Supporting information

Enantioselective Electrosynthesis of Inherently Chiral Calix[4]arenes via a Cobalt-Catalyzed Aryl C-H Acyloxylation

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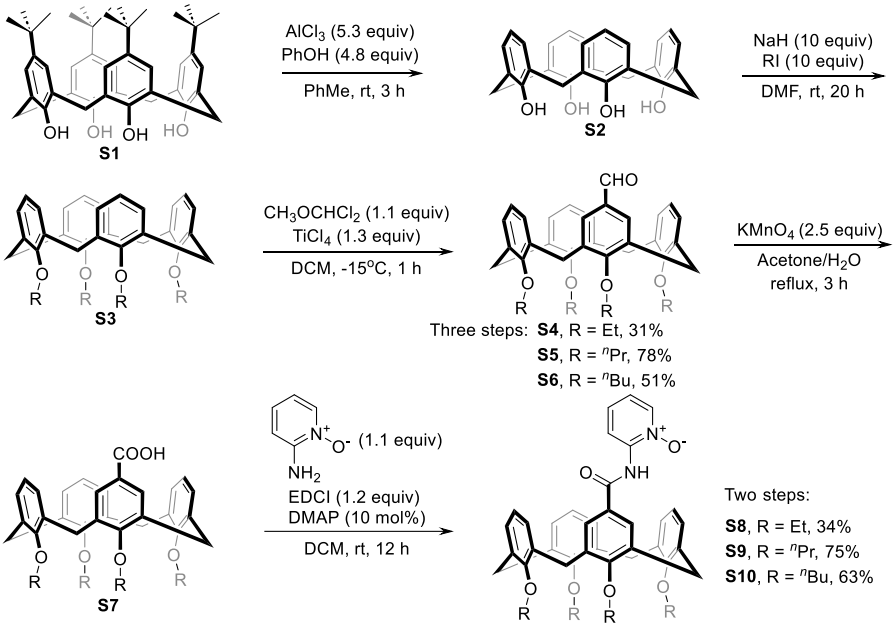
1. General information

The commercial reagent-grade chemicals were used directly without further treatment unless noted. The reactions were carried out in commercially available analytical solvents under air atmosphere unless otherwise noted, and monitored with analytical thin-layer chromatography (TLC) on silica gel 60 F254 plates and visualized under UV (254nm, 365nm). The purifications were implemented by flash column chromatography on silica gel (200-300 mesh) as stationary phase.

NMR spectra were recorded on Bruker Avance 400 (400 MHz for ^{1}H and 100 MHz for ^{13}C) and Bruker Avance 600 (600 MHz for ^{1}H, 564 MHz for ^{19}F and 150 MHz for ^{13}C) spectrometers at 295 K. Chemical shifts were reported in part per million relative to residual peak ($CDCl_3$: ^{1}H δ 7.26 ppm, ^{13}C δ 77.16 ppm; DMSO-D6: ^{1}H δ 2.50 ppm, ^{13}C δ 39.52 ppm; CD_3OD: ^{1}H δ 3.31 ppm, ^{13}C δ 49.00 ppm). The mentioned abbreviations are as follows: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), br (broad). The melting points were measured on a WC-1 instrument. The cyclic voltammetry analysis was conducted on an electrochemical workstation (CHI 760E). The enantiomeric excess (ee) of the products were measured by high-performance liquid chromatography (Agilent 1260 Infinity LC instrument) equipped with chiral columns (Daicel chiral technologies, China). High-Resolution mass spectra (HRMS) were measured on a Waters ACQUITYUPLC 1-Class PLUS liquid chromatogram coupled with a Waters Xevo G2-XS QTof mass spectrometer. The single crystal diffraction was performed on the Oxford (Agilent Gemini E, USA) X-ray diffractometer. The specific rotations were measured on a WZZ-3A polarimeter.

2. Experimental procedures

2.1 Preparation of substrates



To a solution of compound **S1** (25 g, 38.5 mmol) and phenol (4.8 equiv) in toluene (250 mL) was added $AlCl_3$ (5.3 equiv) within 10 minutes at room temperature. The mixture was stirred at this temperature for 3-5 h until **S1** was totally consumed detected by TLC. The reaction was quenched by addition of 0.2 M HCl, leading to white precipitate as the good product **S2**. After filtration, the filtrate was extracted with ethyl acetate. The organic phase was concentrated to remove 80 % of the solvent under reduced pressure, giving a white precipitate as the good product **S2**.

To a solution of **S2** in DMF (250 mL) was added NaH (10 equiv) within 20 minutes in an ice-water bath. After stirring at this temperature for 1 h, the alkyl halides (10 equiv) were added dropwise. Then the mixture was stirred at room temperature for 20 h, following by quenching with 1 M HCl. The pH value was adjusted to 1-2 with 1 M HCl. The resulting white precipitate was collected by filtration, leading to a white powder as the good product **S3**.

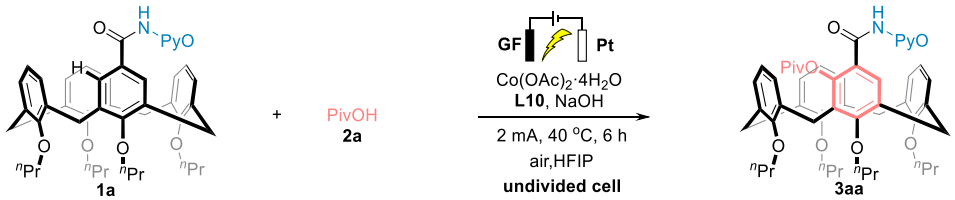
To a solution of the obtained white powder in anhydrous CH_2Cl_2 was added 1,1-dichloromethyl ether (1.1 equiv) at -15 ºC under Ar atmosphere. Then the reaction was stirred at this temperature for 30 minutes, followed by the slow addition of $TiCl_4$ (1.3 equiv). Subsequently, the mixture was stirred at -10 ºC for 1-1.5 h until the total consumption of the starting material monitored by TLC. The reaction was quenched by slow addition of water. The resulting mixture was extracted with ethyl acetate. The organic phase was collected and concentrated on a rotovap under reduced pressure to give a residue, which was purified by flash column chromatography

(petroleum ether/ethyl ether = 20/1), leading to the desired compound as a yellow solid, **S4** (R = Et), **S5** (R = ^{n}Pr), **S6** ((R = ^{n}Bu).

To a solution of the yellow solid in acetone (100 mL) was added the aqueous of $KMnO_4$ (2.5 equiv). The reaction was refluxed in an oil bath (105 °C) for 3-5 h. After dilution with ethyl acetate, the pH value was adjusted to 1-2 with 1 M HCl. Then the mixture was extracted with ethyl acetate. The organic phase was collected and concentrated under reduced pressure, leading to the good product as a yellow solid (**S7**).

To a solution of the acid in CH_2Cl_2 (40 mL) was added 2-pyridinamine 1-oxide (1.1 equiv), DMAP (0.1 equiv), EDCI (1.2 equiv). The reaction was stirred at room temperature for 12 h. Then the mixture was extracted with ethyl acetate and washed with Na_2CO_3 aqueous solution. The organic phase was concentrated under reduced pressure. The residue was purified by flash column chromatography, leading to the final product as a white solid, **S8** (R = Et), **S9** (R = ^{n}Pr), **S10** ((R = ^{n}Bu).

2.2 Experimental procedures of electrolysis



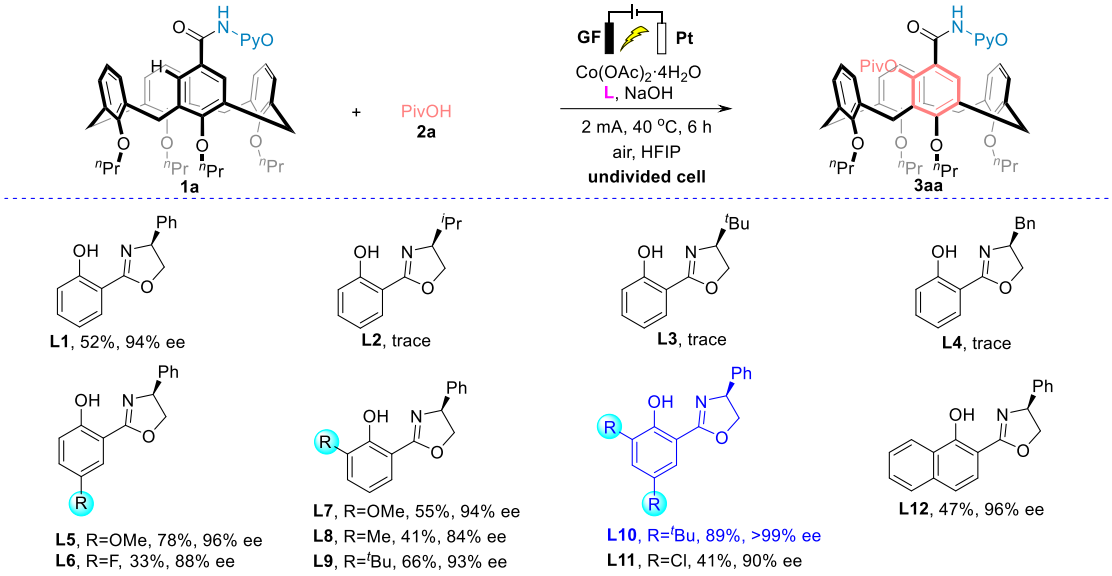
The electrolysis of **1a** and **2a** leading to **3aa** was set as the template reaction.

To a flask (30 mL) was added **1a** (0.1 mmol), $Co(OAc)_2·4H_2O$ (10 mol%), **L10** (20 mol%), NaOH (2 equiv), pivalic acid (2 equiv) and hexafluoroisopropanol (5 mL). It was then equipped with a graphite anode (15 mm×10 mm×6 mm) and a platinum cathode (10 mm×10 mm×0.1 mm) in an undivided cell. The reaction was conducted at a constant current model (2 mA) and stirred at 40 °C for 6 h. After the total consumption of **1a** monitored by TLC, the reaction was diluted with CH_2Cl_2 and washed with $NaHCO_3$ aqueous solution. The organic phase was concentrated under reduced pressure to give a residue, which was purified by flash column chromatography to furnish the desired product **3aa**.

3. Optimization of reaction conditions

3.1 Screening of the ligands

Table S1. Screening of the ligands[a,b]

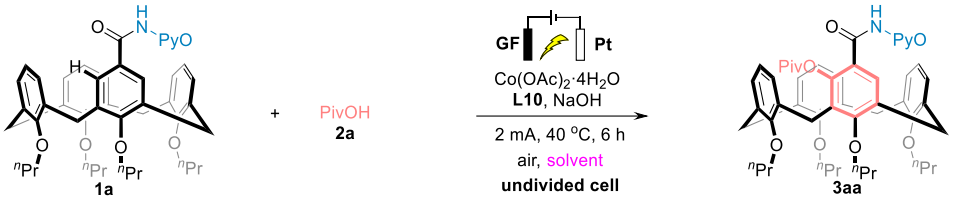


[a]Undivided cell, GF anode (15 mm × 10 mm × 6 mm submerged), platinum plated cathode (10 mm × 10 mm × 0.1 mm submerged), constant current of 2 mA; isolated yields are indicated; ee values were determined by chiral HPLC analysis.

[b]**1a** (0.1 mmol), **2a** (0.2 mmol), $Co(OAc)_2 \cdot 4H_2O$ (10 mol%), **Ligand** (20 mol%), NaOH (2 equiv), 40 °C, 6 h, air, HFIP (5 mL).

3.2 Screening of the solvents

Table S2. Screening of the solvents[a,b]



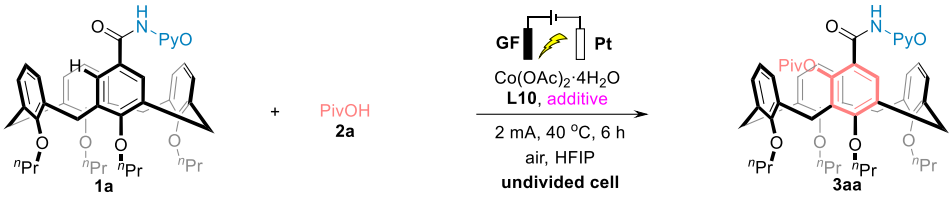
Entry	Solvents	Yield (%)	ee (%)
1	CH_3OH	Trace	-
2	EtOH	Trace	-
3	TFE	Trace	-
4	IPrOH	Trace	-
5	HFIP	89	>99
6	CH_2Cl_2	Trace	-
7	CH_3CN	45	78
8	Acetone	Trace	-
9	Ethyl acetate	Trace	-
10	DMF	Trace	-
11	EA:HFIP = 1:5	61	91
12	Acetone:HFIP = 1:5	Trace	-
13	CH_2Cl_2:HFIP = 1:5	57	93
14	CH_3CN:HFIP = 1:5	Trace	-

[a]Undivided cell, GF anode (15 mm × 10 mm × 6 mm submerged), platinum plated cathode (10 mm × 10 mm × 0.1 mm submerged), constant current of 2 mA; isolated yields are indicated; ee values were determined by chiral HPLC analysis.

[b]**1a** (0.1 mmol), **2a** (0.2 mmol), $Co(OAc)_2·4H_2O$ (10 mol%), **L10** (20 mol%), NaOH (2 equiv), 40 °C, 6 h, air, Solvent (5 mL).

3.3 Screening of the additives

Table S3. Screening of the additives[a,b]



Entry	Additives	Yield (%)	ee (%)
1	-	41	98
2	AcOH	trace	-
3	Na_2CO_3	69	94
4	$NaHCO_3$	51	92
5	DBU	85	96
6	Pyridine	11	89
7	DMAP	27	89
8	Et_3N	71	98
9	^{t}BuONa	85	>99
10	NaOH	89	>99
11[c]	NaOH	86	>99

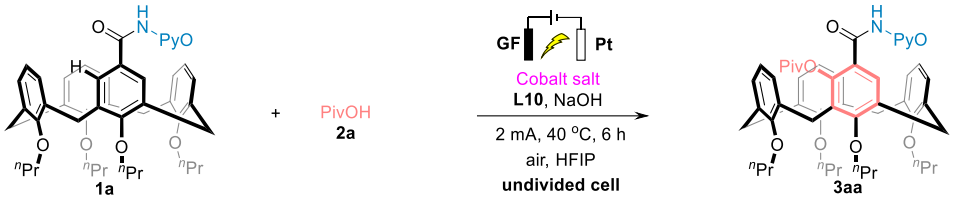
[a]Undivided cell, GF anode (15 mm × 10 mm × 6 mm submerged), platinum plated cathode (10 mm × 10 mm × 0.1 mm submerged), constant current of 2 mA; isolated yields are indicated; ee values were determined by chiral HPLC analysis.

[b]**1a** (0.1 mmol), **2a** (0.2 mmol), $Co(OAc)_2·4H_2O$ (10 mol%), **L10** (20 mol%), additive (2 equiv), 40 °C, 6 h, air, HFIP (5 mL).

[c]Tetrabutylammonium hexafluorophosphate (2 equiv).

3.4 Screening of the cobalt salts

Table S4. Screening of the cobalt salts[a,b]



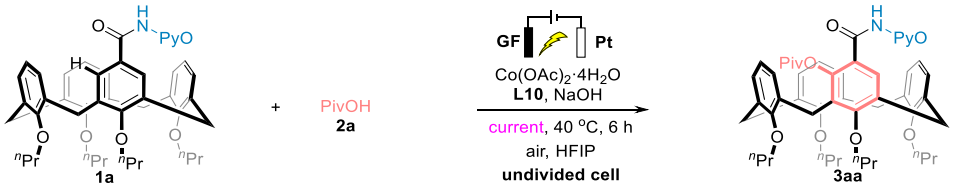
Entry	Cobalt salts	Yield (%)	ee (%)
1	$Co(OAc)_2·4H_2O$	89	>99
2	$Co(OAc)_2$	85	>99
3	$Co(OOCC_6H_5)_2$	55	96
4	$Co(acac)_2$	Trace	-
5	$Co(ONO_3)_2·6H_2O$	Trace	-
6	CoF_2	14	93
7	$CoBr_2$	23	92
8	$CoSO_4·H_2O$	Trace	-
9	$Co(BF_4)_2·6H_2O$	trace	-

[a]Undivided cell, GF anode (15 mm × 10 mm × 6 mm submerged), platinum plated cathode (10 mm × 10 mm × 0.1 mm submerged), constant current of 2 mA; isolated yields are indicated; ee values were determined by chiral HPLC analysis.

[b]**1a** (0.1 mmol), **2a** (0.2 mmol), Cobalt salt (10 mol%), **L10** (20 mol%), NaOH (2 equiv), 40 °C, 6 h, air, HFIP (5 mL).

3.5 Screening of the current

Table S5. Screening of the current[a,b]



Entry	Current (mA)	Yield (%)	ee (%)
1	1	39	99
2[c]	1	43	99
3	2	89	>99
4[d]	3	69	98
5[d]	4	59	98

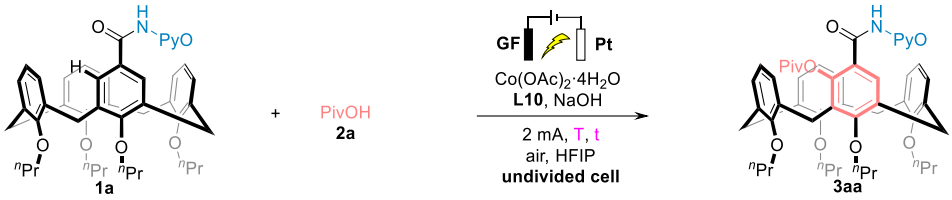
[a]Undivided cell, GF anode (15 mm × 10 mm × 6 mm submerged), platinum plated cathode (10 mm × 10 mm × 0.1 mm submerged), constant current of 2 mA; isolated yields are indicated; ee values were determined by chiral HPLC analysis.

[b]**1a** (0.1 mmol), **2a** (0.2 mmol), Cobalt salt (10 mol%), **L10** (20 mol%), NaOH (2 equiv), 40 °C, 6 h, air, HFIP (5 mL).

[c]8 h. [d]5 h.

3.6 Screening of the reaction time and temperature

Table S6. Screening of the reaction time and temperature[a,b]



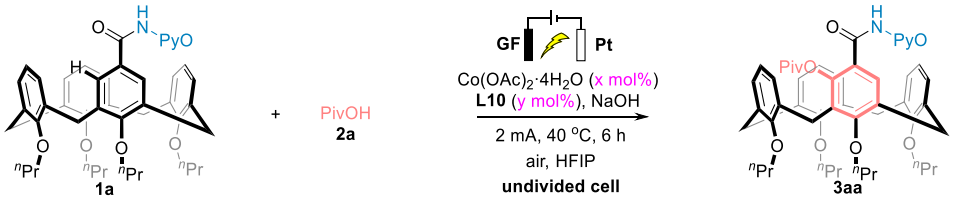
Entry	T (°C)	t (h)	Yield (%)	ee (%)
1	80	6	86	98
2	60	6	88	99
3	40	6	89	>99
4	25	6	48	98
5	40	2	44	98
6	40	4	67	99
7	40	6	89	>99
8	40	8	79	>99
9	40	10	65	99

[a]Undivided cell, GF anode (15 mm × 10 mm × 6 mm submerged), platinum plated cathode (10 mm × 10 mm × 0.1 mm submerged), constant current of 2 mA; isolated yields are indicated; ee values were determined by chiral HPLC analysis.

[b]**1a** (0.1 mmol), **2a** (0.2 mmol), Cobalt salt (10 mol%), **L10** (20 mol%), NaOH (2 equiv), air, HFIP (5 mL).

3.7 Screening of the dosages of catalysts and ligands

Table S7. Screening of the dosages of catalysts and ligands[a,b]



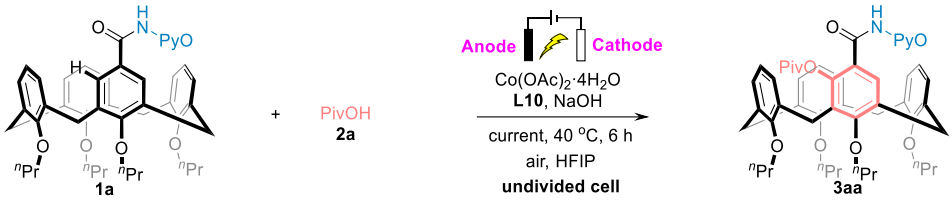
Entry	x	y	Yield (%)	ee (%)
1	10	20	89	>99
2	10	15	64	94
3	10	10	53	93
4	5	10	23	92

[a]Undivided cell, GF anode (15 mm × 10 mm × 6 mm submerged), platinum plated cathode (10 mm × 10 mm × 0.1 mm submerged), constant current of 2 mA; isolated yields are indicated; ee values were determined by chiral HPLC analysis.

[b]**1a** (0.1 mmol), **2a** (0.2 mmol), Cobalt salt (x mol%), **L10** (y mol%), NaOH (2 equiv), 2 mA, 40 °C, 6 h, air, HFIP (5 mL).

3.8 Screening of the electrode materials

Table S8. Screening of the electrode materials[a,b]



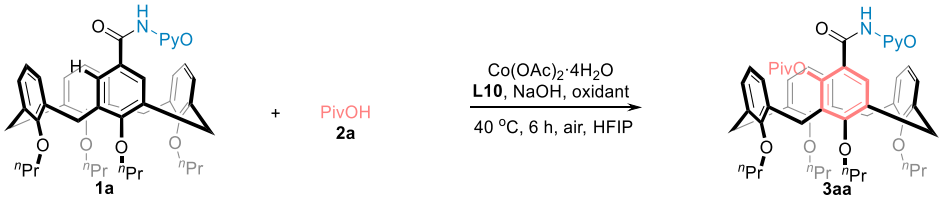
Entry	Anode	Cathode	Yield (%)	ee (%)
1	GF	Pt	89	>99
2	Pt	Pt	23	94
3	GF	GF	36	93
4	GF	Pt	44	98
5	GF	Stainless steel	54	95

[a]Undivided cell, anode (15 mm × 10 mm × 6 mm submerged), cathode (10 mm × 10 mm × 0.1 mm submerged), constant current of 2 mA; isolated yields are indicated; ee values were determined by chiral HPLC analysis.

[b]**1a** (0.1 mmol), **2a** (0.2 mmol), Cobalt salt (10 mol%), **L10** (20 mol%), NaOH (2 equiv), 2 mA, 40 °C, 6 h, air, HFIP (5 mL).

Screening of the oxidants

Table S8-1. Screening of the oxidants[a]



Entry	Oxidant	Yield (%)	ee (%)
1	Ag_2CO_3	18	>99
2	$Mn(OAc)_3·2H_2O$	trace	-
3	O_2	-[b]	-
4[c]	O_2	-[b]	-
5[d]	O_2	-[b]	-

[a]**1a** (0.1 mmol), **2a** (0.2 mmol), $Co(OAc)_2·4H_2O$ (10 mol%), **L10** (20 mol%), NaOH (2 equiv), oxidant (1.0 equiv.), 40 °C, 6 h, air, HFIP (5 mL). [b]No desired compound was observed. [c]60 °C. [d]100 °C

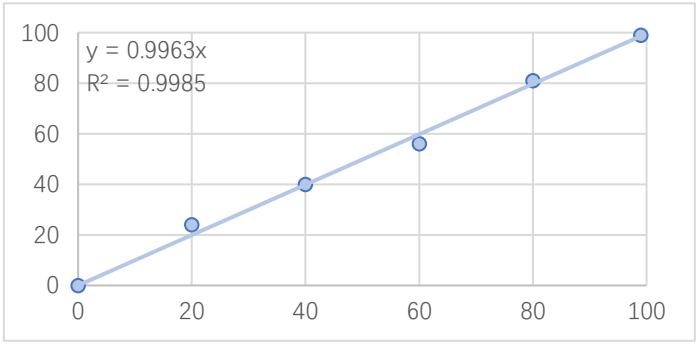
4. Mechanistic studies

4.1 Non-linear effect studies

The synthesis of compound **3aa** was set as the template reaction to conduct the non-linear effect studies. The ligand **L10** samples with various ee values were prepared by rational mixture of enantiopure and racemic **L10** as shown in Table xx. Then the obtained ligands were subjected to the synthesis of compound **3aa** under standard conditions, leading to the corresponding products which were purified by flash column chromatography (PE:EA = 2:1). The ee values were determined by chiral HPLC analysis. The results indicated the linearity between the ee values of **L10** and product **3aa**.

Table S9. Non-linear effect studies

L10 ee (%)	0	20	40	60	80	99
3aa ee (%)	0	24	40	56	81	99



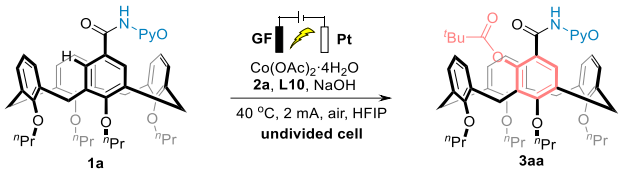
4.2 Dynamic electrode potential analysis

The synthesis of compound **3aa** was set as the template reaction to conduct the dynamic electrode potential analysis. The silver wire (100 mm×1 mm) was used as the reference electrode. A steady potential was observed.

4.3 Cyclic voltammetry studies

The cyclic voltammetry analysis was conducted on an electrochemical workstation (CHI 760E). The electrolysis experiments were implemented with a glassy carbon electrode, Pt electrode and Ag/AgCl reference electrode. The electrolysis was irreversible since the value of ipa/ipc was far less than 1.

5. Application studies

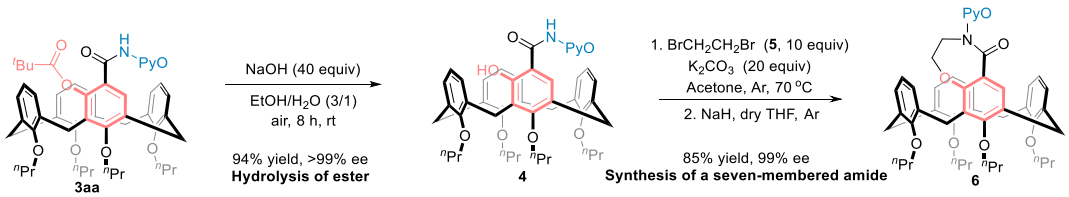


*Scale-up synthesis of **3aa**: 0.3 mmol*

To a flash (30 mL) was added **1a** (0.3 mmol), $Co(OAc)_2·4H_2O$ (10 mol%), **L10** (20 mol%), NaOH (2 equiv), pivalic acid (2 equiv) and hexafluoroisopropanol (20 mL). It was then equipped with a graphite anode (20 mm×20 mm×6 mm) and a platinum cathode (10 mm×10 mm×0.1 mm) in an undivided cell. The reaction was conducted at a constant current model (2 mA) and stirred at 40 °C for 10 h. After the total consumption of 1a monitored by TLC, the reaction was diluted with CH_2Cl_2 and washed with $NaHCO_3$ aqueous solution. The organic phase was concentrated under reduced pressure to give a residue, which was purified by flash column chromatography (petroleum ether/ethyl acetate = 2/1) to furnish the desired product as a light grey solid (0.18 g, 72%, 99% ee).

*Scale-up synthesis of **3aa**: 1.0 mmol*

To a flash (100 mL) was added **1a** (1.0 mmol), $Co(OAc)_2·4H_2O$ (10 mol%), **L10** (20 mol%), NaOH (2 equiv), pivalic acid (2 equiv) and hexafluoroisopropanol (80 mL). It was then equipped with a graphite anode (45 mm×20 mm×6 mm) and a platinum cathode (45 mm×20 mm×0.1 mm) in an undivided cell. The reaction was conducted at a constant current model (2 mA) and stirred at 40 °C for 72 h. After the total consumption of **1a** monitored by TLC, the reaction was diluted with CH_2Cl_2 and washed with $NaHCO_3$ aqueous solution. The organic phase was concentrated under reduced pressure to give a residue, which was purified by flash column chromatography (petroleum ether/ethyl acetate = 2/1) to furnish the desired product as a light grey solid (0.662 g, 80%, 99% ee).



Hydrolysis of the ester motif:

To a flask was added **3aa** (0.1 mmol), NaOH (40 equiv) and $EtOH/H_2O$ (3 mL/1 mL). The mixture was stirred at 25 °C for 8 h under Ar atmosphere. After the total consumption of **3aa** monitored by TLC, the reaction was diluted with CH_2Cl_2 and washed with 1 N HCl aqueous solution. The organic phase was concentrated under reduced pressure to give a residue, which

was purified by flash column chromatography (petroleum ether/ethyl acetate = 1/1) to furnish the desired product **4** as a white solid (70.0 mg, 94%, >99% ee).

Synthesis of an inherently chiral calix[4]arenes embedded with a seven-membered amide:

To a dry flask was added the previously obtained compound **4** (0.1 mmol), **5** (1 mmol, 10 equiv), K_2CO_3 (2 mmol, 20 equiv) and acetone (5 mL). The mixture was stirred in an oil bath (70 °C) for 12 h. After the total consumption of **4** monitored by TLC, the solvent was removed with a rotovap under reduced pressure to give a residue, which was dissolved in dry THF (5 mL) in a dry flash under Ar atmosphere. After the addition of NaH (0.23 mmol) at 0 °C, the reaction was stirred at room tempersture for 6 h, followed by quenching with NH_4Cl aqueous solution. The mixture was extracted with DCM. The organic phase was washed with brine and dried over Na_2SO_4. After concentration under reduced pressure, the resulting residue was purified by flash column chromatography (petroleum ether/ethyl acetate = 1/2) to furnish the desired product **6** as a white solid (66 mg, 85%, 99% ee).

6. X-ray diffraction analysis

Preparation of crystal

The single crystal of **3al** was obtained by slow evaporation at room temperature from a mixed solvent of isopropanol and hexane (1:2).

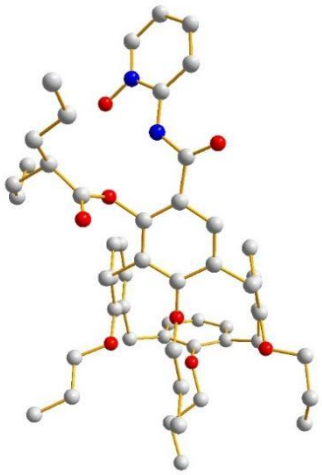


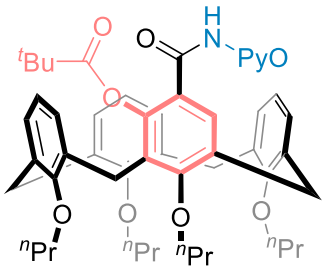
Table S10. Crystal data and structure refinement for **3al** (CCDC 2339942).

Identification code	3al
Empirical formula	$C_{54}H_{65}N_2O_8$
Formula weight	870.08
Temperature/K	200.00(10)
Crystal system	orthorhombic
Space group	$P2_12_12_1$

a/Å	9.7335(2)
b/Å	13.0898(4)
c/Å	38.4467(11)
α/°	90
β/°	90
γ/°	90
Volume/$Å^3$	4898.5(2)
Z	4
$\rho_{calc}g/cm^3$	1.180
μ/mm^{-1}	0.627
F(000)	1868.0
Crystal size/mm^3	0.2 × 0.12 × 0.11
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	7.134 to 149.194
Index ranges	$-12 \leq h \leq 6$, $-16 \leq k \leq 15$, $-46 \leq l \leq 47$
Reflections collected	54476
Independent reflections	9828 [R_{int} = 0.0611, R_{sigma} = 0.0393]
Data/restraints/parameters	9828/0/583
Goodness-of-fit on F^2	1.051
Final R indexes [I>=2σ (I)]	R_1 = 0.0552, wR_2 = 0.1530
Final R indexes [all data]	R_1 = 0.0666, wR_2 = 0.1636
Largest diff. peak/hole / e $Å^{-3}$	0.58/-0.47
Flack parameter	-0.04(8)

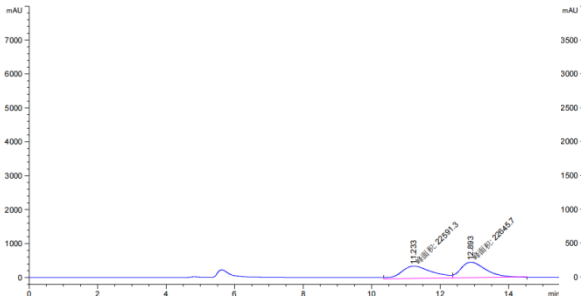
7. NMR data of the acyloxylated inherently chiral calix[4]arenes

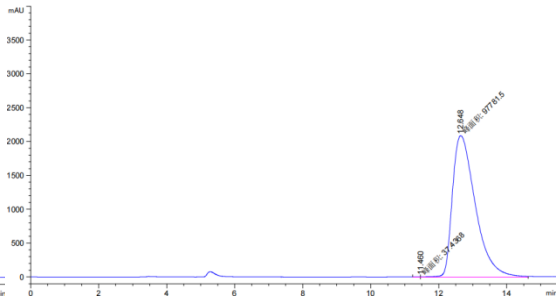
2-(1⁴-(pivaloyloxy)-1²,3²,5²,7²-tetrapropoxy-1,3,5,7(1,3)-tetrabenzenacyclooctaphane-1⁵-carboxamido)pyridine 1-oxide (3aa)



Yield: 73.7 mg (89%), Grey solid, mp: 115 °C.

^{1}H NMR (600 MHz, $CDCl_3$) δ 10.53 (s, 1H), 8.58 (dd, *J* = 8.5, 1.6 Hz, 1H), 8.28 (dd, *J* = 6.5, 1.0 Hz, 1H), 7.56 (s, 1H), 7.41-7.35 (m, 1H), 7.11 (dd, *J* = 7.4, 2.6 Hz, 2H), 7.05-6.98 (m, 1H), 6.92 (t, *J* = 7.4 Hz, 1H), 6.26 (ddd, *J* = 26.0, 16.8, 7.4 Hz, 3H), 6.15 (dd, *J* = 17.1, 7.8 Hz, 3H), 4.51-4.39 (m, 3H), 4.21-4.09 (m, 3H), 4.06-3.95 (m, 2H), 3.74-3.59 (m, 4H), 3.40 (d, *J* = 13.9 Hz, 1H), 3.24 (d, *J* = 13.7 Hz, 1H), 3.15 (dd, *J* = 13.4, 4.9 Hz, 2H), 1.98 (dt, *J* = 15.4, 7.5 Hz, 4H), 1.87 (ddd, *J* = 14.1, 7.1, 4.6 Hz, 4H), 1.36 (s, 9H), 1.09 (dd, *J* = 16.1, 7.5 Hz, 6H), 0.90 (dt, *J* = 10.5, 7.5 Hz, 6H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 176.9, 164.8, 162.3, 157.9, 157.2, 155.2, 155.1, 146.5, 144.7, 137.1, 137.0, 136.9, 134.9, 133.4, 133.3, 131.9, 131.81, 131.78, 128.97, 128.94, 128.3, 128.0, 127.9, 127.86, 127.82, 127.5, 127.3, 122.7, 122.4, 121.9, 121.7, 118.5, 77.1, 77.0, 76.5, 39.3, 31.0, 30.7, 27.2, 23.5, 23.49, 23.1, 23.0, 10.8, 10.77, 9.8, 9.76. **HRMS** (ESI): m/z $[M+H]^+$calcd for $[C_{51}H_{61}N_2O_8]^+$ requires 829.4428, found 829.4435. $[\alpha]_D^{25}$ = +27 (c = 0.1, CH_2Cl_2). The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (CHIRALPAK AD-H, hexane/*i*-PrOH =95/5, detector: 254 nm, T = 25 °C, flow rate: 1 mL/min), t_1 (major) = 12.648 min, t_2 (minor) = 11.460 min.

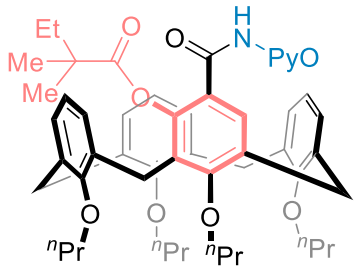




Peak	RetTime	Area	Height	Area
1	11.233	22591.3	363.82465	49.9398
2	12.893	22645.7	454.52222	50.0602

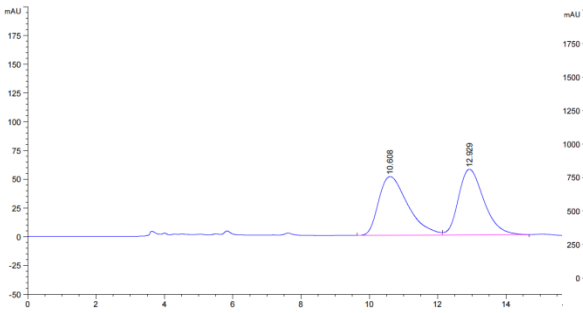
Peak	RetTime	Area	Height	Area
1	11.460	37.4368	3.01561	0.0383
2	12.648	97781.5	2088.38672	99.9617

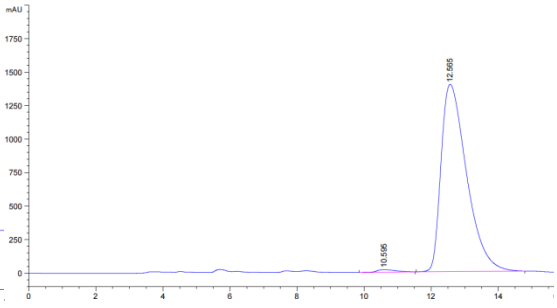
2-(1^4-((2,2-dimethylbutanoyl)oxy)-1^2,3^2,5^2,7^2-tetrapropoxy-1,3,5,7(1,3)-tetrabenzenacyclooctaphane-1^5-carboxamido)pyridine 1-oxide (3ab)



Yield: 71.6 mg (85%), Grey solid, mp: 118 °C.

^{1}H NMR (600 MHz, $CDCl_3$) δ 10.53 (s, 1H), 8.59 (dd, *J* = 8.5, 1.4 Hz, 1H), 8.30 (d, *J* = 6.1 Hz, 1H), 7.56 (s, 1H), 7.38 (t, *J* = 8.0 Hz, 1H), 7.13 (d, *J* = 7.5 Hz, 2H), 7.07-6.98 (m, 1H), 6.93 (t, *J* = 7.4 Hz, 1H), 6.25 (ddd, *J* = 33.9, 20.7, 7.4 Hz, 3H), 6.13 (dd, *J* = 13.7, 6.4 Hz, 3H), 4.51-4.39 (m, 3H), 4.22-4.05 (m, 3H), 4.01 (dd, *J* = 9.8, 6.7 Hz, 2H), 3.83-3.50 (m, 4H), 3.42 (d, *J* = 13.9 Hz, 1H), 3.24 (d, *J* = 13.7 Hz, 1H), 3.15 (dd, *J* = 13.4, 4.2 Hz, 2H), 2.05-1.90 (m, 4H), 1.87 (dt, *J* = 14.0, 7.0 Hz, 4H), 1.73 (dt, *J* = 13.9, 6.5 Hz, 2H), 1.33 (d, *J* = 3.5 Hz, 6H), 1.10 (dd, *J* = 16.2, 7.5 Hz, 6H), 0.94-0.85 (m, 9H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 176.5, 164.9, 162.3, 157.9, 155.2, 155.0, 146.4, 144.7, 137.1, 137.03, 137.01, 134.9, 133.3, 133.2, 131.88, 131.82, 131.80, 129.0, 128.9, 128.3, 128.0, 127.82, 127.78, 127.5, 127.3, 122.7, 122.4, 121.9, 118.5, 114.9, 77.1, 76.9, 76.5, 43.0, 33.0, 31.0, 30.7, 24.6, 24.4, 23.5, 23.1, 23.0, 22.7, 10.83, 10.81, 9.8, 9.7, 9.0. **HRMS** (ESI): m/z $[M+H]^+$ calcd for $[C_{52}H_{63}N_2O_8]^+$ requires 843.4584, found 843.4587. $[\alpha]_D^{25}$ = +11 (c = 0.1, CH_2Cl_2). The product was analyzed by HPLC to determine the enantiomeric excess: 98% ee (CHIRALPAK AD-H, hexane/*i*-PrOH =95/5, detector: 254 nm, T = 25 °C, flow rate: 1 mL/min), t_1 (major) = 12.565 min, t_2 (minor) = 10.595 min.

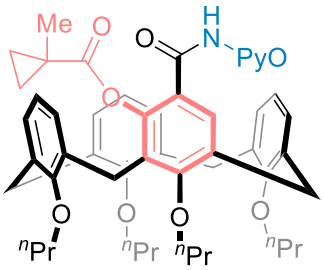




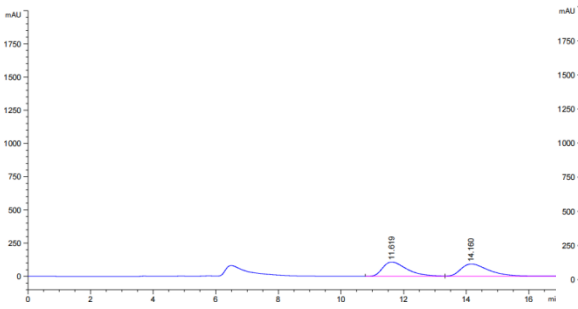
Peak	RetTime	Area	Height	Area
1	10.608	3022.96216	50.99575	50.6313
2	12.929	2947.57495	57.03631	49.3687

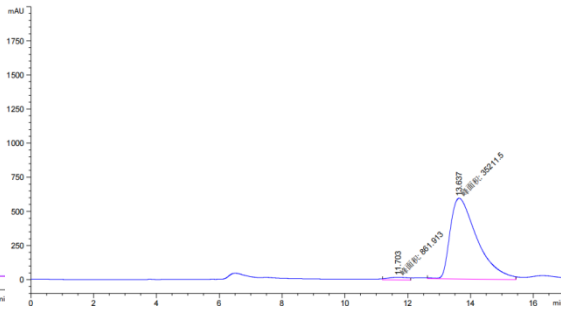
Peak	RetTime	Area	Height	Area
1	10.595	734.93353	19.47724	0.9855
2	12.565	73836.4	1396.22437	99.0145

2-(1^4-((1-methylcyclopropane-1-carbonyl)oxy)-1^2,3^2,5^2,7^2-tetrapropoxy-1,3,5,7(1,3)-tetrabenzenacyclooctaphane-1^5-carboxamido)pyridine 1-oxide (3ac)



Yield: 66.9 mg (81%), White solid, mp: 117 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 10.53 (s, 1H), 8.59 (dd, *J* = 8.5, 1.6 Hz, 1H), 8.34-8.29 (m, 1H), 7.50 (s, 1H), 7.42-7.29 (m, 1H), 7.00 (ddd, *J* = 18.5, 12.5, 4.6 Hz, 3H), 6.83 (t, *J* = 7.4 Hz, 1H), 6.42 – 6.19 (m, 6H), 4.51-4.40 (m, 3H), 4.19 (d, *J* = 13.9 Hz, 1H), 4.15-4.04 (m, 2H), 3.97 (dd, *J* = 9.2, 6.8 Hz, 2H), 3.79-3.53 (m, 4H), 3.38 (d, *J* = 13.9 Hz, 1H), 3.24 (d, *J* = 13.7 Hz, 1H), 3.16 (dd, *J* = 13.4, 4.2 Hz, 2H), 2.12-1.72 (m, 8H), 1.47 (s, 3H), 1.07 (q, *J* = 7.3 Hz, 6H), 1.03-0.88 (m, 8H), 0.79 (d, *J* = 2.8 Hz, 2H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 174.5, 165.6, 164.6, 162.1, 157.5, 156.8, 155.5, 155.4, 146.5, 144.7, 137.1, 136.5, 136.4, 135.9, 135.7, 134.8, 134.6, 134.5, 133.9, 133.8, 132.4, 132.1, 131.2, 128.87, 128.81, 128.7, 128.3, 128.1, 128.0, 127.6, 127.58, 122.5, 122.4, 122.3, 122.0, 121.9, 121.4, 118.5, 115.0, 114.6, 76.9, 76.7, 76.5, 31.0, 30.98, 30.7, 24.3, 23.5, 23.4, 23.2, 23.1, 23.0, 19.4, 19.0, 17.7, 17.6 10.7, 10.6, 10.0, 9.9. **HRMS** (ESI): m/z $[M+H]^+$calcd for $[C_{51}H_{59}N_2O_8]^+$ requires 827.4271, found 827.4280. $[\alpha]_D^{25}$ = +36 (c = 0.1, CH_2Cl_2). The product was analyzed by HPLC to determine the enantiomeric excess: 95% ee (CHIRALPAK AD-H, hexane/*i*-PrOH =95/5, detector: 254 nm, T = 25 °C, flow rate: 1 mL/min), t_1 (major) = 13.637 min, t_2 (minor) = 11.703 min.

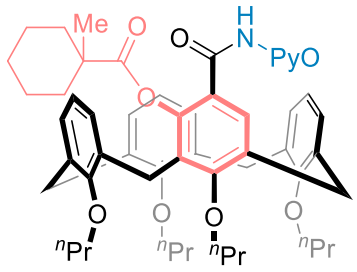




Peak	RetTime	Area	Height	Area
1	11.619	5802.98096	107.56480	49.9495
2	14.160	5814.71924	92.69424	50.0505

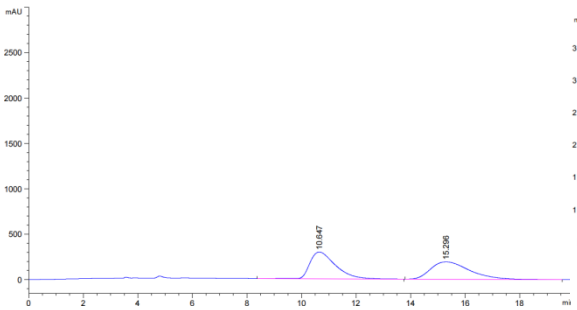
Peak	RetTime	Area	Height	Area
1	11.703	861.91315	19.45750	2.3893
2	13.637	35211.5	590.70612	97.6107

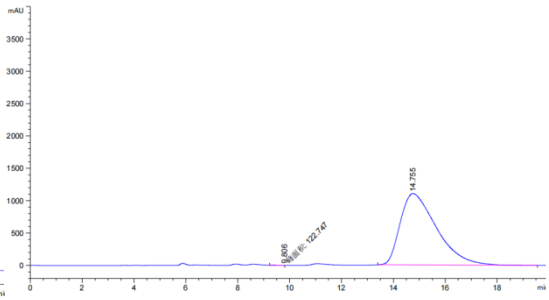
2-(1^4-((1-methylcyclohexane-1-carbonyl)oxy)-1^2,3^2,5^2,7^2-tetrapropoxy-1,3,5,7(1,3)-tetrabenzenacyclooctaphane-1^5-carboxamido)pyridine 1-oxide (3ad)



Yield: 62.5 mg (82%), White solid, mp: 128 °C.

^{1}H NMR (600 MHz, $CDCl_3$) δ 10.51 (s, 1H), 8.58 (dd, *J* = 8.5, 1.5 Hz, 1H), 8.32 – 8.27 (m, 1H), 7.54 (s, 1H), 7.42-7.35 (m, 1H), 7.13 (d, *J* = 7.5 Hz, 2H), 7.05-6.98 (m, 1H), 6.94 (t, *J* = 7.4 Hz, 1H), 6.30-6.23 (m, 2H), 6.19 (d, *J* = 7.4, 1H), 6.15-6.09 (m, 3H), 4.51-4.40 (m, 3H), 4.22-4.07 (m, 3H), 4.02 (dd, *J* = 10.0, 6.6 Hz, 2H), 3.74-3.59 (m, 4H), 3.45 (d, *J* = 13.9 Hz, 1H), 3.24 (d, *J* = 13.7 Hz, 1H), 3.15 (dd, *J* = 13.4, 5.1 Hz, 2H), 2.22-2.04 (m, 2H), 1.98 (dt, *J* = 9.7, 7.9 Hz, 4H), 1.87 (dt, *J* = 14.5, 7.2 Hz, 4H), 1.56-1.31 (m, 8H), 1.34 (s, 3H), 1.10 (dt, *J* = 10.0, 7.4 Hz, 6H), 0.90 (dt, *J* = 12.4, 7.5 Hz, 6H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 176.4, 165.0, 162.2, 157.9, 155.1, 155.0, 146.2, 144.7, 137.1, 137.06, 137.03, 134.9, 133.3, 133.2, 131.9, 131.86, 131.81, 129.0, 128.99, 128.2, 128.0, 127.8, 127.77, 127.4, 127.2, 122.7, 122.4, 122.2, 121.9, 118.5, 114.8, 77.1, 77.0, 76.5, 43.3, 35.3, 35.1, 31.0, 30.7, 25.7, 23.5, 23.49, 23.1, 23.0, 22.6, 22.5, 10.84, 10.82, 9.8, 9.7. **HRMS** (ESI): m/z $[M+H]^+$calcd for $[C_{54}H_{65}N_2O_8]^+$ requires 869.4741, found 869.4744. $[\alpha]_D^{25}$ = +39 (c = 0.1, CH_2Cl_2). The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (CHIRALPAK AD-H, hexane/*i*-PrOH =95/5, detector: 254 nm, T = 25 °C, flow rate: 1 mL/min), t_1 (major) = 14.755 min, t_2 (minor) = 9.806 min.

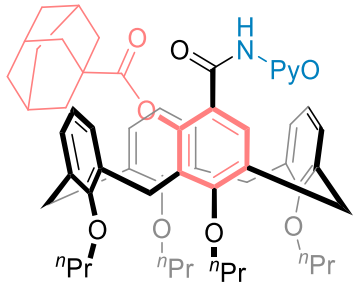




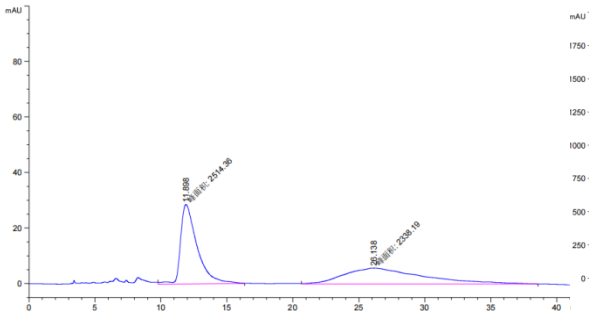
Peak	RetTime	Area	Height	Area
1	10.647	19289.3	294.47650	50.5300
2	15.296	18884.6	190.42738	49.4700

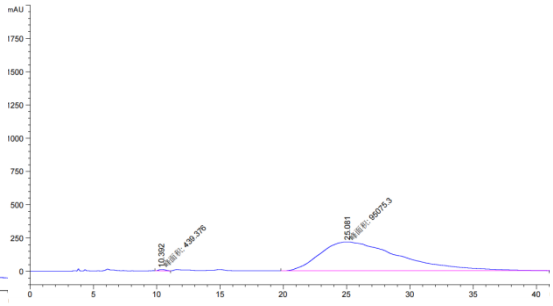
Peak	RetTime	Area	Height	Area
1	9.806	122.74713	4.35635	0.1144
2	14.755	10718.8	1103.01733	99.8856

2-(1^4-((adamantane-1-carbonyl)oxy)-1^2,3^2,5^2,7^2-tetrapropoxy-1,3,5,7(1,3)-tetrabenzenacyclooctaphane-1^5-carboxamido)pyridine 1-oxide (3ae)



Yield: 65.3 mg (72%), White solid, mp: 130 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 10.51 (s, 1H), 8.58 (d, *J* = 8.3 Hz, 1H), 8.29 (d, *J* = 6.4 Hz, 1H), 7.55 (s, 1H), 7.39 (t, *J* = 8.0 Hz, 1H), 7.12 (d, *J* = 7.4 Hz, 2H), 7.02 (t, *J* = 7.0 Hz, 1H), 6.93 (t, *J* = 7.4 Hz, 1H), 6.35-6.21 (m, 3H), 6.21-6.09 (m, 3H), 4.46 (dd, *J* = 20.8, 13.5 Hz, 3H), 4.21-4.06 (m, 3H), 4.01 (td, *J* = 7.1, 2.7 Hz, 2H), 3.73-3.51 (m, 4H), 3.40 (d, *J* = 13.8 Hz, 1H), 3.24 (d, *J* = 13.7 Hz, 1H), 3.15 (dd, *J* = 13.4, 4.6 Hz, 2H), 2.07 (s, 6H), 2.04-1.92 (m, 7H), 1.88 (dd, *J* = 14.2, 7.1 Hz, 4H), 1.71 (s, 6H), 1.10 (td, *J* = 7.4, 3.5 Hz, 6H), 0.90 (dd, *J* = 17.0, 7.6 Hz, 6H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 176.0, 164.7, 162.3, 157.9, 155.2, 155.1, 146.5, 144.7, 137.05, 137.01, 136.9, 134.8, 133.4, 133.3, 131.9, 131.8, 129.0, 128.9, 128.3, 128.0, 127.86, 127.80, 127.5, 127.3, 122.7, 122.4, 121.9, 121.8, 118.5, 114.9, 77.1, 77.0, 76.5, 41.2, 38.6, 36.4, 31.0, 30.7, 27.9, 23.5, 23.4, 23.1, 23.0, 10.82, 10.80, 9.83, 9.77. **HRMS** (ESI): m/z $[M+H]^+$calcd for $[C_{57}H_{67}N_2O_8]^+$ requires 907.4897, found 907.4896. $[\alpha]_D^{25}$ = +11 (c = 0.1, CH_2Cl_2). The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (CHIRALPAK AD-H, hexane/*i*-PrOH = 95/5, detector: 254 nm, T = 25 °C, flow rate: 1 mL/min), t_1 (major) = 25.081 min, t_2 (minor) = 10.392 min.

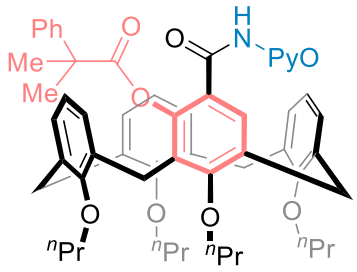




Peak	RetTime	Area	Height	Area
1	11.898	2514.36499	28.62598	51.8153
2	26.138	2338.18677	5.70910	48.1847

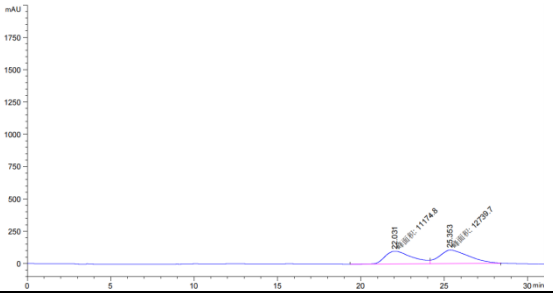
Peak	RetTime	Area	Height	Area
1	10.392	439.37561	10.97502	0.4600
2	25.081	95075.3	216.49039	99.5400

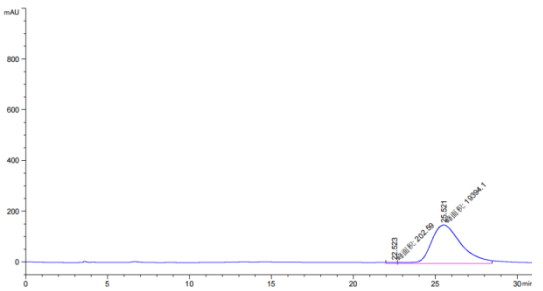
2-(1^4-((2-methyl-2-phenylpropanoyl)oxy)-1^2,3^2,5^2,7^2-tetrapropoxy-1,3,5,7(1,3)-tetrabenzenacyclooctaphane-1^5-carboxamido)pyridine 1-oxide (3af)



Yield: 62.3 mg (70%), White solid, mp: 128 °C.

^{1}H NMR (600 MHz, $CDCl_3$) δ 10.41 (s, 1H), 8.48 (dd, *J* = 8.4, 1.4 Hz, 1H), 8.26 (d, *J* = 6.3 Hz, 1H), 7.50 (s, 1 H), 7.49-7.44 (m, 2H), 7.37 (t, *J* = 8.0 Hz, 1H), 7.17-7.05 (m, 4H), 7.05-6.98 (m, 2H), 6.90 (t, *J* = 7.4 Hz, 1H), 6.25 (dt, *J* = 11.7, 7.6 Hz, 2H), 6.13 (dd, *J* = 16.1, 6.7 Hz, 3H), 6.01 (d, *J* = 7.4 Hz, 1H), 4.47-4.37 (m, 3H), 4.07 (qd, *J* = 11.0, 2.8 Hz, 2H), 4.01-3.93 (m, 2H), 3.85 (d, *J* = 13.9 Hz, 1H), 3.72-3.54 (m, 3H), 3.54-3.44 (m, 1H), 3.21 (d, *J* = 13.7 Hz, 1H), 3.14 (dd, *J* = 13.4, 6.7 Hz, 2H), 2.83 (d, *J* = 14.0 Hz, 1H), 1.93 (dt, *J* = 15.5, 7.6 Hz, 4H), 1.88-1.79 (m, 4H), 1.78 (s, 3H), 1.75 (s, 3H), 1.07 (dt, *J* = 14.7, 7.4 Hz, 6H), 0.88 (td, *J* = 7.4, 2.9 Hz, 6H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 174.8, 164.7, 162.3, 157.9, 155.1, 146.2, 144.5, 143.3, 137.1, 137.0, 136.9, 135.0, 133.4 133.0, 131.9, 131.85, 131.82, 128.93, 128.92, 128.3, 128.1, 127.9, 127.8, 127.6, 127.5, 127.3, 126.7, 126.1, 122.5, 122.4, 121.9, 121.6, 118.5, 115.0, 76.9, 76.5, 46.6, 31.0, 30.9, 30.7, 26.3, 25.8, 23.5, 23.4, 23.2, 23.05, 23.0, 10.8, 10.7, 9.8, 9.7. **HRMS** (ESI): m/z $[M+H]^+$calcd for $[C_{56}H_{63}N_2O_8]^+$ requires 891.4584, found 891.4589. $[\alpha]_D^{25}$ = +28 (c = 0.1, CH_2Cl_2). The product was analyzed by HPLC to determine the enantiomeric excess: 98% ee (CHIRALPAK AD-H, hexane/*i*-PrOH = 95/5, detector: 254 nm, T = 25 °C, flow rate: 1 mL/min), t_1 (major) = 25.521 min, t_2 (minor) = 22.523 min.

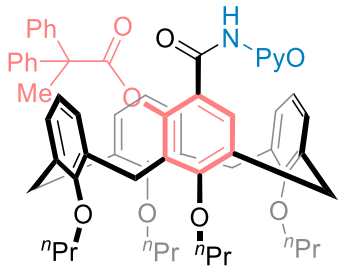




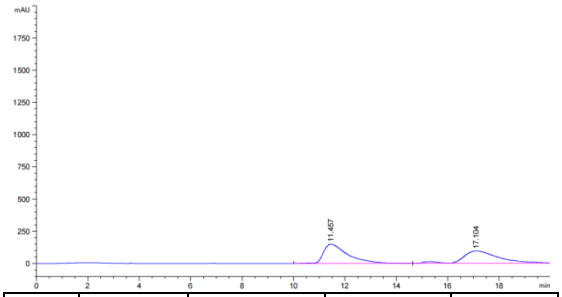
Peak	RetTime	Area	Height	Area
1	22.031	11174.8	98.36907	46.7282
2	25.353	12739.7	104.82605	53.2718

Peak	RetTime	Area	Height	Area
1	22.523	202.59038	4.91290	1.0338
2	25.521	19394.1	151.93422	98.9662

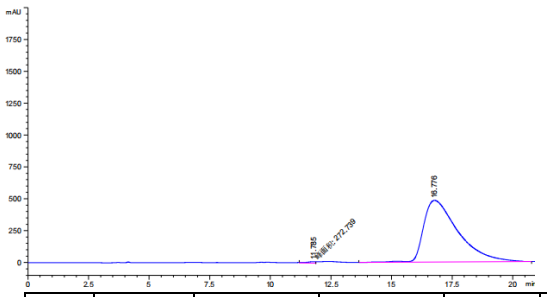
2-(1^4-((2,2-diphenylpropanoyl)oxy)-1^2,3^2,5^2,7^2-tetrapropoxy-1,3,5,7(1,3)-tetrabenzenacyclooctaphane-1^5-carboxamido)pyridine 1-oxide (3ag)



Yield: 75.2 mg (79%), White solid, mp: 134 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 10.47 (s, 1H), 8.56 (dd, *J* = 8.5, 1.4 Hz, 1H), 8.29 (d, *J* = 6.3 Hz, 1H), 7.52 (s, 1H), 7.41 (t, *J* = 8.0 Hz, 1H), 7.35 (dd, *J* = 7.2, 5.0 Hz, 4H), 7.21 (t, *J* = 7.5 Hz, 2H), 7.18-7.10 (m, 3H), 7.10-7.05 (m, 3H), 7.05-6.99 (m, 1H), 6.90 (t, *J* = 7.4 Hz, 1H), 6.27 (t, *J* = 7.5 Hz, 1H), 6.22 (t, *J* = 7.6 Hz, 1H), 6.17-6.09 (m, 3H), 6.01 (d, *J* = 7.4 Hz, 1H), 4.50-4.35 (m, 3H), 4.07 (td, *J* = 11.4, 3.3 Hz, 2H), 4.02-3.92 (m, 2H), 3.83 (d, *J* = 14.0 Hz, 1H), 3.72-3.57 (m, 3H), 3.47 (dt, *J* = 9.4, 6.9 Hz, 1H), 3.23 (d, *J* = 13.8 Hz, 1H), 3.15 (dd, *J* = 13.4, 5.9 Hz, 2H), 2.80 (d, *J* = 14.1 Hz, 1H), 2.18 (s, 3H), 1.98-1.76 (m, 8H), 1.08 (dt, *J* = 14.9, 7.4 Hz, 6H), 0.89 (t, *J* = 7.4 Hz, 6H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 173.4, 164.7, 162.4, 157.9, 155.2, 155.1, 146.3, 144.72, 144.68, 155.5, 143.7, 143.6, 137.2, 137.0, 136.8, 135.1, 133.5, 133.0, 132.0, 131.9, 131.8, 128.93, 128.91, 128.29, 128.25, 128.18, 128.01, 128.00, 127.9, 127.8, 127.6, 127.5, 127.4, 126.9, 126.8, 126.7, 122.4, 121.9, 121.4, 118.5, 115.0, 76.7, 76.5, 56.9, 31.0, 30.9, 30.7, 26.7, 23.5, 23.35, 23.30, 23.05, 23.01, 10.8, 10.76, 9.9, 9.8. **HRMS** (ESI): m/z $[M+H]^+$calcd for $[C_{61}H_{65}N_2O_8]^+$ requires 953.4741, found 953.4749. $[\alpha]_D^{25}$ = +30 (c = 0.1, CH_2Cl_2). The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (CHIRALPAK AD-H, hexane/*i*-PrOH = 95/5, detector: 254 nm, T = 25 °C, flow rate: 1 mL/min), t_1 (major) = 16.776 min, t_2 (minor) = 11.785 min.

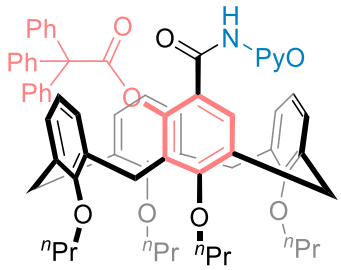


Peak	RetTime	Area	Height	Area
1	11.457	9718.85449	149.14948	51.0610
2	17.104	9314.95117	95.91405	48.9390



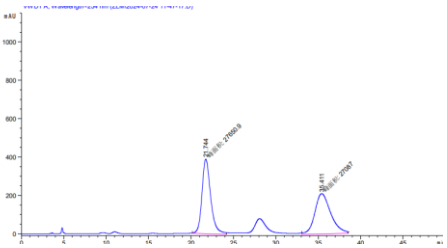
Peak	RetTime	Area	Height	Area
1	11.785	272.73871	10.67186	0.5661
2	16.776	17803.8	483.92004	99.4339

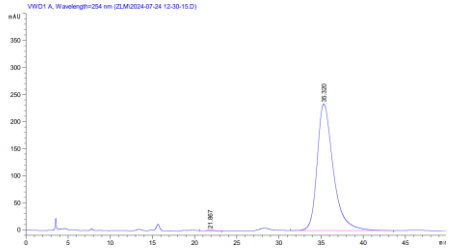
2-(1^2,3^2,5^2,7^2-tetrapropoxy-1^4-(2,2,2-triphenylacetoxy)-1,3,5,7(1,3)-tetrabenzenacyclooctaphane-1^5-carboxamido)pyridine 1-oxide (3ah)



Yield: 76.1 mg (75%), White solid, mp: 145 °C.

^{1}H NMR (600 MHz, $CDCl_3$) δ 10.34 (s, 1H), 8.48 (d, *J* = 7.2 Hz, 1H), 8.23 (d, *J* = 3.0 Hz, 1H), 7.44 (s, 1H), 7.40 (d, *J* = 7.8 Hz, 6H), 7.36 (br, 1H), 7.20-6.91 (m, 12H), 6.87 (t, *J* = 7.4 Hz, 1H), 6.28 (t, *J* = 7.5 Hz, 1H), 6.15 (dd, *J* = 14.1, 7.0 Hz, 3H), 6.08 (d, *J* = 7.3 Hz, 1H), 5.83 (d, *J* = 7.3 Hz, 1H), 4.50-4.34 (m, 3H), 4.04 (dt, *J* = 8.7, 5.7 Hz, 2H), 3.95 (dd, *J* = 8.9, 7.3 Hz, 2H), 3.79-3.53 (m, 4H), 3.51-3.39 (m, 1H), 3.27-3.15 (m, 1H), 3.12 (d, *J* = 13.1 Hz, 2H), 2.69 (d, *J* = 14.0 Hz, 1H), 1.97-1.71 (m, 8H), 1.06 (dt, *J* = 15.1, 7.4 Hz, 6H), 0.88 (td, *J* = 7.4, 3.0 Hz, 6H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 171.9, 165.1, 162.0, 157.8, 155.1, 155.0, 146.1, 144.4, 142.3, 137.0, 136.8, 136.7, 134.9, 133.4, 132.9, 131.9, 131.83, 131.80, 130.6, 128.9, 128.8, 128.7, 127.8, 127.7, 127.5, 127.4, 127.39, 126.8, 122.3, 122.2, 121.9, 121.8, 118.4, 114.8, 76.9, 76.6, 76.4, 68.1, 60.3, 30.89, 30.85, 30.6, 23.4, 23.2, 23.1, 22.95, 22.92, 10.68, 10.67, 9.8, 9.7. **HRMS** (ESI): m/z $[M+H]^+$calcd for $[C_{66}H_{67}N_2O_8]^+$ requires 1015.4897, found 1015.4895. $[\alpha]_D^{25}$ = +27 (c = 0.1, CH_2Cl_2). The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (CHIRALPAK IC-H, hexane/*i*-PrOH = 90/10, detector: 254 nm, T = 25 °C, flow rate: 1 mL/min), t_1 (major) = 35.320 min, t_2 (minor) = 25.867 min

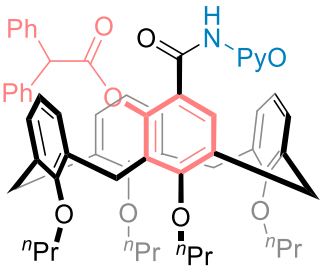




Peak	RetTime	Area	Height	Area
1	21.743	25996.6	382.59467	49.9607
2	35.411	26037.5	205.55771	50.0393

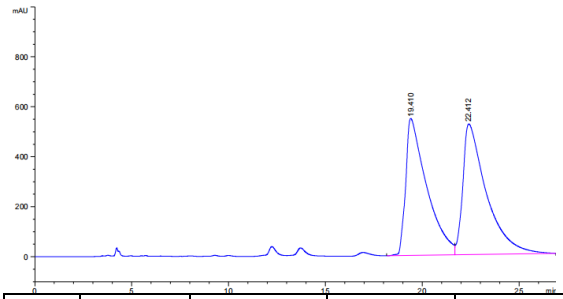
Peak	RetTime	Area	Height	Area
1	21.867	64.27583	1.03985	0.2067
2	35.320	31032.4	233.59198	99.7933

2-(1^4-(2,2-diphenylacetoxy)-1^2,3^2,5^2,7^2-tetrapropoxy-1,3,5,7(1,3)-tetrabenzenacyclooctaphane-1^5-carboxamido)pyridine 1-oxide (3ai)

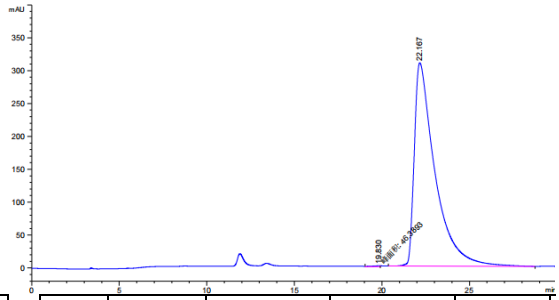


Yield: 66.6 mg (71%), White solid, mp: 132 °C.

^{1}H NMR (600 MHz, $CDCl_3$) δ 10.90 (s, 1H), 8.42 (d, *J* = 7.6 Hz, 1H), 8.22 (d, *J* = 6.1 Hz, 1H), 7.55 (s, 1H), 7.44 (d, *J* = 7.3 Hz, 2H), 7.35 (d, *J* = 7.4 Hz, 2H), 7.27 (dt, *J* = 30.1, 7.6 Hz, 4H), 7.17 (t, *J* = 7.3 Hz, 1H), 7.08 (dt, *J* = 25.0, 7.1 Hz, 3H), 7.00-6.85 (m, 3H), 6.78 (t, *J* = 7.3 Hz, 1H), 6.31 (ddt, *J* = 21.5, 15.5, 7.9 Hz, 5H), 6.11 (d, *J* = 7.3 Hz, 1H), 5.67 (s, 1H), 4.48-4.37 (m, 3H), 4.12-3.99 (m, 2H), 3.93 (dd, *J* = 15.1, 7.4 Hz, 3H), 3.81-3.59 (m, 3H), 3.53 (dd, *J* = 16.6, 7.2 Hz, 1H), 3.24 (d, *J* = 13.7 Hz, 1H), 3.15 (d, *J* = 12.9 Hz, 2H), 2.94 (d, *J* = 14.1 Hz, 1H), 2.00-1.83 (m, 6H), 1.88-1.69 (m, 2H), 1.26 (s, 1H), 1.03 (dt, *J* = 26.6, 7.4 Hz, 6H), 0.91 (t, *J* = 7.5 Hz, 6H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 171.1, 164.1, 162.3, 157.6, 155.5, 155.4, 145.7, 144.5, 138.1, 137.8, 136.9, 136.6, 136.3, 135.1, 134.0, 133.6, 132.5, 132.1, 131.3, 129.7, 128.9, 128.7, 128.67, 128.59, 128.2, 128.0, 127.9, 127.77, 127.72, 127.6, 127.4, 127.1, 122.4, 122.2, 121.9, 120.9, 118.4, 115.0, 76.9, 76.6, 76.5, 56.4, 31.0, 30.9, 30.7, 23.4, 23.2, 23.1, 23.0, 10.6, 10.5, 10.0, 9.9. **HRMS** (ESI): m/z $[M+H]^+$calcd for $[C_{60}H_{63}N_2O_8]^+$ requires 939.4584, found 939.4585. $[\alpha]_D^{25}$ = +39 (c = 0.1, CH_2Cl_2). The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (CHIRALPAK AD-H, hexane/*i*-PrOH = 95/5, detector: 254 nm, T = 25 °C, flow rate: 1 mL/min), t_1 (major) = 22.167 min, t_2 (minor) = 19.830 min.

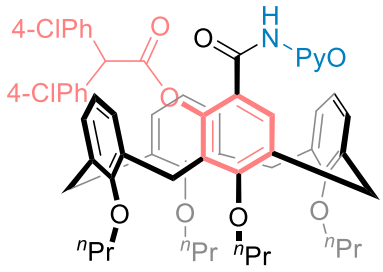


Peak	RetTime	Area	Height	Area
1	19.410	41406.4	548.28741	48.4082
2	22.412	44129.5	522.83246	51.5918



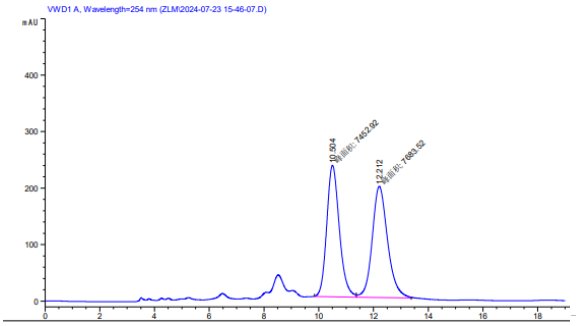
Peak	RetTime	Area	Height	Area
1	19.830	46.38927	1.32011	0.1822
2	22.167	25418.0	309.09381	99.8178

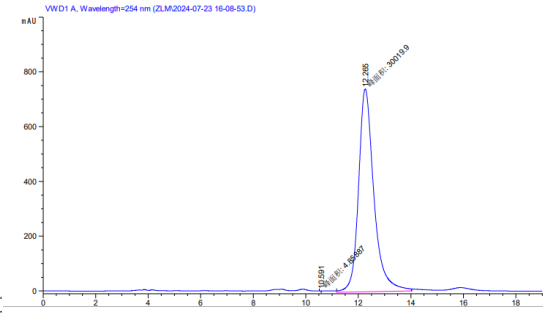
2-(1^4-(2,2-bis(4-chlorophenyl)acetoxy)-1^2,3^2,5^2,7^2-tetrapropoxy-1,3,5,7(1,3)-tetrabenzenacyclooctaphane-1^5-carboxamido)pyridine 1-oxide (3aj)



Yield: 73.5 mg (73%), White solid, mp: 140 °C.

^{1}H NMR (600 MHz, $CDCl_3$) δ 10.80 (s, 1H), 8.37 (d, *J* = 8.1 Hz, 1H), 8.18 (d, *J* = 6.3 Hz, 1H), 7.52 (s, 1H), 7.34 (dd, *J* = 15.5, 7.8 Hz, 3H), 7.27 (s, 1H), 7.22 (d, *J* = 8.2 Hz, 2H), 7.07 (d, *J* = 8.4 Hz, 2H), 7.01-6.92 (m, 2H), 6.90 (s, 1H), 6.77 (d, *J* = 6.9 Hz, 1H), 6.38 (t, *J* = 7.4 Hz, 1H), 6.31 (dd, *J* = 16.7, 8.0 Hz, 4H), 6.13 (d, *J* = 7.2 Hz, 1H), 5.57 (s, 1H), 4.50-4.38 (m, 3H), 4.03 (ddd, *J* = 31.6, 17.8, 8.0 Hz, 3H), 3.96-3.88 (m, 2H), 3.88-3.63 (m, 3H), 3.56 (dd, *J* = 16.5, 7.1 Hz, 1H), 3.24 (d, *J* = 13.7 Hz, 1H), 3.16 (dd, *J* = 13.5, 6.7 Hz, 2H), 2.89 (d, *J* = 14.1 Hz, 1H), 1.98-1.75 (m, 8H), 1.04 (dt, *J* = 17.8, 7.4 Hz, 6H), 0.92 (t, *J* = 7.4 Hz, 6H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 170.4, 163.9, 162.3, 157.4, 155.6, 145.3, 144.2, 136.9, 136.4, 136.2, 135.9, 135.2, 134.2, 134.0, 133.7, 133.3, 132.5, 131.9, 131.0, 130.1, 129.9, 129.8, 128.9, 128.7, 128.6, 128.1, 127.9, 127.6, 127.5, 122.4, 122.2, 122.0, 120.8, 118.5, 114.9, 76.7, 76.6, 55.0, 31.0, 30.9, 30.7, 23.4, 23.2, 23.10, 23.09, 10.63, 10.59, 10.0, 9.9. **HRMS** (ESI): m/z $[M+H]^+$calcd for $[C_{60}H_{61}Cl_2N_2O_8]^+$ requires 1007.3808, found 1007.3815. $[\alpha]_D^{25}$ = +34 (c = 0.1, CH_2Cl_2). The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (CHIRALPAK IC-H, hexane/*i*-PrOH = 90/10, detector: 254 nm, T = 25 °C, flow rate: 1 mL/min), t_1 (major) = 12.265 min, t_2 (minor) = 10.591 min.

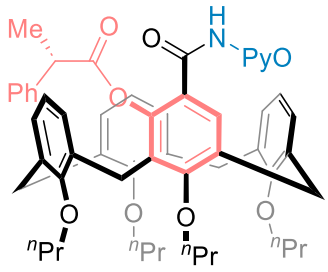




Peak	RetTime	Area	Height	Area
1	10.504	7452.92139	232.79915	49.2383
2	35.320	7683.51904	196.75146	50.7617

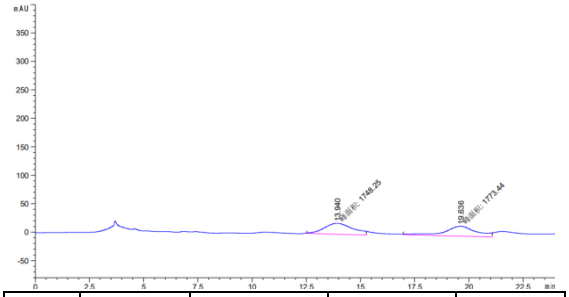
Peak	RetTime	Area	Height	Area
1	10.591	4.85887	1.68972	0.0162
2	12.265	30019.9	740.54327	99.9838

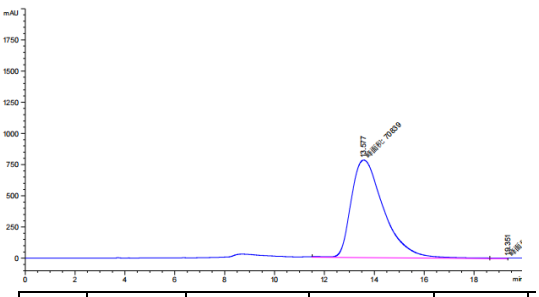
(S)-2-(1^4-((2-phenylpropanoyl)oxy)-1^2,3^2,5^2,7^2-tetrapropoxy-1,3,5,7(1,3)-tetrabenzenacyclooctaphane-1^5-carboxamido)pyridine 1-oxide (3ak)



Yield: 56.1 mg (64%), White solid, mp: 123 °C.

^{1}H NMR (600 MHz, $CDCl_3$) δ 10.72 (s, 1H), 8.46 (d, *J* = 8.1 Hz, 1H), 8.27 (d, *J* = 6.2 Hz, 1H), 7.59 (s, 1H), 7.34 (dd, *J* = 10.9, 3.8 Hz, 3H), 7.18-6.96 (m, 6H), 6.85 (t, *J* = 7.4 Hz, 1H), 6.31 (dt, *J* = 11.1, 7.6 Hz, 2H), 6.19 (dd, *J* = 22.7, 6.8 Hz, 4H), 4.48-4.38 (m, 3H), 4.30 (d, *J* = 7.1 Hz, 1H), 4.05 (dt, *J* = 10.7, 5.3 Hz, 2H), 3.94 (dd, *J* = 16.5, 8.5 Hz, 3H), 3.74-3.58 (m, 3H), 3.53 (d, *J* = 8.6 Hz, 1H), 3.24 (d, *J* = 13.8 Hz, 1H), 3.15 (d, *J* = 13.5 Hz, 2H), 2.94 (d, *J* = 13.2 Hz, 1H), 2.00-1.75 (m, 8H), 1.60 (d, *J* = 7.1 Hz, 3H), 1.05 (dt, *J* = 19.9, 7.4 Hz, 6H), 0.89 (dd, *J* = 13.1, 7.4 Hz, 6H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 173.0, 164.2, 162.5, 157.8, 155.34, 155.32, 145.9, 144.6, 139.7, 137.0, 136.8, 136.6, 135.1, 133.7, 133.3, 132.3, 131.9, 131.4, 129.4, 128.9, 128.8, 128.3, 127.91, 127.89, 127.84, 127.80, 127.6, 127.4, 127.1, 122.45, 122.42, 121.9, 120.9, 118.4, 115.1, 77.0, 76.7, 76.6, 45.5, 31.0, 30.9, 30.7, 23.5, 23.3, 23.0, 18.5, 10.73, 10.67, 9.9, 9.8. **HRMS** (ESI): m/z $[M+H]^+$calcd for $[C_{55}H_{61}N_2O_8]^+$ requires 877.4428, found 877.4426. $[\alpha]_D^{25}$ = +89 (c = 0.1, CH_2Cl_2). The product was analyzed by HPLC to determine the enantiomeric excess: >99% de (CHIRALPAK AD-H, hexane/*i*-PrOH = 95/5, detector: 254 nm, T = 25 °C, flow rate: 1 mL/min), t_1 (major) = 19.351 min, t_2 (minor) = 13.577 min.

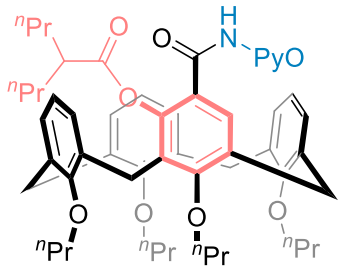




Peak	RetTime	Area	Height	Area
1	13.940	1748.24609	19.17593	49.6423
2	19.636	1773.43933	17.45177	50.3577

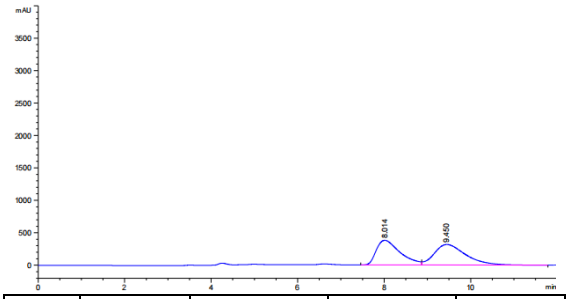
Peak	RetTime	Area	Height	Area
1	13.577	70839.0	780.81744	99.7801
2	19.351	156.12155	3.82582	0.2199

2-(1^2,3^2,5^2,7^2-tetrapropoxy-1^4-((2-propylpentanoyl)oxy)-1,3,5,7(1,3)-tetrabenzenacyclooctaphane-1^5-carboxamido)pyridine 1-oxide (3al)

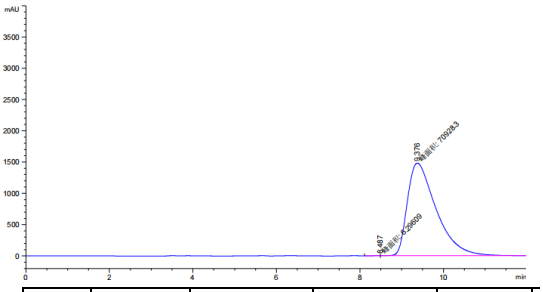


Yield: 67.9 mg (78%), White solid, mp: 116 °C.

^{1}H NMR (600 MHz, $CDCl_3$) δ 10.87 (s, 1H), 8.62 (dd, *J* = 8.5, 1.6 Hz, 1H), 8.29 (dd, *J* = 6.5, 0.9 Hz, 1H), 7.65 (s, 1H), 7.41-7.34 (m, 1H), 7.09 (d, *J* = 7.5 Hz, 2H), 7.04-6.98 (m, 1H), 6.91 (t, *J* = 7.4 Hz, 1H), 6.28 (t, *J* = 7.6 Hz, 2H), 6.21 (d, *J* = 7.2 Hz, 1H), 6.15 (t, *J* = 6.6 Hz, 3H), 4.50-4.40 (m, 3H), 4.21-4.06 (m, 3H), 4.03-3.96 (m, 2H), 3.75-3.59 (m, 4H), 3.39 (d, *J* = 14.0 Hz, 1H), 3.26 (d, *J* = 13.7 Hz, 1H), 3.16 (dd, *J* = 13.4, 2.3 Hz, 2H), 3.02-2.91 (m, 1H), 2.02-1.81 (m, 8H), 1.80-1.66 (m, 2H), 1.61-1.45 (m, 2H), 1.45-1.18 (m, 4H), 1.08 (dt, *J* = 16.4, 7.4 Hz, 6H), 0.94-0.85 (m, 6H), 0.82 (t, *J* = 7.3 Hz, 3H), 0.75 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 174.8, 164.6, 162.5, 157.9, 155.2, 155.1, 146.0, 144.8, 137.1, 136.95, 136.90, 135.1, 133.5, 133.3, 132.0, 131.8, 131.7, 129.3, 128.9, 127.9, 127.8, 127.4, 127.3, 122.5, 122.4, 121.9, 121.3, 118.5, 115.0, 76.95, 76.93, 76.5, 44.7, 33.8, 33.5, 31.0, 30.9, 30.7, 23.9, 23.5, 23.4, 23.1, 23.0, 20.4, 20.3, 14.0, 13.98, 10.8, 10.7, 9.87, 9.80. **HRMS** (ESI): m/z $[M+H]^+$calcd for $[C_{54}H_{67}N_2O_8]^+$ requires 871.4897, found 871.4893. $[\alpha]_D^{25}$ = +42 (c = 0.1, CH_2Cl_2). The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (CHIRALPAK AD-H, hexane/*i*-PrOH = 95/5, detector: 254 nm, T = 25 °C, flow rate: 1 mL/min), t_1 (major) = 9.376 min, t_2 (minor) = 8.487 min.

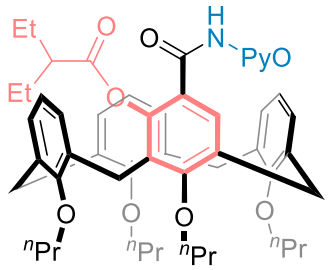


Peak	RetTime	Area	Height	Area
1	8.014	14455.6	379.66327	47.4317
2	9.450	16021.1	316.71589	52.5683



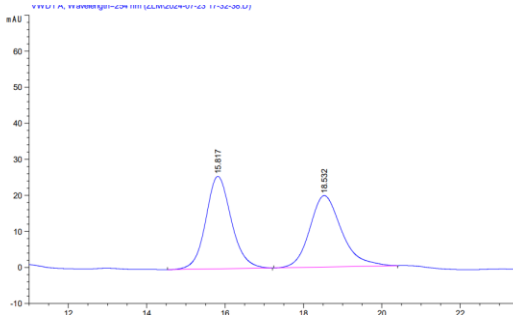
Peak	RetTime	Area	Height	Area
1	8.487	6.29609	1.14416	0.0089
2	9.376	70928.3	1479.99548	99.9911

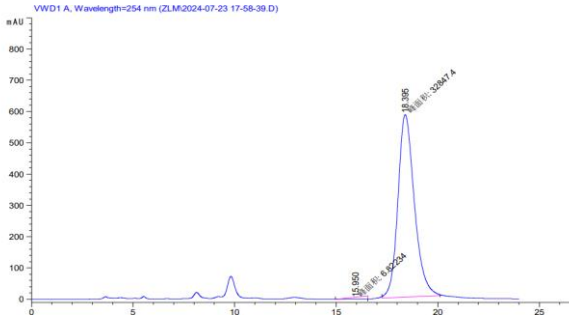
2-(1^4-((2-ethylbutanoyl)oxy)-1^2,3^2,5^2,7^2-tetrapropoxy-1,3,5,7(1,3)-tetrabenzenacyclooctaphane-1^5-carboxamido)pyridine 1-oxide (3am)



Yield: 57.3 mg (54%), White solid, mp: 112 °C.

^{1}H NMR (600 MHz, $CDCl_3$) δ 10.90 (s, 1H), 8.63 (dd, *J* = 8.5, 1.5 Hz, 1H), 8.34 (dd, *J* = 6.5, 0.8 Hz, 1H), 7.68 (s, 1H), 7.44-7.35 (m, 1H), 7.09 (d, *J* = 7.4 Hz, 2H), 7.08-6.98 (m, 1H), 6.91 (t, *J* = 7.5 Hz, 1H), 6.28 (td, *J* = 7.6, 2.4 Hz, 2H), 6.22 (d, *J* = 7.0 Hz, 1H), 6.16 (dd, *J* = 8.2, 3.8 Hz, 3H), 4.52-4.40 (m, 3H), 4.26-4.08 (m, 3H), 4.01 (dd, *J* = 15.0, 6.8 Hz, 2H), 3.85-3.50 (m, 4H), 3.41 (d, *J* = 14.0 Hz, 1H), 3.27 (d, *J* = 13.7 Hz, 1H), 3.16 (dd, *J* = 13.4, 4.1 Hz, 2H), 2.93-2.81 (m, 1H), 1.97 (dp, *J* = 15.3, 7.7 Hz, 4H), 1.92-1.84 m, 4H), 1.83-1.73 (m, 2H), 1.71-1.59 (m, 2H), 1.13-1.05 (m, 6 H), 0.97-0.87 (m, 9H), 0.84 (t, *J* = 7.5 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 174.4, 164.5, 162.5, 157.8, 155.2, 155.1, 146.0, 144.7, 137.1, 136.84, 136.82, 135.0, 133.4, 133.3, 132.0, 131.8, 131.6, 129.4, 128.9, 128.2, 127.80, 127.77, 127.4, 127.3, 122.5, 122.3, 121.9, 121.0, 118.5, 115.0, 76.5, 47.7, 30.92, 30.90, 30.7, 29.5, 27.1, 24.1, 23.9, 23.7, 23.44, 23.37, 23.0, 22.9, 11.4, 11.3, 10.7, 10.7, 9.8, 9.7. **HRMS** (ESI): m/z $[M+H]^+$calcd for $[C_{52}H_{63}N_2O_8]^+$ requires 843.4584, found 843.4602. $[\alpha]_D^{25}$ = +23 (c = 0.1, CH_2Cl_2). The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (CHIRALPAK IC-H, hexane/*i*-PrOH = 90/10, detector: 254 nm, T = 25 °C, flow rate: 1 mL/min), t_1 (major) = 18.395 min, t_2 (minor) = 15.950 min.

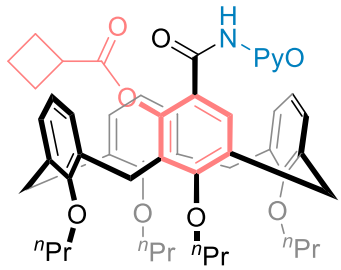




Peak	RetTime	Area	Height	Area
1	15.817	1162.22095	25.67161	51.0181
2	18.532	1115.83569	19.79645	48.9819

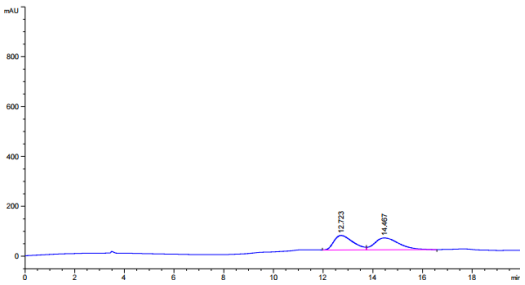
Peak	RetTime	Area	Height	Area
1	15.950	6.82234	4.84642	0.0208
2	18.395	32847.4	583.38098	99.9792

2-(1^4-((cyclobutanecarbonyl)oxy)-1^2,3^2,5^2,7^2-tetrapropoxy-1,3,5,7(1,3)-tetrabenzenacyclooctaphane-1^5-carboxamido)pyridine 1-oxide (3an)

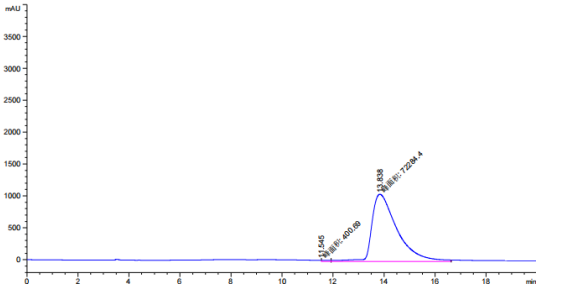


Yield: 44.6 mg (54%), White solid, mp: 118 °C.

^{1}H NMR (600 MHz, $CDCl_3$) δ 10.83 (s, 1H), 8.60 (d, *J* = 8.0 Hz, 1H), 8.28 (d, *J* = 6.3 Hz, 1H), 7.59 (s, 1H), 7.36 (t, *J* = 7.9 Hz, 1H), 7.06-6.90 (m, 3H), 6.81 (t, *J* = 7.4 Hz, 1H), 6.36 (dd, *J* = 15.2, 7.6 Hz, 2H), 6.31-6.22 (m, 4H), 4.51-4.39 (m, 3H), 4.19 (d, *J* = 13.9 Hz, 1H), 4.15-4.03 (m, 2H), 3.96 (dd, *J* = 8.9, 7.1 Hz, 2H), 3.78-3.63 (m, 4H), 3.34 (d, *J* = 14.0 Hz, 1H), 3.25 (d, *J* = 13.7 Hz, 1H), 3.16 (dd, *J* = 13.4, 5.2 Hz, 2H), 2.49-2.17 (m, 4H), 2.12-1.77 (m, 11H), 1.06 (dt, *J* = 11.6, 7.4 Hz, 6H), 0.93 (q, *J* = 7.4 Hz, 6H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 173.9, 164.3, 162.3, 157.5, 155.55, 155.52, 146.1, 144.8, 137.0, 136.5, 136.4, 134.7, 134.0, 133.9, 132.5, 132.1, 131.1, 129.6, 128.7, 128.1, 128.08, 127.9, 127.6, 122.38, 122.36, 122.0, 120.7, 118.5, 115.1, 76.8, 76.5, 38.1, 31.0, 30.98, 30.7, 25.5, 25.1, 23.5, 23.3, 23.2, 23.08, 18.5, 10.7, 10.6, 10.0, 9.9. **HRMS** (ESI): m/z $[M+H]^+$calcd for $[C_{51}H_{59}N_2O_8]^+$ requires 827.4271, found 827.4281. $[\alpha]_D^{25}$ = +18 (c = 0.1, CH_2Cl_2). The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (CHIRALPAK AD-H, hexane/*i*-PrOH = 95/5, detector: 254 nm, T = 25 °C, flow rate: 1 mL/min), t_1 (major) = 13.838 min, t_2 (minor) = 11.545 min.

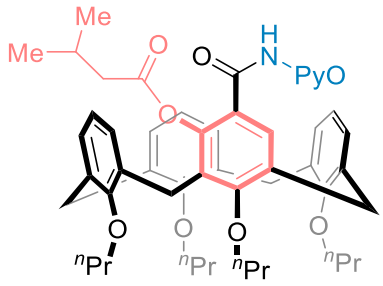


Peak	RetTime	Area	Height	Area
1	12.723	3079.31274	57.93274	48.9534
2	14.467	3210.98706	47.29651	51.0466



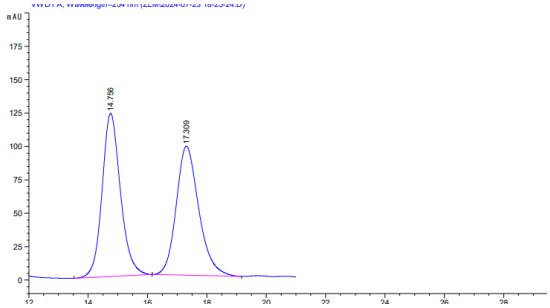
Peak	RetTime	Area	Height	Area
1	11.545	400.68967	17.87404	0.5513
2	13.838	72284.4	1052.42639	99.4487

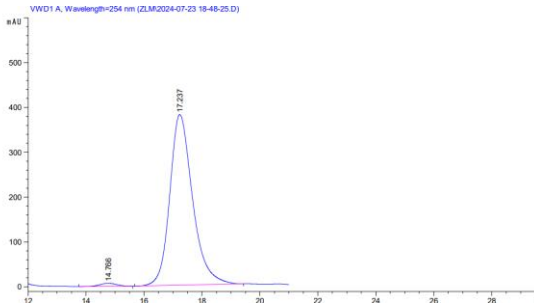
2-(1^4-((3-methylbutanoyl)oxy)-1^2,3^2,5^2,7^2-tetrapropoxy-1,3,5,7(1,3)-tetrabenzenacyclooctaphane-1^5-carboxamido)pyridine 1-oxide (3ao)



Yield: 43.8 mg (52%), White solid, mp: 113 °C.

^{1}H NMR (600 MHz, $CDCl_3$) δ 10.90 (s, 1H), 8.60 (d, *J* = 8.2 Hz, 1H), 8.29 (d, *J* = 6.0 Hz, 1H), 7.61 (s, 1H), 7.36 (t, *J* = 7.9 Hz, 1H), 7.08-6.91 (m, 3H), 6.81 (t, *J* = 7.5 Hz, 1H), 6.43-6.09 (m, 6H), 4.51-4.39 (m, 3H), 4.19 (d, *J* = 14.0 Hz, 1H), 4.15-4.03 (m, 2H), 3.96 (dd, *J* = 13.9, 6.0 Hz, 2H), 3.89-3.49 (m, 4H), 3.34 (d, *J* = 14.0 Hz, 1H), 3.26 (d, *J* = 13.6 Hz, 1H), 3.16 (dd, *J* = 13.5, 2.8 Hz, 2H), 2.81-2.60 (m, 2H), 2.25-2.15 (m, 1 H), 2.01-1.80 (m, 8H), 1.10-1.02 (m, 6H), 1.00-0.89 (m, 12H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 171.6, 164.3, 162.4, 157.5, 155.53, 155.51, 146.2, 144.8, 137.0, 136.5, 136.4, 134.7, 134.0, 133.9, 132.5, 132.1, 131.1, 129.7, 128.8, 128.1, 128.0, 127.9, 127.6, 122.4, 122.3, 122.0, 120.6, 118.5, 115.0, 76.6, 42.9, 31.00, 30.98, 30.7, 29.4, 25.1, 24.7, 23.5, 23.3, 23.1, 23.08, 22.5, 22.3, 10.7, 10.6, 10.0, 9.9. **HRMS** (ESI): m/z $[M+H]^+$calcd for $[C_{51}H_{61}N_2O_8]^+$ requires 829.4428, found 829.4430. $[\alpha]_D^{25}$ = +34 (c = 0.1, CH_2Cl_2). The product was analyzed by HPLC to determine the enantiomeric excess: 97% ee (CHIRALPAK IC-H, hexane/*i*-PrOH = 90/10, detector: 254 nm, T = 25 °C, flow rate: 1 mL/min), t_1 (major) = 17.237 min, t_2 (minor) = 14.766 min.

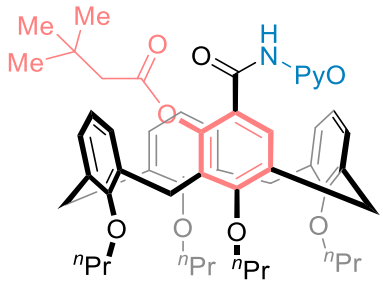




Peak	RetTime	Area	Height	Area
1	14.756	5249.07471	122.06635	50.7166
2	17.309	5100.74316	96.54317	49.2834

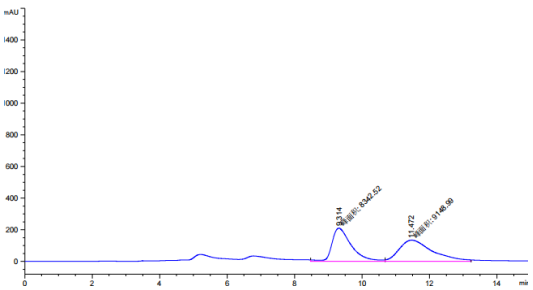
Peak	RetTime	Area	Height	Area
1	14.766	67.21010	6.95162	1.3322
2	17.237	20810.3	380.16534	98.6678

2-(1^4-((3,3-dimethylbutanoyl)oxy)-1^2,3^2,5^2,7^2-tetrapropoxy-1,3,5,7(1,3)-tetrabenzenacyclooctaphane-1^5-carboxamido)pyridine 1-oxide (3ap)

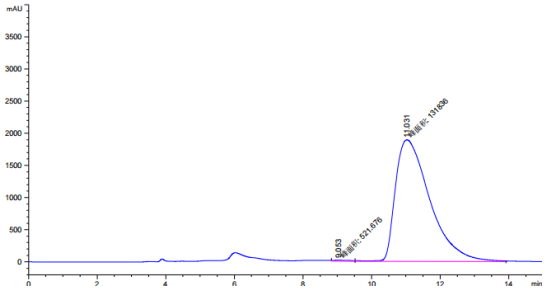


Yield: 52.2 mg (62%), White solid, mp: 116 °C.

^{1}H NMR (600 MHz, $CDCl_3$) δ 10.88 (s, 1H), 8.61 (d, *J* = 8.2 Hz, 1H), 8.29 (d, *J* = 6.2 Hz, 1H), 7.61 (s, 1H), 7.36 (t, *J* = 7.9 Hz, 1H), 6.99 (dd, *J* = 13.7, 6.6 Hz, 3H), 6.82 (t, *J* = 7.4 Hz, 1H), 6.40-6.18 (m, 6H), 4.51-4.39 (m, 3H), 4.19 (d, *J* = 14.0 Hz, 1H), 4.15-4.04 (m, 2H), 4.00-3.92 (m, 2H), 3.81-3.57 (m, 4H), 3.39 (d, *J* = 14.0 Hz, 1H), 3.25 (d, *J* = 13.7 Hz, 1H), 3.16 (dd, *J* = 13.4, 3.9 Hz, 2H), 2.72 (dd, *J* = 40.3, 16.0 Hz, 2H), 2.03-1.81 (m, 8H), 1.11 -0.99 (m, 16H), 0.97-0.87 (m, 6H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 170.6, 164.4, 162.4, 157.6, 155.5, 155.4, 146.1, 144.8, 137.0, 136.6, 136.5, 134.7, 133.9, 133.8, 132.4, 132.0, 131.2, 129.6, 128.8, 128.05, 128.02, 127.9, 127.6, 122.4, 122.0, 120.8, 118.5, 115.0, 76.9, 76.6, 46.9, 31.00, 30.98, 30.7, 30.5, 29.4, 24.7, 23.5, 23.3, 23.1, 23.07, 10.7, 10.6, 10.0, 9.9. **HRMS** (ESI): m/z $[M+H]^+$calcd for $[C_{52}H_{63}N_2O_8]^+$ requires 843.4584, found 843.4593. $[\alpha]_D^{25}$ = +28 (c = 0.1, CH_2Cl_2). The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (CHIRALPAK AD-H, hexane/*i*-PrOH = 97/3, detector: 254 nm, T = 25 °C, flow rate: 1 mL/min), t_1 (major) = 11.031 min, t_2 (minor) = 9.053 min.

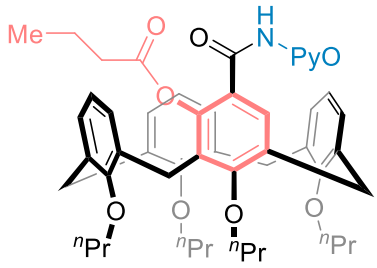


Peak	RetTime	Area	Height	Area
1	9.314	8342.52148	209.35483	47.6947
2	11.472	9148.99121	133.59216	52.3053



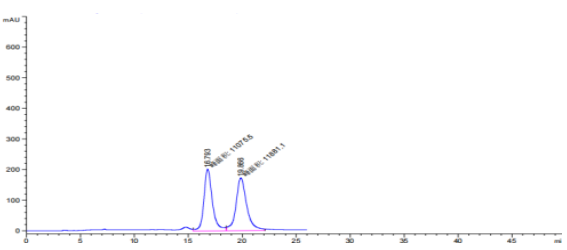
Peak	RetTime	Area	Height	Area
1	9.053	521.67603	14.43180	0.3941
2	11.031	131836	1889.42798	99.6059

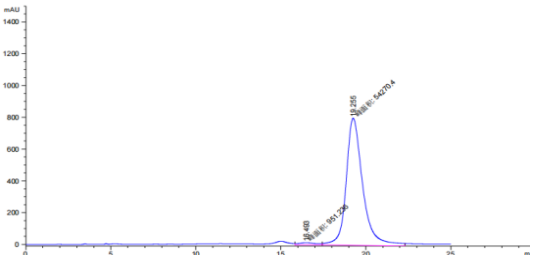
2-(14-(butyryloxy)-12,32,52,72-tetrapropoxy-1,3,5,7(1,3)-tetrabenzenacyclooctaphane-15-carboxamido)pyridine 1-oxide (3aq)



Yield: 25 mg (31%), Yellow solid, mp: 142 °C

^{1}H NMR (600 MHz, $CDCl_3$) δ 10.91 (s, 1H), 8.61 (dd, *J* = 8.5, 1.9 Hz, 1H), 8.29 (dd, *J* = 6.5, 1.5 Hz, 1H), 7.61 (s, 1H), 7.40–7.32 (m, 1H), 7.01–6.98 (m, 1H), 6.97–6.92 (m, 2H), 6.79 (t, *J* = 7.5 Hz, 1H), 6.40–6.35 (m, 2H), 6.33–6.27 (m, 4H), 4.49–4.42 (m, 3H), 4.20 (d, *J* = 14.0 Hz, 1H), 4.14–4.03 (m, 2H), 3.95 (t, *J* = 8.0 Hz, 2H), 3.78–3.70 (m, 4H), 3.32 (d, *J* = 14.0 Hz, 1H), 3.26 (d, *J* = 13.7 Hz, 1H), 3.17 (d, *J* = 3.8 Hz, 1H), 3.15 (d, *J* = 3.9 Hz, 1H), 2.85–2.72 (m, 2H), 1.97–1.92 (m, 4H), 1.90–1.85 (m, 4H), 1.78–1.72 (m, 2H), 1.08–1.03 (m, 6H), 0.9 –0.91 (m, 9H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 172.2, 164.2, 162.4, 157.5, 155.6, 146.2, 144.8, 137.1, 136.4, 136.3, 134.7, 134.1, 134.0, 132.6, 132.1, 131.1, 129.8, 128.7, 128.7, 128.5, 128.1, 128.0, 127.7, 122.4, 122.3, 122.0 120.5, 118.5, 115.1, 36.0, 31.0, 30.99, 30.7, 24.8, 23.4, 23.3, 23.2, 23.1, 18.0, 13.6, 10.6, 10.57, 10.0, 9.9. **HRMS** (ESI): m/z $[M+K]^+$calcd for $[C_{50}H_{58}N_2O_8K]^+$ requires 853.3825, found 853.3832. $[\alpha]_D^{25}$ = +22 (c = 0.1, CH_2Cl_2). The product was analyzed by HPLC to determine the enantiomeric excess: 96% ee (CHIRALPAK IC-H, hexane/*i*-PrOH = 90/10, detector: 254 nm, T = 25 °C, flow rate: 1 mL/min), t_1 (major) = 19.255 min, t_2 (minor) = 16.493 min.

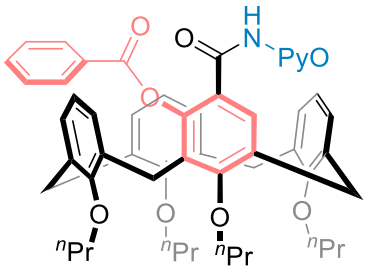




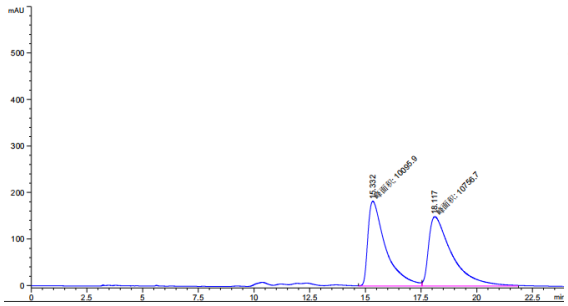
Peak	RetTime	Area	Height	Area
1	16.793	11075.5	201.88644	48.2453
2	19.866	11881.1	172.20108	51.7547

Peak	RetTime	Area	Height	Area
1	16.493	951.23639	13.37805	1.7226
2	19.255	54270.4	799.63397	98.2774

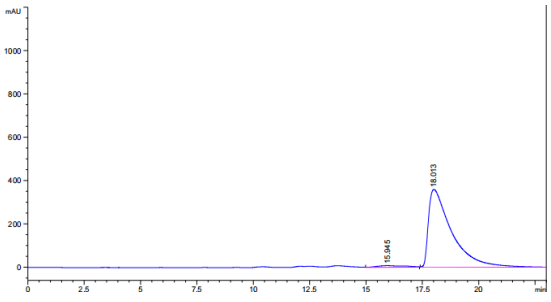
2-(1^4-(benzoyloxy)-1^2,3^2,5^2,7^2-tetrapropoxy-1,3,5,7(1,3)-tetrabenzenacyclooctaphane-1^5-carboxamido)pyridine 1-oxide (3ar)



Yield: 85.6 mg (72%), grey solid, mp: 121 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 10.58 (s, 1H), 8.43 (dd, *J* = 8.5, 1.5 Hz, 1H), 8.25-8.16 (m, 3H), 7.61 (t, *J* = 7.4 Hz, 1H), 7.49 (t, *J* = 7.8 Hz, 2H), 7.42 (s, 1H), 7.27-7.20 (m, 1H), 6.95-6.81 (m, 3H), 6.73 (t, *J* = 7.4 Hz, 1H), 6.60-6.24 (m, 6H), 4.56-4.40 (m, 3H), 4.25 (d, *J* = 13.9 Hz, 1H), 4.13-3.97 (m, 2H), 3.92 (dd, *J* = 8.3, 7.2 Hz, 2H), 3.88-3.67 (m, 4H), 3.38 (d, *J* = 14.0 Hz, 1H), 3.27 (d, *J* = 13.6 Hz, 1H), 3.18 (t, *J* = 13.0 Hz, 2H), 2.00-1.81 (m, 8H), 1.05 (t, *J* = 7.4 Hz, 3H), 0.97 (ddd, *J* = 20.0, 10.0, 4.4 Hz, 9H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 165.0, 164.4, 161.8, 157.2, 155.9, 155.88, 146.4, 144.6, 136.9, 136.0, 135.8, 134.6, 134.4, 133.6, 133.0, 132.4, 131.0, 130.8, 129.0, 128.96, 128.6, 128.5, 128.48, 128.33, 128.30, 127.9, 127.6, 122.4, 122.2, 122.1, 121.5, 118.3, 114.9, 77.2, 76.7, 76.6, 31.1, 31.0, 30.7, 25.3, 23.4, 23.2, 23.17, 23.14, 10.5, 10.4, 10.1, 10.0. **HRMS** (ESI): m/z $[M+H]^+$calcd for $[C_{53}H_{57}N_2O_8]^+$ requires 849.4115, found 849.4122. $[\alpha]_D^{25}$ = +32 (c = 0.1, CH_2Cl_2). The product was analyzed by HPLC to determine the enantiomeric excess: 95% ee (CHIRALPAK IB-H, hexane/*i*-PrOH = 90/10, detector: 254 nm, T = 25 °C, flow rate: 1 mL/min), t_1 (major) = 18.013 min, t_2 (minor) = 15.945 min.

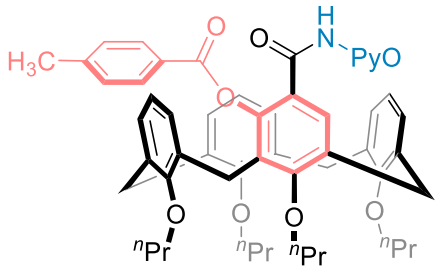


Peak	RetTime	Area	Height	Area
1	15.332	10095.9	182.10905	48.4155
2	18.117	10756.7	148.70924	51.5845

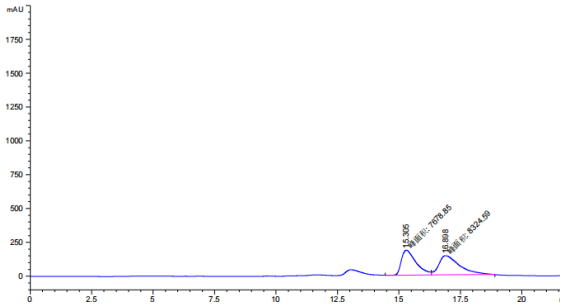


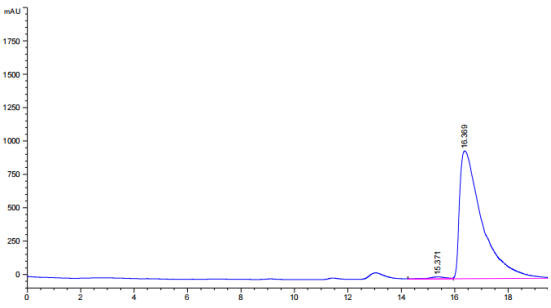
Peak	RetTime	Area	Height	Area
1	15.945	682.77399	7.81819	2.6288
2	18.013	25289.6	357.24292	97.3712

2-(1^4-((4-methylbenzoyl)oxy)-1^2,3^2,5^2,7^2-tetrapropoxy-1,3,5,7(1,3)-tetrabenzenacyclooctaphane-1^5-carboxamido)pyridine 1-oxide (3as)



Yield: 73.7 mg (61%), white solid, mp: 122 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 10.61 (s, 1H), 8.45 (dd, *J* = 8.5, 1.4 Hz, 1H), 8.19 (d, *J* = 5.9 Hz, 1H), 8.12 (d, *J* = 8.1 Hz, 2H), 7.45 (s, 1H), 7.29 (d, *J* = 8.1 Hz, 2H), 7.23 (dd, *J* = 12.3, 4.7 Hz, 1H), 6.95-6.82 (m, 3H), 6.75 (t, *J* = 7.4 Hz, 1H), 6.58-6.29 (m, 6H), 4.56-4.40 (m, 3H), 4.24 (d, *J* = 13.9 Hz, 1H), 4.07 (dtd, *J* = 18.6, 10.3, 8.1 Hz, 2H), 3.93 (dd, *J* = 8.3, 7.2 Hz, 2H), 3.86-3.67 (m, 4H), 3.38 (d, *J* = 13.9 Hz, 1H), 3.28 (d, *J* = 13.6 Hz, 1H), 3.18 (t, *J* = 12.8 Hz, 2H), 2.43 (s, 3H), 2.01-1.81 (m, 8H), 1.06 (t, *J* = 7.4 Hz, 3H), 1.02-0.93 (m, 9H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 165.1, 164.4, 161.9, 157.2, 155.9, 155.8, 146.5, 144.6, 144.5, 136.9, 136.1, 135.9, 134.6, 134.4, 134.36, 133.0, 132.4, 131.0, 130.8, 129.2, 129.1, 128.6, 128.5, 128.3, 128.28, 127.9, 127.6, 126.2, 122.4, 122.3, 122.1, 121.5, 118.3, 115.0, 77.2, 76.7, 76.6, 31.07, 31.03, 30.7, 25.3, 23.4, 23.2, 23.16, 21.8, 10.6, 10.4, 10.1, 10.0. **HRMS** (ESI): m/z $[M+H]^+$calcd for $[C_{53}H_{57}N_2O_8]^+$ requires 863.4271, found 863.4275. $[\alpha]_D^{25}$ = +26 (c = 0.1, CH_2Cl_2). The product was analyzed by HPLC to determine the enantiomeric excess: 98% ee (CHIRALPAK IB-H, hexane/*i*-PrOH = 90/10, detector: 254 nm, T = 25 °C, flow rate: 1 mL/min), t_1 (major) = 16.369 min, t_2 (minor) = 15.371 min.

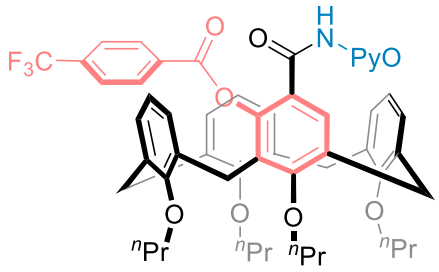




Peak	RetTime	Area	Height	Area
1	15.305	7678.85352	184.89508	47.9825
2	16.898	8324.58594	141.65874	52.0175

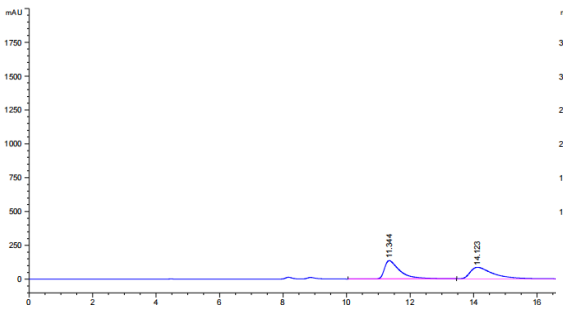
Peak	RetTime	Area	Height	Area
1	15.371	655.08588	14.85541	1.1636
2	16.369	55643.4	957.31390	98.8364

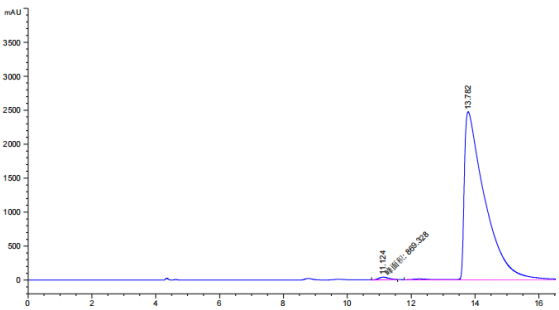
2-(1^2,3^2,5^2,7^2-tetrapropoxy-1^4-((4-(trifluoromethyl)benzoyl)oxy)-1,3,5,7(1,3)-tetrabenzenacyclooctaphane-1^5-carboxamido)pyridine 1-oxide (3at)



Yield: 66.7 mg (52%). White solid, mp: 126 °C.

^{1}H NMR (600 MHz, $CDCl_3$) δ 10.48 (s, 1H), 8.38 (dd, *J* = 8.5, 1.6 Hz, 1H), 8.29 (d, *J* = 8.2 Hz, 2H), 8.20 (dd, *J* = 6.5, 0.9 Hz, 1H), 7.75 (d, *J* = 8.3 Hz, 2H), 7.33 (s, 1H), 7.26-7.20 (m, 1H), 6.95-6.89 (m, 1H), 6.78 (dd, *J* = 15.8, 7.1 Hz, 2H), 6.67 (t, *J* = 7.4 Hz, 1H), 6.62-6.47 (m, 4H), 6.44-6.30 (m, 2H), 4.56-4.41 (m, 3H), 4.29 (d, *J* = 14.0 Hz, 1H), 4.13-3.95 (m, 2H), 3.89 (dd, *J* = 16.8, 9.4 Hz, 3H), 3.78 (tt, *J* = 17.5, 8.7 Hz, 3H), 3.33 (d, *J* = 14.0 Hz, 1H), 3.27 (d, *J* = 13.6 Hz, 1H), 3.19 (t, *J* = 13.1 Hz, 2H), 2.23-1.70 (m, 8H), 0.99 (ddt, *J* = 22.4, 9.9, 7.4 Hz, 12H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 164.2, 163.8, 161.6, 156.9, 156.2, 156.1, 146.1, 144.4, 136.9, 135.7, 135.4, 135.1, 135.0, 134.97, 134.8, 134.4, 133.2, 132.4, 132.37, 131.1, 130.6, 128.8, 128.6, 128.57, 128.48, 128.37, 127.9, 127.7, 125.5, 125.48, 124.5, 122.7, 122.5, 122.15, 122.1, 121.2, 118.4, 114.8, 76.6, 76.6, 31.1, 31.0, 30.7, 25.8, 23.4, 23.3, 23.2, 23.0, 10.4, 10.3, 10.2, 10.1. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -63.1. **HRMS** (ESI): m/z $[M+H]^+$calcd for $[C_{54}H_{56}F_3N_2O_8]^+$ requires 917.3989, found 917.3992. $[\alpha]_D^{25}$ = +37 (c = 0.1, CH_2Cl_2). The product was analyzed by HPLC to determine the enantiomeric excess: 98% ee (CHIRALPAK IB-H, hexane/*i*-PrOH = 90/10, detector: 254 nm, T = 25 °C, flow rate: 1 mL/min), t_1 (major) = 13.782 min, t_2 (minor) = 11.124 min.

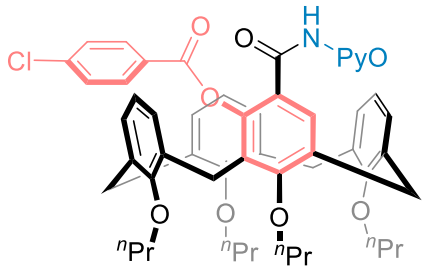




Peak	RetTime	Area	Height	Area
1	11.344	4534.72559	135.12341	51.2788
2	14.123	4308.55127	84.81761	48.7212

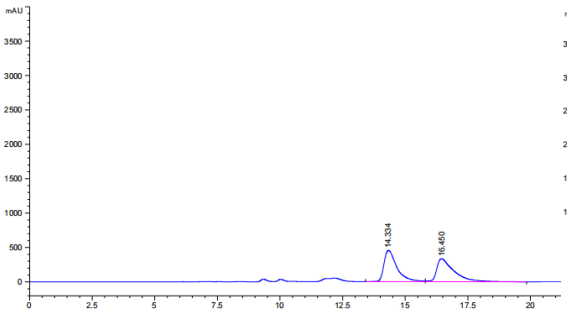
Peak	RetTime	Area	Height	Area
1	11.124	869.32764	39.08958	0.7849
2	13.782	109889	2471.35986	99.2151

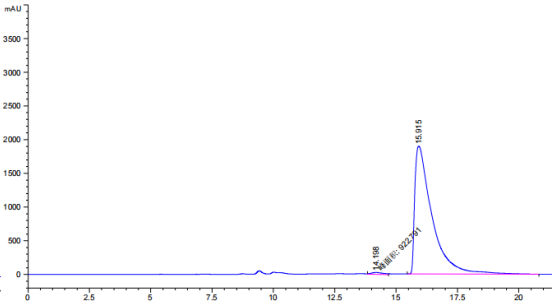
2-(1^4-((4-chlorobenzoyl)oxy)-1^2,3^2,5^2,7^2-tetrapropoxy-1,3,5,7(1,3)-tetrabenzenacyclooctaphane-1^5-carboxamido)pyridine 1-oxide (3au)



Yield: 69.2 mg (56%). White solid, mp: 126 °C.

^{1}H NMR (600 MHz, $CDCl_3$) δ 10.52 (s, 1H), 8.41 (d, *J* = 8.4 Hz, 1H), 8.20 (d, *J* = 6.3 Hz, 1H), 8.13 (d, *J* = 8.5 Hz, 2H), 7.46 (d, *J* = 8.5 Hz, 2H), 7.37 (s, 1H), 7.23 (d, *J* = 7.8 Hz, 1H), 6.92 (dd, *J* = 10.1, 3.9 Hz, 1H), 6.80 (s, 2H), 6.69 (t, *J* = 7.3 Hz, 1H), 6.53 (d, *J* = 32.3 Hz, 4H), 6.42 (s, 2H), 4.58-4.40 (m, 3H), 4.28 (d, *J* = 14.0 Hz, 1H), 4.04 (qd, *J* = 17.5, 9.2 Hz, 2H), 3.89 (dt, *J* = 16.9, 7.4 Hz, 3H), 3.77 (tt, *J* = 17.5, 8.7 Hz, 3H), 3.34 (d, *J* = 13.9 Hz, 1H), 3.27 (d, *J* = 13.6 Hz, 1H), 3.19 (dd, *J* = 13.2, 10.9 Hz, 2H), 1.92 (dddd, *J* = 50.9, 28.9, 14.8, 7.4 Hz, 8H), 1.07-0.94 (m, 12H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 164.2, 164.18, 161.7, 157.0, 156.1, 156.0, 146.2, 144.5, 140.2, 136.9, 135.8, 135.6, 134.9, 134.8, 134.4, 133.2, 132.5, 132.2, 130.7, 128.9, 128.8, 128.56, 128.5, 128.4, 127.9, 127.7, 127.5, 122.5, 122.1, 121.3, 118.4, 114.9, 77.2, 76.7, 76.6, 31.1, 31.0, 30.7, 25.6, 23.4, 23.3, 23.2, 23.1, 10.5, 10.3, 10.2, 10.1. **HRMS** (ESI): m/z $[M+H]^+$calcd for $[C_{53}H_{56}ClN_2O_8]^+$ requires 883.3725, found 883.3734. $[\alpha]_D^{25}$ = +22 (c = 0.1, CH_2Cl_2). The product was analyzed by HPLC to determine the enantiomeric excess: 98% ee (CHIRALPAK IB-H, hexane/*i*-PrOH = 90/10, detector: 254 nm, T = 25 °C, flow rate: 1 mL/min), t_1 (major) = 15.915 min, t_2 (minor) = 14.198 min.

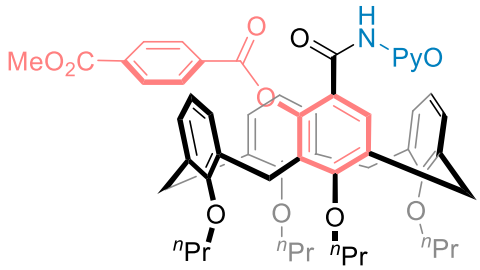




Peak	RetTime	Area	Height	Area
1	14.334	16424.6	455.62845	50.6299
2	16.450	16016.0	334.29611	49.3701

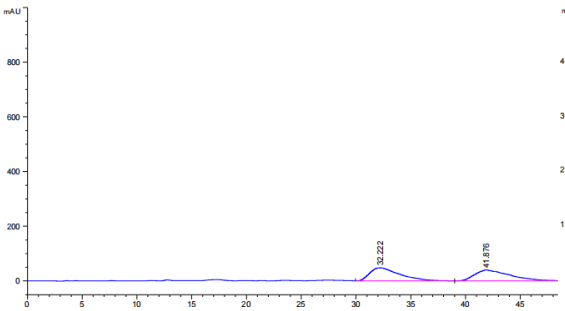
Peak	RetTime	Area	Height	Area
1	14.198	922.79138	27.01377	0.9853
2	15.915	92730.6	1897.02051	99.0147

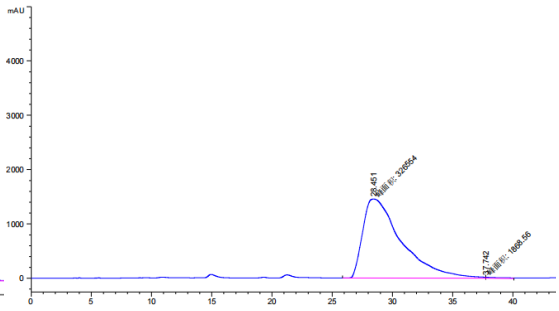
2-(1^4-((4-(methoxycarbonyl)benzoyl)oxy)-1^2,3^2,5^2,7^2-tetrapropoxy-1,3,5,7(1,3)-tetrabenzenacyclooctaphane-1^5-carboxamido)pyridine 1-oxide (3av)



Yield: 77.4 mg (61%). White solid, mp: 128 °C.

^{1}H NMR (600 MHz, $CDCl_3$) δ 10.49 (s, 1H), 8.39 (dd, *J* = 8.5, 1.3 Hz, 1H), 8.25 (d, *J* = 8.4 Hz, 2H), 8.22-8.17 (m, 1H), 8.15 (d, *J* = 8.5 Hz, 2H), 7.34 (s, 1H), 7.22 (dd, *J* = 12.3, 4.7 Hz, 1H), 6.95-6.87 (m, 1H), 6.79 (dd, *J* = 13.0, 7.2 Hz, 2H), 6.68 (t, *J* = 7.4 Hz, 1H), 6.65-6.44 (m, 4H), 6.44-6.30 (m, 2H), 4.56-4.41 (m, 3H), 4.28 (d, *J* = 14.0 Hz, 1H), 4.03 (dtd, *J* = 18.2, 9.9, 8.2 Hz, 2H), 3.96 (s, 3H), 3.92-3.84 (m, 3H), 3.83-3.72 (m, 3H), 3.35 (d, *J* = 14.0 Hz, 1H), 3.27 (d, *J* = 13.6 Hz, 1H), 3.18 (t, *J* = 13.5 Hz, 2H), 2.00-1.81 (m, 8H), 1.60-0.93 (m, 12H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 166.3, 164.2, 161.7, 156.9, 156.2, 156.1, 146.2, 144.5, 136.9, 135.7, 135.5, 135.0, 134.8, 134.4, 134.3, 133.2, 132.9, 132.4, 130.7, 129.6, 128.8, 128.6, 128.5, 128.48, 128.41, 127.9, 127.7, 122.4, 122.1, 121.2, 118.4, 114.8, 76.6, 52.5, 31.1, 31.0, 30.6, 23.4, 23.3, 23.2, 23.1, 10.5, 10.3, 10.2, 10.1. **HRMS** (ESI): m/z $[M+H]^+$calcd for $[C_{55}H_{59}ClN_2O_{10}]^+$ requires 907.4169, found 907.4171. $[\alpha]_D^{25}$ = +34 (c = 0.1, CH_2Cl_2). The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (CHIRALPAK AD-H, hexane/*i*-PrOH = 95/5, detector: 254 nm, T = 25 °C, flow rate: 1 mL/min), t_1 (major) = 28.451 min, t_2 (minor) = 37.742 min.

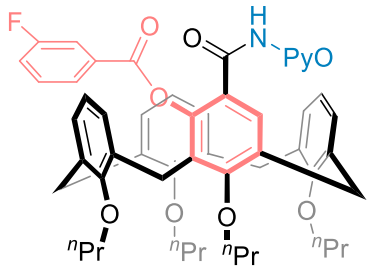




Peak	RetTime	Area	Height	Area
1	32.222	8986.64648	47.40490	50.2234
2	41.876	8906.68164	39.96460	49.7766

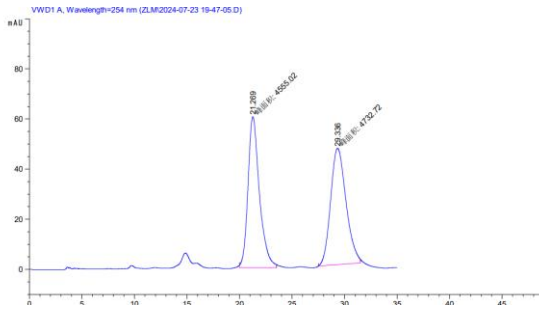
Peak	RetTime	Area	Height	Area
1	28.451	326554	1455.48901	99.4311
2	37.742	1868.55835	20.38922	0.5689

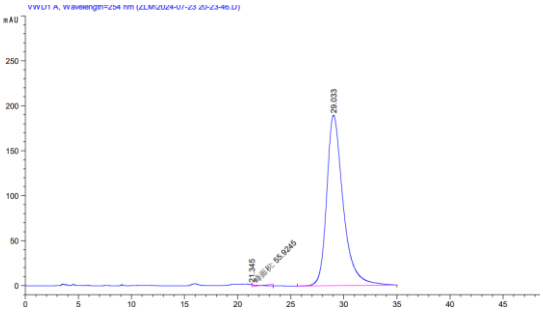
2-(1^4-((3-fluorobenzoyl)oxy)-1^2,3^2,5^2,7^2-tetrapropoxy-1,3,5,7(1,3)-tetrabenzenacyclooctaphane-1^5-carboxamido)pyridine 1-oxide (3aw)



Yield: 69.1 mg (57%). White solid, mp: 122 °C.

^{1}H NMR (600 MHz, $CDCl_3$) δ 10.49 (s, 1H), 8.41 (dd, *J* = 8.5, 1.4 Hz, 1H), 8.24-8.19 (m, 1H), 8.00 (d, *J* = 7.8 Hz, 1H), 7.86 (d, *J* = 9.0 Hz, 1H), 7.47 (td, *J* = 8.0, 5.5 Hz, 1H), 7.34 (s, 1 H), 7.33-7.28 (m, 1H), 7.24 (dd, *J* = 12.4, 4.9 Hz, 1H), 6.97-6.89 (m, 1H), 6.81 (dd, *J* = 13.1, 7.4 Hz, 2H), 6.69 (t, *J* = 7.4 Hz, 1H), 6.52 (dd, *J* = 32.1, 6.9 Hz, 4H), 6.45-6.35 (m, 2H), 4.56-4.41 (m, 3H), 4.27 (d, *J* = 14.0 Hz, 1H), 4.04 (dtd, *J* = 18.4, 10.1, 8.1 Hz, 2H), 3.88 (dt, *J* = 17.0, 7.4 Hz, 3H), 3.82 -3.71 (m, 3H), 3.35 (d, *J* = 14.0 Hz, 1H), 3.27 (d, *J* = 13.6 Hz, 1H), 3.18 (t, *J* = 13.0 Hz, 2H), 2.00-1.89 (m, 6H), 1.87-1.82 (m, 2 H), 1.08-1.01 (m, 3 H), 1.00-0.92 (m, 9H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 164.2, 163.9, 163.3, 161.7, 157.0, 156.1, 156.0, 146.2, 144.5, 136.9, 135.8, 135.6, 134.9, 134.8, 134.4, 133.1, 132.4, 131.2, 130.7, 130.10, 130.05, 128.7, 128.5, 128.5, 128.4, 127.9, 127.6, 126.4, 122.4, 122.1, 121.2, 120.7, 120.6, 118.4, 117.6, 117.5, 114.8, 76.7, 76.6, 31.1, 31.0, 30.7, 23.4, 23.3, 23.2, 23.1 10.5, 10.3, 10.2, 10.1. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -112.1. **HRMS** (ESI): m/z $[M+H]^+$calcd for $[C_{53}H_{56}FN_2O_8]^+$ requires 867.4020, found 867.4032. $[\alpha]_D^{25}$ = +21 (c = 0.1, CH_2Cl_2). The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (CHIRALPAK IC-H, hexane/*i*-PrOH = 90/10, detector: 254 nm, T = 25 °C, flow rate: 1 mL/min), t_1 (major) = 29.033 min, t_2 (minor) = 21.345 min.

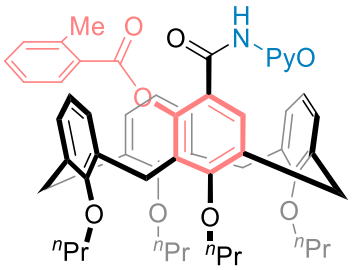




Peak	RetTime	Area	Height	Area
1	21.269	4555.01611	60.24934	49.0742
2	29.336	4726.88574	46.57448	50.9258

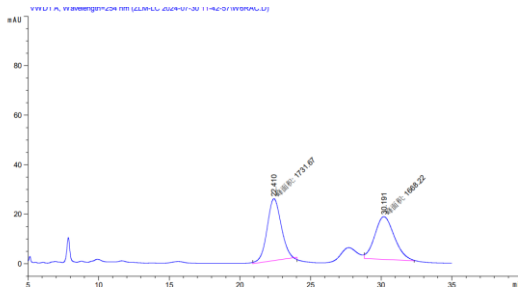
Peak	RetTime	Area	Height	Area
1	21.345	55.92454	2.03804	0.2739
2	29.033	20365.1	189.51302	99.7261

2-(1^4-((2-methylbenzoyl)oxy)-1^2,3^2,5^2,7^2-tetrapropoxy-1,3,5,7(1,3)-tetrabenzenacyclooctaphane-1^5-carboxamido)pyridine 1-oxide (3ax)

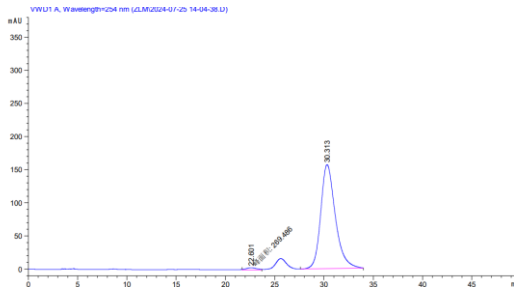


Yield: 82.1 mg (68%). White solid, mp: 122 °C.

^{1}H NMR (600 MHz, $CDCl_3$) δ 10.55 (s, 1H), 8.46 (dd, *J* = 8.5, 1.5 Hz, 1H), 8.25 (d, *J* = 7.7 Hz, 1H), 8.21 (d, *J* = 6.5 Hz, 1H), 7.50-7.41 (m, 2H), 7.31 (t, *J* = 7.6 Hz, 1H), 7.28-7.24 (m, 2H), 6.97-6.86 (m, 3H), 6.78 (t, *J* = 7.4 Hz, 1H), 6.50-6.44 (m, 1H), 6.41 (d, *J* = 7.3 Hz, 2H), 6.35 (dt, *J* = 20.3, 7.2 Hz, 3H), 4.55-4.40 (m, 3H), 4.23 (d, *J* = 13.9 Hz, 1H), 4.08 (ddd, *J* = 15.6, 10.4, 2.5 Hz, 2H), 3.98-3.90 (m, 2H), 3.82-3.68 (m, 4H), 3.44 (d, *J* = 14.0 Hz, 1H), 3.27 (d, *J* = 13.6 Hz, 1H), 3.17 (dd, *J* = 16.0, 13.7 Hz, 2H), 2.62 (s, 3H), 2.15-1.66 (m, 8H), 1.06 (t, *J* = 7.4 Hz, 3H), 1.02-0.91 (m, 9H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 165.3, 164.5, 162.0, 157.3, 155.7, 146.6, 144.6, 141.7, 136.9, 136.2, 136.1, 134.4, 134.2, 132.8, 132.7, 132.3, 131.9, 131.7, 128.64, 128.61, 128.59, 128.3, 128.2, 128.1, 127.8, 127.7, 125.9, 122.4, 122.3, 122.0, 121.5, 118.3, 114.8, 77.1, 76.7, 76.6, 31.02, 30.97, 30.7, 23.4, 23.2, 23.1, 22.0, 10.6, 10.5, 10.1, 10.0. **HRMS** (ESI): m/z $[M+H]^+$calcd for $[C_{54}H_{59}N_2O_8]^+$ requires 863.4271, found 863.4278 $[\alpha]_D^{25}$ = +22 (c = 0.1, CH_2Cl_2). The product was analyzed by HPLC to determine the enantiomeric excess: 96% ee (CHIRALPAK IC-H, hexane/*i*-PrOH = 90/10, detector: 254 nm, T = 25 °C, flow rate: 1 mL/min), t_1 (major) = 30.313 min, t_2 (minor) = 22.601 min.

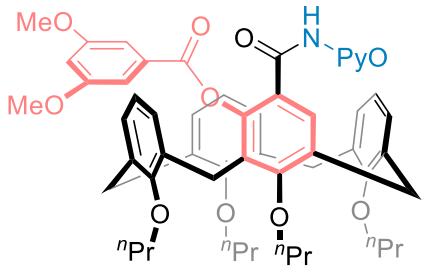


Peak	RetTime	Area	Height	Area
1	22.410	1731.66602	25.00605	50.9331
2	30.191	1668.21912	17.30567	49.0669



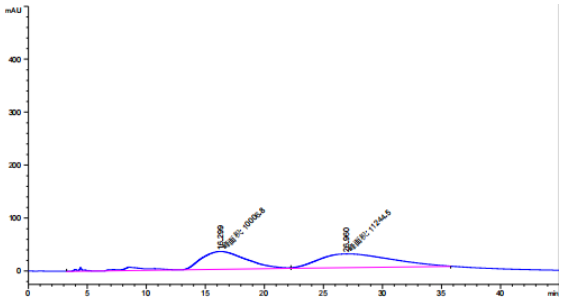
Peak	RetTime	Area	Height	Area
1	22.601	269.48618	3.41499	1.6424
2	30.313	16138.4	156.92009	98.3576

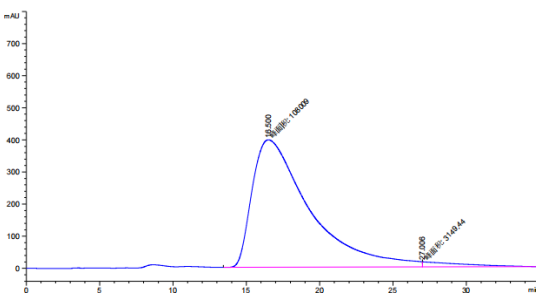
2-(1^4-((3,5-dimethoxybenzoyl)oxy)-1^2,3^2,5^2,7^2-tetrapropoxy-1,3,5,7(1,3)-tetrabenzenacyclooctaphane-1^5-carboxamido)pyridine 1-oxide (3ay)



Yield: 76.3 mg (60%). White solid, mp: 129 °C.

^{1}H NMR (600 MHz, $CDCl_3$) δ 10.52 (s, 1H), 8.43 (dd, *J* = 8.5, 1.5 Hz, 1H), 8.23-8.17 (m, 1H), 7.36 (t, *J* = 7.3 Hz, 3H), 7.26-7.22 (m, 1H), 6.96-6.89 (m, 1H), 6.83 (d, *J* = 7.3 Hz, 2H), 6.77-6.67 (m, 2H), 6.57-6.49 (m, 3H), 6.48-6.39 (m, 3H), 4.55-4.41 (m, 3H), 4.26 (d, *J* = 14.0 Hz, 1H), 4.05 (ddd, *J* = 18.5, 10.5, 5.1 Hz, 2H), 3.94-3.89 (m, 2H), 3.86 (s, 6H), 3.83-3.70 (m, 4H), 3.36 (d, *J* = 14.0 Hz, 1H), 3.27 (d, *J* = 13.6 Hz, 1H), 3.23-3.13 (m, 2H), 2.03-1.79 (m, 8H), 1.05 (t, *J* = 7.4 Hz, 3H), 1.01-0.93 (m, 9H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 164.8, 164.3, 161.7, 160.6, 157.0, 156.0, 155.9, 146.3, 144.5, 136.9, 135.9, 135.6, 134.7, 134.5, 134.3, 133.1, 132.4, 130.9, 130.7, 128.8, 128.5, 128.39, 128.35, 128.3, 127.9, 127.6, 122.4, 122.2, 122.1, 121.5, 118.3, 114.9, 108.0, 107.0, 77.1, 76.6, 76.6, 55.6, 31.1, 31.0, 30.7, 23.4, 23.2, 23.19, 23.11, 10.5, 10.4, 10.2, 10.1. **HRMS** (ESI): m/z $[M+H]^+$calcd for $[C_{55}H_{61}N_2O_{10}]^+$ requires 909.4326, found 909.4330. $[\alpha]_D^{25}$ = +34 (c = 0.1, CH_2Cl_2). The product was analyzed by HPLC to determine the enantiomeric excess: 94% ee (CHIRALPAK AS-H, hexane/*i*-PrOH = 95/5, detector: 254 nm, T = 25 °C, flow rate: 1 mL/min), t_1 (major) = 16.500 min, t_2 (minor) = 27.006 min.

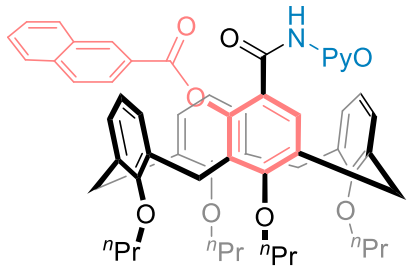




Peak	RetTime	Area	Height	Area
1	16.299	10006.8	33.81359	47.0880
2	26.960	11244.5	26.27214	52.9120

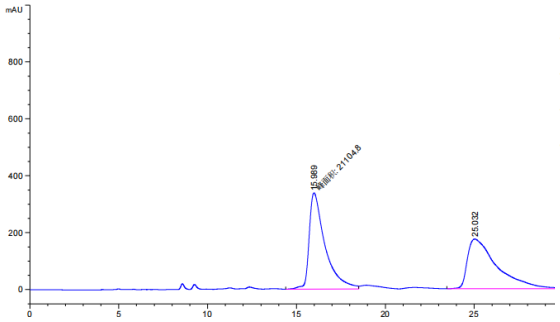
Peak	RetTime	Area	Height	Area
1	16.500	108009	397.15347	97.1667
2	27.006	3149.44019	16.17056	2.8333

2-(1^4-((2-naphthoyl)oxy)-1^2,3^2,5^2,7^2-tetrapropoxy-1,3,5,7(1,3)-tetrabenzenacyclooctaphane-1^5-carboxamido)pyridine 1-oxide (3az)

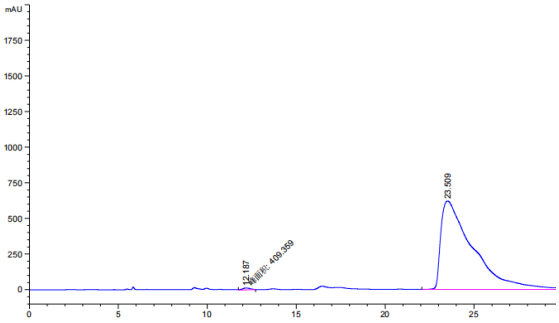


Yield: 78.0 mg (62%). White solid, mp: 167 °C.

^{1}H NMR (600 MHz, $CDCl_3$) δ 10.59 (s, 1H), 8.84 (s, 1H), 8.40 (d, *J* = 8.3 Hz, 1H), 8.19 (d, *J* = 8.6 Hz, 1H), 8.15 (d, *J* = 6.3 Hz, 1H), 8.05 (d, *J* = 8.1 Hz, 1H), 7.91 (dd, *J* = 16.3, 8.4 Hz, 2H), 7.62 (t, *J* = 7.4 Hz, 1H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.39 (s, 1H), 7.18 (t, *J* = 8.0 Hz, 1H), 6.92-6.76 (m, 3H), 6.71 (t, *J* = 7.4 Hz, 1H), 6.63-6.47 (m, 5H), 6.42 (t, *J* = 7.3 Hz, 1H), 4.59-4.42 (m, 3H), 4.30 (d, *J* = 14.0 Hz, 1H), 4.07 (dtd, *J* = 18.0, 10.0, 8.0 Hz, 2H), 3.90 (dt, *J* = 16.8, 7.4 Hz, 3H), 3.85-3.70 (m, 3H), 3.43 (d, *J* = 14.0 Hz, 1H), 3.29 (d, *J* = 13.5 Hz, 1H), 3.20 (dd, *J* = 13.5, 9.2 Hz, 2H), 2.05-1.81 (m, 8H), 1.05 (t, *J* = 7.4 Hz, 3H), 1.03-0.92 (m, 9H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 165.1, 164.4, 161.7, 157.0, 156.1 156.07, 146.4, 144.5, 136.9, 135.9, 135.87, 135.6, 134.9, 134.7, 134.3, 133.3, 132.9, 132.6, 132.5, 130.9, 129.8, 129.0, 128.6, 128.5, 128.48, 128.43, 128.2, 128.0, 127.7, 127.6, 126.6, 126.1, 125.9, 122.5, 122.2, 122.1, 121.6, 118.3, 114.9, 77.2, 76.7, 76.6, 31.1, 31.0, 30.7, 23.4, 23.3, 23.2, 23.1, 10.5, 10.3, 10.2, 10.1. **HRMS** (ESI): m/z $[M+H]^+$calcd for $[C_{57}H_{59}N_2O_8]^+$ requires 899.4271, found 899.4271. $[\alpha]_D^{25}$ = 113 (c = 0.1, CH_2Cl_2). The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (CHIRALPAK IB-H, hexane/*i*-PrOH = 90/10, detector: 254 nm, T = 25 °C, flow rate: 1 mL/min), t_1 (major) = 23.509 min, t_2 (minor) = 12.187 min.

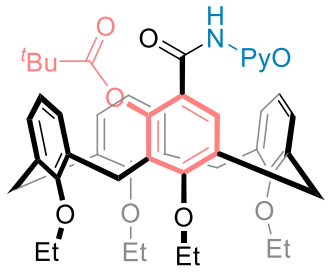


Peak	RetTime	Area	Height	Area
1	15.989	21104.8	338.73370	50.6295
2	25.032	20580.0	174.72723	49.3705



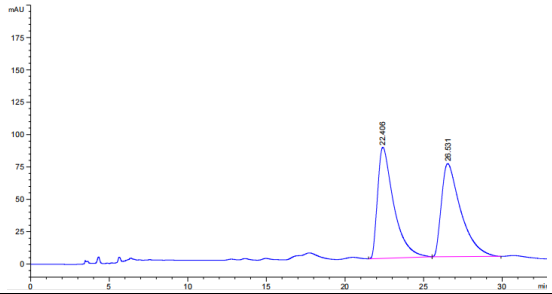
Peak	RetTime	Area	Height	Area
1	12.187	409.35950	13.44584	0.5246
2	23.509	77628.4	620.25781	99.4754

2-(1^2,3^2,5^2,7^2-tetraethoxy-1^4-(pivaloyloxy)-1,3,5,7(1,3)-tetrabenzenacyclooctaphane-1^5-carboxamido)pyridine 1-oxide (3ba)

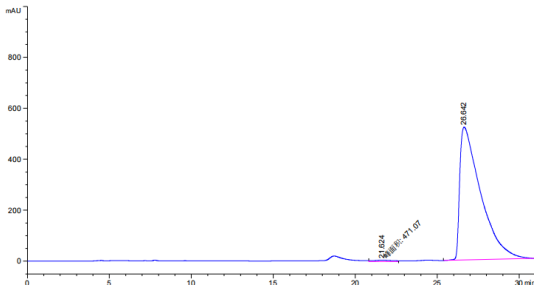


Yield: 60.2 mg (78%). White solid, mp: 112 °C.

^{1}H NMR (600 MHz, $CDCl_3$) δ 10.54 (s, 1H), 8.58 (dd, *J* = 8.4, 1.3 Hz, 1H), 8.29 (d, *J* = 6.3 Hz, 1H), 7.58 (s, 1H), 7.38 (t, *J* = 7.8 Hz, 1H), 7.17-7.09 (m, 2H), 7.06-6.98 (m, 1H), 6.93 (t, *J* = 7.4 Hz, 1H), 6.30 (dt, *J* = 22.0, 7.5 Hz, 3H), 6.26-6.15 (m, 3H), 4.51-4.39 (m, 3H), 4.31 (tdd, *J* = 11.0, 7.1, 3.9 Hz, 2H), 4.17 (dt, *J* = 20.4, 10.3 Hz, 3H), 3.85-3.74 (m, 4H), 3.39 (d, *J* = 13.7 Hz, 1H), 3.24 (d, *J* = 13.5 Hz, 1H), 3.15 (dd, *J* = 13.2, 3.9 Hz, 2H), 1.51-1.43 (m, 12H), 1.37 (s, 9H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 176.9, 164.8, 161.9, 157.4, 154.9, 154.8, 146.4, 144.7, 137.2, 137.19, 137.12, 135.3, 133.5, 133.47, 132.1, 132.06, 131.9, 128.9, 128.8, 128.2, 128.1, 127.9, 127.8, 127.5, 127.3, 122.7, 122.4, 122.1, 121.9, 118.5, 114.9, 70.6, 70.57, 70.53, 69.8, 39.3, 31.0, 30.8, 27.2, 23.6, 15.72, 15.70 15.65. **HRMS** (ESI): m/z $[M+H]^+$calcd for $[C_{47}H_{53}N_2O_8]^+$ requires 773.3802 found 773.3807. $[\alpha]_D^{25}$ = +40 (c = 0.1, CH_2Cl_2). The product was analyzed by HPLC to determine the enantiomeric excess: 98% ee (CHIRALPAK IB-H, hexane/*i*-PrOH = 95/5, detector: 254 nm, T = 25 °C, flow rate: 1 mL/min), t_1 (major) = 26.642 min, t_2 (minor) = 21.624 min.

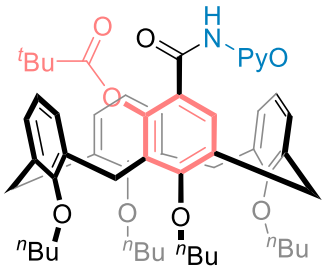


Peak	RetTime	Area	Height	Area
1	22.406	6136.39697	85.81712	49.9065
2	26.531	6159.38037	71.86004	50.0935



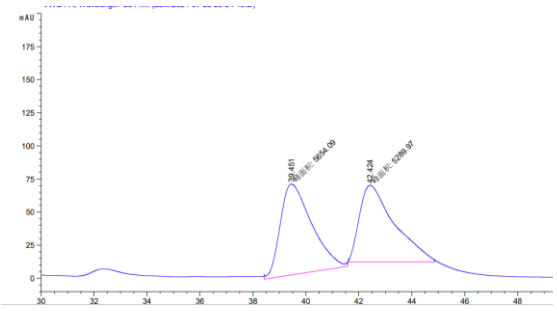
Peak	RetTime	Area	Height	Area
1	21.624	471.07001	5.58379	1.0060
2	26.642	46355.5	521.71954	98.9940

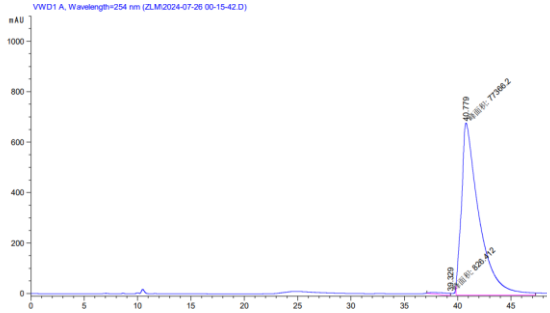
2-(1^2,3^2,5^2,7^2-tetrabutoxy-1^4-(pivaloyloxy)-1,3,5,7(1,3)-tetrabenzenacyclooctaphane-1^5-carboxamido)pyridine 1-oxide (3ca)



Yield: 83.1 mg (94%). White solid, mp: 122 °C.

^{1}H NMR (600 MHz, $CDCl_3$) δ 10.53 (s, 1H), 8.61-8.55 (m, 1H), 8.29 (d, *J* = 6.4 Hz, 1H), 7.57 (s, 1H), 7.38 (t, *J* = 8.0 Hz, 1H), 7.12 (dd, *J* = 7.4, 2.4 Hz, 2H), 7.05-6.97 (m, 1H), 6.93 (t, *J* = 7.4 Hz, 1H), 6.27 (ddd, *J* = 29.8, 14.9, 7.4 Hz, 3H), 6.19-6.09 (m, 3H), 4.51-4.39 (m, 3H), 4.26-4.11 (m, 3H), 4.06 (dd, *J* = 10.2, 6.3 Hz, 2H), 3.71 (dt, *J* = 9.6, 6.5 Hz, 4H), 3.40 (d, *J* = 13.9 Hz, 1H), 3.25 (d, *J* = 13.7 Hz, 1H), 3.16 (dd, *J* = 13.4, 5.0 Hz, 2H), 1.95 (dd, *J* = 16.1, 7.9 Hz, 4H), 1.85 (dd, *J* = 14.6, 6.7 Hz, 4H), 1.60 (ddd, *J* = 15.2, 11.0, 7.6 Hz, 4H), 1.35 (d, *J* = 19.0 Hz, 9H), 1.31 (ddd, *J* = 15.5, 10.1, 7.7 Hz, 4H), 1.05-0.94 (m, 12H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 176.9, 164.8, 162.3, 157.8, 155.2, 155.1, 146.5, 144.7, 137.1, 137.0, 136.9, 134.9, 133.4, 133.3, 131.9, 131.8, 129.0, 128.9, 128.3, 128.0, 127.9, 127.8, 127.4, 127.2, 122.7, 122.4, 121.9, 121.7, 118.5, 114.9, 75.3, 75.1, 75.0, 74.8, 39.3, 32.6, 32.5, 32.1, 32.0, 30.9, 30.7, 27.2, 23.4, 19.7, 19.6, 19.1, 18.9, 14.2, 14.1, 14.0. **HRMS** (ESI): m/z $[M+H]^+$calcd for $[C_{55}H_{69}N_2O_8]^+$ requires 885.5054 found 885.5064. $[\alpha]_D^{25}$ = +28 (c = 0.1, CH_2Cl_2). The product was analyzed by HPLC to determine the enantiomeric excess: 98% ee (CHIRALPAK IB-H, hexane/*i*-PrOH = 96/4, detector: 254 nm, T = 25 °C, flow rate: 0.5 mL/min), t_1 (major) = 40.779 min, t_2 (minor) = 39.329 min.

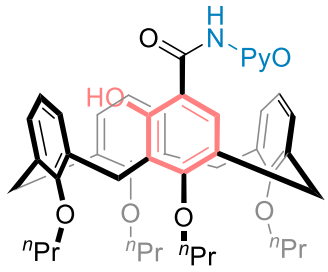




Peak	RetTime	Area	Height	Area
1	39.451	5654.09082	68.64409	51.6636
2	42.424	5289.96924	57.83927	48.3364

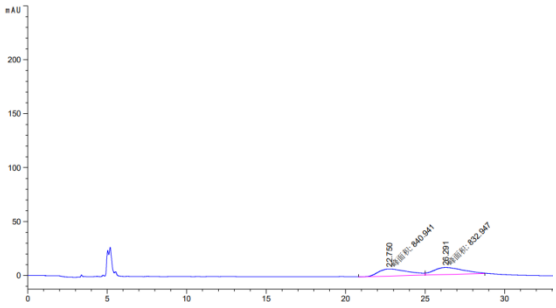
Peak	RetTime	Area	Height	Area
1	39.329	826.41180	9.16060	1.0569
2	40.779	77366.2	681.06677	98.9431

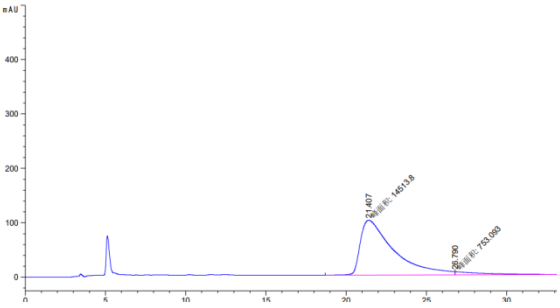
2-(1^2,3^2,5^2,7^2-tetrabutoxy-1^4-hydroxy-1,3,5,7(1,3)-tetrabenzenacyclooctaphane-1^5-carboxamido)pyridine 1-oxide (4)



Yield: 70.0 mg (94%). White solid, mp: 145 °C.

^{1}H NMR (600 MHz, $CDCl_3$) δ 11.94 (s, 1H), 10.97 (s, 1H), 8.56 (s, 1H), 8.32 (s, 1H), 7.50-7.30 (m, 2H), 7.19-6.95 (m, 3H), 6.80 (t, *J* = 7.4 Hz, 1H), 6.41 (d, *J* = 7.1 Hz, 1H), 6.33 (dt, *J* = 13.4, 6.7 Hz, 2H), 6.24 (t, *J* = 6.8 Hz, 3H), 4.50-4.38 (m, 3H), 4.21-3.87 (m, 6H), 3.87-3.60 (m, 4H), 3.18 (dd, *J* = 28.3, 13.5 Hz, 3H), 2.04-1.93 (m, 4H), 1.92-1.83 (m, 4H), 1.08 (dt, *J* = 12.3, 7.4 Hz, 6H), 0.93 (dt, *J* = 14.8, 7.5 Hz, 6H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 169.2, 164.7, 160.7, 157.6, 155.5, 155.4, 136.7, 136.5, 133.9, 133.4, 133.2, 132.7, 128.75, 128.72, 128.5, 127.9, 127.64, 127.61, 127.4, 126.2, 125.6, 122.25, 122.20, 121.8, 107.9, 76.9, 76.5, 31.0, 30.9, 30.6, 23.5, 23.4, 23.3, 23.0, 22.5, 10.73, 10.71, 10.0, 9.9. **HRMS** (ESI): m/z $[M+H]^+$calcd for $[C_{46}H_{53}N_2O_7]^+$ requires 745.3863 found 745.3863. $[\alpha]_D^{25}$ = +26 (c = 0.1, CH_2Cl_2). The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (CHIRALPAK IB-H, hexane/*i*-PrOH = 95/5, detector: 254 nm, T = 25 °C, flow rate: 1 mL/min), t_1 (major) = 21.407 min, t_2 (minor) = 26.790 min.

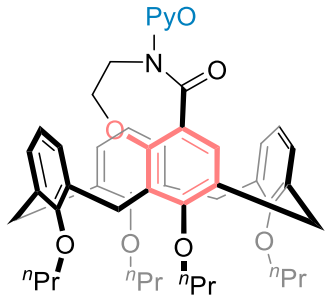




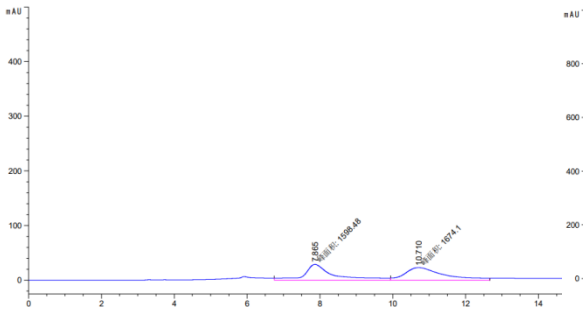
Peak	RetTime	Area	Height	Area
1	22.750	840.94098	6.48589	50.2388
2	26.291	832.94678	6.51230	49.7612

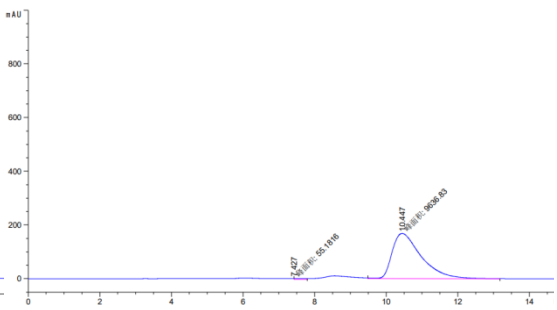
Peak	RetTime	Area	Height	Area
1	21.407	14513.8	100.68847	95.0671
2	26.790	753.09338	5.70805	4.9329

2-(1^5-oxo-1^8,3^2,5^2,7^2-tetrapropoxy-1^2,1^3,1^4,1^5-tetrahydro-1(7,9)-benzo[*f*][1,4]oxazepina-3,5,7(1,3)-tribenzenacyclooctaphane-1^4-yl)pyridine 1-oxide (6)



Yield: 65.5 mg (85%). White solid, mp: 136 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.25 (d, *J* = 6.5 Hz, 1H), 7.48 (dd, *J* = 8.1, 1.6 Hz, 1H), 7.29 (dd, *J* = 11.3, 4.4 Hz, 1H), 7.21 – 7.15 (m, 1H), 7.01 (s, 1H), 6.90 – 6.78 (m, 3H), 6.78 – 6.69 (m, 2H), 6.63 (m, 3H), 6.45 (d, *J* = 6.4 Hz, 1H), 4.49 (d, *J* = 13.3 Hz, 2H), 4.42 (d, *J* = 13.2 Hz, 1H), 4.26 (d, *J* = 13.4 Hz, 1H), 4.10 – 3.88 (m, 5H), 3.87 – 3.71 (m, 4H), 3.66 (d, *J* = 16.1 Hz, 1H), 3.54 (d, *J* = 13.4 Hz, 1H), 3.45 – 3.32 (m, 1H), 3.20 (m, 3H), 3.07 (s, 1H), 1.97 – 1.88 (m, 8H), 1.03 (m, 6H), 0.95 (m, 6H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 170.2, 161.0, 157.1, 156.7, 156.3, 150.6, 145.7, 140.4, 136.1, 135.4, 134.9, 134.8, 134.7, 134.5, 131.0, 129.9, 129.6, 128.7, 128.41, 128.35, 128.3, 127.9, 127.5, 126.9, 126.0, 123.8, 122.6, 122.4, 122.0, 121.9, 77.2, 76.9, 76.3, 71.8, 45.3, 31.4, 31.0, 30.6, 23.3, 23.3, 23.2, 22.6, 10.5, 10.4, 10.2, 10.0. **HRMS** (ESI): m/z $[M+H]^+$calcd for $[C_{48}H_{55}N_2O_7]^+$ requires 771.4004 found 771.4012. $[\alpha]_D^{25}$ = +42 (c = 0.1, CH_2Cl_2). The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (CHIRALPAK AD-H, hexane/*i*-PrOH = 90/10, detector: 254 nm, T = 25 °C, flow rate: 1 mL/min), t_1 (major) = 10.447 min, t_2 (minor) = 7.427 min.



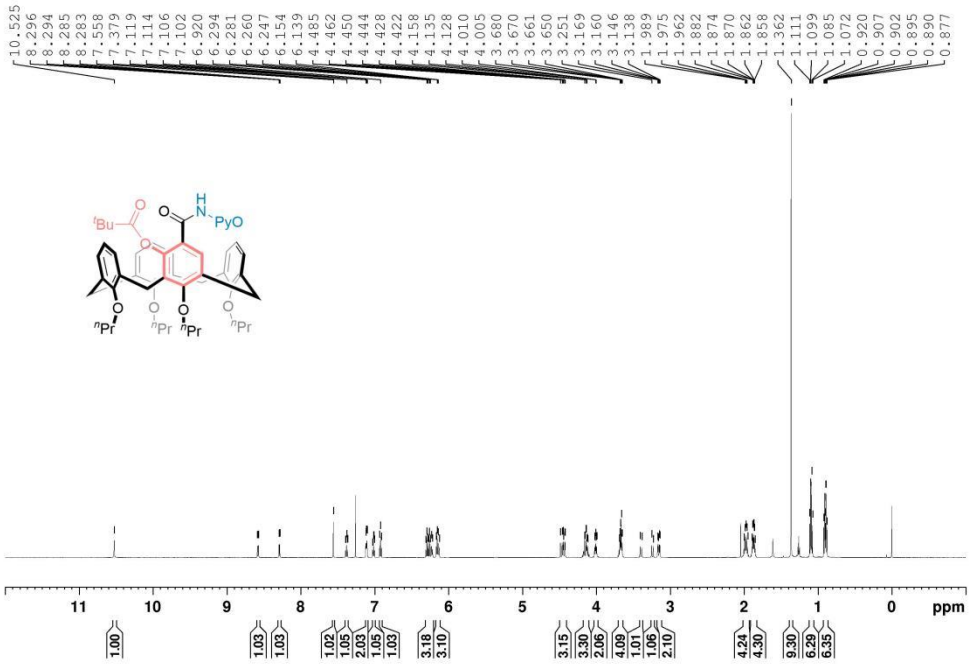


Peak	RetTime	Area	Height	Area
1	7.865	1598.48083	28.66329	48.8446
2	10.710	1674.10315	22.89644	51.1554

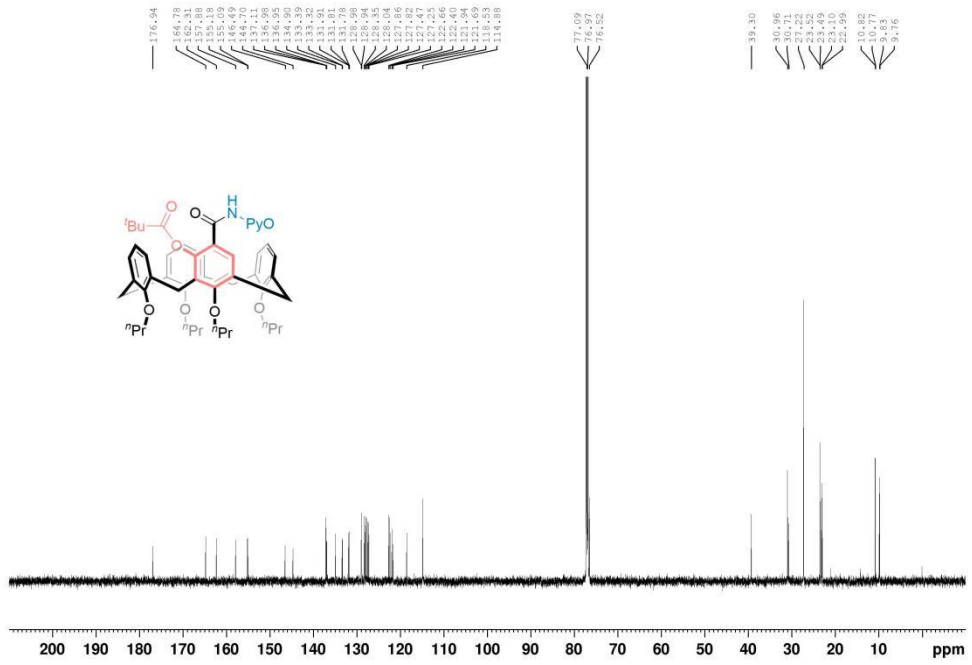
Peak	RetTime	Area	Height	Area
1	7.427	55.18159	2.72039	0.5694
2	10.447	9636.82617	168.70889	99.4306

8. Copies of NMR spectra of the acyloxylated inherently chiral calix[4]arenes

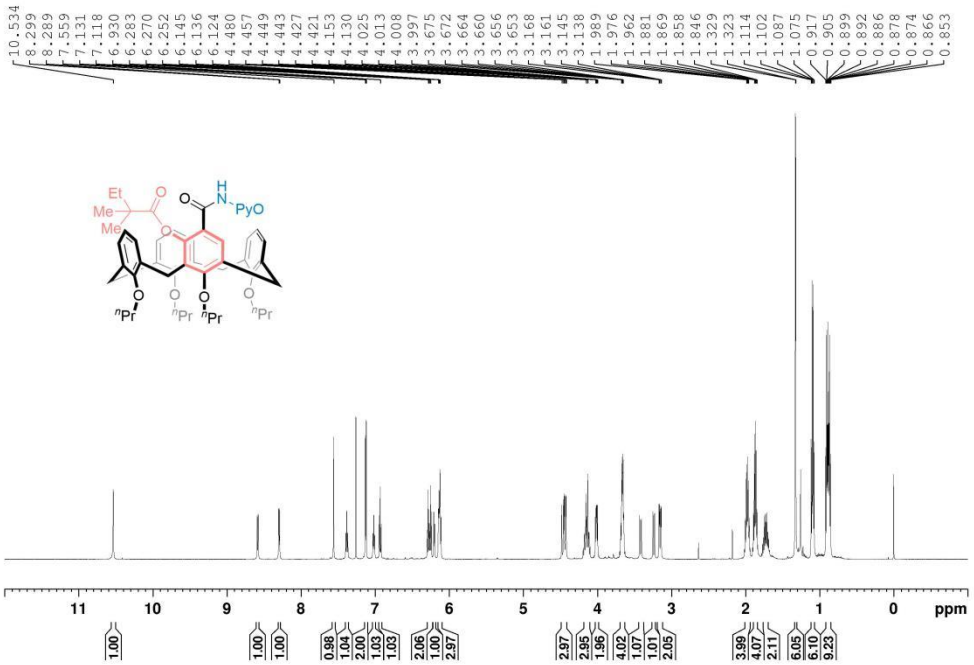
^{1}H NMR (600 MHz, $CDCl_3$), **3aa**



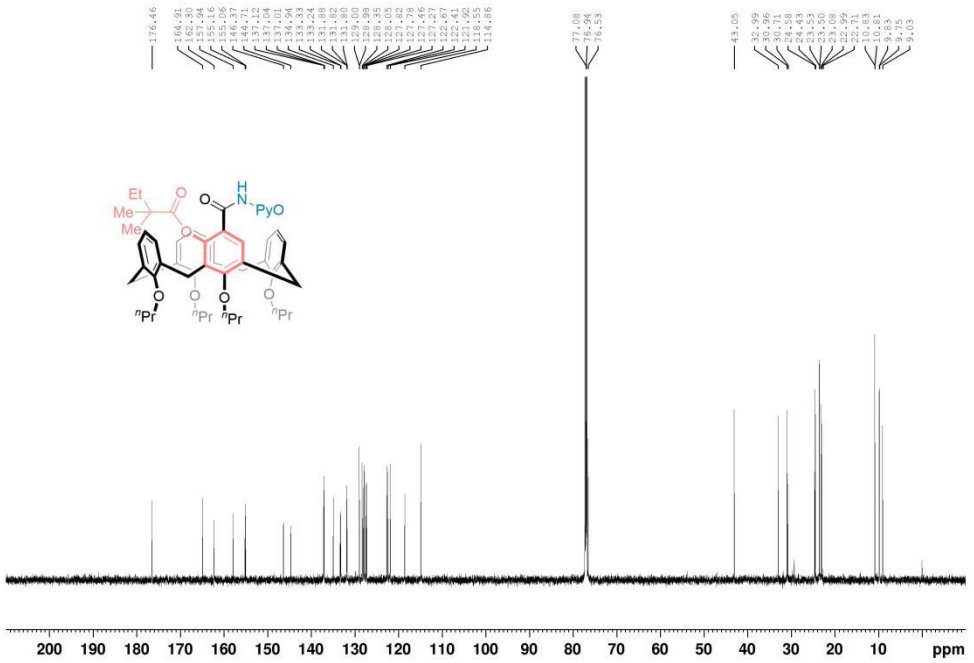
^{13}C NMR (151 MHz, $CDCl_3$), **3aa**



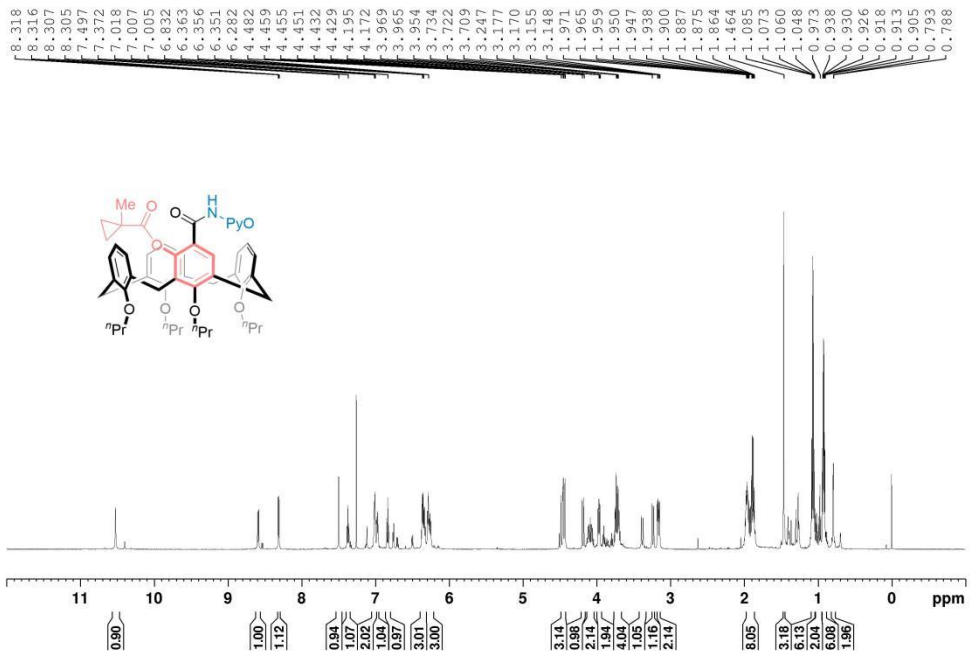
^{1}H NMR (600 MHz, $CDCl_3$), **3ab**



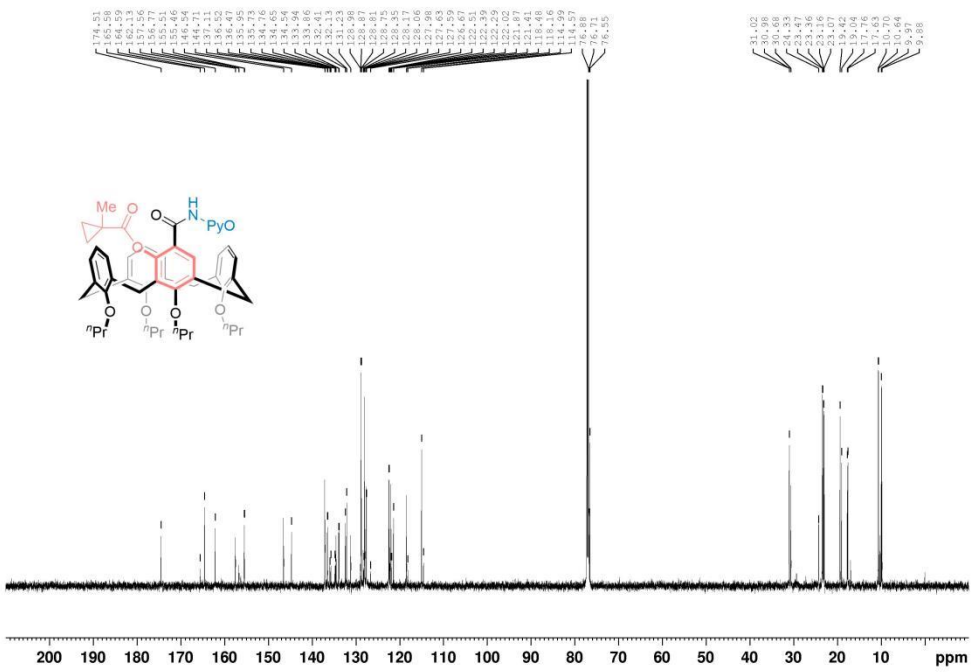
^{13}C NMR (151 MHz, $CDCl_3$), **3ab**



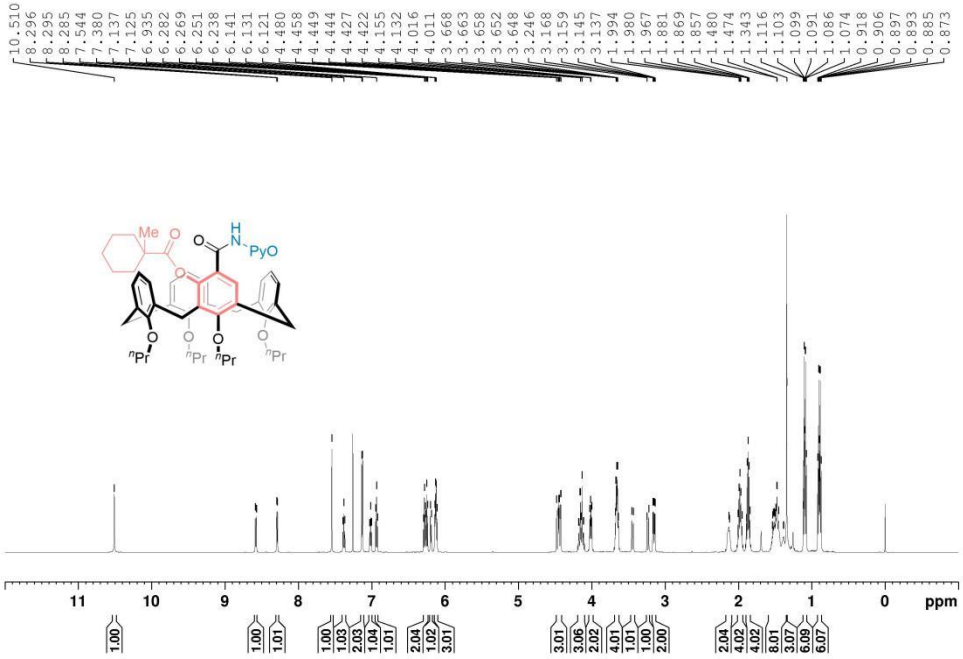
^{1}H NMR (600 MHz, $CDCl_3$), **3ac**



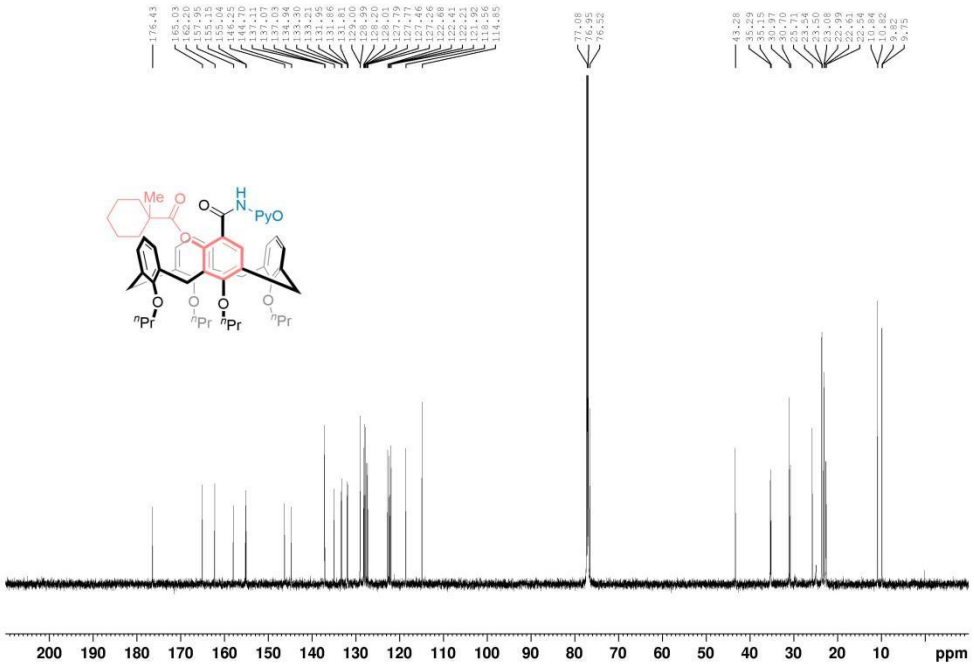
^{13}C NMR (151 MHz, $CDCl_3$), **3ac**



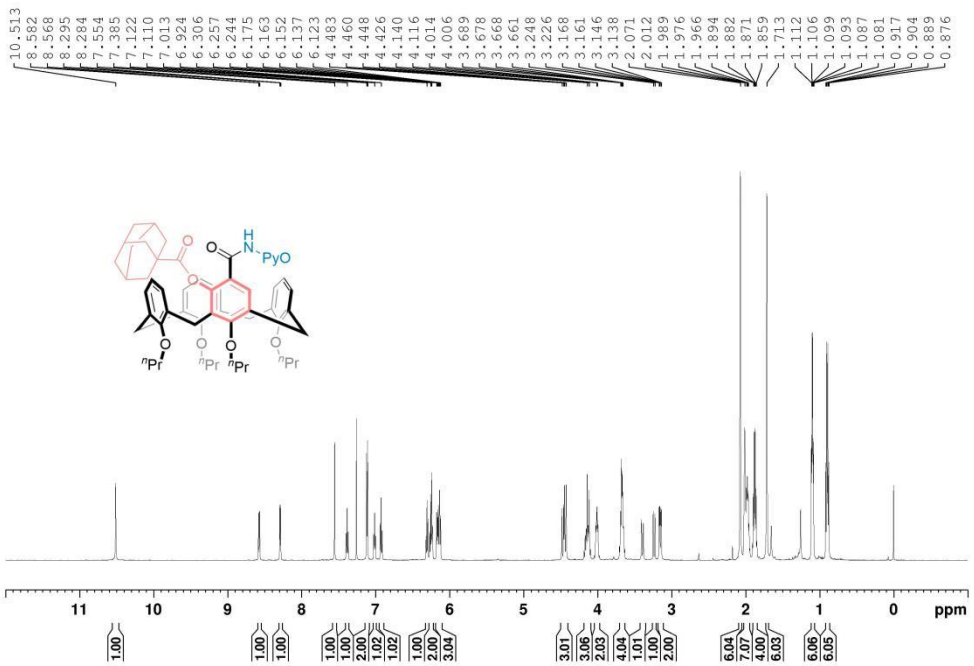
^{1}H NMR (600 MHz, $CDCl_3$), **3ad**



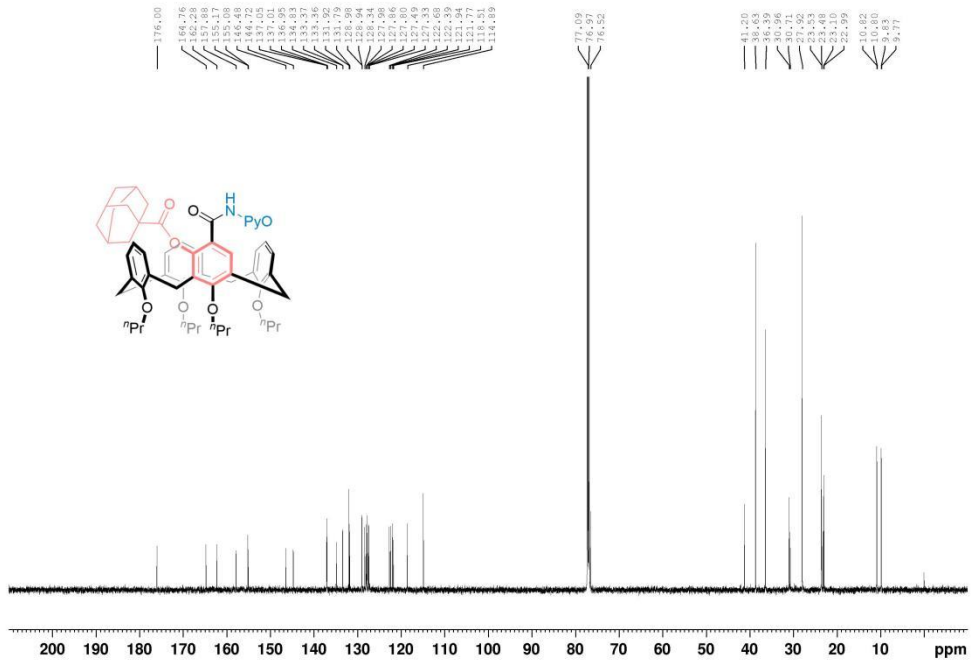
^{13}C NMR (151 MHz, $CDCl_3$), **3ad**



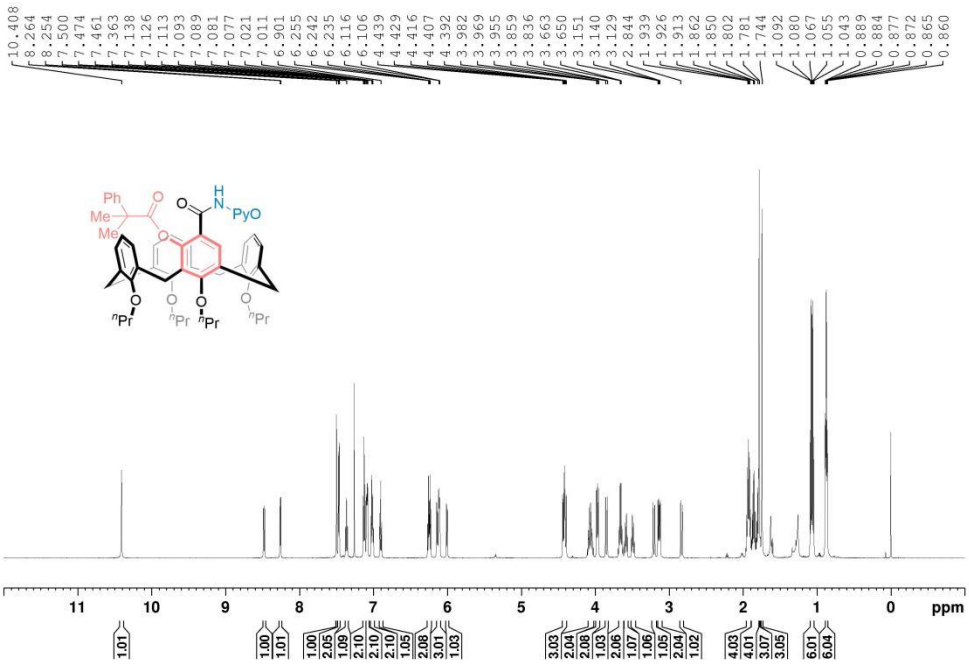
^{1}H NMR (600 MHz, $CDCl_3$), **3ae**



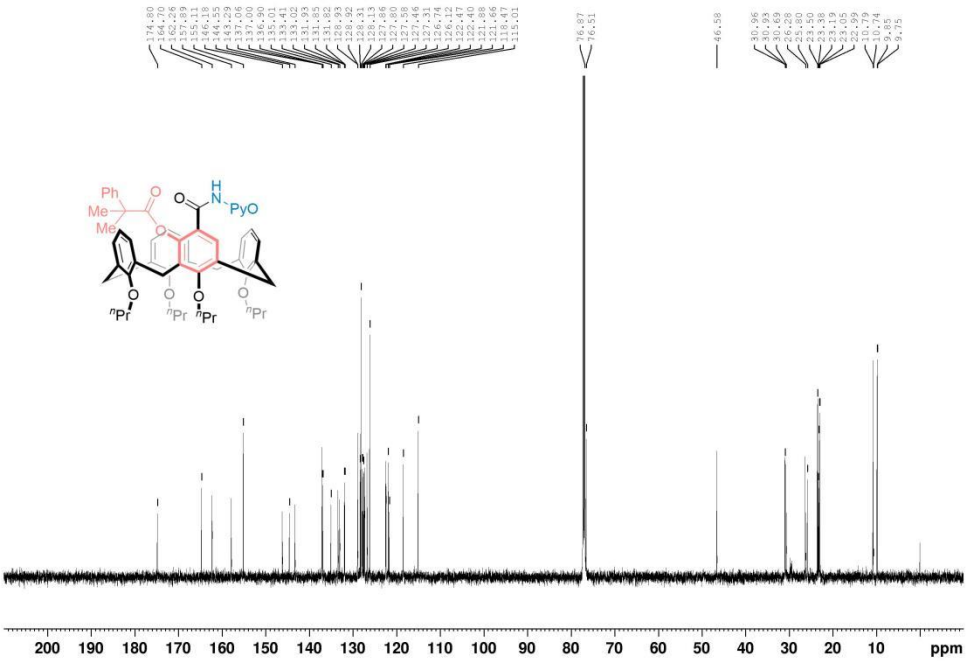
^{13}C NMR (151 MHz, $CDCl_3$), **3ae**



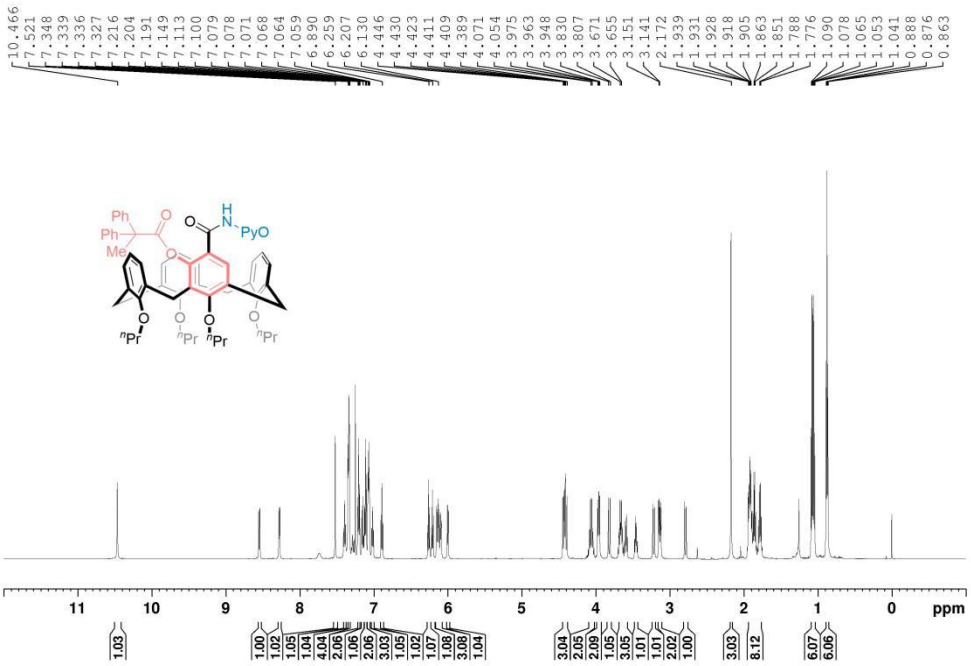
^{1}H NMR (600 MHz, $CDCl_3$), **3af**



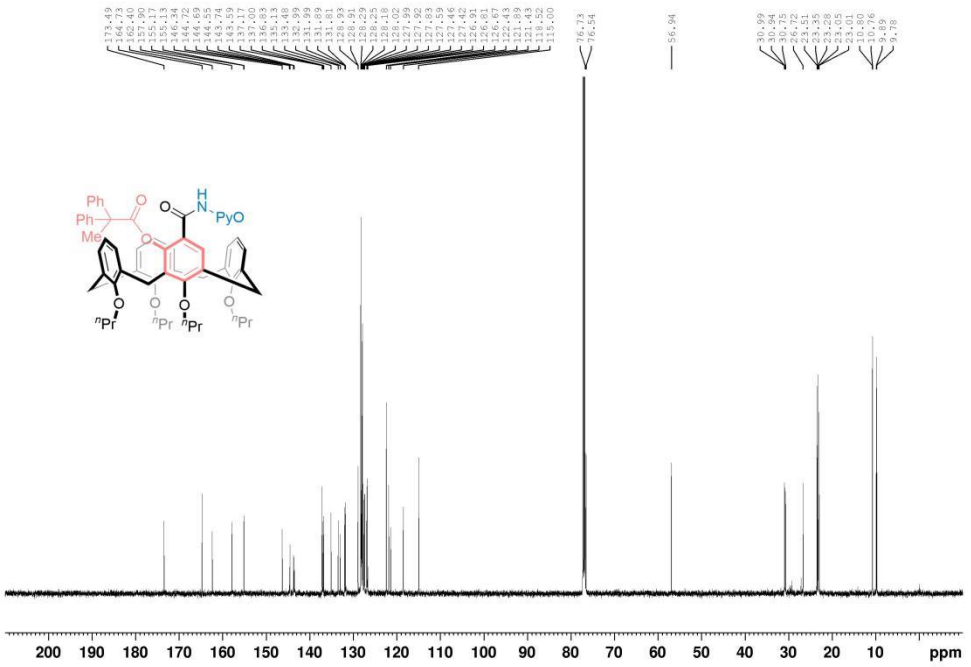
^{13}C NMR (151 MHz, $CDCl_3$), **3af**



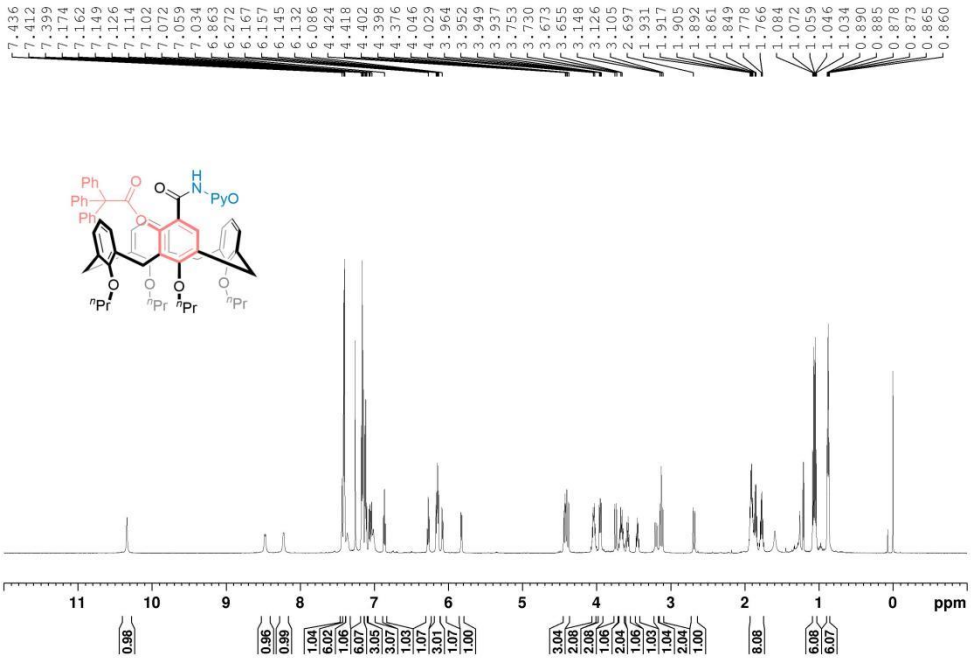
^{1}H NMR (600 MHz, $CDCl_3$), **3ag**



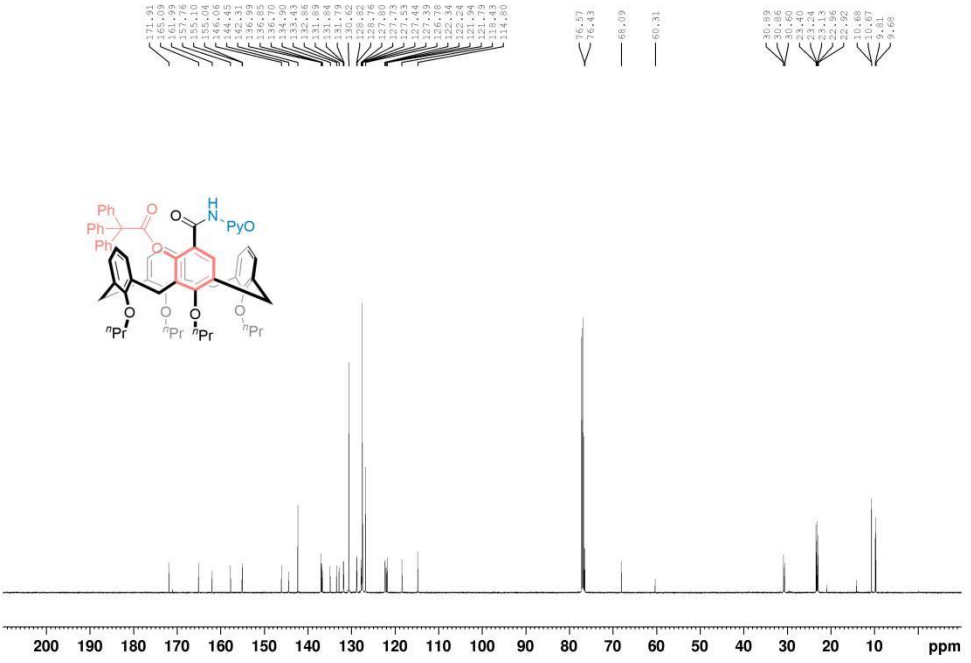
^{13}C NMR (151 MHz, $CDCl_3$), **3ag**



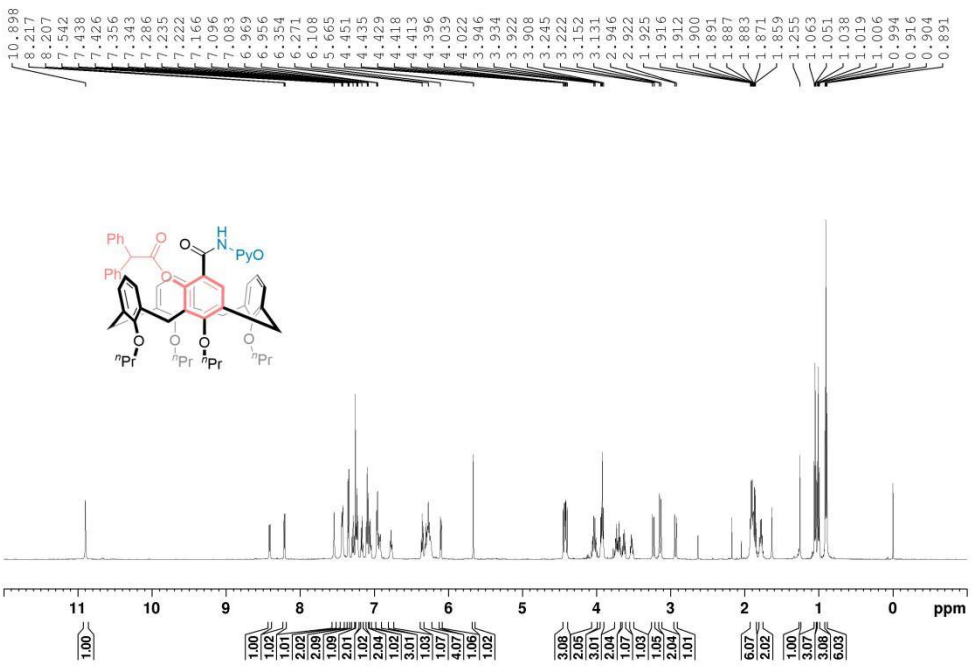
^{1}H NMR (600 MHz, $CDCl_3$), **3ah**



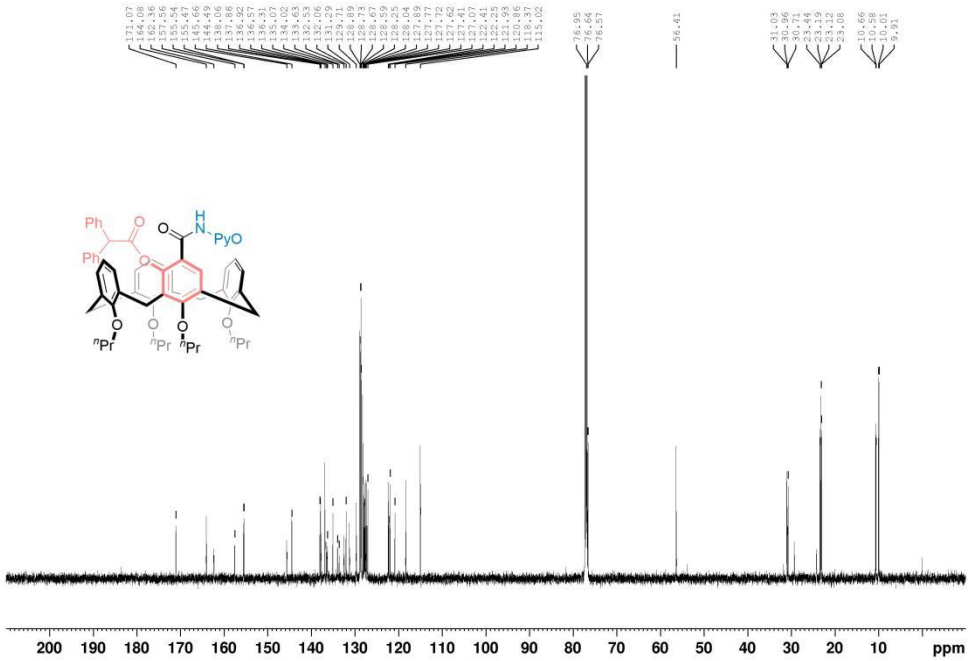
^{13}C NMR (151 MHz, $CDCl_3$), **3ah**



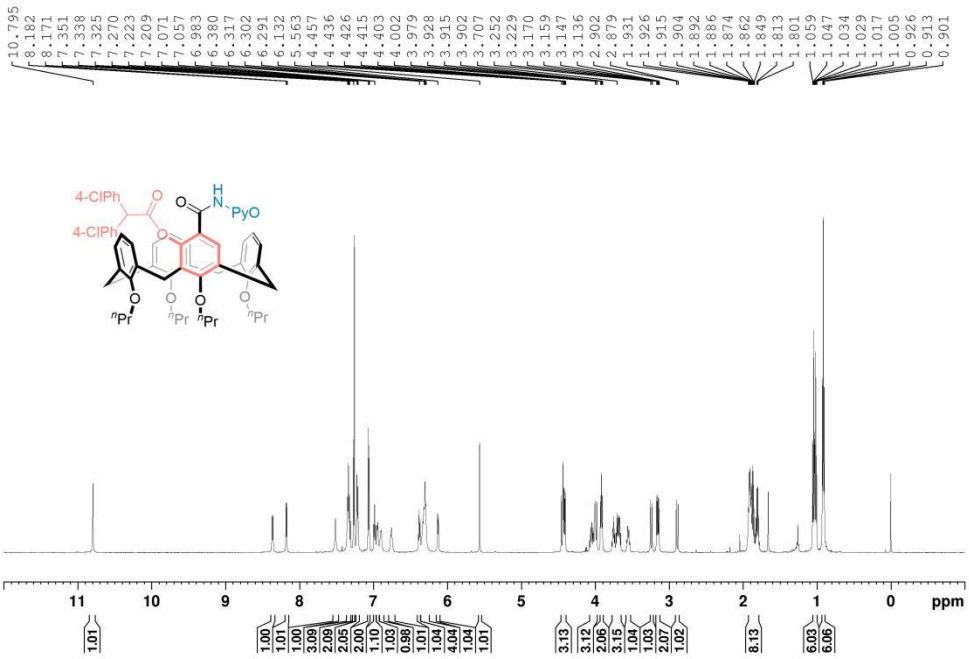
^{1}H NMR (600 MHz, $CDCl_3$), **3ai**



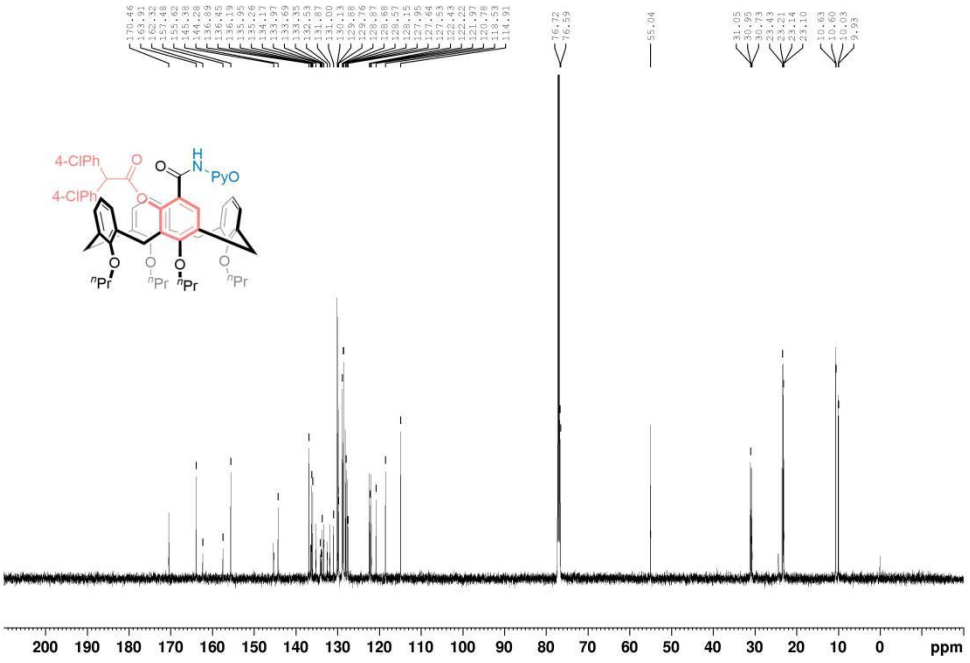
^{13}C NMR (151 MHz, $CDCl_3$), **3ai**



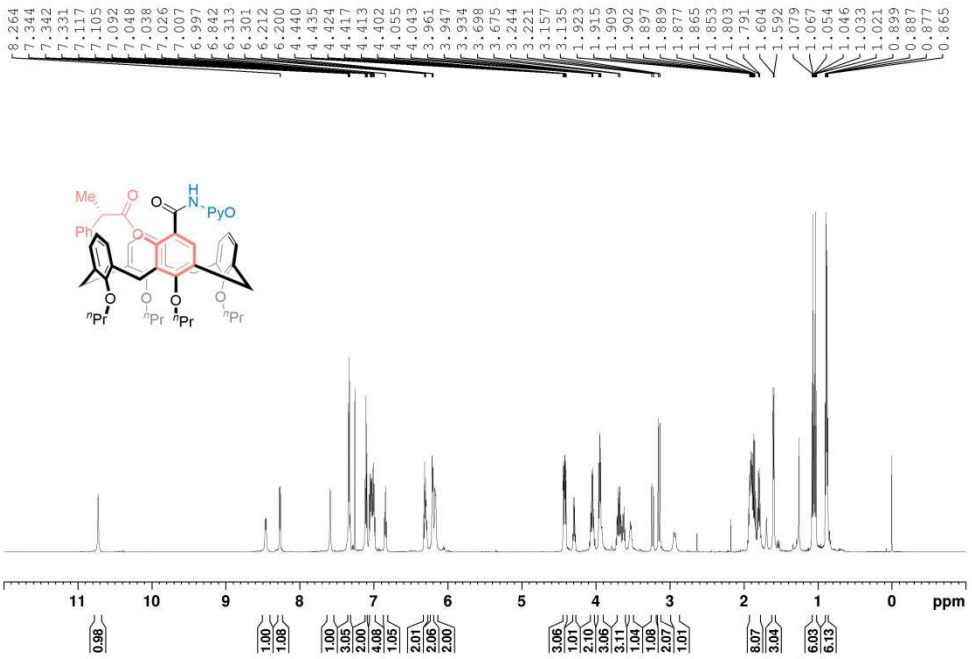
^{1}H NMR (600 MHz, $CDCl_3$), **3aj**



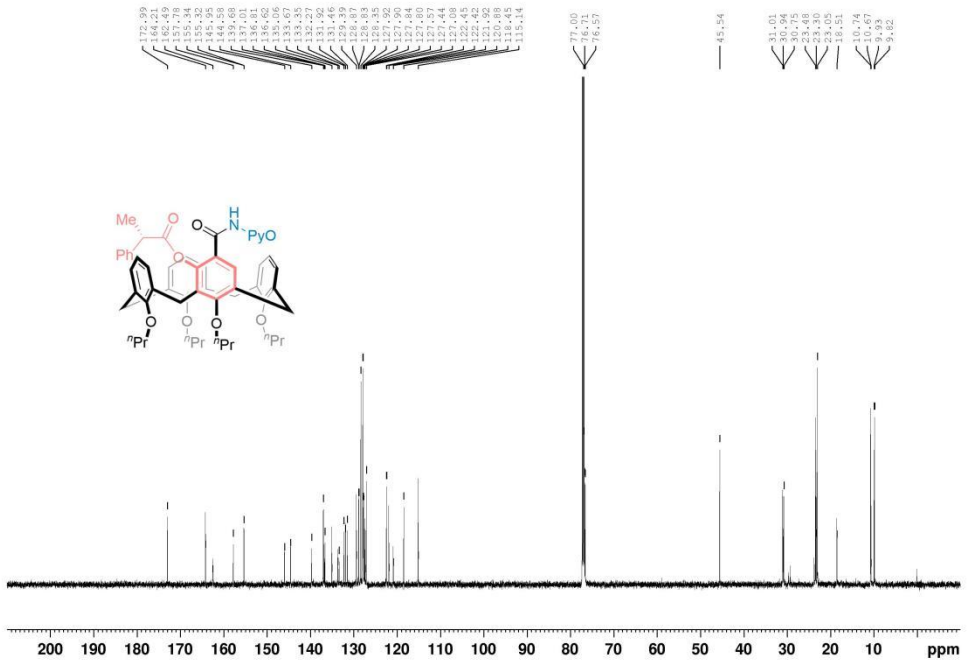
^{13}C NMR (151 MHz, $CDCl_3$), **3aj**



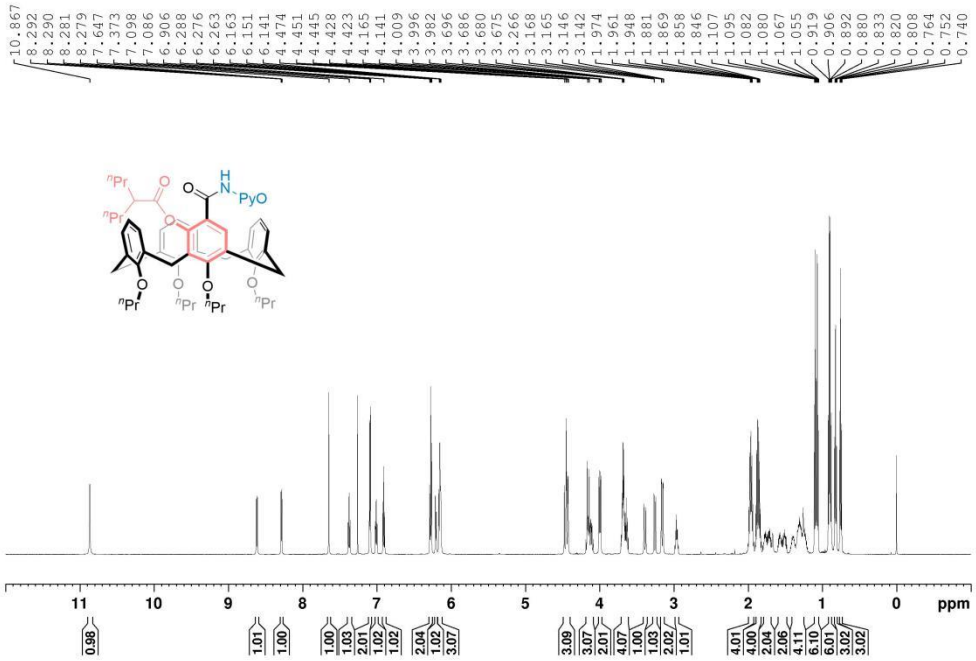
^{1}H NMR (600 MHz, $CDCl_3$), **3ak**



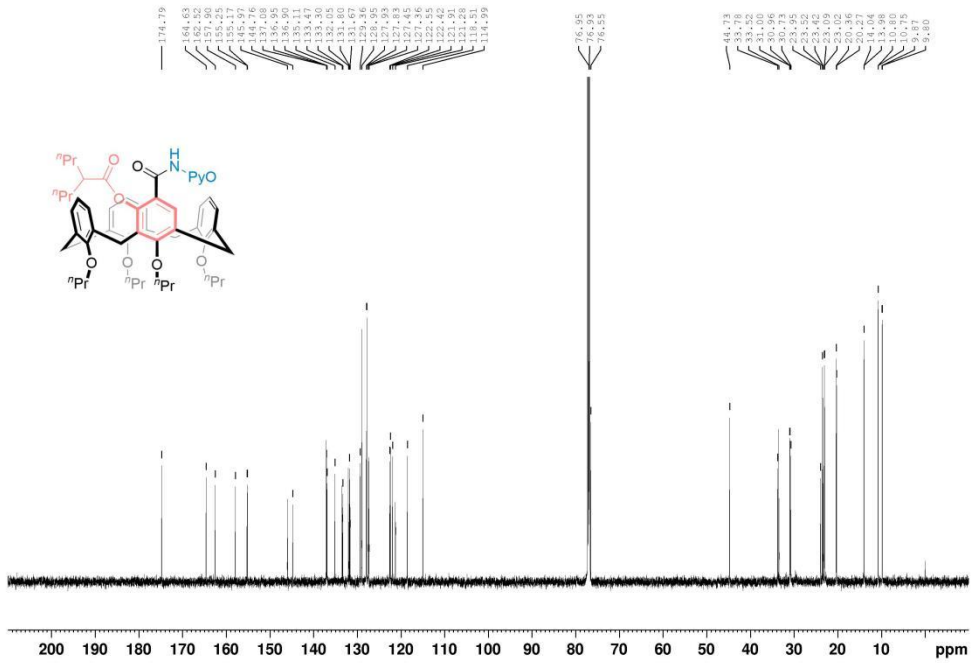
^{13}C NMR (151 MHz, $CDCl_3$), **3ak**



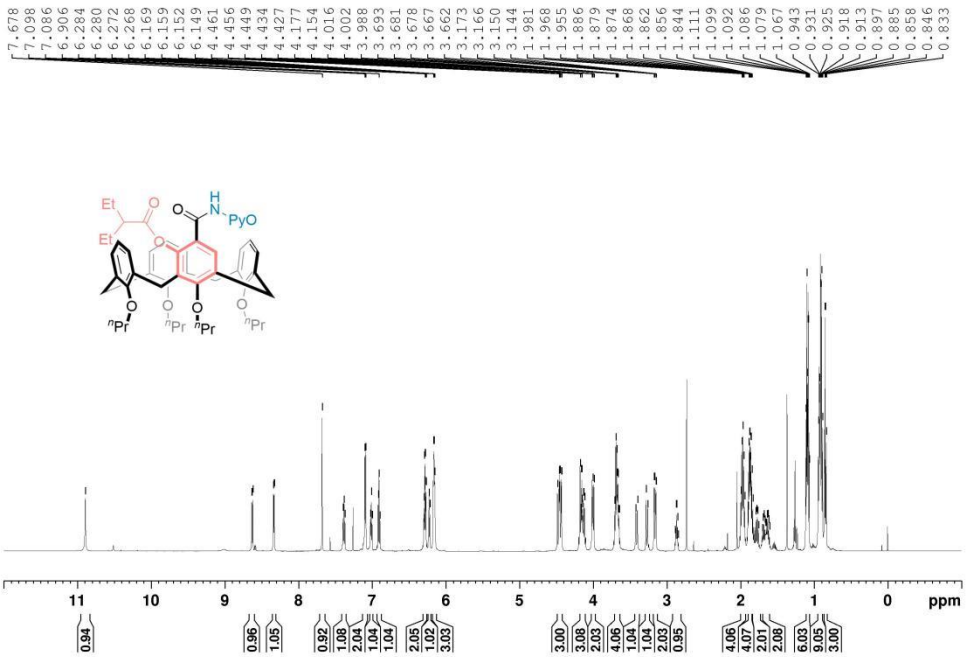
^{1}H NMR (600 MHz, $CDCl_3$), **3al**



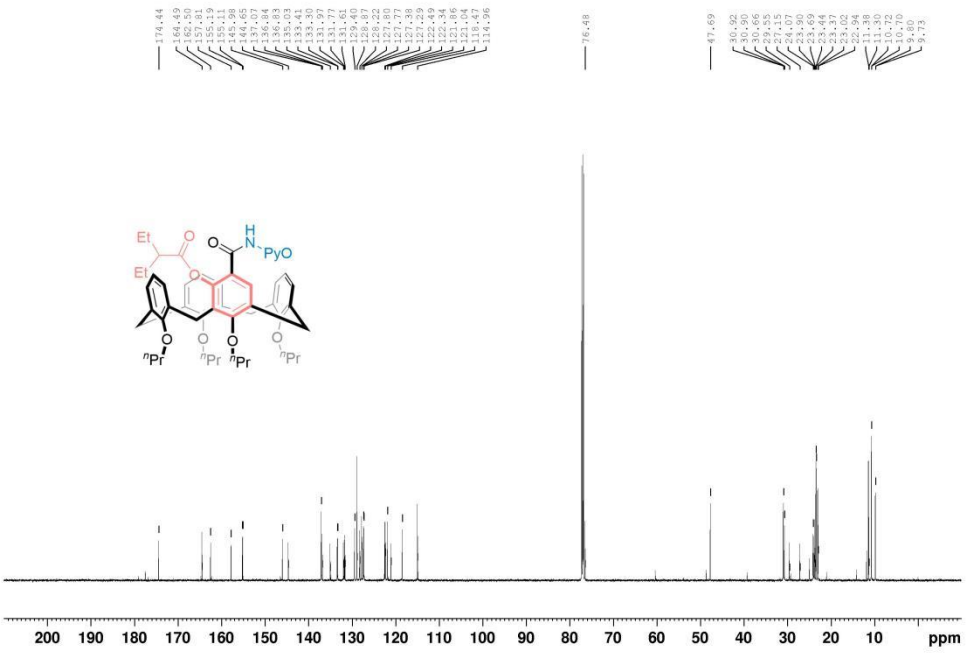
^{13}C NMR (151 MHz, $CDCl_3$), **3al**



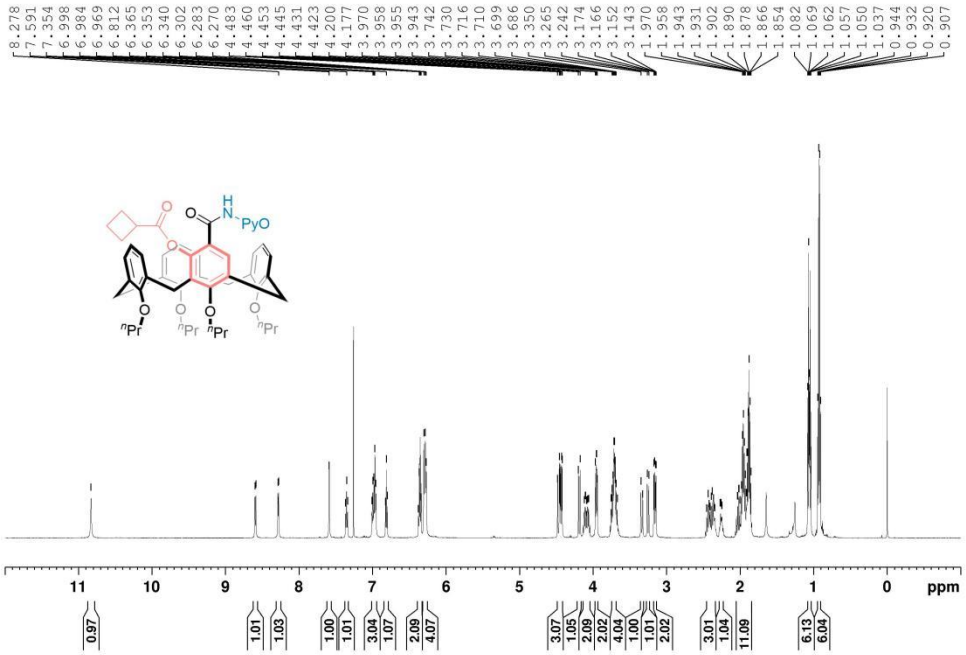
^{1}H NMR (600 MHz, $CDCl_3$), **3am**



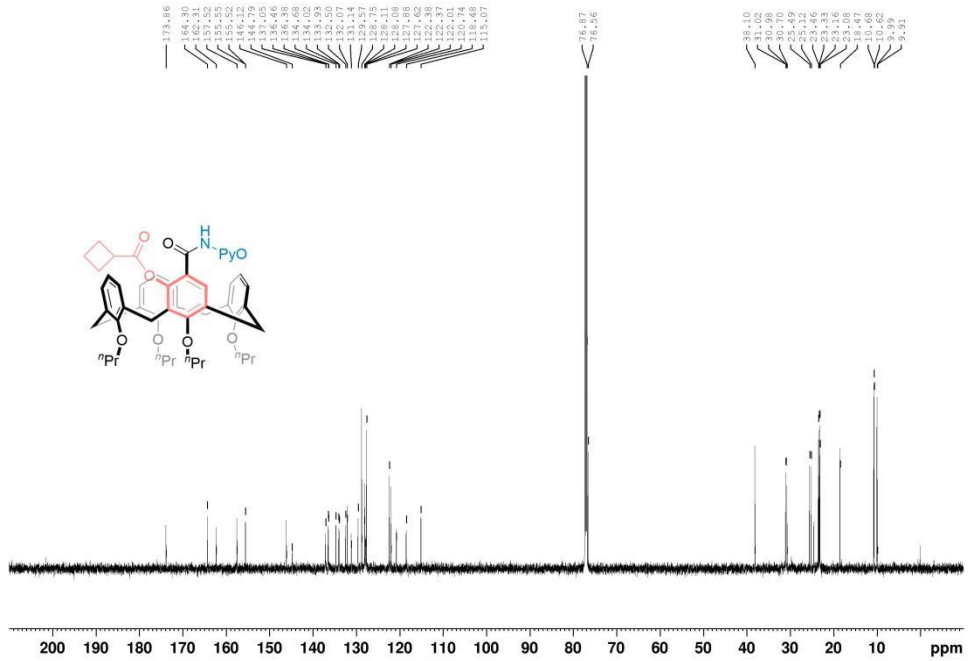
^{13}C NMR (151 MHz, $CDCl_3$), **3am**



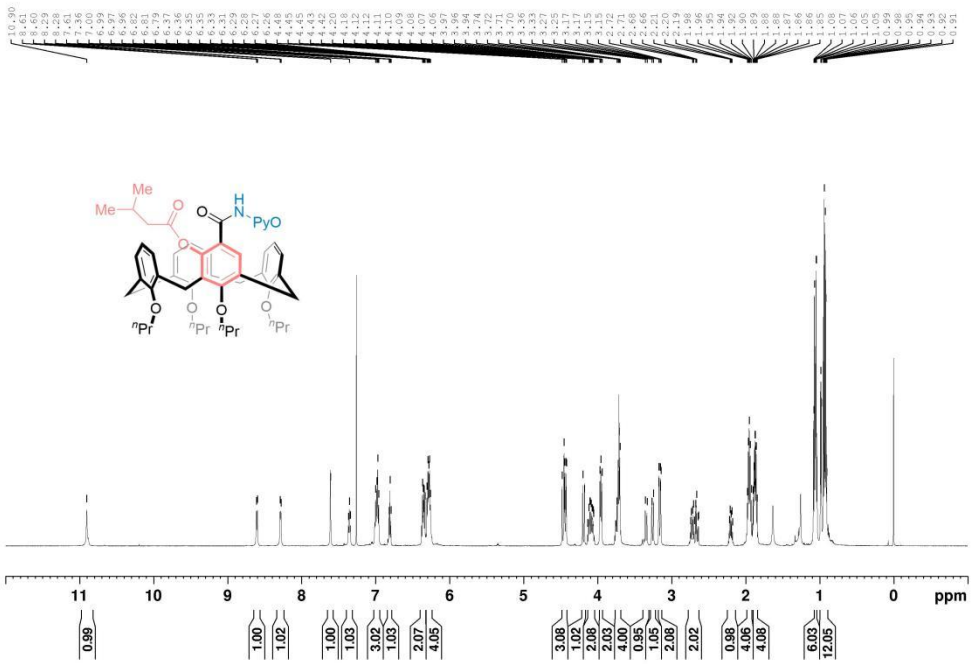
^{1}H NMR (600 MHz, $CDCl_3$), **3an**



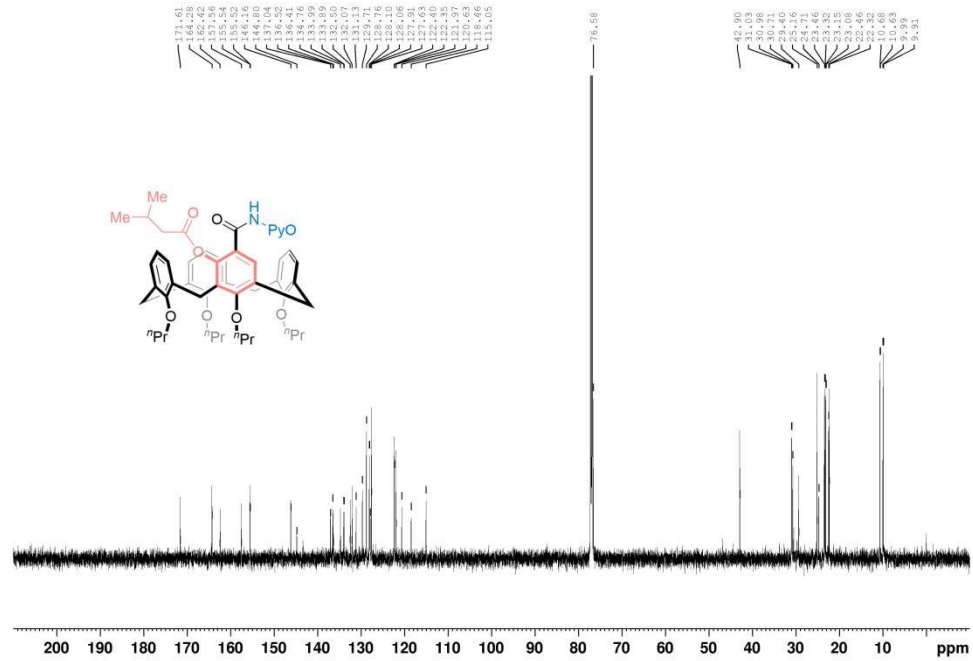
^{13}C NMR (151 MHz, $CDCl_3$), **3an**



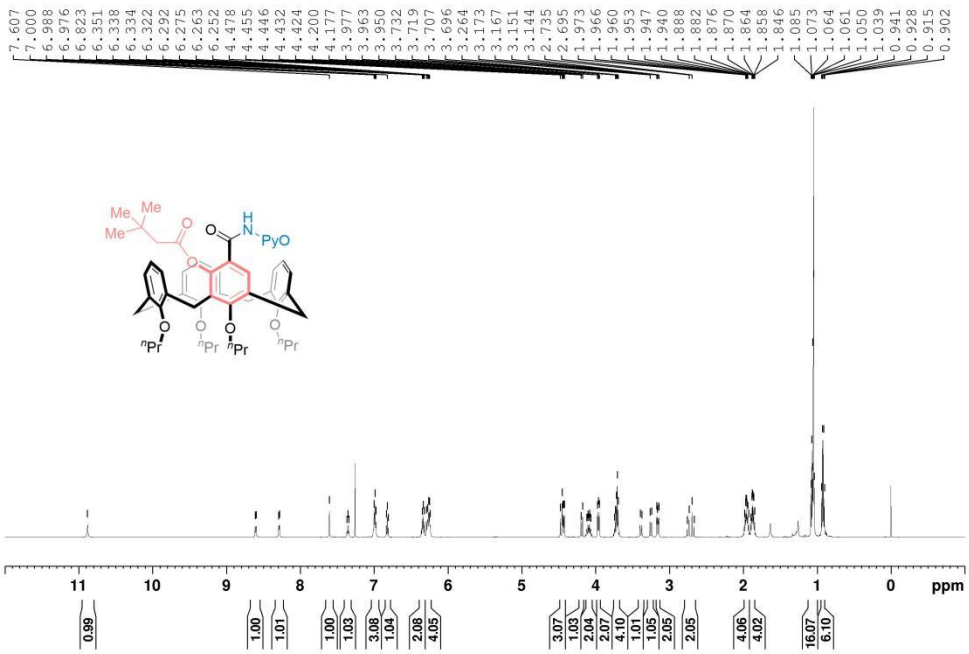
^{1}H NMR (600 MHz, $CDCl_3$), **3ao**



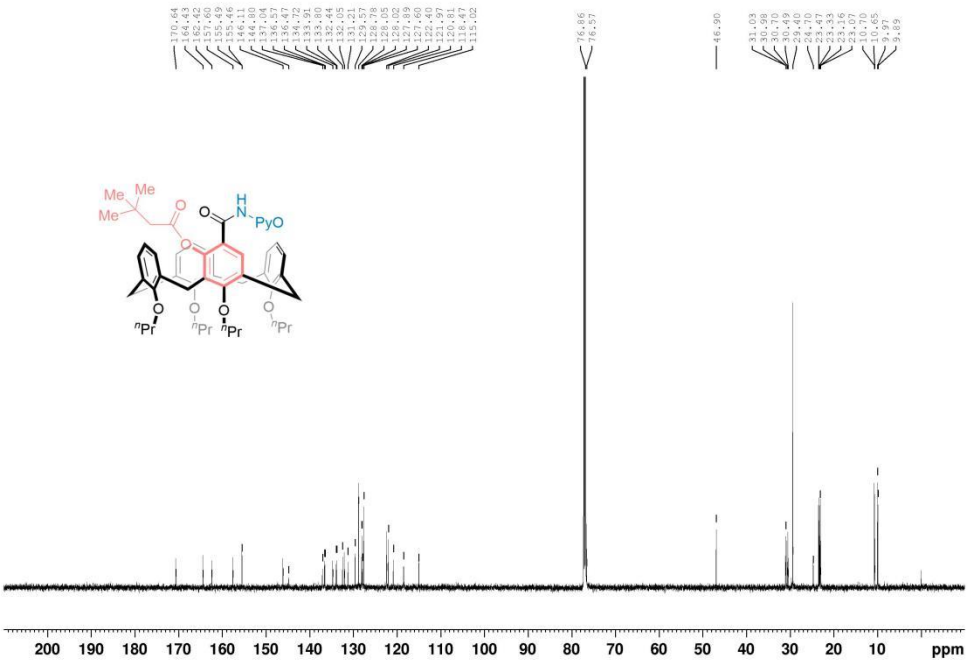
^{13}C NMR (151 MHz, $CDCl_3$), **3ao**



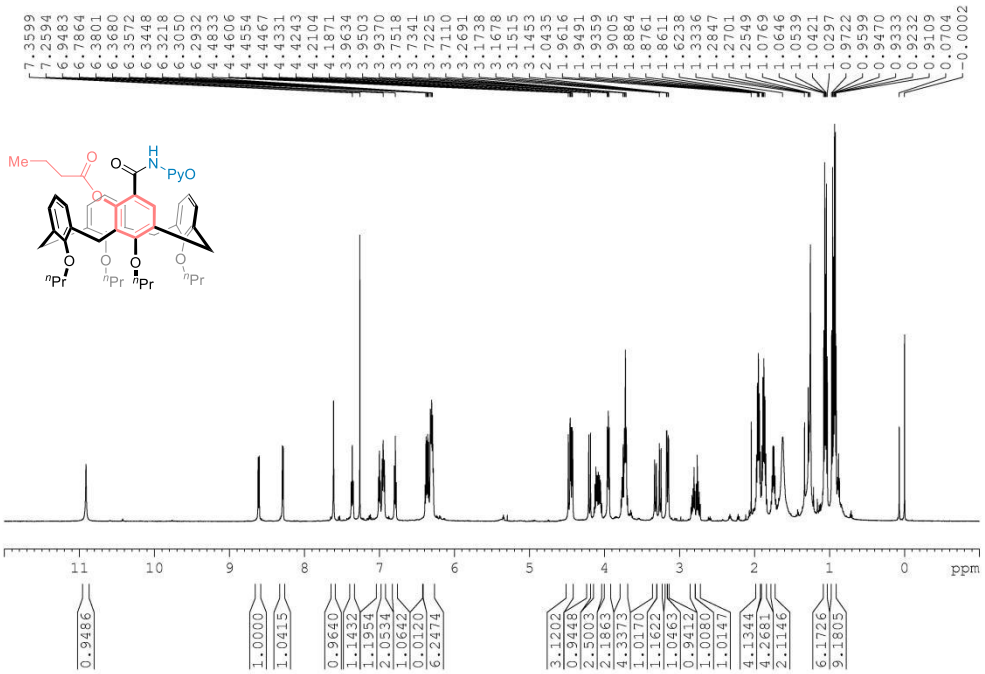
^{1}H NMR (600 MHz, $CDCl_3$), **3ap**



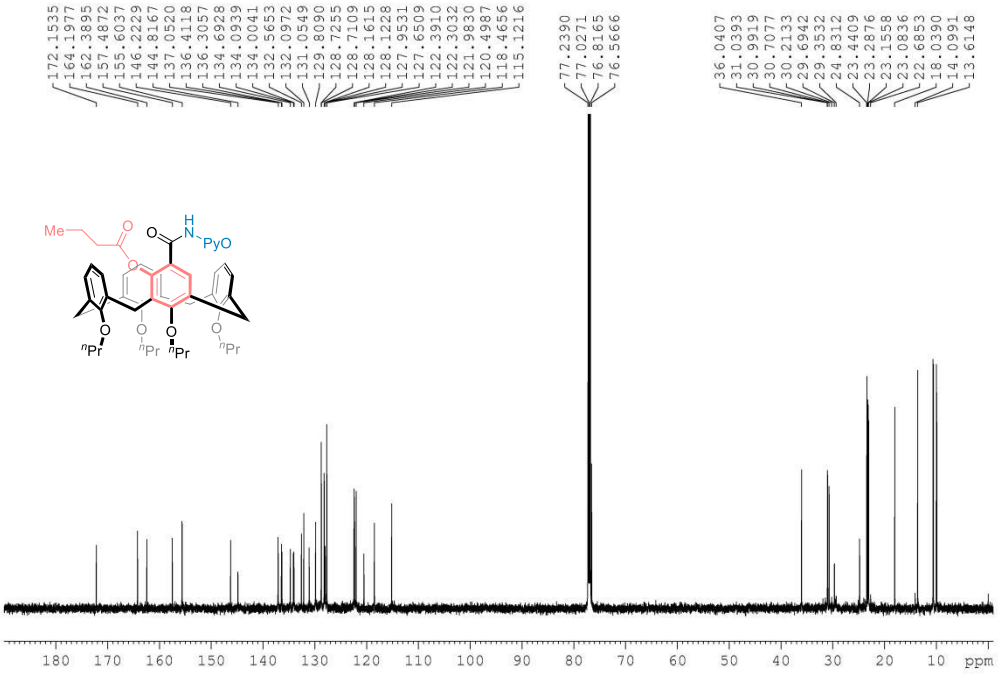
^{13}C NMR (151 MHz, $CDCl_3$), **3ap**



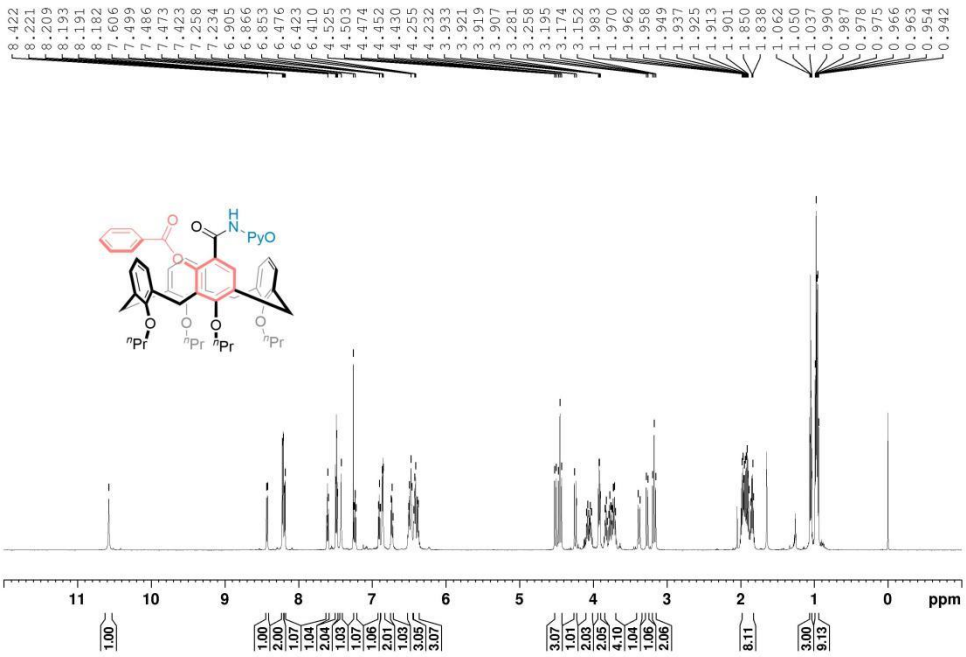
^{1}H NMR (600 MHz, $CDCl_3$), **3aq**



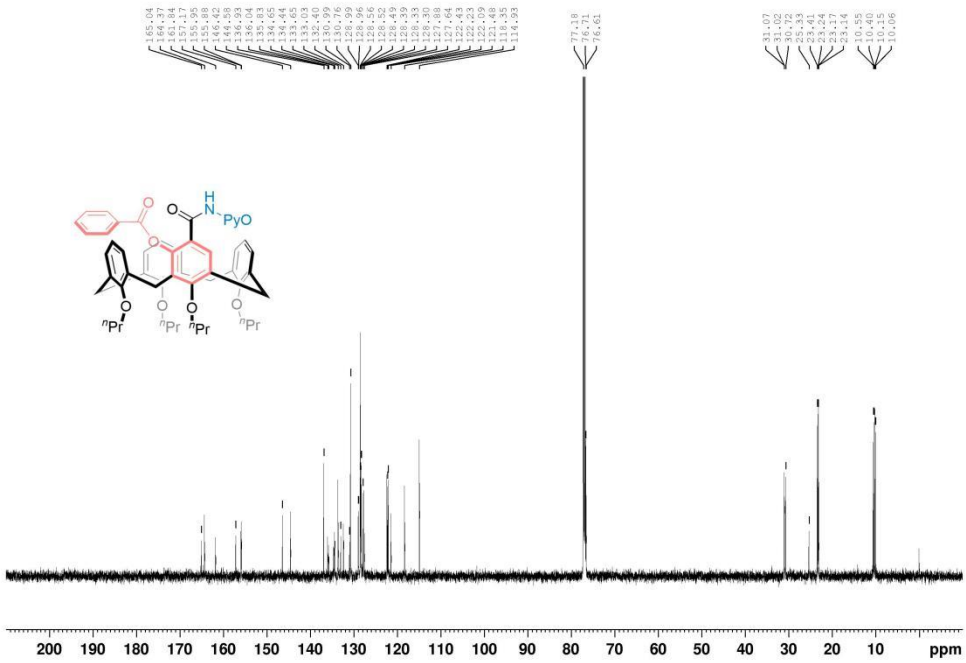
^{13}C NMR (151 MHz, $CDCl_3$), **3aq**



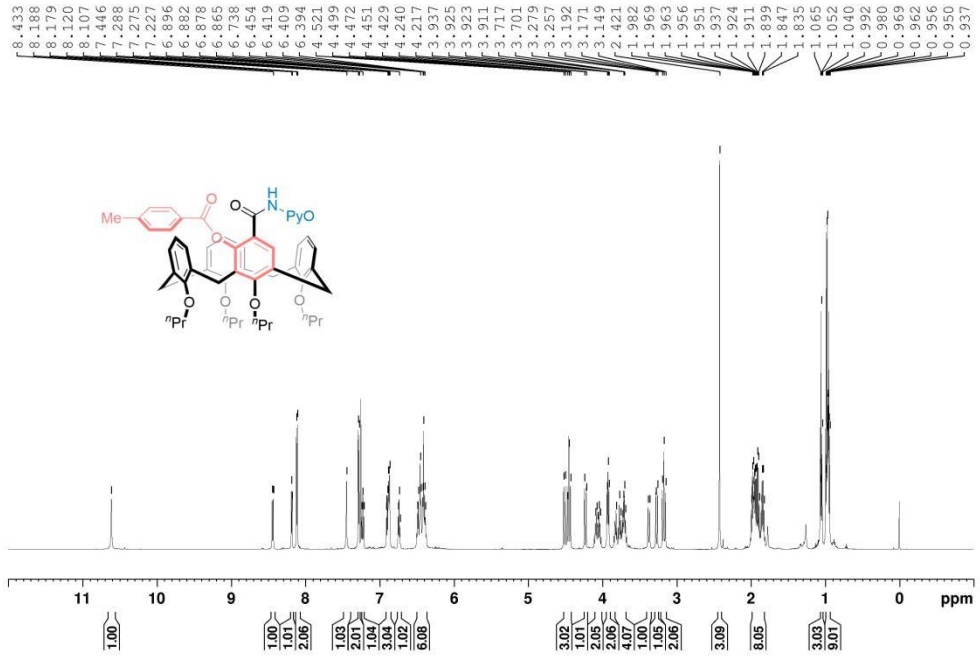
^{1}H NMR (600 MHz, $CDCl_3$), **3ar**



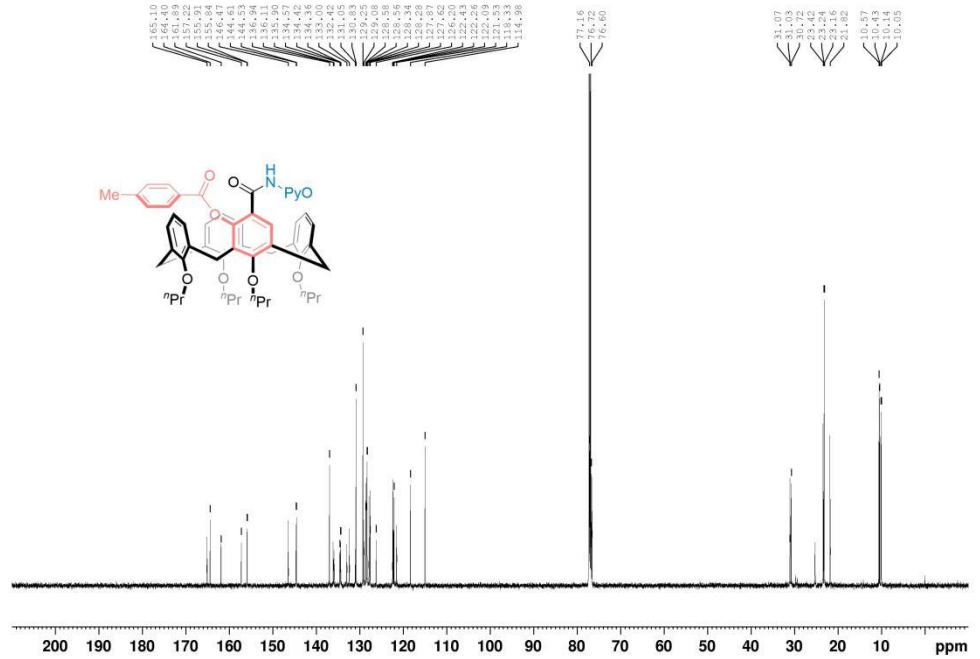
^{13}C NMR (151 MHz, $CDCl_3$), **3ar**



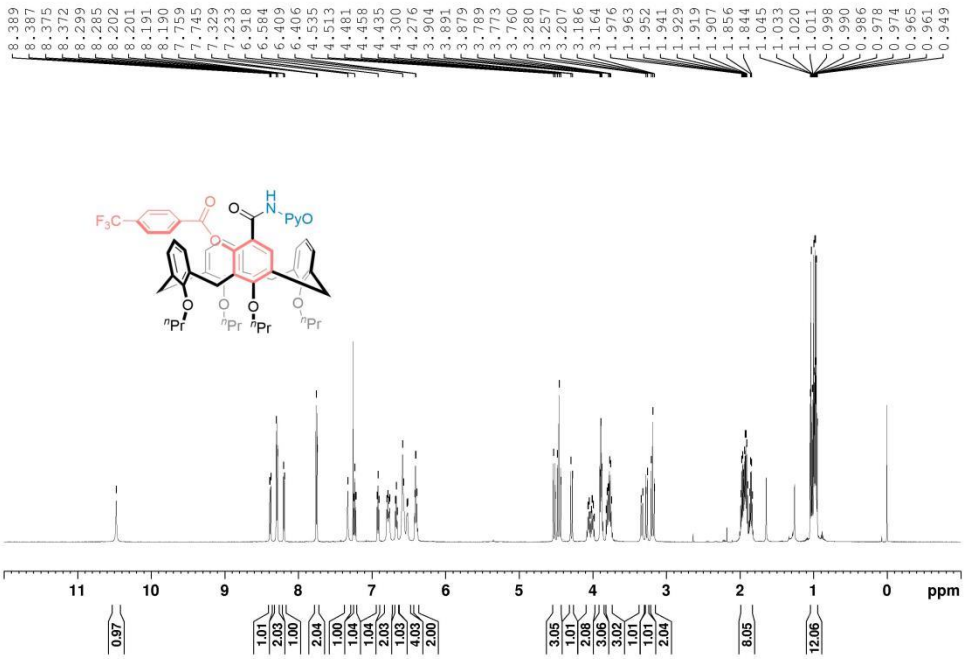
^{1}H NMR (600 MHz, $CDCl_3$), **3as**



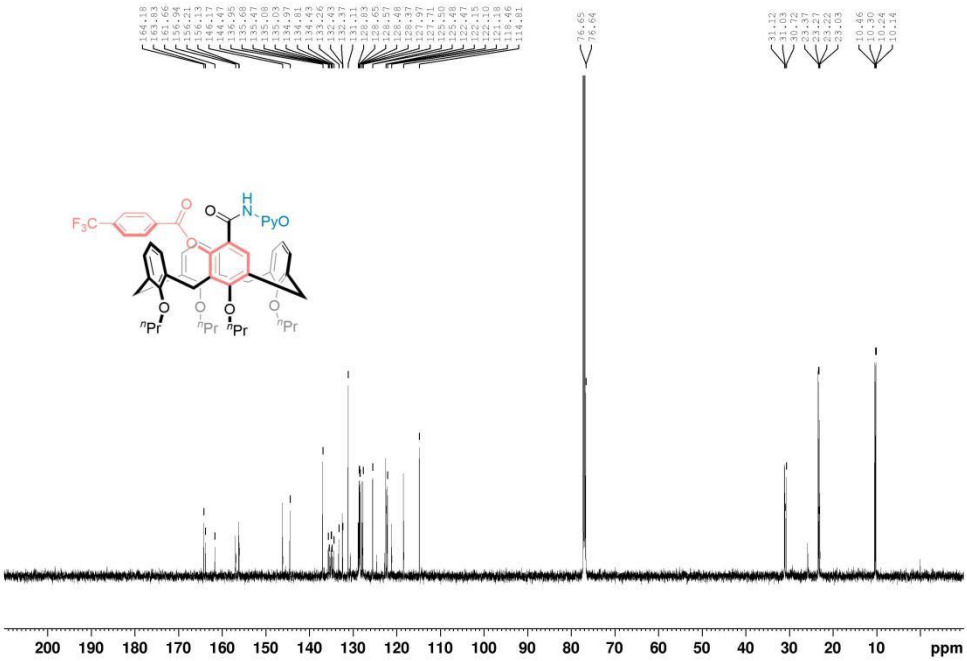
^{13}C NMR (151 MHz, $CDCl_3$), **3as**



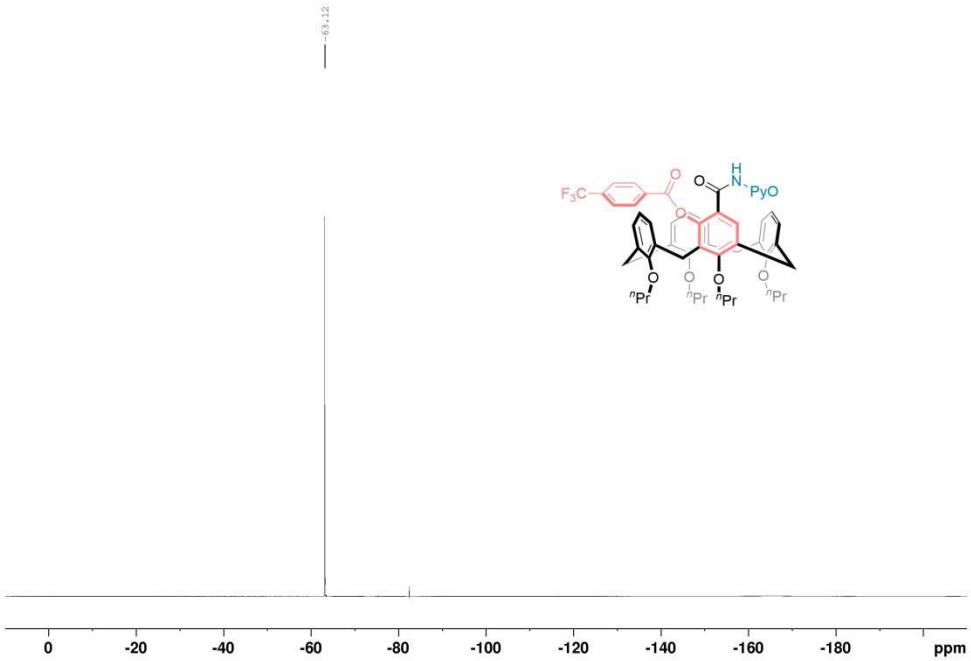
^{1}H NMR (600 MHz, $CDCl_3$), **3at**



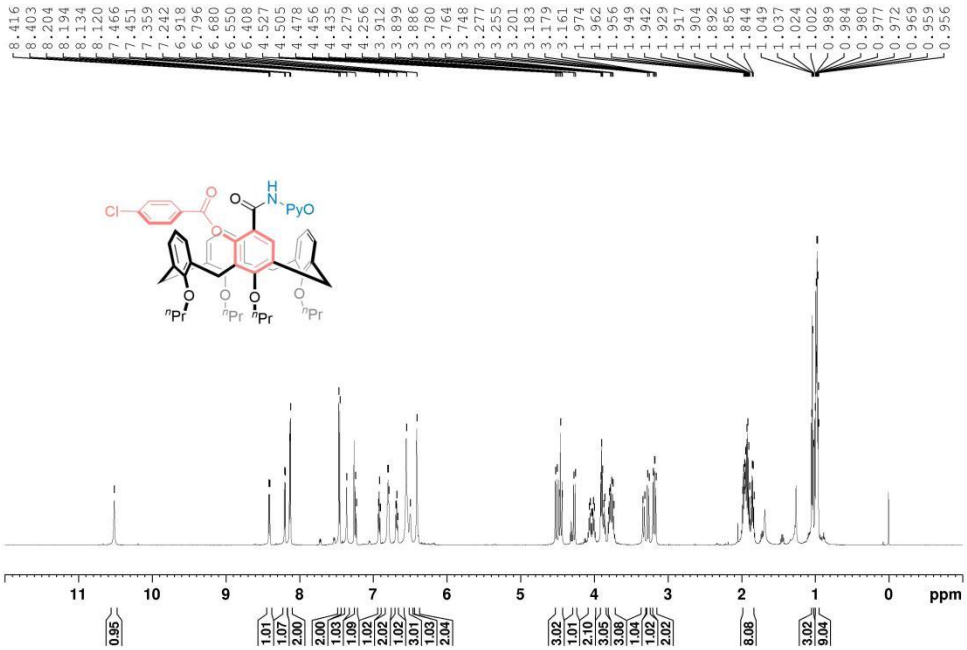
^{13}C NMR (151 MHz, $CDCl_3$), **3at**



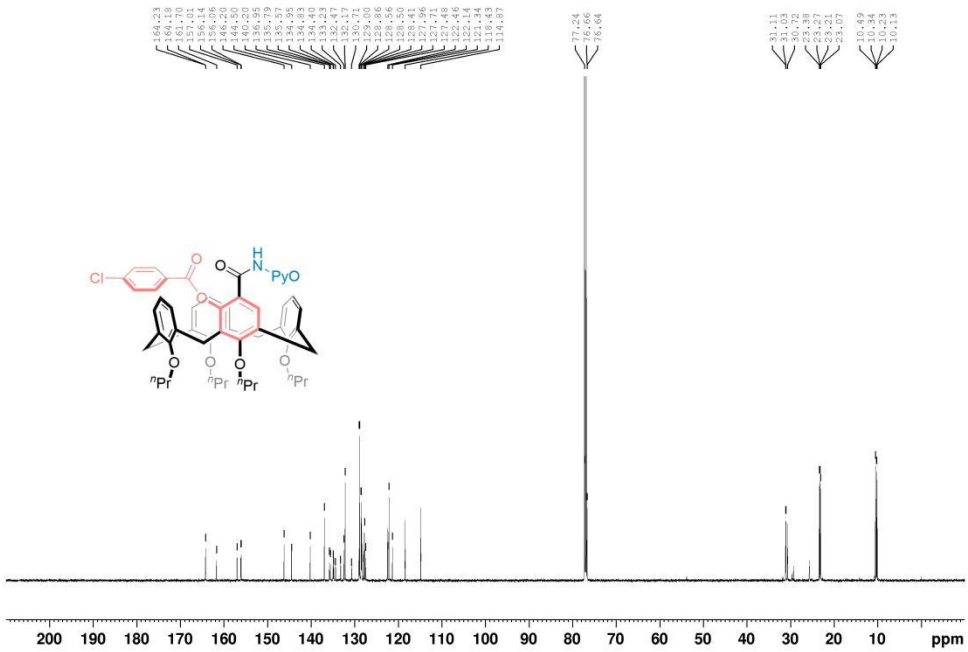
^{19}F NMR (565 MHz, $CDCl_3$), **3at**



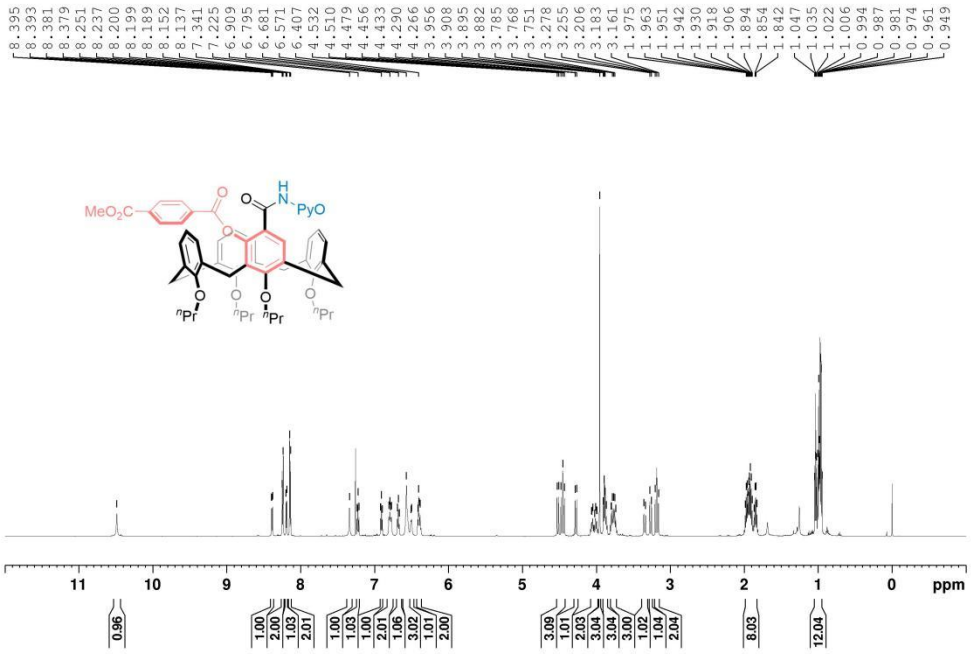
^{1}H NMR (600 MHz, $CDCl_3$), **3au**



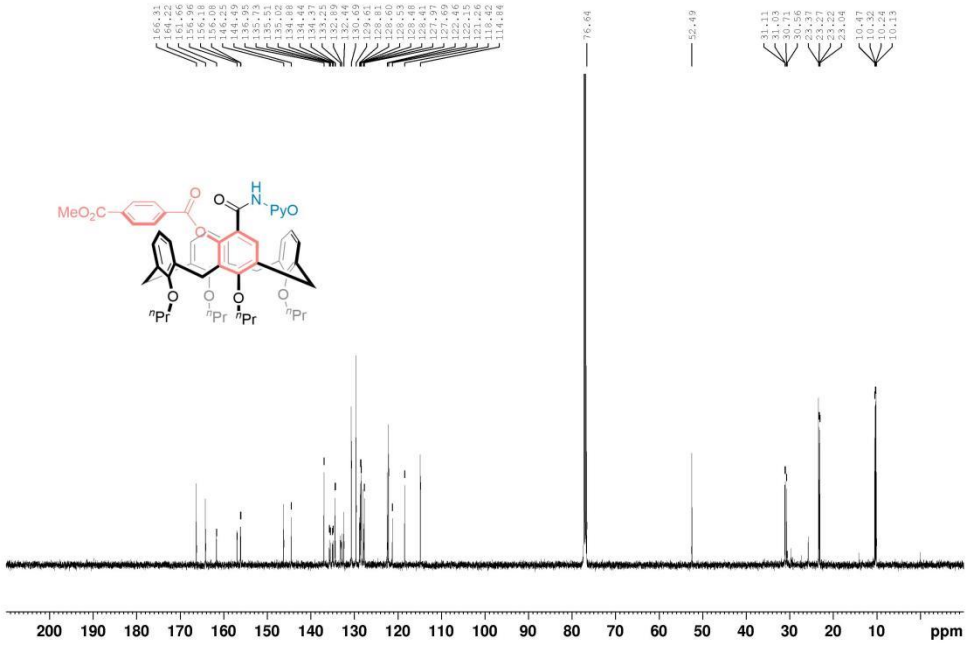
^{13}C NMR (151 MHz, $CDCl_3$), **3au**



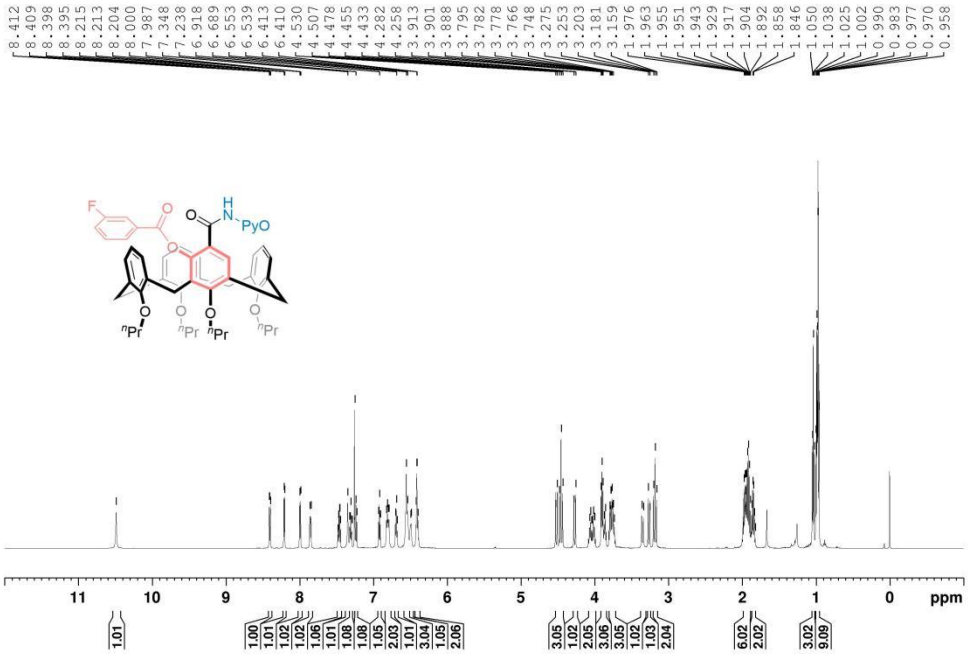
^{1}H NMR (600 MHz, $CDCl_3$), **3av**



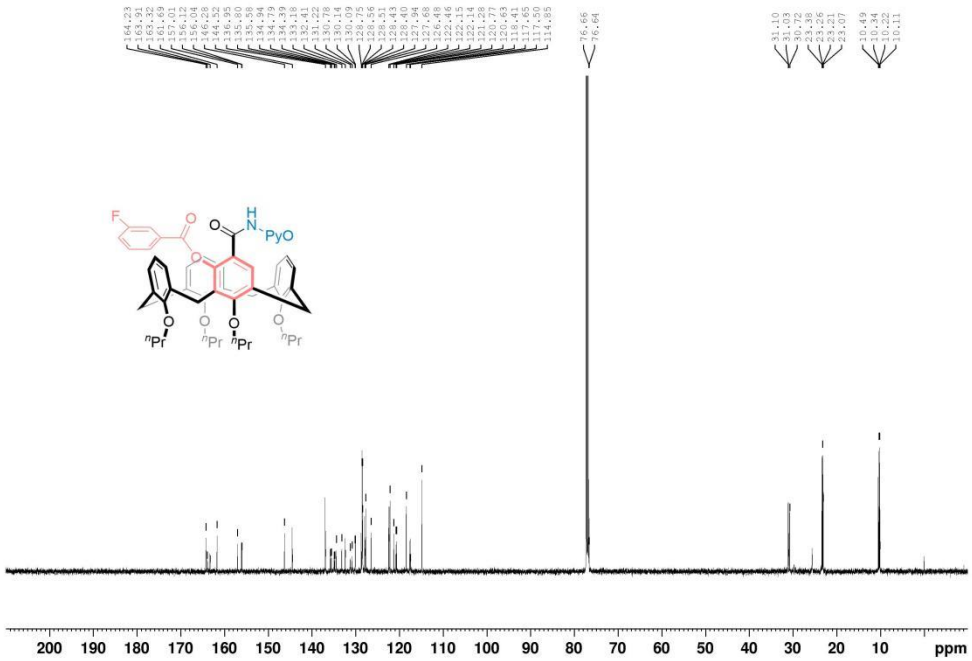
^{13}C NMR (151 MHz, $CDCl_3$), **3av**



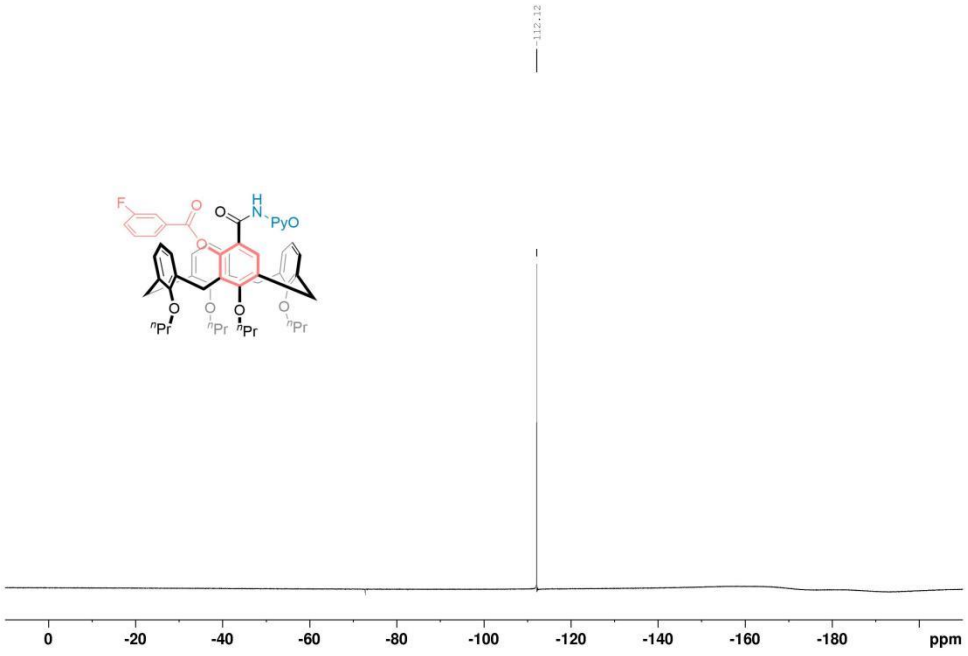
^{1}H NMR (600 MHz, $CDCl_3$), **3aw**



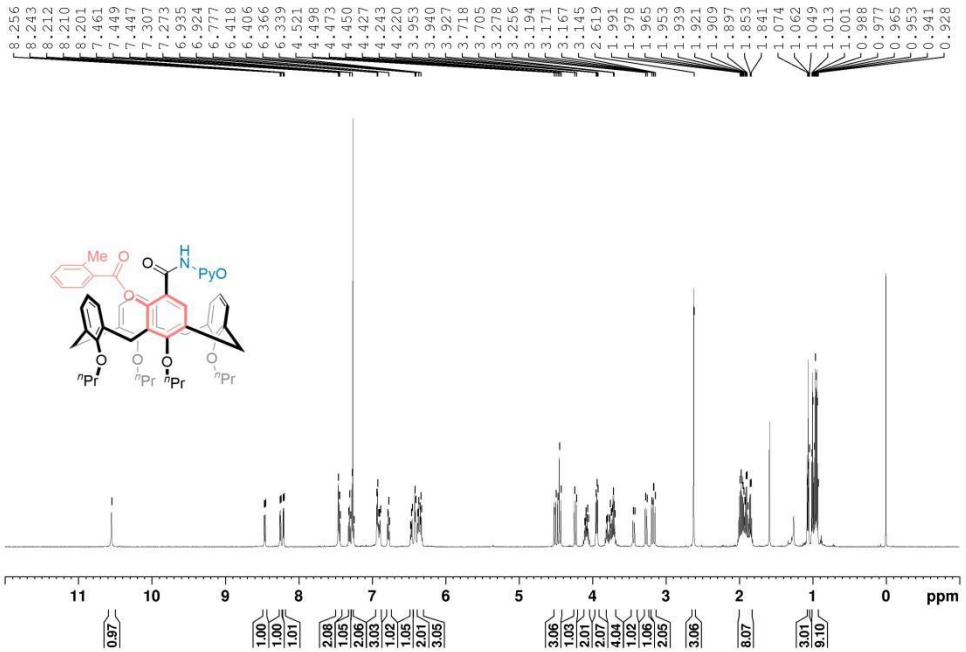
^{13}C NMR (151 MHz, $CDCl_3$), **3aw**



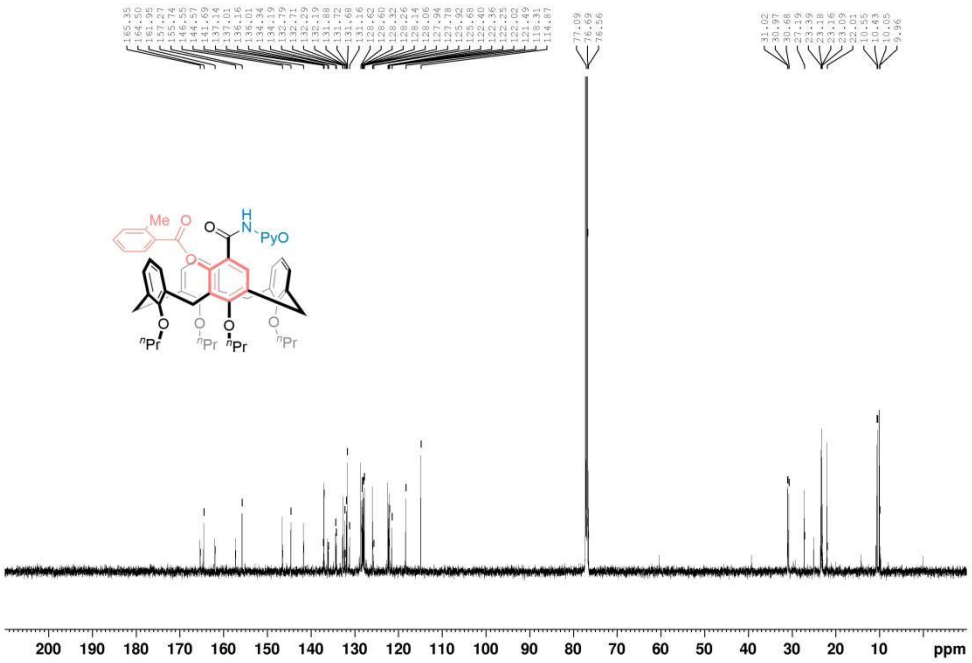
^{19}F NMR (565 MHz, $CDCl_3$), **3aw**



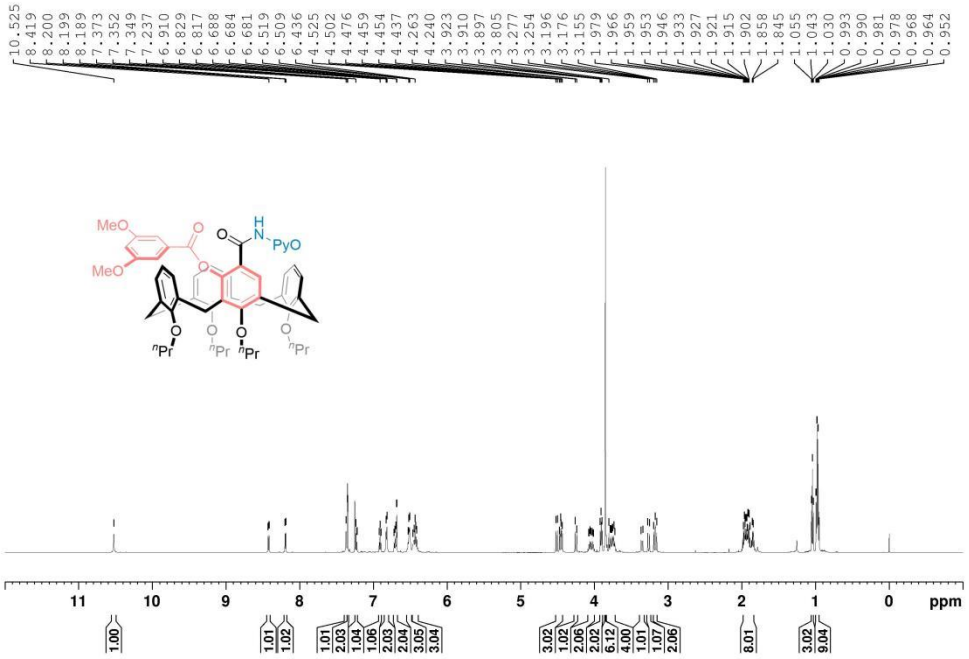
^{1}H NMR (600 MHz, $CDCl_3$), **3ax**



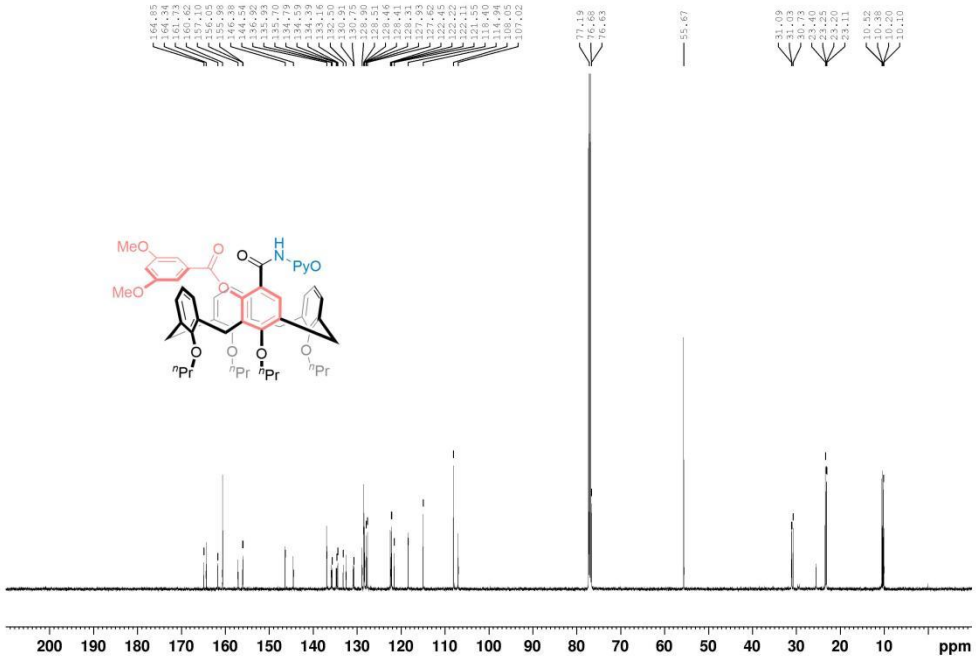
^{13}C NMR (151 MHz, $CDCl_3$), **3ax**



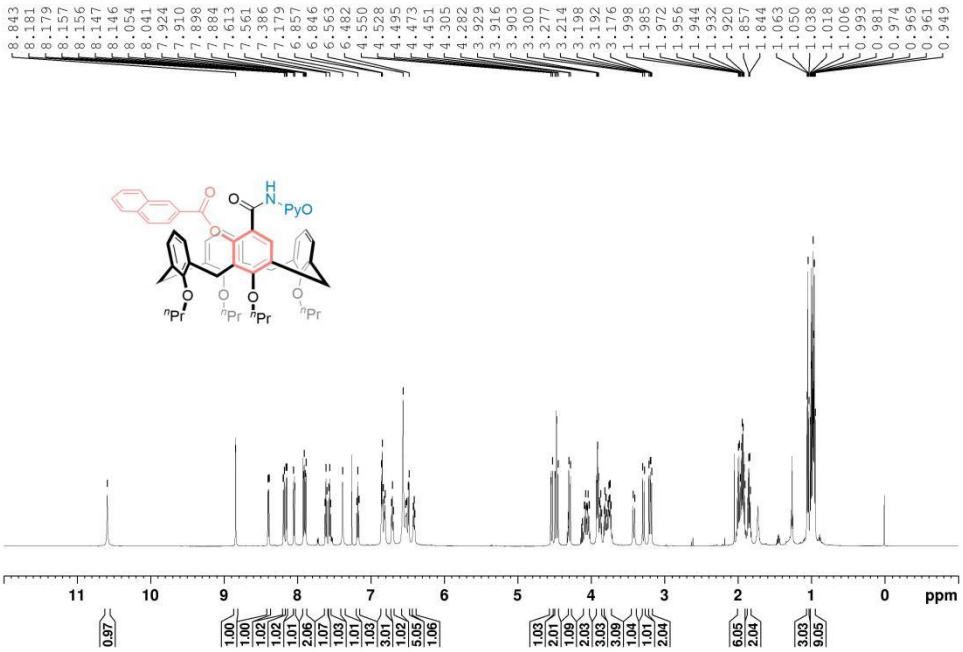
^{1}H NMR (600 MHz, $CDCl_3$), **3ay**



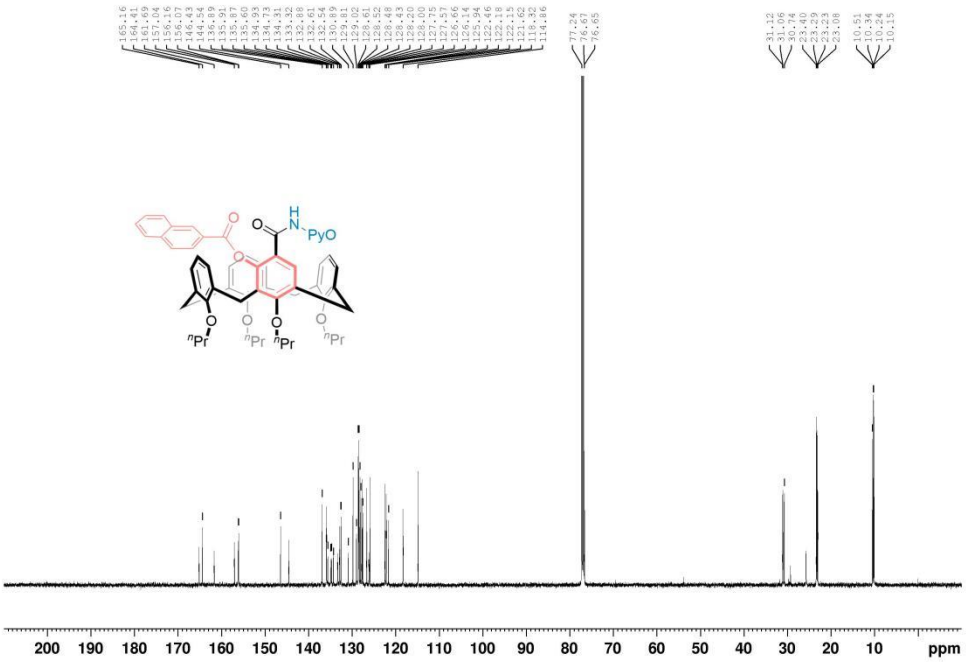
^{13}C NMR (151 MHz, $CDCl_3$), **3ay**



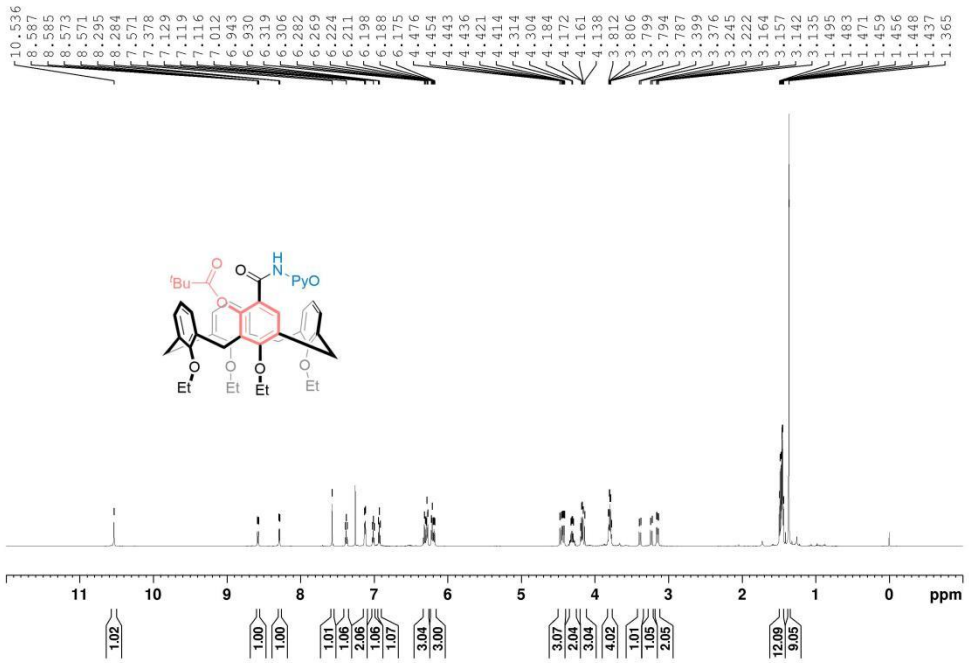
^{1}H NMR (600 MHz, $CDCl_3$), **3az**



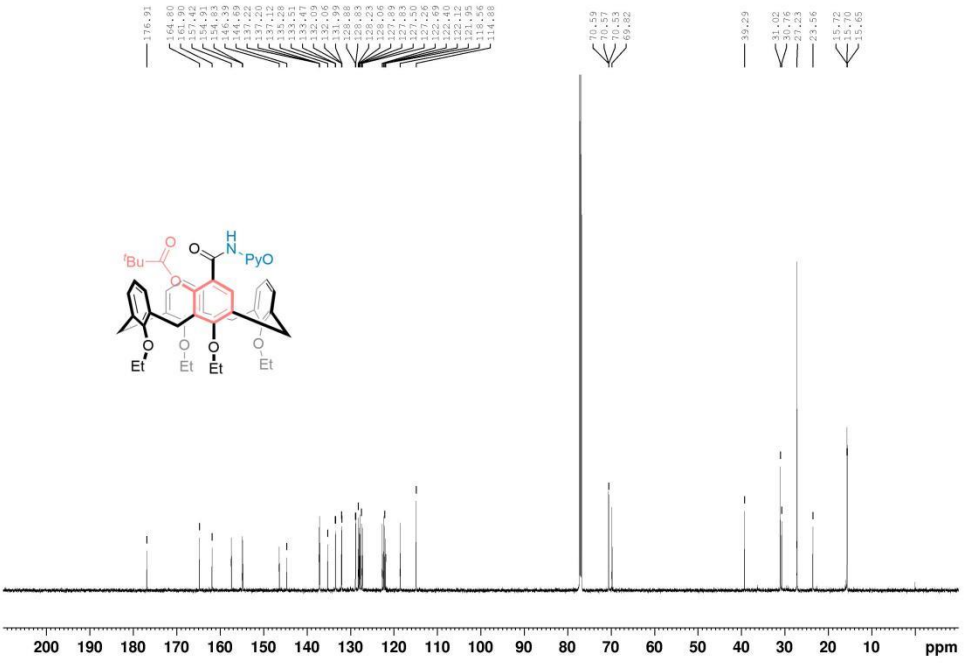
^{13}C NMR (151 MHz, $CDCl_3$), **3az**



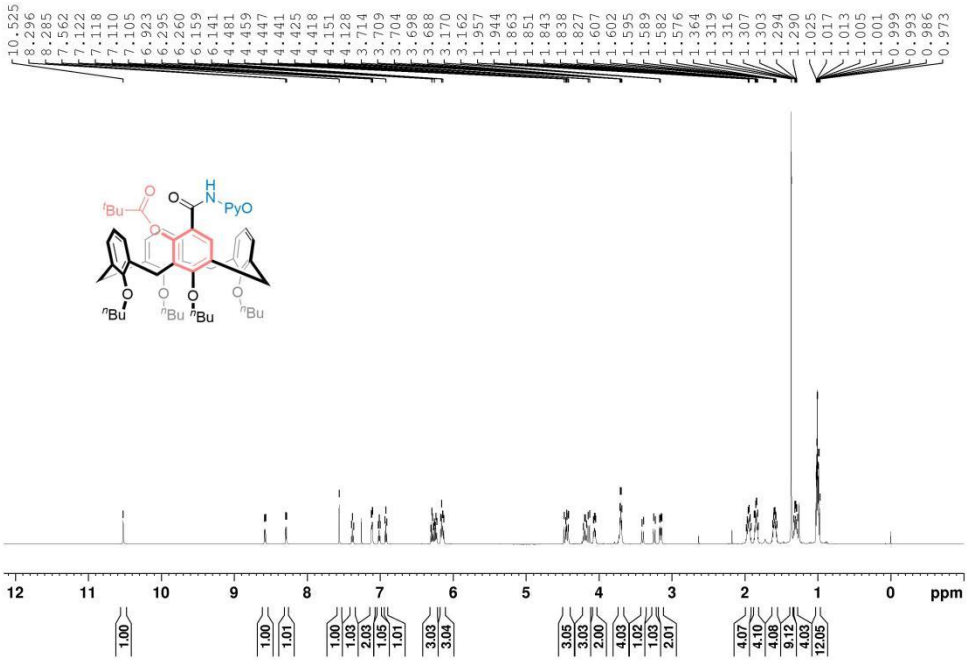
^{1}H NMR (600 MHz, $CDCl_3$), **3ba**



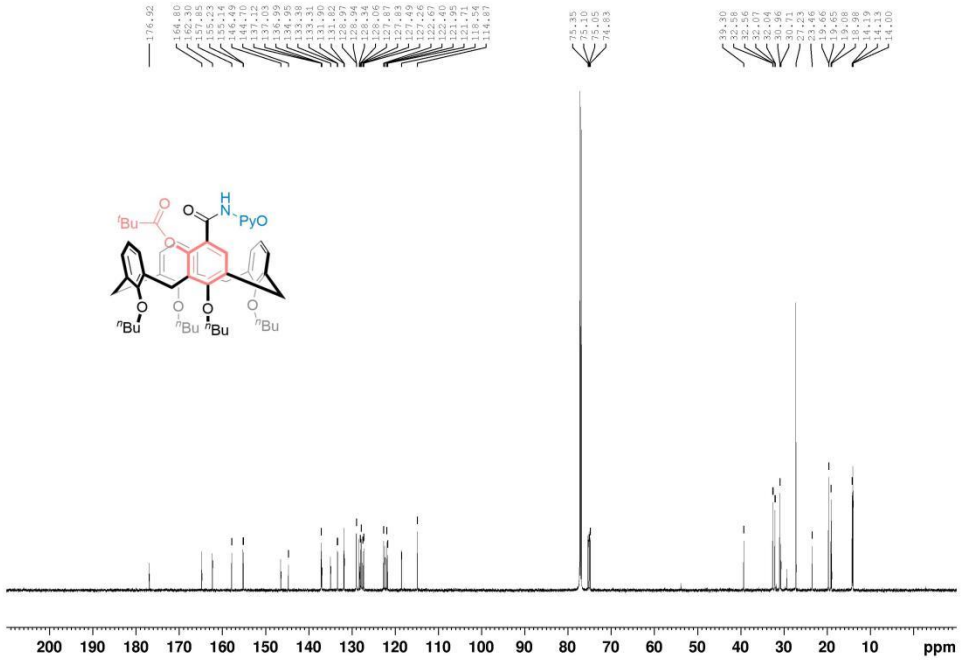
^{13}C NMR (151 MHz, $CDCl_3$), **3ba**



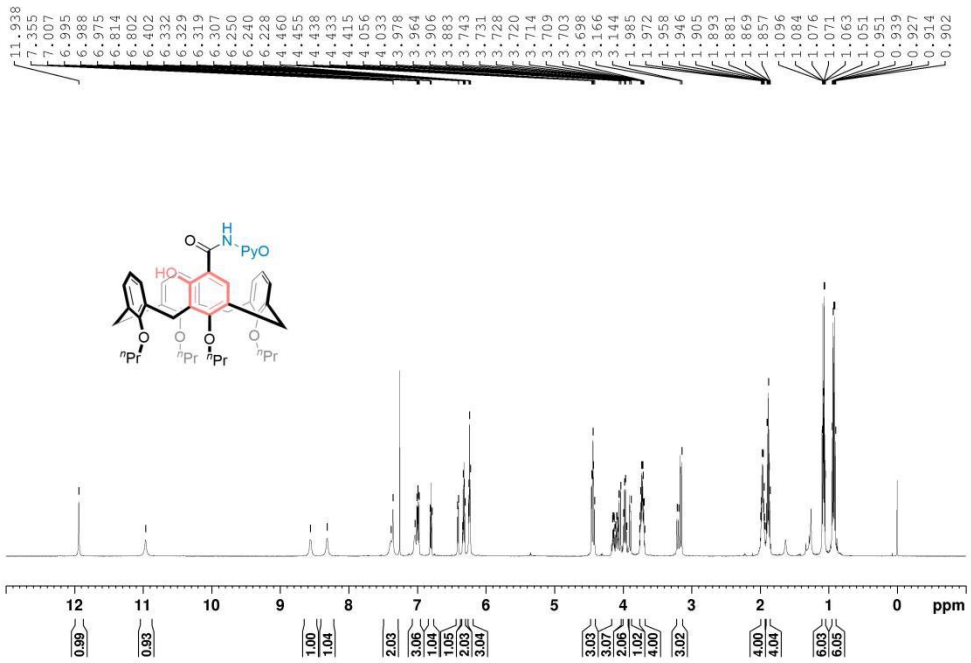
^{1}H NMR (600 MHz, $CDCl_3$), **3ca**



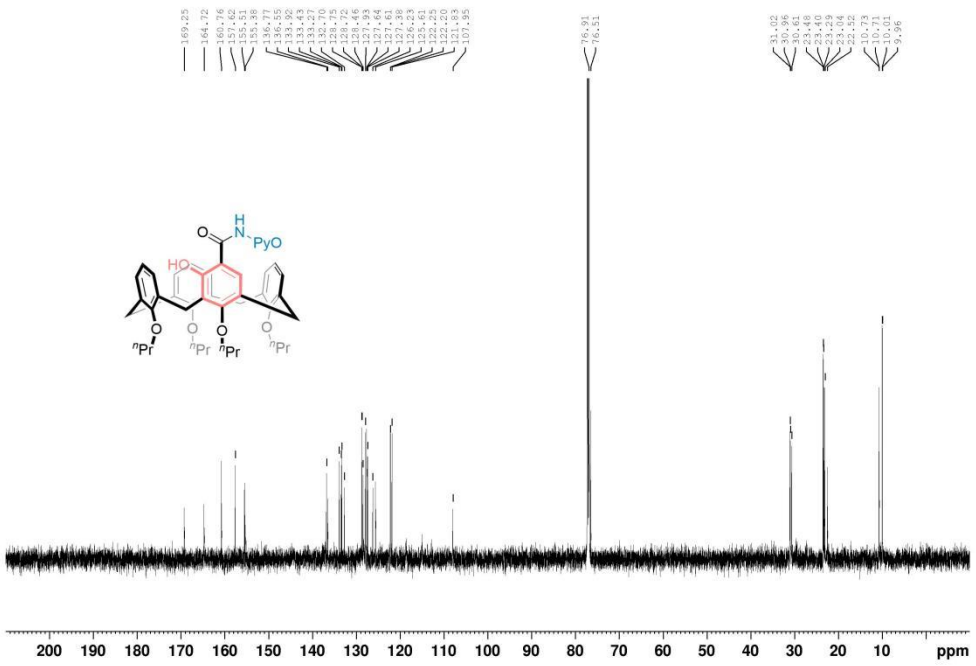
^{13}C NMR (151 MHz, $CDCl_3$), **3ca**



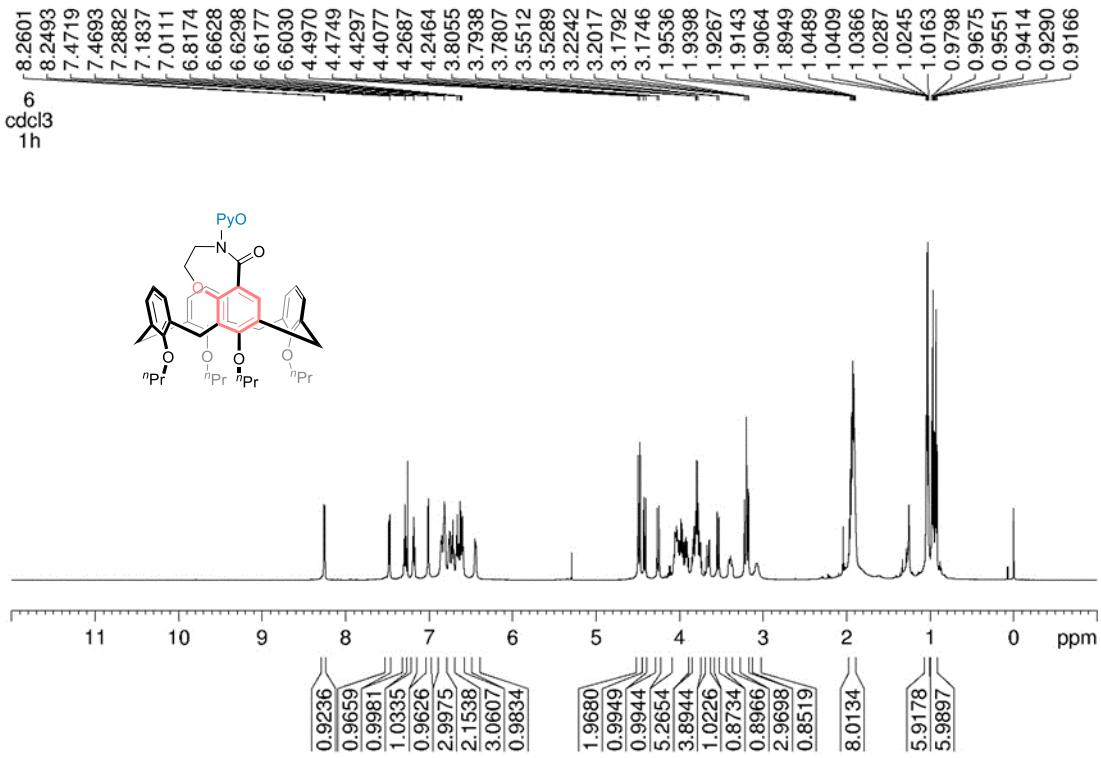
^{1}H NMR (600 MHz, $CDCl_3$), **4**



^{13}C NMR (151 MHz, $CDCl_3$), **4**



^{1}H NMR (600 MHz, $CDCl_3$), **6**



^{13}C NMR (151 MHz, $CDCl_3$), **6**