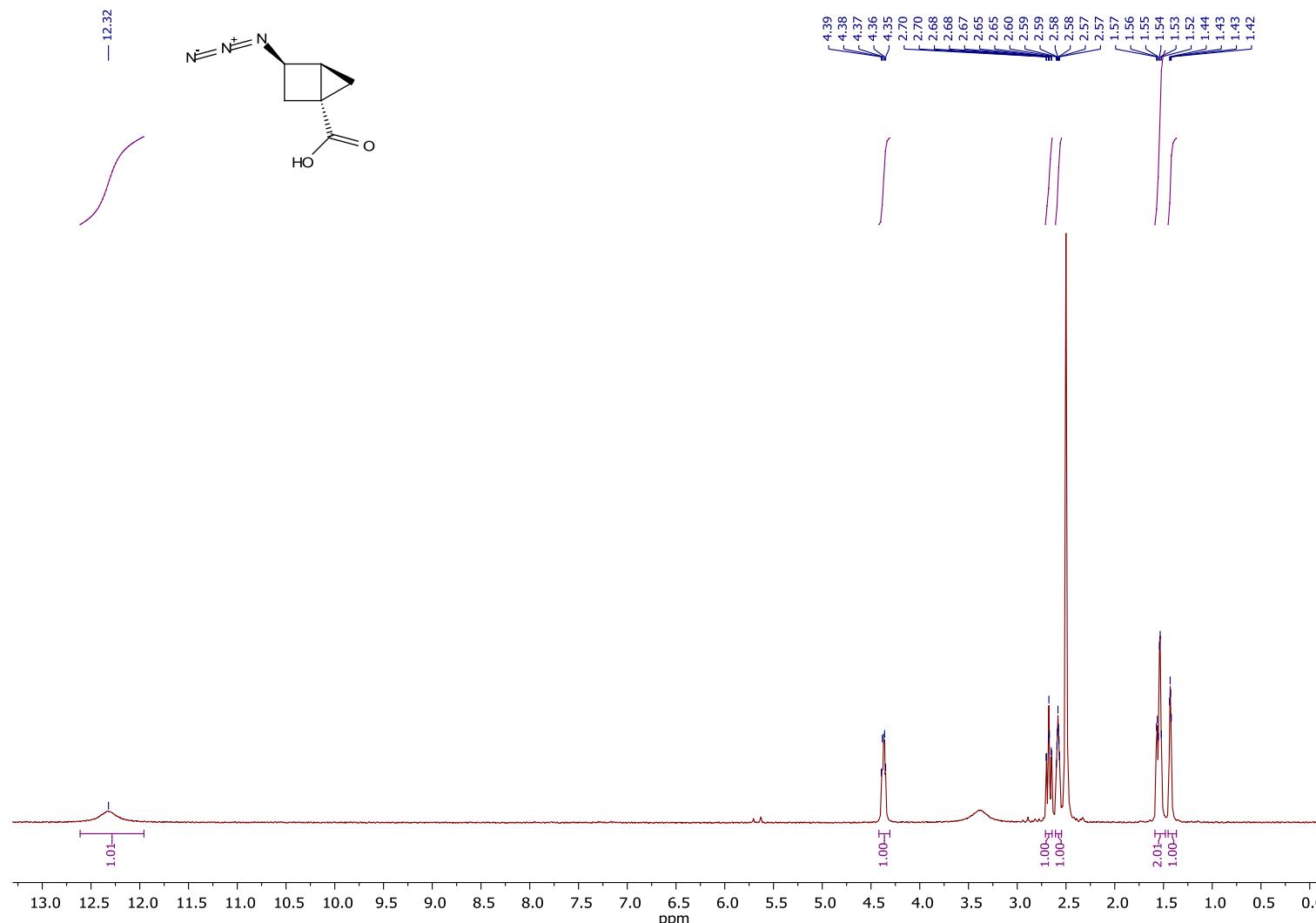
(1*R*^{*,3*R*^{*,4*R*^{*}})-3-Phenylbicyclo[2.1.0]pentane-1-carboxylic acid (17b) (HSQC) (DMSO-*d*₆)}



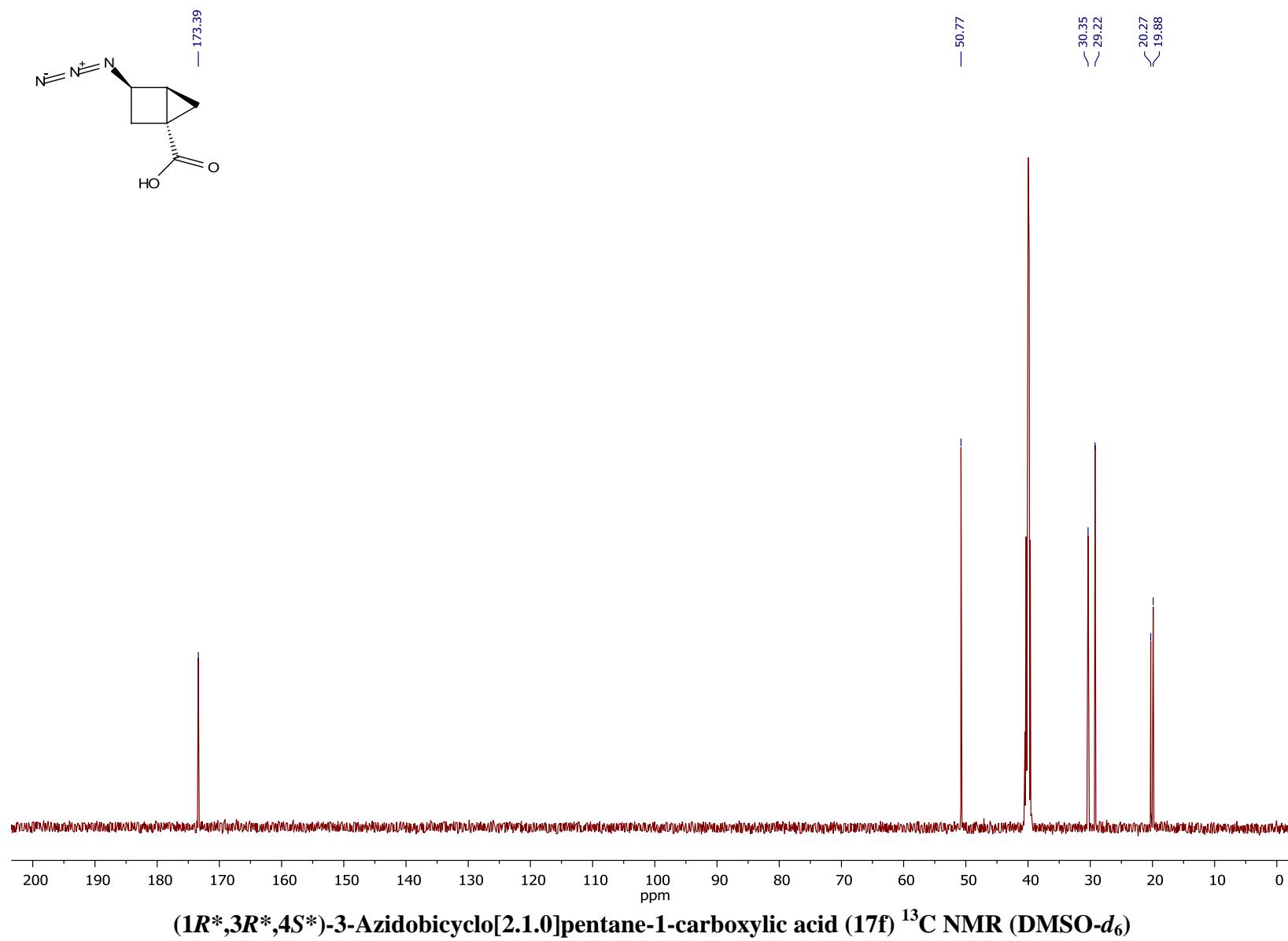
(1*R*^{*},3*R*^{*},4*R*^{*})-3-Phenylbicyclo[2.1.0]pentane-1-carboxylic acid (17b) (H–H NOE) (DMSO-*d*₆)

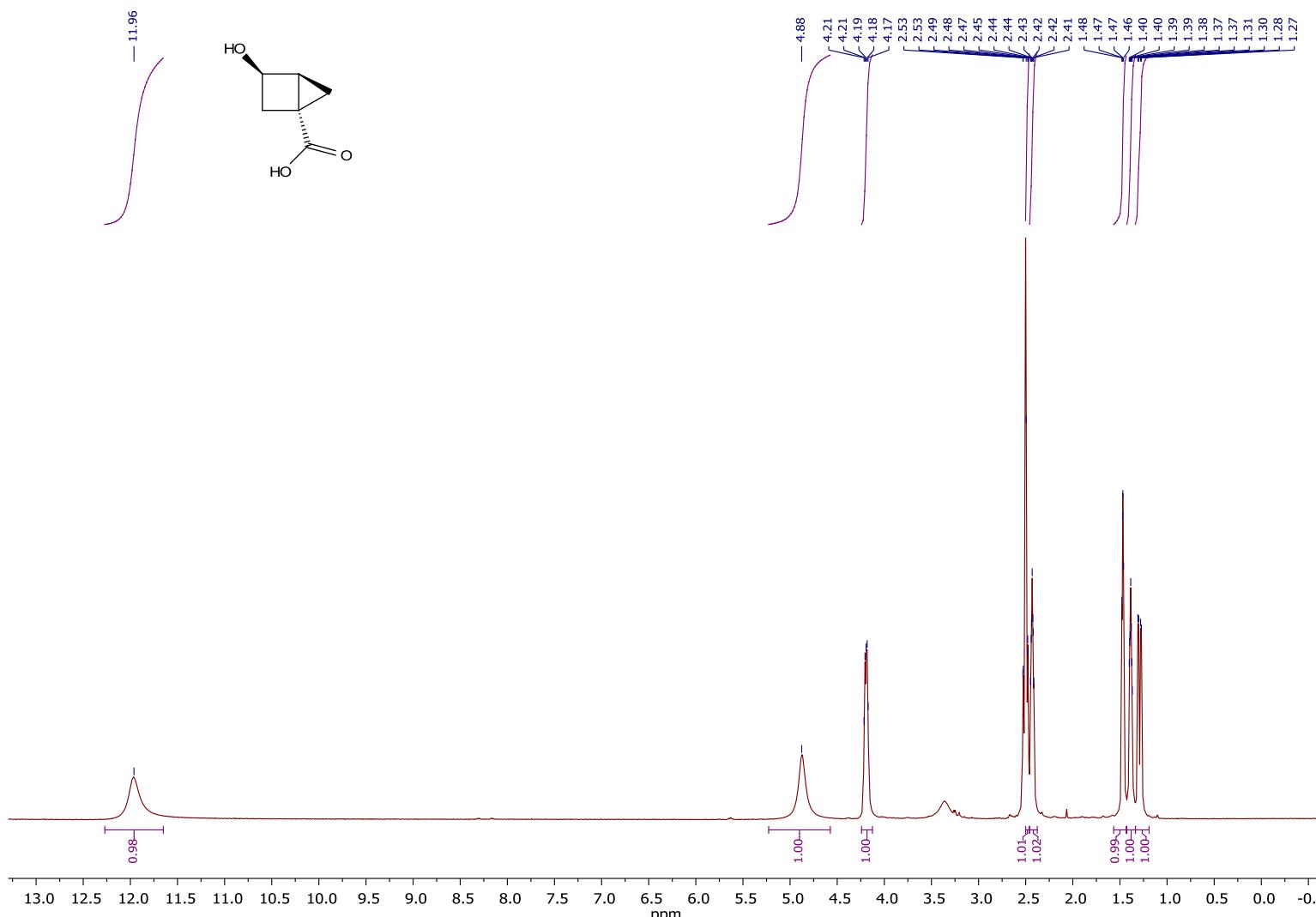




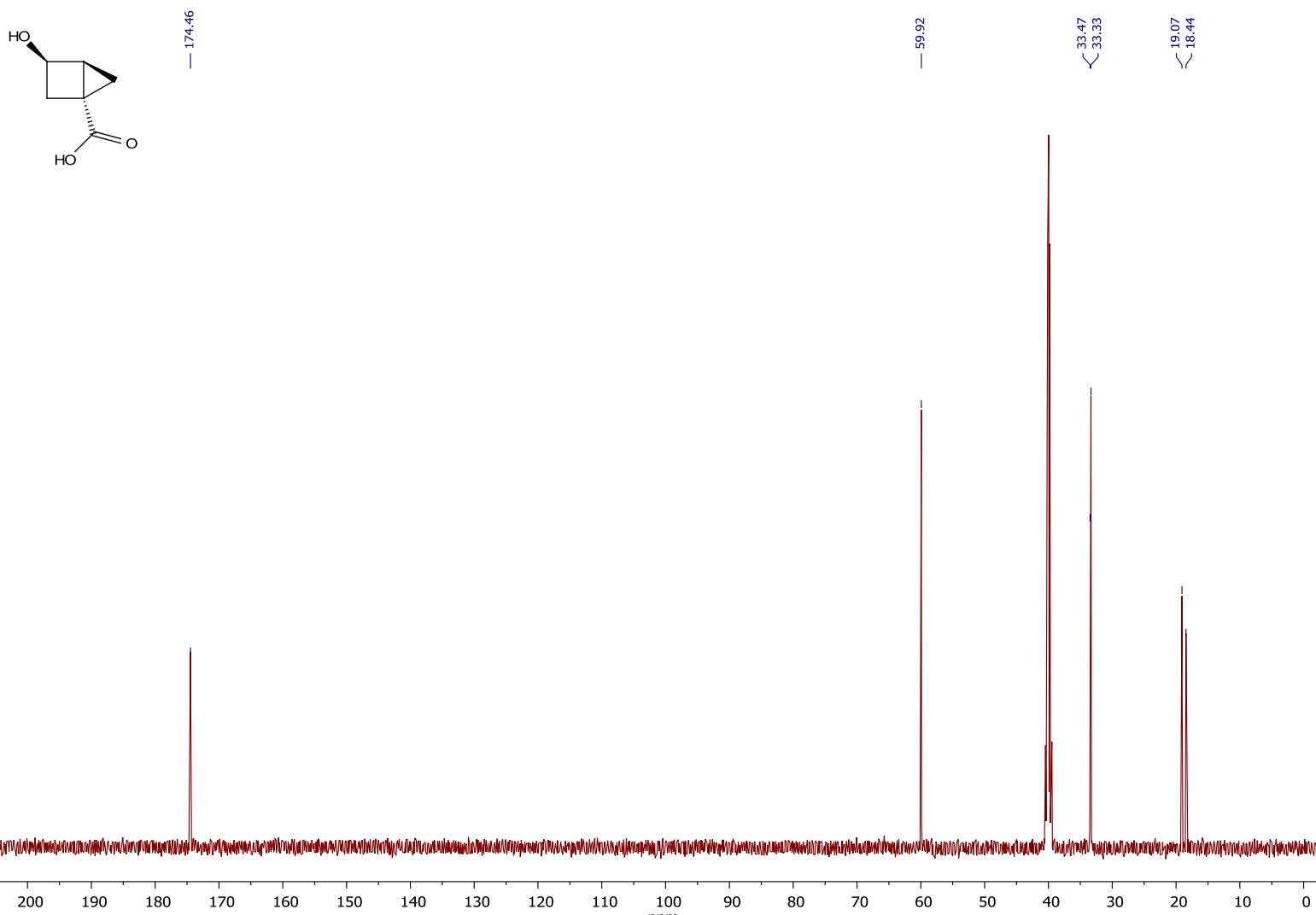


(1R*,3R*,4S*)-3-Azidobicyclo[2.1.0]pentane-1-carboxylic acid (17f) ^1H NMR (DMSO- d_6)

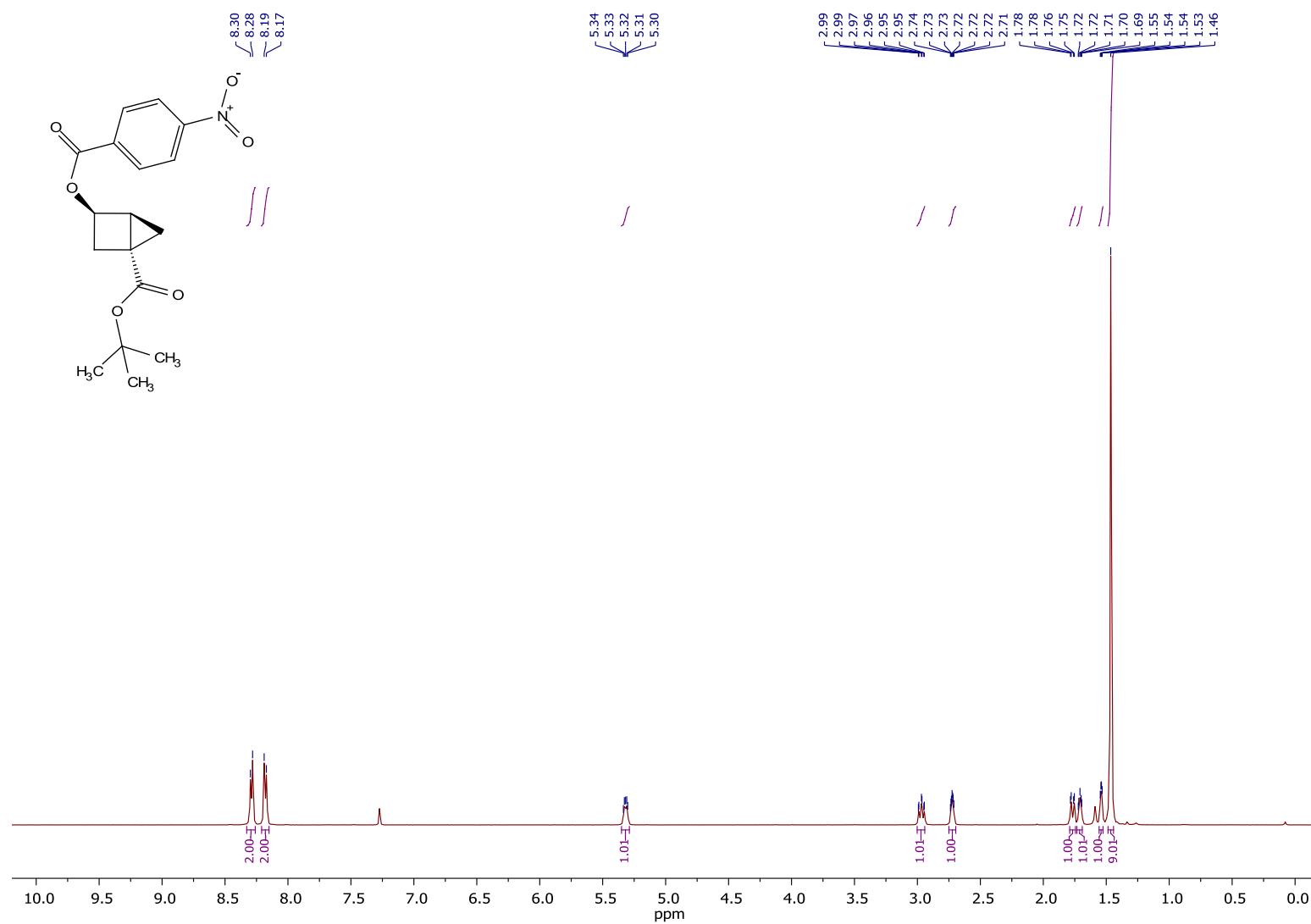


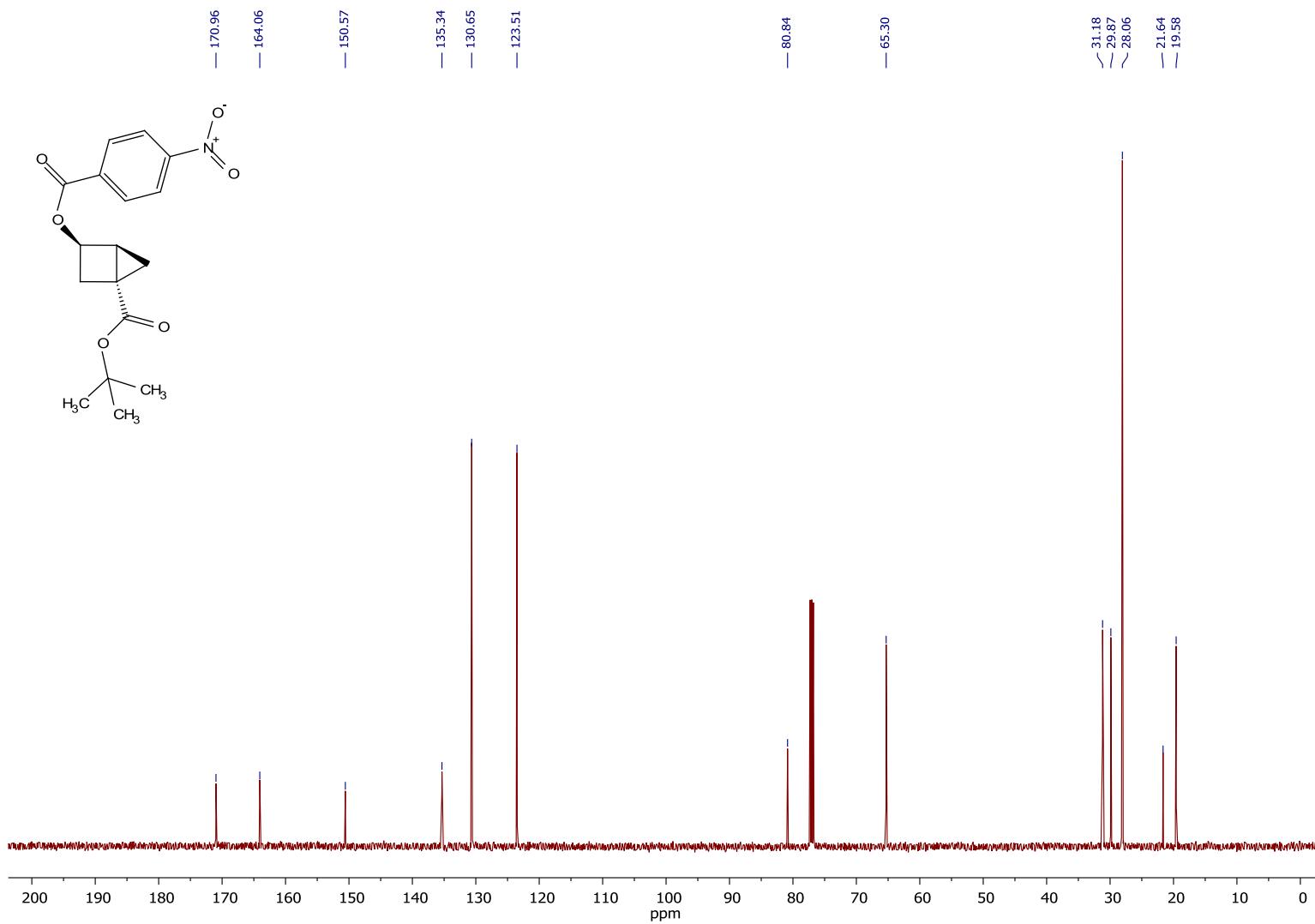


(1*R*^{*},3*R*^{*},4*S*^{*})-3-Hydroxybicyclo[2.1.0]pentane-1-carboxylic acid (17g) ^1H NMR (DMSO-*d*₆)

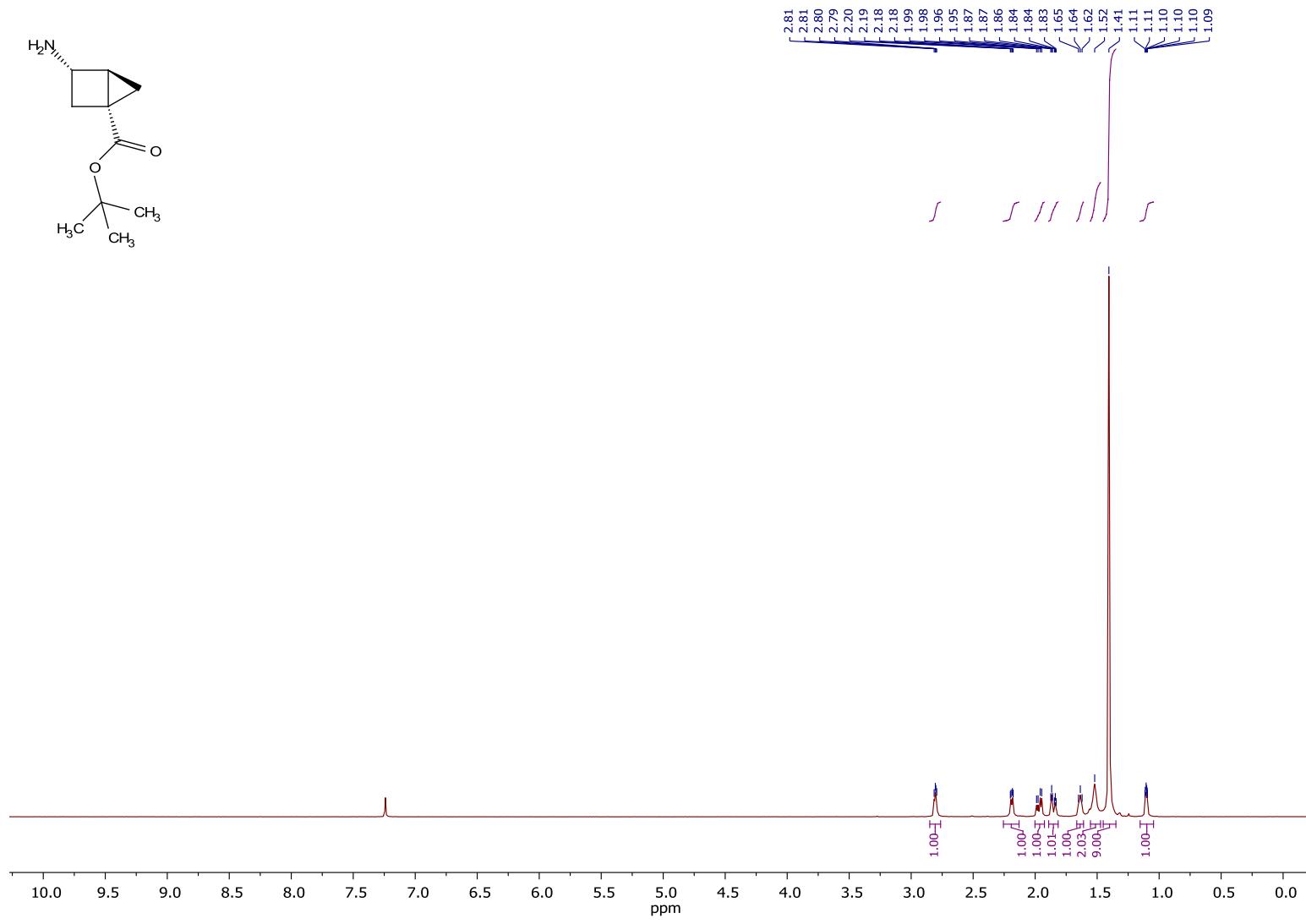
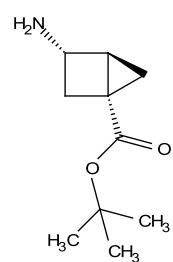


(1*R*^{*,3*R*^{*,4*S*^{*}}-3-Hydroxybicyclo[2.1.0]pentane-1-carboxylic acid (17g) ^{13}C NMR (DMSO-*d*₆)}

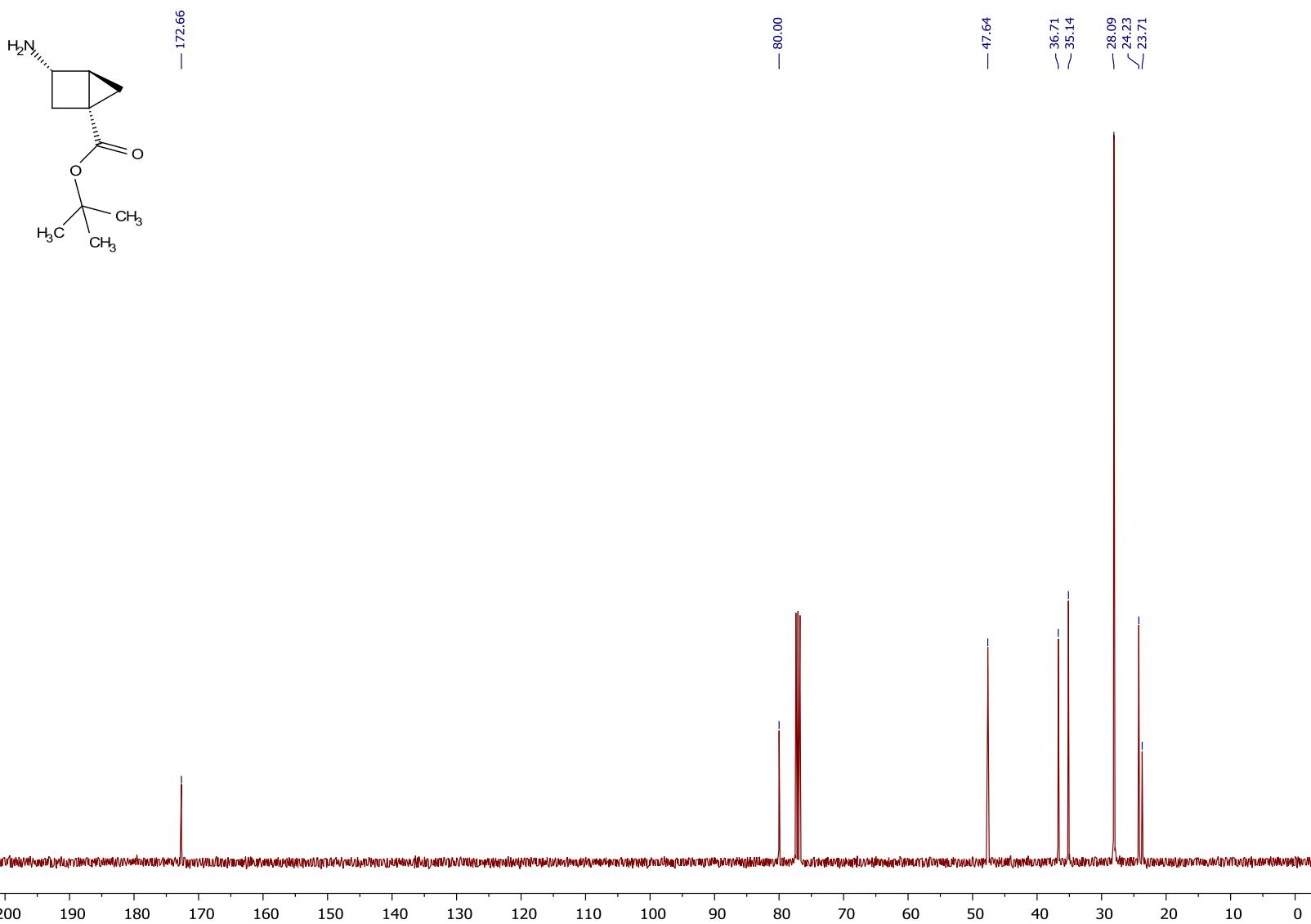




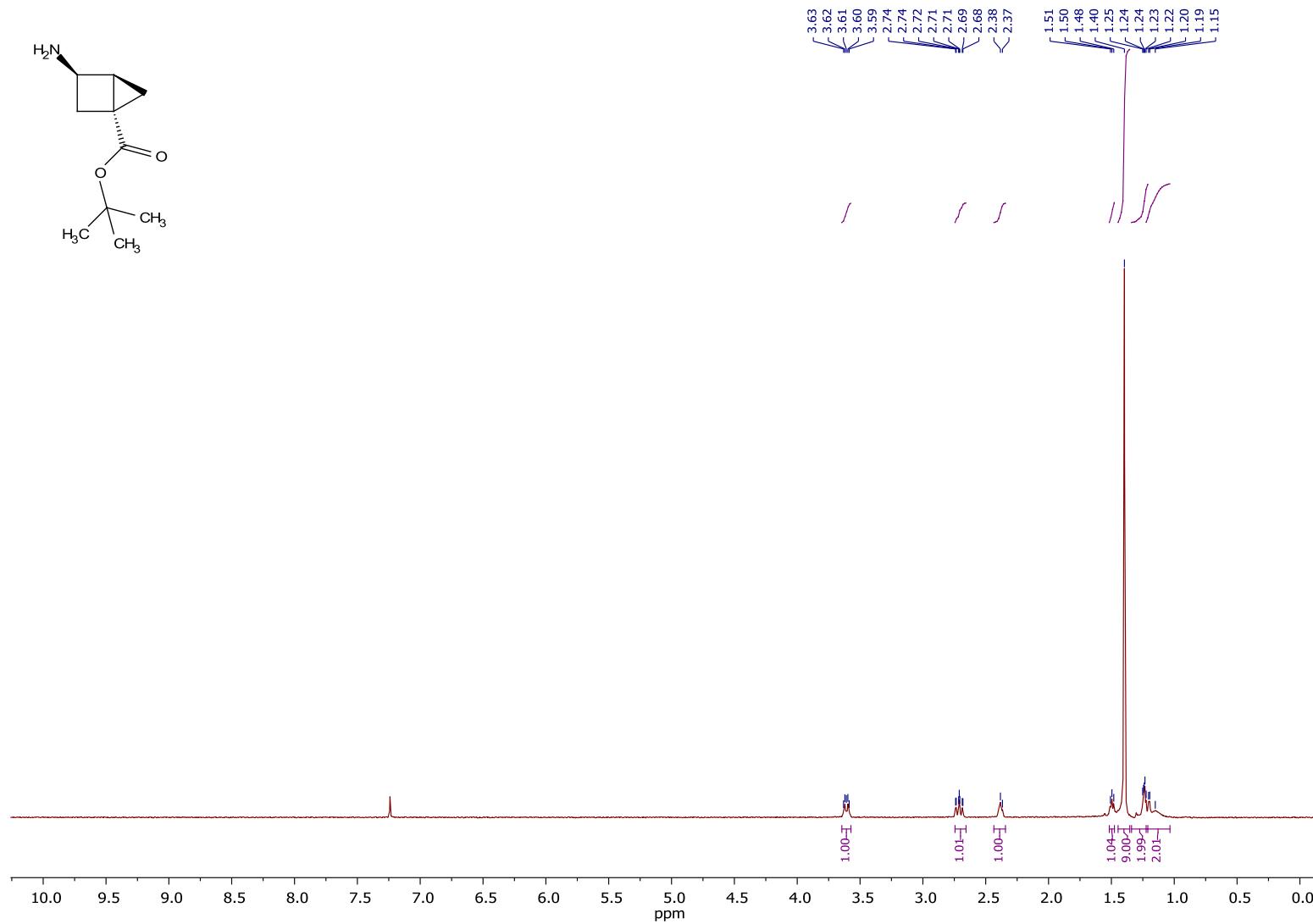
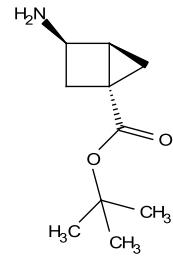
(1*R*^{*,3*R*^{*,4*S*^{*}-*tert*-Butyl 3-((4-nitrobenzoyl)oxy)bicyclo[2.1.0]pentane-1-carboxylate (18g) ^{13}C NMR (CDCl_3)}}



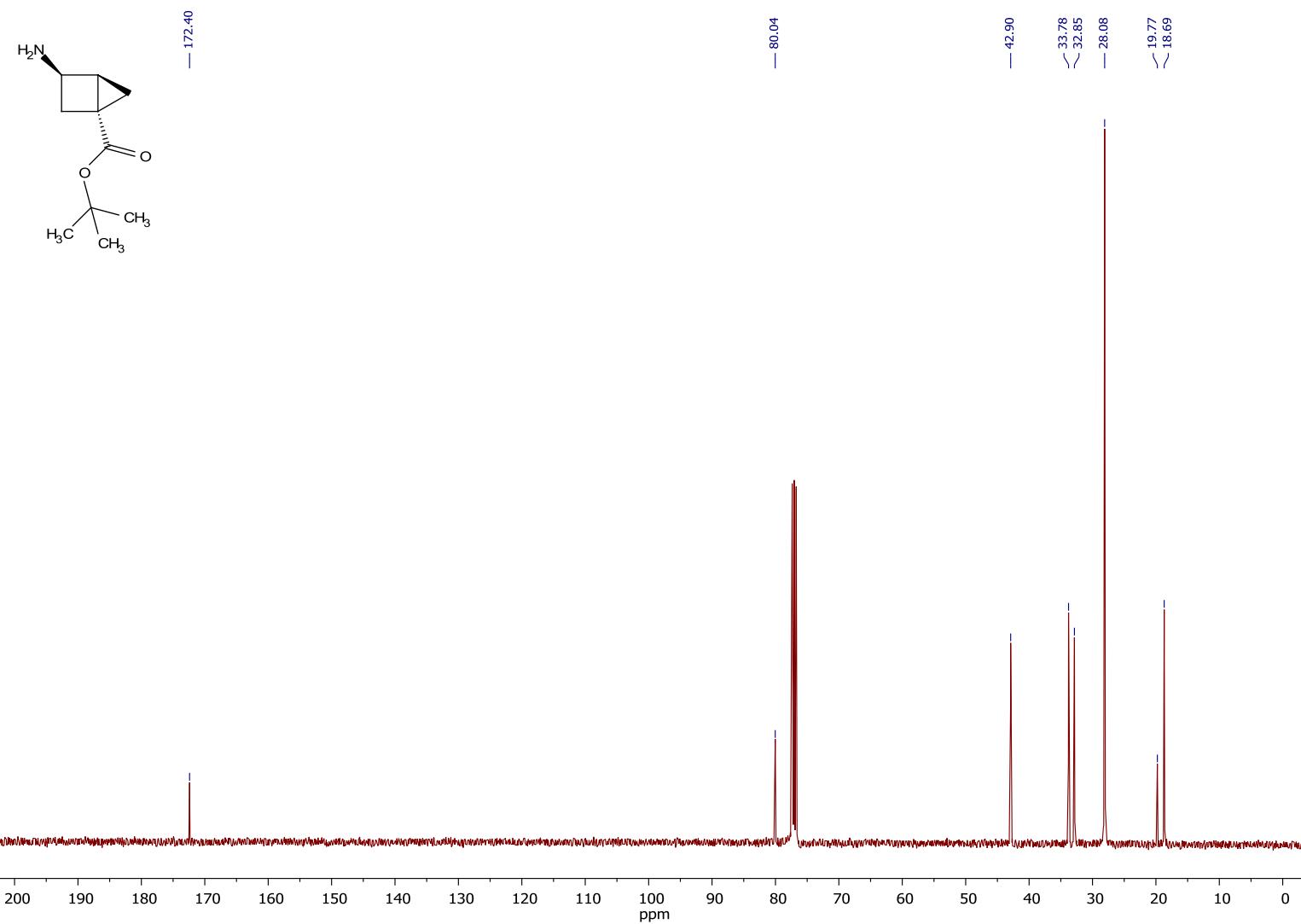
(1*R*^{*,3*S*^{*,4*S*^{*}})-*tert*-Butyl 3-aminobicyclo[2.1.0]pentane-1-carboxylate (19) ^1H NMR (CDCl_3)}

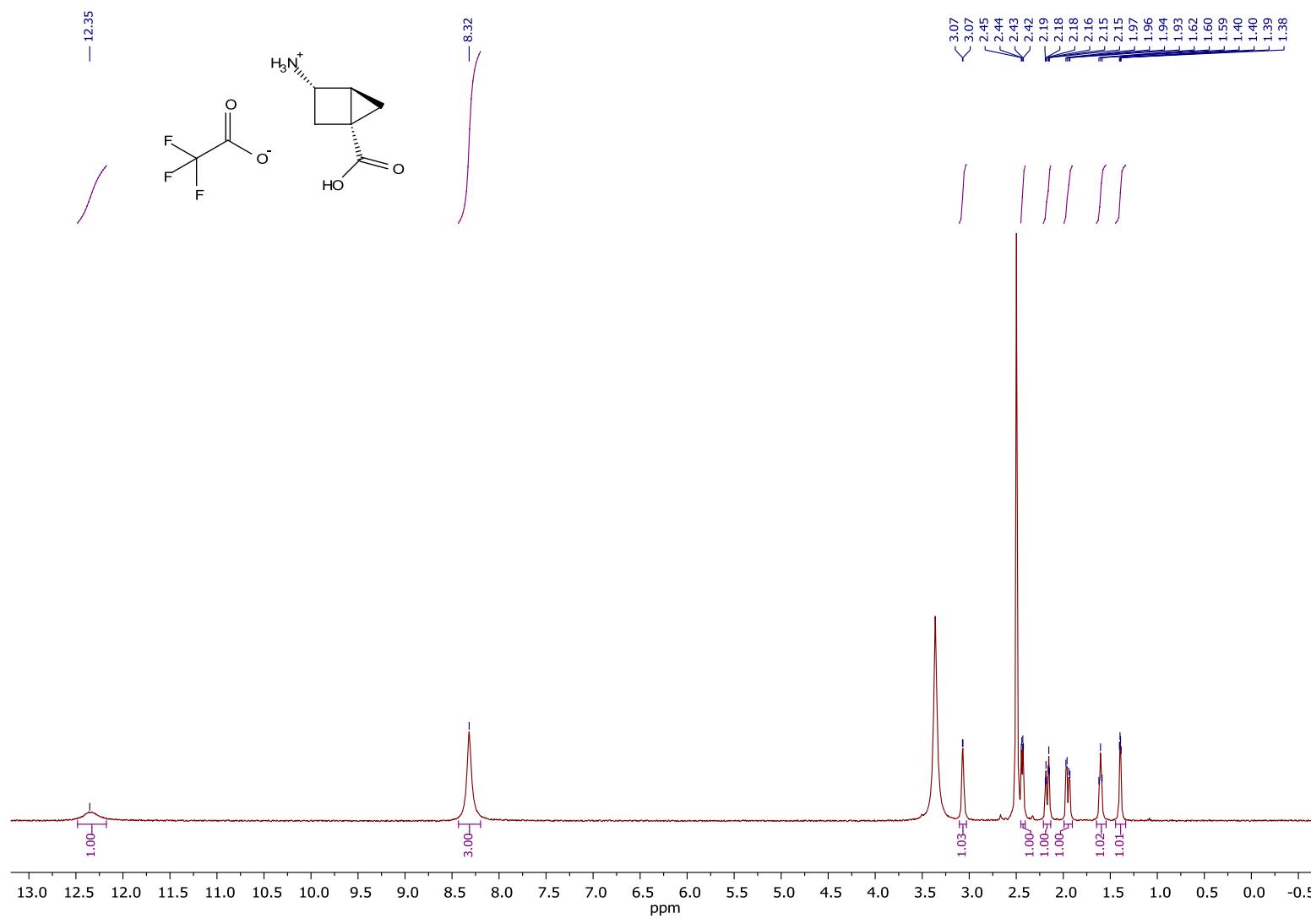


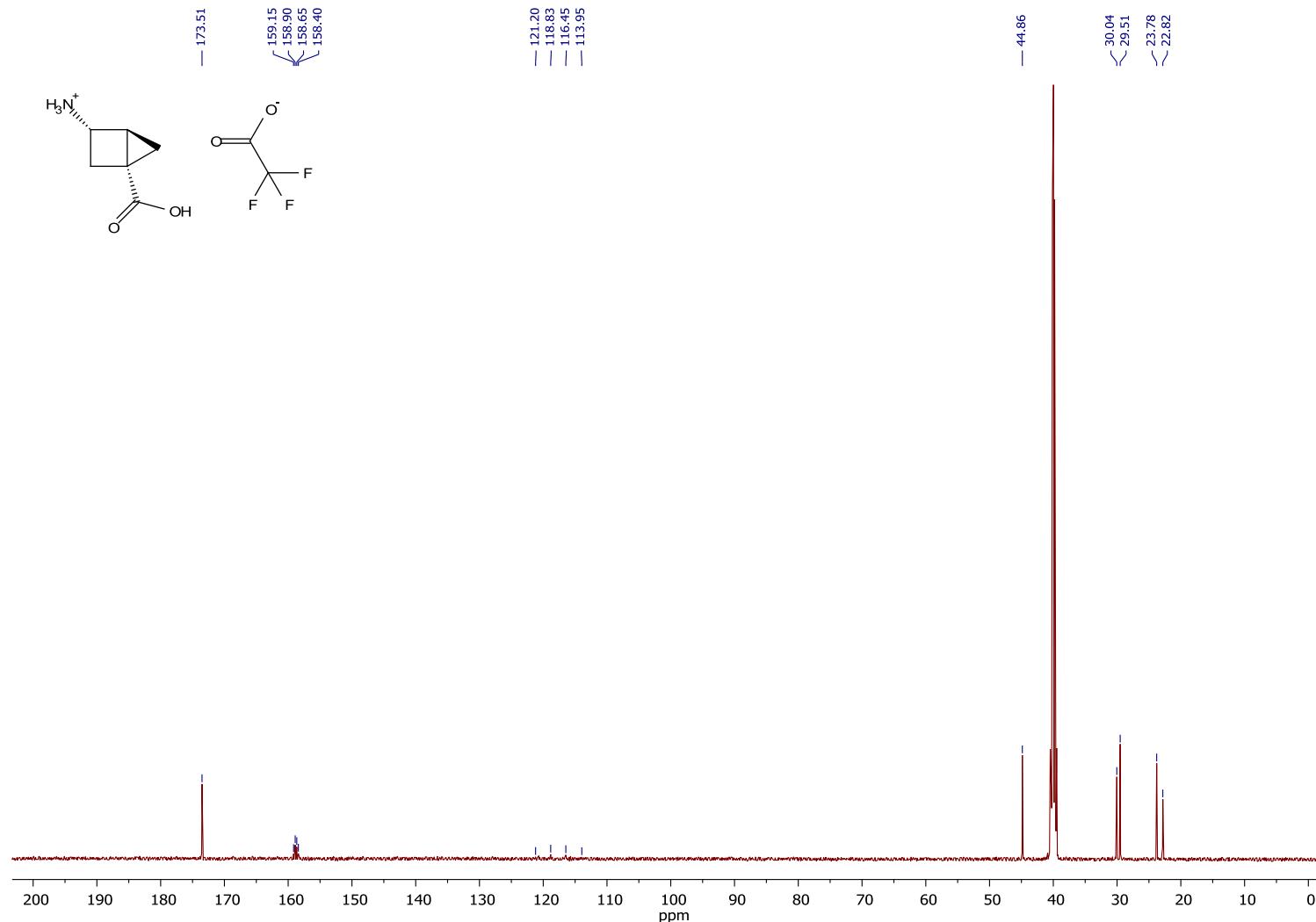
(1*R*^{*,3*S*^{*,4*S*^{*}}-tert-Butyl 3-aminobicyclo[2.1.0]pentane-1-carboxylate (19) ^{13}C NMR (CDCl_3)}



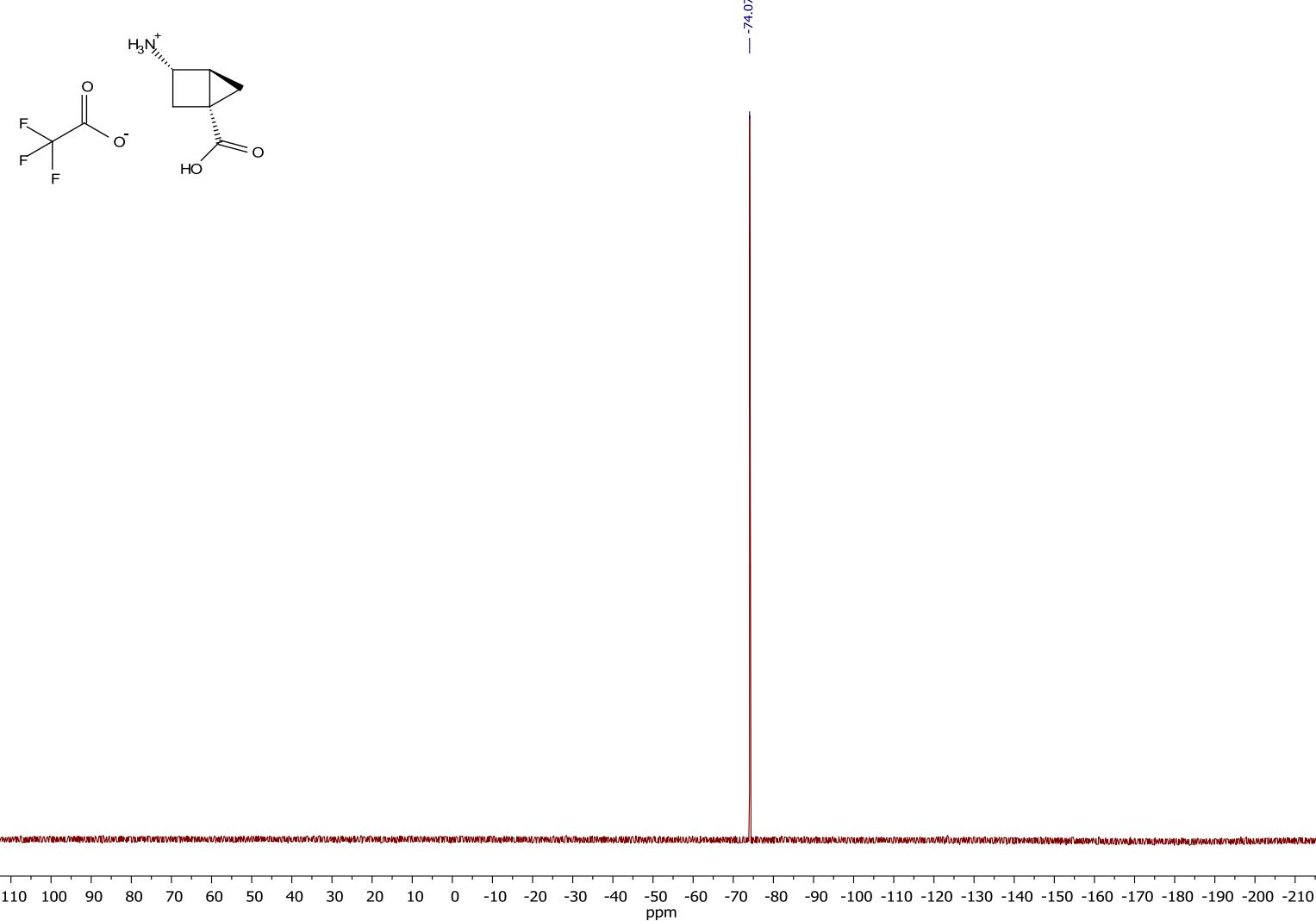
(1*R*^{*},3*R*^{*},4*S*^{*})-*tert*-Butyl 3-aminobicyclo[2.1.0]pentane-1-carboxylate (20) ¹H NMR (CDCl₃)





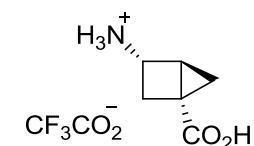
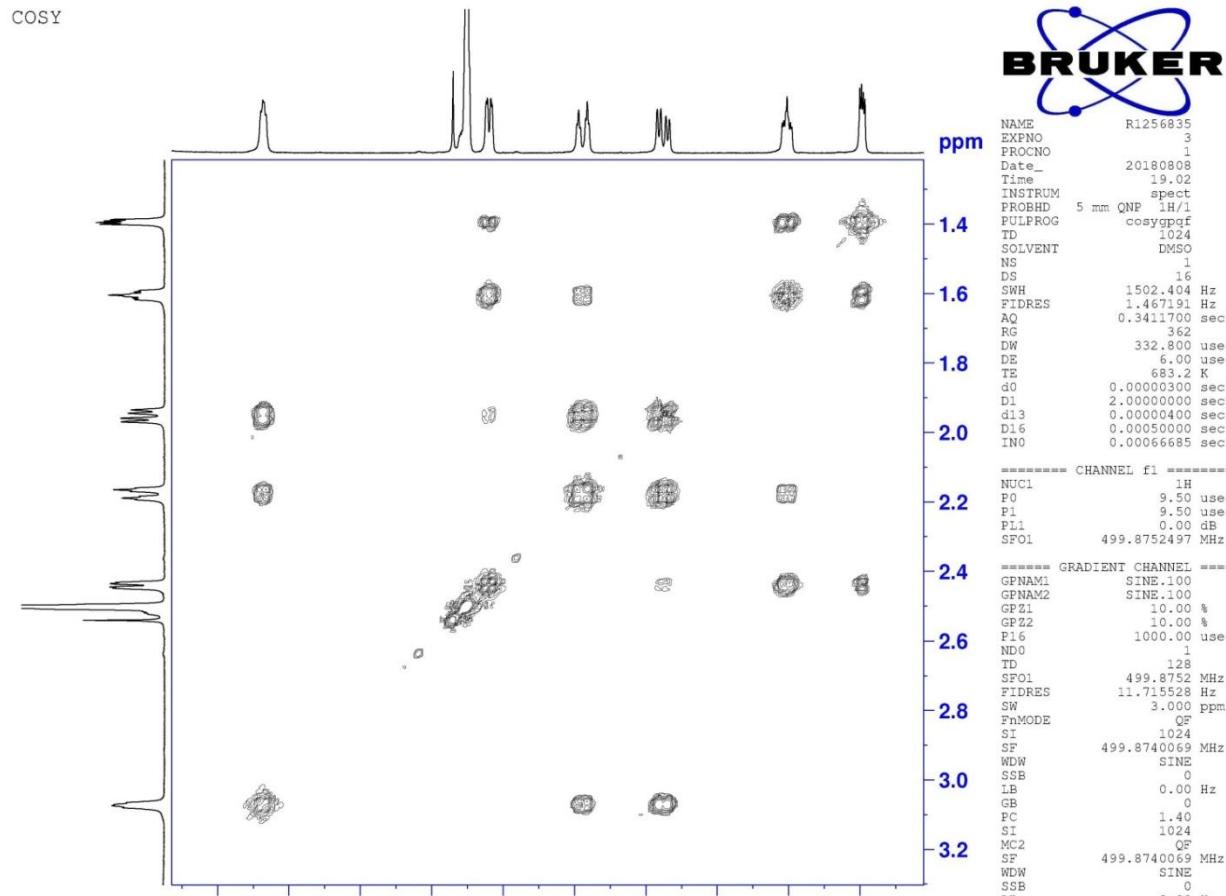


(*1S*,2S*,4R**)-4-Carboxybicyclo[2.1.0]pentan-2-aminium 2,2,2-trifluoroacetate (**21**) ^{13}C NMR (DMSO-*d*₆)



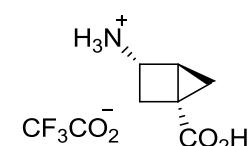
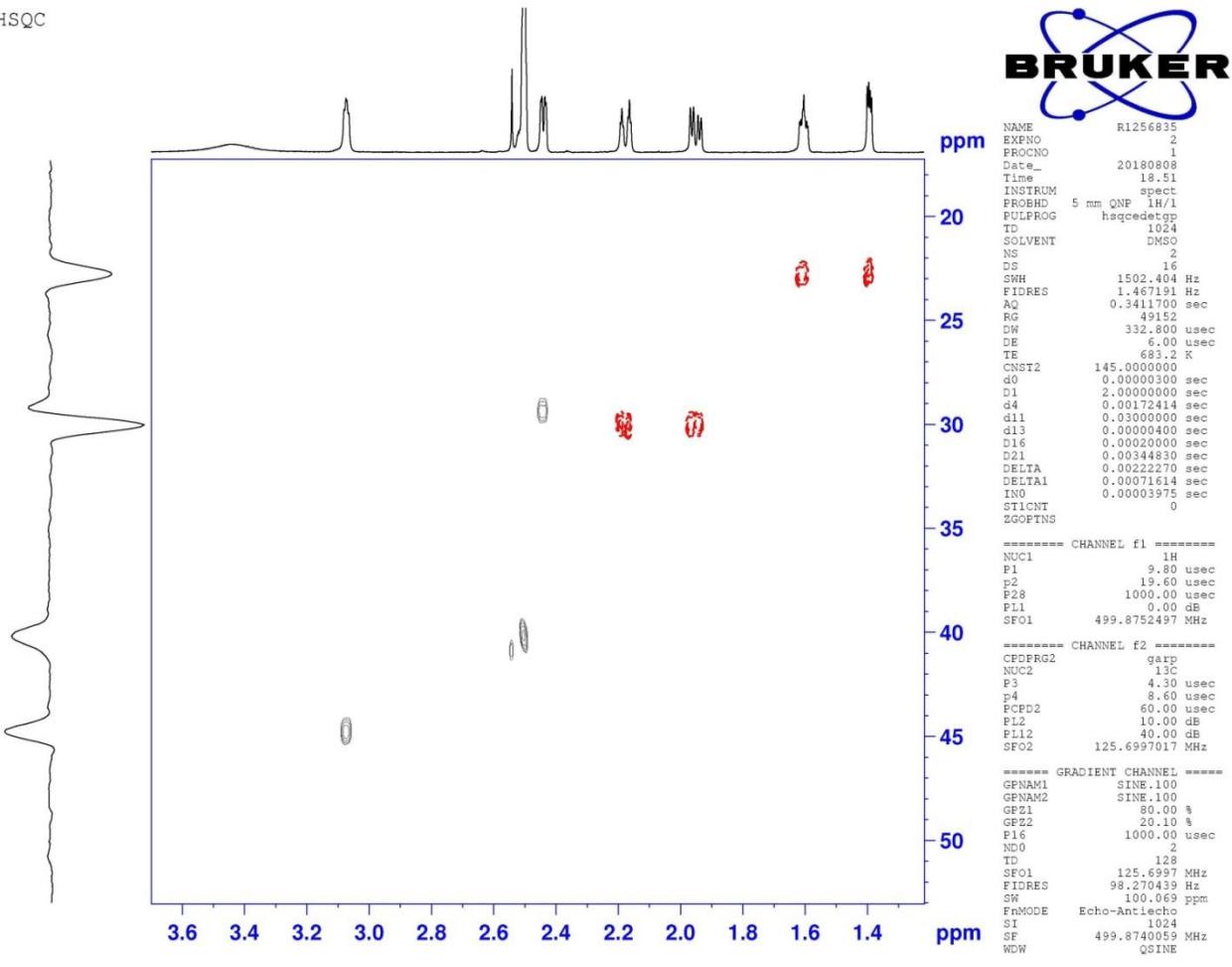
($1S^*,2S^*,4R^*$)-4-Carboxybicyclo[2.1.0]pentan-2-aminium 2,2,2-trifluoroacetate (21) ^{19}F NMR (DMSO- d_6)

COSY

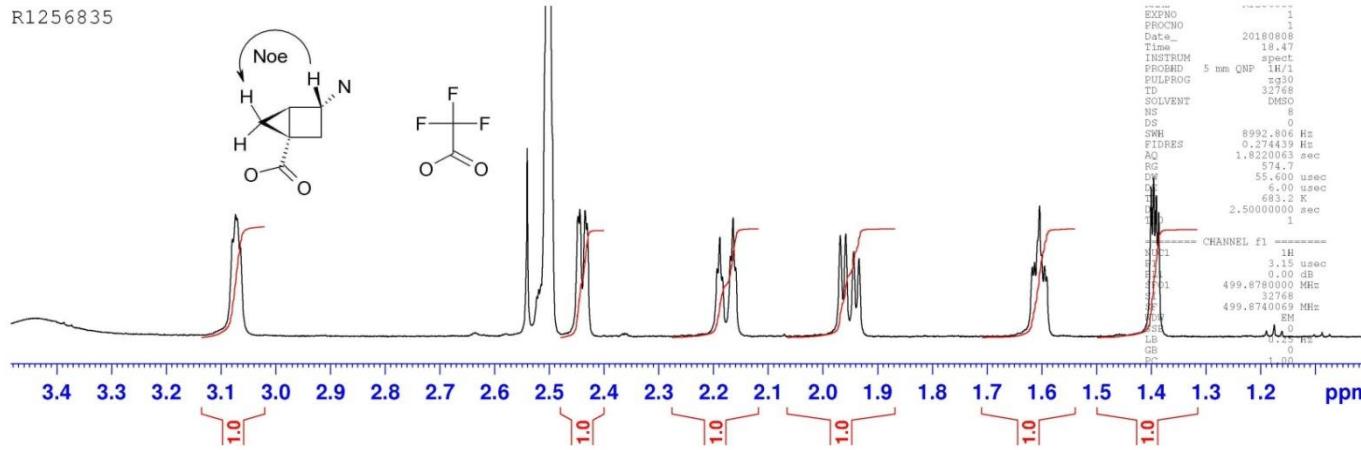


(1*S*^{*,}2*S*^{*,}4*R*^{*})-4-Carboxybicyclo[2.1.0]pentan-2-aminium 2,2,2-trifluoroacetate (**21**) (COSY) (DMSO-*d*₆)

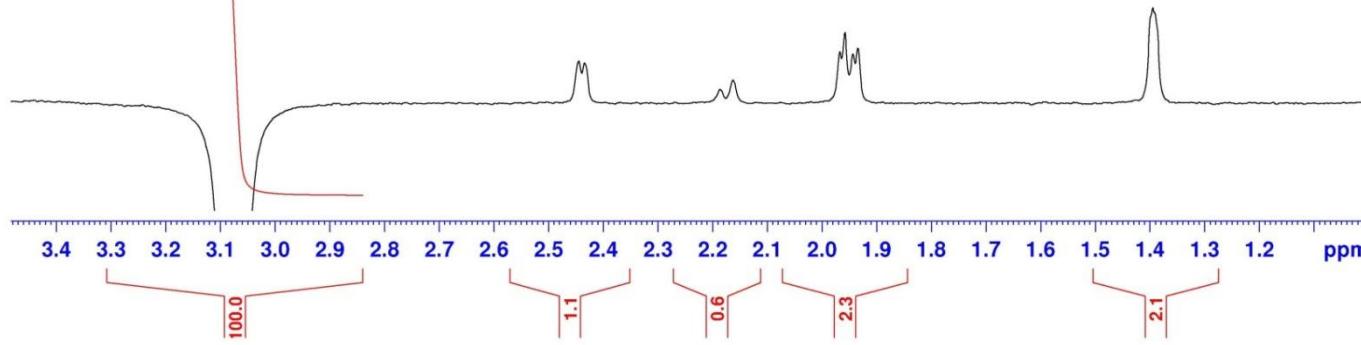
HSQC

(1*S**,2*S**,4*R**)-4-Carboxybicyclo[2.1.0]pentan-2-aminium 2,2,2-trifluoroacetate (**21**) (HSQC) (DMSO-*d*₆)

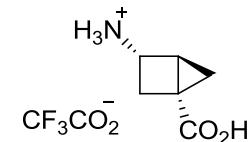
R1256835

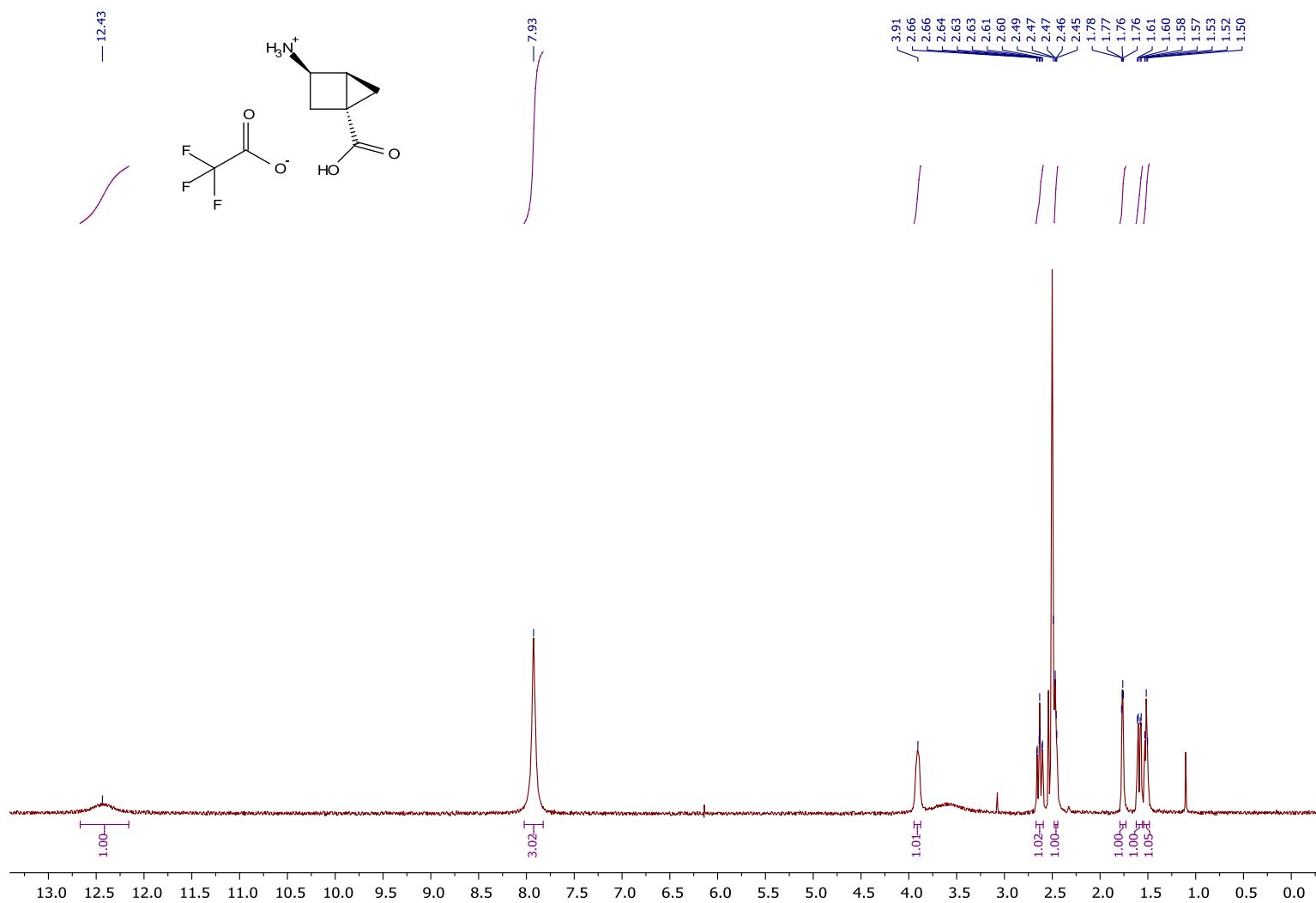


SELNOESY

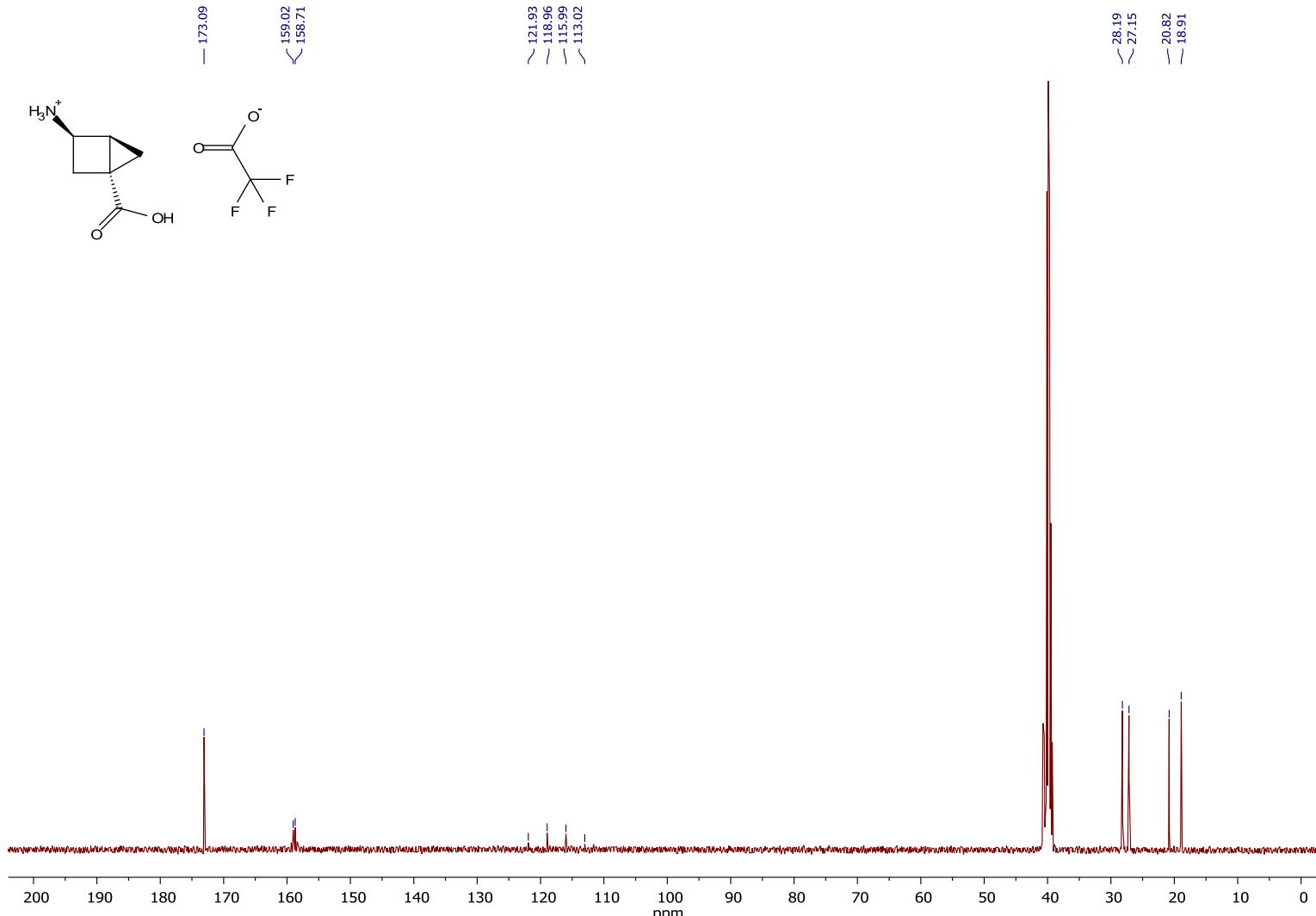


(1*S,2*S**,4*R**)-4-Carboxybicyclo[2.1.0]pentan-2-aminium 2,2,2-trifluoroacetate (21) (H–H NOE) (DMSO-*d*₆)**

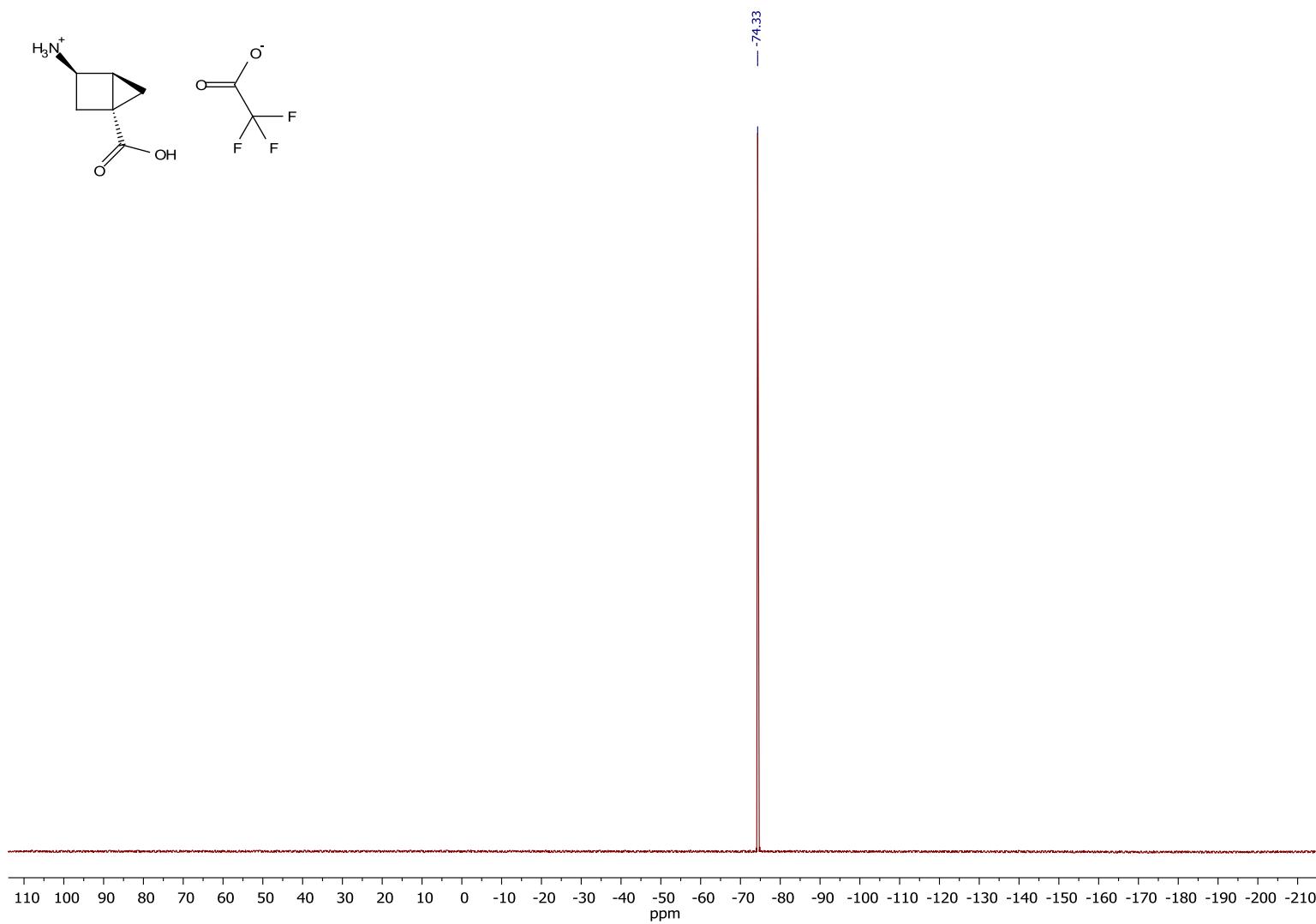
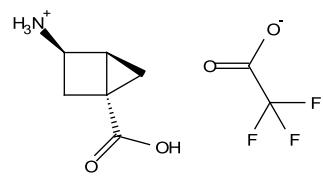




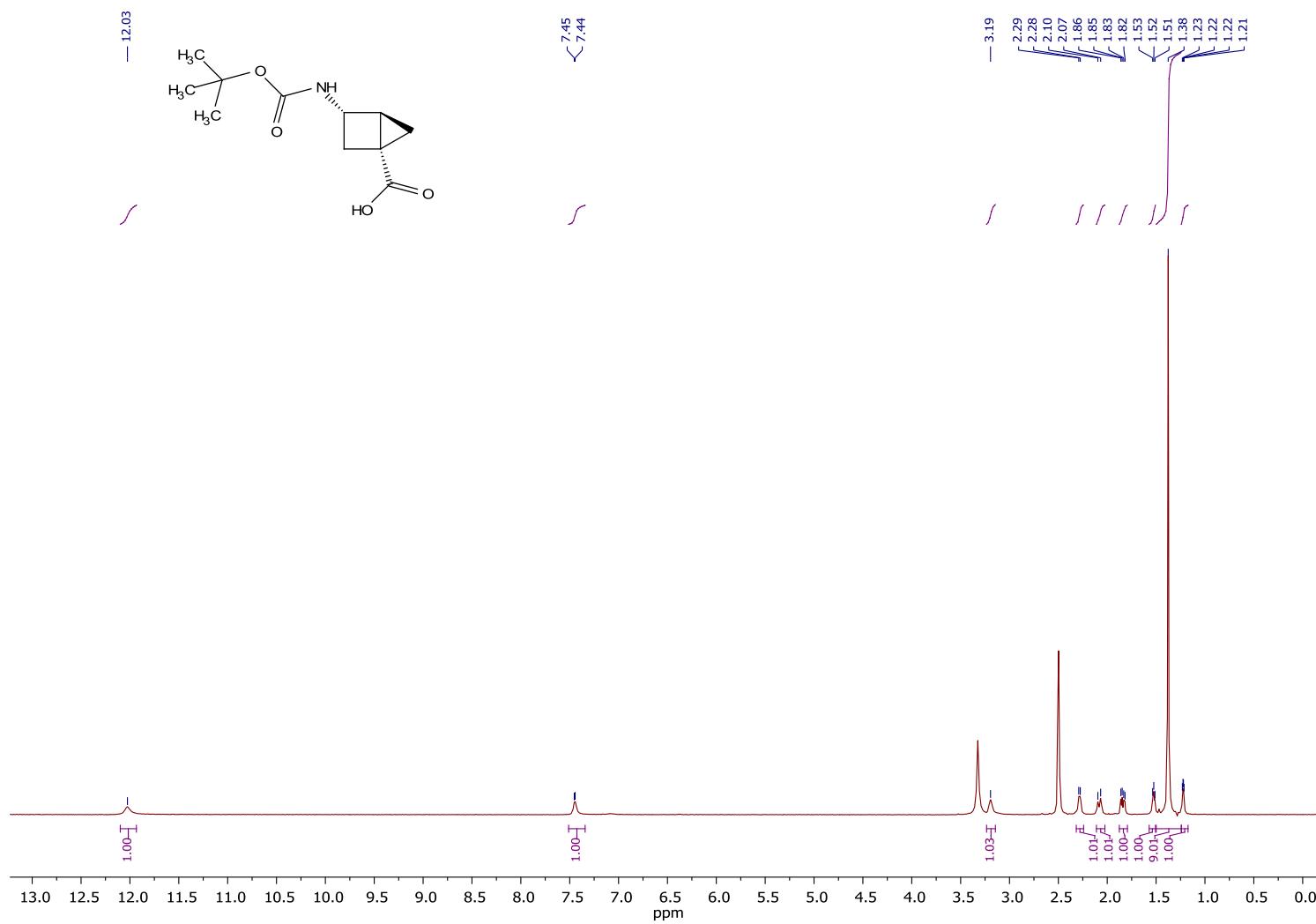
(1*S*^{*,}2*R*^{*,}4*R*^{*})-4-Carboxybicyclo[2.1.0]pentan-2-aminium 2,2,2-trifluoroacetate (22) ¹H NMR (DMSO-*d*₆)



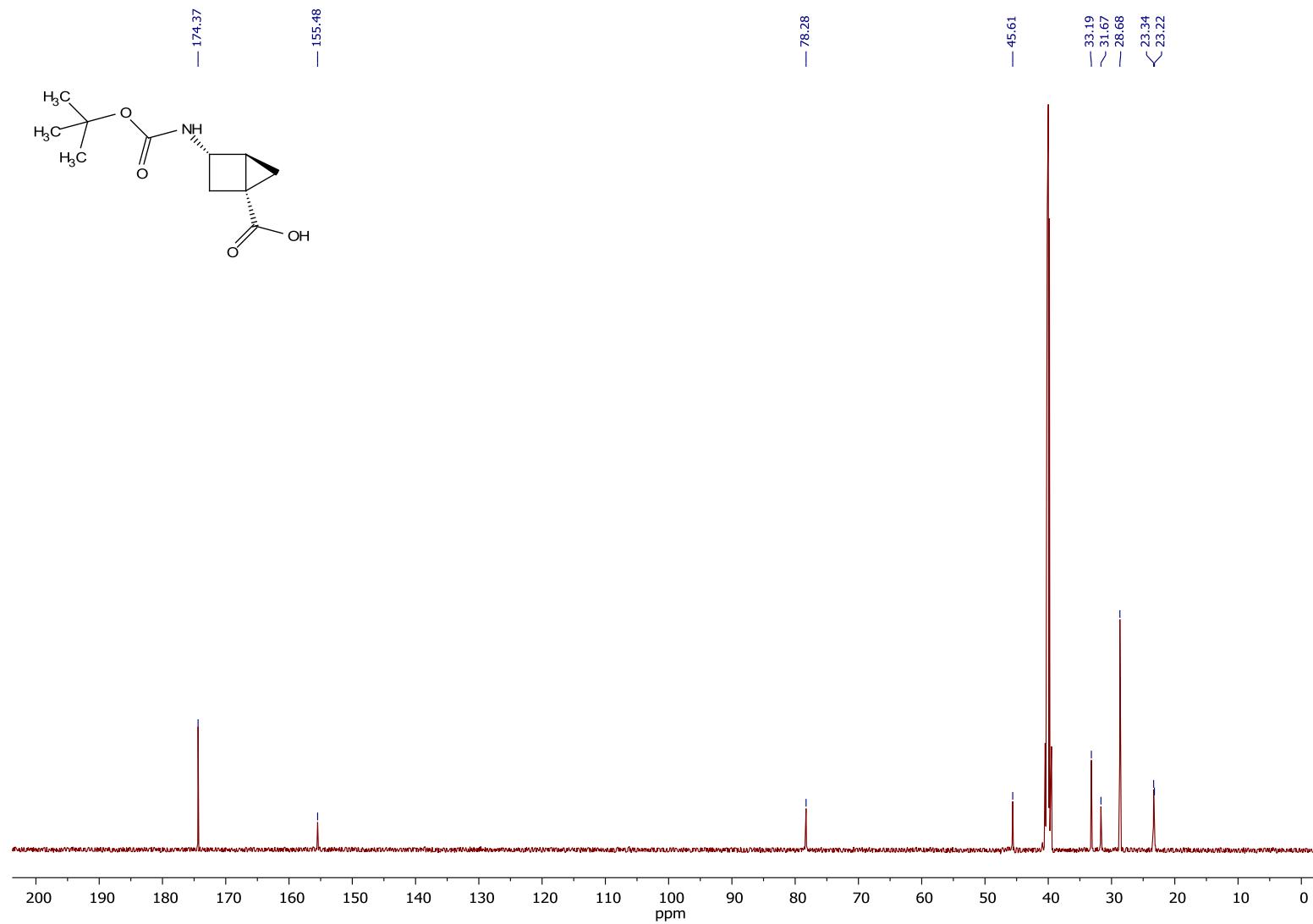
(1*S*^{*,}2*R*^{*,}4*R*^{*})-4-Carboxybicyclo[2.1.0]pentan-2-aminium 2,2,2-trifluoroacetate (**22**) ^{13}C NMR (DMSO-*d*₆)



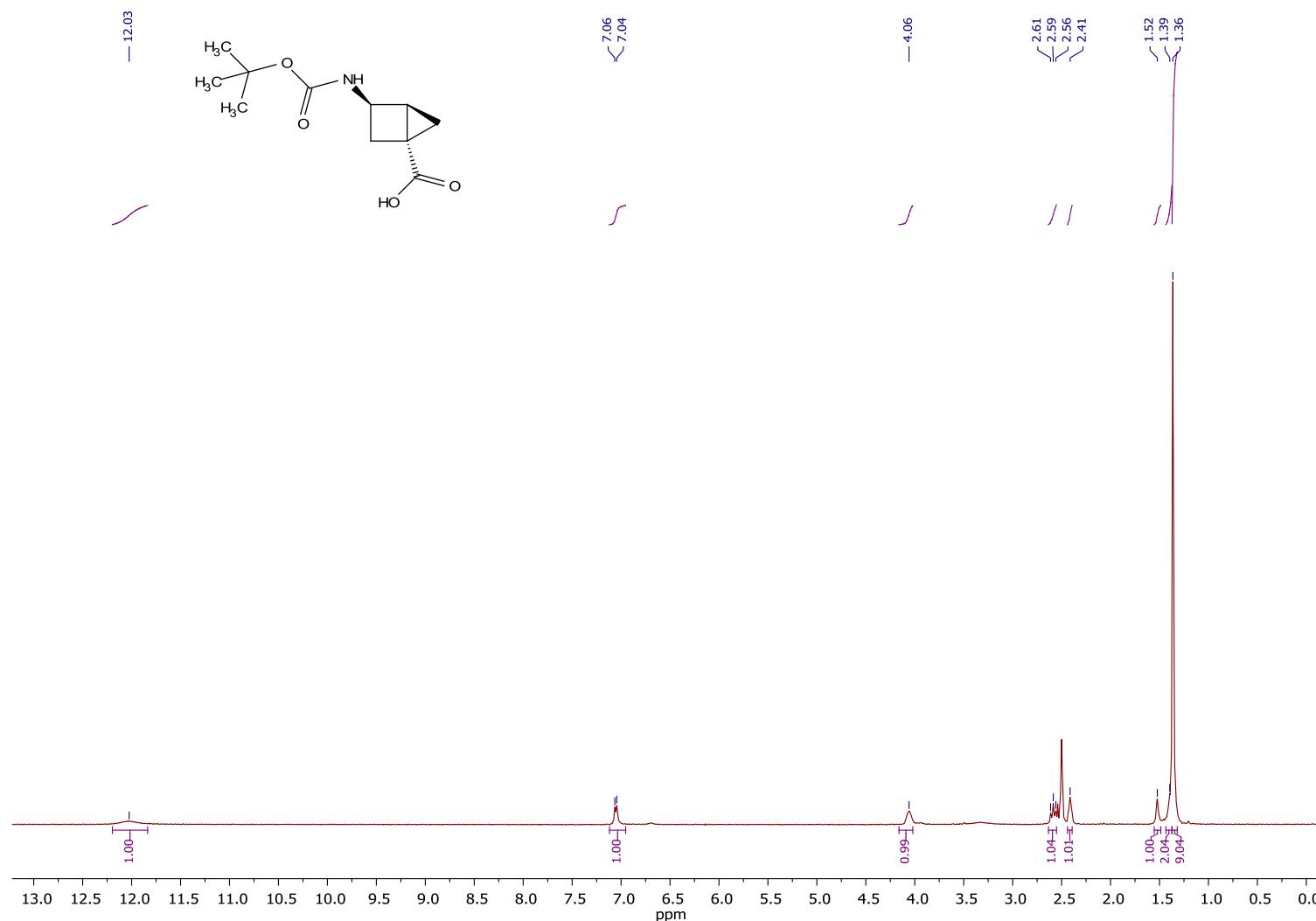
($1S^*,2R^*,4R^*$)-4-Carboxybicyclo[2.1.0]pentan-2-aminium 2,2,2-trifluoroacetate (22) ${}^{19}\text{F}$ NMR (DMSO- d_6)



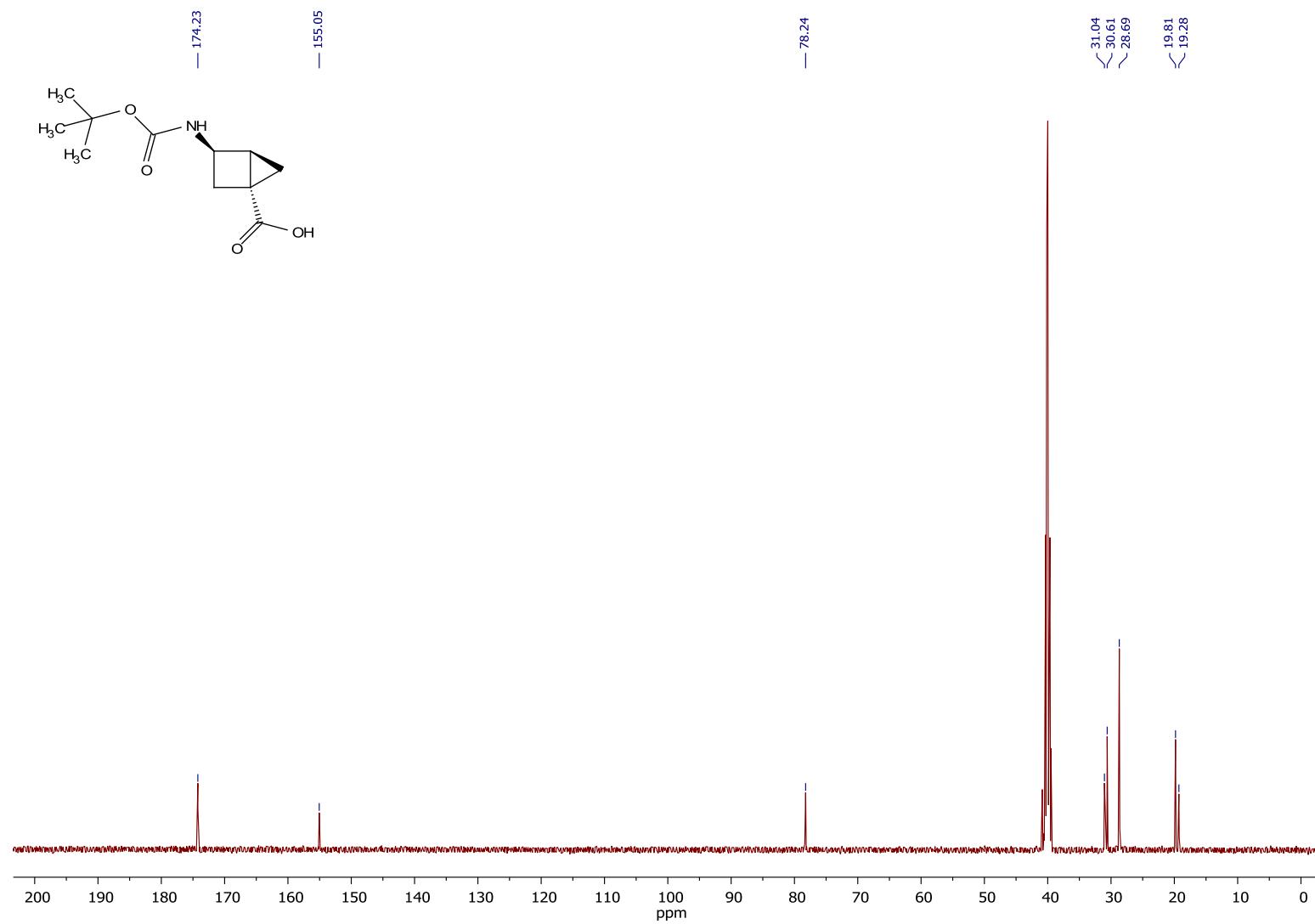
(1*R*^{*},3*S*^{*},4*S*^{*})-3-((*tert*-Butoxycarbonyl)amino)bicyclo[2.1.0]pentane-1-carboxylic acid (23) ¹H NMR (DMSO-*d*₆)



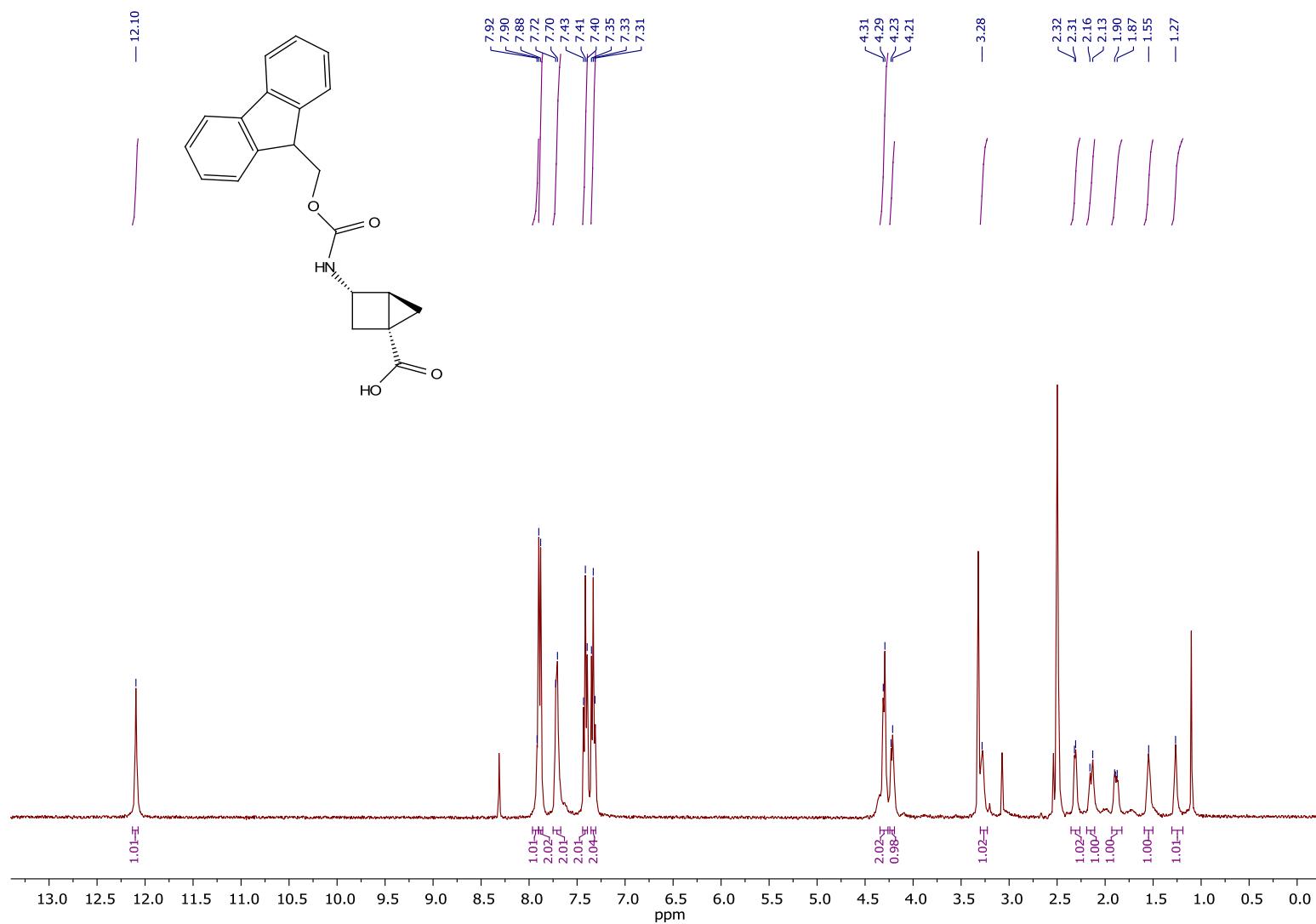
(1*R*^{*,3*S*^{*,4*S*^{*}})-3-((*tert*-Butoxycarbonyl)amino)bicyclo[2.1.0]pentane-1-carboxylic acid (23) ^{13}C NMR (DMSO- d_6)}



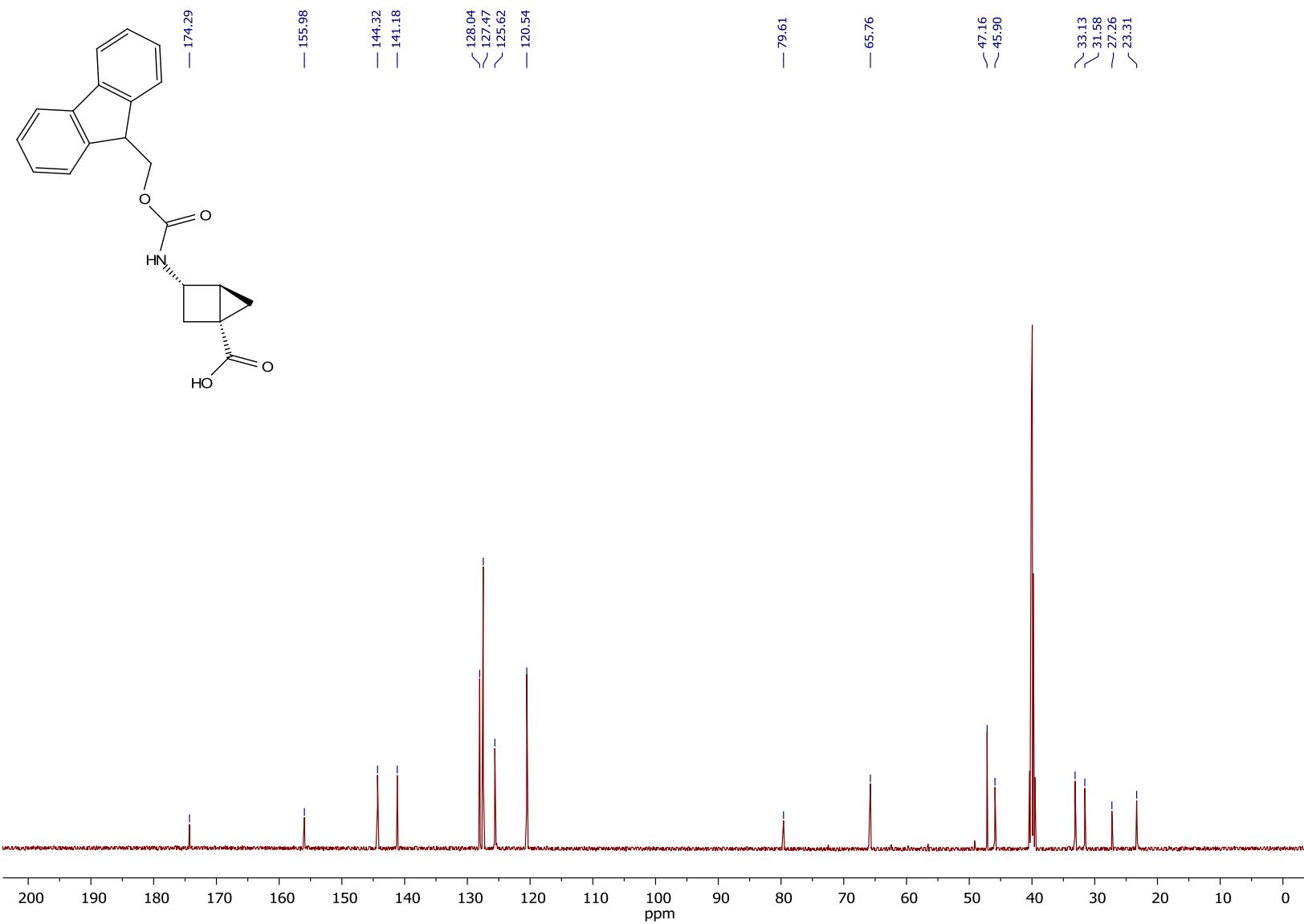
(1*R*^{*,3*R*^{*,4*S*^{*})-3-((tert-Butoxycarbonyl)amino)bicyclo[2.1.0]pentane-1-carboxylic acid (24) ¹H NMR (DMSO-*d*₆)}}



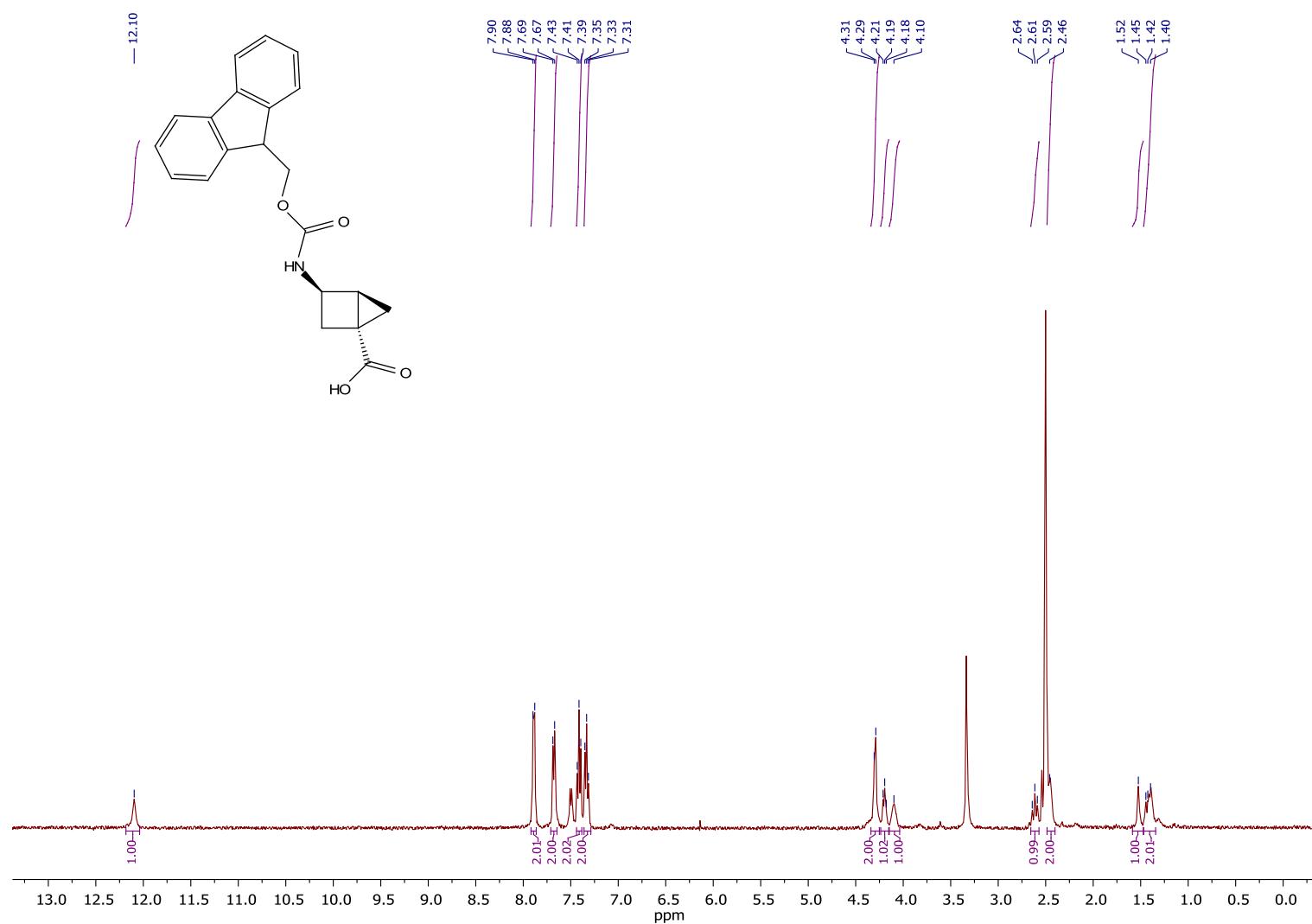
(1*R*^{*,3*R*^{*,4*S*^{*})-3-((*tert*-Butoxycarbonyl)amino)bicyclo[2.1.0]pentane-1-carboxylic acid (24) ^{13}C NMR (DMSO-*d*₆)}}



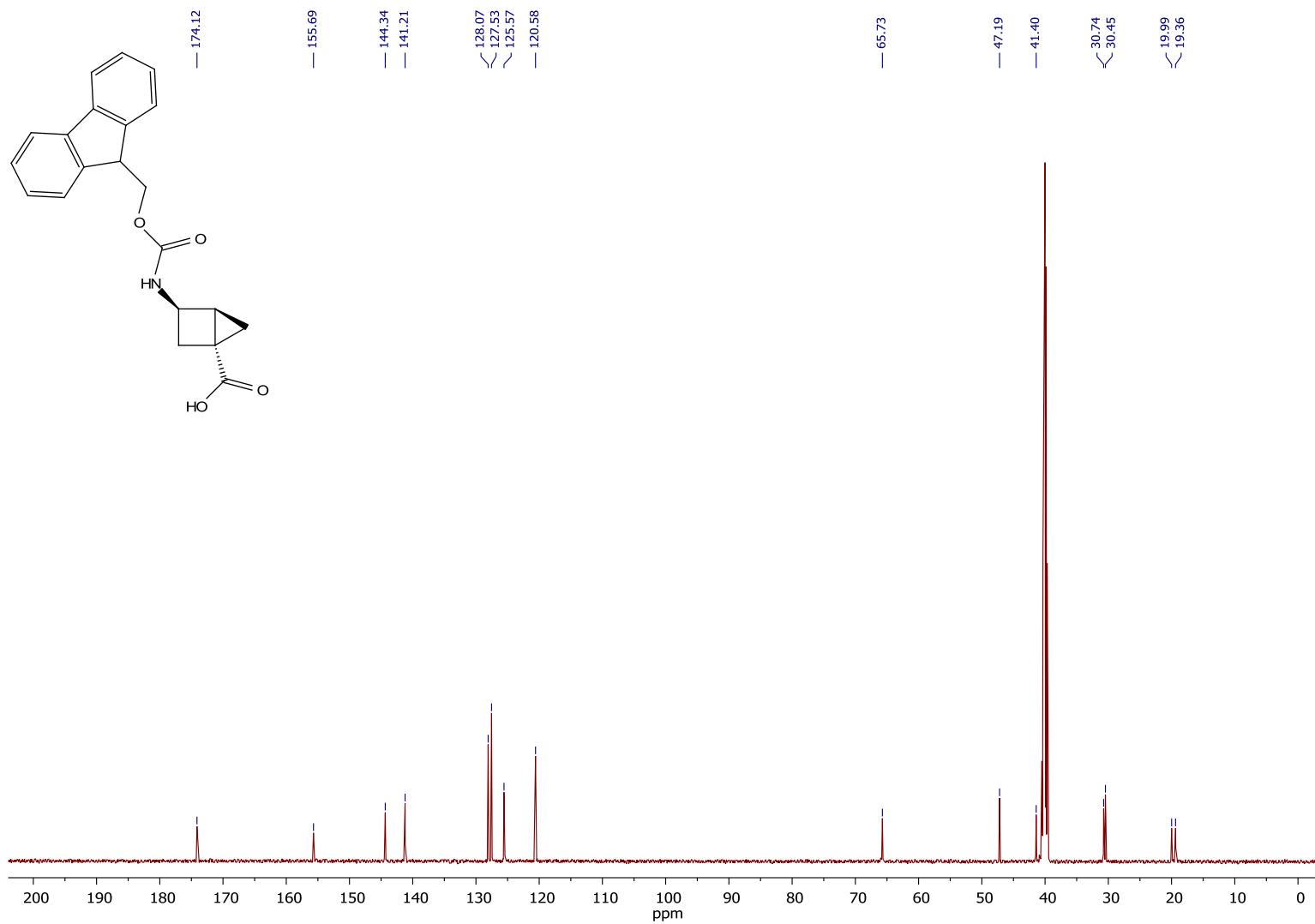
(1*R*^{*},3*S*^{*},4*S*^{*})-3-(((9*H*-Fluoren-9-yl)methoxy)carbonyl)amino)bicyclo[2.1.0]pentane-1-carboxylic acid (25) ¹H NMR (DMSO-*d*₆)



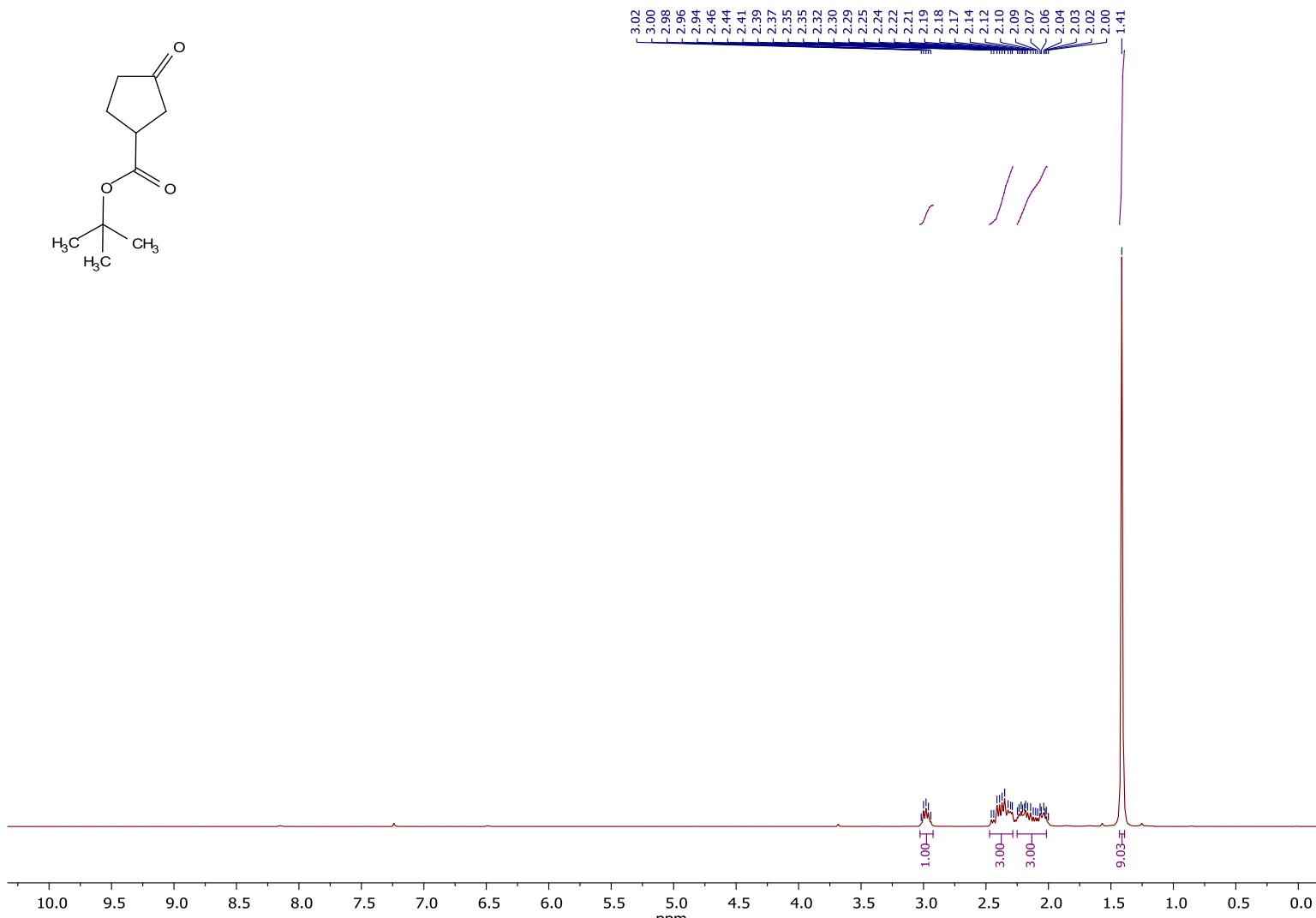
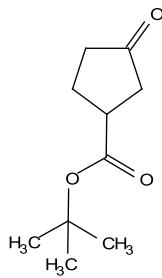
(1*R,3*S**,4*S**)-3-(((9*H*-Fluoren-9-yl)methoxy)carbonyl)amino)bicyclo[2.1.0]pentane-1-carboxylic acid (25) ^{13}C NMR (DMSO-*d*₆)**



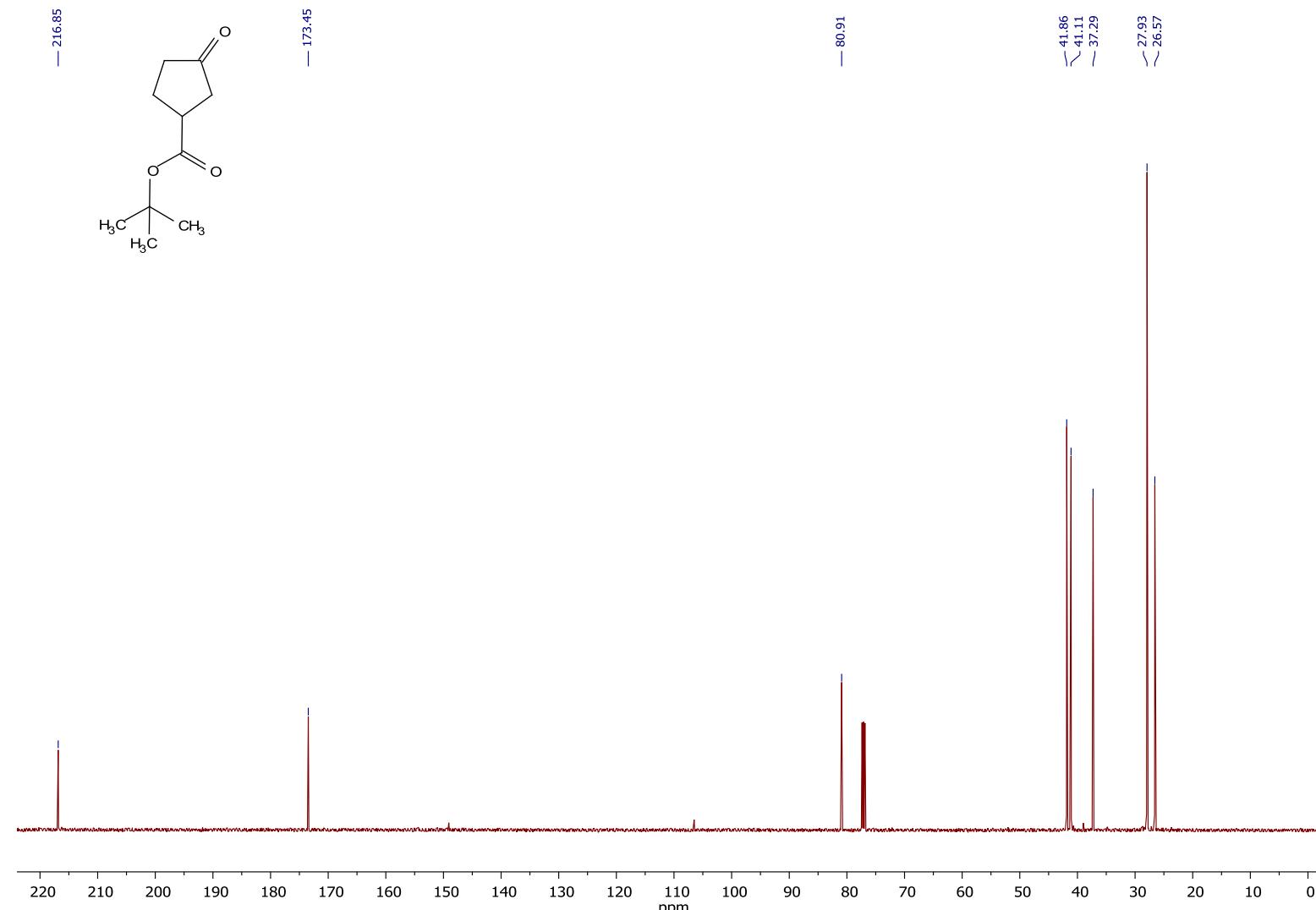
(1*R*^{*,3*R*^{*,4*S*^{*})-3-(((9*H*-fluoren-9-yl)methoxy)carbonyl)amino)bicyclo[2.1.0]pentane-1-carboxylic acid (**26**) ¹H NMR (DMSO-*d*₆)}}



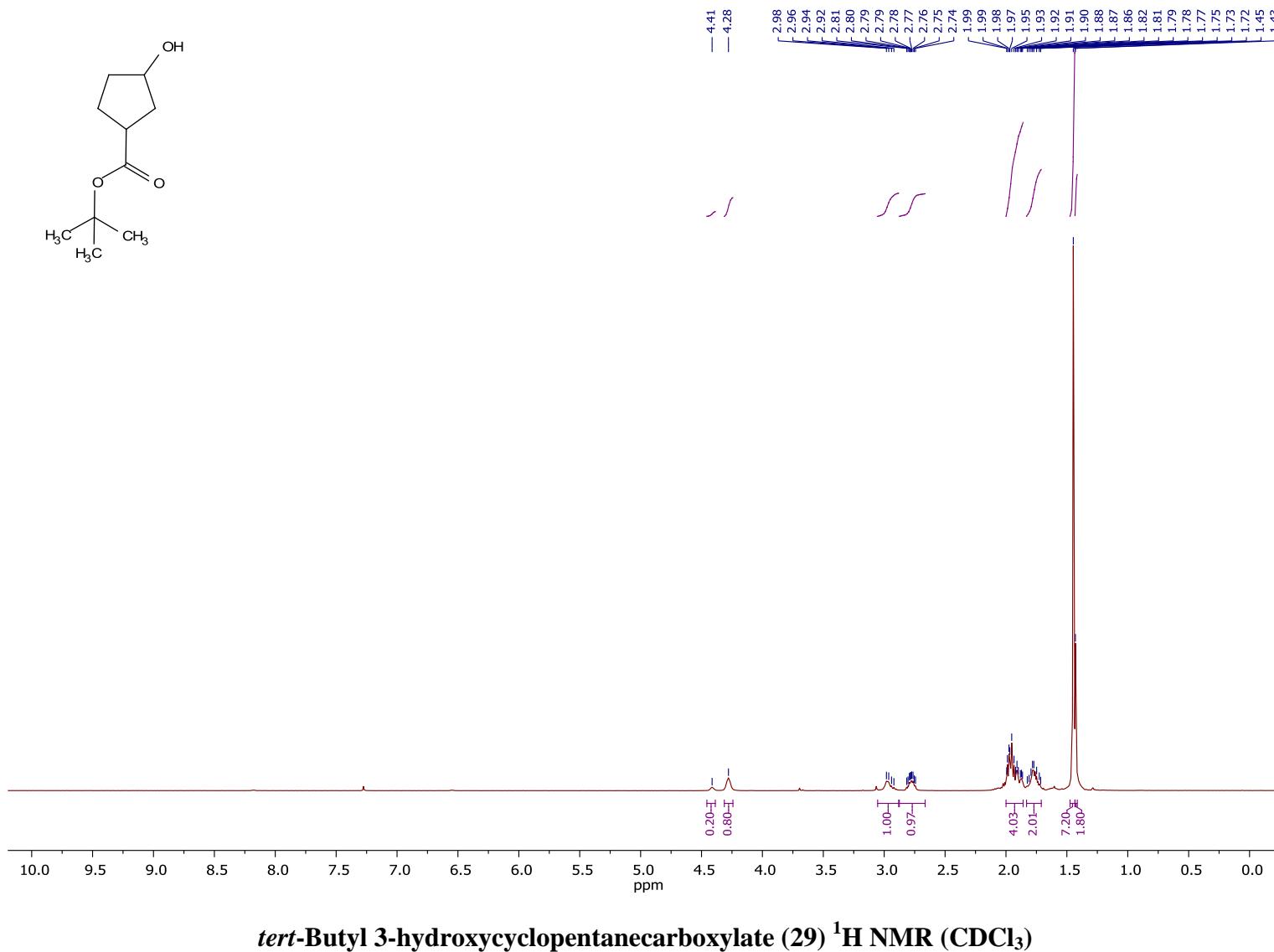
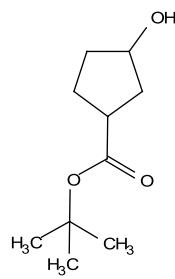
(1*R*^{*,3*R*^{*,4*S*^{*})-3-(((9*H*-fluoren-9-yl)methoxy)carbonyl)amino)bicyclo[2.1.0]pentane-1-carboxylic acid (26)}} ¹³C NMR (DMSO-*d*₆)

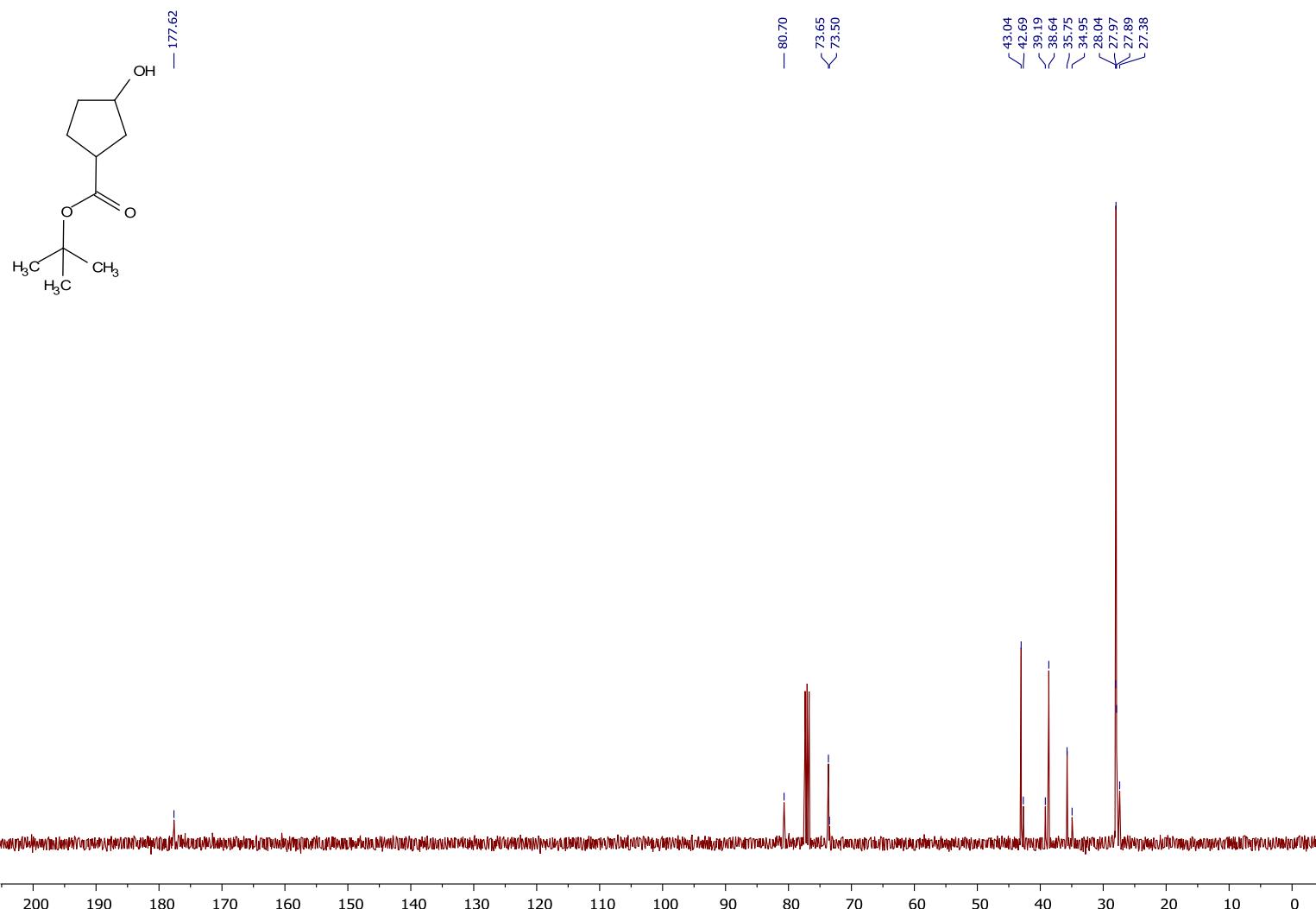


tert-Butyl 3-oxocyclopentanecarboxylate (28) ¹H NMR (CDCl₃)

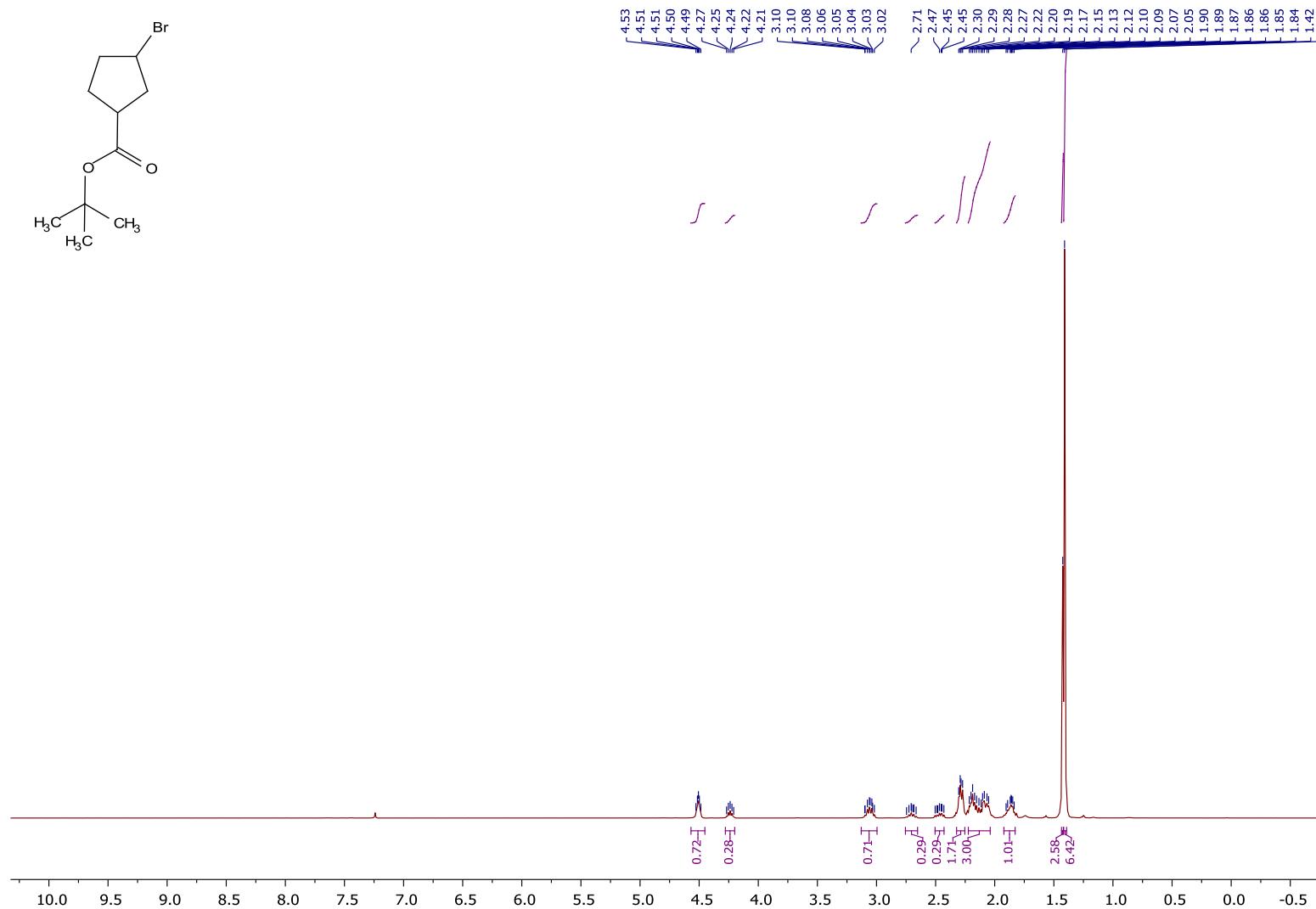
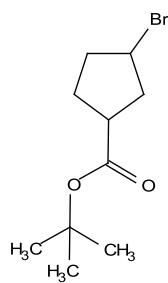


tert-Butyl 3-oxocyclopentanecarboxylate (28) ^{13}C NMR (CDCl_3)

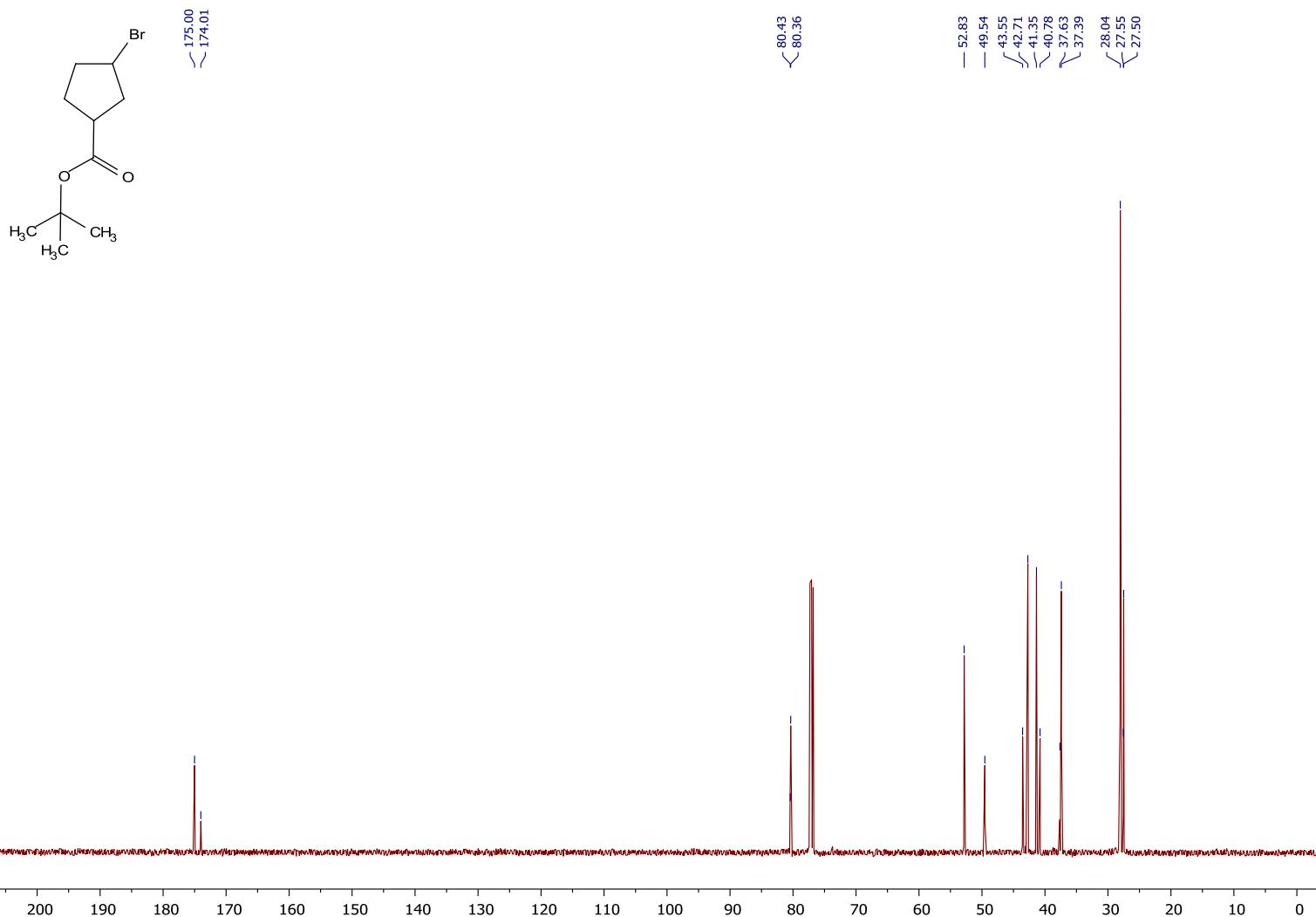


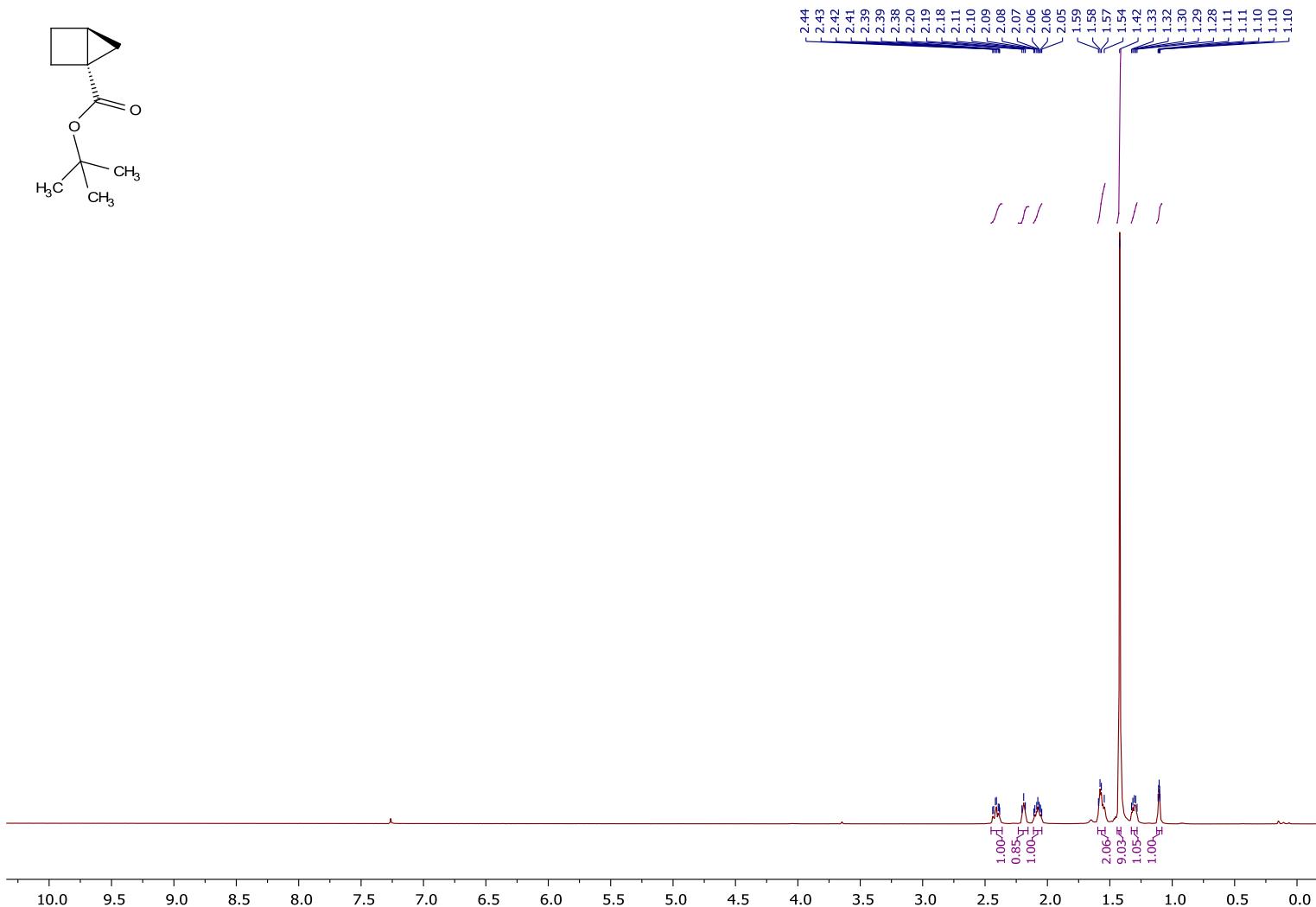
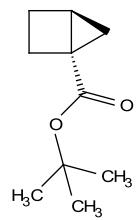


tert-Butyl 3-hydroxycyclopentanecarboxylate (**29**) ^{13}C NMR (CDCl_3)

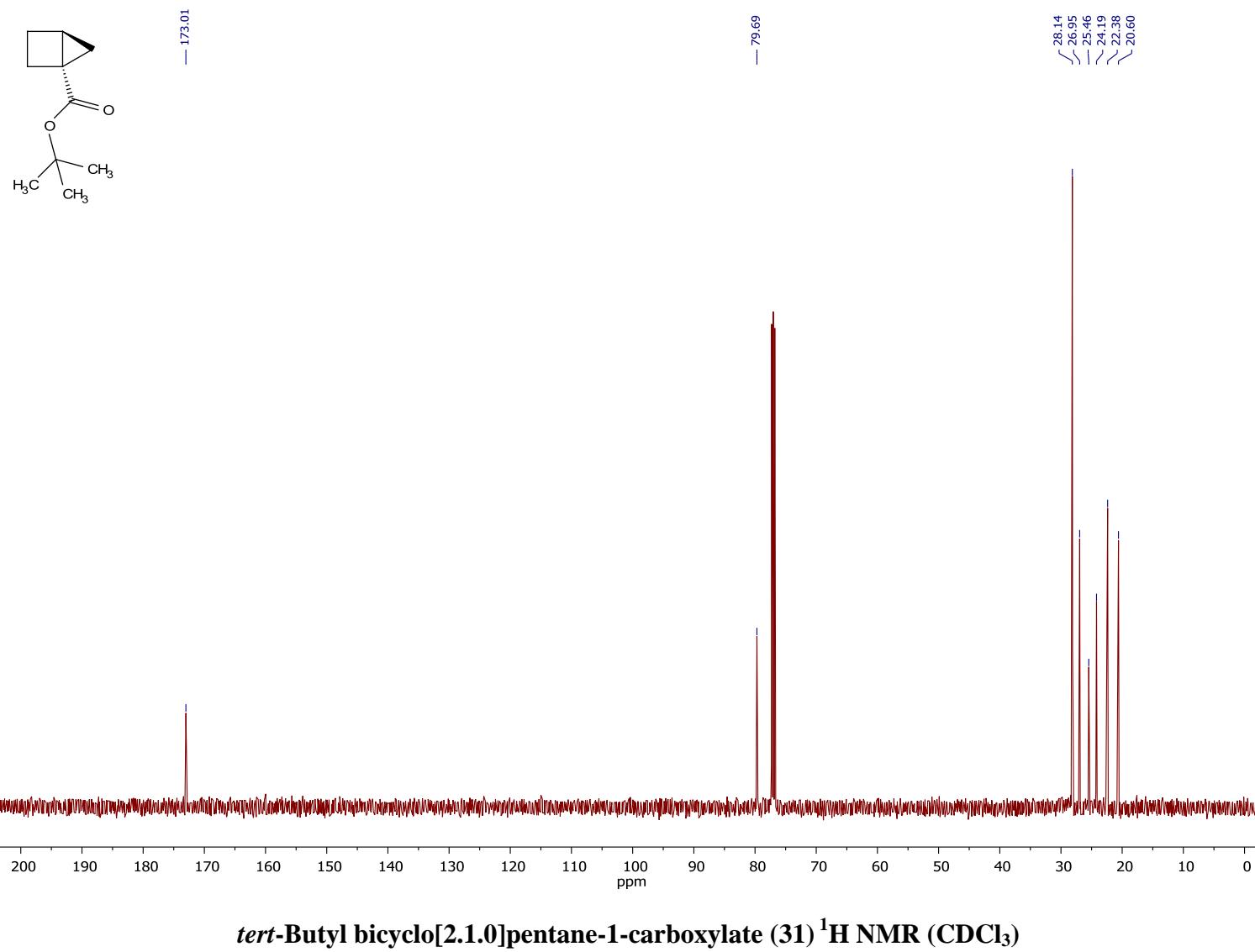


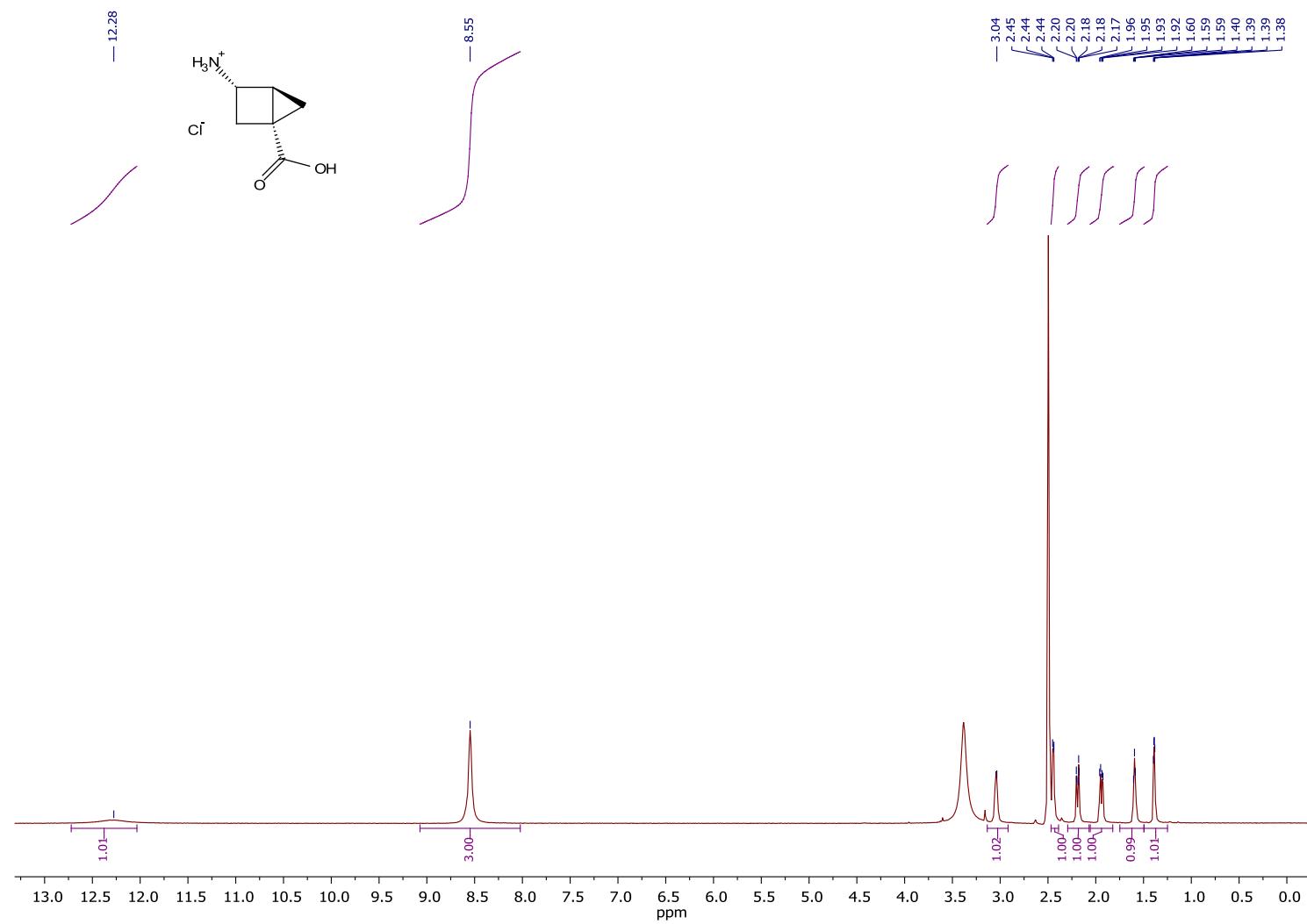
tert-Butyl 3-bromocyclopentanecarboxylate (30) ^1H NMR (CDCl_3)



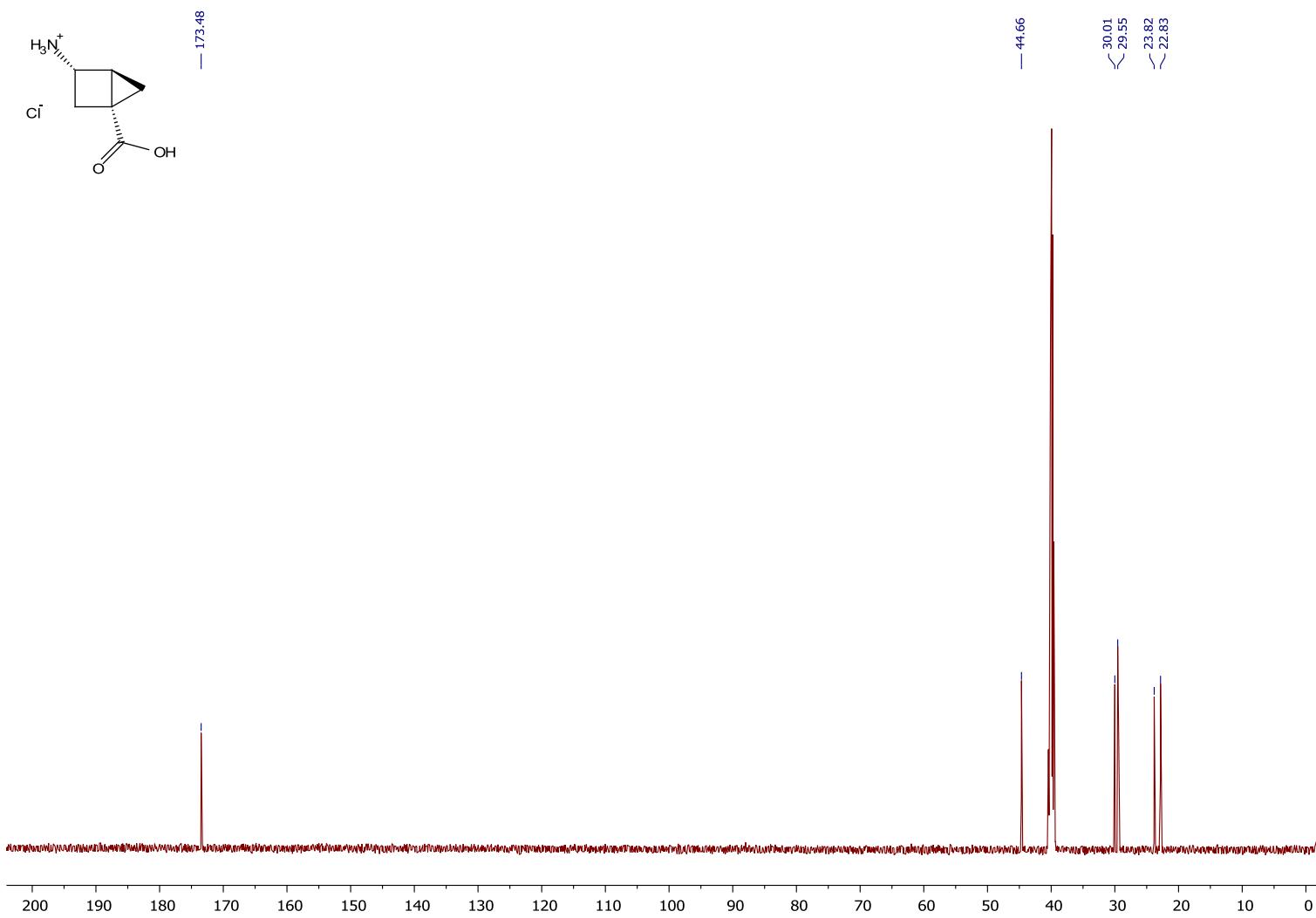


tert-Butyl bicyclo[2.1.0]pentane-1-carboxylate (31) ^1H NMR (CDCl_3)

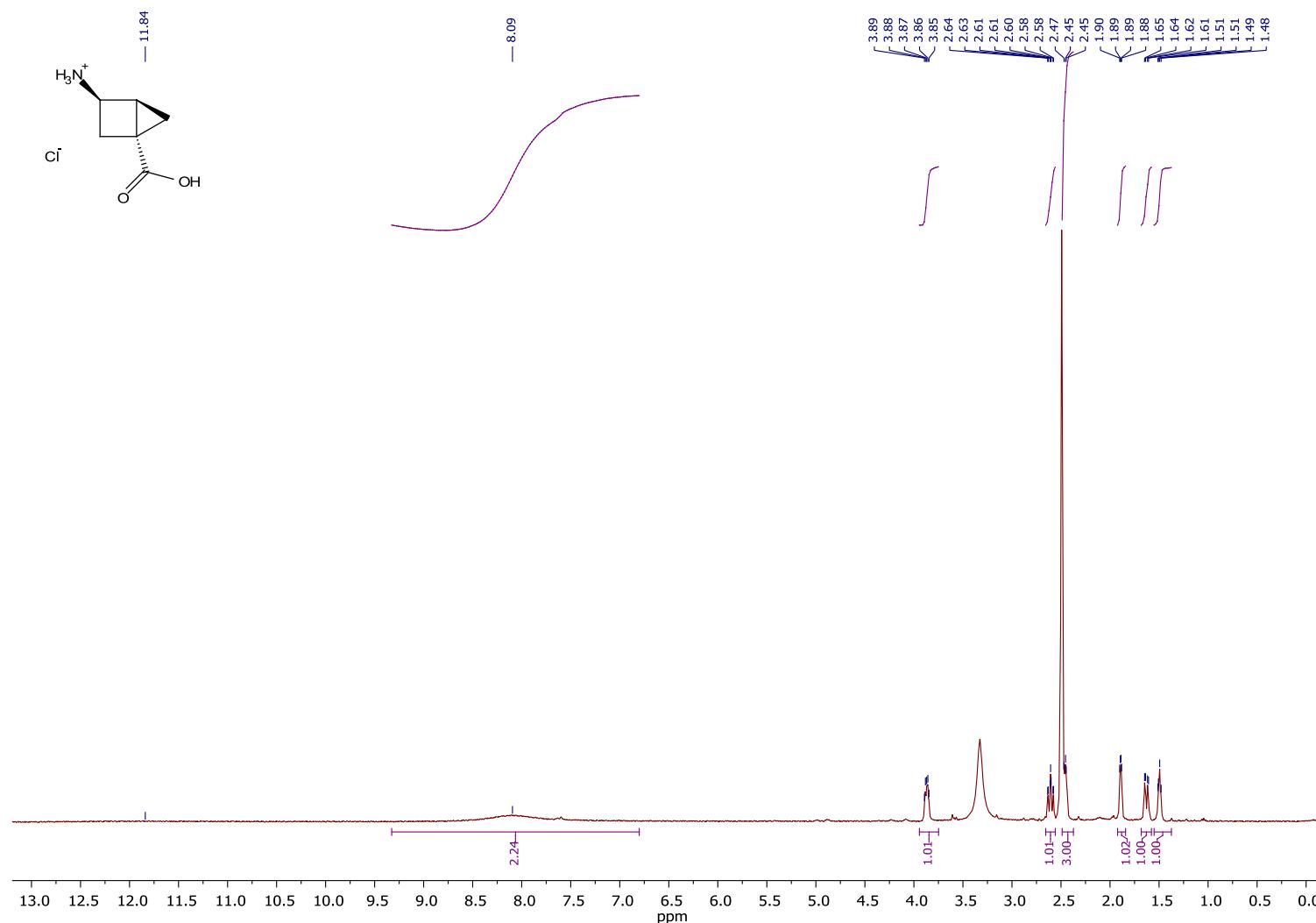


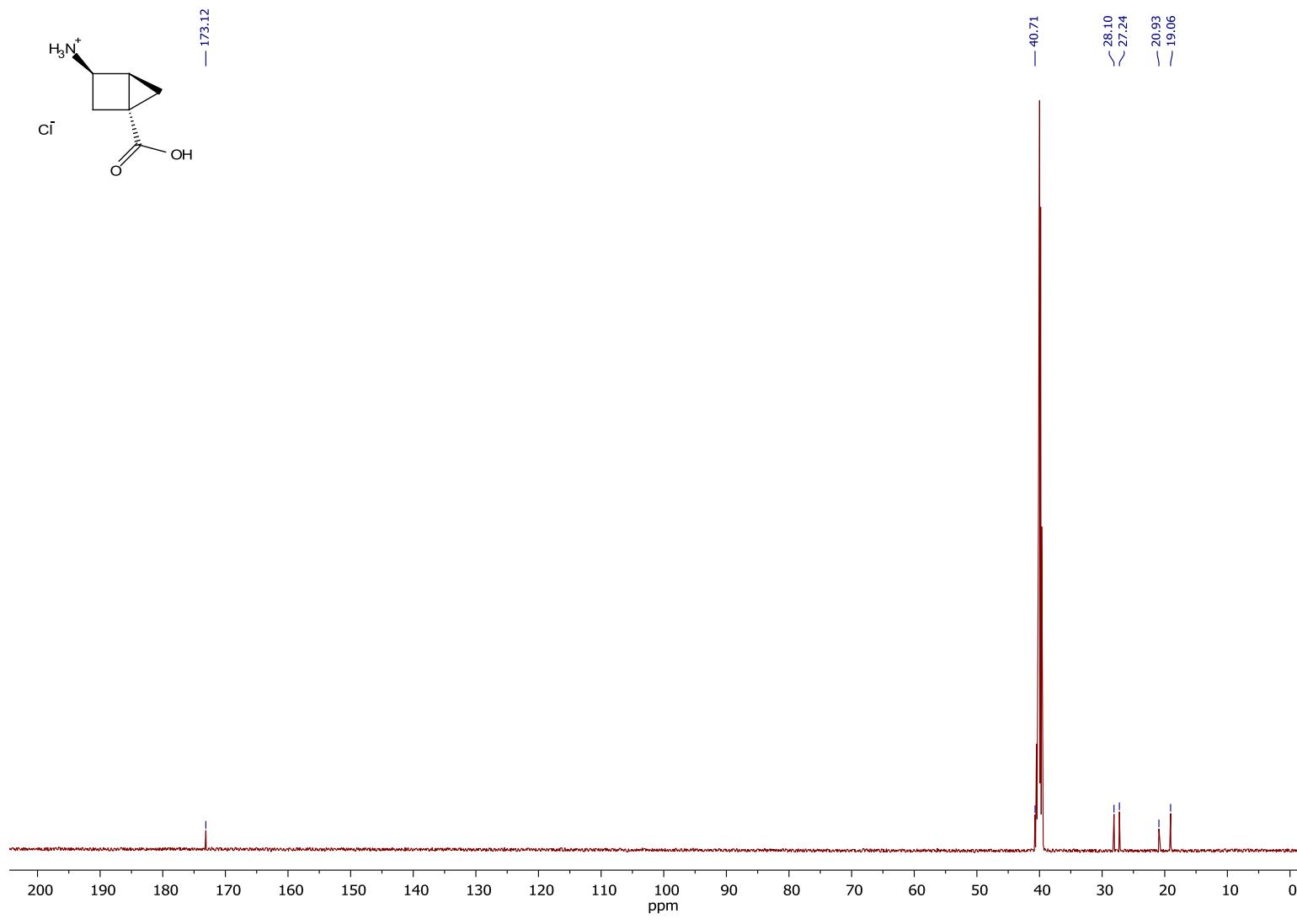


cis-4-Carboxybicyclo[2.1.0]pentan-2-aminium chloride ((1*S*,2*S*,4*R*)-23 or (1*R*,2*R*,4*S*)-23) ^1H NMR (DMSO- d_6)

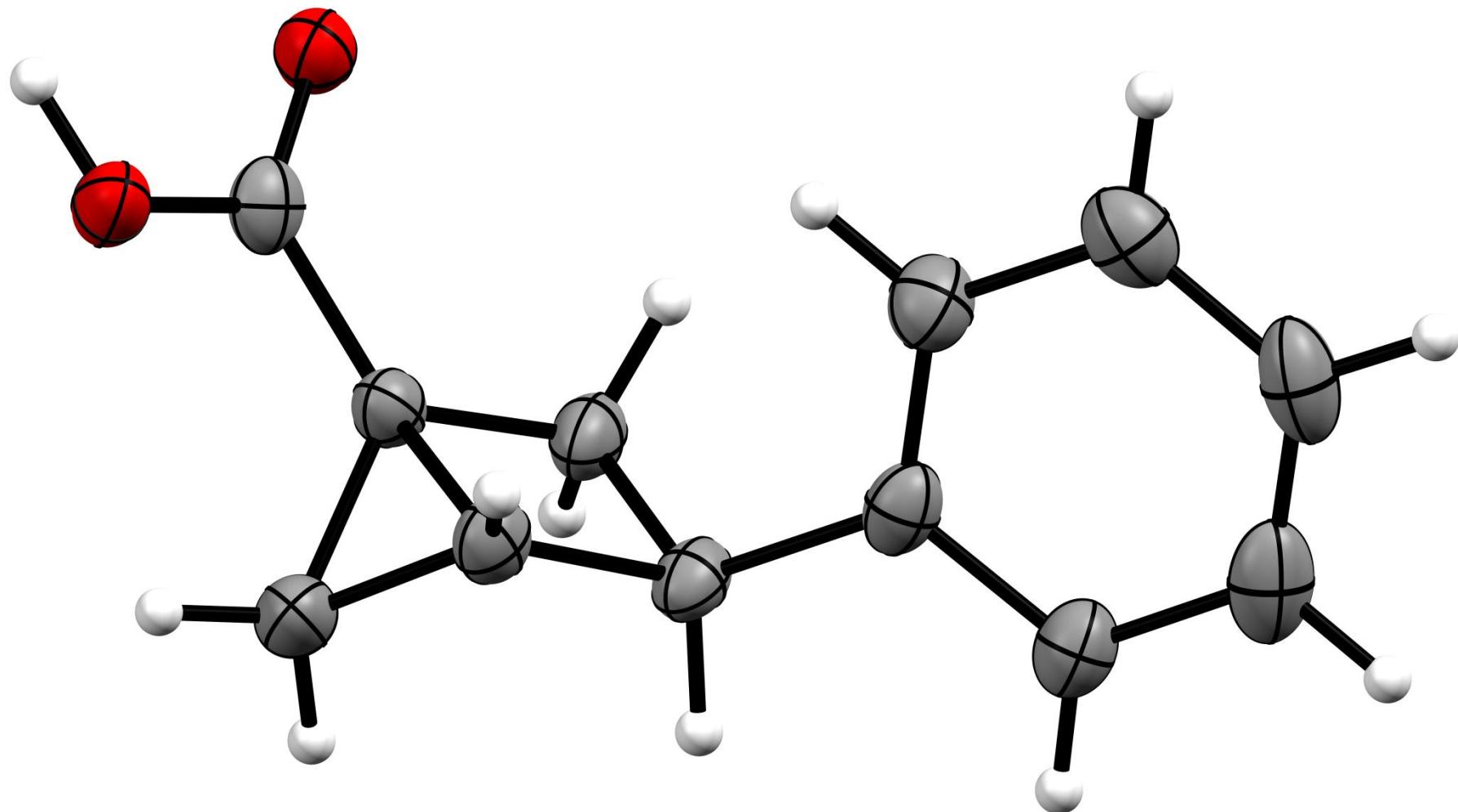


cis-4-Carboxybicyclo[2.1.0]pentan-2-aminium chloride ((1*S*,2*S*,4*R*)-23 or (1*R*,2*R*,4*S*)-23) ^{13}C NMR (DMSO- d_6)

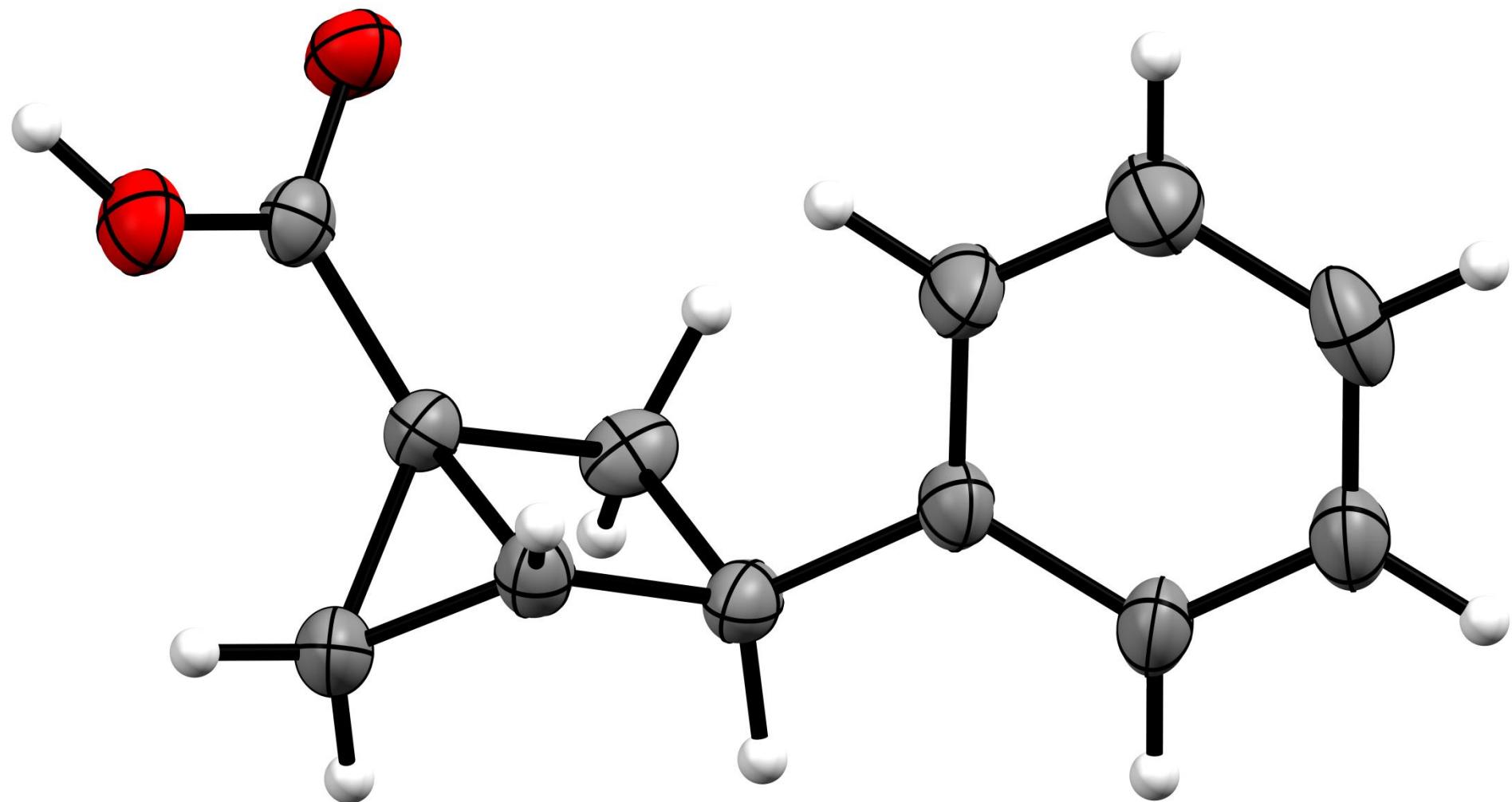




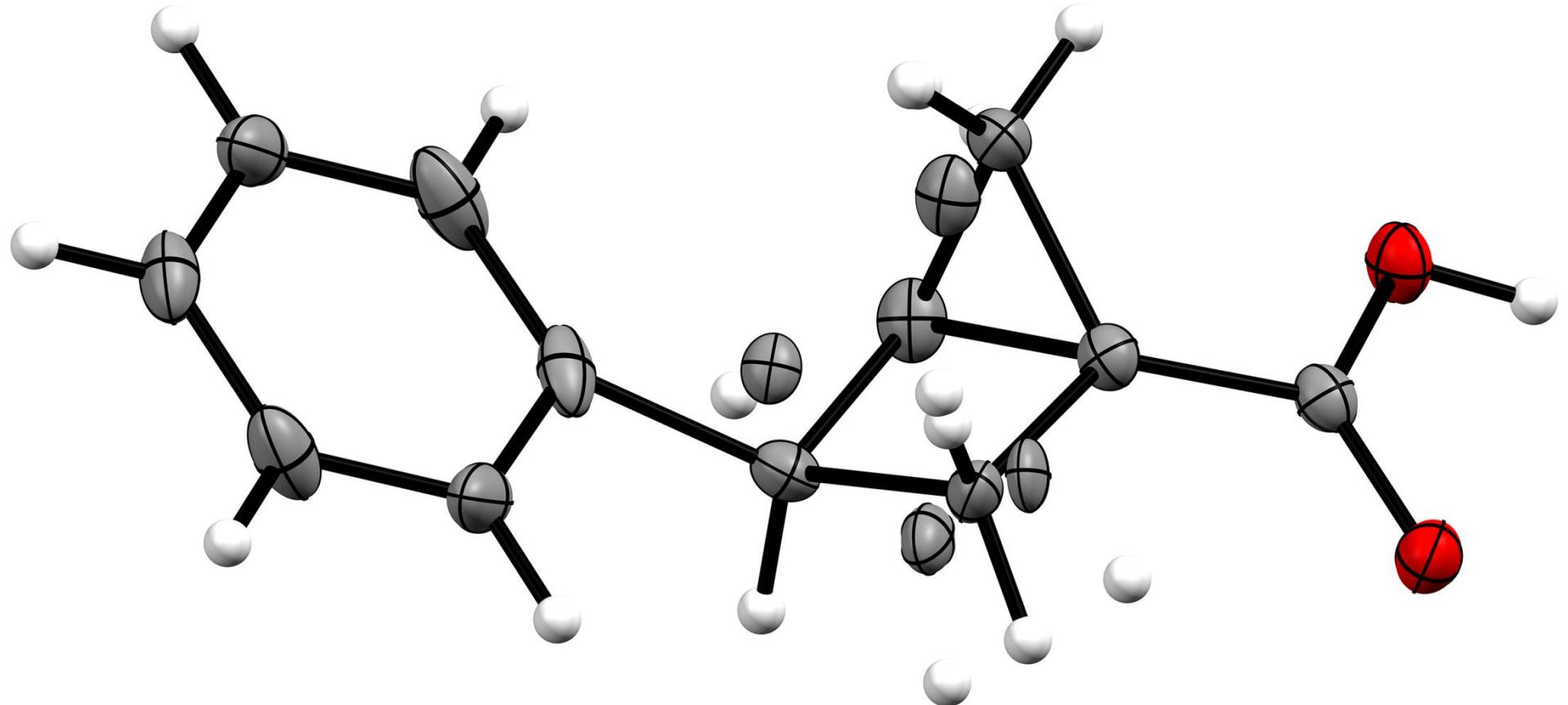
trans-4-Carboxybicyclo[2.1.0]pentan-2-aminium chloride ((1*S*,2*R*,4*R*)-24 or (1*R*,2*S*,4*S*)-24) ^{13}C NMR (DMSO-*d*₆)



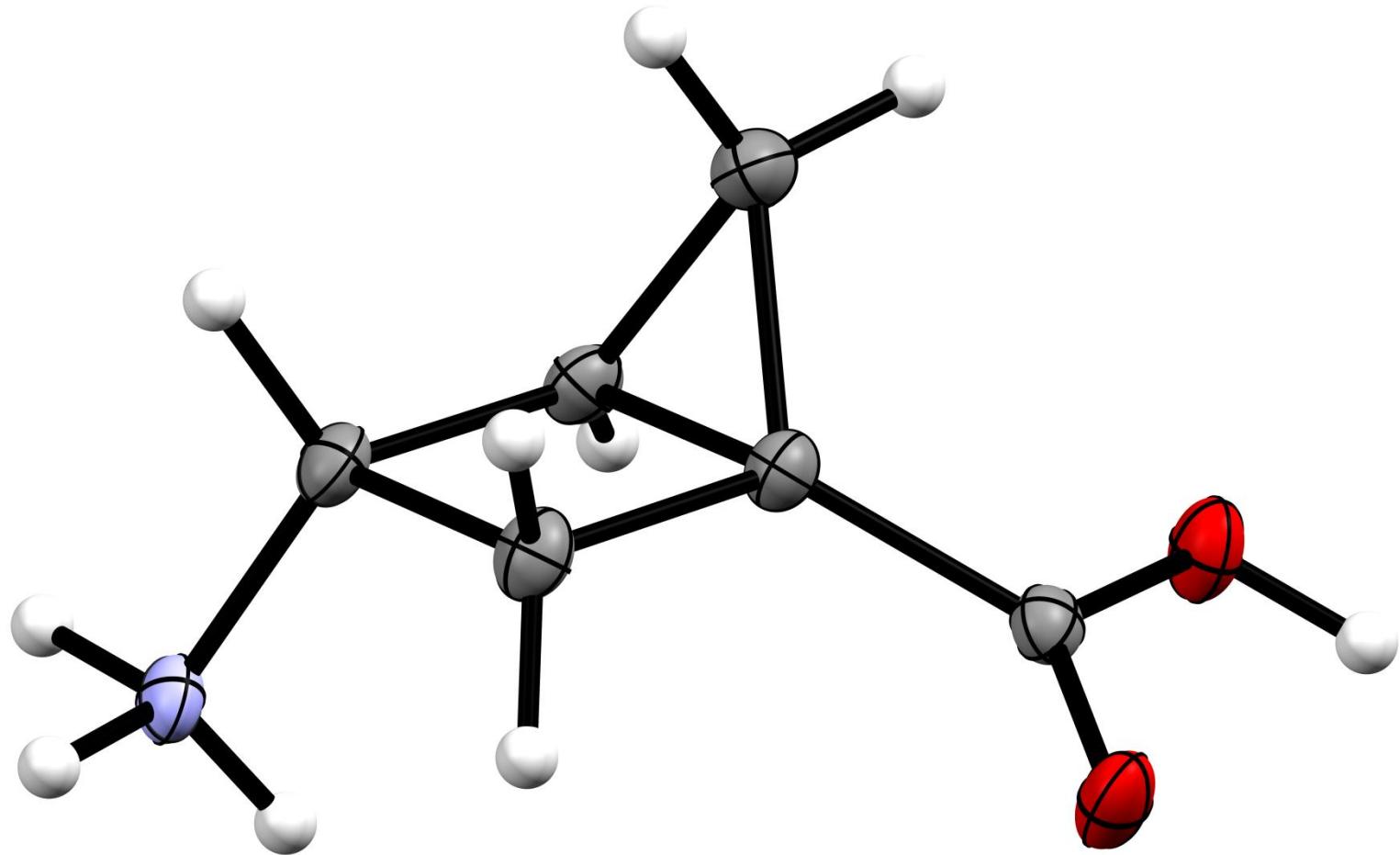
(1*R,3*S**,4*R**)-3-phenylbicyclo[2.1.0]pentane-1-carboxylic acid 10b (conformer A) ORTEP diagrams**
(Thermal ellipsoids are shown at 30% probability level)



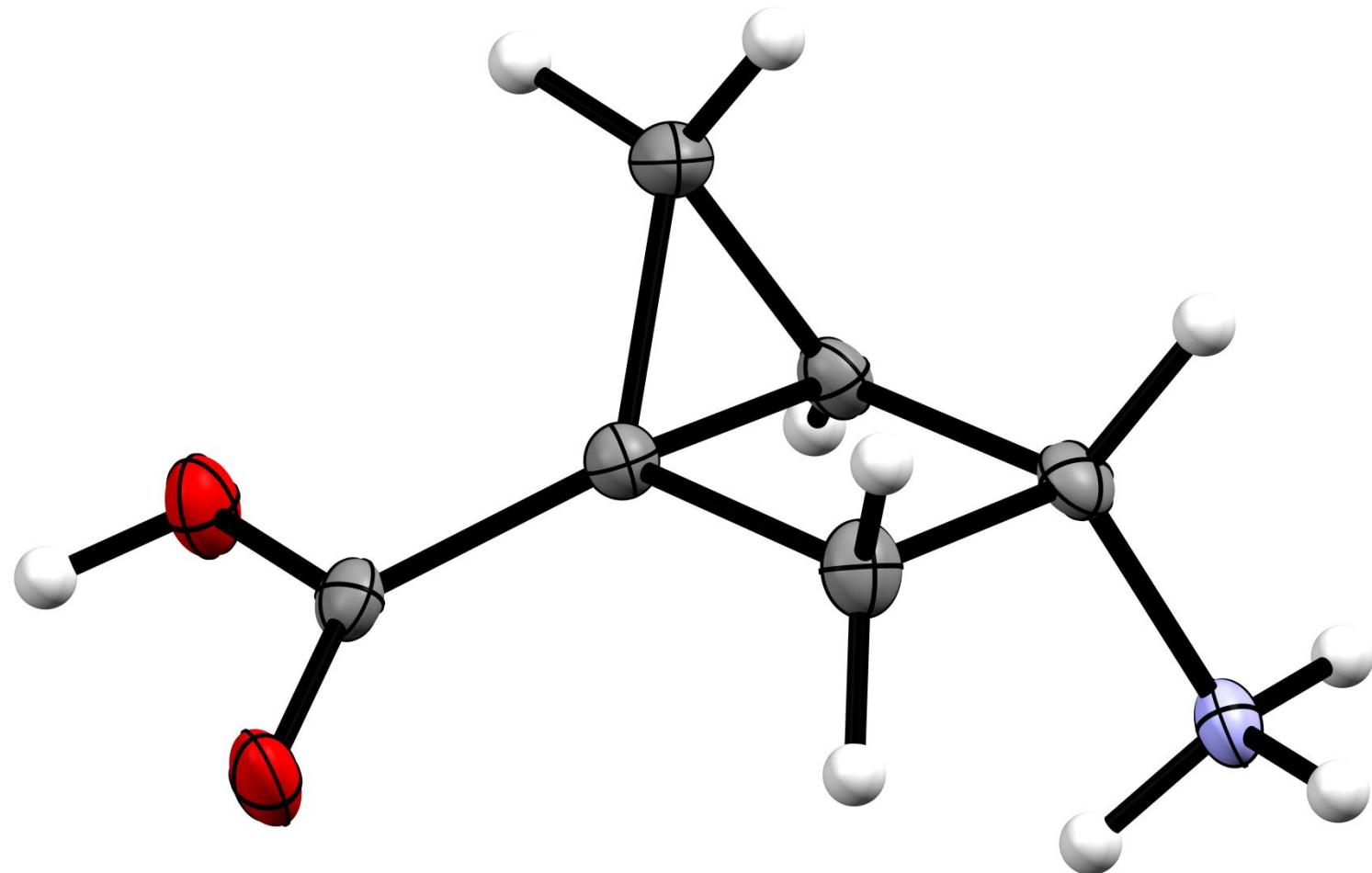
(1*R*^{*},3*S*^{*},4*R*^{*})-3-phenylbicyclo[2.1.0]pentane-1-carboxylic acid 10b (conformer B) ORTEP diagrams
(Thermal ellipsoids are shown at 30% probability level)



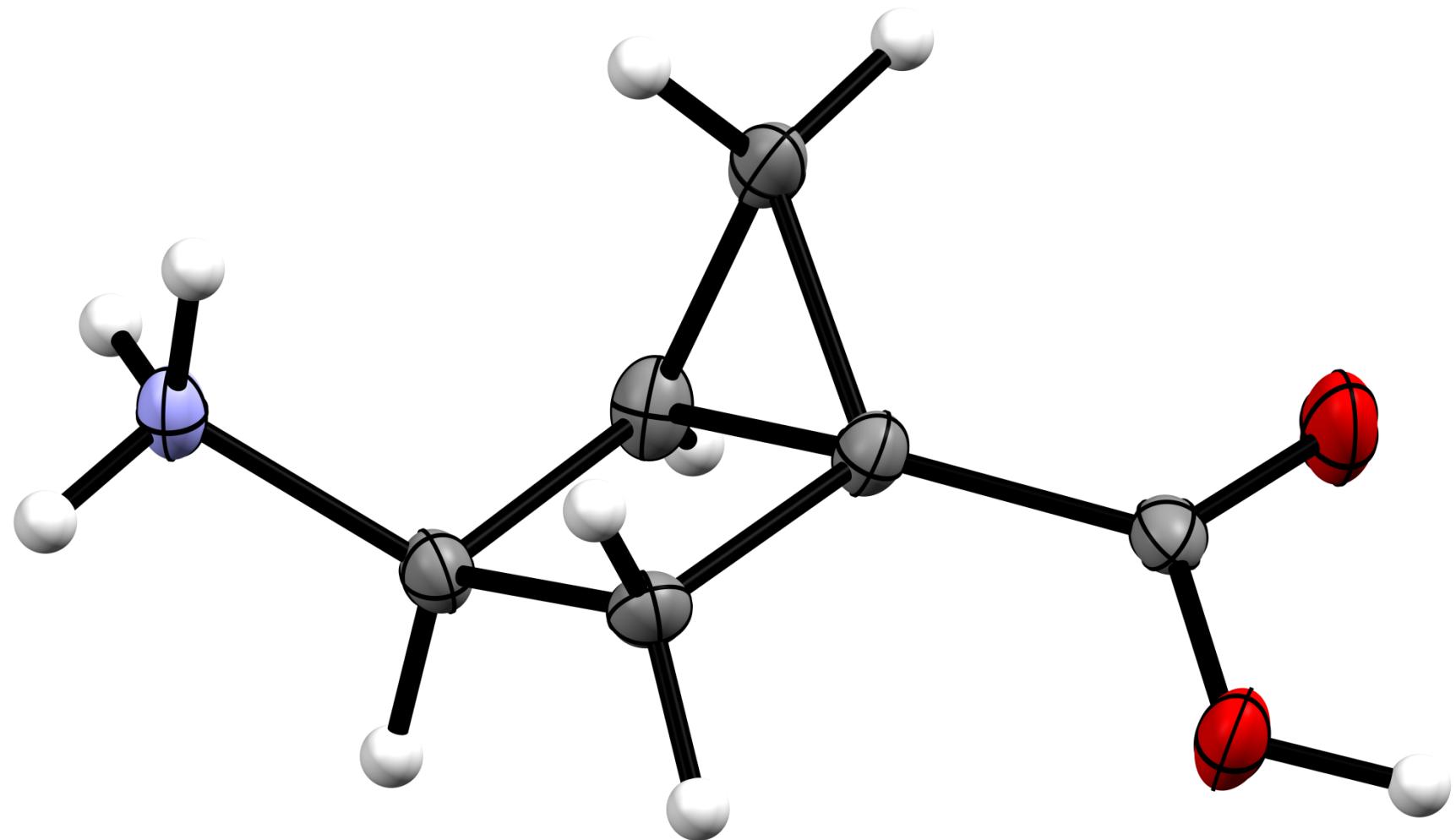
(*1R*,3R*,4R**)-3-phenylbicyclo[2.1.0]pentane-1-carboxylic acid **17b** ORTEP diagrams
(Thermal ellipsoids are shown at 30% probability level)



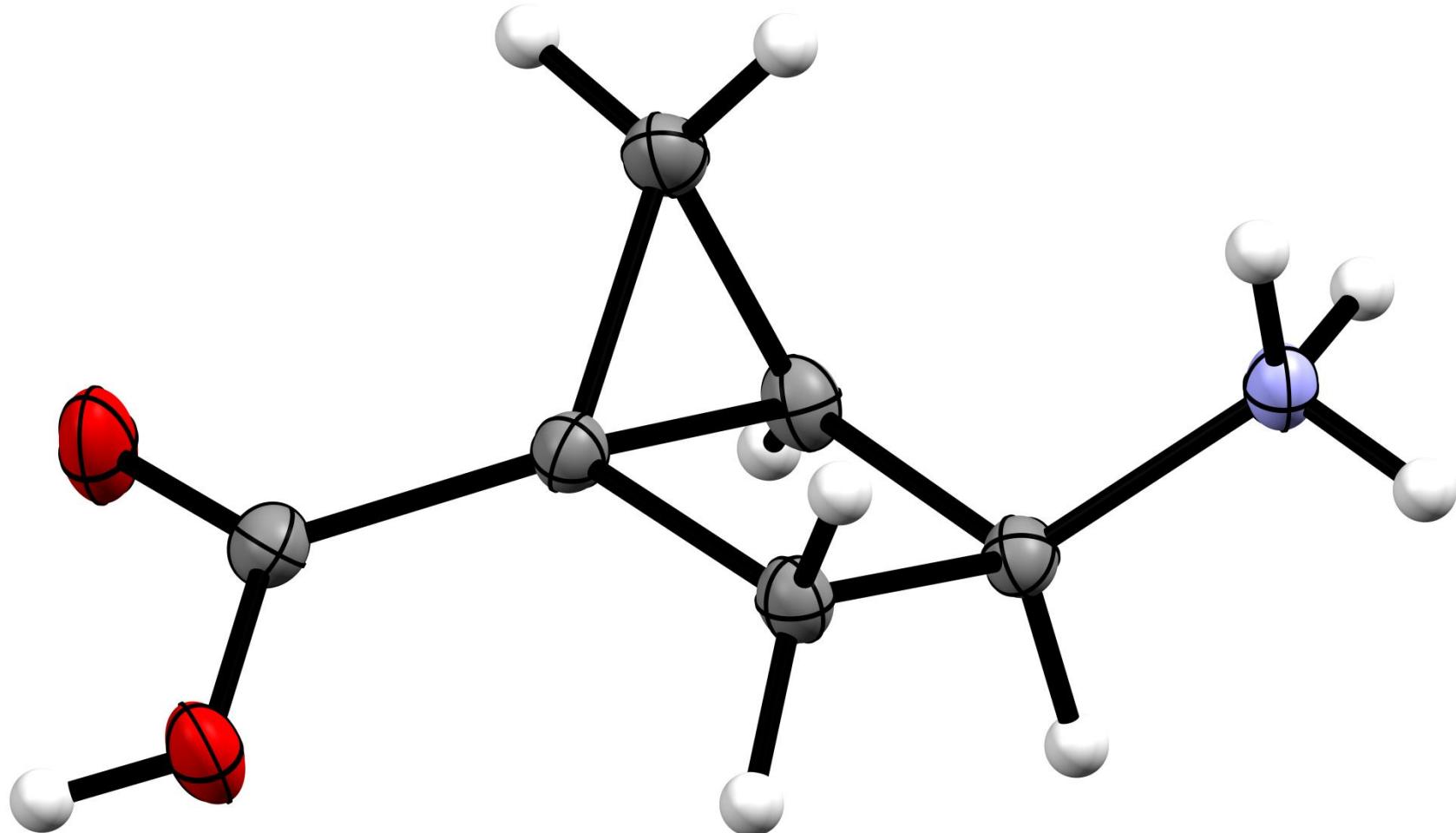
(1*S*,2*S*,4*R*)-3-aminobicyclo[2.1.0]pentane-1-carboxylic acid hydrochloride (1*S*,2*S*,4*R*)-21 ORTEP diagrams
(Thermal ellipsoids are shown at 30% probability level)



(1*R*,2*R*,4*S*)-3-aminobicyclo[2.1.0]pentane-1-carboxylic acid hydrochloride (1*R*,2*R*,4*S*)-21 ORTEP diagrams
(Thermal ellipsoids are shown at 30% probability level)



(1*S*,2*R*,4*R*)-3-aminobicyclo[2.1.0]pentane-1-carboxylic acid hydrochloride (1*S*,2*R*,4*R*)-21 ORTEP diagrams
(Thermal ellipsoids are shown at 30% probability level)



(1*R*,2*S*,4*S*)-3-aminobicyclo[2.1.0]pentane-1-carboxylic acid hydrochloride (1*R*,2*S*,4*S*)-21 ORTEP diagrams
(Thermal ellipsoids are shown at 30% probability level)

X-Ray diffraction studies of **10b, **17b**, (**1S,2S,4R**)-**21**, (**1R,2R,4S**)-**21**, (**1S,2R,4R**)-**22**, (**1R,2S,4S**)-**22**.**

X-Ray diffraction studies were performed with single crystals of housane derivatives obtained by slow evaporation of their solutions: *cis* phenyl-substituted housane carboxylic acid **10b** (in *t*-BuOMe – hexanes), its *trans* isomer **17b** (in THF), as well as amino acid hydrochlorides (**1S,2S,4R**)-**21**·HCl·H₂O (in MeCN – EtOH), (**1R,2R,4S**)-**21**·HCl·H₂O (in MeCN – EtOH); (**1S,2R,4R**)-**22**·HCl (in MeOH), and (**1R,2S,4S**)-**22**·HCl (in MeCN – EtOH).

X-Ray diffraction studies were performed on an automatic diffractometer (graphite monochromated MoK_α radiation, CCD-detector, ω -scanning, $2\theta_{\max} = 60^\circ$). The structure was solved by direct method using SHELXTL package. The crystallographic data and experimental parameters are listed in Table S1. Final atomic coordinates, geometrical parameters and crystallographic data have been deposited with the Cambridge Crystallographic Data Centre, 11 Union Road, Cambridge, CB2 1EZ, UK (fax: +44 1223 336033; e-mail: deposit@ccdc.cam.ac.uk). The deposition numbers are given in Table S1.

Table S1. The crystallographic data and experimental parameters for compounds

10b, **17b**, (**1S,2S,4R**)-**21**, (**1R,2R,4S**)-**21**, (**1S,2R,4R**)-**22**, (**1R,2S,4S**)-**22**

Parameter	10b	17b	(1S,2S,4R)- 21)	(1R,2R,4S)- 21)	(1S,2R,4R)- 22)	(1R,2S,4S)- 22)
Unit cell dimensions						
a, Å	10.7797(7)	6.0802(8)	6.3744(2)	6.3758(4)	6.1443(5)	6.1463(3)
b, Å	21.3371(13)	7.0558(8)	6.7541(2)	6.7491(5)	7.4411(7)	7.4304(3)
c, Å	8.7302(5)	22.379(3)	20.5822(6)	20.5738(12)	8.5384(7)	8.5433(4)
α , °	90	90	90	90	90	90
β , °	102.503(3)	93.955(4)	90	90	93.856(4)	93.884(3)
γ , °	90	90	90	90	90	90
V, Å ³	1960.4(2)	957.8(2)	886.13(5)	885.31(10)	389.49(6)	389.27(3)

Parameter	10b	17b	(1S,2S,4R)-21	(1R,2R,4S)-21	(1S,2R,4R)-22	(1R,2S,4S)-22
F(000)	800	400	384	384	172	172
Crystal system	Monoclinic	Monoclinic	Orthorhombic	Orthorhombic	Monoclinic	Monoclinic
Space group	Cc	P2 ₁ /c	P2 ₁ 2 ₁ 2 ₁	P2 ₁ 2 ₁ 2 ₁	P2 ₁	P2 ₁
Z	8	4	4	4	2	2
μ , mm ⁻¹	0.086	0.088	0.393	0.394	0.430	0.430
D _{calc} , g/cm ³	1.275	1.305	1.361	1.363	1.395	1.396
2 Θ _{max} , grad	60	60	60	60	60	60
Measured reflections	10554	5491	8559	8695	3927	5467
F>4 σ (F) reflections	1890	1235	1685	1636	1374	1466
Parameters	262	168	124	124	107	107
R ₁	0.0632	0.0575	0.0392	0.0538	0.0609	0.0339
wR ₂	0.1442	0.1246	0.0915	0.1469	0.1239	0.0741
S	1.027	1.053	1.016	1.010	1.009	1.032
CCDC number	1961442	1961443	1961444	1961445	1961446	1961447