Supporting Information

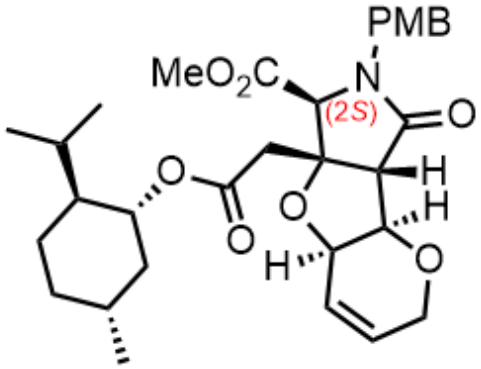
Menthyl esterification allows chiral resolution for synthesis of artificial glutamate analogs

Kenji Morokuma,[1] Shuntaro Tsukamoto,[1] Kei Miyako,[2]
Ryuichi Sakai,[2] Raku Irie,[1] Masato Oikawa[1,*]

[1] Yokohama City University, Seto 22-2, Kanazawa-ku,
Yokohama 236-0027, Japan
[2] Faculty of Fisheries Sciences, Hokkaido University,
Hakodate 041-8611, Japan

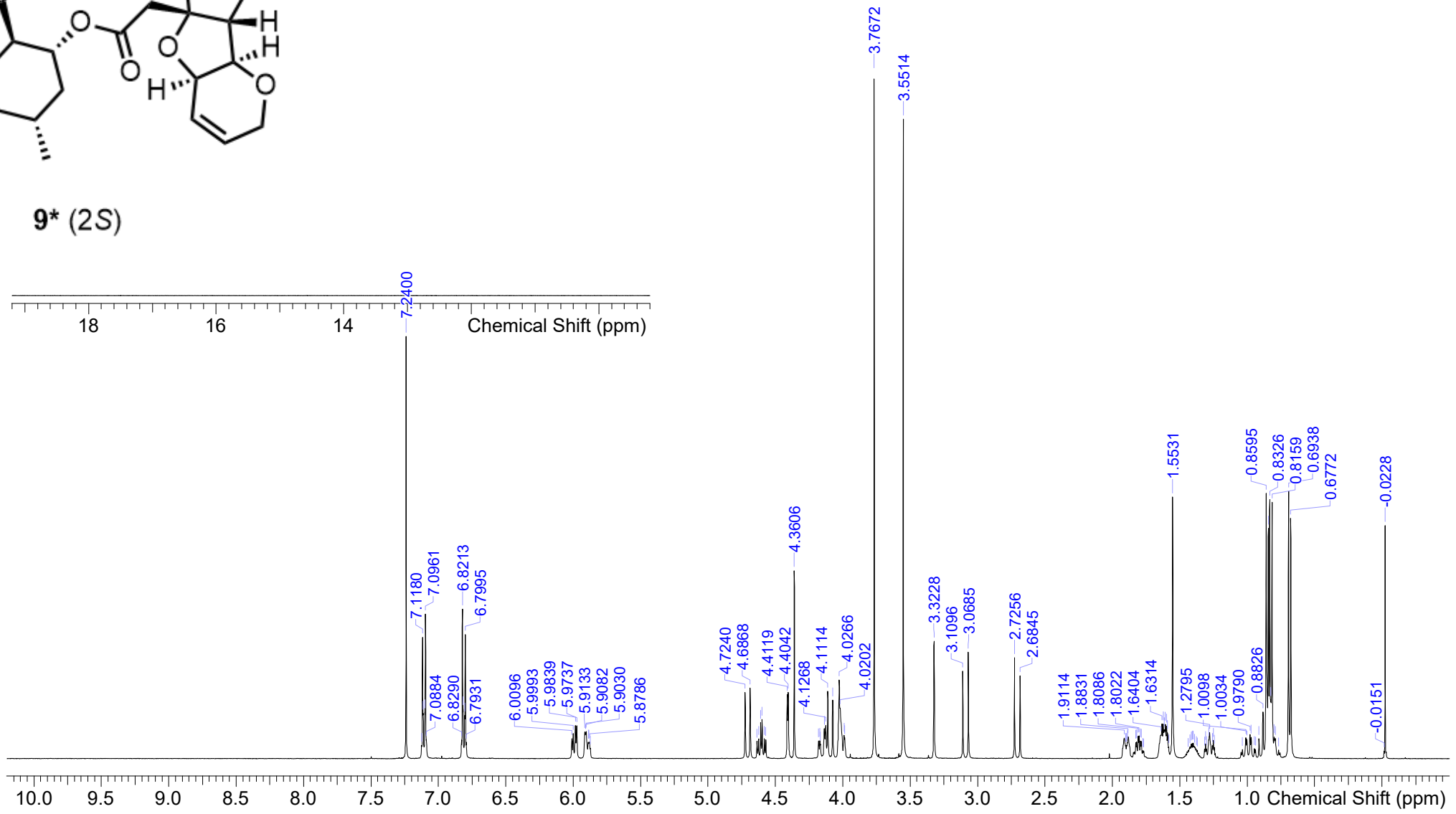
E-mail: moikawa@yokohama-cu.ac.jp

Contents:



9* (2*S*)

SII-2

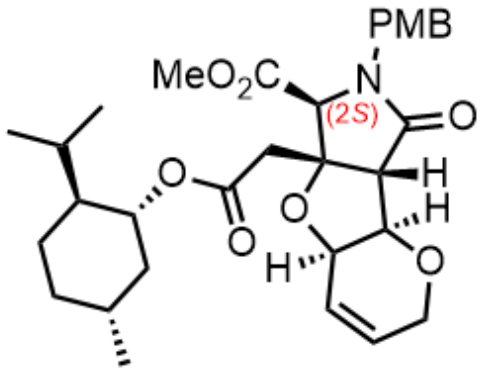


Date 29 Jan 2018 20:04:36

AA70061-002

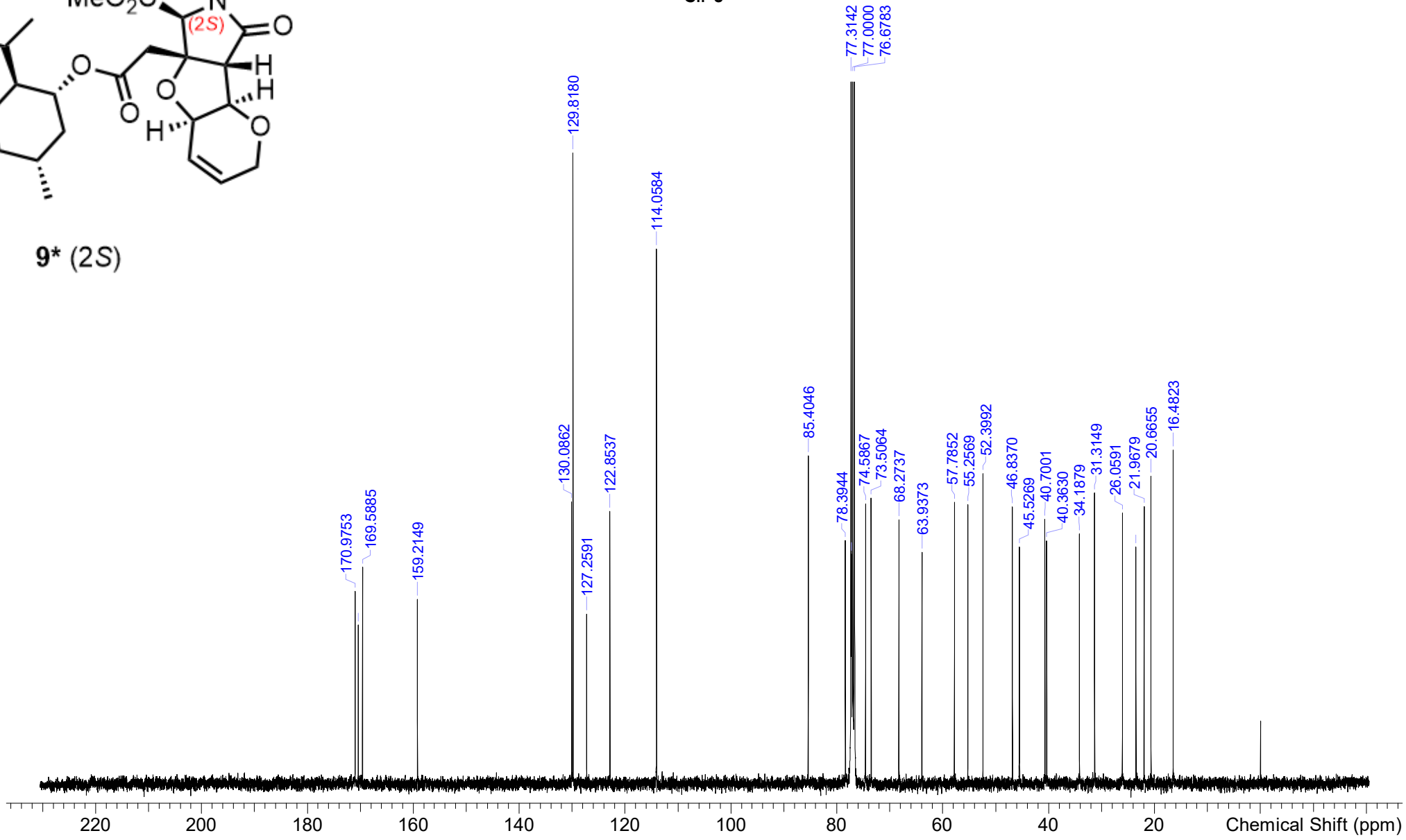
Nucleus	1H
Pulse Sequence	zg30_Bruker_AVIIIHD400N-2
Solvent	CHLOROFORM-d

Acquisition Time (sec)	1.9464
Frequency (MHz)	400.0300
Receiver Gain	202.37
Sweep Width (Hz)	8417.00



9* (2*S*)

SII-3

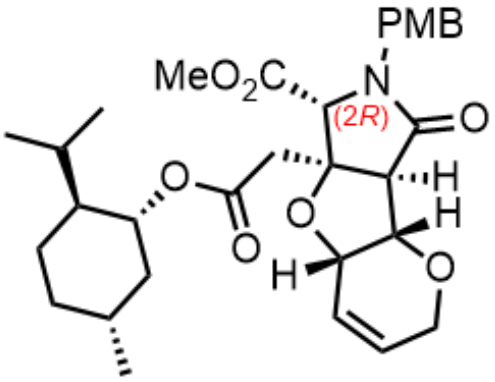


Date 30 Jan 2018 07:23:28

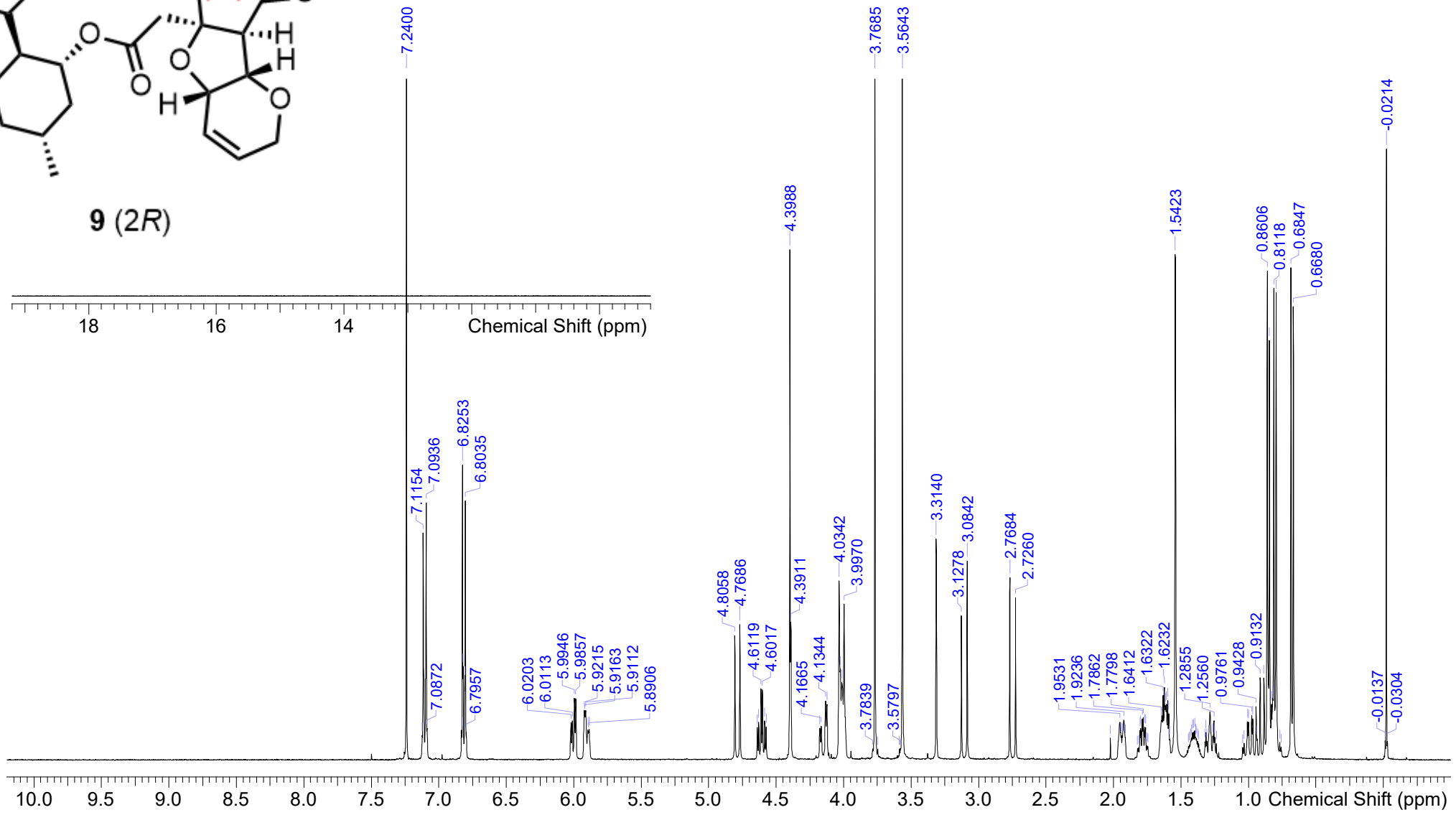
AA70061-002

Nucleus	13C
Pulse Sequence	zgpg30_Bruker_AVIIIHD400N-2
Solvent	CHLOROFORM-d

Acquisition Time (sec)	1.2976
Frequency (MHz)	100.5876
Receiver Gain	202.37
Sweep Width (Hz)	25251.75



9 (2*R*)

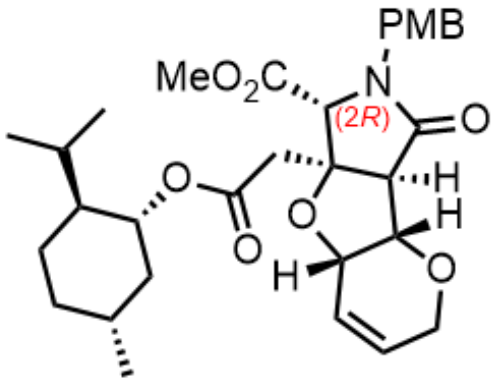


Date 23 Jan 2018 17:52:55

AA70051-003

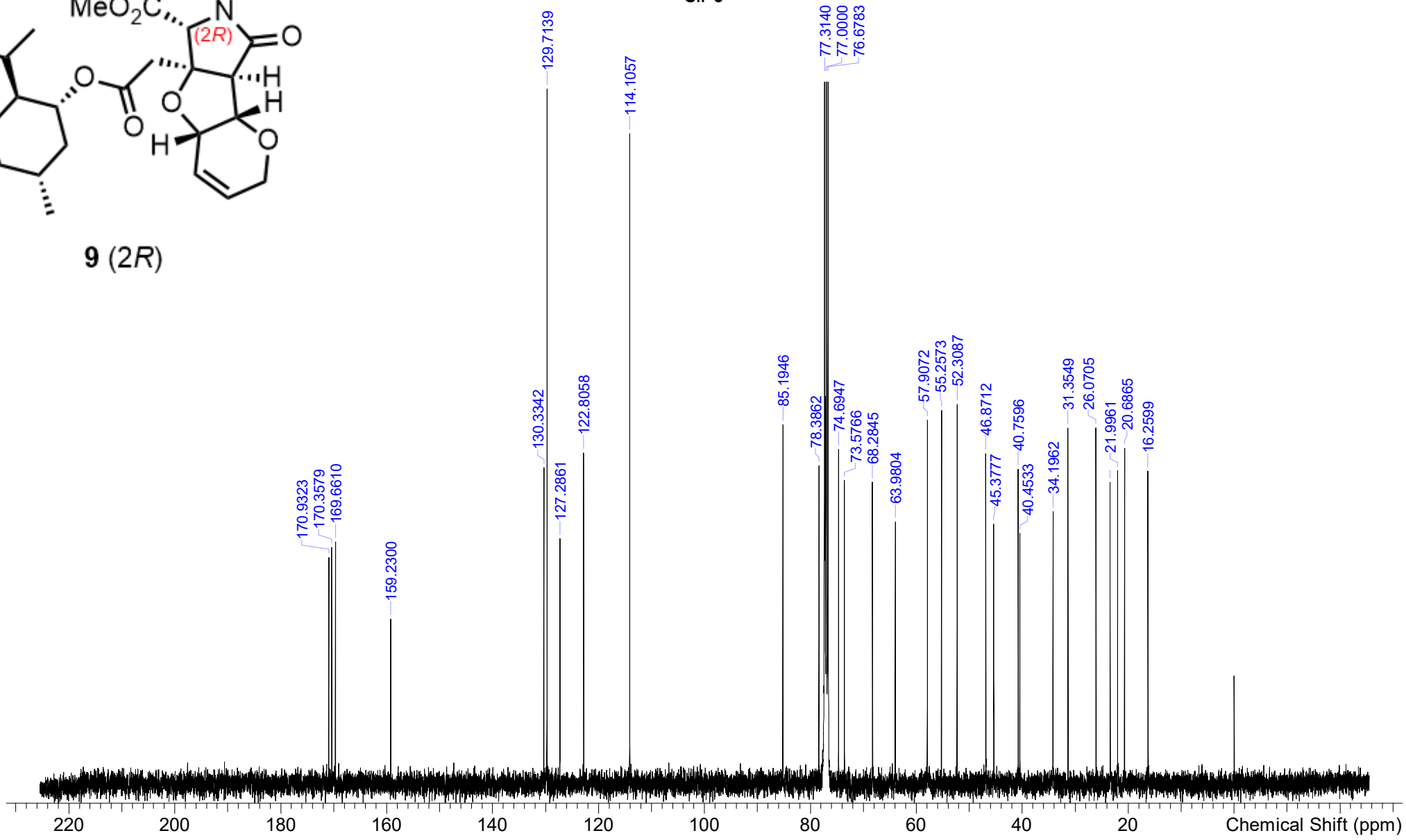
Nucleus	1H
Pulse Sequence	zg30_Bruker_AVANCEIII400N
Solvent	CHLOROFORM-d

Acquisition Time (sec)	1.9464
Frequency (MHz)	400.1800
Receiver Gain	128.00
Sweep Width (Hz)	8417.00



9 (2*R*)

SII-5

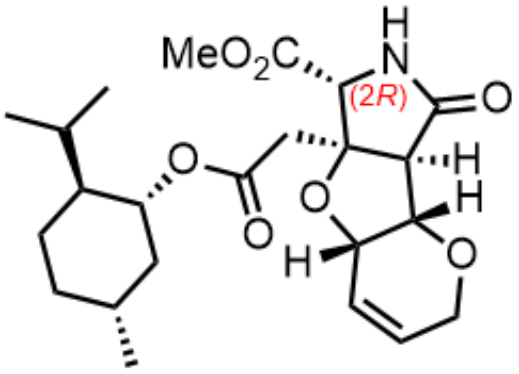


Date 24 Jan 2018 05:25:30

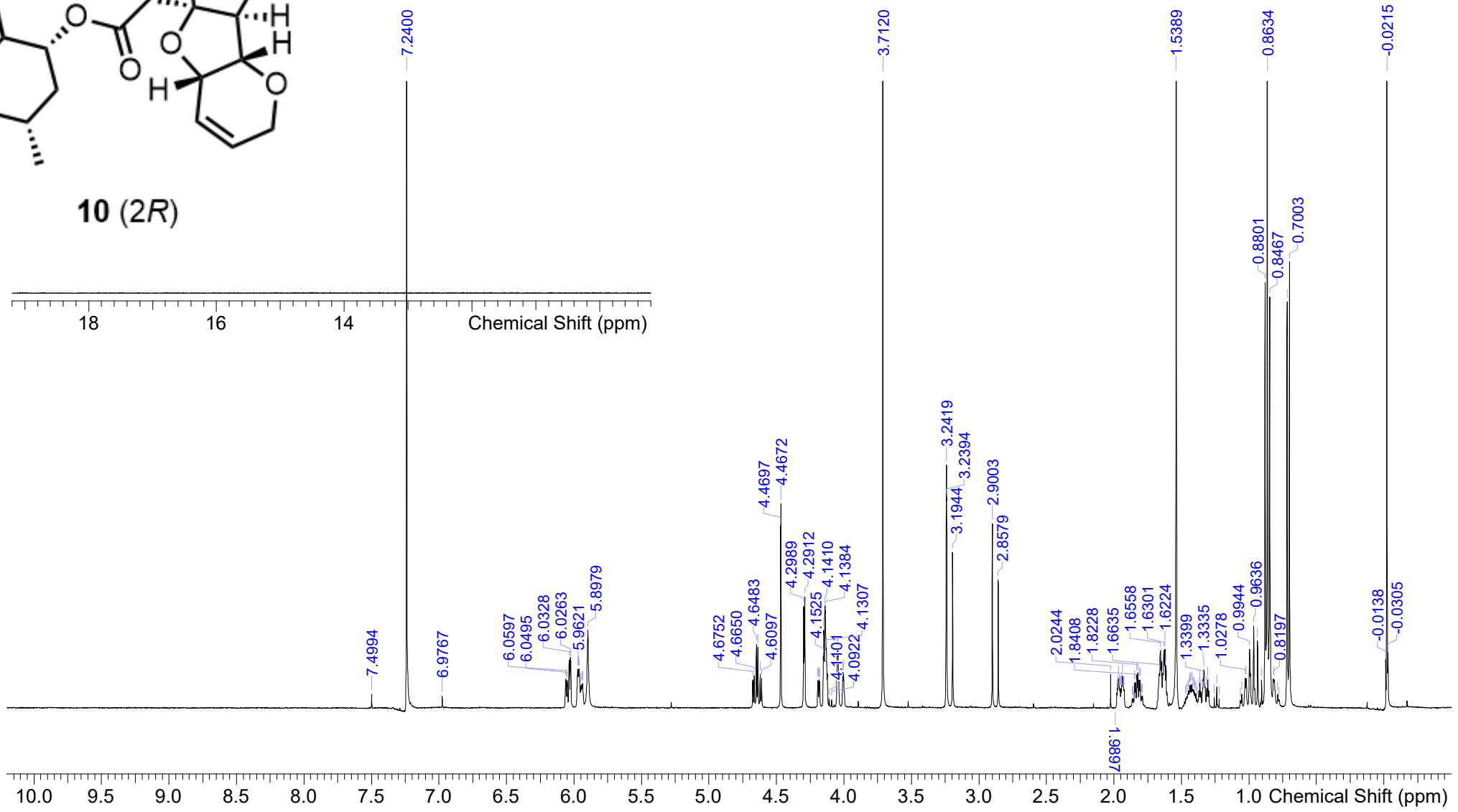
AA70051-003

Nucleus	13C
Pulse Sequence	zgpg30_Bruker_AVANCEIII400N
Solvent	CHLOROFORM-d

Acquisition Time (sec)	1.2976
Frequency (MHz)	100.6253
Receiver Gain	203.00
Sweep Width (Hz)	25251.75



10 (2*R*)

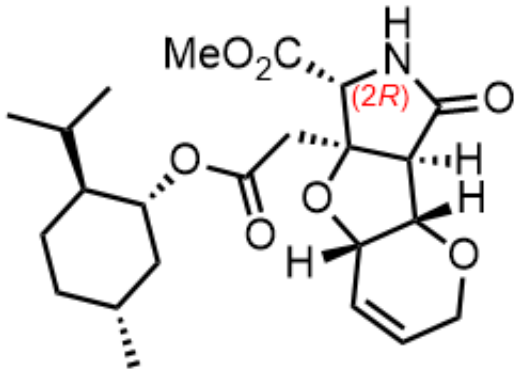


Date 11 Dec 2017 18:57:11

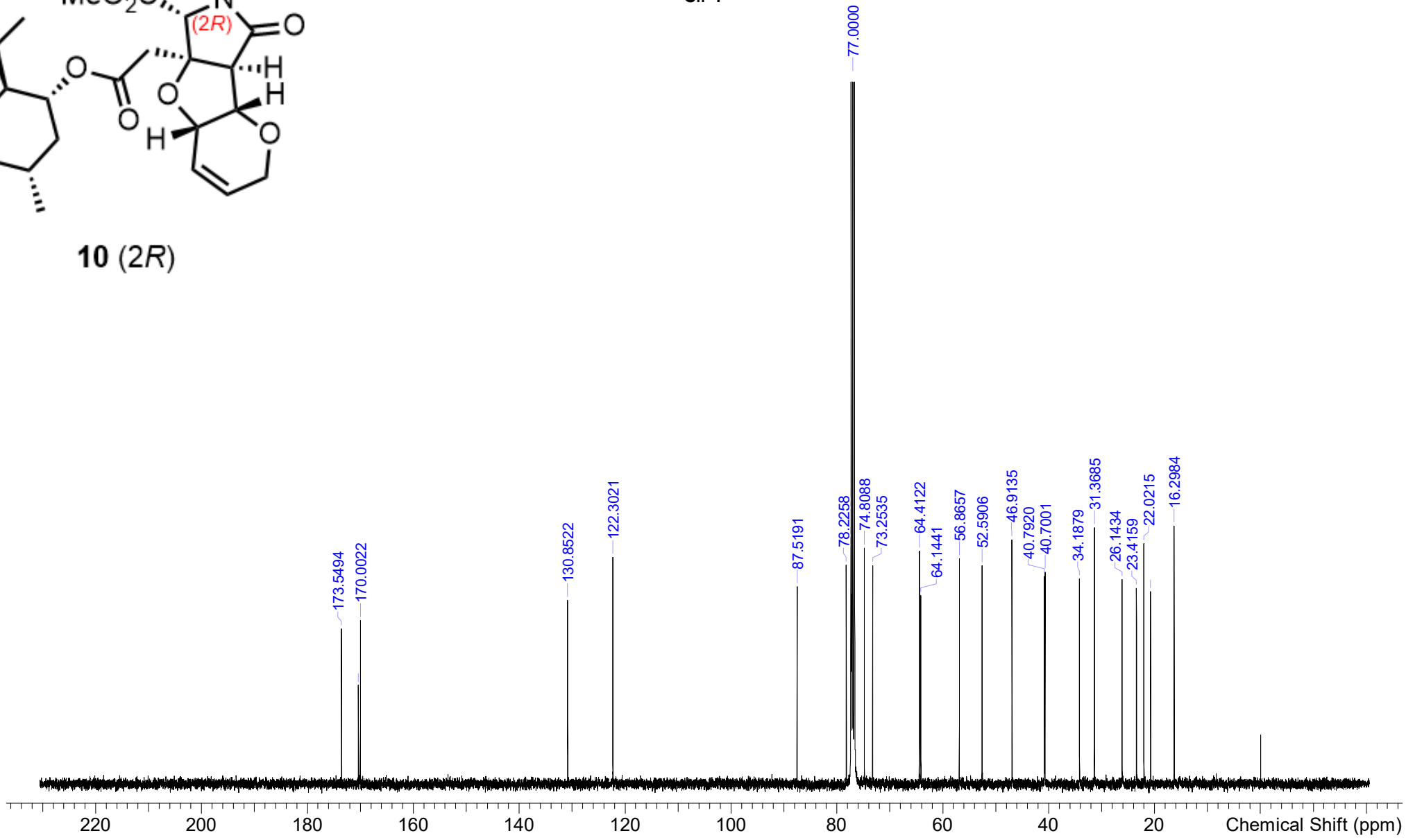
AA70065-002

Nucleus	1H
Pulse Sequence	zg30_Bruker_AVIIIHD400N-2
Solvent	CHLOROFORM-d

Acquisition Time (sec)	1.9464
Frequency (MHz)	400.0300
Receiver Gain	202.37
Sweep Width (Hz)	8417.00



10 (2*R*)

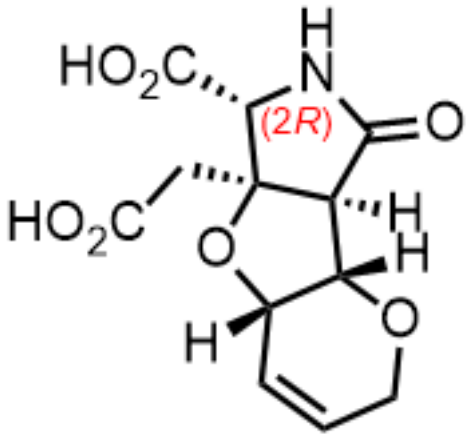


Date 24 Jan 2018 07:30:39

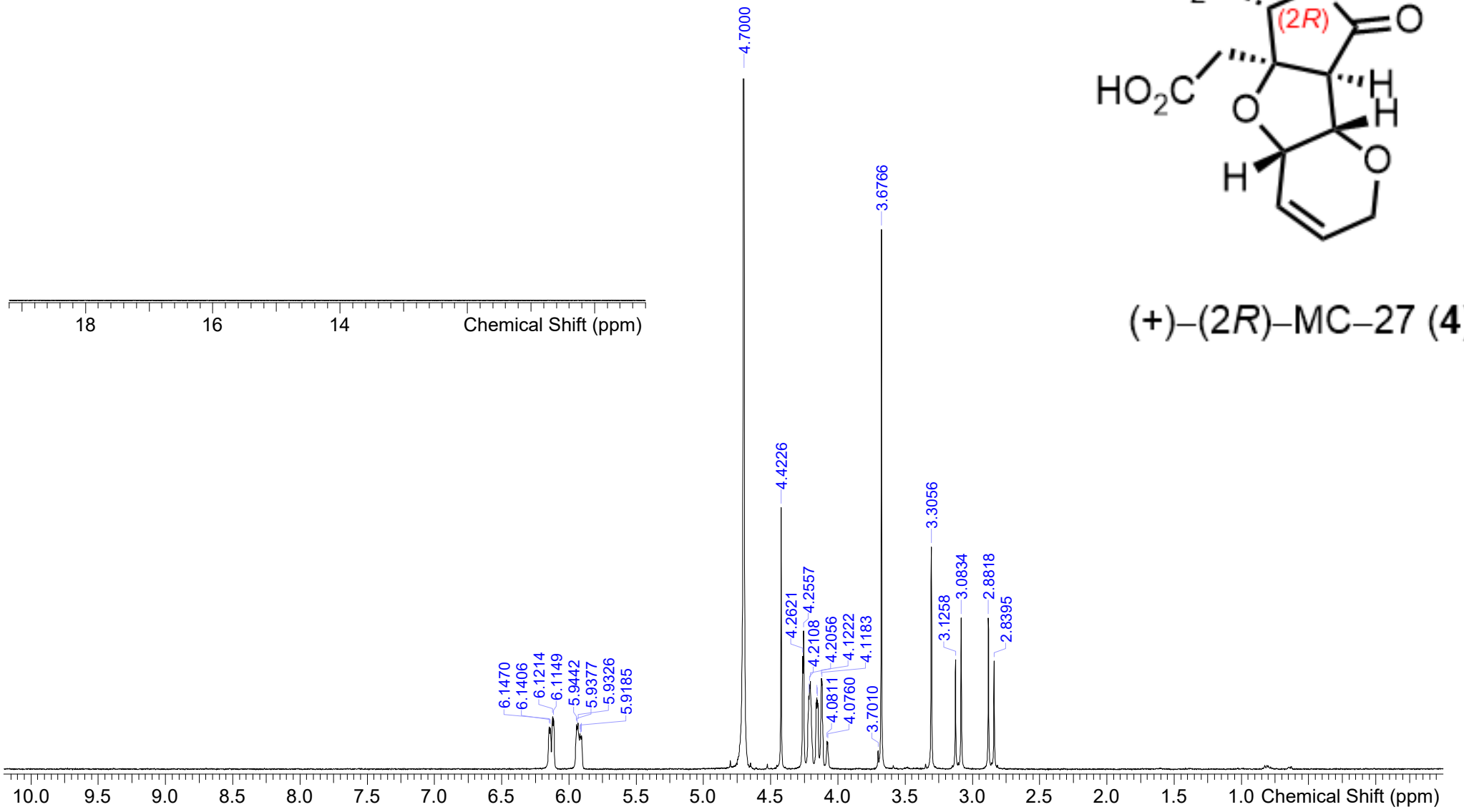
AA70052-001

Nucleus	13C
Pulse Sequence	zgpg30_Bruker_AVIIIHD400N-2
Solvent	CHLOROFORM-d

Acquisition Time (sec)	1.2976
Frequency (MHz)	100.5876
Receiver Gain	202.37
Sweep Width (Hz)	25251.75



(+)–(2*R*)–MC–27 (**4**)

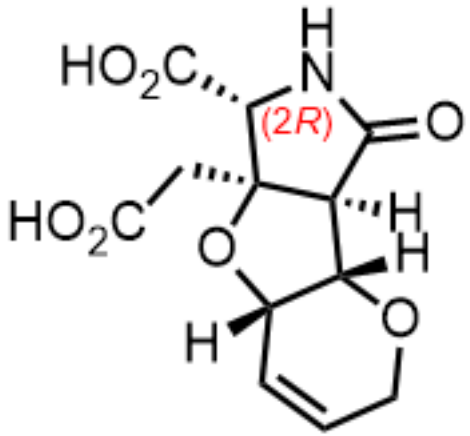


Date 23 Jan 2018 13:48:34

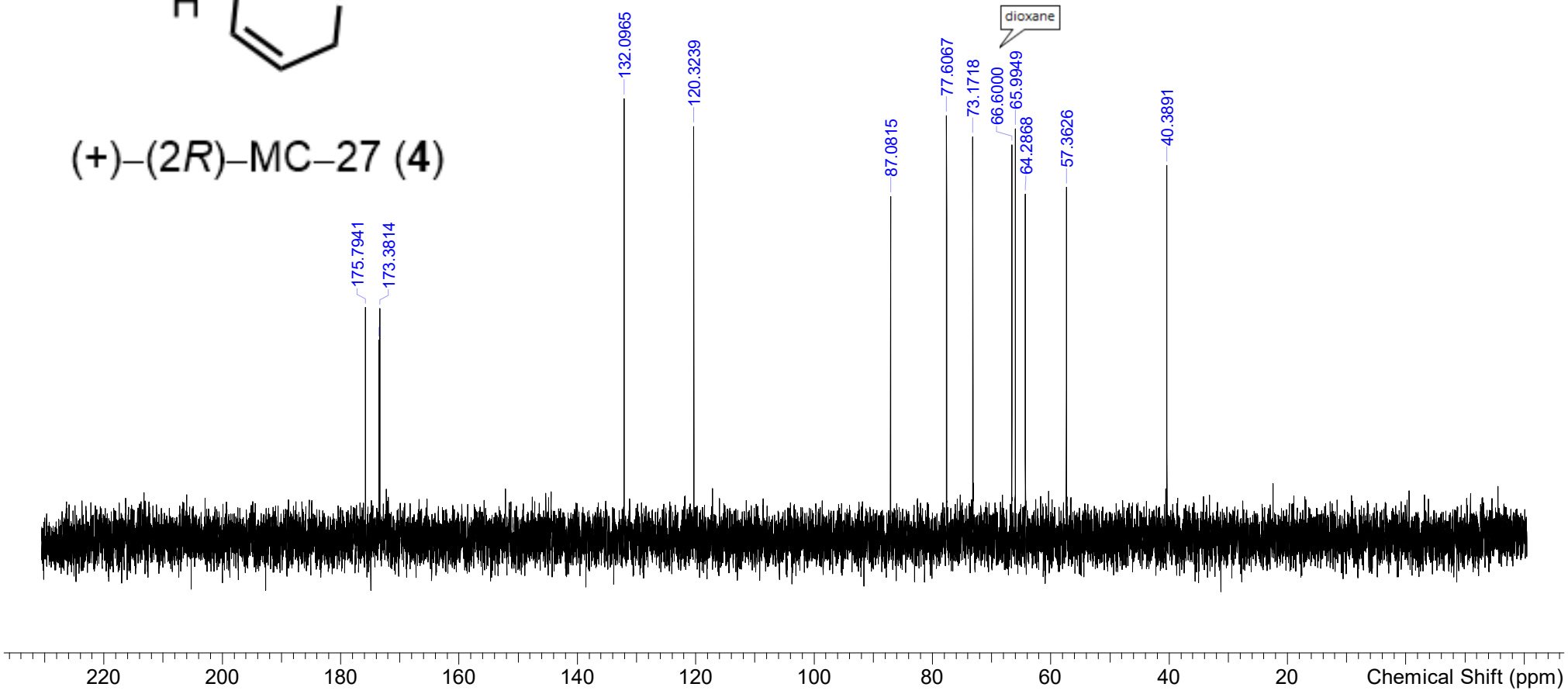
AA70069-001

Nucleus	1H
Pulse Sequence	zg30_Bruker_AVANCEII400M
Solvent	DEUTERIUM OXIDE

Acquisition Time (sec)	1.9464
Frequency (MHz)	400.1300
Receiver Gain	724.00
Sweep Width (Hz)	8417.00



(+)–(2*R*)–MC–27 (**4**)

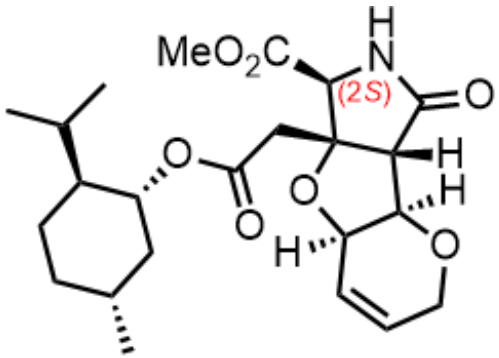


Date 24 Jan 2018 07:32:40

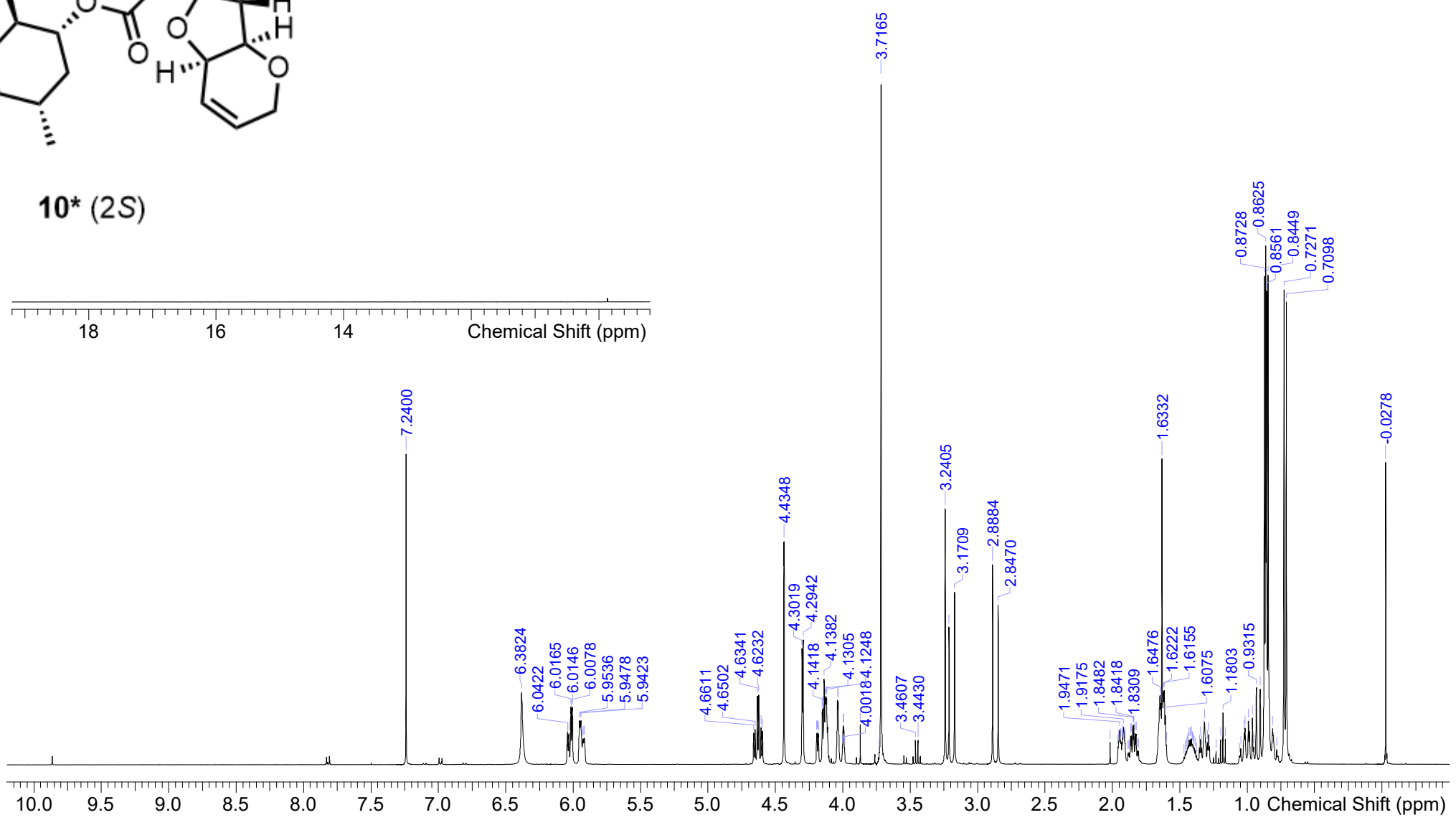
AA70069-001

Nucleus	13C
Pulse Sequence	zgpg30_Bruker_AVANCEII400M
Solvent	DEUTERIUM OXIDE

Acquisition Time (sec)	1.2976
Frequency (MHz)	100.6128
Receiver Gain	2050.00
Sweep Width (Hz)	25251.75



10* (2*S*)

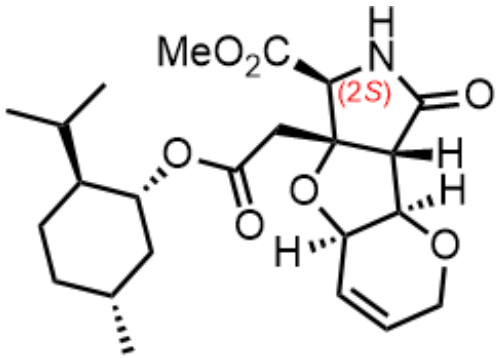


Date 20 Mar 2019 20:14:25

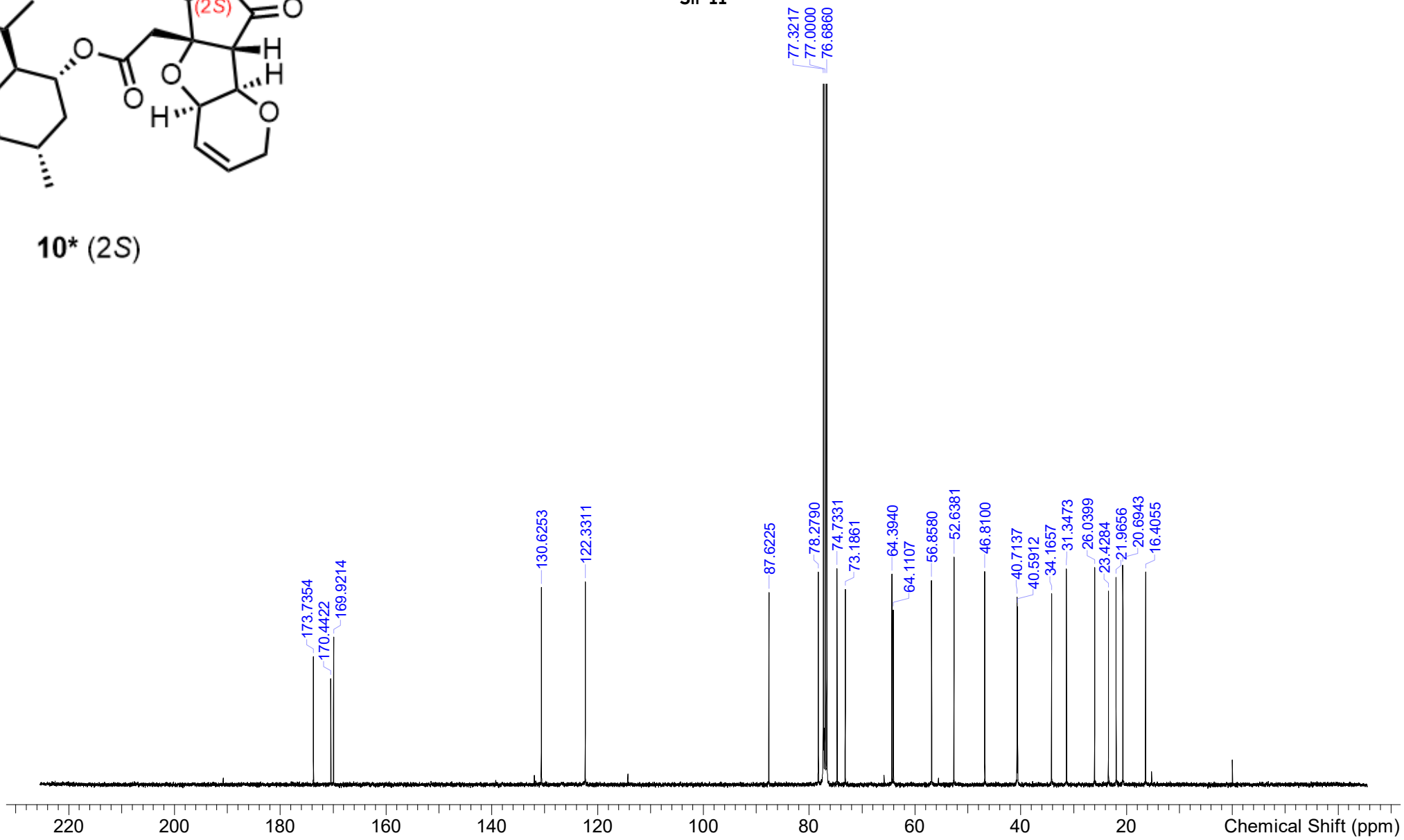
Kenji Morokuma
AA70071-001

Nucleus	1H
Pulse Sequence	zg30_Bruker_AVANCEIII400N
Solvent	CHLOROFORM-d

Acquisition Time (sec)	3.8928	**D1**	1
Frequency (MHz)	400.1800	**NS**	16
Receiver Gain	71.80	**SI**	65536
Sweep Width (Hz)	8417.38	**TD**	65536



10* (2*S*)

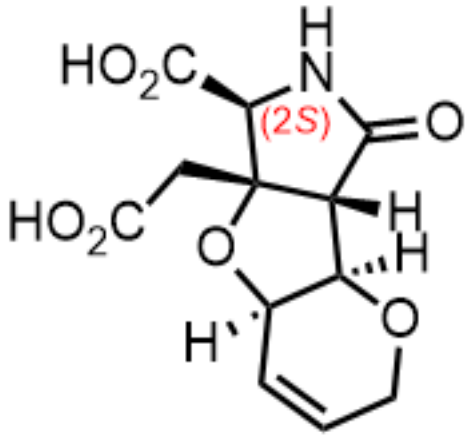


Date 21 Mar 2019 03:54:45

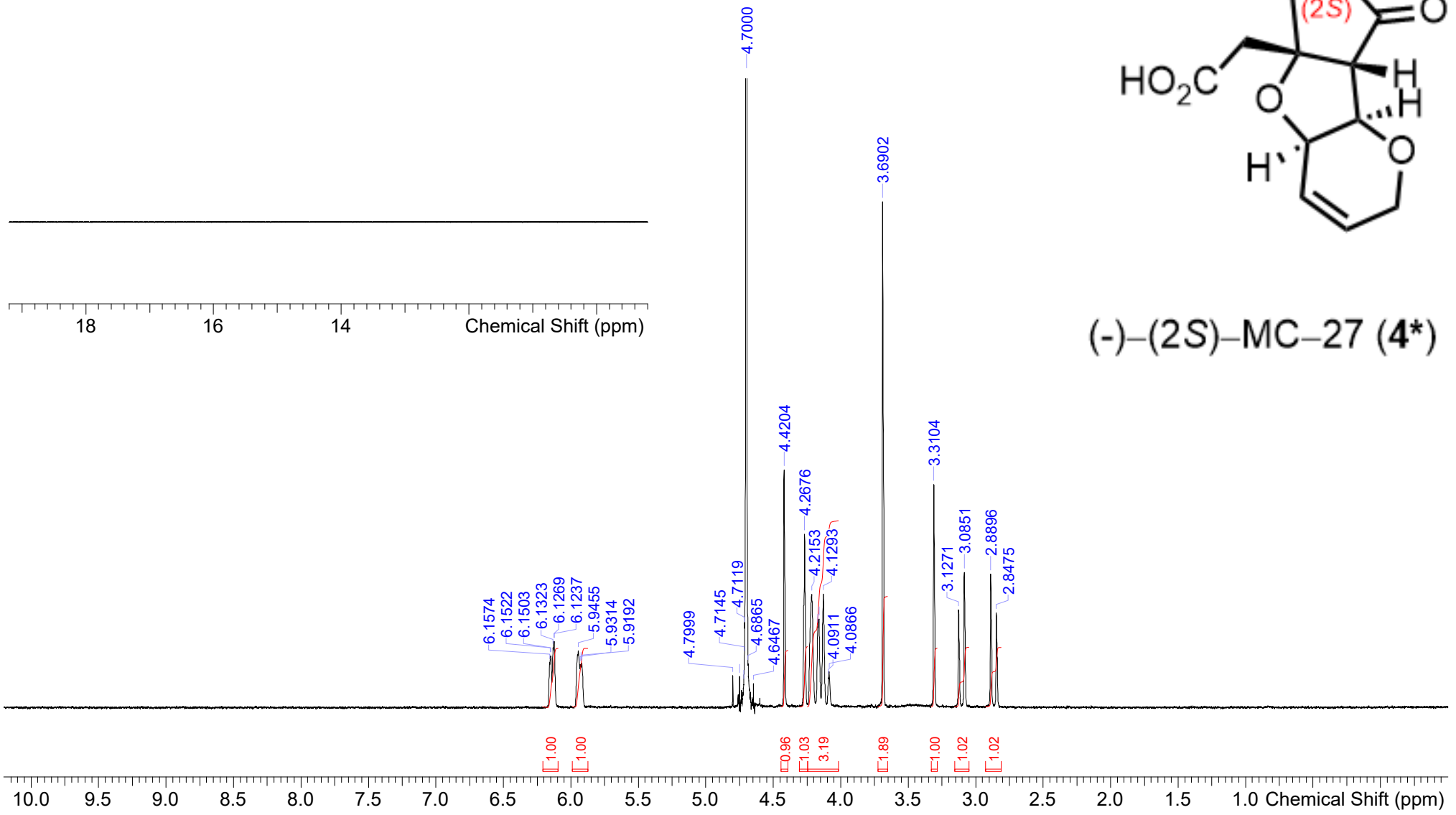
Kenji Morokuma
AA70071-001

Nucleus	13C
Pulse Sequence	zgpg30_Bruker_AVANCEIII400N
Solvent	CHLOROFORM-d

Acquisition Time (sec)	1.2976	**D1**	2
Frequency (MHz)	100.6253	**NS**	8192
Receiver Gain	203.00	**SI**	32768
Sweep Width (Hz)	25251.75	**TD**	65536



(-)–(2*S*)–MC–27 (**4***)

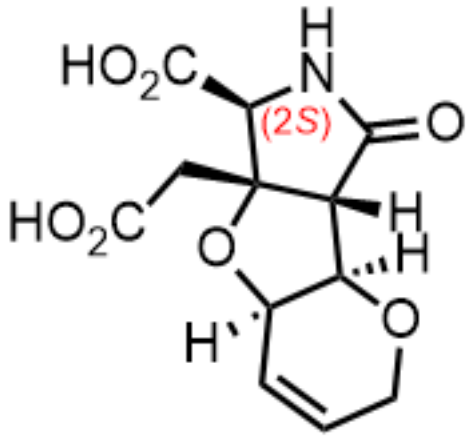


Date 17 Jan 2020 16:34:58

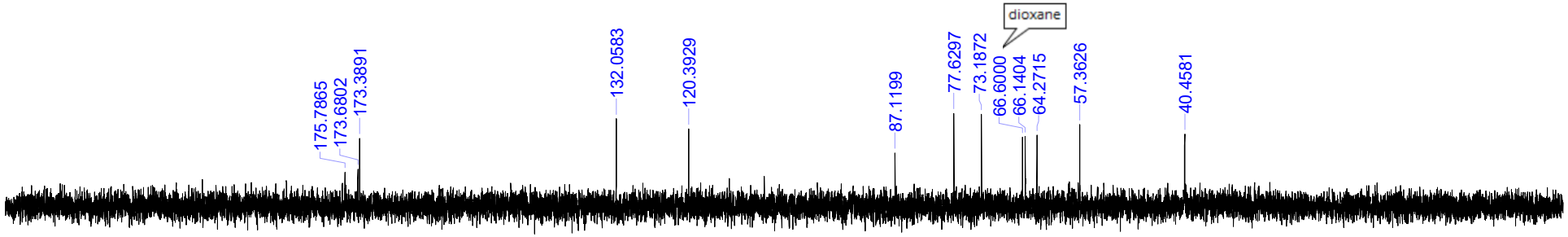
Kenji Morokuma
AA70073-001

Nucleus	1H
Pulse Sequence	zg30_Bruker_AVANCEII400M
Solvent	DEUTERIUM OXIDE

Acquisition Time (sec)	3.8928	**D1**	1
Frequency (MHz)	400.1300	**NS**	64
Receiver Gain	724.00	**SI**	65536
Sweep Width (Hz)	8417.38	**TD**	65536



(-)–(2*S*)–MC–27 (**4***)



Date 18 Jan 2020 01:55:11

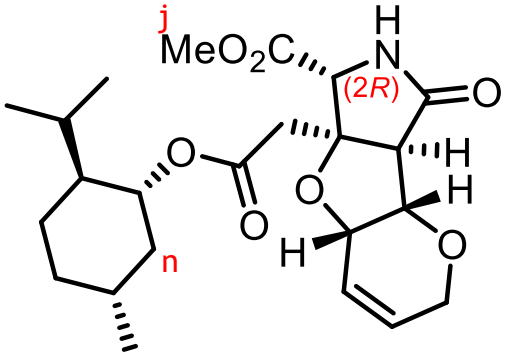
Kenji Morokuma
AA70073-001

Nucleus	13C
Pulse Sequence	zgpg30_Bruker_AVANCEII400M
Solvent	DEUTERIUM OXIDE

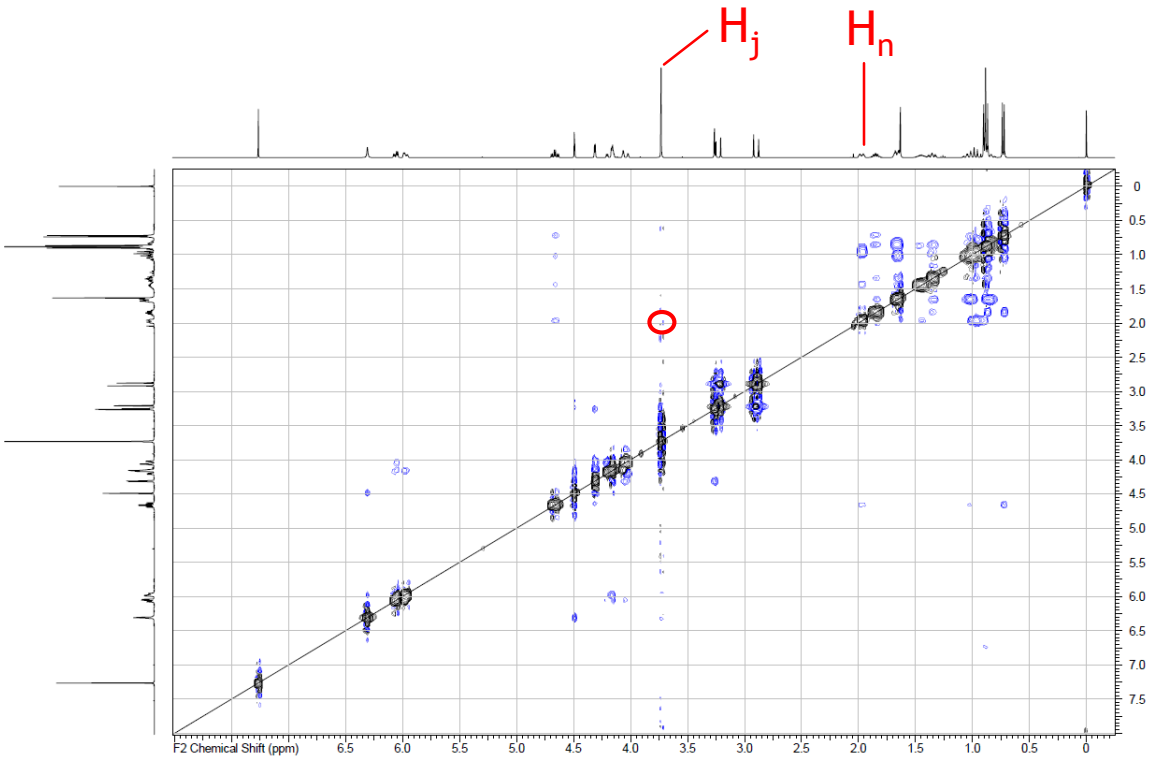
Acquisition Time (sec)	1.2976	**D1**	2
Frequency (MHz)	100.6128	**NS**	4096
Receiver Gain	512.00	**SI**	32768
Sweep Width (Hz)	25251.75	**TD**	65536

NOESY spectrum of menthyl ester diastereomer 10

fig. NOESY (400 MHz, CDCl3)

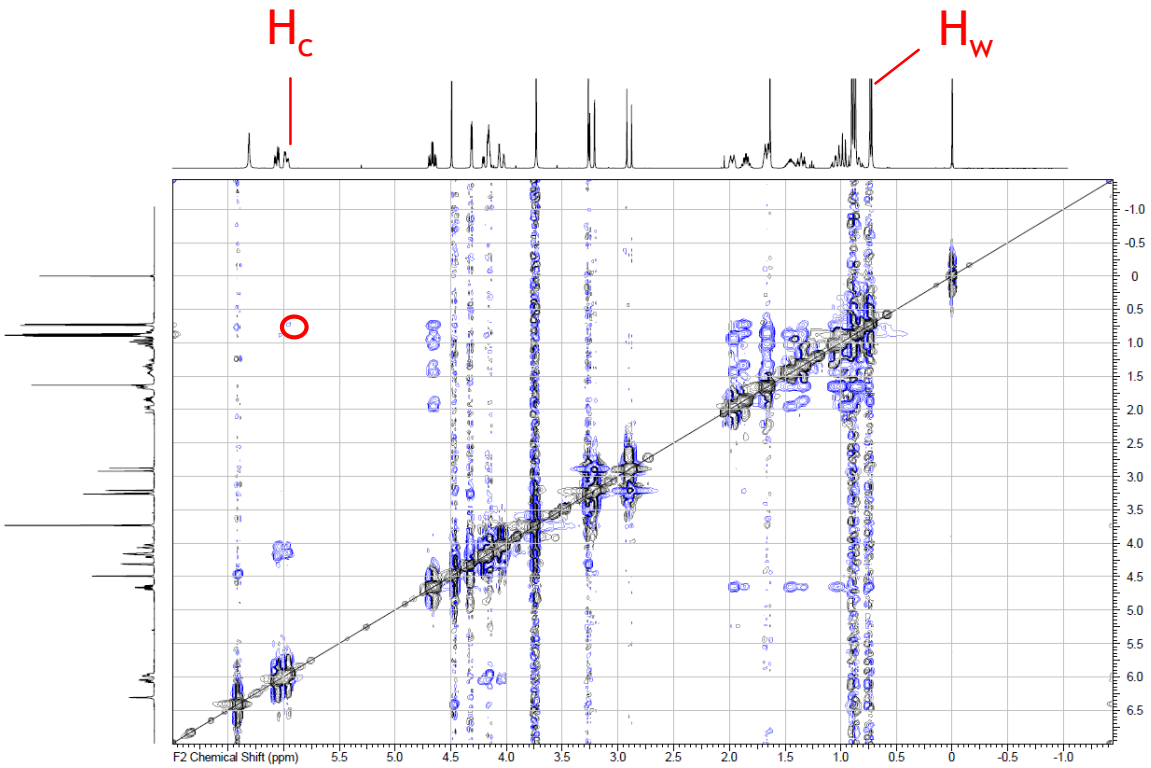


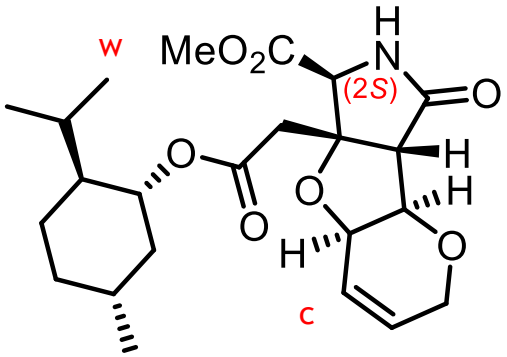
10 (2*R*)



NOESY spectrum of menthyl ester diastereomer 10*

fig. NOESY (400 MHz, CDCl3)

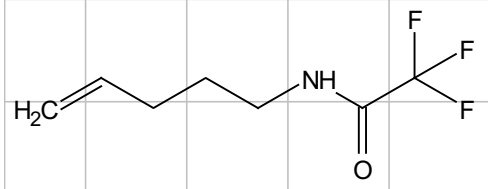




10* (2*S*)

ST-I-116-2.10.fid
1H ST-I-116-2
^{1}H NMR (400 MHz, $CDCl_3$)

SII-16

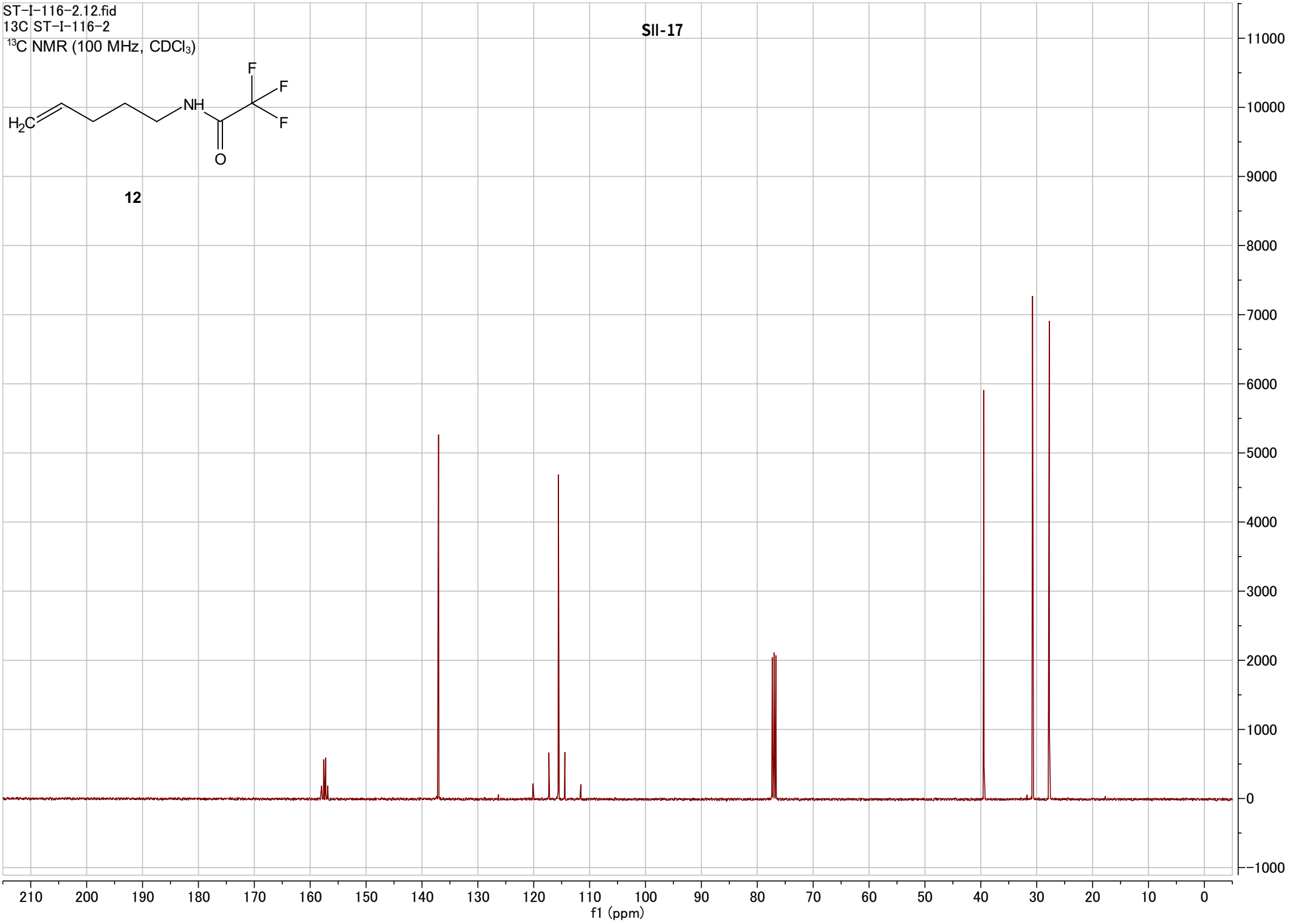


12

ST-I-116-2.12.fid
13C ST-I-116-2

^{13}C NMR (100 MHz, $CDCl_3$)

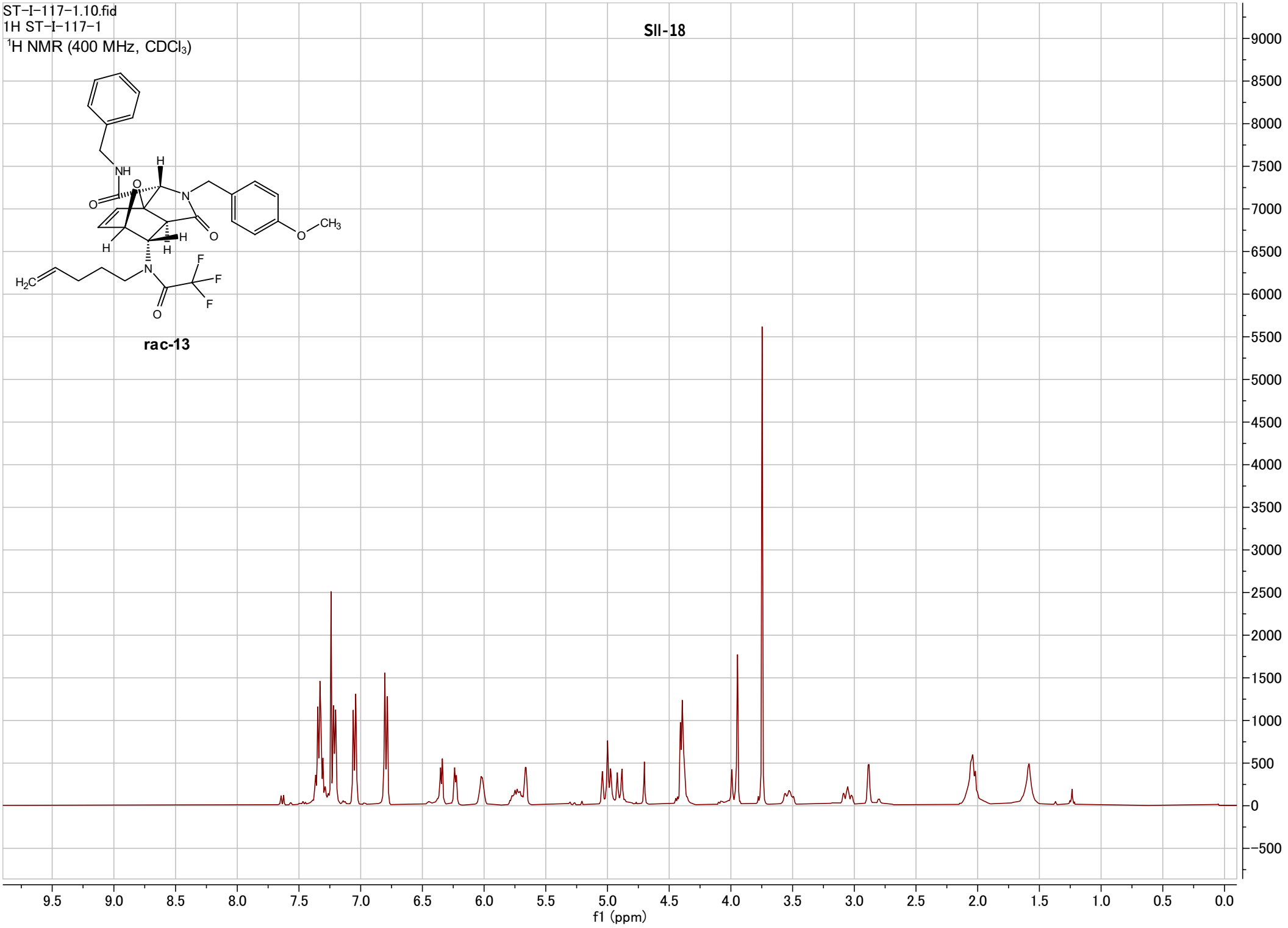
12



ST-I-117-1.10.fid
1H ST-I-117-1
^{1}H NMR (400 MHz, $CDCl_3$)

SII-18

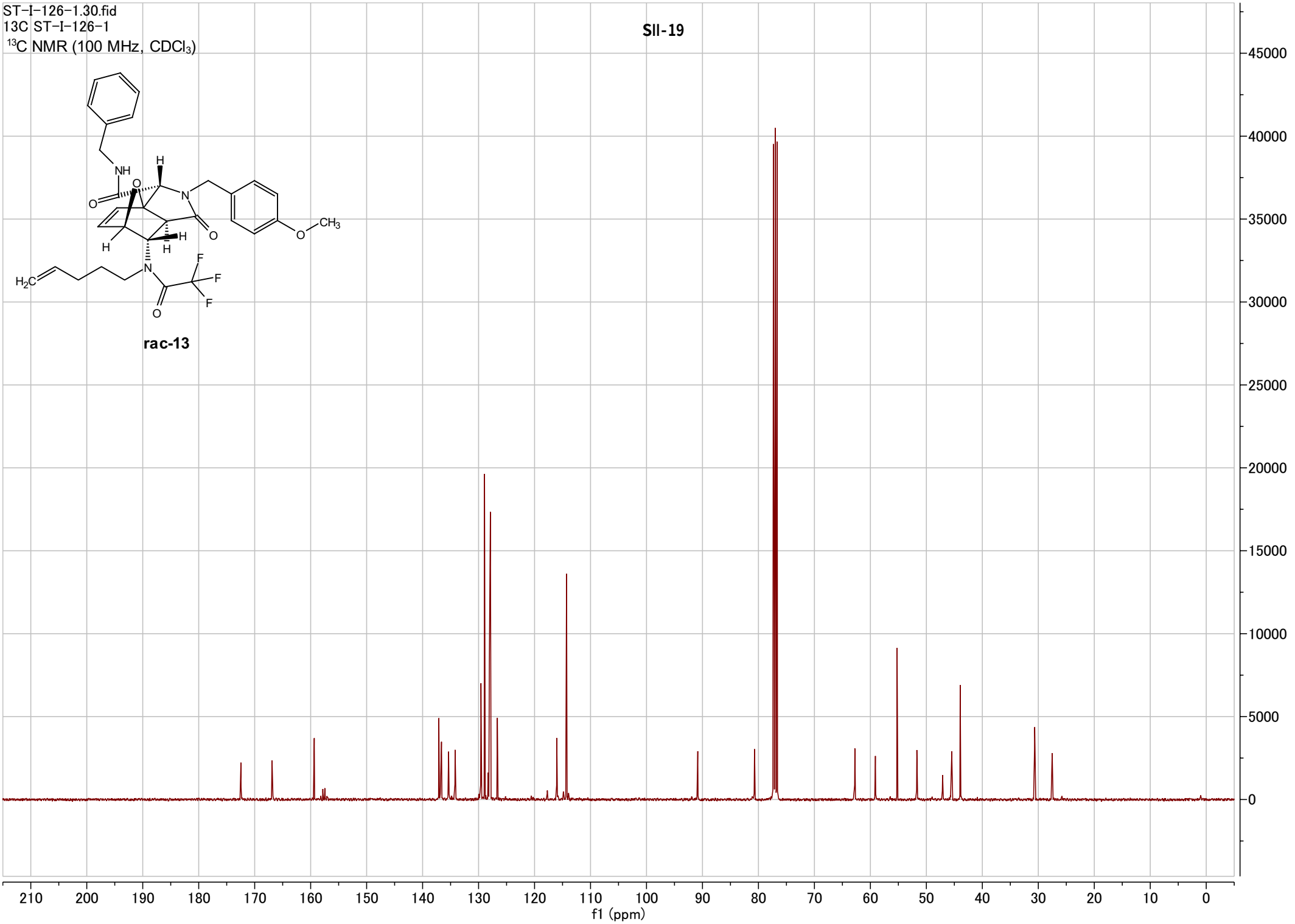
rac-13



ST-I-126-1.30.fid
13C ST-I-126-1
^{13}C NMR (100 MHz, $CDCl_3$)

SII-19

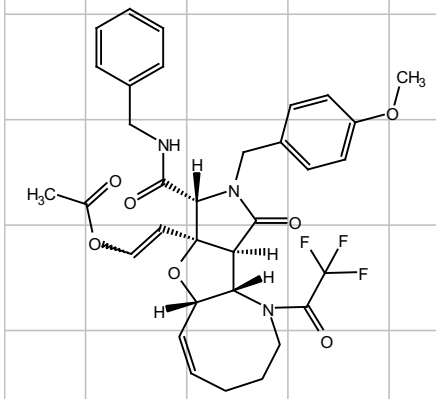
rac-13



ST-I-118-1.10.fid
1H ST-I-118-1

SII-20

^{1}H NMR (400 MHz, $CDCl_3$)

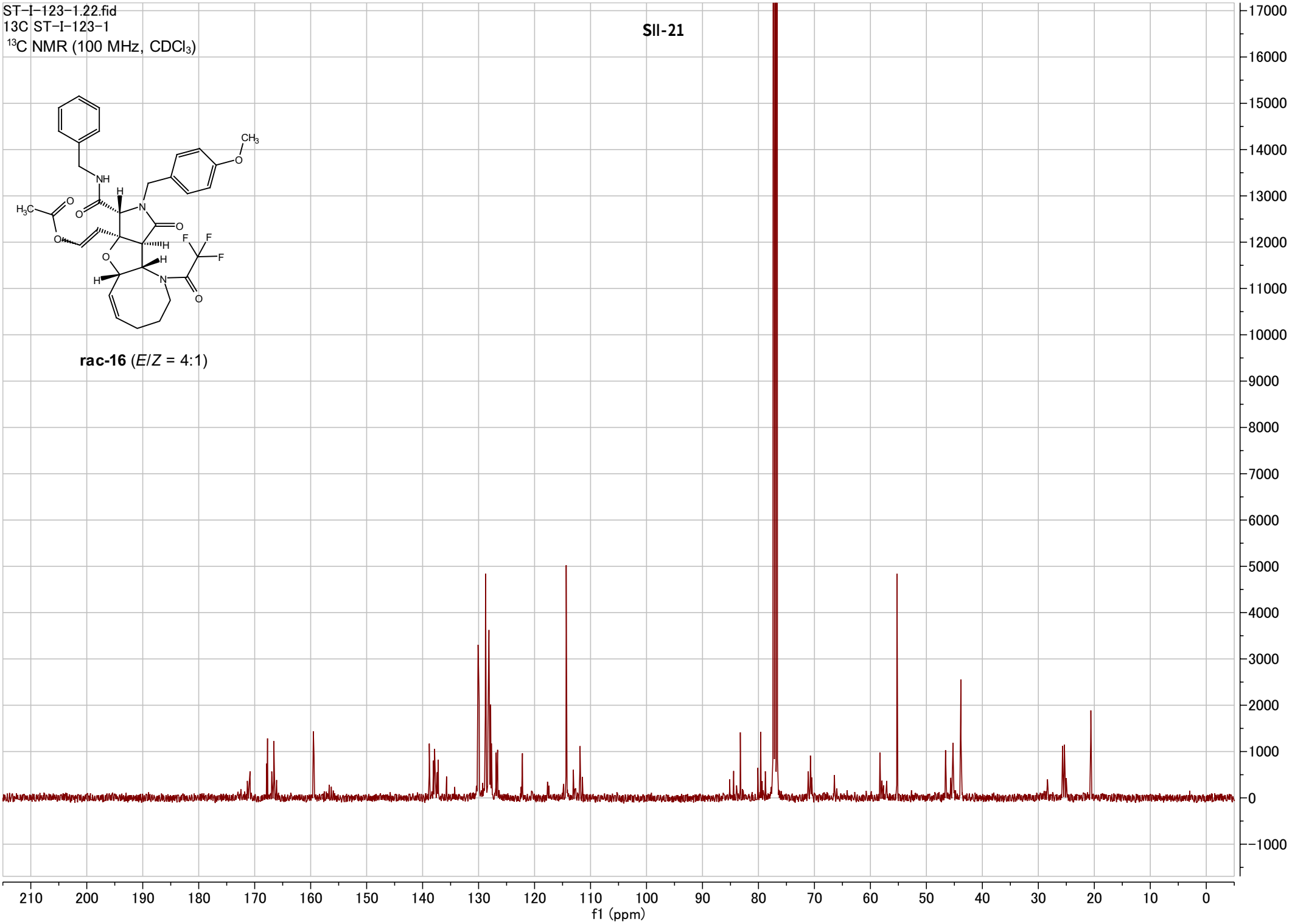


rac-16 (*E*/*Z* = 4:1)

ST-I-123-1.22.fid
13C ST-I-123-1
^{13}C NMR (100 MHz, $CDCl_3$)

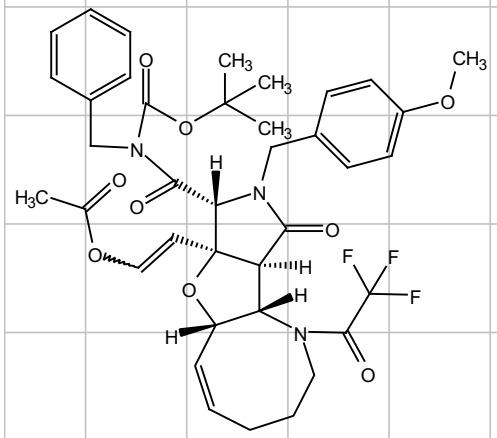
SII-21

rac-16 (*E*/*Z* = 4:1)



ST-I-124-1.10.fid
1H ST-I-124-1
^{1}H NMR (400 MHz, $CDCl_3$)

SII-22

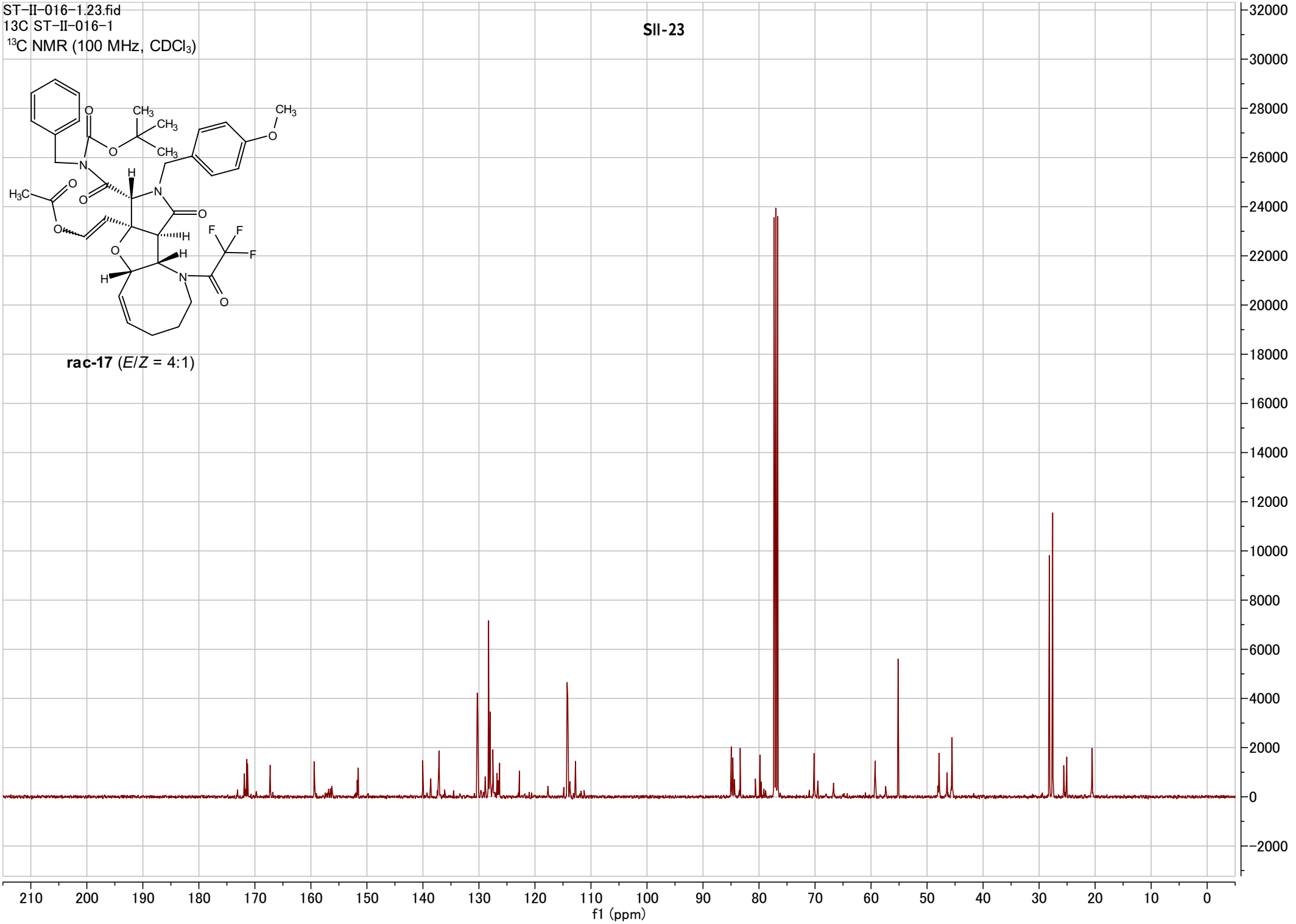


rac-17 (*E*/*Z* = 4:1)

ST-II-016-1.23.fid
13C ST-II-016-1
^{13}C NMR (100 MHz, $CDCl_3$)

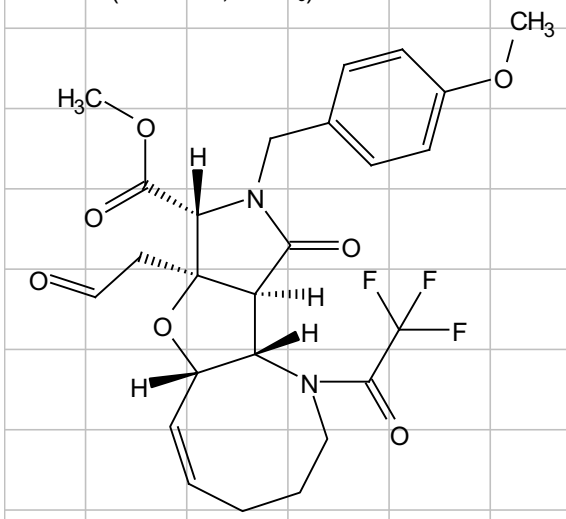
SII-23

rac-17 (*E*/*Z* = 4:1)



ST-II-017-3.10.fid
1H ST-II-017-3
^{1}H NMR (400 MHz, $CDCl_3$)

SII-24



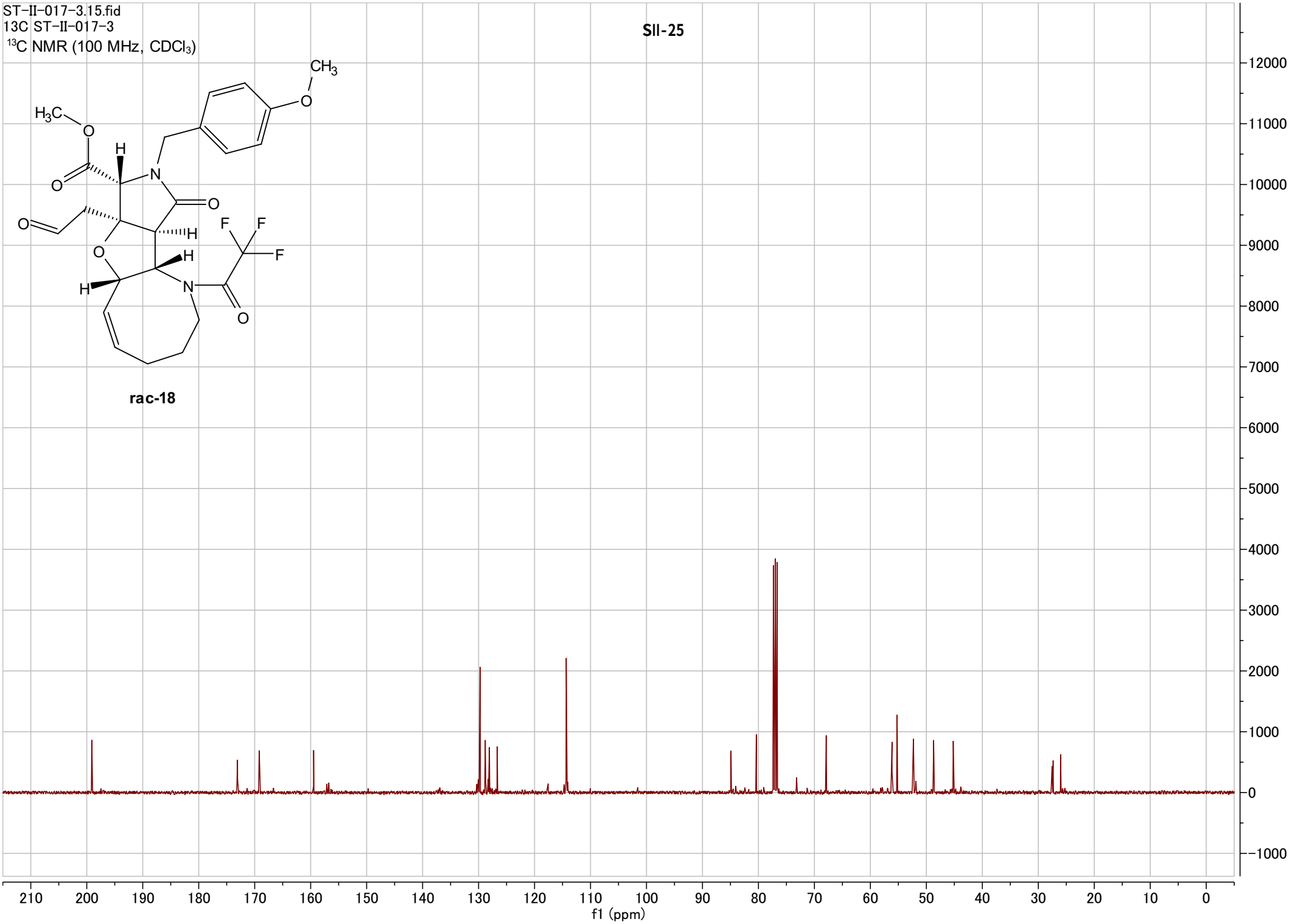
rac-18

f1 (ppm)

ST-II-017-3.15.fid
13C ST-II-017-3
^{13}C NMR (100 MHz, $CDCl_3$)

SII-25

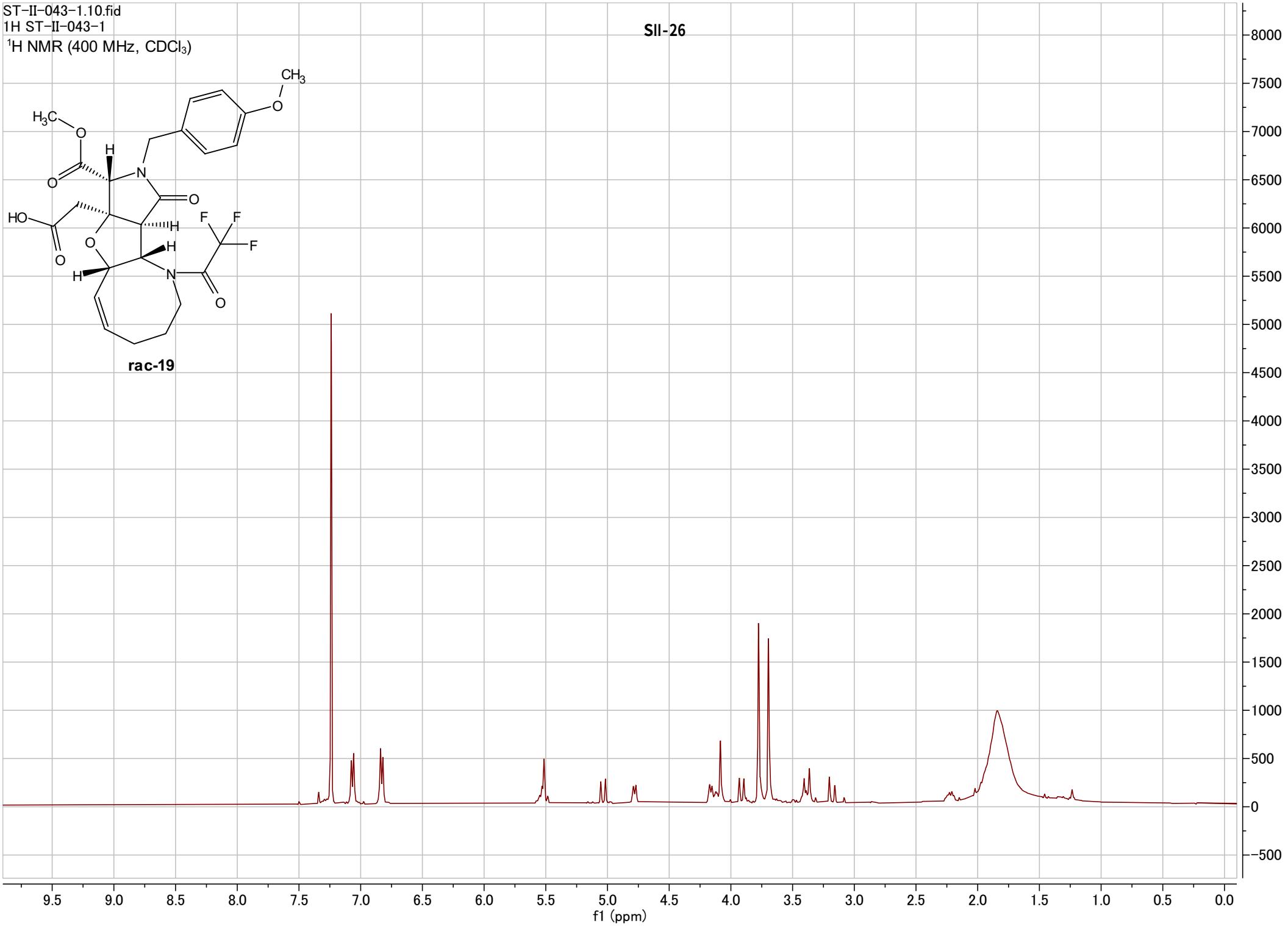
rac-18



ST-II-043-1.10.fid
1H ST-II-043-1
^{1}H NMR (400 MHz, $CDCl_3$)

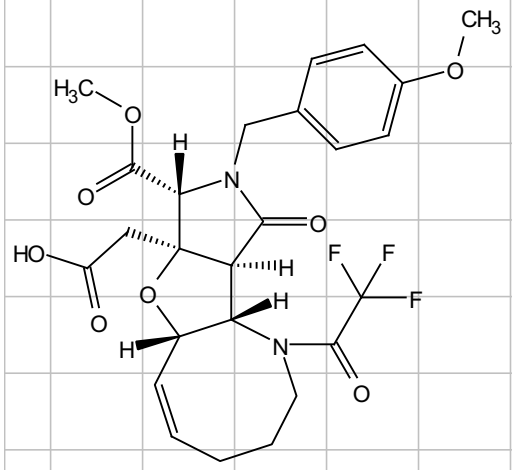
SII-26

rac-19



ST-II-019-2_MeOD.15.fid
13C ST-II-019-2_MeOD
^{13}C NMR (100 MHz, CD_3OD)

SII-27

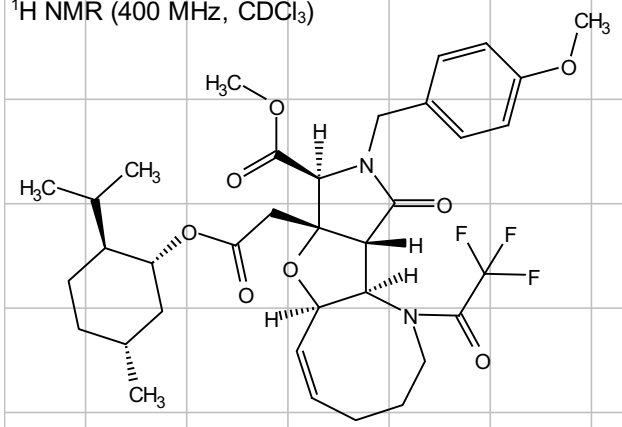


rac-19

ST-II-048-1.10.fid
1H ST-II-048-1

^{1}H NMR (400 MHz, $CDCl_3$)

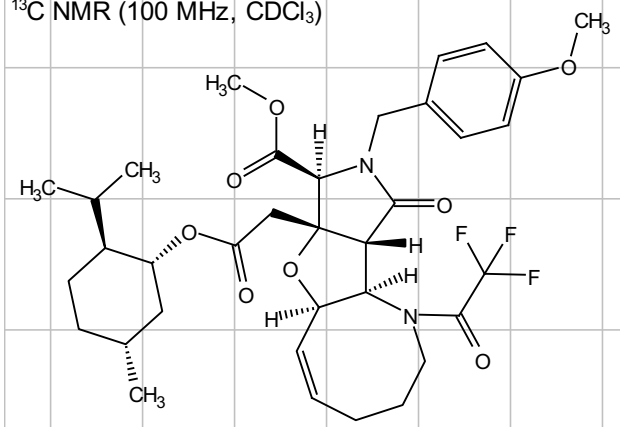
SII-28



20* (2S)

ST-II-048-1.11.fid
13C ST-II-048-1
^{13}C NMR (100 MHz, $CDCl_3$)

SII-29

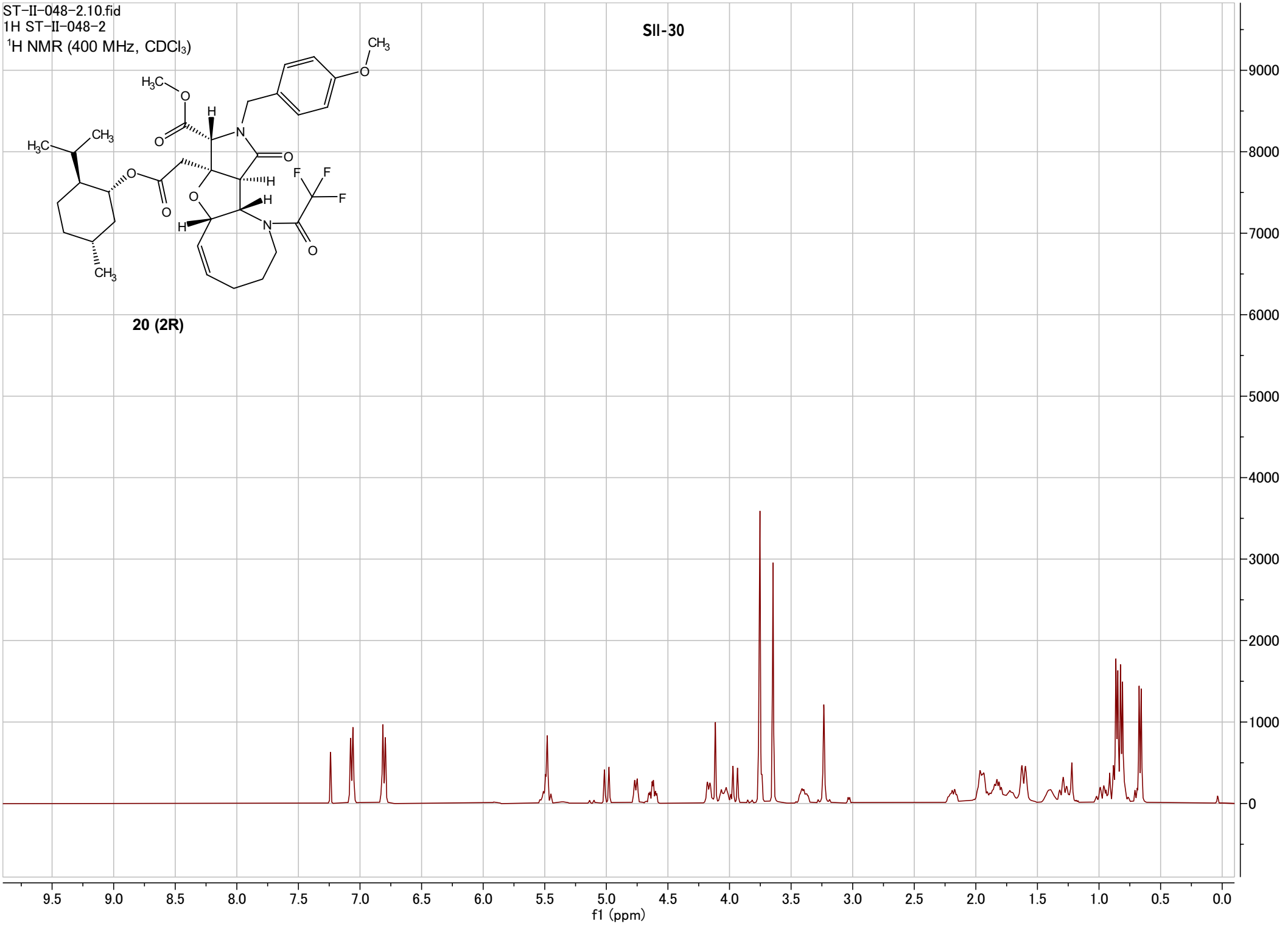


20* (2S)

ST-II-048-2.10.fid
1H ST-II-048-2
^{1}H NMR (400 MHz, $CDCl_3$)

SII-30

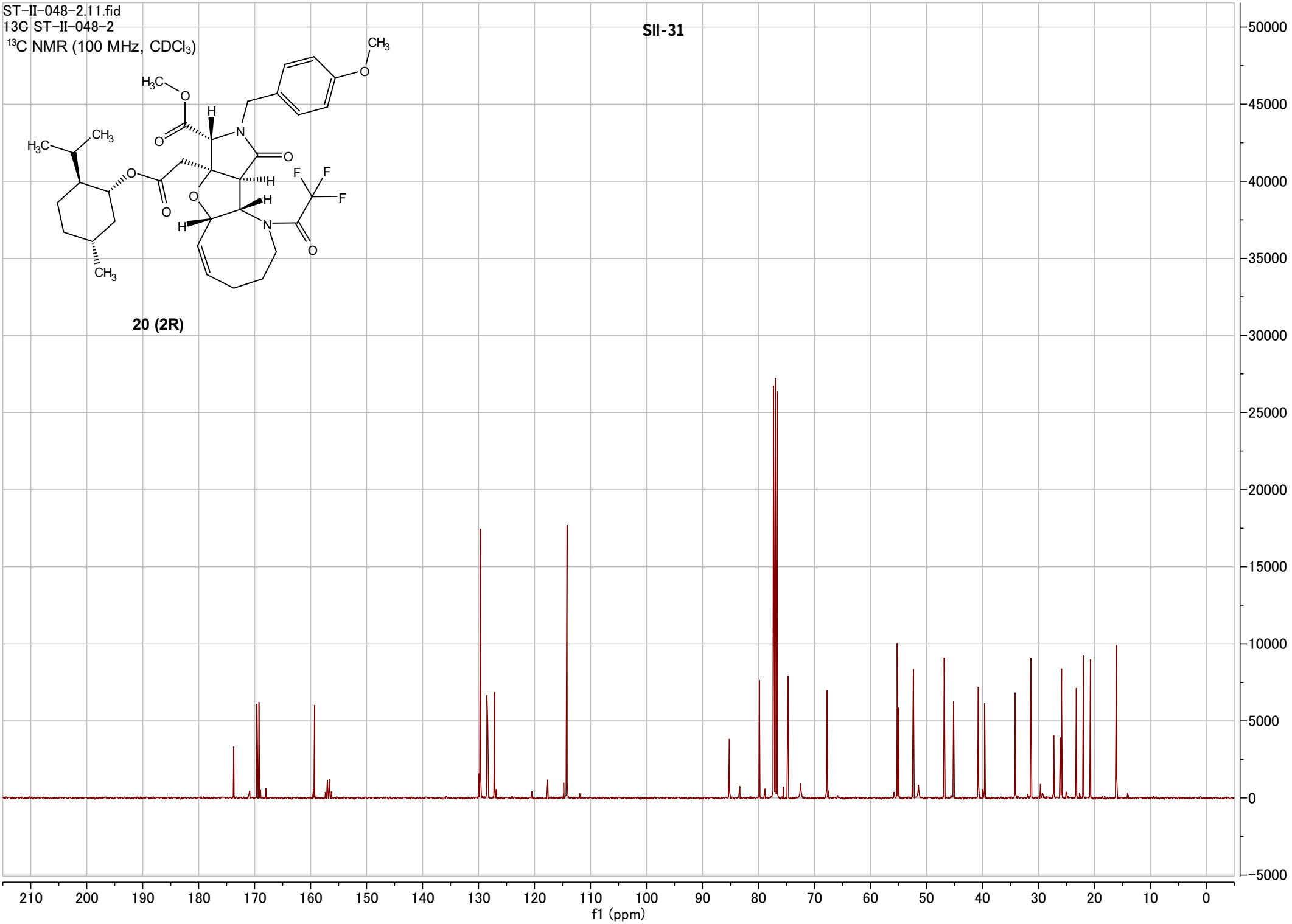
20 (2R)



ST-II-048-2.11.fid
13C ST-II-048-2
^{13}C NMR (100 MHz, $CDCl_3$)

SII-31

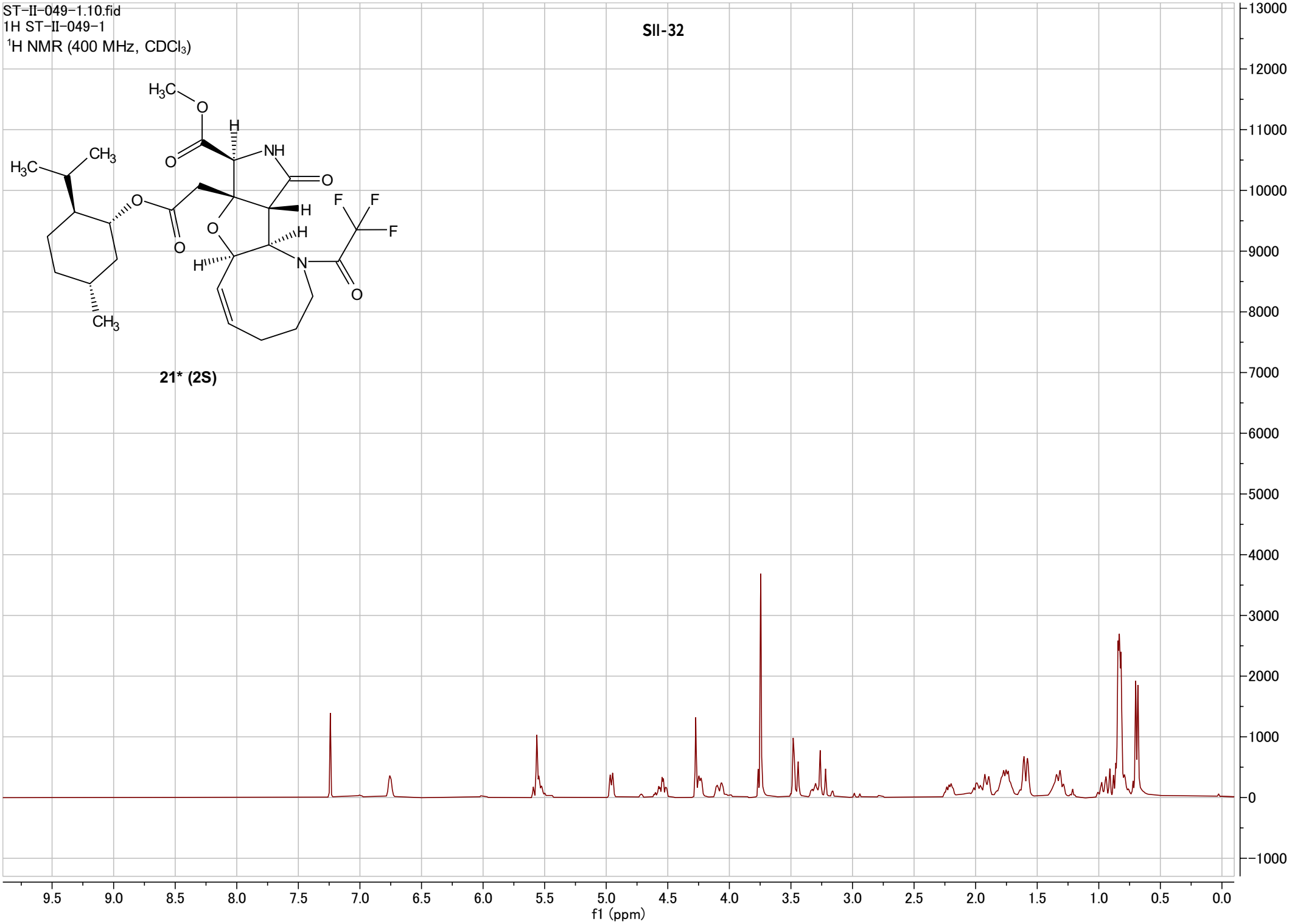
20 (2R)



ST-II-049-1.10.fid
1H ST-II-049-1
^{1}H NMR (400 MHz, $CDCl_3$)

SII-32

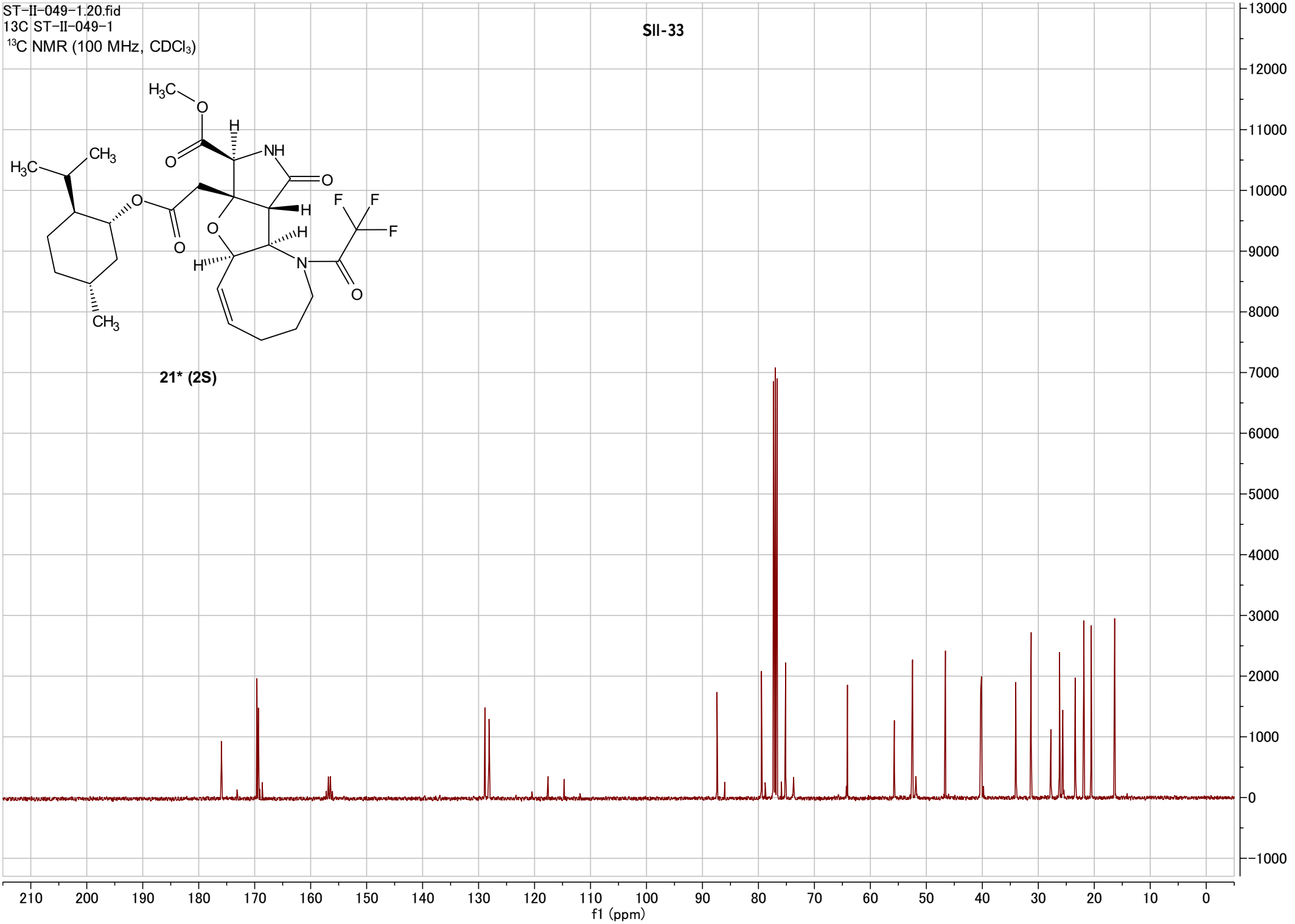
21* (2S)



ST-II-049-1.20.fid
13C ST-II-049-1
^{13}C NMR (100 MHz, $CDCl_3$)

SII-33

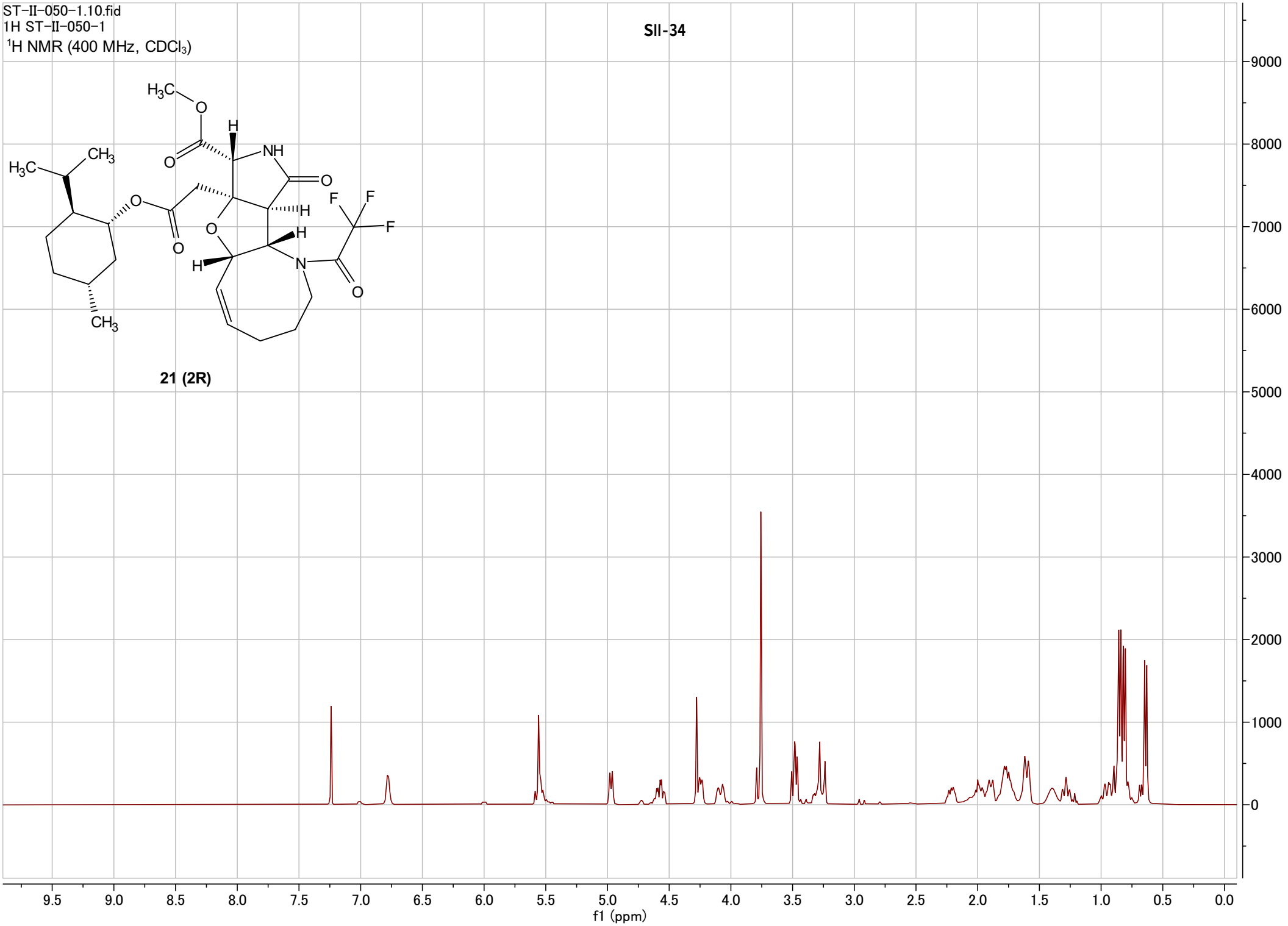
21* (2S)



ST-II-050-1.10.fid
1H ST-II-050-1
^{1}H NMR (400 MHz, $CDCl_3$)

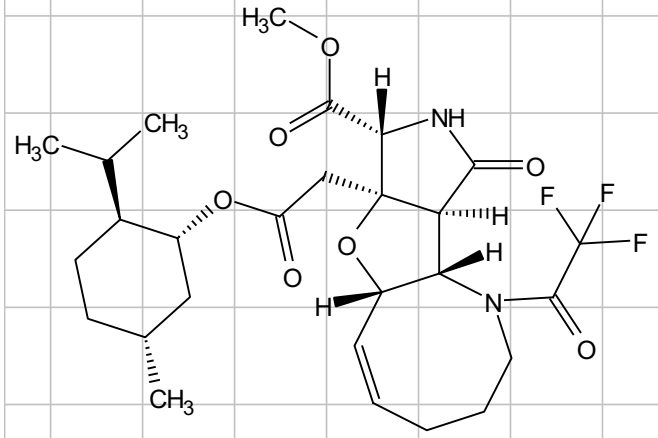
SII-34

21 (2R)



ST-II-050-1.13.fid
13C ST-II-050-1
^{13}C NMR (100 MHz, $CDCl_3$)

SII-35

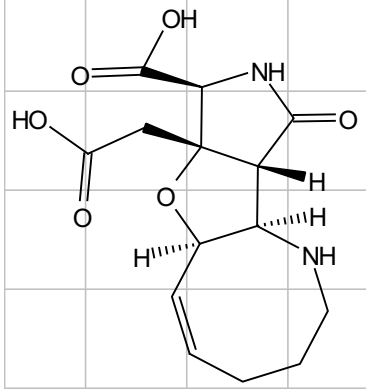


21 (2R)

f1 (ppm)

RS1-50-2
single_pulse
^{1}H NMR (400 MHz, D_2O)

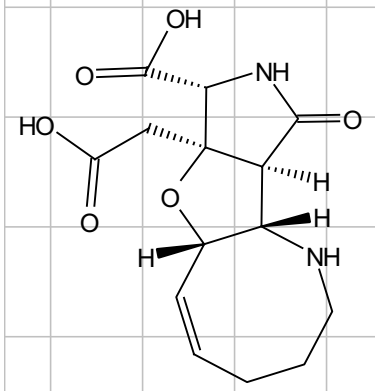
SII-36



(2*S*)–TKM–38 (3*, TFA salt)

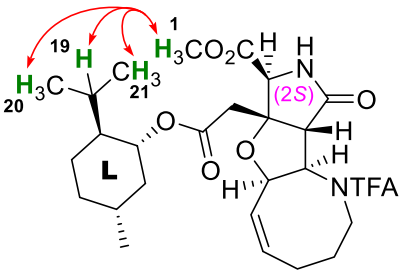
ST-II-052-1.11.fid
13C ST-II-052-1
^{13}C NMR (100 MHz, D_2O)

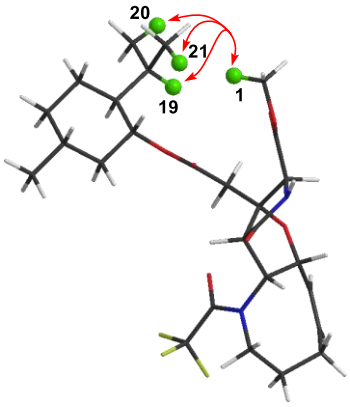
SII-37



(2*R*)–TKM–38 (3)

21* (2*S*)



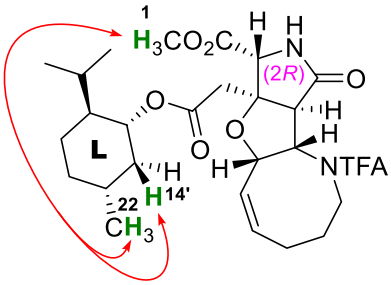


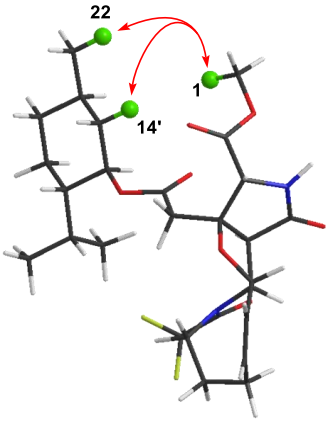
NOESY

CONFLEX
population 89%

---

21 (2*R*)

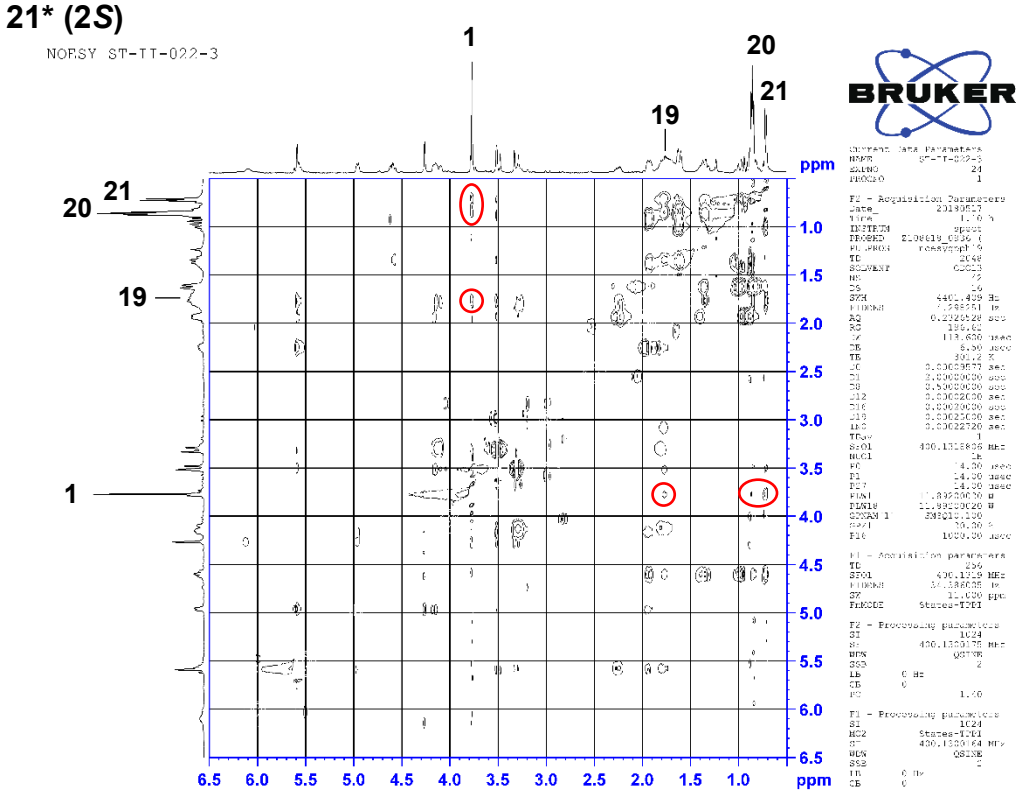




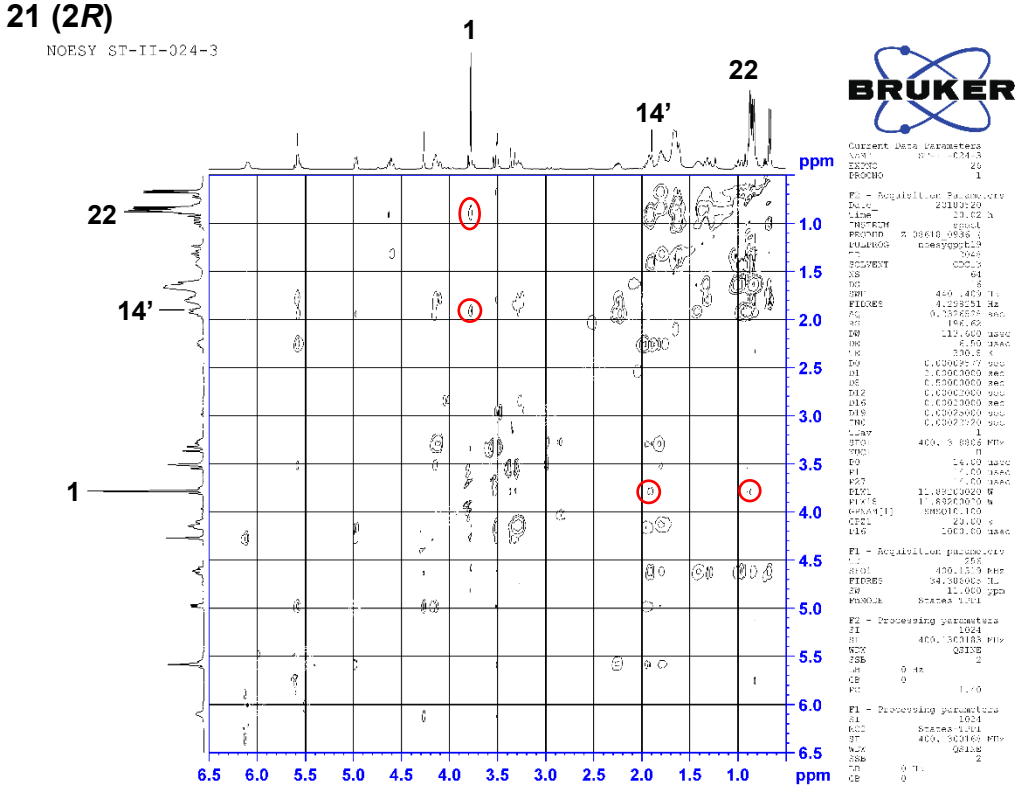
NOESY

CONFLEX
population 77%

21* (2*S*)


NOESY ST-TT-022-3
1
20
21
19
ppm
6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
BRUKER

21 (2*R*)


NOESY ST-TT-024-3
1
22
14'
ppm
6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
BRUKER