



Title	Chiral diversification through the assembly of achiral phenylacetylene macrocycles with a two-fold bridge
Author(s)	Katoono, Ryo; Kusaka, Keiichi; Saito, Yuki et al.
Citation	Chemical Science, 10(18), 4782-4791 https://doi.org/10.1039/C9SC00972H
Issue Date	2019-05-14
Doc URL	https://hdl.handle.net/2115/75085
Rights(URL)	https://creativecommons.org/licenses/by-nc/3.0/
Type	journal article
File Information	Supplementary Information.pdf

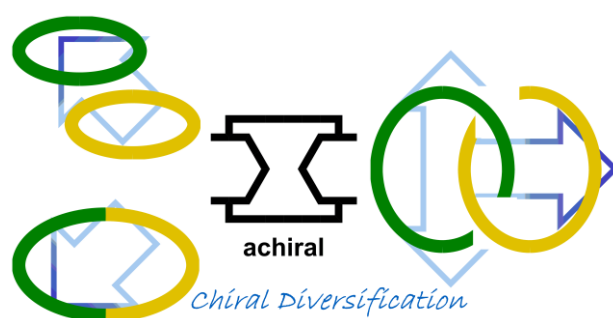


Supplementary Information 2

Chiral diversification through the assembly of achiral phenylacetylene macrocycles with a two-fold bridge

Ryo Katoono,* Keiichi Kusaka, Yuki Saito, Kazuki Sakamoto and Takanori Suzuki

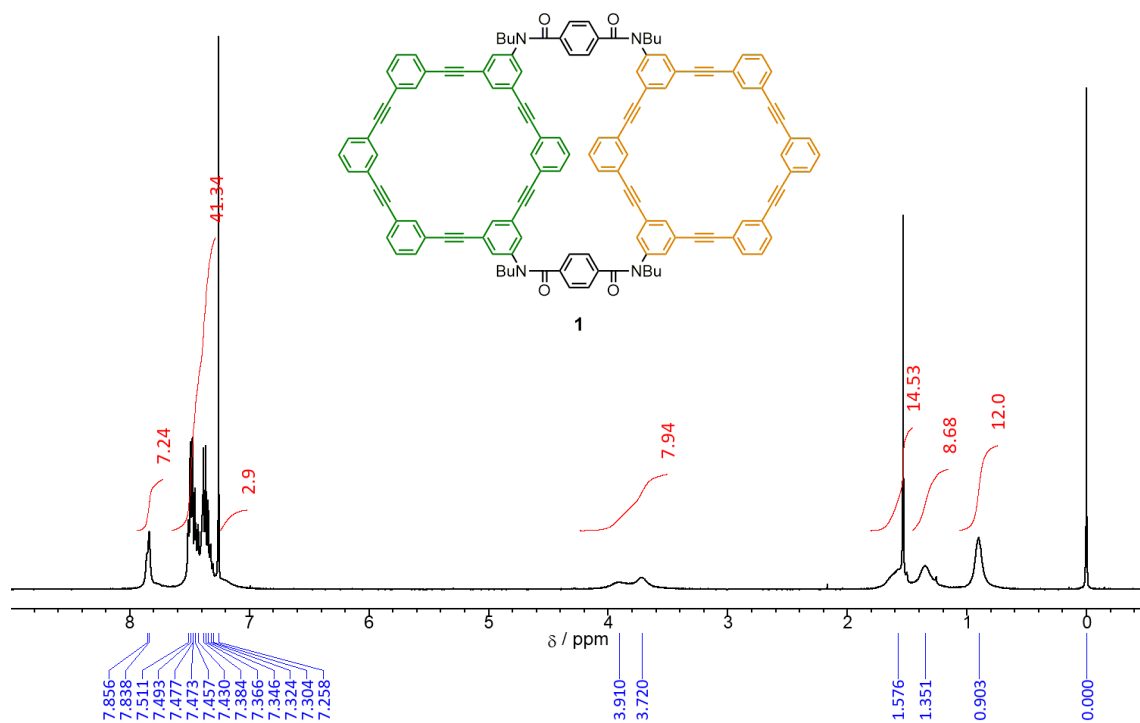
Department of Chemistry, Faculty of Science, Hokkaido University



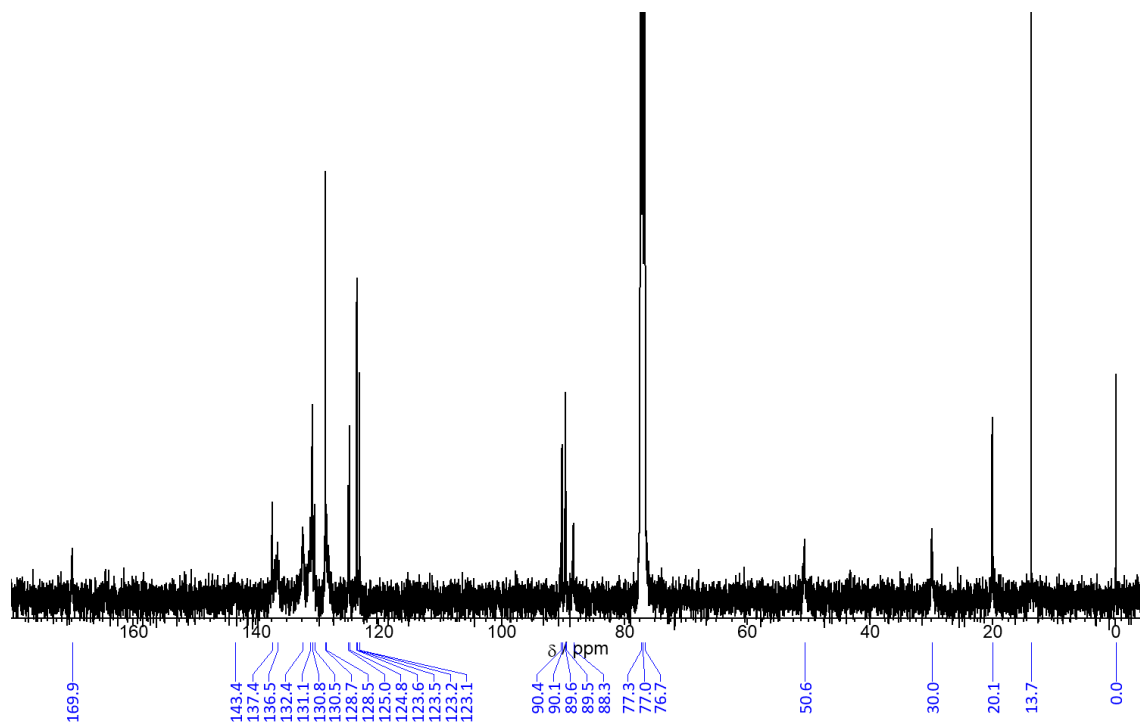
Contents

Copies of ^1H and ^{13}C NMR, IR and mass spectra of new compounds	S40-S76
Copies of ^1H NMR spectrum of new compounds (unprotected)	S77-S80
Copies of chiral HPLC traces	S81

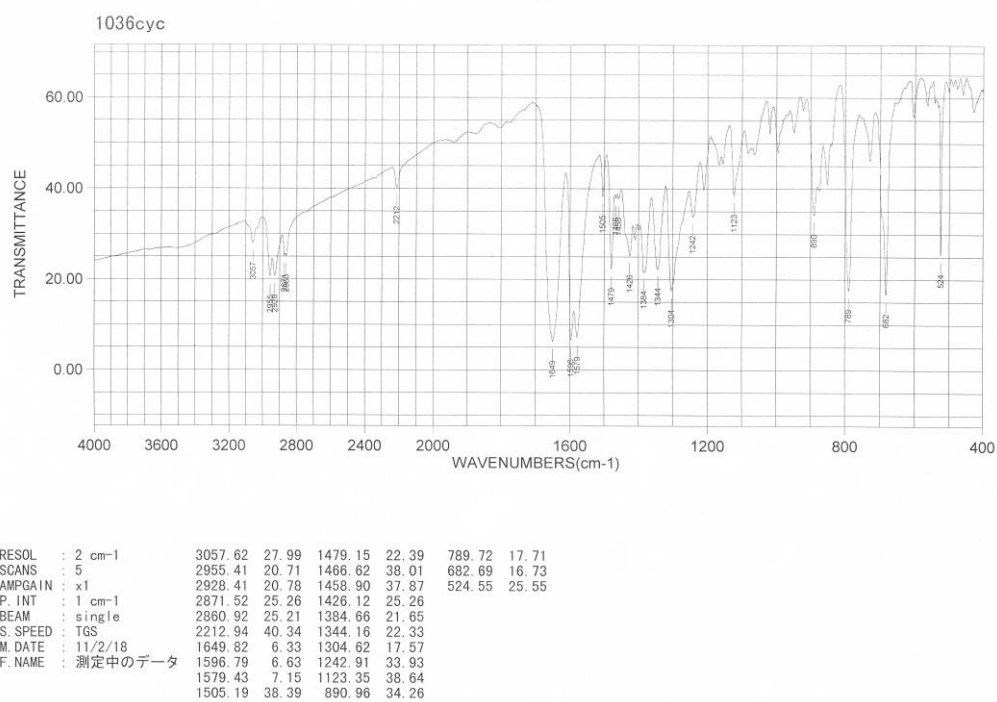
^1H and ^{13}C NMR spectra of new compounds



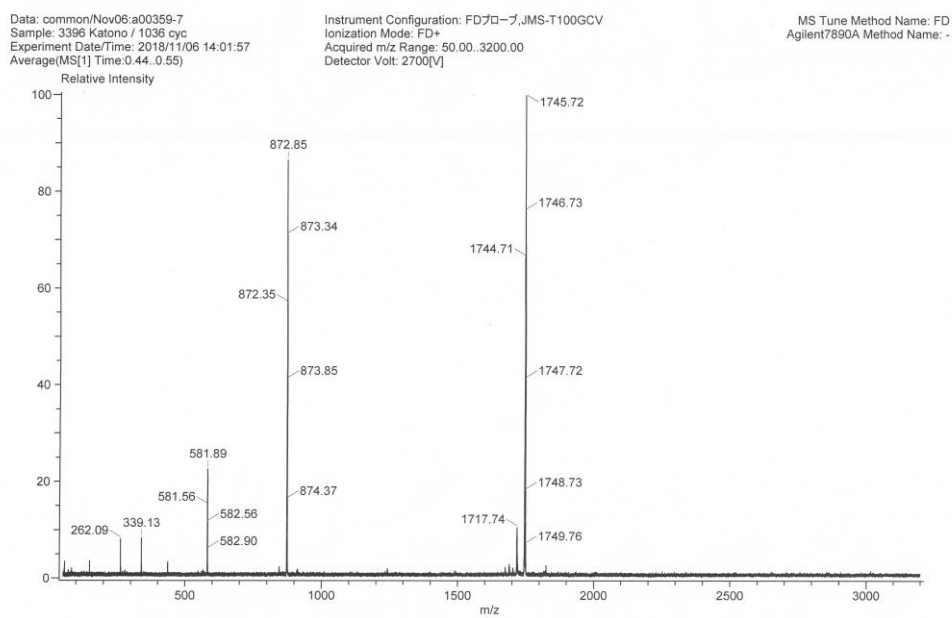
^1H NMR (400 MHz) spectrum of **1**, measured in CDCl_3 at 303 K.



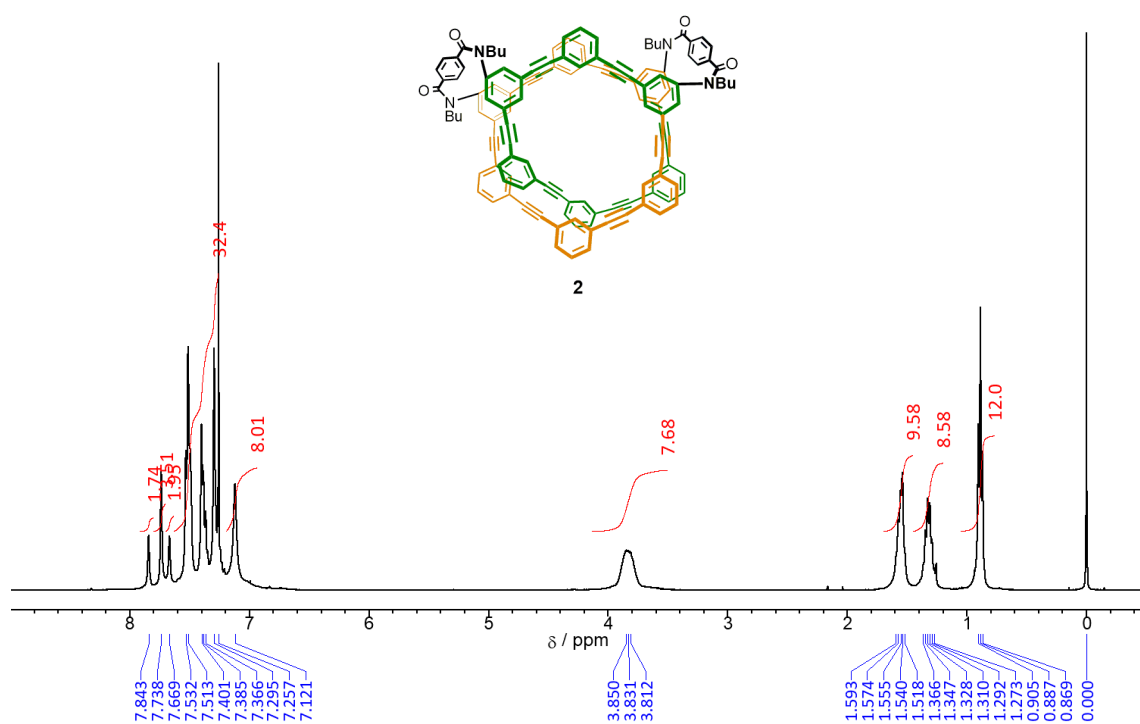
^{13}C NMR (100 MHz) spectrum of **1**, measured in CDCl_3 at 313 K.



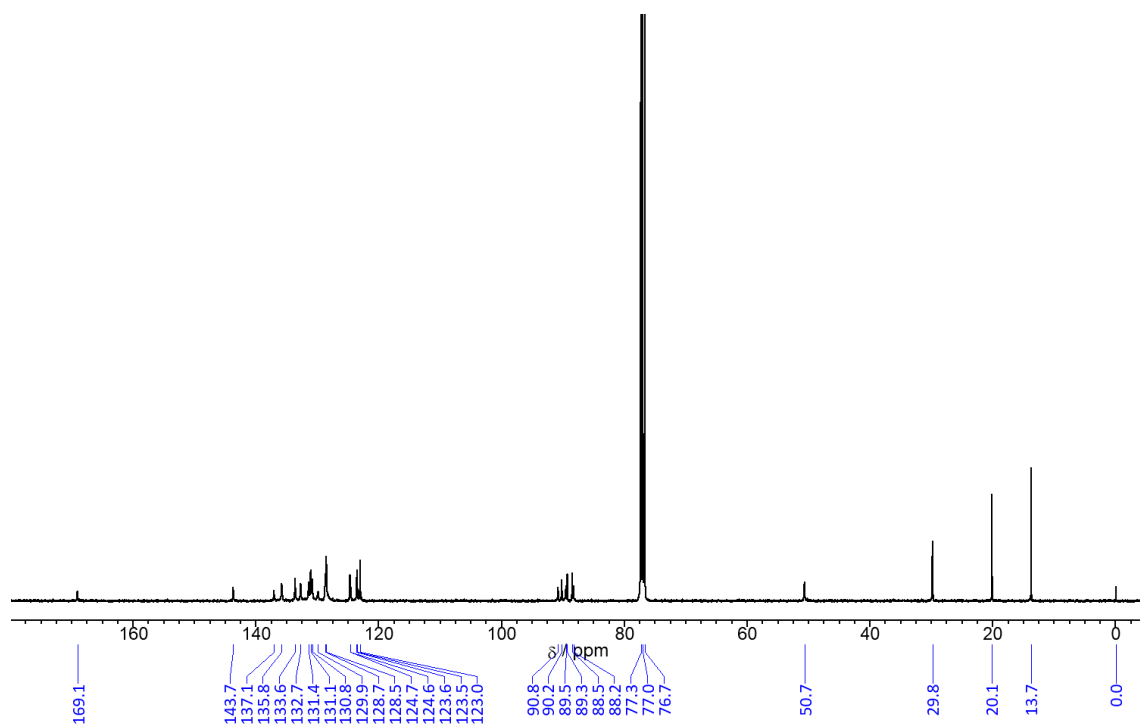
IR spectrum of **1**.



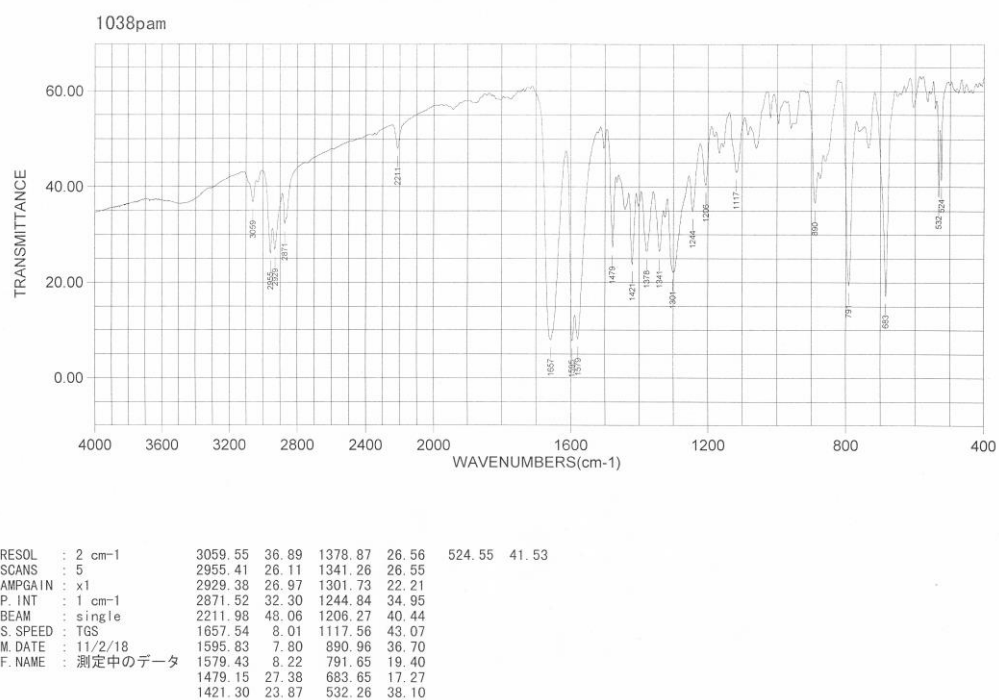
Mass spectrum (FD) of **1**.



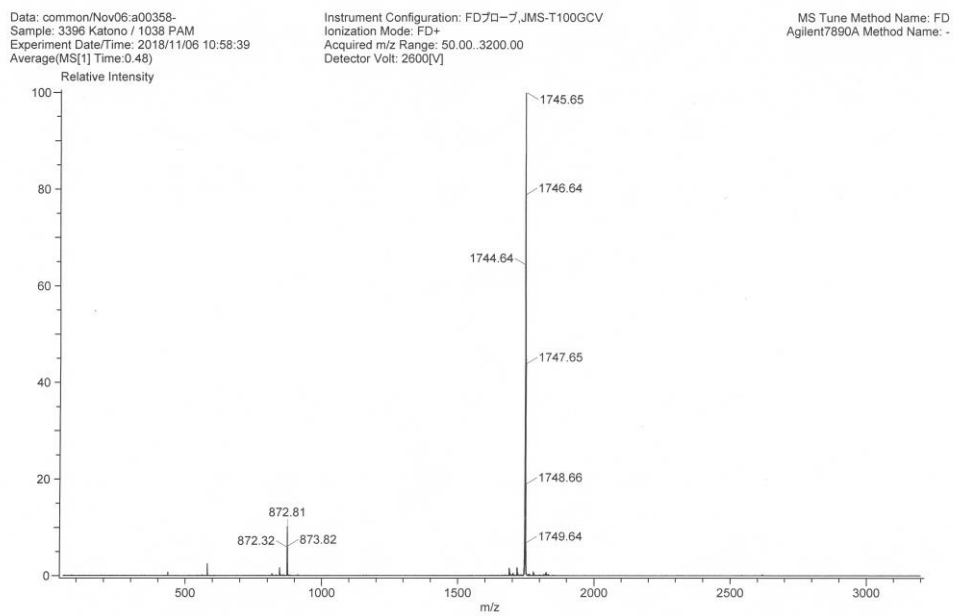
¹H NMR (400 MHz) spectrum of **2**, measured in chloroform-*d* at 303 K.



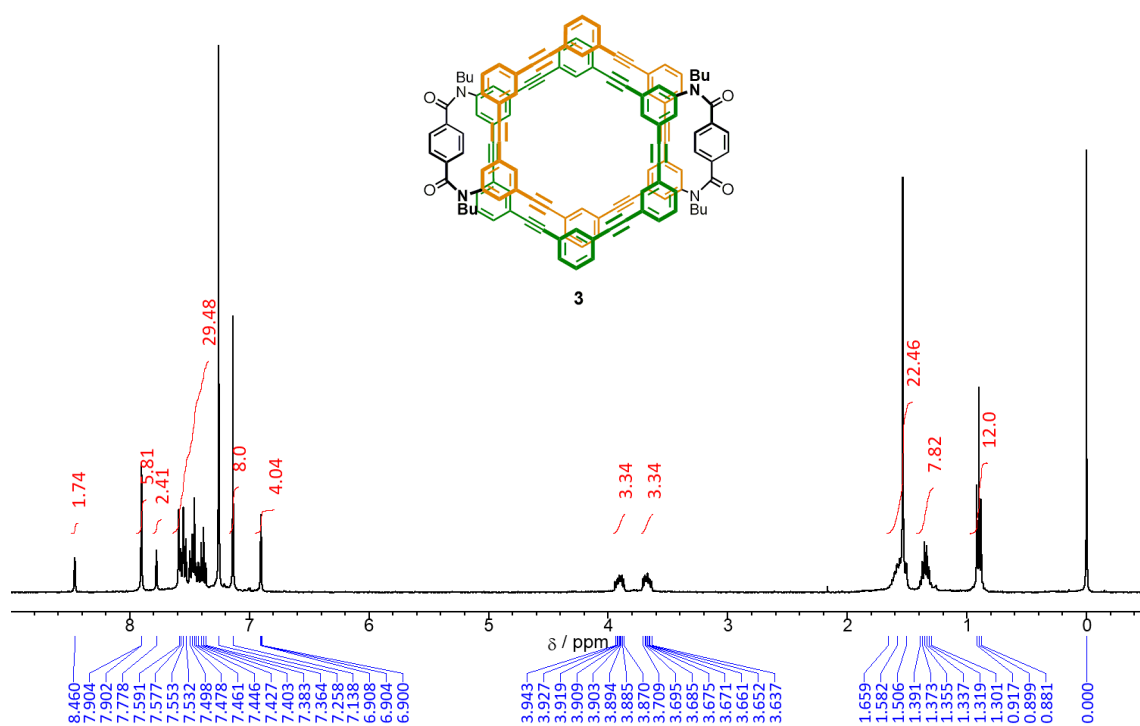
¹³C NMR (100 MHz) spectrum of **2**, measured in chloroform-*d* at 313 K.



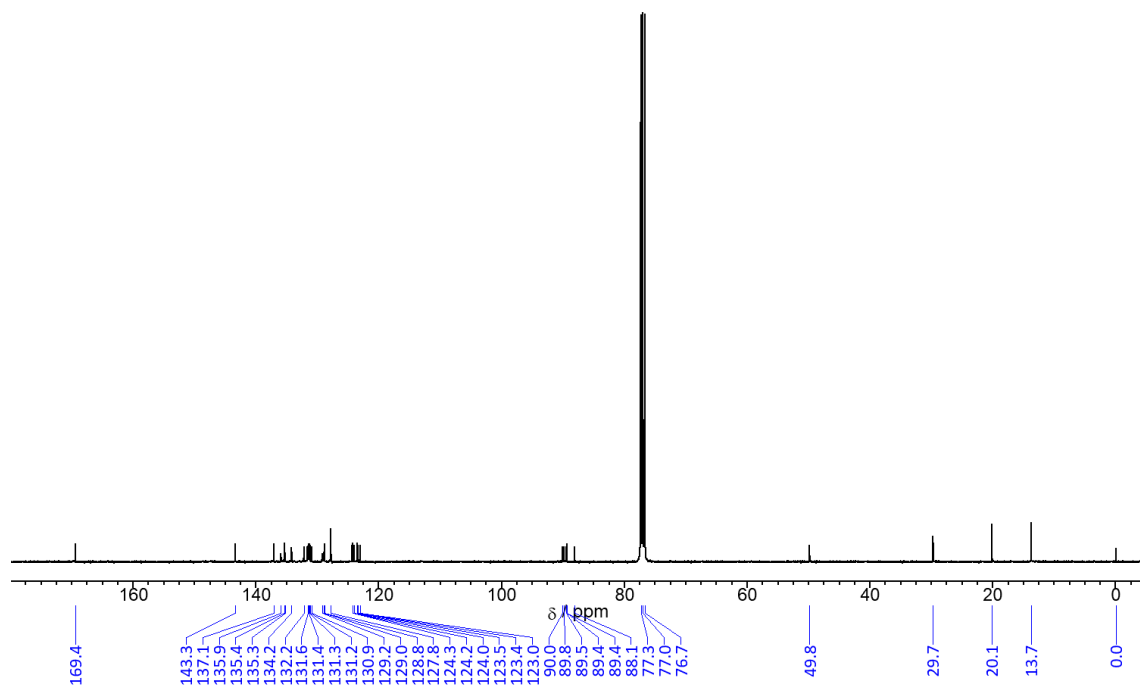
IR spectrum of 2.



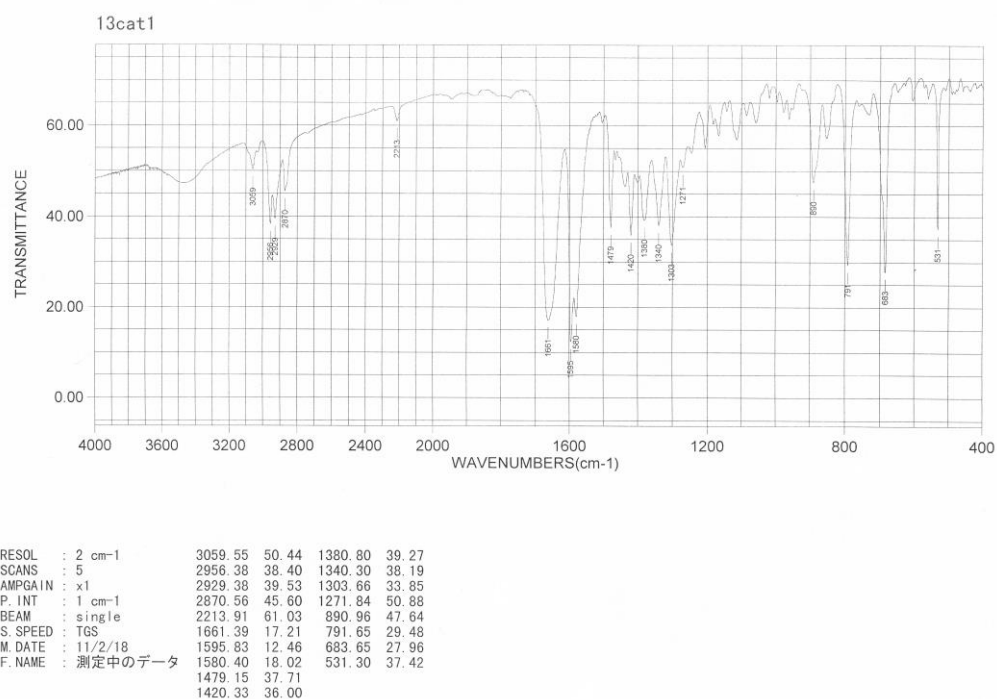
Mass spectrum (FD) of 2.



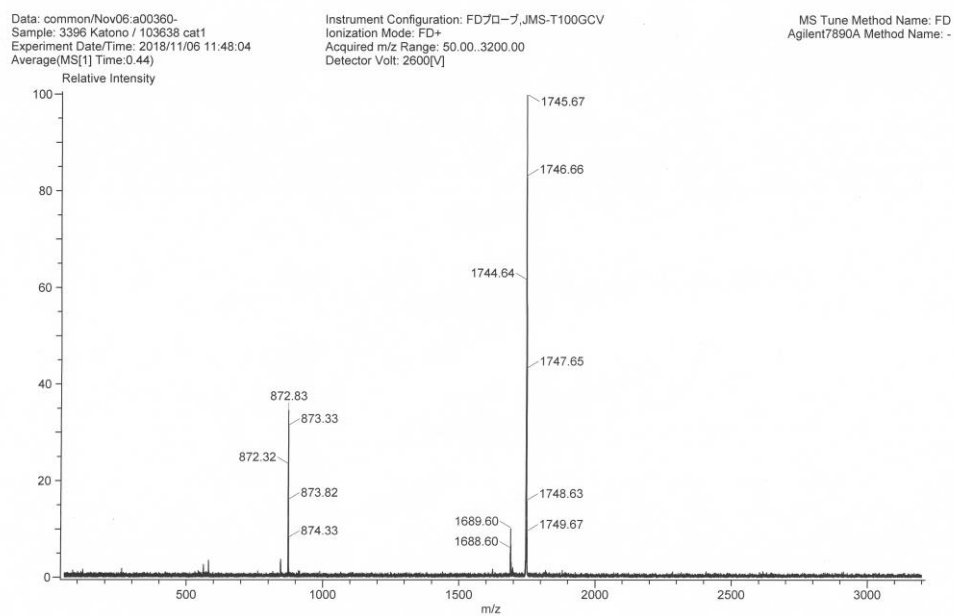
¹H NMR (400 MHz) spectrum of (±)-**3**, measured in chloroform-*d* at 303 K.



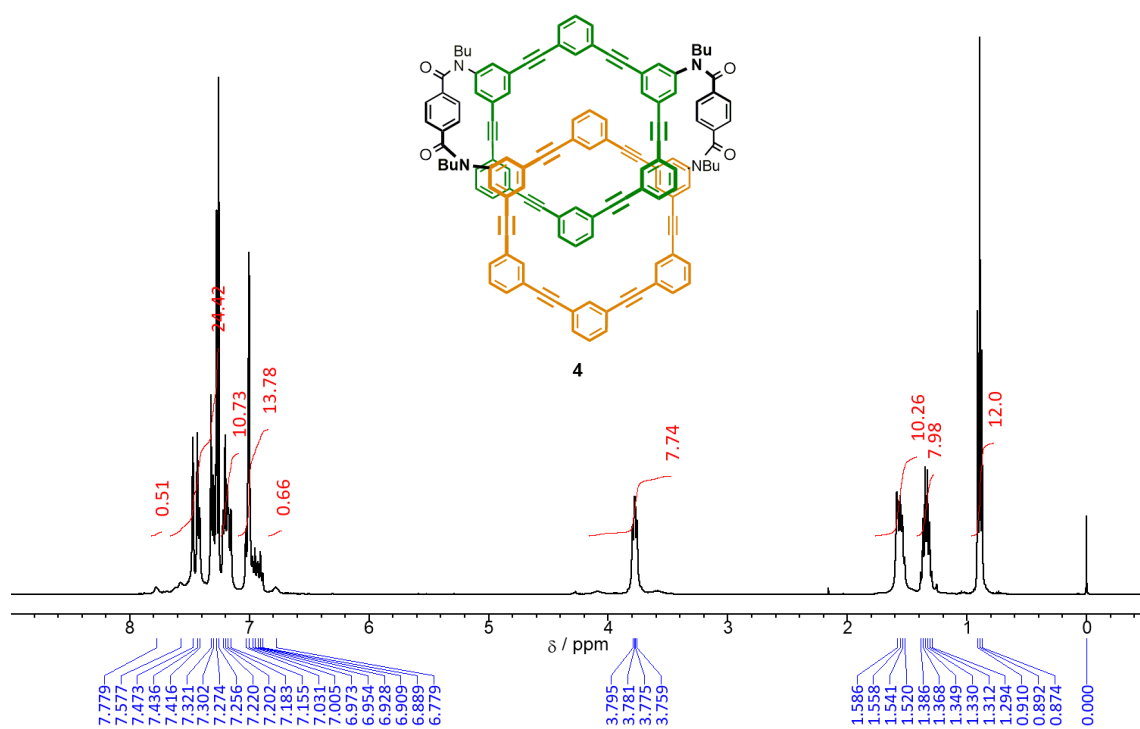
¹³C NMR (100 MHz) spectrum of (±)-**3**, measured in chloroform-*d* at 313 K.



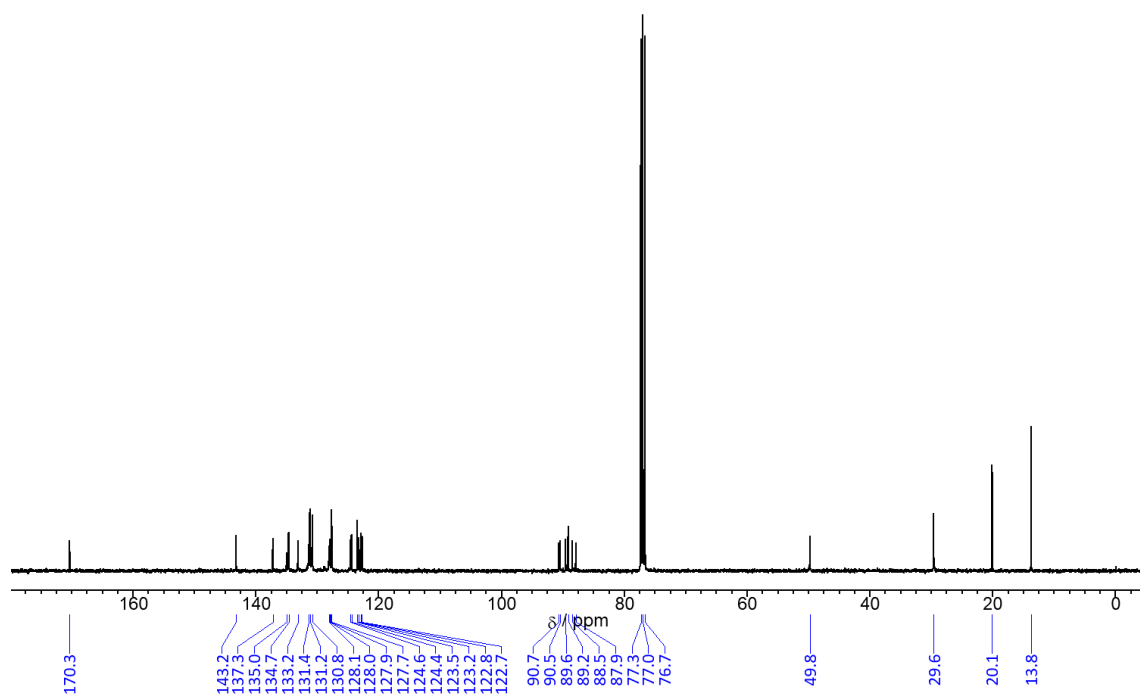
IR spectrum of (±)-3.



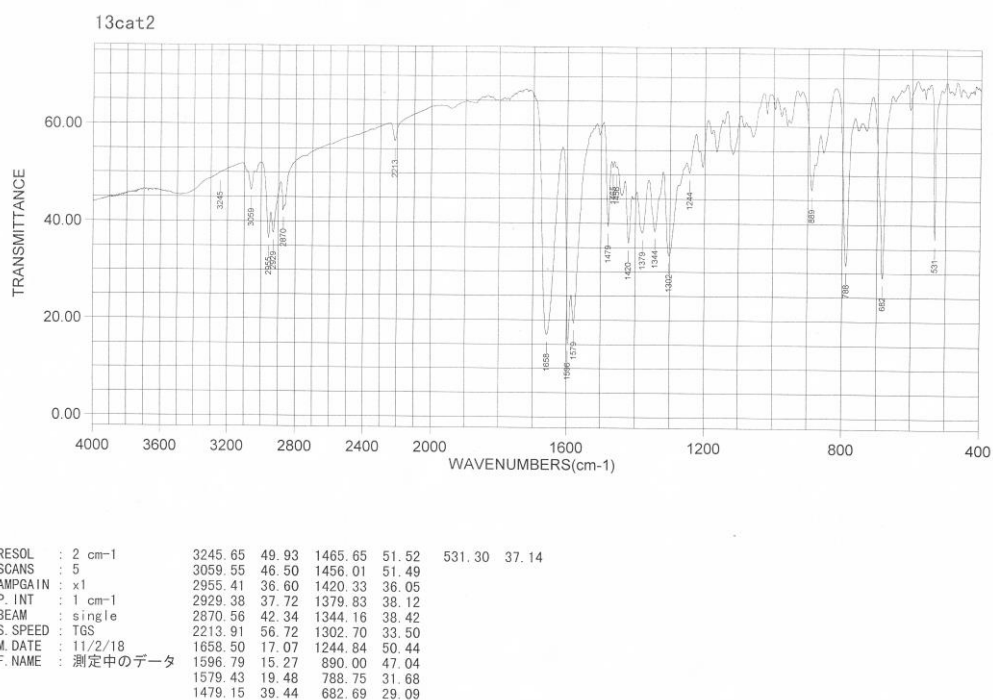
Mass spectrum (FD) of (±)-3.



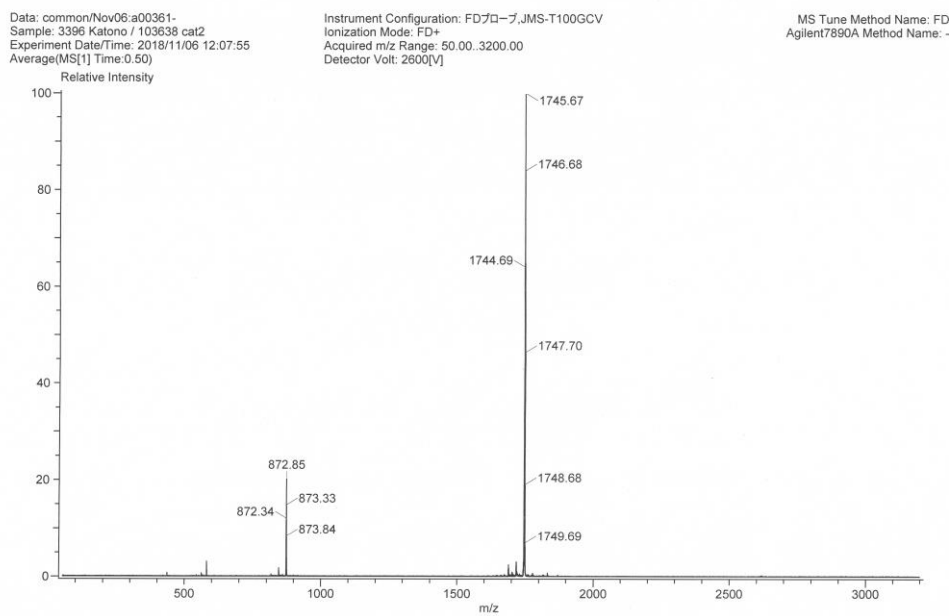
¹H NMR (400 MHz) spectrum of (±)-**4**, measured in chloroform-*d* at 303 K.



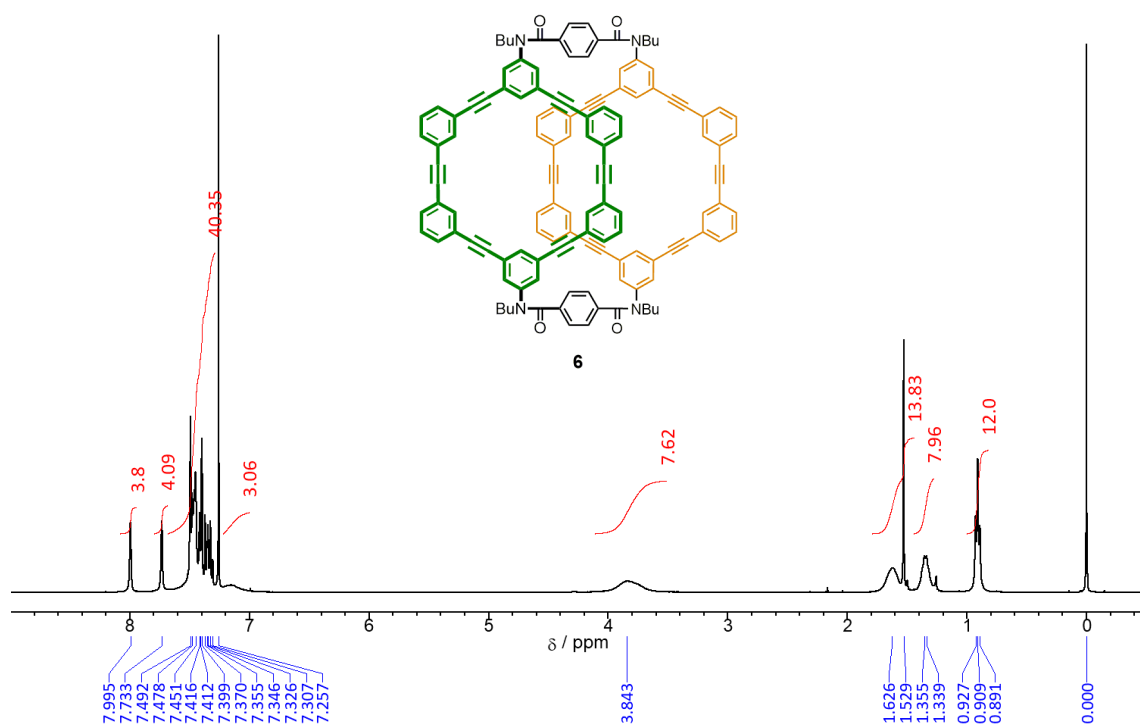
¹³C NMR (100 MHz) spectrum of (±)-**4**, measured in chloroform-*d* at room temperature.



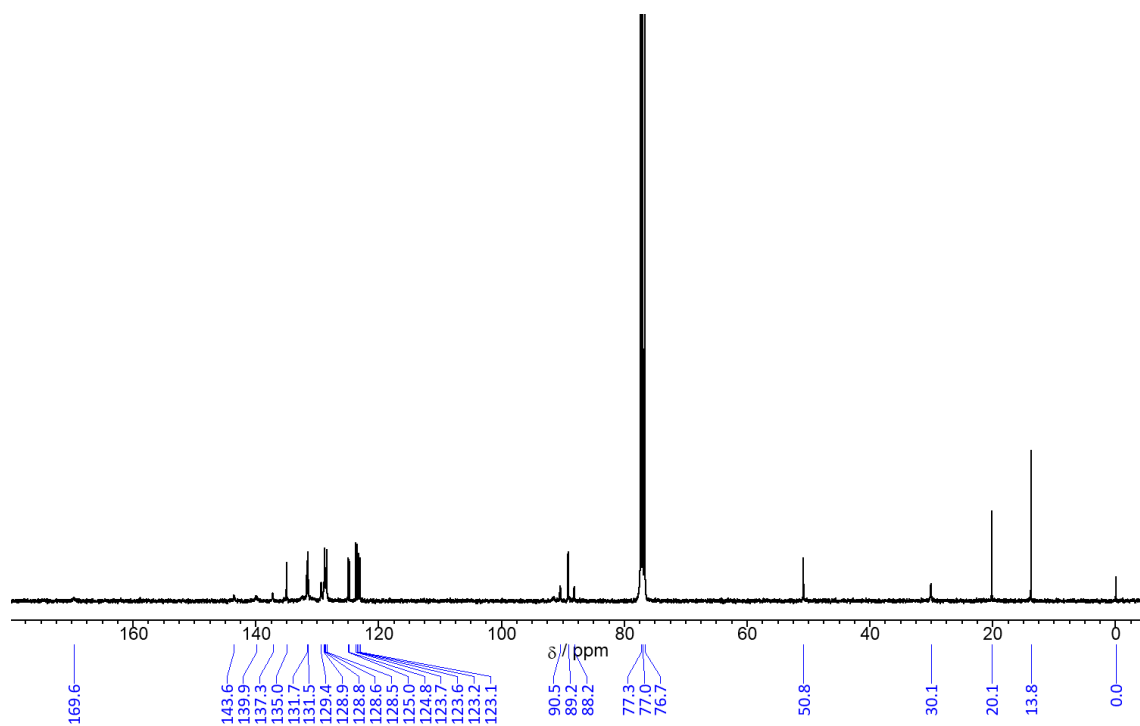
IR spectrum of (±)-4.



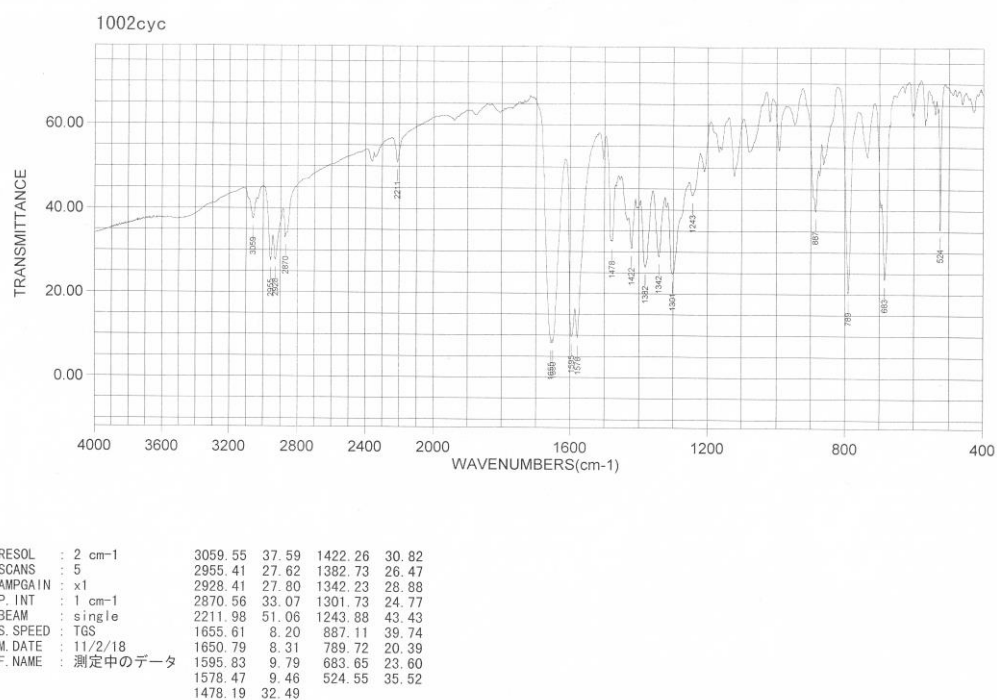
Mass spectrum (FD) of (±)-4.



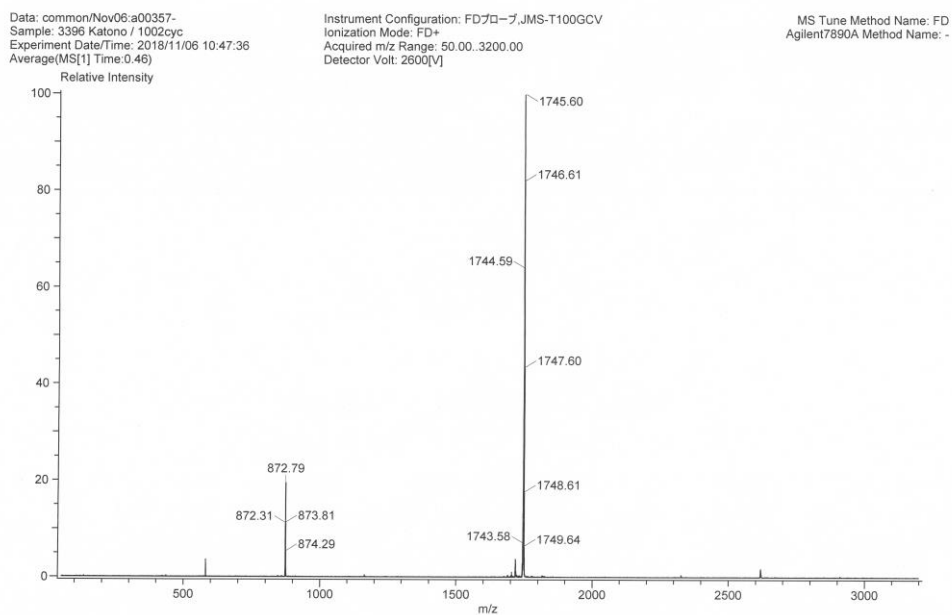
¹H NMR (400 MHz) spectrum of **6**, measured in chloroform-*d* at 303 K.



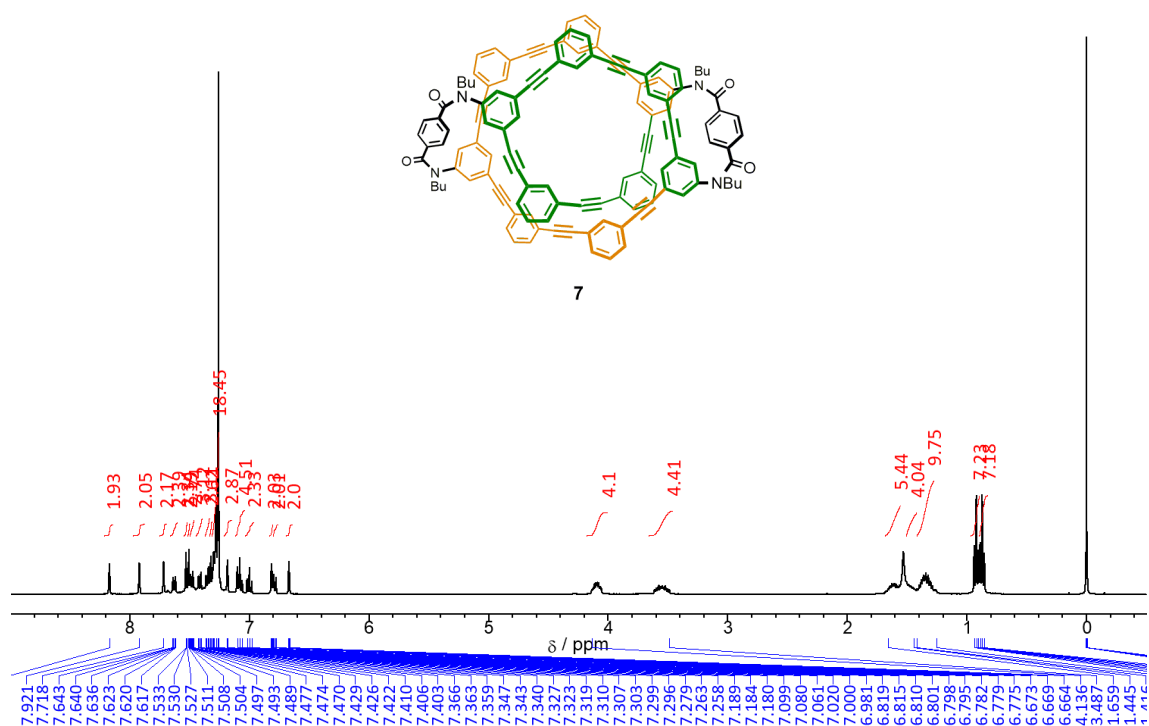
¹³C NMR (100 MHz) spectrum of **6**, measured in chloroform-*d* at 313 K.



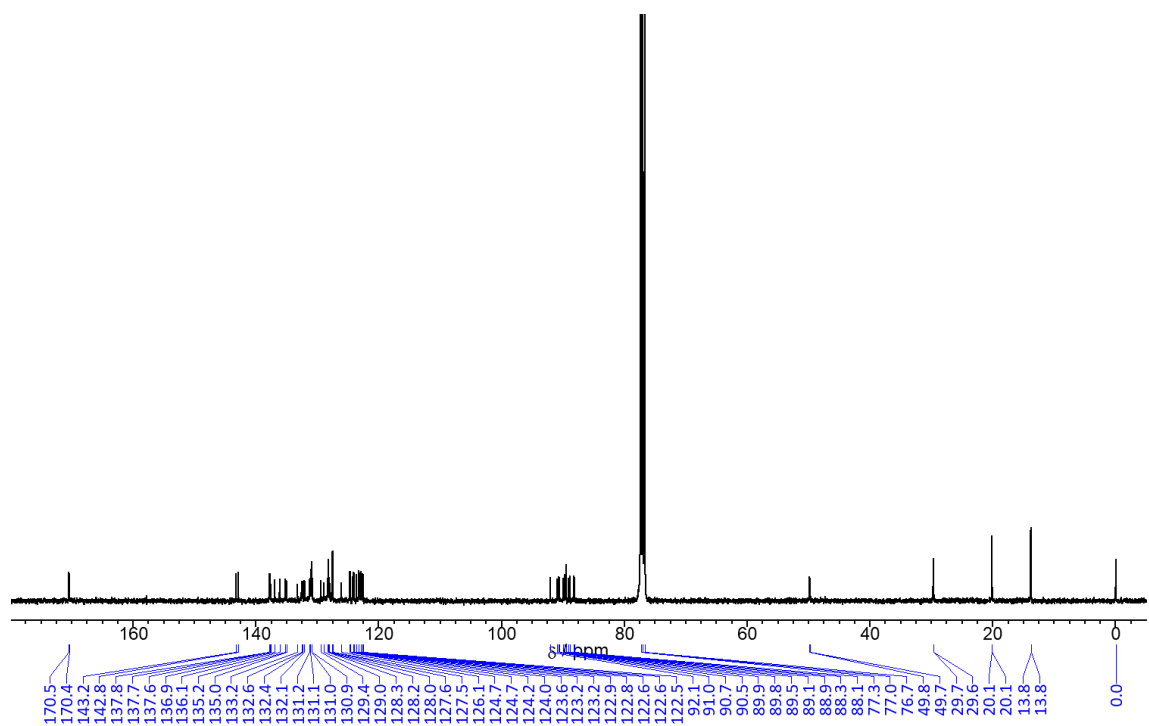
IR spectrum of **6**.



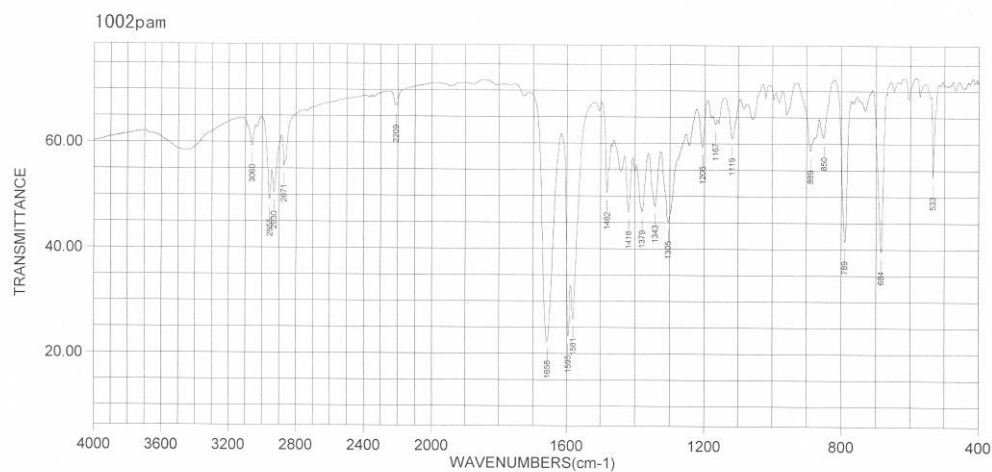
Mass spectrum (FD) of **6**.



¹H NMR (400 MHz) spectrum of **7**, measured in chloroform-*d* at 303 K.

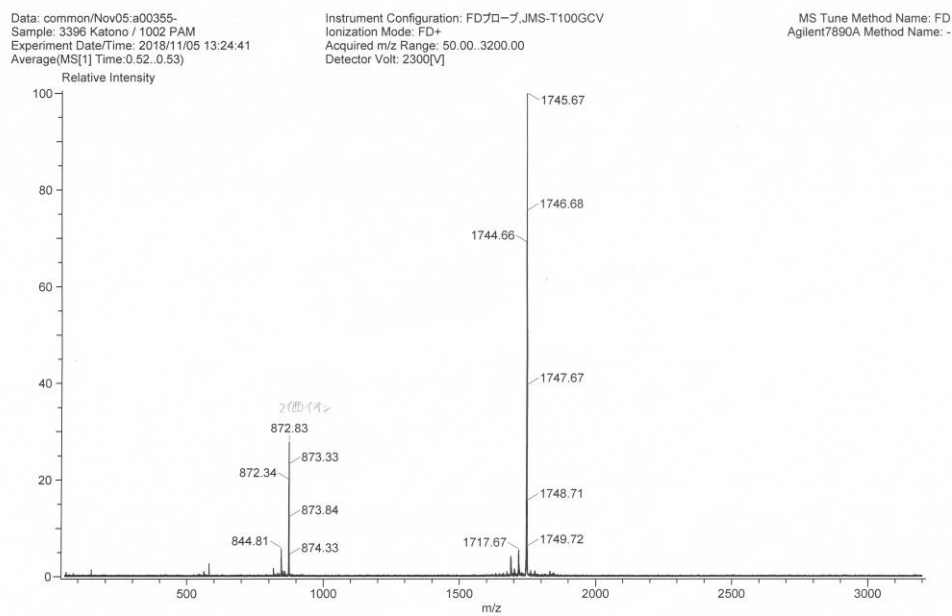


¹³C NMR (100 MHz) spectrum of **7**, measured in chloroform-*d* at room temperature.

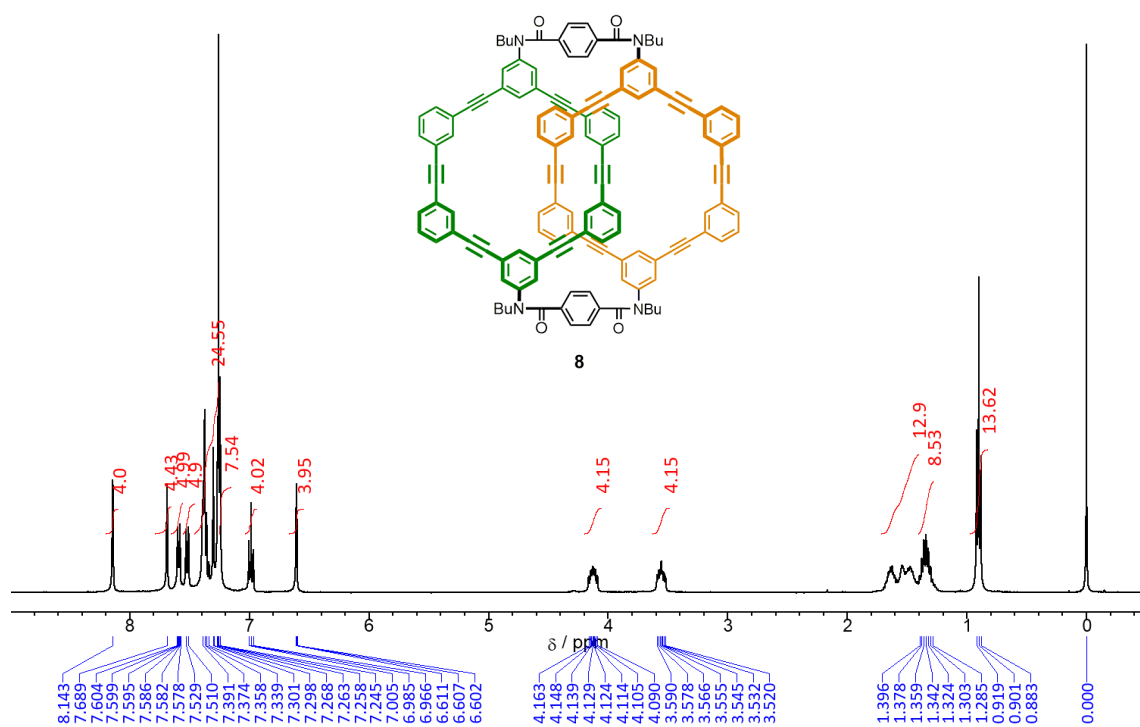


RESOL	: 2 cm-1	3060.51	59.30	1379.83	47.11	533.23	53.77
SCANS	: 5	2955.41	49.06	1343.19	48.12		
AMPGAIN	: x1	2930.34	50.44	1305.59	45.14		
P. INT	: 1 cm-1	2871.52	55.52	1206.27	59.32		
BEAM	: single	2209.09	66.94	1167.70	63.71		
S. SPEED	: TGS	1658.50	22.31	1119.49	60.97		
M. DATE	: 11/2/18	1595.83	23.34	889.03	58.69		
F. NAME	: 測定中のデータ	1581.36	26.55	850.46	61.14		
		1482.04	50.58	789.72	41.46		
		1418.40	47.01	684.61	39.52		

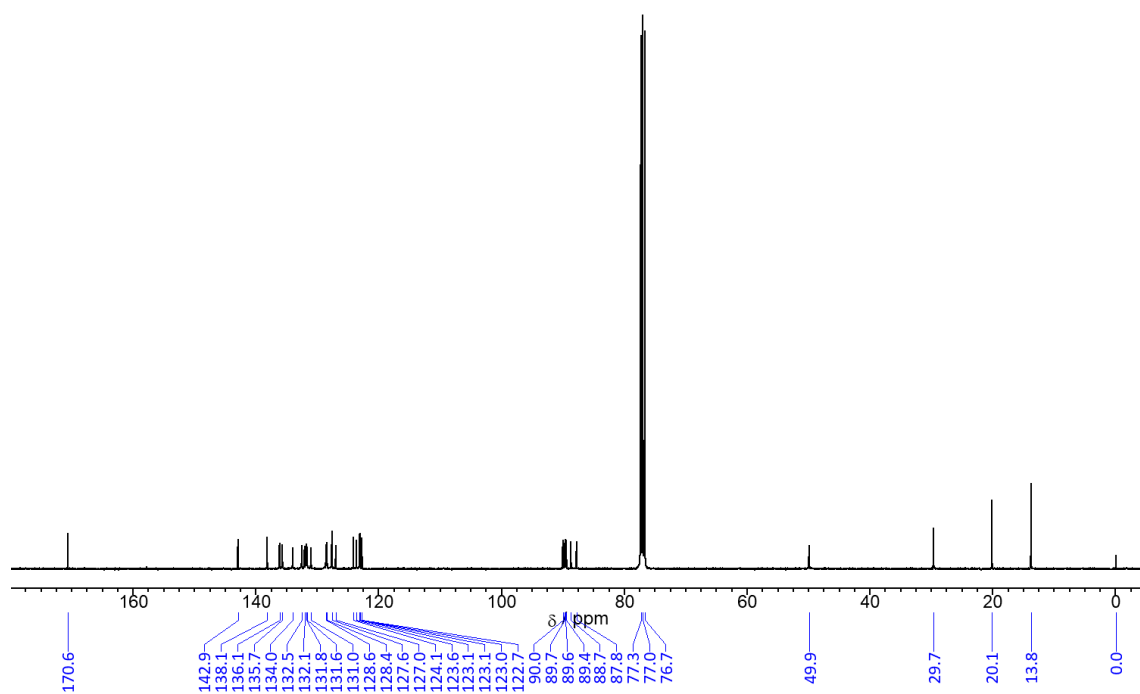
IR spectrum of 7.



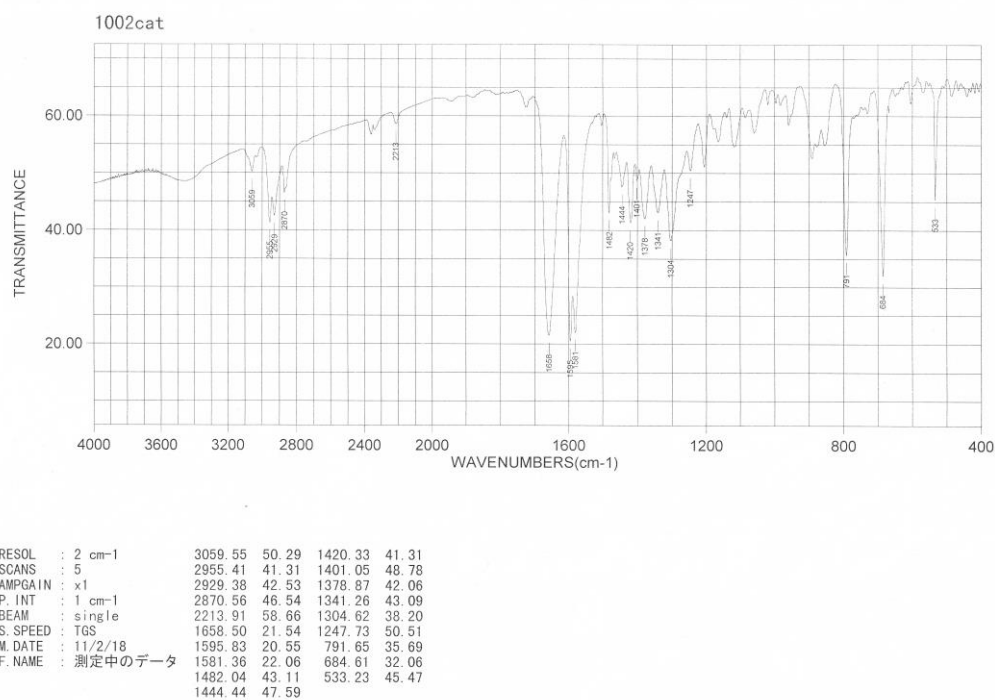
Mass spectrum (FD) of 7.



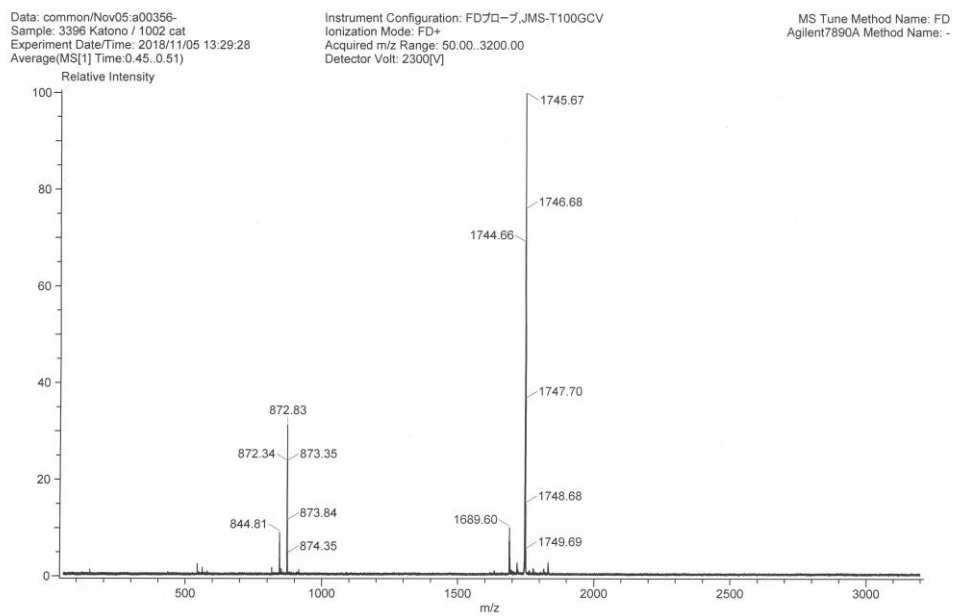
¹H NMR (400 MHz) spectrum of (±)-**8**, measured in chloroform-*d* at 303 K.



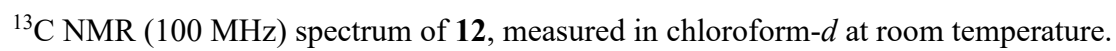
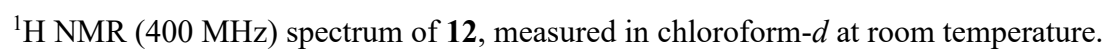
¹³C NMR (100 MHz) spectrum of (±)-**8**, measured in chloroform-*d* at room temperature.

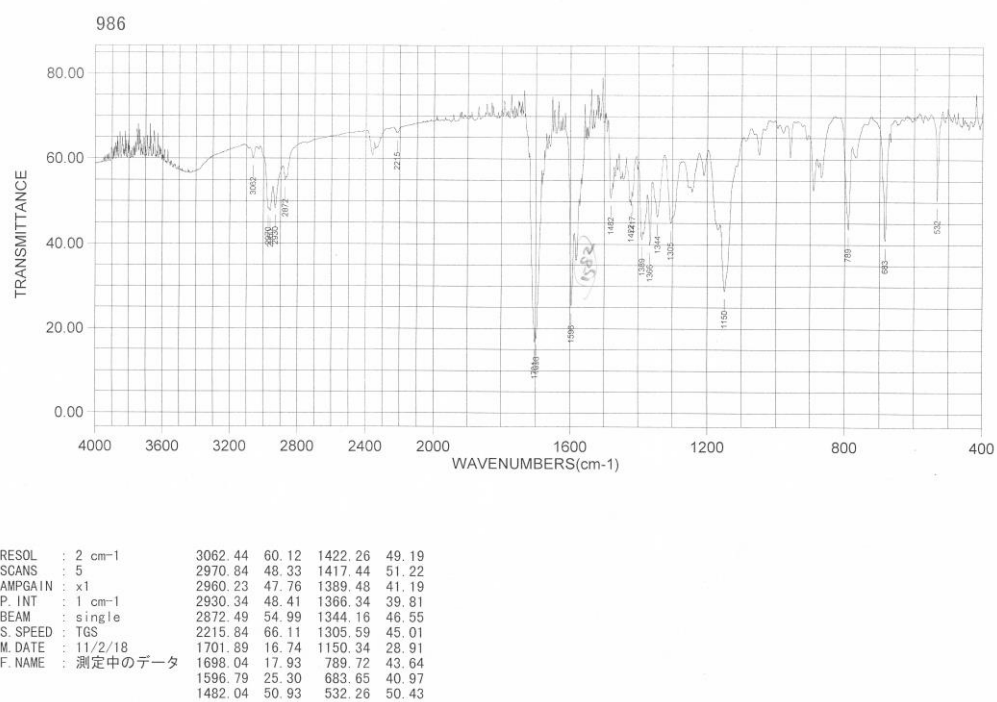


IR spectrum of (±)-8.

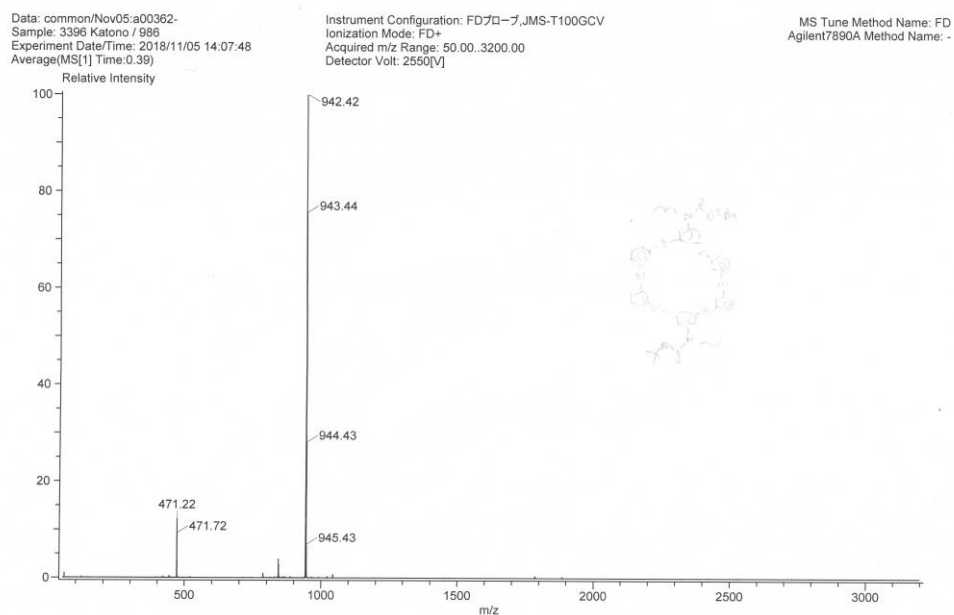


Mass spectrum (FD) of (±)-8.

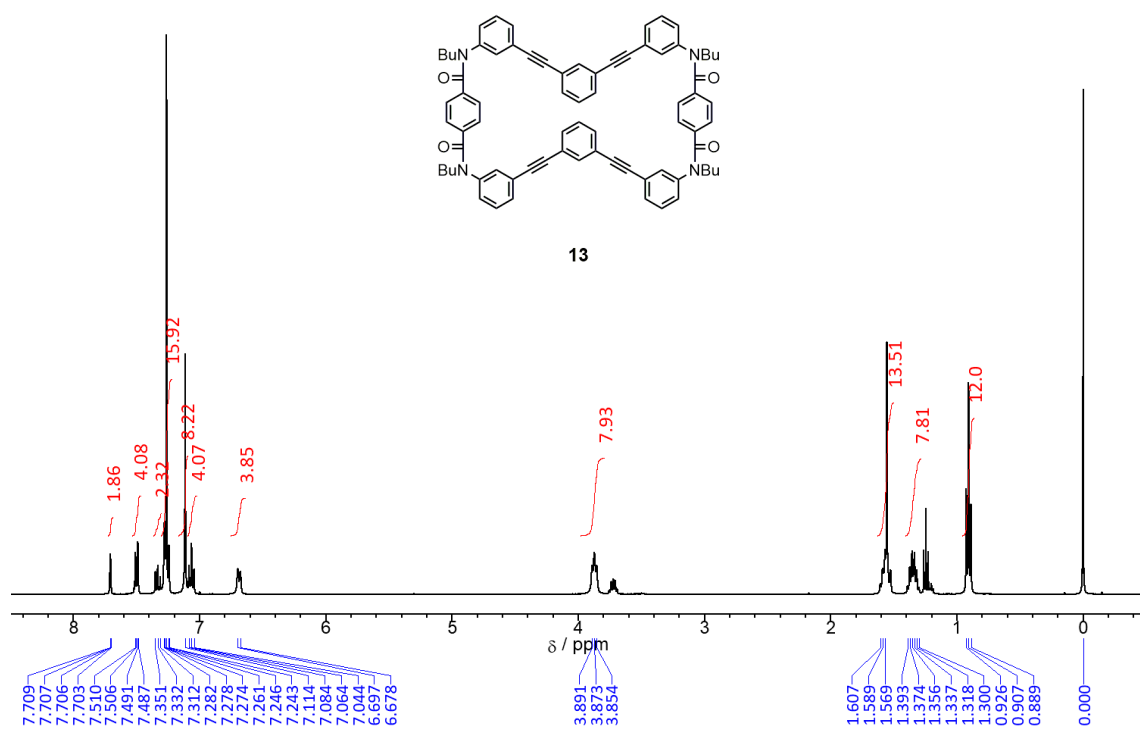




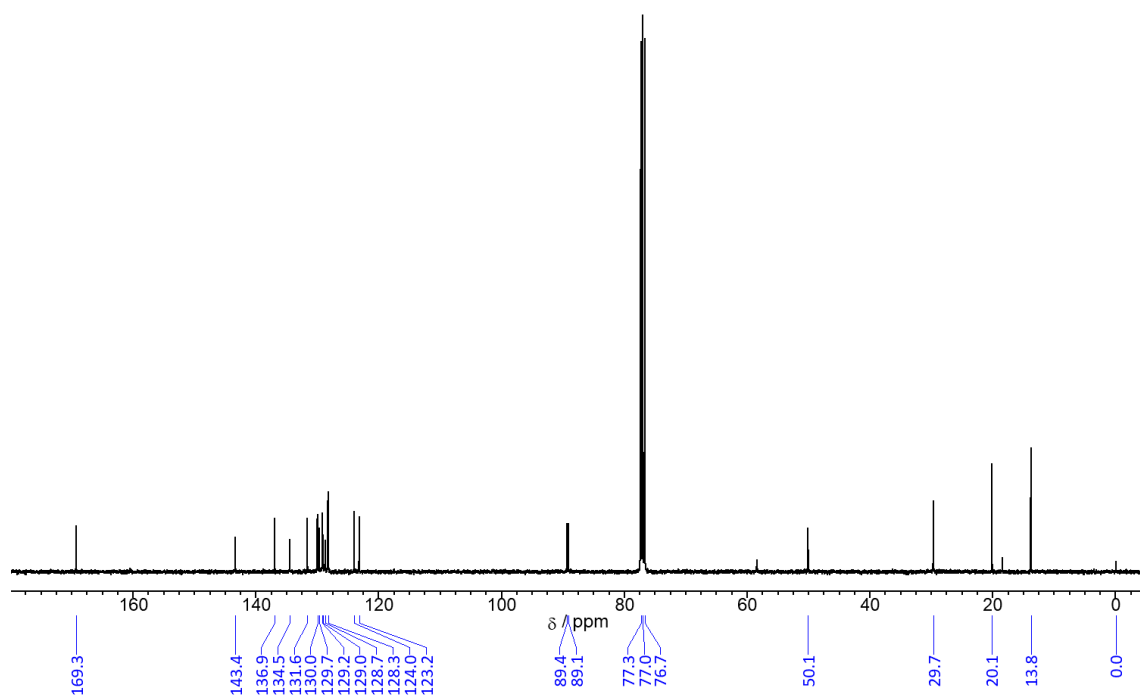
IR spectrum of **12**.



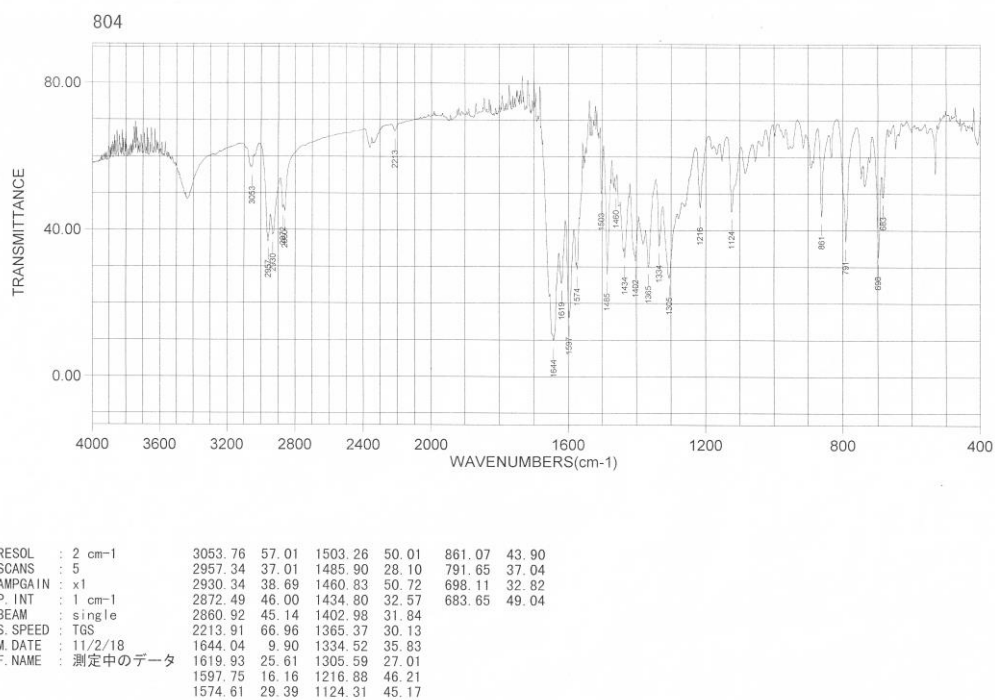
Mass spectrum (FD) of **12**.



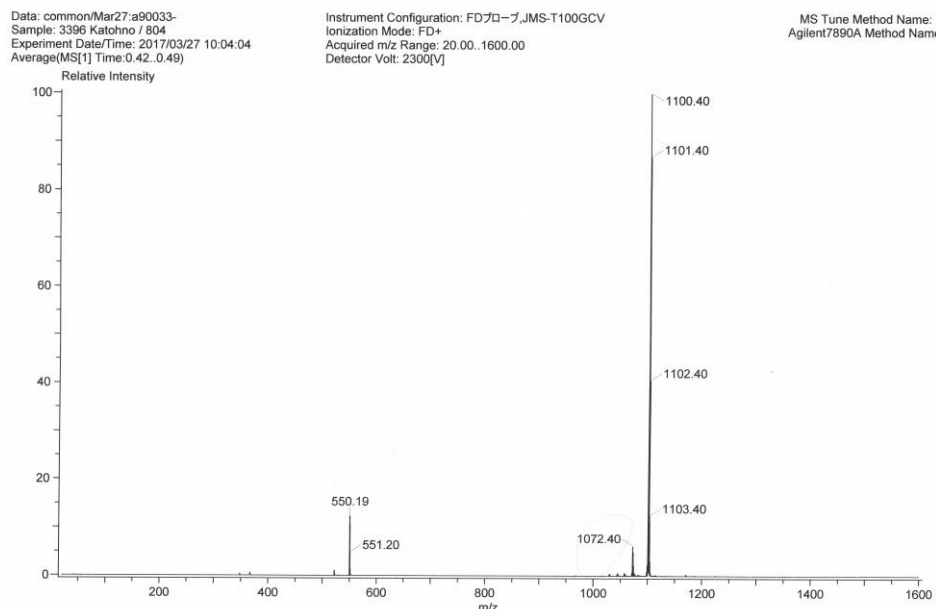
¹H NMR (400 MHz) spectrum of **13** cont. ethanol, measured in chloroform-*d* at room temperature.



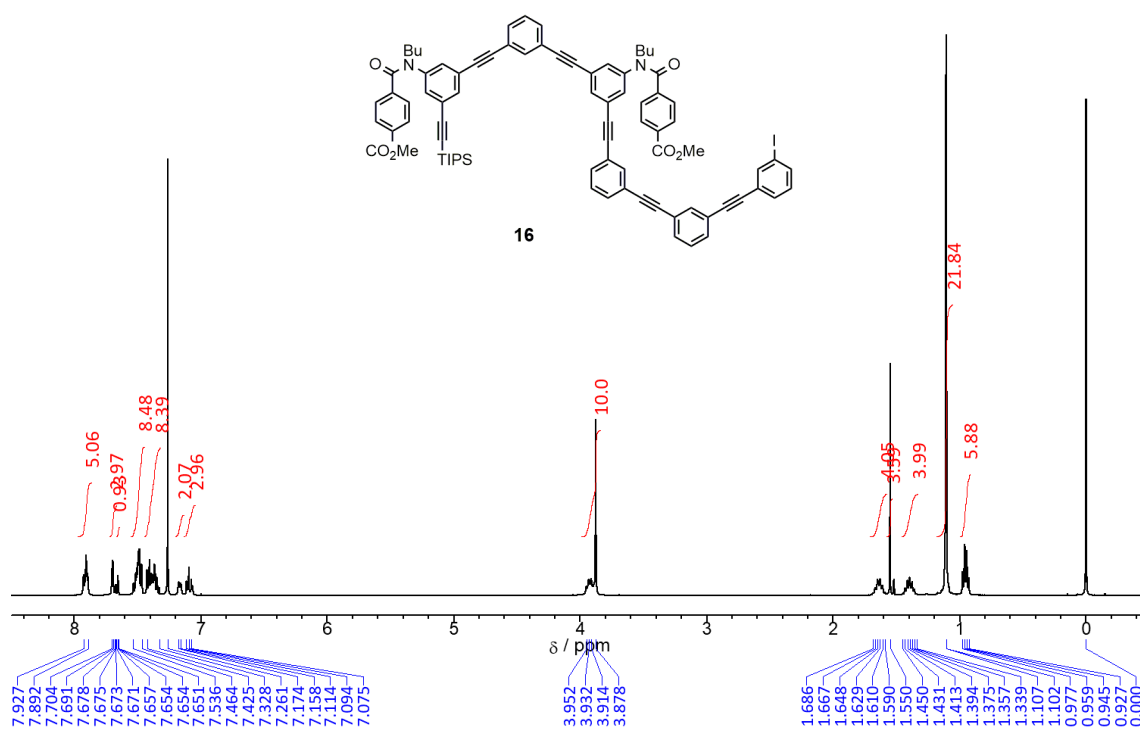
¹³C NMR (100 MHz) spectrum of **13** cont. ethanol, measured in chloroform-*d* at room temperature.



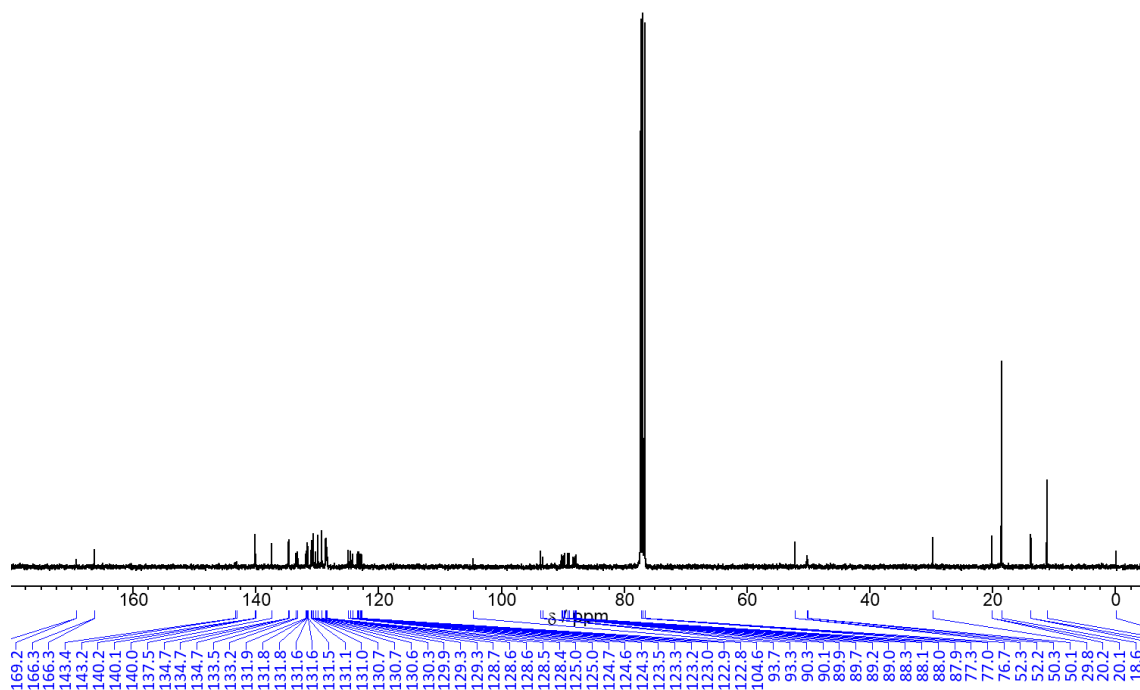
IR spectrum of **13**.



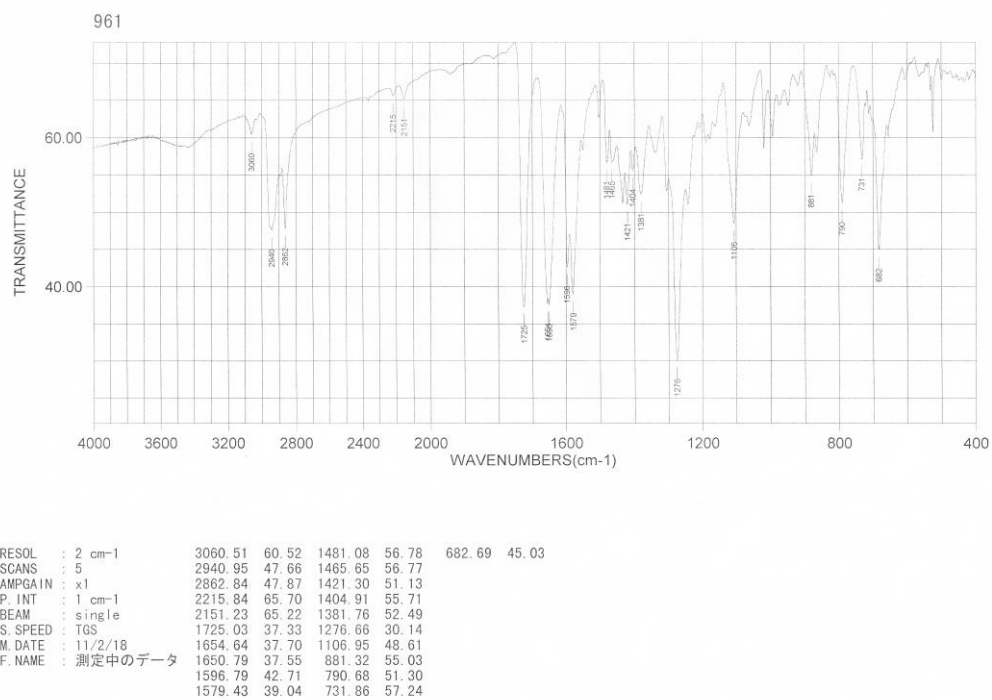
Mass spectrum (FD) of **13**.



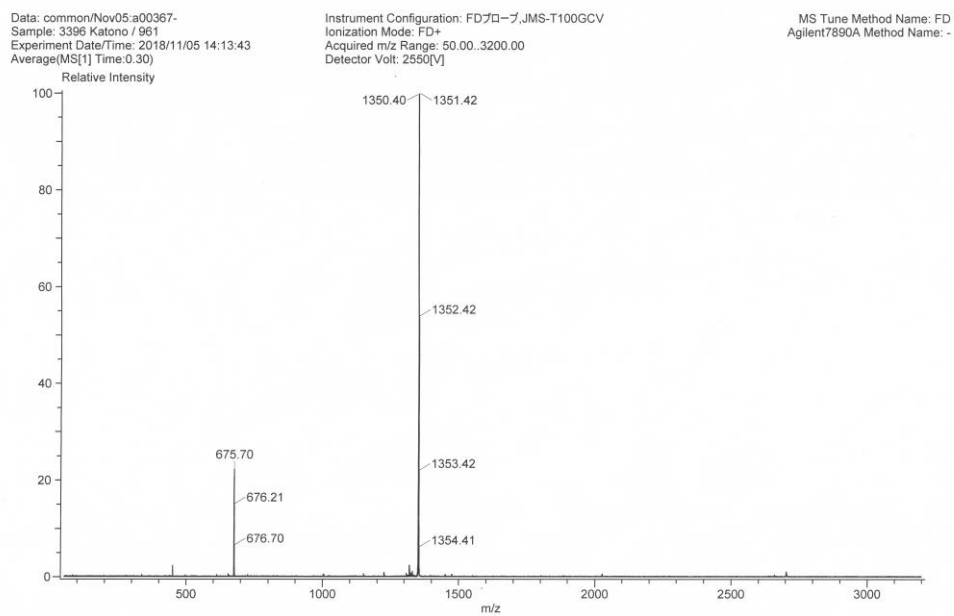
¹H NMR (400 MHz) spectrum of **16**, measured in chloroform-*d* at room temperature.



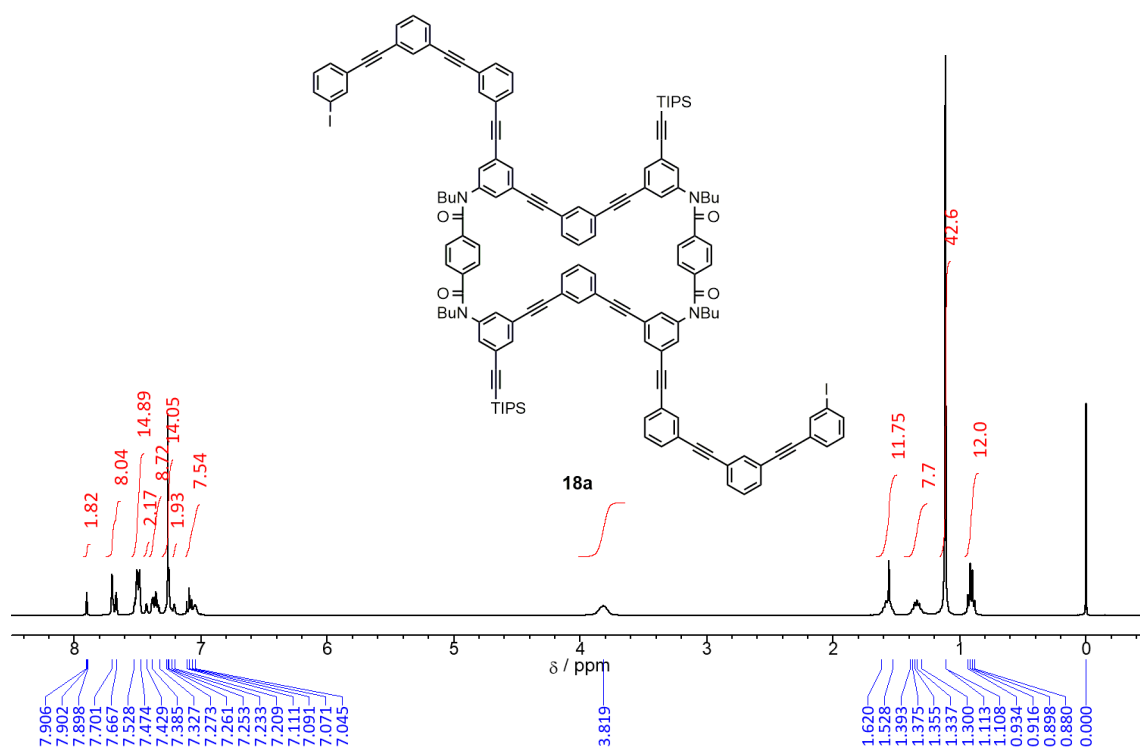
¹³C NMR (100 MHz) spectrum of **16**, measured in chloroform-*d* at room temperature.



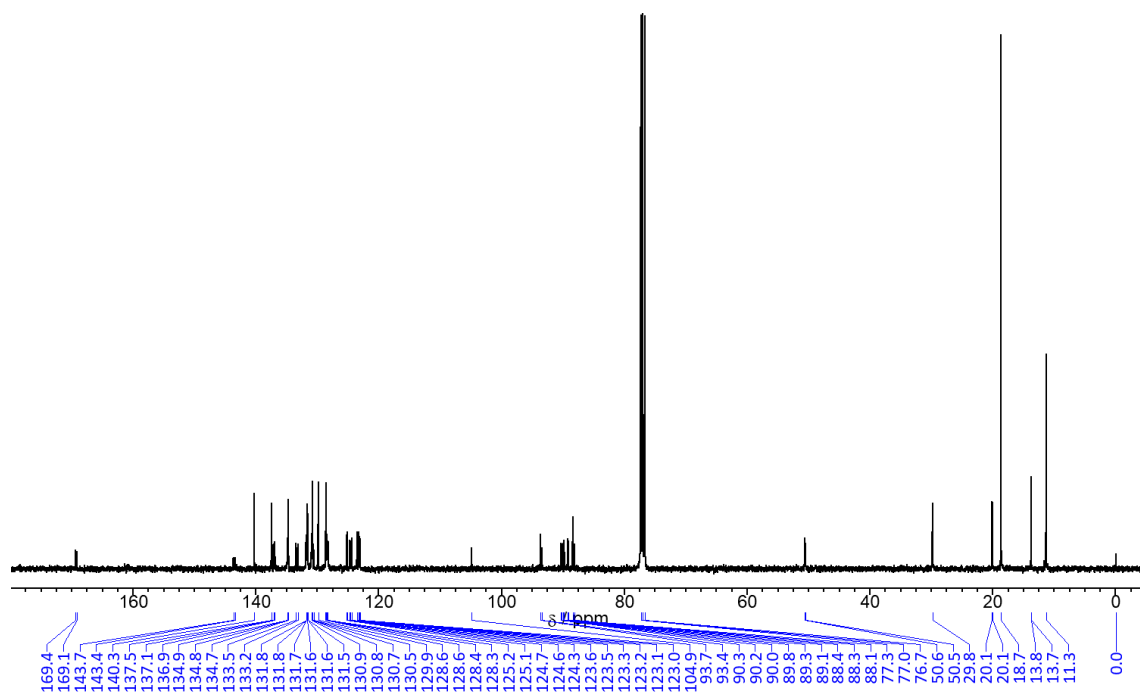
IR spectrum of 16.



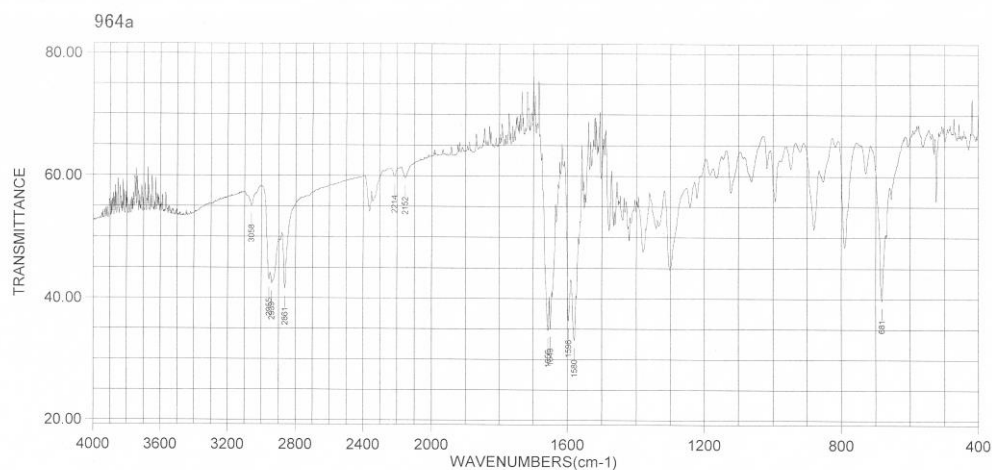
Mass spectrum (FD) of 16.



¹H NMR (400 MHz) spectrum of **18a**, measured in chloroform-*d* at room temperature.

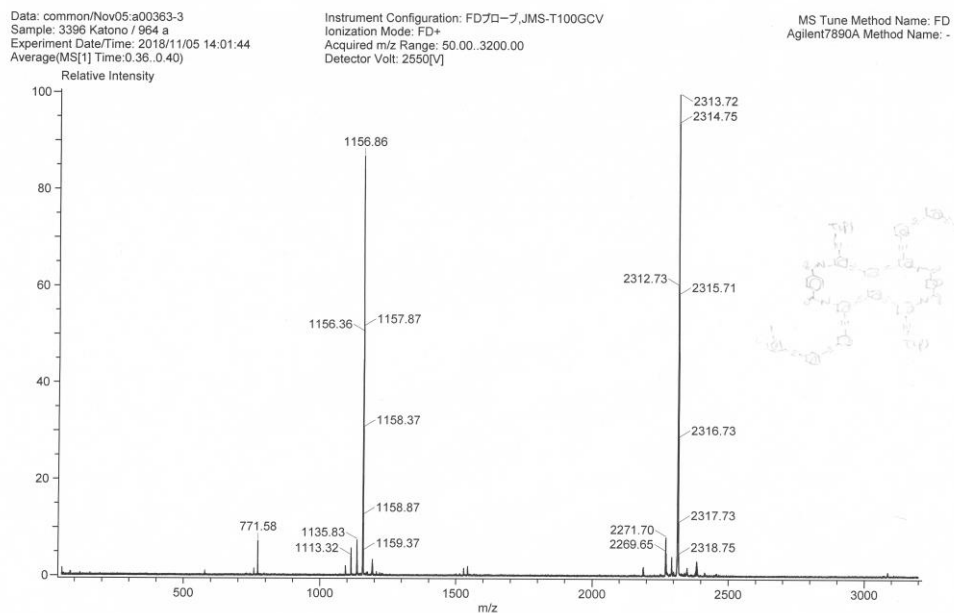


¹³C NMR (100 MHz) spectrum of **18a**, measured in chloroform-*d* at 313 K.

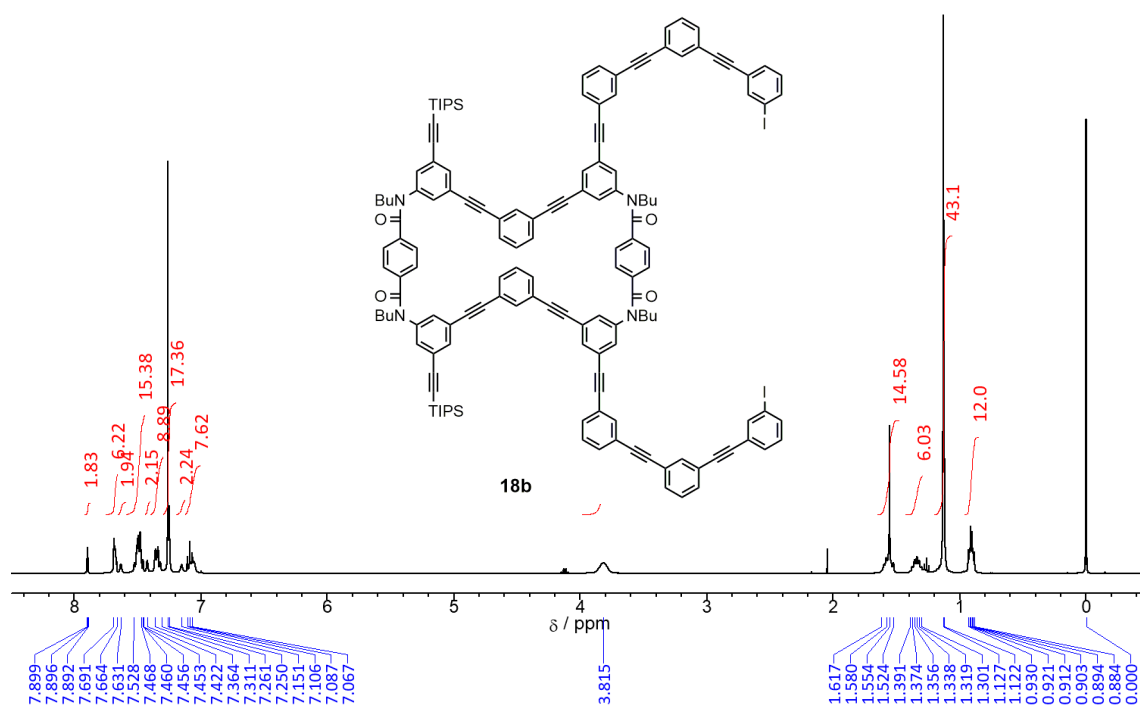


RESOL	: 2 cm-1	3058.59	55.16	681.72	39.70
SCANS	: 5	2955.41	43.06		
AMPGAIN	: x1	2939.02	42.42		
P. INT	: 1 cm-1	2861.88	41.55		
BEAM	: single	2214.87	59.94		
S. SPEED	: TGS	2152.20	59.71		
M. DATE	: 11/2/18	1656.57	34.77		
F. NAME	: 測定中のデータ	1649.82	34.99		
		1596.79	36.35		
		1580.40	33.13		

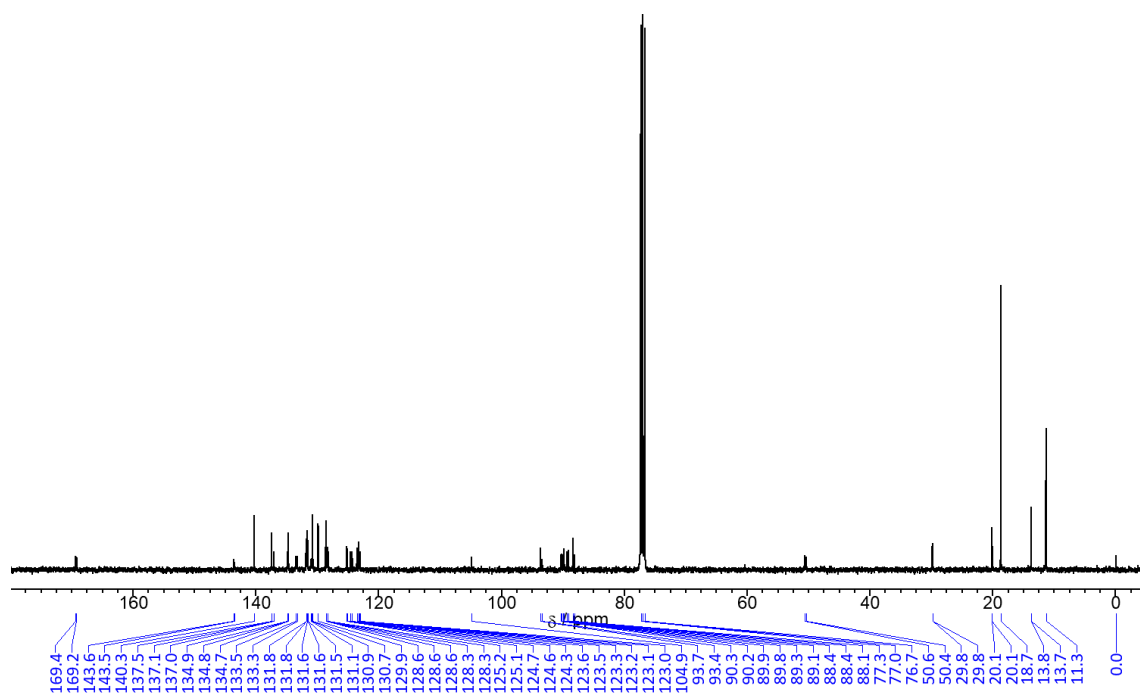
IR spectrum of **18a**.



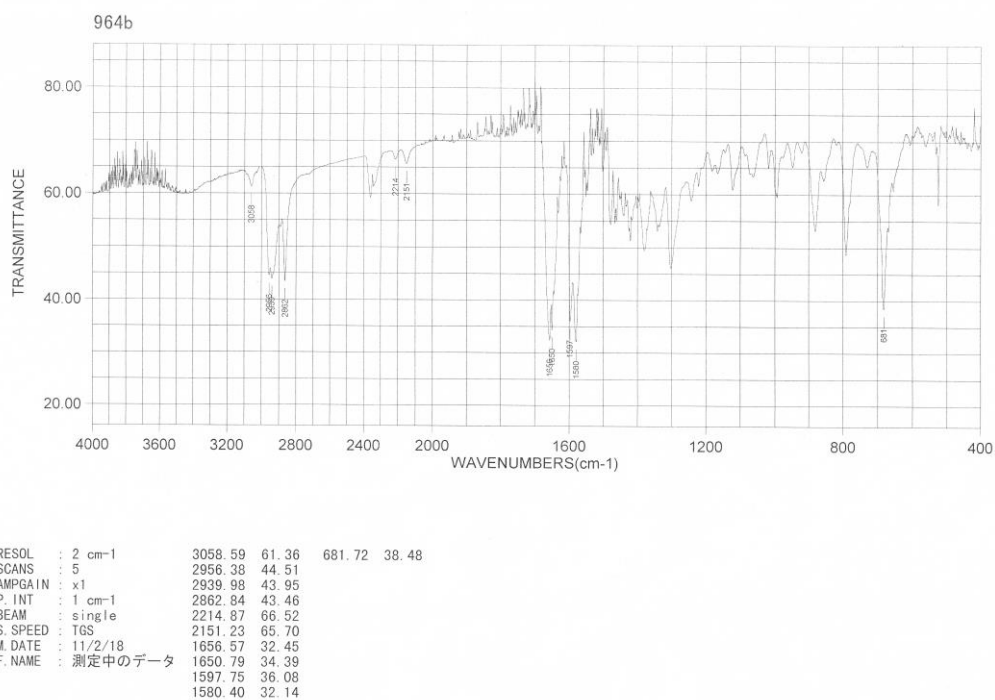
Mass spectrum (FD) of **18a**.



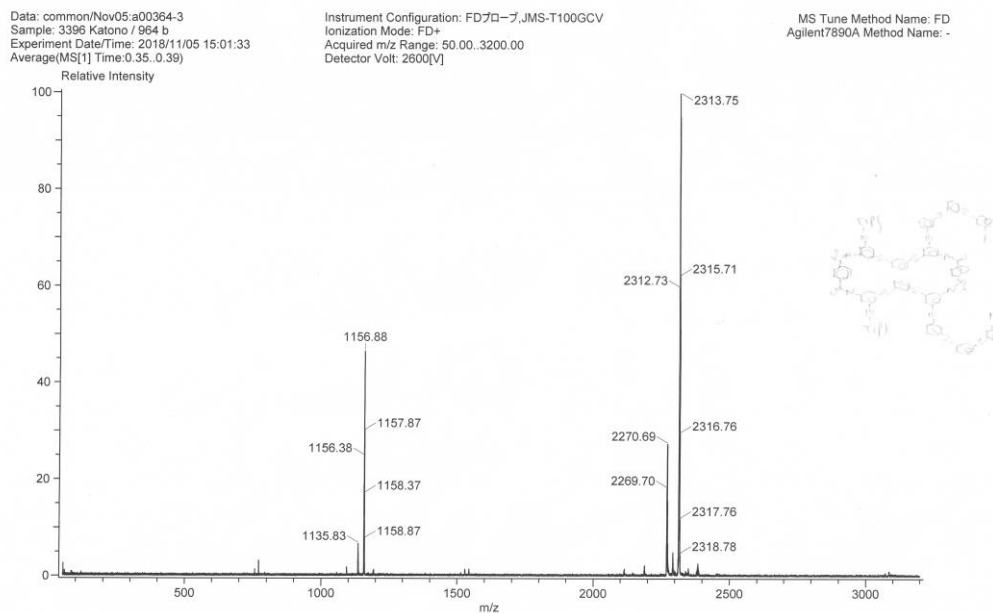
¹H NMR (400 MHz) spectrum of **18b** cont. residual solvent (ethyl acetate), measured in chloroform-*d* at room temperature.



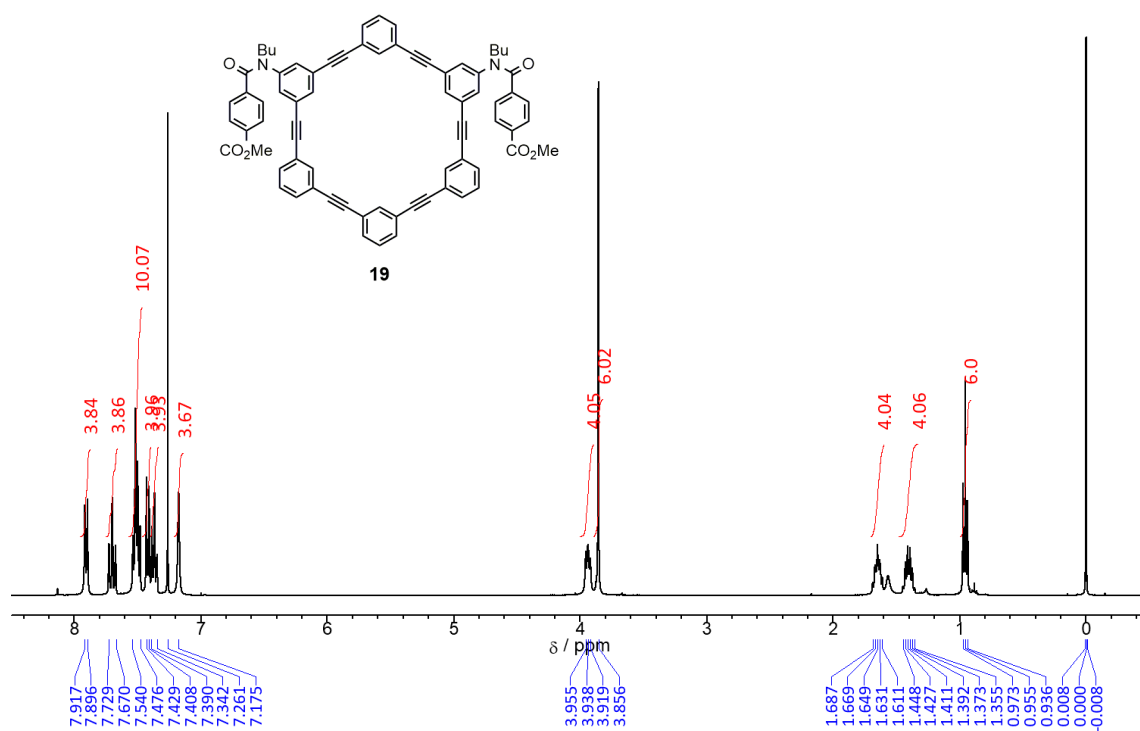
¹³C NMR (100 MHz) spectrum of **18b**, measured in chloroform-*d* at 313 K.



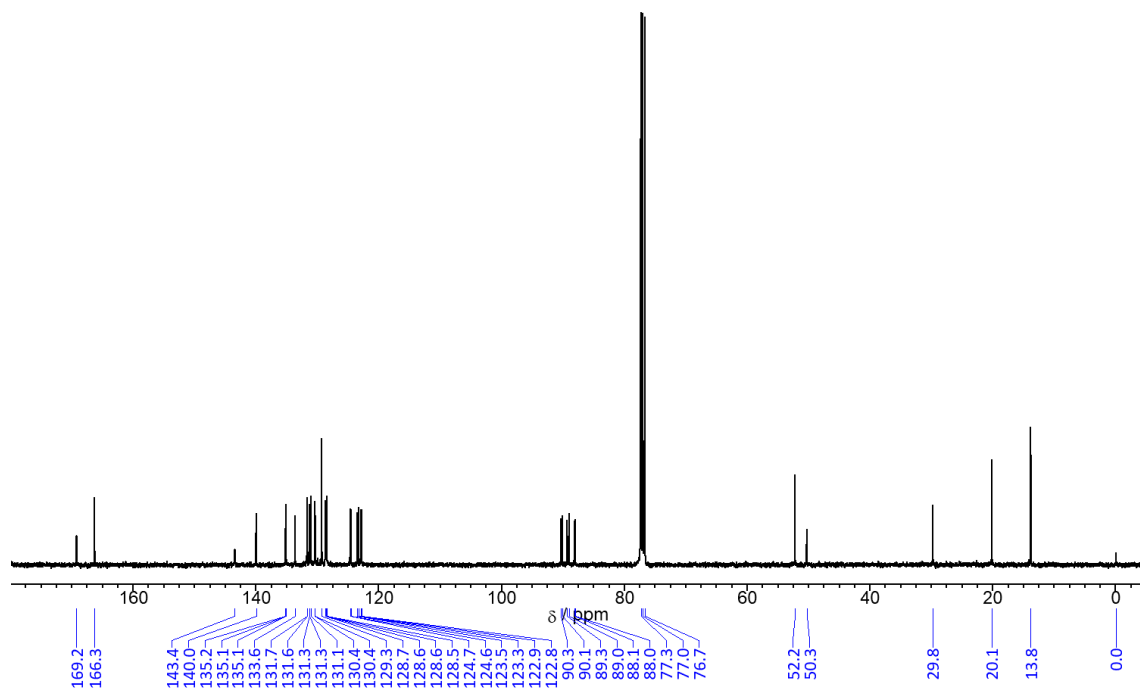
IR spectrum of **18b**.



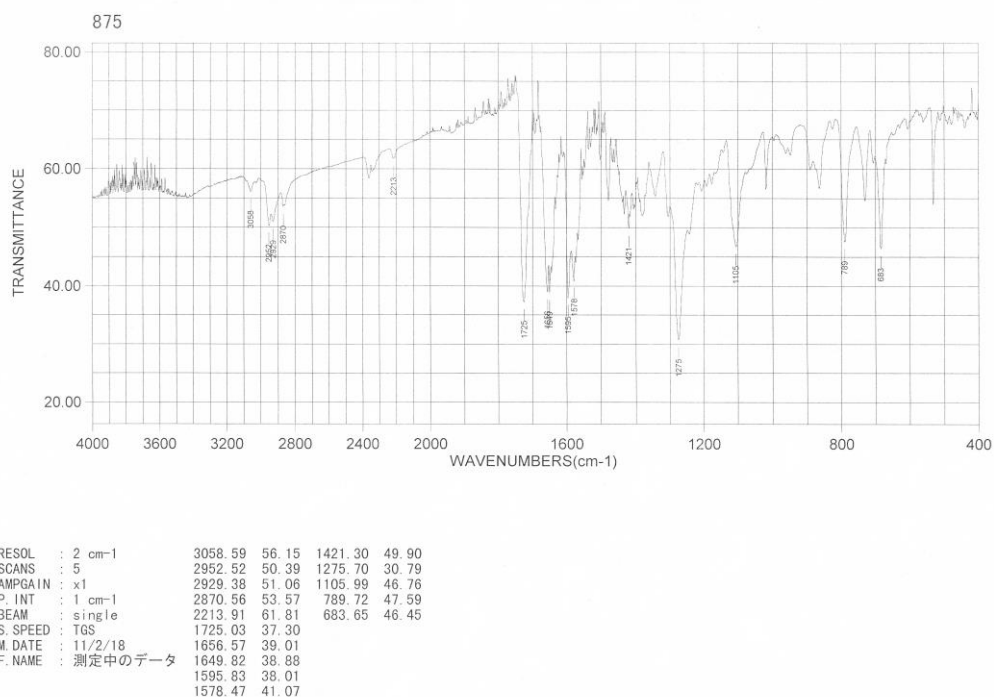
Mass spectrum (FD) of **18b**.



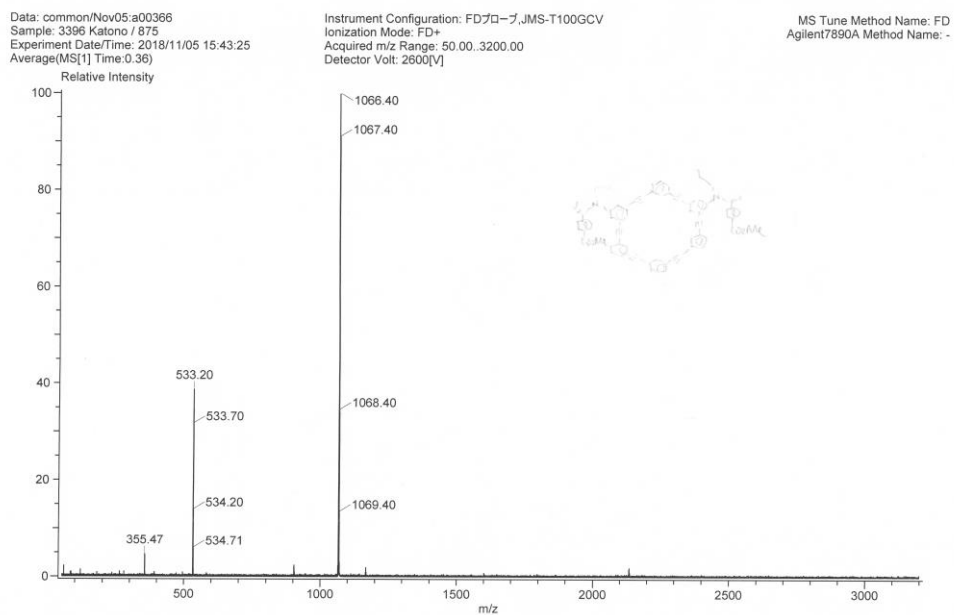
^1H NMR (400 MHz) spectrum of **19**, measured in chloroform-*d* at room temperature.



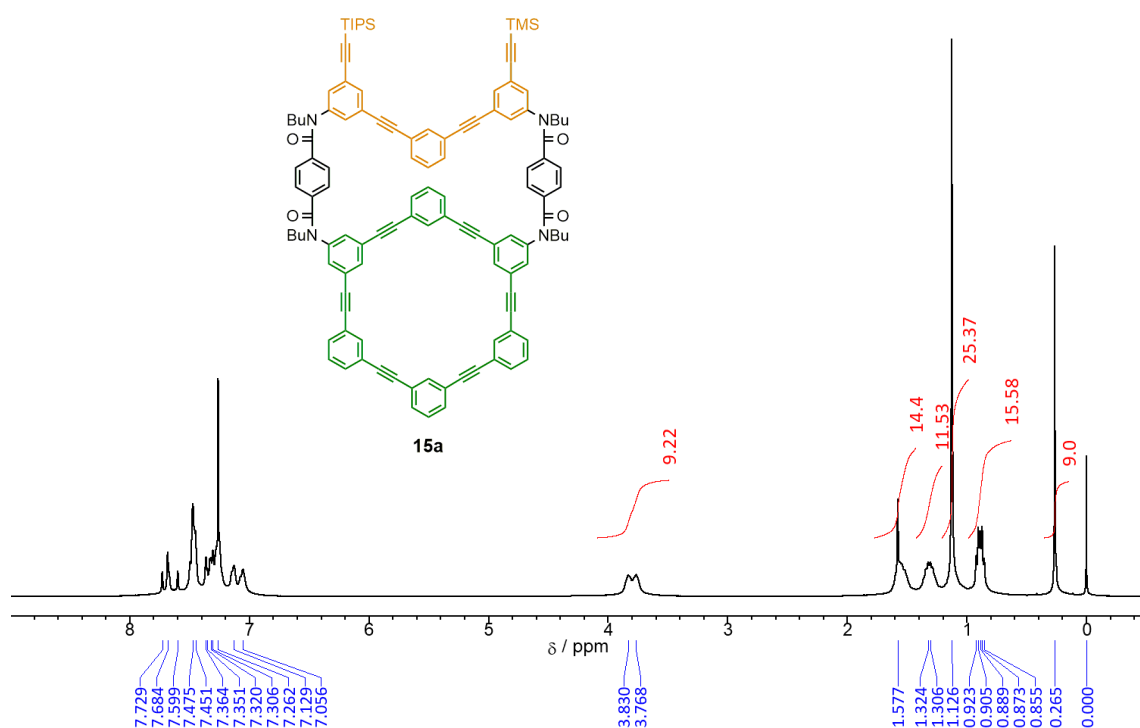
^{13}C NMR (100 MHz) spectrum of **19**, measured in chloroform-*d* at room temperature.



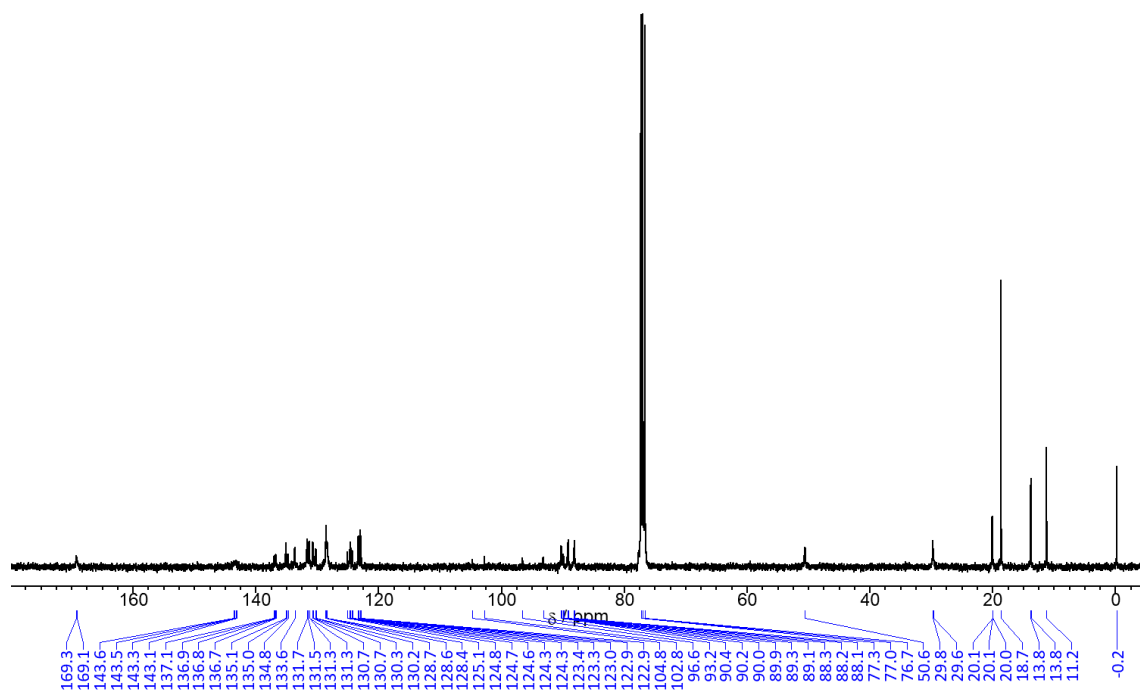
IR spectrum of **19**.



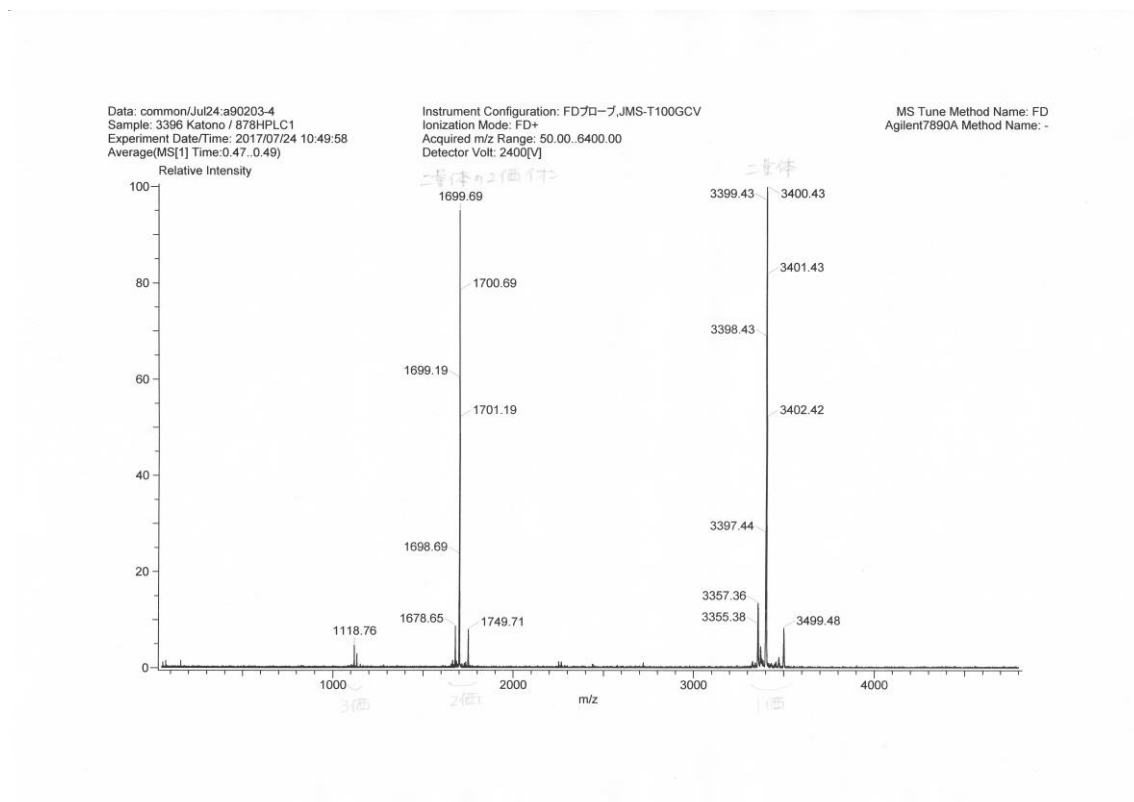
Mass spectrum (FD) of **19**.



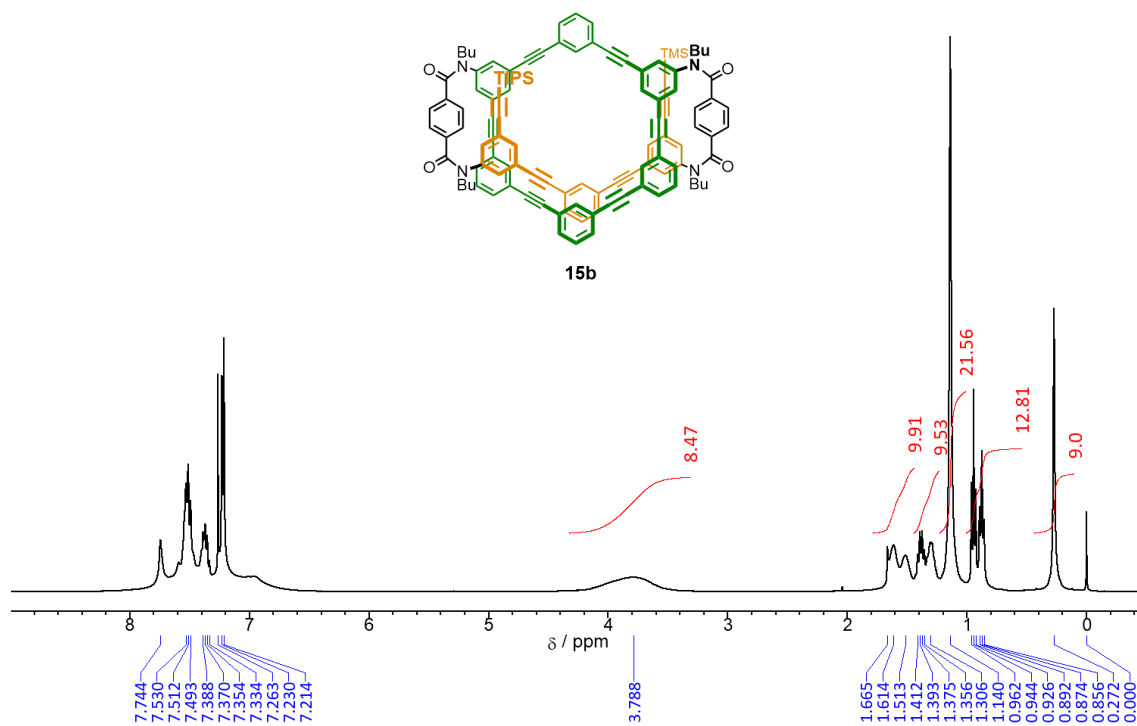
¹H NMR (400 MHz) spectrum of **15a**, measured in chloroform-*d* at room temperature.



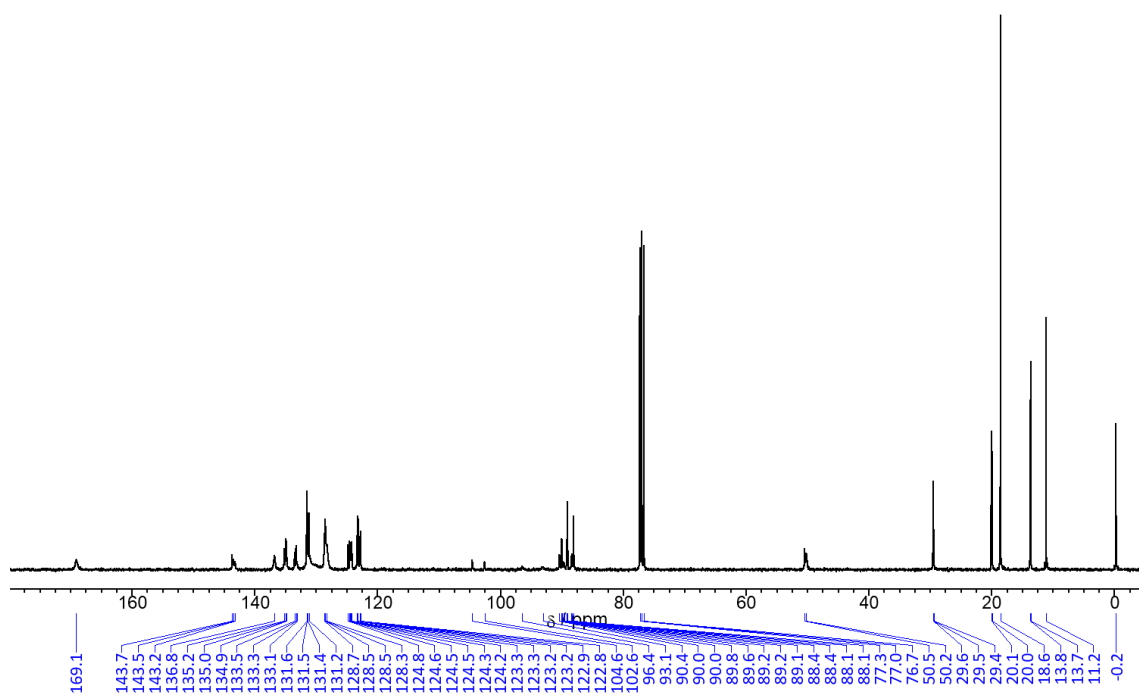
¹³C NMR (100 MHz) spectrum of **15a**, measured in chloroform-*d* at room temperature.



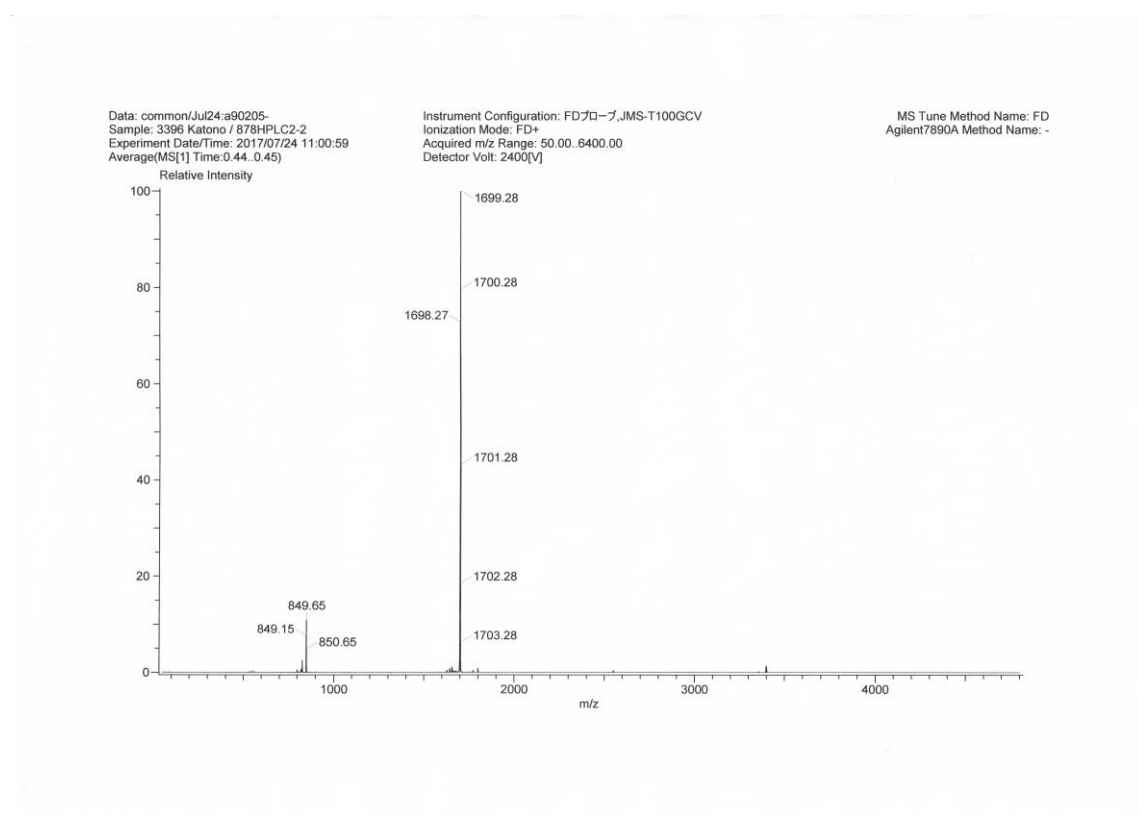
Mass spectrum (FD) of **15a**.



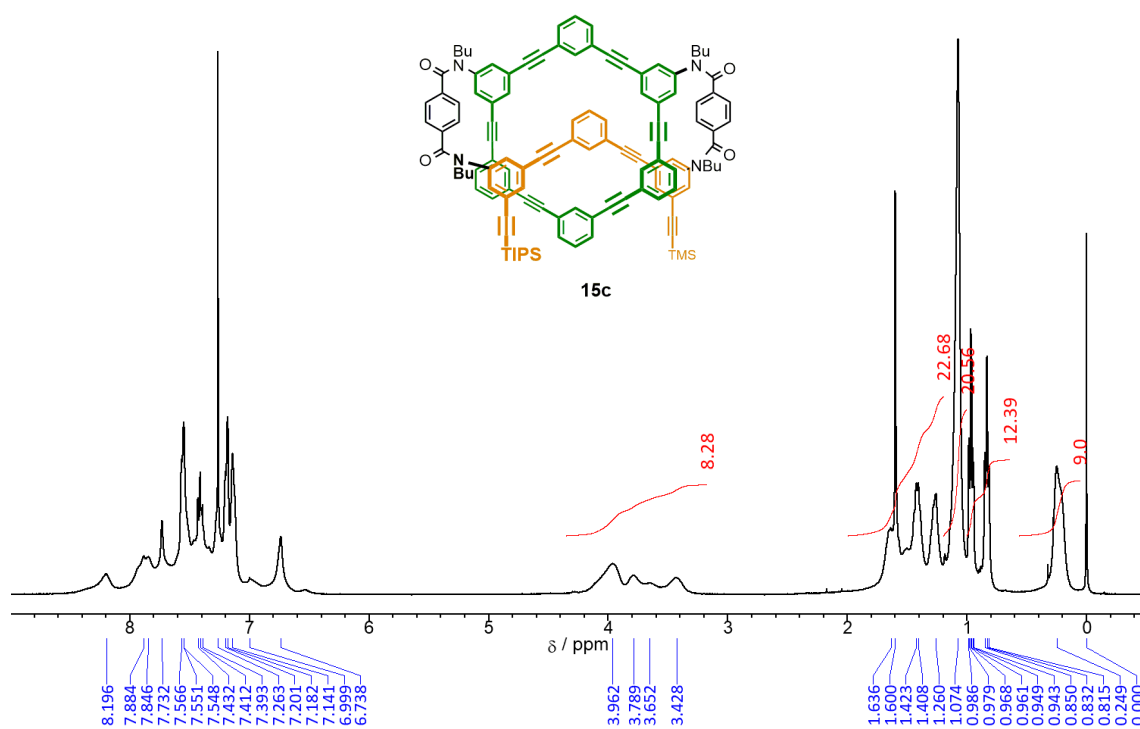
^1H NMR (400 MHz) spectrum of **15b**, measured in chloroform-*d* at room temperature.



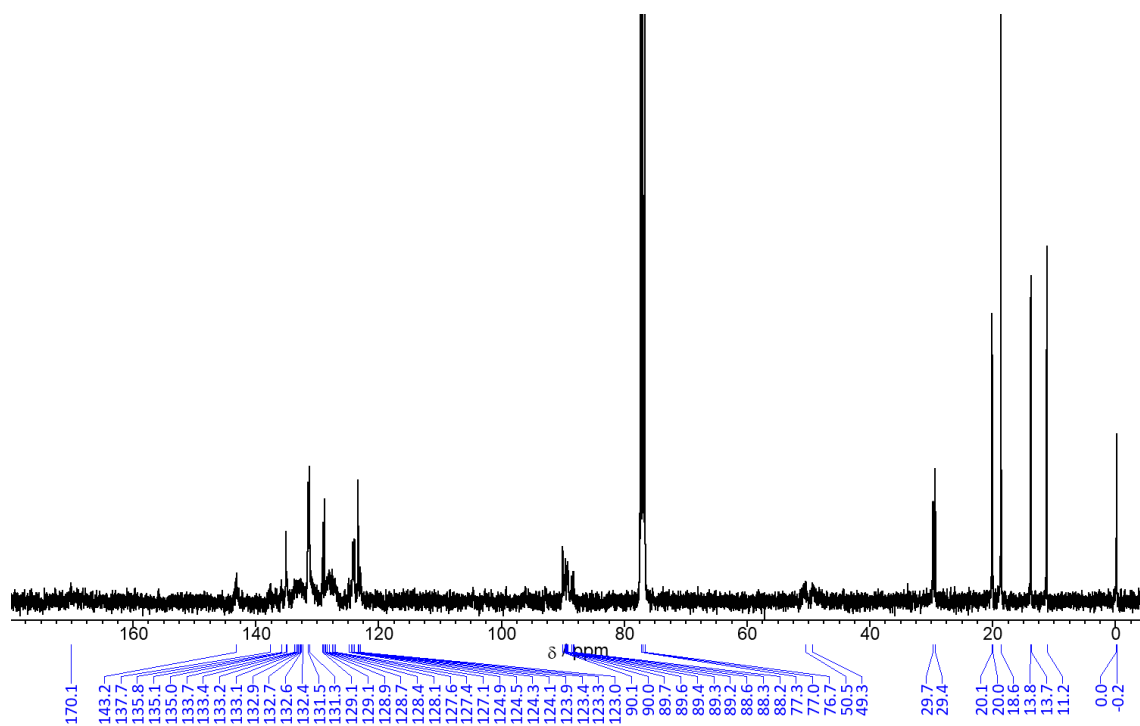
^{13}C NMR (100 MHz) spectrum of **15b**, measured in chloroform-*d* at room temperature.



Mass spectrum (FD) of **15b**.



¹H NMR (400 MHz) spectrum of **15c**, measured in chloroform-*d* at room temperature.

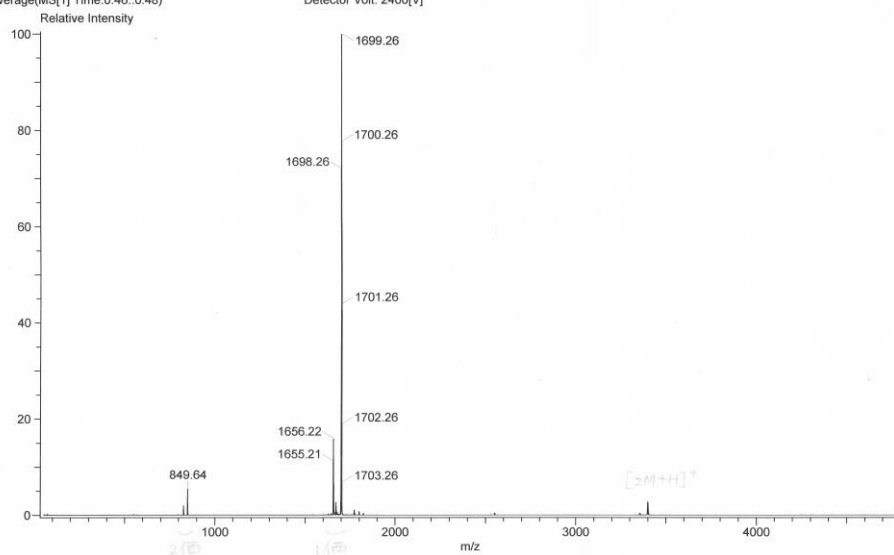


¹³C NMR (100 MHz) spectrum of **15c**, measured in chloroform-*d* at room temperature.

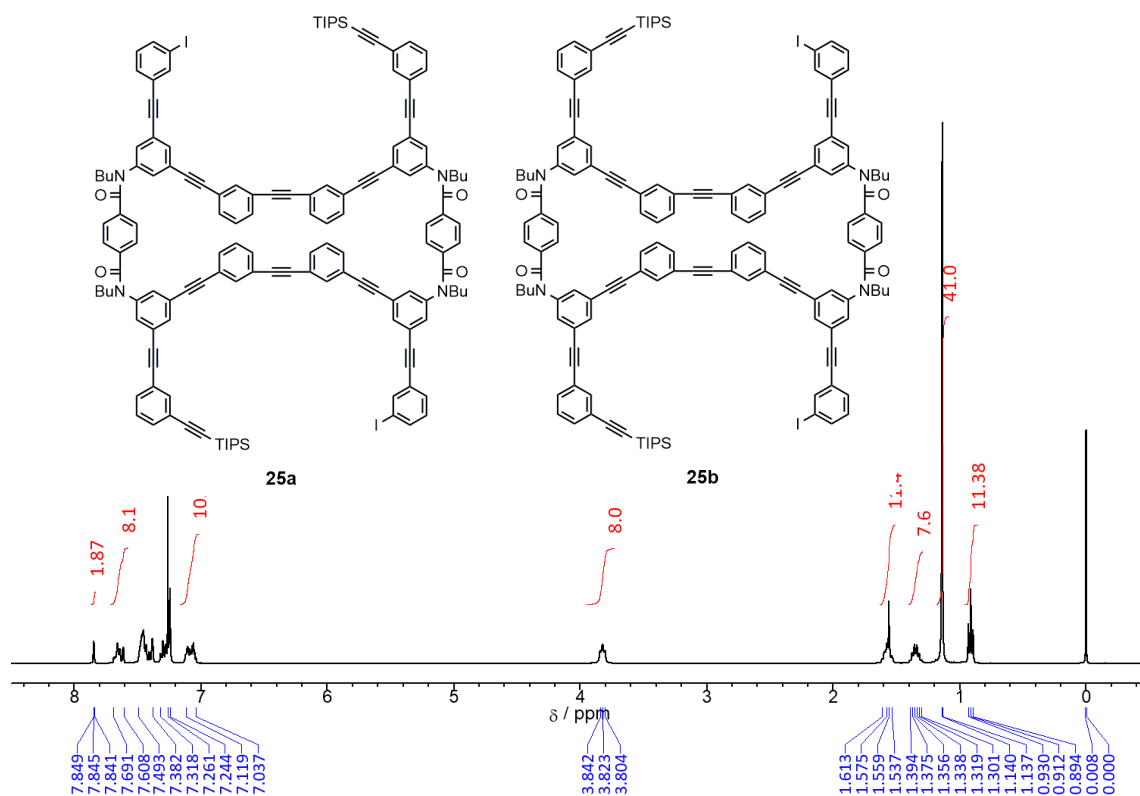
Data: common/Jul24-s90204-
Sample: 3396 Katono / 878HPLC2-1
Experiment Date/Time: 2017/07/24 10:54:58
Average(MS[1] Time:0.46..0.48)

Instrument Configuration: FDプローブ,JMS-T100GCV
Ionization Mode: FD+
Acquired m/z Range: 50.00..6400.00
Detector Volt: 2400[V]

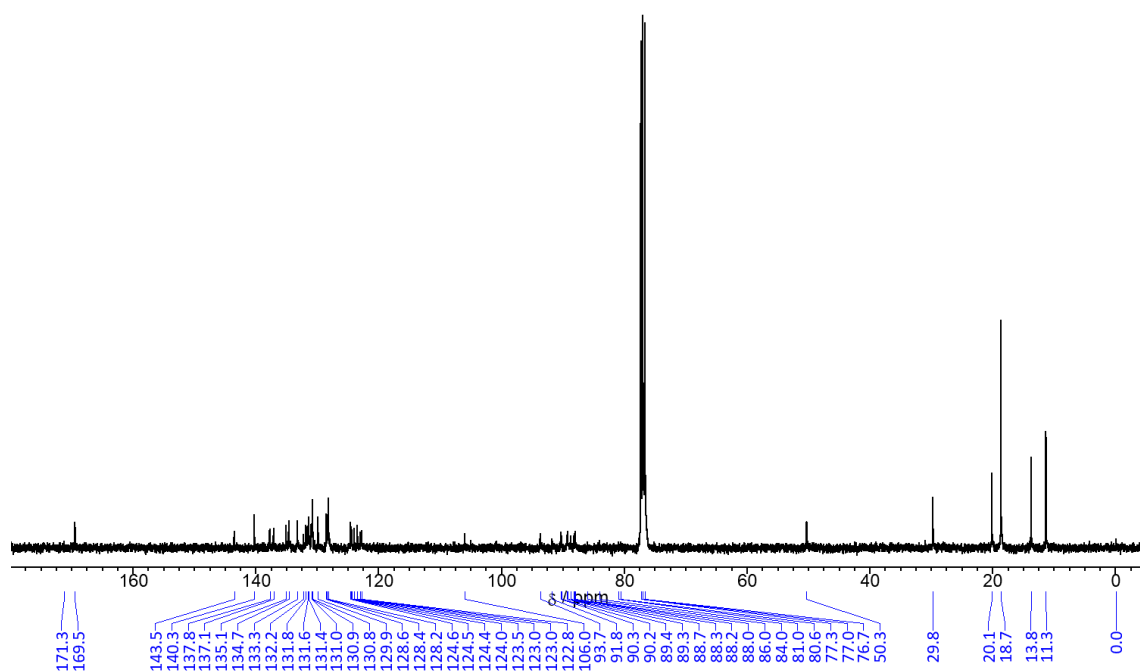
MS Tune Method Name: FD
Agilent7890A Method Name: -



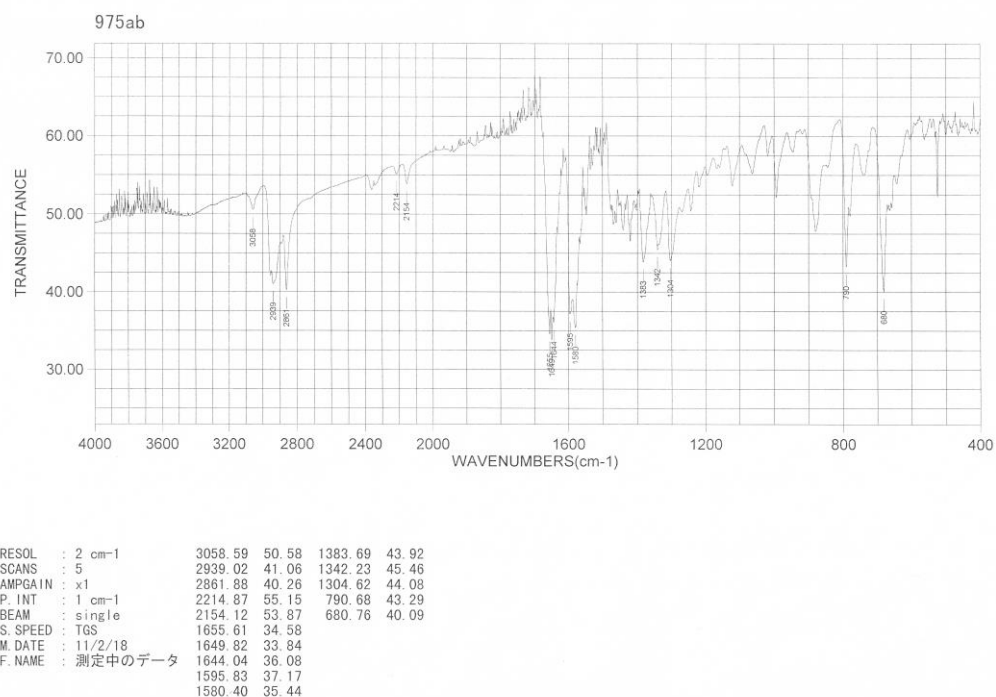
Mass spectrum (FD) of **15c**.



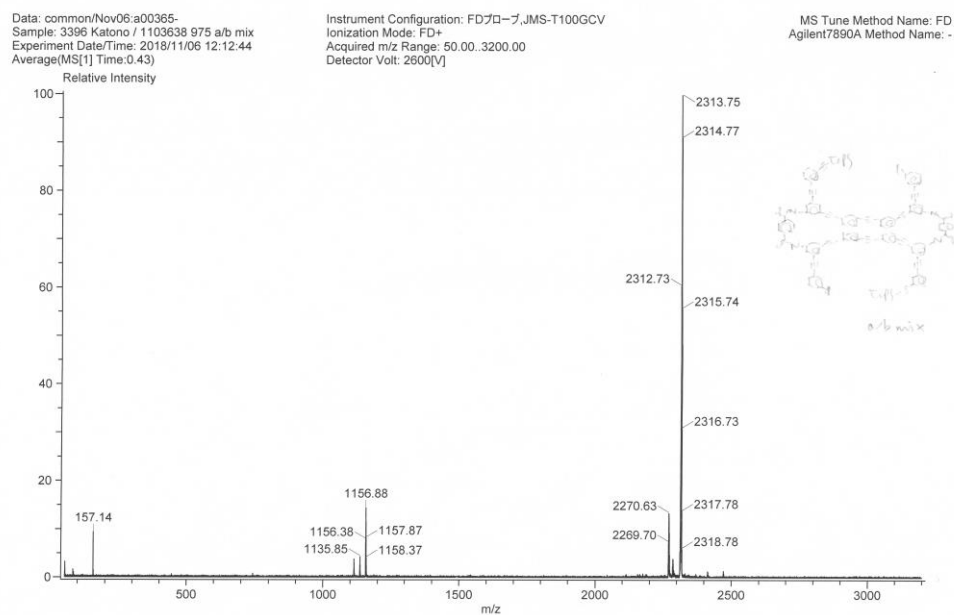
^1H NMR (400 MHz) spectrum of a mixture of **25a** and **25b**, measured in chloroform-*d* at room temperature.



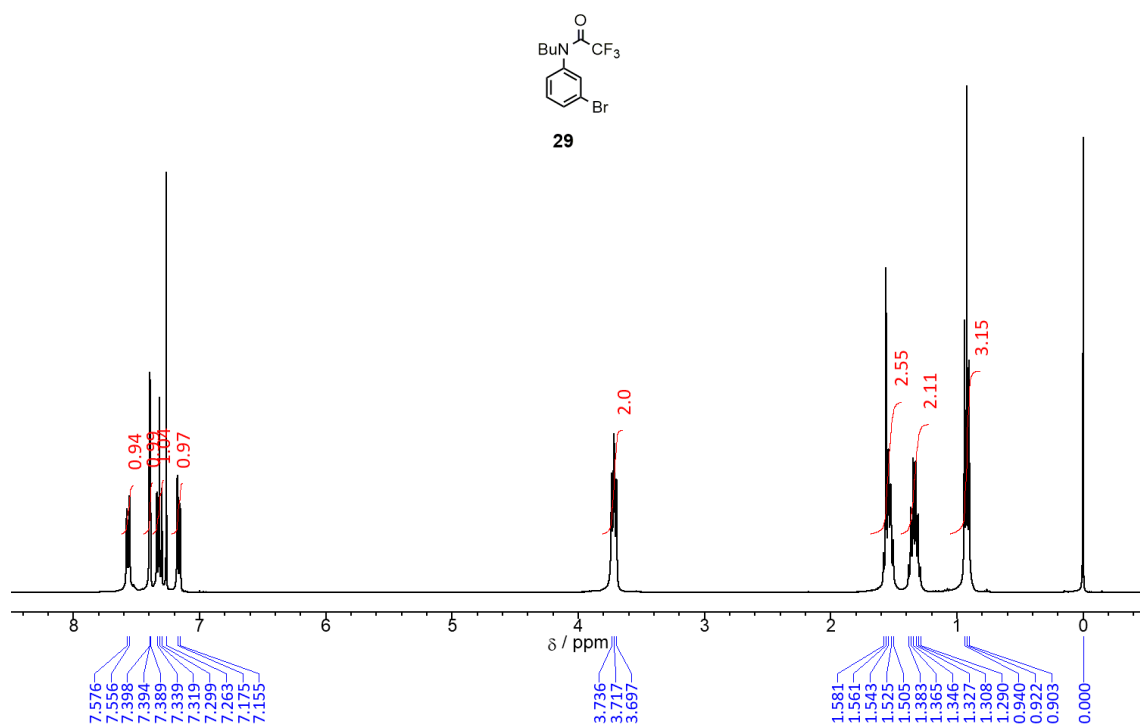
^{13}C NMR (100 MHz) spectrum of a mixture of **25a** and **25b**, measured in chloroform-*d* at 308 K.



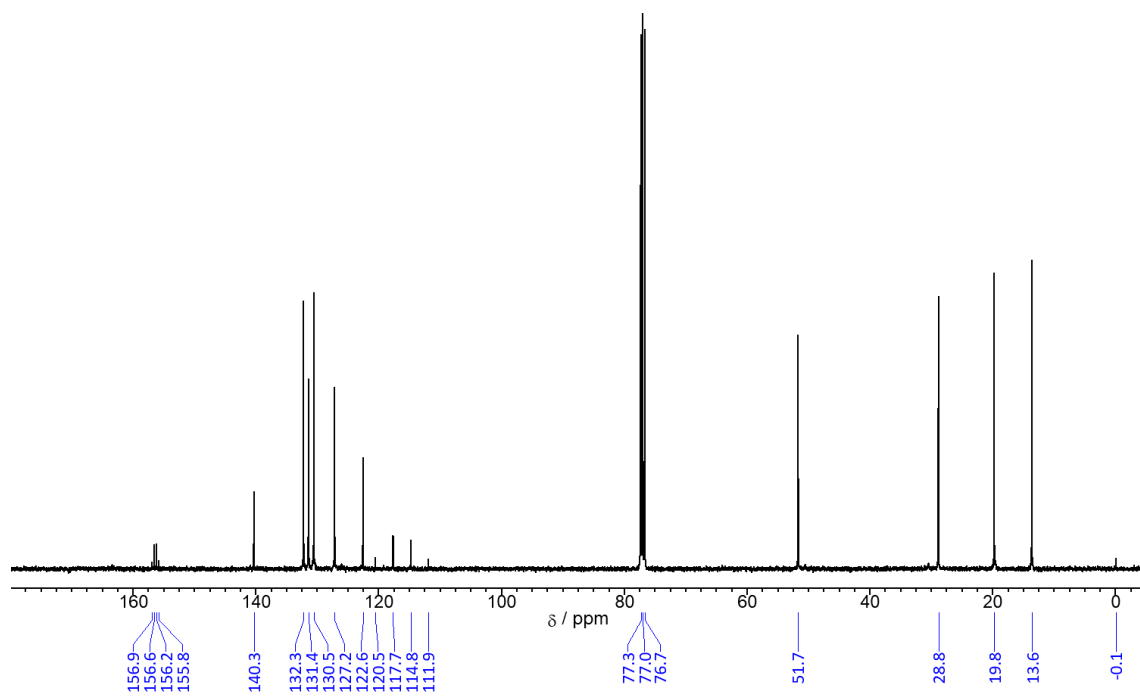
IR spectrum of a mixture of **25a** and **25b**.



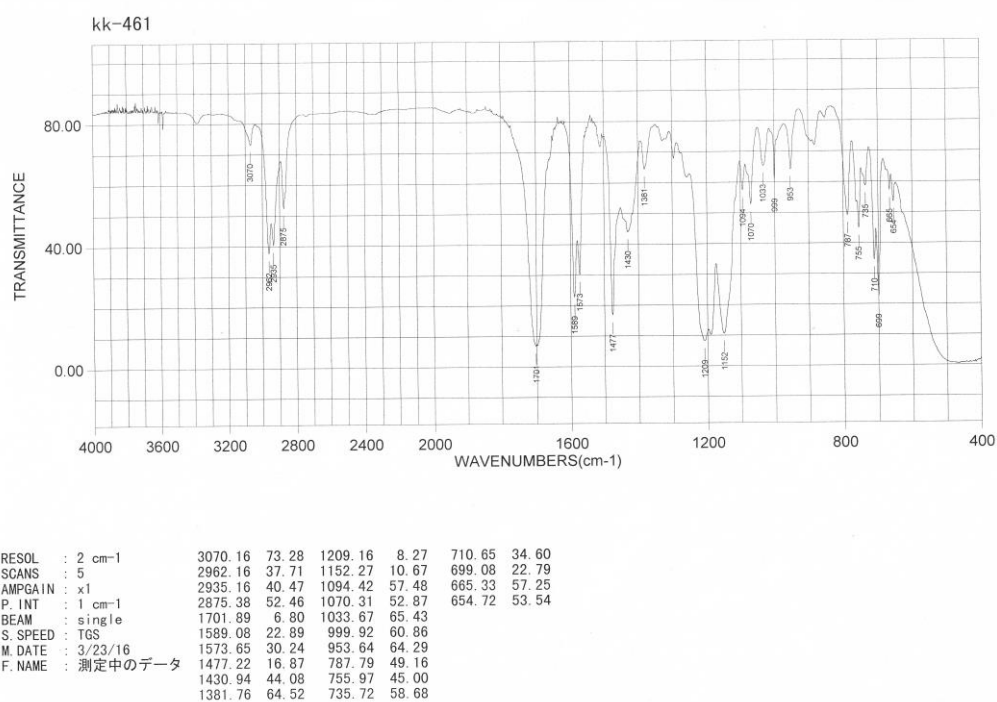
Mass spectrum (FD) of a mixture of **25a** and **25b**.



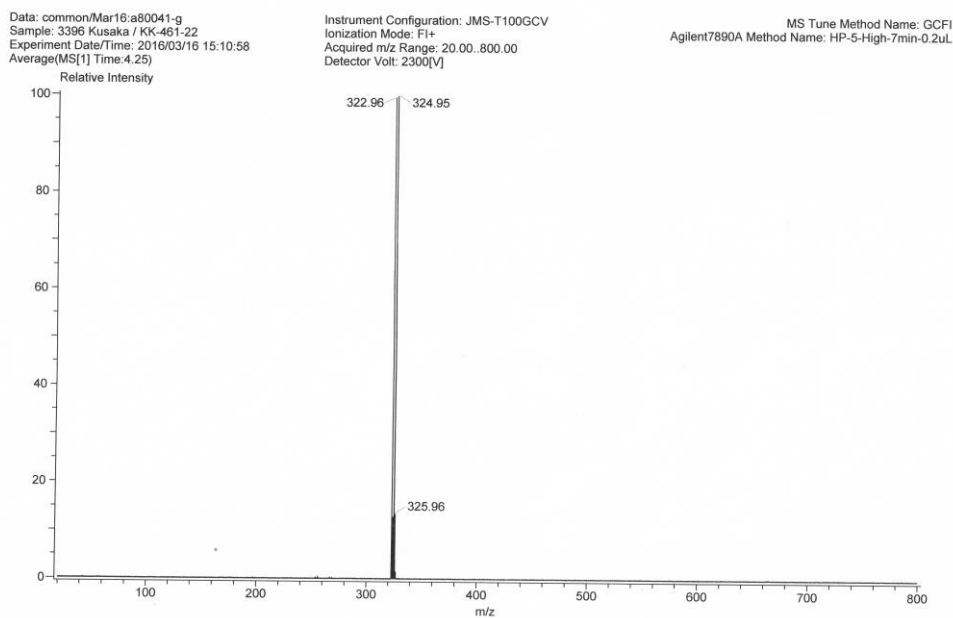
^1H NMR (400 MHz) spectrum of **29**, measured in chloroform-*d* at room temperature.



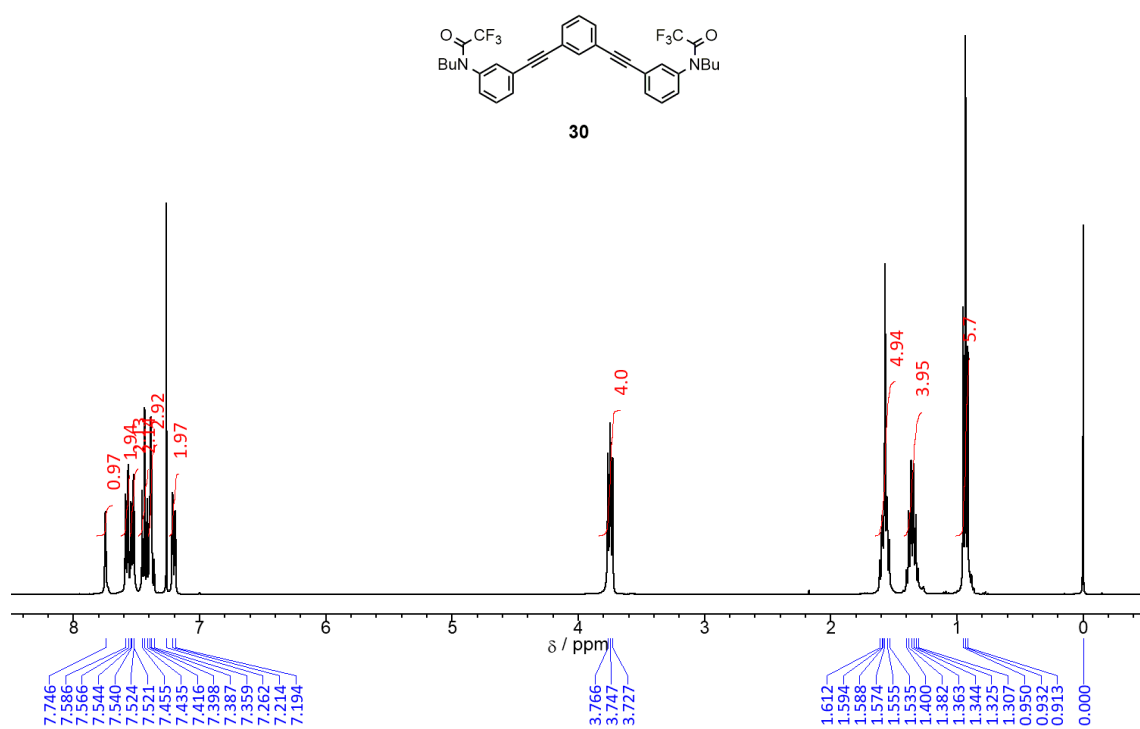
^{13}C NMR (100 MHz) spectrum of **29**, measured in chloroform-*d* at room temperature.



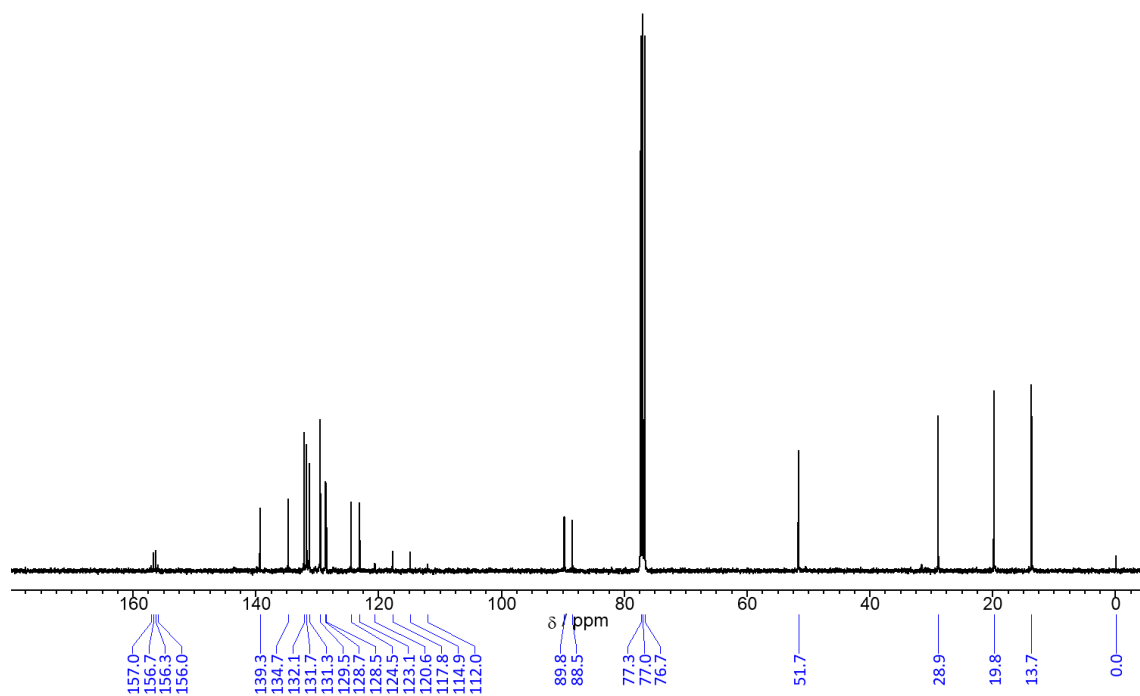
IR spectrum of **29**.



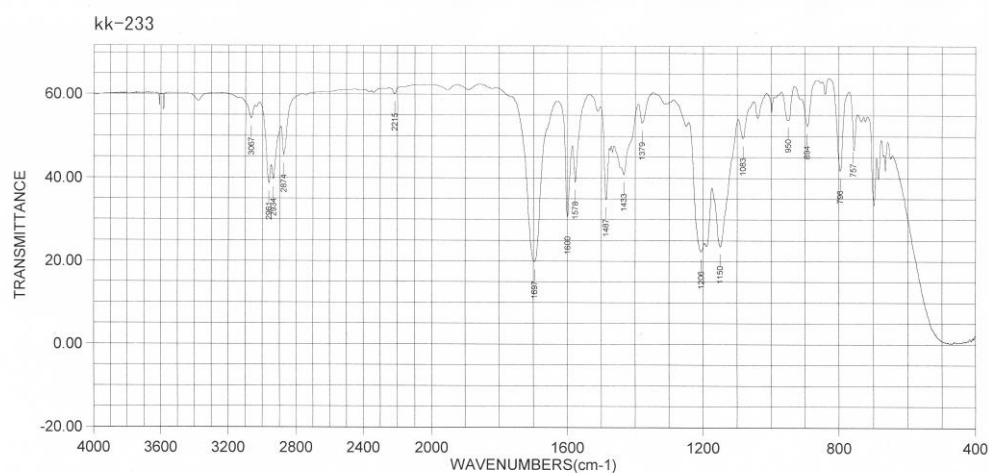
Mass spectrum (FD) of **29**.



^1H NMR (400 MHz) spectrum of **30**, measured in chloroform-*d* at room temperature.

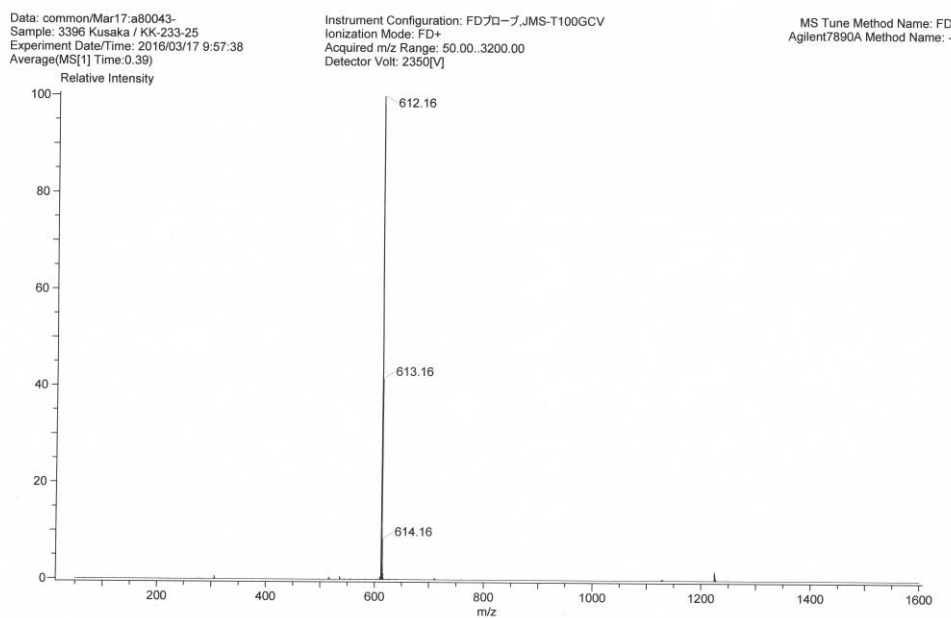


^{13}C NMR (100 MHz) spectrum of **30**, measured in chloroform-*d* at room temperature.



RESOL	: 2	cm-1	3067.26	54.15	1379.83	53.19
SCANS	: 5		2961.20	38.66	1206.27	22.34
AMPGAIN	: x1		2934.20	39.61	1150.34	23.59
P. INT	: 1	cm-1	2874.41	45.30	1083.81	49.51
BEAM	: single		2215.84	60.07	950.75	54.01
S. SPEED	: TGS		1697.07	19.87	894.82	52.48
M. DATE	: 3/27/16		1600.65	30.56	796.47	41.82
F. NAME	: 測定中のデータ		1578.47	38.97	757.90	48.17
			1487.83	34.83		
			1433.83	40.86		

IR spectrum of **30**.



Mass spectrum (FD) of **30**.

[illegible]

Chemical structure of compound **9b** is shown above the ^1H NMR spectrum. The spectrum displays peaks corresponding to the structure, with chemical shifts (δ / ppm) and integration values indicated.

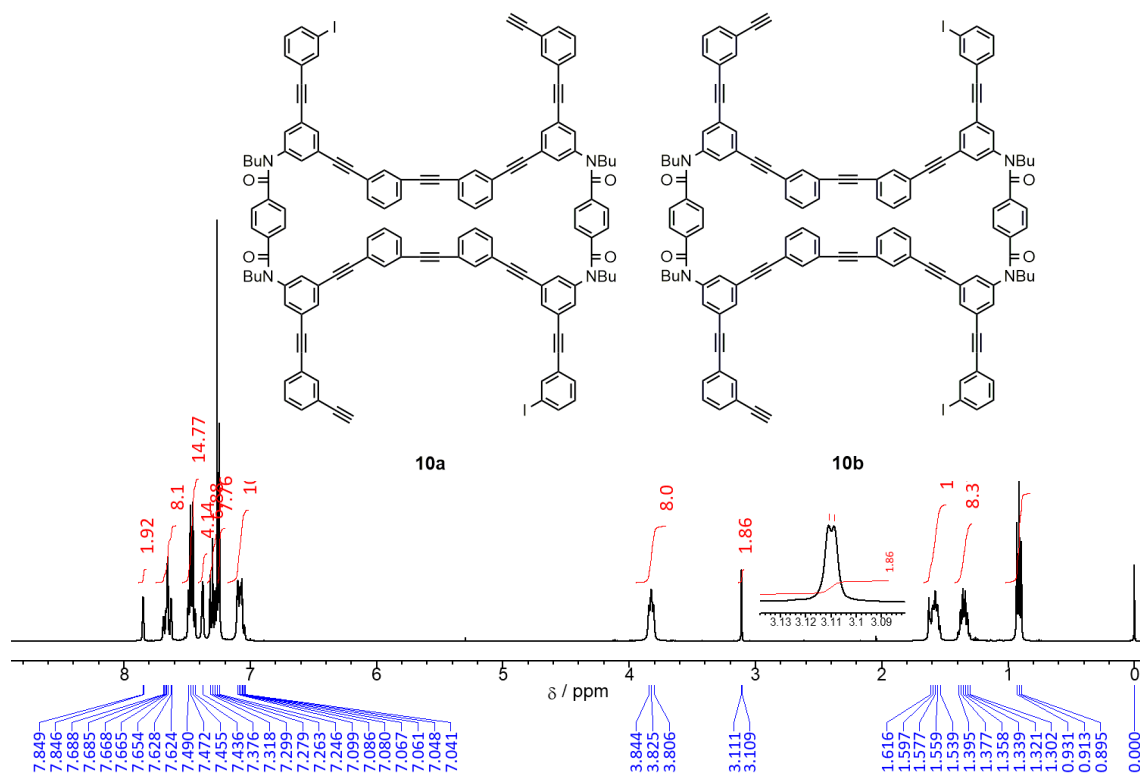
Chemical shifts (δ / ppm) and integration values (from left to right):

- 1.83
- 7.75
- 16.72
- 2.16
- 1.86
- 7.87
- 1.86
- 7.68
- 7.64
- 12.0
- 3.107

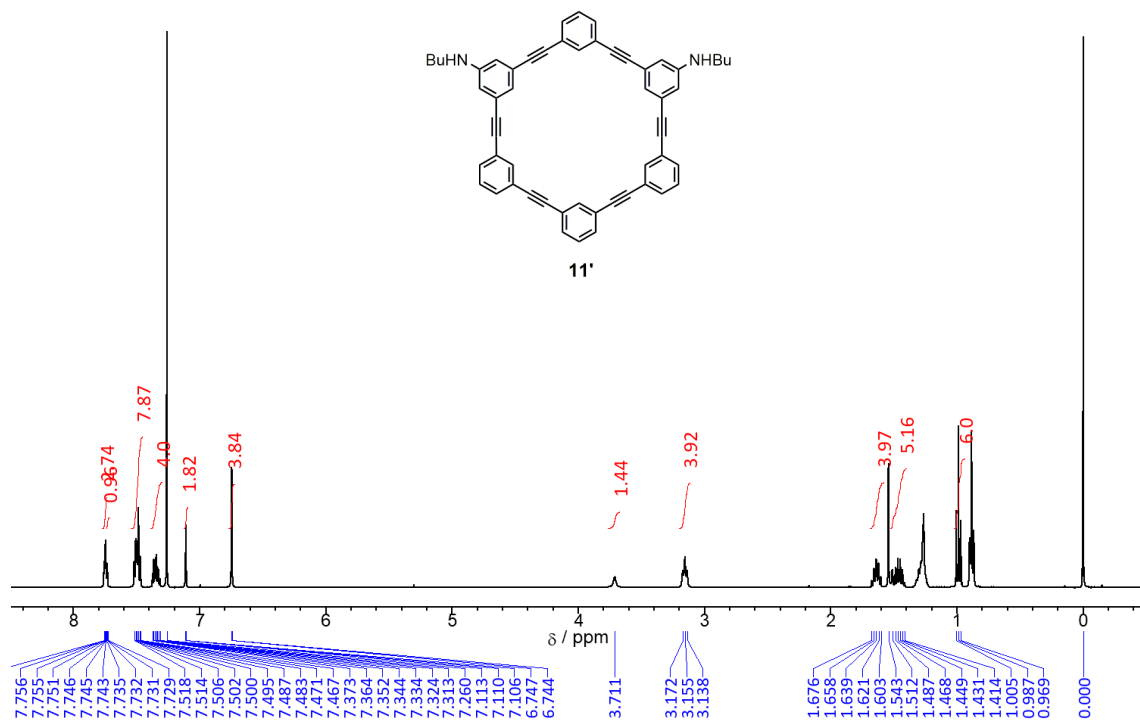
Chemical shifts (δ / ppm) listed at the bottom of the spectrum:

7.890, 7.886, 7.884, 7.679, 7.664, 7.659, 7.657, 7.636, 7.493, 7.440, 7.391, 7.346, 7.326, 7.306, 7.286, 7.263, 7.252, 7.192, 7.147, 7.096, 7.076, 7.056, 6.988, 3.845, 3.822, 3.804, 3.784, 3.107, 1.626, 1.605, 1.570, 1.538, 1.520, 1.402, 1.384, 1.365, 1.346, 1.328, 1.309, 1.293, 0.938, 0.927, 0.920, 0.909, 0.901, 0.891, 0.000.

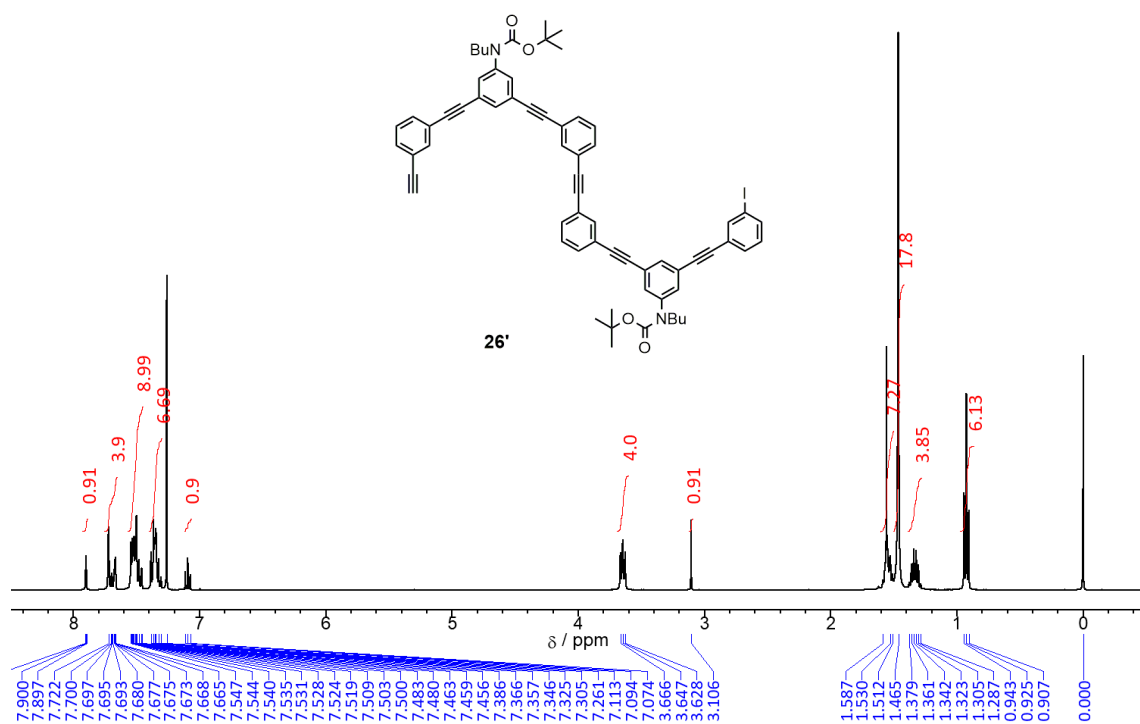
S77



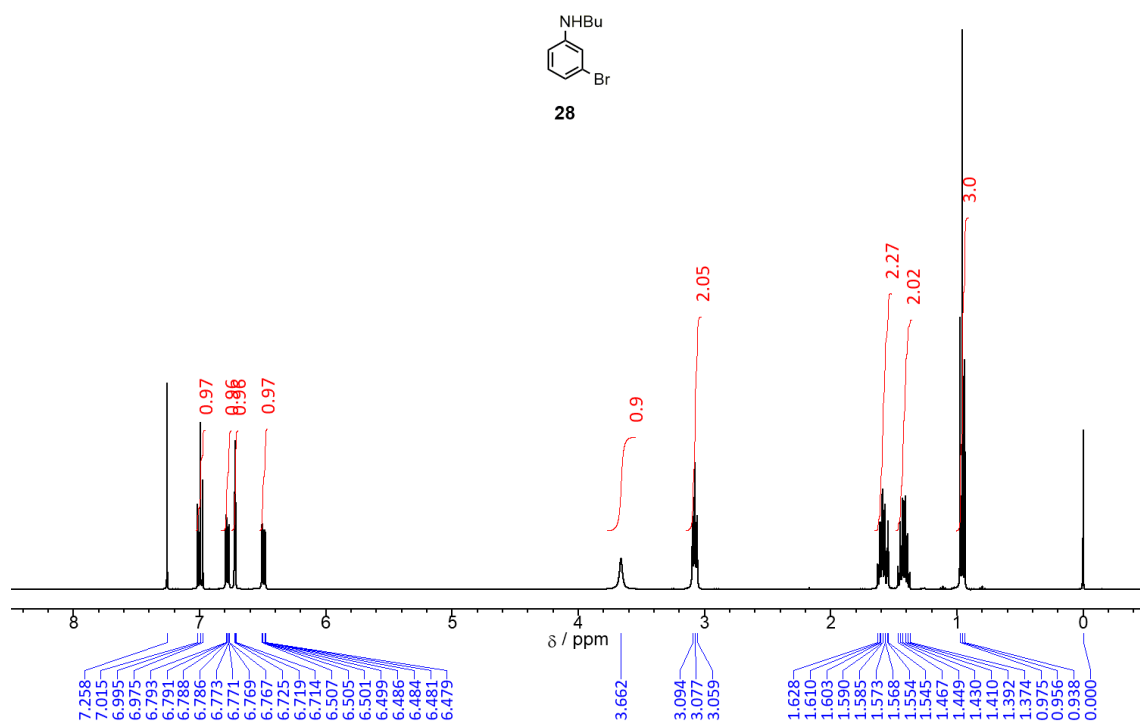
^1H NMR spectrum (400 MHz) of **10a** and **10b**, measured in chloroform-*d* at room temperature.



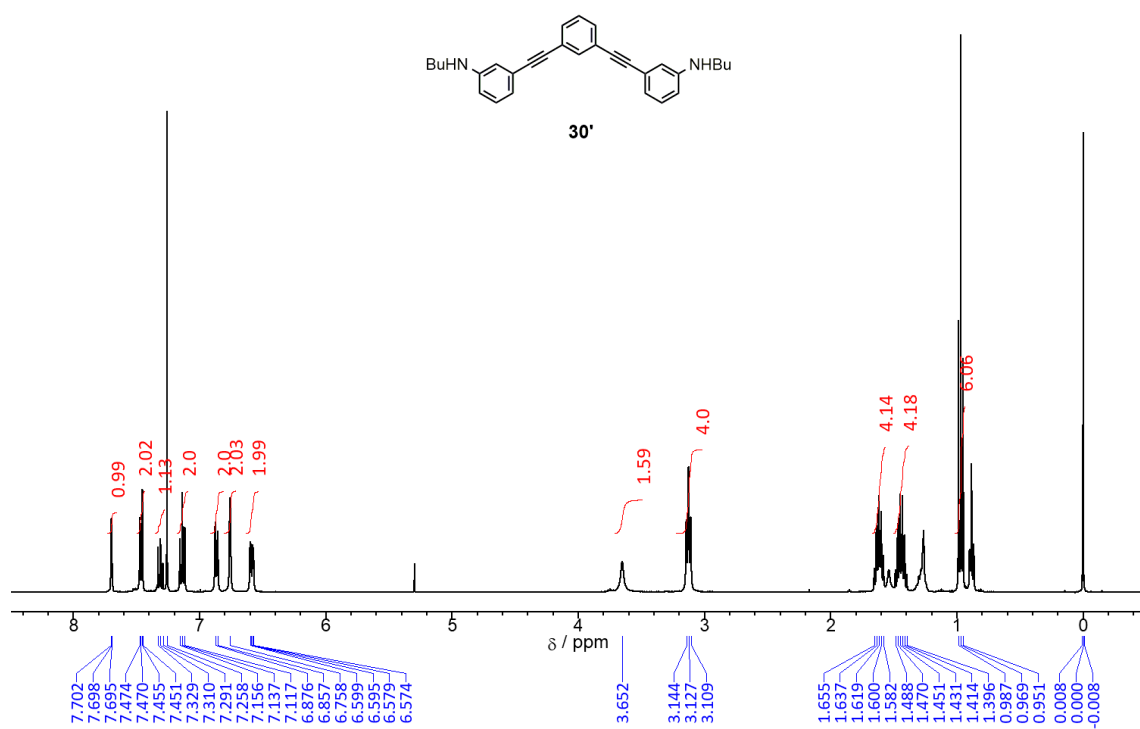
^1H NMR spectrum (400 MHz) of **11'** cont. residual solvents (hexane), measured in chloroform-*d* at room temperature.



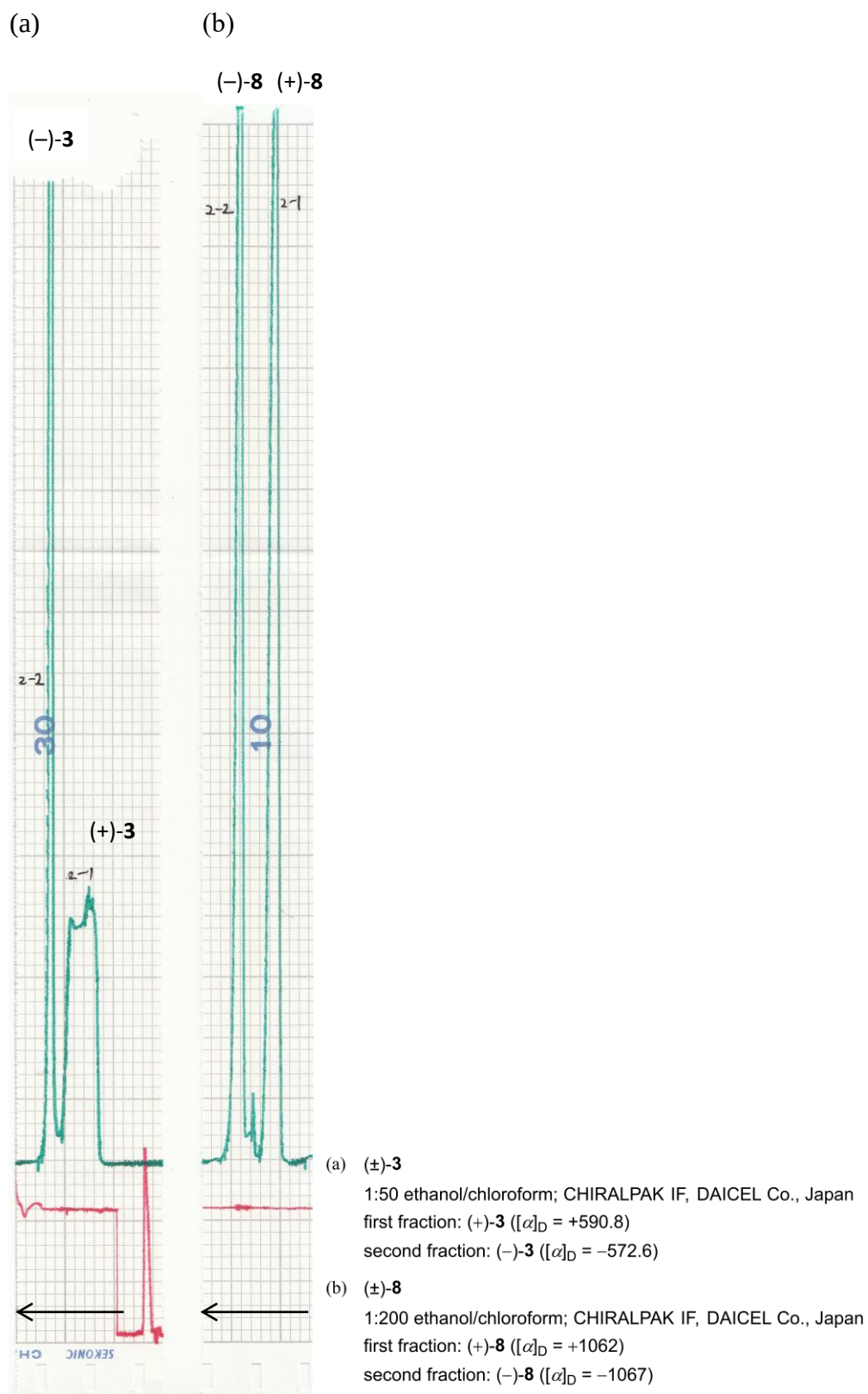
^1H NMR spectrum (400 MHz) of **26'**, measured in chloroform-*d* at room temperature.



^1H NMR spectrum (400 MHz) of **28**, measured in chloroform-*d* at room temperature.



^1H NMR spectrum (400 MHz) of **30'** cont. residual solvents (dichloromethane and hexane), measured in chloroform-*d* at room temperature.



Chiral HPLC traces, detected by UV at 254 nm (green lines).