STRUCTURAL CHEMISTRY

Volume 76 (2020)

Supporting information for article:

Dibenzoate esters of *cis*-tetralin-2,3-diol as analogs of (–)-epigallocatechin gallate: synthesis and crystal structure of anti-cancer drug candidates

Ryan Noboru Rutherford, Shinji Ura, Tak-Hang Chan, Kozo Fukumoto, Takanori Nishioka and Andrea Renzetti

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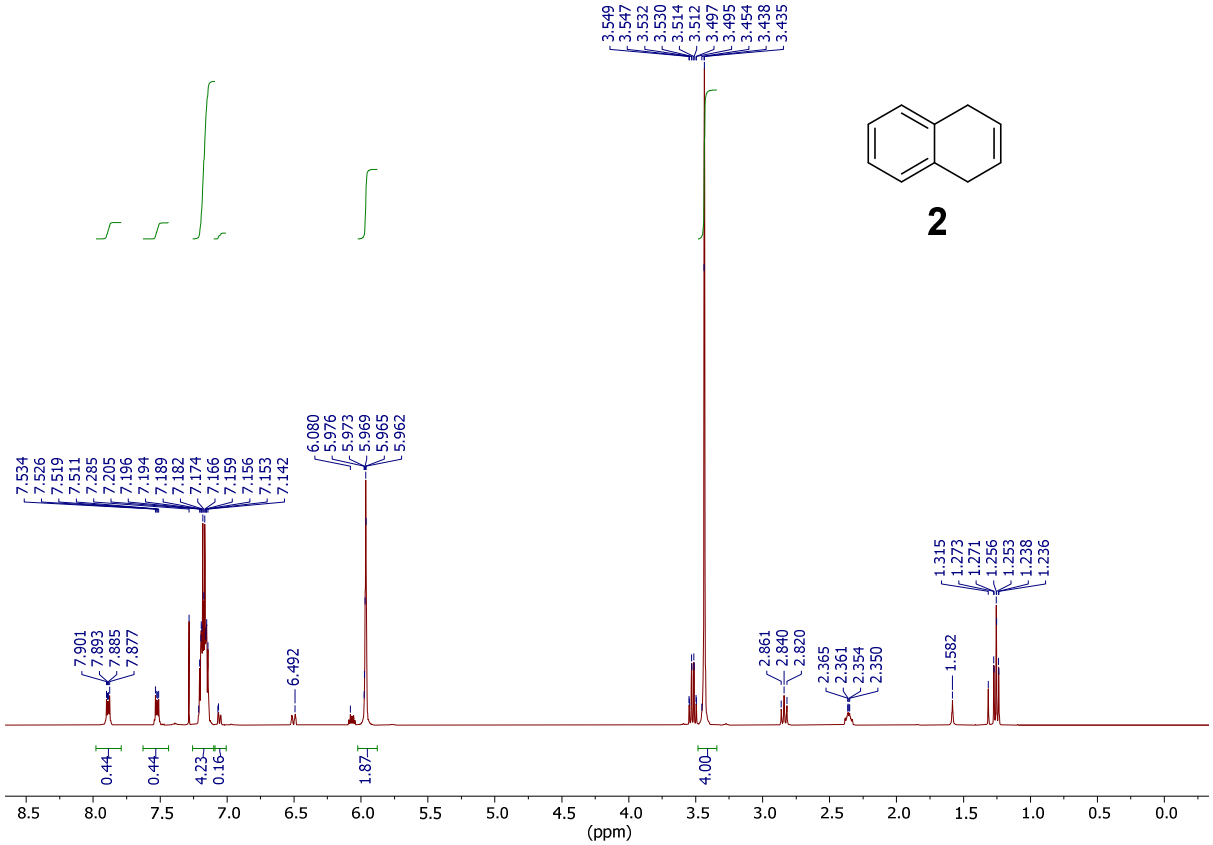
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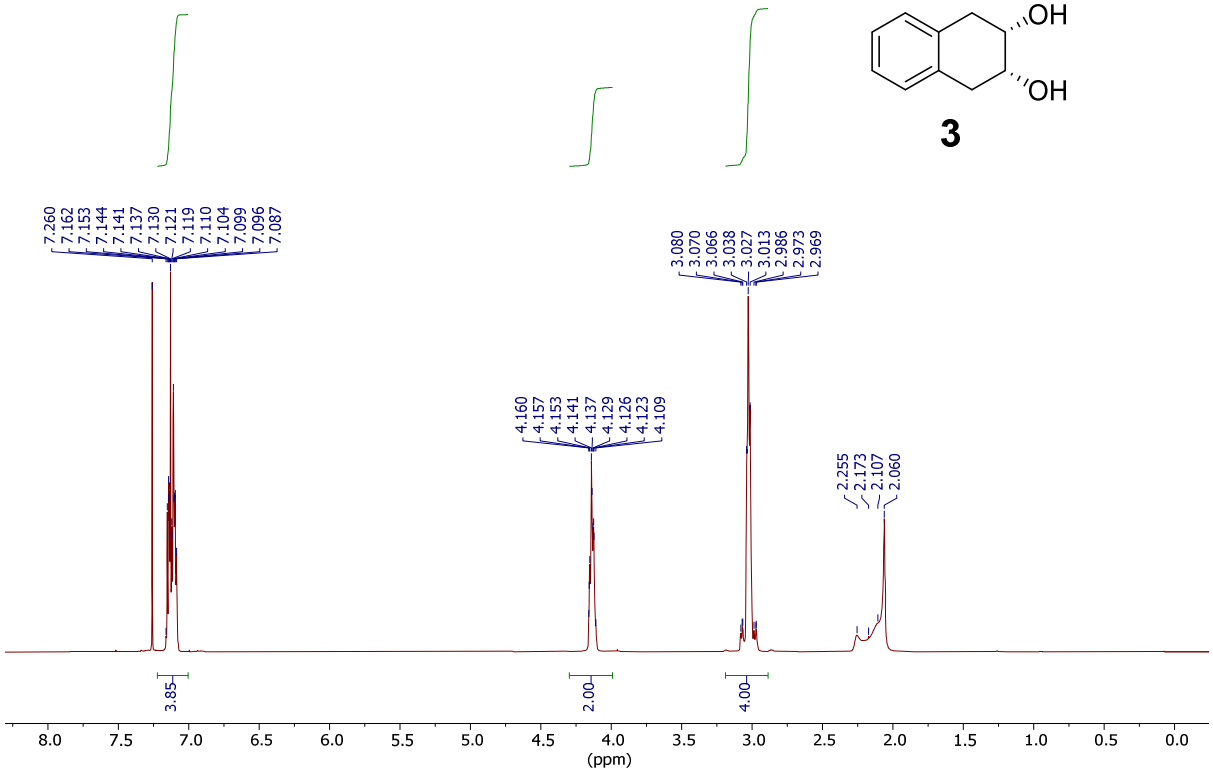
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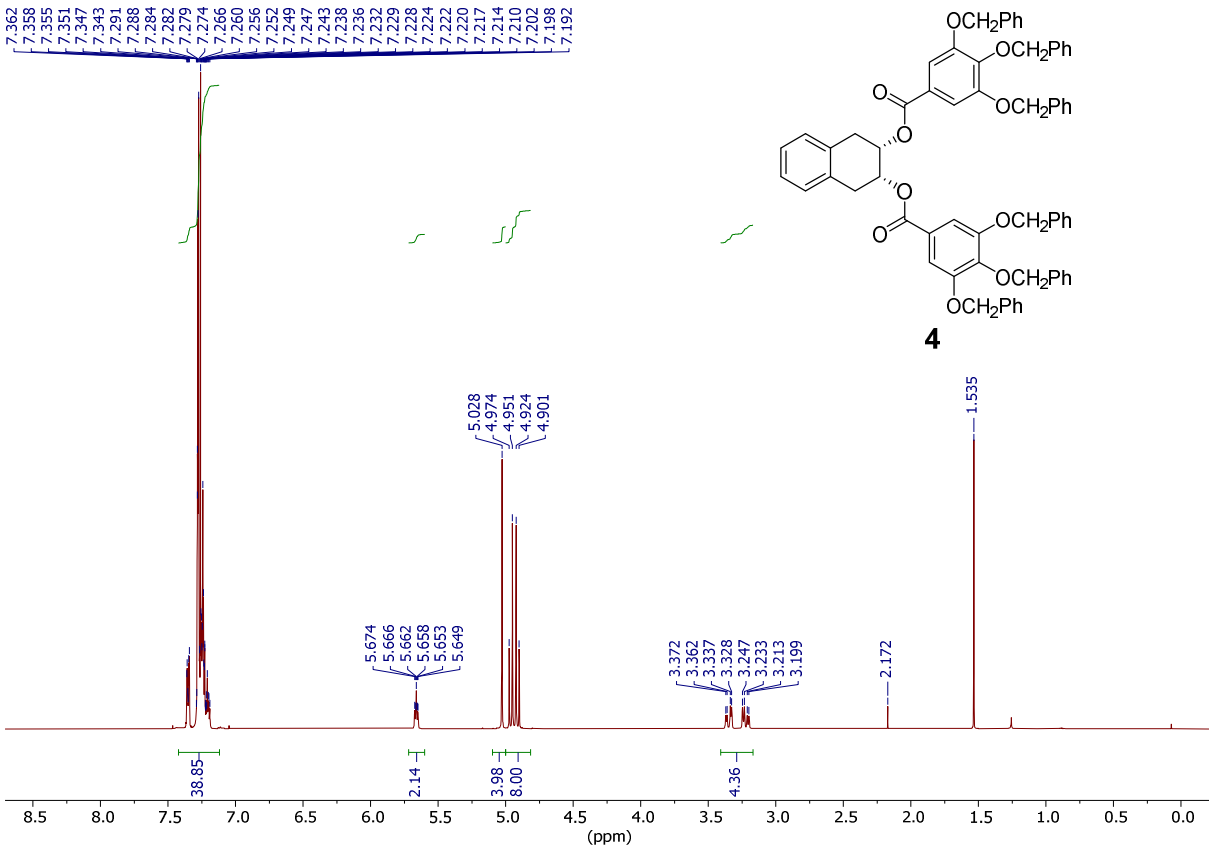
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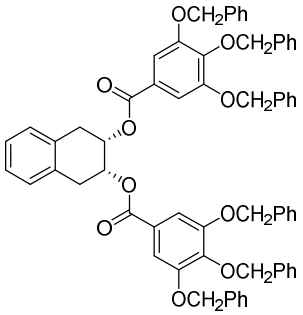
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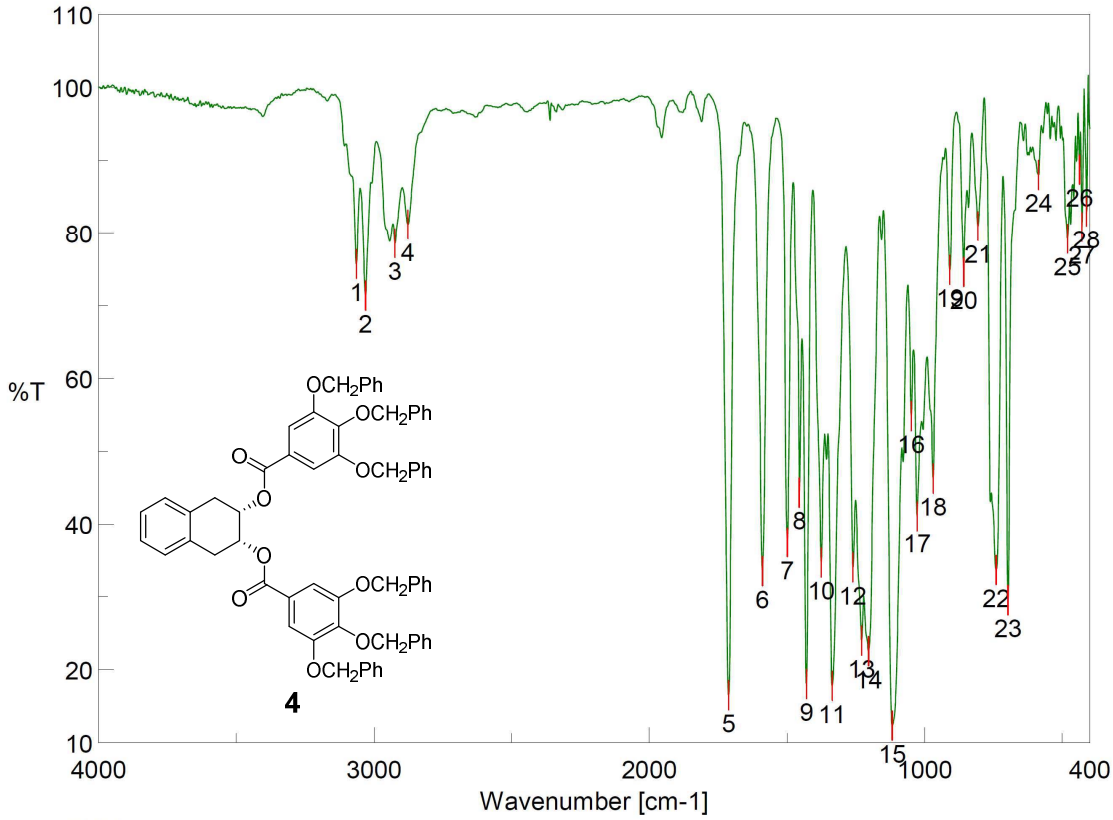
IR spectrum – AR-1



General information

Sample name	AR-1
Comments	Compound **4**
Measurer	Andrea Renzetti
Affiliation	University of the Ryukyus

Data information

Date and time	2019/09/03 11:27

Data type

Horizontal axis	Wavenumber (cm^{-1})
Vertical axis	%T
Start	399.193 cm^{-1}
End	4000.6 cm^{-1}
Data interval	0.964233 cm^{-1}
Number of data	3736

Measurements information

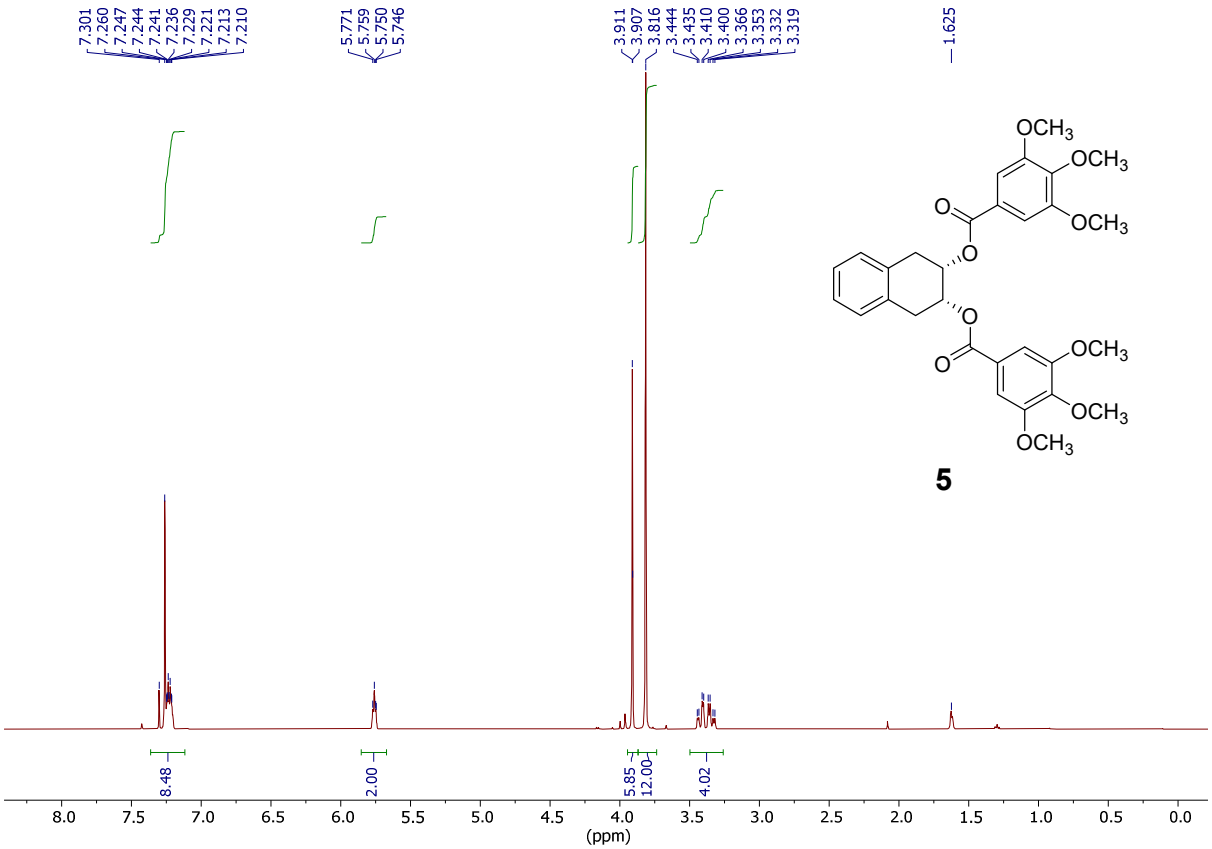
Model name	FT/IR-6100typeA
Serial number	A042161020
Date and time	2019/09/03 11:17
Source	Standard source
Detector	TGS
Integration number	16
Resolution	4 cm^{-1}
Zero filling	On
Apodization	Cosine
Gain	Auto (16)
Aperture	Auto (7.1 mm)
Scan speed	Auto (2 mm/sec)
Filter	Auto (10000 Hz)

Peak detection

No.	$\tilde{\nu}$ (cm^{-1})	%T	No.	$\tilde{\nu}$ (cm^{-1})	%T
1	3063.37	75.6359	2	3030.59	71.2904
3	2923.56	78.525	4	2876.31	81.1464
5	1711.51	16.4878	6	1589.06	33.4091
7	1498.42	37.4207	8	1455.03	44.2878
9	1428.99	18.0347	10	1375	34.6097
11	1335.46	17.76	12	1260.25	33.9445
13	1228.43	23.9432	14	1203.36	22.5075

Peak detection

No.	$\tilde{\nu}$ (cm^{-1})	%T	No.	$\tilde{\nu}$ (cm^{-1})	%T
15	1117.55	12.3208	16	1048.12	54.7741
17	1027.87	41.0743	18	969.055	46.2331
19	908.308	74.8272	20	858.168	74.538
21	806.099	80.8786	22	740.531	33.5755
23	697.141	29.4706	24	586.254	87.9624
25	481.153	79.1519	26	437.762	88.7063
27	428.12	80.7806	28	411.728	82.9518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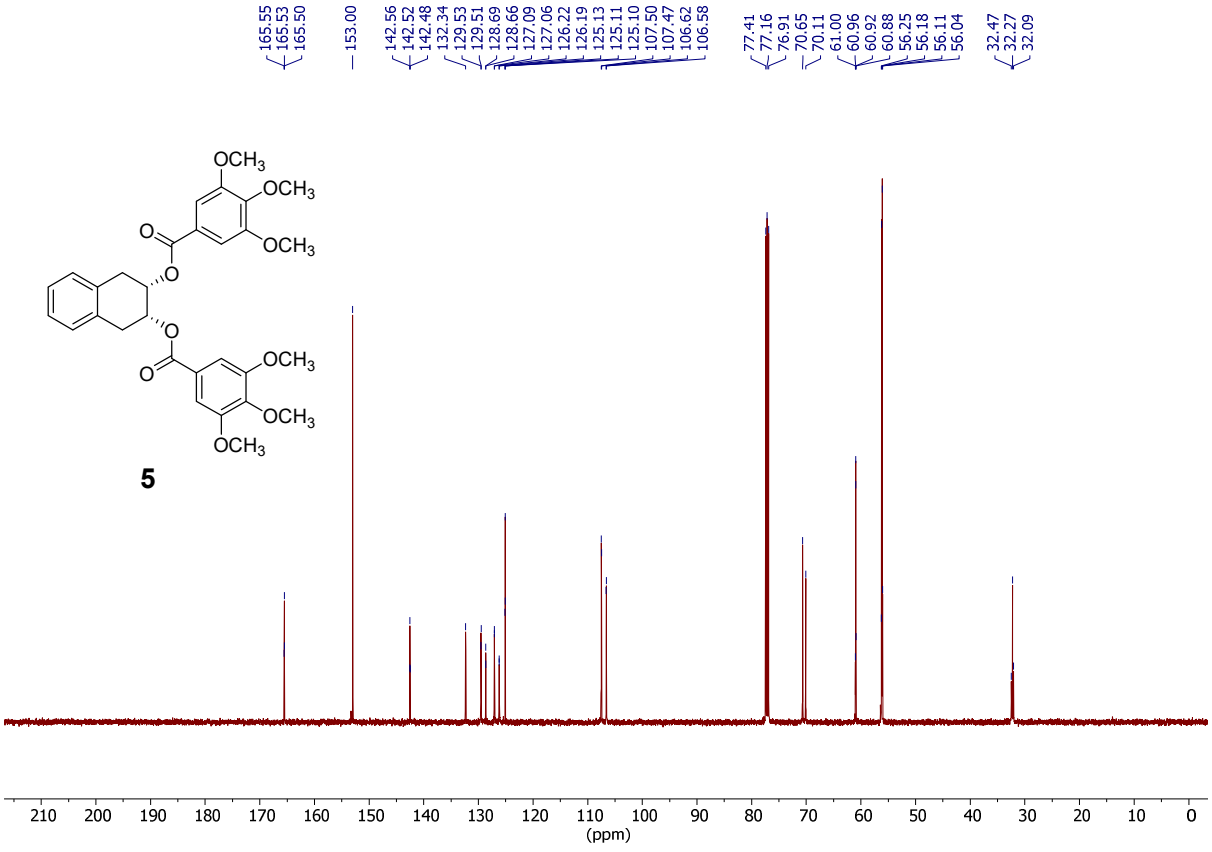
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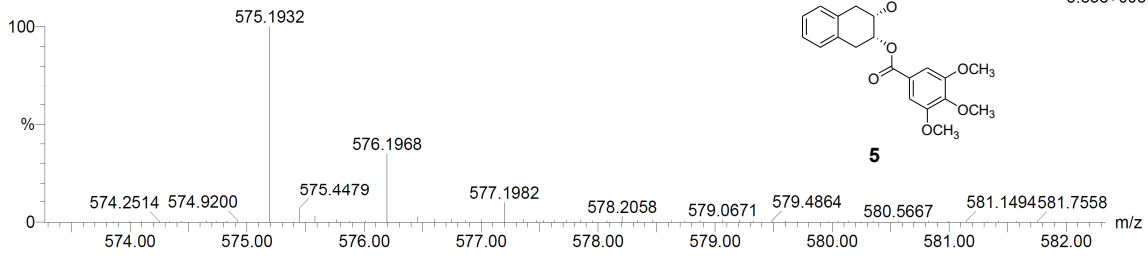
Tolerance = 10.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
2162 formula(e) evaluated with 49 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 1-40 H: 1-60 N: 0-20 O: 0-10 Na: 0-1

AR-30-A
190822_Fukumoto14 50 (0.291)



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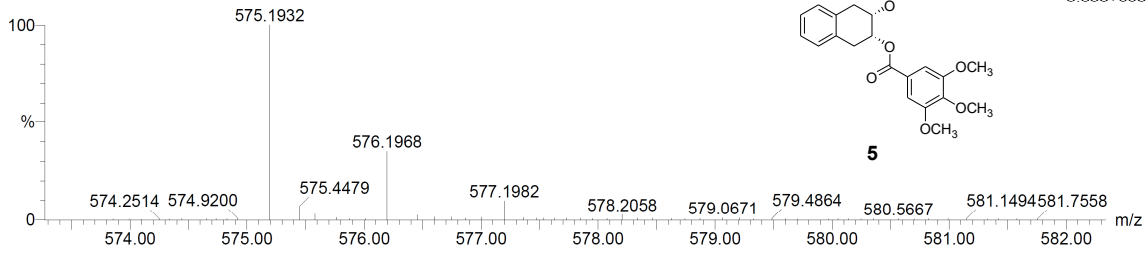
3.83e+003

Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
Minimum:	80.00				-1.5			
Maximum:	100.00		10.0	10.0	50.0			
575.1932	100.00	575.1931	0.1	0.2	22.5	114.5	2.2	C33 H27 N4 O6
		575.1936	-0.4	-0.7	15.5	119.3	7.0	C18 H23 N16 O7
		575.1938	-0.6	-1.0	11.5	118.5	6.2	C20 H28 N10 O9 Na
		575.1925	0.7	1.2	17.5	120.1	7.9	C17 H20 N20 O3 Na
		575.1920	1.2	2.1	24.5	115.6	3.3	C32 H24 N8 O2 Na
		575.1944	-1.2	-2.1	27.5	116.0	3.7	C34 H23 N8 O2
		575.1917	1.5	2.6	17.5	113.1	0.8	C32 H31 O10
		575.1947	-1.5	-2.6	23.5	115.9	3.6	C36 H28 N2 O4 Na
		575.1917	1.5	2.6	28.5	116.1	3.8	C30 H19 N14
		575.1949	-1.7	-3.0	20.5	119.2	6.9	C19 H19 N20 O3
		575.1912	2.0	3.5	12.5	120.4	8.1	C16 H24 N16 O7 Na
		575.1952	-2.0	-3.5	16.5	118.5	6.2	C21 H24 N14 O5 Na
		575.1907	2.5	4.3	19.5	114.8	2.5	C31 H28 N4 O6 Na
		575.1904	2.8	4.9	23.5	115.7	3.4	C29 H23 N10 O4
		575.1960	-2.8	-4.9	28.5	117.3	5.0	C37 H24 N6 Na
		575.1962	-3.0	-5.2	14.5	118.0	5.7	C22 H27 N10 O9
		575.1965	-3.3	-5.7	21.5	118.6	6.4	C22 H20 N18 O Na
		575.1895	3.7	6.4	11.5	121.8	9.5	C13 H23 N18 O9
		575.1893	3.9	6.8	14.5	114.7	2.5	C30 H32 O10 Na
		575.1971	-3.9	-6.8	26.5	117.4	5.1	C38 H27 N2 O4
		575.1893	3.9	6.8	25.5	117.2	4.9	C28 H20 N14 Na
		575.1890	4.2	7.3	18.5	115.7	3.5	C28 H27 N6 O8
		575.1976	-4.4	-7.6	19.5	118.1	5.8	C23 H23 N14 O5
		575.1979	-4.7	-8.2	15.5	117.6	5.3	C25 H28 N8 O7 Na
		575.1984	-5.2	-9.0	31.5	118.8	6.5	C39 H23 N6
		575.1984	-5.2	-9.0	8.5	123.0	10.7	C10 H24 N20 O8 Na
		575.1880	5.2	9.0	20.5	117.4	5.1	C27 H24 N10

Tolerance = 10.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
2162 formula(e) evaluated with 49 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 1-40 H: 1-60 N: 0-20 O: 0-10 Na: 0-1
AR-30-A
190822_Fukumoto14 50 (0.291)



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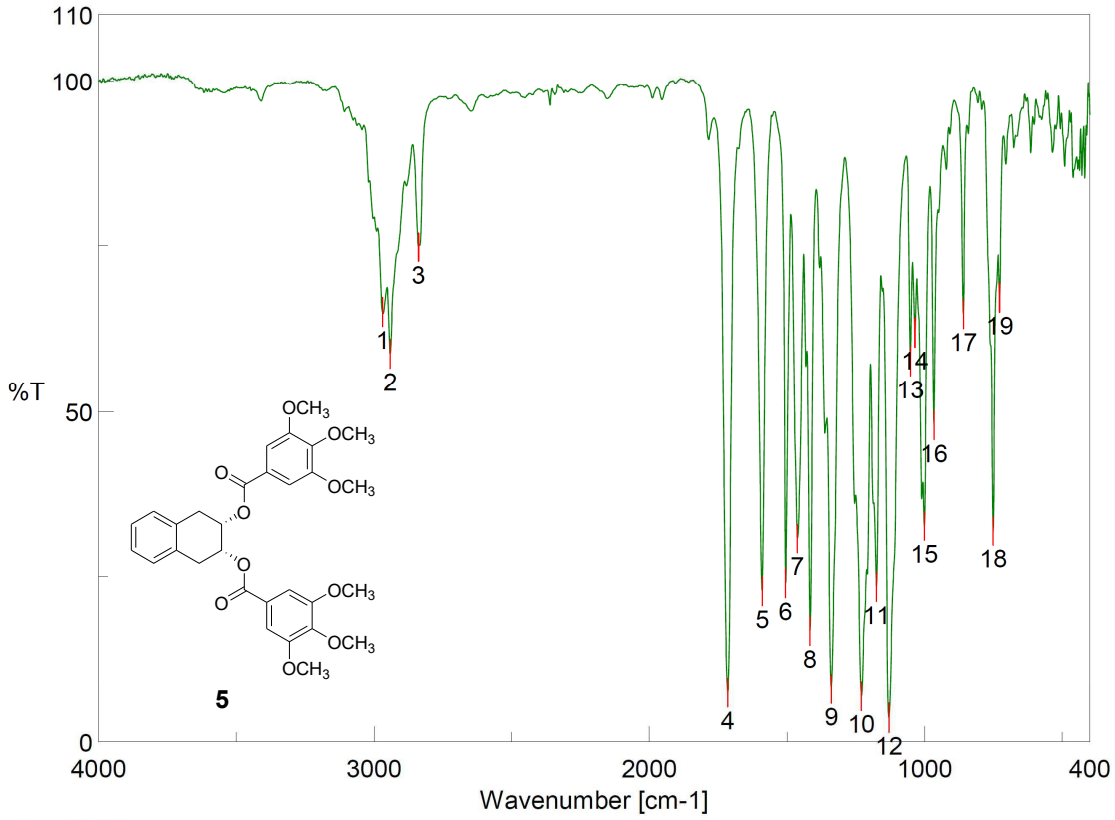
1: TOF MS ES+
3.83e+003

Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
Minimum:		80.00			-1.5			
Maximum:		100.00	10.0	10.0	50.0			
		575.1877	5.5	9.6	24.5	118.3	6.0	C25 H19 N16 O4 Na
		575.1989	-5.7	-9.9	24.5	118.5	6.2	C24 H19 N18 O2
		575.1992	-6.0	-10.4	20.5	117.9	5.7	C26 H24 N12 O
		575.1872	6.0	10.4	31.5	119.2	7.0	C40 H23 N4 O3 Na
		575.1871	6.1	10.6	8.5	123.3	11.0	C11 H24 N18 O
		575.1866	6.6	11.5	15.5	118.1	5.8	C26 H28 N6 O9 Na
		575.1864	6.8	11.8	19.5	118.9	6.6	C24 H23 N12 O8 Na
		575.2003	-7.1	-12.3	18.5	117.4	5.1	C27 H27 N8 O6
		575.1858	7.4	12.9	26.5	119.0	6.7	C39 H27 O5 O7
		575.2006	-7.4	-12.9	14.5	116.9	4.6	C29 H32 N2 O9 Na
		575.2008	-7.6	-13.2	11.5	122.9	10.6	C12 H23 N20 O8
		575.2011	-7.9	-13.7	7.5	122.5	10.2	C14 H28 N14 O10 Na
		575.1853	7.9	13.7	21.5	120.2	7.9	C23 H20 N16 O2 Na
		575.1850	8.2	14.3	14.5	119.8	7.5	C23 H27 N8 O10
		575.1848	8.4	14.6	28.5	119.6	7.3	C38 H24 N4 O Na
		575.2016	-8.4	-14.6	23.5	118.1	5.8	C28 H23 N12 O3
		575.2019	-8.7	-15.1	19.5	117.6	5.4	C30 H28 N6 O5 Na
		575.2024	-9.2	-16.0	12.5	122.7	10.4	C15 H24 N18 O6 Na
		575.1839	9.3	16.2	16.5	120.9	8.6	C22 H24 N12 O6 Na
		575.1837	9.5	16.5	20.5	121.6	9.3	C20 H19 N18 O4
		575.2030	-9.8	-17.0	17.5	116.7	4.4	C31 H31 N2 O9
		575.1834	9.8	17.0	23.5	119.0	6.7	C37 H28 O5 Na

IR spectrum – AR-30-A



General information

Sample name	AR-30-A
Comments	Compound **5**
Measurer	Andrea Renzetti
Affiliation	University of the Ryukyus

Data information

Date and time	2019/09/03 11:42

Data type

Horizontal axis	Wavenumber (cm^{-1})
Vertical axis	%T
Start	399.193 cm^{-1}
End	4000.6 cm^{-1}
Data interval	0.964233 cm^{-1}
Number of data	3736

Measurements information

Model name	FT/IR-6100typeA
Serial number	A042161020
Date and time	2019/09/03 11:41
Source	Standard source
Detector	TGS
Integration number	16
Resolution	4 cm^{-1}
Zero filling	On
Apodization	Cosine
Gain	Auto (16)
Aperture	Auto (7.1 mm)
Scan speed	Auto (2 mm/sec)
Filter	Auto (10000 Hz)

Peak detection

No.	$\tilde{\nu}$ (cm^{-1})	%T	No.	$\tilde{\nu}$ (cm^{-1})	%T
1	2968.87	64.9939	2	2940.91	58.6731
3	2837.74	74.8071	4	1715.37	7.46308
5	1590.02	22.7817	6	1505.17	23.9678
7	1461.78	30.7328	8	1416.46	16.8059
9	1339.32	8.01426	10	1229.4	6.9283
11	1174.44	23.4186	12	1130.08	3.6124
13	1051.98	57.5464	14	1034.62	61.8778

Peak detection

No.	$\tilde{\nu}$ (cm^{-1})	%T	No.	$\tilde{\nu}$ (cm^{-1})	%T
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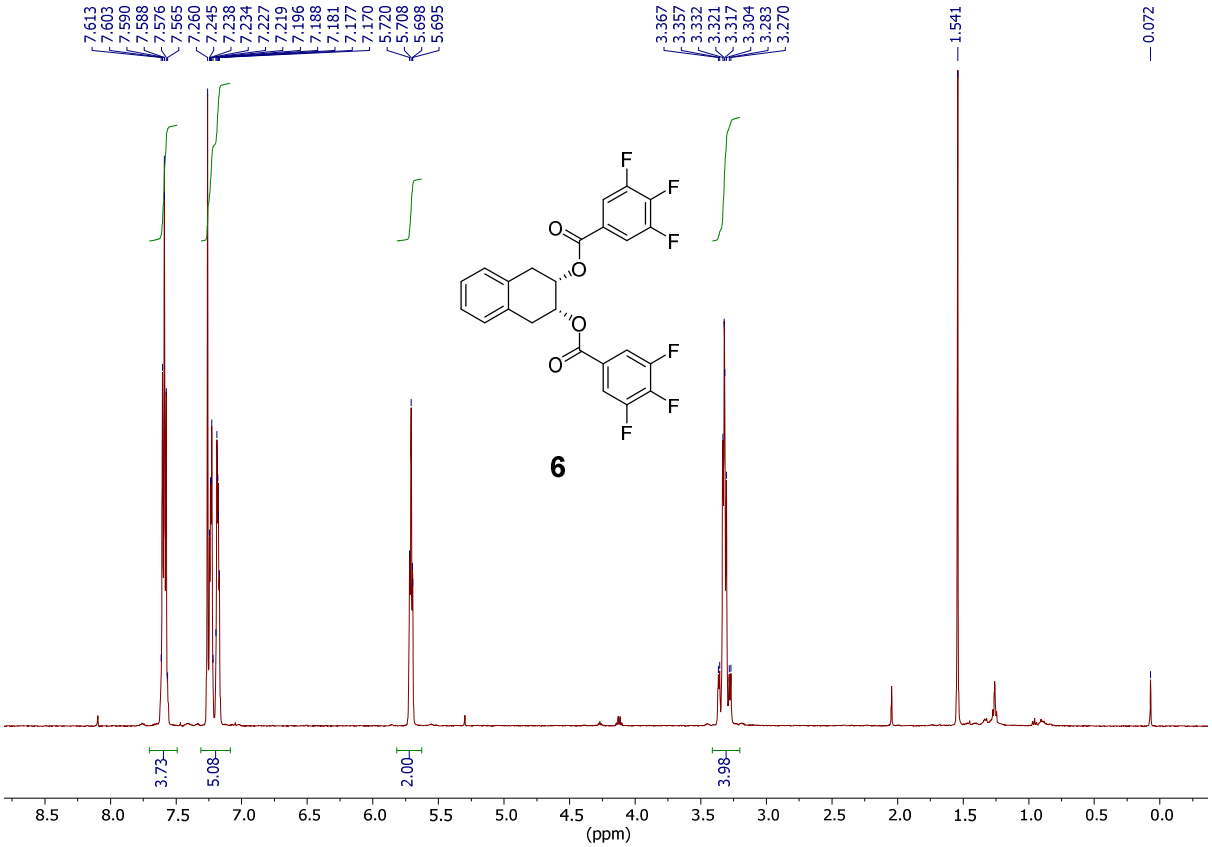
17	859.132	64.6765	18	751.138	31.8623
19	727.996	67.1578			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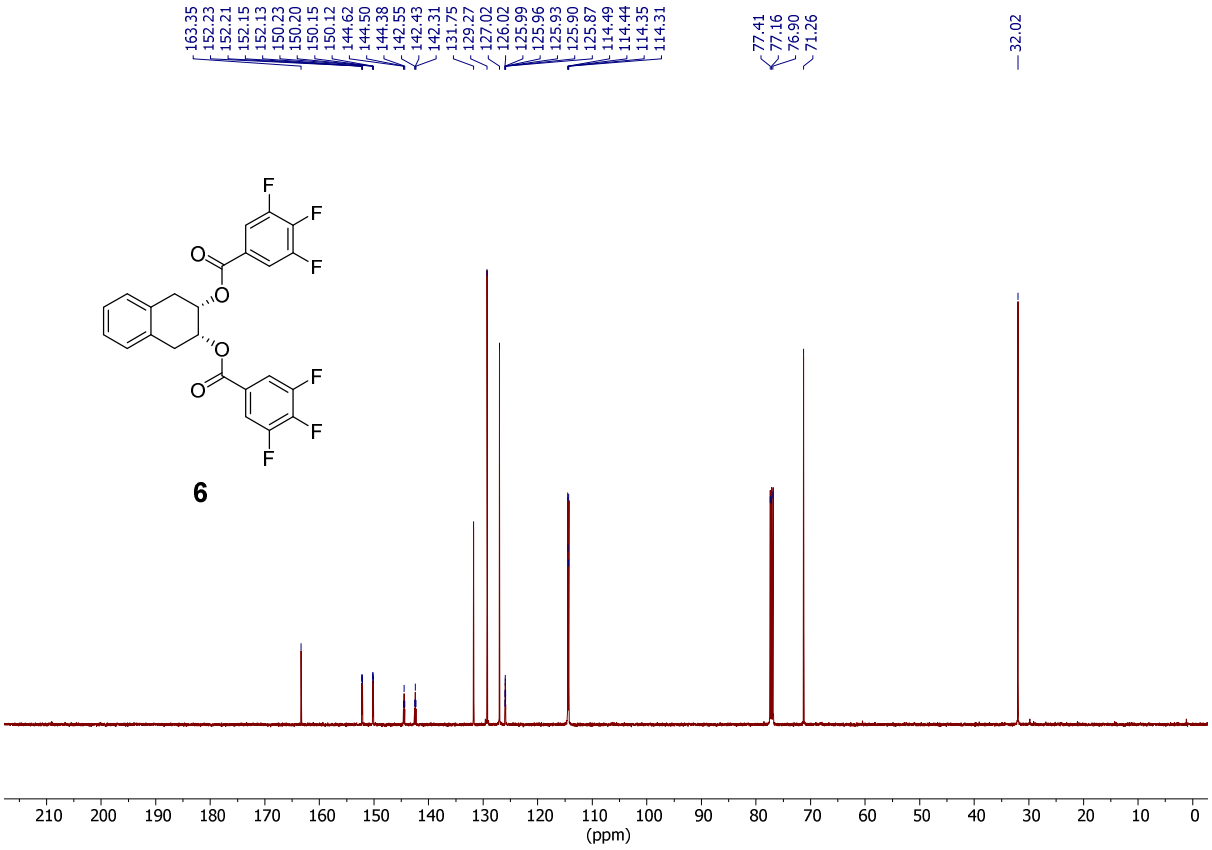
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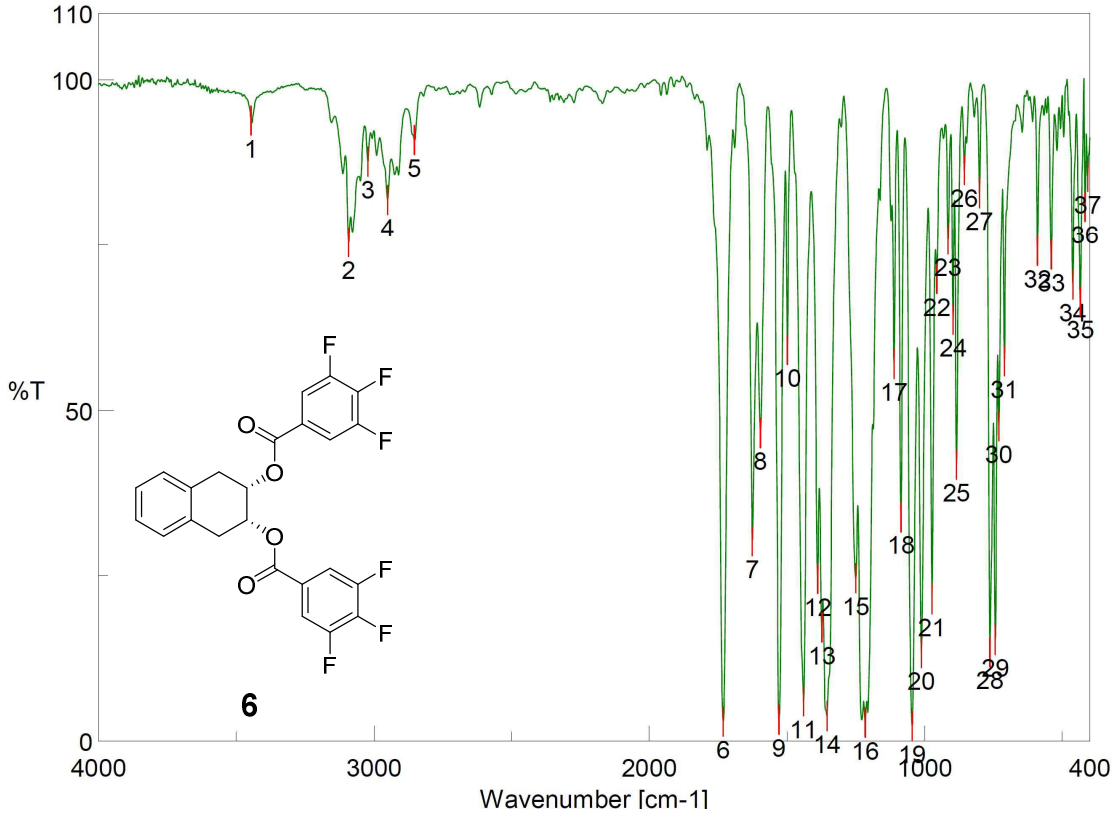
IR spectrum – AR-51-A



General information

Sample name	AR-51-A
Comments	Compound **6**
Measurer	Andrea Renzetti
Affiliation	University of the Ryukyus

Data information

Date and time	2019/09/03 12:17

Data type

Horizontal axis	Wavenumber (cm^{-1})
Vertical axis	%T
Start	399.193 cm^{-1}
End	4000.6 cm^{-1}
Data interval	0.964233 cm^{-1}
Number of data	3736

Measurements information

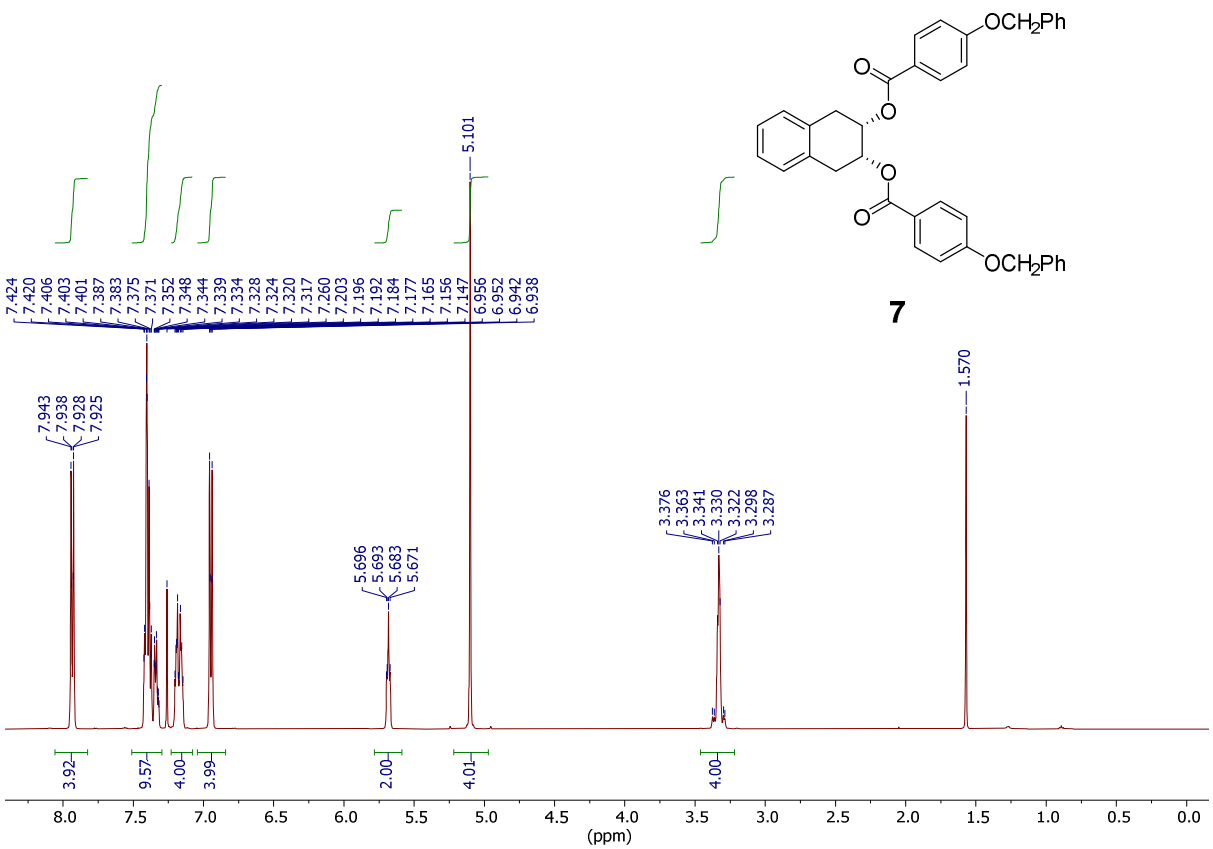
Model name	FT/IR-6100typeA
Serial number	A042161020
Date and time	2019/09/03 12:15
Source	Standard source
Detector	TGS
Integration number	16
Resolution	4 cm^{-1}
Zero filling	On
Apodization	Cosine
Gain	Auto (16)
Aperture	Auto (7.1 mm)
Scan speed	Auto (2 mm/sec)
Filter	Auto (10000 Hz)

Peak detection

No.	$\tilde{\nu}$ (cm^{-1})	%T	No.	$\tilde{\nu}$ (cm^{-1})	%T
1	3446.17	93.8185	2	3092.3	75.3546
3	3021.91	87.625	4	2950.55	81.8391
5	2853.17	90.8851	6	1731.76	2.9149
7	1625.7	30.1227	8	1595.81	46.5586
9	1529.27	3.19284	10	1498.42	59.0774
11	1439.6	5.98753	12	1388.5	24.4244
13	1373.07	17.1192	14	1354.75	3.76519

Peak detection

No.	$\tilde{\nu}$ (cm^{-1})	%T	No.	$\tilde{\nu}$ (cm^{-1})	%T
15	1249.65	24.5559	16	1215.9	2.74906
17	1110.8	56.9923	18	1085.73	33.8509
19	1045.23	2.24461	20	1011.48	13.2487
21	972.912	21.3706	22	955.555	69.8162
23	915.058	75.7671	24	896.737	63.6515
25	884.202	41.814	26	855.275	86.3407
27	800.314	82.867	28	762.709	13.3954
29	743.424	15.2528	30	729.925	47.6103
31	710.64	57.4183	32	590.111	74.0651
33	539.971	73.4724	34	461.868	68.9536
35	433.905	66.3997	36	417.513	80.7787
37	407.871	85.3274			


OCH2Ph
O
O
O
O
OCH2Ph
7
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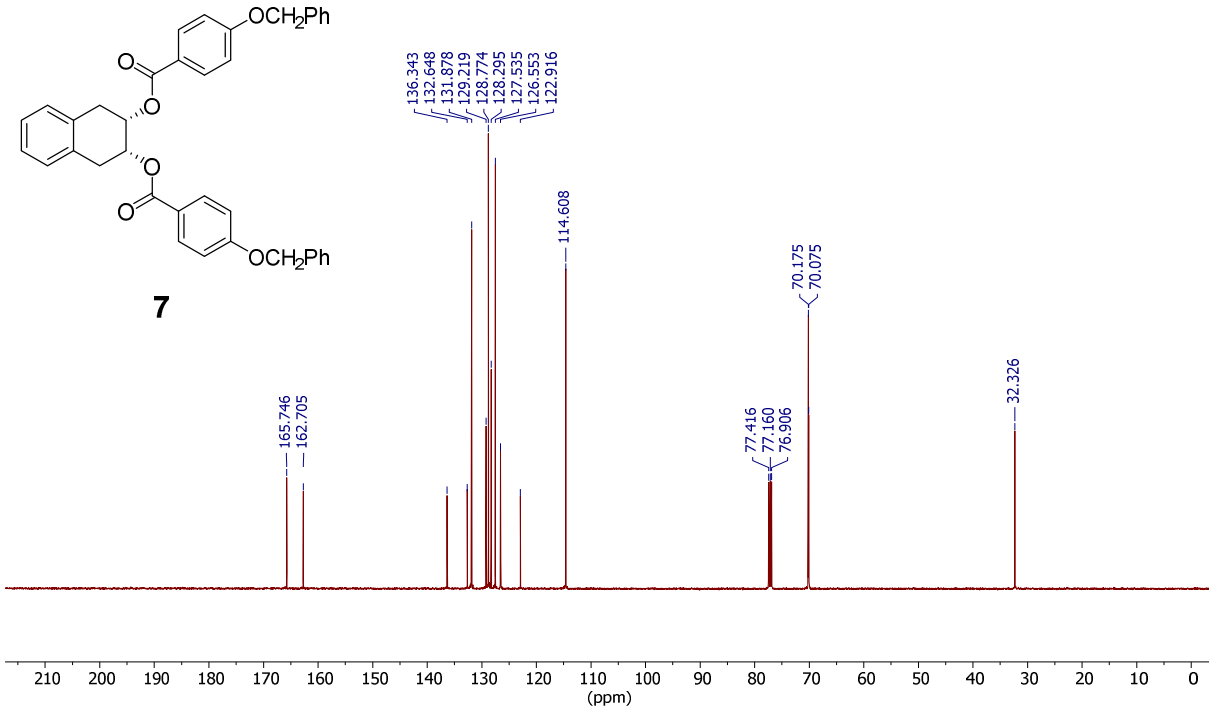
7.420
7.406
7.403
7.401
7.387
7.383
7.375
7.371
7.352
7.348
7.344
7.339
7.334
7.328
7.324
7.320
7.317
7.260
7.203
7.196
7.192
7.184
7.177
7.165
7.156
7.147
6.956
6.952
6.942
6.938
5.101
7.943
7.938
7.928
7.925
5.696
5.693
5.683
5.671
3.376
3.363
3.341
3.330
3.322
3.298
3.287
1.570
3.92
9.57
4.00
3.99
2.00
4.01
4.00
(ppm)


OCH2Ph
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O
O
O
OCH2Ph
7
136.343
132.648
131.878
129.219
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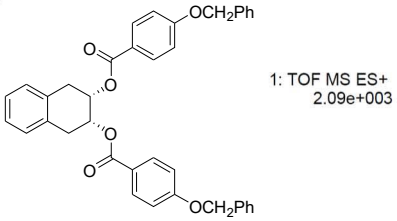
127.535
126.553
122.916
114.608
70.175
70.075
32.326
77.416
77.160
76.906
165.746
162.705
(ppm)

Tolerance = 10.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
60 formula(e) evaluated with 2 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 1-40 H: 1-60 O: 0-10 Na: 0-1

USJ-1-AB-A
190822_Fukumoto18 290 (1.600)

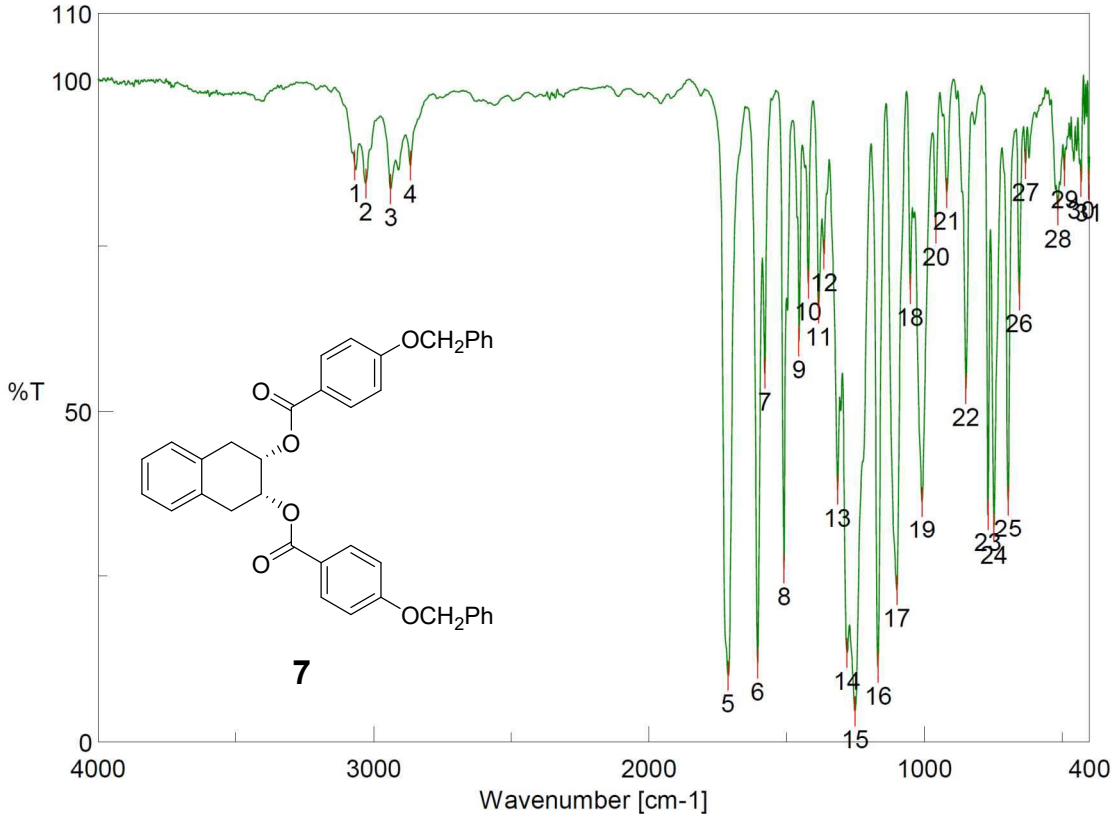


1: TOF MS ES+
2.09e+003

7

Minimum:	80.00				-1.5			
Maximum:	100.00		10.0	10.0	50.0			
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
607.2130	100.00	607.2121	0.9	1.5	25.5	76.1	1.0	C40 H31 O6
		607.2097	3.3	5.4	22.5	75.6	0.5	C38 H32 O6 Na

IR spectrum – USJ-1AA



General information

Sample name	USJ-1AA
Comments	Compound **7**
Measurer	Andrea Renzetti
Affiliation	University of the Ryukyus

Data information

Date and time	2019/09/03 11:54

Data type

Horizontal axis	Wavenumber (cm^{-1})
Vertical axis	%T
Start	399.193 cm^{-1}
End	4000.6 cm^{-1}
Data interval	0.964233 cm^{-1}
Number of data	3736

Measurements information

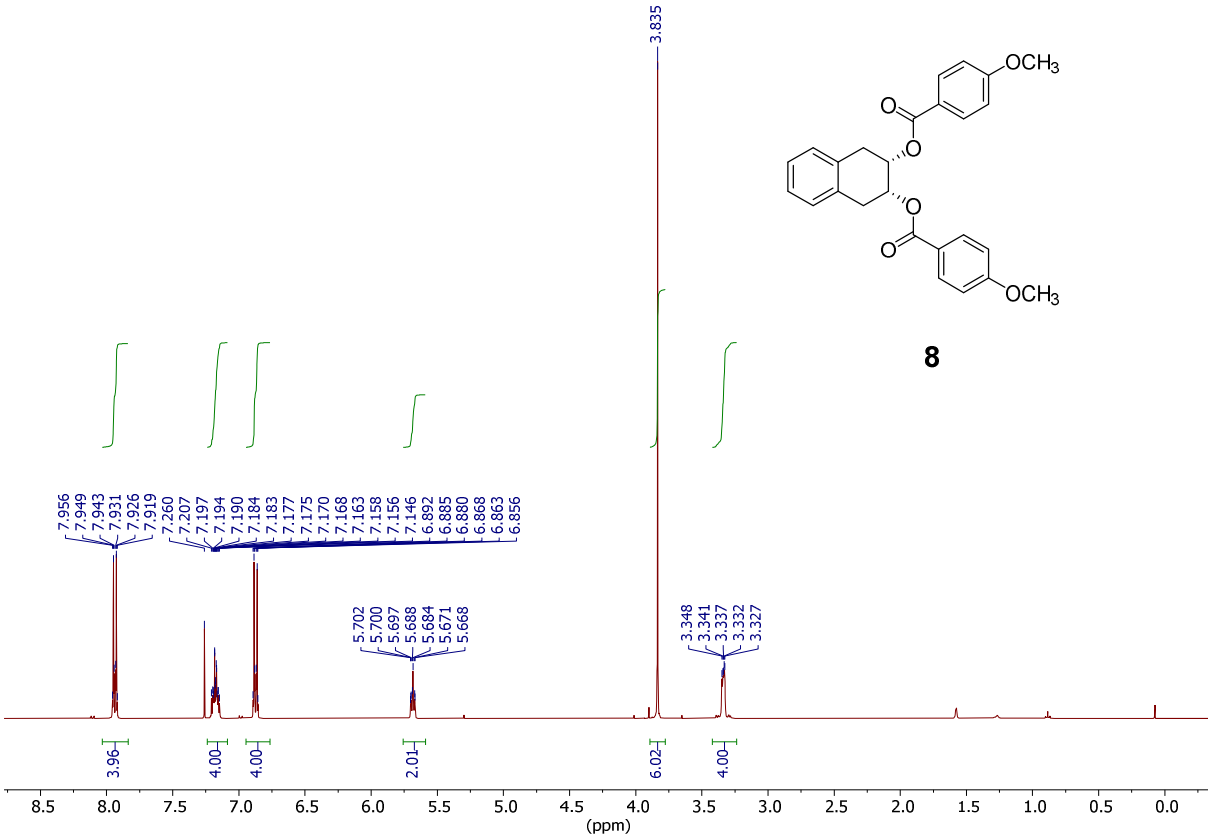
Model name	FT/IR-6100typeA
Serial number	A042161020
Date and time	2019/09/03 11:53
Source	Standard source
Detector	TGS
Integration number	16
Resolution	4 cm^{-1}
Zero filling	On
Apodization	Cosine
Gain	Auto (16)
Aperture	Auto (7.1 mm)
Scan speed	Auto (2 mm/sec)
Filter	Auto (10000 Hz)

Peak detection

No.	$\tilde{\nu}$ (cm^{-1})	%T	No.	$\tilde{\nu}$ (cm^{-1})	%T
1	3068.19	87.0638	2	3028.66	84.3569
3	2938.02	83.5007	4	2866.67	87.0308
5	1712.48	9.95607	6	1605.45	11.7984
7	1578.45	55.6571	8	1509.03	26.1503
9	1455.03	60.4924	10	1421.28	69.2694
11	1383.68	65.4828	12	1364.39	73.7049
13	1315.21	38.1684	14	1280.5	13.417

Peak detection

No.	$\tilde{\nu}$ (cm^{-1})	%T	No.	$\tilde{\nu}$ (cm^{-1})	%T
15	1251.58	4.62286	16	1168.65	11.1696
17	1100.19	22.8709	18	1051.01	68.4285
19	1008.59	36.3294	20	958.448	77.5429
21	918.914	82.9291	22	848.525	53.3246
23	768.494	34.336	24	747.281	32.3957
25	696.177	36.4834	26	655.679	67.4747
27	632.537	87.3124	28	514.901	80.2664
29	491.759	86.2249	30	431.012	84.5836
31	403.05	84.136			


3.835
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7.943
7.931
7.926
7.919
7.260
7.207
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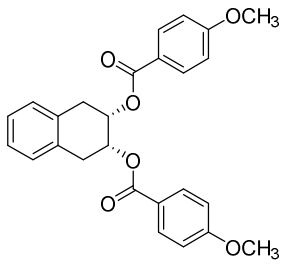
7.183
7.177
7.175
7.170
7.168
7.163
7.158
7.156
7.146
6.892
6.885
6.880
6.868
6.863
6.856
5.702
5.700
5.697
5.688
5.684
5.671
5.668
3.348
3.341
3.337
3.332
3.327
3.96
4.00
4.00
2.01
6.02
4.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
(ppm)


OCH3
O
O
O
O
OCH3

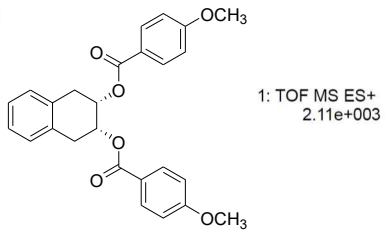
8

165.81
163.55
132.66
131.86
129.23
126.55
122.68
113.73
77.48
77.16
76.84
70.05
55.52
32.34
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)

8

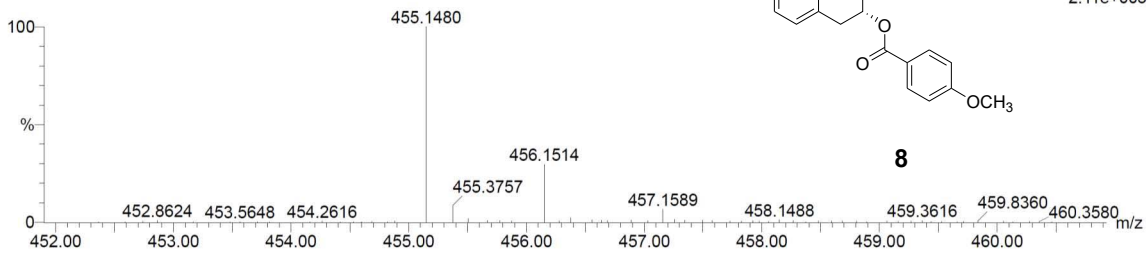
Tolerance = 10.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
104 formula(e) evaluated with 4 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 1-40 H: 1-60 O: 0-10 Na: 0-1
USJ-2-B
190822_Fukumoto17 346 (1.907)



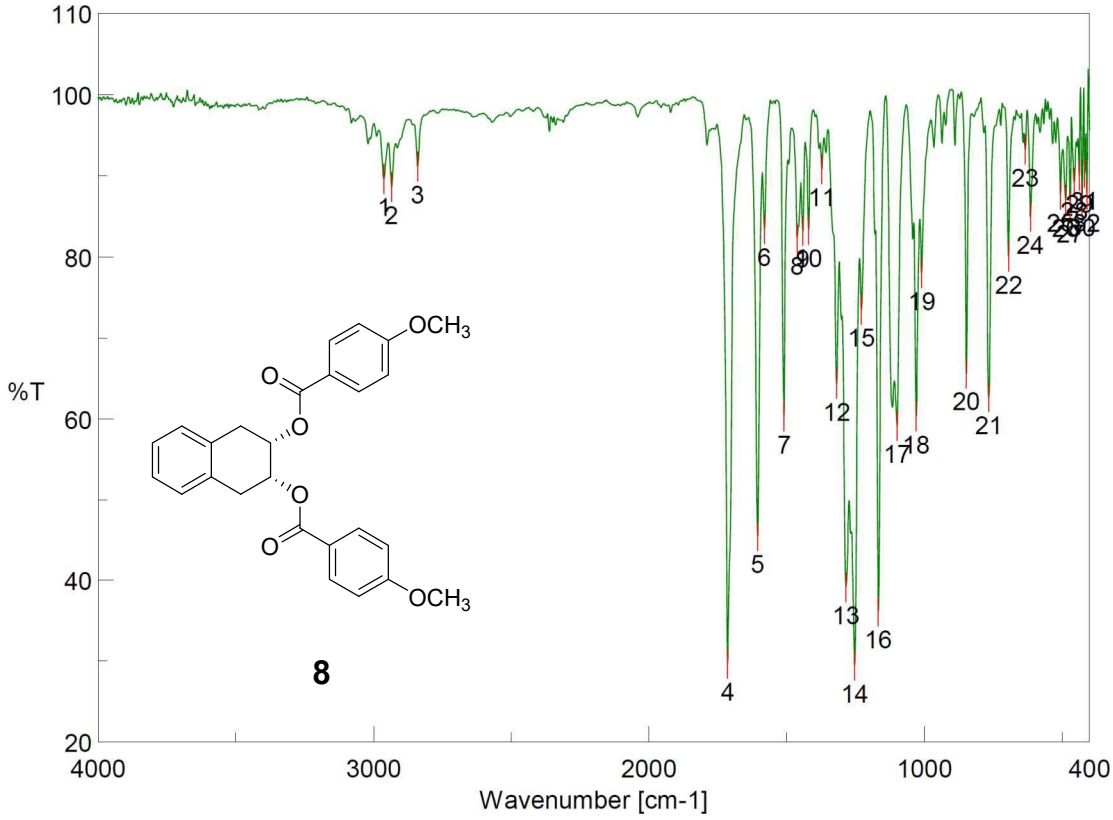
1: TOF MS ES+
2.11e+003

8



Minimum:	80.00				-1.5			
Maximum:	100.00		10.0	10.0	50.0			
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
455.1480	100.00	455.1471	0.9	2.0	14.5	108.3	0.7	C26 H24 O6 Na
		455.1495	-1.5	-3.3	17.5	108.2	0.7	C28 H23 O6
		455.1436	4.4	9.7	26.5	113.3	5.7	C35 H19 O
		455.1412	6.8	14.9	23.5	113.0	5.5	C33 H20 O Na

IR spectrum – USJ-2A



General information

Sample name	USJ-2A
Comments	Compound **8**
Measurer	Andrea Renzetti
Affiliation	University of the Ryukyus

Data information

Date and time	2019/09/03 12:45

Data type

Horizontal axis	Wavenumber (cm^{-1})
Vertical axis	%T
Start	399.193 cm^{-1}
End	4000.6 cm^{-1}
Data interval	0.964233 cm^{-1}
Number of data	3736

Measurements information

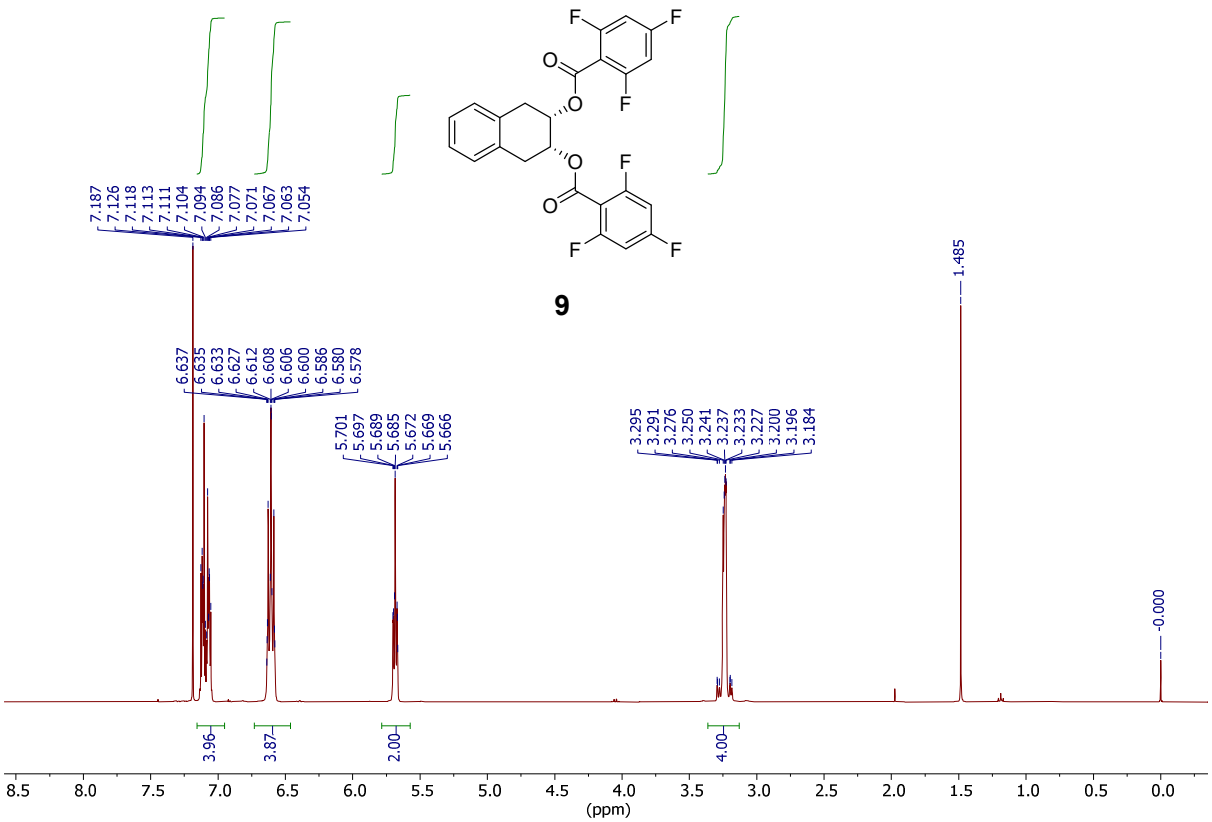
Model name	FT/IR-6100typeA
Serial number	A042161020
Date and time	2019/09/03 12:44
Source	Standard source
Detector	TGS
Integration number	16
Resolution	4 cm^{-1}
Zero filling	On
Apodization	Cosine
Gain	Auto (16)
Aperture	Auto (7.1 mm)
Scan speed	Auto (2 mm/sec)
Filter	Auto (10000 Hz)

Peak detection

No.	$\tilde{\nu}$ (cm^{-1})	%T	No.	$\tilde{\nu}$ (cm^{-1})	%T
1	2962.13	89.5975	2	2934.16	88.5431
3	2839.67	91.1005	4	1714.41	29.6584
5	1605.45	45.3801	6	1580.38	83.3749
7	1509.03	60.2256	8	1461.78	82.3189
9	1441.53	83.1758	10	1420.32	83.2689
11	1372.1	90.7768	12	1319.07	64.3088
13	1284.36	39.1033	14	1252.54	29.3942

Peak detection

No.	$\tilde{\nu}$ (cm^{-1})	%T	No.	$\tilde{\nu}$ (cm^{-1})	%T
15	1228.43	73.4256	16	1166.72	36.0628
17	1099.23	59.1191	18	1029.8	60.2671
19	1009.55	77.96	20	847.561	65.5823
21	766.566	62.6079	22	694.248	79.9425
23	633.501	93.1877	24	614.217	84.8875
25	505.258	87.6056	26	486.938	86.9747
27	470.546	86.2639	28	456.082	89.0988
29	437.762	89.9688	30	427.155	87.0327
31	417.513	90.3058	32	409.799	87.607


9
7.187
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7.118
7.113
7.111
7.104
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7.086
7.077
7.071
7.067
7.063
7.054
6.637
6.635
6.633
6.627
6.612
6.608
6.606
6.600
6.586
6.580
6.578
5.701
5.697
5.689
5.685
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3.295
3.291
3.276
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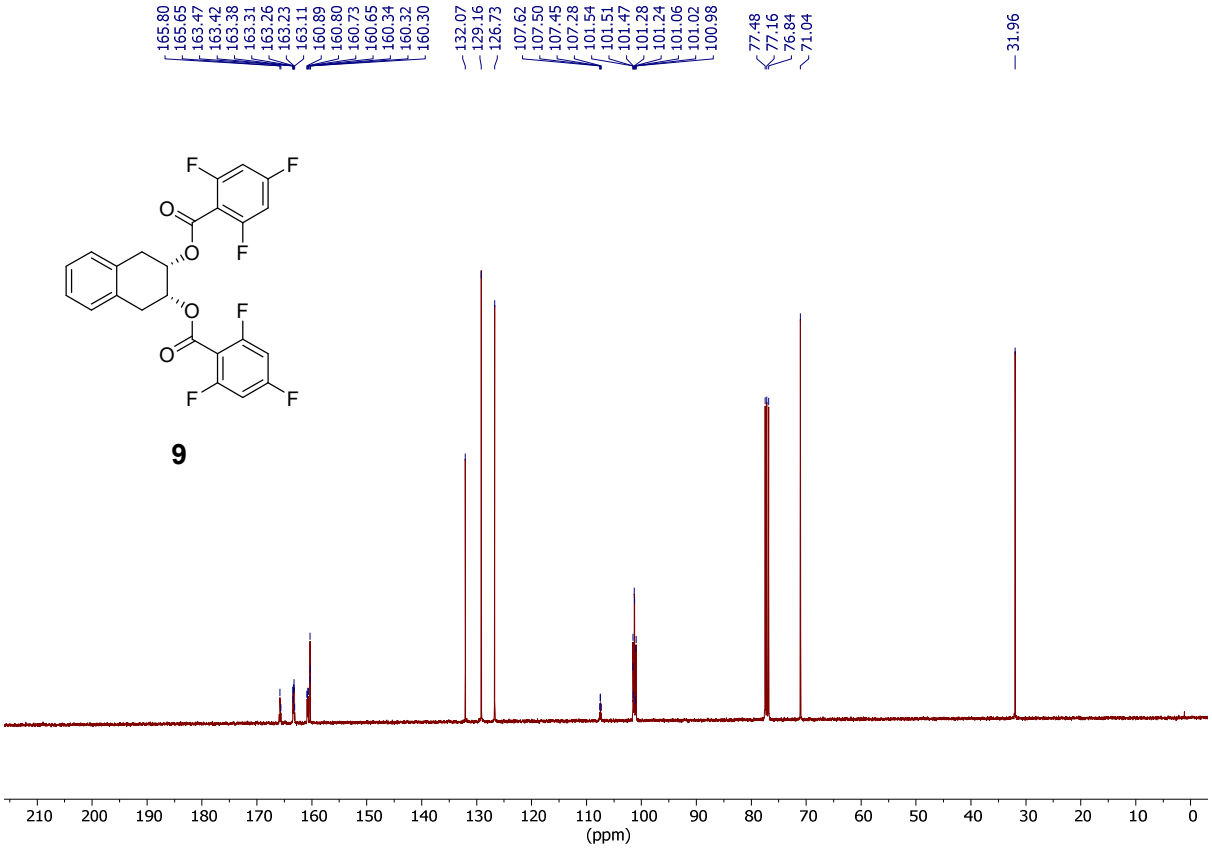
4.00
(ppm)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163.38
163.31
163.26
163.23
163.11
160.89
160.80
160.73
160.65
160.34
160.32
160.30
132.07
129.16
126.73
107.62
107.50
107.45
107.28
101.54
101.51
101.47
101.28
101.24
101.06
101.02
100.98
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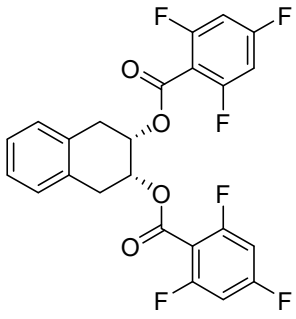
77.16
76.84
71.04
31.96
(ppm)

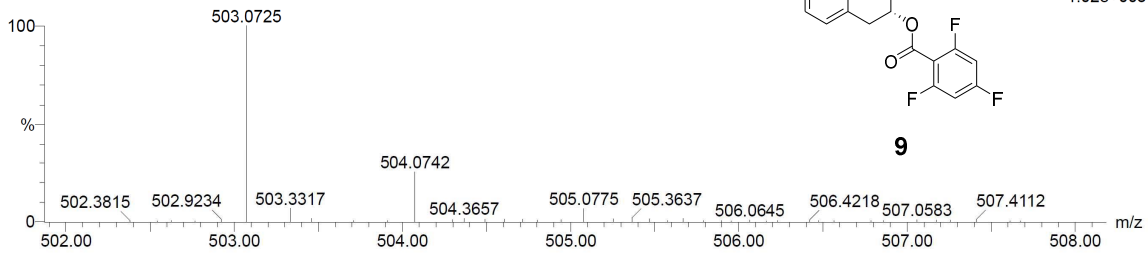

F
F
O
O
F
O
F
O
F
F

9

-101.32
-101.35
-101.37
-101.39
-101.42
-105.32
-105.32
-105.33
-105.36
-105.38
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
(ppm)

Tolerance = 10.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
1016 formula(e) evaluated with 48 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 1-40 H: 1-60 O: 0-10 F: 0-10 Na: 0-1
RNR-2-A
190822_Fukumoto16 48 (0.280)



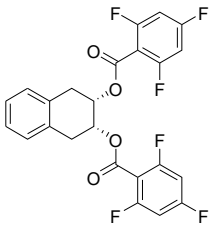
9

1: TOF MS ES+
1.02e+003

Minimum:	80.00				-1.5			
Maximum:	100.00		10.0	10.0	50.0			
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
503.0725	100.00	503.0728	-0.3	-0.6	2.5	83.3	6.1	C15 H17 O7 F9 Na
		503.0729	-0.4	-0.8	13.5	80.1	2.8	C23 H14 O5 F7
		503.0730	-0.5	-1.0	13.5	79.5	2.2	C23 H16 O7 F4 Na
		503.0720	0.5	1.0	28.5	83.2	5.9	C34 H12 O4 F
		503.0731	-0.6	-1.2	24.5	82.1	4.8	C31 H13 O5 F2
		503.0718	0.7	1.4	17.5	79.9	2.6	C26 H15 O6 F3 Na
		503.0718	0.7	1.4	17.5	80.2	3.0	C26 H13 O4 F6
		503.0717	0.8	1.6	6.5	82.1	4.9	C18 H16 O6 F8 Na
		503.0740	-1.5	-3.0	-1.5	84.5	7.2	C12 H18 O8 F10 Na
		503.0741	-1.6	-3.2	9.5	81.3	4.0	C20 H15 O6 F8
		503.0741	-1.6	-3.2	9.5	80.7	3.5	C20 H17 O8 F5 Na
		503.0708	1.7	3.4	32.5	84.1	6.8	C37 H11 O3
		503.0742	-1.7	-3.4	20.5	80.9	3.6	C28 H14 O6 F3
		503.0707	1.8	3.6	21.5	81.3	4.0	C29 H14 O5 F2 Na
		503.0707	1.8	3.6	21.5	81.4	4.2	C29 H12 O3 F5
		503.0743	-1.8	-3.6	20.5	80.8	3.6	C28 H16 O8 Na
		503.0705	2.0	4.0	10.5	81.0	3.7	C21 H15 O5 F7 Na
		503.0705	2.0	4.0	10.5	81.5	4.3	C21 H13 O3 F10
		503.0752	-2.7	-5.4	5.5	82.9	5.6	C17 H16 O7 F9
		503.0753	-2.8	-5.6	5.5	82.5	5.3	C17 H18 O9 F6 Na
		503.0696	2.9	5.8	25.5	82.8	5.5	C32 H13 O4 F Na
		503.0754	-2.9	-5.8	16.5	79.6	2.3	C25 H17 O9 F Na
		503.0754	-2.9	-5.8	16.5	79.9	2.6	C25 H15 O7 F4
		503.0695	3.0	6.0	25.5	82.8	5.5	C32 H11 O2 F4
		503.0694	3.1	6.2	14.5	80.5	3.2	C24 H14 O4 F6 Na
		503.0694	3.1	6.2	14.5	81.1	3.8	C24 H12 O2

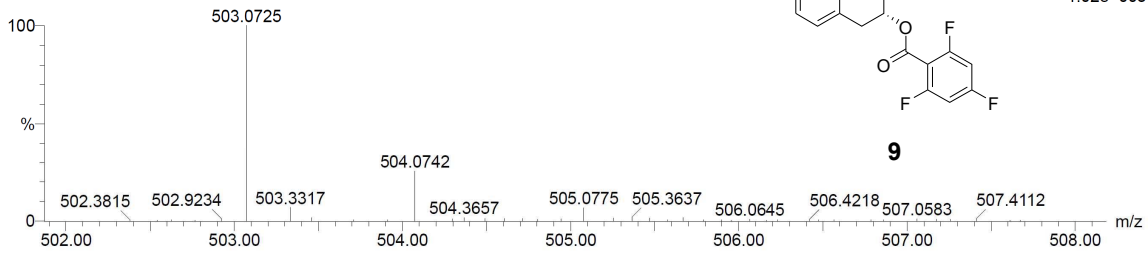
Tolerance = 10.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
1016 formula(e) evaluated with 48 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 1-40 H: 1-60 O: 0-10 F: 0-10 Na: 0-1
RNR-2-A
190822_Fukumoto16 48 (0.280)



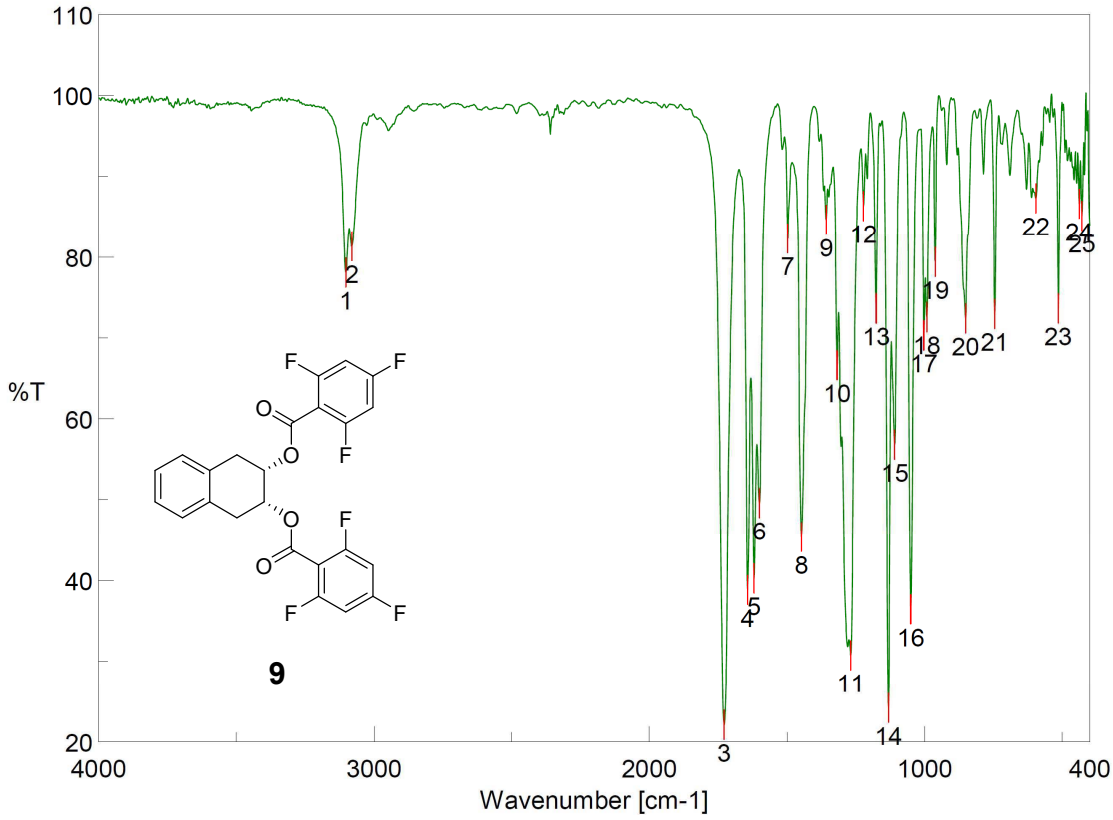
9

1: TOF MS ES+
1.02e+003



Minimum:	80.00				-1.5			
Maximum:	100.00		10.0	10.0	50.0			
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
		503.0764	-3.9	-7.8	1.5	84.2	6.9	F9 C14 H19 O10
		503.0764	-3.9	-7.8	1.5	84.4	7.1	F7 Na C14 H17 O8
		503.0765	-4.0	-8.0	12.5	80.3	3.1	F10 C22 H16 O8
		503.0684	4.1	8.1	29.5	84.0	6.8	F5 C35 H12 O3
		503.0766	-4.1	-8.1	12.5	79.8	2.5	Na C22 H18 O10
		503.0684	4.1	8.1	29.5	84.0	6.8	F2 Na C35 H10 O
		503.0683	4.2	8.3	18.5	81.3	4.0	F3 C27 H13 O3
		503.0767	-4.2	-8.3	23.5	82.6	5.3	F5 Na C30 H15 O8
		503.0682	4.3	8.5	18.5	81.7	4.4	C27 H11 O
		503.0681	4.4	8.7	7.5	83.0	5.7	F8 C19 H14 O3
		503.0777	-5.2	-10.3	8.5	82.2	5.0	F10 Na C19 H17 O9
		503.0672	5.3	10.5	33.5	85.2	7.9	F6 C38 H9 F2
		503.0778	-5.3	-10.5	19.5	81.4	4.1	C27 H16 O9
		503.0671	5.4	10.7	22.5	82.9	5.7	F C30 H10 F7
		503.0671	5.4	10.7	22.5	82.8	5.6	C30 H12 O2
		503.0670	5.5	10.9	11.5	82.4	5.1	F4 Na C22 H13 O2
		503.0788	-6.3	-12.5	4.5	84.2	7.0	F9 Na C16 H18 O10
		503.0790	-6.5	-12.9	15.5	80.5	3.2	F7 C24 H17 O10
		503.0660	6.5	12.9	26.5	84.3	7.1	F2 C33 H11 O
		503.0658	6.7	13.3	15.5	82.5	5.2	F3 Na C25 H12 O
		503.0648	7.7	15.3	30.5	85.7	8.4	F8 Na C36 H10 F2
		503.0647	7.8	15.5	19.5	83.4	6.1	Na C28 H11 F7 Na

IR spectrum – RNR-2A



General information

Sample name	RNR-2A
Comments	Compound **9**
Measurer	Andrea Renzetti
Affiliation	University of the Ryukyus

Data information

Date and time	2019/09/03 12:06

Data type

Horizontal axis	Wavenumber (cm^{-1})
Vertical axis	%T
Start	399.193 cm^{-1}
End	4000.6 cm^{-1}
Data interval	0.964233 cm^{-1}
Number of data	3736

Measurements information

Model name	FT/IR-6100typeA
Serial number	A042161020
Date and time	2019/09/03 12:05
Source	Standard source
Detector	TGS
Integration number	16
Resolution	4 cm^{-1}
Zero filling	On
Apodization	Cosine
Gain	Auto (16)
Aperture	Auto (7.1 mm)
Scan speed	Auto (2 mm/sec)
Filter	Auto (10000 Hz)

Peak detection

No.	$\tilde{\nu}$ (cm^{-1})	%T	No.	$\tilde{\nu}$ (cm^{-1})	%T
1	3101.94	78.0381	2	3079.76	81.3024
3	1727.91	22.0974	4	1643.05	38.8043
5	1618.95	40.2132	6	1599.66	49.5664
7	1497.45	82.2692	8	1447.31	45.3321
9	1357.64	84.6048	10	1318.11	66.5898
11	1267.97	30.6943	12	1221.68	86.274
13	1176.36	73.598	14	1131.05	24.2093

Peak detection

No.	$\tilde{\nu}$ (cm^{-1})	%T	No.	$\tilde{\nu}$ (cm^{-1})	%T
15	1108.87	56.7846	16	1050.05	36.4009
17	1002.8	70.2665	18	992.196	72.4931
19	961.341	79.3367	20	851.418	72.3608
21	745.352	72.9018	22	595.896	87.2575
23	513.936	73.5919	24	438.726	86.561
25	428.12	85.0798			

Refinement of disordered fragment in compound 4

There is a positional disorder for the benzyl group bonded to O4, where the split phenyl group moieties lie at a slightly shifted site. The site occupation factor of the major part (C26A - C31A) was refined to 51(2)%. Geometries of the disordered fragments were restrained using DFIX, DANG, and SIMU in refinement.

Computational details

Electrostatic potential maps were created using *ArgusLab 4.0.1* software (Intel). For each compound, crystal structure was imported, then ground state electron density and electrostatic potential were calculated using the HOMO and LUMO at the Parameterized Model three (PM3) level of theory. A maximum number of 200 iterations and an energy convergence value of 10^{-10} kcal mol^{-1} were selected for the determination of the wave function and the energy of the system in the Hartree-Fock method (SCF).