

## Content

1.	General information .....	2
2.	Optimization of the reactions .....	3
a.	$\alpha$ -amino carboxylates .....	3
b.	$\alpha$ -amino phosphonates .....	6
3.	General procedure for cyclopropanation .....	9
4.	Characterization of new compounds .....	10
5.	Post-functionalization reactions .....	26
6.	NMR spectra .....	29
7.	HPLC chromatograms .....	83
8.	X-ray crystallographic data of 1 (CCDC 2177765) .....	123
9.	X-ray crystallographic data of 27 (CCDC 2350765) .....	131
10.	X-ray crystallographic data of 28 (CCDC 2350766) .....	144
11.	References .....	160

## 1. General information

All manipulations were carried out using flame dried glassware and magnetic stirring under Argon atmosphere.  $\text{Rh}_2(\text{S}-(\text{TCPTAD}))_4$ ,  $\text{Rh}_2(\text{R}-(\text{BTPCB}))_4$ ,  $\text{Rh}_2(\text{esp})_2$  and  $\text{Rh}_2(\text{R}-(\text{p-PhTPCP}))_4$  were purchased from Sigma Aldrich. Tetrahydrofuran (THF) and toluene (tol.) were distilled over sodium and benzophenone and dichloromethane ( $\text{CH}_2\text{Cl}_2$ ) and chloroform ( $\text{CHCl}_3$ ) were distilled over calcium hydride. Other solvents were purchased over molecular sieves and commercially available reagents were purchased from standard chemical suppliers (Sigma Aldrich and Fisher Scientific) and used as received without further purification. Column chromatographies were carried out using silica gel (40-63  $\mu\text{m}$ ) supplied by VWR and analytical thin layer chromatographies (TLC) performed on pre-coated silica gel aluminum plates (60 F254 from Merck) were visualized by UV light (254 nm) and/or stained with ninhydrin.

$^1\text{H}$  (300 MHz),  $^{13}\text{C}$  (75 MHz) and  $^{19}\text{F}$  (282 MHz) NMR spectra were recorded on a Bruker Avance 300 in  $\text{CDCl}_3$  unless otherwise noted.  $^1\text{H}$  (400 MHz),  $^{13}\text{C}$  (101 MHz),  $^{19}\text{F}$  (376 MHz) and  $^{31}\text{P}$  (162 MHz) NMR spectra were recorded on a Bruker Ascend Evo 400 in  $\text{CDCl}_3$  unless otherwise noted. Chemical shifts  $\delta$  are quoted in ppm relatively to the residual solvent peak for  $\text{CDCl}_3$  ( $\delta_{\text{H}} = 7.26$  ppm and  $\delta_{\text{C}} = 77.16$  ppm). Coupling constants  $J$  are expressed in Hertz (Hz) and the following abbreviations for multiplicities are used: s for singlet, br s for broad singlet, d for doublet, t for triplet, q for quartet, dd for doublet of doublets and m for multiplet. HPLC analysis were performed on a ThermoFisher UHPLC system Ultimate RSLC with a PDA detector and columns with chiral stationary phase from Daicel (250 mm x 4.6 mm, 5  $\mu\text{m}$  particle size) at 20 °C. Optical rotations were recorded on a PerkinElmer Polarimeter 341 at 20 °C in  $\text{CHCl}_3$  or  $\text{CH}_2\text{Cl}_2$ . Melting points were recorded on a Stuart SMP3 melting point apparatus. High-resolution mass spectra (HRMS) were recorded on Waters LCT Premier. IR spectra were recorded on a PerkinElmer Spectrum 100.

Methyl-2-(di(*tert*-butoxycarbonyl)amino)but-2-enoate<sup>1</sup> (**I**) was synthesized following reported procedures. Diazo compounds were synthesized according to the literature.<sup>2-7</sup>  $\text{Rh}_2(\text{S}-(\text{p-PhTPCP}))_4$  was synthesized following reported procedures.<sup>8</sup>

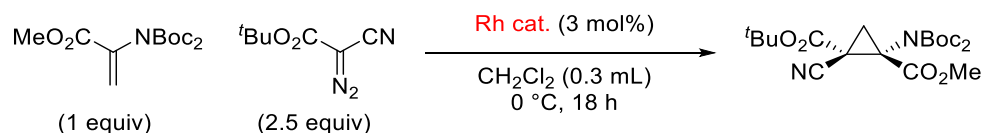
Diethyl [1-(*tert*butoxycarbonylamino)ethen-1-yl]phosphonate (**II**) and Diethyl [1-(benzyloxycarbonylamino)ethen-1-yl]phosphonate (**III**) were synthesized according to the literature.<sup>9</sup>

## 2. Optimization of the reactions

### a. $\alpha$ -amino carboxylates

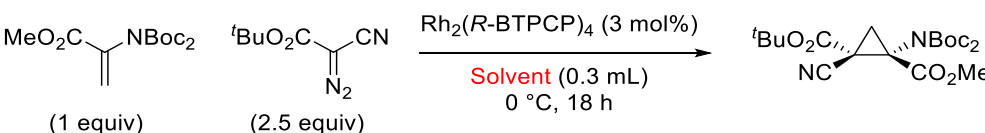
Methyl-2-(di(*tert*-butoxycarbonyl)amino)but-2-enoate **I** and *tert*-butyl 2-cyano-2-diazoacetate were initially utilized to find optimal conditions in terms of catalyst, solvent, temperature, concentration, and reaction stoichiometry (Table S1-5).

**Table S1.** Catalyst evaluation

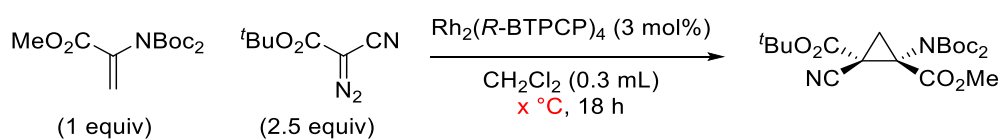


Entry	Rh cat.	NMR yield (%)	ee (%)
1	Rh <sub>2</sub> ( <i>R</i> -BTPCP) <sub>4</sub>	93	94
2	Rh <sub>2</sub> ( <i>S</i> -TCPTAD) <sub>4</sub>	70	52
3	Rh <sub>2</sub> ( <i>S</i> -DOSP) <sub>4</sub>	/	/
4	Rh <sub>2</sub> ( <i>S</i> -IBAZ) <sub>4</sub>	32	/
5	Rh <sub>2</sub> ( <i>S</i> -TCPTTL) <sub>4</sub>	<5	/

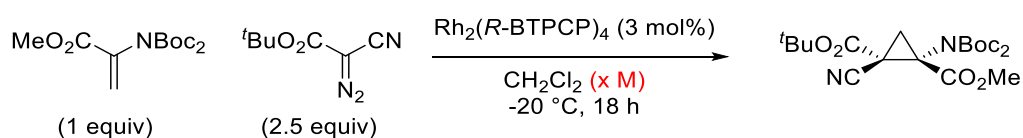
**Table S2.** Solvent evaluation



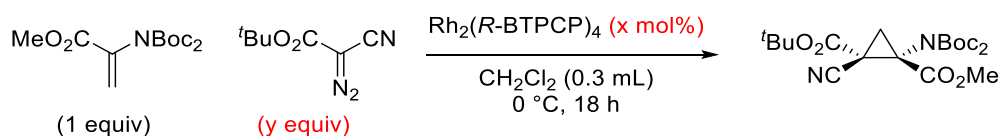
Entry	Solvent	NMR yield (%)	ee (%)
1	CH <sub>2</sub> Cl <sub>2</sub>	93	94
2	Toluene	/	/
3	MeCN	37	/
4	Pentane	63	90
5	Et <sub>2</sub> O	<5	/
6	CHCl <sub>3</sub>	41	/
7	Heptane	83	92

**Table S3.** Temperature evaluation

Entry	T ( $^\circ\text{C}$ )	NMR yield (%)	ee (%)
1	-20	36	/
2	0	93	94
3	25	96	95

**Table S4.** Concentration evaluation

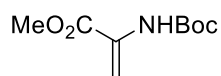
Entry	Alkene (M)	Diazo (M)	NMR yield (%)	ee (%)
1	0.17	0.42	26	/
2	0.33	0.83	36	/
3	0.66	1.67	14	/

**Table S5.** Reaction stoichiometry

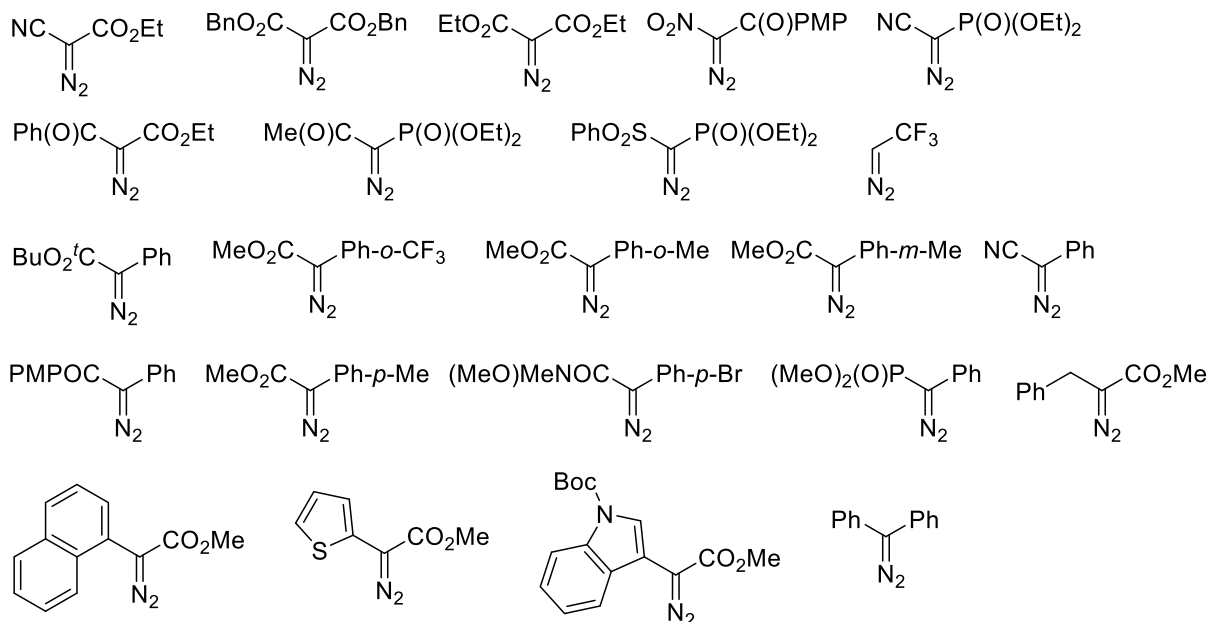
Entry	x;y	NMR yield (%)	ee (%)
1	3;2.5	93	94
2	2;2.5	94	96
3	1.5;2.5	93	94
4	1;2.5	83	/
5	3;2.0	84	/
6	3;1.5	69	/

**Table S6.** Unsuccessful partners in the cyclopropanation reaction

**Alkenes**



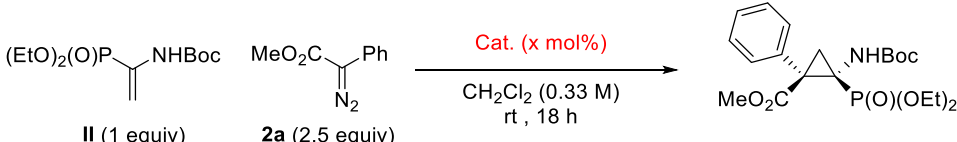
**Diazo reagents**



## b. $\alpha$ -amino phosphonates

Diethyl [1-(tertbutoxycarbonylamino)ethen-1-yl]phosphonate (**II**) and methyl 2-diazo-2-phenylacetate were initially utilized to find optimal conditions in terms of catalyst, solvent, temperature, concentration, and reaction stoichiometry (Table S6-11).

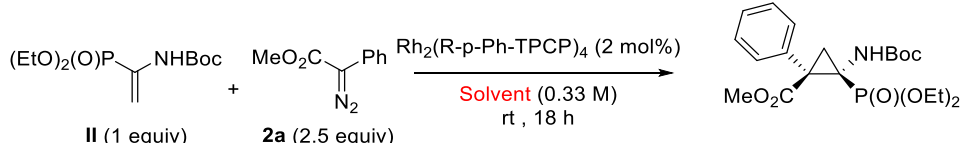
**Table S7.** Catalyst evaluation



Entry	Cat. (mol%)	Conv (%)	Yield. % <sup>a</sup> ( ) <sup>d</sup>	dr <sup>b</sup>	ee <sup>c</sup>
1	Rh <sub>2</sub> ( <i>R</i> -BTPCP) <sub>4</sub> (2)	92	57 (34)	88:12	26
2	Rh <sub>2</sub> ( <i>R</i> - <i>p</i> -Ph-TPCP) <sub>4</sub> (2)	89	67 (63)	99:1	85
3	Rh <sub>2</sub> ( <i>S</i> -PTTL) <sub>4</sub> (2)	55	27 (22)	87:13	8
4	Rh <sub>2</sub> ( <i>S</i> - <i>tert</i> -PTTL) <sub>4</sub> (2)	84	26 (30)	79:21	26
5	Rh <sub>2</sub> ( <i>S</i> -TBPTTL) <sub>4</sub> (2)	58	20	80:20	80
6	Rh <sub>2</sub> ( <i>S</i> -TCPTTL) <sub>4</sub> (2)	97	48 (36)	nd	71
7	Rh <sub>2</sub> ( <i>R</i> -PTAD) <sub>4</sub> (2)	72	53	89:11	30
8	Rh <sub>2</sub> ( <i>S</i> -TCPTAD) <sub>4</sub> (2)	100	58 (55)	nd	75
9	Ru(II)-( <i>S</i> )-Pheox	0	-	-	-

(a): <sup>1</sup>H NMR yield (triphenylmethane as IS), (b): determined by <sup>31</sup>P NMR, (c): determined by HPLC, (d): isolated yield

**Table S8.** Solvent evaluation



Entry	Solvent	Conv (%)	Yield. % <sup>a</sup> ( ) <sup>d</sup>	dr <sup>b</sup>	ee (%) <sup>c</sup>
1	DCM	89	67 (63)	99:1	85
2	CHCl <sub>3</sub>	50	6	50:50	nd
3	PhCH <sub>3</sub>	58	26	83:17	70
4	DCE	82	56 (47)	95:5	82
5	MeCN	52	30 (27)	96:4	87
6	THF	0	0	-	-
7	MeOH	0	0	-	-
8	EtOAc	75	38 (34)	88:12	80
9	Et <sub>2</sub> O	60	28 (nc)	90:10	73

(a): <sup>1</sup>H NMR yield (triphenylmethane as IS), (b): determined by <sup>31</sup>P NMR, (c): determined by HPLC, (d): isolated yield

**Table S9.** Temperature evaluation

Entry	T °C	Conv (%) <sup>a</sup>	Yield. % <sup>a</sup> (e)	dr <sup>b</sup>	ee (%) <sup>c</sup>
1	rt	89	67 (63)	99:1	85
2	0	94	56 (48)	n.d	84
3	-10	88	56 (56)	97:3	87
4 <sup>d</sup>	-20	89	60 (55)	96:4	86
5 <sup>d</sup>	-30	83	46 (45)	92:8	86

(a): <sup>1</sup>H NMR yield (triphenylmethane as IS), (b) : determined by <sup>31</sup>P NMR, (c): determined by HPLC, (d): addition in 5 h, (e) : isolated yield

**Table S10.** Concentration evaluation

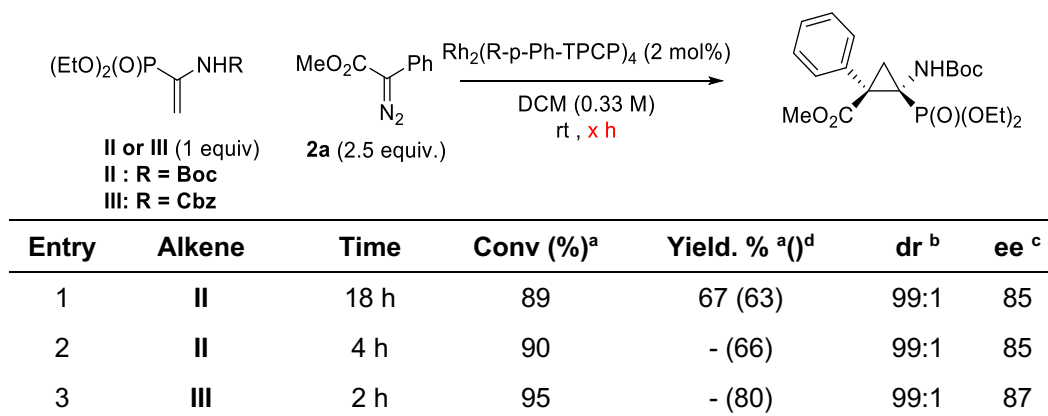
Entry	Conc (M)	Conv (%) <sup>a</sup>	Yield. % <sup>a</sup> (b)	dr <sup>c</sup>	ee <sup>d</sup>
1	0.33	89	67 (63)	99:1	85
2	0.2	89	51 (42)	93:7	74

(a): <sup>1</sup>H NMR yield (triphenylmethane), (b) : isolated yield, (c) : determined by <sup>31</sup>P NMR, (d): determined by HPLC

**Table S11.** Diazo equivalent

Entry	2a (x eq)	Conv (%) <sup>a</sup>	Yield. % <sup>a</sup> (d)	dr <sup>b</sup>	ee <sup>c</sup>
1	2.5	89	67 (63)	99:1	85
2	3	94	73 (66)	98:2	83
3	3.5	100	84 (75)	98:2	84

(a): <sup>1</sup>H NMR yield (triphenylmethane), (b) : determined by <sup>31</sup>P NMR, (c): determined by HPLC, (d) : isolated yield

**Table S12.** Time and alkene evaluation

(a): <sup>1</sup>H NMR yield (triphenylmethane), (b) : determined by <sup>31</sup>P NMR,  
(c): determined by HPLC, (d) : isolated yield



### 3. General procedure for cyclopropanation

#### General procedure A for the synthesis of racemic $\alpha$ -amino carboxylates.

In a dry microwave tube, the alkene (100.0  $\mu\text{mol}$ ) and  $\text{Rh}(\text{esp})_2$  (3.0  $\mu\text{mol}$ ) were dissolved in  $\text{CH}_2\text{Cl}_2$  (150.0  $\mu\text{L}$ ) under argon. The tube was inserted in an ice bath. The diazo (250.0  $\mu\text{mol}$ ) was then added as a solution in  $\text{CH}_2\text{Cl}_2$  (150.0  $\mu\text{L}$ ) over 1 h. The reaction mixture was stirred at 25  $^\circ\text{C}$  for 18 h. An aliquot was analyzed by  $^1\text{H}$  NMR. The mixture was concentrated and purified by column chromatography on silica gel (Pentane: $\text{Et}_2\text{O}$ ) to give the pure product.

#### General procedure B for the synthesis of $\alpha$ -amino carboxylates.

In a dry microwave tube, the alkene (100.0  $\mu\text{mol}$ ) and  $\text{Rh}_2(\text{R-BTPCP})_4$  (2.0  $\mu\text{mol}$ ) were dissolved in  $\text{CH}_2\text{Cl}_2$  (150.0  $\mu\text{L}$ ) under argon. The tube was inserted in a bath at 0  $^\circ\text{C}$ . The diazo (250.0  $\mu\text{mol}$ ) was then added as a solution in  $\text{CH}_2\text{Cl}_2$  (150.0  $\mu\text{L}$ ) over 5 h and stirred at 0  $^\circ\text{C}$  for 18 h. An aliquot was analyzed by  $^1\text{H}$  NMR. The mixture was concentrated and purified by column chromatography on silica gel (Pentane: $\text{Et}_2\text{O}$ ) to give the pure product.

#### General procedure C for the synthesis of racemic $\alpha$ -amino phosphonates.

In a dry microwave tube, the alkene (100.0  $\mu\text{mol}$ ) and  $\text{Rh}(\text{esp})_2$  (2.0  $\mu\text{mol}$ ) were dissolved in  $\text{CH}_2\text{Cl}_2$  (150.0  $\mu\text{L}$ ) under argon. The diazo (250.0  $\mu\text{mol}$ ) was then added as a solution in  $\text{CH}_2\text{Cl}_2$  (150.0  $\mu\text{L}$ ) over 10 minutes. The reaction mixture was stirred at 25  $^\circ\text{C}$  for 2 h. An aliquot was analyzed by  $^1\text{H}$  NMR. The mixture was concentrated and purified by column chromatography on silica gel (Petroleum ether: $\text{EtOAc}$ ) to give the pure product.

#### General procedure D for the synthesis of enantiopure $\alpha$ -amino phosphonates.

In a dry microwave tube, the alkene (100.0  $\mu\text{mol}$ ) and  $\text{Rh}_2(\text{R-p-PhTPCP})_4$  (2.0  $\mu\text{mol}$ ) were dissolved in  $\text{CH}_2\text{Cl}_2$  (150.0  $\mu\text{L}$ ) under argon. The diazo (250.0  $\mu\text{mol}$ ) was then added as a solution in  $\text{CH}_2\text{Cl}_2$  (150.0  $\mu\text{L}$ ) over 30 minutes and stirred at 25  $^\circ\text{C}$  for 1h30. An aliquot was analyzed by  $^1\text{H}$  NMR. The mixture was concentrated and purified by column chromatography on silica gel (Petroleum ether: $\text{EtOAc}$ ) to give the pure product.

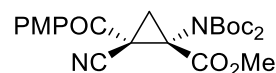
#### 4. Characterization of new compounds

**Compound 1** : 2-(tert-butyl 1-methyl (1*S*,2*R*)-1-(bis(tert-butoxycarbonyl)amino)-2-cyanocyclopropane-1,2-dicarboxylate



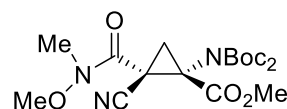
Compound **1** was prepared according to the general procedure B and obtained as a pale orange solid in 92% yield, 99:1 d.r., 98:2 e.r..  $^1\text{H NMR}$  (300 MHz,  $\text{CDCl}_3$ )  $\delta$  3.83 (s, 3H), 2.64 (d,  $J = 6.8$  Hz, 1H), 2.30 (d,  $J = 6.8$  Hz, 1H), 1.50 (s, 18H), 1.48 (s, 9H).  $^{13}\text{C NMR}$  (75 MHz,  $\text{CDCl}_3$ )  $\delta$  166.66 (s, 1C), 161.53 (s, 1C), 151.41 (s, 1C), 150.72 (s, 1C), 114.96 (s, 1C), 85.01 (s, 1C), 84.25 (s, 1C), 83.98 (s, 1C), 53.72 (s, 1C), 50.26 (s, 1C), 32.28 (s, 1C), 28.74 (s, 1C), 28.10 (s, 3C), 27.99 (s, 3C), 27.83 (s, 3C). **HR-MS** (APCI)  $m/z$  calcd for  $\text{C}_{21}\text{H}_{31}\text{N}_2\text{O}_8$   $[\text{M}-\text{H}]^-$  439.2080, found 439.2081. **IR** ( $\text{cm}^{-1}$ ) 2989, 2934, 2848, 2259, 1761, 1742, 1721, 1459, 1396, 1368, 1274, 1251, 1145, 1117, 963. **HPLC** Daicel Chiralpak IC-3, 150 mm,  $\varnothing$  4.6 mm, Heptane/iso-propanol = 90/10, 1.0 mL/min,  $\lambda = 220$  nm,  $t_{\text{major}} = 17.23$  min,  $t_{\text{minor}} = 14.12$  min.  $[\alpha]_{\text{D}}^{20} -2.1$  (c 1.00,  $\text{CHCl}_3$ ). **MP** 130-132 °C.

**Compound 2** : methyl (1*S*,2*R*)-1-(bis(tert-butoxycarbonyl)amino)-2-cyano-2-(4-methoxybenzoyl)cyclopropane-1-carboxylate



Compound **2** was prepared according to the general procedure B and obtained as a colorless oil in 20% yield, 99:1 d.r., 93.5:6.5 e.r..  $^1\text{H NMR}$  (300 MHz,  $\text{CDCl}_3$ )  $\delta$  8.36 – 7.77 (m, 2H), 7.04 – 6.74 (m, 2H), 3.89 (s, 3H), 3.87 (s, 3H), 2.78 (d,  $J = 6.3$  Hz, 1H), 2.60 (d,  $J = 6.3$  Hz, 1H), 1.50 (s, 9H), 1.25 (s, 9H).  $^{13}\text{C NMR}$  (75 MHz,  $\text{CDCl}_3$ )  $\delta$  184.97 (s, 1C), 166.61 (s, 1C), 163.77 (s, 1C), 151.63 (s, 2C), 132.95 (s, 2C), 127.99 (s, 1C), 117.65 (s, 1C), 113.06 (s, 2C), 83.93 (s, 2C), 55.50 (s, 1C), 53.59 (s, 1C), 52.14 (s, 1C), 33.47 (s, 1C), 28.92 (s, 1C), 27.70 (s, 6C). **HR-MS** (APCI)  $m/z$  calcd for  $\text{C}_{24}\text{H}_{30}\text{N}_2\text{O}_8$   $[\text{M}]$  474.2002, found 474.2007. **IR** ( $\text{cm}^{-1}$ ) 2982, 2919, 2850, 2244, 1743, 1712, 1601, 1512, 1366, 1249, 1171, 1117, 1022, 974. **HPLC** Daicel Chiralpak IC-3, 150 mm,  $\varnothing$  4.6 mm, Heptane/iso-propanol = 70/30, 1.0 mL/min,  $\lambda = 220$  nm,  $t_{\text{major}} = 7.11$  min,  $t_{\text{minor}} = 9.25$  min.  $[\alpha]_{\text{D}}^{20} -12.6$  (c 1.00,  $\text{CHCl}_3$ ).

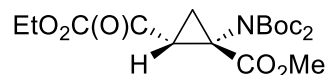
**Compound 3** : methyl (1*S*,2*R*)-1-(bis(tert-butoxycarbonyl)amino)-2-cyano-2-(methoxy(methyl)carbamoyl)cyclopropane-1-carboxylate



Compound **3** was prepared according to the general procedure B and obtained as a colorless oil in 37% yield, 99:1 d.r., 96:4 e.r..  $^1\text{H NMR}$  (300 MHz,  $\text{CDCl}_3$ )  $\delta$  3.85 (s, 3H), 3.83 (s, 3H), 3.22 (s, 3H), 2.59 (d,  $J = 6.5$  Hz, 1H), 2.55 (d,  $J = 6.5$  Hz, 1H), 1.48 (s, 18H).  $^{13}\text{C NMR}$  (75 MHz,  $\text{CDCl}_3$ )  $\delta$  166.60 (s, 1C), 151.66 (s, 2C), 115.83 (s, 1C), 83.82 (s, 2C), 61.20 (s, 1C), 53.64 (s, 1C), 50.31 (s, 1C), 28.77 (s, 1C), 28.61 (s, 1C), 28.03 (s, 6C),  $\text{CH}_3$  and  $\text{CON}$  not visible due to broad signals, see HSQC. **HR-MS** (APCI)  $m/z$  calcd for  $\text{C}_{19}\text{H}_{30}\text{N}_3\text{O}_8$   $[\text{M}+\text{H}]^+$  428.2033, found 428.2027. **IR** ( $\text{cm}^{-1}$ ) 3116, 2929, 2854, 2244, 1791, 1739, 1714, 1661, 1438,

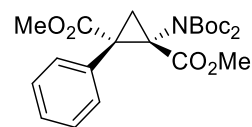
1366, 1299, 1255, 1213, 1153, 1090, 937. **HPLC** Daicel Chiralpak IC-3, 150 mm, Ø 4.6 mm, Heptane/iso-propanol = 70/30, 1.0 mL/min,  $\lambda = 220$  nm,  $t_{\text{major}} = 8.39$  min,  $t_{\text{minor}} = 13.83$  min.  $[\alpha]_{\text{D}}^{20} -27.3$  (c 1.00,  $\text{CHCl}_3$ ).

**Compound 4** : methyl (1*S*,2*R*)-1-(bis(tert-butoxycarbonyl)amino)-2-(2-ethoxy-2-oxoacetyl)cyclopropane-1-carboxylate



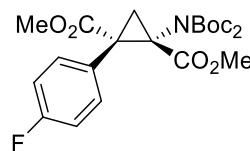
Compound **4** was prepared according to the general procedure B and obtained as a colorless oil in 22% yield, 99:1 d.r., 72:28 e.r..  $^1\text{H NMR}$  (300 MHz,  $\text{CDCl}_3$ )  $\delta$  4.48 – 4.22 (m, 2H), 3.96 (t,  $J = 8.3$  Hz, 1H), 3.74 (s, 3H), 2.14 (dd,  $J = 8.6, 5.7$  Hz, 1H), 1.98 (dd,  $J = 8.0, 5.7$  Hz, 1H), 1.51 (s, 9H), 1.42 (s, 9H), 1.36 (t,  $J = 7.1$  Hz, 3H).  $^{13}\text{C NMR}$  (75 MHz,  $\text{CDCl}_3$ )  $\delta$  185.88 (s, 1C), 169.90 (s, 1C), 160.68 (s, 1C), 152.89 (s, 1C), 152.42 (s, 1C), 83.79 (s, 1C), 83.44 (s, 1C), 62.68 (s, 1C), 53.30 (s, 1C), 47.57 (s, 1C), 33.91 (s, 1C), 28.11 (s, 3C), 27.99 (s, 3C), 23.91 (s, 1C), 14.12 (s, 1C). **HR-MS** (ESI)  $m/z$  calcd for  $\text{C}_{19}\text{H}_{29}\text{NO}_9\text{K}$   $[\text{M}+\text{K}]^+$  454.1479, found 454.1503. **IR** ( $\text{cm}^{-1}$ ) 2982, 2936, 1738, 1721, 1706, 1439, 1366, 1272, 1248, 1155, 1116, 1087, 1058, 982. **HPLC** Daicel Chiralpak IB-3, 150 mm, Ø 4.6 mm, Heptane/iso-propanol = 95/5, 1.0 mL/min,  $\lambda = 220$  nm,  $t_{\text{major}} = 6.24$  min,  $t_{\text{minor}} = 5.71$  min.  $[\alpha]_{\text{D}}^{20} +3.9$  (c 1.00,  $\text{CHCl}_3$ ).

**Compound 5** : dimethyl (1*S*,2*S*)-1-(bis(tert-butoxycarbonyl)amino)-2-phenylcyclopropane-1,2-dicarboxylate



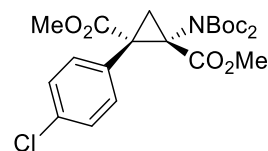
Compound **5** was prepared according to the general procedure B and obtained as a colorless oil in 78% yield, 99:1 d.r., 99:1 e.r..  $^1\text{H NMR}$  (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.47 (d,  $J = 6.4$  Hz, 2H), 7.38 – 7.17 (m, 3H), 3.59 (s, 3H), 3.27 (s, 3H), 2.57 (d,  $J = 6.0$  Hz, 1H), 2.25 (d,  $J = 6.0$  Hz, 1H), 1.56 (s, 9H), 1.51 (s, 9H).  $^{13}\text{C NMR}$  (75 MHz,  $\text{CDCl}_3$ )  $\delta$  169.84 (s, 1C), 168.30 (s, 1C), 152.98 (s, 1C), 152.60 (s, 1C), 135.83 (s, 1C), 130.28 (s, 2C), 128.02 (s, 2C), 127.72 (s, 1C), 83.01 (s, 2C), 53.10 (s, 1C), 52.47 (s, 1C), 50.44 (s, 1C), 45.10 (s, 1C), 28.18 (s, 3C), 28.15 (s, 3C), 26.95 (s, 1C). **HR-MS** (ESI)  $m/z$  calcd for  $\text{C}_{23}\text{H}_{31}\text{NO}_8\text{K}$   $[\text{M}+\text{K}]^+$  488.1687, found 488.1685. **IR** ( $\text{cm}^{-1}$ ) 2982, 2952, 1730, 1708, 1435, 1366, 1274, 1228, 1164, 1116, 1065, 1017, 956. **HPLC** Daicel Chiralpak IE-3, 150 mm, Ø 4.6 mm, Heptane/iso-propanol = 95/5, 1.0 mL/min,  $\lambda = 220$  nm,  $t_{\text{major}} = 11.92$  min,  $t_{\text{minor}} = 13.73$  min.  $[\alpha]_{\text{D}}^{20} -19.9$  (c 1.00,  $\text{CHCl}_3$ ).

**Compound 6:** dimethyl (1S,2S)-1-(bis(tert-butoxycarbonyl)amino)-2-(4-fluorophenyl)cyclopropane-1,2-dicarboxylate



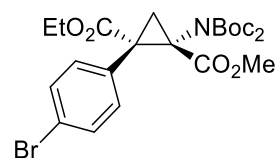
Compound **6** was prepared according to the general procedure B and obtained as a colorless oil in 77% yield, 99:1 d.r., 98:2 e.r..  $^1\text{H NMR}$  (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.45 (s, 2H), 6.98 (t,  $J = 8.8$  Hz, 2H), 3.59 (s, 3H), 3.32 (s, 3H), 2.54 (d,  $J = 6.1$  Hz, 1H), 2.25 (d,  $J = 6.1$  Hz, 1H), 1.55 (s, 9H), 1.51 (s, 9H).  $^{13}\text{C NMR}$  (75 MHz,  $\text{CDCl}_3$ )  $\delta$  169.64 (s, 1C), 168.18 (s, 1C), 162.28 (d,  $J = 246.2$  Hz, 1C), 153.05 (s, 1C), 152.49 (s, 1C), 131.98 (d,  $J = 8.1$  Hz, 2C), 131.62 (d,  $J = 3.3$  Hz, 1C), 114.97 (d,  $J = 21.5$  Hz, 2C), 83.09 (s, 2C), 53.12 (s, 1C), 52.61 (s, 1C), 50.45 (s, 1C), 44.35 (s, 1C), 28.16 (s, 6C), 27.02 (s, 1C).  $^{19}\text{F NMR}$  (282 MHz,  $\text{CDCl}_3$ )  $\delta$  -114.34 (s). **HR-MS** (ESI)  $m/z$  calcd for  $\text{C}_{23}\text{H}_{30}\text{NO}_8\text{NaF}$   $[\text{M}+\text{Na}]^+$  490.1853, found 490.1845. **IR** ( $\text{cm}^{-1}$ ) 2985, 1739, 1725, 1707, 1607, 1515, 1438, 1393, 1364, 1324, 1291, 1275, 1226, 1161, 1125, 1080, 1067, 1022, 968. **HPLC** Daicel Chiralpak IE-3, 150 mm,  $\varnothing$  4.6 mm, Heptane/iso-propanol = 95/5, 1.0 mL/min,  $\lambda = 220$  nm,  $t_{\text{major}} = 7.96$  min,  $t_{\text{minor}} = 8.49$  min.  $[\alpha]_{\text{D}}^{20}$  -27.6 (c 1.00,  $\text{CHCl}_3$ ).

**Compound 7:** dimethyl (1S,2S)-1-(bis(tert-butoxycarbonyl)amino)-2-(4-chlorophenyl)cyclopropane-1,2-dicarboxylate



Compound **7** was prepared according to the general procedure B and obtained as a colorless solid in 97% yield, 99:1 d.r., 97:3 e.r..  $^1\text{H NMR}$  (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.41 (d,  $J = 8.7$  Hz, 2H), 7.26 (d,  $J = 8.8$  Hz, 2H), 3.59 (s, 3H), 3.33 (s, 3H), 2.53 (d,  $J = 6.1$  Hz, 1H), 2.25 (d,  $J = 6.1$  Hz, 1H), 1.54 (s, 9H), 1.50 (s, 9H).  $^{13}\text{C NMR}$  (75 MHz,  $\text{CDCl}_3$ )  $\delta$  169.40 (s, 1C), 168.10 (s, 1C), 153.03 (s, 1C), 152.43 (s, 1C), 134.38 (s, 1C), 133.59 (s, 1C), 131.69 (s, 1C), 129.37 (s, 1C), 128.24 (s, 2C), 83.12 (s, 2C), 53.13 (s, 1C), 52.65 (s, 1C), 50.38 (s, 1C), 44.44 (s, 1C), 28.14 (s, 6C), 27.00 (s, 1C). **HR-MS** (ESI)  $m/z$  calcd for  $\text{C}_{23}\text{H}_{30}\text{NO}_8\text{NaCl}$   $[\text{M}+\text{Na}]^+$  506.1558, found 506.1552. **IR** ( $\text{cm}^{-1}$ ) 2981, 2953, 1731, 1707, 1494, 1435, 1366, 1275, 1227, 1164, 1116, 1091, 1013, 956. **HPLC** Daicel Chiralpak IE-3, 150 mm,  $\varnothing$  4.6 mm, Heptane/iso-propanol = 95/5, 1.0 mL/min,  $\lambda = 220$  nm,  $t_{\text{major}} = 9.58$  min,  $t_{\text{minor}} = 10.44$  min.  $[\alpha]_{\text{D}}^{20}$  -44.4 (c 1.00,  $\text{CHCl}_3$ ).

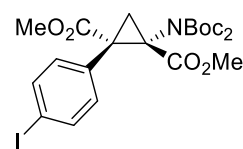
**Compound 8:** 2-ethyl 1-methyl (1S,2S)-1-(4-bromophenyl)-2-(bis(tert-butoxycarbonyl)amino)cyclopropane-1,2-dicarboxylate



Compound **8** was prepared according to the general procedure B and obtained as a colorless oil in 85% yield, 99:1 d.r., 99.5:0.5 e.r..  $^1\text{H NMR}$  (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.41 (d,  $J = 8.9$  Hz, 2H), 7.35 (d,  $J = 8.4$  Hz, 2H), 4.04 (q,  $J = 7.1$  Hz, 2H), 3.33 (s, 3H), 2.53 (d,  $J = 6.2$  Hz, 1H), 2.24 (d,  $J = 6.2$  Hz, 1H), 1.54 (s, 9H), 1.51 (s, 9H), 1.16 (t,  $J = 7.1$  Hz, 3H).  $^{13}\text{C NMR}$  (75 MHz,  $\text{CDCl}_3$ )  $\delta$  168.67 (s, 1C), 168.16 (s, 1C), 152.93 (s, 1C), 152.57 (s, 1C), 135.05 (s, 1C),

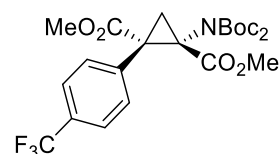
132.05 (s, 1C), 131.10 (s, 2C), 129.20 (s, 1C), 121.75 (s, 1C), 83.09 (s, 2C), 62.05 (s, 1C), 52.63 (s, 1C), 50.26 (s, 1C), 44.72 (s, 1C), 28.18 (s, 3C), 28.13 (s, 3C), 26.58 (s, 1C), 14.00 (s, 1C). **HR-MS** (ESI)  $m/z$  calcd for  $C_{24}H_{32}NO_8K$   $[M+K]^+$  580.0948, found 580.0952. **IR** ( $cm^{-1}$ ) 2981, 2932, 1728, 1709, 1489, 1365, 1275, 1223, 1166, 1116, 1065, 1009, 912. **HPLC** Daicel Chiralpak IE-3, 150 mm,  $\varnothing$  4.6 mm, Heptane/iso-propanol = 90/10, 1.0 mL/min,  $\lambda$  = 220 nm,  $t_{major}$  = 6.97 min,  $t_{minor}$  = 8.48 min.  $[\alpha]_D^{20}$  -47.4 (c 1.00,  $CHCl_3$ ).

**Compound 9: dimethyl (1S,2S)-1-(bis(tert-butoxycarbonyl)amino)-2-(4-iodophenyl)cyclopropane-1,2-dicarboxylate**



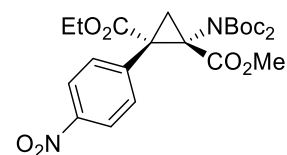
Compound **9** was prepared according to the general procedure B and obtained as a colorless solid in 96% yield, 99:1 d.r., 98:2 e.r..  **$^1H$  NMR** (300 MHz,  $CDCl_3$ )  $\delta$  7.62 (d,  $J$  = 8.6 Hz, 2H), 7.22 (d,  $J$  = 7.7 Hz, 2H), 3.59 (s, 3H), 3.34 (s, 3H), 2.52 (d,  $J$  = 6.1 Hz, 1H), 2.25 (d,  $J$  = 6.1 Hz, 1H), 1.54 (s, 9H), 1.51 (s, 9H).  **$^{13}C$  NMR** (75 MHz,  $CDCl_3$ )  $\delta$  169.28 (s, 1C), 168.08 (s, 1C), 153.00 (s, 1C), 152.42 (s, 1C), 138.30 (s, 1C), 137.16 (s, 2C), 135.59 (s, 1C), 132.28 (s, 1C), 129.43 (s, 1C), 83.13 (s, 2C), 53.15 (s, 1C), 52.67 (s, 1C), 50.31 (s, 1C), 44.63 (s, 1C), 28.14 (s, 6C), 26.94 (s, 1C). **HR-MS** (ESI)  $m/z$  calcd for  $C_{23}H_{30}NO_8NaI$   $[M+Na]^+$  598.0914, found 598.0888. **IR** ( $cm^{-1}$ ) 2977, 2951, 1731, 1695, 1484, 1436, 1363, 1241, 1221, 1158, 1120, 1080, 1005, 948. **HPLC** Daicel Chiralpak IE-3, 150 mm,  $\varnothing$  4.6 mm, Heptane/iso-propanol = 95/5, 1.0 mL/min,  $\lambda$  = 220 nm,  $t_{major}$  = 10.21 min,  $t_{minor}$  = 11.42 min.  $[\alpha]_D^{20}$  -40.0 (c 1.00,  $CHCl_3$ ).

**Compound 10: dimethyl (1S,2S)-1-(bis(tert-butoxycarbonyl)amino)-2-(4-(trifluoromethyl)phenyl)cyclopropane-1,2-dicarboxylate**



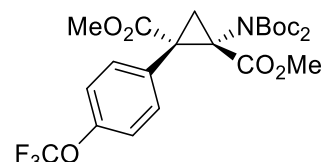
Compound **10** was prepared according to the general procedure B and obtained as a colorless oil in 52% yield, 99:1 d.r., 99.5:0.5 e.r..  $^1\text{H NMR}$  (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.64 – 7.52 (m, 4H), 3.60 (s, 3H), 3.30 (s, 3H), 2.58 (d,  $J = 6.2$  Hz, 1H), 2.30 (d,  $J = 6.2$  Hz, 1H), 1.55 (s, 9H), 1.51 (s, 9H).  $^{13}\text{C NMR}$  (75 MHz,  $\text{CDCl}_3$ )  $\delta$  169.09 (s, 1C), 168.00 (s, 1C), 153.06 (s, 1C), 152.39 (s, 1C), 139.92 (d,  $J = 1.3$  Hz, 1C), 130.75 (s, 2C), 129.82 (q,  $J = 32.3$  Hz, 1C), 124.94 (q,  $J = 3.7$  Hz, 2C), 124.28 (q,  $J = 272.1$  Hz, 1C), 122.48 (s, 1C), 83.23 (s, 2C), 53.18 (s, 1C), 52.61 (s, 1C), 50.32 (s, 1C), 44.69 (s, 1C), 28.14 (s, 6C), 27.00 (s, 1C).  $^{19}\text{F NMR}$  (282 MHz,  $\text{CDCl}_3$ )  $\delta$  -62.51 (s). **HR-MS** (ESI)  $m/z$  calcd for  $\text{C}_{24}\text{H}_{30}\text{NO}_8\text{F}_3\text{Na}$   $[\text{M}+\text{Na}]^+$  540.1821, found 540.1786. **IR** ( $\text{cm}^{-1}$ ) 2980, 2950, 1735, 1698, 1620, 1438, 1365, 1326, 1271, 1224, 1163, 1132, 1102, 1061, 1014, 949. **HPLC** Daicel Chiralpak IC-3, 150 mm,  $\text{O}$  4.6 mm, Heptane/iso-propanol = 90/10, 1.0 mL/min,  $\lambda = 220$  nm,  $t_{\text{major}} = 4.30$  min,  $t_{\text{minor}} = 8.41$  min.  $[\alpha]_{\text{D}}^{20}$  -29.6 ( $c$  1.00,  $\text{CHCl}_3$ ).

**Compound 11: 2-ethyl 1-methyl (1S,2S)-1-((tert-butoxycarbonyl)amino)-2-(4-nitrophenyl)cyclopropane-1,2-dicarboxylate**



Compound **11** was prepared according to the general procedure B and obtained as a colorless oil in 24% yield, 99:1 d.r., 99.5:0.5 e.r..  $^1\text{H NMR}$  (300 MHz,  $\text{CDCl}_3$ )  $\delta$  8.17 (d,  $J = 8.9$  Hz, 2H), 7.68 (d,  $J = 7.6$  Hz, 2H), 4.05 (q,  $J = 7.1$  Hz, 2H), 3.36 (s, 3H), 2.59 (d,  $J = 6.3$  Hz, 1H), 2.34 (d,  $J = 6.3$  Hz, 1H), 1.55 (s, 9H), 1.52 (s, 9H), 1.15 (t,  $J = 7.1$  Hz, 3H).  $^{13}\text{C NMR}$  (75 MHz,  $\text{CDCl}_3$ )  $\delta$  168.00 (s, 1C), 167.96 (s, 1C), 153.07 (s, 1C), 152.36 (s, 1C), 147.41 (s, 1C), 143.52 (s, 2C), 131.41 (s, 1C), 123.18 (s, 2C), 83.37 (s, 1C), 83.31 (s, 1C), 62.34 (s, 1C), 52.85 (s, 1C), 50.28 (s, 1C), 44.80 (s, 1C), 28.18 (s, 3C), 28.15 (s, 3C), 26.98 (s, 1C), 13.97 (s, 1C). **HR-MS** (ESI)  $m/z$  calcd for  $\text{C}_{24}\text{H}_{32}\text{N}_2\text{O}_{10}\text{Na}$   $[\text{M}+\text{Na}]^+$  531.1955, found 531.1946. **IR** ( $\text{cm}^{-1}$ ) 2982, 2933, 1729, 1708, 1602, 1522, 1366, 1347, 1274, 1224, 1163, 1116, 1066, 1010, 969. **HPLC** Daicel Chiralpak IE-3, 150 mm,  $\text{O}$  4.6 mm, Heptane/iso-propanol = 80/20, 1.0 mL/min,  $\lambda = 220$  nm,  $t_{\text{major}} = 7.50$  min,  $t_{\text{minor}} = 8.37$  min.  $[\alpha]_{\text{D}}^{20}$  -19.3 ( $c$  1.00,  $\text{CHCl}_3$ ).

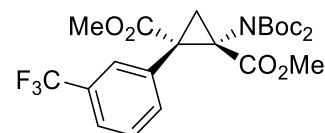
**Compound 12: dimethyl (1S,2S)-1-(bis(tert-butoxycarbonyl)amino)-2-(4-(trifluoromethoxy)phenyl)cyclopropane-1,2-dicarboxylate**



Compound **12** was prepared according to the general procedure B and obtained as a colorless oil in 75% yield, 99:1 d.r., 98.5:1.5 e.r..  $^1\text{H NMR}$  (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.52 (d,  $J = 7.9$  Hz, 2H), 7.14 (d,  $J = 8.0$  Hz, 2H), 3.60 (s, 3H), 3.27 (s, 3H), 2.55 (d,  $J = 6.2$  Hz, 1H), 2.27 (d,  $J = 6.2$  Hz, 1H), 1.55 (s, 9H), 1.50 (s, 9H).  $^{13}\text{C NMR}$  (75 MHz,  $\text{CDCl}_3$ )  $\delta$  169.36 (s, 1C), 168.06 (s, 1C), 153.05 (s, 1C), 152.45 (s, 1C), 148.64 (q,  $J = 1.7$  Hz, 1C), 134.62 (s,

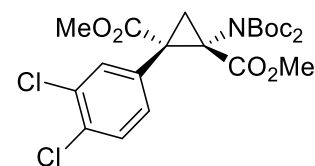
1C), 131.79 (s, 2C), 120.59 (q,  $J = 257.0$  Hz, 1C), 120.43 (s, 2C), 83.15 (s, 2C), 53.15 (s, 1C), 52.47 (s, 1C), 50.41 (s, 1C), 44.28 (s, 1C), 28.13 (s, 6C), 26.87 (s, 1C).  **$^{19}\text{F}$  NMR** (282 MHz,  $\text{CDCl}_3$ )  $\delta$  -57.84 (s). **HR-MS** (ESI)  $m/z$  calcd for  $\text{C}_{24}\text{H}_{30}\text{NO}_9\text{F}_3\text{K}$   $[\text{M}+\text{K}]^+$  572.1510, found 572.1489. **IR** ( $\text{cm}^{-1}$ ) 2983, 2957, 1734, 1708, 1511, 1436, 1367, 1252, 1222, 1160, 1116, 1068, 1016, 958. **HPLC** Daicel Chiralpak IC-3, 150 mm,  $\text{O}$  4.6 mm, Heptane/iso-propanol = 90/10, 1.0 mL/min,  $\lambda = 220$  nm,  $t_{\text{major}} = 4.16$  min,  $t_{\text{minor}} = 8.50$  min.  $[\alpha]_{\text{D}}^{20}$  -18.1 ( $c$  1.00,  $\text{CHCl}_3$ ).

**Compound 13 : dimethyl (1S,2S)-1-(bis(tert-butoxycarbonyl)amino)-2-(3-(trifluoromethyl)phenyl)cyclopropane-1,2-dicarboxylate**



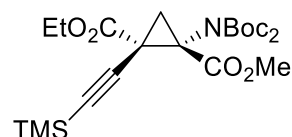
Compound **13** was prepared according to the general procedure B and obtained as a colorless oil in 81% yield, 99:1 d.r., 97.5:2.5 e.r..  **$^1\text{H}$  NMR** (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.69 (d,  $J = 9.9$  Hz, 2H), 7.53 (d,  $J = 7.8$  Hz, 1H), 7.43 (t,  $J = 7.7$  Hz, 1H), 3.60 (s, 3H), 3.31 (s, 3H), 2.57 (d,  $J = 6.2$  Hz, 1H), 2.30 (d,  $J = 6.2$  Hz, 1H), 1.56 (s, 9H), 1.51 (s, 9H).  **$^{13}\text{C}$  NMR** (75 MHz,  $\text{CDCl}_3$ )  $\delta$  168.98 (s, 1C), 167.85 (s, 1C), 152.75 (s, 1C), 152.22 (s, 1C), 136.87 (s, 1C), 133.89 (d,  $J = 2.1$  Hz, 1C), 130.25 (q,  $J = 32.3$  Hz, 1C), 128.38 (s, 1C), 126.81 (dd,  $J = 5.8, 3.3$  Hz, 1C), 124.45 (s, q,  $J = 3.8$  Hz, 1C), 124.09 (q,  $J = 272.1$  Hz, 1C), 83.17 (s, 1C), 83.06 (s, 1C), 53.06 (s, 1C), 52.50 (s, 1C), 50.20 (s, 1C), 44.59 (s, 1C), 28.01 (s, 6C), 26.92 (s, 1C).  **$^{19}\text{F}$  NMR** (282 MHz,  $\text{CDCl}_3$ )  $\delta$  -62.43 (s). **HR-MS** (ESI)  $m/z$  calcd for  $\text{C}_{24}\text{H}_{30}\text{NO}_8\text{F}_3\text{K}$   $[\text{M}+\text{K}]^+$  556.1561, found 556.1548. **IR** ( $\text{cm}^{-1}$ ) 2983, 1733, 1709, 1436, 1367, 1325, 1273, 1227, 1163, 1116, 1069, 958. **HPLC** Daicel Chiralpak IE-3, 150 mm,  $\text{O}$  4.6 mm, Heptane/iso-propanol = 95/5, 1.0 mL/min,  $\lambda = 220$  nm,  $t_{\text{major}} = 7.21$  min,  $t_{\text{minor}} = 7.84$  min.  $[\alpha]_{\text{D}}^{20}$  -30.9 ( $c$  1.00,  $\text{CHCl}_3$ ).

**Compound 14 : dimethyl (1S,2S)-1-(bis(tert-butoxycarbonyl)amino)-2-(3,4-dichlorophenyl)cyclopropane-1,2-dicarboxylate**



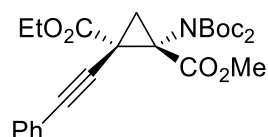
Compound **14** was prepared according to the general procedure B and obtained as a colorless oil in 71% yield, 99:1 d.r., 98:2 e.r..  **$^1\text{H}$  NMR** (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.54 (s, 1H), 7.36 (s, 2H), 3.60 (s, 3H), 3.40 (s, 3H), 2.52 (d,  $J = 6.2$  Hz, 1H), 2.26 (d,  $J = 6.2$  Hz, 1H), 1.55 (s, 9H), 1.51 (s, 9H).  **$^{13}\text{C}$  NMR** (75 MHz,  $\text{CDCl}_3$ )  $\delta$  168.94 (s, 1C), 167.98 (s, 1C), 152.96 (s, 1C), 152.28 (s, 1C), 136.15 (s, 1C), 132.16 (s, 1C), 132.01 (s, 1C), 131.90 (s, 1C), 130.02 (s, 1C), 129.91 (s, 1C), 83.32 (s, 1C), 83.25 (s, 1C), 53.25 (s, 1C), 52.86 (s, 1C), 50.31 (s, 1C), 44.16 (s, 1C), 28.16 (s, 6C), 27.22 (s, 1C). **HR-MS** (ESI)  $m/z$  calcd for  $\text{C}_{23}\text{H}_{29}\text{NO}_8\text{Cl}_2\text{Na}$   $[\text{M}+\text{Na}]^+$  540.1168, found 540.1148. **IR** ( $\text{cm}^{-1}$ ) 2982, 2954, 1732, 1708, 1473, 1435, 1366, 1275, 1227, 1165, 1116, 1067, 1032, 957. **HPLC** Daicel Chiralpak IE-3, 150 mm,  $\text{O}$  4.6 mm, Heptane/iso-propanol = 95/5, 1.0 mL/min,  $\lambda = 220$  nm,  $t_{\text{major}} = 7.96$  min,  $t_{\text{minor}} = 8.98$  min.  $[\alpha]_{\text{D}}^{20}$  -58.8 ( $c$  1.00,  $\text{CHCl}_3$ ).

**Compound 15**: 2-ethyl 1-methyl (1*S*,2*S*)-1-(bis(tert-butoxycarbonyl)amino)-2-((trimethylsilyl)ethynyl)cyclopropane-1,2-dicarboxylate



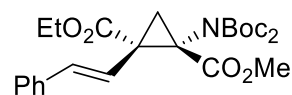
Compound **15** was prepared according to the general procedure B and obtained as a colorless oil in 68% yield, 99:1 d.r., 93:7 e.r..  $^1\text{H NMR}$  (300 MHz,  $\text{CDCl}_3$ )  $\delta$  4.40 – 3.96 (m, 2H), 3.75 (s, 3H), 2.54 (d,  $J = 6.2$  Hz, 1H), 2.15 (d,  $J = 6.2$  Hz, 1H), 1.48 (s, 9H), 1.47 (s, 9H), 1.29 (t,  $J = 7.2$  Hz, 3H), 0.14 (s, 9H).  $^{13}\text{C NMR}$  (75 MHz,  $\text{CDCl}_3$ )  $\delta$  167.18 (s, 1C), 166.67 (s, 1C), 151.76 (s, 1C), 150.85 (s, 1C), 99.43 (s, 1C), 89.27 (s, 1C), 83.34 (s, 1C), 83.11 (s, 1C), 62.49 (s, 1C), 52.70 (s, 1C), 51.95 (s, 1C), 34.65 (s, 1C), 30.00 (s, 1C), 28.12 (s, 3C), 27.93 (s, 3C), 13.96 (s, 1C), -0.06 (s, 3C). **HR-MS** (ESI)  $m/z$  calcd for  $\text{C}_{23}\text{H}_{37}\text{NO}_8\text{SiNa}$   $[\text{M}+\text{Na}]^+$  506.2186, found 506.2178. **IR** ( $\text{cm}^{-1}$ ) 2981, 2258, 2179, 1798, 1732, 1457, 1368, 1277, 1249, 1213, 1156, 1121, 1097, 1066, 977. **HPLC** Daicel Chiralpak IE-3, 150 mm,  $\varnothing$  4.6 mm, Heptane/iso-propanol = 90/10, 1.0 mL/min,  $\lambda = 220$  nm,  $t_{\text{major}} = 9.10$  min,  $t_{\text{minor}} = 8.59$  min.  $[\alpha]_{\text{D}}^{20} -17.4$  (c 1.00,  $\text{CHCl}_3$ ).

**Compound 16**: 2-ethyl 1-methyl (1*S*,2*S*)-1-(bis(tert-butoxycarbonyl)amino)-2-(phenylethynyl)cyclopropane-1,2-dicarboxylate



Compound **16** was prepared according to the general procedure B and obtained as a colorless oil in 31% yield, 99:1 d.r., 98:2 e.r..  $^1\text{H NMR}$  (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.41 (dd,  $J = 6.7$ , 3.0 Hz, 2H), 7.28 (dd,  $J = 4.9$ , 1.8 Hz, 3H), 4.35 – 4.07 (m, 2H), 3.76 (s, 3H), 2.62 (d,  $J = 6.2$  Hz, 1H), 2.27 (d,  $J = 6.2$  Hz, 1H), 1.51 (s, 9H), 1.50 (s, 9H), 1.33 (t,  $J = 7.2$  Hz, 3H).  $^{13}\text{C NMR}$  (75 MHz,  $\text{CDCl}_3$ )  $\delta$  167.41 (s, 1C), 166.84 (s, 1C), 151.87 (s, 1C), 151.12 (s, 1C), 131.85 (s, 2C), 128.32 (s, 3C), 123.16 (s, 1C), 84.10 (s, 1C), 83.72 (s, 1C), 83.39 (s, 1C), 83.22 (s, 1C), 62.59 (s, 1C), 53.03 (s, 1C), 51.95 (s, 1C), 34.65 (s, 1C), 30.25 (s, 1C), 28.15 (s, 3C), 27.96 (s, 3C), 14.10 (s, 1C). **HR-MS** (ESI)  $m/z$  calcd for  $\text{C}_{26}\text{H}_{33}\text{NO}_8\text{K}$   $[\text{M}+\text{K}]^+$  526.1843, found 526.1843. **IR** ( $\text{cm}^{-1}$ ) 2981, 2936, 1797, 1732, 1443, 1368, 1275, 1234, 1154, 1119, 1097, 1065, 973. **HPLC** Daicel Chiralpak IE-3, 150 mm,  $\varnothing$  4.6 mm, Heptane/iso-propanol = 90/10, 1.0 mL/min,  $\lambda = 220$  nm,  $t_{\text{major}} = 12.91$  min,  $t_{\text{minor}} = 12.20$  min.  $[\alpha]_{\text{D}}^{20} -9.4$  (c 1.00,  $\text{CHCl}_3$ ).

**Compound 17**: 2-ethyl 1-methyl (1*S*,2*S*)-1-(bis(tert-butoxycarbonyl)amino)-2-((*E*)-styryl)cyclopropane-1,2-dicarboxylate

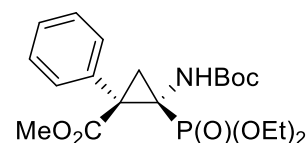


Compound **17** was prepared according to the general procedure B and obtained as a colorless oil in 55% yield, 99:1 d.r., 97:3 e.r..  $^1\text{H NMR}$  (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.39 (dd,  $J = 8.3$ , 1.4 Hz, 2H), 7.35 – 7.22 (m, 3H), 6.53 (s, 2H), 4.14 (ddd,  $J = 25.7$ , 10.8, 7.1 Hz, 2H), 3.60 (s, 3H), 2.55 (d,  $J = 6.5$  Hz, 1H), 2.17 (d,  $J = 6.5$  Hz, 1H), 1.52 (s, 18H), 1.27 (t,  $J = 7.1$  Hz, 3H).  $^{13}\text{C NMR}$  (75 MHz,  $\text{CDCl}_3$ )  $\delta$  168.88 (s, 1C), 167.73 (s, 1C), 152.14 (s, 1C), 151.93 (s, 1C),



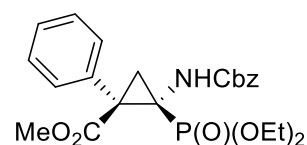
136.86 (s, 1C), 134.51 (s, 1C), 128.65 (s, 2C), 127.88 (s, 1C), 126.72 (s, 2C), 121.55 (s, 1C), 83.06 (s, 1C), 82.92 (s, 1C), 61.91 (s, 1C), 52.74 (s, 1C), 50.37 (s, 1C), 43.43 (s, 1C), 28.15 (s, 3C), 28.06 (s, 3C), 25.77 (s, 1C), 14.12 (s, 1C). **HR-MS** (ESI)  $m/z$  calcd for  $C_{26}H_{35}NO_8K$   $[M+K]^+$  528.2000, found 528.2014. **IR** ( $cm^{-1}$ ) 2969, 1794, 1741, 1717, 1446, 1368, 1257, 1154, 1092, 1021, 975. **HPLC** Daicel Chiralpak IC-3, 150 mm,  $\varnothing$  4.6 mm, Heptane/iso-propanol = 70/30, 1.0 mL/min,  $\lambda$  = 220 nm,  $t_{major}$  = 4.77 min,  $t_{minor}$  = 16.58 min.  $[\alpha]_D^{20}$  -12.5 (c 1.00,  $CHCl_3$ ).

**Compound 18** : methyl (1*S*,2*R*)-2-((tert-butoxycarbonyl)amino)-2-(diethoxyphosphoryl)-1-phenyl cyclopropane-1-carboxylate



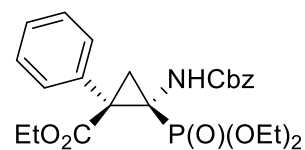
Compound **18** was prepared according to the general procedure D and obtained as an amorphous solid in 63% yield, 92.5:7.5 e.r..  **$^1H$  NMR** (300 MHz,  $CDCl_3$ )  $\delta$  7.50 – 7.42 (m, 2H), 7.41 – 7.28 (m, 3H), 4.43 (s, 1H), 4.33 – 4.12 (m, 4H), 3.69 (s, 3H), 2.52 (ddd,  $J$  = 14.1, 6.9, 0.9 Hz, 1H), 2.29 (t,  $J$  = 6.9 Hz, 1H), 1.44 – 1.37 (m, 3H), 1.35 – 1.23 (m, 12H).  **$^{13}C$  NMR** (75 MHz,  $CDCl_3$ )  $\delta$  169.54 (1C, d,  $J$  = 4.0 Hz), 154.42 (1C, s), 133.72 (1C, s), 130.14 (2C, s), 128.83 (2C, s), 128.34 (1C, s), 80.04 (1C, s), 63.34 (1C, d,  $J$  = 3.8 Hz), 63.03 (1C, d,  $J$  = 6.1 Hz), 52.92 (1C, s), 42.07 (1C, s), 36.69 (1C, d,  $J$  = 217.4 Hz), 28.21 (3C, s), 21.41 (1C, s), 16.69 (1C, d,  $J$  = 6.0 Hz), 16.47 (1C, d,  $J$  = 6.6 Hz).  **$^{31}P$  NMR** (121 MHz,  $CDCl_3$ )  $\delta$  21.17. **HR-MS** (ESI+)  $m/z$  calcd for  $C_{20}H_{30}NO_7PNa$   $[M+Na]^+$  450.1658. found 450.1654. **IR** ( $cm^{-1}$ ) 2983, 1728, 1498, 1363, 1240, 1160, 1051, 1022, 977. **HPLC** Daicel Chiralpak IE, 150 mm,  $\varnothing$  4.6 mm, Heptane/iso-propanol = 80/20, 1.0 mL/min,  $\lambda$  = 210 nm,  $t_{minor}$  = 11.80 min,  $t_{major}$  = 14.54 min.  $[\alpha]_D^{20}$  +24.6 (c 1.00,  $CH_2Cl_2$ ).

**Compound 19** : methyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-phenylcyclopropane-1-carboxylate



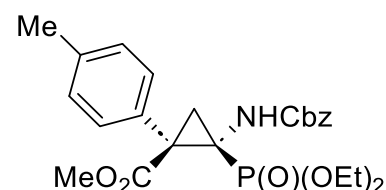
Compound **19** was prepared according to the general procedure D and obtained as an amorphous solid in 80% yield, 93.5:6.5 e.r..  **$^1H$  NMR** (400 MHz,  $CDCl_3$ )  $\delta$  7.50 – 7.42 (m, 2H), 7.38 – 7.32 (m, 4H), 7.31 – 7.27 (m, 2H), 7.17 – 7.04 (m, 2H), 4.91 (dd,  $J$  = 41.2, 12.4 Hz, 2H), 4.71 (s, 1H), 4.22 (tq,  $J$  = 14.1, 7.0 Hz, 4H), 3.70 (s, 3H), 2.55 (dd,  $J$  = 13.8, 6.9 Hz, 1H), 2.28 (t,  $J$  = 6.8 Hz, 1H), 1.39 (t,  $J$  = 7.1 Hz, 3H), 1.28 (t,  $J$  = 7.1 Hz, 4H).  **$^{13}C$  NMR** (101 MHz,  $CDCl_3$ )  $\delta$  169.32 (1C, s), 154.94 (1C, s), 136.27 (1C, s), 133.52 (1C, s), 130.16 (2C, s), 129.01 (2C, s), 128.56 (2C, s), 128.53 (1C, s), 128.18 (1C, s), 127.74 (2C, s), 66.72 (1C, s), 63.49 (1C, d,  $J$  = 5.3 Hz), 63.08 (1C, d,  $J$  = 6.4 Hz), 52.99 (1C, s), 42.23 (1C, s), 36.61 (1C, d,  $J$  = 218.7 Hz), 21.47 (1C, s), 16.67 (1C, d,  $J$  = 6.1 Hz), 16.46 (1C, d,  $J$  = 6.3 Hz).  **$^{31}P$  NMR** (162 MHz,  $CDCl_3$ )  $\delta$  20.62. **HR-MS** (ESI+)  $m/z$  calcd for  $C_{23}H_{29}NO_7P$   $[M+H]^+$  462.1676. found 462.1678. **IR** ( $cm^{-1}$ ) 2983, 1730, 1495, 1450, 1303, 1222, 1172, 1057, 1020, 972, 746. **HPLC** Daicel Chiralpak IB, 250 mm,  $\varnothing$  4.6 mm, Heptane/iso-propanol = 90/10, 1.0 mL/min,  $\lambda$  = 210 nm,  $t_{minor}$  = 8.21 min,  $t_{major}$  = 8.95 min.  $[\alpha]_D^{20}$  +20.1 (c 1.00,  $CHCl_3$ ).

**Compound 20**: ethyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-phenyl cyclopropane-1-carboxylate



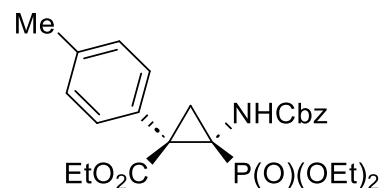
Compound **20** was prepared according to the general procedure D and obtained as an amorphous solid in 41% yield, 89.5:10.5 e.r.. **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.50 – 7.40 (m, 2H), 7.39 – 7.31 (m, 4H), 7.30 – 7.26 (m, 2H), 7.18 – 7.02 (m, 2H), 4.91 (dd, *J* = 40.8, 12.4 Hz, 2H), 4.70 (s, 1H), 4.26 – 4.17 (m, 4H), 4.13 (dd, *J* = 14.2, 7.1 Hz, 2H), 2.52 (dd, *J* = 13.8, 6.9 Hz, 1H), 2.28 (t, *J* = 6.7 Hz, 1H), 1.38 (t, *J* = 7.1 Hz, 3H), 1.26 (dt, *J* = 14.2, 7.1 Hz, 6H). **<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 168.79 (1C, s), 154.95 (1C, s), 136.31 (1C, s), 133.69 (1C, s), 130.14 (2C, s), 128.97 (2C, s), 128.56 (2C, s), 128.45 (1C, s), 128.16 (1C, s), 127.71 (2C, s), 66.68 (1C, s), 63.44 (1C, d, *J* = 5.1 Hz), 63.01 (1C, d, *J* = 6.3 Hz), 62.01 (1C, s), 42.44 (1C, s), 36.59 (1C, d, *J* = 218.7 Hz), 21.40 (1C, s), 16.65 (1C, d, *J* = 6.1 Hz), 16.45 (1C, d, *J* = 6.4 Hz), 14.00 (1C, s). **<sup>31</sup>P NMR** (162 MHz, CDCl<sub>3</sub>) δ 20.63. **HR-MS** (ESI+) *m/z* calcd for C<sub>24</sub>H<sub>31</sub>NO<sub>7</sub>P [M+H]<sup>+</sup> 476.1833. found 476.1833. **IR** (cm<sup>-1</sup>) 2991, 2364, 1728, 1519, 1294, 1224, 1188, 1051, 1022, 970. **HPLC** Daicel Chiralpak IB, 250 mm, Ø 4.6 mm, Heptane/Ethanol = 95/5, 1.0 mL/min, λ = 210 nm, *t*<sub>minor</sub> = 7.22 min, *t*<sub>major</sub> = 7.68 min. [α]<sub>D</sub><sup>20</sup> +18.3 (*c* 1.00, CH<sub>2</sub>Cl<sub>2</sub>).

**Compound 21**: methyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-(*p*-tolyl) cyclopropane-1-carboxylate



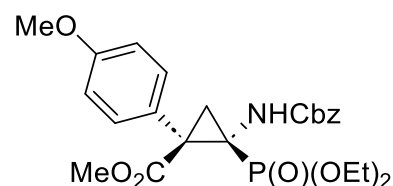
Compound **21** was prepared according to the general procedure D and obtained as an amorphous solid in 72% yield, 95:5 e.r.. **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.35 – 7.26 (m, 5H), 7.20 – 7.04 (m, 4H), 4.92 (dd, *J* = 40.7, 12.4 Hz, 2H), 4.70 (s, 1H), 4.28 – 4.12 (m, 4H), 3.68 (s, 3H), 2.52 (dd, *J* = 13.9, 6.9 Hz, 1H), 2.33 (s, 3H), 2.25 (t, *J* = 6.6 Hz, 1H), 1.38 (t, *J* = 7.1 Hz, 3H), 1.28 (t, *J* = 7.1 Hz, 3H). **<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 169.44 (1C, s), 154.97 (1C, s), 138.33 (1C, s), 136.32 (1C, s), 130.41 (1C, s), 129.96 (2C, s), 129.72 (2C, s), 128.53 (2C, s), 128.15 (1C, s), 127.77 (2C, s), 66.67 (1C, s), 63.44 (1C, d, *J* = 4.4 Hz), 63.04 (1C, d, *J* = 6.4 Hz), 52.92 (1C, s), 41.93 (1C, s), 36.55 (1C, d, *J* = 217.9 Hz), 21.51 (1C, s), 21.28 (1C, s), 16.65 (1C, d, *J* = 6.0 Hz), 16.44 (1C, d, *J* = 6.4 Hz). **<sup>31</sup>P NMR** (162 MHz, CDCl<sub>3</sub>) δ 20.75. **HR-MS** (ESI+) *m/z* calcd for C<sub>24</sub>H<sub>31</sub>NO<sub>7</sub>P [M+H]<sup>+</sup> 476.1833. found 476.1831. **IR** (cm<sup>-1</sup>) 2987, 2364, 1737, 1512, 1429, 1224, 1170, 1060, 1028, 970. **HPLC** Daicel Chiralpak IA, 250 mm, Ø 4.6 mm, Heptane/Ethanol = 95/5, 1.0 mL/min, λ = 210 nm, *t*<sub>minor</sub> = 15.48 min, *t*<sub>major</sub> = 18.16 min. [α]<sub>D</sub><sup>20</sup> +28.7 (*c* 1.00, CH<sub>2</sub>Cl<sub>2</sub>).

**Compound 22**: ethyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-(*p*-tolyl) cyclopropane-1-carboxylate



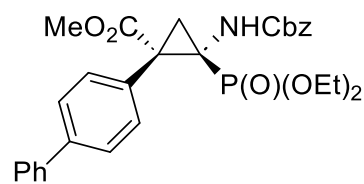
Compound **22** was prepared according to the general procedure D and obtained as an amorphous solid in 48% yield, 97:3 e.r.. **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.38 – 7.27 (m, 5H), 7.20 – 7.05 (m, 4H), 4.92 (dd, *J* = 39.3, 12.5 Hz, 2H), 4.69 (s, 1H), 4.27 – 4.16 (m, 4H), 4.12 (q, *J* = 7.2 Hz, 2H), 2.50 (dd, *J* = 13.8, 6.8 Hz, 1H), 2.34 (s, 3H), 2.24 (t, *J* = 6.5 Hz, 1H), 1.37 (t, *J* = 7.1 Hz, 4H), 1.26 (dt, *J* = 14.2, 7.0 Hz, 6H). **<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 168.81 (1C, s), 154.86 (1C, s), 138.13 (1C, s), 136.24 (1C, s), 130.46 (1C, s), 129.81 (2C, s), 129.57 (2C, s), 128.41 (2C, s), 128.03 (1C, s), 127.60 (2C, s), 66.52 (1C, s), 63.30 (1C, s), 62.86 (1C, d, *J* = 6.1 Hz), 61.81 (1C, s), 42.03 (1C, s), 36.41 (1C, d, *J* = 219.6 Hz), 21.32 (1C, s), 21.17 (1C, s), 16.52 (1C, d, *J* = 6.2 Hz), 16.32 (1C, d, *J* = 6.3 Hz), 13.88 (1C, s). **<sup>31</sup>P NMR** (162 MHz, CDCl<sub>3</sub>) δ 20.78. **HR-MS** (ESI+) *m/z* calcd for C<sub>25</sub>H<sub>33</sub>NO<sub>7</sub>P [M+H]<sup>+</sup> 490.1989. found 490.1989. **IR** (cm<sup>-1</sup>) 2926, 2357, 1737, 1519, 1462, 1301, 1228, 1097, 1060, 1018, 970. **HPLC** Daicel Chiralpak IB, 250 mm, Ø 4.6 mm, Heptane/Ethanol = 98/2, 1.0 mL/min, λ = 210 nm, *t*<sub>minor</sub> = 14.52 min, *t*<sub>major</sub> = 15.36 min. [α]<sub>D</sub><sup>20</sup> +28.5 (c 1.00, CH<sub>2</sub>Cl<sub>2</sub>).

**Compound 23**: methyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-(4-methoxyphenyl)cyclopropane-1-carboxylate



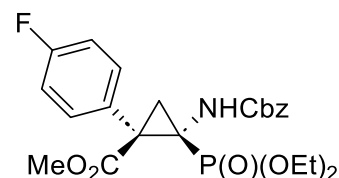
Compound **23** was prepared according to the general procedure D and obtained as an amorphous solid in 85% yield, 91:9 e.r.. **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.45 – 7.31 (m, 2H), 7.33 – 7.22 (m, 3H), 7.19 – 7.08 (m, 2H), 6.90 – 6.83 (m, 2H), 4.92 (dd, *J* = 41.3, 12.4 Hz, 2H), 4.71 (s, 1H), 4.28 – 4.13 (m, 4H), 3.79 (s, 3H), 3.69 (s, 3H), 2.51 (dd, *J* = 13.9, 6.9 Hz, 1H), 2.23 (t, *J* = 6.6 Hz, 1H), 1.38 (t, *J* = 7.1 Hz, 3H), 1.28 (t, *J* = 7.0 Hz, 3H). **<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 169.45 (1C, d, *J* = 4.2 Hz), 159.60 (1C, s), 154.88 (1C, s), 136.18 (1C, s), 131.18 (2C, s), 128.44 (2C, s), 128.07 (1C, s), 127.66 (2C, s), 125.24 (1C, s), 114.28 (2C, s), 66.59 (1C, s), 63.31 (1C, d, *J* = 4.5 Hz), 62.93 (1C, d, *J* = 6.4 Hz), 55.29 (1C, s), 52.79 (1C, s), 42.03 – 41.50 (1C, m), 36.54 (1C, d, *J* = 218.3 Hz), 21.45 (1C, s), 16.53 (1C, d, *J* = 6.1 Hz), 16.33 (1C, d, *J* = 6.4 Hz). **<sup>31</sup>P NMR** (162 MHz, CDCl<sub>3</sub>) δ 20.79. **HR-MS** (ESI+) *m/z* calcd for C<sub>24</sub>H<sub>31</sub>NO<sub>8</sub>P [M+H]<sup>+</sup> 492.1782 found 492.1783. **IR** (cm<sup>-1</sup>) 2991, 2360, 1734, 1516, 1446, 1429, 1247, 1224, 1171, 1056, 1022. **HPLC** Daicel Chiralpak IA, 250 mm, Ø 4.6 mm, Heptane/iso-propanol = 80/20, 1.0 mL/min, λ = 210 nm, *t*<sub>minor</sub> = 8.75 min, *t*<sub>major</sub> = 7.89 min. [α]<sub>D</sub><sup>20</sup> +31.4 (c 1.00, CH<sub>2</sub>Cl<sub>2</sub>).

**Compound 24** : methyl (1*R*,2*S*)-1-([1,1'-biphenyl]-4-yl)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)cyclopropane-1-carboxylate



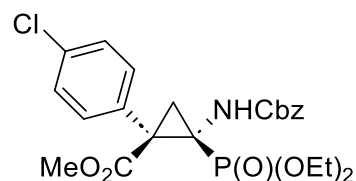
Compound **24** was prepared according to the general procedure D using  $\text{Rh}_2((S)\text{-p-PhTPCP})_4$  and obtained as an amorphous solid in 57% yield, 3:97 e.r..  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.60 – 7.55 (m, 4H), 7.54 – 7.48 (m, 2H), 7.48 – 7.43 (m, 2H), 7.39 – 7.33 (m, 2H), 7.25 – 7.18 (m, 2H), 7.17 – 7.08 (m, 2H), 4.92 (dd,  $J = 39.2, 12.4$  Hz, 2H), 4.77 (s, 1H), 4.23 (ddd,  $J = 10.5, 9.9, 4.9$  Hz, 4H), 3.72 (s, 3H), 2.58 (dd,  $J = 13.9, 6.9$  Hz, 1H), 2.34 (t,  $J = 6.8$  Hz, 1H), 1.40 (t,  $J = 7.1$  Hz, 3H), 1.30 (t,  $J = 7.0$  Hz, 3H).  $^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  169.32 (1C, d,  $J = 4.3$  Hz), 155.01 (1C, s), 141.33 (1C, s), 140.44 (2C, s), 136.21 (1C, s), 132.44 (1C, s), 130.54 (1C, s), 128.97 (2C, s), 128.56 (1C, s), 128.21 (1C, s), 127.82 (2C, s), 127.72 (1C, s), 127.64 (2C, s), 127.20 (2C, s), 66.80 (1C, s), 63.52 (1C, d,  $J = 4.9$  Hz), 63.13 (1C, d,  $J = 6.2$  Hz), 53.04 (1C, s), 41.97 (1C, s), 36.84 (1C, d,  $J = 218.0$  Hz), 21.50 (1C, s), 16.67 (1C, d,  $J = 6.1$  Hz), 16.47 (1C, d,  $J = 6.3$  Hz).  $^{31}\text{P NMR}$  (162 MHz,  $\text{CDCl}_3$ )  $\delta$  20.59. **HR-MS** (ESI+)  $m/z$  calcd for  $\text{C}_{29}\text{H}_{33}\text{NO}_7\text{P}$   $[\text{M}+\text{H}]^+$  538.1989 found 538.1990. **IR** ( $\text{cm}^{-1}$ ) 2987, 2364, 1734, 1487, 1232, 1170, 1110, 1051, 1024, 977. **HPLC** Daicel Chiralpak IB, 250 mm,  $\varnothing$  4.6 mm, Heptane/Ethanol = 95/5, 1.0 mL/min,  $\lambda = 210$  nm,  $t_{\text{major}} = 11.33$  min,  $t_{\text{minor}} = 12.45$  min.  $[\alpha]_{\text{D}}^{20} -36.9$  (c 1.00,  $\text{CH}_2\text{Cl}_2$ ).

**Compound 25** : methyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-(4-fluoro phenyl)cyclopropane-1-carboxylate



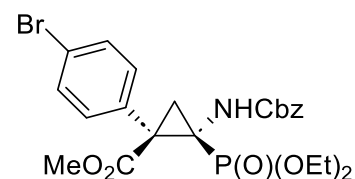
Compound **25** was prepared according to the general procedure D and obtained as an amorphous solid in 78% yield, 90:10 e.r..  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.42 – 7.30 (m, 2H), 7.28 – 7.20 (m, 3H), 7.13 – 7.01 (m, 2H), 7.00 – 6.92 (m, 2H), 4.85 (dd,  $J = 38.2, 12.4$  Hz, 2H), 4.64 (s, 1H), 4.21 – 4.05 (m, 4H), 3.62 (s, 3H), 2.47 (dd,  $J = 14.1, 7.0$  Hz, 1H), 2.20 (t,  $J = 6.8$  Hz, 1H), 1.31 (t,  $J = 7.1$  Hz, 3H), 1.23 – 1.17 (m, 3H).  $^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  169.09 (1C, s), 162.66 (1C, d,  $J = 247.9$  Hz), 154.82 (1C, s), 136.04 (1C, s), 131.86 (2C, d,  $J = 8.2$  Hz), 129.24 (1C, s), 128.47 (2C, s), 128.16 (1C, s), 127.71 (2C, s), 115.80 (2C, d,  $J = 21.7$  Hz), 66.71 (1C, s), 63.40 (1C, d,  $J = 5.4$  Hz), 63.00 (1C, d,  $J = 6.3$  Hz), 52.91 (1C, s), 41.40 (1C, s), 36.80 (1C, d,  $J = 217.7$  Hz), 21.36 (1C, s), 16.52 (1C, d,  $J = 5.9$  Hz), 16.33 (1C, d,  $J = 6.3$  Hz).  $^{19}\text{F NMR}$  (377 MHz,  $\text{CDCl}_3$ )  $\delta$  -113.13 (s).  $^{31}\text{P NMR}$  (162 MHz,  $\text{CDCl}_3$ )  $\delta$  20.41. **HR-MS** (ESI+)  $m/z$  calcd for  $\text{C}_{23}\text{H}_{28}\text{FNO}_7\text{P}$   $[\text{M}+\text{H}]^+$  480.1582 found 480.1581. **IR** ( $\text{cm}^{-1}$ ) 2999, 2360, 1734, 1512, 1442, 1305, 1220, 1163, 1064, 1028, 977. **HPLC** Daicel Chiralpak IB, 250 mm,  $\varnothing$  4.6 mm, Heptane/Ethanol = 95/5, 1.0 mL/min,  $\lambda = 210$  nm,  $t_{\text{minor}} = 9.12$  min,  $t_{\text{major}} = 9.89$  min.  $[\alpha]_{\text{D}}^{20} +17.7$  (c 1.00,  $\text{CH}_2\text{Cl}_2$ ).

**Compound 26**: methyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-1-(4-chlorophenyl)-2-(diethoxy phosphoryl)cyclopropane-1-carboxylate



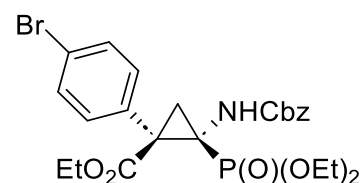
Compound **26** was prepared according to the general procedure D and obtained as an amorphous solid in 55% yield, 94.5:5.5 e.r..  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.43 – 7.36 (m, 2H), 7.35 – 7.29 (m, 5H), 7.19 – 7.08 (m, 2H), 4.93 (dd,  $J = 38.1, 12.4$  Hz, 2H), 4.69 (s, 1H), 4.20 (dp,  $J = 21.1, 7.0$  Hz, 4H), 3.70 (s, 3H), 2.55 (dd,  $J = 14.1, 7.0$  Hz, 1H), 2.27 (t,  $J = 6.8$  Hz, 1H), 1.38 (t,  $J = 7.1$  Hz, 3H), 1.28 (t,  $J = 7.0$  Hz, 3H).  $^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  168.88 (1C, s), 154.81 (1C, s), 136.01 (1C, s), 134.49 (1C, s), 131.98 (1C, s), 131.45 (2C, s), 129.02 (2C, s), 128.49 (2C, s), 128.18 (1C, s), 127.73 (2C, s), 66.74 (1C, s), 63.41 (1C, d,  $J = 5.2$  Hz), 63.02 (1C, d,  $J = 6.4$  Hz), 52.96 (1C, s), 41.50 (1C, s), 36.91 (1C, d,  $J = 217.2$  Hz), 21.23 (1C, s), 16.52 (1C, d,  $J = 6.1$  Hz), 16.33 (1C, d,  $J = 6.4$  Hz).  $^{31}\text{P NMR}$  (162 MHz,  $\text{CDCl}_3$ )  $\delta$  20.27. **HR-MS** (ESI+)  $m/z$  calcd for  $\text{C}_{23}\text{H}_{28}^{35}\text{ClNO}_7\text{P}$   $[\text{M}+\text{H}]^+$  496.1286 found 496.1287. **IR** ( $\text{cm}^{-1}$ ) 2987, 2364, 1734, 1494, 1232, 1093, 1022, 977. **HPLC** Daicel Chiralpak IB, 150 mm,  $\varnothing$  4.6 mm, Heptane/Ethanol = 97/3, 1.0 mL/min,  $\lambda = 210$  nm,  $t_{\text{minor}} = 13.54$  min,  $t_{\text{major}} = 14.80$  min.  $[\alpha]_{\text{D}}^{20} +28.2$  (c 1.00,  $\text{CH}_2\text{Cl}_2$ ).

**Compound 27**: methyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-1-(4-bromophenyl)-2-(diethoxy phosphoryl)cyclopropane-1-carboxylate



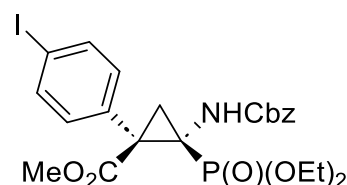
Compound **27** was prepared according to the general procedure D and obtained as a white solid in 89% yield, 89.5:10.5 e.r..  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.50 – 7.40 (m, 2H), 7.41 – 7.29 (m, 5H), 7.20 – 7.08 (m, 2H), 4.93 (dd,  $J = 39.2, 12.4$  Hz, 2H), 4.72 (s, 1H), 4.20 (dp,  $J = 21.2, 7.1$  Hz, 4H), 3.69 (s, 3H), 2.54 (dd,  $J = 14.1, 7.0$  Hz, 1H), 2.27 (t,  $J = 6.8$  Hz, 1H), 1.37 (t,  $J = 7.1$  Hz, 3H), 1.28 (t,  $J = 7.1$  Hz, 3H).  $^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  168.82 (1C, d,  $J = 4.0$  Hz), 154.82 (1C, s), 136.01 (1C, s), 132.51 (1C, s), 131.97 (2C, s), 131.78 (2C, s), 128.51 (2C, s), 128.18 (1C, s), 127.74 (2C, s), 122.70 (1C, s), 66.75 (1C, s), 63.43 (1C, d,  $J = 5.0$  Hz), 63.04 (1C, d,  $J = 6.3$  Hz), 52.97 (1C, s), 41.58 (1C, s), 36.90 (1C, d,  $J = 217.5$  Hz), 21.20 (1C, s), 16.52 (1C, d,  $J = 5.9$  Hz), 16.33 (1C, d,  $J = 6.4$  Hz).  $^{31}\text{P NMR}$  (162 MHz,  $\text{CDCl}_3$ )  $\delta$  20.25. **HR-MS** (ESI+)  $m/z$  calcd for  $\text{C}_{23}\text{H}_{28}^{79}\text{BrNO}_7\text{P}$   $[\text{M}+\text{H}]^+$  540.0781 found 540.0783. **IR** ( $\text{cm}^{-1}$ ) 2983, 1728, 1527, 1490, 1294, 1232, 1166, 1101, 1060, 1022, 974. **HPLC** Daicel Chiralpak IE, 150 mm,  $\varnothing$  4.6 mm, Heptane/iso-propanol = 70/30, 1.0 mL/min,  $\lambda = 220$  nm,  $t_{\text{minor}} = 9.82$  min,  $t_{\text{major}} = 12.31$  min.  $[\alpha]_{\text{D}}^{20} +30.3$  (c 1.00,  $\text{CH}_2\text{Cl}_2$ ). **MP** 140-143  $^{\circ}\text{C}$ .

**Compound 28**: ethyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-1-(4-bromophenyl)-2-(diethoxyphosphoryl)cyclopropane-1-carboxylate



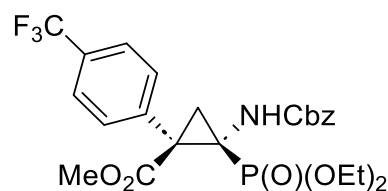
Compound **28** was prepared according to the general procedure D and obtained as a white solid in 84% yield, 97:3 e.r..  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.51 – 7.42 (m, 2H), 7.38 – 7.27 (m, 5H), 7.20 – 7.04 (m, 2H), 4.93 (dd,  $J = 38.3, 12.4$  Hz, 2H), 4.71 (s, 1H), 4.26 – 4.10 (m, 6H), 2.52 (dd,  $J = 14.1, 7.0$  Hz, 1H), 2.26 (t,  $J = 6.8$  Hz, 1H), 1.41 – 1.33 (m, 3H), 1.31 – 1.20 (m, 6H).  $^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  168.42 (s), 154.92 (s), 136.15 (s), 132.81 (s), 132.04 (s), 131.86 (s), 128.62 (s), 128.28 (s), 127.82 (s), 122.73 (s), 66.82 (s), 63.48 (d,  $J = 5.3$  Hz), 63.06 (d,  $J = 6.4$  Hz), 62.16 (s), 41.89 (s), 36.96 (d,  $J = 217.6$  Hz), 21.24 (s), 16.62 (d,  $J = 6.2$  Hz), 16.43 (d,  $J = 6.4$  Hz), 13.98 (s).  $^{31}\text{P NMR}$  (162 MHz,  $\text{CDCl}_3$ )  $\delta$  20.26. **HR-MS** (ESI+)  $m/z$  calcd for  $\text{C}_{24}\text{H}_{30}^{79}\text{BrNO}_7\text{P}$   $[\text{M}+\text{H}]^+$  554.0938 found 554.0939. **IR** ( $\text{cm}^{-1}$ ) 2976, 2364, 1724, 1519, 1494, 1298, 1224, 1178, 1064, 1022, 989. **HPLC** Daicel Chiralpak ID, 150 mm,  $\varnothing$  4.6 mm, Heptane/iso-propanol = 80/20, 1.0 mL/min,  $\lambda = 220$  nm,  $t_{\text{minor}} = 13.91$  min,  $t_{\text{major}} = 16.22$  min.  $[\alpha]_{\text{D}}^{20} +22.1$  (c 1.00,  $\text{CH}_2\text{Cl}_2$ ). **MP** 145-148 °C.

**Compound 29**: methyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-(4-iodophenyl)cyclopropane-1-carboxylate



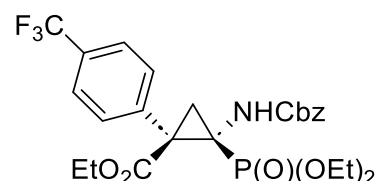
Compound **29** was prepared according to the general procedure D and obtained as an amorphous solid in 71% yield, 96:4 e.r..  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.70 – 7.62 (m, 2H), 7.31 (d,  $J = 6.8$  Hz, 3H), 7.17 (dd,  $J = 20.0, 7.3$  Hz, 4H), 4.93 (dd,  $J = 36.6, 12.4$  Hz, 2H), 4.70 (s, 1H), 4.20 (dt,  $J = 15.4, 6.4$  Hz, 4H), 3.69 (s, 3H), 2.53 (dd,  $J = 14.1, 7.0$  Hz, 1H), 2.26 (t,  $J = 6.8$  Hz, 1H), 1.37 (t,  $J = 7.1$  Hz, 3H), 1.28 (t,  $J = 7.0$  Hz, 3H).  $^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  168.92 (1C, d,  $J = 4.0$  Hz), 154.94 (1C, s), 138.07 (2C, s), 136.13 (1C, s), 133.32 (1C, s), 132.08 (2C, s), 128.66 (2C, s), 128.31 (1C, s), 127.87 (2C, s), 94.58 (1C, s), 66.88 (1C, s), 63.55 (1C, d,  $J = 5.4$  Hz), 63.14 (1C, d,  $J = 6.4$  Hz), 53.09 (1C, s), 41.80 (1C, s), 36.99 (1C, d,  $J = 217.0$  Hz), 21.24 (1C, s), 16.64 (1C, d,  $J = 6.0$  Hz), 16.44 (1C, d,  $J = 6.3$  Hz).  $^{31}\text{P NMR}$  (162 MHz,  $\text{CDCl}_3$ )  $\delta$  20.22. **HR-MS** (ESI+)  $m/z$  calcd for  $\text{C}_{23}\text{H}_{28}^{127}\text{INO}_7\text{P}$   $[\text{M}+\text{H}]^+$  588.0643 found 588.0643. **IR** ( $\text{cm}^{-1}$ ) 2991, 2364, 1728, 1519, 1433, 1298, 1224, 1166, 1097, 1051, 1018, 977. **HPLC** Daicel Chiralpak IB, 250 mm,  $\varnothing$  4.6 mm, Heptane/Ethanol = 95/5, 1.0 mL/min,  $\lambda = 210$  nm,  $t_{\text{minor}} = 9.60$  min,  $t_{\text{major}} = 10.34$  min.  $[\alpha]_{\text{D}}^{20} +32.1$  (c 1.00,  $\text{CH}_2\text{Cl}_2$ ).

**Compound 30**: methyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-(4-(trifluoromethyl)phenyl)cyclopropane-1-carboxylate



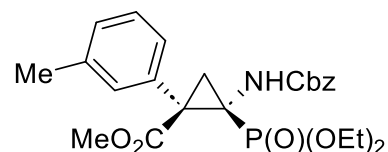
Compound **30** was prepared according to the general procedure D and obtained as an amorphous solid in 71% yield, 98.5:1.5 e.r..  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.67 – 7.57 (m, 4H), 7.35 – 7.26 (m, 3H), 7.19 – 7.06 (m, 2H), 4.92 (dd,  $J = 33.0, 12.3$  Hz, 2H), 4.71 (s, 1H), 4.20 (td,  $J = 14.4, 7.1$  Hz, 4H), 3.70 (s, 3H), 2.59 (dd,  $J = 14.1, 7.1$  Hz, 1H), 2.34 (t,  $J = 6.9$  Hz, 1H), 1.38 (t,  $J = 7.1$  Hz, 3H), 1.28 (t,  $J = 7.0$  Hz, 3H).  $^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  168.73 (1C, d,  $J = 4.0$  Hz), 154.92 (1C, s), 137.64 (1C, s), 136.03 (1C, s), 130.68 (2C, s), 128.61 (2C, s), 128.36 (1C, s), 127.89 (2C, s), 125.83 (2C, d,  $J = 3.5$  Hz), 124.04 (1C, q,  $J = 272.1$  Hz), 66.95 (1C, s), 63.62 (1C, d,  $J = 5.2$  Hz), 63.19 (1C, d,  $J = 6.3$  Hz), 53.18 (1C, s), 41.89 (1C, s), 37.27 (1C, d,  $J = 217.2$  Hz), 21.30 (1C, s), 16.63 (1C, d,  $J = 5.9$  Hz), 16.45 (1C, d,  $J = 6.2$  Hz).  $^{19}\text{F NMR}$  (377 MHz,  $\text{CDCl}_3$ )  $\delta$  -62.69 (s).  $^{31}\text{P NMR}$  (162 MHz,  $\text{CDCl}_3$ )  $\delta$  20.01. **HR-MS** (ESI+)  $m/z$  calcd for  $\text{C}_{24}\text{H}_{28}\text{F}_3\text{NO}_7\text{P}$   $[\text{M}+\text{H}]^+$  530.1550 found 530.1552. **IR** ( $\text{cm}^{-1}$ ) 2991, 2368, 1728, 1519, 1323, 1224, 1166, 1122, 1053, 1018, 977. **HPLC** Daicel Chiralpak IB, 250 mm,  $\varnothing$  4.6 mm, Heptane/Ethanol = 98/2, 1.0 mL/min,  $\lambda = 210$  nm,  $t_{\text{minor}} = 18.58$  min,  $t_{\text{major}} = 20.03$  min.  $[\alpha]_{\text{D}}^{20} +17.6$  (c 1.00,  $\text{CH}_2\text{Cl}_2$ ).

**Compound 31**: ethyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-(4-(trifluoromethyl)phenyl)cyclopropane-1-carboxylate



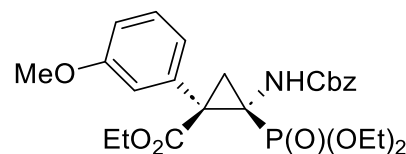
Compound **31** was prepared according to the general procedure D and obtained as an amorphous solid in 60% yield, 99:1 e.r..  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.64 – 7.55 (m, 4H), 7.36 – 7.27 (m, 3H), 7.18 – 7.05 (m, 2H), 4.92 (dd,  $J = 31.6, 12.3$  Hz, 2H), 4.69 (s, 1H), 4.25 – 4.12 (m, 6H), 2.57 (dd,  $J = 14.1, 7.0$  Hz, 1H), 2.33 (t,  $J = 6.8$  Hz, 1H), 1.38 (t,  $J = 7.1$  Hz, 3H), 1.27 (dt,  $J = 14.2, 7.1$  Hz, 6H).  $^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  168.07 (1C, d,  $J = 3.9$  Hz), 154.80 (1C, s), 137.70 (1C, s), 135.94 (1C, s), 130.51 (2C, s), 128.48 (2C, s), 128.22 (1C, s), 127.74 (2C, s), 125.67 (2C, d,  $J = 3.4$  Hz), 123.94 (1C, q,  $J = 271.9$  Hz), 66.79 (1C, s), 63.45 (1C, d,  $J = 5.2$  Hz), 63.00 (1C, d,  $J = 6.3$  Hz), 62.18 (1C, s), 41.96 (1C, s), 37.10 (1C, d,  $J = 217.1$  Hz), 21.12 (1C, s), 16.50 (1C, d,  $J = 6.1$  Hz), 16.32 (1C, d,  $J = 6.4$  Hz), 13.85 (1C, s).  $^{19}\text{F NMR}$  (377 MHz,  $\text{CDCl}_3$ )  $\delta$  -62.67 (s).  $^{31}\text{P NMR}$  (162 MHz,  $\text{CDCl}_3$ )  $\delta$  20.03. **HR-MS** (ESI+)  $m/z$  calcd for  $\text{C}_{25}\text{H}_{30}\text{F}_3\text{NO}_7\text{P}$   $[\text{M}+\text{H}]^+$  544.1707 found 544.1708. **IR** ( $\text{cm}^{-1}$ ) 2987, 2360, 1728, 1519, 1327, 1228, 1170, 1126, 1060, 1018, 977. **HPLC** Daicel Chiralpak IA, 250 mm,  $\varnothing$  4.6 mm, Heptane/Ethanol = 95/5, 1.0 mL/min,  $\lambda = 210$  nm,  $t_{\text{minor}} = 14.19$  min,  $t_{\text{major}} = 15.49$  min.  $[\alpha]_{\text{D}}^{20} +21.2$  (c 1.00,  $\text{CH}_2\text{Cl}_2$ ).

**Compound 32**: methyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-(*m*-tolyl)cyclopropane-1-carboxylate



Compound **32** was prepared according to the general procedure D and obtained as an amorphous solid in 82% yield, 90:10 e.r..  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.41 – 7.26 (m, 3H), 7.26 – 7.20 (m, 3H), 7.18 – 7.07 (m, 3H), 4.92 (dd,  $J = 46.2, 12.4$  Hz, 2H), 4.73 (s, 1H), 4.27 – 4.15 (m, 4H), 3.69 (s, 3H), 2.52 (dd,  $J = 13.6, 6.8$  Hz, 1H), 2.34 (s, 3H), 2.25 (t,  $J = 6.2$  Hz, 1H), 1.38 (t,  $J = 7.1$  Hz, 3H), 1.28 (t,  $J = 6.9$  Hz, 3H).  $^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  169.43 (1C, d,  $J = 4.1$  Hz), 155.04 (1C, s), 138.76 (1C, s), 136.29 (1C, s), 133.34 (1C, s), 130.72 (1C, s), 129.38 (1C, s), 128.88 (1C, s), 128.57 (2C, s), 128.20 (1C, s), 127.77 (2C, s), 127.21 (1C, s), 66.74 (1C, s), 63.50 (1C, d,  $J = 4.4$  Hz), 63.11 (1C, d,  $J = 6.4$  Hz), 52.98 (1C, s), 42.21 (1C, s), 36.51 (1C, d,  $J = 218.0$  Hz), 21.61 (1C, s), 16.67 (1C, d,  $J = 5.9$  Hz), 16.46 (1C, d,  $J = 6.3$  Hz).  $^{31}\text{P NMR}$  (162 MHz,  $\text{CDCl}_3$ )  $\delta$  20.65. **HR-MS** (ESI+)  $m/z$  calcd for  $\text{C}_{24}\text{H}_{31}\text{NO}_7\text{P}$   $[\text{M}+\text{H}]^+$  476.1833. found 476.1832. **IR** ( $\text{cm}^{-1}$ ) 2983, 2364, 1734, 1523, 1429, 1305, 1220, 1159, 1051, 1022, 977. **HPLC** Daicel Chiralpak IB, 250 mm,  $\varnothing$  4.6 mm, Heptane/Ethanol = 95/5, 1.0 mL/min,  $\lambda = 210$  nm,  $t_{\text{minor}} = 7.89$  min,  $t_{\text{major}} = 8.51$  min.  $[\alpha]_{\text{D}}^{20} +12.6$  (c 1.00,  $\text{CH}_2\text{Cl}_2$ ).

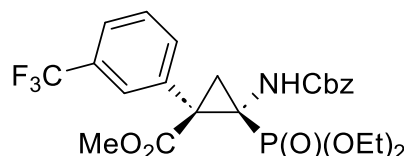
**Compound 33**: ethyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-(3-methoxyphenyl)cyclopropane-1-carboxylate



Compound **33** was prepared according to the general procedure D and obtained as an amorphous solid in 78% yield, 96.5:3.5 e.r..  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.36 – 7.26 (m, 4H), 7.18 – 7.08 (m, 2H), 7.08 – 6.98 (m, 2H), 6.89 – 6.81 (m, 1H), 4.93 (dd,  $J = 41.5, 12.5$  Hz, 2H), 4.74 (s, 1H), 4.18 (ddd,  $J = 18.9, 12.2, 5.1$  Hz, 6H), 3.79 (s, 3H), 2.50 (dd,  $J = 13.7, 6.9$  Hz, 1H), 2.24 (t,  $J = 6.6$  Hz, 1H), 1.37 (t,  $J = 7.1$  Hz, 3H), 1.27 (dd,  $J = 10.2, 7.0$  Hz, 6H).  $^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  168.69 (1C, d,  $J = 4.3$  Hz), 159.91 (1C, s), 154.98 (1C, s), 136.32 (1C, s), 135.06 (1C, s), 129.93 (1C, s), 128.55 (2C, s), 128.15 (1C, s), 127.69 (2C, s), 122.35 (1C, s), 115.89 (1C, s), 113.89 (1C, s), 66.68 (1C, s), 63.42 (1C, s), 62.97 (1C, d,  $J = 6.4$  Hz), 62.01 (1C, s), 55.36 (1C, s), 42.42 (1C, s), 36.54 (1C, d,  $J = 218.8$  Hz), 21.55 (1C, s), 16.63 (1C, d,  $J = 6.2$  Hz), 16.43 (1C, d,  $J = 6.5$  Hz), 14.00 (1C, s).  $^{31}\text{P NMR}$  (162 MHz,  $\text{CDCl}_3$ )  $\delta$  20.27. **HR-MS** (ESI+)  $m/z$  calcd for  $\text{C}_{25}\text{H}_{33}\text{NO}_8\text{P}$   $[\text{M}+\text{H}]^+$  506.1938 found 506.1936. **IR** ( $\text{cm}^{-1}$ ) 2991, 2360, 1724, 1593, 1487, 1286, 1232, 1163, 1018, 970. **HPLC** Daicel Chiralpak IB, 250 mm,  $\varnothing$  4.6 mm, Heptane/Ethanol = 97/3, 1.0 mL/min,  $\lambda = 210$  nm,  $t_{\text{minor}} = 12.25$  min,  $t_{\text{major}} = 13.29$  min.  $[\alpha]_{\text{D}}^{20} +27.8$  (c 1.00,  $\text{CH}_2\text{Cl}_2$ ).

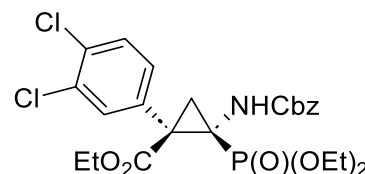


**Compound 34** : methyl (1*S*,2*R*)-2-  
(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-  
(3-(trifluoromethyl)phenyl)cyclopropane-1-carboxylate



Compound **34** was prepared according to the general procedure D and obtained as an amorphous solid in 87% yield, 56.5:43.5 e.r..  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.73 – 7.67 (m, 2H), 7.58 (d,  $J$  = 7.8 Hz, 1H), 7.47 (t,  $J$  = 7.7 Hz, 1H), 7.34 – 7.27 (m, 3H), 7.19 – 7.07 (m, 2H), 4.92 (dd,  $J$  = 43.4, 12.3 Hz, 2H), 4.70 (s, 1H), 4.20 (qd,  $J$  = 14.4, 7.2 Hz, 4H), 3.70 (d,  $J$  = 6.4 Hz, 3H), 2.60 (dd,  $J$  = 14.2, 7.1 Hz, 1H), 2.33 (t,  $J$  = 6.9 Hz, 1H), 1.38 (t,  $J$  = 7.1 Hz, 3H), 1.29 (t,  $J$  = 7.0 Hz, 3H).  $^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  168.82 (1C, d,  $J$  = 3.9 Hz), 154.99 (1C, s), 136.06 (1C, s), 134.75 (1C, s), 133.80 (1C, s), 129.39 (1C, s), 128.62 (2C, s), 128.33 (1C, s), 127.89 (2C, s), 126.90 (1C, s), 125.42 (1C, d,  $J$  = 2.9 Hz), 123.95 (1C, q,  $J$  = 273.8 Hz), 66.99 (1C, s), 63.63 (1C, d,  $J$  = 6.1 Hz), 63.25 (1C, d,  $J$  = 6.5 Hz), 53.20 (1C, s), 41.86 (1C, s), 37.38 (1C, d,  $J$  = 217.3 Hz), 21.40 (1C, s), 16.63 (1C, d,  $J$  = 5.9 Hz), 16.46 (1C, d,  $J$  = 6.3 Hz).  $^{19}\text{F NMR}$  (377 MHz,  $\text{CDCl}_3$ )  $\delta$  -62.55 (s).  $^{31}\text{P NMR}$  (162 MHz,  $\text{CDCl}_3$ )  $\delta$  20.27. **HR-MS** (ESI+)  $m/z$  calcd for  $\text{C}_{24}\text{H}_{28}\text{F}_3\text{NO}_7\text{P}$   $[\text{M}+\text{H}]^+$  530.1550 found 530.1551. **IR** ( $\text{cm}^{-1}$ ) 2983, 2364, 1737, 1512, 1442, 1338, 1232, 1163, 1130, 1055, 1021, 977. **HPLC** Daicel Chiralpak IB, 250 mm,  $\varnothing$  4.6 mm, Heptane/Ethanol = 95/5, 1.0 mL/min,  $\lambda$  = 210 nm,  $t_{\text{minor}}$  = 7.51 min,  $t_{\text{major}}$  = 8.25 min.  $[\alpha]_{\text{D}}^{20}$  0.4 (c 1.00,  $\text{CH}_2\text{Cl}_2$ ).

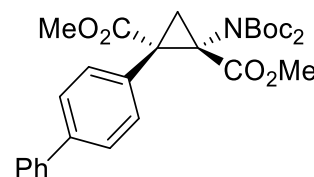
**Compound 35** : ethyl (1*S*,2*R*)-2-  
(((benzyloxy)carbonyl)amino)-1-(3,4-dichlorophenyl)-2-  
(diethoxyphosphoryl)cyclopropane-1-carboxylate



Compound **35** was prepared according to the general procedure D and obtained as an amorphous solid in 80% yield, 91.5:8.5 e.r..  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.56 (s, 1H), 7.37 (dd,  $J$  = 10.5, 7.2 Hz, 2H), 7.30 (d,  $J$  = 6.7 Hz, 4H), 7.16 (d,  $J$  = 5.6 Hz, 2H), 4.95 (dd,  $J$  = 47.7, 12.3 Hz, 2H), 4.76 (s, 1H), 4.22 – 4.10 (m, 6H), 2.53 (dd,  $J$  = 14.1, 7.0 Hz, 1H), 2.25 (t,  $J$  = 6.8 Hz, 1H), 1.36 (t,  $J$  = 7.1 Hz, 3H), 1.30 – 1.23 (m, 6H).  $^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  168.08 (1C, d,  $J$  = 3.7 Hz), 154.99 (1C, s), 133.99 (1C, s), 132.87 (1C, d,  $J$  = 2.4 Hz), 132.15 (1C, s), 130.72 (1C, s), 129.61 (1C, s), 128.64 (2C, s), 128.35 (1C, s), 127.90 (2C, s), 66.98 (1C, s), 63.55 (1C, d,  $J$  = 5.1 Hz), 63.15 (1C, d,  $J$  = 6.4 Hz), 62.33 (1C, s), 41.53 (1C, s), 37.40 (1C, d,  $J$  = 216.8 Hz), 21.29 (1C, s), 16.61 (1C, d,  $J$  = 6.0 Hz), 16.43 (1C, d,  $J$  = 6.3 Hz), 13.97 (1C, s).  $^{31}\text{P NMR}$  (162 MHz,  $\text{CDCl}_3$ )  $\delta$  20.27. **HR-MS** (ESI+)  $m/z$  calcd for  $\text{C}_{24}\text{H}_{29}^{35}\text{Cl}_2\text{NO}_7\text{P}$   $[\text{M}+\text{H}]^+$  544.1053 found 544.1057. **IR** ( $\text{cm}^{-1}$ ) 2983, 2364, 1728, 1470, 1300, 1224, 1047, 1018, 977. **HPLC** Daicel Chiralpak IB, 250 mm,  $\varnothing$  4.6 mm, Heptane/Ethanol = 97/3, 1.0 mL/min,  $\lambda$  = 210 nm,  $t_{\text{minor}}$  = 10.23 min,  $t_{\text{major}}$  = 11.14 min.  $[\alpha]_{\text{D}}^{20}$  +15.6 (c 1.00,  $\text{CH}_2\text{Cl}_2$ ).

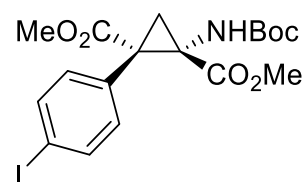
## 5. Post-functionalization reactions

### **Compound 36**: dimethyl (1S,2S)-1-([1,1'-biphenyl]-4-yl)-2-(bis(tert-butoxycarbonyl)amino)cyclopropane-1,2-dicarboxylate



The cyclopropane **9** (20.0 mg; 34.8  $\mu\text{mol}$ ), phenyl boronic acid (5.1 mg; 41.7  $\mu\text{mol}$ ) and  $\text{K}_2\text{CO}_3$  (9.6 mg; 69.5  $\mu\text{mol}$ ) were dissolved in THF:H<sub>2</sub>O (1.0:0.3 mL). The solution was degassed with argon. Then, bistriphenylphosphinepalladium dichloride (1.2 mg; 1.7  $\mu\text{mol}$ ) was added and the mixture heated at 80 °C for 16 h. The aqueous phase was extracted with EtOAc (3 x 3.0 mL). The organic phase was dried, filtered and concentrated. The crude product was purified by column chromatography on silica gel (Pentane:Et<sub>2</sub>O 30%) to give the pure product as a colorless oil in 77% yield, 99:1 d.r., 98:2 e.r.. **<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.63 – 7.57 (m, 2H), 7.54 (s, 4H), 7.43 (dd,  $J$  = 8.2, 6.7 Hz, 2H), 7.37 – 7.30 (m, 1H), 3.62 (s, 3H), 3.29 (s, 3H), 2.61 (d,  $J$  = 6.1 Hz, 1H), 2.28 (d,  $J$  = 6.1 Hz, 1H), 1.57 (s, 9H), 1.52 (s, 9H). **<sup>13</sup>C NMR** (75 MHz, CDCl<sub>3</sub>)  $\delta$  169.81 (s, 1C), 168.29 (s, 1C), 153.01 (s, 1C), 152.59 (s, 1C), 140.95 (s, 1C), 140.38 (s, 1C), 134.87 (s, 1C), 130.68 (s, 2C), 128.85 (s, 2C), 127.39 (s, 1C), 127.20 (s, 2C), 126.71 (s, 2C), 83.05 (s, 2C), 53.16 (s, 1C), 52.53 (s, 1C), 50.49 (s, 1C), 44.84 (s, 1C), 28.20 (s, 3C), 28.16 (s, 3C), 26.96 (s, 1C). **HR-MS** (ESI)  $m/z$  calcd for C<sub>29</sub>H<sub>35</sub>NO<sub>8</sub>K [M+K]<sup>+</sup> 564.2000, found 564.2006. **IR** (cm<sup>-1</sup>) 2981, 2952, 1731, 1708, 1488, 1435, 1366, 1275, 1230, 1165, 1116, 1076, 1014, 957. **HPLC** Daicel Chiralpak IE-3, 150 mm,  $\varnothing$  4.6 mm, Heptane/iso-propanol = 90/10, 1.0 mL/min,  $\lambda$  = 220 nm,  $t_{\text{major}}$  = 9.48 min,  $t_{\text{minor}}$  = 12.58 min.  **$[\alpha]_D^{20}$**  -14.1 (c 1.00, CHCl<sub>3</sub>).

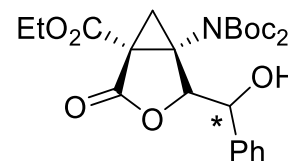
### **Compound 37**: dimethyl (1S,2S)-1-((tert-butoxycarbonyl)amino)-2-(4-iodophenyl)cyclopropane-1,2-dicarboxylate



The cyclopropane **9** (20.0 mg; 34.8  $\mu\text{mol}$ ) was dissolved in MeCN (300.0  $\mu\text{L}$ ). The solution was cooled to 0 °C and Yb(OTf)<sub>3</sub> (2.2 mg; 3.5  $\mu\text{mol}$ ) was added. The mixture was stirred at 25 °C for 1 h. Water (2.0 mL) and CH<sub>2</sub>Cl<sub>2</sub> (2.0 mL) were added and the aqueous phase was extracted with CH<sub>2</sub>Cl<sub>2</sub> (2 x 2.0 mL). The organic phases were dried, filtered and concentrated. The crude product was purified by column chromatography on silica gel (Pentane:Et<sub>2</sub>O 30 to 50%) to give the pure product as a colorless oil in 73% yield, 99:1 d.r., 98.5:1.5 e.r.. **<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.62 (d,  $J$  = 8.5 Hz, 2H), 7.15 (d,  $J$  = 8.2 Hz, 2H), 5.35 (s, 1H), 3.65 (s, 3H), 3.38 (s, 3H), 2.39 (d,  $J$  = 5.8 Hz, 1H), 2.24 (s, 1H), 1.46 (s, 9H). **<sup>13</sup>C NMR** (75 MHz, CDCl<sub>3</sub>)  $\delta$  168.87 (s, 2C), 155.66 (s, 1C), 137.45 (s, 2C), 134.54 (s, 1C), 132.19 (s, 2C), 94.06 (s, 1C), 80.70 (s, 1C), 53.27 (s, 1C), 52.64 (s, 2C), 44.92 (s, 1C), 28.37 (s, 3C), 24.78 (s, 1C). **HR-MS** (ESI)  $m/z$  calcd for C<sub>18</sub>H<sub>23</sub>NO<sub>6</sub>I [M+H]<sup>+</sup> 476.0570, found 476.0576. **IR** (cm<sup>-1</sup>) 3373, 3100, 2954, 2845, 1712, 1569, 1517, 1484, 1438, 1392, 1351, 1230, 1206, 1165, 1089, 1054, 1006, 953. **HPLC** Daicel Chiralpak IC-3, 150 mm,  $\varnothing$  4.6 mm, Heptane/iso-

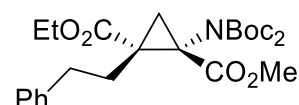
propanol = 80/20, 1.0 mL/min,  $\lambda = 220$  nm,  $t_{\text{major}} = 7.42$  min,  $t_{\text{minor}} = 10.04$  min.  $[\alpha]_{\text{D}}^{20} -18.1$  (c 1.00,  $\text{CHCl}_3$ ).

**Compound 38**: ethyl (1*R*,2*S*,5*S*)-5-(bis(tert-butoxycarbonyl)amino)-2-(hydroxy(phenyl)methyl)-4-oxo-3-oxabicyclo[3.1.0]hexane-1-carboxylate



The cyclopropane **17** (15.0 mg; 31.0  $\mu\text{mol}$ ) was dissolved in acetone and  $\text{H}_2\text{O}$  (700.0  $\mu\text{L}$ ; 4:1). NMO (6.8 mg; 67.4  $\mu\text{mol}$ ) and  $\text{OsO}_4$  (9.4  $\mu\text{L}$ ; 0.9  $\mu\text{mol}$ , 4wt% in  $\text{H}_2\text{O}$ ) were added and the reaction was stirred at 25  $^\circ\text{C}$  for 16 h. The reaction was quenched and filtrated over celite. The organic phase was extracted with  $\text{CH}_2\text{Cl}_2$  (3 x 2.0 mL). The organic phases were dried, filtered and concentrated. The crude product was purified by column chromatography on silica gel (Pentane: $\text{Et}_2\text{O}$  40%) to give the pure product as a colorless oil in 63% yield, 99:1 d.r., 98.5:1.5 e.r..  $^1\text{H NMR}$  (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.53 – 7.29 (m, 5H), 5.18 (d,  $J = 6.5$  Hz, 1H), 4.79 (dd,  $J = 6.4, 3.5$  Hz, 1H), 4.06 – 3.65 (m, 2H), 2.51 (d,  $J = 3.5$  Hz, 1H), 2.46 (d,  $J = 6.8$  Hz, 1H), 2.23 (d,  $J = 6.9$  Hz, 1H), 1.54 (s, 9H), 1.42 (s, 9H), 1.03 (t,  $J = 7.1$  Hz, 3H).  $^{13}\text{C NMR}$  (75 MHz,  $\text{CDCl}_3$ )  $\delta$  170.22 (s, 1C), 165.69 (s, 1C), 150.86 (s, 1C), 150.68 (s, 1C), 138.26 (s, 1C), 128.88 (s, 1C), 128.72 (s, 2C), 126.77 (s, 2C), 84.51 (s, 1C), 84.03 (s, 1C), 80.75 (s, 1C), 74.59 (s, 1C), 61.98 (s, 1C), 50.13 (s, 1C), 37.89 (s, 1C), 28.11 (s, 3C), 27.82 (s, 3C), 23.53 (s, 1C), 13.86 (s, 1C).  $^1\text{H NMR}$  in  $\text{CDCl}_3/\text{D}_2\text{O}$  (9:1) and HSQC can be found in the spectra section, confirming the structure. **HR-MS** (ESI)  $m/z$  calcd for  $\text{C}_{25}\text{H}_{33}\text{NO}_9\text{K}$  [ $\text{M}+\text{K}$ ] $^+$  530.1792, found 530.1804. **IR** ( $\text{cm}^{-1}$ ) 3513, 2980, 2922, 2856, 1789, 1729, 1455, 1369, 1275, 1249, 1156, 1116, 1099, 1025, 998. **HPLC** Daicel Chiralpak IE-3, 150 mm,  $\varnothing$  4.6 mm, Heptane/iso-propanol = 80/20, 1.0 mL/min,  $\lambda = 220$  nm,  $t_{\text{major}} = 8.81$  min,  $t_{\text{minor}} = 11.91$  min.  $[\alpha]_{\text{D}}^{20} +1.8$  (c 1.00,  $\text{CHCl}_3$ ).

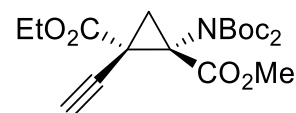
**Compound 39**: 2-ethyl 1-methyl (1*S*,2*R*)-1-(bis(tert-butoxycarbonyl)amino)-2-phenethylcyclopropane-1,2-dicarboxylate



The cyclopropane **17** (22.0 mg; 50.0  $\mu\text{mol}$ ) was dissolved in  $\text{EtOAc}$  (1.5 mL).  $\text{Pd}(\text{OH})_2$  (3.2 mg; 4.5  $\mu\text{mol}$ ) was added. The atmosphere was filled with  $\text{H}_2$  and the mixture stirred at 25  $^\circ\text{C}$  for 24 h. The reaction was filtered over a pad of celite and concentrated to give the pure product as a colorless oil in 91% yield, 99:1 d.r., 96.6:3.5 e.r..  $^1\text{H NMR}$  (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.33 – 7.23 (m, 2H), 7.22 – 7.11 (m, 3H), 4.33 – 3.96 (m, 2H), 3.73 (s, 3H), 2.72 (dt,  $J = 11.3, 5.6$  Hz, 2H), 2.41 (ddd,  $J = 15.5, 10.3, 5.5$  Hz, 1H), 2.03 (ddd,  $J = 13.9, 10.3, 6.8$  Hz, 1H), 1.98 – 1.91 (m, 2H), 1.49 (s, 9H), 1.46 (s, 9H), 1.29 (t,  $J = 7.2$  Hz, 3H).  $^{13}\text{C NMR}$  (75 MHz,  $\text{CDCl}_3$ )  $\delta$  169.73 (s, 1C), 169.33 (s, 1C), 152.26 (s, 1C), 151.85 (s, 1C), 142.01 (s, 1C), 128.67 (s, 2C), 128.46 (s, 2C), 125.97 (s, 1C), 82.91 (s, 1C), 82.76 (s, 1C), 61.62 (s, 1C), 52.97 (s, 1C), 48.92 (s, 1C), 42.19 (s, 1C), 33.76 (s, 1C), 29.68 (s, 1C), 28.13 (s, 3C), 28.01 (s, 3C), 27.37 (s, 1C), 14.13

(s, 1C). **HR-MS** (ES)  $m/z$  calcd for  $C_{26}H_{37}NO_8K$   $[M+K]^+$  530.2156, found 530.2161. **IR** ( $cm^{-1}$ ) 2981, 2936, 2258, 1794, 1725, 1455, 1367, 1251, 1212, 1158, 1096, 1062, 1030, 912. **HPLC** Daicel Chiralpak IC-3, 150 mm,  $\varnothing$  4.6 mm, Heptane/iso-propanol = 90/10, 1.0 mL/min,  $\lambda$  = 220 nm,  $t_{major}$  = 5.08 min,  $t_{minor}$  = 22.11 min.  $[\alpha]_D^{20}$  +0.6 (c 1.00,  $CHCl_3$ ).

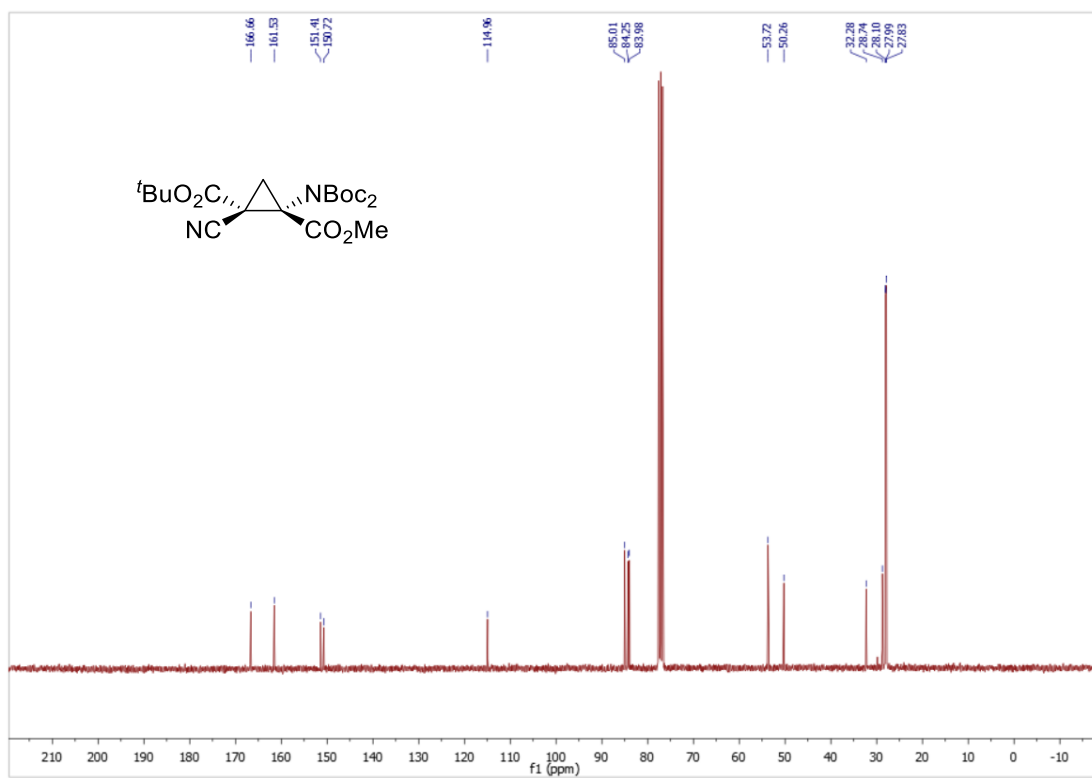
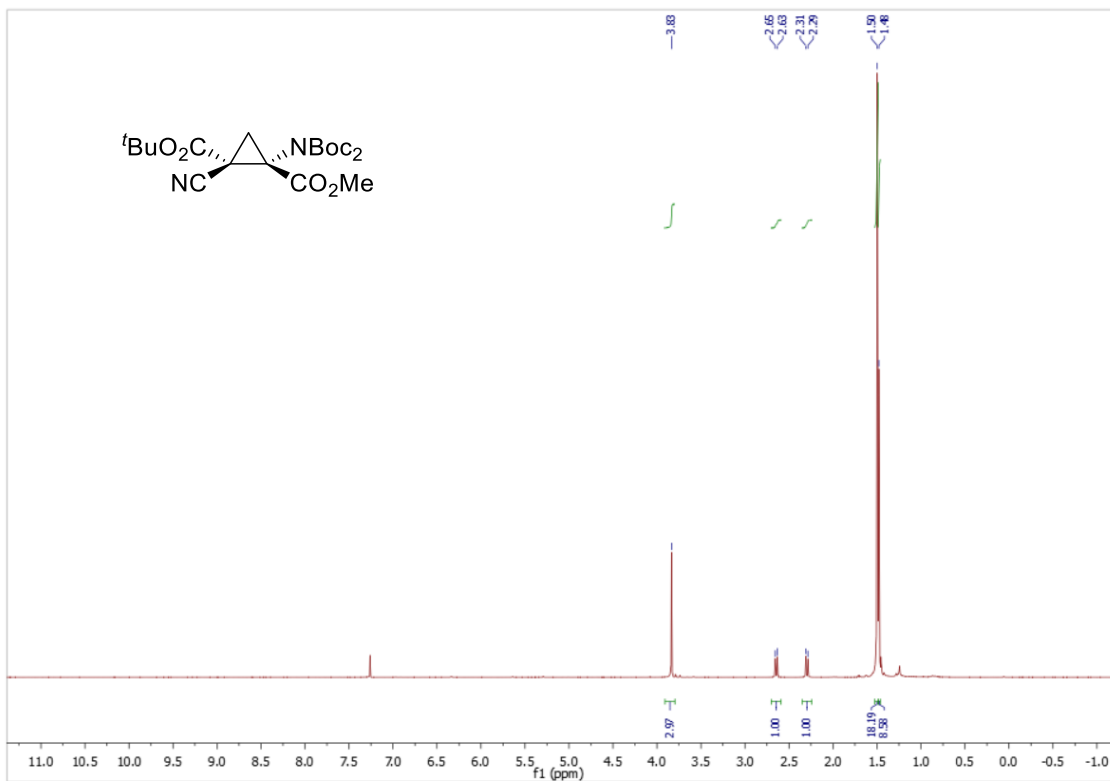
**Compound 40**: 2-ethyl 1-methyl (1*S*,2*S*)-1-(bis(tert-butoxycarbonyl)amino)-2-ethynylcyclopropane-1,2-dicarboxylate



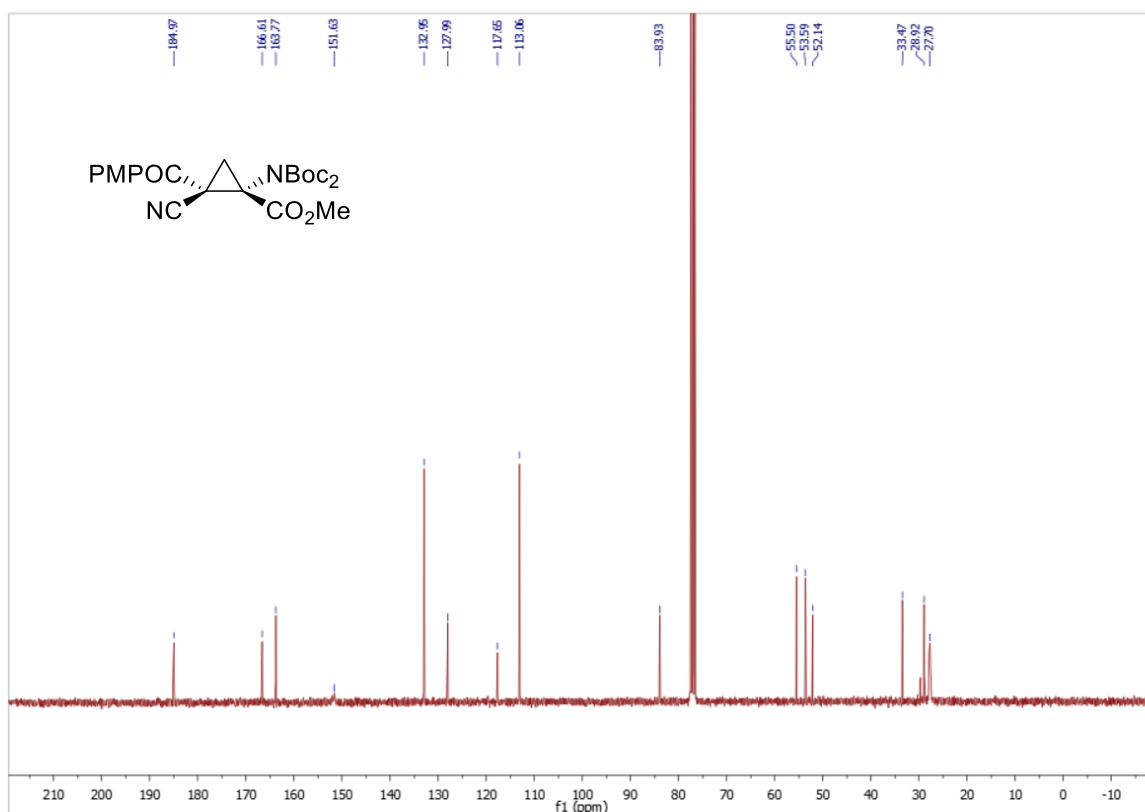
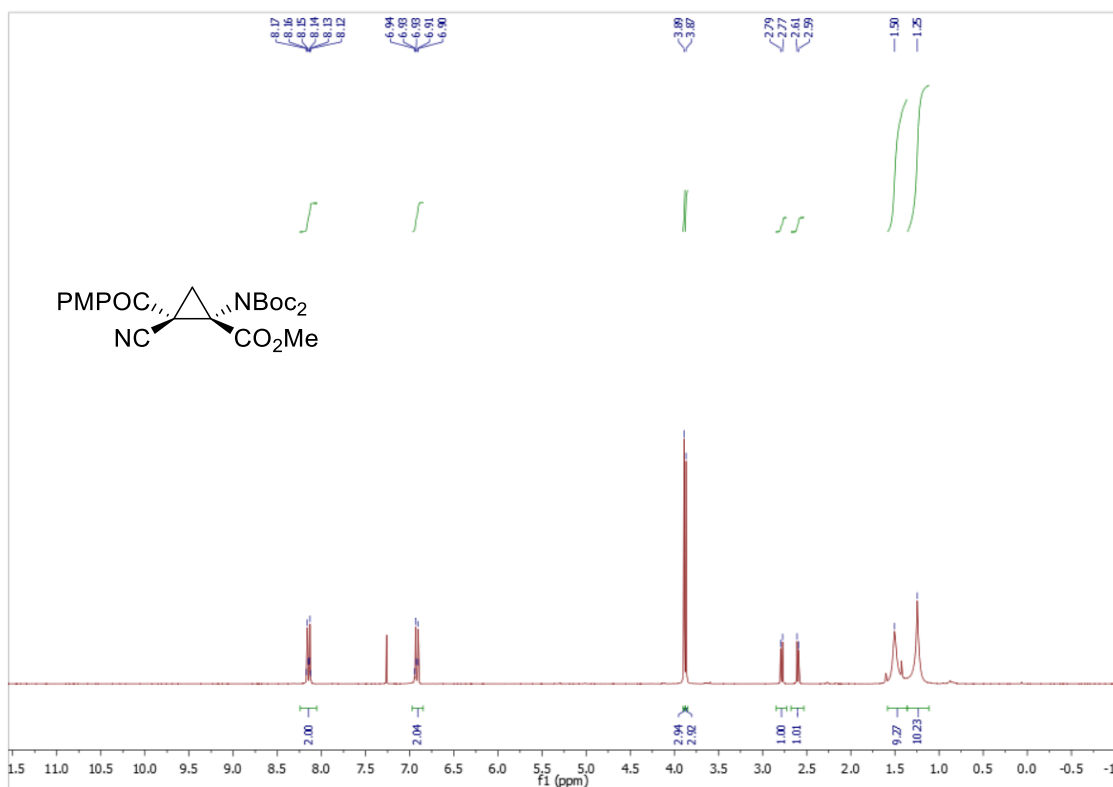
The cyclopropane **15** (15.0 mg; 31.0  $\mu$ mol) was dissolved in dry THF (200.0  $\mu$ L). TBAF (43.4  $\mu$ L; 43.4  $\mu$ mol, 1 M in THF) was added and the reaction stirred at 25  $^{\circ}C$  for 1 h. The solution was filtrated over a small pad of silica and eluted with  $Et_2O$  to give the pure product as a colorless oil in 96% yield, 99:1 d.r., 92:8 e.r..  **$^1H$  NMR** (300 MHz,  $CDCl_3$ )  $\delta$  4.32 – 3.99 (m, 2H), 3.77 (s, 3H), 2.52 (d,  $J$  = 6.3 Hz, 1H), 2.34 (s, 1H), 2.19 (d,  $J$  = 6.3 Hz, 1H), 1.49 (s, 9H), 1.48 (s, 9H), 1.30 (t,  $J$  = 7.2 Hz, 3H).  **$^{13}C$  NMR** (75 MHz,  $CDCl_3$ )  $\delta$  167.27 (s, 1C), 166.50 (s, 1C), 151.81 (s, 1C), 151.21 (s, 1C), 83.45 (s, 1C), 83.32 (s, 1C), 77.77 (s, 1C), 72.68 (s, 1C), 62.70 (s, 1C), 52.99 (s, 1C), 51.39 (s, 1C), 33.80 (s, 1C), 29.81 (s, 1C), 28.13 (s, 3C), 27.95 (s, 3C), 14.06 (s, 1C). **HR-MS** (ESI)  $m/z$  calcd for  $C_{20}H_{30}NO_8$   $[M+H]^+$  412.1971, found 412.1963. **IR** ( $cm^{-1}$ ) 3293, 3262, 2984, 2918, 2850, 1743, 1727, 1458, 1430, 1395, 1369, 1340, 1285, 1250, 1208, 1121, 1088, 1065, 1034, 1015, 967. **HPLC** Daicel Chiralpak IB-3, 150 mm,  $\varnothing$  4.6 mm, Heptane/iso-propanol = 98/2, 15  $^{\circ}C$ , 1.0 mL/min,  $\lambda$  = 220 nm,  $t_{major}$  = 7.26 min,  $t_{minor}$  = 8.07 min.  $[\alpha]_D^{20}$  -6.2 (c 1.00,  $CHCl_3$ ).

## 6. NMR spectra

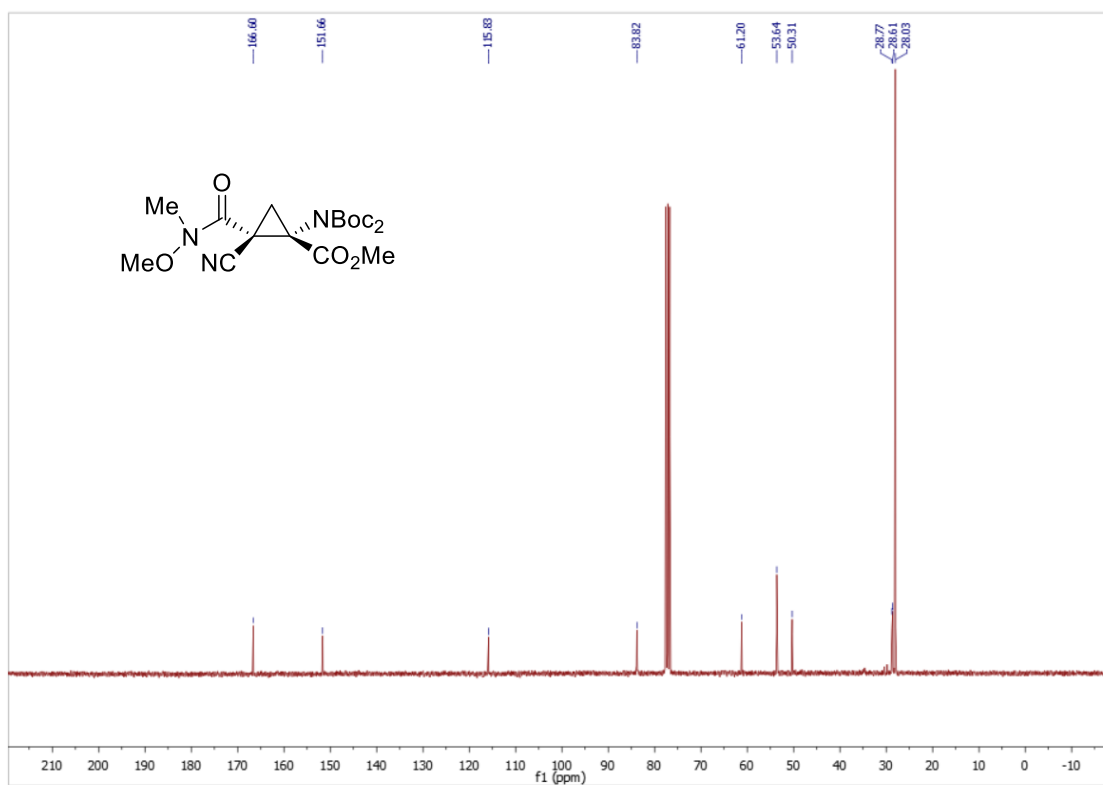
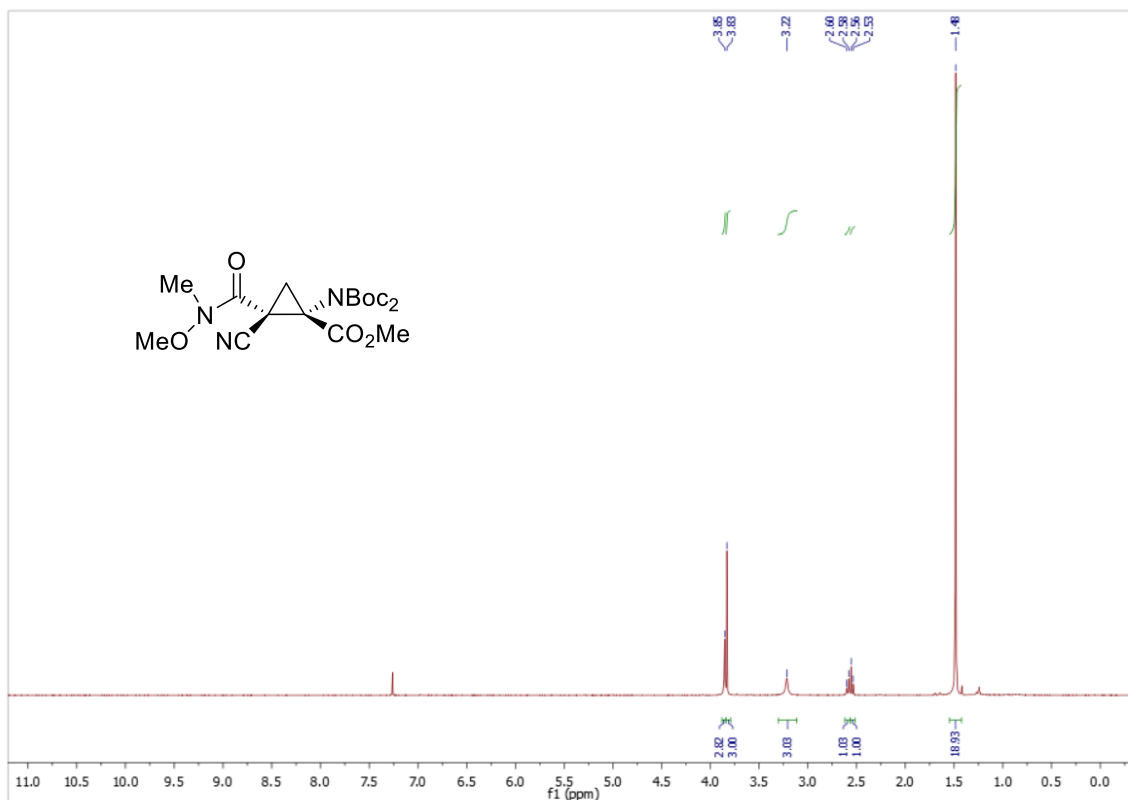
**Compound 1** : 2-(tert-butyl) 1-methyl (1*S*,2*R*)-1-(bis(tert-butoxycarbonyl)amino)-2-cyanocyclopropane-1,2-dicarboxylate

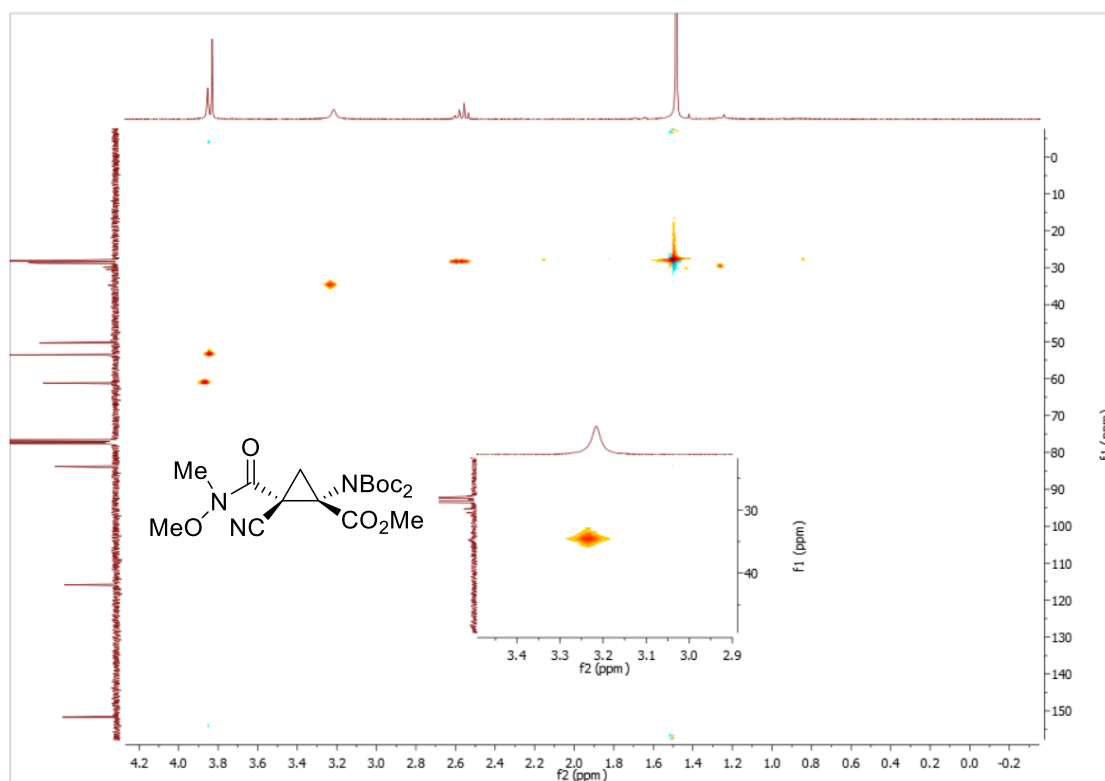


**Compound 2**: methyl (1*S*,2*R*)-1-(bis(tert-butoxycarbonyl)amino)-2-cyano-2-(4-methoxybenzoyl)cyclopropane-1-carboxylate

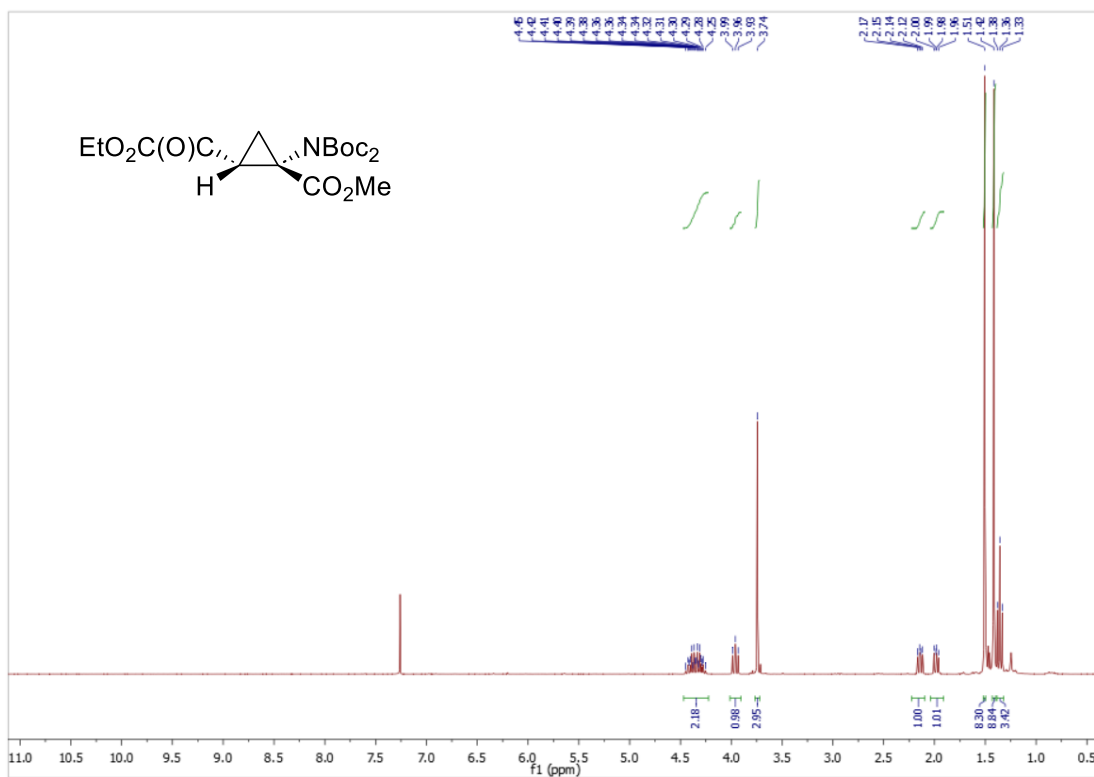


**Compound 3**: methyl (1*S*,2*R*)-1-(bis(tert-butoxycarbonyl)amino)-2-cyano-2-(methoxy(methyl)carbamoyl)cyclopropane-1-carboxylate

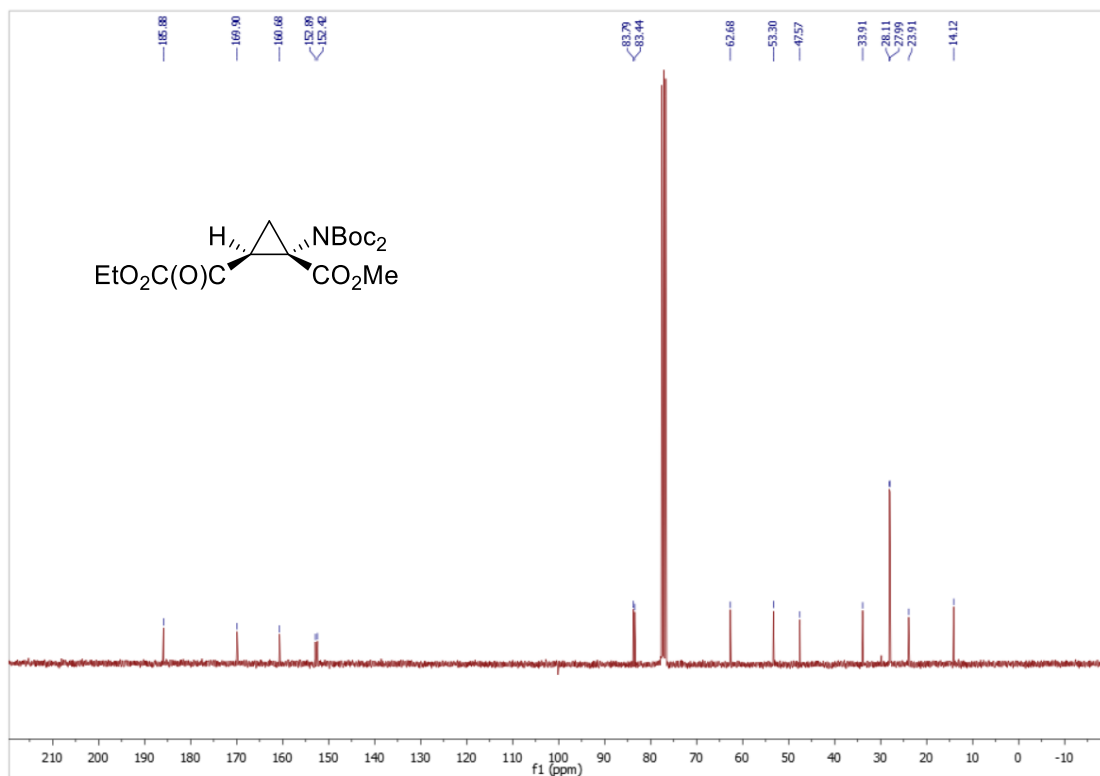




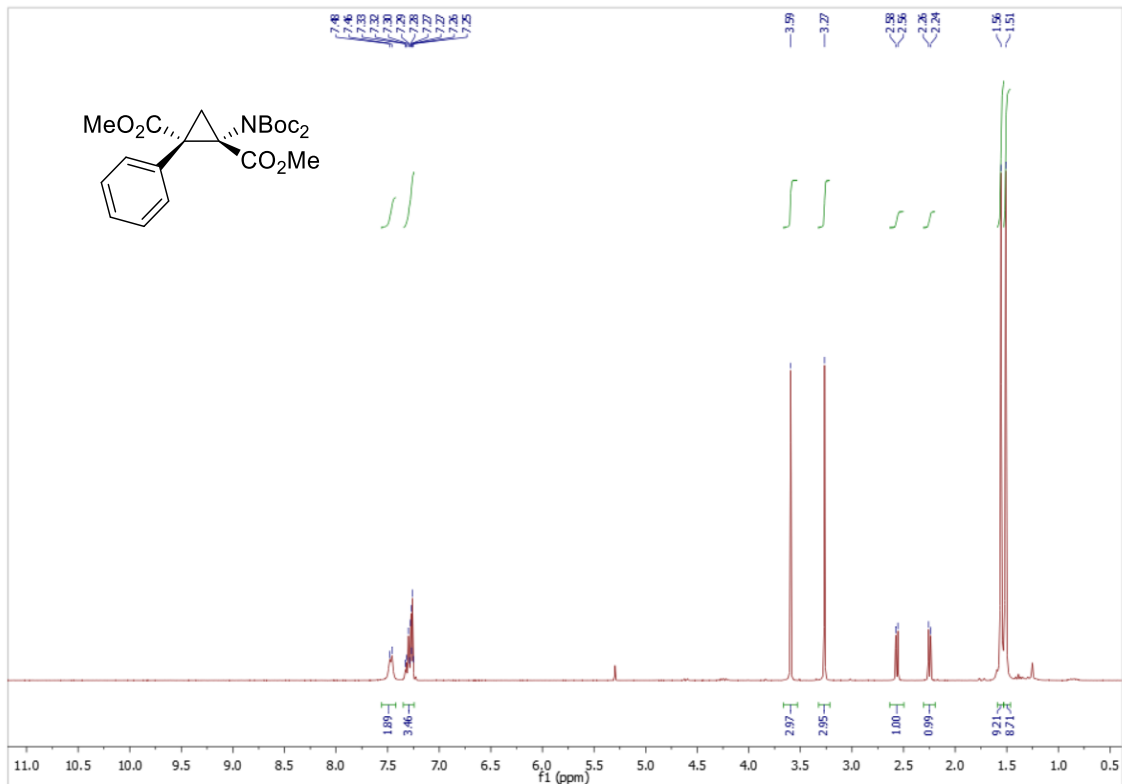
**Compound 4** : methyl (1*S*,2*R*)-1-(bis(tert-butoxycarbonyl)amino)-2-(2-ethoxy-2-oxoacetyl)cyclopropane-1-carboxylate

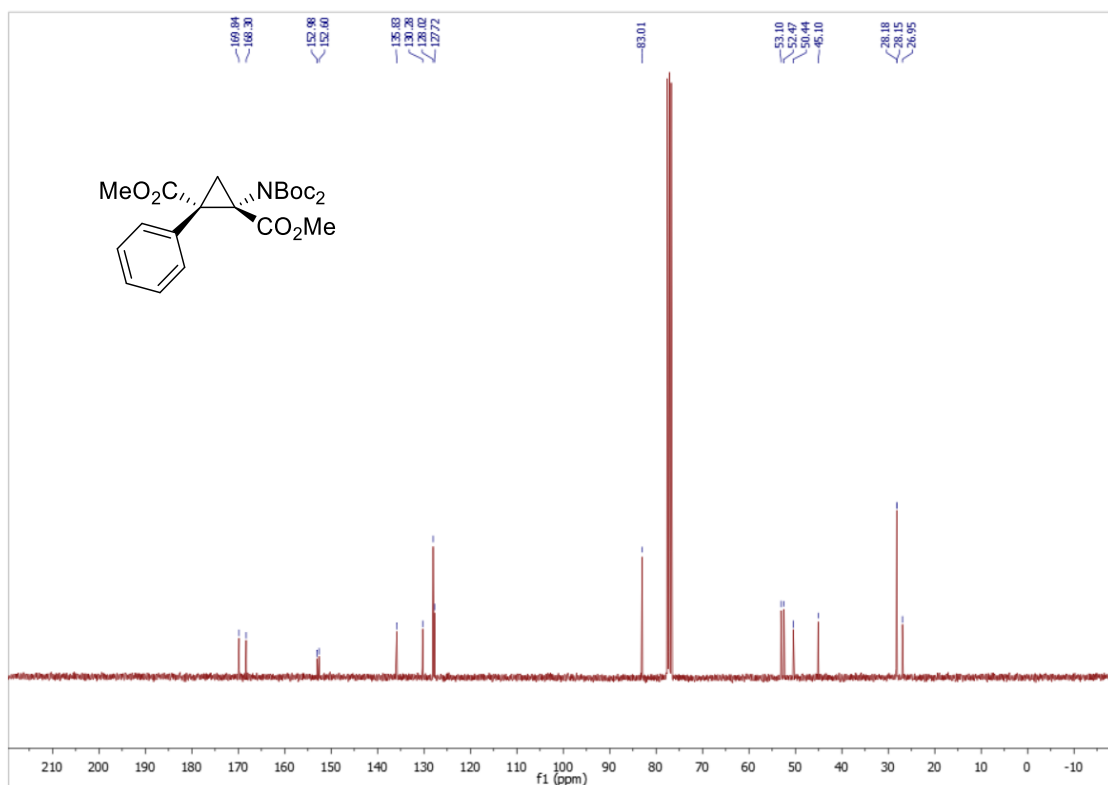




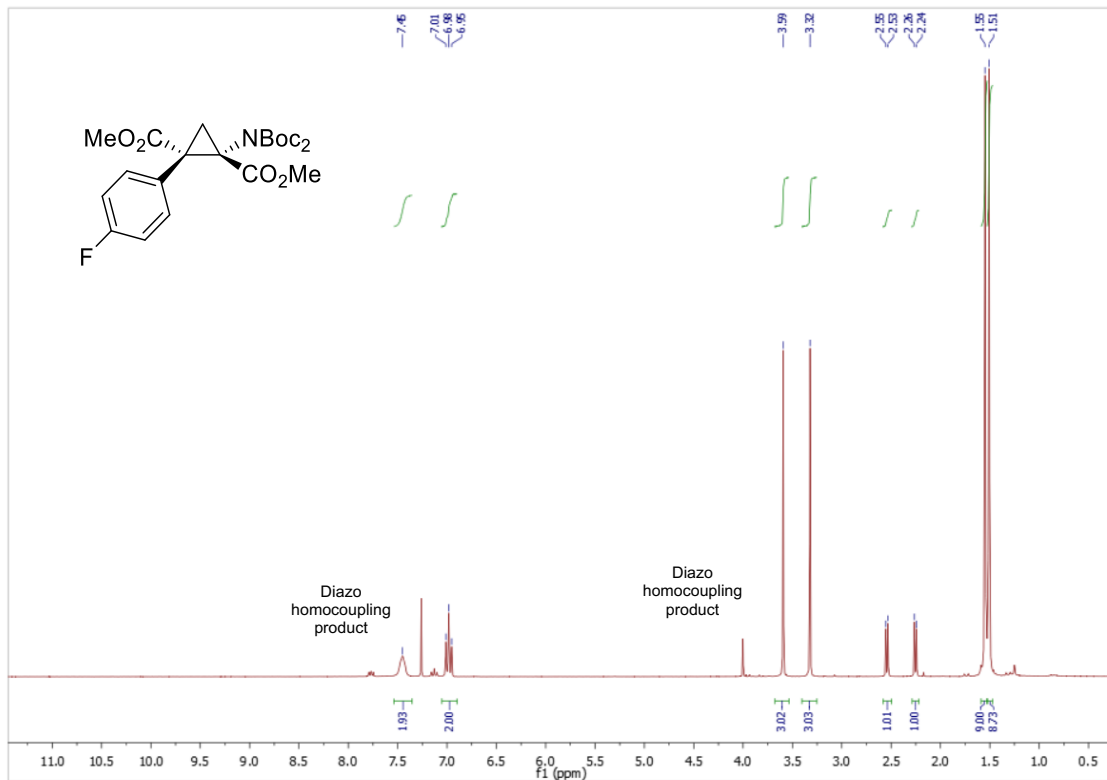


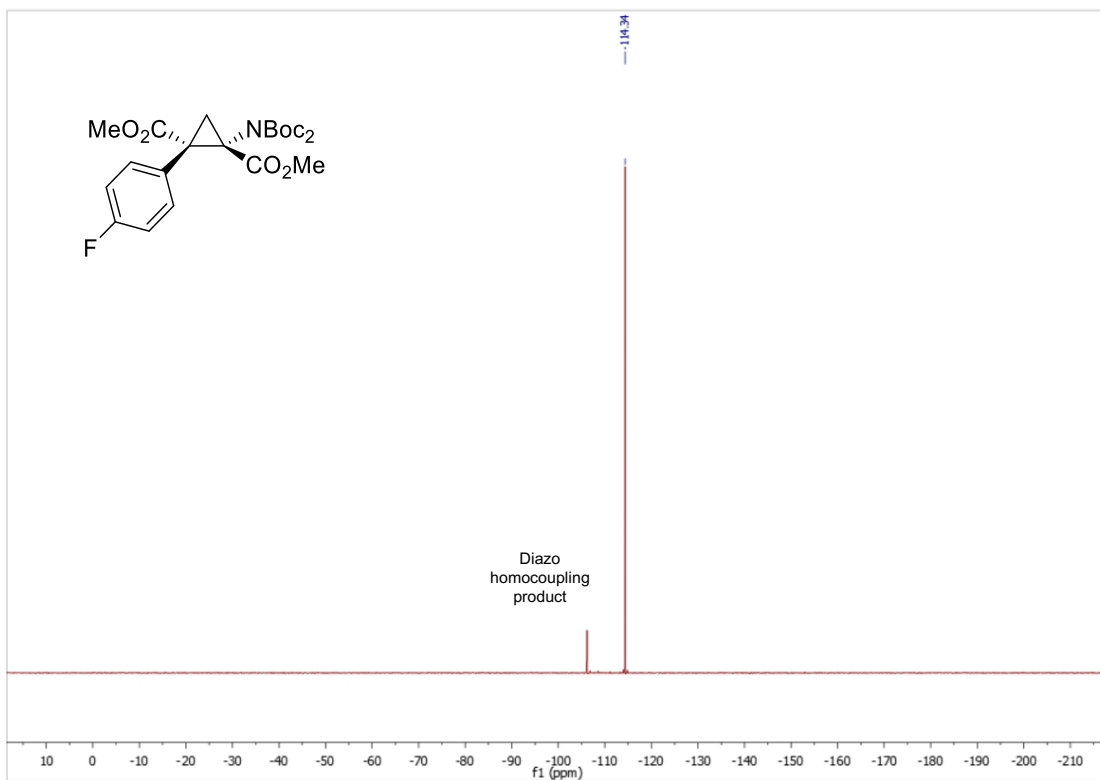
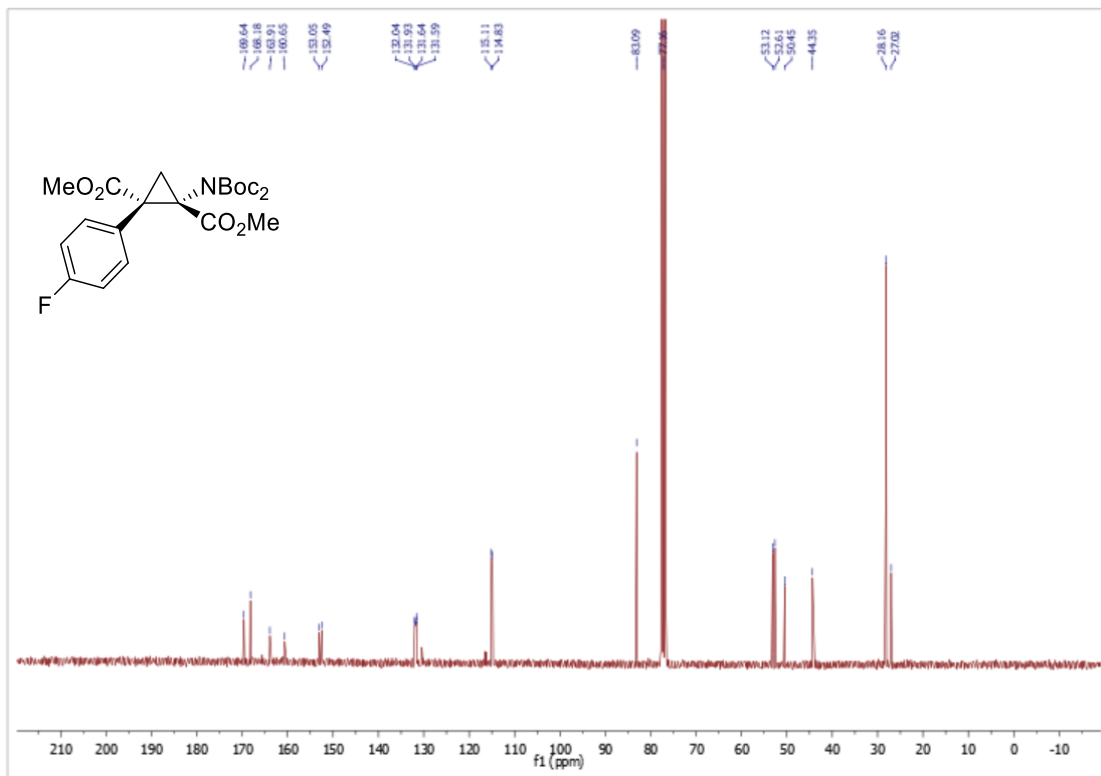
**Compound 5**: dimethyl (1S,2S)-1-(bis(tert-butoxycarbonyl)amino)-2-phenylcyclopropane-1,2-dicarboxylate



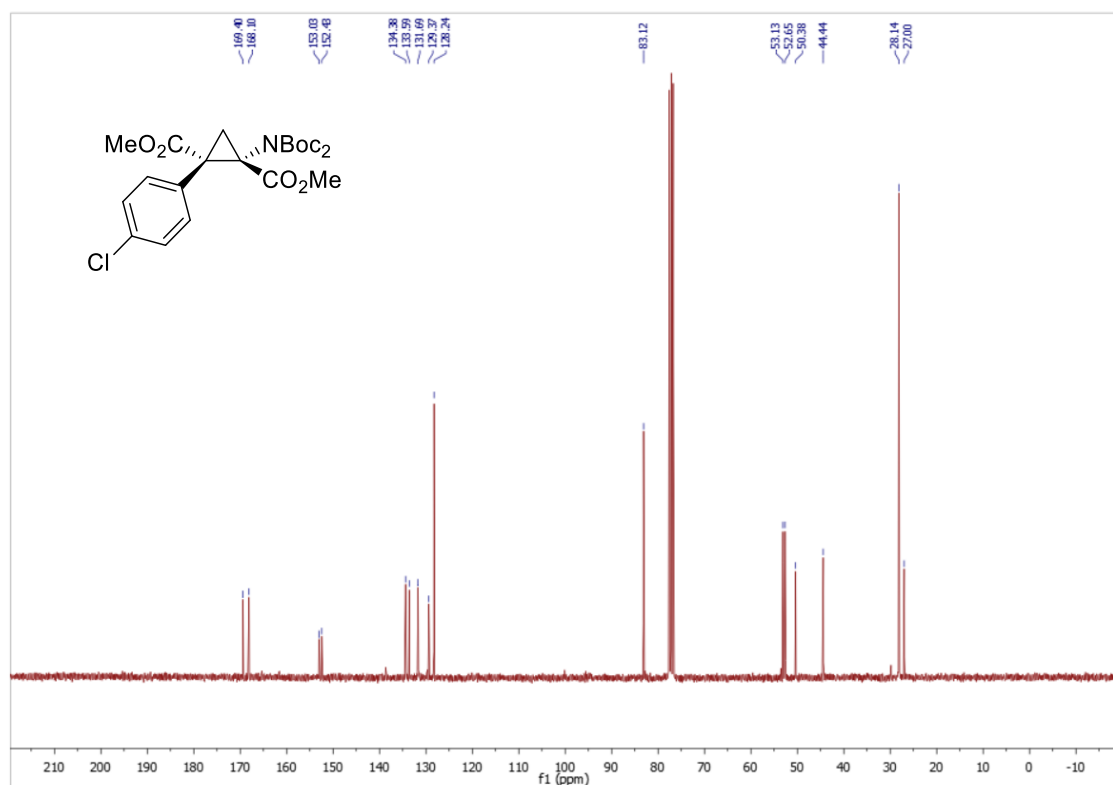
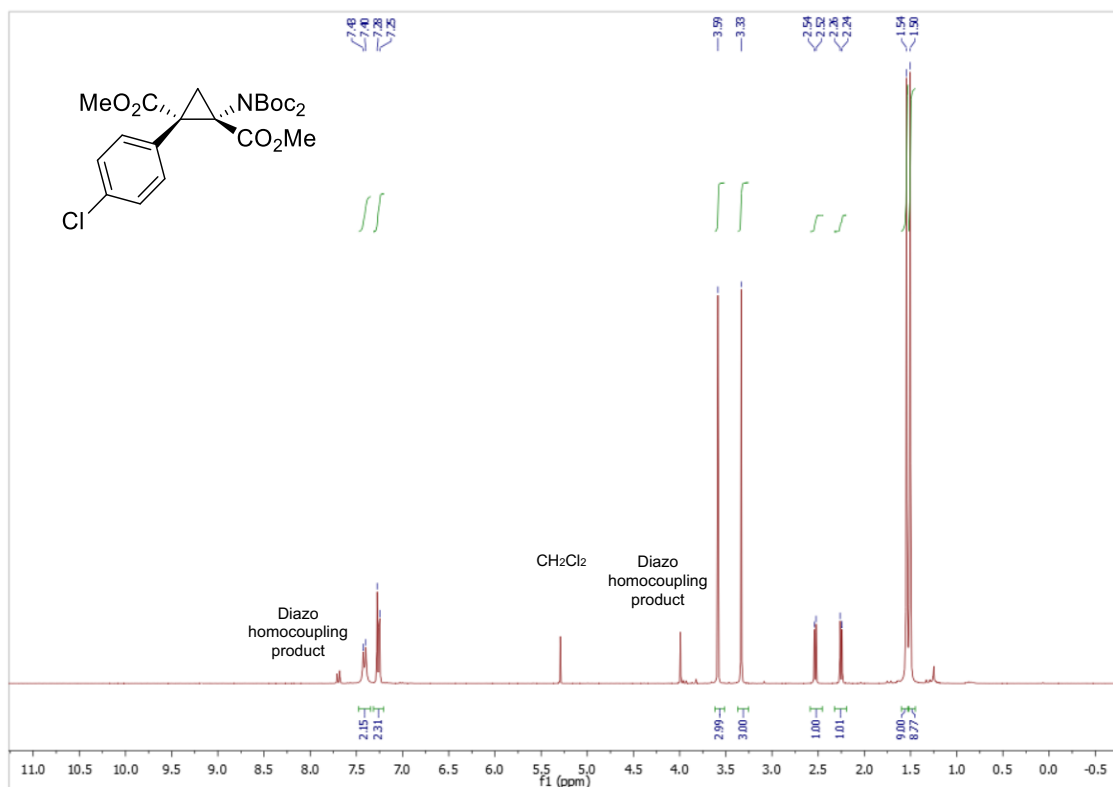


**Compound 6**: dimethyl (1S,2S)-1-(bis(tert-butoxycarbonyl)amino)-2-(4-fluorophenyl)cyclopropane-1,2-dicarboxylate

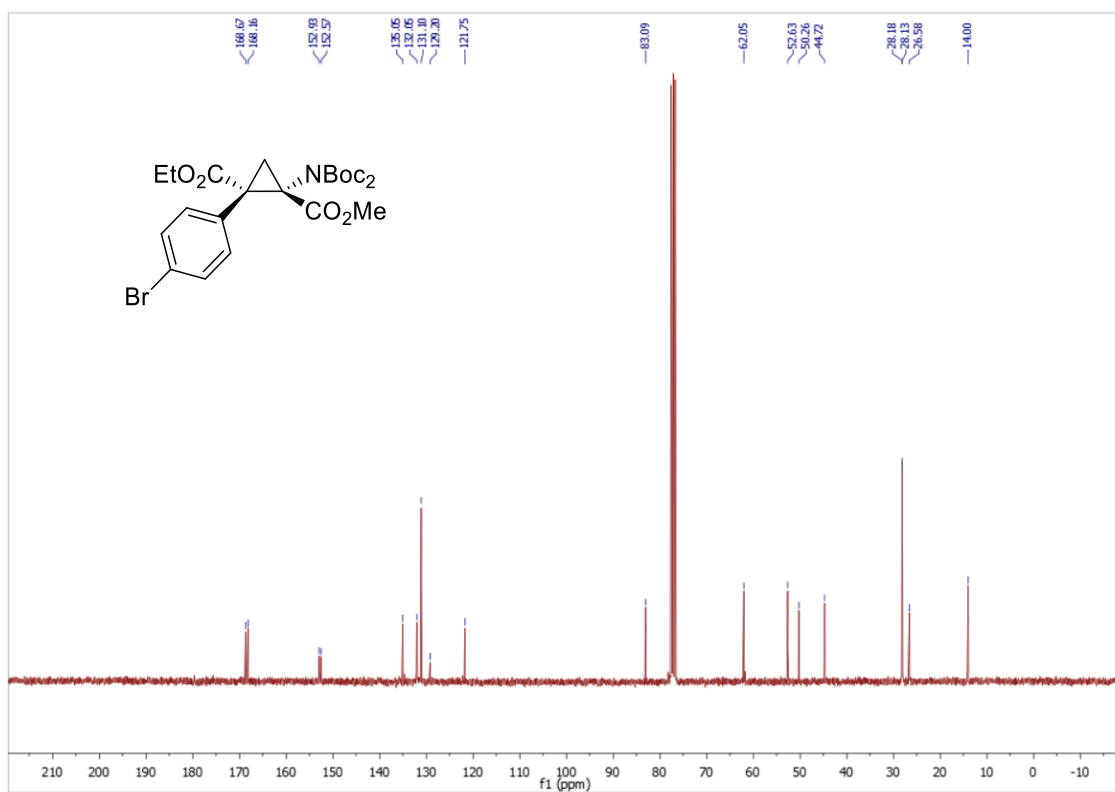
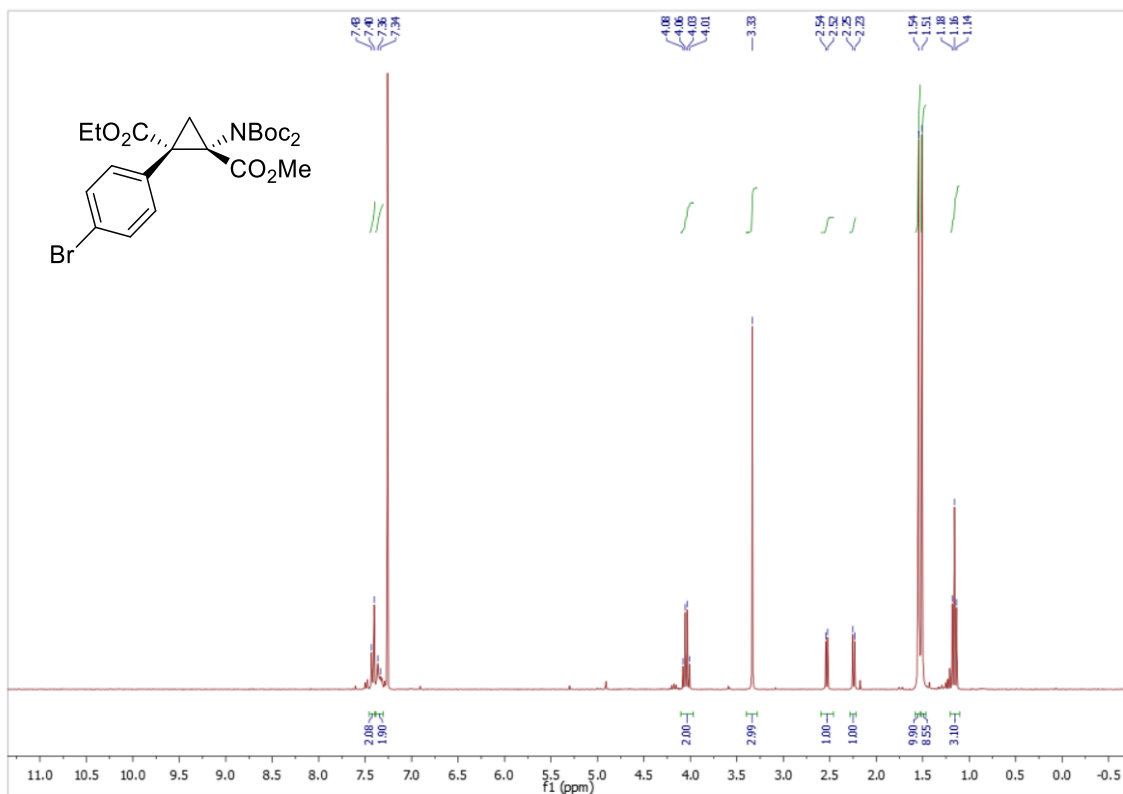




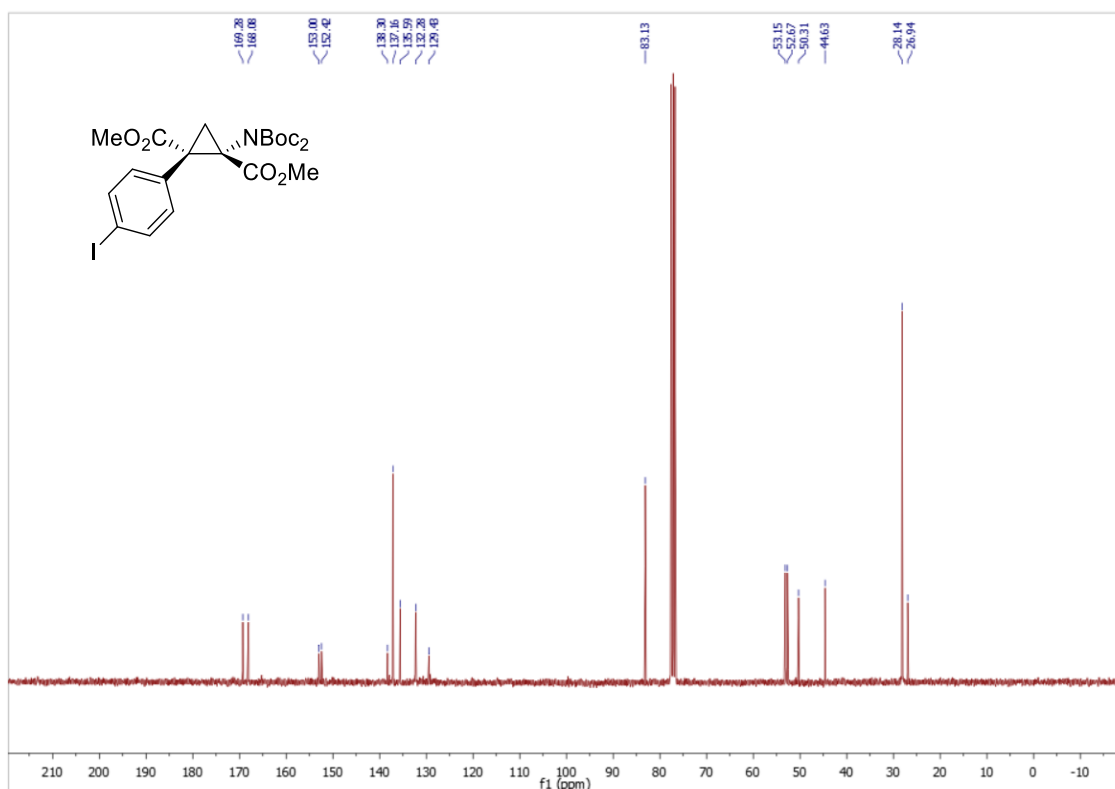
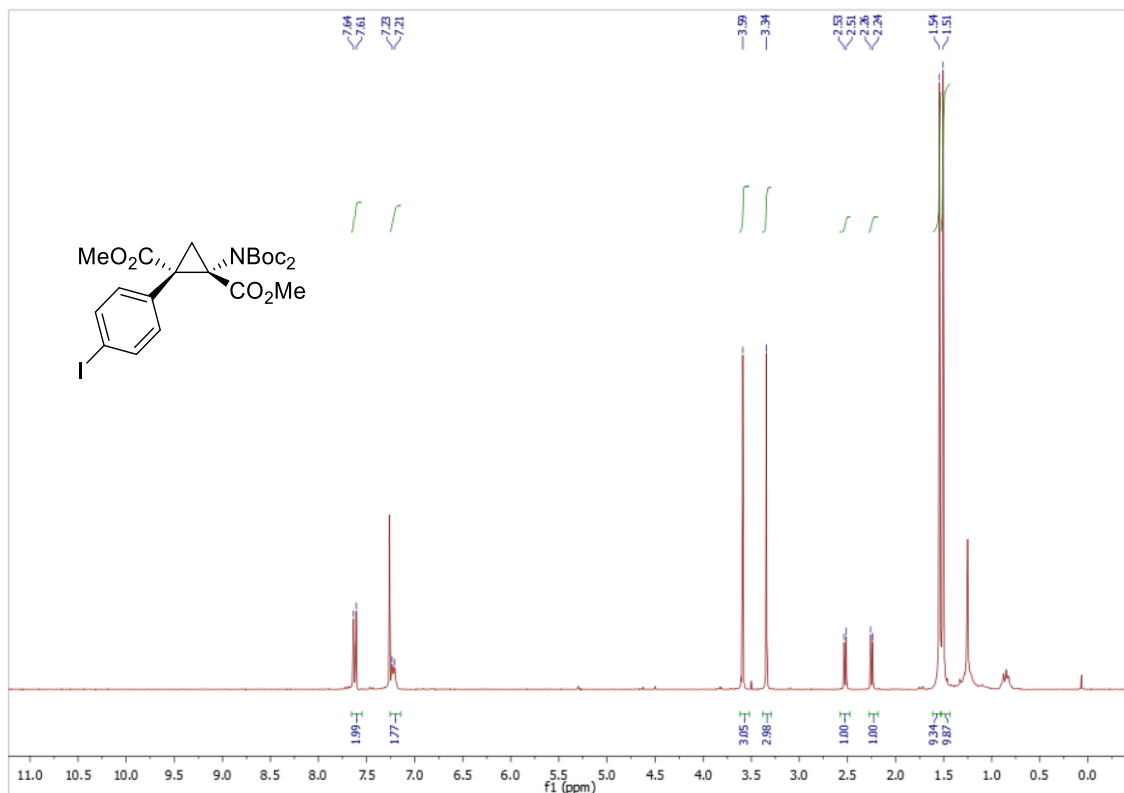
**Compound 7**: dimethyl (1*S*,2*S*)-1-(bis(tert-butoxycarbonyl)amino)-2-(4-chlorophenyl)cyclopropane-1,2-dicarboxylate



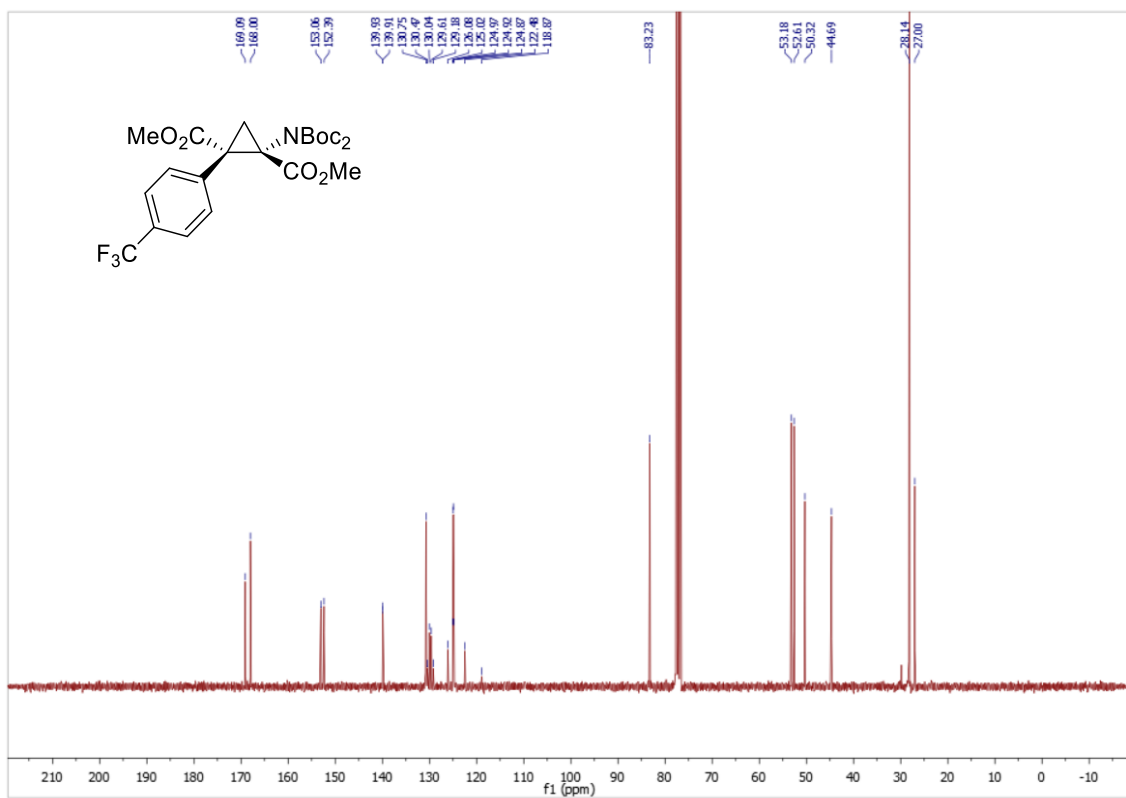
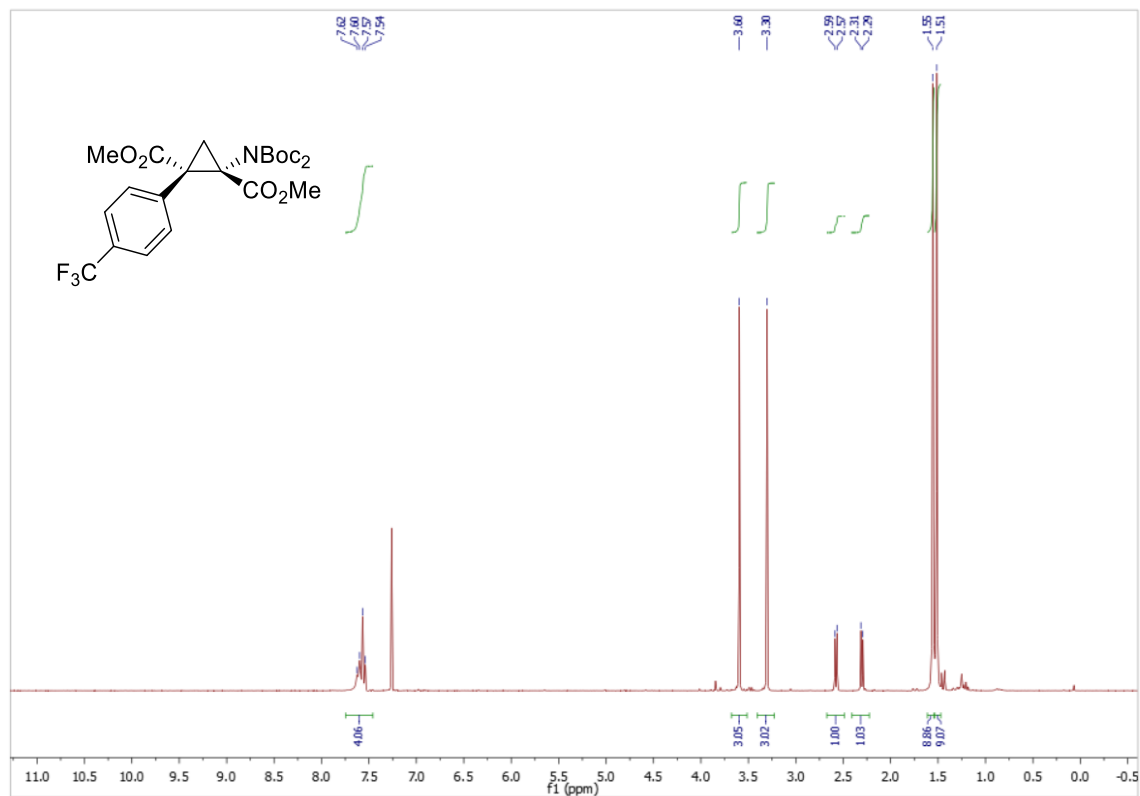
**Compound 8**: 1-ethyl 2-methyl (1S,2S)-1-(4-bromophenyl)-2-(bis(tert-butoxycarbonyl)amino)cyclopropane-1,2-dicarboxylate

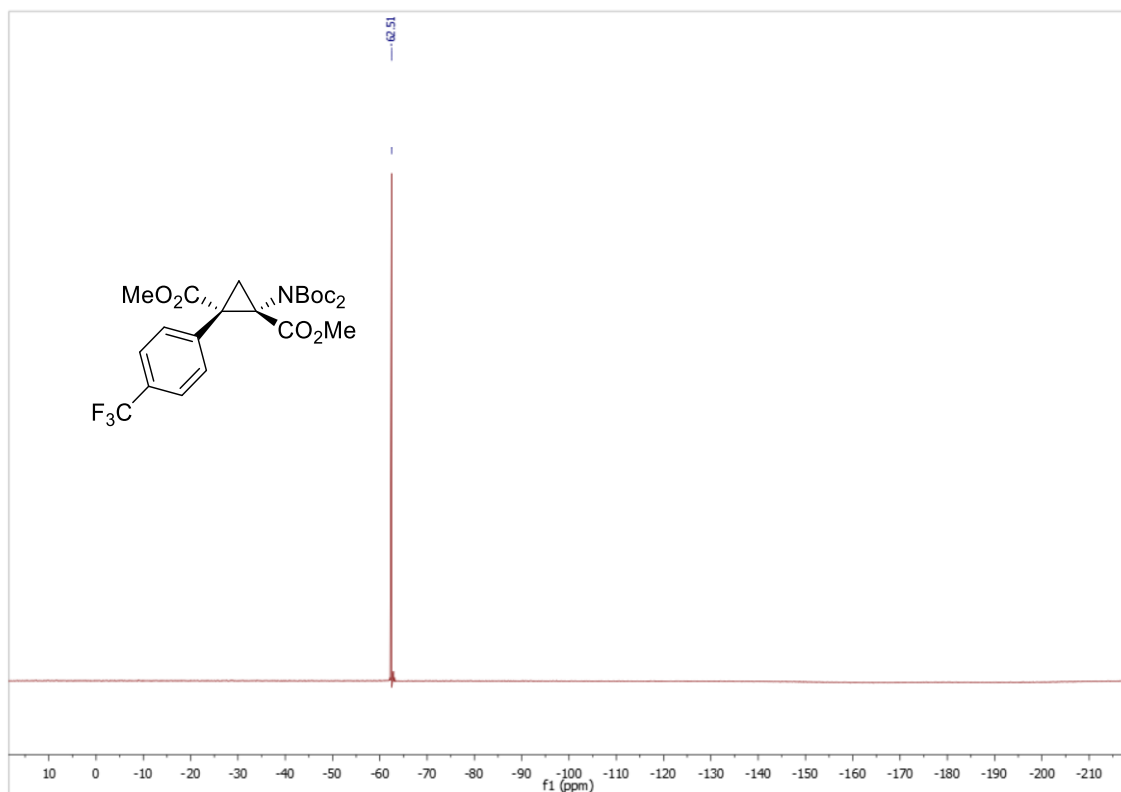


**Compound 9**: dimethyl (1*S*,2*S*)-1-(bis(tert-butoxycarbonyl)amino)-2-(4-iodophenyl)cyclopropane-1,2-dicarboxylate

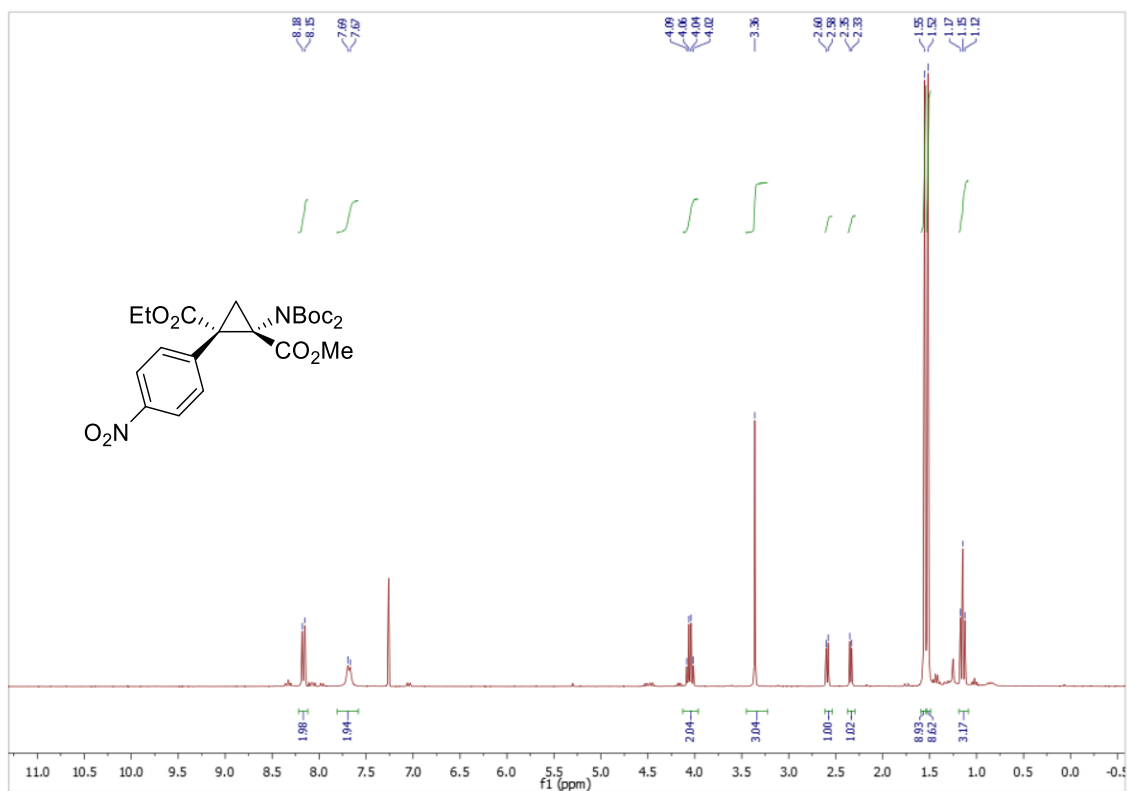


**Compound 10** : dimethyl (1*S*,2*S*)-1-(bis(tert-butoxycarbonyl)amino)-2-(4-(trifluoromethyl)phenyl)cyclopropane-1,2-dicarboxylate

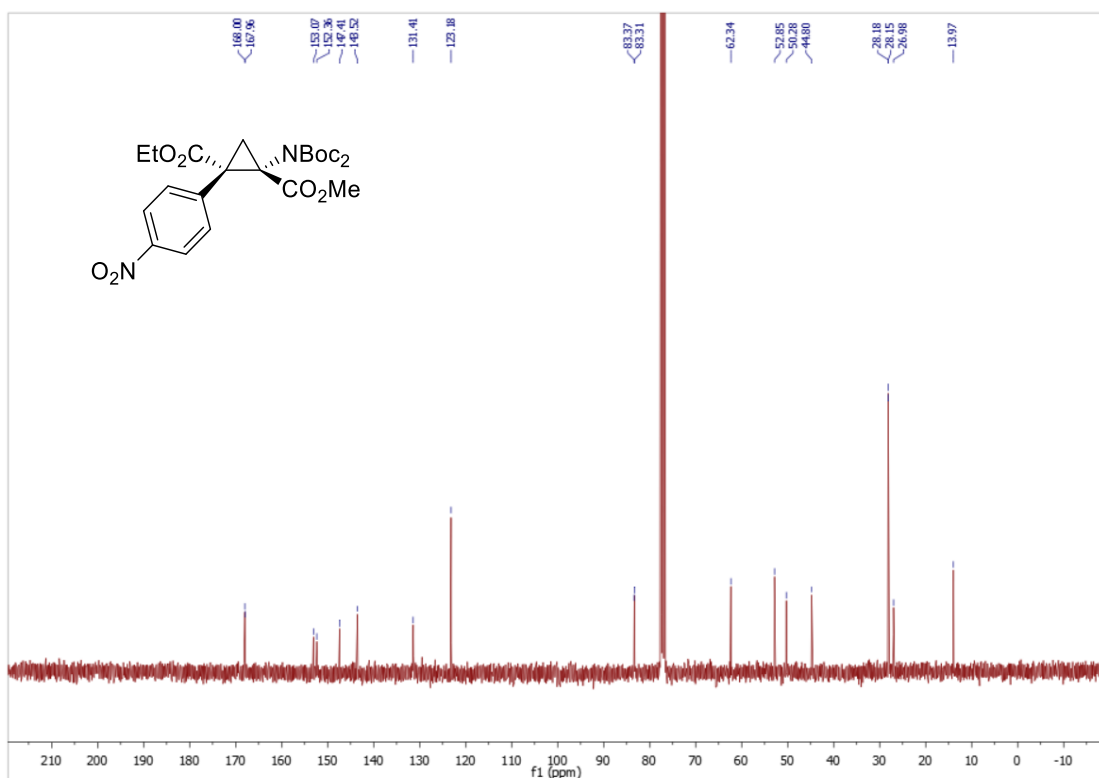




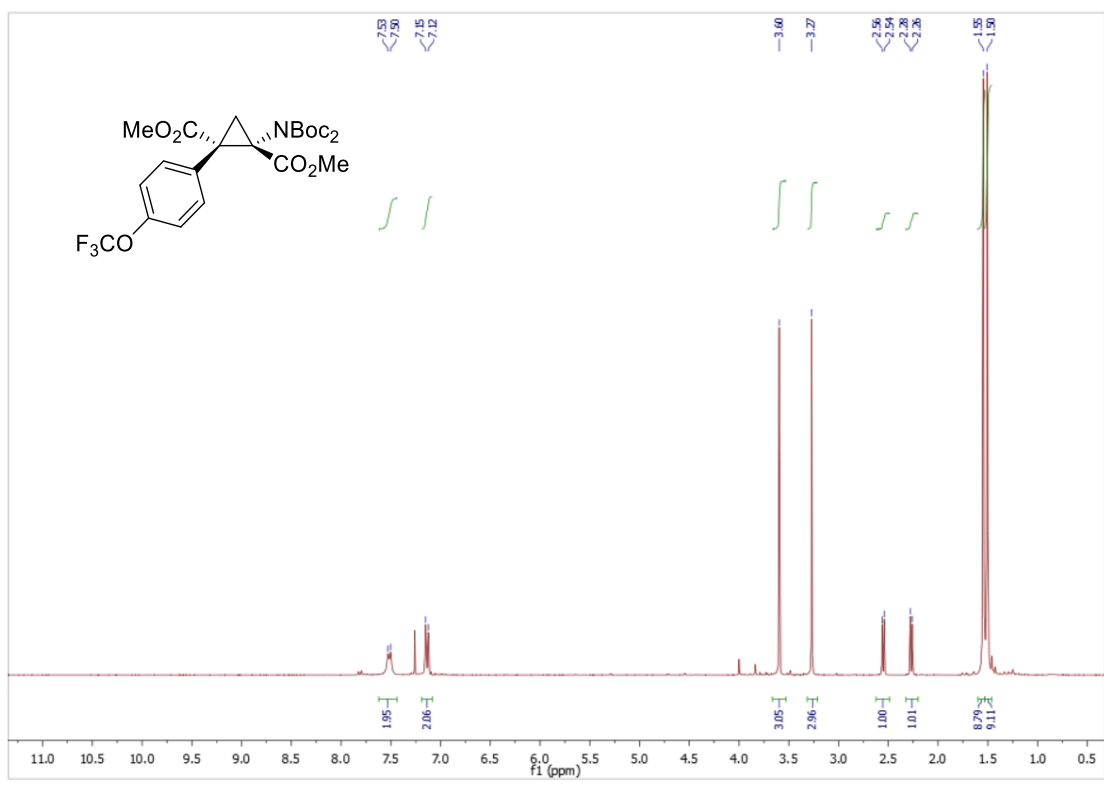
**Compound 11**: 2-ethyl 1-methyl (1*S*,2*S*)-1-(bis(tert-butoxycarbonyl)amino)-2-(4-nitrophenyl)cyclopropane-1,2-dicarboxylate

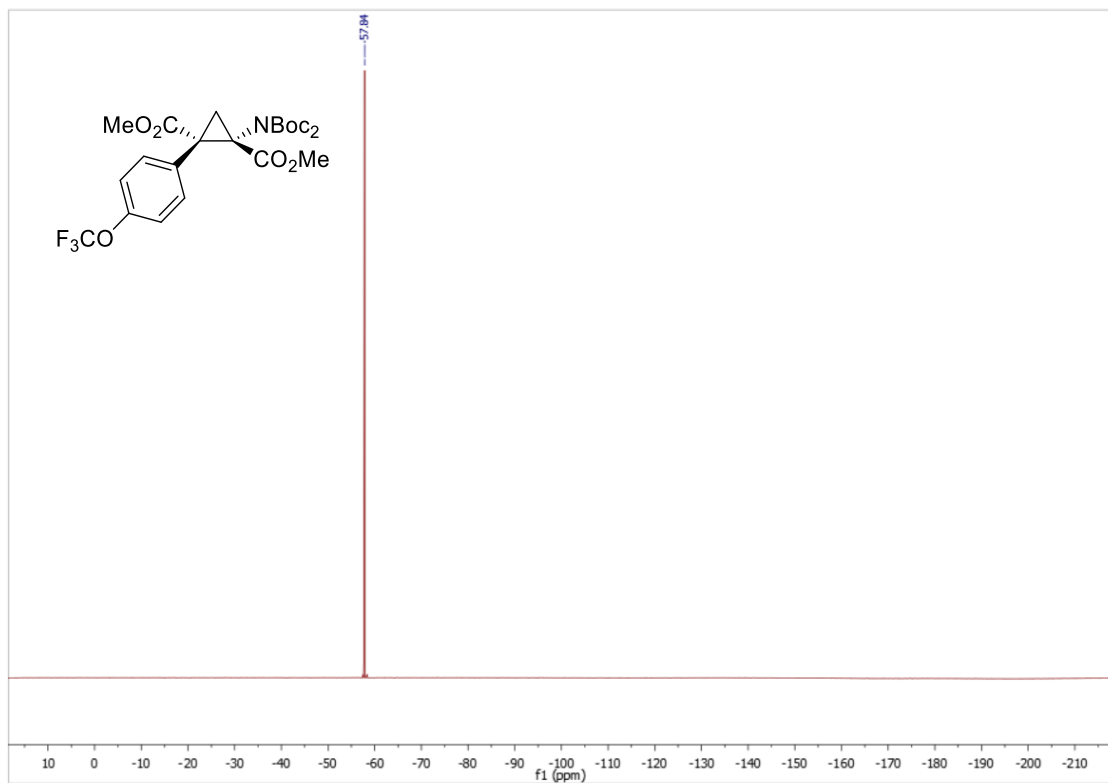
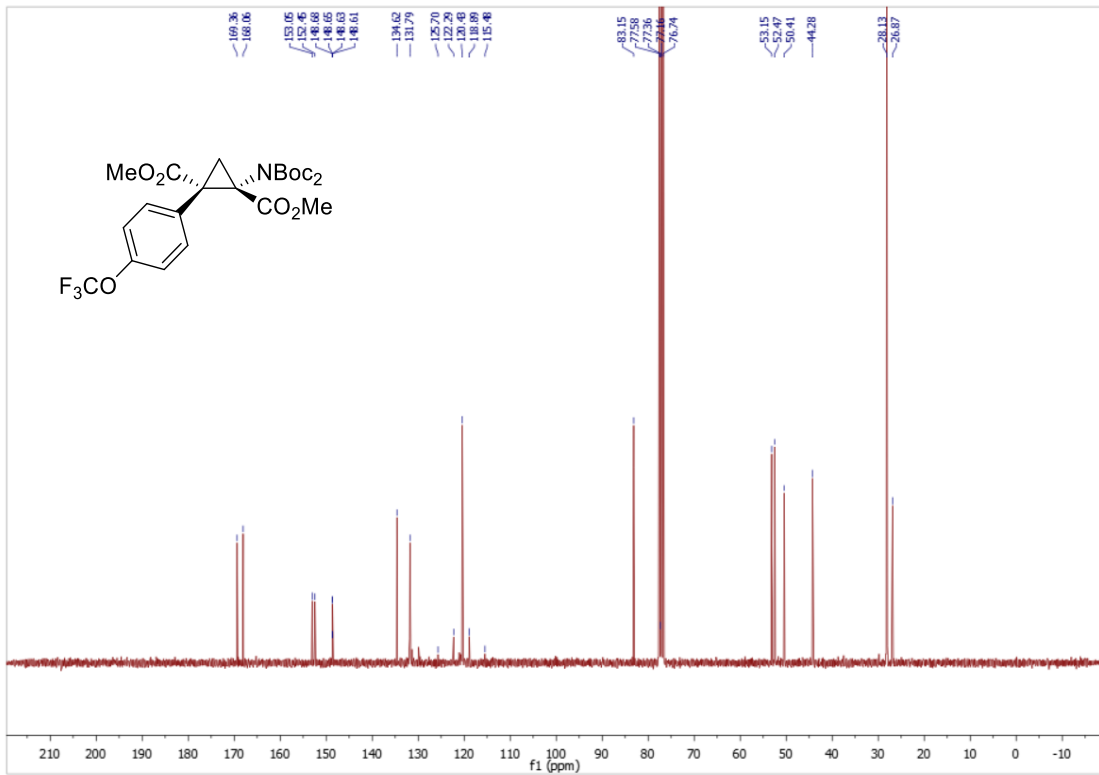




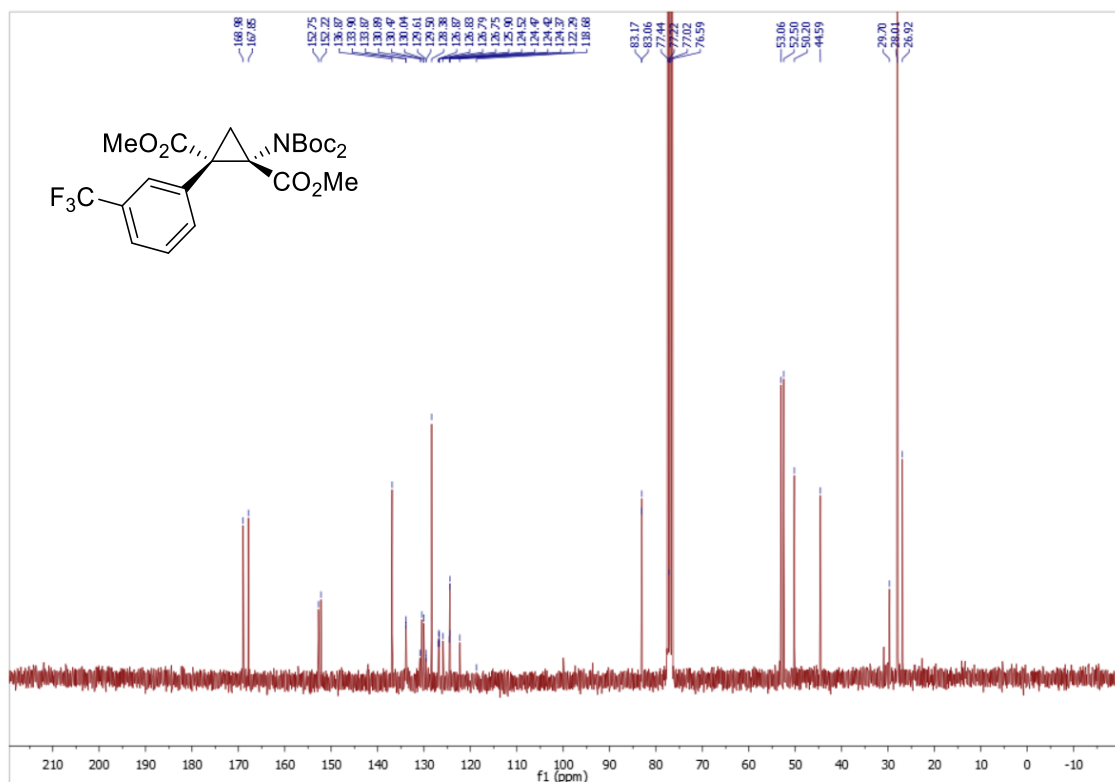
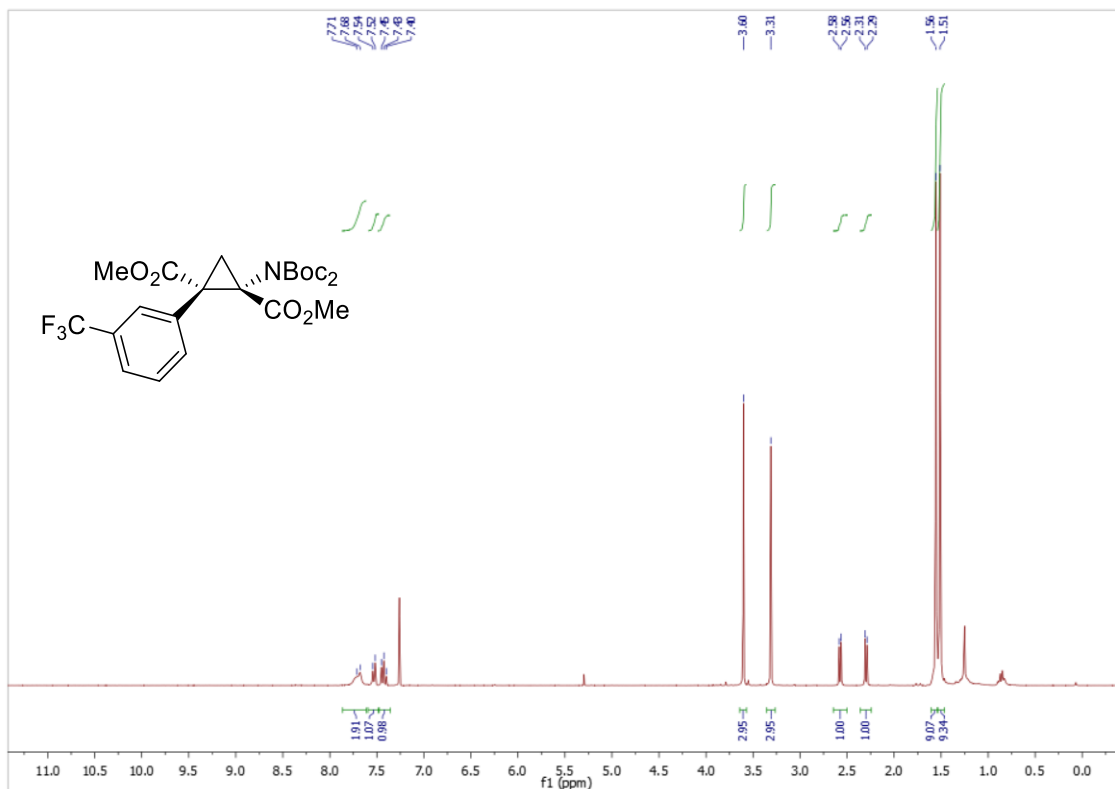


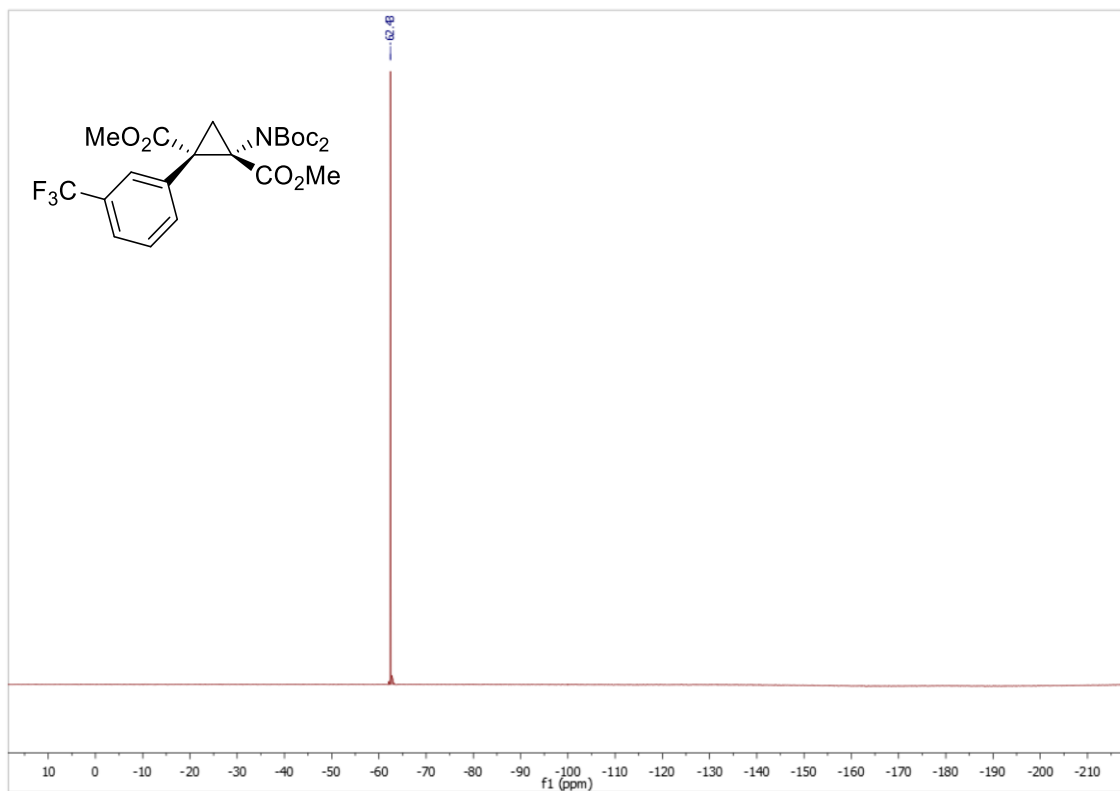
**Compound 12:** dimethyl (1S,2S)-1-(bis(tert-butoxycarbonyl)amino)-2-(4-(trifluoromethoxy)phenyl)cyclopropane-1,2-dicarboxylate



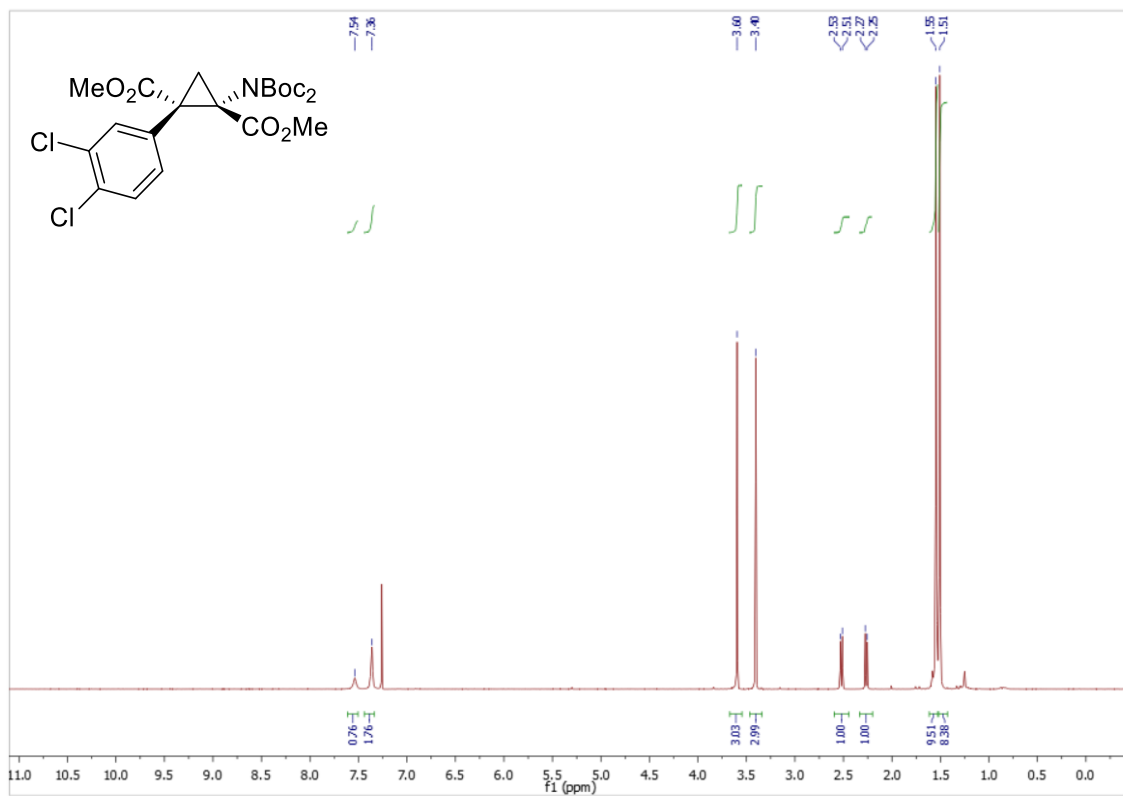


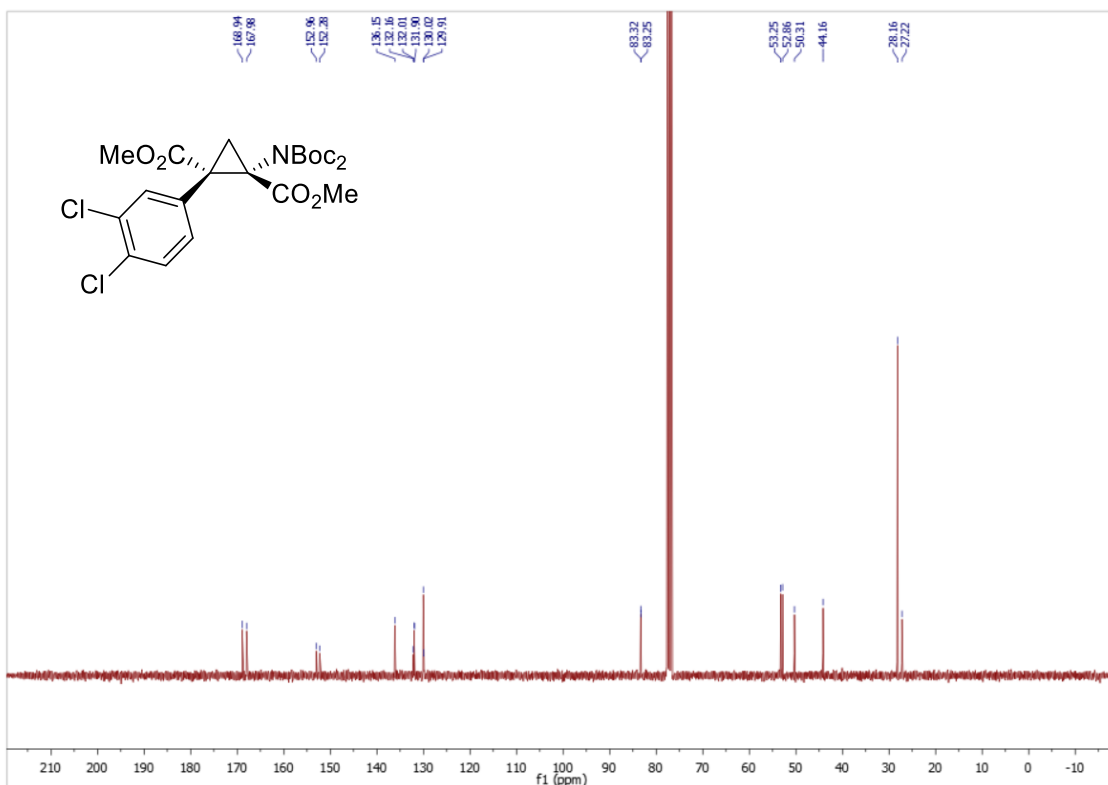
**Compound 13**: dimethyl (1*S*,2*S*)-1-(bis(tert-butoxycarbonyl)amino)-2-(3-(trifluoromethyl)phenyl)cyclopropane-1,2-dicarboxylate



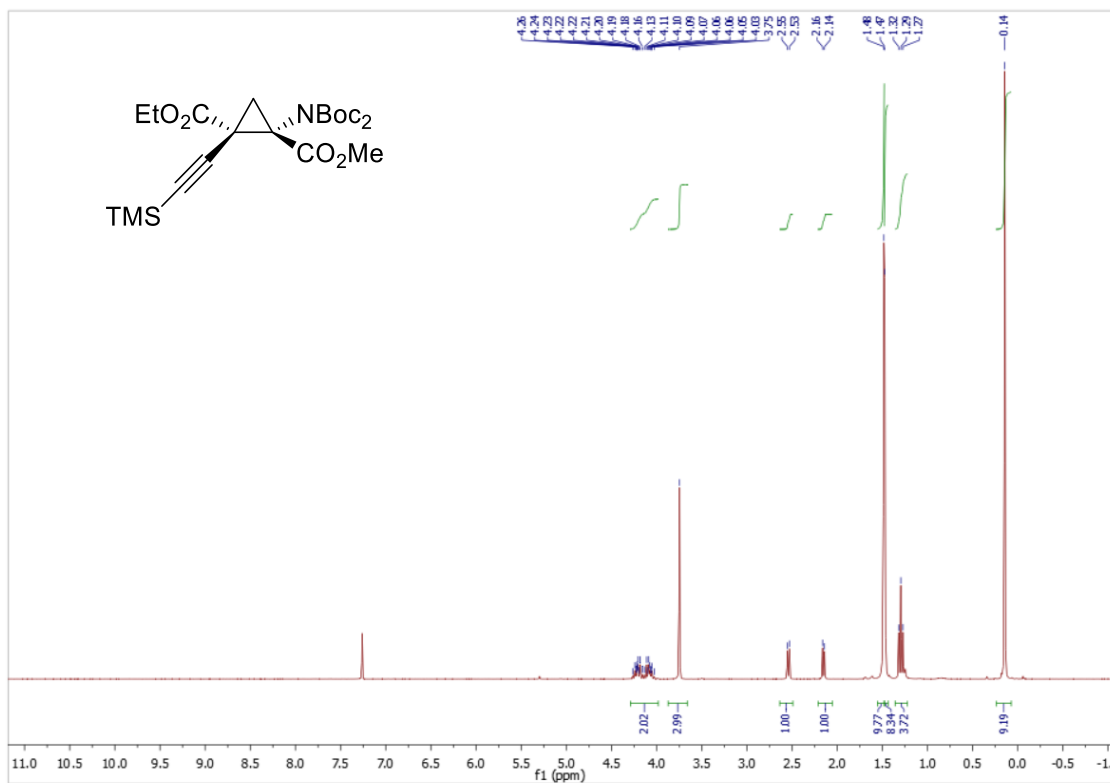


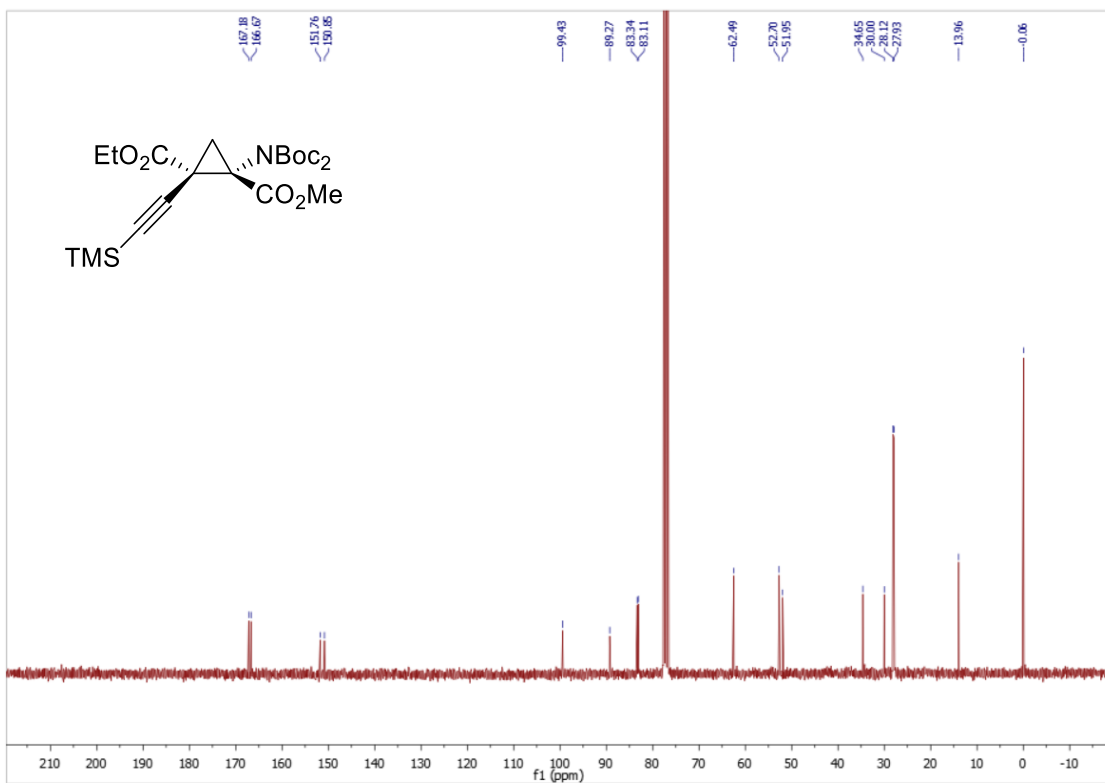
**Compound 14**: dimethyl (1*S*,2*S*)-1-(bis(tert-butoxycarbonyl)amino)-2-(3,4-dichlorophenyl)cyclopropane-1,2-dicarboxylate



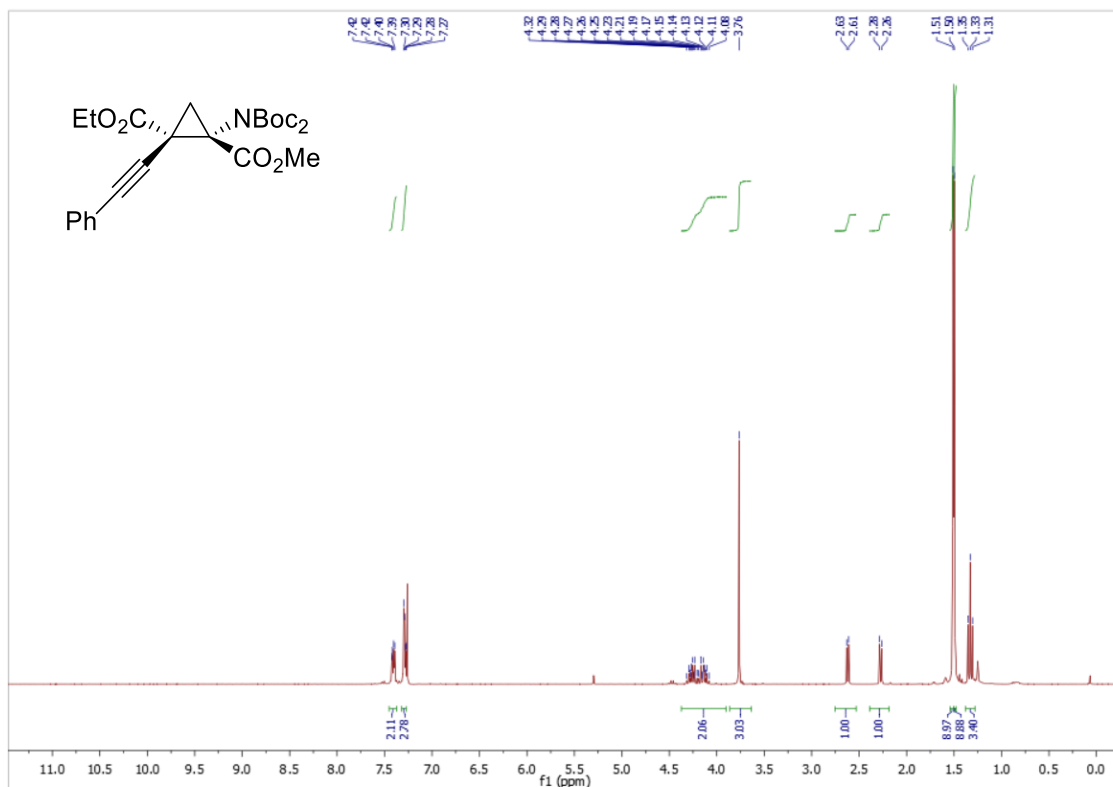


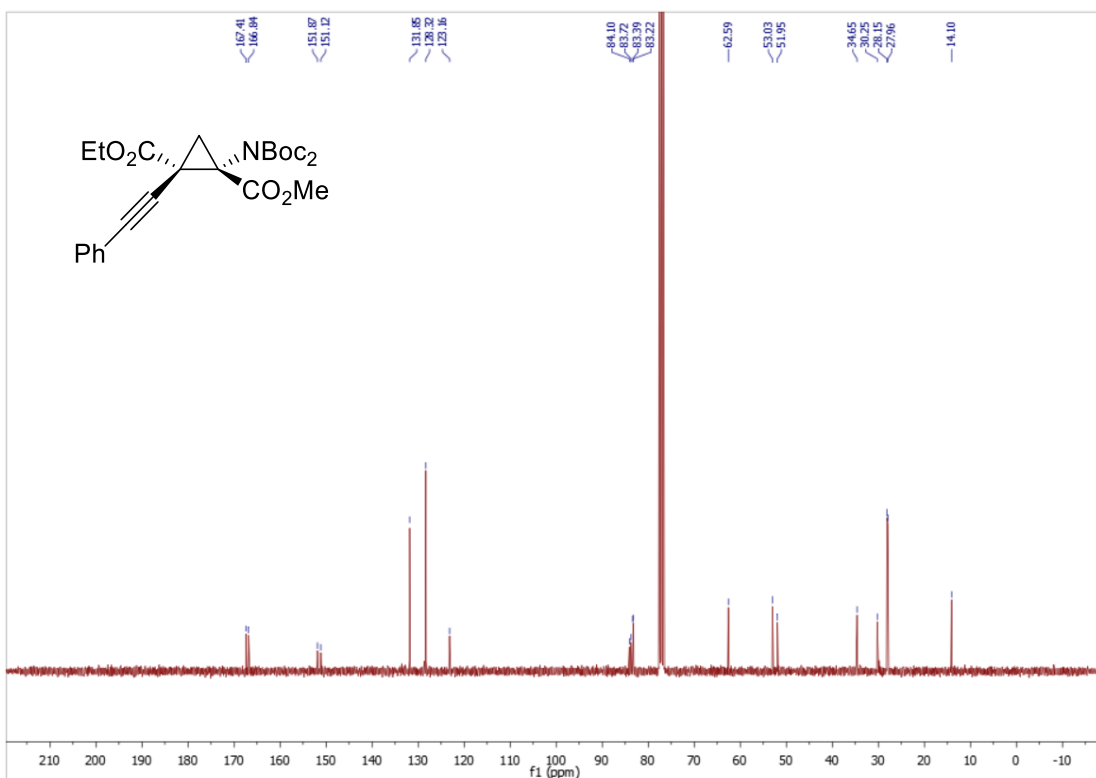
**Compound 15**: 2-ethyl 1-methyl (1*S*,2*S*)-1-(bis(tert-butoxycarbonyl)amino)-2-((trimethylsilyl)ethynyl)cyclopropane-1,2-dicarboxylate



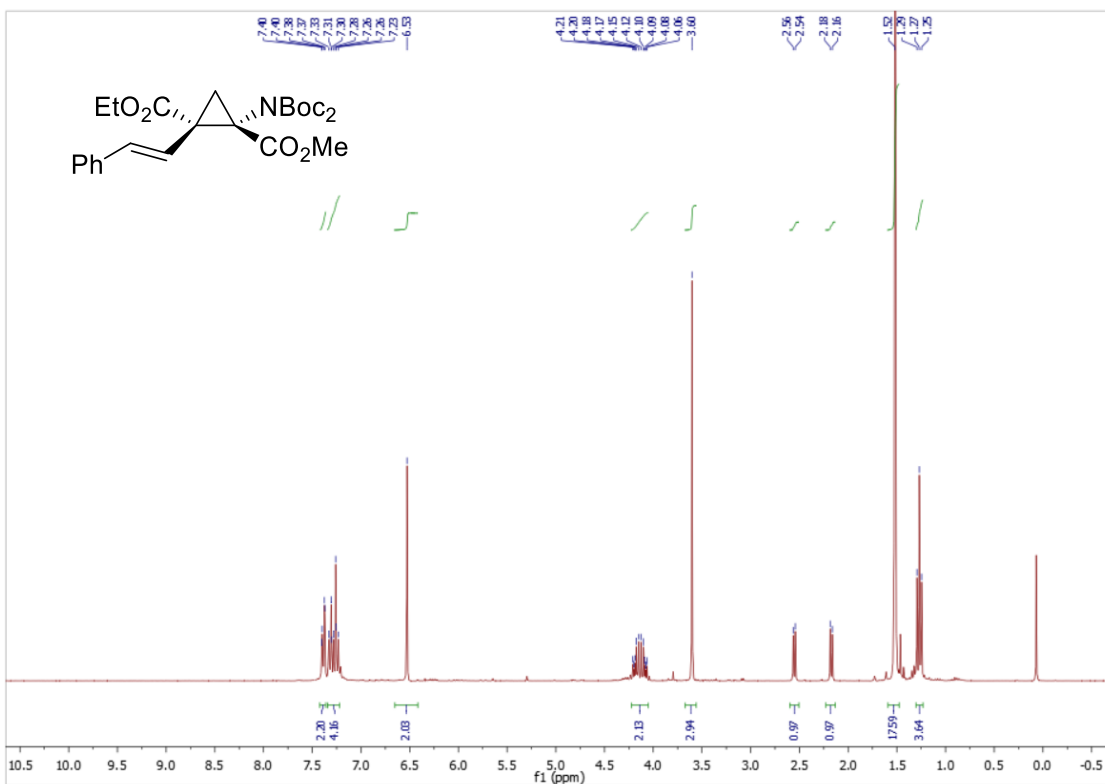


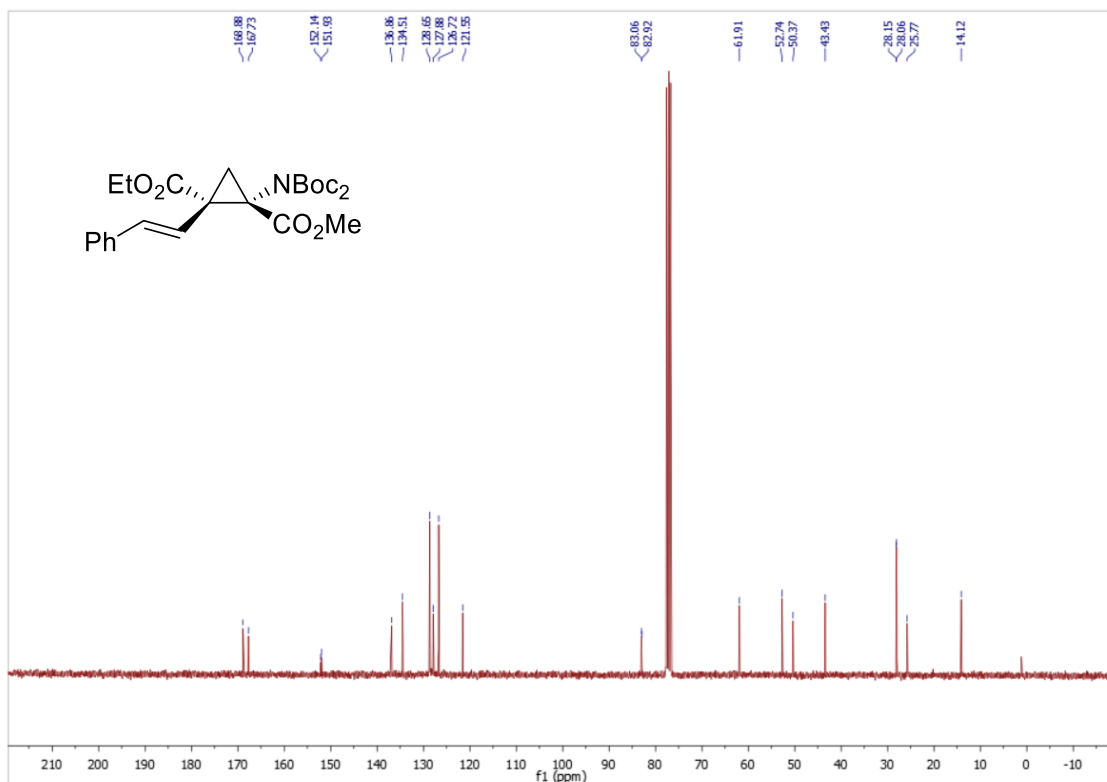
**Compound 16**: 2-ethyl 1-methyl (1*S*,2*S*)-1-(bis(tert-butoxycarbonyl)amino)-2-(phenylethynyl)cyclopropane-1,2-dicarboxylate



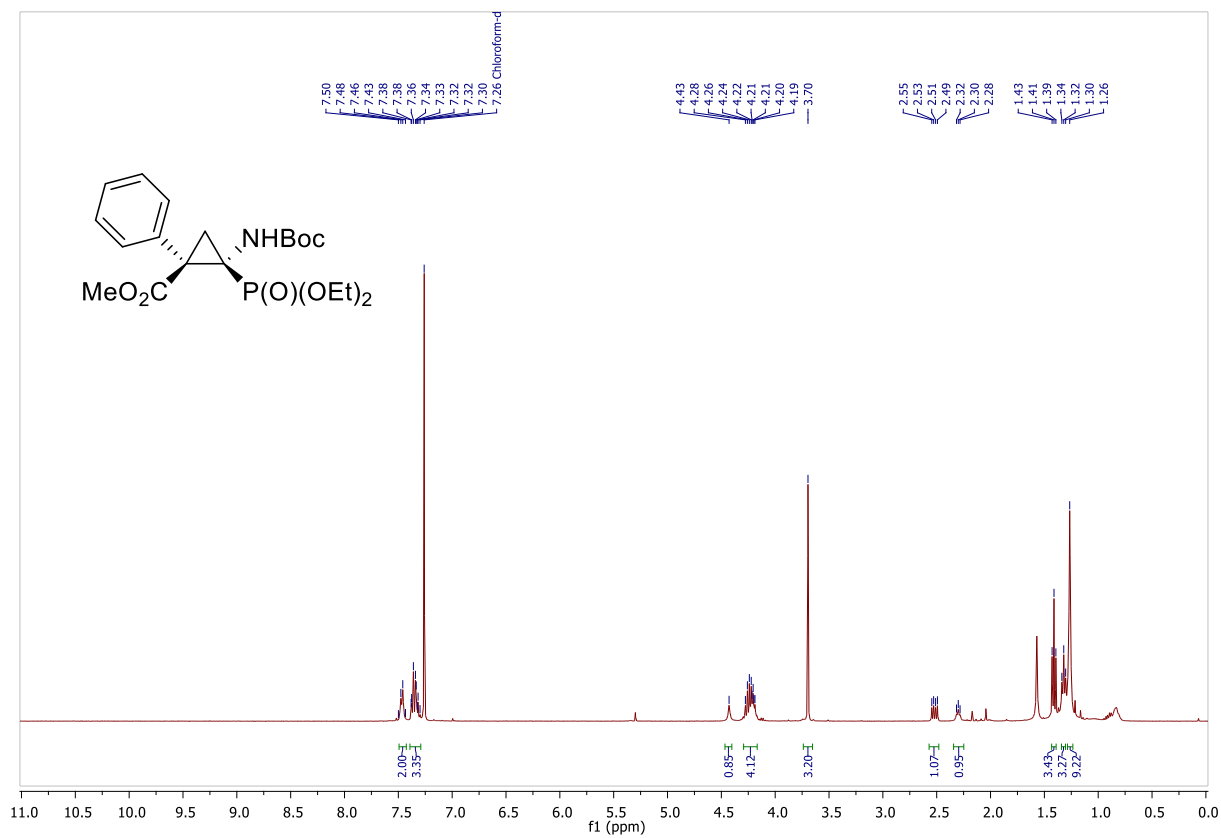


**Compound 17**: 2-ethyl 1-methyl (1*S*,2*S*)-1-(bis(tert-butoxycarbonyl)amino)-2-((*E*)-styryl)cyclopropane-1,2-dicarboxylate

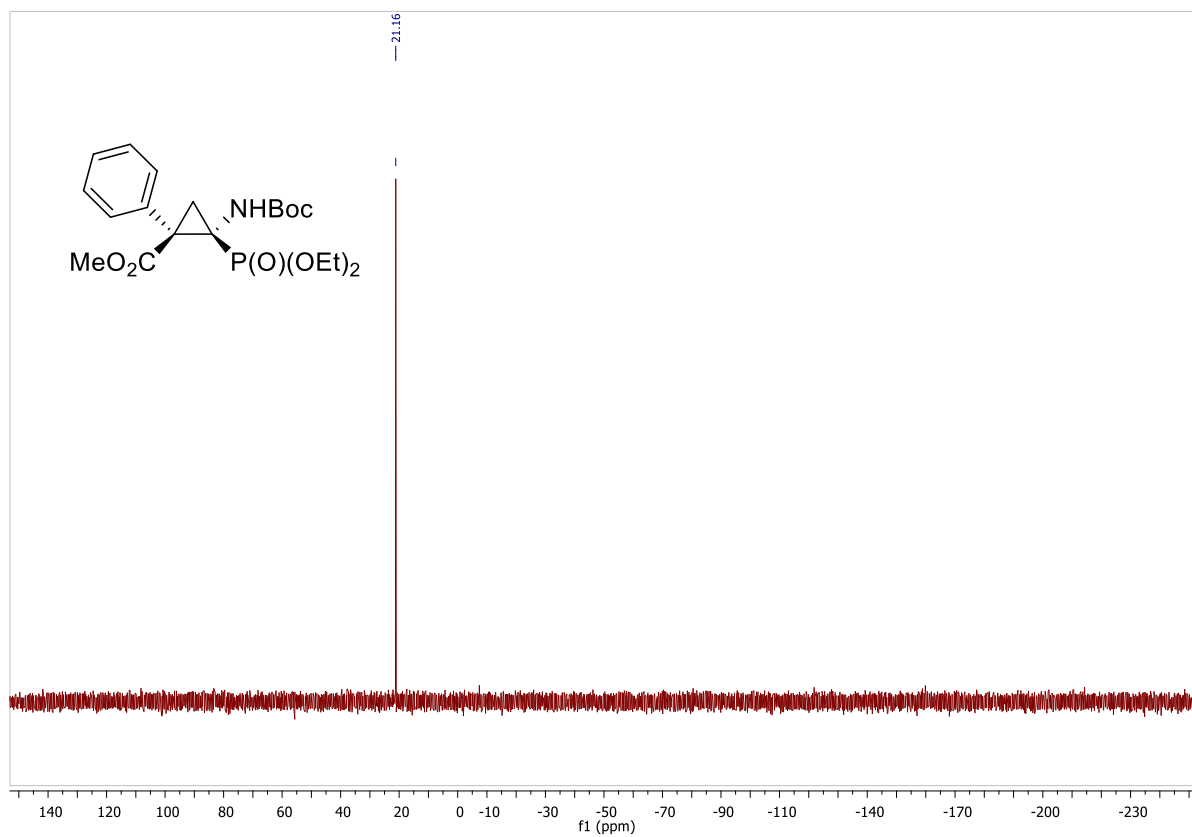
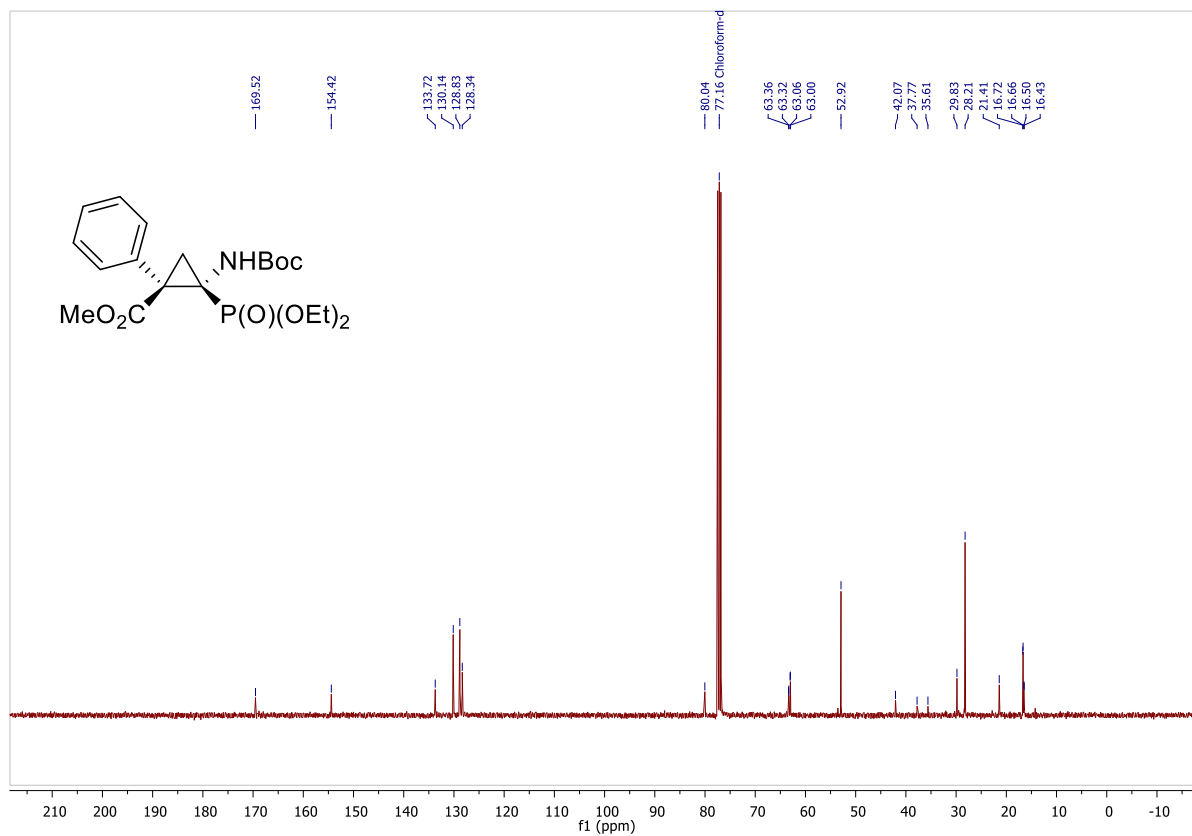




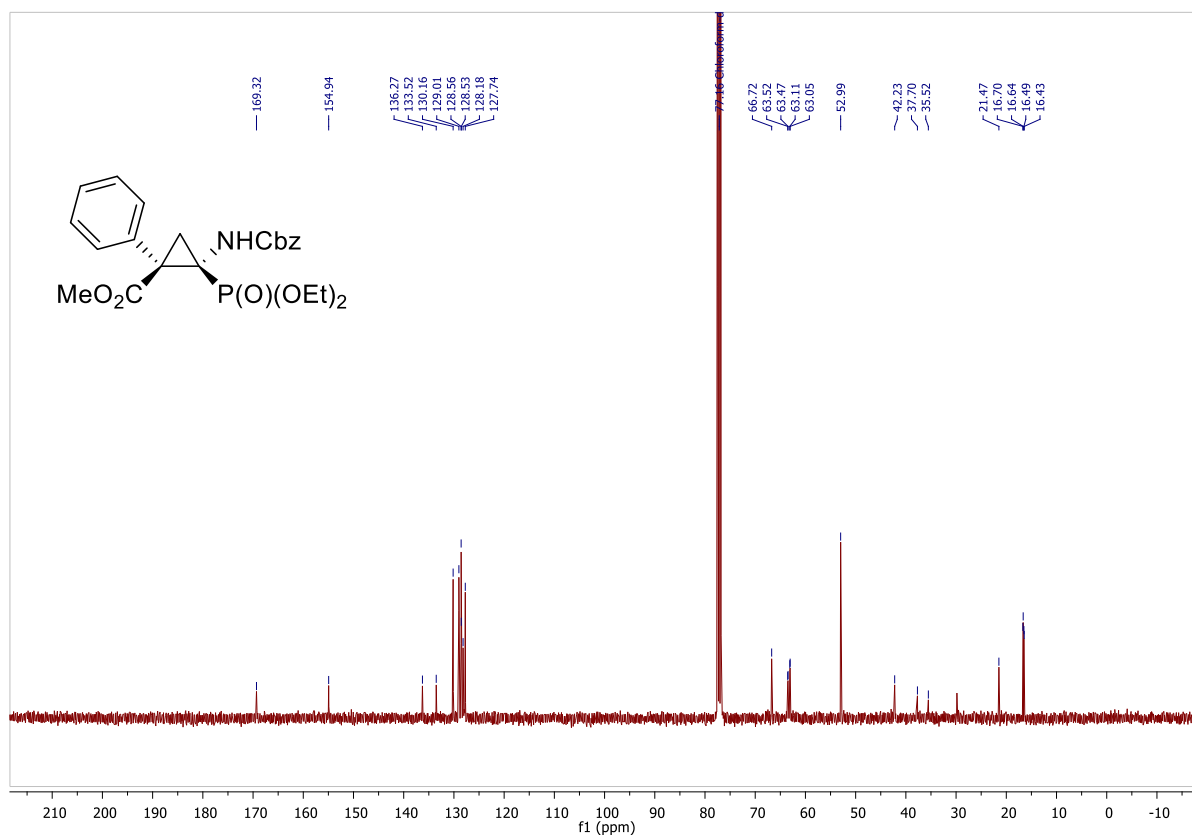
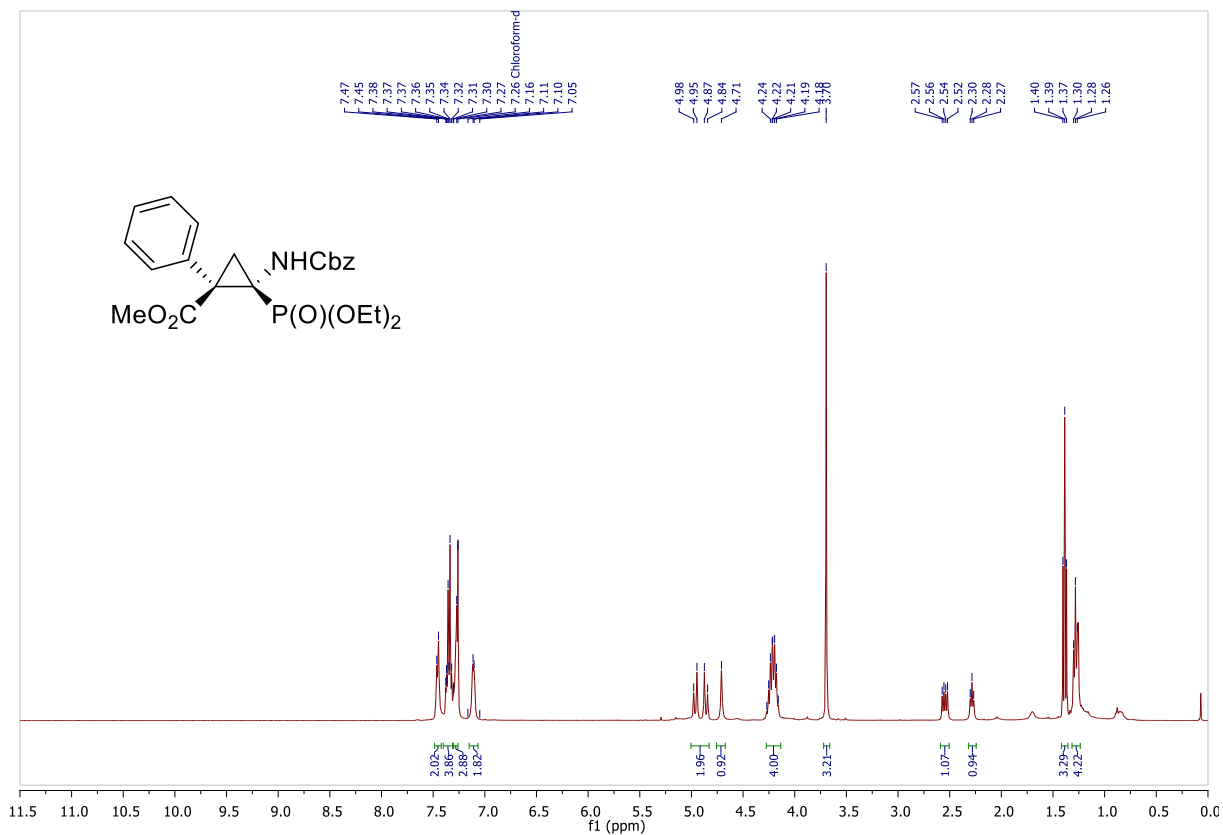
**Compound 18** : methyl (1*S*,2*R*)-2-((tert-butoxycarbonyl)amino)-2-(diethoxyphosphoryl)-1-phenyl cyclopropane-1-carboxylate

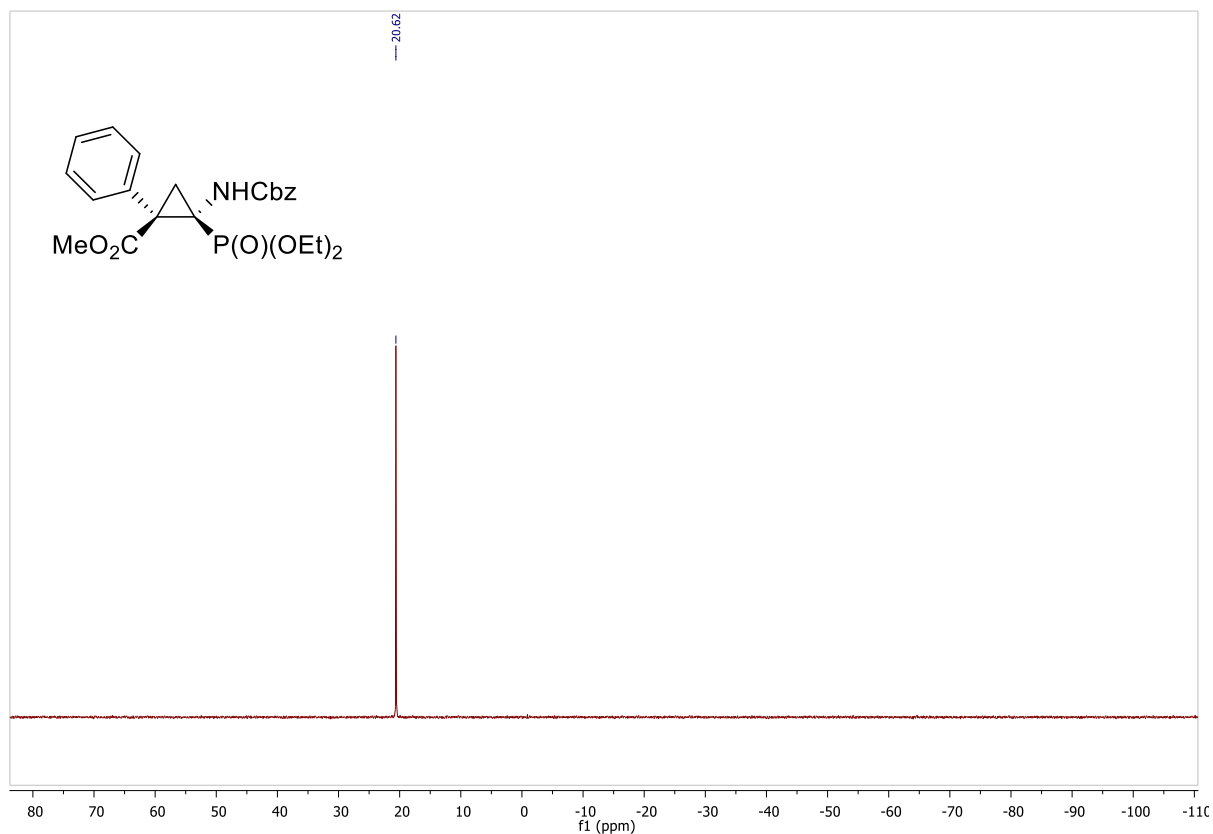




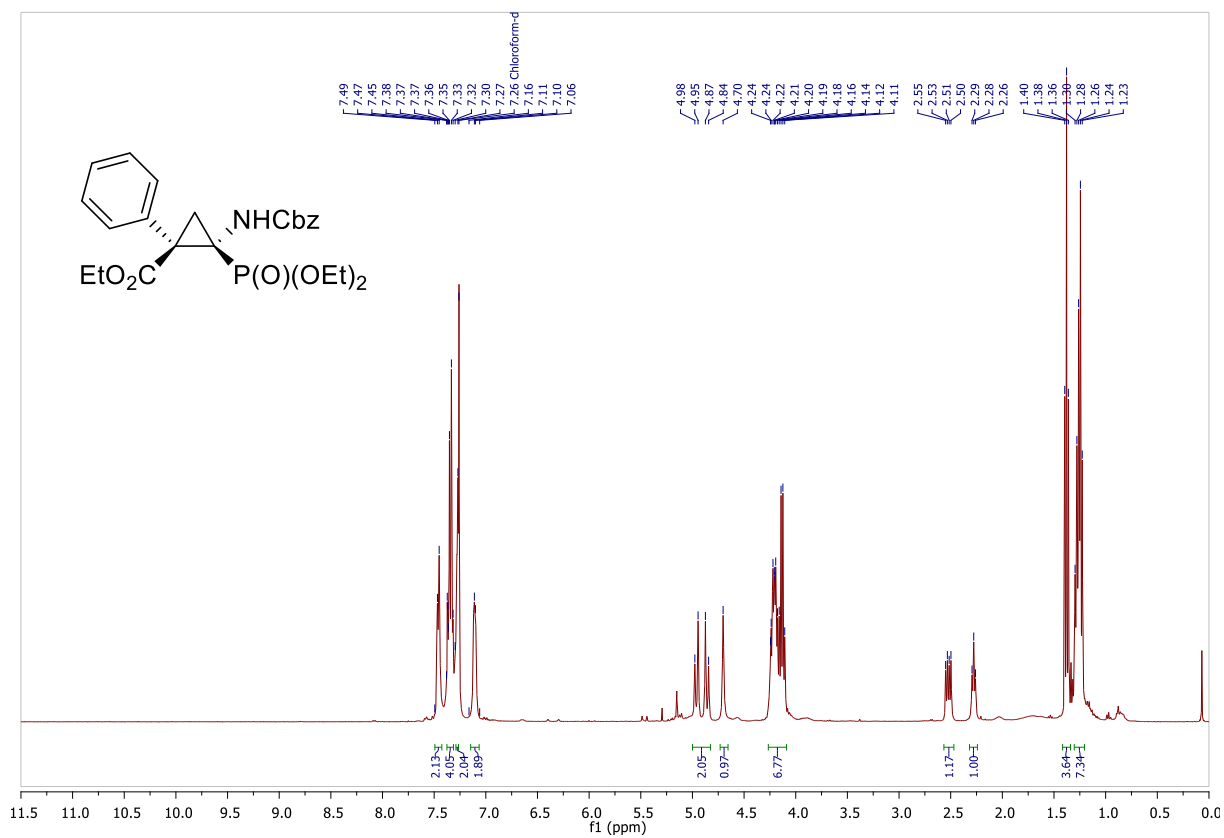


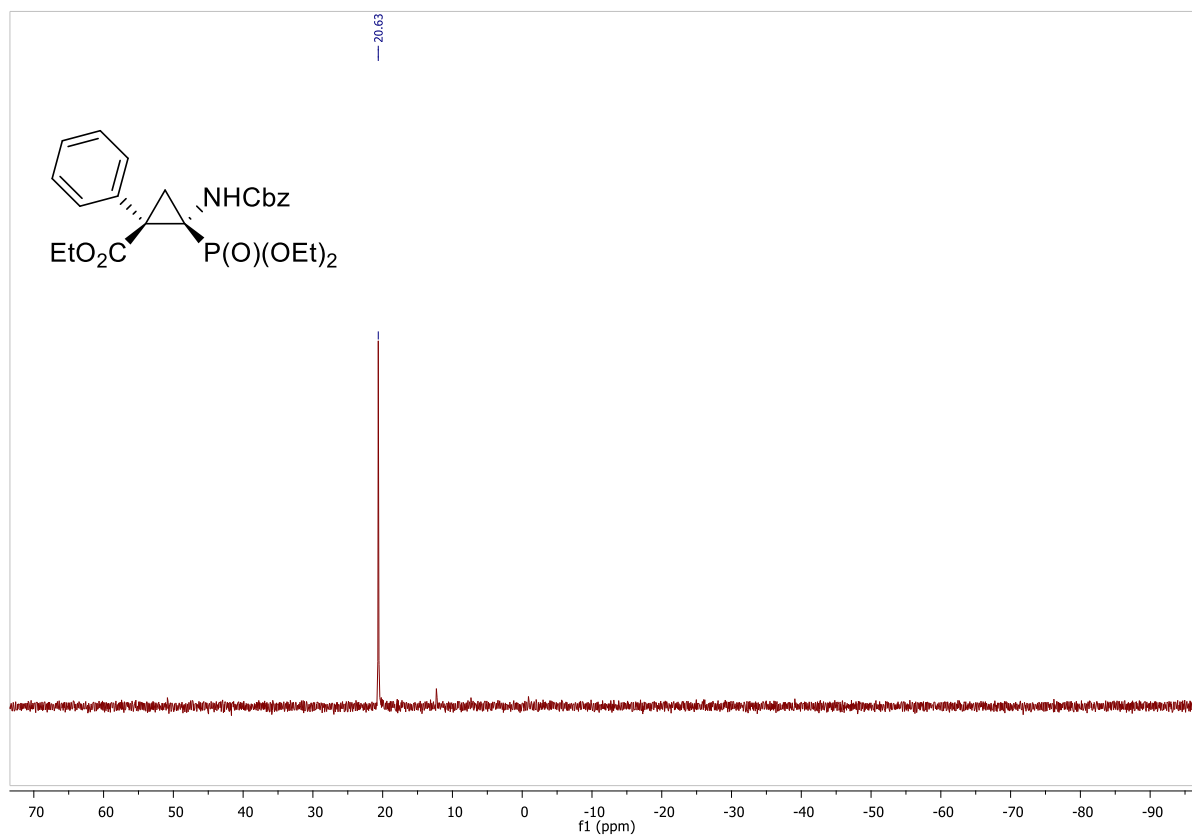
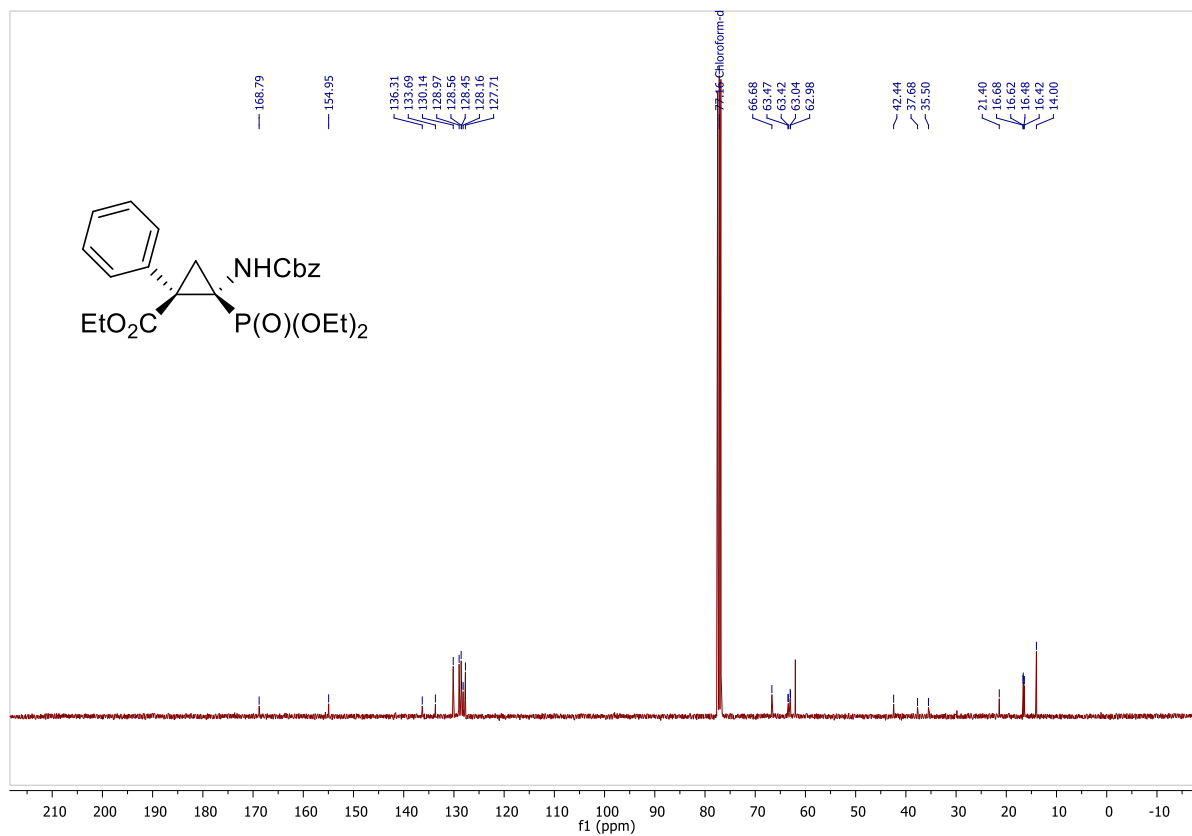
**Compound 19** : methyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-phenylcyclopropane-1-carboxylate



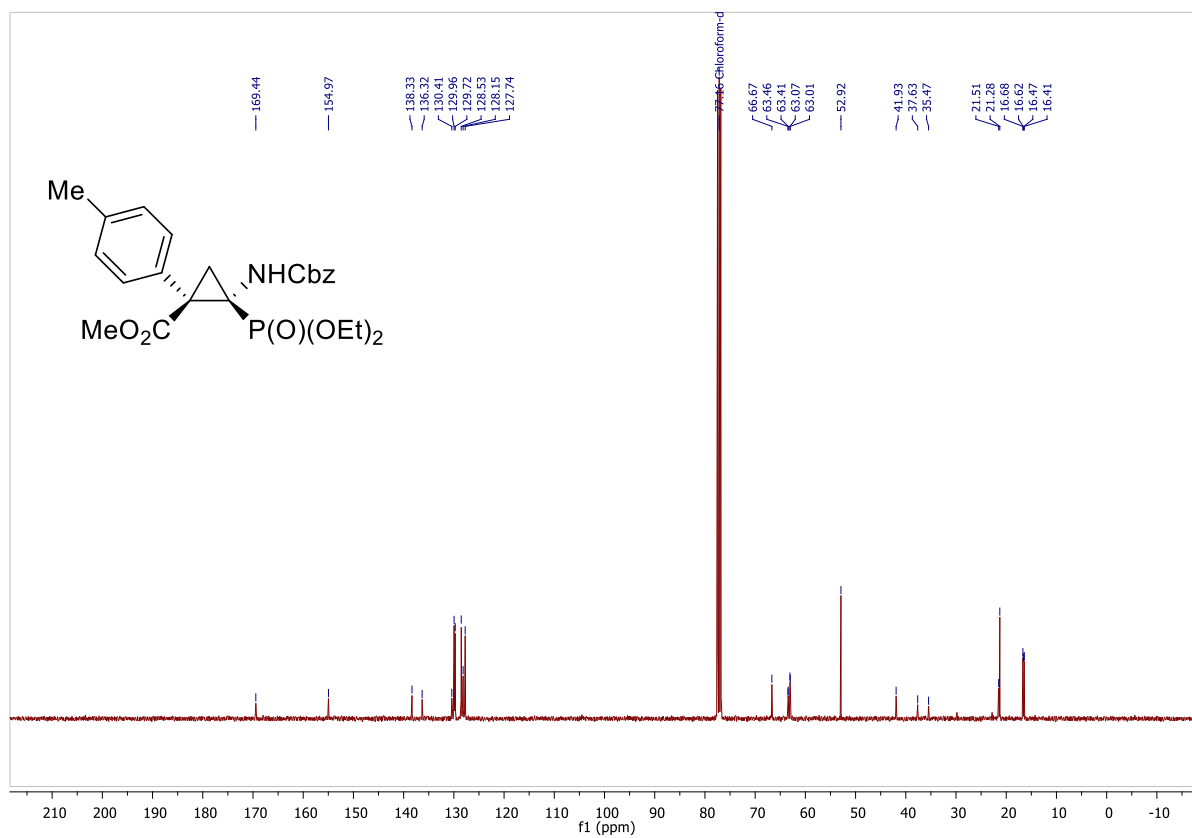
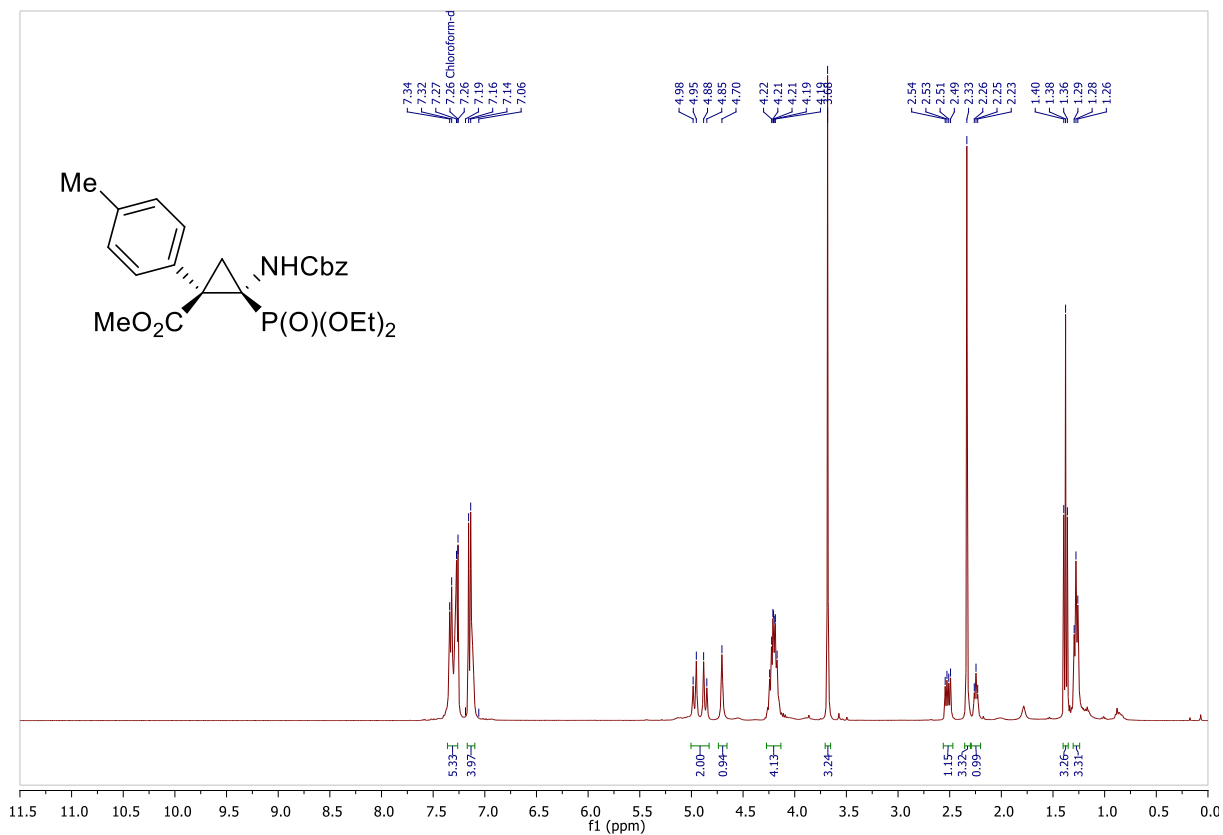


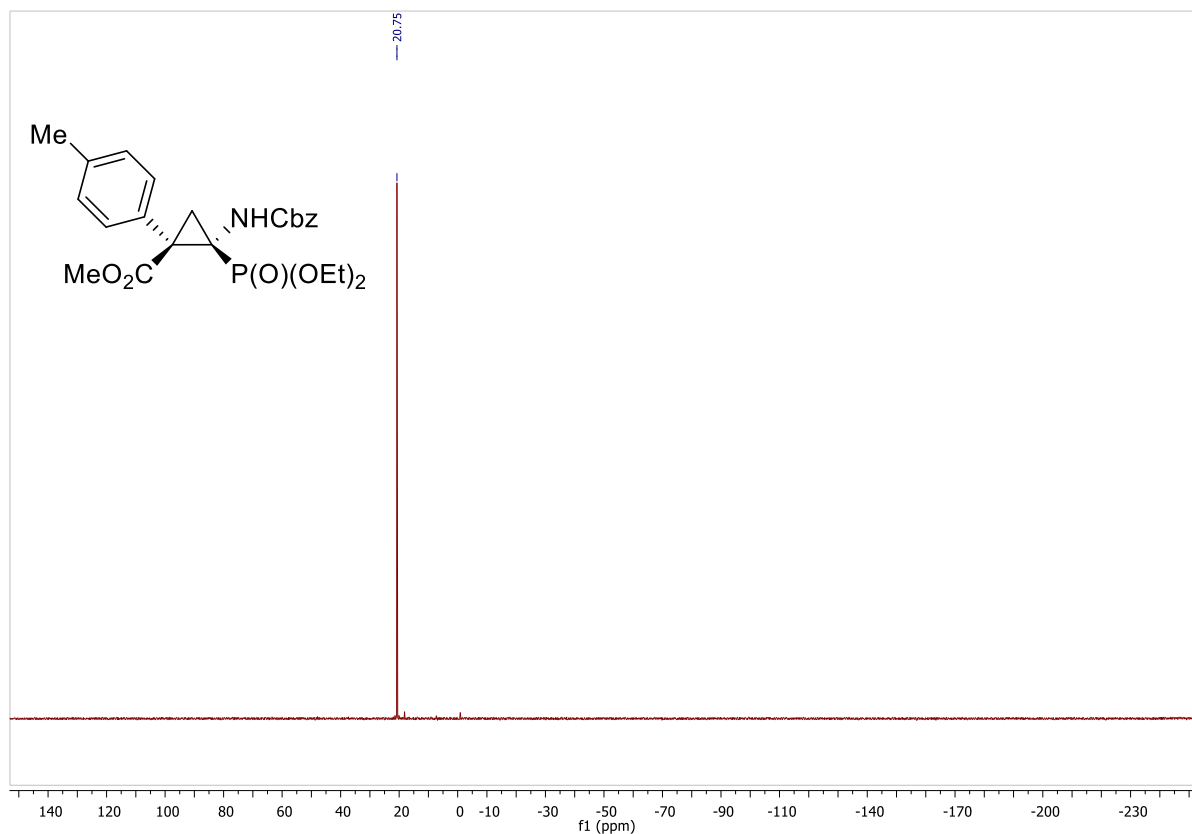
**Compound 20 : ethyl (1S,2R)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-phenyl cyclopropane-1-carboxylate**



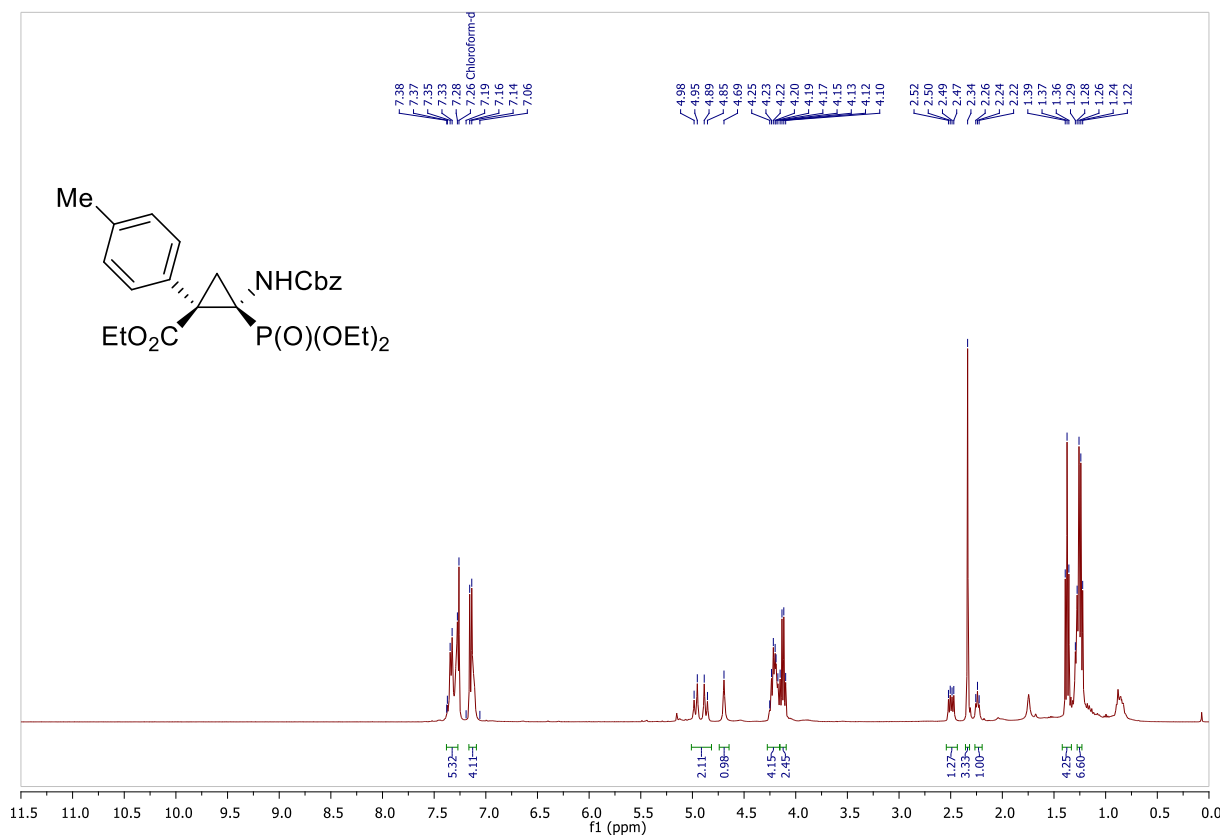


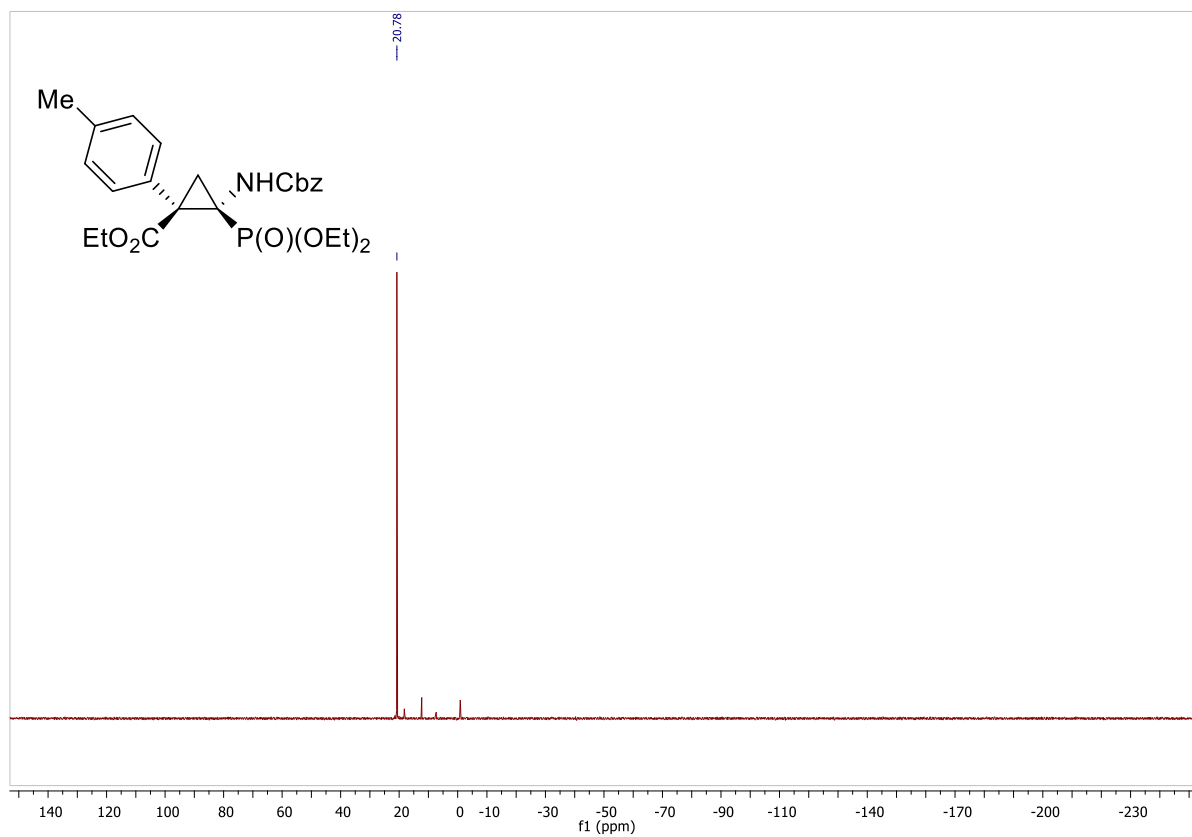
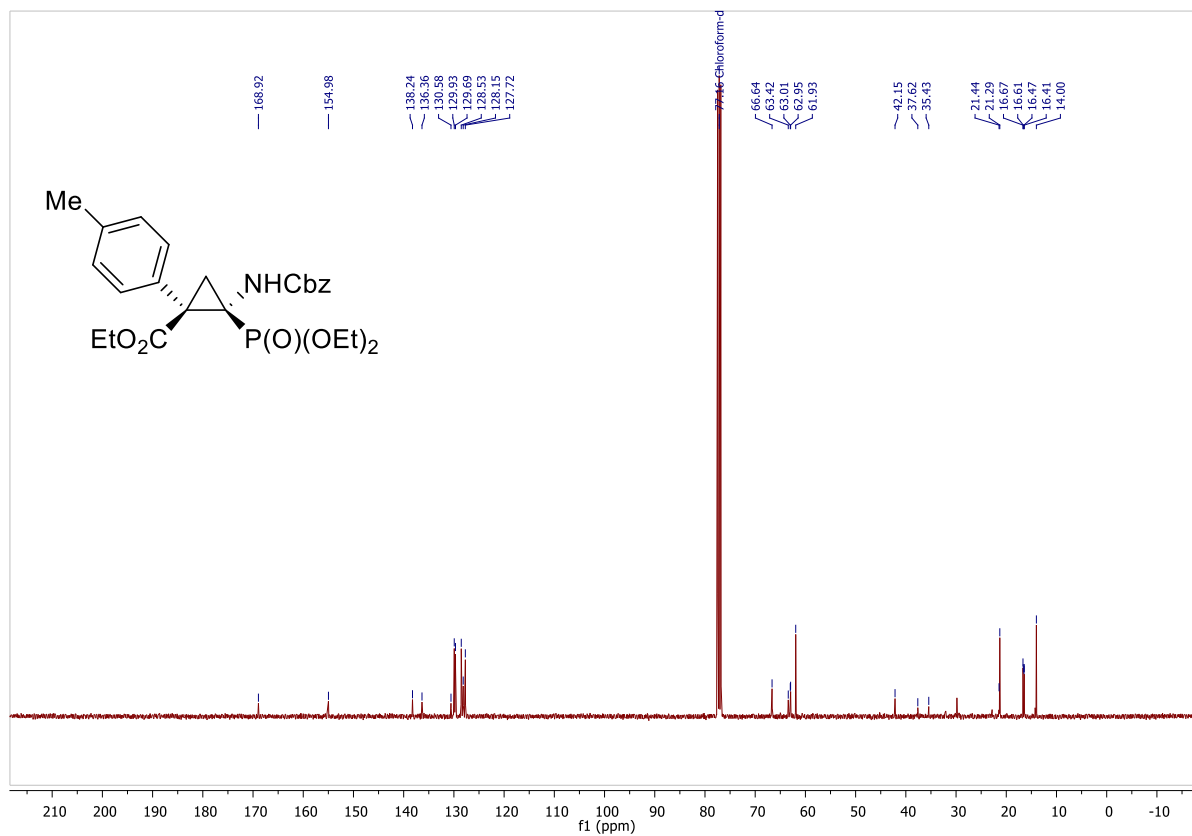
**Compound 21:** methyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-(*p*-tolyl) cyclopropane-1-carboxylate



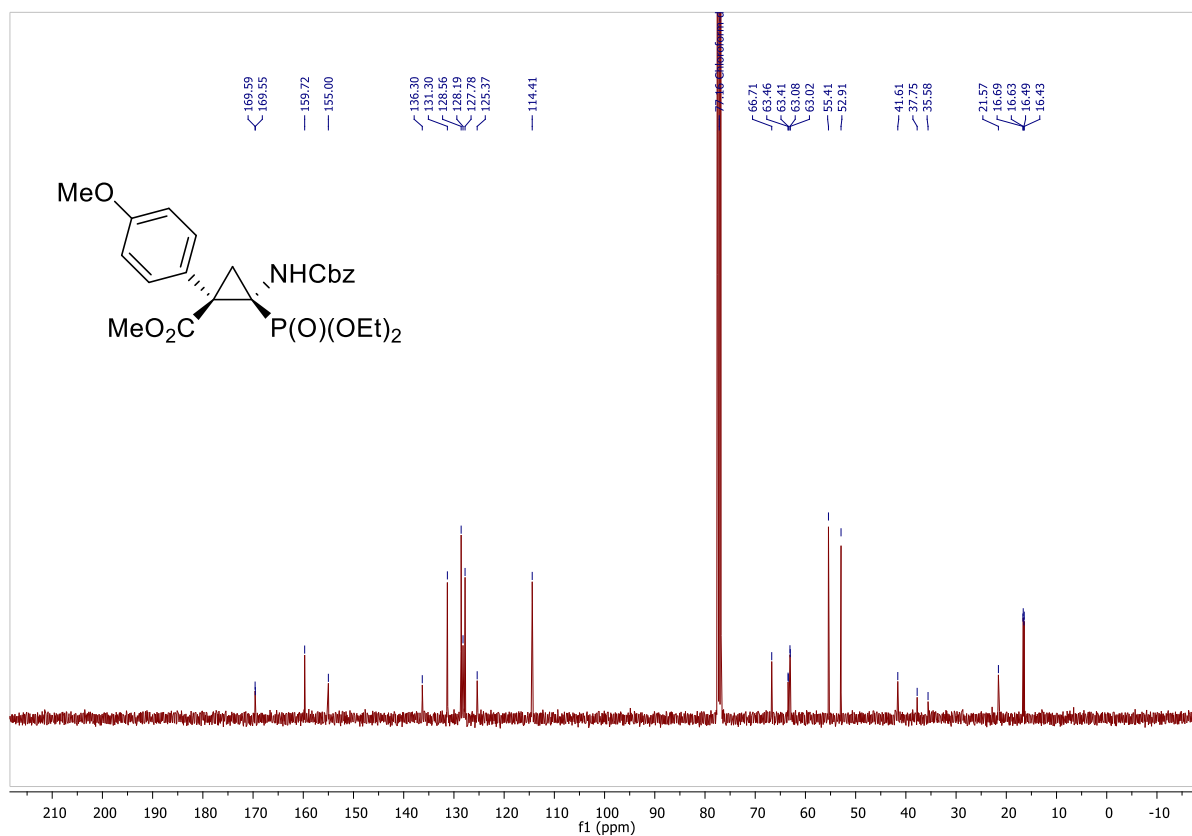
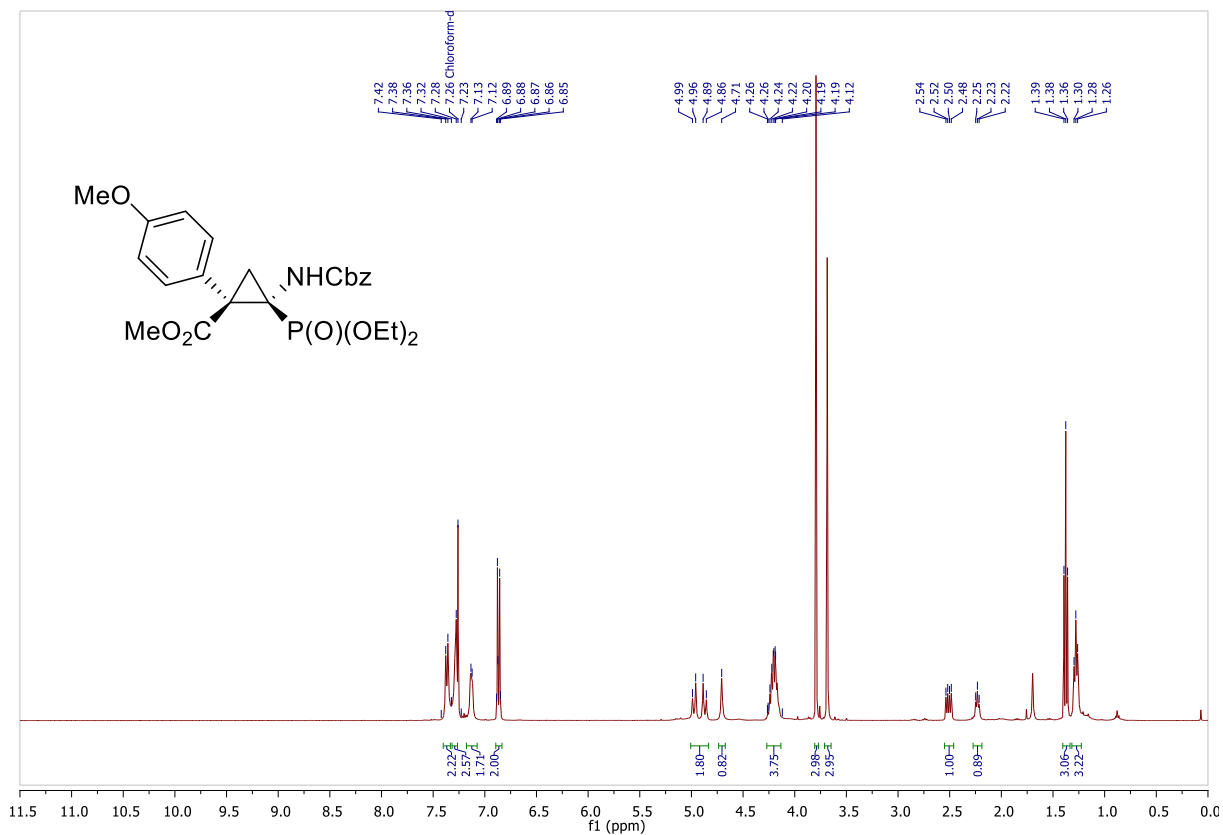


**Compound 22** : ethyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-(*p*-tolyl) cyclopropane-1-carboxylate

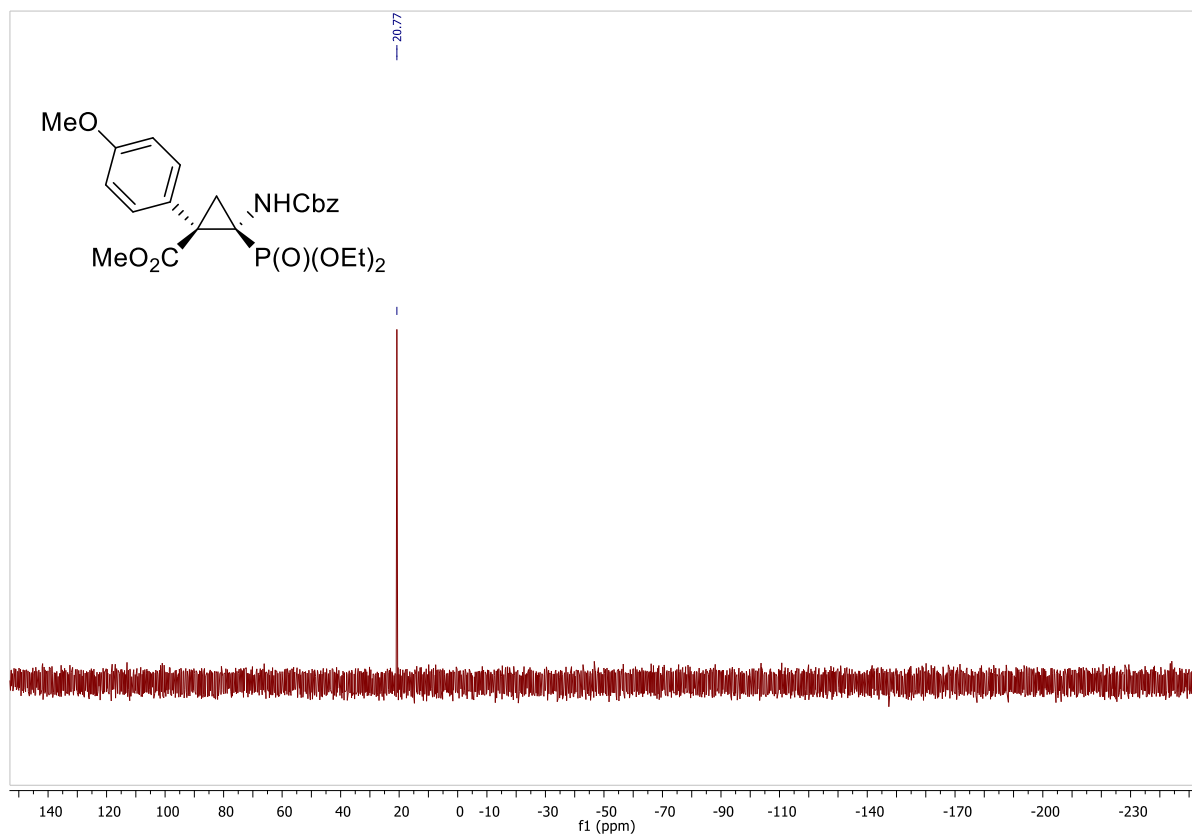




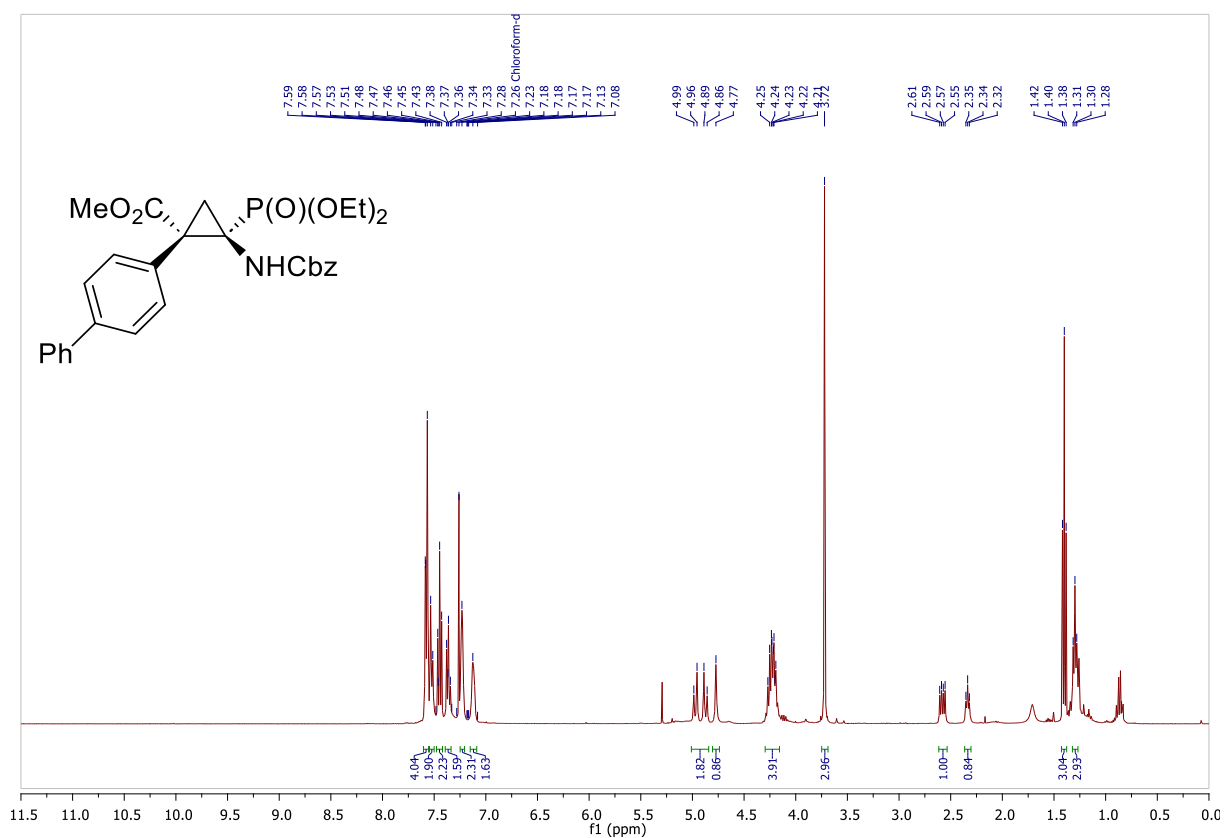
**Compound 23 : methyl (1S,2R)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-(4-methoxyphenyl)cyclopropane-1-carboxylate**

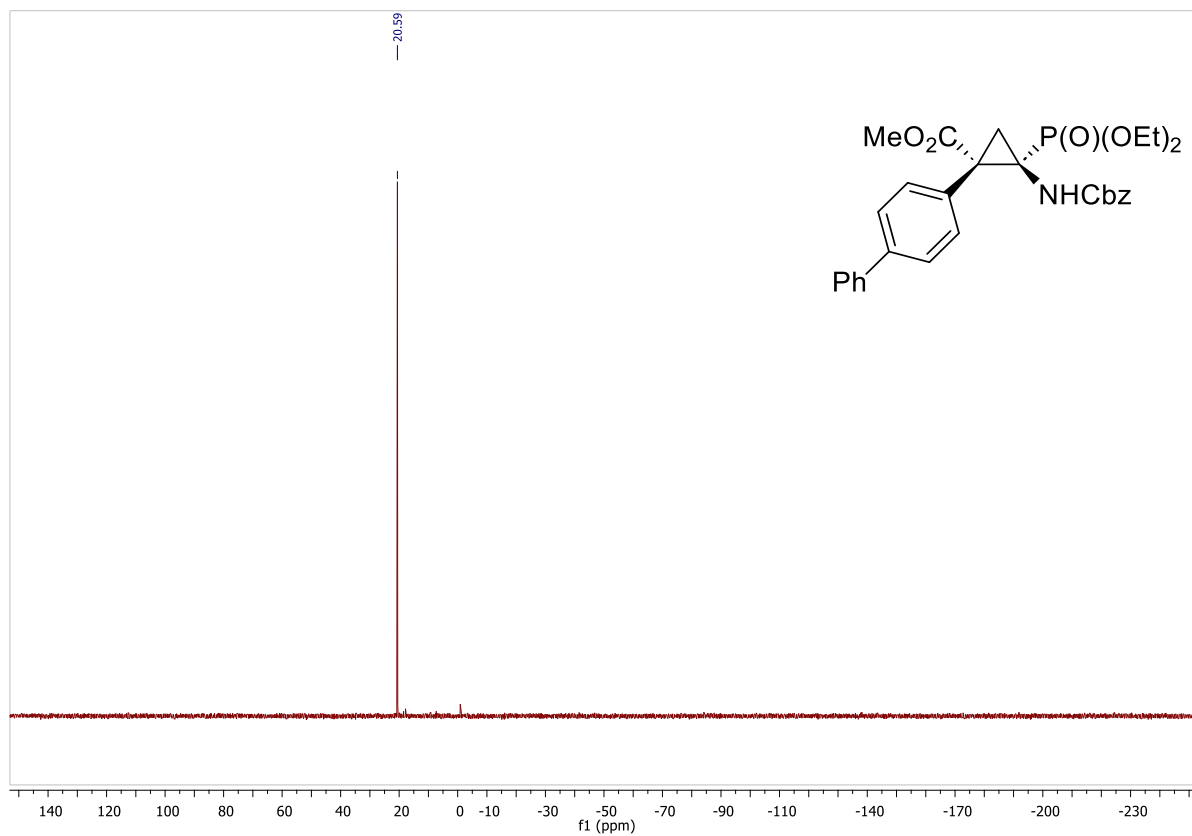
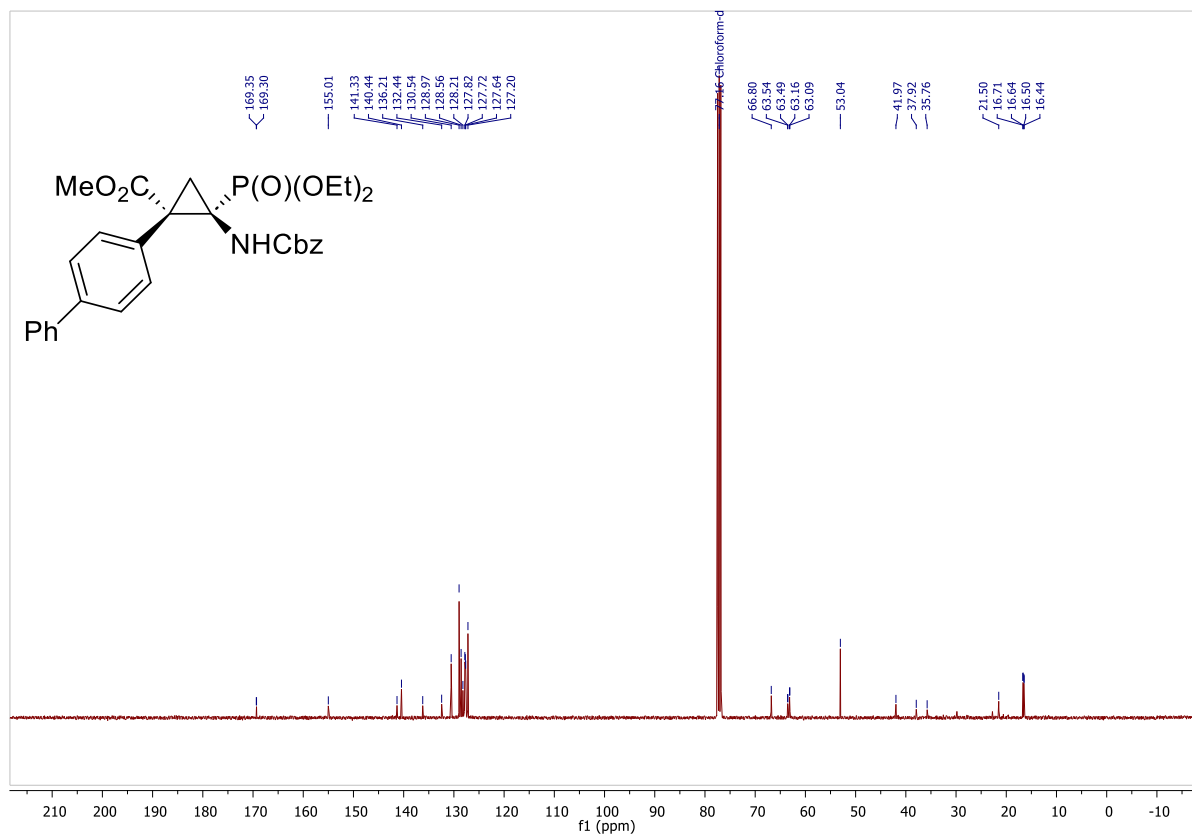




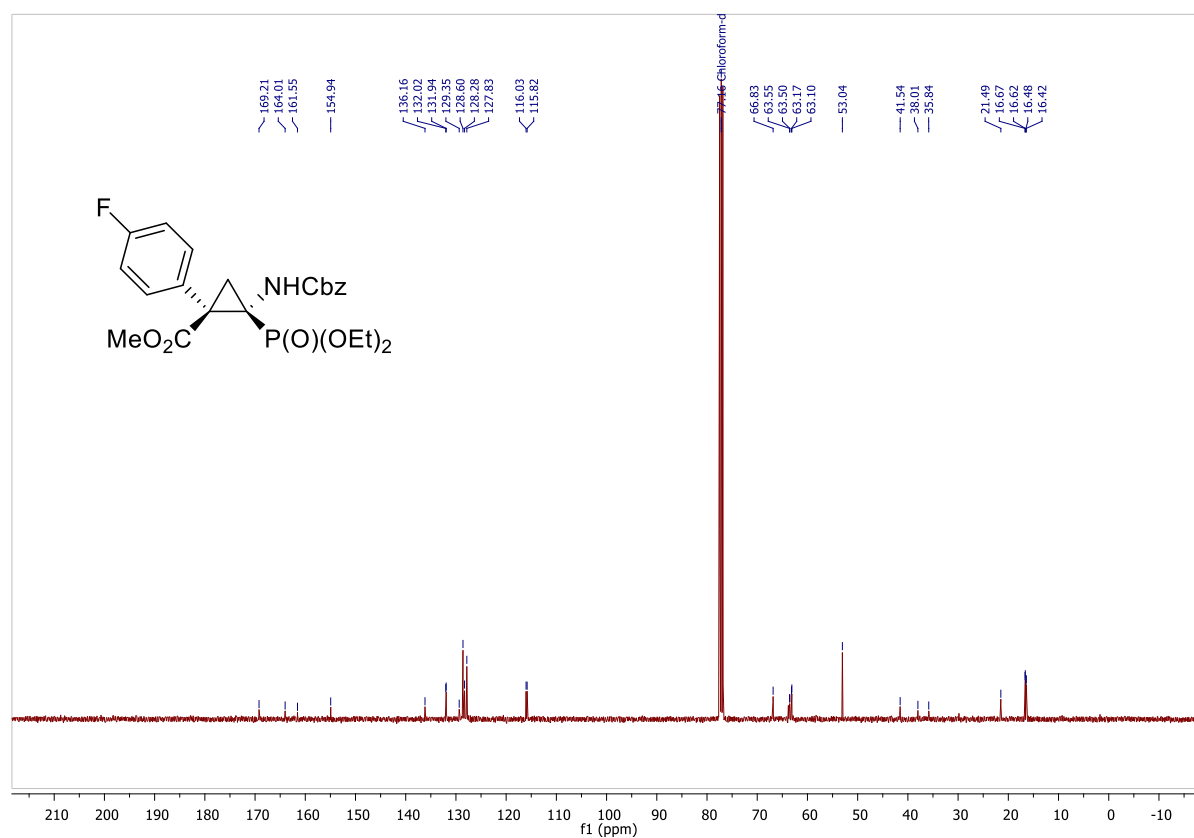
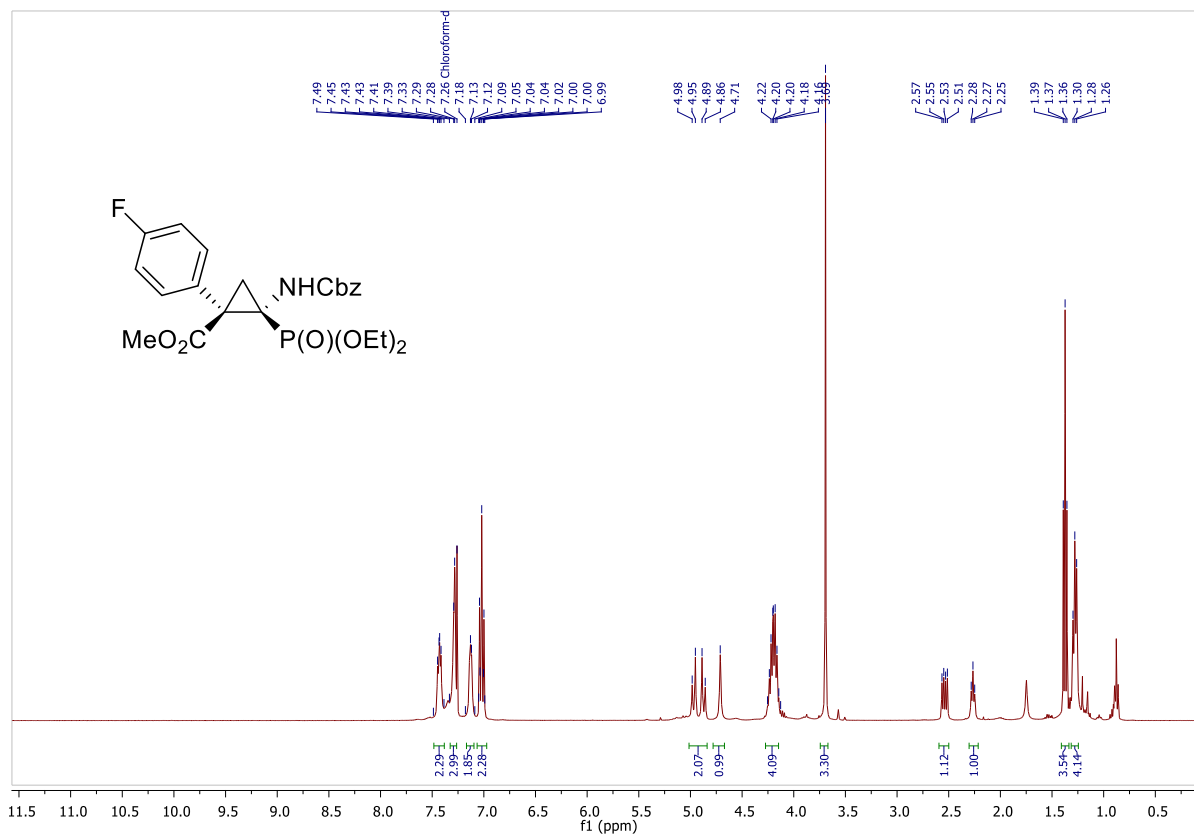


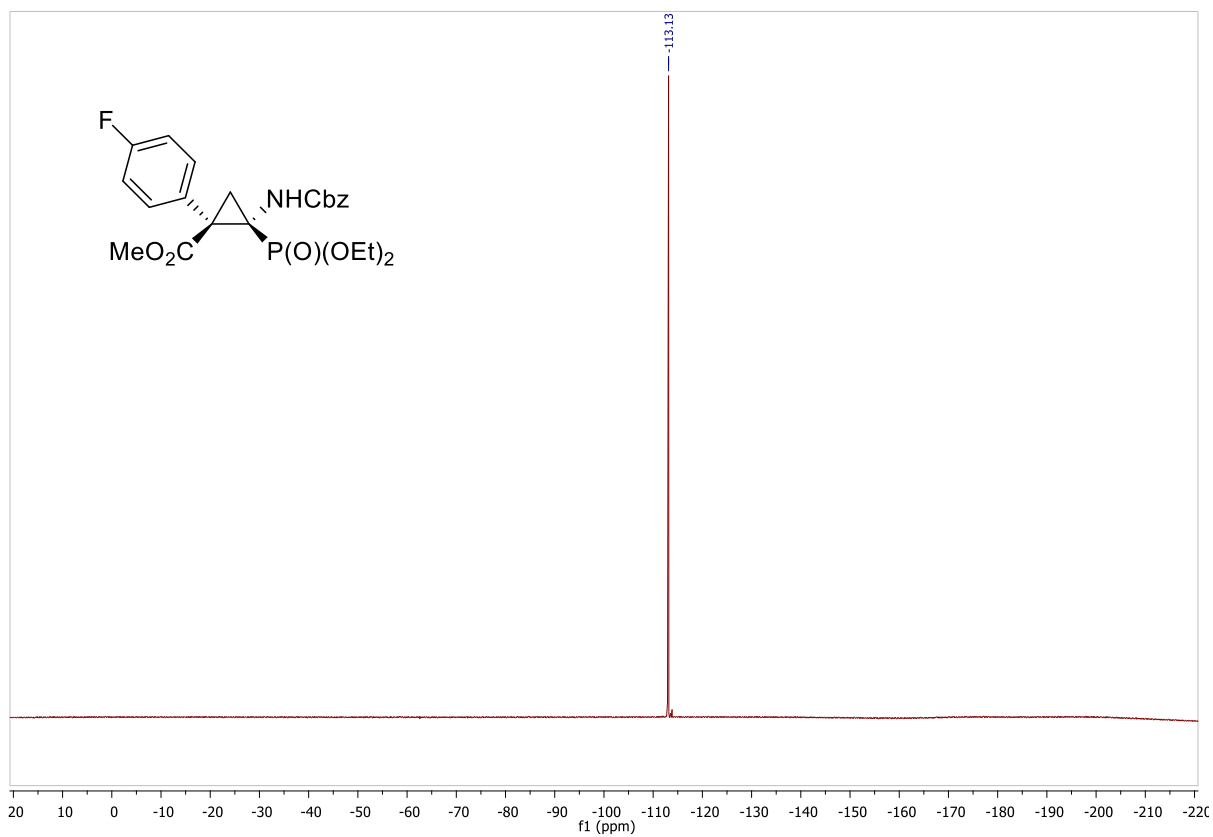
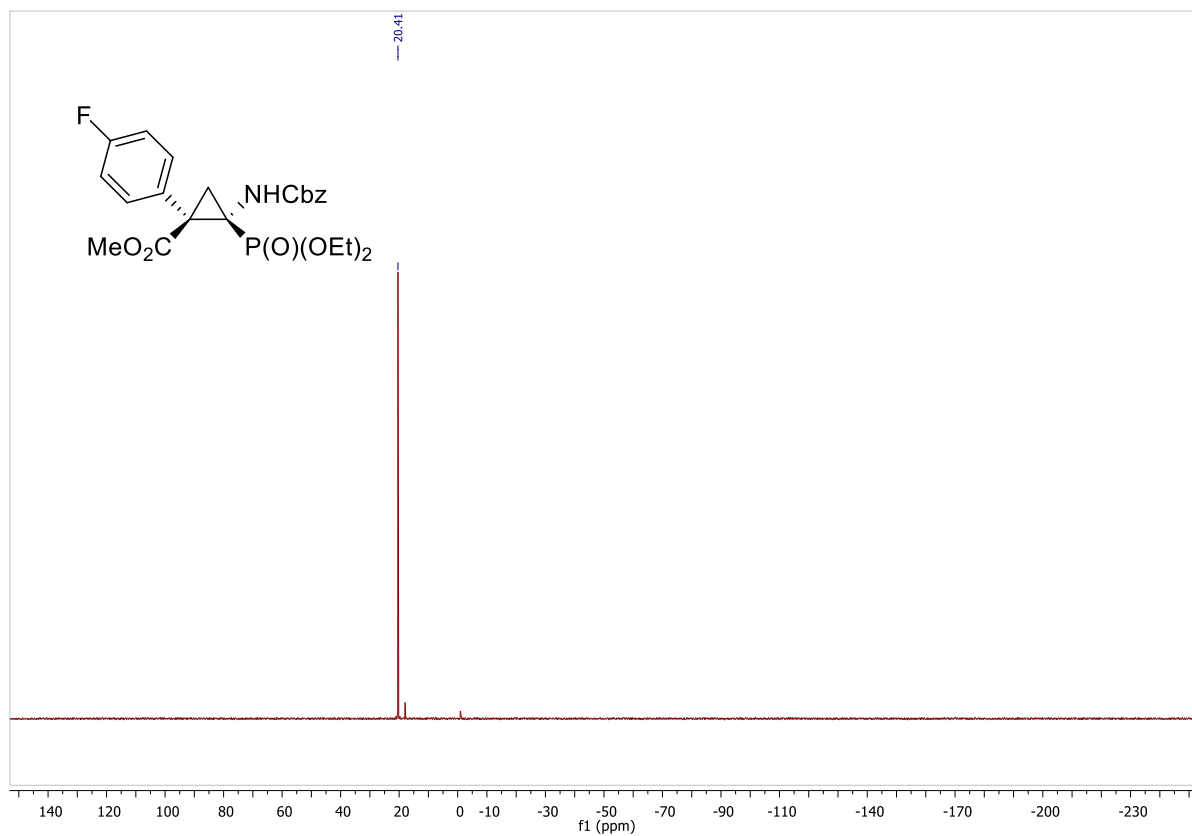
**Compound 24** : methyl (1R,2S)-1-([1,1'-biphenyl]-4-yl)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)cyclopropane-1-carboxylate



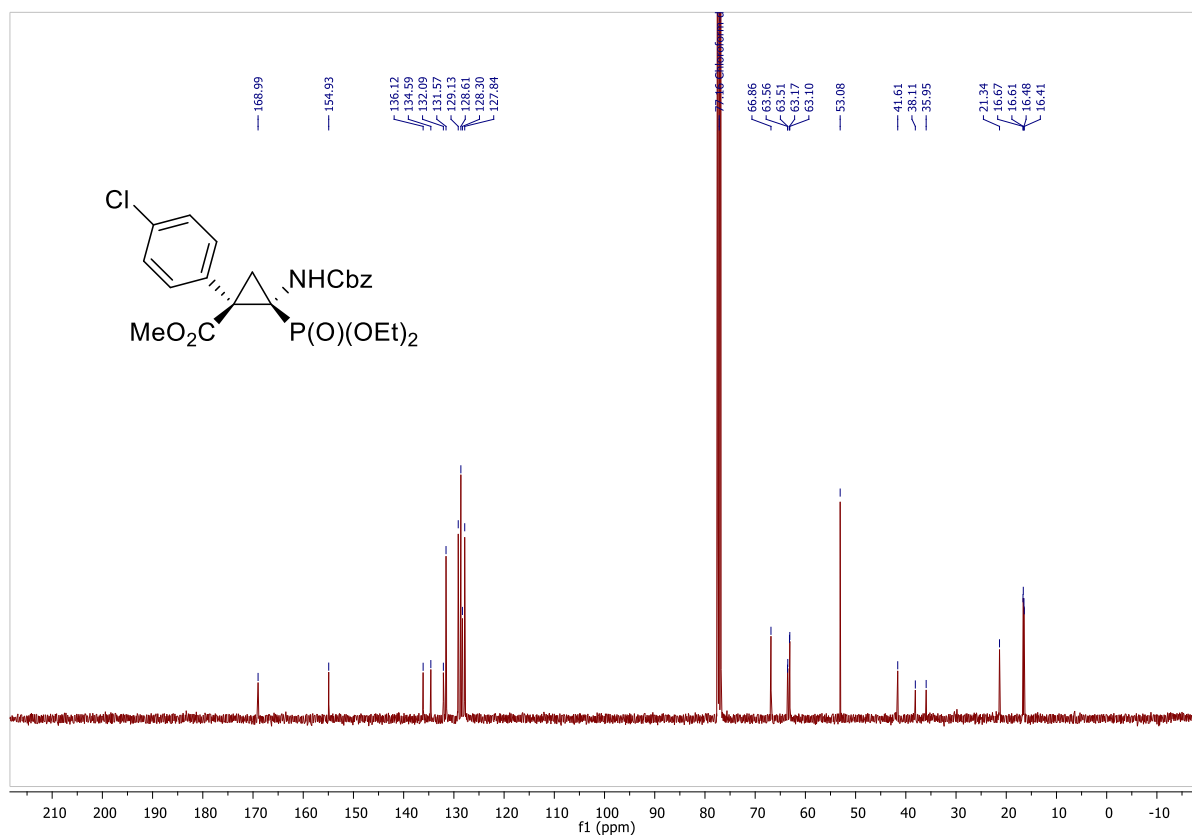
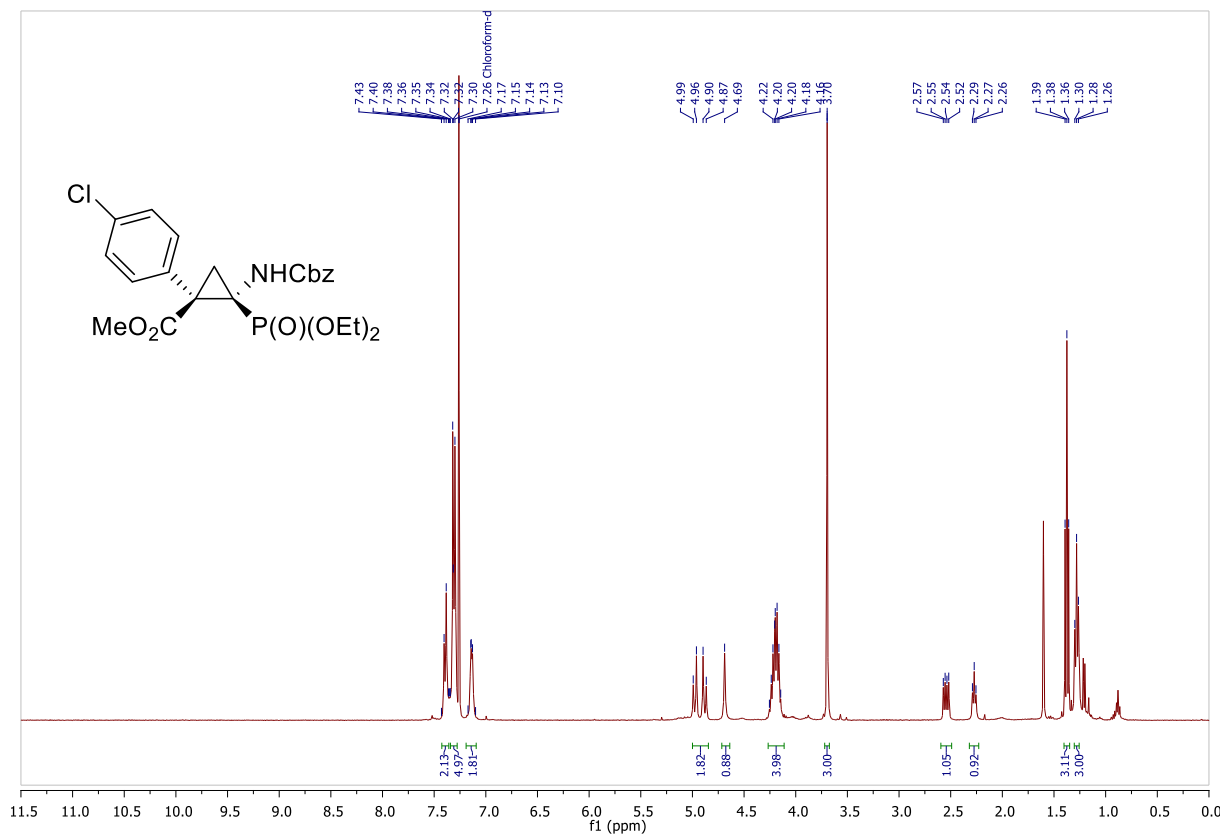


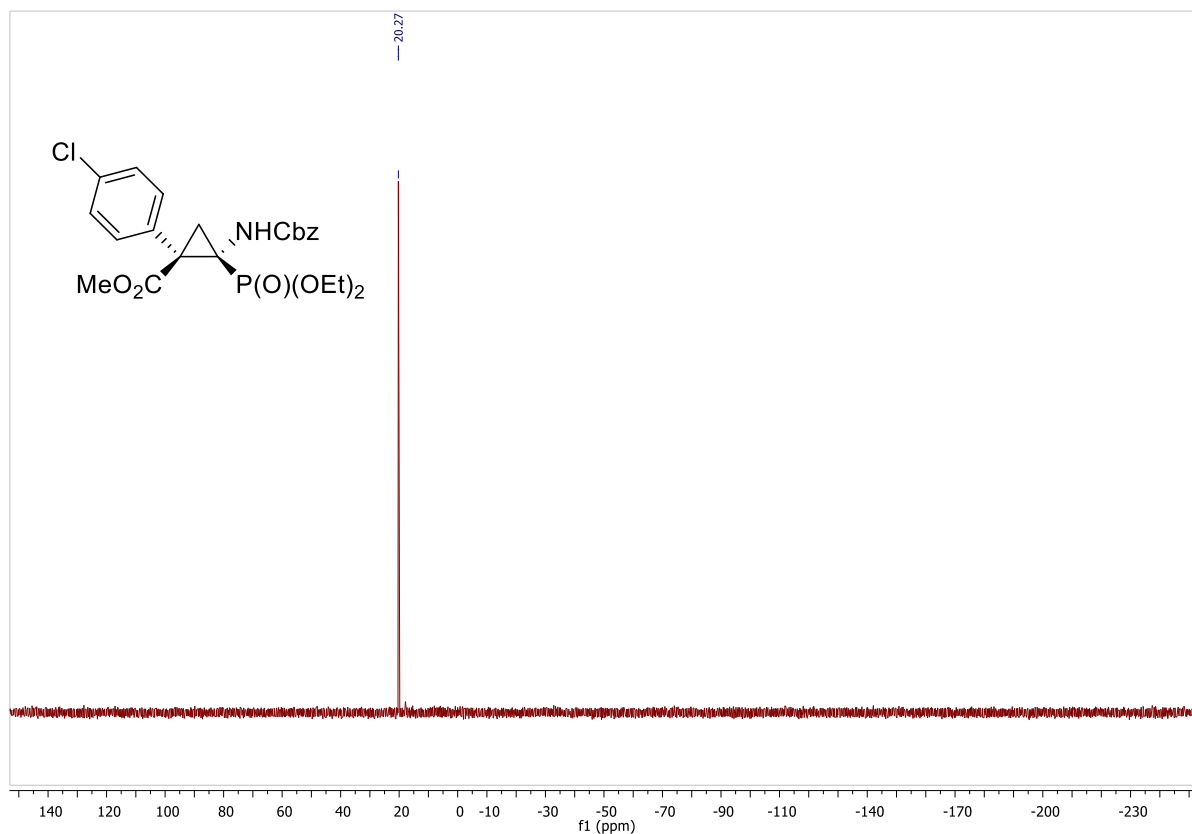
**Compound 25 : methyl (1S,2R)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-(4-fluoro phenyl)cyclopropane-1-carboxylate**



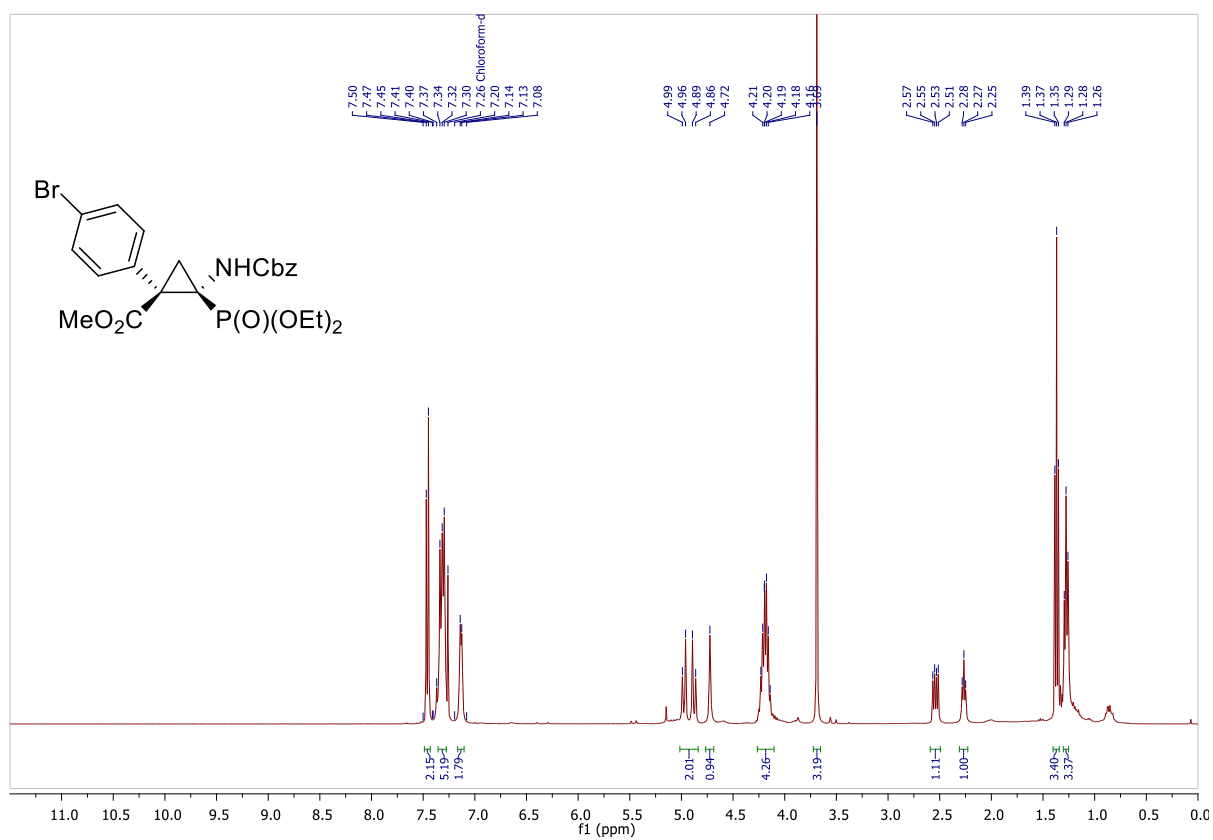


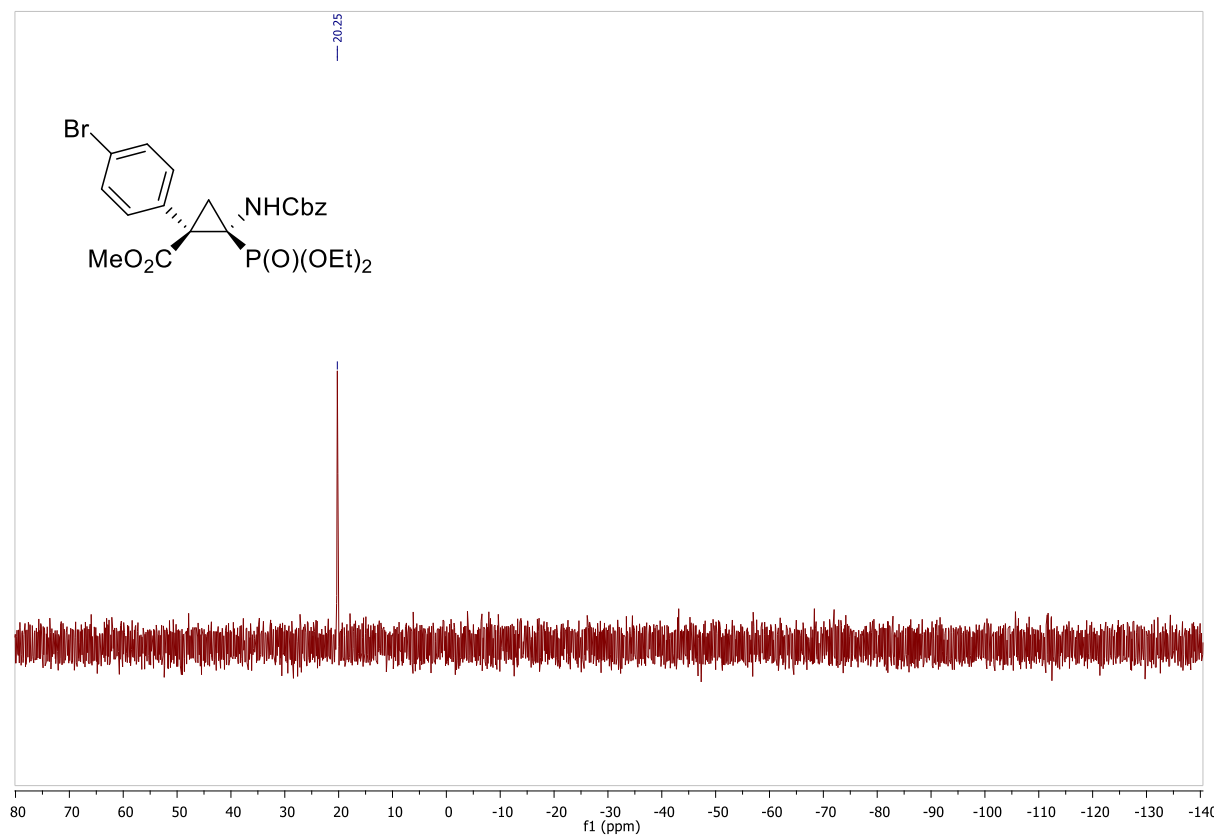
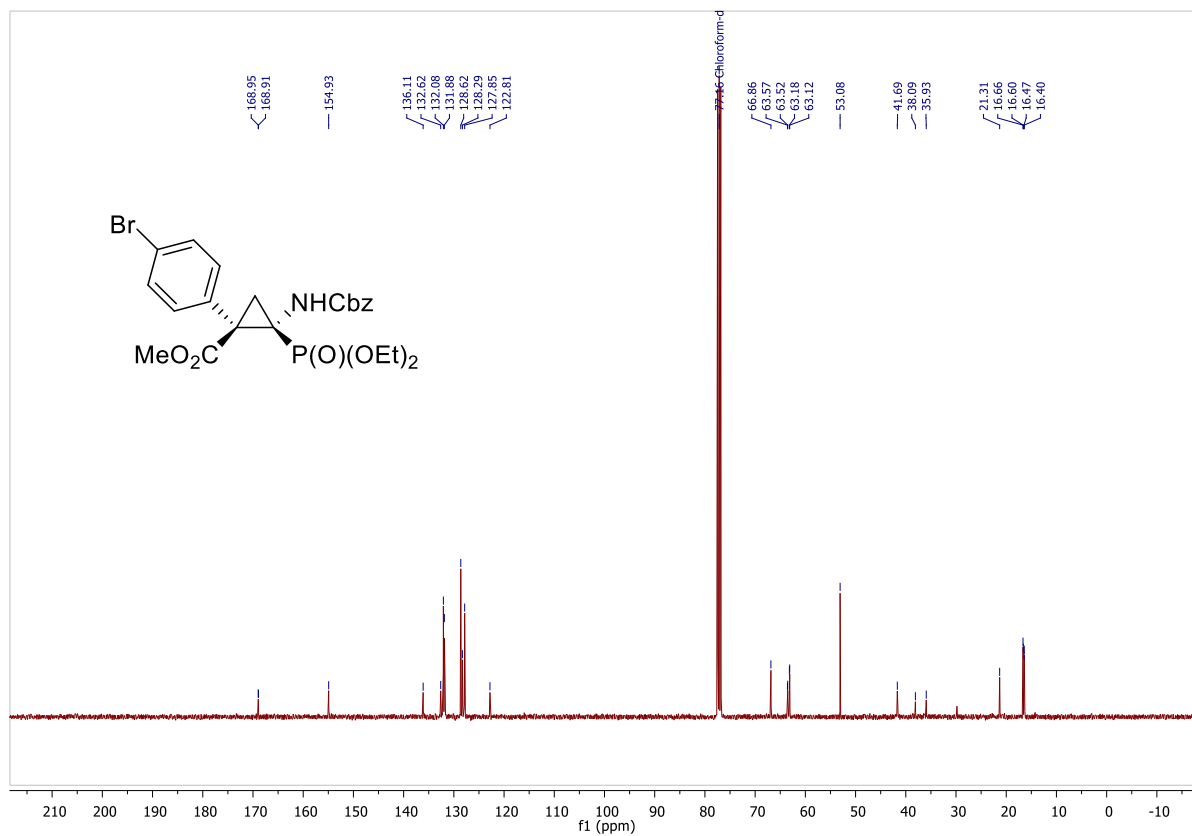
**Compound 26** : methyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-1-(4-chlorophenyl)-2-(diethoxy phosphoryl)cyclopropane-1-carboxylate



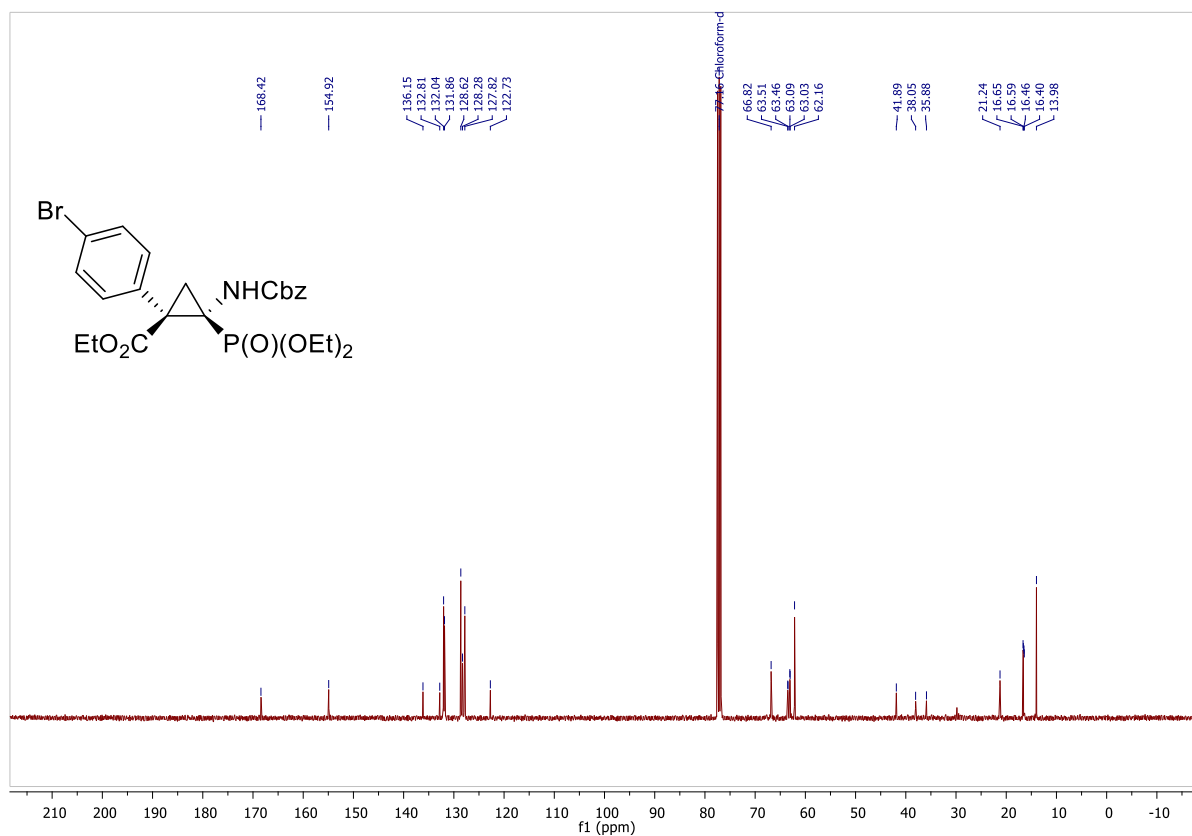
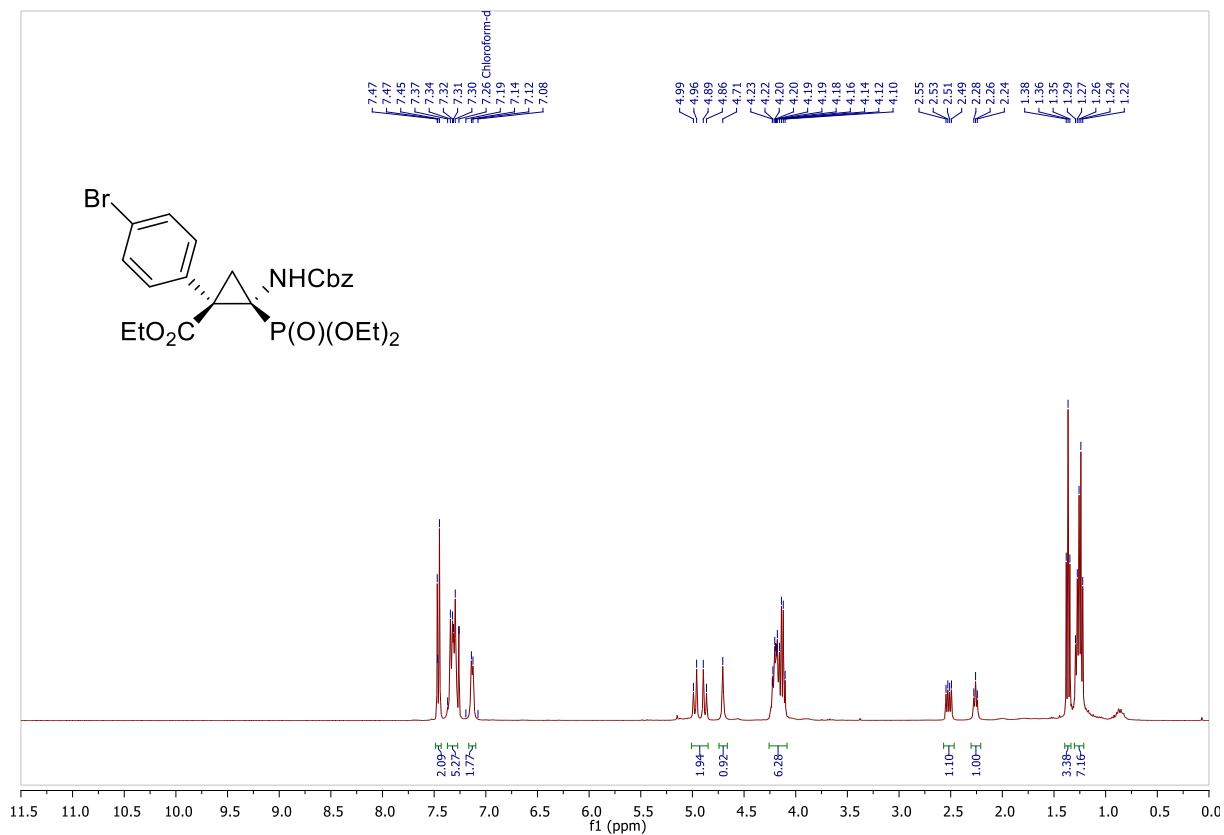


**Compound 27** : methyl (1S,2R)-2-(((benzyloxy)carbonyl)amino)-1-(4-bromophenyl)-2-(diethoxy phosphoryl)cyclopropane-1-carboxylate

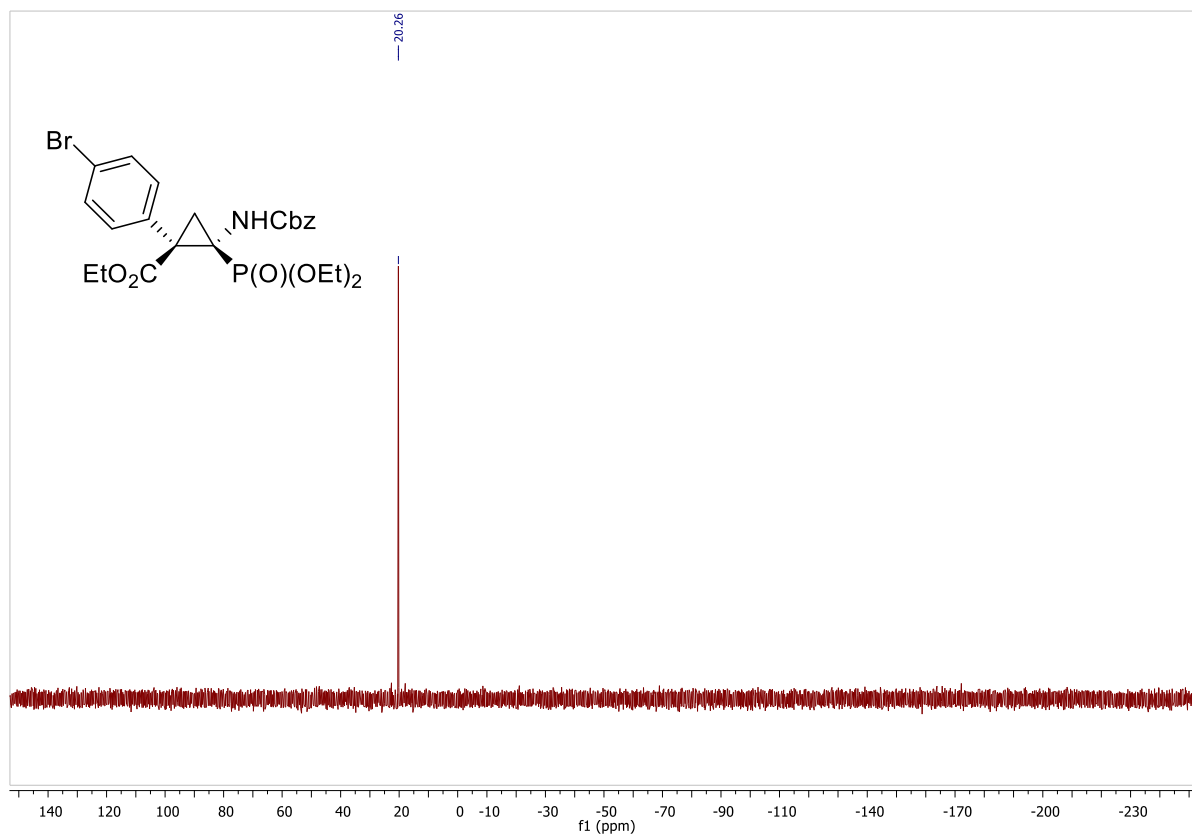




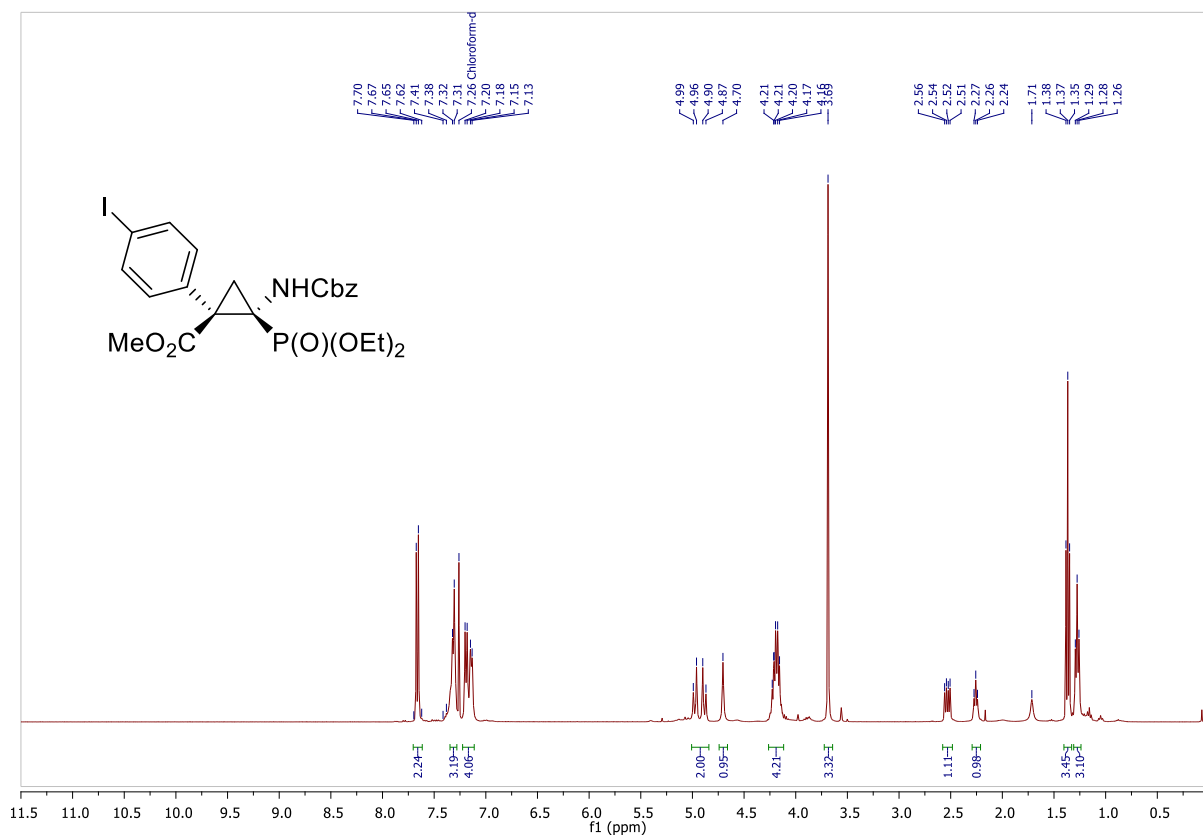
**Compound 28**: ethyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-1-(4-bromophenyl)-2-(diethoxyphosphoryl)cyclopropane-1-carboxylate

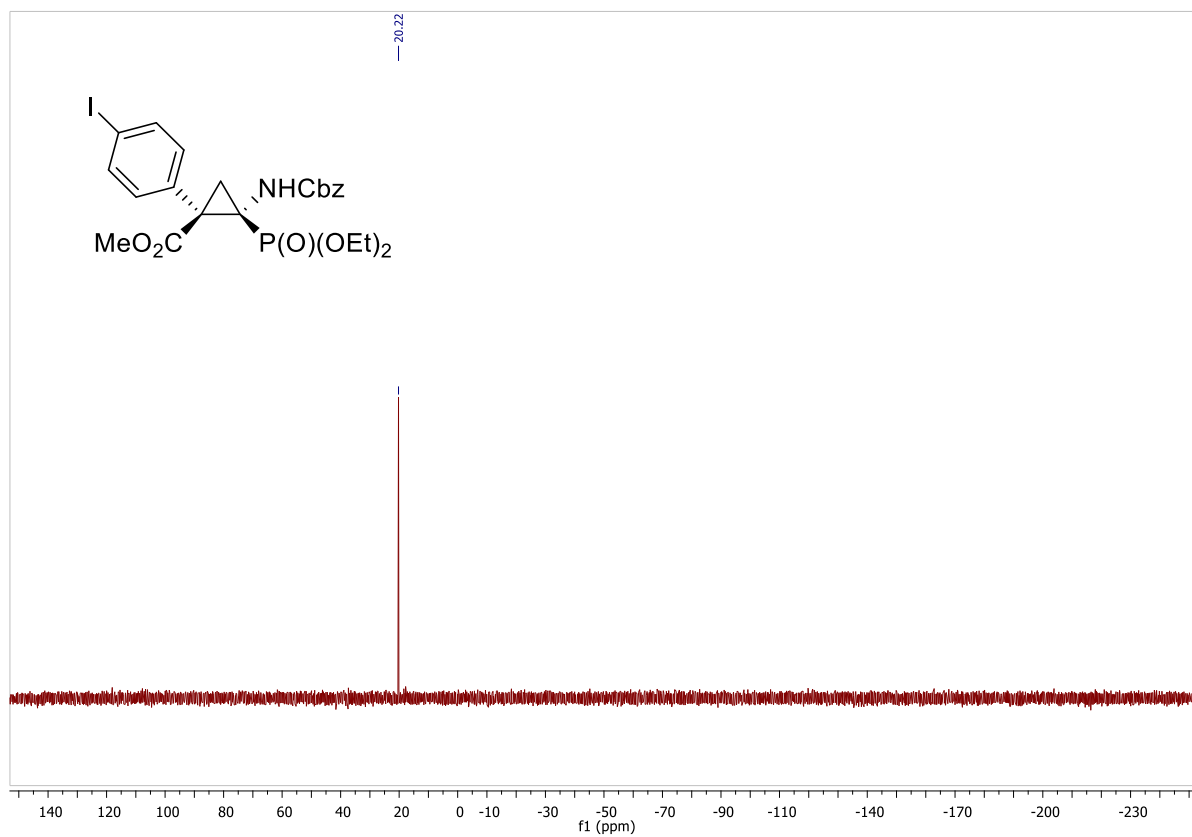
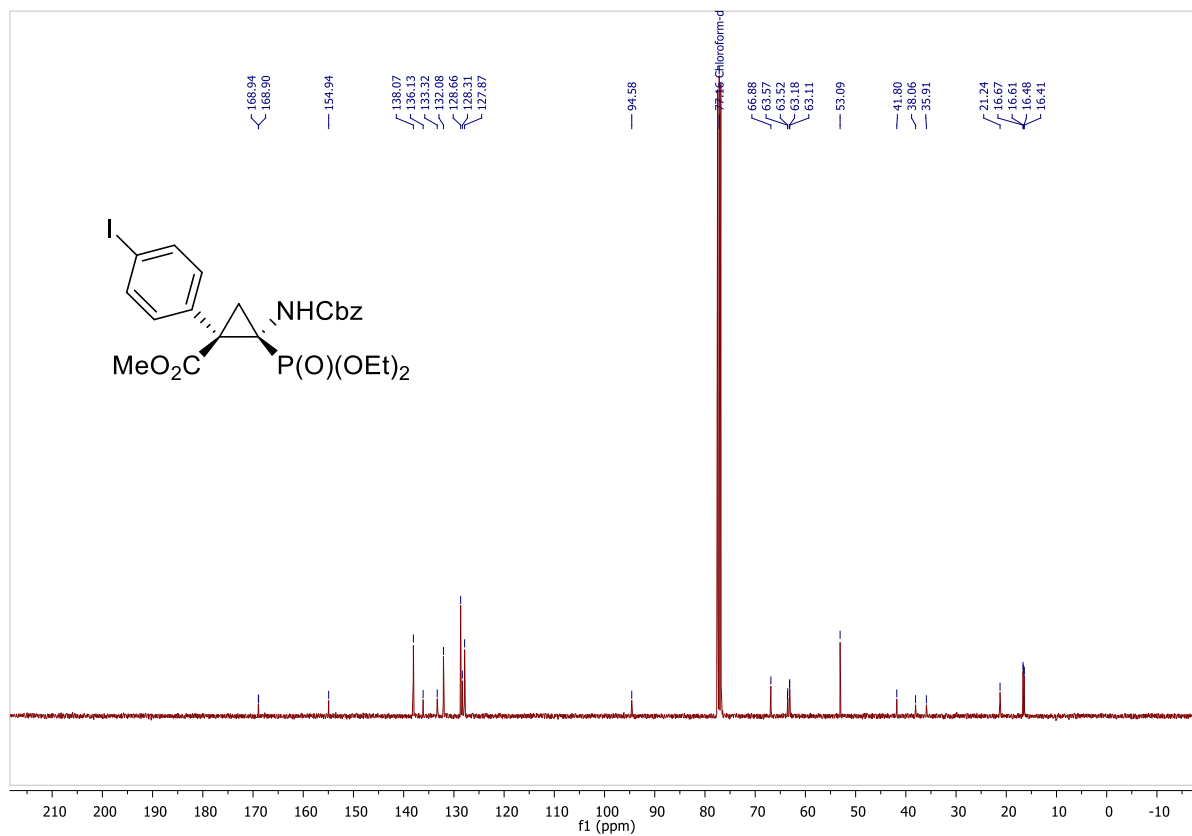




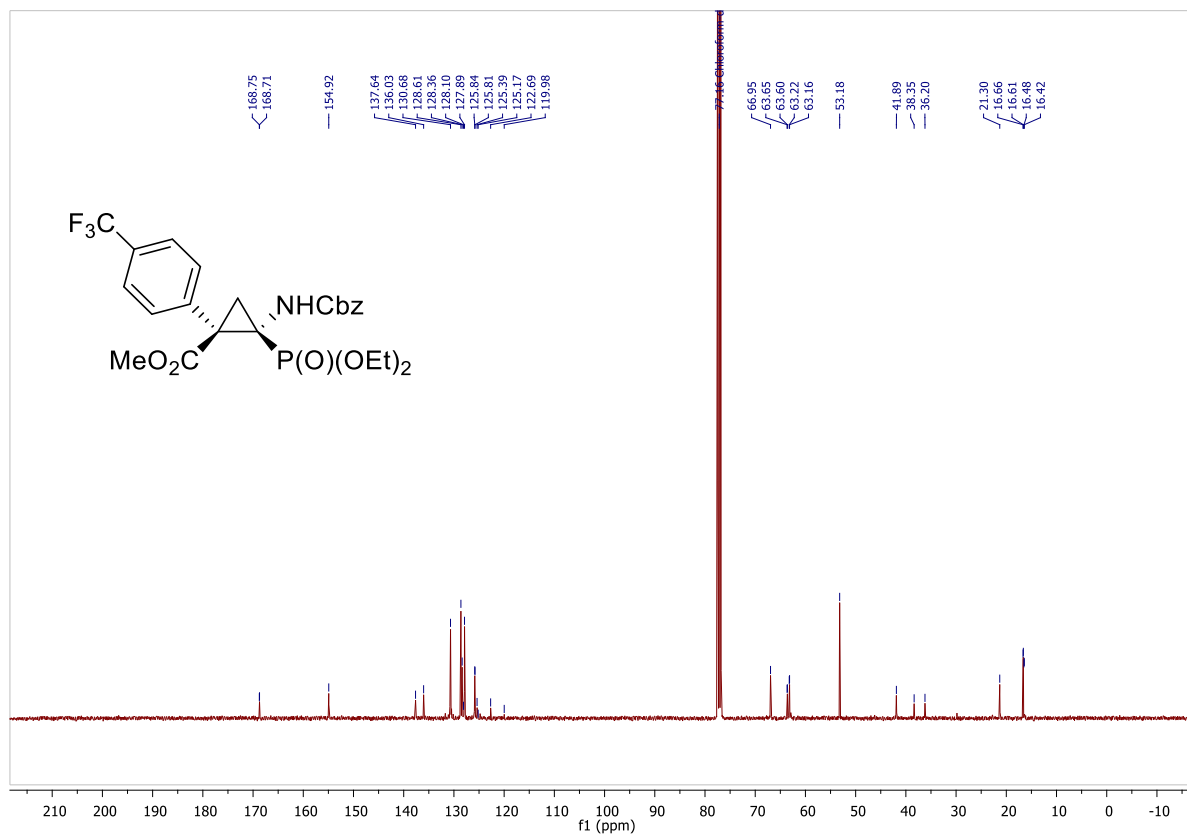
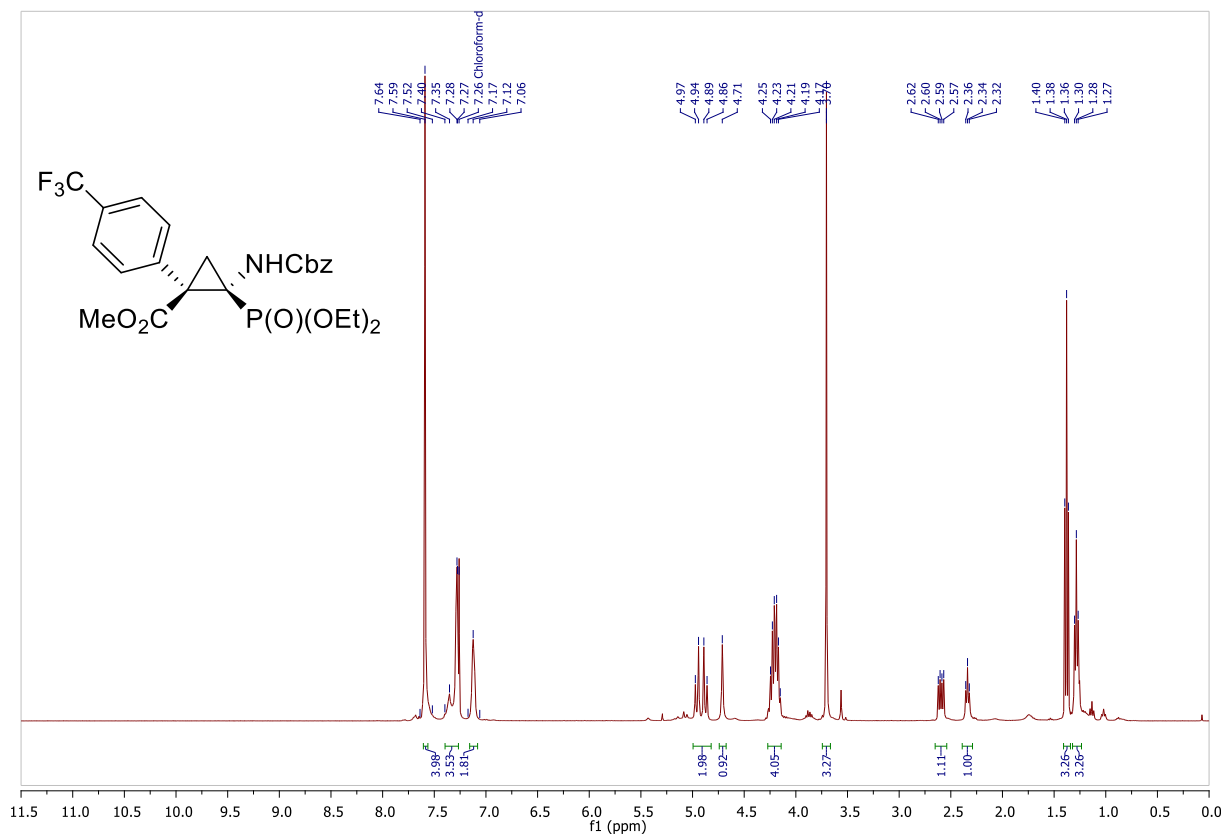


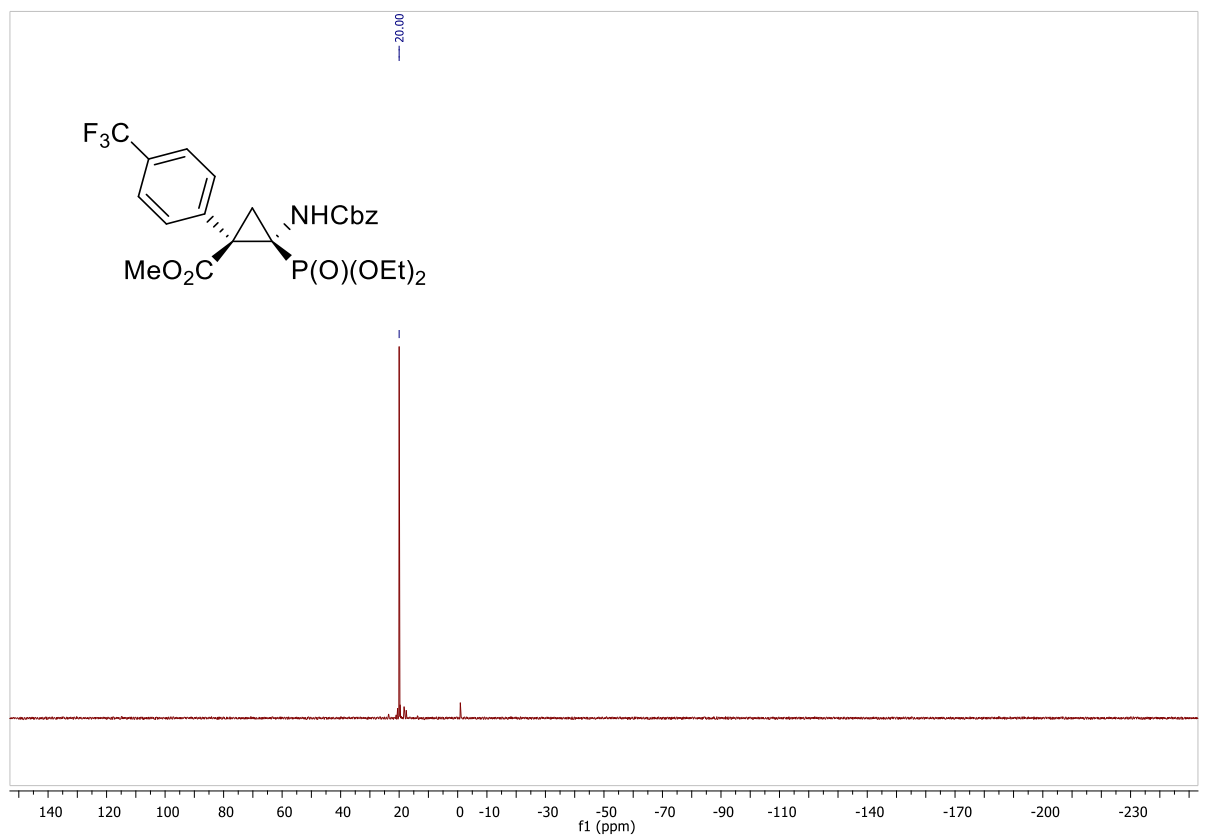
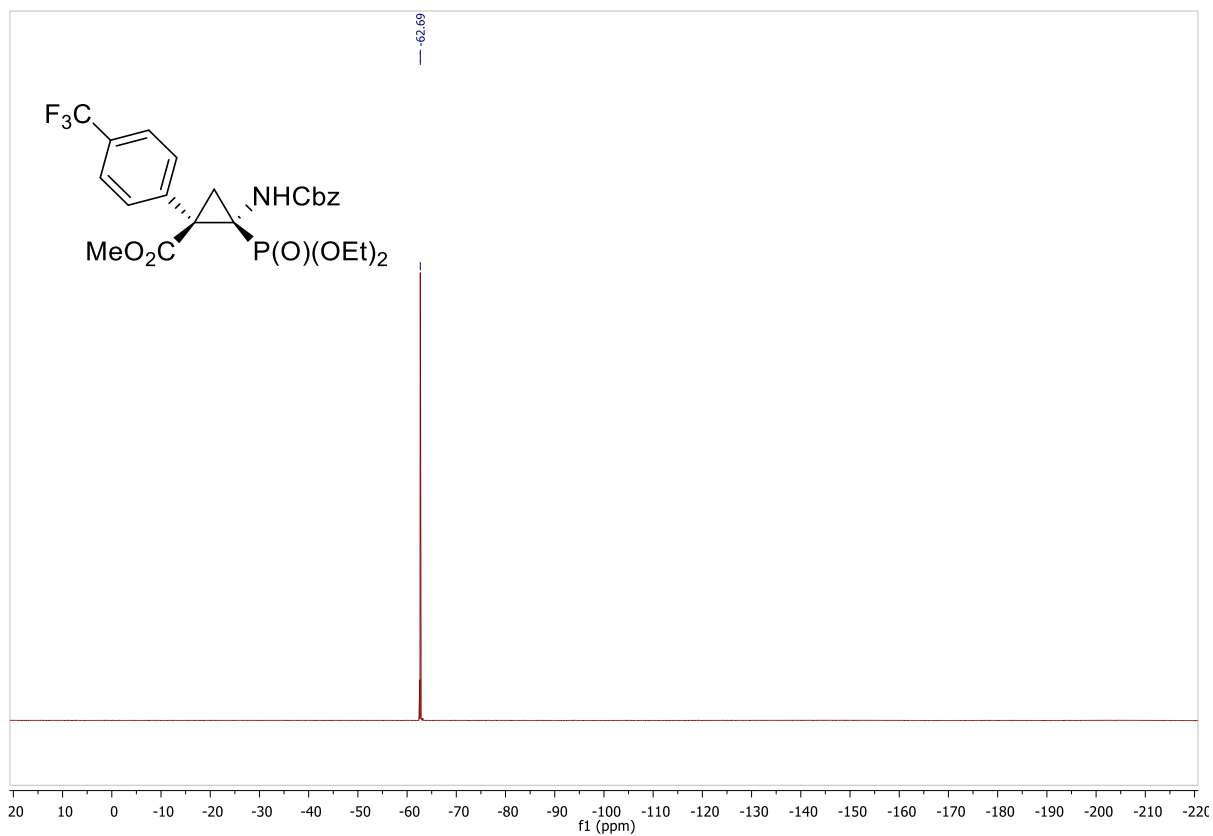
**Compound 29** : methyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-(4-iodophenyl)cyclopropane-1-carboxylate



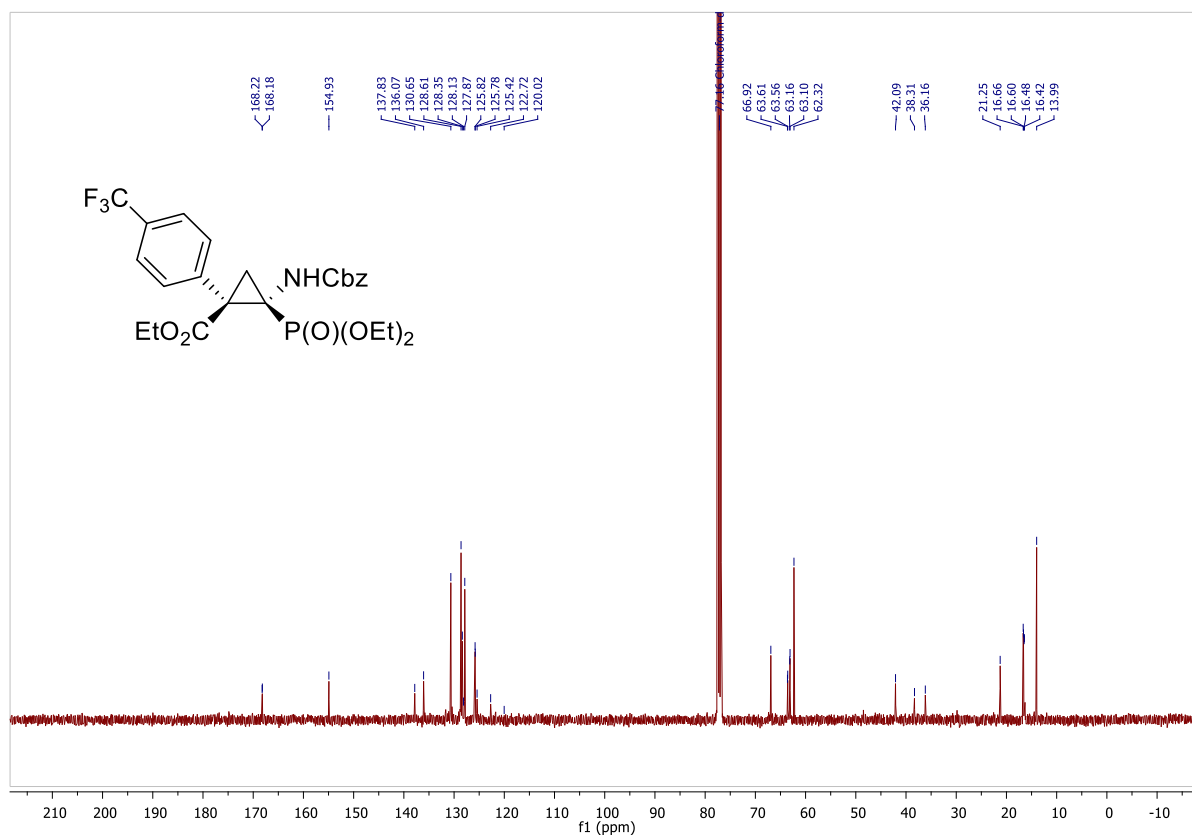
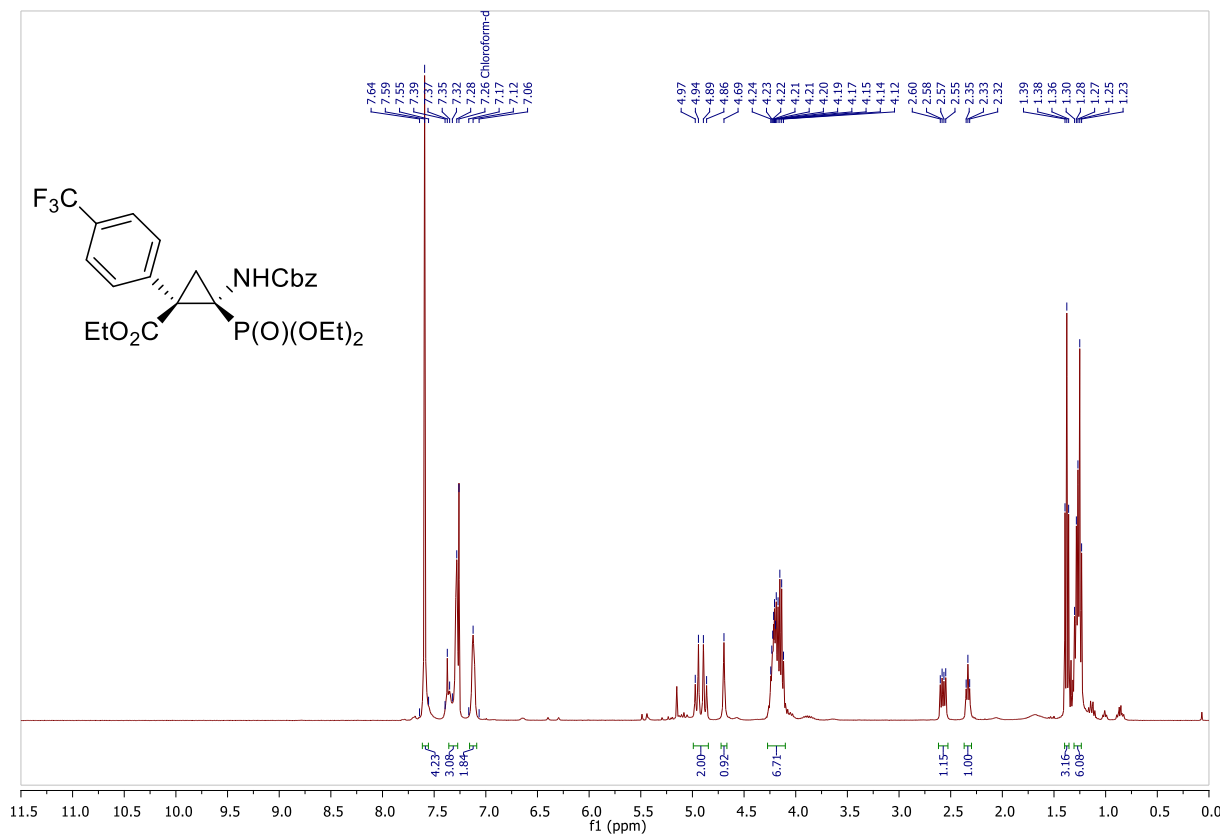


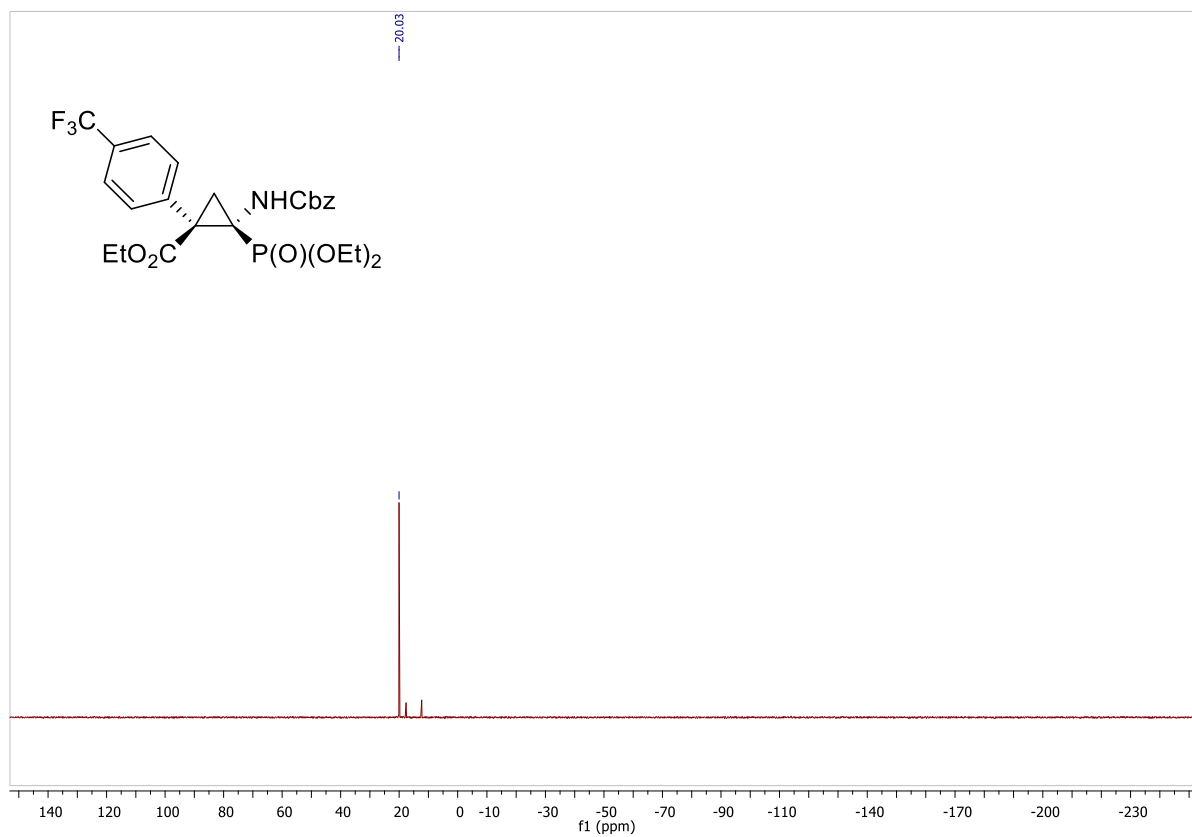
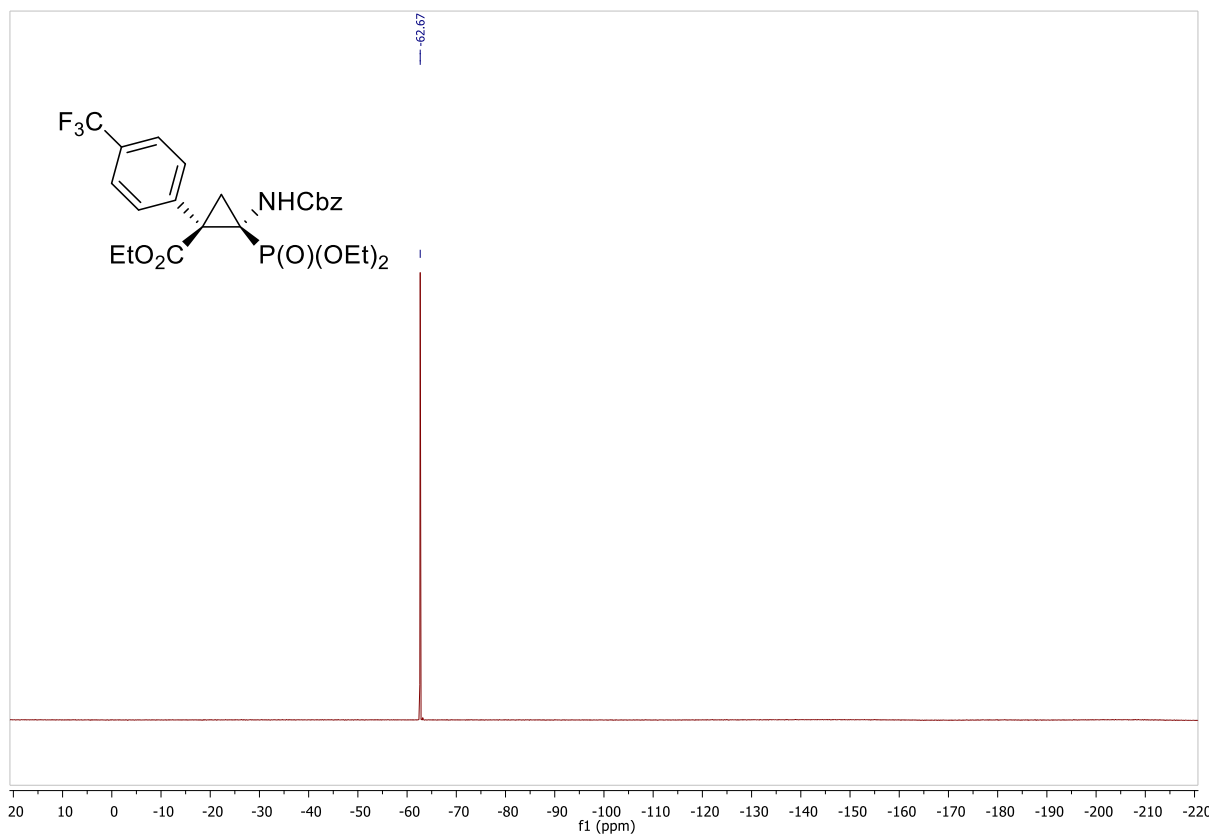
**Compound 30:** methyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-(4-(trifluoromethyl)phenyl)cyclopropane-1-carboxylate



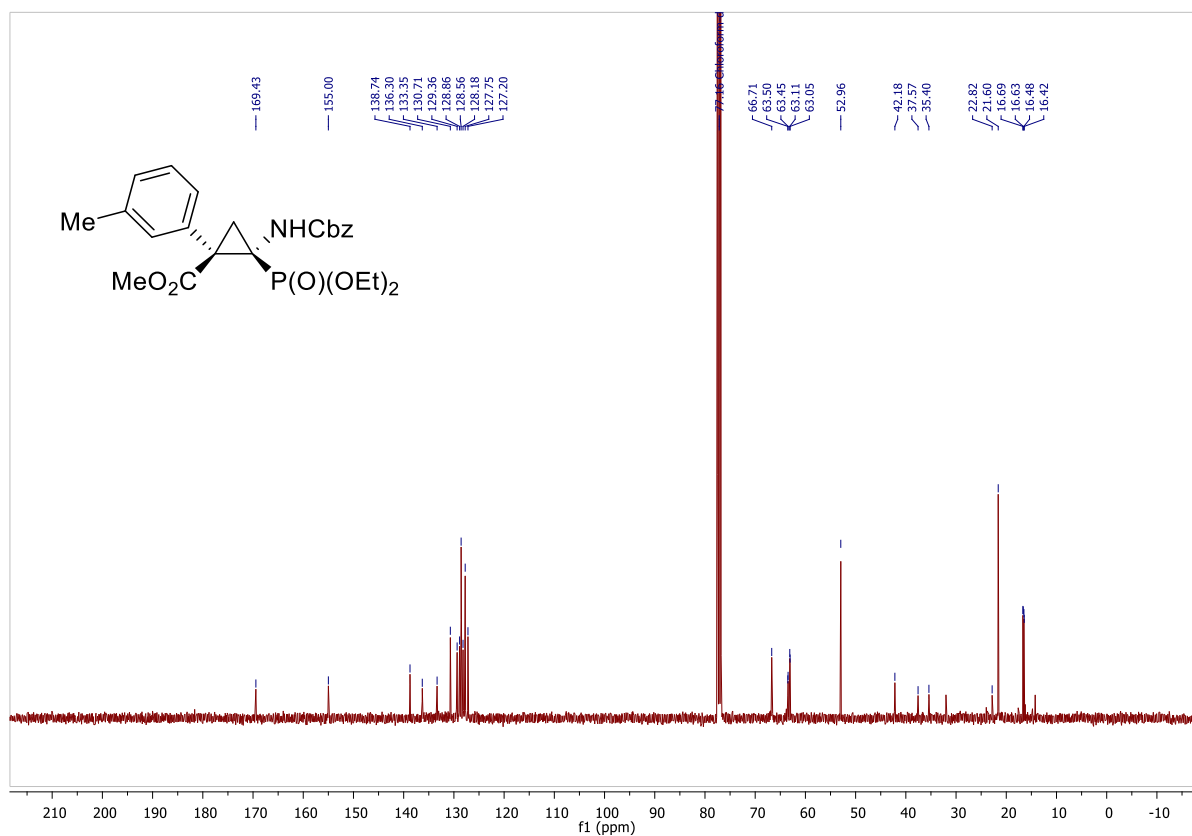
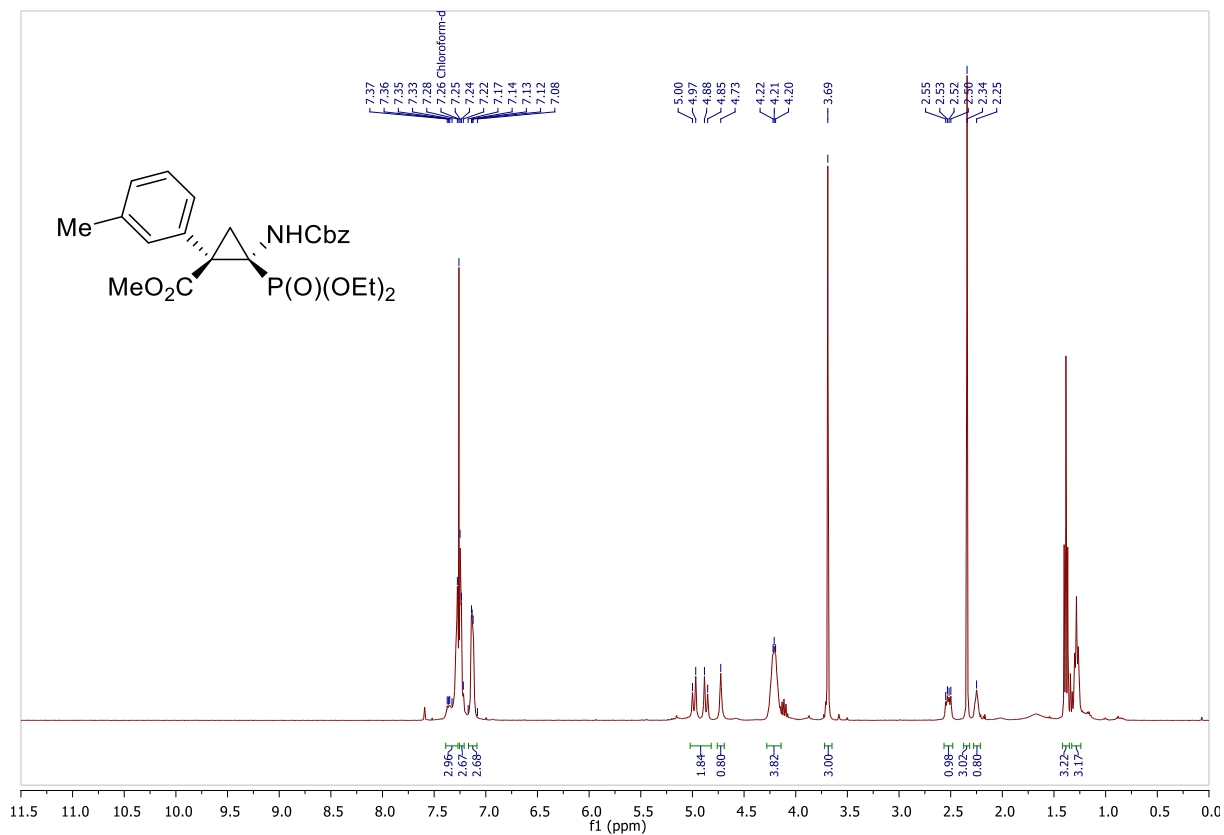


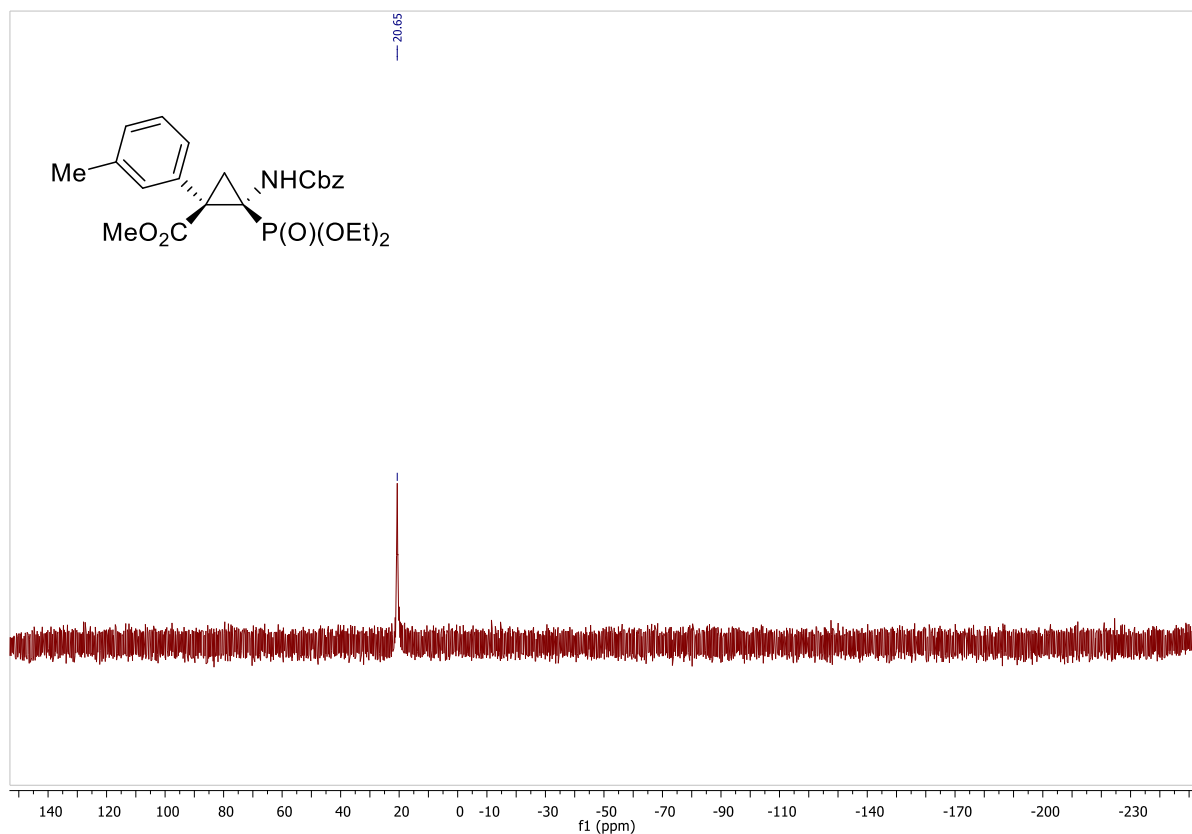
**Compound 31** : ethyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-(4-(trifluoromethyl)phenyl)cyclopropane-1-carboxylate



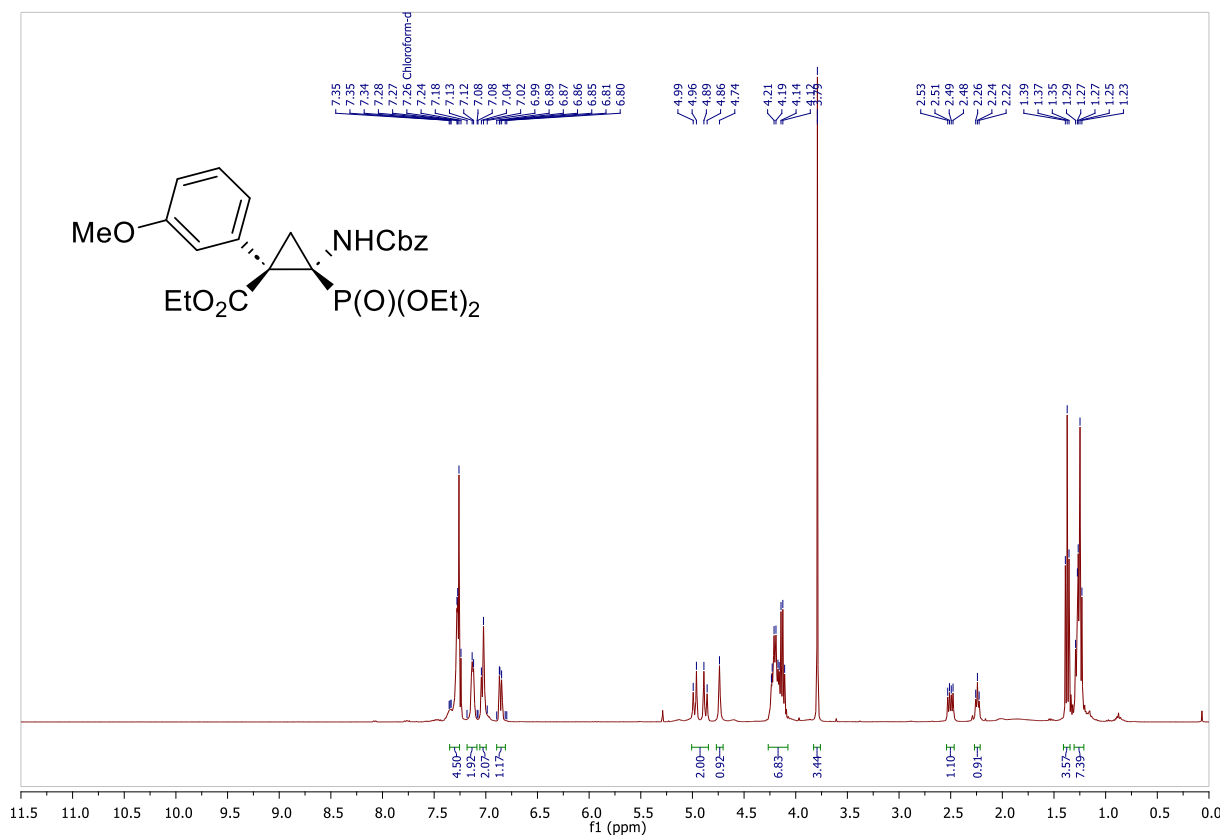


**Compound 32** : methyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-(*m*-tolyl)cyclopropane-1-carboxylate

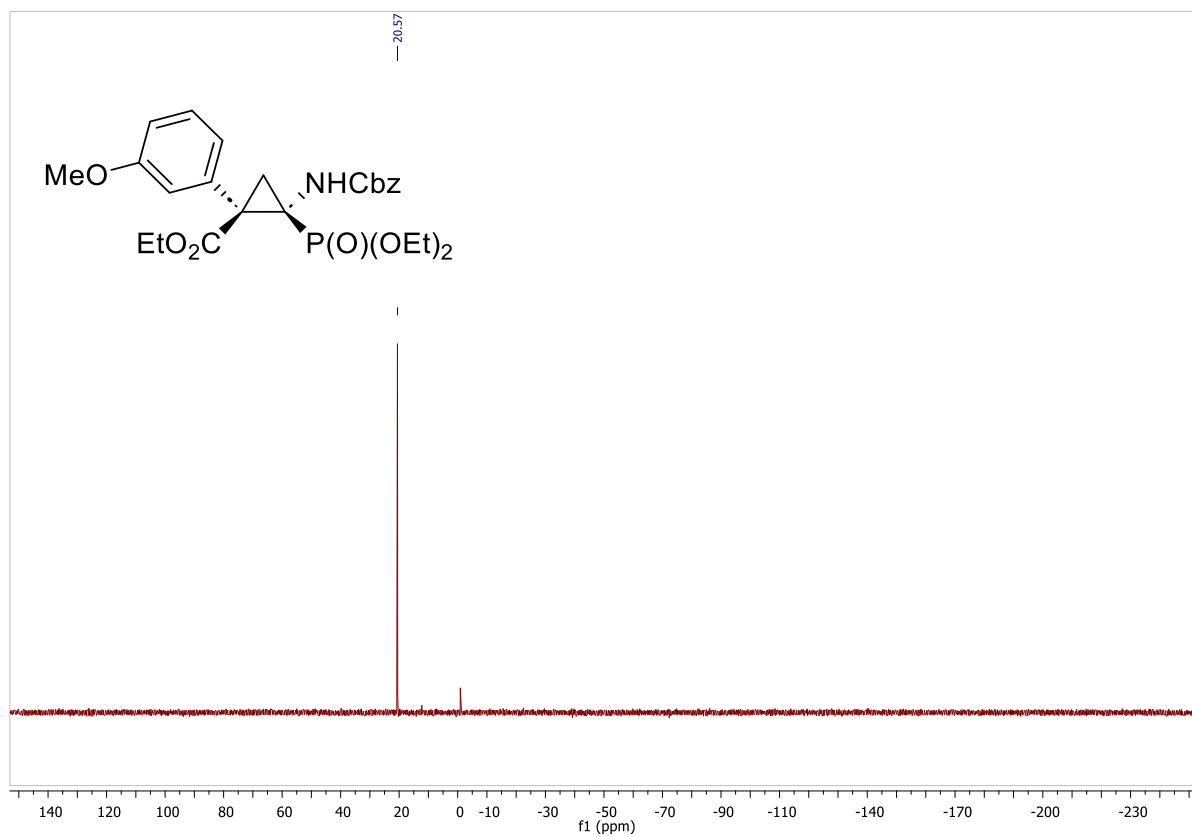
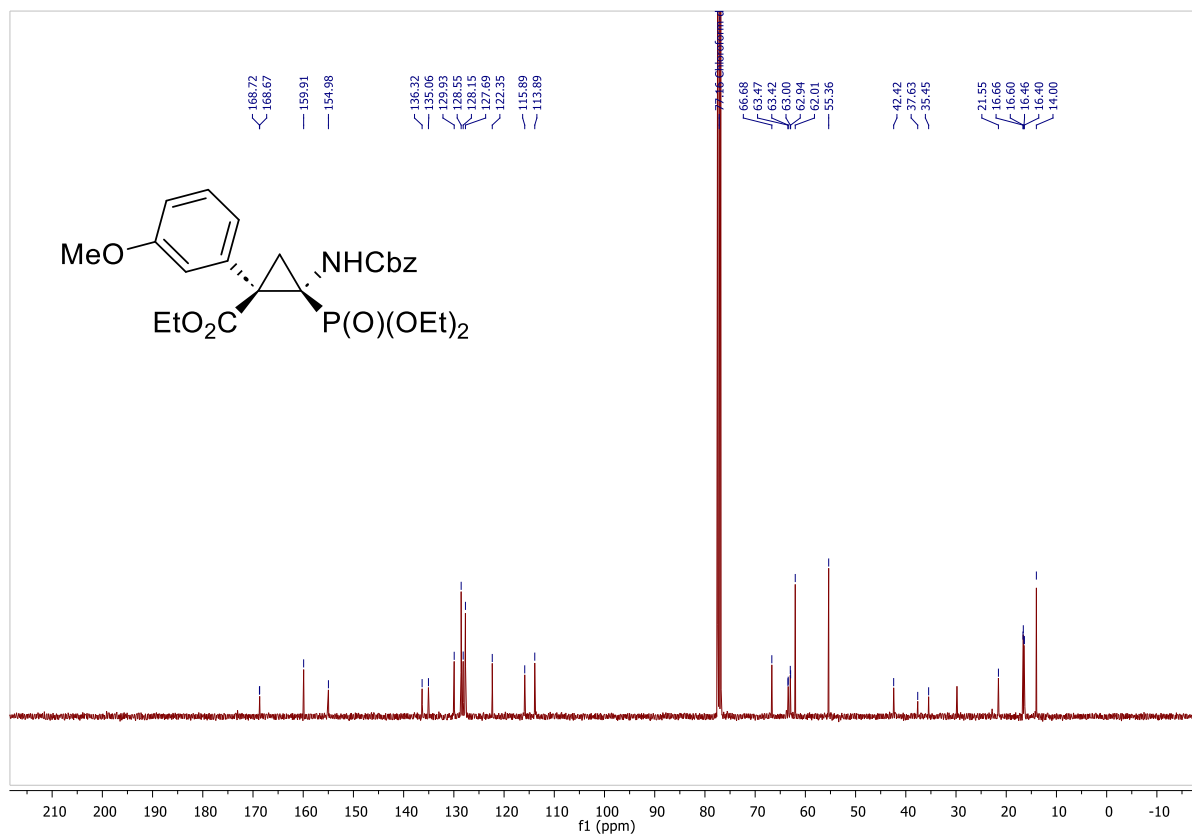




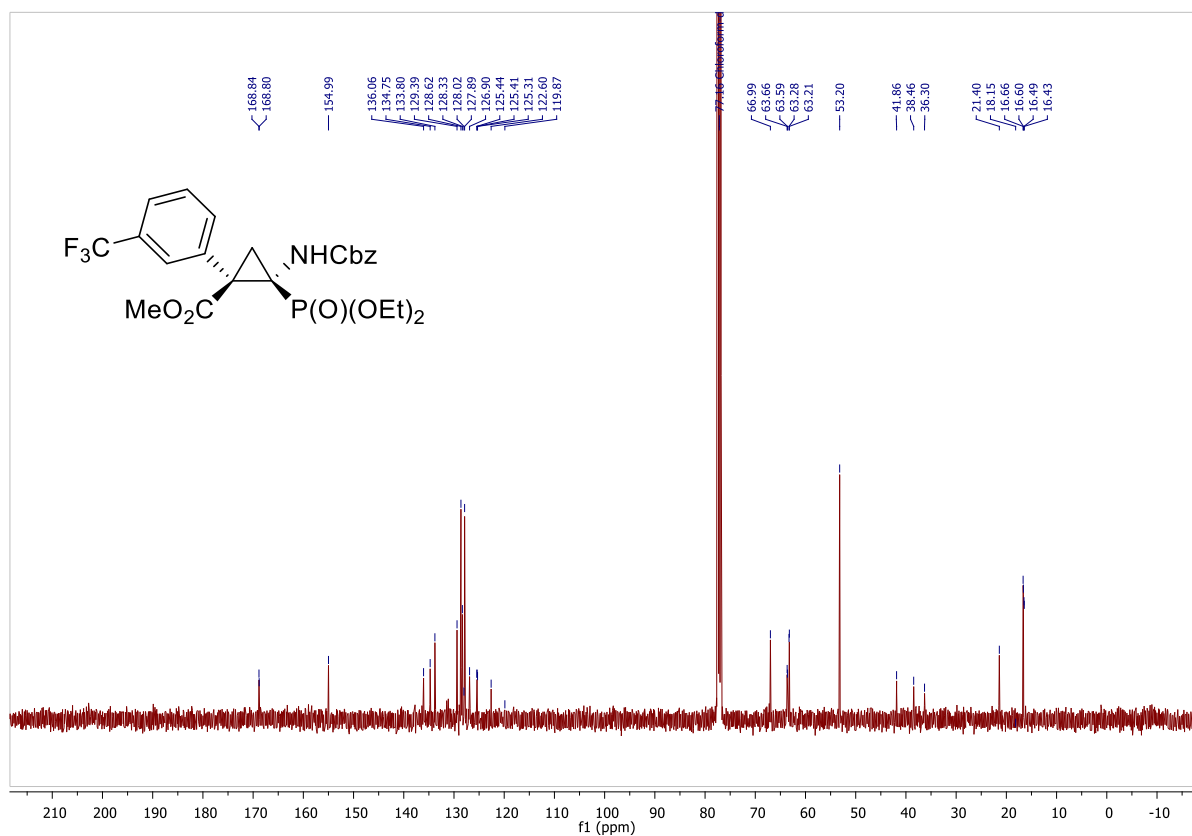
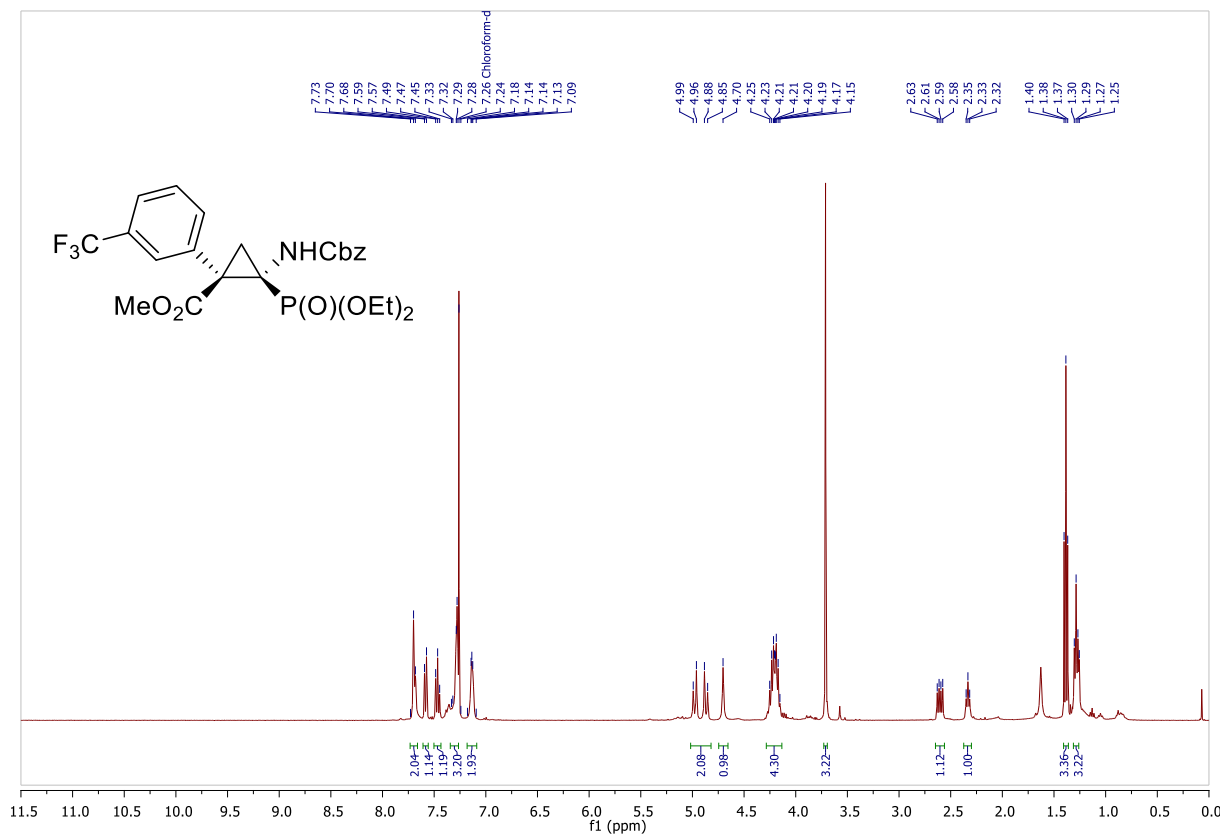
**Compound 33** : ethyl (1S,2R)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-(3-methoxyphenyl)cyclopropane-1-carboxylate

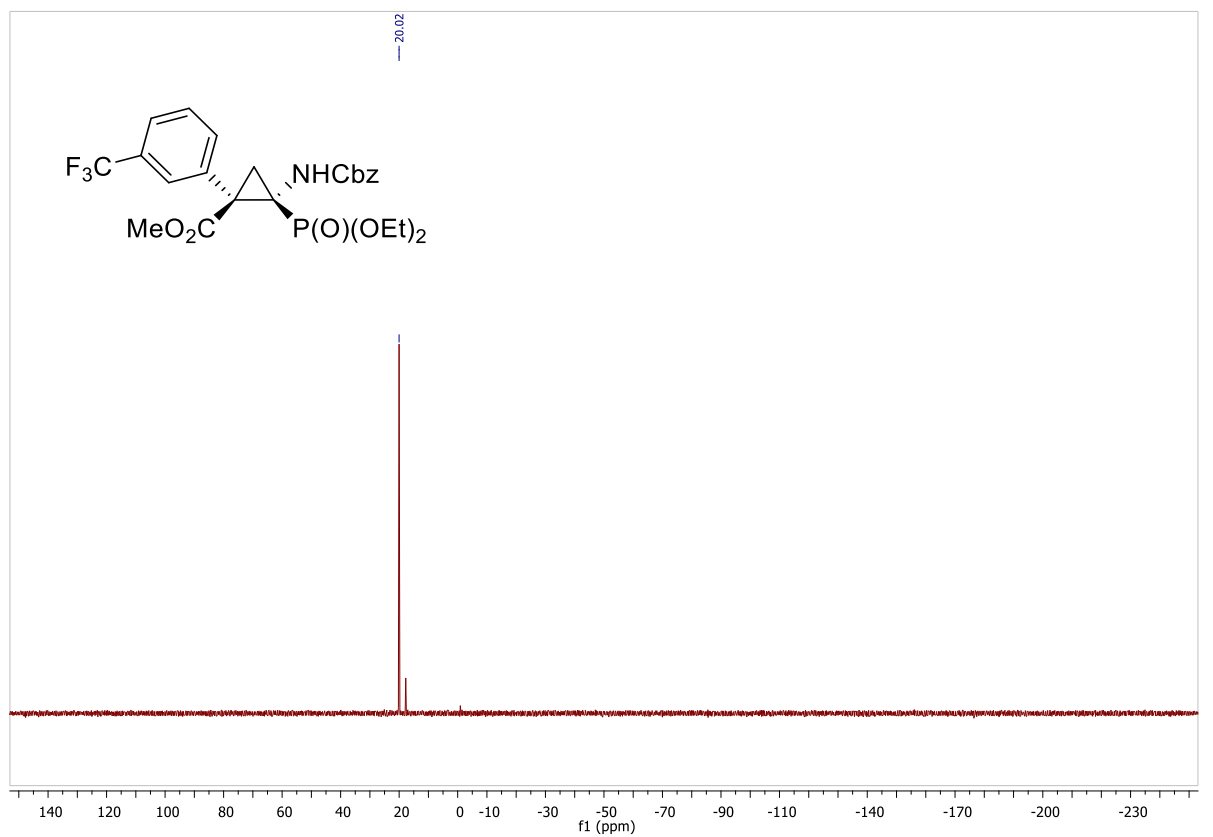
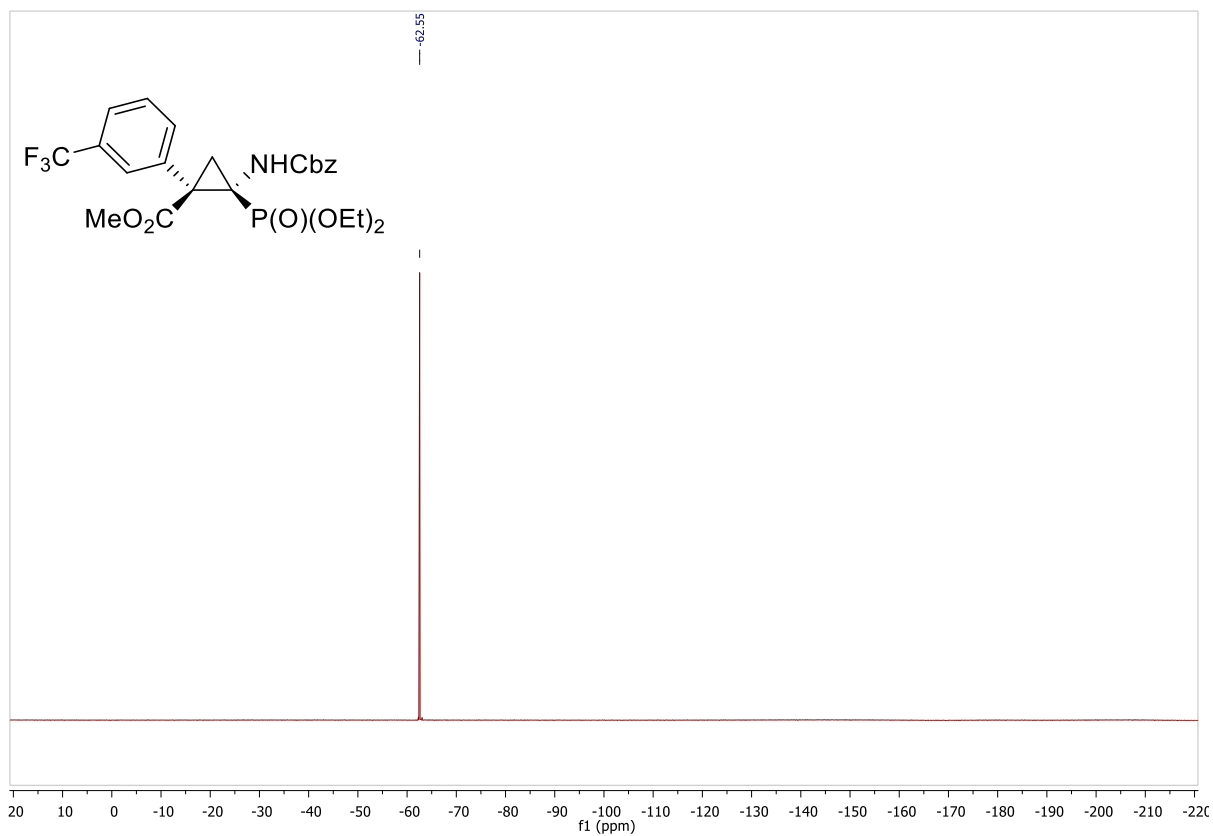




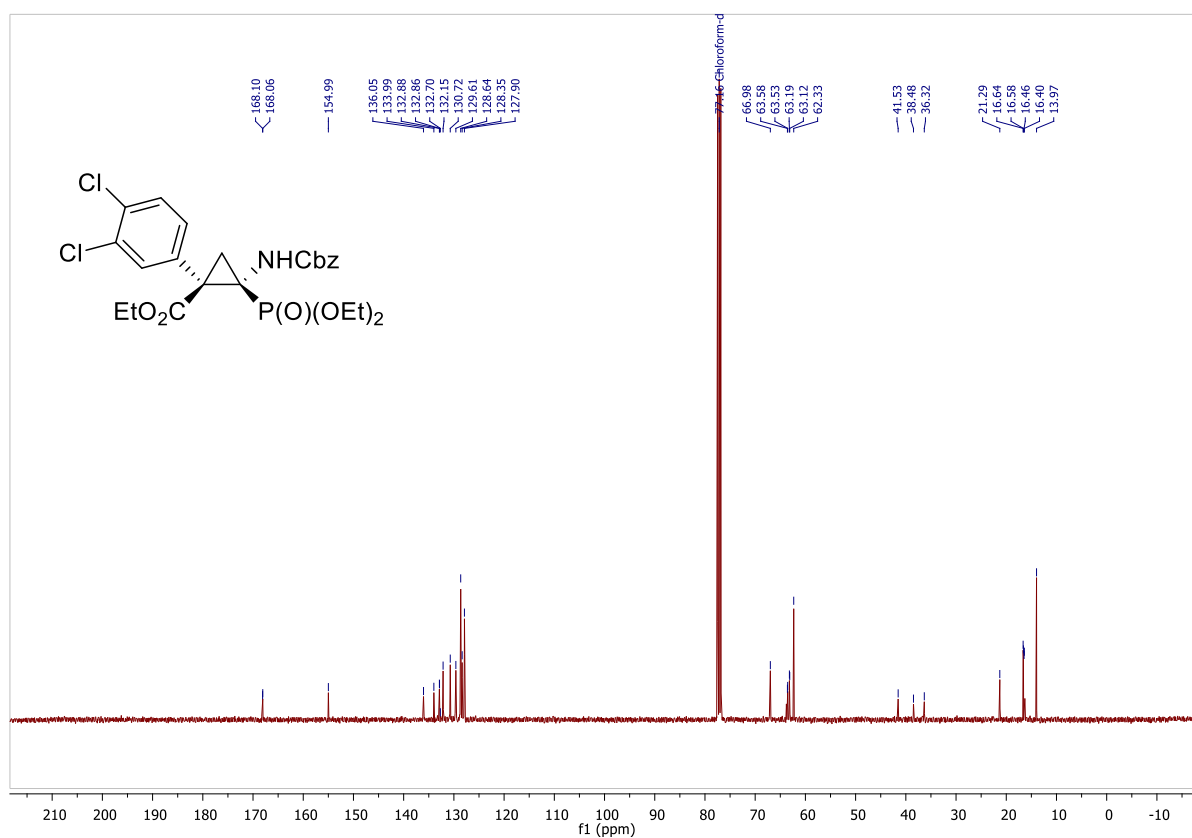
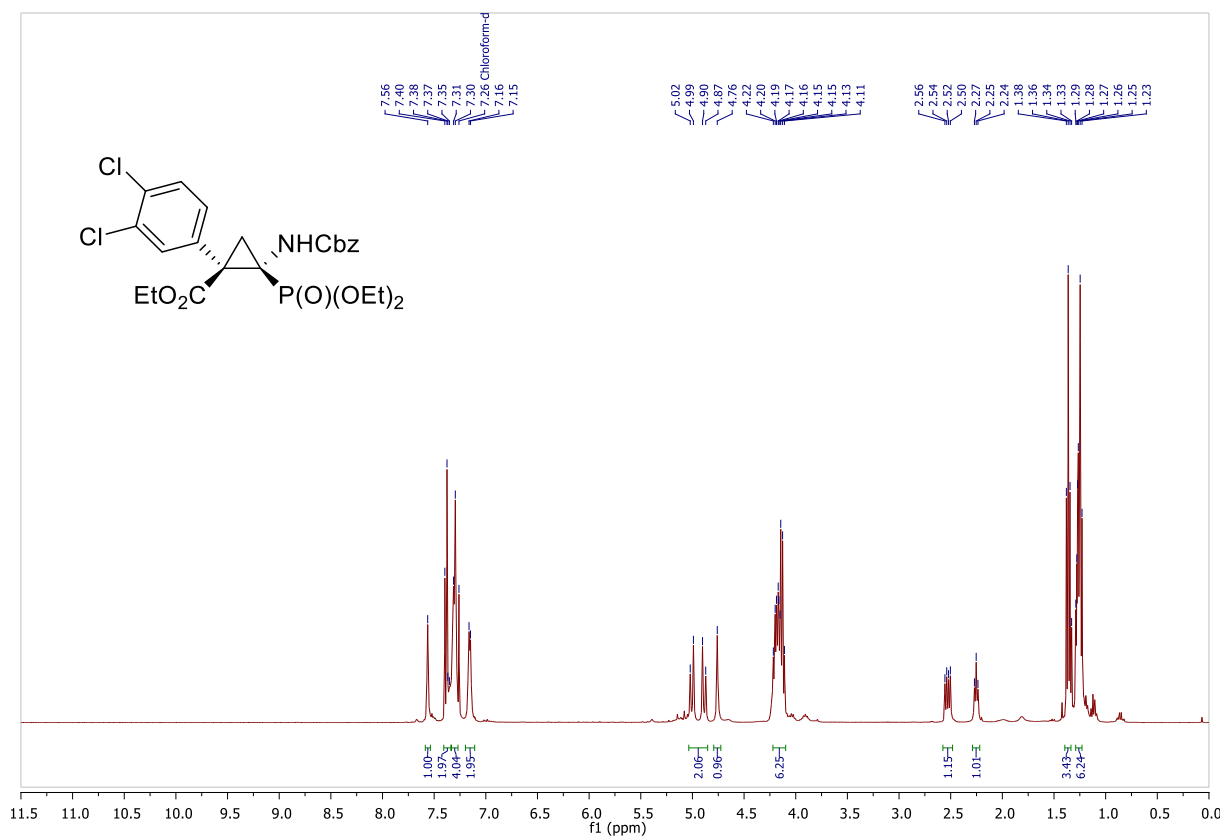


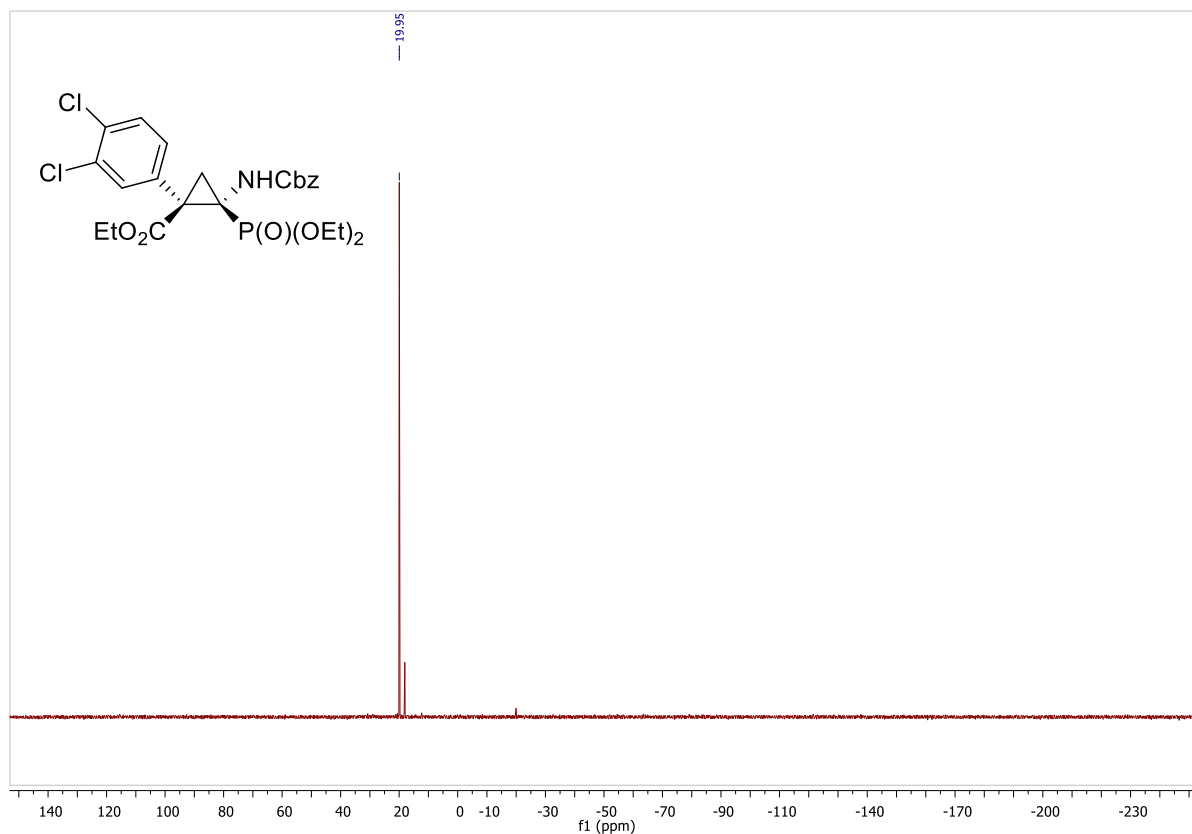
**Compound 34 : methyl (1S,2R)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-(3-(trifluoromethyl)phenyl)cyclopropane-1-carboxylate**



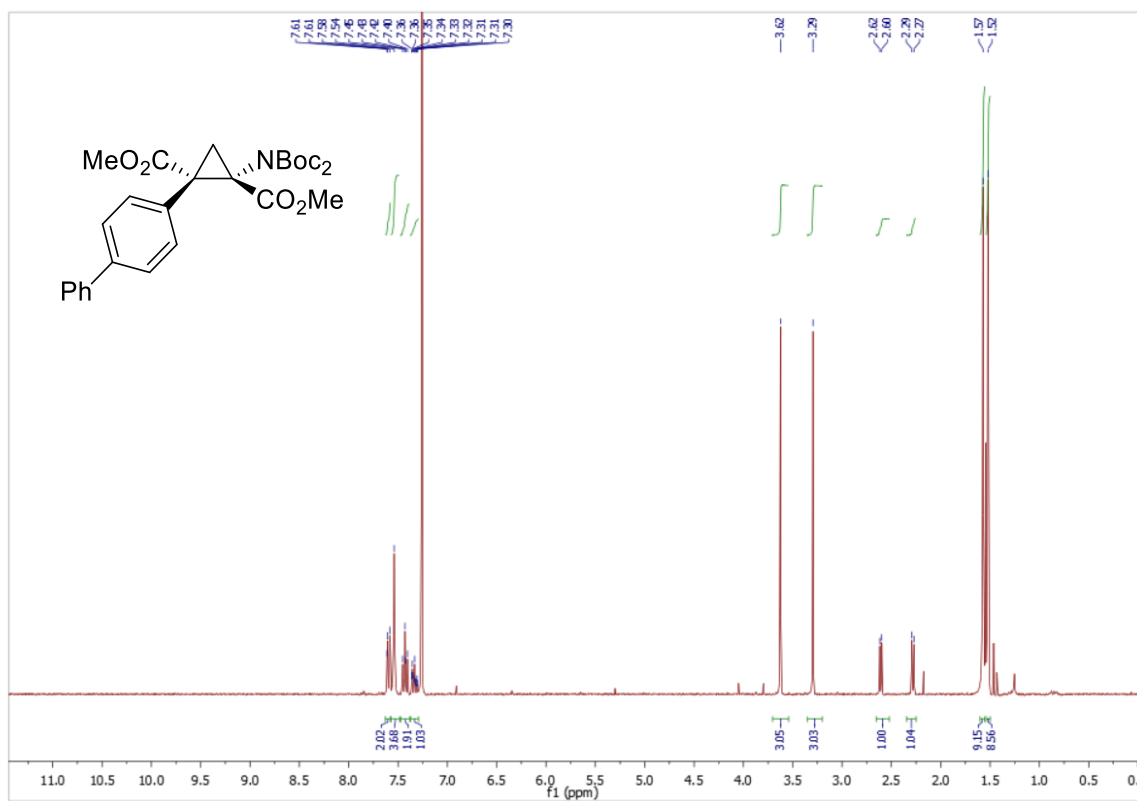


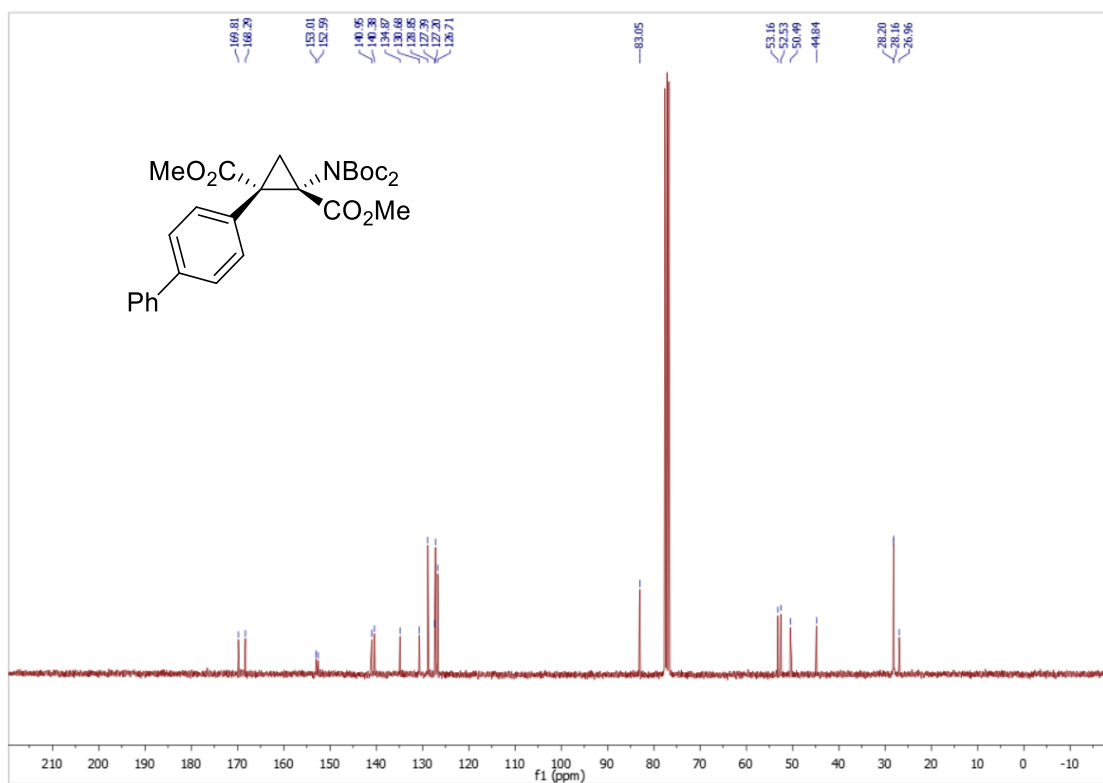
**Compound 35:** ethyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-1-(3,4-dichlorophenyl)-2-(diethoxyphosphoryl)cyclopropane-1-carboxylate



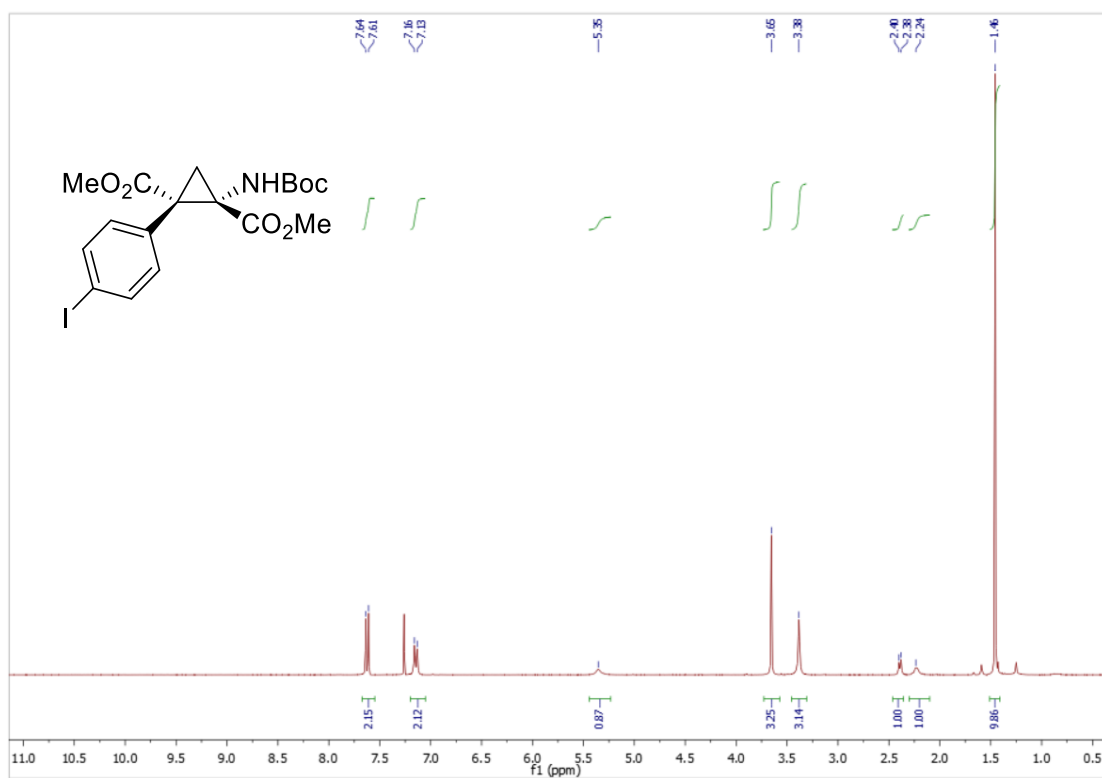


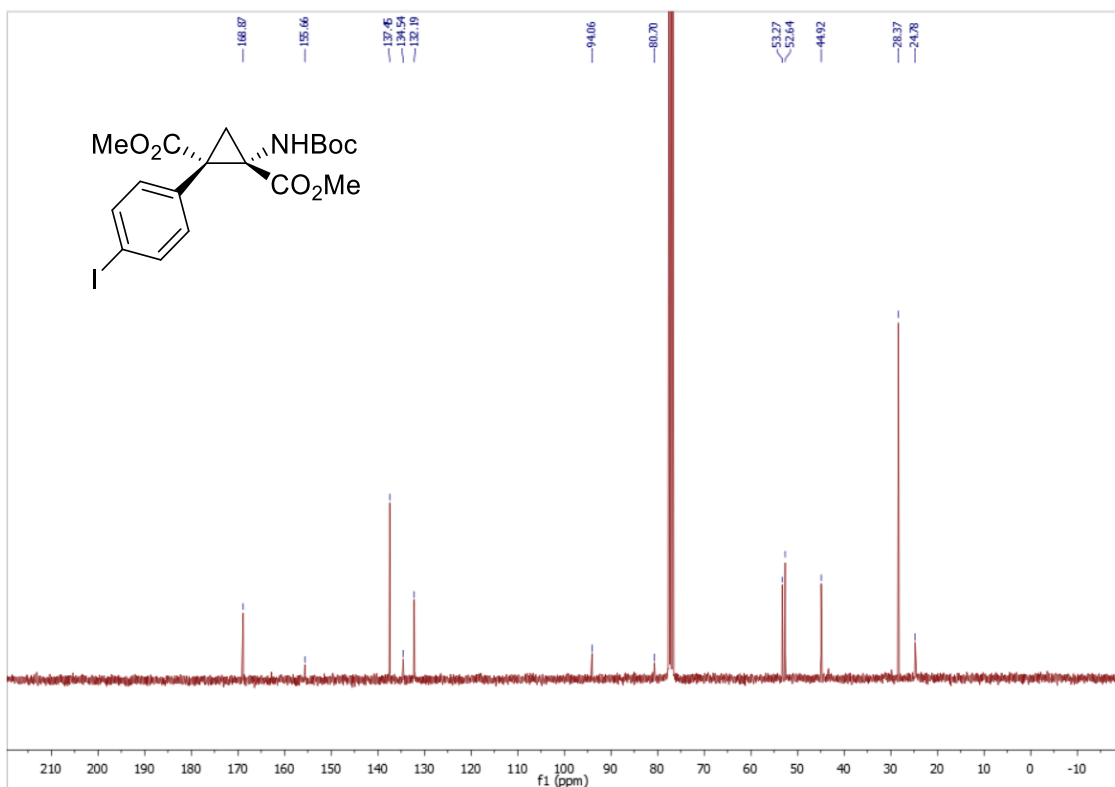
**Compound 36** : dimethyl (1S,2S)-1-([1,1'-biphenyl]-4-yl)-2-(bis(tert-butoxycarbonyl)amino)cyclopropane-1,2-dicarboxylate



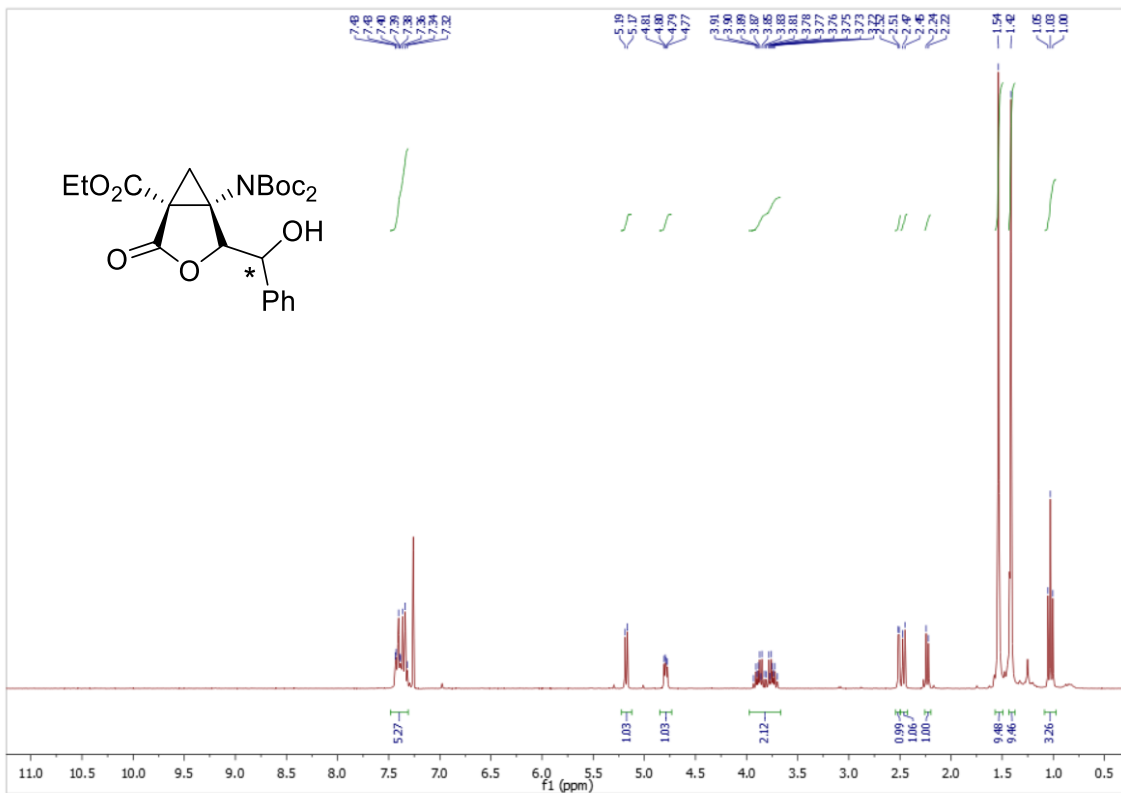


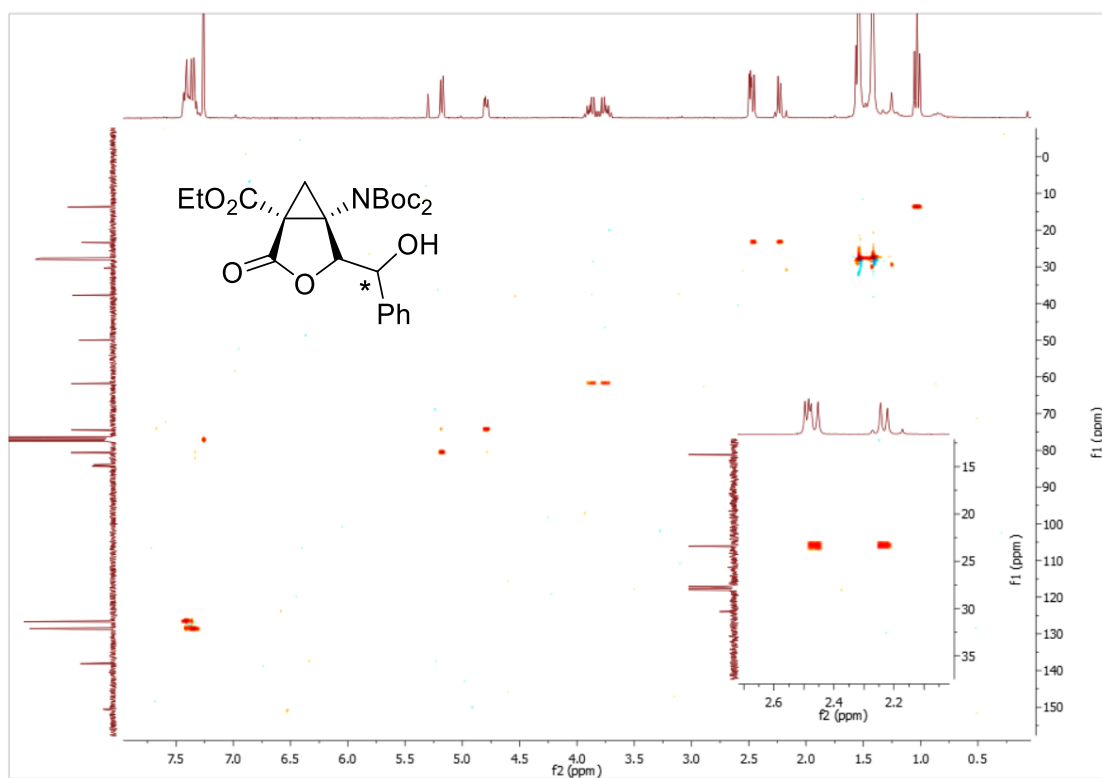
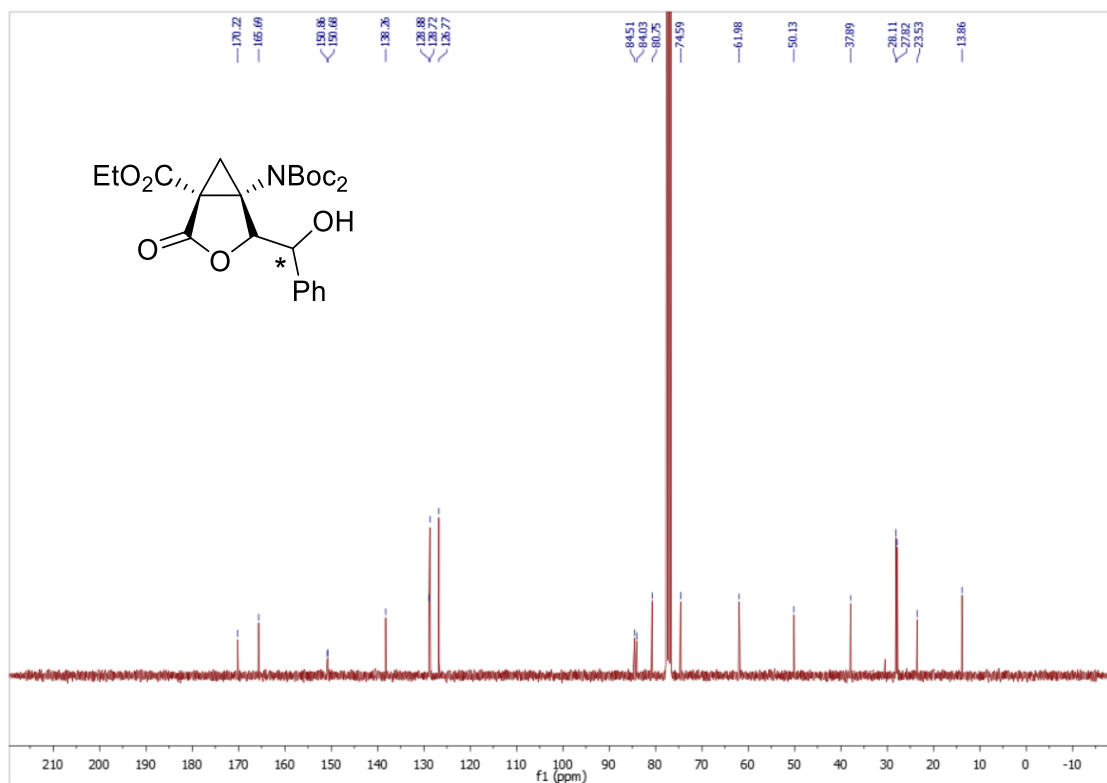
**Compound 37**: dimethyl (1*S*,2*S*)-1-((tert-butoxycarbonyl)amino)-2-(4-iodophenyl)cyclopropane-1,2-dicarboxylate





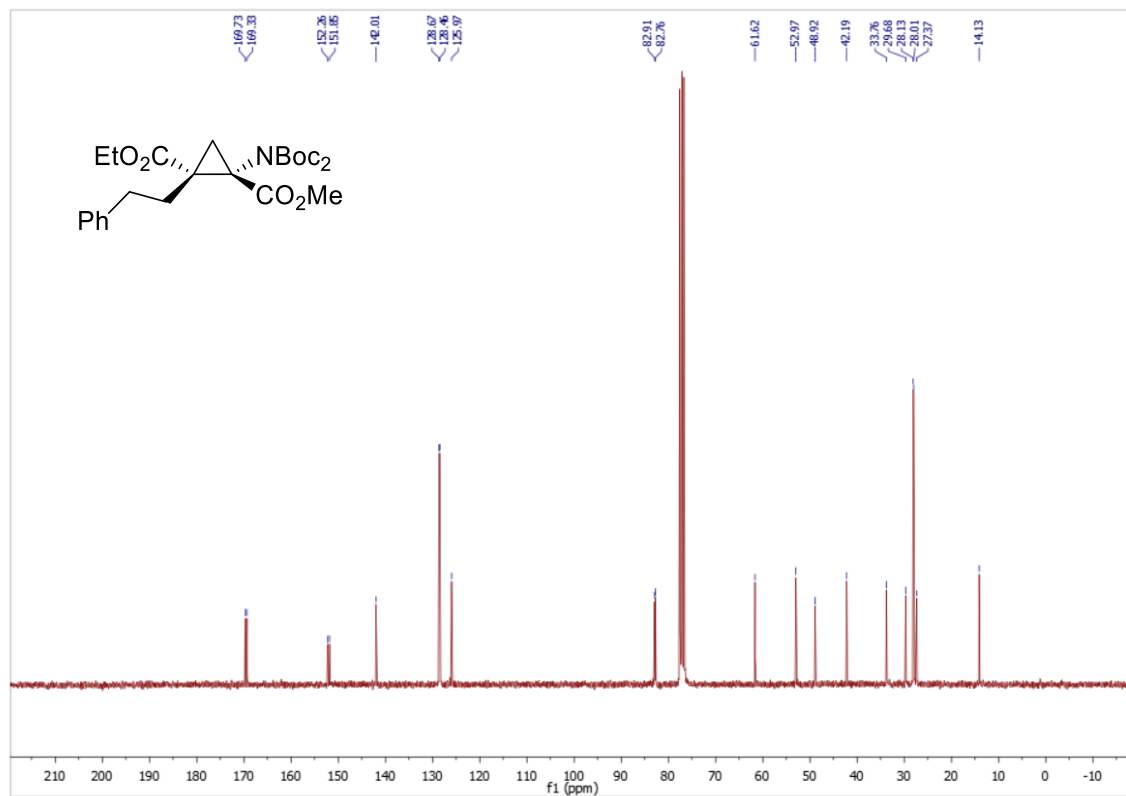
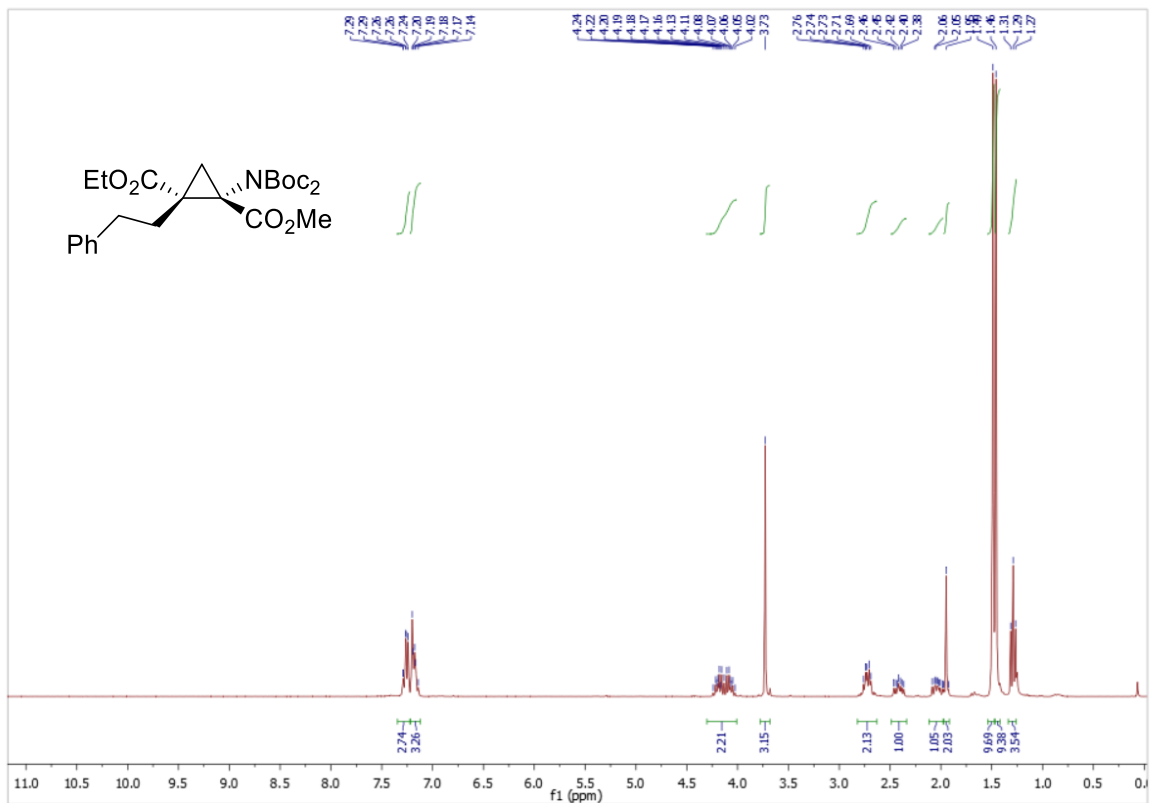
**Compound 38**: ethyl (1*R*,2*S*,5*S*)-5-(bis(tert-butoxycarbonyl)amino)-2-(hydroxy(phenyl)methyl)-4-oxo-3-oxabicyclo[3.1.0]hexane-1-carboxylate



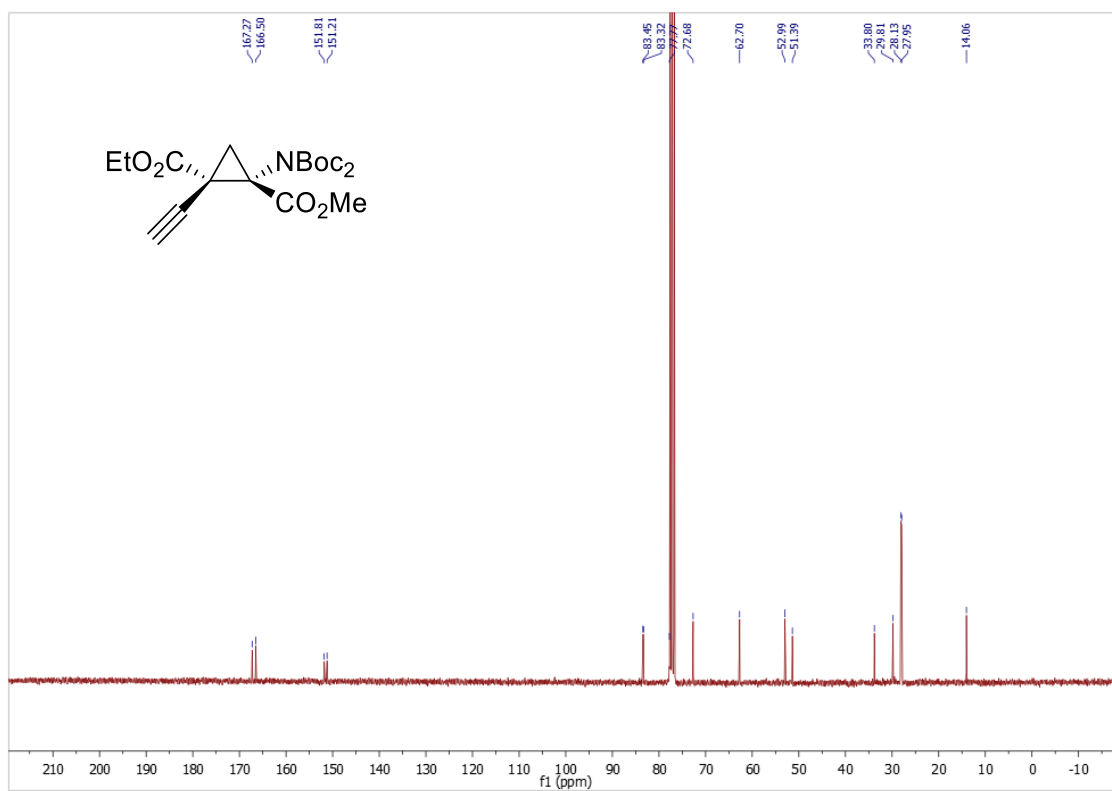
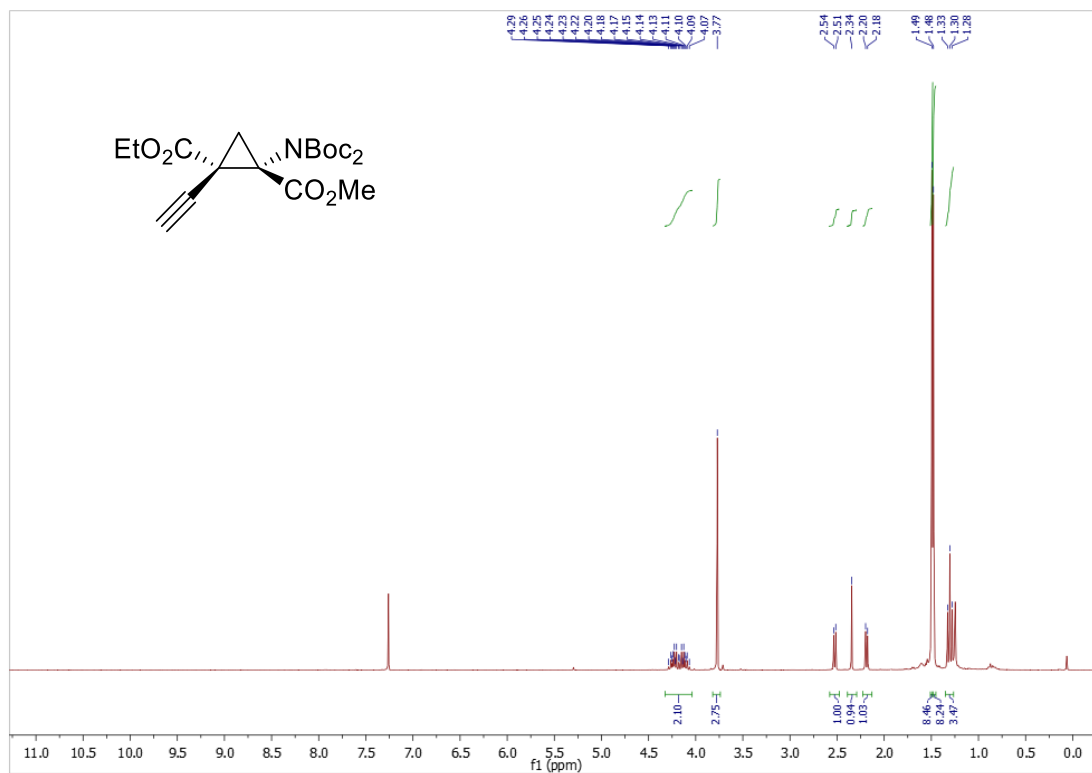




**Compound 39**: 2-ethyl 1-methyl (1*S*,2*R*)-1-(bis(tert-butoxycarbonyl)amino)-2-phenethylcyclopropane-1,2-dicarboxylate



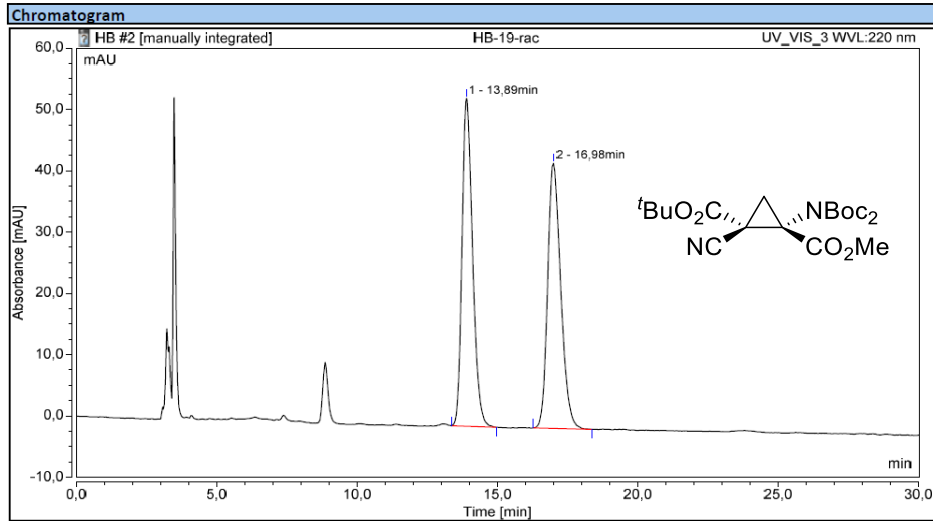
**Compound 40**: 2-ethyl 1-methyl (1*S*,2*S*)-1-(bis(tert-butoxycarbonyl)amino)-2-ethynylcyclopropane-1,2-dicarboxylate



## 7. HPLC chromatograms

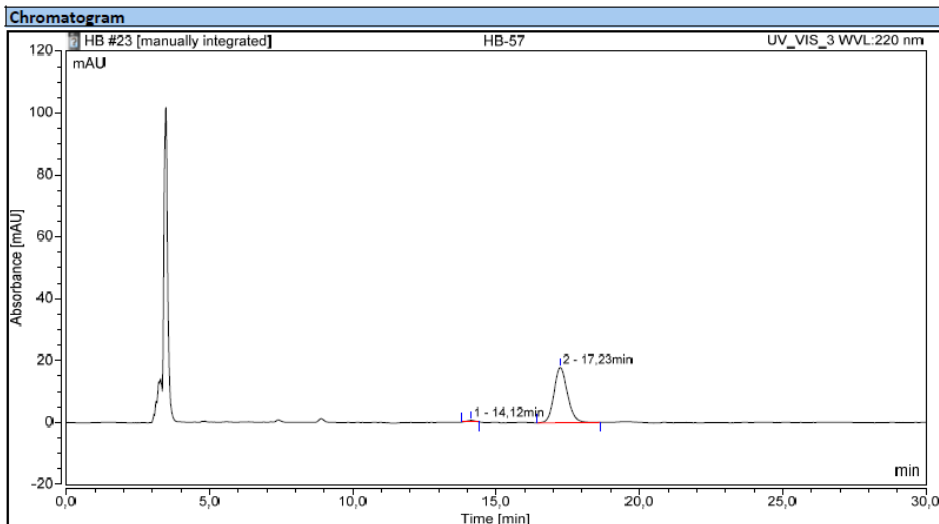
### **Compound 1** : 2-(tert-butyl) 1-methyl (1S,2R)-1-(bis(tert-butoxycarbonyl)amino)-2-cyanocyclopropane-1,2-dicarboxylate

Injection Name:	HB-78-rac	Run Time (min):	30,00
Vial Number:	RE1	Injection Volume:	10,00
Injection Type:	Unknown	Channel:	UV_VIS_3
Comment:	IC_Hept:iPrOH9:1	Wavelength:	220
Instrument Method:	90A-10C	Bandwidth:	4
Processing Method:	methode traitement	Dilution Factor:	1,0000
Injection Date/Time:	07/déc./21 09:58	Sample Weight:	1,0000



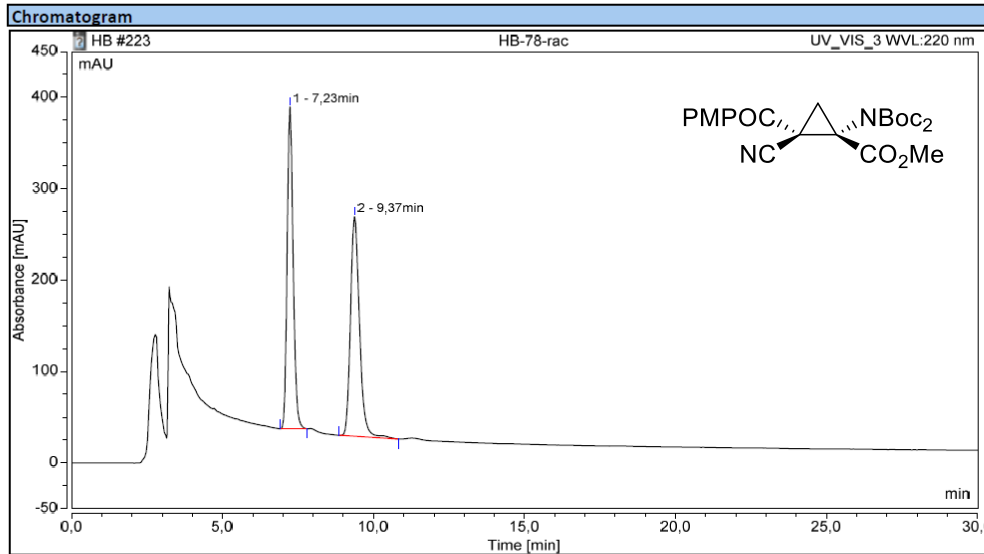
**Injection Details**

Injection Name:	HB-78-rac	Run Time (min):	30,00
Vial Number:	RB8	Injection Volume:	10,00
Injection Type:	Unknown	Channel:	UV_VIS_3
Comment:	IC_Hept:iPrOH9:1	Wavelength:	220
Instrument Method:	90A-10C	Bandwidth:	4
Processing Method:	methode traitement	Dilution Factor:	1,0000
Injection Date/Time:	20/déc./21 15:57	Sample Weight:	1,0000



**Compound 2** : methyl (1S,2R)-1-(bis(tert-butoxycarbonyl)amino)-2-cyano-2-(4-methoxybenzoyl)cyclopropane-1-carboxylate

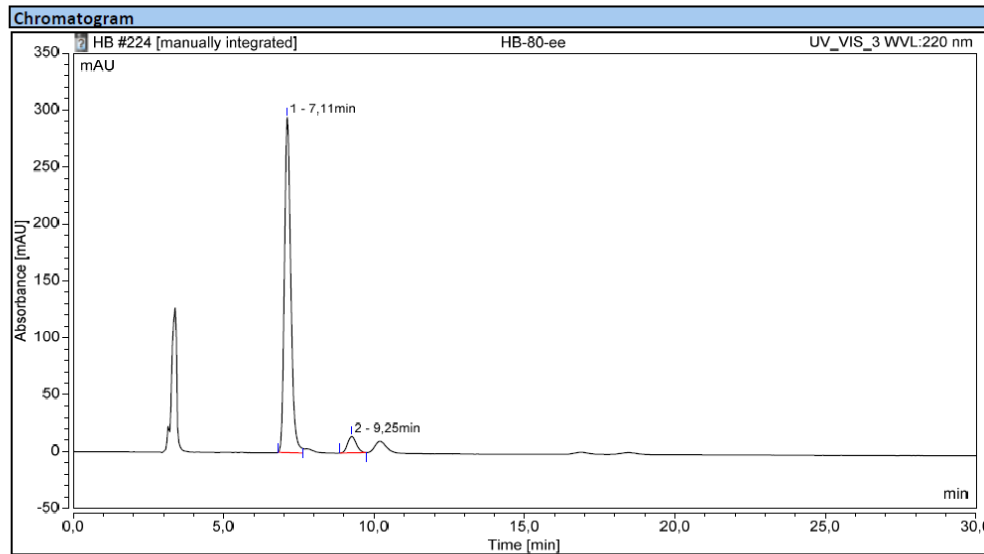
Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	RC6	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IC_Hept:iPrOH 70/30 20°C	Wavelength: 220
Instrument Method:	70A-30C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	24/mars/22 12:59	Sample Weight: 1,0000



**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		7,23	80,969	352,318	49,22	59,44
2		9,37	83,519	240,394	50,78	40,56
<b>Total:</b>			<b>164,489</b>	<b>592,712</b>	<b>100,00</b>	<b>100,00</b>

Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	RC7	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IC_Hept:iPrOH 70/30 20°C	Wavelength: 220
Instrument Method:	70A-30C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	24/mars/22 13:31	Sample Weight: 1,0000

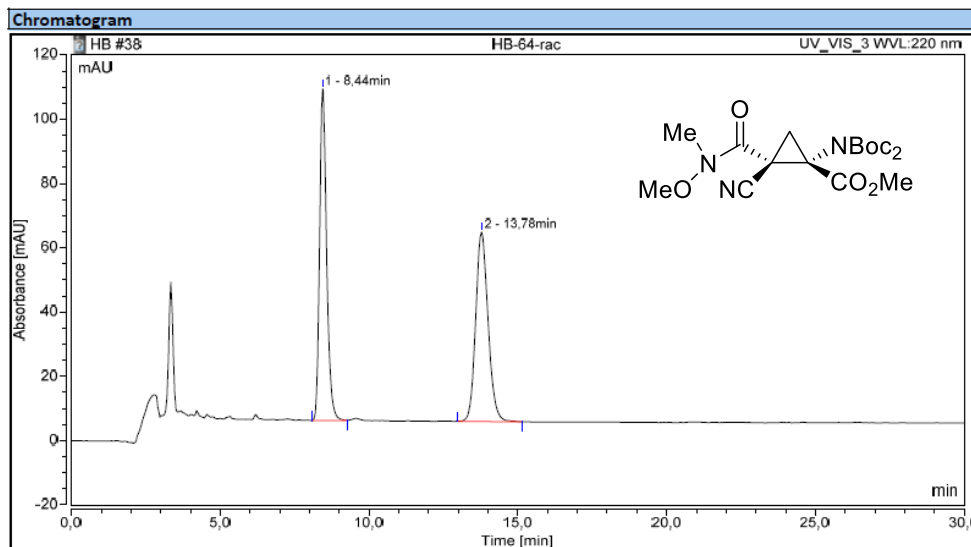


**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		7,11	71,868	294,625	93,64	95,29
2		9,25	4,881	14,573	6,36	4,71
<b>Total:</b>			<b>76,749</b>	<b>309,198</b>	<b>100,00</b>	<b>100,00</b>

**Compound 3** : methyl (1*S*,2*R*)-1-(bis(tert-butoxycarbonyl)amino)-2-cyano-2-(methoxy(methyl)carbamoyl)cyclopropane-1-carboxylate

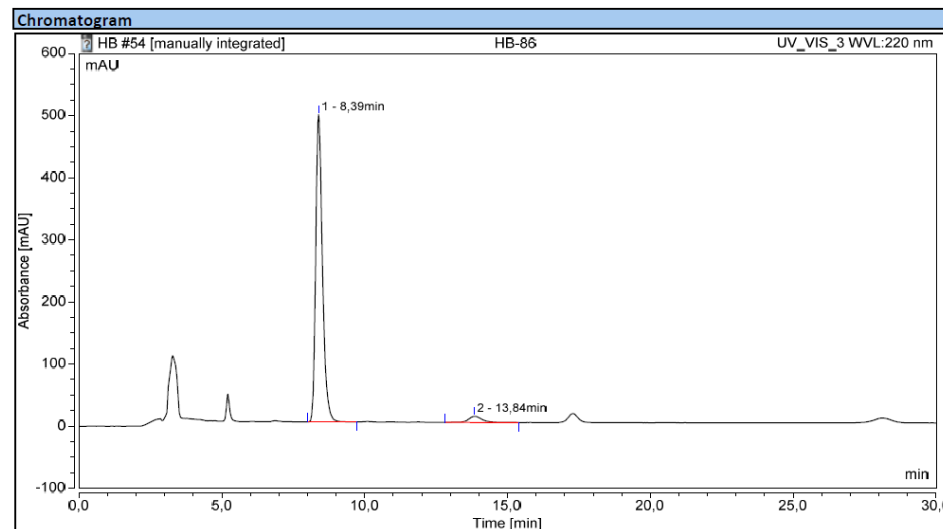
Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	RB2	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IC_Hept:iPrOH7:3	Wavelength: 220
Instrument Method:	70A-30C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	17janv./22 14:07	Sample Weight: 1,0000



**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		8,44	28,150	103,168	49,78	63,62
2		13,78	28,402	58,986	50,22	36,38
<b>Total:</b>			<b>56,552</b>	<b>162,154</b>	<b>100,00</b>	<b>100,00</b>

Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	RB7	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IC_Hept:iPrOH7:3	Wavelength: 220
Instrument Method:	70A-30C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	24/janv./22 12:02	Sample Weight: 1,0000

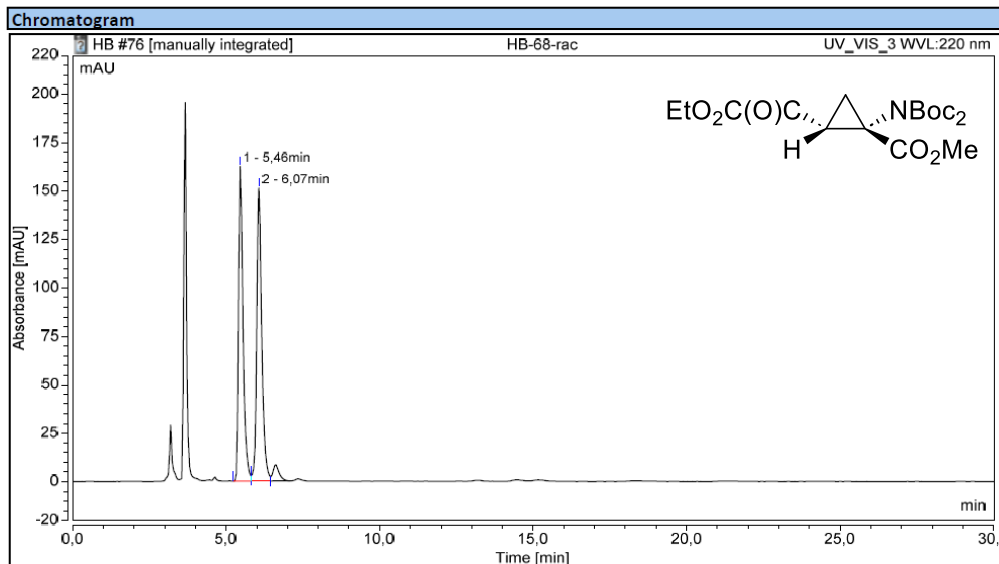


**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		8,39	135,251	494,755	96,14	98,03
2		13,84	5,425	9,958	3,86	1,97
<b>Total:</b>			<b>140,675</b>	<b>504,714</b>	<b>100,00</b>	<b>100,00</b>

**Compound 4** : methyl (1S,2R)-1-(bis(tert-butoxycarbonyl)amino)-2-(2-ethoxy-2-oxoacetyl)cyclopropane-1-carboxylate

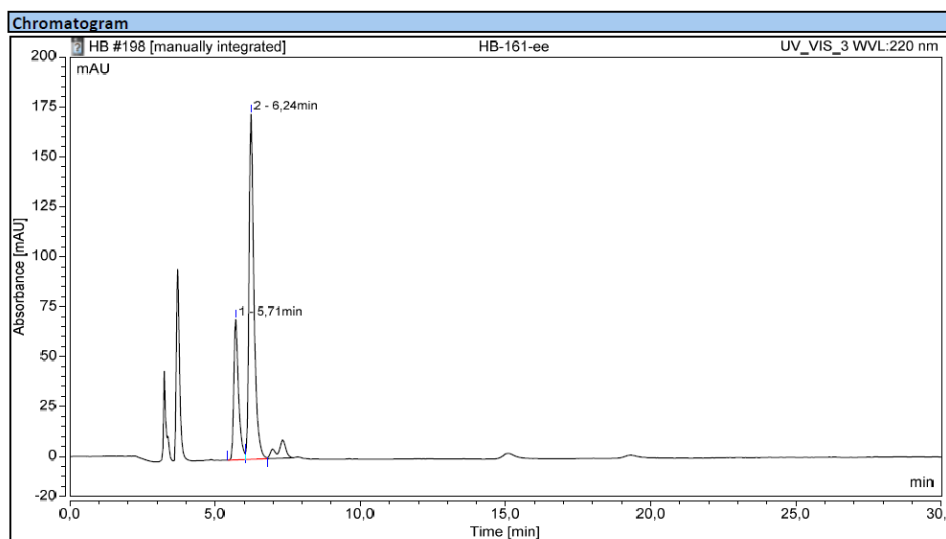
Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	RD2	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IB_Hept:iPrOH 5:0.5	Wavelength: 220
Instrument Method:	95A-5C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	26/janv./22 12:47	Sample Weight: 1,0000



**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		5,46	27,564	162,488	49,04	51,79
2		6,07	28,646	151,279	50,96	48,21
<b>Total:</b>			<b>56,210</b>	<b>313,767</b>	<b>100,00</b>	<b>100,00</b>

Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	RB4	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IB_Hept:iPrOH 95/5 20°C	Wavelength: 220
Instrument Method:	95A-5C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	10/mars/22 10:01	Sample Weight: 1,0000

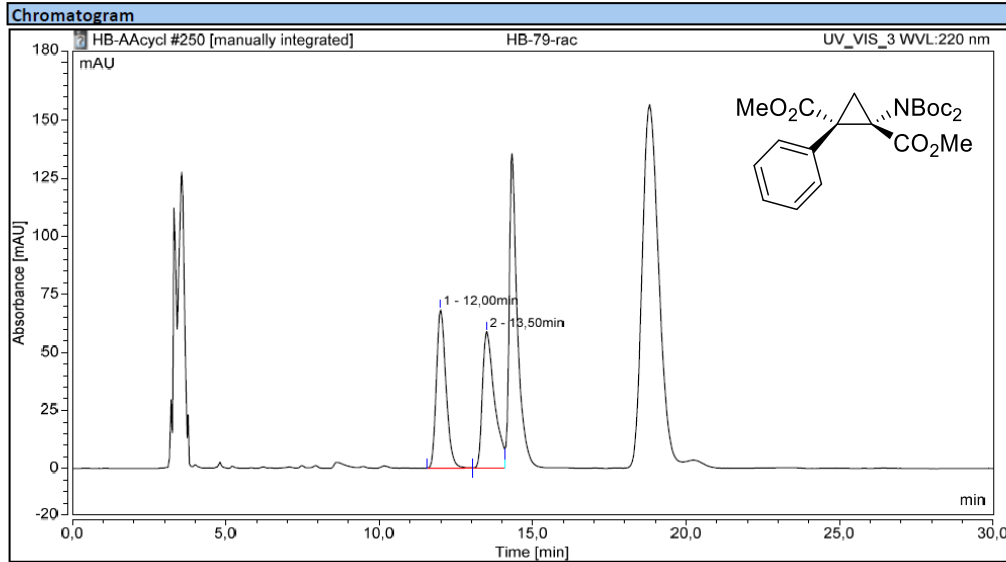


**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		5,71	13,243	70,357	27,84	28,94
2		6,24	34,326	172,788	72,16	71,06
<b>Total:</b>			<b>47,568</b>	<b>243,145</b>	<b>100,00</b>	<b>100,00</b>

**Compound 5** : dimethyl (1S,2S)-1-(bis(tert-butoxycarbonyl)amino)-2-phenylcyclopropane-1,2-dicarboxylate

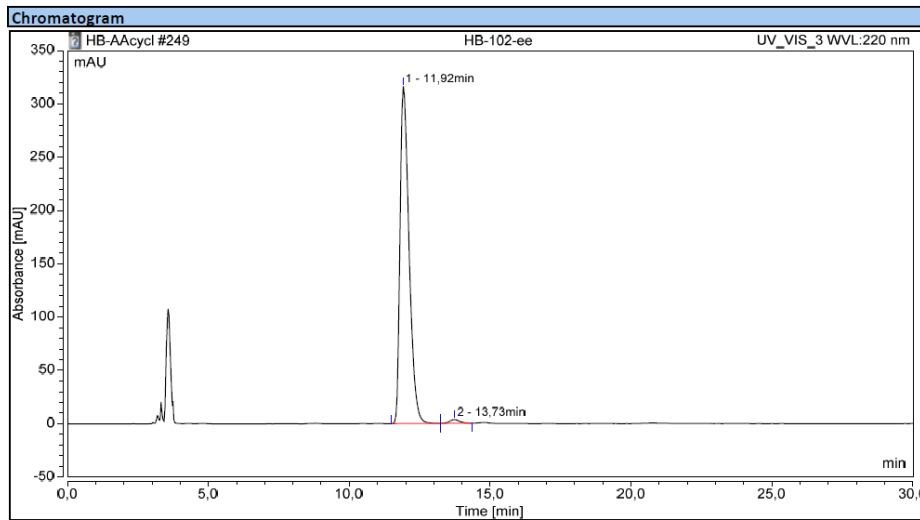
Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	GB4	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IE_Hept:iPrOH 95/5 20°C	Wavelength: 220
Instrument Method:	95A-5C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	04/avr./22 16:18	Sample Weight: 1,0000



**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		12,00	24,830	68,403	48,31	53,74
2		13,50	26,568	58,885	51,69	46,26
<b>Total:</b>			<b>51,398</b>	<b>127,288</b>	<b>100,00</b>	<b>100,00</b>

Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	GB3	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IE_Hept:iPrOH 95/5 20°C	Wavelength: 220
Instrument Method:	95A-5C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	04/avr./22 15:46	Sample Weight: 1,0000

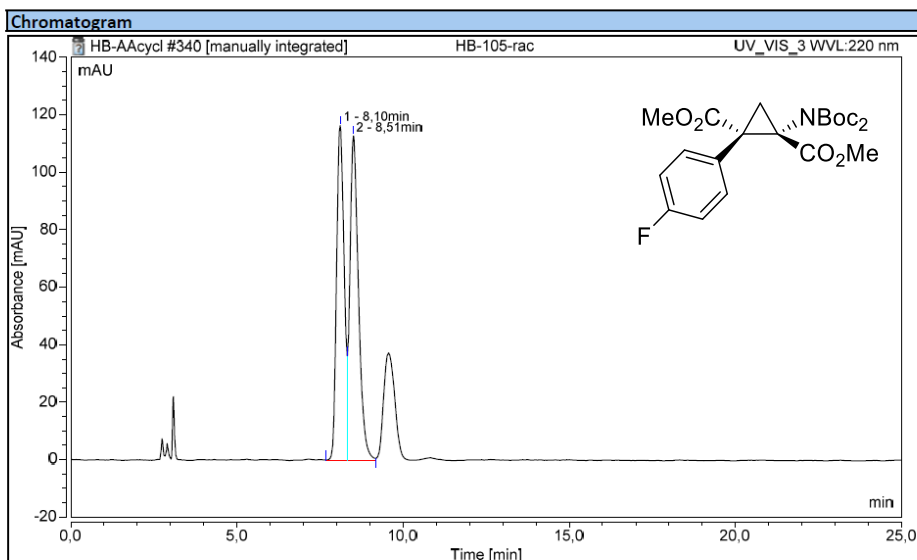


**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		11,92	116,743	316,115	98,76	98,92
2		13,73	1,461	3,465	1,24	1,08
<b>Total:</b>			<b>118,204</b>	<b>319,580</b>	<b>100,00</b>	<b>100,00</b>

**Compound 6: dimethyl (1S,2S)-1-(bis(tert-butoxycarbonyl)amino)-2-(4-fluorophenyl)cyclopropane-1,2-dicarboxylate**

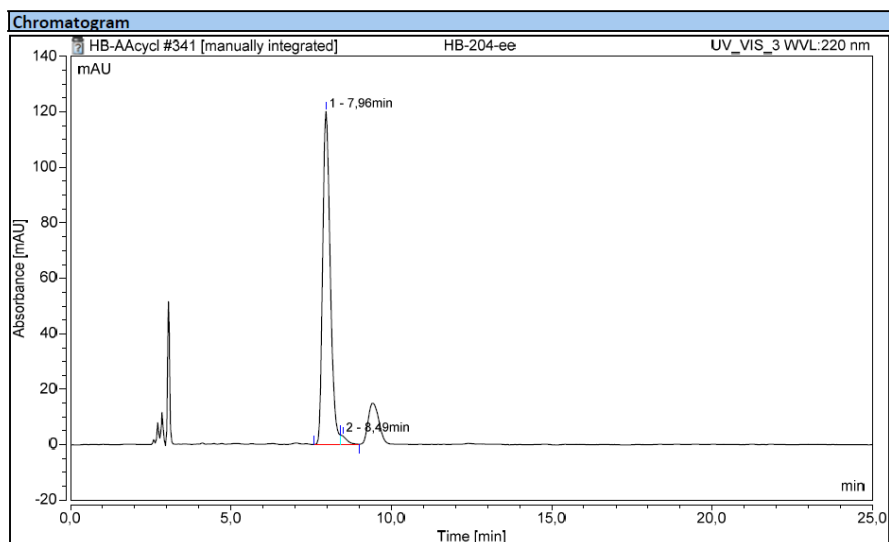
Injection Name:	HB-78-rac	Run Time (min):	25,00
Vial Number:	RC3	Injection Volume:	10,00
Injection Type:	Unknown	Channel:	UV_VIS_3
Comment:	IE_Hept:iPrOH 95/5 20°C	Wavelength:	220
Instrument Method:	95A-5C	Bandwidth:	4
Processing Method:	methode traitement	Dilution Factor:	1,0000
Injection Date/Time:	25/mal/22 12:35	Sample Weight:	1,0000



**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Resolution (EP)
1		8,10	30,537	116,436	47,45	50,79	0,89
2		8,51	33,814	112,827	52,55	49,21	n.a.
<b>Total:</b>			<b>64,351</b>	<b>229,263</b>	<b>100,00</b>	<b>100,00</b>	

Injection Name:	HB-78-rac	Run Time (min):	25,00
Vial Number:	RC4	Injection Volume:	10,00
Injection Type:	Unknown	Channel:	UV_VIS_3
Comment:	IE_Hept:iPrOH 95/5 20°C	Wavelength:	220
Instrument Method:	95A-5C	Bandwidth:	4
Processing Method:	methode traitement	Dilution Factor:	1,0000
Injection Date/Time:	25/mal/22 13:02	Sample Weight:	1,0000



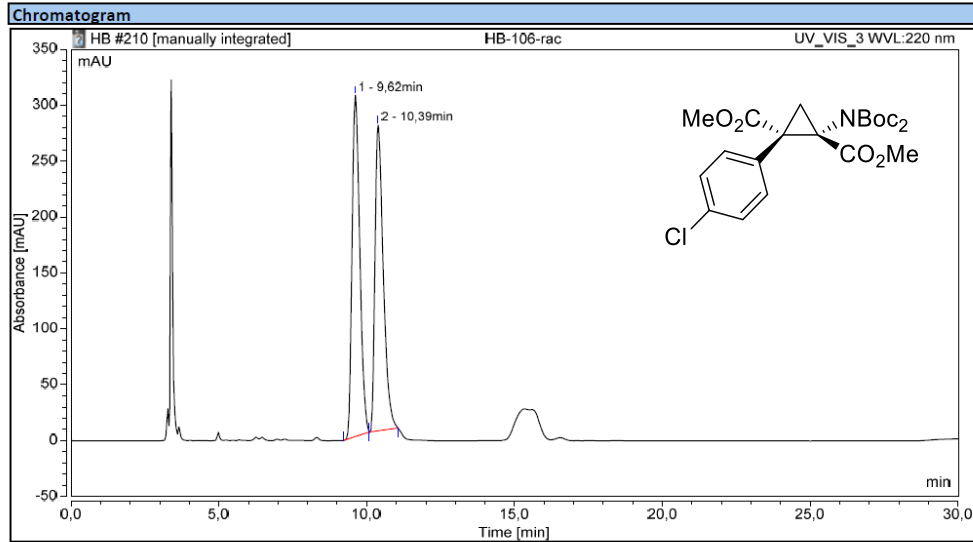
**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Resolution (EP)
1		7,96	32,829	120,075	97,75	97,49	n.a.
2		8,49	0,757	3,087	2,25	2,51	n.a.
<b>Total:</b>			<b>33,587</b>	<b>123,162</b>	<b>100,00</b>	<b>100,00</b>	



**Compound 7** : dimethyl (1S,2S)-1-(bis(tert-butoxycarbonyl)amino)-2-(4-chlorophenyl)cyclopropane-1,2-dicarboxylate

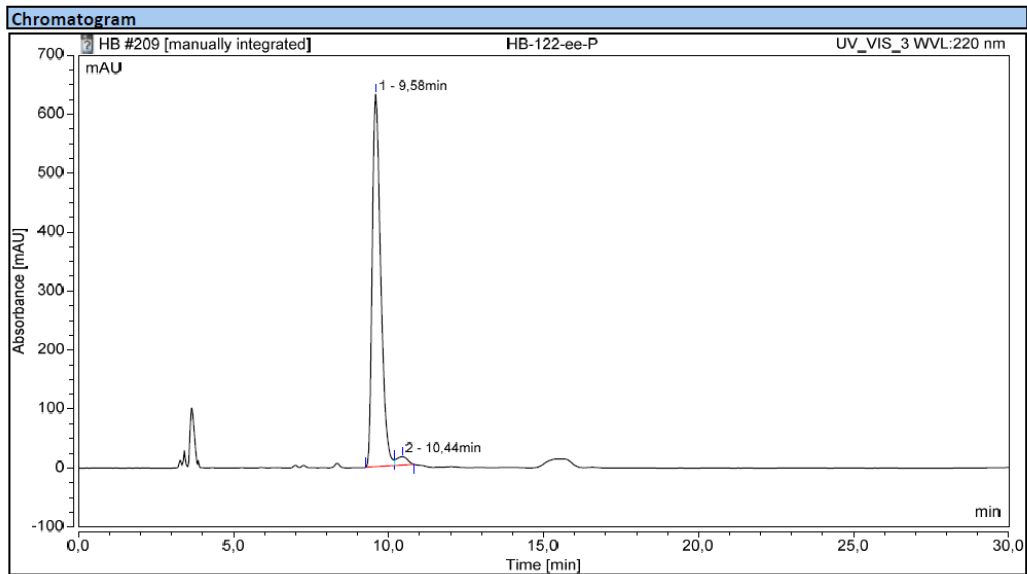
Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	GC7	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IE_Hept:iPrOH 95/5 20°C	Wavelength: 220
Instrument Method:	95A-5C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	10/mars/22 20:03	Sample Weight: 1,0000



**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		9,62	91,443	305,229	50,69	52,75
2		10,39	88,938	273,436	49,31	47,25
<b>Total:</b>			<b>180,381</b>	<b>578,664</b>	<b>100,00</b>	<b>100,00</b>

Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	GC6	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IE_Hept:iPrOH 95/5 20°C	Wavelength: 220
Instrument Method:	95A-5C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	10/mars/22 19:32	Sample Weight: 1,0000

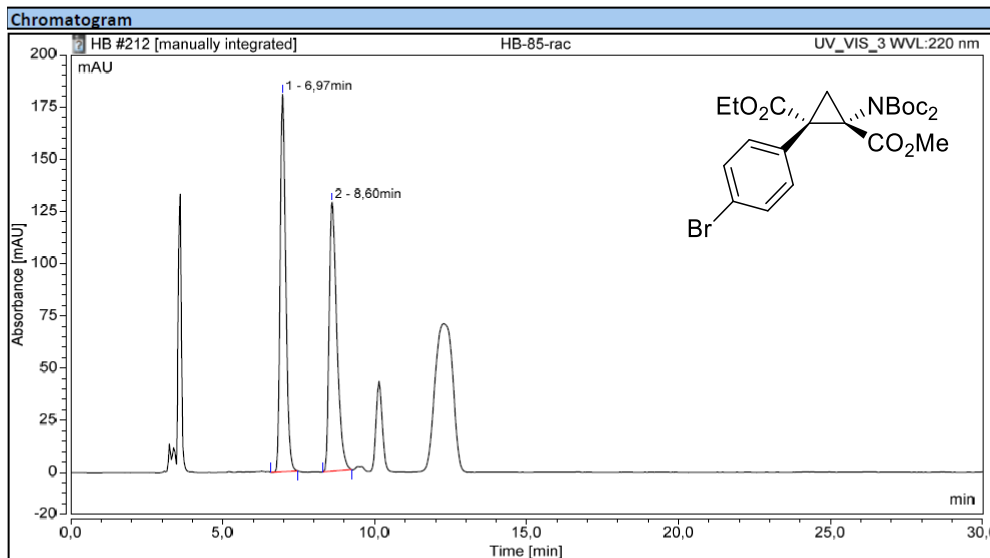


**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		9,58	199,668	631,596	97,06	97,77
2		10,44	6,054	14,420	2,94	2,23
<b>Total:</b>			<b>205,723</b>	<b>646,016</b>	<b>100,00</b>	<b>100,00</b>

**Compound 8 : 1-ethyl 2-methyl (1S,2S)-1-(4-bromophenyl)-2-(bis(tert-butoxycarbonyl)amino)cyclopropane-1,2-dicarboxylate**

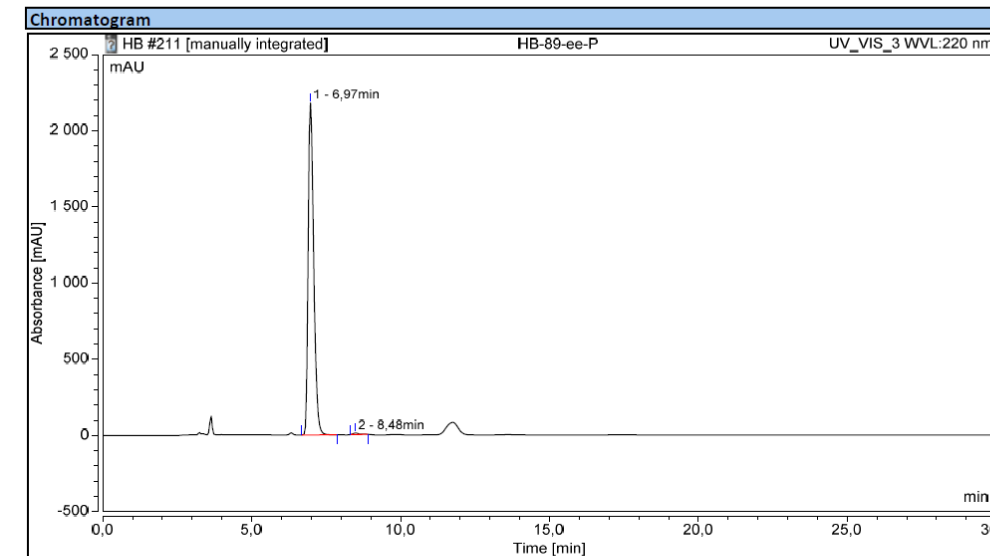
Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	GD2	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IE_Hept:iPrOH 90/10 20°C	Wavelength: 220
Instrument Method:	90A-10C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	10/mars/22 21:06	Sample Weight: 1,0000



**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		6,97	38,432	180,564	50,33	58,37
2		8,60	37,928	128,795	49,67	41,63
<b>Total:</b>			<b>76,360</b>	<b>309,359</b>	<b>100,00</b>	<b>100,00</b>

Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	GD1	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IE_Hept:iPrOH 90/10 20°C	Wavelength: 220
Instrument Method:	90A-10C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	10/mars/22 20:34	Sample Weight: 1,0000

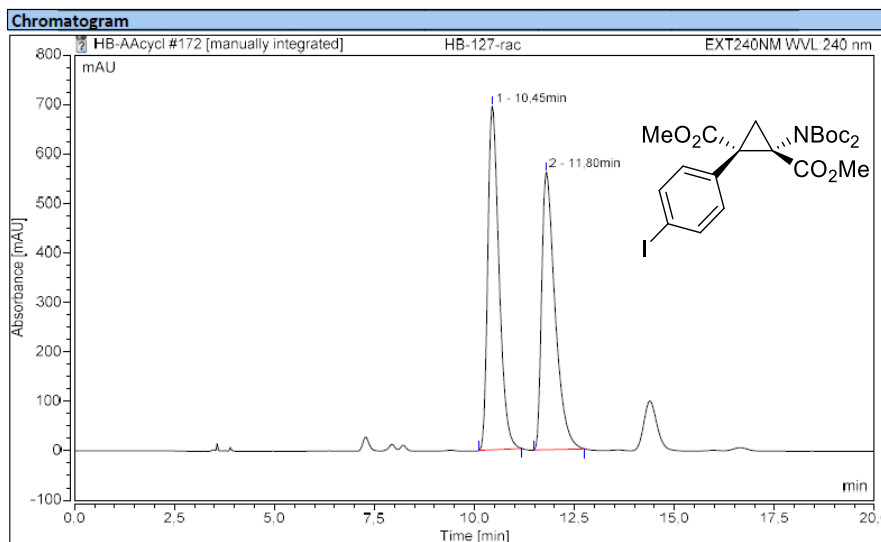


**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		6,97	472,997	2179,787	99,50	99,55
2		8,48	2,393	9,899	0,50	0,45
<b>Total:</b>			<b>475,390</b>	<b>2189,686</b>	<b>100,00</b>	<b>100,00</b>

**Compound 9: dimethyl (1S,2S)-1-(bis(tert-butoxycarbonyl)amino)-2-(4-iodophenyl)cyclopropane-1,2-dicarboxylate**

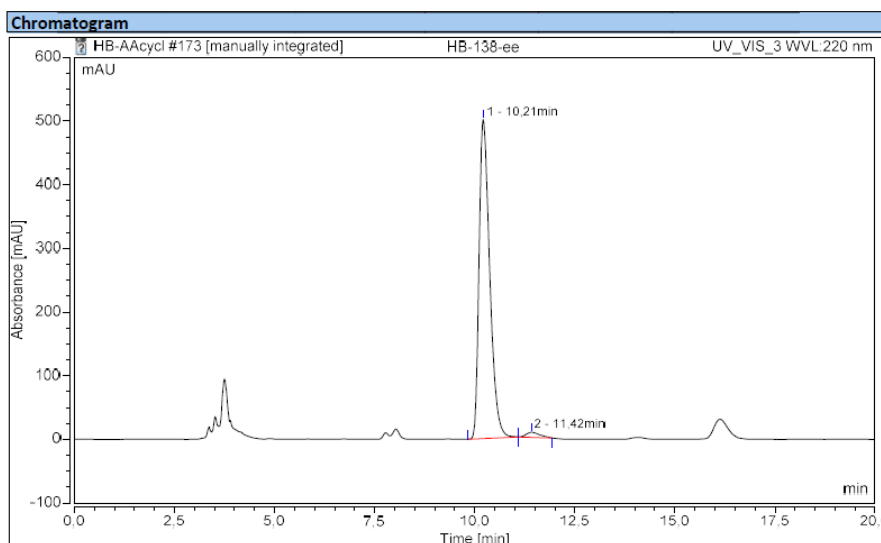
Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 20,00
Vial Number:	RB5	Injection Volume: 10,00
Injection Type:	Unknown	Channel: EXT240NM
Comment:	IE_Hept:IPrOH 95/5 20°C	Wavelength: n.a.
Instrument Method:	95A-5C	Bandwidth: n.a.
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	28/fevr.122 15:42	Sample Weight: 1,0000



**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Resolution (EP)
1		10.45	226,586	694,690	50,88	55,30	2,44
2		11.80	218,747	561,586	49,12	44,70	n.a.
<b>Total:</b>			<b>445,333</b>	<b>1256,276</b>	<b>100,00</b>	<b>100,00</b>	

Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 20,00
Vial Number:	RC8	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IE_Hept:IPrOH 95/5 20°C	Wavelength: 220
Instrument Method:	95A-5C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	28/fevr.122 16:03	Sample Weight: 1,0000

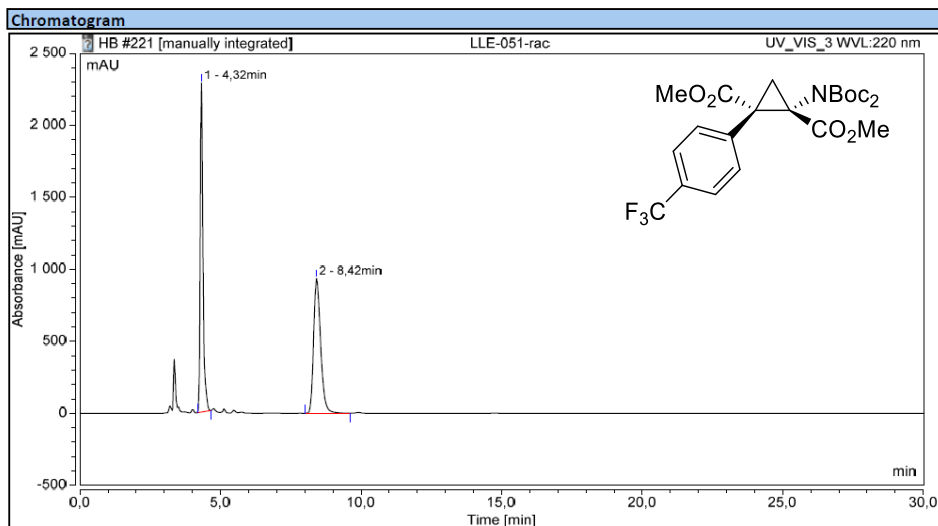


**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Resolution (EP)
1		10.21	153,845	501,117	98,01	98,41	2,22
2		11.42	3,130	8,099	1,99	1,59	n.a.
<b>Total:</b>			<b>156,975</b>	<b>509,216</b>	<b>100,00</b>	<b>100,00</b>	

**Compound 10**: dimethyl (1S,2S)-1-(bis(tert-butoxycarbonyl)amino)-2-(4-(trifluoromethyl)phenyl)cyclopropane-1,2-dicarboxylate

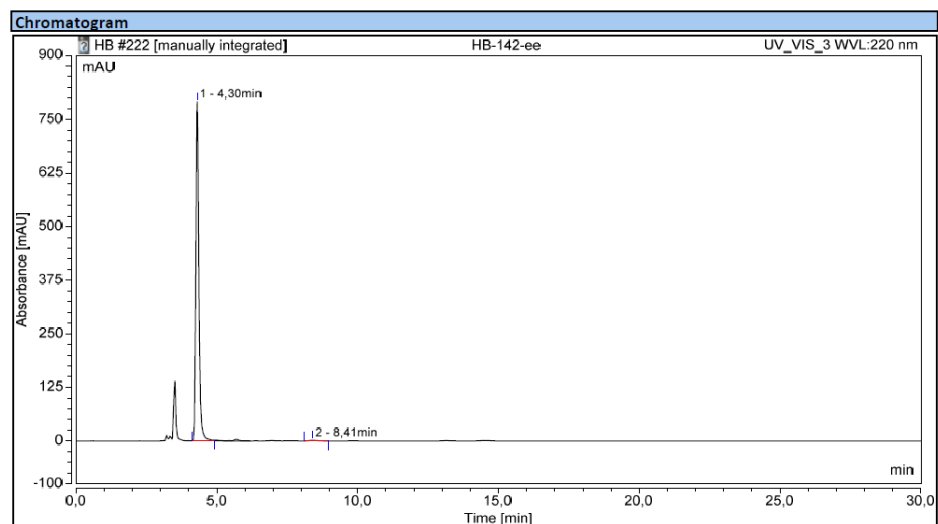
Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	RC4	Injection Volume: 15,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IC_Hept:iPrOH 90/10 20°C	Wavelength: 220
Instrument Method:	90A-10C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	24/mars/22 11:57	Sample Weight: 1,0000



**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		4,32	263,844	2285,850	49,26	70,94
2		8,42	271,733	936,449	50,74	29,06
Total:			535,576	3222,300	100,00	100,00

Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	RC5	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IC_Hept:iPrOH 90/10 20°C	Wavelength: 220
Instrument Method:	90A-10C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	24/mars/22 12:28	Sample Weight: 1,0000

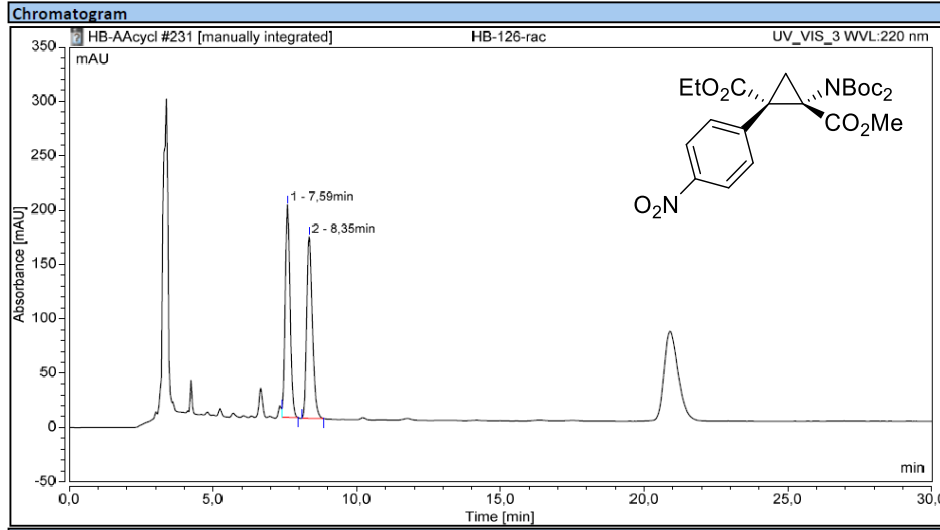


**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		4,30	97,571	791,290	99,46	99,78
2		8,41	0,526	1,773	0,54	0,22
Total:			98,097	793,063	100,00	100,00

**Compound 11 : 2-ethyl 1-methyl (1S,2S)-1-(bis(tert-butoxycarbonyl)amino)-2-(4-nitrophenyl)cyclopropane-1,2-dicarboxylate**

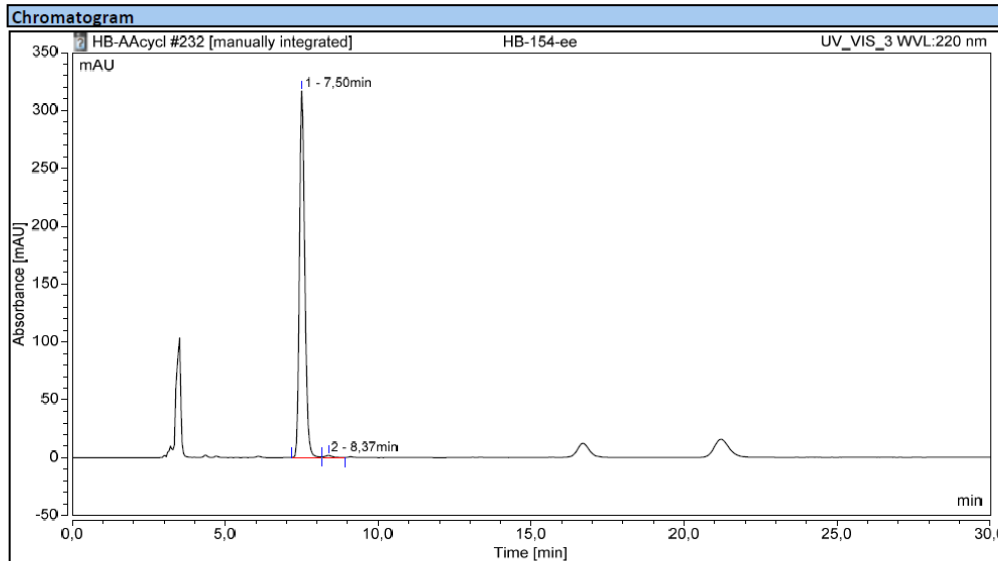
Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	RA4	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IE_Hept:iPrOH 80/20 20°C	Wavelength: 220
Instrument Method:	80A-20C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	29/mars/22 10:12	Sample Weight: 1,0000



**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		7.59	38,176	195,761	50,03	53,95
2		8.35	38,135	167,120	49,97	46,05
<b>Total:</b>			<b>76,312</b>	<b>362,881</b>	<b>100,00</b>	<b>100,00</b>

Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	RA5	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IE_Hept:iPrOH 80/20 20°C	Wavelength: 220
Instrument Method:	80A-20C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	29/mars/22 10:43	Sample Weight: 1,0000

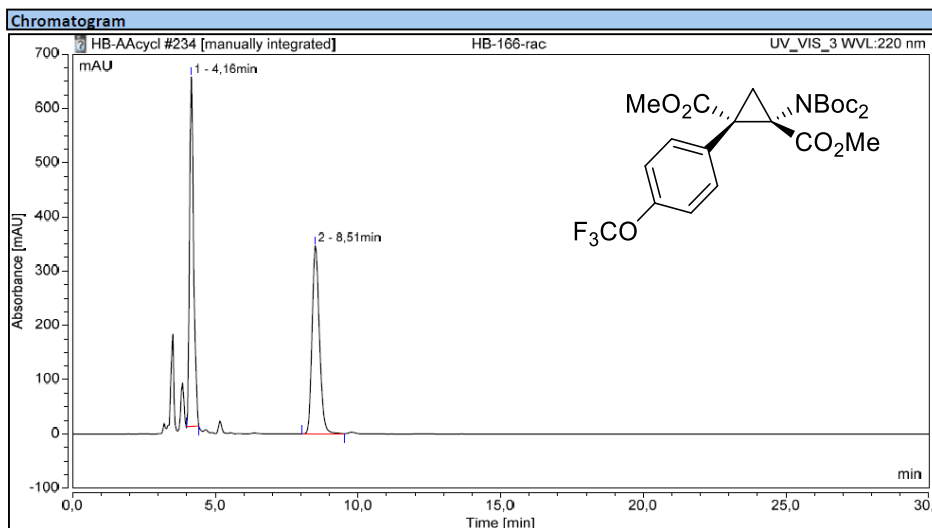


**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		7,50	64,165	316,760	99,32	99,46
2		8,37	0,442	1,736	0,68	0,54
<b>Total:</b>			<b>64,607</b>	<b>318,495</b>	<b>100,00</b>	<b>100,00</b>

**Compound 12: dimethyl (1S,2S)-1-(bis(tert-butoxycarbonyl)amino)-2-(4-(trifluoromethoxy)phenyl)cyclopropane-1,2-dicarboxylate**

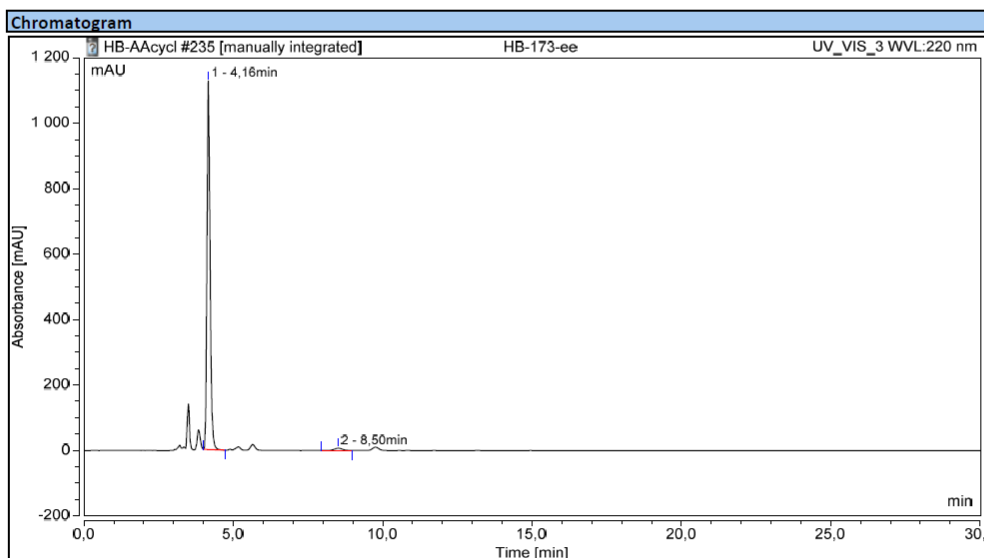
Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	RA6	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IC_Hept:iPrOH 90/10 20°C	Wavelength: 220
Instrument Method:	90A-10C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	29/mars/22 11:59	Sample Weight: 1,0000



**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		4,16	107,189	644,293	50,85	64,99
2		8,51	103,624	347,091	49,15	35,01
<b>Total:</b>			<b>210,813</b>	<b>991,384</b>	<b>100,00</b>	<b>100,00</b>

Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	RA7	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IC_Hept:iPrOH 90/10 20°C	Wavelength: 220
Instrument Method:	90A-10C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	29/mars/22 12:30	Sample Weight: 1,0000

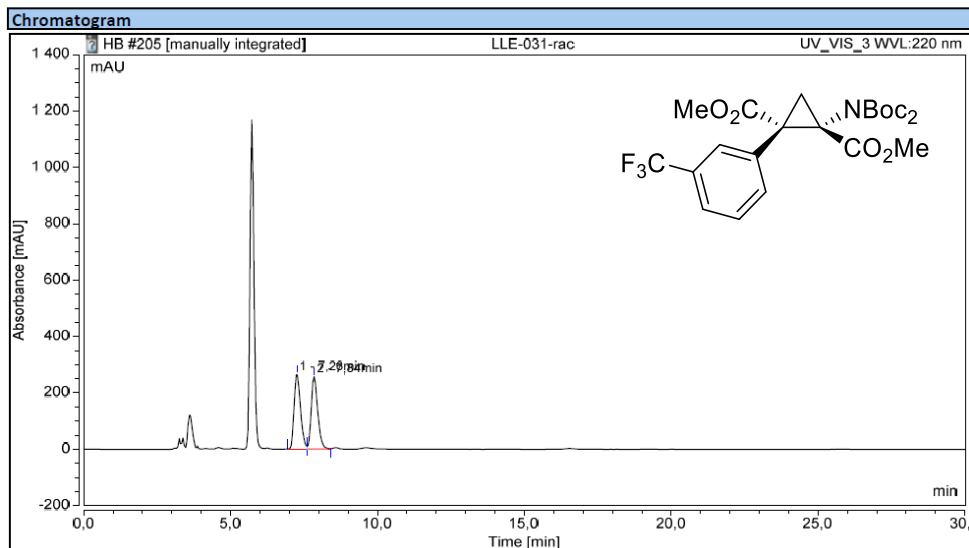


**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		4,16	136,765	1126,583	98,51	99,41
2		8,50	2,063	6,728	1,49	0,59
<b>Total:</b>			<b>138,828</b>	<b>1133,311</b>	<b>100,00</b>	<b>100,00</b>

**Compound 13**: dimethyl (1S,2S)-1-(bis(tert-butoxycarbonyl)amino)-2-(3-(trifluoromethyl)phenyl)cyclopropane-1,2-dicarboxylate

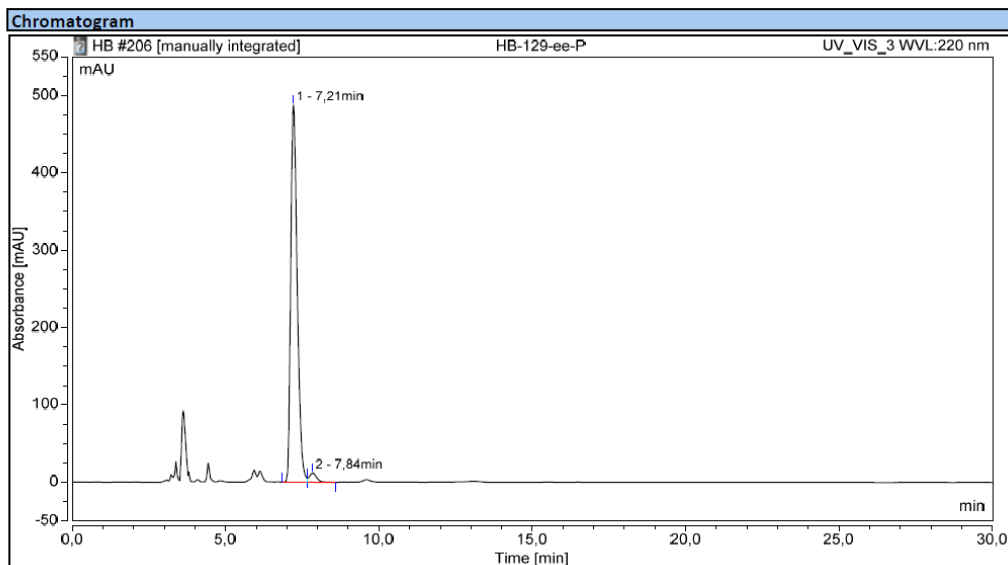
Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	GC2	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IE_Hept:iPrOH 95/5 20°C	Wavelength: 220
Instrument Method:	95A-5C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	10/mars/22 17:26	Sample Weight: 1,0000



**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		7,26	66,436	264,437	50,21	50,69
2		7,84	65,872	257,239	49,79	49,31
<b>Total:</b>			<b>132,308</b>	<b>521,676</b>	<b>100,00</b>	<b>100,00</b>

Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	GC3	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IE_Hept:iPrOH 95/5 20°C	Wavelength: 220
Instrument Method:	95A-5C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	10/mars/22 17:57	Sample Weight: 1,0000

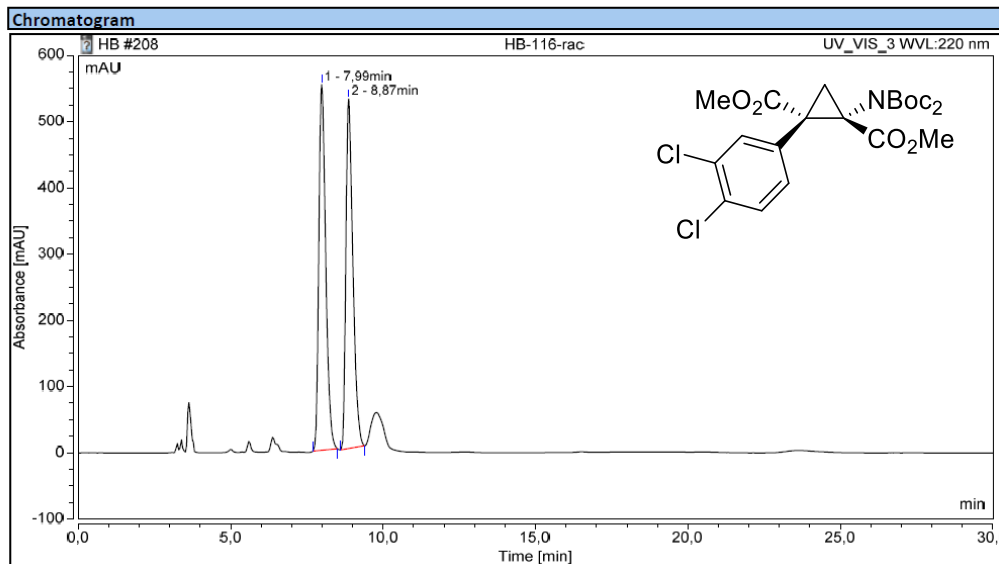


**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		7,21	119,870	487,972	97,30	97,56
2		7,84	3,320	12,196	2,70	2,44
<b>Total:</b>			<b>123,190</b>	<b>500,168</b>	<b>100,00</b>	<b>100,00</b>

**Compound 14**: dimethyl (1S,2S)-1-(bis(tert-butoxycarbonyl)amino)-2-(3,4-dichlorophenyl)cyclopropane-1,2-dicarboxylate

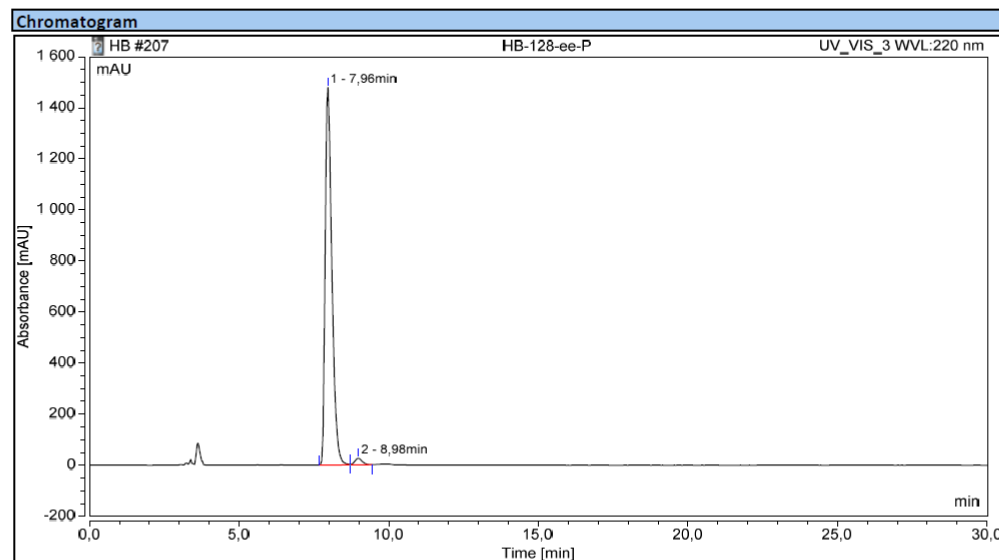
Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	GC5	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IE_Hept:iPrOH 95/5 20°C	Wavelength: 220
Instrument Method:	95A-5C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	10/mars/22 19:00	Sample Weight: 1,0000



**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		7,99	143,794	552,107	50,61	51,13
2		8,87	140,300	527,601	49,39	48,87
<b>Total:</b>			<b>284,094</b>	<b>1079,708</b>	<b>100,00</b>	<b>100,00</b>

Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	GC4	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IE_Hept:iPrOH 95/5 20°C	Wavelength: 220
Instrument Method:	95A-5C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	10/mars/22 18:29	Sample Weight: 1,0000



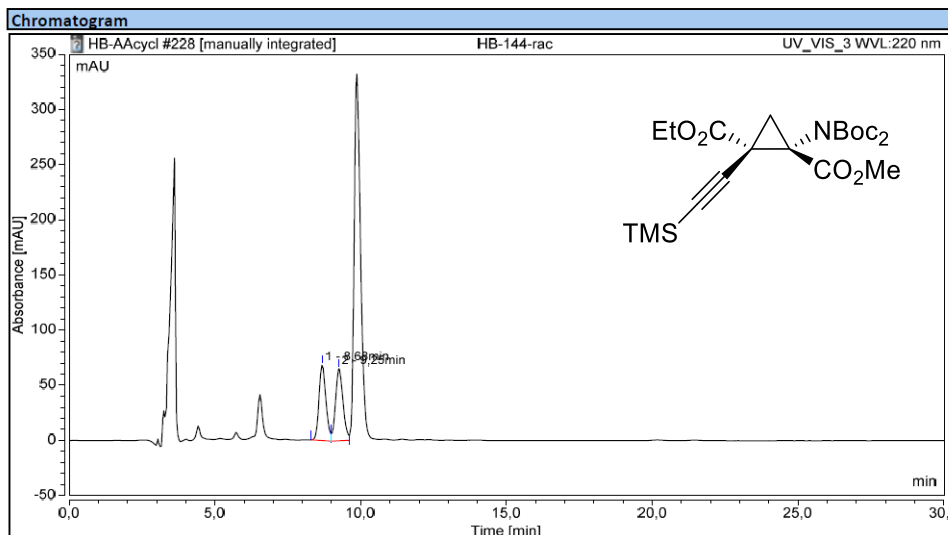
**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		7,96	385,356	1478,988	98,10	98,26
2		8,98	7,477	26,140	1,90	1,74
<b>Total:</b>			<b>392,833</b>	<b>1505,128</b>	<b>100,00</b>	<b>100,00</b>



**Compound 15: 2-ethyl 1-methyl (1S,2S)-1-(bis(tert-butoxycarbonyl)amino)-2-((trimethylsilyl)ethynyl)cyclopropane-1,2-dicarboxylate**

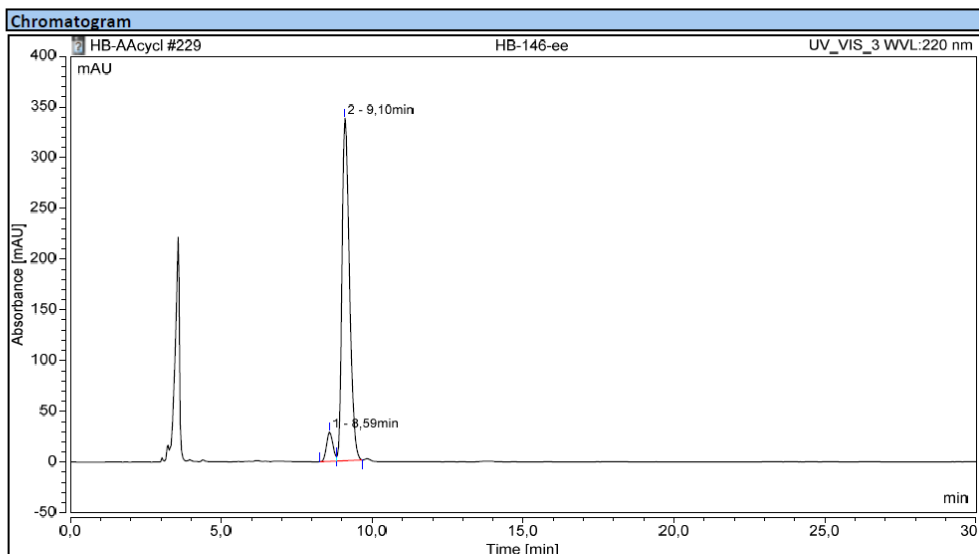
Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	RA1	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IE_Hept:iPrOH 90/10 20°C	Wavelength: 220
Instrument Method:	90A-10C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	29/mars/22 08:38	Sample Weight: 1,0000



**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		8,68	18,954	68,663	49,61	51,24
2		9,25	19,253	65,351	50,39	48,76
<b>Total:</b>			<b>38,207</b>	<b>134,014</b>	<b>100,00</b>	<b>100,00</b>

Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	RA2	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IE_Hept:iPrOH 90/10 20°C	Wavelength: 220
Instrument Method:	90A-10C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	29/mars/22 09:09	Sample Weight: 1,0000

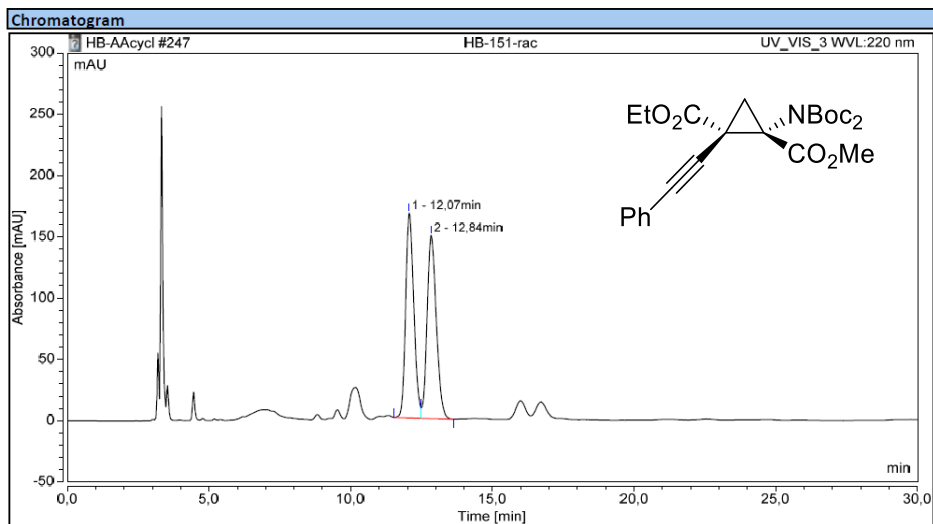


**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		8,59	7,096	29,026	6,83	7,92
2		9,10	96,734	337,587	93,17	92,08
<b>Total:</b>			<b>103,829</b>	<b>366,614</b>	<b>100,00</b>	<b>100,00</b>

**Compound 16** : 2-ethyl 1-methyl (1S,2S)-1-(bis(tert-butoxycarbonyl)amino)-2-(phenylethynyl)cyclopropane-1,2-dicarboxylate

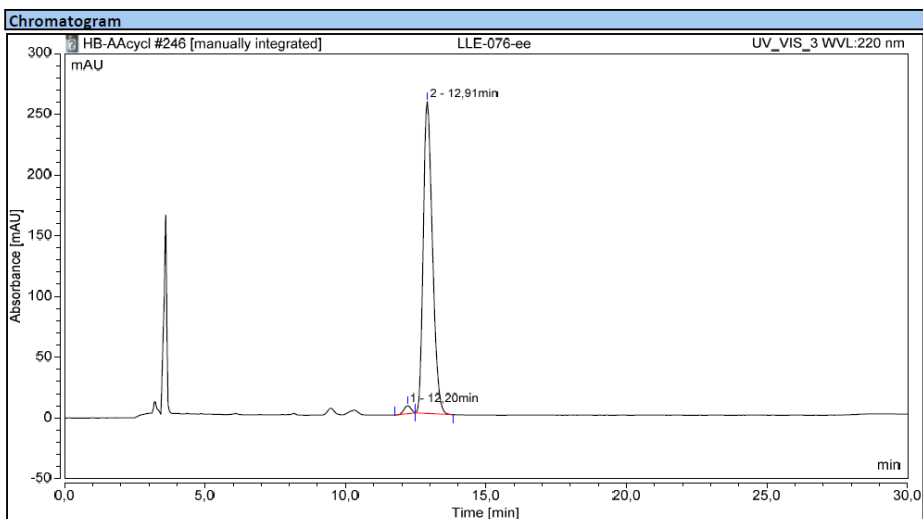
Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	GB2	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IE_Hept:iPrOH 90/10 20°C	Wavelength: 220
Instrument Method:	90A-10C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	04/avr./22 13:53	Sample Weight: 1,0000



**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		12,07	56,890	167,253	49,76	52,79
2		12,84	56,432	149,545	50,24	47,21
<b>Total:</b>			<b>112,322</b>	<b>316,797</b>	<b>100,00</b>	<b>100,00</b>

Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	GA5	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IE_Hept:iPrOH 90/10 20°C	Wavelength: 220
Instrument Method:	90A-10C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	04/avr./22 13:22	Sample Weight: 1,0000

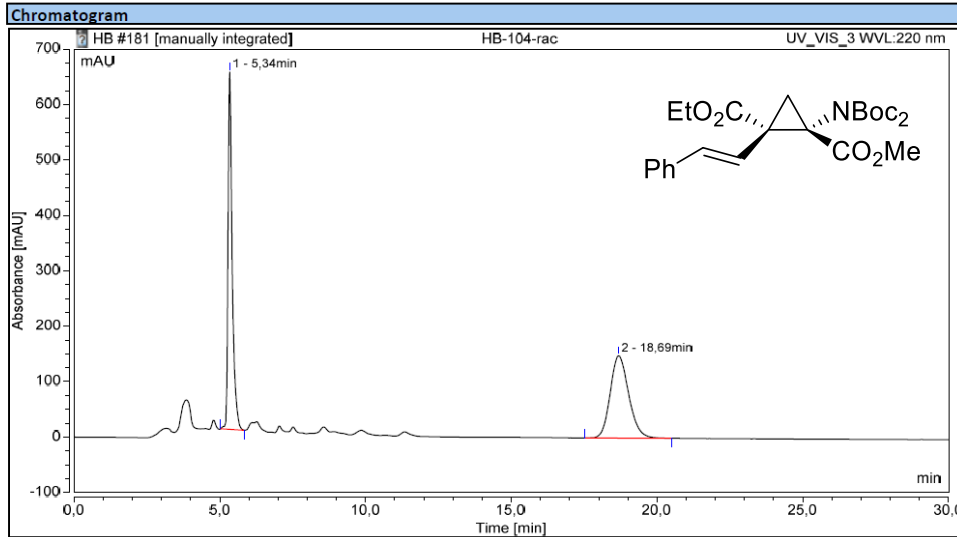


**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		12,20	1,898	6,761	1,91	2,57
2		12,91	97,370	256,824	98,09	97,43
<b>Total:</b>			<b>99,268</b>	<b>263,585</b>	<b>100,00</b>	<b>100,00</b>

**Compound 17 : 2-ethyl 1-methyl (1S,2S)-1-(bis(tert-butoxycarbonyl)amino)-2-((E)-styryl)cyclopropane-1,2-dicarboxylate**

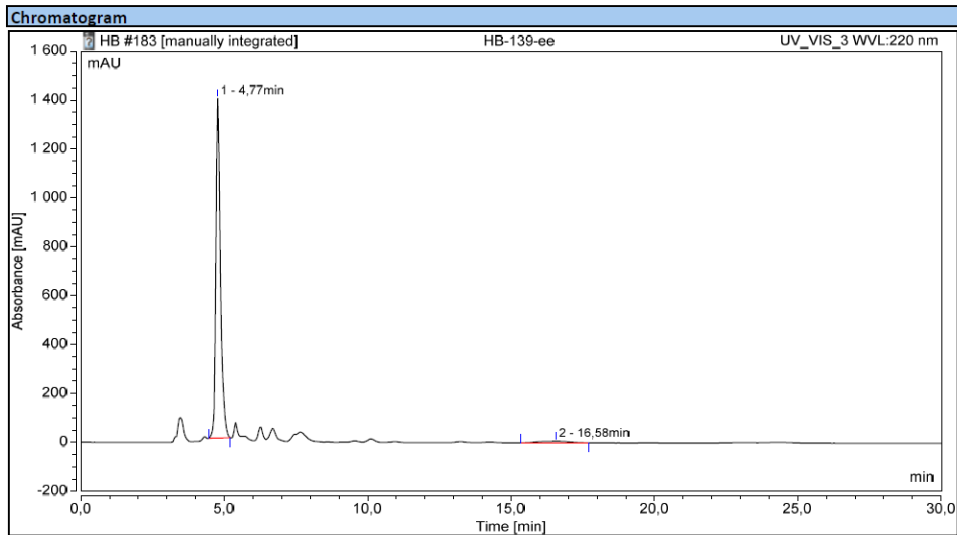
Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	RB5	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IC_Hept:iPrOH 70/30 20°C	Wavelength: 220
Instrument Method:	70A-30C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	03/mars/22 08:39	Sample Weight: 1,0000



**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		5,34	106,999	644,855	49,48	81,26
2		18,69	109,250	148,679	50,52	18,74
Total:			216 249	793 535	100 00	100 00

Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	RB6	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IC_Hept:iPrOH 70/30 20°C	Wavelength: 220
Instrument Method:	70A-30C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	03/mars/22 09:10	Sample Weight: 1,0000

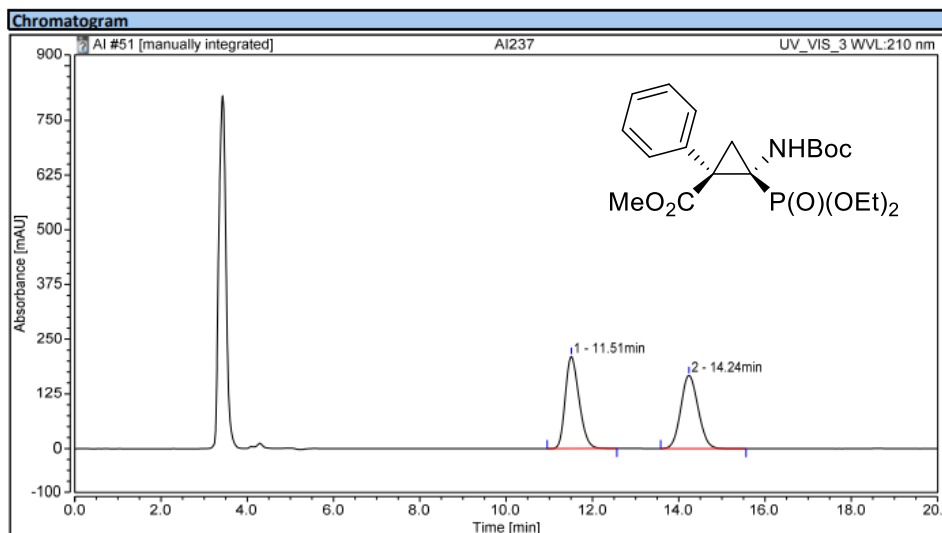


**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		4,77	253,923	1390,154	96,97	99,49
2		16,58	7,935	7,061	3,03	0,51
Total:			261,858	1397,216	100,00	100,00

**Compound 18**: methyl (1*S*,2*R*)-2-((tert-butoxycarbonyl)amino)-2-(diethoxyphosphoryl)-1-phenyl cyclopropane-1-carboxylate

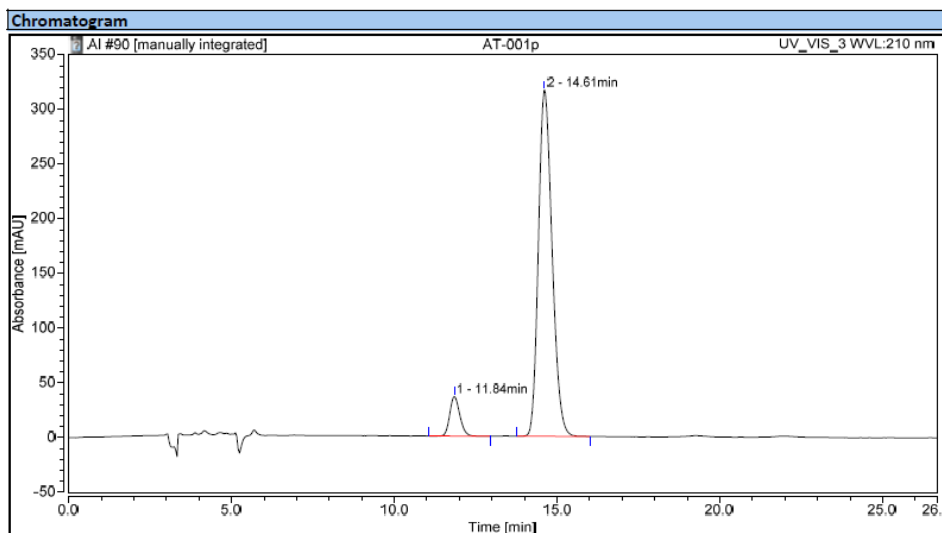
Injection Details		
Injection Name:	AI237	Run Time (min): 20.00
Vial Number:	BC3	Injection Volume: 15.00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IE hept/oprOH	Wavelength: 210
Instrument Method:	80A-20C	Bandwidth: 2
Processing Method:	methode traitement2	Dilution Factor: 1.0000
Injection Date/Time:	22/sept/23 16:57	Sample Weight: 1.0000



**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		11.51	78.271	210.862	50.01	55.74
2		14.24	78.247	167.453	49.99	44.26
<b>Total:</b>			<b>156.518</b>	<b>378.315</b>	<b>100.00</b>	<b>100.00</b>

Injection Details		
Injection Name:	AT-001p	Run Time (min): 26.67
Vial Number:	GE1	Injection Volume: 10.00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IE hept/oprOH	Wavelength: 210
Instrument Method:	80A-20C	Bandwidth: 2
Processing Method:	methode traitement2	Dilution Factor: 1.0000
Injection Date/Time:	23/nov./23 15:55	Sample Weight: 1.0000

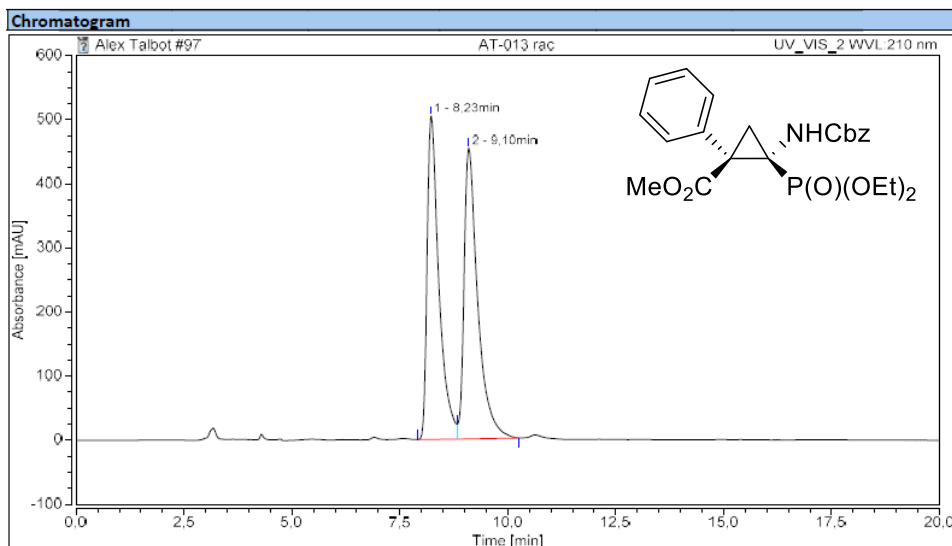


**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		11.84	12.892	36.166	7.74	10.25
2		14.61	153.586	316.615	92.26	89.75
<b>Total:</b>			<b>166.479</b>	<b>352.781</b>	<b>100.00</b>	<b>100.00</b>

**Compound 19** : methyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-phenylcyclopropane-1-carboxylate

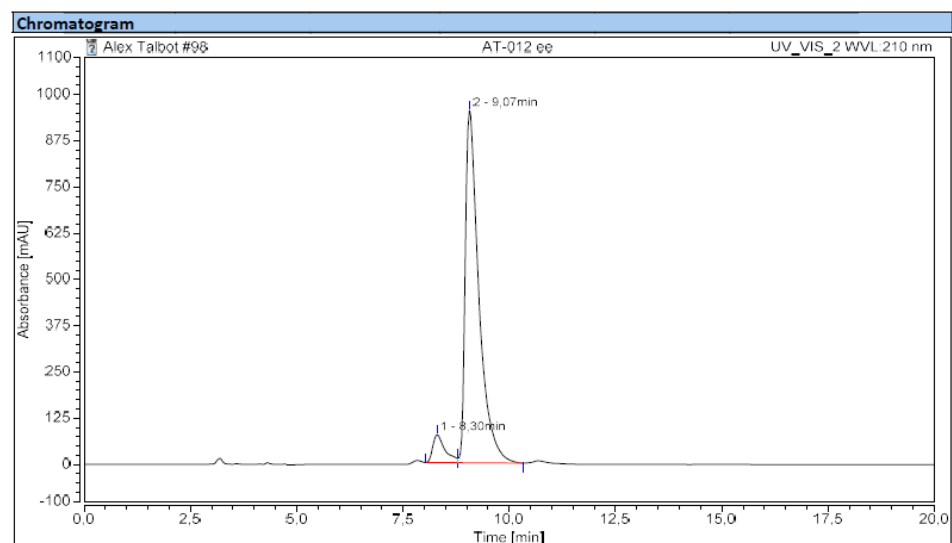
Injection Details		
Injection Name:	AT-013 rac	Run Time (min): 20,00
Vial Number:	BA3	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_2
Comment:	IB 250*4.6 v5µm A: heptane D:heptane:iprOH 9:1 20°C 1ml/min	Wavelength: 210
Instrument Method:	90A-10C20min	Bandwidth: 2
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	20/juin/24 16:01	Sample Weight: 1,0000



**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		8,23	159,380	504,526	48,89	52,65
2		9,10	166,629	453,667	51,11	47,35
<b>Total:</b>			<b>326,009</b>	<b>958,193</b>	<b>100,00</b>	<b>100,00</b>

Injection Details		
Injection Name:	AT-012 ee	Run Time (min): 20,00
Vial Number:	BA4	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_2
Comment:	IB 250*4.6 v5µm A: heptane D:heptane:iprOH 9:1 20°C 1ml/min	Wavelength: 210
Instrument Method:	90A-10C20min	Bandwidth: 2
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	20/juin/24 16:21	Sample Weight: 1,0000

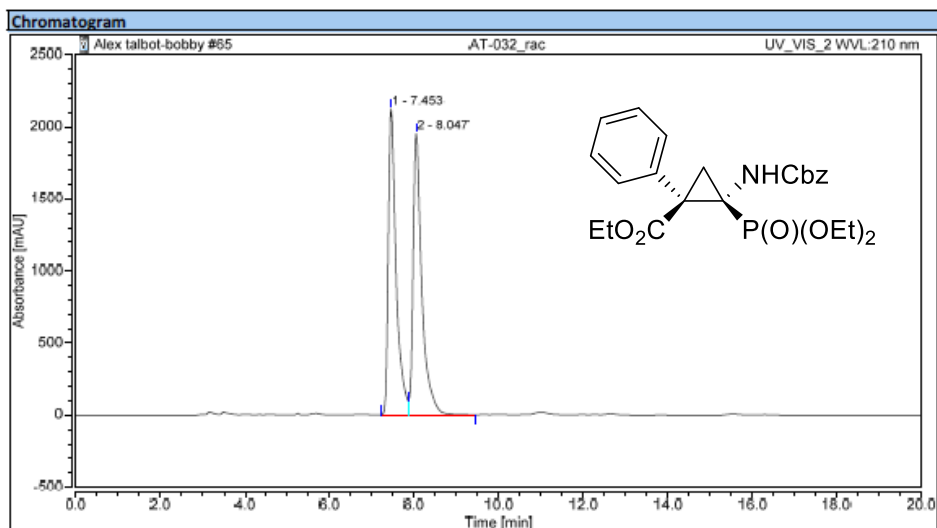


**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		8,30	25,364	75,370	6,72	7,33
2		9,07	352,197	952,717	93,28	92,67
<b>Total:</b>			<b>377,561</b>	<b>1028,086</b>	<b>100,00</b>	<b>100,00</b>

**Compound 20**: ethyl (1S,2R)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-phenyl cyclopropane-1-carboxylate

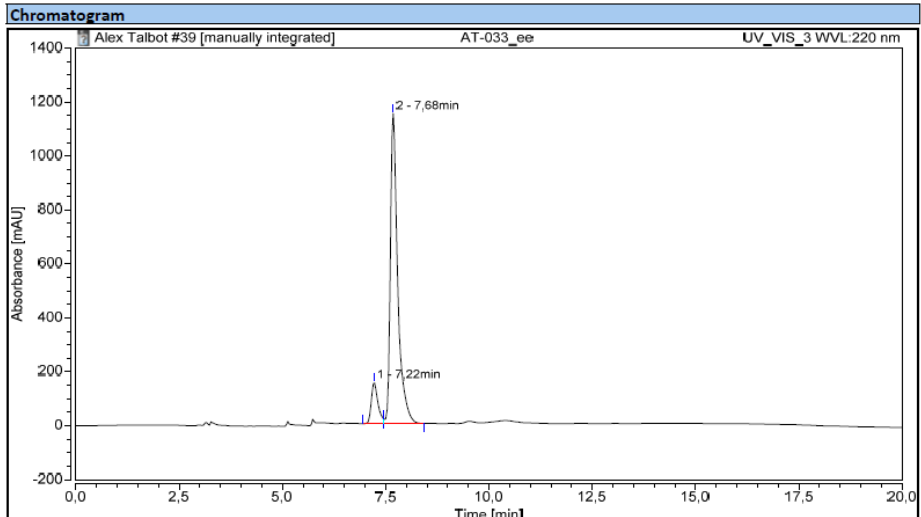
Injection Details		
Injection Name:	AT-032_rac	Run Time (min): 20.00
Vial Number:	BE1	Injection Volume: 10.00
Injection Type:	Unknown	Channel: UV_VIS_2
Comment:	IB 250*4.6 v5µm A: heptane C:EtOH 20°C 1ml/min	Wavelength: 210
Instrument Method:	95A-5B 20	Bandwidth: 2
Processing Method:	methode traitement	Dilution Factor: 1.0000
Injection Date/Time:	31/janv./24 10:51	Sample Weight: 1.0000



**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		7.453	455.288	2125.404	49.30	52.13	n.a.
2		8.047	468.230	1952.024	50.70	47.87	n.a.
<b>Total:</b>			<b>923.518</b>	<b>4077.428</b>	<b>100.00</b>	<b>100.00</b>	

Injection Details		
Injection Name:	AT-033_ee	Run Time (min): 20.00
Vial Number:	GC2	Injection Volume: 15.00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IB 250*4.6 v5µm A: heptane B:etOH 20°C 1ml/min	Wavelength: 220
Instrument Method:	95A-5B 25	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1.0000
Injection Date/Time:	05/janv./24 15:00	Sample Weight: 1.0000

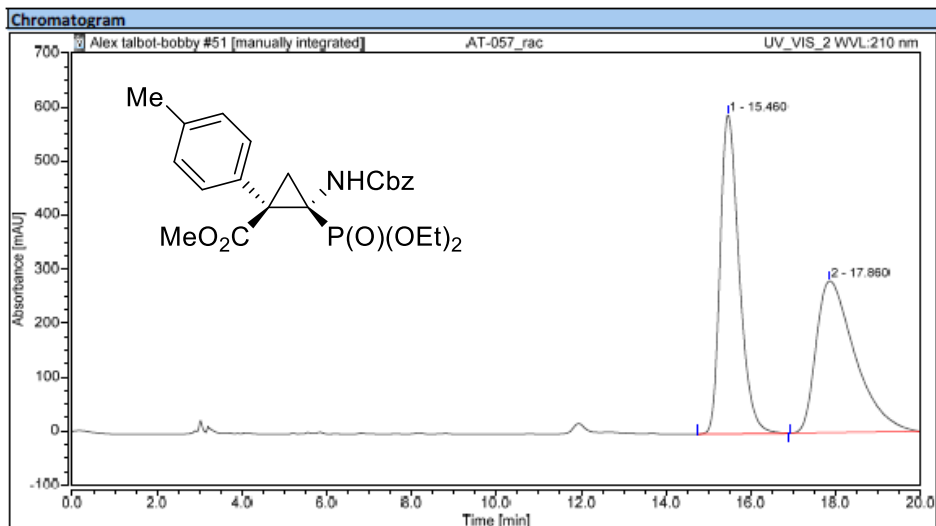


**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		7.22	28.186	152.959	10.62	11.74
2		7.68	237.236	1149.513	89.38	88.26
<b>Total:</b>			<b>265.422</b>	<b>1302.472</b>	<b>100.00</b>	<b>100.00</b>

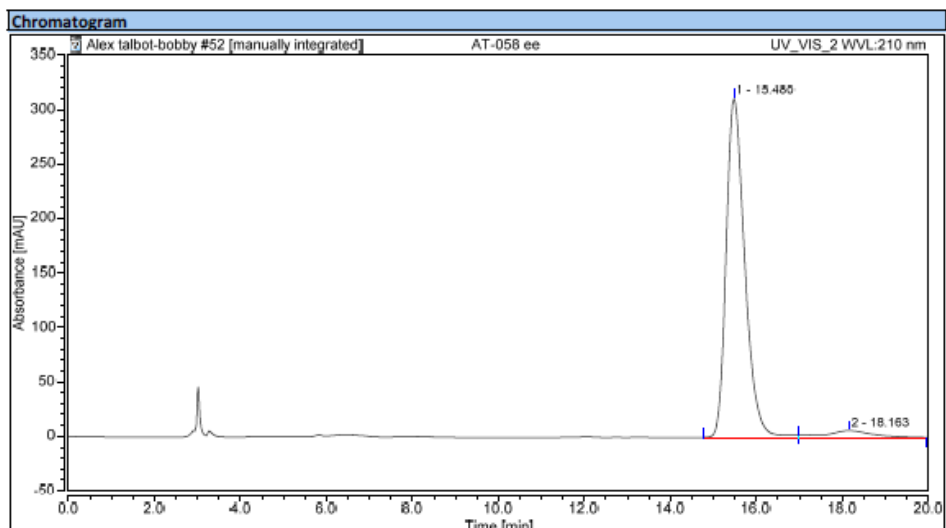
**Compound 21** : methyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-(*p*-tolyl) cyclopropane-1-carboxylate

Injection Details		
Injection Name:	AT-057_rac	Run Time (min): 20.00
Vial Number:	BE3	Injection Volume: 15.00
Injection Type:	Unknown	Channel: UV_VIS_2
Comment:	IA 250*4.6 v5µm A: heptane C:EtOH 20°C 1ml/min	Wavelength: 210
Instrument Method:	95A-5B 20	Bandwidth: 2
Processing Method:	methode traitement	Dilution Factor: 1.0000
Injection Date/Time:	30/janv./24 12:00	Sample Weight: 1.0000



No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		15.460	306.302	590.851	50.66	67.78	n.a.
2		17.860	298.281	280.906	49.34	32.22	n.a.
<b>Total:</b>			<b>604.583</b>	<b>871.757</b>	<b>100.00</b>	<b>100.00</b>	

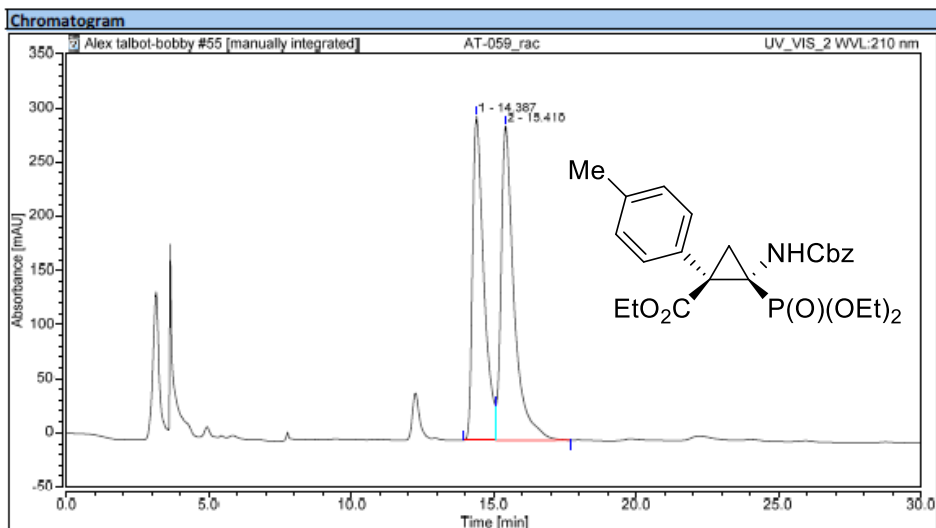
Injection Details		
Injection Name:	AT-058 ee	Run Time (min): 20.00
Vial Number:	BD3	Injection Volume: 15.00
Injection Type:	Unknown	Channel: UV_VIS_2
Comment:	IA 250*4.6 v5µm A: heptane C:EtOH 20°C 1ml/min	Wavelength: 210
Instrument Method:	95A-5B 20	Bandwidth: 2
Processing Method:	methode traitement	Dilution Factor: 1.0000
Injection Date/Time:	30/janv./24 12:22	Sample Weight: 1.0000



No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		15.480	155.796	311.227	95.21	98.02	n.a.
2		18.163	7.841	6.288	4.79	1.98	n.a.
<b>Total:</b>			<b>163.637</b>	<b>317.515</b>	<b>100.00</b>	<b>100.00</b>	

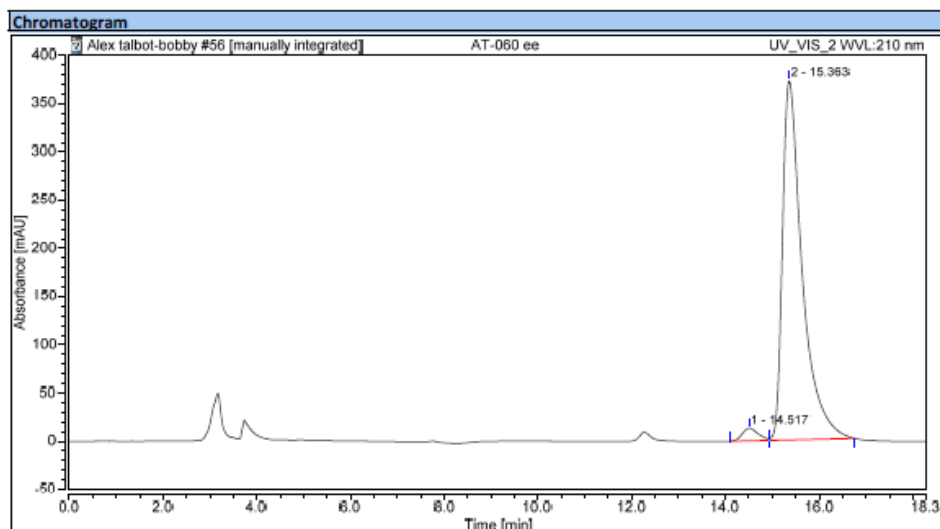
**Compound 22**: ethyl (1S,2R)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-(p-tolyl) cyclopropane-1-carboxylate

Injection Details		
Injection Name:	AT-059_rac	Run Time (min): 30.00
Vial Number:	BE4	Injection Volume: 15.00
Injection Type:	Unknown	Channel: UV_VIS_2
Comment:	IB 250*4.6 v5µm A: heptane C:EtOH 20°C 1ml/min	Wavelength: 210
Instrument Method:	98A-2B 20C-30min	Bandwidth: 2
Processing Method:	methode traitement	Dilution Factor: 1.0000
Injection Date/Time:	30/janv./24 14:42	Sample Weight: 1.0000



No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		14.387	137.039	299.227	47.09	50.77	n.a.
2		15.410	153.984	290.167	52.91	49.23	n.a.
<b>Total:</b>			<b>291.023</b>	<b>589.394</b>	<b>100.00</b>	<b>100.00</b>	

Injection Details		
Injection Name:	AT-060 ee	Run Time (min): 18.29
Vial Number:	BD4	Injection Volume: 15.00
Injection Type:	Unknown	Channel: UV_VIS_2
Comment:	IB 250*4.6 v5µm A: heptane C:EtOH 20°C 1ml/min	Wavelength: 210
Instrument Method:	98A-2B 20C-30min	Bandwidth: 2
Processing Method:	methode traitement	Dilution Factor: 1.0000
Injection Date/Time:	30/janv./24 15:14	Sample Weight: 1.0000

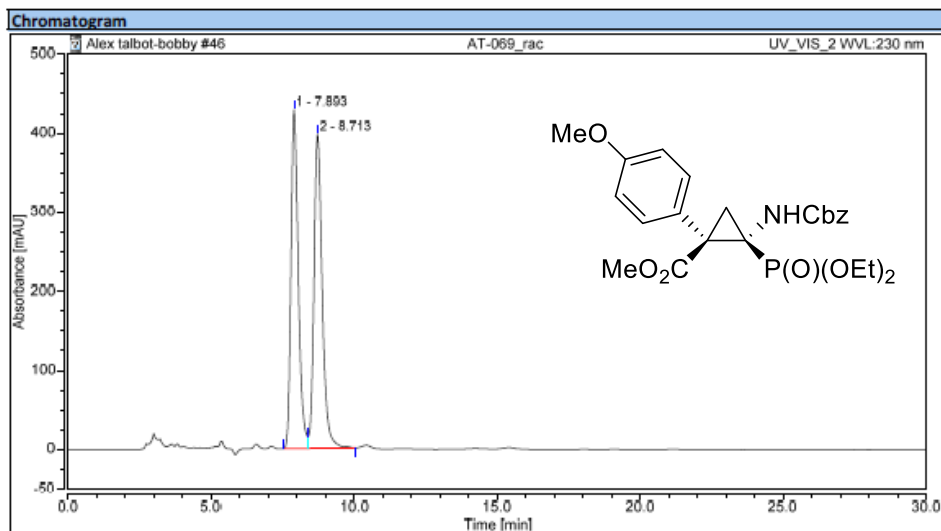


No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		14.517	5.154	13.122	2.76	3.40	n.a.
2		15.363	181.568	372.684	97.24	96.60	n.a.
<b>Total:</b>			<b>186.722</b>	<b>385.806</b>	<b>100.00</b>	<b>100.00</b>	



**Compound 23**: methyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-(4-methoxyphenyl)cyclopropane-1-carboxylate

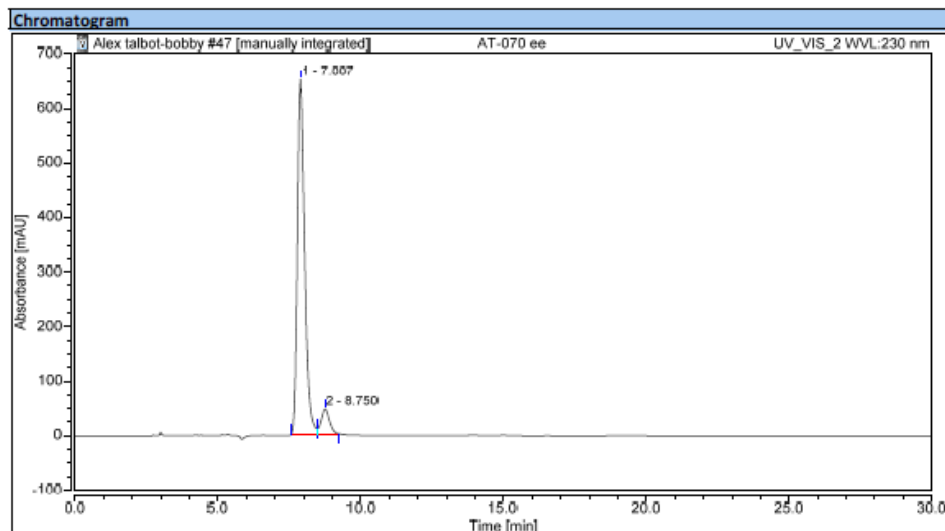
Injection Details		
Injection Name:	AT-069_rac	Run Time (min): 30.00
Vial Number:	BE8	Injection Volume: 15.00
Injection Type:	Unknown	Channel: UV_VIS_2
Comment:	IA 250*4.6 v5µm A: heptane C:iPrOH 20°C 1ml/min	Wavelength: 230
Instrument Method:	80A-20C	Bandwidth: 2
Processing Method:	methode traitement	Dilution Factor: 1.0000
Injection Date/Time:	30/janv./24 09:53	Sample Weight: 1.0000



**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		7.893	126.942	428.643	48.85	51.89	n.a.
2		8.713	132.899	397.480	51.15	48.11	n.a.
<b>Total:</b>			<b>259.841</b>	<b>826.123</b>	<b>100.00</b>	<b>100.00</b>	

Injection Details		
Injection Name:	AT-070 ee	Run Time (min): 30.00
Vial Number:	BD8	Injection Volume: 15.00
Injection Type:	Unknown	Channel: UV_VIS_2
Comment:	IA 250*4.6 v5µm A: heptane C:iPrOH 20°C 1ml/min	Wavelength: 230
Instrument Method:	80A-20C	Bandwidth: 2
Processing Method:	methode traitement	Dilution Factor: 1.0000
Injection Date/Time:	30/janv./24 10:25	Sample Weight: 1.0000

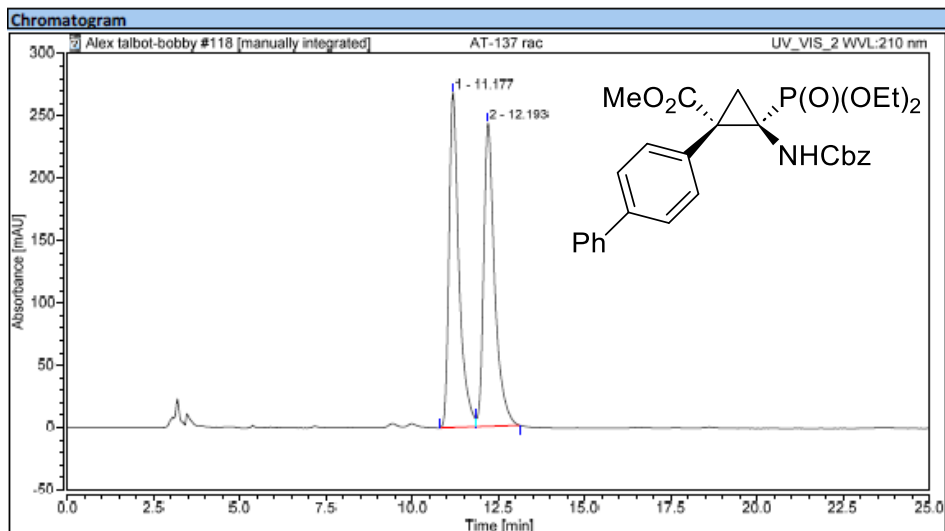


**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		7.887	191.352	649.658	92.71	93.45	n.a.
2		8.750	15.051	45.544	7.29	6.55	n.a.
<b>Total:</b>			<b>206.403</b>	<b>695.201</b>	<b>100.00</b>	<b>100.00</b>	

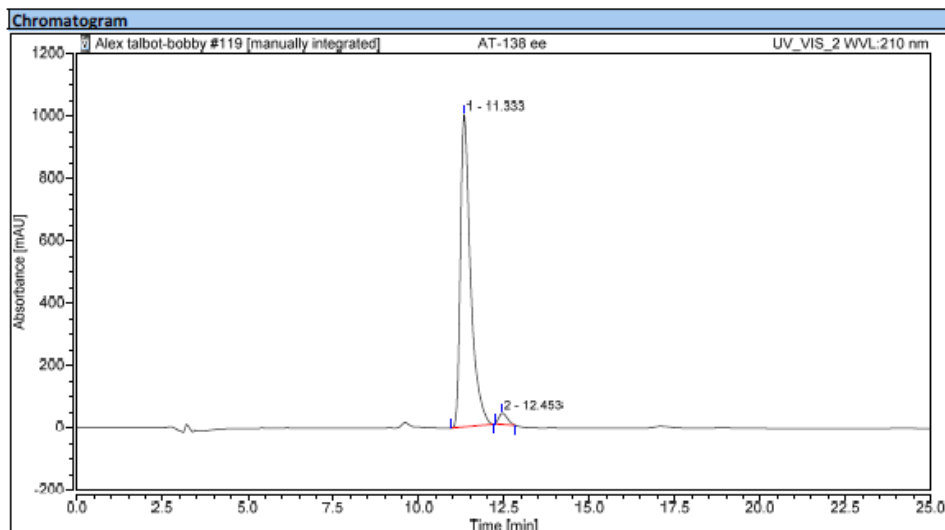
**Compound 24** : methyl (1R,2S)-1-([1,1'-biphenyl]-4-yl)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)cyclopropane-1-carboxylate

Injection Details		
Injection Name:	AT-137 rac	Run Time (min): 25.00
Vial Number:	BE7	Injection Volume: 15.00
Injection Type:	Unknown	Channel: UV_VIS_2
Comment:	IB 250*4.6 v5µm A: heptane B:EtOH 20°C 1ml/min	Wavelength: 210
Instrument Method:	95A-5B 20 25 min	Bandwidth: 2
Processing Method:	methode traitement	Dilution Factor: 1.0000
Injection Date/Time:	23/avr/24 15:37	Sample Weight: 1.0000



Integration Results							
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		11.177	89.623	268.671	49.91	52.48	n.a.
2		12.193	89.947	243.245	50.09	47.52	n.a.
<b>Total:</b>			<b>179.570</b>	<b>511.916</b>	<b>100.00</b>	<b>100.00</b>	

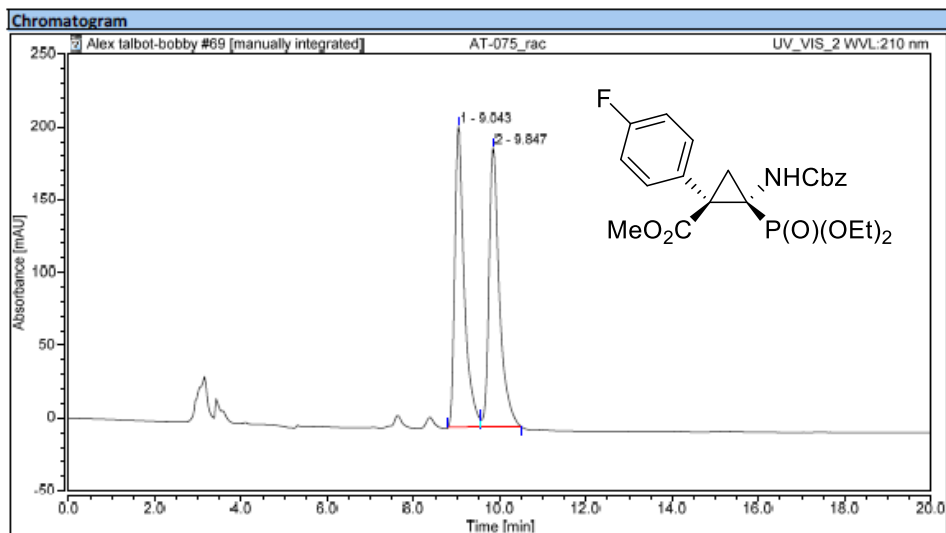
Injection Details		
Injection Name:	AT-138 ee	Run Time (min): 25.00
Vial Number:	BE8	Injection Volume: 15.00
Injection Type:	Unknown	Channel: UV_VIS_2
Comment:	IB 250*4.6 v5µm A: heptane B:EtOH 20°C 1ml/min	Wavelength: 210
Instrument Method:	95A-5B 20 25 min	Bandwidth: 2
Processing Method:	methode traitement	Dilution Factor: 1.0000
Injection Date/Time:	23/avr/24 16:13	Sample Weight: 1.0000



Integration Results							
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		11.333	345.657	1003.433	97.00	96.50	n.a.
2		12.453	10.705	36.340	3.00	3.50	n.a.
<b>Total:</b>			<b>356.362</b>	<b>1039.773</b>	<b>100.00</b>	<b>100.00</b>	

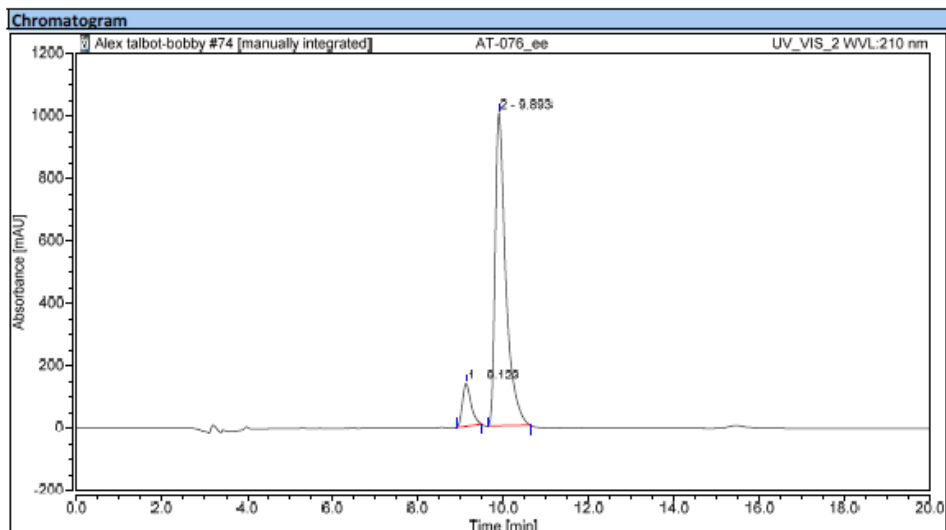
**Compound 25**: methyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-(4-fluoro phenyl)cyclopropane-1-carboxylate

Injection Details		
Injection Name:	AT-075_rac	Run Time (min): 20.00
Vial Number:	BE1	Injection Volume: 15.00
Injection Type:	Unknown	Channel: UV_VIS_2
Comment:	IB 250*4.6 v5µm A: heptane B: EtOH 20°C 1ml/min	Wavelength: 210
Instrument Method:	95A-5B 20	Bandwidth: 2
Processing Method:	methode traitement	Dilution Factor: 1.0000
Injection Date/Time:	02/fevr./24 08:46	Sample Weight: 1.0000



Integration Results							
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		9.043	52.494	206.753	49.55	51.88	n.a.
2		9.847	53.456	191.749	50.45	48.12	n.a.
<b>Total:</b>			<b>105.950</b>	<b>398.502</b>	<b>100.00</b>	<b>100.00</b>	

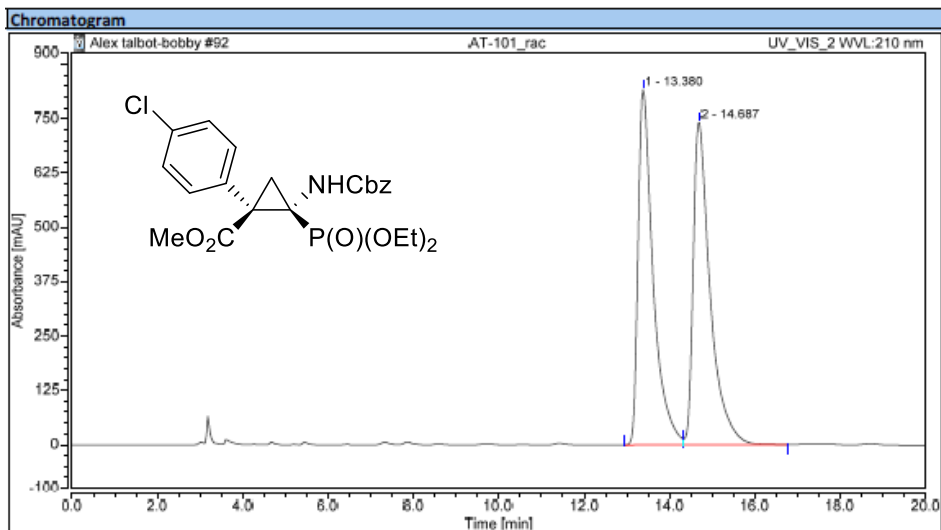
Injection Details		
Injection Name:	AT-076_ee	Run Time (min): 20.00
Vial Number:	BE6	Injection Volume: 15.00
Injection Type:	Unknown	Channel: UV_VIS_2
Comment:	IB 250*4.6 v5µm A: heptane B: EtOH 20°C 1ml/min	Wavelength: 210
Instrument Method:	95A-5B 20	Bandwidth: 2
Processing Method:	methode traitement	Dilution Factor: 1.0000
Injection Date/Time:	02/fevr./24 10:41	Sample Weight: 1.0000



Integration Results							
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		9.123	31.929	137.831	9.95	12.06	n.a.
2		9.893	288.841	1004.842	90.05	87.94	n.a.
<b>Total:</b>			<b>320.770</b>	<b>1142.674</b>	<b>100.00</b>	<b>100.00</b>	

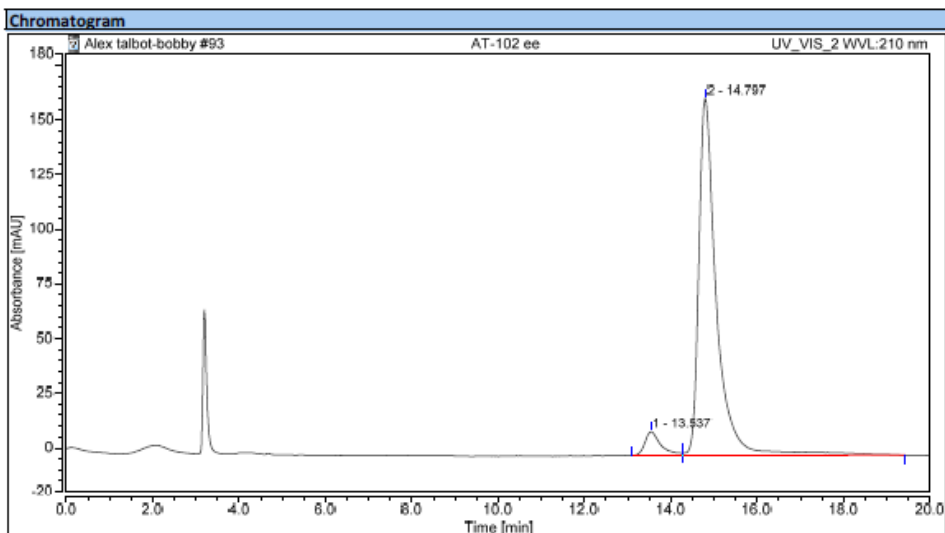
**Compound 26**: methyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-1-(4-chlorophenyl)-2-(diethoxy phosphoryl)cyclopropane-1-carboxylate

Injection Details		
Injection Name:	AT-101_rac	Run Time (min): 20.00
Vial Number:	BE1	Injection Volume: 15.00
Injection Type:	Unknown	Channel: UV_VIS_2
Comment:	IB 250*4.6 v5µm A: heptane B:EtOH 20°C 1ml/min	Wavelength: 210
Instrument Method:	97A-3B 20	Bandwidth: 2
Processing Method:	methode traitement	Dilution Factor: 1.0000
Injection Date/Time:	27/fevr./24 09:47	Sample Weight: 1.0000



Integration Results							
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		13.380	334.152	817.276	49.45	52.42	n.a.
2		14.687	341.520	741.926	50.55	47.58	n.a.
<b>Total:</b>			<b>675.672</b>	<b>1559.202</b>	<b>100.00</b>	<b>100.00</b>	

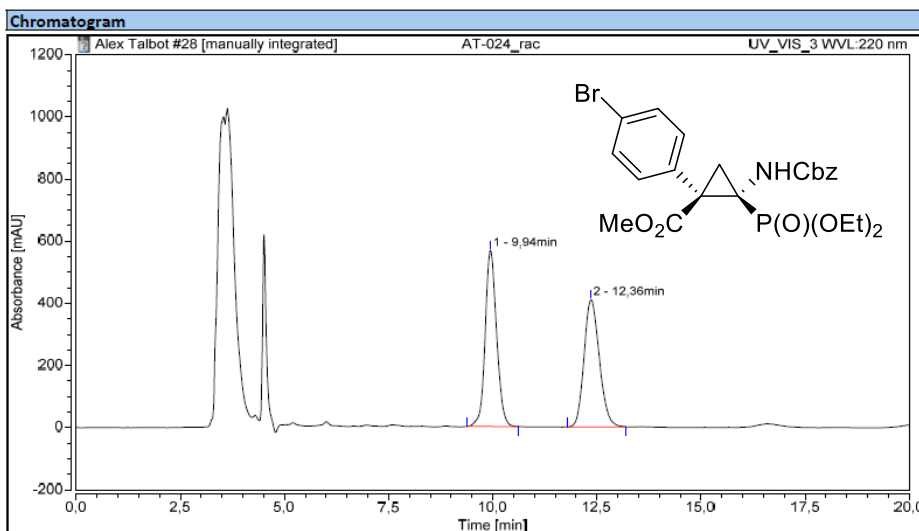
Injection Details		
Injection Name:	AT-102 ee	Run Time (min): 20.00
Vial Number:	BE2	Injection Volume: 15.00
Injection Type:	Unknown	Channel: UV_VIS_2
Comment:	IB 250*4.6 v5µm A: heptane B:EtOH 20°C 1ml/min	Wavelength: 210
Instrument Method:	97A-3B 20	Bandwidth: 2
Processing Method:	methode traitement	Dilution Factor: 1.0000
Injection Date/Time:	27/fevr./24 10:08	Sample Weight: 1.0000



Integration Results							
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		13.537	4.626	10.783	5.61	6.19	n.a.
2		14.797	77.900	163.314	94.39	93.81	n.a.
<b>Total:</b>			<b>82.526</b>	<b>174.097</b>	<b>100.00</b>	<b>100.00</b>	

**Compound 27** : methyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-1-(4-bromophenyl)-2-(diethoxy phosphoryl)cyclopropane-1-carboxylate

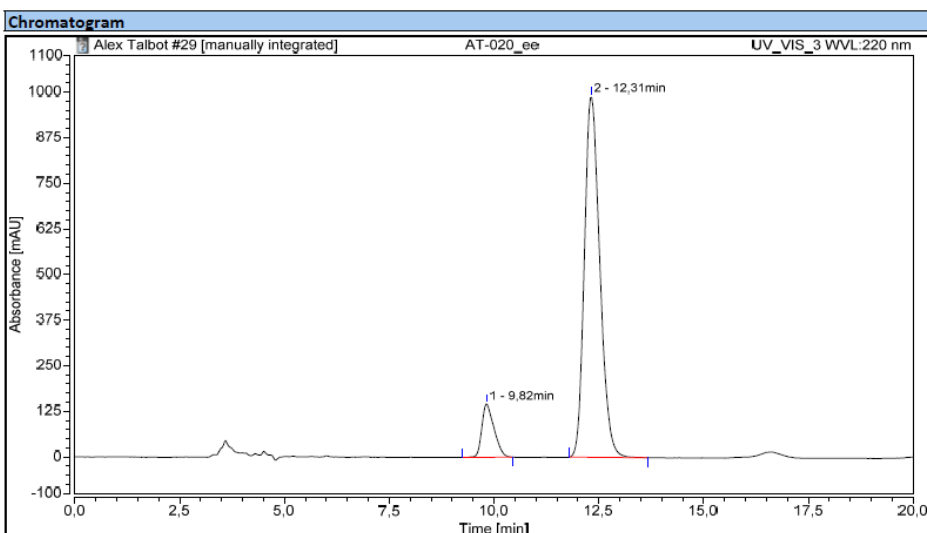
Injection Details		
Injection Name:	AT-024_rac	Run Time (min): 20,00
Vial Number:	GE2	Injection Volume: 20,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IE 250*4.6 v5µm A: heptane c:1prOH 20°C 1ml/min	Wavelength: 220
Instrument Method:	70A-30C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	22/déc.23 10:35	Sample Weight: 1,0000



**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		9,94	179,611	567,446	50,72	58,07
2		12,36	174,486	409,813	49,28	41,93
Total:			354,097	977,259	100,00	100,00

Injection Details		
Injection Name:	AT-020_ee	Run Time (min): 20,00
Vial Number:	GE3	Injection Volume: 20,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IE 250*4.6 v5µm A: heptane c:1prOH 20°C 1ml/min	Wavelength: 220
Instrument Method:	70A-30C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	22/déc.23 10:57	Sample Weight: 1,0000

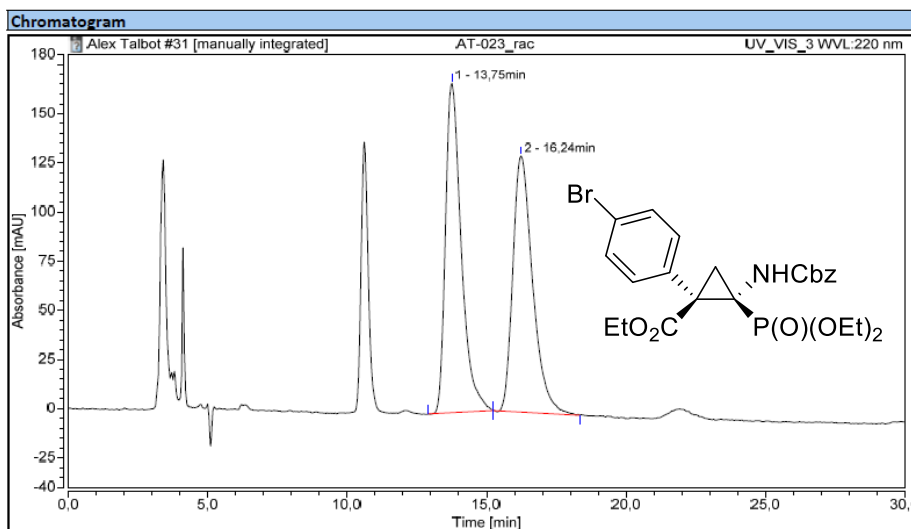


**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		9,82	49,357	146,822	10,46	12,92
2		12,31	422,660	989,930	89,54	87,08
Total:			472,018	1136,753	100,00	100,00

**Compound 28**: ethyl (1S,2R)-2-(((benzyloxy)carbonyl)amino)-1-(4-bromophenyl)-2-(diethoxyphosphoryl)cyclopropane-1-carboxylate

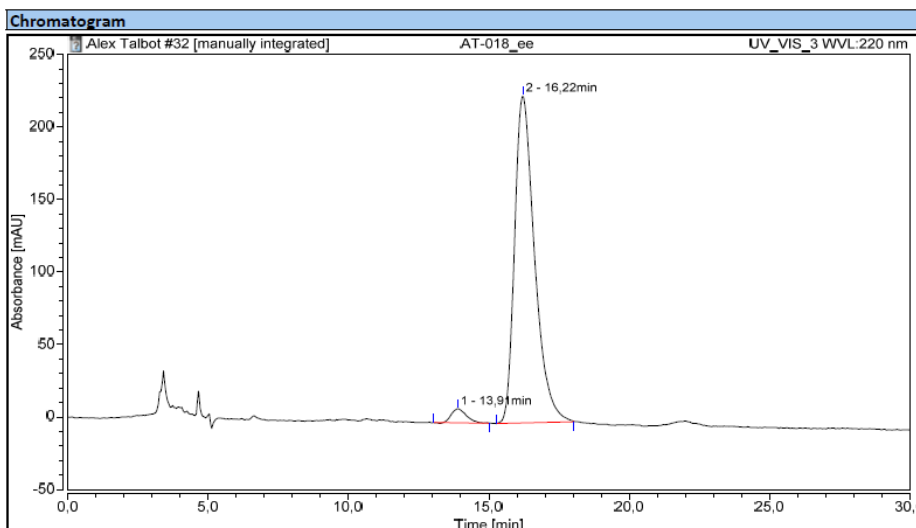
Injection Details		
Injection Name:	AT-023_rac	Run Time (min): 30,00
Vial Number:	GE1	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	ID 250*4.6 v5µm A: heptane c:lprOH 20°C 1ml/min	Wavelength: 220
Instrument Method:	80A-20C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	22/déc./23 12:41	Sample Weight: 1,0000



**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		13,75	109,727	167,500	50,64	56,25
2		16,24	106,955	130,296	49,36	43,75
Total:			216,682	297,797	100,00	100,00

Injection Details		
Injection Name:	AT-018_ee	Run Time (min): 30,00
Vial Number:	GE4	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	ID 250*4.6 v5µm A: heptane c:lprOH 20°C 1ml/min	Wavelength: 220
Instrument Method:	80A-20C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	22/déc./23 13:12	Sample Weight: 1,0000

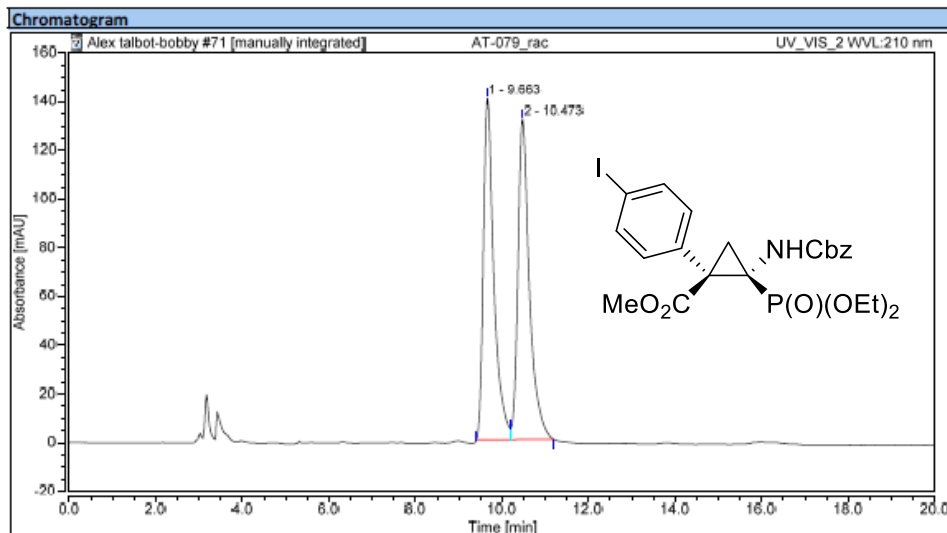


**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		13,91	5,882	9,655	3,06	4,10
2		16,22	186,471	225,584	96,94	95,90
Total:			192,353	235,239	100,00	100,00

**Compound 29** : methyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-(4-iodophenyl)cyclopropane-1-carboxylate

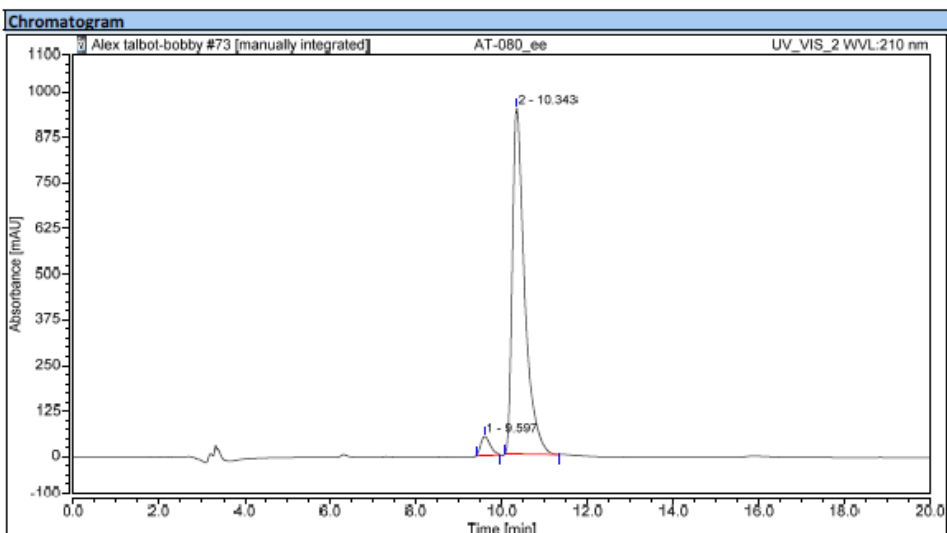
Injection Details			
Injection Name:	AT-079_rac	Run Time (min):	20.00
Vial Number:	BE3	Injection Volume:	15.00
Injection Type:	Unknown	Channel:	UV_VIS_2
Comment:	IB 250*4.6 v5µm A: heptane B:EtOH 20°C 1ml/min	Wavelength:	210
Instrument Method:	95A-5B 20	Bandwidth:	2
Processing Method:	methode traitement	Dilution Factor:	1.0000
Injection Date/Time:	02/fevr./24 09:28	Sample Weight:	1.0000



**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		9.663	39.201	140.311	49.24	51.56	n.a.
2		10.473	40.413	131.802	50.76	48.44	n.a.
<b>Total:</b>			<b>79.614</b>	<b>272.113</b>	<b>100.00</b>	<b>100.00</b>	

Injection Details			
Injection Name:	AT-080_ee	Run Time (min):	20.00
Vial Number:	BE5	Injection Volume:	15.00
Injection Type:	Unknown	Channel:	UV_VIS_2
Comment:	IB 250*4.6 v5µm A: heptane B:EtOH 20°C 1ml/min	Wavelength:	210
Instrument Method:	95A-5B 20	Bandwidth:	2
Processing Method:	methode traitement	Dilution Factor:	1.0000
Injection Date/Time:	02/fevr./24 10:18	Sample Weight:	1.0000

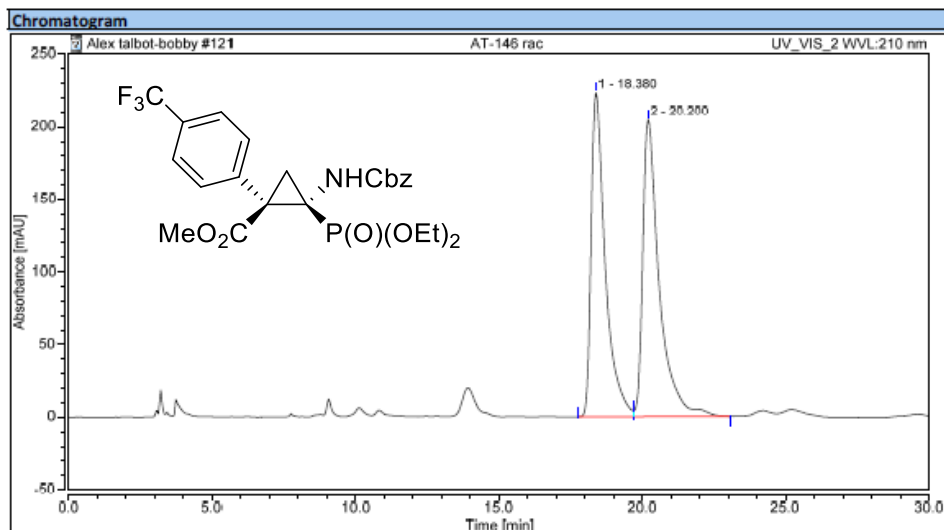


**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		9.597	13.392	52.338	4.08	5.23	n.a.
2		10.343	314.515	947.854	95.92	94.77	n.a.
<b>Total:</b>			<b>327.907</b>	<b>1000.192</b>	<b>100.00</b>	<b>100.00</b>	

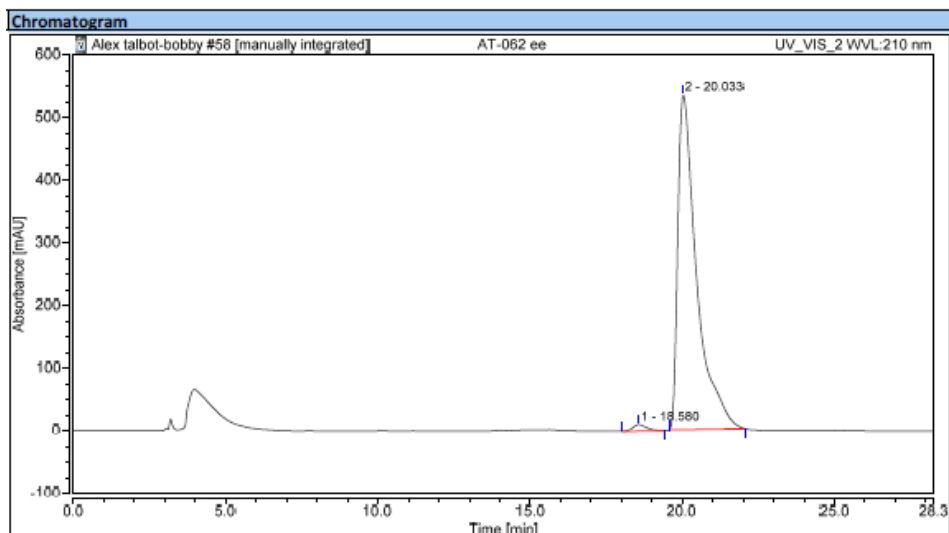
**Compound 30** : methyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-(4-(trifluoromethyl)phenyl)cyclopropane-1-carboxylate

Injection Details		
Injection Name:	AT-146 rac	Run Time (min): 30.00
Vial Number:	BD7	Injection Volume: 15.00
Injection Type:	Unknown	Channel: UV_VIS_2
Comment:	IB 250*4.6 v5µm A: heptane B:EtOH 20°C 1ml/min	Wavelength: 210
Instrument Method:	98A-2B 20C-30min	Bandwidth: 2
Processing Method:	methode traitement	Dilution Factor: 1.0000
Injection Date/Time:	23/avr/24 17:01	Sample Weight: 1.0000



Integration Results							
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		18.380	127.413	223.419	48.30	52.15	n.a.
2		20.200	136.396	205.021	51.70	47.85	n.a.
<b>Total:</b>			<b>263.809</b>	<b>428.440</b>	<b>100.00</b>	<b>100.00</b>	

Injection Details		
Injection Name:	AT-062 ee	Run Time (min): 28.26
Vial Number:	BD5	Injection Volume: 15.00
Injection Type:	Unknown	Channel: UV_VIS_2
Comment:	IB 250*4.6 v5µm A: heptane C:EtOH 20°C 1ml/min	Wavelength: 210
Instrument Method:	98A-2B 20C-30min	Bandwidth: 2
Processing Method:	methode traitement	Dilution Factor: 1.0000
Injection Date/Time:	30/janv./24 16:06	Sample Weight: 1.0000

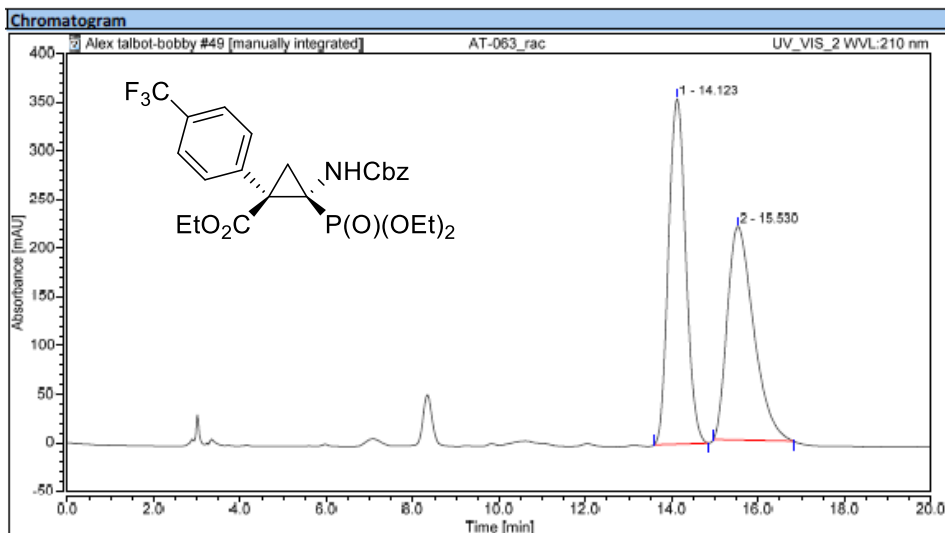


Integration Results							
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		18.580	5.645	10.377	1.45	1.90	n.a.
2		20.033	383.217	534.818	98.55	98.10	n.a.
<b>Total:</b>			<b>388.862</b>	<b>545.195</b>	<b>100.00</b>	<b>100.00</b>	



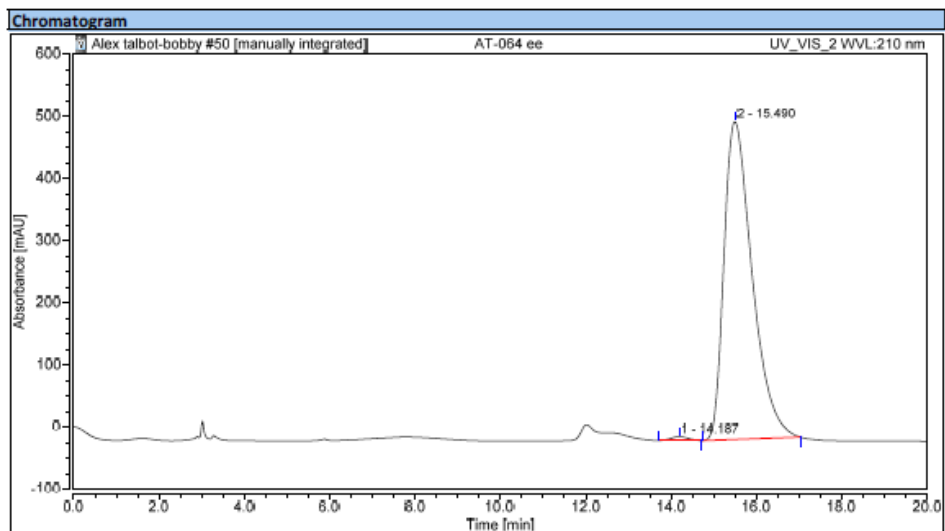
**Compound 31** : ethyl (1S,2R)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-(4-(trifluoromethyl)phenyl)cyclopropane-1-carboxylate

Injection Details		
Injection Name:	AT-063_rac	Run Time (min): 20.00
Vial Number:	BE6	Injection Volume: 15.00
Injection Type:	Unknown	Channel: UV_VIS_2
Comment:	IA 250*4.6 v5µm A: heptane C:EtOH 20°C 1ml/min	Wavelength: 210
Instrument Method:	95A-5B 20	Bandwidth: 2
Processing Method:	methode traitement	Dilution Factor: 1.0000
Injection Date/Time:	30 janv ./24 11:17	Sample Weight: 1.0000



No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		14.123	164.214	355.384	51.35	61.78	n.a.
2		15.530	155.561	219.822	48.65	38.22	n.a.
<b>Total:</b>			<b>319.776</b>	<b>575.206</b>	<b>100.00</b>	<b>100.00</b>	

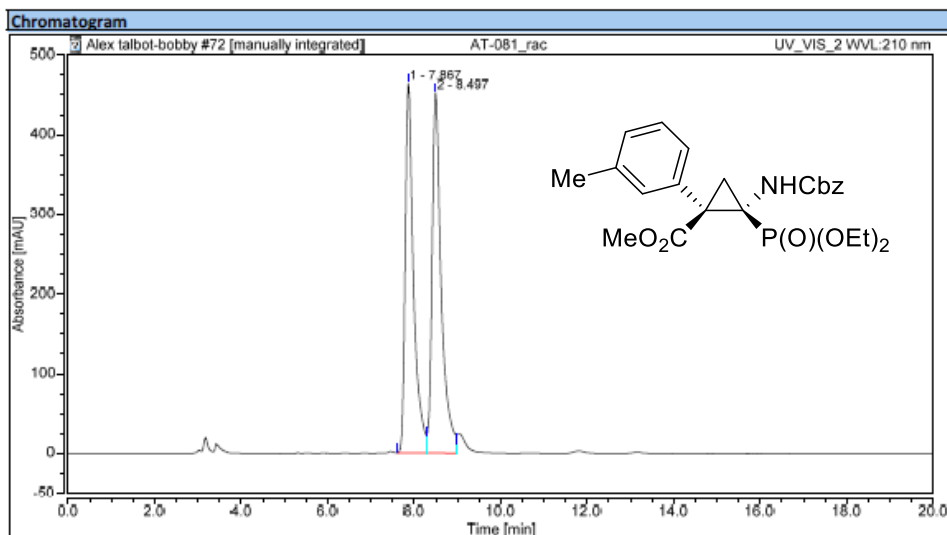
Injection Details		
Injection Name:	AT-064 ee	Run Time (min): 20.00
Vial Number:	BD6	Injection Volume: 15.00
Injection Type:	Unknown	Channel: UV_VIS_2
Comment:	IA 250*4.6 v5µm A: heptane C:EtOH 20°C 1ml/min	Wavelength: 210
Instrument Method:	95A-5B 20	Bandwidth: 2
Processing Method:	methode traitement	Dilution Factor: 1.0000
Injection Date/Time:	30 janv ./24 11:39	Sample Weight: 1.0000



No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		14.187	3.033	6.182	0.77	1.19	n.a.
2		15.490	390.858	511.754	99.23	98.81	n.a.
<b>Total:</b>			<b>393.891</b>	<b>517.936</b>	<b>100.00</b>	<b>100.00</b>	

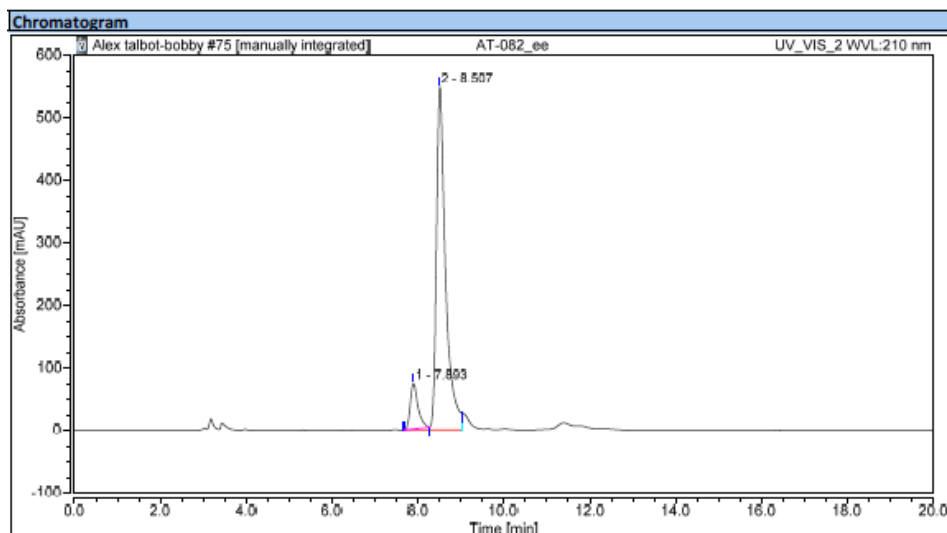
**Compound 32**: methyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-(*m*-tolyl)cyclopropane-1-carboxylate

Injection Details		
Injection Name:	AT-081_rac	Run Time (min): 20.00
Vial Number:	BE4	Injection Volume: 15.00
Injection Type:	Unknown	Channel: UV_VIS_2
Comment:	IB 250*4.6 v5µm A: heptane B:EtOH 20°C 1ml/min	Wavelength: 210
Instrument Method:	95A-5B 20	Bandwidth: 2
Processing Method:	methode traitement	Dilution Factor: 1.0000
Injection Date/Time:	02/fevr./24 09:50	Sample Weight: 1.0000



Integration Results							
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		7.867	103.204	464.007	48.18	50.62	n.a.
2		8.497	110.985	452.560	51.82	49.38	n.a.
<b>Total:</b>			<b>214.189</b>	<b>916.567</b>	<b>100.00</b>	<b>100.00</b>	

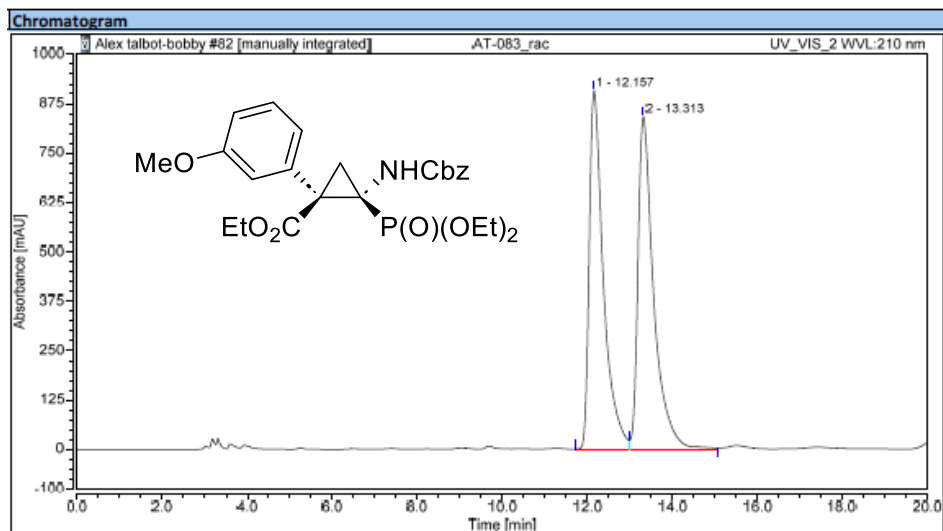
Injection Details		
Injection Name:	AT-082_ee	Run Time (min): 20.00
Vial Number:	BE7	Injection Volume: 15.00
Injection Type:	Unknown	Channel: UV_VIS_2
Comment:	IB 250*4.6 v5µm A: heptane B:EtOH 20°C 1ml/min	Wavelength: 210
Instrument Method:	95A-5B 20	Bandwidth: 2
Processing Method:	methode traitement	Dilution Factor: 1.0000
Injection Date/Time:	02/fevr./24 11:02	Sample Weight: 1.0000



Integration Results							
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		7.893	15.143	73.571	10.00	11.79	n.a.
2		8.507	136.286	550.482	90.00	88.21	n.a.
<b>Total:</b>			<b>151.429</b>	<b>624.053</b>	<b>100.00</b>	<b>100.00</b>	

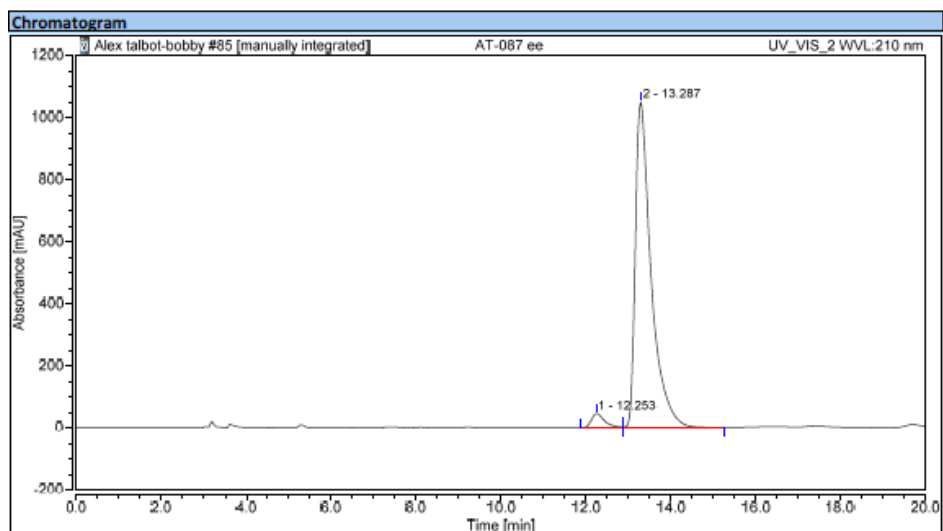
**Compound 33**: ethyl (1S,2R)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-(3-methoxyphenyl)cyclopropane-1-carboxylate

Injection Details		
Injection Name:	AT-083_rac	Run Time (min): 20.00
Vial Number:	BE1	Injection Volume: 15.00
Injection Type:	Unknown	Channel: UV_VIS_2
Comment:	IB 250*4.6 v5µm A: heptane B:EtOH 20°C 1ml/min	Wavelength: 210
Instrument Method:	97A-3B 20	Bandwidth: 2
Processing Method:	methode traitement	Dilution Factor: 1.0000
Injection Date/Time:	16/fevr./24 09:59	Sample Weight: 1.0000



Integration Results							
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		12.157	348.444	907.635	49.24	51.90	n.a.
2		13.313	359.197	841.324	50.76	48.10	n.a.
<b>Total:</b>			<b>707.641</b>	<b>1748.960</b>	<b>100.00</b>	<b>100.00</b>	

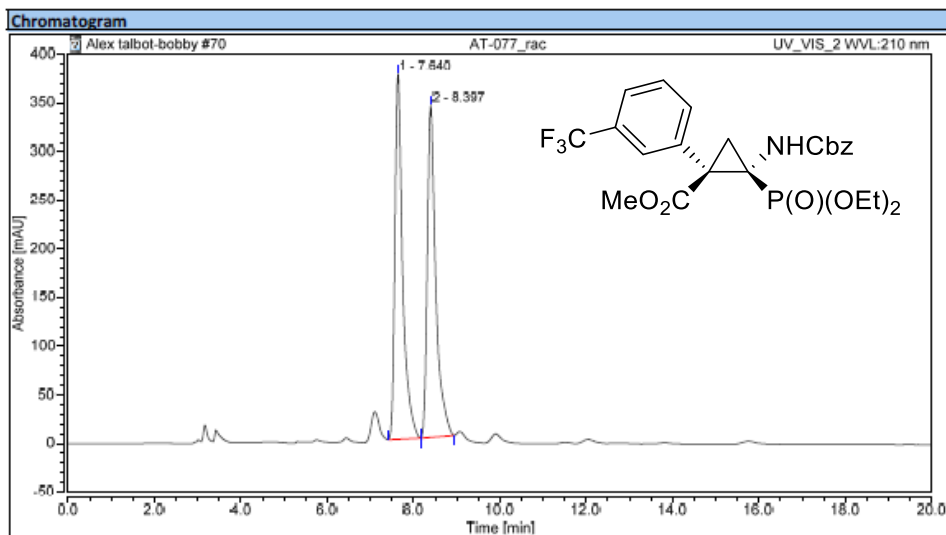
Injection Details		
Injection Name:	AT-087 ee	Run Time (min): 20.00
Vial Number:	BE4	Injection Volume: 15.00
Injection Type:	Unknown	Channel: UV_VIS_2
Comment:	IB 250*4.6 v5µm A: heptane B:EtOH 20°C 1ml/min	Wavelength: 210
Instrument Method:	97A-3B 20	Bandwidth: 2
Processing Method:	methode traitement	Dilution Factor: 1.0000
Injection Date/Time:	16/fevr./24 11:03	Sample Weight: 1.0000



Integration Results							
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		12.253	16.614	46.184	3.55	4.21	n.a.
2		13.287	451.086	1050.477	96.45	95.79	n.a.
<b>Total:</b>			<b>467.700</b>	<b>1096.660</b>	<b>100.00</b>	<b>100.00</b>	

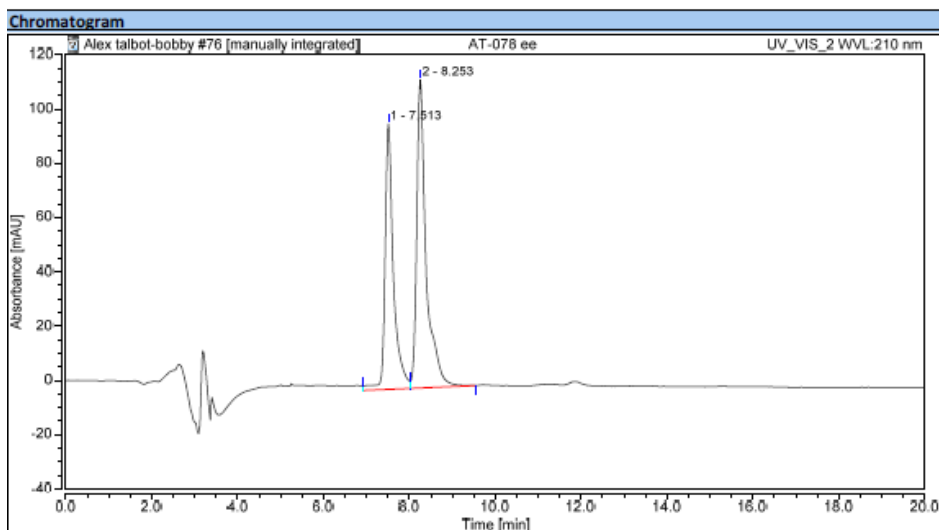
**Compound 34** : methyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-2-(diethoxyphosphoryl)-1-(3-(trifluoromethyl)phenyl)cyclopropane-1-carboxylate

Injection Details		
Injection Name:	AT-077_rac	Run Time (min): 20.00
Vial Number:	BE2	Injection Volume: 15.00
Injection Type:	Unknown	Channel: UV_VIS_2
Comment:	IB 250*4.6 v5µm A: heptane B:EtOH 20°C 1ml/min	Wavelength: 210
Instrument Method:	95A-5B 20	Bandwidth: 2
Processing Method:	methode traitement	Dilution Factor: 1.0000
Injection Date/Time:	02/fevr./24 09:07	Sample Weight: 1.0000



Integration Results							
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount
1		7.640	78.256	376.034	50.20	52.37	n.a.
2		8.397	77.636	342.018	49.80	47.63	n.a.
<b>Total:</b>			<b>155.892</b>	<b>718.052</b>	<b>100.00</b>	<b>100.00</b>	

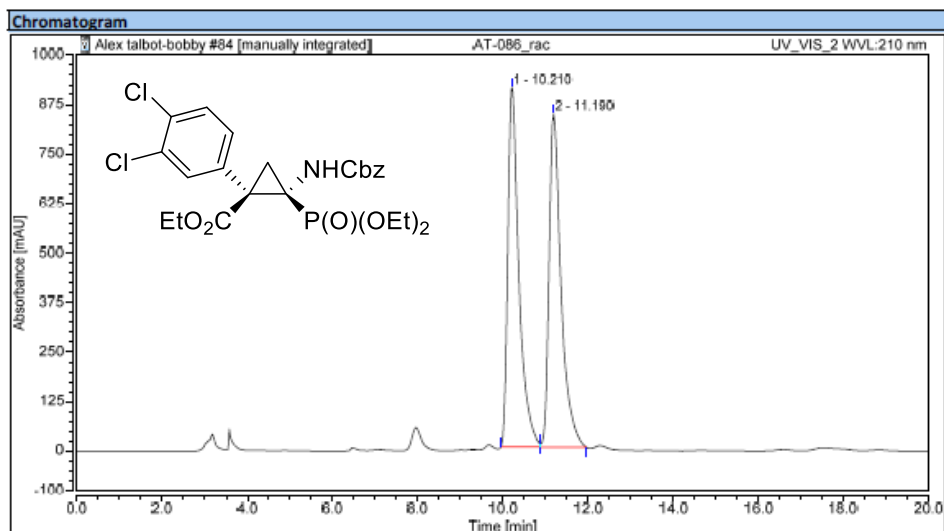
Injection Details		
Injection Name:	AT-078 ee	Run Time (min): 20.00
Vial Number:	BE8	Injection Volume: 15.00
Injection Type:	Unknown	Channel: UV_VIS_2
Comment:	IB 250*4.6 v5µm A: heptane B:EtOH 20°C 1ml/min	Wavelength: 210
Instrument Method:	95A-5B 20	Bandwidth: 2
Processing Method:	methode traitement	Dilution Factor: 1.0000
Injection Date/Time:	02/fevr./24 11:57	Sample Weight: 1.0000



Integration Results							
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount
1		7.513	22.031	97.794	43.34	46.29	n.a.
2		8.253	28.799	113.492	56.66	53.71	n.a.
<b>Total:</b>			<b>50.830</b>	<b>211.285</b>	<b>100.00</b>	<b>100.00</b>	

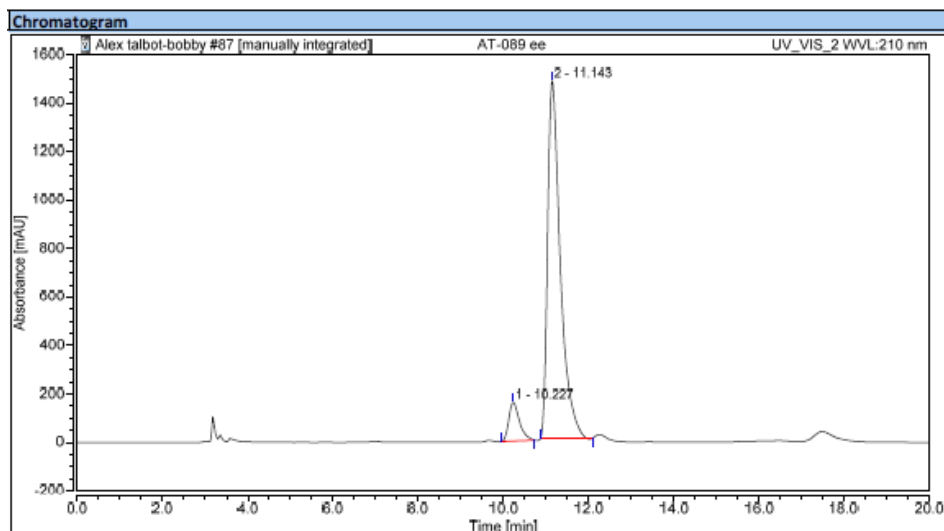
**Compound 35**: ethyl (1*S*,2*R*)-2-(((benzyloxy)carbonyl)amino)-1-(3,4-dichlorophenyl)-2-(diethoxyphosphoryl)cyclopropane-1-carboxylate

Injection Details			
Injection Name:	AT-086_rac	Run Time (min):	20.00
Vial Number:	BE3	Injection Volume:	15.00
Injection Type:	Unknown	Channel:	UV_VIS_2
Comment:	IB 250*4.6 v5µm A: heptane B:EtOH 20°C 1ml/min	Wavelength:	210
Instrument Method:	97A-3B 20	Bandwidth:	2
Processing Method:	methode traitement	Dilution Factor:	1.0000
Injection Date/Time:	16/fevr./24 10:42	Sample Weight:	1.0000



Integration Results							
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		10.210	273.590	907.890	50.02	51.89	n.a.
2		11.190	273.403	841.702	49.98	48.11	n.a.
<b>Total:</b>			<b>546.994</b>	<b>1749.592</b>	<b>100.00</b>	<b>100.00</b>	

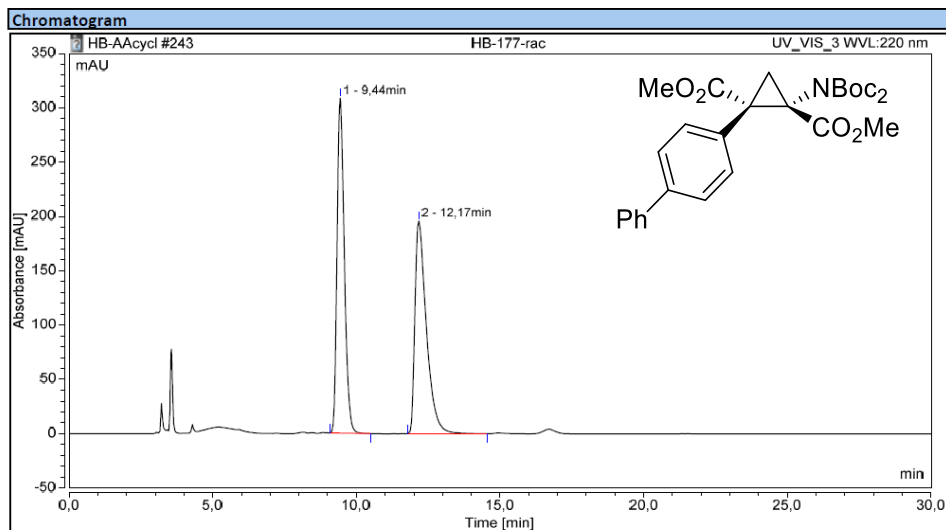
Injection Details			
Injection Name:	AT-089 ee	Run Time (min):	20.00
Vial Number:	BE6	Injection Volume:	15.00
Injection Type:	Unknown	Channel:	UV_VIS_2
Comment:	IB 250*4.6 v5µm A: heptane B:EtOH 20°C 1ml/min	Wavelength:	210
Instrument Method:	97A-3B 20	Bandwidth:	2
Processing Method:	methode traitement	Dilution Factor:	1.0000
Injection Date/Time:	16/fevr./24 11:46	Sample Weight:	1.0000



Integration Results							
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		10.227	46.414	160.694	8.36	9.81	n.a.
2		11.143	508.909	1477.161	91.64	90.19	n.a.
<b>Total:</b>			<b>555.323</b>	<b>1637.856</b>	<b>100.00</b>	<b>100.00</b>	

**Compound 36**: dimethyl (1S,2S)-1-([1,1'-biphenyl]-4-yl)-2-(bis(tert-butoxycarbonyl)amino)cyclopropane-1,2-dicarboxylate

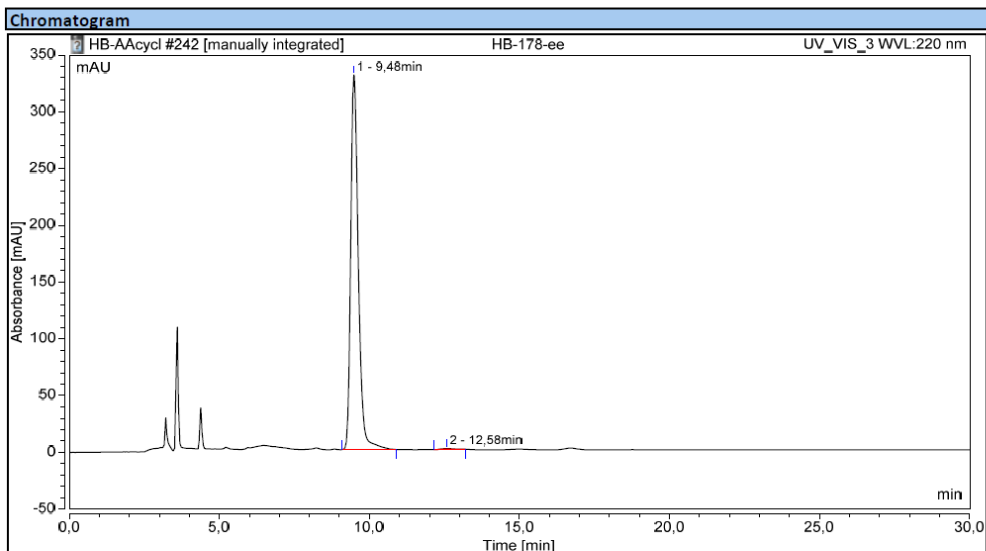
Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	GB6	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IE_Hept:iPrOH 90/10 20°C	Wavelength: 220
Instrument Method:	90A-10C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	04/avr./22 11:48	Sample Weight: 1,0000



**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		9,44	90,145	308,466	49,96	61,16
2		12,17	90,289	195,881	50,04	38,84
<b>Total:</b>			<b>180,434</b>	<b>504,346</b>	<b>100,00</b>	<b>100,00</b>

Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	GB5	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IE_Hept:iPrOH 90/10 20°C	Wavelength: 220
Instrument Method:	90A-10C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	04/avr./22 11:16	Sample Weight: 1,0000

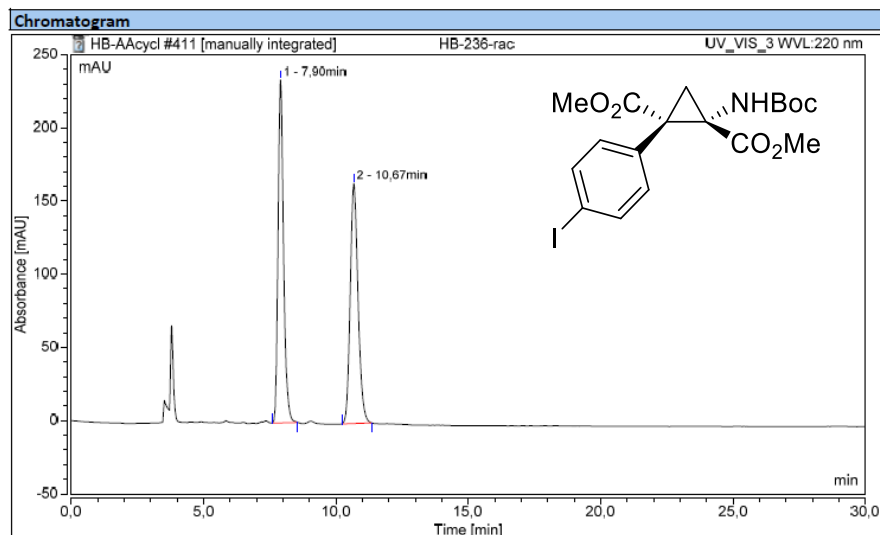


**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %
1		9,48	97,273	330,135	99,65	99,78
2		12,58	0,345	0,737	0,35	0,22
<b>Total:</b>			<b>97,618</b>	<b>330,872</b>	<b>100,00</b>	<b>100,00</b>

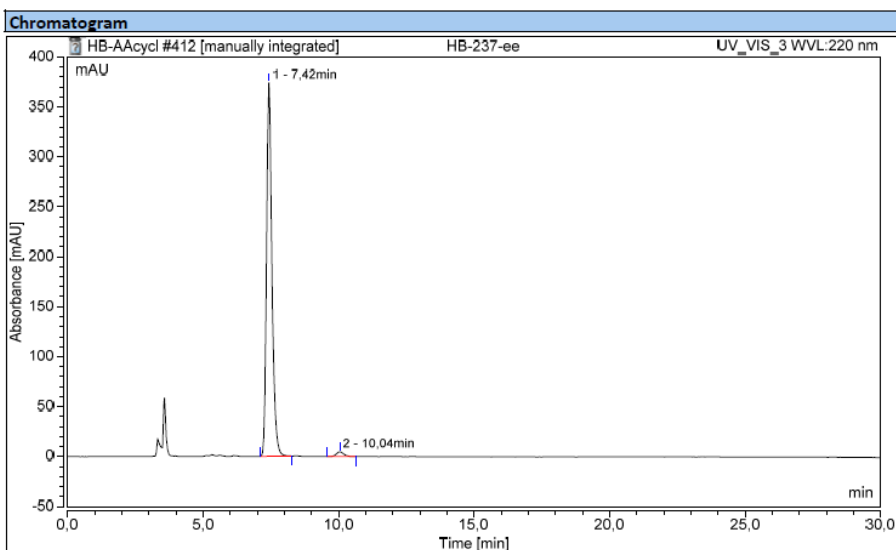
**Compound 37**: dimethyl (1S,2S)-1-((tert-butoxycarbonyl)amino)-2-(4-iodophenyl)cyclopropane-1,2-dicarboxylate

Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	RE3	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IC_Hept:iPrOH 80/20 20°C	Wavelength: 220
Instrument Method:	80A-20C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	17/juin/22 08:17	Sample Weight: 1,0000



Integration Results							
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Resolution (EP)
1		7,90	56,505	234,258	50,05	58,81	6,15
2		10,67	56,385	164,044	49,95	41,19	n.a.
Total:			112,890	398,303	100,00	100,00	

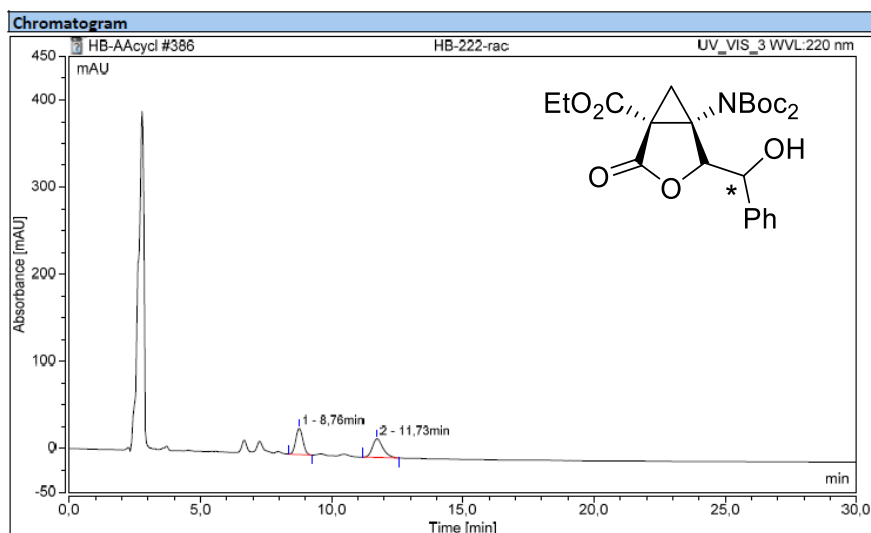
Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	RE4	Injection Volume: 10,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IC_Hept:iPrOH 80/20 20°C	Wavelength: 220
Instrument Method:	80A-20C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	17/juin/22 08:49	Sample Weight: 1,0000



Integration Results							
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Resolution (EP)
1		7,42	86,695	373,859	98,32	98,78	6,08
2		10,04	1,484	4,617	1,68	1,22	n.a.
Total:			88,179	378,476	100,00	100,00	

**Compound 38: ethyl (1R,2S,5S)-5-(bis(tert-butoxycarbonyl)amino)-2-(hydroxy(phenyl)methyl)-4-oxo-3-oxabicyclo[3.1.0]hexane-1-carboxylate**

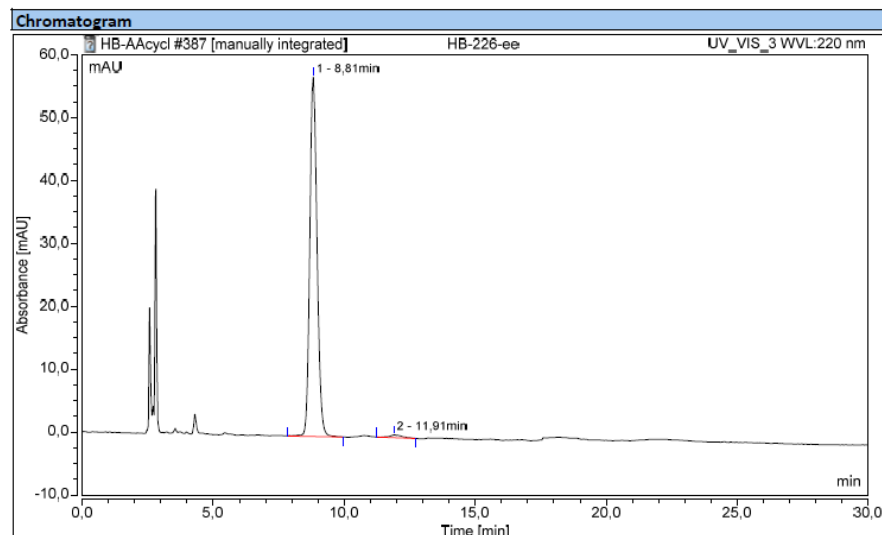
Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	RA1	Injection Volume: 15,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IE_Hept:PrOH 80/20 20°C 1.3ml/min	Wavelength: 220
Instrument Method:	75A-25C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	13/juin/22 08:52	Sample Weight: 1,0000



**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Resolution (EP)
1		8,76	9,391	30,271	48,48	58,41	4,94
2		11,73	9,979	21,558	51,52	41,59	n.a.
Total:			19,370	51,829	100,00	100,00	

Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	RA2	Injection Volume: 1,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IE_Hept:PrOH 80/20 20°C 1.3ml/min	Wavelength: 220
Instrument Method:	75A-25C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	13/juin/22 09:23	Sample Weight: 1,0000



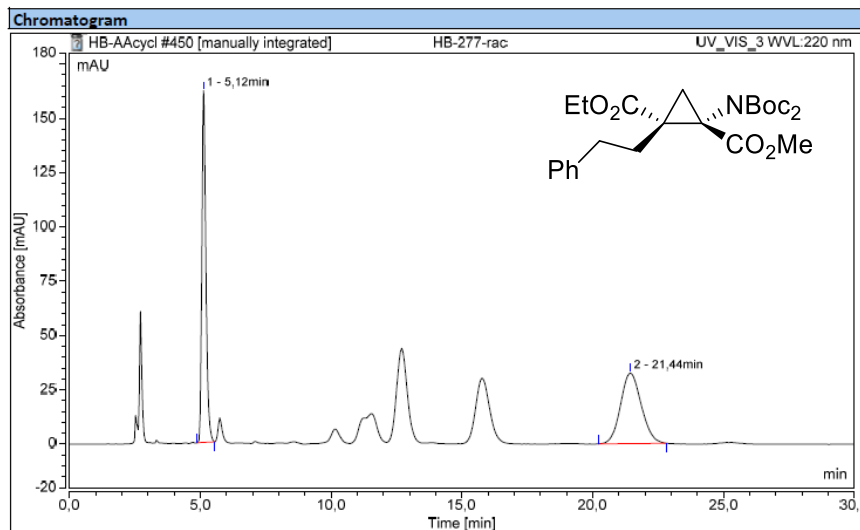
**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Resolution (EP)
1		8,81	18,361	57,132	98,66	99,20	4,78
2		11,91	0,249	0,459	1,34	0,80	n.a.
Total:			18,610	57,592	100,00	100,00	



**Compound 39 : 2-ethyl 1-methyl (1S,2R)-1-(bis(tert-butoxycarbonyl)amino)-2-phenethylcyclopropane-1,2-dicarboxylate**

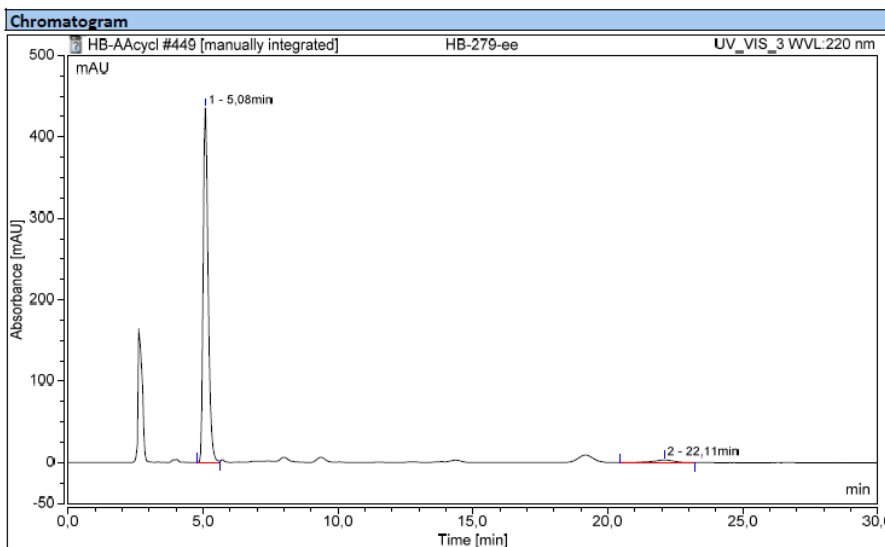
Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	RA7	Injection Volume: 20,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IC_Hept:iPrOH 90/10 20°C	Wavelength: 220
Instrument Method:	90A-10C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	18/juil./22 11:48	Sample Weight: 1,0000



**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Resolution (EP)
1		5,12	28,821	161,967	49,20	83,31	18,98
2		21,44	29,755	32,455	50,80	16,69	n.a.
Total:			58,576	194,421	100,00	100,00	

Injection Details		
Injection Name:	HB-78-rac	Run Time (min): 30,00
Vial Number:	RA8	Injection Volume: 20,00
Injection Type:	Unknown	Channel: UV_VIS_3
Comment:	IC_Hept:iPrOH 90/10 20°C	Wavelength: 220
Instrument Method:	90A-10C	Bandwidth: 4
Processing Method:	methode traitement	Dilution Factor: 1,0000
Injection Date/Time:	18/juil./22 11:17	Sample Weight: 1,0000

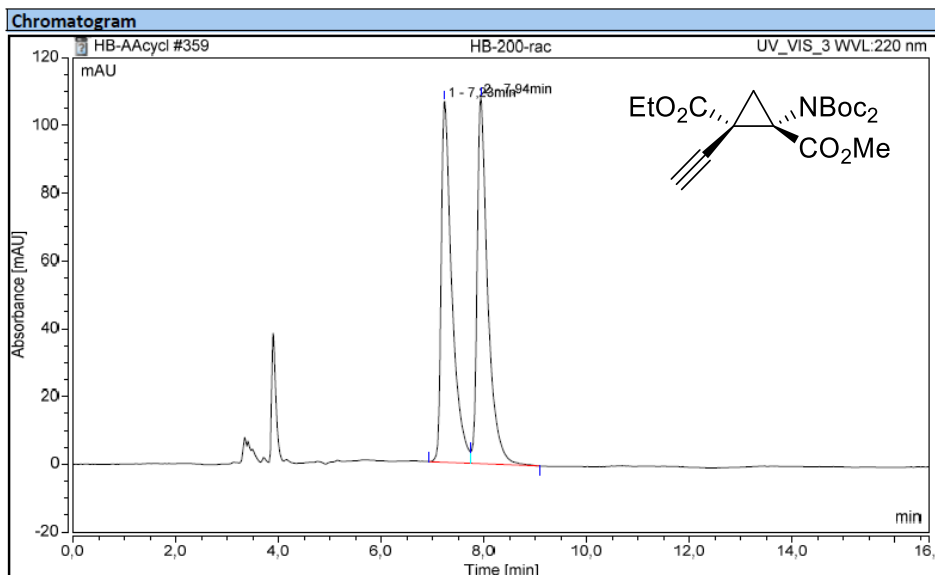


**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Resolution (EP)
1		5,08	92,309	435,270	96,63	99,25	19,77
2		22,11	3,215	3,299	3,37	0,75	n.a.
Total:			95,524	438,569	100,00	100,00	

**Compound 40 : 2-ethyl 1-methyl (1S,2S)-1-(bis(tert-butoxycarbonyl)amino)-2-ethynylcyclopropane-1,2-dicarboxylate**

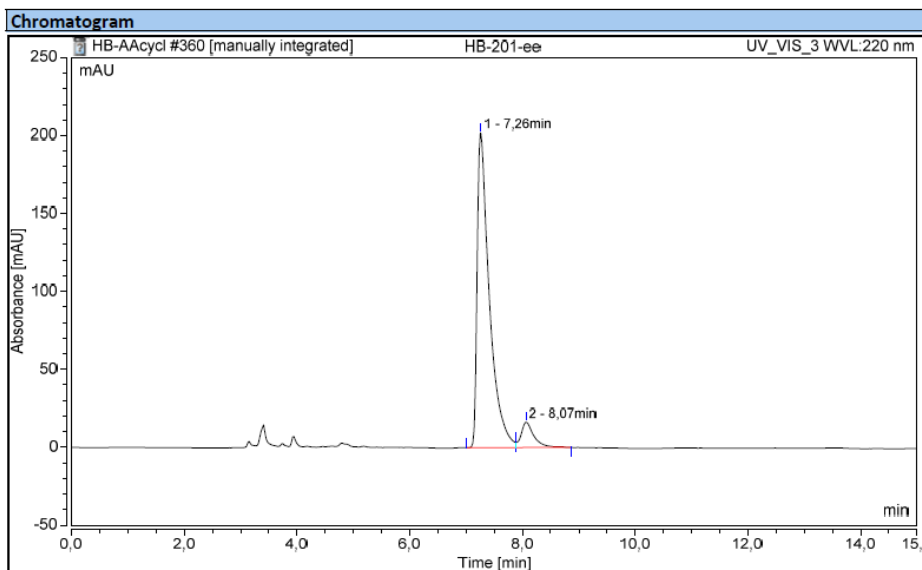
Injection Name:	HB-78-rac	Run Time (min):	16,67
Vial Number:	RB6	Injection Volume:	10,00
Injection Type:	Unknown	Channel:	UV_VIS_3
Comment:	IB_Hept:iPrOH 98/2 15°C	Wavelength:	220
Instrument Method:	98A-2C_20min	Bandwidth:	4
Processing Method:	methode traitement	Dilution Factor:	1,0000
Injection Date/Time:	31/mai/22 09:40	Sample Weight:	1,0000



**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Resolution (EP)
1		7,23	25,729	106,568	49,81	49,66	2,04
2		7,94	25,924	108,007	50,19	50,34	n.a.
<b>Total:</b>			<b>51,653</b>	<b>214,575</b>	<b>100,00</b>	<b>100,00</b>	

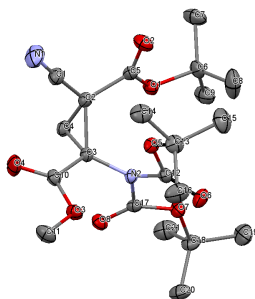
Injection Name:	HB-78-rac	Run Time (min):	15,00
Vial Number:	RB7	Injection Volume:	10,00
Injection Type:	Unknown	Channel:	UV_VIS_3
Comment:	IB_Hept:iPrOH 98/2 15°C	Wavelength:	220
Instrument Method:	98A-2C_20min	Bandwidth:	4
Processing Method:	methode traitement	Dilution Factor:	1,0000
Injection Date/Time:	31/mai/22 09:59	Sample Weight:	1,0000



**Integration Results**

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Resolution (EP)
1		7,26	50,589	201,889	92,15	92,50	2,21
2		8,07	4,312	16,367	7,85	7,50	n.a.
<b>Total:</b>			<b>54,901</b>	<b>218,256</b>	<b>100,00</b>	<b>100,00</b>	

## 8. X-ray crystallographic data of 1 (CCDC 2177765)



**Table 1.** Crystal data and structure refinement for M22\_05\_Beucher\_23-1\_A1.

Identification code	M22_05_Beucher_23-1_A1
Empirical formula	C <sub>21</sub> H <sub>32</sub> N <sub>2</sub> O <sub>8</sub>
Formula weight	440.48
Temperature/K	100.00(10)
Crystal system	orthorhombic
Space group	P2 <sub>1</sub> 2 <sub>1</sub> 2 <sub>1</sub>
a/Å	8.9833(2)
b/Å	13.0769(2)
c/Å	20.1678(3)
α/°	90
β/°	90
γ/°	90
Volume/Å <sup>3</sup>	2369.19(7)
Z	4
ρ <sub>calc</sub> /cm <sup>3</sup>	1.235
μ/mm <sup>-1</sup>	0.791
F(000)	944.0
Crystal size/mm <sup>3</sup>	0.394 × 0.107 × 0.081
Radiation	Cu Kα (λ = 1.54184)
2θ range for data collection/°	8.058 to 153.018
Index ranges	-10 ≤ h ≤ 11, -16 ≤ k ≤ 15, -24 ≤ l ≤ 25
Reflections collected	22611
Independent reflections	4942 [R <sub>int</sub> = 0.0240, R <sub>sigma</sub> = 0.0156]
Data/restraints/parameters	4942/0/377
Goodness-of-fit on F <sup>2</sup>	1.023
Final R indexes [I ≥ 2σ (I)]	R <sub>1</sub> = 0.0256, wR <sub>2</sub> = 0.0669
Final R indexes [all data]	R <sub>1</sub> = 0.0262, wR <sub>2</sub> = 0.0674
Largest diff. peak/hole / e Å <sup>-3</sup>	0.19/-0.18
Flack parameter	0.00(4)

**Table 2.** Fractional Atomic Coordinates ( $\times 10^4$ ) and Equivalent Isotropic Displacement Parameters ( $\text{\AA}^2 \times 10^3$ ) for M22\_05\_Beucher\_23-1\_A1.  $U_{eq}$  is defined as 1/3 of the trace of the orthogonalised  $U_{ij}$  tensor.

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> (eq)
O1	1558.2(12)	5393.2(8)	4865.1(5)	22.5(2)
O5	4582.9(11)	4750.7(7)	3823.3(5)	19.4(2)
O3	6785.2(11)	6363.0(8)	3998.3(5)	22.6(2)
O8	5485.0(12)	6498.4(8)	5679.0(5)	22.3(2)
O7	4862.7(12)	4832.2(8)	5815.4(5)	22.0(2)
O2	1088.2(13)	5208.0(9)	3769.4(5)	28.2(2)
O6	5532.2(13)	3864.7(8)	4694.4(5)	26.9(2)
O4	5431.2(14)	7762.3(9)	3768.6(6)	32.2(3)
N2	4726.9(13)	5535.3(8)	4796.2(5)	16.0(2)
C10	5568.1(17)	6940.4(10)	4029.6(7)	19.8(3)
C17	5090.0(14)	5675.8(10)	5475.7(7)	17.1(3)
C12	5001.1(15)	4616.9(10)	4450.4(7)	18.3(3)
C5	1699.9(15)	5613.0(11)	4228.8(7)	19.6(3)
C3	4356.0(15)	6447.1(10)	4436.0(6)	16.6(3)
C4	3210.3(16)	7161.6(10)	4708.8(7)	19.7(3)
N1	2422(2)	7263.5(13)	2956.6(8)	41.2(4)
C2	2755.0(15)	6507.7(11)	4130.1(7)	18.7(3)
C1	2599.3(18)	6944.8(12)	3476.2(8)	25.0(3)
C18	5318.9(17)	4750.5(13)	6524.5(7)	24.9(3)
C6	684.0(17)	4488.0(12)	5095.7(8)	26.1(3)
C13	5022.0(18)	4003.9(12)	3301.0(7)	25.6(3)
C20	6996.5(19)	4868.3(16)	6571.5(8)	32.3(4)
C11	7965.0(19)	6759.9(14)	3577.3(8)	29.1(3)
C21	4501(2)	5538.7(16)	6942.6(8)	33.9(4)
C14	4594(2)	4572.4(14)	2674.6(8)	31.4(4)
C7	-927.6(19)	4609.3(18)	4891.1(9)	36.9(4)
C9	852(2)	4575.5(16)	5842.5(9)	36.2(4)
C16	6687(2)	3813.1(16)	3332.1(9)	36.7(4)
C19	4840(2)	3666.9(16)	6691.6(9)	39.5(4)
C15	4100(3)	3038.9(14)	3382.6(10)	40.4(4)
C8	1412(3)	3533.8(16)	4832.4(13)	49.4(6)

**Table 3.** Anisotropic Displacement Parameters ( $\text{\AA}^2 \times 10^3$ ) for M22\_05\_Beucher\_23-1\_A1.  
 The Anisotropic displacement factor exponent takes the form:  $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\dots]$ .

Atom	$U_{11}$	$U_{22}$	$U_{33}$	$U_{23}$	$U_{13}$	$U_{12}$
O1	22.3(5)	24.0(5)	21.2(5)	4.5(4)	-1.2(4)	-6.4(4)
O5	23.5(5)	18.7(5)	15.9(4)	-3.3(4)	-1.0(4)	2.4(4)
O3	18.7(5)	25.7(5)	23.3(5)	1.3(4)	2.7(4)	-3.0(4)
O8	25.2(5)	22.9(5)	18.8(4)	-1.6(4)	-2.1(4)	-4.6(4)
O7	26.5(5)	23.8(5)	15.6(4)	4.7(4)	-2.1(4)	-4.1(4)
O2	25.9(5)	35.4(6)	23.3(5)	-4.1(4)	-1.4(4)	-7.9(5)
O6	37.1(6)	19.8(5)	24.0(5)	1.6(4)	-2.2(5)	7.5(5)
O4	34.3(6)	22.3(5)	40.1(6)	11.2(5)	7.8(5)	-1.3(5)
N2	18.8(5)	15.1(5)	14.0(5)	0.6(4)	-0.1(4)	0.0(4)
C10	23.4(7)	18.5(6)	17.5(6)	-1.0(5)	0.1(5)	-3.1(5)
C17	14.3(6)	21.3(6)	15.8(6)	0.3(5)	0.1(5)	0.3(5)
C12	18.7(6)	17.9(6)	18.2(6)	-0.2(5)	0.8(5)	-0.4(5)
C5	16.8(6)	19.5(6)	22.4(6)	0.1(5)	-0.5(5)	1.7(5)
C3	19.0(6)	15.3(6)	15.7(6)	0.4(5)	0.0(5)	-0.5(5)
C4	22.1(7)	16.5(6)	20.4(7)	-1.4(5)	-0.2(5)	1.8(5)
N1	59.1(10)	37.2(8)	27.5(8)	10.5(6)	-10.2(7)	-3.7(8)
C2	19.9(7)	18.0(6)	18.1(6)	1.5(5)	-0.7(5)	1.1(5)
C1	29.5(7)	22.2(7)	23.3(7)	2.0(6)	-2.4(6)	-0.7(6)
C18	25.0(7)	35.3(8)	14.4(6)	6.8(6)	-1.4(5)	-0.3(6)
C6	22.6(7)	25.6(8)	30.2(8)	6.2(6)	2.7(6)	-7.6(6)
C13	31.2(8)	25.0(7)	20.8(7)	-10.4(6)	-0.1(6)	3.4(6)
C20	24.5(7)	46.6(10)	25.8(8)	8.2(7)	-3.9(6)	4.0(7)
C11	23.5(7)	37.2(9)	26.6(8)	-1.6(7)	6.6(6)	-8.2(7)
C21	32.1(9)	51.8(11)	17.9(7)	2.4(7)	4.7(6)	4.1(8)
C14	35.4(9)	39.6(9)	19.1(7)	-6.8(6)	-1.0(6)	1.8(8)
C7	23.3(8)	55.8(12)	31.6(9)	7.2(8)	-1.5(7)	-13.1(8)
C9	30.6(8)	47.9(10)	30.2(8)	15.0(8)	-4.2(7)	-15.4(8)
C16	34.8(9)	46.1(10)	29.4(8)	-12.8(8)	-0.2(7)	14.5(8)
C19	48.8(11)	41.2(10)	28.5(8)	17.1(8)	-6.8(8)	-8.0(9)
C15	59.2(12)	25.9(8)	36.1(10)	-10.4(7)	-1.2(9)	-7.1(8)
C8	58.2(13)	24.8(8)	65.3(14)	8.5(9)	26.8(12)	-3.1(9)

**Table 4.** Bond Lengths for M22\_05\_Beucher\_23-1\_A1.

<b>Atom</b>	<b>Atom</b>	<b>Length/Å</b>	<b>Atom</b>	<b>Atom</b>	<b>Length/Å</b>
O1	C5	1.3211(17)	C5	C2	1.5189(19)
O1	C6	1.4947(17)	C3	C4	1.4950(19)
O5	C12	1.3310(17)	C3	C2	1.5669(18)
O5	C13	1.4895(16)	C4	C2	1.5035(19)
O3	C10	1.3303(18)	N1	C1	1.139(2)
O3	C11	1.4538(18)	C2	C1	1.444(2)
O8	C17	1.2046(17)	C18	C20	1.518(2)
O7	C17	1.3146(17)	C18	C21	1.521(2)
O7	C18	1.4915(16)	C18	C19	1.519(2)
O2	C5	1.2003(18)	C6	C7	1.514(2)
O6	C12	1.1989(18)	C6	C9	1.518(2)
O4	C10	1.2030(18)	C6	C8	1.506(3)
N2	C17	1.4205(17)	C13	C14	1.515(2)
N2	C12	1.4105(17)	C13	C16	1.518(2)
N2	C3	1.4355(17)	C13	C15	1.518(2)
C10	C3	1.5078(19)			

**Table 5.** Bond Angles for M22\_05\_Beucher\_23-1\_A1.

<b>Atom</b>	<b>Atom</b>	<b>Atom</b>	<b>Angle/°</b>	<b>Atom</b>	<b>Atom</b>	<b>Atom</b>	<b>Angle/°</b>
C5	O1	C6	121.68(12)	C5	C2	C3	118.83(11)
C12	O5	C13	120.74(11)	C4	C2	C5	120.40(12)
C10	O3	C11	115.10(12)	C4	C2	C3	58.23(9)
C17	O7	C18	121.15(11)	C1	C2	C5	111.35(12)
C17	N2	C3	115.73(11)	C1	C2	C3	117.94(12)
C12	N2	C17	123.16(11)	C1	C2	C4	120.67(13)
C12	N2	C3	119.84(10)	N1	C1	C2	176.96(18)
O3	C10	C3	112.12(12)	O7	C18	C20	108.99(12)
O4	C10	O3	124.77(14)	O7	C18	C21	110.50(12)
O4	C10	C3	123.11(14)	O7	C18	C19	101.64(12)
O8	C17	O7	128.15(13)	C20	C18	C21	112.11(15)
O8	C17	N2	120.76(12)	C20	C18	C19	111.23(15)
O7	C17	N2	111.01(11)	C19	C18	C21	111.87(15)
O5	C12	N2	107.98(11)	O1	C6	C7	109.55(13)
O6	C12	O5	127.60(13)	O1	C6	C9	101.34(12)
O6	C12	N2	124.42(12)	O1	C6	C8	108.56(13)
O1	C5	C2	110.80(12)	C7	C6	C9	110.97(14)
O2	C5	O1	127.56(14)	C8	C6	C7	113.97(18)
O2	C5	C2	121.63(13)	C8	C6	C9	111.66(17)
N2	C3	C10	117.57(12)	O5	C13	C14	101.59(12)
N2	C3	C4	119.52(11)	O5	C13	C16	109.85(13)
N2	C3	C2	117.02(11)	O5	C13	C15	108.90(13)
C10	C3	C2	115.29(11)	C14	C13	C16	111.40(15)
C4	C3	C10	115.45(12)	C14	C13	C15	111.10(15)
C4	C3	C2	58.76(9)	C16	C13	C15	113.34(16)
C3	C4	C2	63.01(9)				

**Table 6.** Torsion Angles for M22\_05\_Beucher\_23-1\_A1.

<b>A</b>	<b>B</b>	<b>C</b>	<b>D</b>	<b>Angle/°</b>	<b>A</b>	<b>B</b>	<b>C</b>	<b>D</b>	<b>Angle/°</b>
O1	C5	C2	C3	54.45(16)	C12	O5	C13	C14	-168.69(13)
O1	C5	C2	C4	-13.60(18)	C12	O5	C13	C16	-50.65(19)
O1	C5	C2	C1	-163.42(12)	C12	O5	C13	C15	74.02(17)
O3	C10	C3	N2	8.35(16)	C12	N2	C17	O8	146.15(13)
O3	C10	C3	C4	158.13(12)	C12	N2	C17	O7	-36.79(17)
O3	C10	C3	C2	-136.10(12)	C12	N2	C3	C10	-70.17(16)
O2	C5	C2	C3	-127.00(15)	C12	N2	C3	C4	141.31(13)
O2	C5	C2	C4	164.95(13)	C12	N2	C3	C2	73.66(16)
O2	C5	C2	C1	15.13(19)	C5	O1	C6	C7	-62.43(18)
O4	C10	C3	N2	-171.53(13)	C5	O1	C6	C9	-179.73(14)
O4	C10	C3	C4	-21.8(2)	C5	O1	C6	C8	62.6(2)
O4	C10	C3	C2	44.02(19)	C3	N2	C17	O8	-20.91(19)
N2	C3	C4	C2	-105.50(13)	C3	N2	C17	O7	156.15(11)
N2	C3	C2	C5	-0.07(18)	C3	N2	C12	O5	-11.24(17)
N2	C3	C2	C4	109.72(13)	C3	N2	C12	O6	168.17(14)
N2	C3	C2	C1	-139.74(13)	C3	C4	C2	C5	107.11(14)
C10	C3	C4	C2	105.34(13)	C3	C4	C2	C1	-105.87(15)
C10	C3	C2	C5	144.58(13)	C4	C3	C2	C5	-109.79(14)
C10	C3	C2	C4	-105.63(13)	C4	C3	C2	C1	110.54(15)
C10	C3	C2	C1	4.91(18)	C18	O7	C17	O8	-10.0(2)
C17	O7	C18	C20	-62.38(18)	C18	O7	C17	N2	173.19(11)
C17	O7	C18	C21	61.23(17)	C6	O1	C5	O2	6.8(2)
C17	O7	C18	C19	-179.89(14)	C6	O1	C5	C2	-174.75(12)
C17	N2	C12	O5	-177.80(11)	C13	O5	C12	O6	-12.5(2)
C17	N2	C12	O6	1.6(2)	C13	O5	C12	N2	166.93(11)
C17	N2	C3	C10	97.36(14)	C11	O3	C10	O4	-4.1(2)
C17	N2	C3	C4	-51.16(17)	C11	O3	C10	C3	176.06(12)
C17	N2	C3	C2	-118.81(13)					



**Table 7.** Hydrogen Atom Coordinates ( $\text{\AA}\times 10^4$ ) and Isotropic Displacement Parameters ( $\text{\AA}^2\times 10^3$ ) for M22\_05\_Beucher\_23-1\_A1.

<b>Atom</b>	<b>x</b>	<b>y</b>	<b>z</b>	<b>U(eq)</b>
H4A	2760(20)	6963(15)	5137(9)	24
H4B	3380(20)	7874(15)	4613(9)	24
H20A	7460(30)	4360(20)	6292(12)	48
H20B	7310(30)	5550(20)	6419(12)	48
H20C	7310(30)	4780(20)	7033(13)	48
H11A	7530(30)	6947(19)	3134(12)	44
H11B	8400(30)	7369(18)	3776(11)	44
H11C	8690(30)	6180(19)	3545(12)	44
H21A	4930(30)	6240(20)	6873(12)	51
H21B	3420(30)	5540(20)	6837(13)	51
H21C	4600(30)	5350(20)	7387(13)	51
H14A	3530(30)	4770(20)	2684(12)	47
H14B	5160(30)	5210(20)	2608(12)	47
H14C	4790(30)	4144(19)	2274(12)	47
H7A	-1380(30)	5300(20)	5065(13)	55
H7B	-1040(30)	4570(20)	4414(14)	55
H7C	-1490(30)	4060(20)	5100(13)	55
H9A	1890(30)	4540(20)	5967(13)	54
H9B	370(30)	5260(20)	6023(13)	54
H9C	360(30)	4010(20)	6068(13)	54
H16A	7250(30)	4460(20)	3336(12)	55
H16B	6930(30)	3400(20)	3725(14)	55
H16C	7000(30)	3410(20)	2924(13)	55
H19A	3720(30)	3630(20)	6682(14)	59
H19B	5350(30)	3160(20)	6376(14)	59
H19C	5140(30)	3510(20)	7166(14)	59
H15A	4280(30)	2710(20)	3822(14)	61
H15B	2940(40)	3220(20)	3329(13)	61
H15C	4370(30)	2570(20)	3019(14)	61
H8A	1300(40)	3510(20)	4361(17)	74
H8B	2490(40)	3520(20)	4949(15)	74
H8C	870(40)	2960(20)	4996(15)	74

## Experimental

Single crystals of  $C_{21}H_{32}N_2O_8$  [M22\_05\_Beucher\_23-1\_A1] were synthesized. A suitable crystal was selected and mounted on a SuperNova, Dual, Cu at home/near, Atlas diffractometer. The crystal was kept at 100.00(10) K during data collection. Using Olex2 [1], the structure was solved with the SHELXT [2] structure solution program using Intrinsic Phasing and refined with the XL [3] refinement package using Least Squares minimisation.

Dolomanov, O.V., Bourhis, L.J., Gildea, R.J., Howard, J.A.K. & Puschmann, H. (2009), *J. Appl. Cryst.* 42, 339-341.

Sheldrick, G.M. (2015). *Acta Cryst.* A71, 3-8.

Sheldrick, G.M. (2008). *Acta Cryst.* A64, 112-122.

Crystal structure determination of [M22\_05\_Beucher\_23-1\_A1]

**Crystal Data** for  $C_{21}H_{32}N_2O_8$  ( $M = 440.48$  g/mol): orthorhombic, space group  $P2_12_12_1$  (no. 19),  $a = 8.9833(2)$  Å,  $b = 13.0769(2)$  Å,  $c = 20.1678(3)$  Å,  $V = 2369.19(7)$  Å<sup>3</sup>,  $Z = 4$ ,  $T = 100.00(10)$  K,  $\mu(\text{Cu K}\alpha) = 0.791$  mm<sup>-1</sup>,  $D_{\text{calc}} = 1.235$  g/cm<sup>3</sup>, 22611 reflections measured ( $8.058^\circ \leq 2\theta \leq 153.018^\circ$ ), 4942 unique ( $R_{\text{int}} = 0.0240$ ,  $R_{\text{sigma}} = 0.0156$ ) which were used in all calculations. The final  $R_1$  was 0.0256 ( $I > 2\sigma(I)$ ) and  $wR_2$  was 0.0674 (all data).

Refinement model description

Number of restraints - 0, number of constraints - unknown.

Details:

1.Fixed Uiso

At 1.2 times of:

All C(H,H) groups

At 1.5 times of:

All C(H,H,H) groups

## 9. X-ray crystallographic data of 27 (CCDC 2350765)

### DATA COLLECTION

The crystal structure of **AT020** [ $C_{23}H_{27}BrNO_7P$ ] has been determined from single crystal X-Ray diffraction. The chosen crystal was stuck on a glass fibre and mounted on a kappa goniometer of a Bruker D8-VENTURE diffractometer equipped with a PHOTON area detector. A series of exposures ( ) were recorded see in annexe 1,

The cell parameters and the orientation matrix of the crystal were preliminary determined by using APEX Software<sup>1</sup>. Data integration and global cell refinement were performed with SAINT Software<sup>2</sup>. Intensities were corrected for Lorentz, polarisation, decay and absorption effects (SADABS Software<sup>2</sup>) and reduced to  $F_o^2$ . The program package WinGX<sup>3</sup> was used for space group determination, structure solution and refinement.

### DATA REFINEMENT

The standard space group  $P2_12_12_1$  ( $n^\circ 19$ ) was determined from systematic extinctions and relative  $F_o^2$  of equivalent reflections. The structure was solved by direct methods<sup>4</sup>. Anisotropic displacement parameters were refined for all non-hydrogen atoms. Every Hydrogen atoms were located from subsequent difference Fourier syntheses and placed with geometrical constraints (SHELXL<sup>4</sup>). The final cycle of full-matrix least-square refinement on  $F^2$  was based on 5510 observed reflections and 301 variable parameters and converged with unweighted and weighted agreement factors of:

$R1 = 0.0446$ ,  $wR2 = 0.1374$  for xxx reflections with  $I > 2\sigma I$  and  $R1 = 0.0466$ ,  $wR2 = 0.1402$  for all data.

The refinement data are summarized in annexe 1 table 2

## CRYSTALLOGRAPHIC DATA AND STRUCTURAL DESCRIPTION

### Crystallographic data

The crystal data are collected in Table 1. The full crystallographic parameters (atomic coordinates, bond length, angles and anisotropic displacements) are reported in annexe 2.

**Table 1: Crystal data**

Chemical Formula	C <sub>23</sub> H <sub>27</sub> BrNO <sub>7</sub> P
Molecular Weight / <i>g.mol</i> <sup>-1</sup>	540.3
Crystal System	Orthorhombic
Space Group	<i>P</i> 2 <sub>1</sub> 2 <sub>1</sub> 2 <sub>1</sub>
Z, Z' (asymmetric units per unit cell)	4, 1
a / Å	8.6222(2)
b / Å	13.3983(3)
c / Å	22.3429(5)
α / °	90
β / °	90
γ / °	90
V / Å <sup>3</sup>	2581.12(1)
d <sub>calc</sub> / <i>g.cm</i> <sup>-3</sup>	1.390
F(000) / e <sup>-</sup>	1112
Absorption coefficient μ (MoKα <sub>1</sub> ) / <i>mm</i> <sup>-1</sup>	3.110

### Structural description

The Asymmetric unit is composed of one molecule of C<sub>23</sub>H<sub>27</sub>BrNO<sub>7</sub>P (Figures 1 and 2). The stereogenic centres are C1 and C2, that exhibit a S and R configuration respectively.

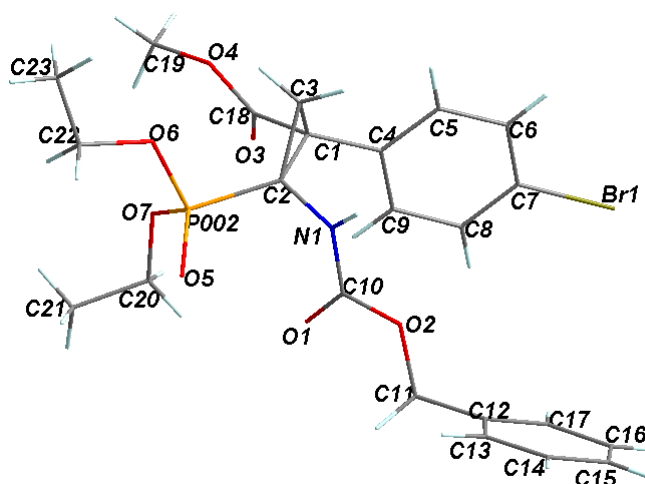


Figure 1: Asymmetric unit with atom labelled.

The stereogenic centres C1 and C2 are S and R respectively.

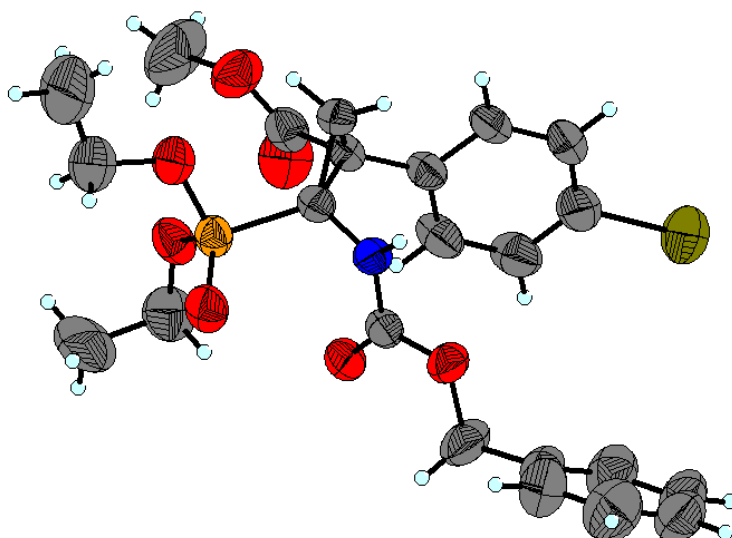


Figure 2 : asymmetric unit in thermal ellipsoidal representation (50% of probability)

Table 1: Hydrogen bond table

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1)...O(5)#1	0.86	1.98	2.795(4)	158

Symmetry transformations used to generate equivalent atoms: #1  $x-1/2, -y+3/2, -z+1$

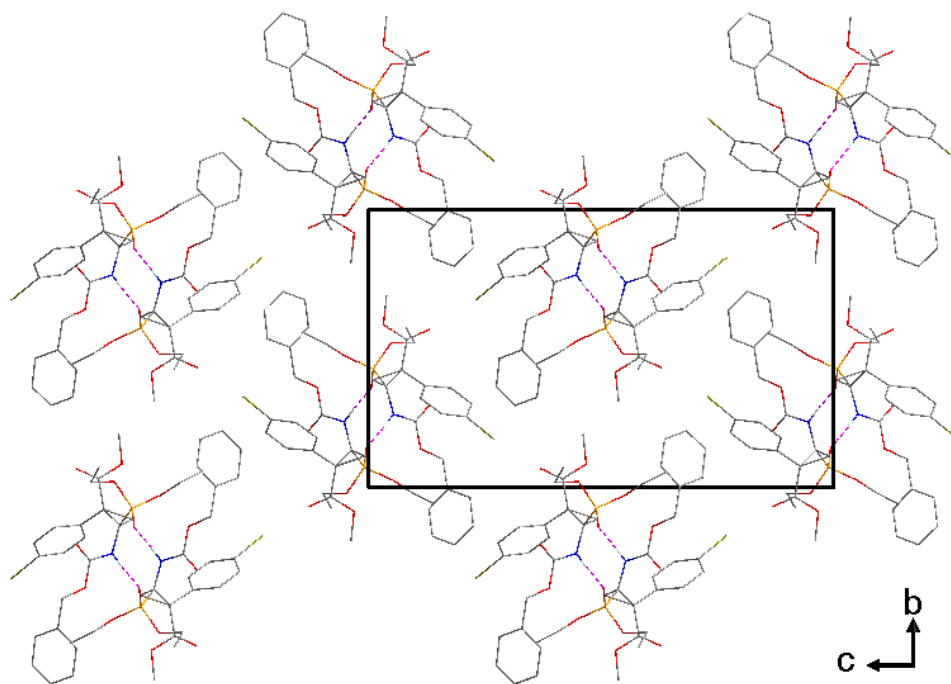


Figure 3: Projection along a axis

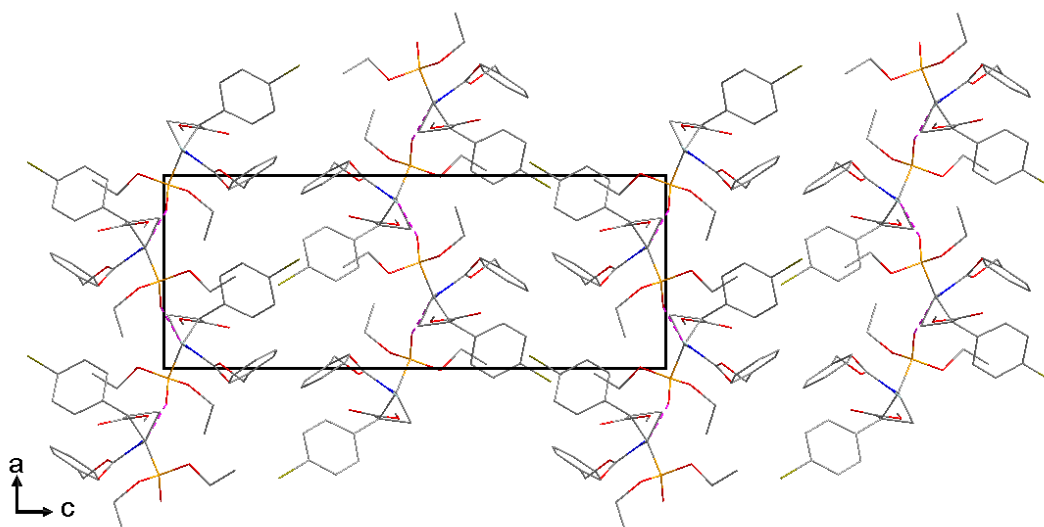


Figure 4: Projection along b axis

Sofwares :

(1)- APEX. V2022.10-1

(2)- SAINT V8.40B Bruker AXS LLC.219

- SADABS – 2016/2 – Bruker AXS area detector scaling and absorption correction

(3)- WinGX: Version 2023.1: An integrated system of Windows Programs for the solution, refinement and analysis of Single Crystal X-Ray Diffraction Data, By Louis J. Farrugia, Dept. of chemistry, University of Glasgow. L. J. Farrugia (2012) J. Appl. Cryst. 45, 849-854.

(4)-include in WinGX suite :

- SIR 92: A. Altomare, G. Cascarano, C. Giacovazzo and A. Guagliardi, J. Appl. Crystallogr., 1994, 27, 435.;

- SHELX & SHELXTL program: G. M. Sheldrick, Acta Crystallogr., Sect. A: Found. Crystallogr., 2008, 64, 112-122..

(6)-PowderCell for Windows (version 2.4) by Kraus W. & Nolze G., Federal institute for materials Research and testing, Rudower Chausse 5, 12489 Berlin Germany.

**ANNEXE 1 :**

**- Table 1 : DATA COLLECTION FOR AT020 -**

**Scan parameters : D=40.0168mm,  $\lambda = 1.54184 \text{ \AA}$**

Axis	$2\theta/^\circ$	$\omega/^\circ$	$\phi/^\circ$	$\chi/^\circ$	Width/ $^\circ$	Frames
Phi	55	0	0	54.74	1	180
Phi	55	0	180	54.74	1	180
Omega	109.52	107.37	-40	-44.5	0.5	216
Omega	109.52	117.3	160	65.5	0.5	245
Omega	-48.14	-153.55	153	44.5	0.5	216
Omega	109.52	107.37	-80	-44.5	0.5	216
Omega	109.52	107.37	0	-44.5	0.5	216
Omega	-48.14	-55.86	0	-65.5	0.5	192
Omega	109.52	117.3	-120	65.5	0.5	245
Omega	109.52	215.37	160	-44.5	0.5	216
Omega	109.52	-5.2	-80	65.5	0.5	245
Omega	109.52	117.3	-40	65.5	0.5	245
Omega	109.52	107.37	-120	-44.5	0.5	216
Omega	109.52	79.76	108	68.5	0.5	162
Omega	109.52	107.37	40	-44.5	0.5	216
Omega	109.52	117.3	120	65.5	0.5	245
Omega	109.52	-5.2	80	65.5	0.5	245
Omega	109.52	79.76	180	68.5	0.5	162
Omega	-48.14	-45.55	51	44.5	0.5	216
Phi	109.52	14.28	-58.34	22	0.5	505
Omega	109.52	107.37	120	-44.5	0.5	216
Omega	109.52	107.37	80	-44.5	0.5	216
Phi	-48.14	-39.51	-261.65	22	0.5	391
Omega	-48.14	-45.55	102	44.5	0.5	216
Omega	109.52	215.37	-160	-44.5	0.5	216
Omega	109.52	117.3	-160	65.5	0.5	245
Omega	-48.14	-45.55	0	44.5	0.5	216
Omega	109.52	-5.2	0	65.5	0.5	245

Unique reflections [ $F_o > 4.0 \sigma(F_o)$ ]	5510
$\theta$ range / $^\circ$	3.847 to 80.173
hkl range	$0 \leq h \leq 10, 0 \leq k \leq 16, -28 \leq l \leq 28$
$R_{int} = \Sigma[ F_o^2 - F_o^2(\text{mean}) ] / \Sigma[F_o^2]$	0.039
Completeness to $\theta = 26.40 / \%$	99.4

**- Table 2 : REFINEMENT DATA FOR AT020**

Number of reflections (n) (with $F_o > 4.0 \sigma(F_o)$ )	5249
Number of refined parameters (p) / restraints	301 / 0
Final R indices [ $I > 2\sigma(I)$ ]	R1 = 0.0446, wR2 = 0.1374
R indices (all data)	R1 = 0.0466, wR2 = 0.1402
Goodness of Fit indicator (Restrained GooF)	1.023
Maximum peak in Final Difference Map / $e \cdot \text{\AA}^{-3}$	0.752
Maximum hole in Final Difference Map / $e \cdot \text{\AA}^{-3}$	-0.313

$$R_1 = \frac{\sum (|F_o| - |F_c|)}{\sum |F_o|}$$

$$wR_2 = \left[ \frac{\sum [w (F_o^2 - F_c^2)^2]}{\sum [w (F_o^2)^2]} \right]^{1/2}$$

$$\text{GooF} = \left[ \frac{\sum [w (F_o^2 - F_c^2)^2]}{(n - p)} \right]^{1/2}$$



## ANNEXE 2 : CRYSTALLOGRAPHIC DATA

**Table 1a:** Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ).

U(eq) is defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor

	x	y	z	U(eq)
C(1)	2421(3)	9219(2)	5707(1)	45(1)
C(2)	3753(4)	8657(2)	5368(1)	42(1)
C(3)	2207(4)	8867(3)	5078(2)	49(1)
C(4)	1651(4)	8667(3)	6209(2)	49(1)
C(5)	234(4)	8209(3)	6118(2)	60(1)
C(6)	-461(5)	7659(4)	6567(2)	73(1)
C(7)	281(5)	7552(4)	7102(2)	70(1)
C(8)	1674(5)	8018(5)	7214(2)	73(1)
C(9)	2352(4)	8585(4)	6767(2)	64(1)
C(10)	4852(4)	7388(2)	6007(1)	45(1)
C(11)	5656(8)	6029(4)	6599(3)	91(2)
C(12)	5229(6)	4950(4)	6698(2)	78(2)
C(13)	5737(9)	4235(5)	6298(3)	97(2)
C(14)	5355(11)	3249(6)	6376(4)	115(2)
C(15)	4534(9)	2955(5)	6846(3)	97(2)
C(16)	4021(10)	3613(6)	7252(3)	108(2)
C(17)	4378(9)	4646(5)	7184(3)	95(2)
C(18)	2610(4)	10324(3)	5823(2)	57(1)
C(19)	2678(13)	11906(4)	5383(4)	128(3)
C(20)	6881(7)	10198(4)	5988(3)	84(1)
C(21)	8353(9)	10726(6)	5811(6)	136(4)
C(22)	6018(8)	10171(7)	4073(4)	114(3)
C(23)	5252(14)	10583(12)	3583(5)	187(6)
Br(1)	-652(1)	6736(1)	7700(1)	103(1)
N(1)	3970(3)	7638(2)	5531(1)	44(1)
O(1)	5661(3)	7946(2)	6293(1)	56(1)
O(2)	4716(3)	6396(2)	6113(1)	57(1)
O(3)	2872(5)	10683(3)	6298(2)	85(1)
O(4)	2424(5)	10848(2)	5316(2)	79(1)
O(5)	6826(3)	8586(2)	5053(1)	62(1)
O(6)	4922(4)	9727(3)	4487(1)	70(1)
O(7)	5818(3)	10216(2)	5485(2)	62(1)
P(002)	5498(1)	9266(1)	5094(1)	48(1)

**Table 1b:** Hydrogen coordinates ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ).U(eq) is defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor

	x	y	z	U(eq)
H(3A)	2179	9369	4765	58
H(3B)	1517	8305	5013	58
H(5)	-257	8272	5749	72
H(6)	-1425	7366	6506	88
H(8)	2153	7953	7585	88
H(9)	3283	8912	6841	76
H(11A)	5461	6414	6959	109
H(11B)	6748	6086	6500	109
H(13)	6344	4424	5973	117
H(14)	5678	2780	6095	138
H(15)	4310	2281	6894	116
H(16)	3435	3397	7577	129
H(17)	4039	5108	7466	114
H(19A)	1803	12201	5581	192
H(19B)	2802	12205	4995	192
H(19C)	3596	12017	5616	192
H(20A)	7108	9514	6098	101
H(20B)	6415	10529	6330	101
H(21A)	8897	10334	5519	205
H(21B)	8996	10814	6158	205
H(21C)	8104	11366	5644	205
H(22A)	6606	10687	4276	137
H(22B)	6741	9665	3936	137
H(23A)	4579	10091	3409	280
H(23B)	5999	10795	3291	280
H(23C)	4650	11146	3711	280
H(1)	3531	7177	5324	53

**Table 2:** Angles (°)

C(3)-C(1)-C(4)	119.6(3)	O(1)-C(10)-O(2)	124.5(3)	O(7)-C(20)-H(20A)	109.9
C(3)-C(1)-C(18)	119.0(3)	N(1)-C(10)-O(2)	109.5(3)	C(21)-C(20)-H(20A)	109.9
C(4)-C(1)-C(18)	113.7(3)	O(2)-C(11)-C(12)	107.5(4)	O(7)-C(20)-H(20B)	109.9
C(3)-C(1)-C(2)	59.0(2)	O(2)-C(11)-H(11A)	110.2	C(21)-C(20)-H(20B)	109.9
C(4)-C(1)-C(2)	116.6(3)	C(12)-C(11)-H(11A)	110.2	H(20A)-C(20)-H(20B)	108.3
C(18)-C(1)-C(2)	118.3(3)	O(2)-C(11)-H(11B)	110.2	C(20)-C(21)-H(21A)	109.5
N(1)-C(2)-C(3)	113.9(3)	C(12)-C(11)-H(11B)	110.2	C(20)-C(21)-H(21B)	109.5
N(1)-C(2)-C(1)	115.7(3)	H(11A)-C(11)-H(11B)	108.5	H(21A)-C(21)-H(21B)	109.5
C(3)-C(2)-C(1)	58.1(2)	C(17)-C(12)-C(13)	118.4(5)	C(20)-C(21)-H(21C)	109.5
N(1)-C(2)-P(002)	114.1(2)	C(17)-C(12)-C(11)	122.1(6)	H(21A)-C(21)-H(21C)	109.5
C(3)-C(2)-P(002)	120.1(2)	C(13)-C(12)-C(11)	119.5(6)	H(21B)-C(21)-H(21C)	109.5
C(1)-C(2)-P(002)	123.6(2)	C(14)-C(13)-C(12)	120.6(7)	C(23)-C(22)-O(6)	110.8(7)
C(1)-C(3)-C(2)	62.9(2)	C(14)-C(13)-H(13)	119.7	C(23)-C(22)-H(22A)	109.5
C(1)-C(3)-H(3A)	117.5	C(12)-C(13)-H(13)	119.7	O(6)-C(22)-H(22A)	109.5
C(2)-C(3)-H(3A)	117.5	C(15)-C(14)-C(13)	121.0(8)	C(23)-C(22)-H(22B)	109.5
C(1)-C(3)-H(3B)	117.5	C(15)-C(14)-H(14)	119.5	O(6)-C(22)-H(22B)	109.5
C(2)-C(3)-H(3B)	117.5	C(13)-C(14)-H(14)	119.5	H(22A)-C(22)-H(22B)	108.1
H(3A)-C(3)-H(3B)	114.5	C(14)-C(15)-C(16)	121.1(6)	C(22)-C(23)-H(23A)	109.5
C(5)-C(4)-C(9)	118.8(3)	C(14)-C(15)-H(15)	119.4	C(22)-C(23)-H(23B)	109.5
C(5)-C(4)-C(1)	120.0(3)	C(16)-C(15)-H(15)	119.4	H(23A)-C(23)-H(23B)	109.5
C(9)-C(4)-C(1)	121.2(3)	C(15)-C(16)-C(17)	119.6(7)	C(22)-C(23)-H(23C)	109.5
C(4)-C(5)-C(6)	120.8(4)	C(15)-C(16)-H(16)	120.2	H(23A)-C(23)-H(23C)	109.5
C(4)-C(5)-H(5)	119.6	C(17)-C(16)-H(16)	120.2	H(23B)-C(23)-H(23C)	109.5
C(6)-C(5)-H(5)	119.6	C(12)-C(17)-C(16)	119.2(7)	C(10)-N(1)-C(2)	120.9(3)
C(7)-C(6)-C(5)	119.3(4)	C(12)-C(17)-H(17)	120.4	C(10)-N(1)-H(1)	119.5
C(7)-C(6)-H(6)	120.3	C(16)-C(17)-H(17)	120.4	C(2)-N(1)-H(1)	119.5
C(5)-C(6)-H(6)	120.3	O(3)-C(18)-O(4)	124.5(4)	C(10)-O(2)-C(11)	114.7(3)
C(6)-C(7)-C(8)	121.3(4)	O(3)-C(18)-C(1)	124.7(4)	C(18)-O(4)-C(19)	114.1(5)
C(6)-C(7)-Br(1)	118.5(4)	O(4)-C(18)-C(1)	110.8(3)	C(22)-O(6)-P(002)	120.4(4)
C(8)-C(7)-Br(1)	120.2(4)	O(4)-C(19)-H(19A)	109.5	C(20)-O(7)-P(002)	122.0(3)
C(7)-C(8)-C(9)	119.2(4)	O(4)-C(19)-H(19B)	109.5	O(5)-P(002)-O(6)	115.99(18)
C(7)-C(8)-H(8)	120.4	H(19A)-C(19)-H(19B)	109.5	O(5)-P(002)-O(7)	113.65(17)
C(9)-C(8)-H(8)	120.4	O(4)-C(19)-H(19C)	109.5	O(6)-P(002)-O(7)	102.55(18)
C(8)-C(9)-C(4)	120.5(4)	H(19A)-C(19)-H(19C)	109.5	O(5)-P(002)-C(2)	112.83(16)
C(8)-C(9)-H(9)	119.8	H(19B)-C(19)-H(19C)	109.5	O(6)-P(002)-C(2)	101.93(16)
C(4)-C(9)-H(9)	119.8	O(7)-C(20)-C(21)	108.8(6)	O(7)-P(002)-C(2)	108.77(15)
O(1)-C(10)-N(1)	126.1(3)				

**Table 3:** Bond lengths (Å)

C(1)-C(3)	1.495(5)	C(14)-C(15)	1.326(12)
C(1)-C(4)	1.499(5)	C(14)-H(14)	0.93
C(1)-C(18)	1.512(5)	C(15)-C(16)	1.341(12)
C(1)-C(2)	1.568(4)	C(15)-H(15)	0.93
C(2)-N(1)	1.425(4)	C(16)-C(17)	1.426(9)
C(2)-C(3)	1.509(4)	C(16)-H(16)	0.93
C(2)-P(002)	1.818(3)	C(17)-H(17)	0.93
C(3)-H(3A)	0.97	C(18)-O(3)	1.188(6)
C(3)-H(3B)	0.97	C(18)-O(4)	1.342(6)
C(4)-C(5)	1.382(5)	C(19)-O(4)	1.442(7)
C(4)-C(9)	1.390(5)	C(19)-H(19A)	0.96
C(5)-C(6)	1.382(6)	C(19)-H(19B)	0.96
C(5)-H(5)	0.93	C(19)-H(19C)	0.96
C(6)-C(7)	1.364(7)	C(20)-O(7)	1.450(7)
C(6)-H(6)	0.93	C(20)-C(21)	1.505(10)
C(7)-C(8)	1.376(7)	C(20)-H(20A)	0.97
C(7)-Br(1)	1.904(4)	C(20)-H(20B)	0.97
C(8)-C(9)	1.383(7)	C(21)-H(21A)	0.96
C(8)-H(8)	0.93	C(21)-H(21B)	0.96
C(9)-H(9)	0.93	C(21)-H(21C)	0.96
C(10)-O(1)	1.206(4)	C(22)-C(23)	1.393(13)
C(10)-N(1)	1.349(4)	C(22)-O(6)	1.450(6)
C(10)-O(2)	1.355(4)	C(22)-H(22A)	0.97
C(11)-O(2)	1.442(5)	C(22)-H(22B)	0.97
C(11)-C(12)	1.508(6)	C(23)-H(23A)	0.96
C(11)-H(11A)	0.97	C(23)-H(23B)	0.96
C(11)-H(11B)	0.97	C(23)-H(23C)	0.96
C(12)-C(17)	1.372(9)	N(1)-H(1)	0.86
C(12)-C(13)	1.381(10)	O(5)-P(002)	1.467(3)
C(13)-C(14)	1.373(10)	O(6)-P(002)	1.569(3)
C(13)-H(13)	0.93	O(7)-P(002)	1.569(3)

**Table 4:** Anisotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) The anisotropic displacement factor exponent takes the form:  $-2 \pi^2 [ h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12} ]$

	U11	U22	U33	U23	U13	U12
C(1)	36(1)	48(2)	51(2)	-5(1)	1(1)	1(1)
C(2)	39(1)	46(2)	41(1)	-2(1)	-1(1)	0(1)
C(3)	45(2)	51(2)	50(2)	3(1)	-8(1)	0(1)
C(4)	36(1)	58(2)	51(2)	-1(1)	2(1)	2(1)
C(5)	38(2)	79(2)	64(2)	11(2)	-6(1)	-7(2)
C(6)	43(2)	93(3)	84(3)	24(2)	-1(2)	-13(2)
C(7)	53(2)	88(3)	69(2)	15(2)	13(2)	4(2)
C(8)	55(2)	112(4)	52(2)	5(2)	2(2)	3(2)
C(9)	43(2)	96(3)	51(2)	-7(2)	-1(1)	-7(2)
C(10)	39(1)	49(2)	46(1)	0(1)	1(1)	-4(1)
C(11)	106(4)	69(3)	97(3)	33(2)	-55(3)	-29(3)
C(12)	83(3)	72(3)	78(3)	30(2)	-41(2)	-27(2)
C(13)	102(4)	88(3)	102(4)	22(3)	6(3)	-15(3)
C(14)	129(6)	85(4)	132(6)	8(4)	0(5)	1(4)
C(15)	105(4)	75(3)	110(4)	29(3)	-32(4)	-16(3)
C(16)	121(5)	114(5)	87(4)	49(4)	-25(4)	-36(4)
C(17)	116(5)	100(4)	70(3)	19(3)	-16(3)	-25(4)
C(18)	48(2)	51(2)	72(2)	-9(2)	8(2)	3(1)
C(19)	178(8)	52(3)	155(7)	12(3)	-30(6)	-11(4)
C(20)	88(3)	67(3)	96(4)	-14(2)	-12(3)	-11(2)
C(21)	83(4)	110(5)	216(10)	11(6)	-56(6)	-33(4)
C(22)	81(4)	154(6)	108(4)	64(5)	21(3)	-7(4)
C(23)	129(7)	284(16)	149(8)	133(10)	12(6)	-42(9)
Br(1)	93(1)	128(1)	89(1)	43(1)	19(1)	-7(1)
N(1)	46(1)	42(1)	45(1)	-2(1)	-5(1)	-3(1)
O(1)	53(1)	58(1)	59(1)	-2(1)	-11(1)	-11(1)
O(2)	62(2)	51(1)	57(1)	10(1)	-16(1)	-9(1)
O(3)	109(3)	65(2)	80(2)	-26(2)	13(2)	-4(2)
O(4)	91(2)	50(2)	94(2)	5(1)	-14(2)	3(2)
O(5)	53(1)	58(1)	73(2)	-1(1)	19(1)	7(1)
O(6)	64(2)	84(2)	63(2)	19(1)	9(1)	-6(2)
O(7)	49(1)	50(1)	88(2)	-11(1)	3(1)	-5(1)
P(002)	44(1)	45(1)	54(1)	0(1)	9(1)	0(1)

**Table 5:** Torsion angles (°)

C(3)-C(1)-C(2)-N(1)	-103.2(3)	C(11)-C(12)-C(17)-C(16)	-179.7(6)
C(4)-C(1)-C(2)-N(1)	6.8(4)	C(15)-C(16)-C(17)-C(12)	0.7(10)
C(18)-C(1)-C(2)-N(1)	148.2(3)	C(3)-C(1)-C(18)-O(3)	-175.6(4)
C(4)-C(1)-C(2)-C(3)	110.0(3)	C(4)-C(1)-C(18)-O(3)	35.1(5)
C(18)-C(1)-C(2)-C(3)	-108.5(4)	C(2)-C(1)-C(18)-O(3)	-107.4(5)
C(3)-C(1)-C(2)-P(002)	107.2(3)	C(3)-C(1)-C(18)-O(4)	5.1(5)
C(4)-C(1)-C(2)-P(002)	-142.7(3)	C(4)-C(1)-C(18)-O(4)	-144.2(3)
C(18)-C(1)-C(2)-P(002)	-1.3(4)	C(2)-C(1)-C(18)-O(4)	73.3(4)
C(4)-C(1)-C(3)-C(2)	-105.1(3)	O(1)-C(10)-N(1)-C(2)	-8.7(5)
C(18)-C(1)-C(3)-C(2)	107.5(3)	O(2)-C(10)-N(1)-C(2)	172.3(3)
N(1)-C(2)-C(3)-C(1)	106.4(3)	C(3)-C(2)-N(1)-C(10)	-149.5(3)
P(002)-C(2)-C(3)-C(1)	-113.1(3)	C(1)-C(2)-N(1)-C(10)	-84.9(4)
C(3)-C(1)-C(4)-C(5)	-32.5(5)	P(002)-C(2)-N(1)-C(10)	67.6(3)
C(18)-C(1)-C(4)-C(5)	116.5(4)	O(1)-C(10)-O(2)-C(11)	-1.3(6)
C(2)-C(1)-C(4)-C(5)	-100.3(4)	N(1)-C(10)-O(2)-C(11)	177.8(4)
C(3)-C(1)-C(4)-C(9)	145.9(4)	C(12)-C(11)-O(2)-C(10)	173.8(5)
C(18)-C(1)-C(4)-C(9)	-65.0(5)	O(3)-C(18)-O(4)-C(19)	4.2(8)
C(2)-C(1)-C(4)-C(9)	78.2(4)	C(1)-C(18)-O(4)-C(19)	-176.5(6)
C(9)-C(4)-C(5)-C(6)	-1.4(6)	C(23)-C(22)-O(6)-P(002)	-176.6(9)
C(1)-C(4)-C(5)-C(6)	177.1(4)	C(21)-C(20)-O(7)-P(002)	105.4(6)
C(4)-C(5)-C(6)-C(7)	-1.4(8)	C(22)-O(6)-P(002)-O(5)	-48.3(6)
C(5)-C(6)-C(7)-C(8)	3.1(8)	C(22)-O(6)-P(002)-O(7)	76.1(6)
C(5)-C(6)-C(7)-Br(1)	-176.9(4)	C(22)-O(6)-P(002)-C(2)	-171.3(5)
C(6)-C(7)-C(8)-C(9)	-1.8(8)	C(20)-O(7)-P(002)-O(5)	-30.6(4)
Br(1)-C(7)-C(8)-C(9)	178.2(4)	C(20)-O(7)-P(002)-O(6)	-156.6(4)
C(7)-C(8)-C(9)-C(4)	-1.2(8)	C(20)-O(7)-P(002)-C(2)	96.0(4)
C(5)-C(4)-C(9)-C(8)	2.8(7)	N(1)-C(2)-P(002)-O(5)	3.3(3)
C(1)-C(4)-C(9)-C(8)	-175.7(4)	C(3)-C(2)-P(002)-O(5)	-137.1(3)
O(2)-C(11)-C(12)-C(17)	-107.1(6)	C(1)-C(2)-P(002)-O(5)	153.3(3)
O(2)-C(11)-C(12)-C(13)	74.6(7)	N(1)-C(2)-P(002)-O(6)	128.4(2)
C(17)-C(12)-C(13)-C(14)	2.1(10)	C(3)-C(2)-P(002)-O(6)	-12.0(3)
C(11)-C(12)-C(13)-C(14)	-179.6(7)	C(1)-C(2)-P(002)-O(6)	-81.6(3)
C(12)-C(13)-C(14)-C(15)	-2.2(13)	N(1)-C(2)-P(002)-O(7)	-123.8(2)
C(13)-C(14)-C(15)-C(16)	1.5(13)	C(3)-C(2)-P(002)-O(7)	95.8(3)
C(14)-C(15)-C(16)-C(17)	-0.8(11)	C(1)-C(2)-P(002)-O(7)	26.2(3)
C(13)-C(12)-C(17)-C(16)	-1.4(9)		

**Table 6:** Calculated reflections from PowderCell\*

h	k	l	2 $\theta$ /°	d/Å	I/rel.	F(hkl)
---	---	---	---------------	-----	--------	--------

h	k	l	2 $\theta$ /°	d/Å	I/rel.	F(hkl)
0	1	1	7.69	11.49	100.00	118.99
0	0	2	7.91	11.17	6.18	43.04
0	1	2	10.30	8.58	8.35	46.18
1	1	0	12.20	7.25	16.72	77.50
1	1	1	12.83	6.90	25.20	70.80
1	0	2	12.96	6.83	23.25	97.19
0	1	3	13.59	6.51	3.33	38.61
0	2	2	15.41	5.75	50.40	170.65
1	0	3	15.71	5.64	35.41	145.90
1	2	0	16.75	5.29	23.16	125.94
1	1	3	17.05	5.20	78.79	167.35
0	1	4	17.19	5.16	19.57	118.89
1	2	1	17.21	5.15	16.68	77.73
0	2	3	17.79	4.98	3.38	51.19
1	2	2	18.54	4.78	5.11	46.44
1	1	4	20.05	4.42	3.99	44.49
1	2	3	20.58	4.31	37.31	139.76
2	0	0	20.59	4.31	15.35	179.33
0	2	4	20.69	4.29	2.76	54.04
2	0	1	20.97	4.23	6.73	85.61
0	3	2	21.41	4.15	3.87	66.31
2	1	1	22.00	4.04	8.68	72.26
2	0	2	22.08	4.02	15.87	138.73
1	0	5	22.39	3.97	6.58	90.60

h	k	l	2 $\theta$ /°	d/Å	I/rel.	F(hkl)
1	3	0	22.40	3.97	10.72	115.68
1	3	1	22.76	3.90	15.73	100.74
2	1	2	23.07	3.85	36.34	155.35
1	2	4	23.14	3.84	40.74	164.98
0	3	3	23.20	3.83	9.21	111.24
0	2	5	23.92	3.72	5.59	89.48
2	2	0	24.54	3.63	4.01	77.87
2	1	3	24.75	3.59	6.57	71.09
0	1	6	24.80	3.59	4.28	81.35
2	2	1	24.86	3.58	6.87	73.03
1	3	3	25.43	3.50	5.31	65.78
0	3	4	25.52	3.49	2.03	57.72
2	2	2	25.82	3.45	5.39	67.32
1	0	6	26.04	3.42	6.09	102.14
2	2	3	27.34	3.26	8.71	90.90
1	3	4	27.56	3.23	3.57	58.70
0	3	5	28.23	3.16	5.11	101.88
0	1	7	28.73	3.10	5.18	104.54
2	3	1	29.04	3.07	9.62	101.88
0	4	3	29.21	3.05	7.07	124.30
1	2	6	29.31	3.05	3.54	62.41
2	2	4	29.35	3.04	5.25	76.14
2	1	5	29.53	3.02	10.50	108.35
1	4	2	29.69	3.01	13.08	121.63

Source: Cu-K $\alpha_1$  ( $\lambda = 1.540598 \text{ \AA}$ )

Condition on reflections:  $l \geq 2$

Range (2 $\theta$ ): From 3° to 30°

\*PowderCell for Windows (version 2.4) by Kraus W. & Nolze G., Federal institute for materials Research and testing, Rudower Chausse 5, 12489 Berlin Germany.

## 10. X-ray crystallographic data of 28 (CCDC 2350766)

### DATA COLLECTION

The crystal structure of **AT018** [ $\text{C}_{24}\text{H}_{29}\text{BrNO}_7\text{P}$ ] has been determined from single crystal X-Ray diffraction. The chosen crystal was stuck on a glass fibre and mounted on a kappa goniometer of a Bruker D8-VENTURE diffractometer equipped with a PHOTON area detector. A series of exposures were recorded see in annexe 1,

The cell parameters and the orientation matrix of the crystal were preliminary determined by using APEX Software<sup>1</sup>. Data integration and global cell refinement were performed with SAINT Software<sup>2</sup>. Intensities were corrected for Lorentz, polarisation, decay and absorption effects (SADABS Software<sup>2</sup>) and reduced to  $F_o^2$ . The program package WinGX<sup>3</sup> was used for space group determination, structure solution and refinement.

### DATA REFINEMENT

The standard space group  $P2_12_12_1$  ( $n^\circ 19$ ) was determined from systematic extinctions and relative  $F_o^2$  of equivalent reflections. The structure was solved by direct methods<sup>4</sup>. Anisotropic displacement parameters were refined for all non-hydrogen atoms. Every Hydrogen atoms were located from subsequent difference Fourier syntheses and placed with geometrical constraints (SHELXL<sup>4</sup>). The final cycle of full-matrix least-square refinement on  $F^2$  was based on 5848 observed reflections and 330 variable parameters and converged with unweighted and weighted agreement factors of:

$R1 = 0.0509$ ,  $wR2 = 0.1496$  for 5023 reflections with  $|I > 2\sigma|$  and  $R1 = 0.0571$ ,  $wR2 = 0.1575$  for all data.

The refinement data are summarized in annexe 1 table 2



## CRYSTALLOGRAPHIC DATA AND STRUCTURAL DESCRIPTION

### *Crystallographic data*

The crystal data are collected in Table 1. The full crystallographic parameters (atomic coordinates, bond length, angles and anisotropic displacements) are reported in annexe 2.

**Table 1: Crystal data**

Chemical Formula	C <sub>24</sub> H <sub>29</sub> BrNO <sub>7</sub> P
Molecular Weight / <i>g.mol</i> <sup>-1</sup>	554.3
Crystal System	Orthorhombic
Space Group	<i>P</i> 2 <sub>1</sub> 2 <sub>1</sub> 2 <sub>1</sub>
Z , Z' (asymmetric units per unit cell)	4,1
a / Å	8.6898(2)
b / Å	14.0318(4)
c / Å	22.1254(6)
α / °	90
β / °	90
γ / °	90
V / Å <sup>3</sup>	2697.83(1)
d <sub>calc</sub> / <i>g.cm</i> <sup>-3</sup>	1.365
F(000) / e <sup>-</sup>	1144
Absolute structure parameter	0.044(9)
Absorption coefficient μ (MoKα <sub>1</sub> ) / <i>mm</i> <sup>-1</sup>	2.989

### *Structural description*

The Asymmetric unit is composed of one molecule of C<sub>24</sub>H<sub>29</sub>BrNO<sub>7</sub>P (Figures 1 and 2). One of the ethyl chain is disordered (light/medium grey on figures 1 and 2) with a statistical occupancy factor of 63/37% (dark/light grey). The stereo genic centres are C1 and C3 , that exhibit a S and R configuration respectively.

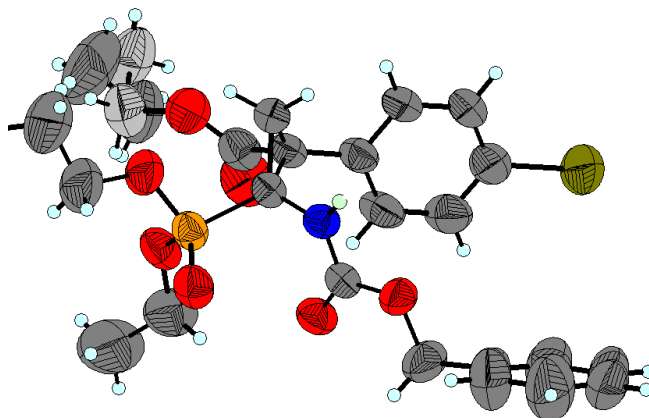


Figure 1: Asymmetric unit in thermal ellipsoidal representation (50% of probability).  
One of the ethyl chain is disordered with 37/63 % of s.o.f. (light/medium grey)

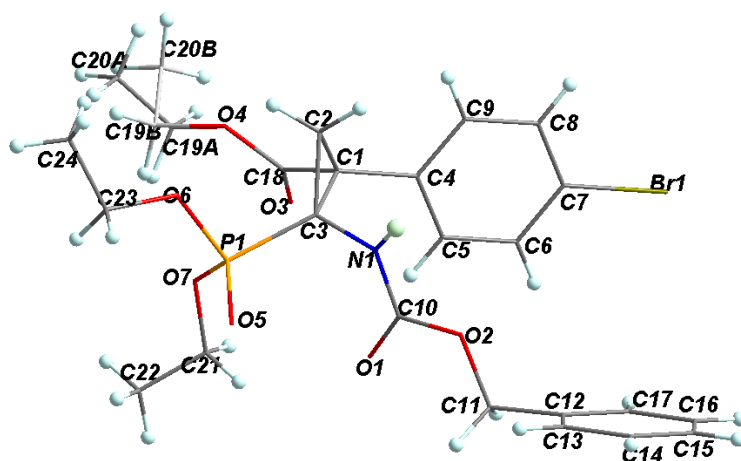


Figure 2: Asymmetric unit with atom labels.  
The Stereogenic centres C1 and C3 are S and R respectively.

Table 1: Hydrogen bond table

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)- H(1)...O(5)#1	0.86	1.99	2.804(4)	157.3

Symmetry transformations used to generate equivalent atoms: #1  $x+1/2, -y+1/2, -z+1$

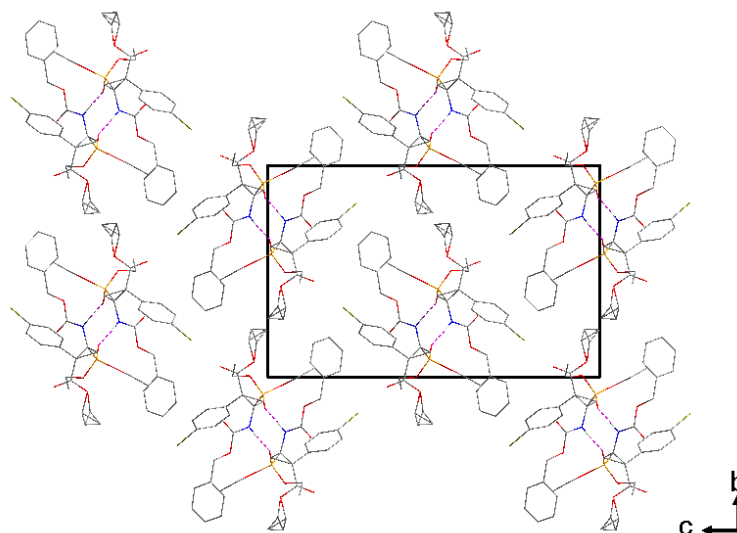


Figure 3 : Projection along a axis

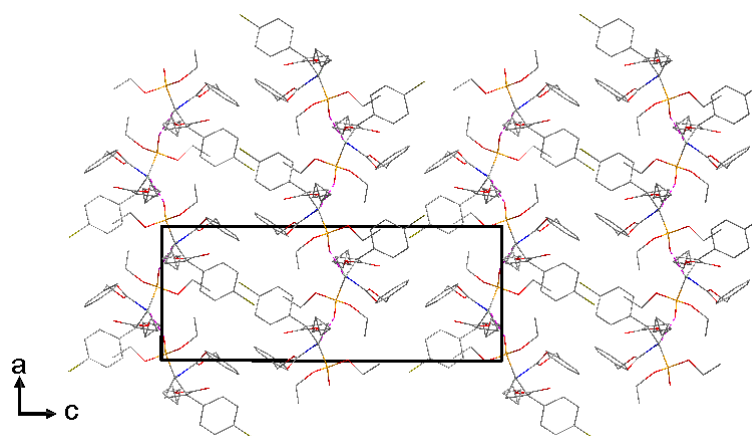


Figure 4: Projection along b axis

Softwares :

(1)- APEX. V2022.10-1

(2)- SAINT V8.40B Bruker AXS LLC.219

- SADABS – 2016/2 – Bruker AXS area detector scaling and absorption correction

(3)- WinGX: Version 2023.1: An integrated system of Windows Programs for the solution, refinement and analysis of Single Crystal X-Ray Diffraction Data, By LouisJ. Farrugia, Dept. of chemistry, University of Glasgow. L. J. Farrugia (2012) J. Appl. Cryst. 45, 849-854.

(4)-include in WinGX suite :

- SIR 92: A. Altomare, G. Cascarano, C. Giacovazzo and A. Guagliardi, J. Appl. Crystallogr., 1994, 27, 435.;

- SHELX & SHELXTL program: G. M. Sheldrick, Acta Crystallogr., Sect. A: Found. Crystallogr., 2008, 64, 112-122..

(6)-PowderCell for Windows (version 2.4) by Kraus W. & Nolze G., Federal institute for materials Research and testing, Rudower Chausse 5, 12489 Berlin Germany.

**ANNEXE 1 :**

**- Table 1 : DATA COLLECTION FOR AT018 -**

**Scan parameters : D=40.068mm,  $\lambda = 1.54184 \text{ \AA}$**

Axis	2 $\theta$ /°	$\omega$ /°	$\phi$ /°	$\chi$ /°	Width/°	Frames	time
Phi	55	0	0	54.74	1	180	1
Phi	55	0	180	54.74	1	180	1
Omega	-48.14	- 153.55	51	44.5	0.5	216	1
Omega	109.52	107.37	120	-44.5	0.5	216	5
Omega	109.52	-5.2	160	65.5	0.5	245	5
Omega	-48.14	40.14	0	-65.5	0.5	192	1
Omega	109.52	- 144.63	40	-44.5	0.5	216	5
Omega	109.52	117.3	40	65.5	0.5	245	5
Omega	109.52	107.37	0	-44.5	0.5	216	5
Omega	109.52	107.37	-80	-44.5	0.5	216	5
Omega	109.52	117.3	-40	65.5	0.5	245	5
Omega	-48.14	- 153.55	-54	44.5	0.5	216	1
Omega	109.52	117.3	0	65.5	0.5	245	5
Omega	109.52	107.37	-120	-44.5	0.5	216	5
Omega	109.52	- 144.63	-40	-44.5	0.5	216	5
Omega	-48.14	- 153.55	0	44.5	0.5	216	1
Omega	109.52	117.3	-80	65.5	0.5	245	5
Omega	109.52	-1.24	324	68.5	0.5	162	5
Omega	109.52	-1.24	180	68.5	0.5	162	5
Omega	109.52	-1.24	108	68.5	0.5	162	5
Phi	109.52	14.28	-58.34	22	0.5	490	5
Omega	109.52	107.37	160	-44.5	0.5	216	5
Omega	109.52	- 144.63	80	-44.5	0.5	216	5
Omega	109.52	117.3	120	65.5	0.5	245	5
Omega	109.52	-5.2	-160	65.5	0.5	245	5
Phi	-48.14	-45.55	117.85	44.5	0.5	391	1
Phi	-48.14	-39.51	- 173.65	22	0.5	391	1

Unique reflections [ $F_o > 4.0 \sigma(F_o)$ ]	5848
$\theta$ range / °	3.730 to 81.236
hkl range	-10<=h<=8, -17<=k<=17, - 28<=l<=28
$R_{int} = \Sigma[ F_o^2 - F_o^2(\text{mean}) ] / \Sigma[F_o^2]$	0.058
Completeness to $\theta = 67.684$ / %	99.9

**- Table 2 : REFINEMENT DATA FOR AT018**

Number of reflections (n) (with $F_o > 4.0 \sigma(F_o)$ )	5023
Number of refined parameters (p) / restraints	330 / 0
Final R indices [ $I > 2\sigma(I)$ ]	R1 = 0.0509, wR2 = 0.1496
R indices (all data)	R1 = 0.0571, wR2 = 0.1575
Goodness of Fit indicator (Restrained GooF)	0.991
Maximum peak in Final Difference Map / $e\text{\AA}^{-3}$	0.647
Maximum hole in Final Difference Map / $e\text{\AA}^{-3}$	-0.458

$$R_1 = \Sigma (||F_o| - |F_c||) / \Sigma |F_o|$$

$$wR_2 = [ \Sigma [ w (F_o^2 - F_c^2)^2 ] / \Sigma [ w (F_o^2)^2 ] ]^{1/2}$$

$$\text{GooF} = [ \Sigma [ w (F_o^2 - F_c^2)^2 ] / (n - p) ]^{1/2}$$

## ANNEXE 2 : CRYSTALLOGRAPHIC DATA

Table 1a: Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ).

U(eq) is defined as one third of the trace of the orthogonalized Uij tensor

	x	y	z	U(eq)
C(1)	7559(4)	866(3)	5749(2)	59(1)
C(2)	7772(4)	1164(3)	5106(2)	62(1)
C(3)	6254(4)	1400(3)	5395(2)	55(1)
C(4)	8359(4)	1419(3)	6241(2)	63(1)
C(5)	7691(5)	1534(5)	6806(2)	78(1)
C(6)	8428(6)	2071(5)	7246(2)	89(2)
C(7)	9819(6)	2480(5)	7120(3)	86(1)
C(8)	10521(5)	2339(5)	6575(3)	87(1)
C(9)	9784(5)	1814(4)	6136(2)	74(1)
C(10)	5196(4)	2639(3)	6019(2)	56(1)
C(11)	4392(9)	3962(4)	6597(3)	102(2)
C(12)	4819(8)	4978(4)	6698(3)	90(2)
C(13)	4286(12)	5662(6)	6292(5)	126(3)
C(14)	4629(15)	6608(7)	6389(6)	152(4)
C(15)	5478(12)	6870(7)	6865(5)	131(3)
C(16)	6021(13)	6236(8)	7264(5)	140(4)
C(17)	5694(13)	5268(6)	7183(4)	123(3)
C(18)	7315(6)	-169(3)	5898(3)	76(1)
C(19A)	7160(30)	- 1719(14)	5559(13)	120(7)
C(20A)	7400(50)	- 2223(11)	5027(15)	200(18)
C(19B)	6910(40)	- 1660(30)	5320(18)	102(9)
C(20B)	8250(40)	- 2272(16)	5475(15)	124(11)
C(22)	1830(20)	- 587(10)	5970(12)	270(14)
C(21)	3133(10)	-11(5)	6050(4)	110(2)
C(23)	3961(10)	27(7)	4088(4)	125(3)
C(24)	4781(17)	- 413(11)	3582(5)	177(6)
Br(1)	10819(1)	3263(1)	7706(1)	118(1)
N(1)	6054(4)	2383(2)	5545(1)	56(1)
O(1)	4389(4)	2119(2)	6318(1)	68(1)
O(2)	5322(4)	3593(2)	6110(2)	69(1)

O(3)	7053(7)	-471(3)	6390(2)	105(1)
O(4)	7433(5)	-709(3)	5400(2)	96(1)
O(5)	3209(4)	1494(2)	5074(2)	76(1)
O(6)	5058(5)	364(3)	4520(2)	88(1)
O(7)	4149(4)	-55(2)	5532(2)	77(1)
P(1)	4503(1)	831(1)	5127(1)	62(1)



**Table 1b:** Hydrogen coordinates ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ). U(eq) is defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor

	x	y	z	U(eq)
H(2A)	7780	666	4802	74
H(2B)	8473	1687	5027	74
H(5)	6748	1250	6890	94
H(6)	7978	2153	7624	106
H(8)	11490	2595	6501	105
H(9)	10256	1724	5763	89
H(11A)	4569	3596	6962	123
H(11B)	3310	3915	6493	123
H(13)	3703	5481	5958	151
H(14)	4268	7067	6122	182
H(15)	5697	7512	6921	158
H(16)	6610	6436	7591	168
H(17)	6068	4821	7456	147
H(19A)	7866	-1926	5871	143
H(19B)	6113	-1810	5702	143
H(20A)	8429	-2104	4884	299
H(20B)	6675	-2022	4728	299
H(20C)	7279	-2893	5103	299
H(19C)	6586	-1762	4906	123
H(19D)	6045	-1789	5586	123
H(20D)	9051	-2180	5182	186
H(20E)	7939	-2929	5475	186
H(20F)	8628	-2105	5869	186
H(22A)	1262	-370	5625	405
H(22B)	1196	-552	6324	405
H(22C)	2150	-1235	5907	405
H(21A)	3686	-216	6407	132
H(21B)	2809	643	6113	132
H(23A)	3339	554	3943	150
H(23B)	3284	-436	4276	150
H(24A)	5723	-690	3726	265
H(24B)	5009	63	3284	265
H(24C)	4149	-900	3406	265
H(1)	6490	2814	5327	67

**Table 2: Bond lengths (Å)**

C(1)-C(2)	1.494(6)	C(16)-H(16)	0.93
C(1)-C(18)	1.504(6)	C(17)-H(17)	0.93
C(1)-C(4)	1.507(6)	C(18)-O(3)	1.190(8)
C(1)-C(3)	1.568(5)	C(18)-O(4)	1.341(8)
C(2)-C(3)	1.503(5)	C(19A)- C(20A)	1.39(4)
C(2)-H(2A)	0.97	C(19A)-O(4)	1.48(2)
C(2)-H(2B)	0.97	C(19A)- H(19A)	0.97
C(3)-N(1)	1.430(5)	C(19A)- H(19B)	0.97
C(3)-P(1)	1.818(4)	C(20A)- H(20A)	0.9893
C(4)-C(9)	1.376(6)	C(20A)- H(20B)	0.9905
C(4)-C(5)	1.388(6)	C(20A)- H(20C)	0.9905
C(5)-C(6)	1.388(8)	C(19B)-O(4)	1.42(4)
C(5)-H(5)	0.93	C(19B)- C(20B)	1.49(5)
C(6)-C(7)	1.366(9)	C(19B)- H(19C)	0.97
C(6)-H(6)	0.93	C(19B)- H(19D)	0.97
C(7)-C(8)	1.367(8)	C(20B)- H(20D)	0.96
C(7)-Br(1)	1.908(5)	C(20B)- H(20E)	0.96
C(8)-C(9)	1.377(7)	C(20B)- H(20F)	0.96
C(8)-H(8)	0.93	C(22)-C(21)	1.400(17)
C(9)-H(9)	0.93	C(22)- H(22A)	0.9601
C(10)-O(1)	1.210(5)	C(22)- H(22B)	0.9601
C(10)-N(1)	1.336(5)	C(22)- H(22C)	0.9601
C(10)-O(2)	1.359(5)	C(21)-O(7)	1.448(9)
C(11)-O(2)	1.443(6)	C(21)- H(21A)	0.97
C(11)-C(12)	1.491(7)	C(21)- H(21B)	0.97
C(11)- H(11A)	0.97	C(23)-O(6)	1.431(8)
C(11)- H(11B)	0.97	C(23)-C(24)	1.463(15)
C(12)-C(17)	1.376(11)	C(23)- H(23A)	0.97
C(12)-C(13)	1.394(12)	C(23)- H(23B)	0.97
C(13)-C(14)	1.377(12)	C(24)- H(24A)	0.96
C(13)-H(13)	0.93	C(24)- H(24B)	0.96
C(14)-C(15)	1.337(16)	C(24)- H(24C)	0.96
C(14)-H(14)	0.93	N(1)-H(1)	0.86
C(15)-C(16)	1.339(16)	O(5)-P(1)	1.463(3)
C(15)-H(15)	0.93	O(6)-P(1)	1.569(4)
C(16)-C(17)	1.398(13)	O(7)-P(1)	1.563(3)

**Table 3: Angles (°)**

C(2)-C(1)-C(18)	4	119.7(4)	C(9)-C(8)-H(8)	120.2	O(3)-C(18)-C(1)	5	124.8(5)	C(22)-C(21)-O(7)	111.6(10)
C(2)-C(1)-C(4)	3	119.1(3)	C(4)-C(9)-C(8)	121.0(5)	O(4)-C(18)-C(1)	4	110.8(4)	C(22)-C(21)-H(21A)	109.3
C(18)-C(1)-C(4)	4	113.8(4)	C(4)-C(9)-H(9)	119.5	C(20A)-C(19A)-O(4)		105(2)	O(7)-C(21)-H(21A)	109.3
C(2)-C(1)-C(3)		58.7(3)	C(8)-C(9)-H(9)	119.5	C(20A)-C(19A)-H(19A)		110.9	C(22)-C(21)-H(21B)	109.3
C(18)-C(1)-C(3)	3	117.9(3)	O(1)-C(10)-N(1)	126.3(4)	O(4)-C(19A)-H(19A)		110.7	O(7)-C(21)-H(21B)	109.3
C(4)-C(1)-C(3)	3	116.6(3)	O(1)-C(10)-O(2)	124.0(4)	C(20A)-C(19A)-H(19B)		110.5	H(21A)-C(21)-H(21B)	107.9
C(1)-C(2)-C(3)		63.1(3)	N(1)-C(10)-O(2)	109.7(3)	O(4)-C(19A)-H(19B)		110.7	O(6)-C(23)-C(24)	109.0(8)
C(1)-C(2)-H(2A)		117.4	O(2)-C(11)-C(12)	108.4(4)	H(19A)-C(19A)-H(19B)		108.8	O(6)-C(23)-H(23A)	109.8
C(3)-C(2)-H(2A)		117.4	O(2)-C(11)-H(11A)	110	C(19A)-C(20A)-H(20A)		112.5	C(24)-C(23)-H(23A)	109.9
C(1)-C(2)-H(2B)		117.4	C(12)-C(11)-H(11A)	110	C(19A)-C(20A)-H(20B)		112.9	O(6)-C(23)-H(23B)	109.9
C(3)-C(2)-H(2B)		117.4	O(2)-C(11)-H(11B)	110	H(20A)-C(20A)-H(20B)		106.1	C(24)-C(23)-H(23B)	109.9
H(2A)-C(2)-H(2B)		114.5	C(12)-C(11)-H(11B)	110	C(19A)-C(20A)-H(20C)		112.9	H(23A)-C(23)-H(23B)	108.3
N(1)-C(3)-C(2)	3	114.7(3)	H(11A)-C(11)-H(11B)	108.4	H(20A)-C(20A)-H(20C)		105.9	C(23)-C(24)-H(24A)	109.5
N(1)-C(3)-C(1)	3	115.7(3)	C(17)-C(12)-C(13)	118.9(7)	H(20B)-C(20A)-H(20C)		105.9	C(23)-C(24)-H(24B)	109.4
C(2)-C(3)-C(1)		58.2(3)	C(17)-C(12)-C(11)	122.5(8)	O(4)-C(19B)-C(20B)		105(2)	H(24A)-C(24)-H(24B)	109.5
N(1)-C(3)-P(1)	3	113.4(3)	C(13)-C(12)-C(11)	118.6(7)	O(4)-C(19B)-H(19C)		110.8	C(23)-C(24)-H(24C)	109.5
C(2)-C(3)-P(1)	3	119.9(3)	C(14)-C(13)-C(12)	119.4(9)	C(20B)-C(19B)-H(19C)		110.8	H(24A)-C(24)-H(24C)	109.5
C(1)-C(3)-P(1)	3	124.0(3)	C(14)-C(13)-H(13)	120.3	O(4)-C(19B)-H(19D)		110.7	H(24B)-C(24)-H(24C)	109.5
C(9)-C(4)-C(5)	4	118.8(4)	C(12)-C(13)-H(13)	120.3	C(20B)-C(19B)-H(19D)		110.6	C(10)-N(1)-C(3)	120.6(3)
C(9)-C(4)-C(1)	4	120.0(4)	C(15)-C(14)-C(13)	120.5(1)	H(19C)-C(19B)-H(19D)		108.8	C(10)-N(1)-H(1)	119.7
C(5)-C(4)-C(1)	4	121.1(4)	C(15)-C(14)-H(14)	119.8	C(19B)-C(20B)-H(20D)		109.4	C(3)-N(1)-H(1)	119.7
C(6)-C(5)-C(4)	4	120.1(4)	C(13)-C(14)-H(14)	119.7	C(19B)-C(20B)-H(20E)		109.5	C(10)-O(2)-C(11)	114.8(3)
C(6)-C(5)-H(5)		120	C(14)-C(15)-C(16)	122.0(8)	H(20D)-C(20B)-H(20E)		109.5	C(18)-O(4)-C(19B)	127.4(18)
C(4)-C(5)-H(5)		119.9	C(14)-C(15)-H(15)	119	C(19B)-C(20B)-H(20F)		109.6	C(18)-O(4)-C(19A)	109.5(12)
C(7)-C(6)-C(5)	5	119.6(5)	C(16)-C(15)-H(15)	119	H(20D)-C(20B)-H(20F)		109.5	C(23)-O(6)-P(1)	120.3(5)
C(7)-C(6)-H(6)		120.2	C(15)-C(16)-C(17)	119.3(9)	H(20E)-C(20B)-H(20F)		109.5	C(21)-O(7)-P(1)	122.7(4)
C(5)-C(6)-H(6)		120.2	C(15)-C(16)-H(16)	120.4	C(21)-C(22)-H(22A)		109.5	O(5)-P(1)-O(7)	113.6(2)
C(6)-C(7)-C(8)	5	120.9(5)	C(17)-C(16)-H(16)	120.3	C(21)-C(22)-H(22B)		109.4	O(5)-P(1)-O(6)	115.7(2)
C(6)-C(7)-Br(1)	4	120.5(4)	C(12)-C(17)-C(16)	120.0(10)	H(22A)-C(22)-H(22B)		109.5	O(7)-P(1)-O(6)	102.6(2)
C(8)-C(7)-Br(1)	4	118.6(4)	C(12)-C(17)-H(17)	120	C(21)-C(22)-H(22C)		109.5	O(5)-P(1)-C(3)	112.98(1)
C(7)-C(8)-C(9)	5	119.5(5)	C(16)-C(17)-H(17)	120	H(22A)-C(22)-H(22C)		109.5	O(7)-P(1)-C(3)	109.02(1)
C(7)-C(8)-H(8)		120.3	O(3)-C(18)-O(4)	124.4(5)	H(22B)-C(22)-H(22C)		109.5	O(6)-P(1)-C(3)	101.87(1)

**Table 4:** Anisotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) The anisotropic displacement factor exponent takes the form:  $-2 \pi^2 [h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12}]$

	U11	U22	U33	U23	U13	U12	
C(1)	44(2)	62(2)	71(2)	3(2)	0(1)	1(1)	
C(2)	54(2)	64(2)	68(2)	-5(2)	5(2)	-2(1)	
C(3)	50(2)	56(2)	59(2)	3(1)	-2(1)	-2(1)	
C(4)	45(2)	74(2)	68(2)	1(2)	-4(1)	2(2)	
C(5)	49(2)	116(4)	69(2)	7(2)	0(2)	-4(2)	
C(6)	67(2)	132(5)	67(2)	-6(3)	-2(2)	8(3)	
C(7)	62(2)	108(4)	87(3)	-	18(3)	-12(2)	4(2)
C(8)	51(2)	109(4)	102(3)	-	28(3)	-1(2)	-12(2)
C(9)	48(2)	91(3)	83(3)	-	11(2)	5(2)	-4(2)
C(10)	47(2)	60(2)	60(2)	2(1)	-1(1)	-5(1)	
C(11)	117(4)	77(3)	113(4)	-	24(3)	56(4)	-24(3)
C(12)	98(4)	78(3)	93(3)	-	25(3)	32(3)	-21(3)
C(13)	139(6)	94(4)	144(7)	-	13(4)	-30(5)	-25(4)
C(14)	171(10)	85(5)	199(11)	-	12(6)	-32(9)	-8(5)
C(15)	132(6)	106(5)	156(8)	-	57(6)	36(6)	-30(5)
C(16)	151(8)	157(9)	112(5)	-	68(6)	27(6)	-53(7)
C(17)	158(7)	119(5)	91(4)	-	23(4)	19(5)	-30(5)
C(18)	62(2)	66(2)	100(3)	12(2)	-12(2)	5(2)	
C(19A)	167(17)	54(6)	138(16)	2(9)	-19(12)	-3(8)	
C(20A)	320(40)	78(8)	200(30)	-	36(12)	50(30)	12(14)
C(19B)	101(15)	86(16)	120(20)	0(16)	-18(14)	15(11)	
C(20B)	160(20)	78(11)	130(20)	-	12(11)	-39(16)	33(12)
C(22)	225(16)	140(9)	450(30)	-	98(15)	200(20)	99(11)
C(21)	113(5)	86(4)	131(6)	20(4)	30(4)	-6(3)	
C(23)	108(5)	139(6)	128(6)	-	50(5)	-34(5)	5(5)
C(24)	167(10)	234(14)	130(7)	-	95(9)	-21(7)	17(10)

Br(1)	95(1)	151(1)	109(1)	- 47(1)	-23(1)	0(1)
N(1)	54(1)	54(2)	60(1)	5(1)	5(1)	-2(1)
O(1)	60(1)	69(2)	76(2)	7(1)	11(1)	-8(1)
O(2)	75(2)	60(1)	73(2)	-9(1)	20(1)	-10(1)
O(3)	121(3)	82(2)	112(3)	37(2)	-12(3)	-1(2)
O(4)	93(2)	59(2)	135(3)	-5(2)	8(2)	2(2)
O(5)	64(2)	69(2)	94(2)	0(2)	-22(2)	10(1)
O(6)	81(2)	96(2)	85(2)	- 26(2)	-18(2)	2(2)
O(7)	59(2)	60(2)	112(2)	10(2)	-3(2)	-7(1)
P(1)	54(1)	58(1)	75(1)	0(1)	-12(1)	1(1)

**Table 5:** Torsion angles (°)

C(18)-C(1)-C(2)-C(3)	106.5(4)	C(5)-C(4)-C(9)-C(8)	-2.0(8)	N(1)-C(10)-O(2)-C(11)	177.5(5)
C(4)-C(1)-C(2)-C(3)	105.2(4)	C(1)-C(4)-C(9)-C(8)	178.6(5)	C(12)-C(11)-O(2)-C(10)	172.0(5)
C(1)-C(2)-C(3)-N(1)	106.0(4)	C(7)-C(8)-C(9)-C(4)	-0.8(9)	O(3)-C(18)-O(4)- C(19B)	17(2)
C(1)-C(2)-C(3)-P(1)	113.7(3)	O(2)-C(11)-C(12)-C(17)	105.1(8)	C(1)-C(18)-O(4)- C(19B)	-163(2)
C(2)-C(1)-C(3)-N(1)	104.3(4)	O(2)-C(11)-C(12)-C(13)	76.2(9)	O(3)-C(18)-O(4)- C(19A)	1.1(14)
C(18)-C(1)-C(3)-N(1)	146.1(4)	C(17)-C(12)-C(13)- C(14)	-0.8(14)	C(1)-C(18)-O(4)- C(19A)	-178.9(12)
C(4)-C(1)-C(3)-N(1)	5.1(5)	C(11)-C(12)-C(13)- C(14)	178.0(9)	C(20B)-C(19B)-O(4)- C(18)	-93(3)
C(18)-C(1)-C(3)-C(2)	109.6(5)	C(12)-C(13)-C(14)- C(15)	0.6(19)	C(20A)-C(19A)-O(4)- C(18)	-176(2)
C(4)-C(1)-C(3)-C(2)	109.4(4)	C(13)-C(14)-C(15)- C(16)	-0.1(19)	C(24)-C(23)-O(6)-P(1)	-177.3(9)
C(2)-C(1)-C(3)-P(1)	106.8(3)	C(14)-C(15)-C(16)- C(17)	-0.1(17)	C(22)-C(21)-O(7)-P(1)	116.3(12)
C(18)-C(1)-C(3)-P(1)	-2.8(5)	C(13)-C(12)-C(17)- C(16)	0.6(13)	C(21)-O(7)-P(1)-O(5)	-32.8(6)
C(4)-C(1)-C(3)-P(1)	143.8(3)	C(11)-C(12)-C(17)- C(16)	178.1(7)	C(21)-O(7)-P(1)-O(6)	-158.4(5)
C(2)-C(1)-C(4)-C(9)	-33.8(6)	C(15)-C(16)-C(17)- C(12)	-0.2(15)	C(21)-O(7)-P(1)-C(3)	94.1(5)
C(18)-C(1)-C(4)-C(9)	116.2(5)	C(2)-C(1)-C(18)-O(3)	175.7(5)	C(23)-O(6)-P(1)-O(5)	-42.5(7)
C(3)-C(1)-C(4)-C(9)	101.2(5)	C(4)-C(1)-C(18)-O(3)	34.4(7)	C(23)-O(6)-P(1)-O(7)	81.7(7)
C(2)-C(1)-C(4)-C(5)	146.8(4)	C(3)-C(1)-C(18)-O(3)	107.7(6)	C(23)-O(6)-P(1)-C(3)	-165.4(6)
C(18)-C(1)-C(4)-C(5)	-63.2(5)	C(2)-C(1)-C(18)-O(4)	4.2(5)	N(1)-C(3)-P(1)-O(5)	3.5(3)
C(3)-C(1)-C(4)-C(5)	79.4(5)	C(4)-C(1)-C(18)-O(4)	145.6(4)	C(2)-C(3)-P(1)-O(5)	-137.2(3)
C(9)-C(4)-C(5)-C(6)	2.6(8)	C(3)-C(1)-C(18)-O(4)	72.3(5)	C(1)-C(3)-P(1)-O(5)	153.1(3)
C(1)-C(4)-C(5)-C(6)	178.0(5)	O(1)-C(10)-N(1)-C(3)	-8.7(6)	N(1)-C(3)-P(1)-O(7)	-123.7(3)
C(4)-C(5)-C(6)-C(7)	-0.5(9)	O(2)-C(10)-N(1)-C(3)	173.0(3)	C(2)-C(3)-P(1)-O(7)	95.5(3)
C(5)-C(6)-C(7)-C(8)	-2.4(10)	C(2)-C(3)-N(1)-C(10)	149.1(3)	C(1)-C(3)-P(1)-O(7)	25.8(4)
C(5)-C(6)-C(7)-Br(1)	177.6(5)	C(1)-C(3)-N(1)-C(10)	-84.1(4)	N(1)-C(3)-P(1)-O(6)	128.3(3)
C(6)-C(7)-C(8)-C(9)	3.0(10)	P(1)-C(3)-N(1)-C(10)	68.1(4)	C(2)-C(3)-P(1)-O(6)	-12.4(4)
Br(1)-C(7)-C(8)-C(9)	176.9(5)	O(1)-C(10)-O(2)-C(11)	-0.8(7)	C(1)-C(3)-P(1)-O(6)	-82.2(4)

**Table 6:** Calculated reflections from PowderCell\*

h	k	l	2 $\theta$ /°	d/Å	I/rel.	F(hkl)	h	k	l	2 $\theta$ /°	d/Å	I/rel.	F(hkl)
0	1	1	7.45	11.85	100.00	124.05	2	0	2	21.96	4.04	11.05	123.71
0	0	2	7.99	11.06	6.05	46.23	0	3	3	22.49	3.95	12.15	132.99
0	1	2	10.17	8.69	9.81	53.15	1	0	5	22.53	3.94	4.04	76.84
1	1	0	11.97	7.39	16.66	81.60	2	1	2	22.87	3.89	36.62	166.13
1	1	1	12.62	7.01	27.72	78.55	1	2	4	22.87	3.89	32.65	156.91
1	0	2	12.94	6.83	24.40	106.94	0	2	5	23.75	3.74	3.27	73.08
0	2	2	14.94	5.92	29.86	136.85	2	2	0	24.07	3.69	2.17	60.39
1	0	3	15.75	5.62	33.38	152.68	2	2	1	24.41	3.64	4.38	61.52
1	2	0	16.22	5.46	23.67	132.56	2	1	3	24.59	3.62	6.49	75.50
1	2	1	16.71	5.30	20.44	89.80	1	3	3	24.74	3.60	7.55	81.94
1	1	3	16.97	5.22	70.80	169.77	0	1	6	24.95	3.57	2.91	72.53
0	1	4	17.22	5.15	16.09	116.14	2	2	2	25.40	3.50	3.73	59.18
0	2	3	17.43	5.08	4.33	61.04	1	0	6	26.23	3.39	6.50	114.31
1	2	2	18.11	4.90	3.39	39.68	1	3	4	26.97	3.30	3.68	62.63
1	1	4	20.04	4.43	3.17	42.60	2	2	3	26.97	3.30	5.04	73.34
1	2	3	20.22	4.39	29.91	132.15	0	3	5	27.73	3.21	3.12	83.95
2	0	0	20.42	4.34	8.88	145.53	2	3	0	28.01	3.18	2.48	75.70
0	2	4	20.43	4.34	6.46	87.78	0	4	3	28.15	3.17	5.09	109.03
0	3	2	20.60	4.31	2.83	58.64	2	3	1	28.30	3.15	11.03	114.15
2	0	1	20.82	4.26	5.10	79.49	1	4	2	28.58	3.12	12.03	120.48
0	1	5	21.03	4.22	2.16	52.28	0	1	7	28.93	3.08	4.03	99.87
1	3	0	21.56	4.12	12.43	128.74	2	2	4	29.05	3.07	6.15	87.60
2	1	1	21.77	4.08	10.74	85.48	1	2	6	29.20	3.06	2.49	56.09
1	3	1	21.93	4.05	13.19	95.47	2	1	5	29.48	3.03	7.64	99.25

Source: Cu-K $\alpha_1$  ( $\lambda = 1.540598 \text{ \AA}$ )

Condition on reflections:  $I \geq 2$

Range (2 $\theta$ ): From 3° to 30°

\*PowderCell for Windows (version 2.4) by Kraus W. & Nolze G., Federal institute for materials Research and testing, Rudower Chausse 5, 12489 Berlin Germany.

## 11. References

- 1) T. Rossolini, J. A. Leitch, R. Grainger, D. J. Dixon *Org. Lett.* **2018**, *20*, 6794–6798.
- 2) Y. Haegawa, T. Cantin, J. Decaens, S. Couve-Bonnaire, A. B. Charette, T. Poisson, P. Jubault *Chem. Eur. J.* **2022**, e202201438.
- 4) Y. Takei, A. Murata, K. Yamagishi, S. Arai, H. Nakamura, T. Inoue, S. Takeoka *Chem. Commun.* **2013**, *49*, 7313-7315.
- 5) S. Yu, R. Hua, X. Fu, G. Liu, D. Zhang, S. Jia, H. Qiu, W. Hu *Org. Lett.* **2019**, *21*, 5737-5741.
- 6) W. Li, L. Zhou *Org. Lett.* **2022**, *24*, 3976-3981
- 7) O. Pavlyuk, H. Teller, M. C. McMills *Tetrahedron Lett.* **2009**, *50*, 2716-2718.
- 8) C. Qin, H. M. L. Davies, *J. Am. Chem. Soc.* **2013**, *135*, 14516–14519.
- 9) W. Wu, Z. Lin, P. Chen, J. Li, H. Jiang, *J. Org. Chem.* **2017**, *82*, 12746-12756.