Supporting Information

Mercuric triflate-catalyzed biomimetic tandem cyclization leading to polycarbocycles

Hiroshi Imagawa, Tomoaki Iyenaga, and Mugio Nishizawa*

Faculty of Pharmaceutical Sciences, Tokushima Bunri University,
Yamashiro-cho, Tokushima 770-8514, Japan

mugi@ph.bunri-u.ac.jp

Spectroscopic Data

1: ^1H HMR (200 MHz in CDCl_3) δ 1.57 (d, $J = 1.2$ Hz, 3H), 1.77 (t, $J = 2.2$ Hz, 3H), 2.16-2.24 (m, 4H), 2.29 (dt, $J = 7.2, 7.6$ Hz, 2H), 2.58 (t, $J = 7.6$ Hz, 2H), 3.78 (s, 6H), 5.22 (brt, $J = 7.2$ Hz, 1H), 6.30 (t, $J = 2.2$ Hz, 1H), 6.36 (d, $J = 2.2$ Hz, 2H); ^{13}C NMR (50 MHz in CDCl_3) δ 3.24 q, 15.66 q, 17.80 t, 29.54 t, 36.18 t, 38.92 t, 55.04 q (2C), 75.57 s, 78.94 s, 97.60 d, 106.43 d, 124.48 d, 134.47 s, 144.69 s, 160.70 s; FTIR 2940, 2838, 2216, 1614, 1471, 1070, 832, 695 cm^{-1} ; MS (EI) m/z 272 (M^+); HRMS (EI) calcd for $\text{C}_{18}\text{H}_{24}\text{O}_2$ (M^+) 272.1776, found 272.1771.

2: ^1H HMR (600 MHz in CDCl_3) δ 1.41 1.41 (ddt, $J = 6.9, 13.2, 13.2$ Hz,

1H), 1.55 (dq, $J = 5.5, 13.2$ Hz, 1H), 1.71 (brs, 3H), 1.97 (ddt, $J = 1.6, 6.6, 13.2$ Hz, 1H), 2.06 (m, 1H), 2.18 (m, 1H), 2.35 (brd, $J = 13.6$ Hz, 1H), 2.80 (dd, $J = 4.1, 17.4$ Hz, 1H), 2.92 (ddd, $J = 6.5, 12.0, 17.4$ Hz, 1H), 3.08 (dd, $J = 6.6, 13.2$ Hz, 1H), 3.74 (d, $J = 5.5$ Hz, 1H), 5.42 (brs, 1H), 6.23 (d, $J = 2.6$ Hz, 1H), 6.30 (d, $J = 2.6$ Hz, 1H); ^{13}C NMR (150 MHz in CDCl_3) δ 17.25 q, 20.28 t, 21.81 q, 23.90 t, 32.41 t, 32.72 t, 36.84 s, 47.16 d, 55.01 q, 55.09 q, 97.40 d, 104.87 d, 121.80 d, 127.87 s, 134.54 s, 138.81 s, 157.92 s, 160.02 s; FTIR 2959, 2838, 1613, 1583, 1463, 1285, 1203, 1155, 1107, 813 cm^{-1} ; MS (EI) m/z 272 (M^+); HRMS (EI) calcd for $\text{C}_{18}\text{H}_{24}\text{O}_2$ (M^+) 272.1776, found 272.1778.

7: ^1H HMR (200 MHz in CDCl_3) δ 1.34 (s, 3H), 1.59 (s, 3H), 1.77 (s, 3H), 2.01-2.20 (m, 8H), 2.45 (s, 3H), 3.79 (d, $J = 8.0$ Hz, 2H), 5.10 (m, 1H), 5.18 (brt, $J = 7.8$ Hz, 1H), 7.33 (d, $J = 8.0$ Hz, 2H), 7.74 (d, $J = 8.0$ Hz, 2H); ^{13}C NMR (50 MHz in CDCl_3) δ 3.17 q, 15.52 q, 15.95 q, 17.65 t, 21.34 q, 25.85 t, 38.76 t, 39.31 t, 55.85 t, 75.46 s, 78.75 s, 110.40 d, 124.19 d, 128.36 d (2C), 129.44 d (2C), 134.23 s, 135.69 s, 144.35 s, 145.92 s; FTIR 2918, 1611, 1597, 1316, 1152, 1088, 899, 816, 746, 629 cm^{-1} ; MS (CI) m/z 345 ($\text{M}^+\text{+H}$); HRMS (CI) calcd for $\text{C}_{21}\text{H}_{29}\text{O}_2\text{S}$ ($\text{M}^+\text{+H}$) 345.1888, found 345.1862.

8: ^1H HMR (200 MHz in CDCl_3) δ 0.63 (s, 3H), 1.09 (ddd, $J = 7.8, 10.0,$

13.2 Hz), 1.37-1.51 (m, 2H), 1.59 (brs, 3H), 1.76 (brs, 3H), 1.92 (m, 2H), 2.13 (m, 2H), 2.45 (s, 3H), 2.64 (brs, 1H), 3.16 (d, $J = 4.4$ Hz, 2H), 5.36 (brs, 1H), 5.50 (brs, 1H), 7.36 (d, $J = 8.4$ Hz, 2H), 7.82 (d, $J = 8.4$ Hz, 2H); ^{13}C NMR (50 MHz in CDCl_3) δ 11.96 q, 20.79 q, 21.61 q, 22.14 q, 22.38 t, 25.93 t, 33.44 t, 35.32 s, 44.25 d, 45.17 d, 54.57 t, 121.64 d, 124.43 d, 128.23 d (2C), 129.89 d (2C), 132.85 s, 134.25 s, 137.41 s, 144.55 s; FTIR 3029, 2963, 2923, 2843, 1597, 1444, 1313, 1301, 1151, 1089, 818 cm^{-1} ; MS (EI) m/z 344 (M^+); HRMS (EI) calcd for $\text{C}_{21}\text{H}_{28}\text{O}_2\text{S}$ (M^+) 344.1810, found 344.1814.

9: ^1H HMR (200 MHz in CDCl_3) δ 1.60 (s, 3H), 1.71 (s, 3H), 1.78 (s, 3H), 2.06 (s, 3H), 2.06-2.17 (m, 1H), 4.59 (d, $J = 7.0$ Hz, 2H), 5.15 (brt, $J = 6.0$ Hz, 1H), 5.35 (brt, $J = 7.0$, 1H); ^{13}C NMR (50 MHz in CDCl_3) δ 2.98 q, 15.43 q, 16.00 q, 17.62 t, 20.53 q, 25.79 t, 38.73 t, 39.09 t, 60.94 t, 75.23 s, 78.62 s, 118.36 d, 124.31 d, 133.99 s, 141.60 s, 170.62 s; FTIR 2914, 1746, 1668, 1444, 1384, 1250, 1023, 836 cm^{-1} ; MS (CI) m/z 249 ($\text{M}^+ + \text{H}$); HRMS (CI) calcd for $\text{C}_{16}\text{H}_{25}\text{O}_2$ ($\text{M}^+ + \text{H}$) 249.1855, found 249.1882.

10: ^1H HMR (400 MHz in CDCl_3) δ 0.74 (s, 3H), 1.25 (ddd, $J = 5.8, 12.0, 12.0$ Hz, 1H), 1.61 (s, 3H), 1.64-1.76 (m, 2H), 1.72 (s, 3H), 1.91 (dd, $J = 5.6, 12.0$ Hz, 1H), 1.98-2.20 (m, 4H), 2.05 (s, 3H), 4.16 (dd, $J = 5.9, 11.7$ Hz, 1H), 4.29 (dd, $J = 3.3, 11.7$ Hz, 1H), 5.39 (brs, 1H), 5.55 (d, $J = 4.4$ Hz,

1H); ^{13}C NMR (50 MHz in CDCl_3) δ 12.36 q, 20.98 q, 21.23 q, 21.89 q, 22.57 t, 25.77 t, 34.03 t, 34.27 s, 44.34 d, 50.02 d, 62.98 t, 121.40 d, 123.87 d, 133.45 s, 134.17 s, 171.19 s; FTIR 2965, 2922, 2844, 1742, 1443, 1364, 1236, 1037, 822 cm^{-1} ; MS (EI) m/z 248 (M^+); HRMS (EI) calcd for $\text{C}_{16}\text{H}_{24}\text{O}_2$ (M^+) 248.1776, found 248.1770.

11

11: ^1H HMR (200 MHz in CDCl_3) δ 0.05 (s, 6H), 0.89 (s, 9H), 1.58 (s, 3H), 1.61 (s, 3H), 1.74 (s, 3H), 1.95-2.19 (m, 8H), 4.17 (d, $J = 6.4$ Hz, 2H), 5.14 (t, $J = 6.0$ Hz, 1H), 5.29 (t, $J = 6.4$ Hz, 1H); ^{13}C NMR (50 MHz in CDCl_3) δ -5.08 s (2C), 3.40 q, 15.78 q, 16.31 q, 17.99 t, 18.38 s, 25.99 q (3C), 26.29 t, 39.09 t, 39.42 t, 60.28 t, 75.51 s, 79.00 s, 124.53 d, 124.93 d, 133.90 s, 136.69 s; FTIR 2956, 1669, 1471, 1384, 1255, 1111, 1069, 841, 778, 664 cm^{-1} ; MS (EI) m/z 320 (M^+); HRMS (EI) calcd for $\text{C}_{20}\text{H}_{36}\text{OSi}$ (M^+) 320.2524, found 320.2535.

12

12: ^1H HMR (200 MHz in CDCl_3) δ 0.05 (s, 3H), 0.72 (s, 3H), 0.89 (s, 9H), 1.24 (m, 1H), 1.59 (s, 3H), 1.76 (s, 3H), 1.70-2.14 (m, 7H), 3.67 (dd, $J = 5.6, 10.2$ Hz, 1H), 3.82 (dd, $J = 3.6, 10.2$ Hz, 1H), 5.36 (brs, 1H), 5.46 (brs, 1H); ^{13}C NMR (50 MHz in CDCl_3) δ -5.46 q (2C), 12.59 q, 18.14 s, 21.01 q, 22.30 q, 22.75 t, 25.94 q (3C), 34.24 t, 34.29 t, 44.59 d, 53.44 d, 61.11 t, 121.28 d, 122.76 d, 134.60 s, 135.30 s; FTIR 2958, 2928, 2855, 1471, 1462, 1438, 1254, 1107, 838, 774 cm^{-1} ; MS (EI) m/z 320 (M^+); HRMS (EI) calcd

for $C_{20}H_{36}OSi$ (M^+) 320.2535, found 320.2538.

13: 1H HMR (200 MHz in $CDCl_3$) δ 1.74 (s, 3H), 1.76 (s, 3H), 2.26 (m, 4H), 3.76 (s, 6H), 4.50 (d, $J = 6.6$ Hz, 2H), 5.52 (t, $J = 6.6$ Hz, 1H), 6.06-6.11 (m, 3H); ^{13}C NMR (50 MHz in $CDCl_3$) δ 3.36 q, 16.45 q, 17.52 t, 38.78 t, 55.24 q (2C), 64.75 t, 76.00 s, 78.45 s, 92.94 d, 93.52 d (2C), 120.31 d, 139.66 s, 160.70 s, 161.44 s (2C); FTIR 2943, 2838, 1596, 1471, 1204, 1148, 1062, 822 cm^{-1} ; MS (EI) m/z 274 (M^+); HRMS (EI) calcd for $C_{17}H_{22}O_3$ (M^+) 274.1569, found 274.1570.

14: 1H HMR (600 MHz in $CDCl_3$) δ 1.36 (s, 3H), 1.76 (d, $J = 1.6$ Hz, 3H), 1.81 (m, 1H), 1.87 (m, 1H), 1.96 (m, 1H), 2.01 (m, 1H), 2.11 (brd, $J = 9.0$ Hz, 1H), 3.74 (s, 3H), 3.77 (s, 3H), 3.84 (dd, $J = 9.0, 10.8$ Hz, 1H), 4.18 (dd, $J = 3.0, 10.8$ Hz, 1H), 5.57 (brs, 1H), 6.01 (d, $J = 2.5$ Hz, 1H), 6.07 (d, $J = 2.5$ Hz, 1H); ^{13}C NMR (150 MHz in $CDCl_3$) δ 22.36 q, 22.59 t, 23.83 q, 29.29 t, 33.58 s, 47.34 d, 55.11 q, 55.14 q, 65.54 t, 92.83 d, 93.56 d, 112.35 s, 124.83 d, 130.10 s, 156.50 s, 158.89 s, 160.56 s; FTIR 2933, 1614, 1585, 1459, 1203, 1153, 814 cm^{-1} ; MS (EI) m/z 274 (M^+); HRMS (EI) calcd for $C_{17}H_{22}O_3$ (M^+) 274.1569, found 274.1562.

15

15: ^1H NMR (200 MHz in CDCl_3) δ 1.18 (s, 3H), 1.45 (m, 1H), 1.67 (t, $J = 1.6$ Hz, 3H), 2.14 (m, 2H), 2.63 (brd, $J = 12.0$ Hz, 1H), 3.02 (ddd, $J = 1.8, 5.8, 12.8$ Hz, 1H), 3.74 (s, 3H), 3.76 (s, 3H), 3.98 (dd, $J = 10.2, 12.0$ Hz, 1H), 4.38 (dd, $J = 3.4, 10.2$ Hz, 1H), 5.46 (brs, 1H), 6.02 (d, $J = 2.6$ Hz, 1H), 6.06 (d, $J = 2.6$ Hz, 1H); ^{13}C NMR (50 MHz in CDCl_3) δ 18.15, 20.65, 23.50, 31.67, 33.54, 44.46, 55.15 (2C), 64.20, 92.21, 93.77, 113.66, 123.42, 129.94, 155.60, 159.23, 160.25; FTIR 2929, 1614, 1584, 1462, 1437, 1420, 1201, 1154, 1105, 814 cm^{-1} ; MS (EI) m/z 274 (M^+); HRMS (EI) calcd for $\text{C}_{17}\text{H}_{22}\text{O}_3$ (M^+) 274.1575, found 274.1562.

16

16: ^1H NMR (200 MHz in CDCl_3) δ 1.82 (d, $J = 1.4$ Hz, 6H), 2.65 (m, 2H), 2.85 (dt, $J = 4.8, 9.4$ Hz, 1H), 3.76 (s, 6H), 3.98 (d, $J = 4.8$ Hz, 2H), 5.62 (brs, 2H), 6.07 (s, 3H); ^{13}C NMR (50 MHz in CDCl_3) δ 22.18 (2C), 27.57, 45.16, 55.34 (2C), 69.43, 92.99, 93.50 (2C), 121.87 (2C), 132.98 (2C), 161.03, 161.44 (2C); MS (CI) m/z 275 ($\text{M}^+ + \text{H}$).

LEE-30-3

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: LEE-30-3-1H061004_23_08

INOVA-600 "NMR"

Relax. delay 1.000 sec

Pulse 31.5 degrees

Acq. time 1.994 sec

Width 3000.3 Hz

32 repetitions

OBSERVE H1, 199.8863580 MHz

DATA PROCESSING

FT size 16384

Total time 1 min, 36 sec

LEE-30-3

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: LEE-30-3-13C061004_23_19

INOVA-600 "NMR"

Pulse 60.0 degrees

Acq. time 1.498 sec

Width 12500.0 Hz

128 repetitions

OBSERVE C13, 50.2614276 MHz

DECOUPLE H1, 199.8873538 MHz

Power 30 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 30 min, 7 sec

LEE-30-3

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: LEE-30-3-13C061004_23_19

INOVA-600 "NMR"

Pulse 60.0 degrees

Acq. time 1.498 sec

Width 12500.0 Hz

128 repetitions

OBSERVE C13, 50.2614276 MHz

DECOUPLE H1, 199.8873538 MHz

Power 30 dB

continuously on

=====

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 30 min, 7 sec

積算回数
 ゼロフィリング
 ゲイン
 測定日時
 測定者
 ファイル名
 サンプル名
 コメント

128
 ON
 Auto (8)
 2004/09/22 20:12
 ienaga
 Memory#2
 LEE-30-3

分解
 アポダイゼーション
 スキャンスピード
 更新日時

4 cm-1
 Cosine
 Auto (2 mm/sec)
 2004/09/22 20:14

No.	cm-1	%R	No.	cm-1	%R	No.	cm-1	%R	No.	cm-1	%R
1	3649.62	95.8483	2	2940.91	7.79518	3	2837.74	9.65318	4	2737.46	83.3655
6	2496.4	92.6543	7	2216.77	91.6143	8	2123.24	88.2272	9	2077.92	88.7475
11	1846.51	95.8319	12	1667.16	77.005	13	1614.13	8.91639	14	1471.42	10.2273
16	1348	13.6682	17	1314.25	16.0063	18	1294	14.8713	19	1257.36	61.1068
21	1162.87	9.65095	22	1070.3	11.4651	23	1028.84	52.8294	24	993.16	62.0262
26	938.199	45.2989	27	924.7	30.5628	28	832.133	10.6506	29	745.352	94.7162
31	851.822	89.9068	32	573.719	72.4297	33	539.007	63.3686	34	504.294	61.971
36	417.513	92.2448							5	2642	93.3936
									10	1980.54	94.6279
									15	1382.71	39.7302
									20	1209.15	9.79309
									25	970.019	73.4791
									30	695.212	17.5108
									35	431.012	93.7707

[Mass Spectrum]

Data : NISHIZAWA-EI.11-JUN-04.001 Date : 11-Jun-2004 12:03

Sample: LEE-30-3

Note : MStation

Inlet : Direct

Ion Mode : EI+

Spectrum Type : Normal Ion [MF-Linear]

RT : 0.67 min Scan# : 21

BP : m/z 152.0000 Int. : 1585.55

Output m/z range : 36.1350 to 503.7611

Cut Level : 0.00 %

1

I Elemental Composition I

Page: 1

Data : NISHIZAWA-EIHR.30-JUN-04.005 Date : 30-Jun-2004 10:08

Sample: LEE-30-3

Note : MStation

Inlet : Direct

RT : 0.96 min

Elements : C 100/0, H 150/0, O 20/0, S 1/0

Mass Tolerance : 5mmu

Unsaturation (U.S.) : 0.0 - 30.0

Ion Mode : EI+

Scan#: 23

Observed m/z	Int%	Err [ppm / mmu]	U.S.	Composition
272.1771	77.6	-1.9 / -0.5	7.0	C 18 H 24 O 2
		-14.2 / -3.9	3.0	C 15 H 28 O 2 S

LEE-31-1-A

exp9 s2pul

SAMPLE		DEC. a VT	
date	Nov 12 2003	dfrq	599.963
solvent	CDCl3	ds	H1
file	/export/home/-	dpwr	45
nmr1/vnarsys/data-		dof	0
/nishizawa/lee311a-		dm	nna
_1b.fid		dmm	c
ACQUISITION		PROCESSING	
sfrq	599.963	dsag	
tn	H1	dres	1.0
at	3.641	homo	n
np	65536	temp	25.0
sw	9000.9		
fb	5000	wtfile	
bs	4	proc	it
tpwr	57	fn	65536
pw	10.3	math	f
dl	1.359		
tof	-600.0	werr	
nt	64	wexp	
ct	64	wba	
alock	n	wnt	wEt
gain	45		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	284.0		
wp	3999.8		
vs	211		
sc	0		
wc	400		
hsmm	10.00		
is	2150.86		
rf1	751.6		
rfp	0		
th	7		
ins	100.000		
nm	ph

LEB-31-1-A

exp8 s2pul

SAMPLE		DEC. & VT	
date	Nov 12 2003	dfrq	599.962
solvent	CDCl3	dn	H1
file	/export/home/-	dpwr	45
nmr1/vnmrpy/data-	doz		-1000.0
/nishiawa/lee311a-	dm	y	
	_13c.fid	dmm	w
ACQUISITION		daf	12500
sfrq	150.877	dseq	garpl
tn	C13	dres	1.0
at	0.905	homo	n
np	65536	temp	25.0
sw	36199.1	PROCESSING	
fb	20000	lb	1.50
bs	16	wtfile	
tpwr	54	proc	ft
pw	7.0	fn	65536
dl	1.200	math	f
tof	3384.3		
nt	65536	warr	
ct	512	wexp	wft
slock	n	wbs	wft
gain	56	wnc	
FLAG			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	13577.1		
wp	13577.3		
vs	90		
sc	0		
nc	320		
hsum	42.43		
is	500.00		
rf1	12528.9		
rfp	11616.2		
ch	6		
ins	100.000		
na	ph

積算回数
ゼロフィリング
ゲイン
測定日時
測定者
ファイル名
サンプル名
コメント

24
ON
Auto (4)
2004/06/18 0:01
ienaga
LEE-31-10-A
LEE-31-10-A

分解
アポダイゼーション
スキャンスピード
更新日時

4 cm-1
Cosine
Auto (2 mm/sec)
2004/09/30 11:08

No.	cm-1	%R	No.	cm-1	%R	No.	cm-1	%R	No.	cm-1	%R
1	2959.23	92.8762	2	2838.7	95.0898	3	1613.16	91.6681	4	1583.27	91.8276
6	1285.32	96.9328	7	1203.36	94.3228	8	1155.15	92.1712	9	1106.94	95.9044
11	612.288	97.5193	12	544.792	96.1801	13	485.009	97.3651	14	475.367	98.1438
16	443.547	98.517	17	430.048	96.7903	18	418.477	87.9063	19	407.871	86.349
									5	1462.74	96.1919
									10	812.849	97.4944
									15	459.939	95.3558

[Mass Spectrum]

Data : NISHIZAWA-EI.06-JUL-04.001 Date : 06-Jul-2004 09:29

Sample: LEE-31-13-A

Note : MStation

Inlet : Direct

Ion Mode : EI+

Spectrum Type : Normal Ion [MF-Linear]

RT : 0.47 min Scan# : 15

BP : m/z 257.0000 Int. : 1599.98

Output m/z range : 37.2700 to 506.0312

Cut Level : 0.00 %

[Elemental Composition]

Page: 1

Data : NISHIZAWA-EIHR.07-JUL-04.001 Date : 07-Jul-2004 13:47

Sample: LEE-31-13-A

Note : MStation

Inlet : Direct

Ion Mode : EI+

RT : 1.09 min

Scan#: 26

Elements : C 100/0, H 150/0, O 20/0, S 1/0

Mass Tolerance : 5mmu

Unsaturation (U.S.) : 0.0 ~ 30.0

Observed m/z	Int%	Err [ppm / mmu]	U.S.	Composition
272.1778	32.5	+0.6 / -11.8	7.0	C 18 H 24 O 2
			3.0	C 15 H 28 O 2 S

LEE-47-1

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: LEE-47-1-1H031204_16_58

INOVA-600 "NMR"

Relax. delay 1.000 sec

Pulse 31.5 degrees

Acq. time 1.994 sec

Width 3000.3 Hz

32 repetitions

OBSERVE H1, 199.8863492 MHz

DATA PROCESSING

FT size 16384

Total time 1 min, 36 sec

LEE-47-1

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: LEE-47-1-13C031204_17_11

INOVA-600 "NMR"

Pulse 60.0 degrees

Acq. time 1.498 sec

Width 12500.0 Hz

192 repetitions

OBSERVE C13, 50.2614310 MHz

DECOUPLE H1, 199.8873538 MHz

Power 30 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 15 min, 3 sec

LEE-47

Pulse Sequence: dept

Solvent: cdcl3

Ambient temperature

File: LEE-47--dept072204_20_50

INOVA-600 "NMR"

Relax. delay 2.000 sec

Pulse 90.0 degrees

Acq. time 2.134 sec

Width 14992.5 Hz

120 repetitions

OBSERVE C13, 50.3073510 MHz

DECOUPLE H1, 200.0697598 MHz

Power 40 dB

on during acquisition

off during delay

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 8 min, 52 sec

7

積算回数
ゼロフィリング
ゲイン
測定日時
測定者
ファイル名
サンプル名
コメント

30
ON
Auto (4)
2004/07/22 22:12
ienaga
Memory#2
LEE-47

分解
アボダイゼーション
スキャンスピード
更新日時

4 cm-1
Cosine
Auto (2 mm/sec)
2004/07/22 22:14

No.	cm-1	%R	No.	cm-1	%R	No.	cm-1	%R	No.	cm-1	%R	No.	cm-1	%R
1	3851.15	94.547	2	3646.73	88.7018	3	3543.56	90.9295	4	2917.77	52.2184	5	1920.75	92.5864
6	1661.37	75.2793	7	1596.77	64.1691	8	1494.56	72.4465	9	1446.35	61.2325	10	1316.18	55.9512
11	1237.11	66.773	12	1152.26	58.6959	13	1087.66	61.4007	14	1019.19	76.225	15	898.666	71.5716
16	815.742	62.9812	17	746.317	61.2312	18	666.285	66.7735	19	628.68	68.8905	20	565.041	61.0811
21	515.865	62.5898	22	464.761	79.9884									

[Mass Spectrum]
Data : NISHIZAWA-CI.23-JUL-04.001 Date : 23-Jul-2004 09:20
Sample: LEE-47(iso-Bu)

Sample: LEE-47(iso-Bu)

Inlet : Direct

Inlet : Direct

Ion Mode : CI+

Spectrum Type : Normal Ion [MF-Linear]

RT : 1.34 min Scan# : 50-k(13)[k=1.0]

BP : m/z	189.0000	Int. : 399.82
----------	----------	---------------

Output m/z range : 36.1350 to 507.1662

Cut Level : 0.00 %

7

[Elemental Composition]

Page: 1

Data : NISHIZAWA-CIHR.24-JUL-04.002 Date : 24-Jul-2004 11:04

Sample: LEE-47

Note : MStation

Inlet : Direct

RT : 1.56 min

Elements : C 100/0, H 150/0, O 20/0, S 1/0

Mass Tolerance : 5mmu

Unsaturation (U.S.) : 0.0 - 30.0

Ion Mode : CI+

Scan#: 40

Observed m/z	Int%	Err [ppm / mmu]	U.S.	Composition
345.1862	11.9	+2.2 / -7.6	12.5	C 24 H 25 O 2
			8.5	C 21 H 29 O 2 S

LEE-50-5-B

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: LEE-50-5-B-1H072904_14_00

INOVA-600 "NMR"

Relax. delay 1.000 sec

Pulse 42.0 degrees

Acq. time 4.000 sec

Width 4000.0 Hz

32 repetitions

OBSERVE H1, 200.0687963 MHz

DATA PROCESSING

FT size 32768

Total time 2 min, 40 sec

LEE-50-5-B

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: LEE-50-5-B-13C072904_14_57

INOVA-600 "NMR"

Relax. delay 1.000 sec

Pulse 31.2 degrees

Acq. time 2.134 sec

Width 14992.5 Hz

736 repetitions

OBSERVE C13, 50.3073373 MHz

DECOUPLE H1, 200.0697598 MHz

Power 40 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 1 hr, 2 min, 51 sec

8

LEE-50-5-B

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: LEE-50-5-B-13C072904_14_57

INOVA-600 "NMR"

Relax. delay 1.000 sec

Pulse 31.2 degrees

Acq. time 2.134 sec

Width 14992.5 Hz

736 repetitions

DECOUPLE H1, 200.0697598 MHz

Power 40 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 1 hr, 2 min, 51 sec

積算回数
ゼロファイリング
ゲイン
測定日時
測定者
ファイル名
サンプル名
コメント

100
ON
Auto (4)
2004/09/10 1:50
IENAGA
Memory#2
lee-50-5-b

分解
アボダイゼーション
スキャンスピード
更新日時

4 cm-1
Cosine
Auto (2 mm/sec)
2004/09/10 1:52

No.	cm-1	%R	No.	cm-1	%R	No.	cm-1	%R	No.	cm-1	%R
1	3537.77	97.0085	2	3028.66	76.8203	3	2963.09	45.3904	4	2922.59	39.8556
6	2363.34	97.3629	7	1666.2	91.5221	8	1596.77	65.1865	9	1493.6	84.5194
11	1407.78	75.8046	12	1378.85	64.4233	13	1313.29	33.9568	14	1301.72	35.2586
16	1230.36	86.216	17	1215.9	83.2674	18	1151.29	24.025	19	1119.48	63.9303
21	1041.37	77.0758	22	1019.19	82.8449	23	996.053	91.1114	24	969.055	94.9069
26	935.306	91.6677	27	886.131	73.2813	28	862.025	84.8685	29	846.597	79.0917
31	764.637	59.977	32	720.282	58.5441	33	670.142	51.2121	34	651.822	68.6315
36	617.109	79.6623	37	556.363	44.9686	38	534.185	53.8463	39	507.187	64.5928
41	464.761	85.1891							40	488.866	82.5647
									5	2842.56	54.1158
									10	1444.42	50.464
									15	1289.18	46.4338
									20	1088.62	42.0961
									25	950.734	93.817
									30	817.67	46.5778
									35	636.394	78.7085

[Mass Spectrum]
Data : NISHIZAWA-EI.10-SEP-04.001 Date : 10-Sep-2004 09:23
Sample: LEE-50-5-B

Sample: LEE-50-5-B

Inlet : Direct

Inlet : Direct

Ion Mode : EI+

Spectrum Type : Normal Ion [MF-Linear]

RT : 0.87 min Scan# : 27

BP : m/z 173.0000 Int. : 1250.81

Output m/z range : 35.0000 to 503.7611

Cut Level : 0.00 %

8

[Elemental Composition]

Page: 1

Data : NISHIZAWA-EIHR.14-SEP-04.003 Date : 14-Sep-2004 11:06

Sample: LEE-50-5-B

Note : MStation

Inlet : Direct

RT : 1.71 min

Elements : C 100/0, H 150/0, O 20/0, S 1/0

Mass Tolerance : 5mmu

Unsaturation (U.S.) : 0.0 - 30.0

Ion Mode : EI+

Scan#: 42

Observed m/z	Int%	Err [ppm / mmu]	U.S.	Composition
344.1814	69.0	+10.9 / +3.7	13.0	C 24 H 24 O 2
		-6.2 / -2.1	4.0	C 17 H 28 O 7
		+1.1 / +0.4	9.0	C 21 H 28 O 2 S

LEE-49-1

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: LEE-49-1-1H031204_17_18

INOVA-600 "NMR"

Relax. delay 1.000 sec

Pulse 31.5 degrees

Acq. time 1.994 sec

Width 3000.3 Hz

32 repetitions

OBSERVE H1, 199.8863529 MHz

DATA PROCESSING

FT size 16384

Total time 1 min, 36 sec

LEE-49-1-13C

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: LEE-49-1-13C031204_17_29

INOVA-600 "NMR"

Pulse 60.0 degrees

Acq. time 1.498 sec

Width 12500.0 Hz

163 repetitions

OBSERVE C13, 50.2614291 MHz

DECOUPLE H1, 199.8873538 MHz

Power 30 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 30 min, 7 sec

9

LEE-49-1-13C

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: LEE-49-1-13C031204_17_29

INOVA-600 "NMR"

Pulse 60.0 degrees

Acq. time 1.498 sec

Width 12500.0 Hz

163 repetitions

OBSERVE C13, 50.2614291 MHz

DECOUPLE H1, 199.8873538 MHz

Power 30 dB

continuously on

~~NAME: 13C-1204_17_29~~

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 30 min, 7 sec

9

積算回数
ゼロフィリング
ゲイン
測定日時
測定者
ファイル名
サンプル名
コメント

30
ON
Auto (32)
2004/07/22 21:58
ienaga
LEE-49.jws
LEE-49

分解
アボタイゼーション
スキャンスピード
更新日時

4 cm-1
Cosine
Auto (2 mm/sec)
2004/07/22 22:06

No.	cm-1	%R	No.	cm-1	%R	No.	cm-1	%R	No.	cm-1	%R
1	3645.77	91.0707	2	3458.71	84.3614	3	2914.88	7.58063	4	2737.46	78.8882
6	2051.89	89.1848	7	1746.23	7.91913	8	1668.12	36.234	9	1444.42	14.789
11	1338.36	46.8382	12	1249.65	13.0686	13	1088.62	59.2589	14	1023.05	18.0599
16	835.99	54.3575	17	646.036	78.3418	18	607.467	49.333	19	575.647	67.7271
21	441.619	68.5637	22	428.12	72.3599	23	419.442	74.9249	5	2466.51	91.8894
									10	1383.68	14.9345
									15	955.555	24.685
									20	501.401	60.5597

[Mass Spectrum]

Data : NISHIZAWA-CI.23-JUL-04.003 Date : 23-Jul-2004 09:29

Sample: LEE-49(iso-Bu)

Note : MStation

Inlet : Direct

Ion Mode : CI+

Spectrum Type : Normal Ion [MF-Linear]

RT : 0.47 min Scan# : 18-k(68)[k=1.0]

BP : m/z 189.0000 Int. : 381.37

Output m/z range : 40.6751 to 501.4911

Cut Level : 0.00 %

9

[Elemental Composition]

Page: 1

Data : NISHIZAWA-CIHR.24-JUL-04.001 Date : 24-Jul-2004 10:53

Sample: LEE-49

Note : MStation

Inlet : Direct

Ion Mode : CI+

RT : 0.78 min

Scan#: 19

Elements : C 100/0, H 150/0, O 20/0, S 1/0

Mass Tolerance : 5mmu

Unsaturation (U.S.) : 0.0 - 30.0

Observed m/z	Int%	Err [ppm / mmu]	U.S.	Composition
249.1882	4.9	+10.9 / -2.6	4.5	C 16 H 25 O 2
			0.5	C 13 H 29 O 2 S

9

----- ACQUISITION PARAMETERS -----

File Name = 1d_spectrum.255
 Author = JEOL LTD.
 Sample ID = LEX-51-1-A
 Content = Sample Pulse Experiment
 Creation Date = 17-MAR-2004 00:06:54
 Revision Date = 17-MAR-2004 00:36:55

Spec Site = JNM-ECX400
 Spec Type = 1D NMR

Data Format = 1D COMPLEX
 Dimensions = 1
 Dim Title = 1H
 Dim Size = 14384
 Dim Units = [ppm]

Scans = 34
 Mod_return = 2
 X_points = 16384
 X_preprocs = 0
 X_domain = 1H
 X_offset = 5 [ppm]
 X_freq = 399.7841973 [MHz]
 X_sweep = 5.9980024 [kHz]
 X_resolution = 0.36616006 [Hz]
 X_acq_duration = 2.7312128 [s]
 Digital Filter = FALSE
 Filter Factor = 1
 Delay of start = 1 [s]
 Actual_start_time = 17-MAR-2004 00:02
 Acq_delay = 0.1641 [ms]
 X90 = 16.5 [us]
 Xr90 = 10 [us]
 Tr90 = 10 [us]
 Qua90 = 10 [us]
 X90_h1 = 11 [us]
 Xr90_h1 = 10 [us]
 Tr90_h1 = 10 [us]
 Qua90_h1 = 10 [us]
 X90_lo = 44 [us]
 Xr90_lo = 10 [us]
 Tr90_lo = 10 [us]
 Qua90_lo = 10 [us]
 Spin_lock_90 = 38 [us]
 Spin_lock_attn = 12 [dB]
 Deut_grad_shim_90 = 10 [us]
 Deut_grad_shim_attn = 61.875 [dB]
 Adv_card = 16/1MHz/20
 Field_strength = 9.389766 [T]
 Filter_mode = BUTTERWORTH
 Filter_width = 2.9836649 [kHz]
 Recvr_gain = 13
 Ixr_code = 210
 Obs_pwidth = 1 [us]
 Obs_noise = WAGGE
 Ixr_pwidth = 1 [us]
 Ixr_noise = WAGGE
 Trl_pwidth = 1 [us]
 Trl_noise = WAGGE
 Qua_pwidth = 1 [us]
 Qua_noise = WAGGE
 Solvent = CHLOROFORM-D
 Lock_strength = 1391
 Lock_level = 180
 Lock_gain = 27
 Lock_psc_offset = 61.6311 [kHz]
 Lock_phase = 9.8 [deg]
 Lock_psc_state = 28 GPC ON
 Lock_state = LOCK ON
 Autolock_level = 180
 Sawtooth_range = 4
 Lock_status = YALE
 Lock_settle_point = 4098
 Lock_acquire_point = 0
 Spin_section = SPIN ON
 Spin_state = SPIN ON
 Spin_status = SPIN ON
 Spin_get = 16 [Hz]
 Spin_set = 15 [Hz]
 Spin_gas_monitor = N2
 Temp_action = TEMP OFF
 Temp_state = TEMP OFF
 Temp_status = TEMP OFF
 Temp_get = 20.3 [deg]
 Temp_set = 25 [deg]
 Temp_comp = 0
 Temp_delay = 0 [s]
 Temp_limit_hi = 150 [deg]
 Temp_limit_lo = -100 [deg]
 Temp_heating = -63 [deg]
 Temp_boiling = 61 [deg]
 Temp_ramp_stop = 0 [deg]
 Temp_ramp_wait = 0 [s]
 Temp_substant = 25 [deg]
 Temp_limit_hi_max = 160 [deg]
 Temp_limit_lo_min = 20 [deg]
 Temp_limit_hi_max = 50 [deg]
 Temp_limit_lo_min = -40 [deg]
 Temp_limit_lo_min = -110 [deg]

X : parts per Million : 1H

LEE-51-3-A

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: LEE-51-3-A-13C081704_21_30

INOVA-600 "NMR"

Relax. delay 1.000 sec

Pulse 31.2 degrees

Acq. time 2.134 sec

Width 14992.5 Hz

443 repetitions

OBSERVE C13, 50.3073373 MHz

DECOUPLE H1, 200.0697598 MHz

Power 40 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 52 min, 23 sec

10

LEE-51-3-A

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: LEE-51-3-A-13C081704_21_30

INOVA-600 "NMR"

Relax. delay 1.000 sec

Pulse 31.2 degrees

Acq. time 2.134 sec

Width 14992.5 Hz

443 repetitions

OBSERVE C13, 50.3073373 MHz

DECOUPLE H1, 200.0697598 MHz

Power 40 dB

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 52 min, 23 sec

10

積算回数
ゼロフィリング
ゲイン
測定日時
測定者
ファイル名
サンプル名
コメント

16
ON
Auto (8)
2004/08/18 12:25
ienaga
LEE-51-3-A
LEE-51-3-A

分解
アボダイゼーション
スキャンスピード
更新日時

4 cm-1
Cosine
Auto (2 mm/sec)
2004/09/29 2:12

No.	cm-1	%R	No.	cm-1	%R	No.	cm-1	%R	No.	cm-1	%R
1	3849.22	97.1682	2	3644.8	95.2122	3	2965.02	72.7182	4	2921.63	71.5981
6	1742.37	57.2078	7	1644.02	95.0223	8	1443.46	79.9033	9	1364.39	75.7399
11	1036.55	76.7466	12	970.983	88.6809	13	822.491	84.9585	14	605.539	90.287
16	514.901	92.2502	17	479.224	94.6793	18	452.225	87.6808	19	439.69	87.0031
21	407.871	70.6984							5	2843.52	76.9687
									10	1236.15	60.1428
									15	539.971	91.7761
									20	429.084	86.7783

[Mass Spectrum]

Data : NISHIZAWA-EI.14-SEP-04.002 Date : 14-Sep-2004 16:24

Sample: LEE-51-3-R

Note : MStation

Inlet : Direct

Ion Mode : EI+

Spectrum Type : Normal Ion [MF-Linear]

RT : 0.47 min Scan# : 15

BP : m/z 149.0000 Int. : 1599.98

Output m/z range : 35.0000 to 501.4911

Cut Level : 0.00 %

10

[Elemental Composition]

Page: 1

Data : NISHIZAWA-EIHR.15-SEP-04.002 Date : 15-Sep-2004 13:28

Sample: LEE-51-3-A

Note : MStation

Inlet : Direct

RT : 0.36 min

Elements : C 100/0, H 150/0, O 20/0, S 1/0

Mass Tolerance : 5mmu

Unsaturation (U.S.) : 0.0 - 30.0

Ion Mode : EI+

Scan#: 9

Observed m/z	Int%	Err [ppm / mmu]	U.S.	Composition
248.1770	100.0	-2.4 / -0.6	5.0	C 16 H 24 O 2
		-16.0 / -4.0	1.0	C 13 H 28 O 2 S

10

LEE-42

Pulse Sequence: s2pul1

Solvent: CDCl3

Ambient temperature

File: LEE-42--1H072204_20_16

INOVA-600 "NMR"

Relax. delay 1.000 sec

Pulse 42.0 degrees

Acq. time 4.000 sec

Width 4000.0 Hz

32 repetitions

OBSERVE H1, 200.0688081 MHz

DATA PROCESSING

FT size 32768

Total time 2 min, 40 sec

11

LEE-42

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: LEE-42--13C072204_20_23

INOVA-600 "NMR"

Relax. delay 1.000 sec

Pulse 31.2 degrees

Acq. time 2.134 sec

Width 14992.5 Hz

120 repetitions

OBSERVE C13, 50.3073377 MHz

DECOUPLE H1, 200.0697598 MHz

Power 40 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 6 min, 17 sec

LEE-42

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: LEE-42--13C072204_20_23

INOVA-600 "NMR"

Relax. delay 1.000 sec

Pulse 31.2 degrees

Acq. time 2.134 sec

Width 14992.5 Hz

120 repetitions

OBSERVE C13, 50.3073377 MHz

DECOUPLE H1, 200.0697598 MHz

Power 40 dB

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 6 min, 17 sec

11

積算回数
ゼロフィリング
ゲイン
測定日時
測定者
ファイル名
サンプル名
コメント

30
ON
Auto (4)
2004/07/22 22:19
ienaga
Memory#2
LEE-42

分解
アポダイゼーション
スキャンスピード
更新日時

4 cm-1
Cosine
Auto (2 mm/sec)
2004/07/22 22:20

No.	cm-1	%R	No.	cm-1	%R	No.	cm-1	%R	No.	cm-1	%R	No.	cm-1	%R
1	2956.34	11.3615	2	2737.46	82.4606	3	2708.53	86.9186	4	1669.09	64.2167	5	1471.42	23.592
6	1383.68	29.5303	7	1360.53	43.2668	8	1337.39	69.6765	9	1255.43	16.1949	10	1196.61	72.4263
11	1110.8	17.3733	12	1069.33	14.9674	13	1005.7	38.0454	14	938.199	68.9388	15	840.812	13.9795
16	778.136	15.555	17	664.357	51.7821	18	571.79	80.0841	19	504.294	75.2524	20	418.477	84.9387

[Mass Spectrum]

Data : NISHIZAWA-EI.23-JUL-04.003 Date : 23-Jul-2004 09:00

Sample: LEE-42

Note : MStation

Inlet : Direct

Ion Mode : EI+

Spectrum Type : Normal Ion [MF-Linear]

RT : 0.44 min Scan# : 14

BP : m/z 75.0000 Int. : 1599.98

Output m/z range : 36.1350 to 502.6261

Cut Level : 0.00 %

11

[Elemental Composition]

Page: 1

Data : NISHIZAWA-EIHR.24-JUL-04.001 Date : 24-Jul-2004 11:24

Sample: LEE-42

Note : MStation

Inlet : Direct

Ion Mode : EI+

RT : 0.79 min

Scan#: 20

Elements : C 100/0, H 150/0, O 20/0, Si 1/0

Mass Tolerance : 5mmu

Unsaturation (U.S.) : 0.0 - 30.0

Observed m/z	Int%	Err [ppm / mmu]	U.S.	Composition
320.2524	16.7	+6.3 / +2.0	9.0	C 24 H 32
		-12.1 / -3.9	0.0	C 17 H 36 O 5
		-3.5 / -1.1	4.0	C 20 H 36 O Si

LEE-43-4

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: LEE-43-4-B072604_23_23

INNOVA-500 "NMR"

Relax. delay 1.000 sec

Pulse 42.0 degrees

Acq. time 4.000 sec

Width 4000.0 Hz

16 repetitions

OBSERVE H1, 200.0688066 MHz

DATA PROCESSING

FT size 32768

Total time 1 min, 20 sec

LEE-43-4

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: LEE-43-4-13C072404_15_34

INOVA-600 "NMR"

Relax. delay 1.000 sec

Pulse 31.2 degrees

Acq. time 2.134 sec

Width 14992.5 Hz

500 repetitions

OBSERVE C13, 50.3073359 MHz

DECOUPLE H1, 200.0697598 MHz

Power 40 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 26 min, 11 sec

12

LEE-43-4

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: LEE-43-4-13C072404_15_34

INOVA-600 "NMR"

12

積算回数
ゼロフィリング
ゲイン
測定日時
測定者
ファイル名
サンプル名
コメント

30
ON
Auto (4)
2004/07/24 17:44
ienaga
Memory#2
LEE-43-4-A

分解
アボタイゼーション
スキャンスピード
更新日時

4 cm-1
Cosine
Auto (2 mm/sec)
2004/07/24 17:46

No.	cm-1	%R	No.	cm-1	%R	No.	cm-1	%R	No.	cm-1	%R
1	3029.62	80.2542	2	2958.27	26.9108	3	2928.38	23.792	4	2855.1	33.4207
6	1461.78	64.4163	7	1437.67	61.7484	8	1375.96	68.3336	9	1360.53	80.3158
11	1216.86	80.2027	12	1181.19	87.5125	13	1130.08	62.6705	14	1106.94	32.4532
16	1047.16	70.9628	17	1005.7	57.4471	18	937.235	79.0066	19	872.631	47.0136
21	774.279	31.0076	22	715.461	83.2135	23	672.071	80.2749	24	618.074	91.872
26	448.369	93.2784	27	420.406	88.7682				25	562.148	91.6956

12

[Mass Spectrum]

Data : NISHIZAWA-EI.26-JUL-04.002 Date : 26-Jul-2004 09:29

Sample: LEE-43-4-A

Note : MStation

Inlet : Direct

Ion Mode : EI+

Spectrum Type : Normal Ion [MF-Linear]

RT : 0.54 min Scan# : 17

BP : m/z 263.0000 Int. : 1599.98

Output m/z range : 35.0000 to 504.8961

Cut Level : 0.00 %

12

[Elemental Composition]

Page: 1

Data : NISHIZAWA-EIHR.27-JUL-04.001 Date : 27-Jul-2004 10:29
 Sample: LEE-43-4-A
 Note : MStation
 Inlet : Direct
 RT : 0.71 min
 Elements : C 100/0, H 150/0, O 20/0, Si 1/0
 Mass Tolerance : 5mmu
 Unsaturation (U.S.) : 0.0 - 30.0

Ion Mode : EI+

Scan#: 18

SI 1/0

Observed m/z	Int%	Err [ppm / mmu]	U.S.	Composition
320.2538	31.6	+10.5 / +3.4	9.0	C 24 H 32
		-7.9 / -2.5	0.0	C 17 H 36 O 5
		+0.7 / +0.2	4.0	C 20 H 36 O Si

12

LEE-58-1-A-a

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: LEE-58-1-A-a-1H042604_21_59

INOVA-600 "NMR"

Relax. delay 1.334 sec

Pulse 45.2 degrees

Acq. time 2.666 sec

Width 3000.3 Hz

32 repetitions

OBSERVE H1, 199.9482341 MHz

DATA PROCESSING

FT size 16384

Total time 2 min, 8 sec

13

LEE-58-1

¹³C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: LEE-31-10-13C063004_21_07

INOVA-600 "NMR"

Relax. delay 1.000 sec

Pulse 31.2 degrees

Acq. time 2.134 sec

Width 14992.5 Hz

161 repetitions

OBSERVE C13, 50.3073400 MHz

DECOUPLE H1, 200.0697598 MHz

Power 40 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 26 min, 11 sec

13

8-1-A
LEE-31-10

Pulse Sequence: dept

Solvent: cdcl3

Ambient temperature

File: LEE-31-10-DEPT063004_21_20

INOVA-600 "NMR"

Relax. delay 2.000 sec

Pulse 90.0 degrees

Acq. time 2.134 sec

Width 14992.5 Hz

163 repetitions

OBSERVE C13, 50.3073400 MHz

DECOUPLE H1, 200.0697598 MHz

Power 40 dB

on during acquisition

off during delay

WALTZ-16 modulated

Time from 0.000000

Line broadening 1.0 Hz

FT size 65536

Total time 11.800 min, 52 sec

積算回数
ゼロフィリング
ゲイン
測定日時
測定者
ファイル名
サンプル名
コメント

24
ON
Auto (8)
2004/06/18 0:17
ienaga
LEE-31-10-B
LEE-31-10-B

分解
アボダイゼーション
スキャンスピード
更新日時

4 cm-1
Cosine
Auto (2 mm/sec)
2004/10/01 1:30

No.	cm-1	%R	No.	cm-1	%R	No.	cm-1	%R	No.	cm-1	%R	No.	cm-1	%R
1	3742.19	98.229	2	3732.55	97.3943	3	3721.94	98.3911	4	3709.41	98.4517	5	3687.23	98.135
6	3666.98	98.7066	7	3646.73	97.4797	8	3626.48	97.9297	9	3616.84	98.6237	10	3585.02	98.6211
11	2942.84	94.7016	12	2837.74	96.0624	13	2355.62	97.5114	14	1789.62	98.3428	15	1769.37	98.3521
16	1730.8	98.9989	17	1714.41	98.638	18	1696.09	97.7564	19	1681.62	96.6697	20	1668.12	96.6576
21	1644.02	96.0841	22	1595.81	86.2373	23	1556.27	94.8998	24	1538.92	95.3279	25	1516.74	95.9305
26	1505.17	94.7842	27	1471.42	89.7869	28	1455.03	90.5602	29	1385.6	93.699	30	1336.43	95.1636
31	1244.83	96.0847	32	1204.33	89.2733	33	1148.4	87.1444	34	1061.62	91.117	35	1026.91	93.4488
36	946.877	96.2779	37	821.527	92.6872	38	680.749	93.8414	39	610.36	96.9333			

[Mass Spectrum]

Data : NISHIZAWA-EI.18-JUN-04.003 Date : 18-Jun-2004 10:34

Sample: LEE-81-12-B

Note : MStation

Inlet : Direct

Ion Mode : EI+

Spectrum Type : Normal Ion [MF-Linear]

RT : 1.27 min Scan# : 39

BP : m/z 154.0000 Int. : 1599.98

Output m/z range : 35.0000 to 504.8961

Cut Level : 0.00 %

17117356

13

[Elemental Composition]

Page: 1

Data : NISHIZAWA-EIHR.01-JUL-04.001 Date : 01-Jul-2004 09:00

Sample: IEE-58-1-A

Note : MStation

Inlet : Direct

RT : 0.87 min

Elements : C 100/0, H 150/0, O 20/0, S 1/0

Mass Tolerance : 5mmu

Unsaturation (U.S.) : 0.0 - 30.0

Ion Mode : EI+

Scan#: 21

Observed m/z	Int%	Err [ppm / mmu]	U.S.	Composition
274.1570	57.9	+0.2 / -12.1	7.0	C 17 H 22 O 3
			3.0	C 14 H 26 O 3 S

LEE-60-1-C

expl s2pul

```

SAMPLES          DEC. & VT
date   Jul 1 2004   dfrq   599.963
solvent   CDCl3   dn      H1
file   /export/home/~ dpwr   45
vnmr1/vnmrsys/data- dof      0
/nishitawa/lee601c- dn      nnn
          _1h.fid   dnm      c
ACQUISITION      dmf      12500
sfrq   599.963   dpcq
to      H1   dres      1.0
at      3.641   homo    n
ap      65536   temp    25.0
aw      9000.9
fb      5000   wtfile
bs      4   proc      ft
tpwr     57   fn      65536
pw      10.3   math     f
dl      1.359
tof     -600.0   werr
nt      32   wexp
ct      32   wbs
alock   n   vnt
gain    43   wlt

FLAGS
il      n
in      n
dp      Y
hs      nn

DISPLAY
sp      453.0
vp      3599.6
vs      250
sc      0
wc      400
hnmn     9.00
is      1337.76
rf1     749.6
rfp      0
th      7
ins     100.000
om      ph
  
```

14

LEE-60-1-C

exp2 s2pul

SAMPLE		DESC. & VT	
date	Jul 1 2004	dirq	599.962
solvent	CDCl3	dn	H1
file	/export/home/-	dpr	45
vnmr1/vnmrsvs/data-	dof	-1000.0	
/nishizawa/lee601c-	dm	y	
	_13c.fid	dm	w
ACQUISITION		dmf	12500
sfrq	150.877	dseq	garpl
tn	C13	drea	1.0
at	0.905	homo	n
np	65536	temp	25.0
sw	36199.1	PROCESSING	
fb	20000	lb	1.50
bs	16	wtFile	
tpwr	54	proc	ft
pw	7.0	fa	65536
d1	1.200	math	f
tof	3384.3		
nt	512	weir	
ot	512	wexp	wft
alock	n	wbs	wft
gain	56	wnt	
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-0.2		
wp	13577.3		
vs	90		
sc	0		
wc	320		
huan	42.43		
is	500.00		
rfl	12527.8		
rtg	11616.2		
th	3		
ins	100.000		
nm	pb

14

LEE-60-1-C

Pulse Sequence: dept

Solvent: CDCl₃

Temp. 25.0 C / 298.1 K

File: lee601c_dept

INOVA-500 "varian"

Relax. delay 2.000 sec

Pulse 90.3 degrees

Acq. time 0.905 sec

Width 36199.1 Hz

54 repetitions

OBSERVE C13, 150.8587729 MHz

DECOUPLE H1, 599.9606994 MHz

Power 45 dB

on during acquisition

off during delay

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.5 Hz

FT size 65536

Total time 12 min, 53 sec

14

CH3 carbons

CH2 carbons

CH carbons

all protonated carbons

[Mass Spectrum]

Data : NISHIZAWA-EI.07-MAY-04.001 Date : 07-May-2004 19:04

Sample: LEE-60-1-C

Note : MStation

Inlet : Direct

Ion Mode : EI+

Spectrum Type : Normal Ion [MF-Linear]

RT : 1.30 min Scan# : 40

BP : m/z 206.0000 Int. : 1599.98

Output m/z range : 35.0000 to 504.8961

Cut Level : 0.00 %

17068734

14

[Elemental Composition]

Page: 1

Data : NISHIZAWA-EIHR.11-JUN-04.002 Date : 11-Jun-2004 11:14
 Sample: LEE-60-1-C
 Note : MStation
 Inlet : Direct
 RT : 1.00 min
 Elements : C 100/0, H 150/0, O 20/0, Br 2/0
 Mass Tolerance : 5mmu
 Unsaturation (U.S.) : 0.0 - 30.0

Ion Mode : EI+
 Scan#: 24

Observed m/z	Int%	Err [ppm / mmu]	U.S. Composition
274.1562	32.9	-2.6 / -0.7	7.0 C 17 H 22 O 3

14

LEE-60-1-D

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: LEE-60-1-D042804_20_57

INOVA-600 "NMR"

Relax. delay 1.334 sec

Pulse 45.2 degrees

Acq. time 2.666 sec

Width 3000.3 Hz

32 repetitions

OBSERVE H1, 199.9482359 MHz

DATA PROCESSING

FT size 16384

Total time 2 min, 8 sec

15

LEE-60-1-D

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: LEE-60-1-D-13C043004_18_06

INOVA-600 "NMR"

Pulse 60.0 degrees

Acq. time 1.498 sec

Width 12500.0 Hz

1200 repetitions

OBSERVE C13, 50.2614223 MHz

DECOUPLE H1, 199.8873538 MHz

Power 30 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 30 min, 7 sec

15

積算回数
ゼロフィリング
ゲイン
測定日時
測定者
ファイル名
サンプル名
コメント

54
ON
Auto (4)
2004/10/01 17:06
ienaga
Memory#2
LEE-60-1-D

分解
アポダイゼーション
スキャンスピード
更新日時

4 cm-1
Cosine
Auto (2 mm/sec)
2004/10/01 17:09

No.	cm-1	%R	No.	cm-1	%R	No.	cm-1	%R	No.	cm-1	%R
1	3449.06	95.9122	2	2929.34	63.6555	3	2838.7	75.1858	4	1670.05	78.5393
6	1584.24	26.1999	7	1486.85	61.066	8	1461.78	47.9212	9	1436.71	51.675
11	1389.46	69.9037	12	1340.28	77.3131	13	1301.72	75.7154	14	1277.61	70.1414
16	1201.43	38.712	17	1176.36	58.6709	18	1154.19	27.0213	19	1105.01	31.6897
21	1053.91	56.6757	22	941.092	81.2507	23	813.813	68.07	24	744.388	93.6918
26	589.147	93.9887	27	471.51	95.9148	28	441.619	95.3357	29	417.513	96.1594
									5	1614.13	25.8956
									10	1420.32	49.6877
									15	1216.86	52.1109
									20	1078.98	65.181
									25	633.501	91.055
									30	408.835	96.5975

[Mass Spectrum]

Data : NISHIZAWA-EI.07-MAY-04.002 Date : 07-May-2004 19:08

Sample: LEE-60-1-D

Note : MStation

Inlet : Direct

Ion Mode : EI+

Spectrum Type : Normal Ion [MF-Linear]

RT : 1.37 min Scan# : 42

BP : m/z 259.0000 Int. : 1599.98

Output m/z range : 35.0000 to 501.4911

Cut Level : 0.00 %

15

[Elemental Composition]

Data : NISHIZAWA-EIHR.11-JUN-04.001 Date : 11-Jun-2004 11:02

Sample: LEE-60-1-D

Note : MStation

Inlet : Direct

RT : 0.83 min

Elements : C 100/0, H 150/0, O 20/0, Br 2/0

Mass Tolerance : 5mmu

Unsaturation (U.S.) : 0.0 ~ 30.0

Ion Mode : EI+

Scan#: 20

Br 2/0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
274.1575	41.9	+2.2 / +0.6	7.0	C 17 H 22 O 3

15

LER-60-1-A

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: LER-60-1-A042804_20_23

INOVA-600 "NMR"

Relax. delay 1.334 sec

Pulse 45.2 degrees

Acq. time 2.666 sec

Width 3000.3 Hz

32 repetitions

OBSERVE H1, 199.9482352 MHz

DATA PROCESSING

FT size 16384

Total time 2 min, 8 sec

16

LEE-60-1-A

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: LEE-60-1-A-13C050104_15_41

INOVA-600 "NMR"

Relax. delay 1.000 sec

Pulse 31.2 degrees

Acq. time 2.134 sec

Width 14992.5 Hz

1626 repetitions

OBSERVE C13, 50.3073364 MHz

DECOUPLE H1, 200.0697598 MHz

Power 40 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 2 hr, 10 min, 58 sec

16

[Mass Spectrum]

Data : NISHIZAWA-CI.11-JUN-04.001 Date : 11-Jun-2004 15:09

Sample: LEE-60-1-A(iso-Bu)

Note : MStation

Inlet : Direct

Ion Mode : CI+

Spectrum Type : Normal Ion [MF-Linear]

RT : 1.12 min Scan# : 42-k(68)[k=1.0]

BP : m/z 275.0000 Int. : 397.82

Output m/z range : 36.1350 to 501.4911

Cut Level : 0.00 %

16

[Mass Spectrum]

Data : NISHIZAWA-EI.07-MAY-04.003 Date : 07-May-2004 19:13

Sample: LEE-60-1-A

Note : MStation

Inlet : Direct

Ion Mode : EI+

Spectrum Type : Normal Ion [MF-Linear]

RT : 0.30 min Scan# : 10

BP : m/z 168.0000 Int. : 1373.88

Output m/z range : 35.0000 to 503.7611

Cut Level : 0.00 %

16