ELECTRONIC SUPPLEMENTARY INFORMATION

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General Methods and Materials

Commercial grade reagents and solvents were used without further purifications.

NMR spectra: ^{1}H-NMR and ^{13}C-NMR ^{19}FNMR spectra were recorded with instruments at 300 MHz (Bruker AMX 300 and Brucker F300). The chemical shifts are reported in ppm (δ), with the solvent reference relative to tetramethylsilane (TMS).

Mass spectra: Mass spectra were registered on an APEX II & Xmass software (Bruker Daltonics) instrument or on a thermo Finnigan LCQ Advantage instrument, equipped with an ESI ion source.

$[\alpha]^{T}_{D}$: Optical rotations were obtained on a Perkin-Elmer 241 polarimeter at 589 nm using a 1 mL cell, with a length of 1 dm.

HPLC: For HPLC analyses on chiral stationary phase, to determine enantiomeric excesses, it was used an Agilent Instrument Series 1100. The specific operative conditions for each product are reported from time to time.

TLC: Reactions and chromatographic purifications were monitored by analytical thin-layer chromatography (TLC) using silica gel 60 F254 pre-coated glass plates and visualized using UV light, phosphomolybdic acid or ninhydrin.

Chromatographic purification: Purification of the products was performed by column chromatography with flash technique (according to the Still method) using as stationary phase silica gel 230-400 mesh (Merck-SIGMA ALDRICH).

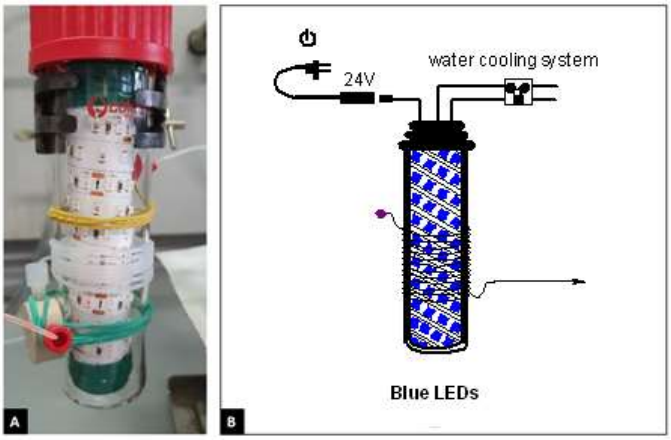
Dry solvents: Dichloromethane (DCM) was dried by distillation under nitrogen atmosphere on CaH2. The other dry solvents used are commercially available and they are stored under nitrogen over molecular sieves (bottles with crown cap).

Reactions work-up: The organic phases, if necessary, were dried over Na_2SO_4. The solvents were removed under reduced pressure and then at high vacuum pump (0.1-0.005 mmHg).

Penn*PhD* Photoreactor m2 with 455 nm LED (3.4 W/cm^2). K-type temperature probe.

MultiVial Photoreactor: home-made, custom-designed; internal diameter 14.4 cm with 12 vial holes. Blue LED stripe 465 nm, 24 W/m, ORBLUF52120812, 44 cm.

Photoreactor #1: For the realization of the home-made photoreactor, see the "making of" video associated to our previous work (Medici, F.; Resta, S.; Presenti, P.; Caruso, L.; Puglisi, A.; Raimondi, L.; Rossi, S.; Benaglia, M. Stereoselective Visible Light Catalytic Cyclization of Bis(Enones): A Viable Approach to the Synthesis of Enantiomerically Enriched Cyclopentane Rings. *European Journal of Organic Chemistry n/a* (n/a). https://doi.org/10.1002/ejoc.202100397). The glassware components are available at Colaver s.r.l (www.colaver.it). A strip of 47 LEDs #1 (39.16 cm) was employed.



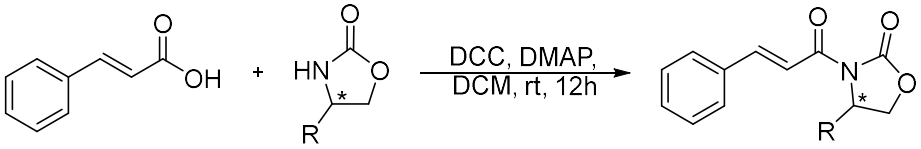
Blue LEDs characterization:

LEDs type-1: Commercially available ORBLUF52120812 120 led/m 12V with IP95 protection skin and a self-adhesive tape were employed in the realization of photoreactor #1. Blue LEDs showed an almost monochromatic emission profile showing a maximum of intensity located at ca. 460 nm with I = 674.0 mW/cm^2

Substrate Synthesis

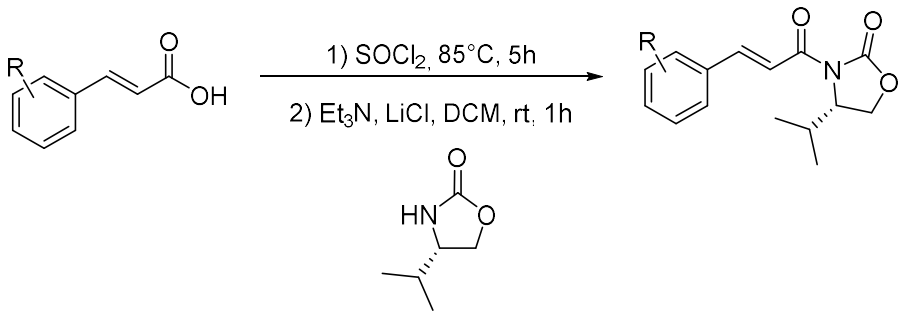
Oxazolidinone Derivatives

General procedure A



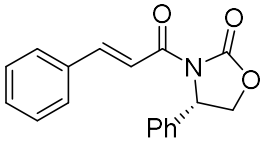
To a suspension of oxazolidinone, DMAP (0.22 eq.) and *trans*-cinnamic acid (1 eq.) in DCM (0.5M) at 0 °C, under argon atmosphere, was added DCC (1 eq.) in one portion. After 10 min the temperature was raised to r.t. and stirring was continued until no starting material has left as confirmed by TLC. The dicyclohexylurea formed was filtered and the precipitate washed with DCM. The filtrate was washed with sat. $NaHCO_3$, dried with $MgSO_4$ and concentrated at reduced pressure to furnish the crude product, which was purified by silica gel chromatography (8:2 Hex/EtOAc).

General procedure B



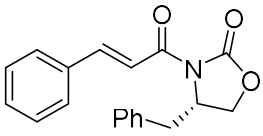
A suspension of the corresponding cinnamic derivatives in $SOCl_2$ was refluxed for 5h. The resulting solution was cooled to r.t. and evaporated several times with CH_2Cl_2 to give a residue free of $SOCl_2$ and HCl. The crude product was then dissolved in DCM and OxaIPr, LiCl and Et_3N were added. The mixture was allowed to stir for 1h, then quenched by adding 1M HCl solution. The organic phase was washed with $NaHCO_3$ sat. sol. and brine then dried over $MgSO_4$. Filtration and evaporation of the solvent gave the crude product, which is purified by flash column chromatography (Hex/AcOEt 8:2)

(S)-3-cinnamoyl-4-phenyloxazolidin-2-one, 2a



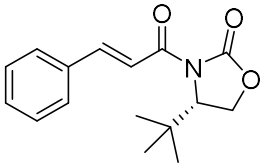
Using General procedure A, starting from (S)-4-phenyloxazolidin-2-one, obtained a white solid in 97% yield.
The data are in accordance with the literature.[1]

(S)-4-benzyl-3-cinnamoyloxazolidin-2-one, 2b



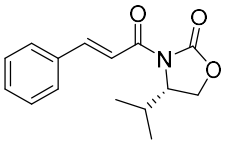
Using General procedure A, starting from (*S*)-4-benzyloxazolidin-2-one, obtained a white solid in 95% yield.
The data are in accordance with the literature.[2]

(S)-4-(tert-butyl)-3-cinnamoyloxazolidin-2-one, 2c



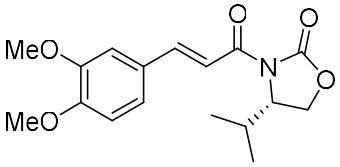
Using General procedure A, starting from (*S*)-4-(tert-butyl)oxazolidin-2-one, obtained a white solid in 90% yield.
The data are in accordance with the literature.[2]

(S)-3-cinnamoyl-4-isopropyloxazolidin-2-one, 2d



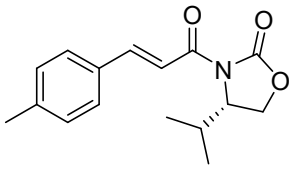
Using General procedure A, starting from (*S*)-4-isopropyloxazolidin-2-one, obtained a white solid in 95% yield.
The data are in accordance with the literature.[3]

(S,E)-3-(3-(3,4-dimethoxyphenyl)acryloyl)-4-isopropyloxazolidin-2-one, 6



Using General procedure B starting from (E)-3-(3,4-dimethoxyphenyl)acrylic acid, obtained a white solid in 87% yield.
^{1}H-NMR ($CDCl_3$) = δ 7.92 (bs, 2H), 7.32 (dd, J = 8.31, 1.94 Hz, 1H), 7.26 (bs, 1H), 7.00 (d, J = 8.25 Hz, 1H), 4.71-4.66 (m, 1H), 4.44 (t, J = 8.63 Hz, 1H), 4.36 (dd, J = 9.08, 3.28 Hz, 1H), 4.04 (d, J = 3.35 Hz, 6H), 2.63-2.53 (m, 1H), 1.06 (dd, J = 11.31, 6.93 Hz, 6H)
^{13}C-NMR ($CDCl_3$) = δ 165.3, 154.3, 151.5, 149.2, 146.2, 127.7, 123.3, 114.8, 111.0, 110.1, 63.4, 58.6, 55.9, 28.6, 18.0, 14.7
HRMS (ESI+): calc. $C_{17}H_{21}NO_5Na$: 342.1317; found: 342.1318

(S,E)-4-isopropyl-3-(3-(p-tolyl)acryloyl)oxazolidin-2-one, 7



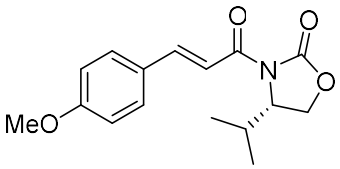
Using General procedure B starting from (E)-3-(p-tolyl)acrylic acid, obtained a white solid in 91% yield.

^{1}H-NMR ($CDCl_3$) = δ 7.85 (q, J = 26.16, 15.73 Hz, 2H), 7.51 (d, J = 8.08, 2H), 7.19 (d, J = 7.19 Hz, 2H), 4.55 (m, 1H), 4.27 (m, 2H), 2.45 (m, 1H), 2.37 (s, 3H), 0.93 (dd, J = 11.25 6.96 Hz, 6H).

^{13}C-NMR ($CDCl_3$) = δ 165.4, 154.3, 146.3, 141.2, 132.0, 129.7, 128.7, 116.1, 63.5, 58.7, 28.7, 21.6, 18.1, 14.8

HRMS (ESI+): calc. $C_{16}H_{19}NO_3Na$: 296.1263 found: 296.1261

(S,E)-4-isopropyl-3-(3-(4-methoxyphenyl)acryloyl)oxazolidin-2-one, 8



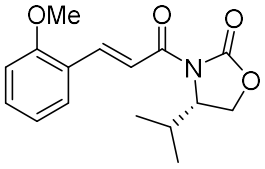
Using General procedure B, starting from (E)-3-(4-methoxyphenyl)acrylic acid, obtained a white solid in 88% yield.

^{1}H-NMR ($CDCl_3$) = δ 7.80 (s, 2H), 7.56 (d, J = 8.78, 2H), 6.89 (d, J = 6.89 Hz, 2H), 4.57-4.52 (m, 1H), 4.32-4.20 (m, 2H), 3.83 (s, 3H), 2.48-2.40 (m, 1H), 0.92 (dd, J = 11.29 7.02 Hz, 6H)).

^{13}C-NMR ($CDCl_3$) = δ 165.5, 161.8, 154.3, 146.1, 130.5, 127.5, 114.7, 114.4, 63.5, 58.8, 55.5, 28.7, 18.1, 14.9

HRMS (ESI+): calc. $C_{16}H_{19}NO_4Na$: 312.1212; found: 312.1210

(S,E)-4-isopropyl-3-(3-(2-methoxyphenyl)acryloyl)oxazolidin-2-one, 9



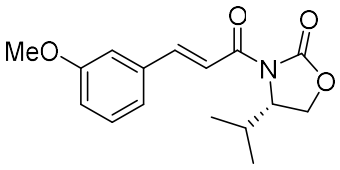
Using General procedure B starting from (E)-3-(2-methoxyphenyl)acrylic acid, obtained a white solid in 95% yield.

^{1}H-NMR ($CDCl_3$) = δ 8.19 (d, J = 15.85 Hz, 1H), 8.00 (d, J = 15.86, 1H), 7.63 (dd, J = 7.73, 1.69Hz, 1H), 7.35 (t, J = 7.87, 1H), 6.99-6.90 (m, 2H), 4.59-4.54 (m, 1H), 4.33 – 4.21 (m, 2H), 3.90 (s, 3H), 2.53-2.41 (m, 1H), 0.96-0.90 (dd, J = 11.51, 6.97 Hz, 6H).

^{13}C-NMR ($CDCl_3$) = δ 165.8, 158.8, 154.3, 141.7, 132.0, 129.5, 123.8, 120.9, 117.6, 111.3, 63.5, 58.8, 55.7, 28.7, 18.2, 14.9

HRMS (ESI+): calc. $C_{16}H_{19}NO_4Na$: 312.1212; found: 312.1210

(S,E)-4-isopropyl-3-(3-(3-methoxyphenyl)acryloyl)oxazolidin-2-one, 10

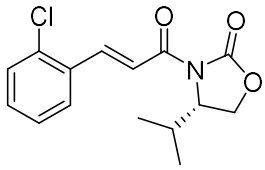


Using General procedure B starting from (E)-3-(3-methoxyphenyl)acrylic acid, obtained a white solid in 90% yield.

^{1}H-NMR ($CDCl_3$) = δ 7.92 (q, J = 34.42 15.69 Hz, 2H), 7.33-7.20 (m, 2H), 7.13-7.11 (m, 1H), 6.94 (dd, J = 8.13, 2.69, 2H), 4.57-4.53 (m, 1H), 4.31-4.22 (m, 2H), 3.83 (s, 3H), 2.49 – 2.43 (m, 1H), 0.93 (dd, J = 11.71 6.98 Hz, 6H)

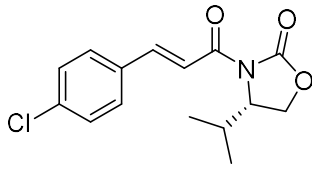
^{13}C-NMR ($CDCl_3$) = δ 165.3, 160.0, 154.3, 146.2, 136.1, 130.0, 121.4, 117.6, 116.7, 113.5, 63.6, 58.8, 55.5, 28.7, 18.2, 14.9
HRMS (ESI+): calc. $C_{16}H_{19}NO_4Na$: 312.1212; found: 312.1210

(S,E)-3-(3-(2-chlorophenyl)acryloyl)-4-isopropyloxazolidin-2-one, 11



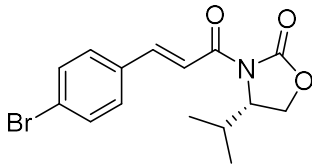
Using General procedure B starting from (E)-3-(2-chlorophenyl)acrylic acid, obtained a white solid in 86% yield.
^{1}H-NMR ($CDCl_3$) = δ 8.18 (d, J = 15.71 Hz, 1H), 7.86 (d, J = 15.74 Hz, 1H), 7.69-7.66 (m, 1H), 7.35 – 7.32 (m, 1H), 7.27-7.18 (m, 2H), 4.53-4.48 (m, 1H), 4.30 – 4.16 (m, 2H), 2.44 – 2.35 (m, 1H), 0.87 (dd, 11.00 6.97 Hz, 6H).
^{13}C-NMR ($CDCl_3$) = δ 164.6, 154.1, 141.4, 135.2, 132.7, 131.3, 130.0, 128.0, 127.1, 119.6, 63.5, 58.6, 28.5, 17.9, 14.7
HRMS (ESI+): calc. $C_{15}H_{16}NO_3NaCl$: 316.0716; found: 316.0714

(S,E)-3-(3-(4-chlorophenyl)acryloyl)-4-isopropyloxazolidin-2-one, 12



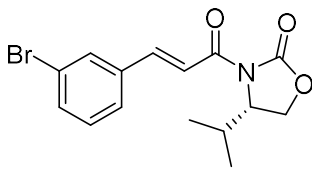
Using General procedure B starting from (E)-3-(4-chlorophenyl)acrylic acid, obtained a white solid in 91% yield.
^{1}H-NMR ($CDCl_3$) = δ 7.92 (d, J = 15.75 Hz, 1H), 7.77 (d, J = 15.73 Hz, 1H), 7.56-7.53 (m, 2H), 7.38 – 7.35 (m, 2H), 4.57-4.53 (m, 1H), 4.35 – 4.23 (m, 2H), 2.51 – 2.40 (m, 1H), 0.93 (dd, 12.55 6.97 Hz, 6H).
^{13}C-NMR ($CDCl_3$) = δ 165.1, 154.3, 144.8, 136.7, 133.3, 129.9, 129.3, 117.8, 63.6, 58.8, 28.7, 18.2, 14.9
HRMS (ESI+): calc. $C_{15}H_{16}NO_3NaCl$: 316.0716; found: 316.0714

(S,E)-3-(3-(4-bromophenyl)acryloyl)-4-isopropyloxazolidin-2-one, 13



Using General procedure B starting from (E)-3-(4-bromophenyl)acrylic acid, obtained a white solid in 86% yield.
^{1}H-NMR ($CDCl_3$) = δ 7.93 (d, J = 15.70 Hz, 1H), 7.75 (d J = 15.75, 1H), 7.53-7.45 (m, 4H), 4.57-4.52 (m, 1H), 4.35 – 4.22 (m, 2H), 2.50 – 2.40 (m, 1H), 0.93 (dd, J = 12.55 6.98 Hz, 6H).
^{13}C-NMR ($CDCl_3$) = δ 165.1, 154.3, 144.8, 133.7, 132.3, 130.1, 125.0, 117.9, 63.6, 28.8, 28.7, 18.2, 14.9.
HRMS (ESI+): calc. $C_{15}H_{16}NO_3NaBr$: 360.0211 found: 360.0208

(S,E)-3-(3-(3-bromophenyl)acryloyl)-4-isopropyloxazolidin-2-one, 14



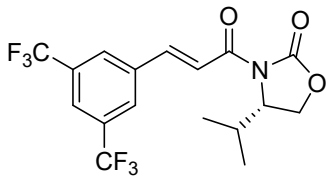
Using General procedure B starting from (E)-3-(3-bromophenyl)acrylic acid, obtained a white solid in 83% yield.
^{1}H-NMR ($CDCl_3$) = δ 7.76 (t, J = 1.92 Hz, 1H), 7.65 (d, J = 8.08 Hz, 1H), 7.55 (d, J = 7.86 Hz, 1H), 7.29 (t, J = 7.89 Hz, 1H), 4.68-4.61 (m, 1H), 4.38

(t, J = 8.85 Hz, 1H), 4.27-4.22 (m, 1H), 2.52 – 2.42 (m, 1H), 0.97 (d, J = 6.74 Hz, 6H).

^{13}C-NMR ($CDCl_3$) = δ 168.4, 153.7, 135.3, 135.2, 131.9, 129.5, 127.6, 122.0, 63.7, 58.8, 26.4, 18.0, 15.2

HRMS (ESI+): calc. $C_{15}H_{16}NO_3NaBr$: 360.0211 found: 360.0208

(S,E)-3-(3-(3,5-bis(trifluoromethyl)phenyl)acryloyl)-4-isopropyloxazolidin-2-one, 15



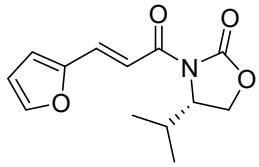
Using General procedure B starting from (E)-3-(3,5-bis(trifluoromethyl)phenyl)acrylic acid, obtained a white solid in 58% yield.

^{1}H-NMR ($CDCl_3$) = δ 8.08-7.81 (m, 5H), 4.60-4.55 (m, 1H), 4.38 – 4.26 (m, 2H), 2.52 – 2.41 (m, 1H), 0.95 (dd, J = 13.13 6.96, 6H).

^{13}C-NMR ($CDCl_3$) = δ 164.4, 154.3, 142.3, 132.6 (q, 33.7 Hz, *C*CF_3), 128.2, 123.7, 123.1 (q, 272.8 Hz, *C*F_3), 121.3, 117.7, 63.8, 28.9, 28.6, 18.1, 14.9.

^{19}FNMR: δ -63.07

(S,E)-3-(3-(furan-2-yl)acryloyl)-4-isopropyloxazolidin-2-one, 16



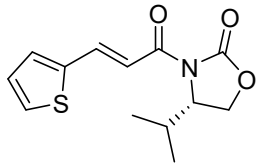
Using General procedure B starting from (E)-3-(furan-2-yl)acrylic acid, obtained a white solid in 67% yield.

^{1}H-NMR ($CDCl_3$) = δ 7.95 (d, J = 15.39 Hz, 1H), 7.73 (d, J = 15.39, 1H), 7.41 (d, J = 5.05 Hz, 1H), 7.33 (d, J = 3.70, 1H), 7.06 (dd, J = 5.08 3.64 Hz, 1H), 4.57-4.52 (m, 1H), 4.33 – 4.21 (m, 2H), 2.50 – 2.39 (m, 1H), 0.92 (dd, J = 11.95 6.98 Hz, 6H).

^{13}C-NMR ($CDCl_3$) = δ 165.1, 154.2, 140.2, 138.6, 131.7, 129.4, 128.8, 115.9, 63.5, 58.8, 28.7, 18.2, 14.9.

HRMS (ESI+): calc. $C_{13}H_{15}NO_4Na$: 272.0899; found: 272.0903

(S,E)-4-isopropyl-3-(3-(thiophen-2-yl)acryloyl)oxazolidin-2-one, 17



Using General procedure B starting from (E)-3-(thiophen-2-yl)acrylic acid, obtained a white solid in 71% yield.

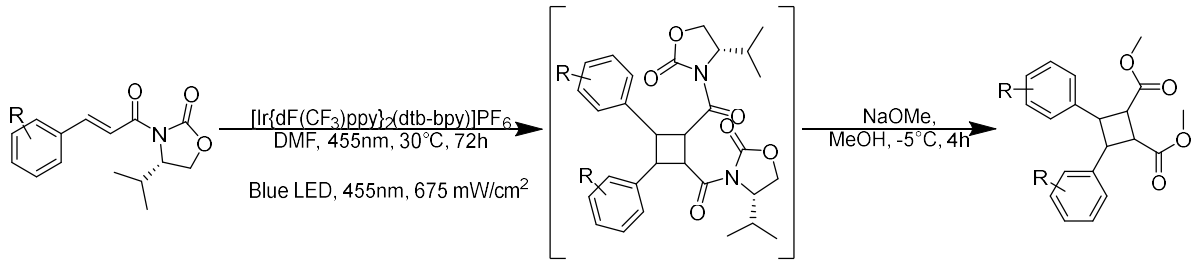
^{1}H-NMR ($CDCl_3$) = δ 7.77 (d, J = 15.43 Hz, 1H), 7.59 (d, J = 15.44, 1H), 7.51 (d, J = 1.77 Hz, 1H), 6.69 (d, J = 3.44, 1H), 6.48 (dd, J = 3.47 1.81 Hz, 1H), 4.57-4.52 (m, 1H), 4.33 – 4.21 (m, 2H), 2.48 – 2.42 (m, 1H), 0.92 (dd, J = 12.36 6.99 Hz, 6H).

^{13}C-NMR ($CDCl_3$) = δ 165.3, 154.2, 151.6, 145.3, 132.3, 116.0, 114.9, 112.6, 63.5, 58.8, 28.7, 18.2, 14.9.

HRMS (ESI+): calc. $C_{13}H_{15}NO_3NaS$: 288.0670; found: 288.0672

Photodimerisation of oxazolidinone derivatives 2, 6-17

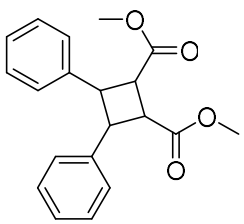
General procedure C



In a sealable vial, to a solution of the oxazolidinone derivative (0.5 mmol) in dry DMF (0.5 M), the Ir catalyst (0.005 mmol, 5.61 mg, 0.01 eq.) was added, and the solution was degassed with N_2 for 20 minutes. The vial was then sealed under N_2 and irradiated for 72h at 30°C. the crude solution was diluted with DCM (10 mL) and washed with brine (5x15mL). The organic phase was dried over $MgSO_4$ then filtered and the solvent evaporated. The crude product was filtered on a short pad of silica gel (Hex/EtOAc 7:3) to eliminate the catalyst and used in the next step without further purification.

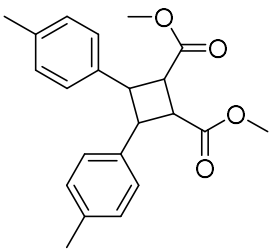
In a heat gun-dried flask under nitrogen the crude product and dry MeOH (0.017 M) were added. The mixture was cooled at -5°C and a freshly prepared solution of NaOMe (2.1 eq.) was added dropwise. The reaction was allowed to stir for 4h at -5°C. The reaction was then quenched with a 0.1 M solution of HCl (1 mL), diluted with EtOAc (10 mL) and washed with brine (3x 15mL) the aqueous phase was then extracted with EtOAc (20 mL). The reunited organic phase was dried over $MgSO_4$, filtered and the solvent evaporated. The crude product was purified by flash chromatography (Hex/EtOAc 8:2) to obtain the pure product.

Dimethyl 3,4-diphenylcyclobutane-1,2-dicarboxylate, 4-5



Using General procedure C starting from (R)-3-cinnamoyl-4-isopropyloxazolidin-2-one, obtained a white solid in 71% yield. Data according to literature.[4]

Dimethyl 3,4-di-p-tolylcyclobutane-1,2-dicarboxylate, 18



Using General procedure C starting from (S,E)-4-isopropyl-3-(3-(p-tolyl)acryloyl)oxazolidin-2-one, obtained a white solid in 93% yield, dr(trans/Cis): ≥ 98:2.

^{1}H-NMR ($CDCl_3$) = δ 7.22-7.12 (m, 8H), 3.73 (s, 6H), 3.68-3.65 (m, 2H), 3.48-3.45 (m, 2H), 2.33 (s, 6H).

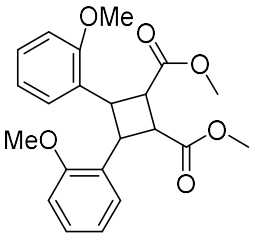
^{13}C-NMR ($CDCl_3$) = δ 173.2, 138.2, 136.9, 129.4, 126.9, 52.3, 47.5, 44.6, 21.2.

HRMS (ESI+): calc. $C_{22}H_{24}O_4Na$: 375.1572; found: 375.1571

HPLC data: Chiralpack OD-H, 98:2 Hex/IPA, 0.8 mL/min; τ: 7 min, 11min; ee%: 95%.

$[\alpha]_D^{20}$ = -4.00; c=0.15 M in acetone

Dimethyl 3,4-bis(2-methoxyphenyl)cyclobutane-1,2-dicarboxylate, 19



Using General procedure C starting from ((S,E)-4-isopropyl-3-(3-(2-methoxyphenyl)acryloyl)oxazolidin-2-one, obtained a white solid in 92% yield, dr(trans/Cis): 70:30.

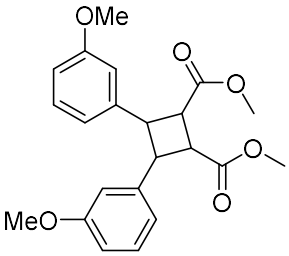
^{1}H-NMR ($CDCl_3$) = δ 7.24(t, J = 7.91Hz, 3H), 7.04-6.97 (m, 2H), 6.91 (td, j = 7.49, 7.48, 1.04, 2H), 6.83 (d, 2H), 6.74 (td, J = 7.50, 7.50, 1.08 Hz, 1H), 6.53 (d, 1H), 4.61-4.59 (m, 1H), 4.20-4.16 (m, 2H), 4.02-3.99 (m, 1H), 3.73 (s, 12H), 3.53 (s, 2H), 3.36-3.33 (m, 2H).

^{13}C-NMR ($CDCl_3$) = δ 173.8, 173.5, 157.5, 157.2, 129.5, 128.2, 128.0, 127.9, 127.7, 127.5, 127.4, 120.5, 119.6, 110.2, 109.7, 55.1, 54.7, 52.0, 51.9, 45.4, 40.8, 40.6

HRMS (ESI+): calc. $C_{22}H_{24}O_6Na$: 407.1471; found: 407.1469

HPLC data: Lux 5u cellulose-3, 98:2 Hex/IPA, 0.8 mL/min; τ: 32 min, 40min; ee%: 79%.

Dimethyl 3,4-bis(3-methoxyphenyl)cyclobutane-1,2-dicarboxylate, 20



Using General procedure C starting from (S,E)-4-isopropyl-3-(3-(3-methoxyphenyl)acryloyl)oxazolidin-2-one, obtained a white solid in 91% yield, dr(trans/Cis): 70:30.

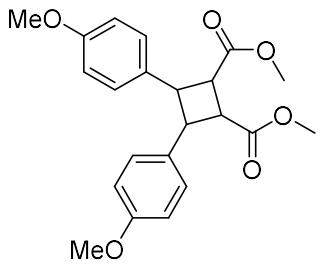
^{1}H-NMR ($CDCl_3$) = δ 7.24(t, J = 7.91Hz, 3H), 7.05 (t, J = 7.92, 1H), 6.90-6.77 (m, 6H), 6.63-6.55 (m, 2H), 6.45 (t, J = 2.07 Hz, 0.74H), 4.37-4.35 (m, 0.77H), 3.83 – 3.81 (m, 1H), 3.79 (s, 6H), 3.75 (s, 3H), 3.74 (s, 6H), 3.71-3.68 (m, 2H), 3.63 (s, 2.33H), 3.49-3.45 (m, 2H).

^{13}C-NMR ($CDCl_3$) = δ 173.1, 160.0, 142.8, 140.3, 129.8, 129.2, 120.3, 119.3, 113.7, 112.9, 112.5, 112.3, 55.3, 55.2, 52.3, 47.4, 45.0, 44.5, 43.5.

HRMS (ESI+): calc. $C_{22}H_{24}O_6Na$: 407.1471; found: 407.1468

HPLC data: Lux 3u cellulose-1, 98:2 Hex/IPA, 0.8 mL/min; τ: 20 min, 27 min; ee%: 89%.

Dimethyl 3,4-bis(4-methoxyphenyl)cyclobutane-1,2-dicarboxylate, 21



Using General procedure C starting from (S,E)-4-isopropyl-3-(3-(4-methoxyphenyl)acryloyl)oxazolidin-2-one, obtained a white solid in 94% yield, dr(trans/Cis): 70:30

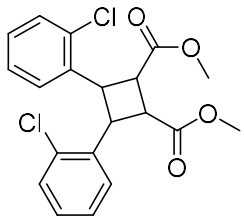
^{1}H-NMR ($CDCl_3$) = δ 7.22-7.19 (m, 4H), 6.88-6.82(m, 7H), 6.68-6.65 (m, 2H), 4.31-4.29 (m, 1H), 3.79-3.71 (m, 22H), 3.62-3.59 (m, 2H), 3.44-3.41 (m, 2H).

^{13}C-NMR ($CDCl_3$) = δ 173.2, 158.9, 158.2, 133.3, 130.9, 129.0, 128.1, 114.2, 113.6, 55.4, 55.3, 52.3, 47.5, 44.7, 44.5, 43.7

HRMS (ESI+): calc. $C_{22}H_{24}O_6Na$: 407.1471; found: 407.1471

HPLC data: Lux 3u cellulose-1, 98:2 Hex/IPA, 0.8 mL/min; τ: 21 min, 35 min; ee%: 95%.

Dimethyl 3,4-bis(2-chlorophenyl)cyclobutane-1,2-dicarboxylate, 22



Using General procedure C starting from (S,E)-3-(3-(2-chlorophenyl)acryloyl)-4-isopropyloxazolidin-2-one, obtained a white solid in 85% yield, dr(trans/Cis): ≥ 98:2.

^{1}H-NMR ($CDCl_3$) = δ 7.58 (d, J = 8.46 Hz, 2H), 7.31 (t, J = 7.90, 4H), 7.18 (td, J = 7.60, 7.57, 1.65 Hz, 2H), 4.33 (m, 2H), 3.73 (s, 6H), 3.45 (m, 2H).

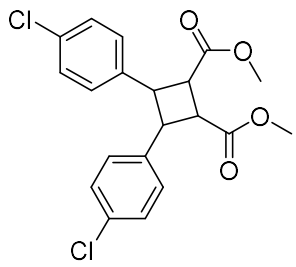
^{13}C-NMR ($CDCl_3$) = δ 172.8, 137.7, 134.0, 129.8, 128.6, 128.5, 127.4, 52.4, 44.8, 43.6.

HRMS (ESI+): calc. $C_{20}H_{18}O_4NaCl_2$: 415.0480; found: 415.0480

HPLC data: Lux 3u amylose-1, 98:2 Hex/IPA, 0.8 mL/min; τ: 20 min, 22 min; ee%: >98%.

$[\alpha]_D^{20}$ = -6.67; c=0.10 M in acetone

Dimethyl 3,4-bis(4-chlorophenyl)cyclobutane-1,2-dicarboxylate, 23



Using General procedure C starting from (S,E)-3-(3-(4-chlorophenyl)acryloyl)-4-isopropyloxazolidin-2-one, obtained a white solid in 86% yield, dr(trans/Cis): ≥ 98:2.

^{1}H-NMR ($CDCl_3$) = δ 7.30 (d, J = 8.46 Hz, 4H), 7.20 (d, J = 8.51 Hz, 4H), 3.74 (s, 6H), 3.64-3.61 (m, 2H), 3.46-3.42 (m, 2H).

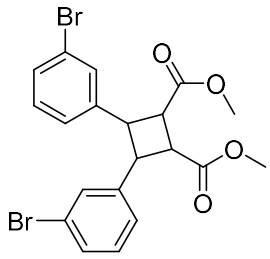
^{13}C-NMR ($CDCl_3$) = δ 172.7, 139.2, 133.4, 129.1, 128.3, 52.5, 47.1, 44.5

HRMS (ESI+): calc. $C_{20}H_{18}O_4NaCl_2$: 415.0480; found: 415.0476

HPLC data: Lux 3u cellulose-1, 98:2 Hex/IPA, 0.8 mL/min; τ: 11 min, 18 min; ee%: 73%.

$[\alpha]_D^{20}$ = -22.50; c=0.20 M in acetone

Dimethyl 3,4-bis(3-bromophenyl)cyclobutane-1,2-dicarboxylate, 24



Using General procedure C starting from (S,E)-3-(3-(3-bromophenyl)acryloyl)-4-isopropyloxazolidin-2-one obtained a white solid in 79% yield, dr(trans/Cis): ≥ 98:2.

^{1}H-NMR ($CDCl_3$) = δ 7.42-7.39 (m, 4H),7.21-7.19 (m, 4H), 3.76 (s, 6H), 3.68-3.65 (m, 2H), 3.46 – 3.45 (m, 2H).

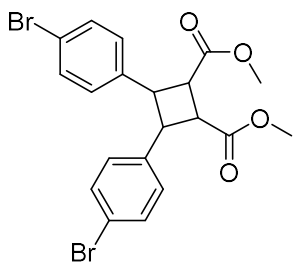
^{13}C-NMR ($CDCl_3$) = δ 172.6, 142.9, 131.9,130.7, 130.5, 130.0, 127.2, 125.7, 123.1, 62.3, 52.5, 46.9, 44.6, 42.8, 29.9

HRMS (ESI+): calc. $C_{20}H_{18}O_4NaBr_2$: 502.9470; found: 502.9470

HPLC data: Lux 3u cellulose-1, 98:2 Hex/IPA, 0.8 mL/min; τ: 14 min, 18 min; ee%: 79%.

$[\alpha]_D^{20}$ = -11.30; c=0.10 M in acetone

Dimethyl 3,4-bis(4-bromophenyl)cyclobutane-1,2-dicarboxylate, 25



Using General procedure C starting from (S,E)-3-(3-(4-bromophenyl)acryloyl)-4-isopropyloxazolidin-2-one, obtained a white solid in 83% yield, dr(trans/Cis): 68:32.

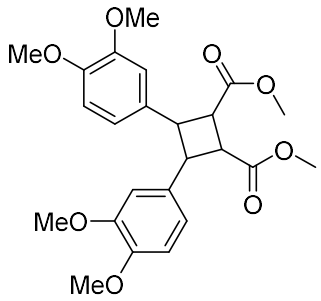
^{1}H-NMR ($CDCl_3$) = δ 7.45 (d, J = 8.39 Hz, 3H), 7.26 (d, J = 8.41, 2H), 7.14 (d, J = 8.44 Hz, 3H), 6.79 (d, J = 8.45, 2H), 4.35-4.33 (m, 1H), 3.78-3.72 (m, 9H), 3.62-3.59 (m, 2H), 3.45-3.42 (m, 2H).

^{13}C-NMR ($CDCl_3$) = δ 172.7, 172.6, 139.6, 137.4, 132.0, 131.5, 129.5, 128.6, 128.5, 121.4, 120.8, 52.5, 52.4, 47.1, 44.4, 43.4.

HRMS (ESI+): calc. $C_{20}H_{18}O_4NaBr_2$: 502.9470; found: 502.9468

HPLC data: Lux 3u cellulose-1, 98:2 Hex/IPA, 0.8 mL/min; τ: 12 min, 21 min; ee%: >98%.

Dimethyl 3,4-bis(3,4-dimethoxyphenyl)cyclobutane-1,2-dicarboxylate, 26



Using General procedure C starting from (S,E)-3-(3-(3,4-dimethoxyphenyl)acryloyl)-4-isopropyloxazolidin-2-one, obtained a white solid in 85% yield, dr(trans/Cis): 70:30.

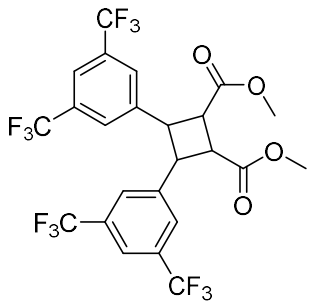
^{1}H-NMR ($CDCl_3$) = δ 7.29 (bs, 4H), 7.23-7.17 (m, 6H), 7.03-6.71 (m 18H), 6.53 (d, J = 8.01, 2H), 4.60-4.57 (m, 2H), 4.18-4.15 (m, 5H), 4.00-3.98 (m, 3H), 3.73 (s, 29H), 3.53 (s, 6H), 3.35-3.32 (m, 5H).

^{13}C-NMR ($CDCl_3$) = δ 173.9, 173.6, 157.6, 157.3, 129.7, 128.4, 128.1, 127.9, 127.5, 120.6, 119.7, 110.3, 109.8, 55.1, 54.9, 52.1, 52.0, 45.5, 42.5, 40.9, 40.7.

HRMS (ESI+): at HRMS MW ion was not visible, only fragmentation is observed

HPLC data: Lux 3u amylose-2, 98:2 Hex/IPA, 0.8 mL/min; τ: 30 min, 34 min; ee%: 83%.

Dimethyl 3,4-bis(3,5-bis(trifluoromethyl)phenyl)cyclobutane-1,2-dicarboxylate, 27



Using General procedure C starting from (S,E)-3-(3-(3,5-bis(trifluoromethyl)phenyl)acryloyl)-4-isopropyloxazolidin-2-one, obtained a white solid in 72% yield, dr(trans/Cis): ≥ 98:2.

^{1}H-NMR ($CDCl_3$) = δ 7.60 (s, 2H), 7.28 (s, 4H), 4.62-4.60 (m, 2H), 3.93-3.91 (m, 2H), 3.80(s, 6H).

^{19}F-NMR ($CDCl_3$) = δ -63.37 (s, 12F).

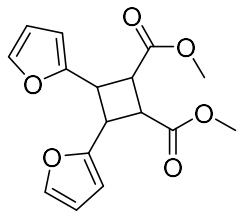
^{13}C-NMR ($CDCl_3$) = δ 171.7, 140.1, 132.1 (q, J = 33.53 Hz, *C*CF_3), 127.8, 124.6 (q, J = 227.68 Hz, *C*F_3), 121.2, 52.8, 44.8, 42.4.

HRMS (ESI+): calc. $C_{24}H_{16}O_4NaF_{12}$: 619.0755; found: 619.0745

HPLC data: Lux 3u cellulose-1, 98:2 Hex/IPA, 0.8 mL/min; τ: 5 min, 7 min; ee%: 91%.

$[\alpha]_D^{20}$ = +103.33; c=0.13 M in acetone

Dimethyl 3,4-di(furan-2-yl)cyclobutane-1,2-dicarboxylate, 28



Using General procedure C starting from (S,E)-3-(3-(furan-2-yl)acryloyl)-4-isopropyloxazolidin-2-one, obtained a white low melting solid in 90% yield, dr: 1:1.

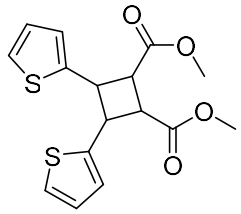
^{1}H-NMR ($CDCl_3$) = δ 7.37-7.36 (m, 1H), 7.23-7.22 (m, 1H), 6.30-6.29 (q, J = 3.20, 1.92 Hz, 1H), 6.21-6.15 (m, 2H), 5.94 (d, J = 3.28 Hz, 1H), 4.26-4.24 (m, 1H), 3.88-3.87 (m, 1H), 3.79-3.76 (m, 2H), 3.73 (s, 7H), 3.56-3.52 (m, 2H).

^{13}C-NMR ($CDCl_3$) = δ 172.4, 172.3, 153.3, 152.7, 142.4, 141.9, 110.5, 110.3, 107.1, 106.8, 52.4, 52.3, 43.2, 43.2, 39.8, 38.6.

HRMS (ESI+): calc. $C_{16}H_{16}O_6Na$: 327.0845; found: 327.0846

HPLC data: Chiralpack OD-H, 98:2 Hex/IPA, 0.8 mL/min; τ: 12 min, 14 min; ee%: 83%.

Dimethyl 3,4-di(thiophen-2-yl)cyclobutane-1,2-dicarboxylate, 29



Using General procedure C starting from(S,E)-4-isopropyl-3-(3-(thiophen-2-yl)acryloyl)oxazolidin-2-one, obtained a white waxy solid in 89% yield dr: 1:1.

^{1}H-NMR ($CDCl_3$) = δ 7.22-7.20 (m, 2H), 7.09 (dd, J = 5.09, 1.13, 2H), 6.98-6.96 (m, 4H), 6.87-6.83 (m, 2H), 6.78 (dd, J = 3.58, 1.08 Hz, 2H), 4.55-4.52 (m, 2H), 3.87 – 3.83 (m, 2H), 3.82-3.80 (m, 2H), 3.75 (d, J = 1.55 Hz, 12H), 3.49-3.46 (m, 2H).

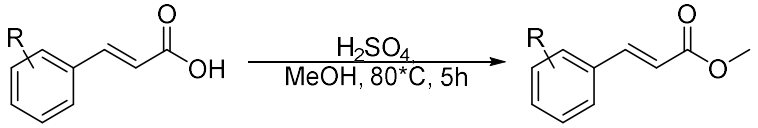
^{13}C-NMR ($CDCl_3$) = δ 172.2, 143.8, 141.7, 127.2, 126.8, 126.7, 125.4, 124.8, 124.7, 124.6, 52.4, 52.3, 45.9, 45.8, 45.1, 41.6.

HRMS (ESI+): calc. $C_{16}H_{16}O_4NaS_2$: 359.0388; found: 359.0385

HPLC data: Lux 3u cellulose-1, 98:2 Hex/IPA, 0.8 mL/min; τ: 19 min, 21min; ee%: >98%.

Cinnamic esters

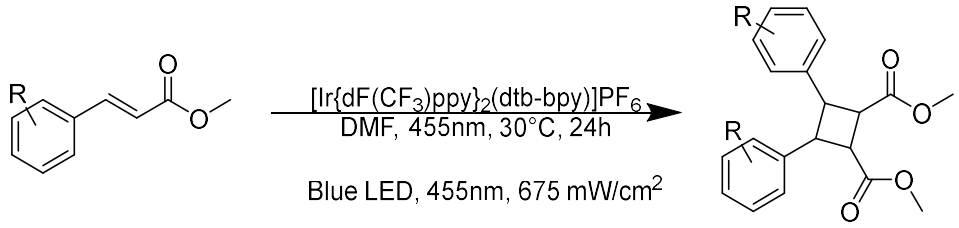
General Procedure D



To a solution of substituted cinnamic acid (5 mmol) in MeOH (0.3M), sulfuric acid, few drops, was added and the solution was stirred at 80°C for 5h. The mixture was then diluted with water (10 mL) and extracted with DCM(3x15 mL). The combined organic phases were washed with aq. sat. sol. $NaHCO_3$ (3x10 mL). The organic phases were then dried over $MgSO_4$, filtered and then evaporated to obtain the pure product. The data from the Me-esters obtained are in agreement with the literature.[5, 6, 7, 8, 9]

Photodimerisation of methyl cinnamate derivatives

General Procedure E



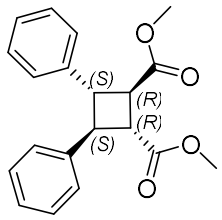
In a sealable vial, to a solution of the cinnamic ester (0.5 mmol) in dry DMF (0.5 M), the Ir catalyst (0.005 mmol, 5.61 mg, 0.01 eq.) was added, and the solution was degassed with N_2 for 20 minutes. The vial was then sealed under N_2 and irradiated for 72h at 30°C. the crude solution was diluted with DCM (10 mL) and washed with brine (5x15mL). The organic phase was dried over $MgSO_4$ then filtered and the solvent evaporated. The crude product was purified by flash chromatography (Hex/EtOAc 8:2) to obtain the pure product. Data were in accordance with the previous obtained enantioenriched compounds

Specific Rotation

An exact amount of the substrate was dissolved in 1 mL of acetone. The solution was then charged in the polarimeter cell, and the specific rotation was measured.

compound **4**: $[\alpha]_D^{20}$ = -8.26 c=0.25 in acetone

According to the comparison with the literature data[10] absolute configuration of **4** was established to be 1*R*, 2*R*, 3*S*, 4*S.*



dimethyl (**1*R*,2*R*,3*S*,4*S***)-3,4-diphenylcyclobutane-1,2-dicarboxylate

Specific rotations were measured for other compounds isolated as >98/2 d.r.

compound **18** : $[\alpha]_D^{20}$ = -4.00 c=0.15 M in acetone

compound **22** : $[\alpha]_D^{20}$ = --6.67 c=0.1 M in acetone

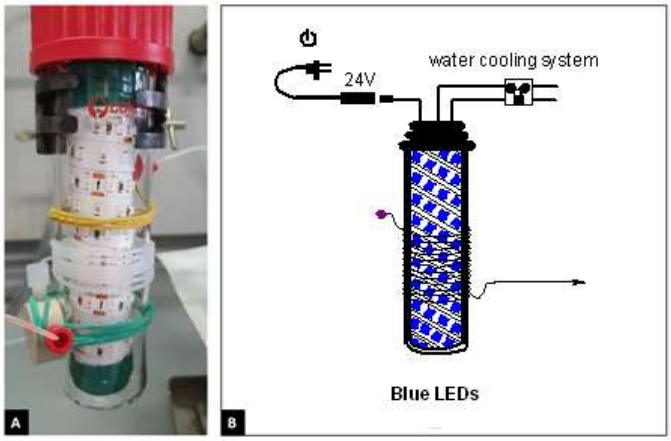
compound **23** : $[\alpha]_D^{20}$ = -22.50 c=0.2 M in acetone

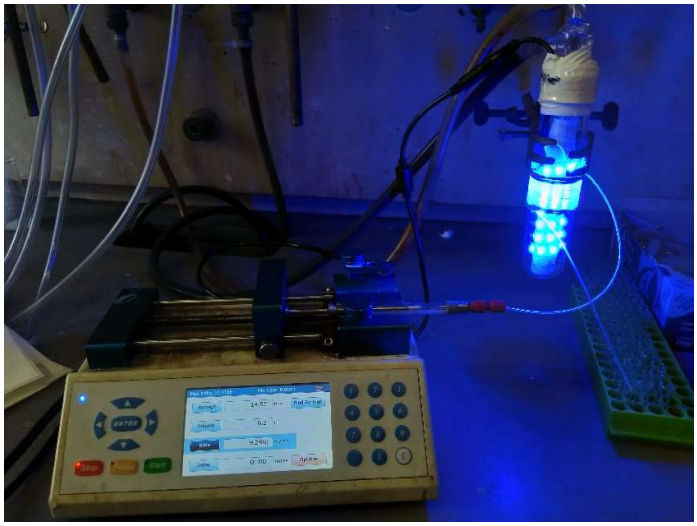
compound **24** : $[\alpha]_D^{20}$ = -11.30 c=0.1 M in acetone

compound **27** : $[\alpha]_D^{20}$ = +103.33 c=0.13 M in acetone

Flow Chemistry

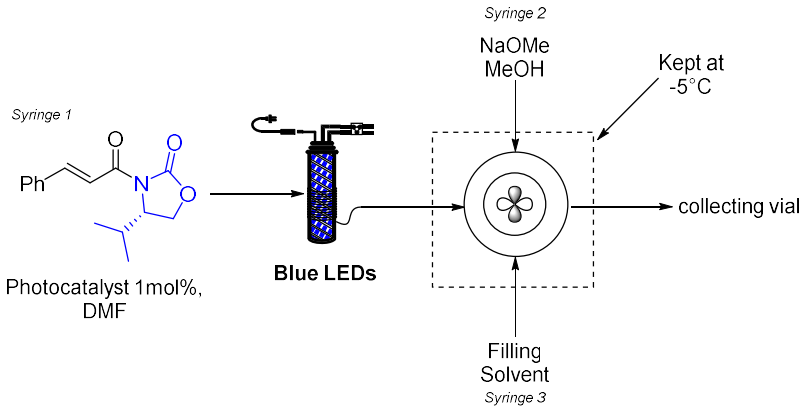
Photocatalysis step





An HPFA tube (ID 0.01 in OD 1/16 in, h: 150cm) was rolled up to the photoreactor and nitrogen was fluxed inside. Meanwhile, in a dried flask were added the Ir catalyst, the substrate, 1,3,5-trimethoxybenzene, and DMF. The solution was then degassed with nitrogen for 15 minutes. The solution was then charged in a SGE syringe and attached to the coil reactor. The coil was filled with the reaction solution, and then the syringe was placed in the syringe pump. After turning on the syringe pump the LEDs were also turned on. After each cycle (1 residence time) the vial was changed. The first two volumes were discarded. To the fraction $CDCl_3$ was added and the mixture was directly analysed by NMR.

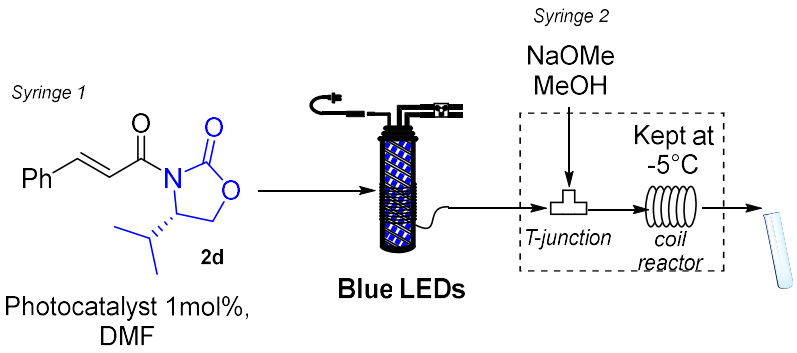
Two step one-pot reaction: CSTR

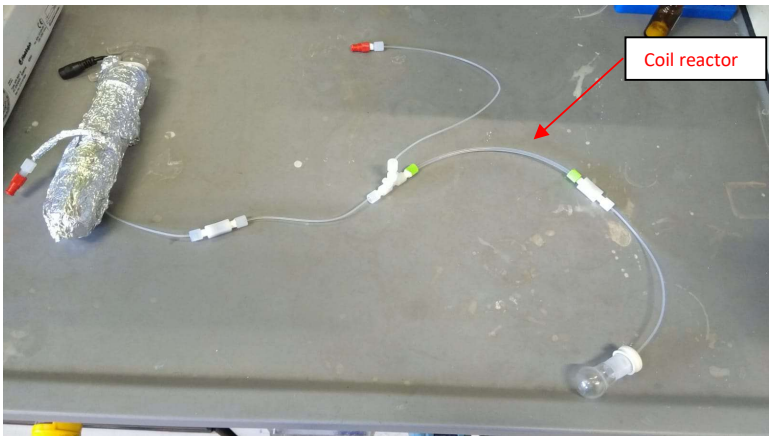


A CSTR (1.5mL volume with the stirring bar) was connected by HPFA tubing to the exit of the photoreactor. A syringe containing a 2M NaOMe solution, and the syringe containing the filling solvent were connected to the CSTR with a T-junction. In a dried flask were added the Ir catalyst (0.00292 mmol, 1 mol%), **2d**(2.92 mmol), 1,3,5-trimethoxybenzene(0.96 mmol, 0.33 eq.), and DMF(4M). The solution was then degassed with nitrogen for 15 minutes, and then charged in a SGE syringe and connected to the photoreactor. The Flow rate were setup to 1.1 uL/min for the syringe 1; 5.5 uL/min for the syringe 2, and 10.06 uL/min for the syringe 3. The total flow rate was 16.66 uL/min and the residence time in the CSTR is 90 minutes. All the tubes were filled up to the CSTR then the syringe pumps and the LEDs were started. Each 167 minutes (dead volumes included) the collecting vial was changed. The first two volumes were discharged. The fractions were quenched by addition of a 0.1M HCl solution, then extracted with EtOAc (3x5 mL); the organic phases were washed with saturated $NaHCO_3$ solution (2 mL) and brine (2 mL). The organic phase was dried over $MgSO_4$ filtered and evaporated to obtain the crude

that was analyzed by NMR. Flash chromatography (Hex/EtOAc 8:2) was performed to obtain the pure product that was analysed by HPLC.

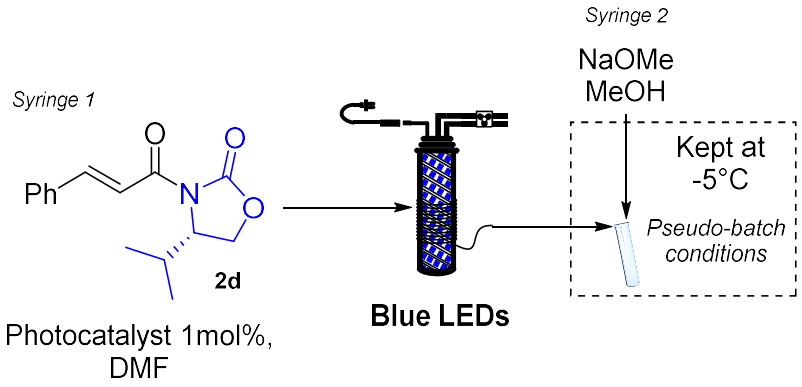
Two step one-pot reaction: Coil reactor





The exit of the photoreactor and a syringe containing a 1M NaOMe solution were connected by a T-junction to a coil reactor (FEP tubing, 19.1 cm, 0,38 mL volume) for the esterification. The coil reactor was immersed in a bath kept at -5°C. All the system was flushed with N_2 for 1h. In a dried flask were added the Ir catalyst (0.00292 mmol, 1 mol%), **2d** (2.92 mmol), 1,3,5-trimethoxybenzene (0.96 mmol, 0.33 eq.), and DMF (4M). The solution was then degassed with nitrogen for 15 minutes, and then charged in a SGE syringe and attached to the photoreactor. The Flow rate were setup to 1.1 uL/min for the syringe 1; 4.31 uL/min for the syringe 2. The total flow rate was 5.41 uL/min and the residence time in the FEP coil reactor was 70 minutes. All the tubes were filled up to the T-junction then the syringe pumps and the LEDs were started. Each 133 minutes (dead volumes included) the collecting vial was changed. After the first volume clogging was observed and the reaction was stopped.

Two step Flow/Batch reaction



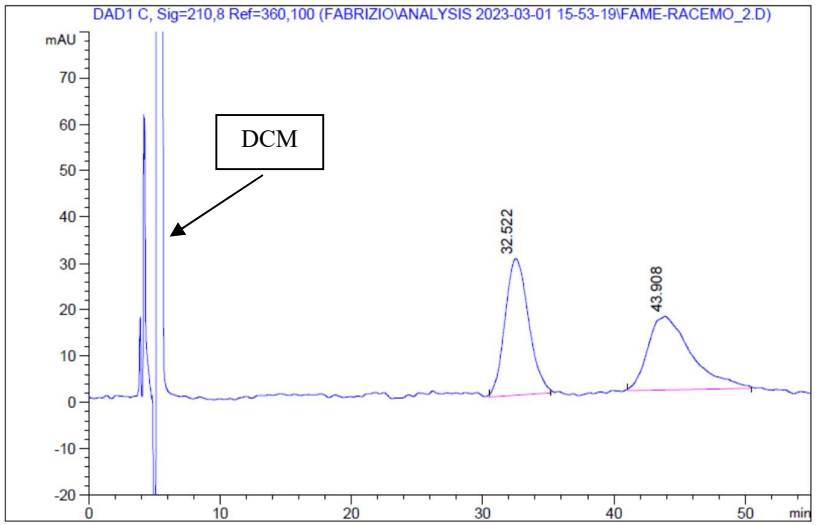
The exit of the photoreactor was collected in a vial filled with 1 mL dry MeOH equipped with a stirring bar and kept a -5°C under nitrogen. A 1 M NaOMe solution fed the vial via a syringe 2. The flow rate for the syringe 1 was 1.1 uL/min (60 minutes residence time in the photoreactor), while the flow rate for syringe 2 was 4.4 uL/min. After each cycle the vial was changed, and the mixture was allowed to stir for additional 1h before the quench. The first two volumes were discarded. The fractions were quenched by addition of a 0.1M HCl solution, then extracted with EtOAc (3x5 mL); the organic phases were washed with saturated $NaHCO_3$ solution (2 mL) and brine (2 mL). The organic phase was dried over $MgSO_4$ filtered and evaporated to obtain the crude that was analyzed by NMR. Flash chromatography (Hex/EtOAc 8:2) were performed to obtain the pure product to analysed by HPLC.

HPLC traces.

The HPLC conditions are indicated on the top of the chromatogram (column, solvent, flow rate, pressure)

- ***Trans-Dimethyl 3,4-diphenylcyclobutane-1,2-dicarboxylate 4: racemic (Left); derived from (S)-3-cinnamoyl-4-isopropyloxazolidin-2-one 2d (right)***

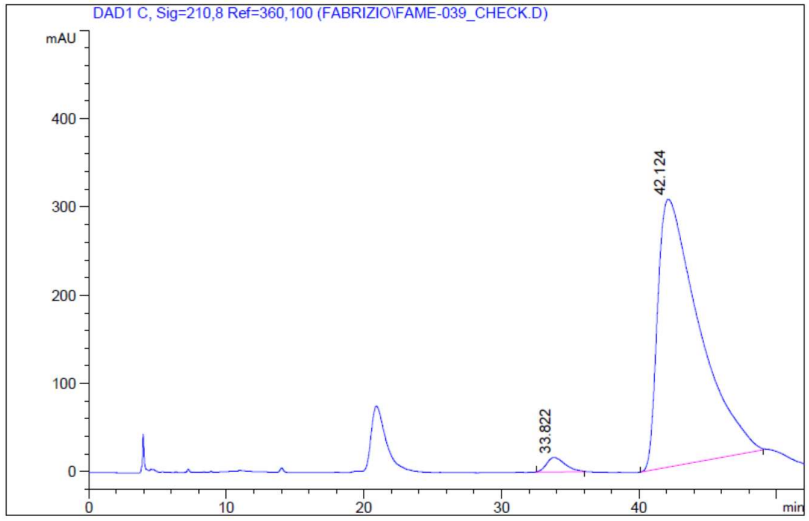
Lux 5u cellulose-3, Hex/IPA 98:2, 0.8 mL/min, P= 33 ba
r



Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area	Area %	Name
1	32.522	MM	2.079	3673.400	50.125	
2	43.908	MM	3.841	3655.116	49.875	

LUX 5u CELLULOSE-3, Hex/IPA 98:2, 0.8 mL/min, P=35 bar

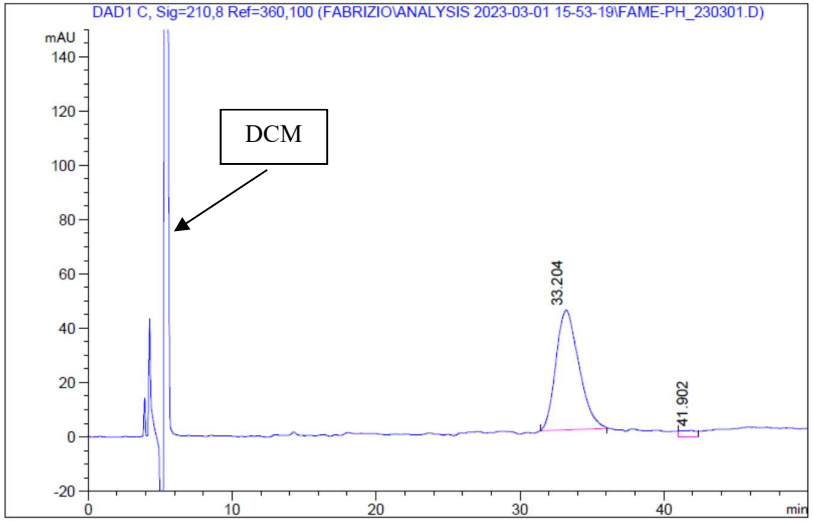


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area	Area %	Name
1	33.822	BB	1.097	1515.486	2.336	
2	42.124	BB	2.817	63350.137	97.664	

- ***Dimethyl 3,4-diphenylcyclobutane-1,2-dicarboxylate 4 from (S)-3-cinnamoyl-4-phenyloxazolidin-2-one 2a***

Lux 5u cellulose-3, Hex/IPA 98:2, 0.8 mL/min, P= 33 ba
r

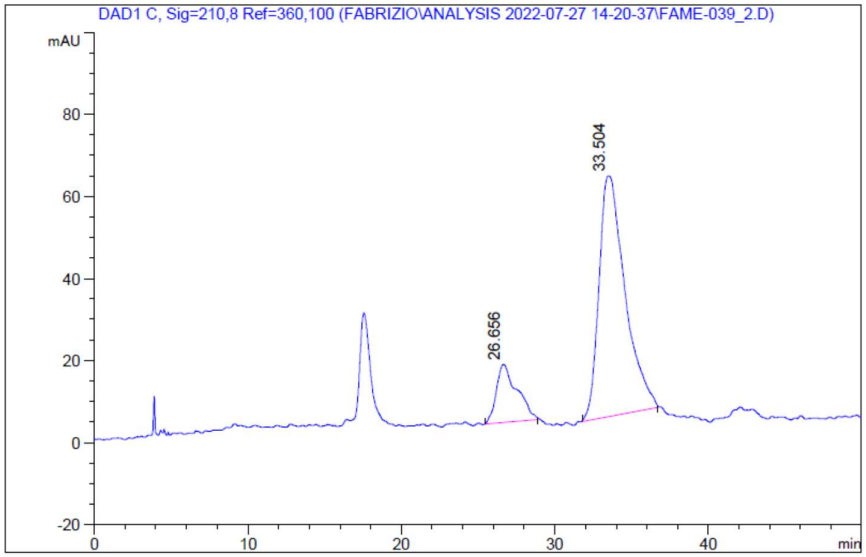


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area	Area %	Name
1	33.204	BB	1.408	4907.896	96.398	
2	41.902	MM	1.310	183.397	3.602	

- ***Dimethyl 3,4-diphenylcyclobutane-1,2-dicarboxylate 4 from (S)-4-benzyl-3-cinnamoyloxazolidin-2-one 2b***

Lux 5u cellulose-3, Hex/IPA 98:2, 0.8 mL/min, P= 40bar



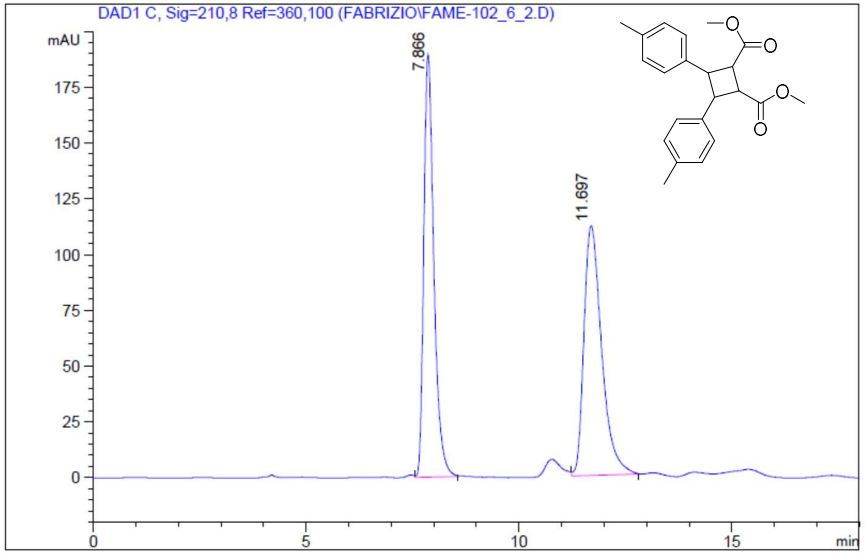
Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area	Area %	Name
1	26.656	BB	1.216	1317.200	16.011	
2	33.504	BB	1.400	6909.724	83.989	

racemic (left); enantiomerically enriched (right)

- ***Dimethyl 3,4-di-p-tolylcyclobutane-1,2-dicarboxylate, 18***

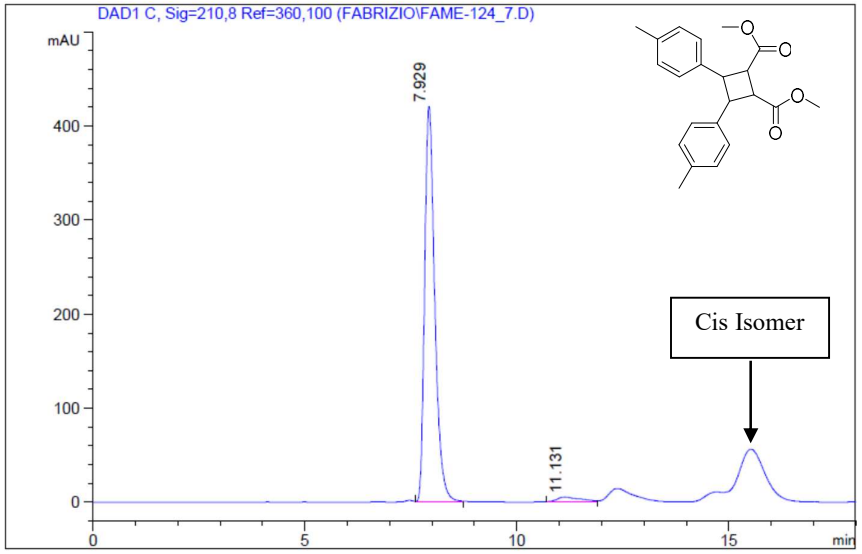
Chiralcell OD-H, Hex:IPA 98:2, 0.8 mL/min, p=32 bar



Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area	Area %	Name
1	7.866	VB	0.252	3111.180	48.951	
2	11.697	VB	0.446	3244.538	51.049	

CHIRALPACK OD-H, Hex/IPA 98:2, 0.8 mL/min, P=34 bar

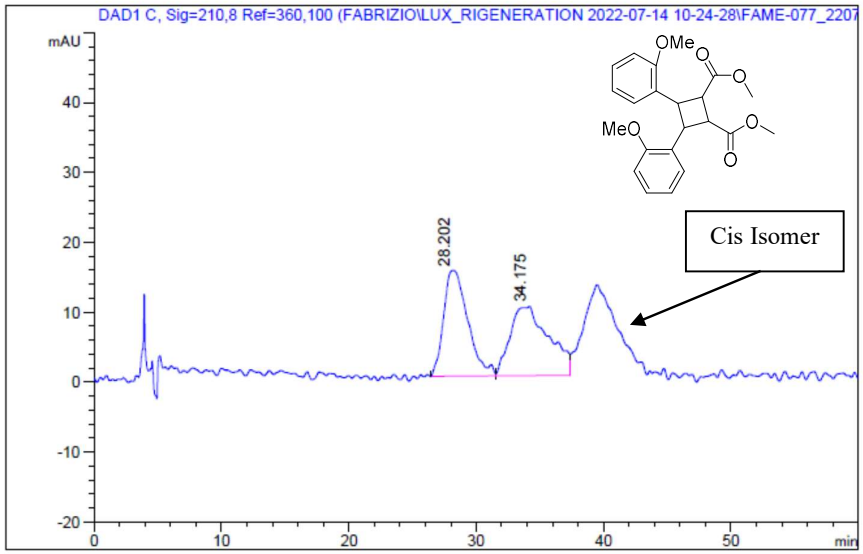


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area	Area %	Name
1	7.929	VB	0.256	6982.550	97.501	
2	11.131	BV	0.530	178.964	2.499	

- ***Dimethyl 3,4-bis(2-methoxyphenyl)cyclobutane-1,2-dicarboxylate, 19***

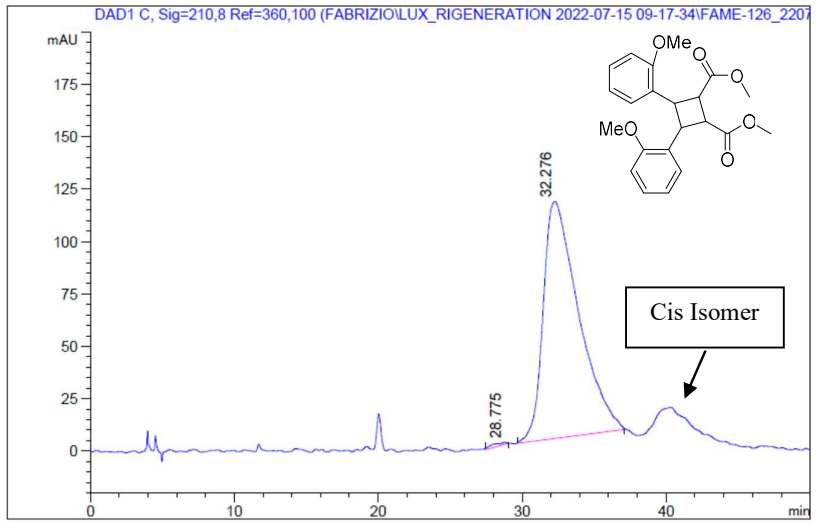
Lux 5u cellulose-3, Hex/IPA, 98:2, 0.6 mL/min, P= 40



Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area	Area %	Name
1	28.202	BV	1.554	2008.148	48.921	
2	34.175	VV	2.490	2096.764	51.079	

Lux 5u cellulose-3, Hex/IPA 98:2, 0.8 mL/min, P= 40bar

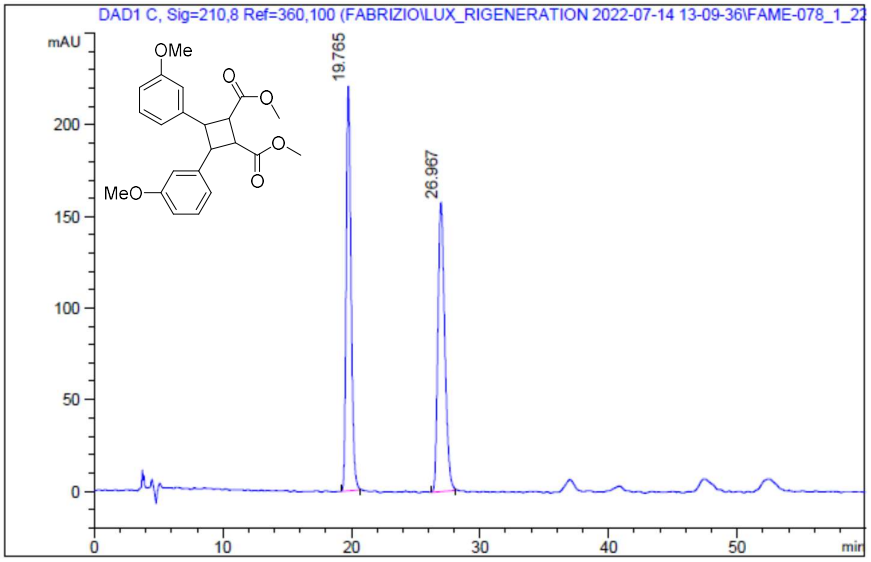


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area	Area %	Name
1	28.775	MM	1.237	103.024	0.532	
2	32.276	BB	2.316	19261.131	99.468	

- ***Dimethyl 3,4-bis(3-methoxyphenyl)cyclobutane-1,2-dicarboxylate, 20***

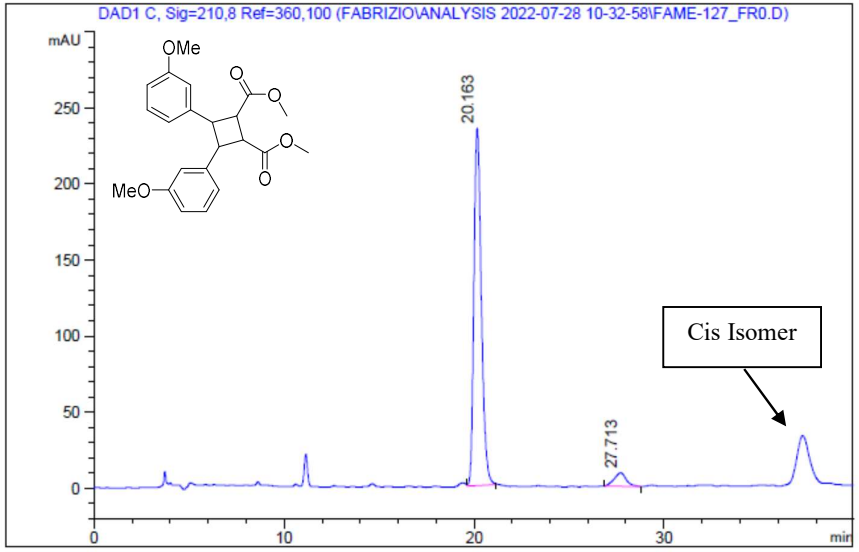
Lux 3u cellulose-1, Hex/IPA, 98:2, 0.8 mL/min, P= 67



Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area	Area %	Name
1	19.765	BB	0.407	5806.403	49.844	
2	26.967	BB	0.571	5842.653	50.156	

Lux 3u cellulose-1, Hex/IPA 98:2, 0.8 mL/min, P= 50bar

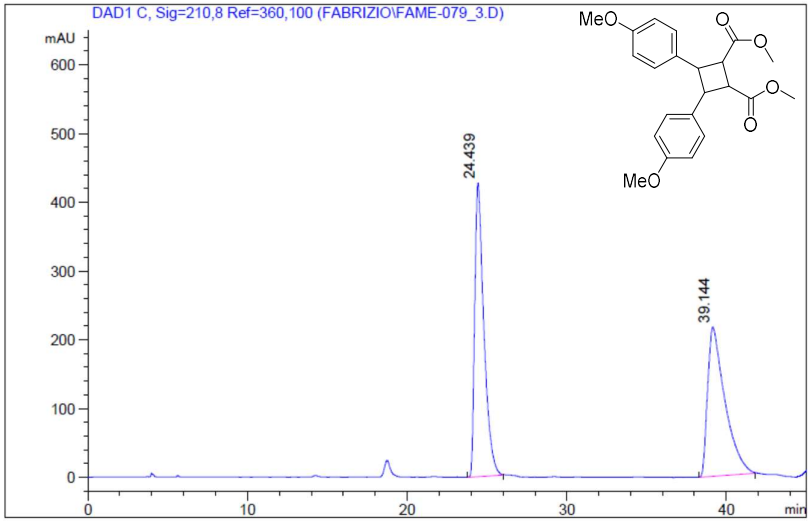


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area	Area %	Name
1	20.163	VB	0.417	6387.538	94.264	
2	27.713	BB	0.624	388.651	5.736	

- ***Dimethyl 3,4-bis(4-methoxyphenyl)cyclobutane-1,2-dicarboxylate, 21***

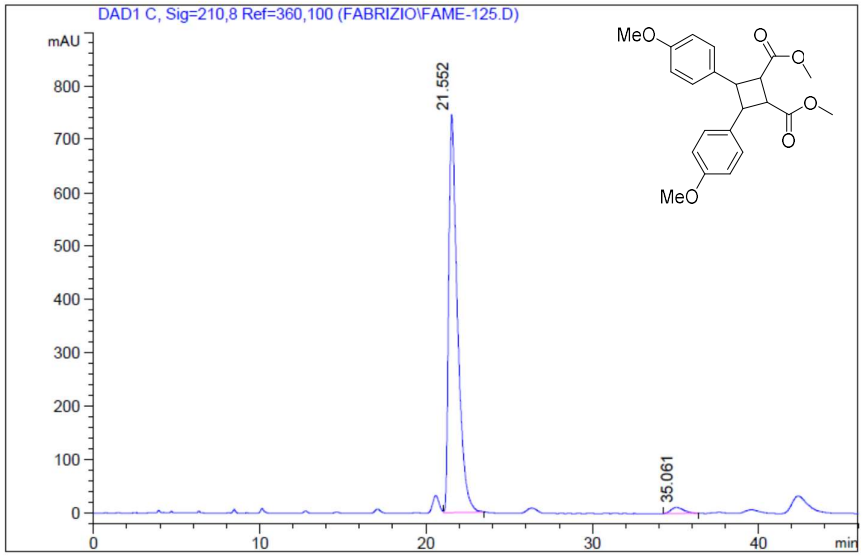
Lux 3u cellulose-1, Hex:IPA 98:2, 0.8 ml/min, p=64 bar



Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area	Area %	Name
1	24.439	BB	0.594	16955.877	50.934	
2	39.144	BB	1.071	16333.938	49.066	

Lux 3u cellulose-1, Hex:IPA 98:1, 0,8 ml/min, p=59 bar

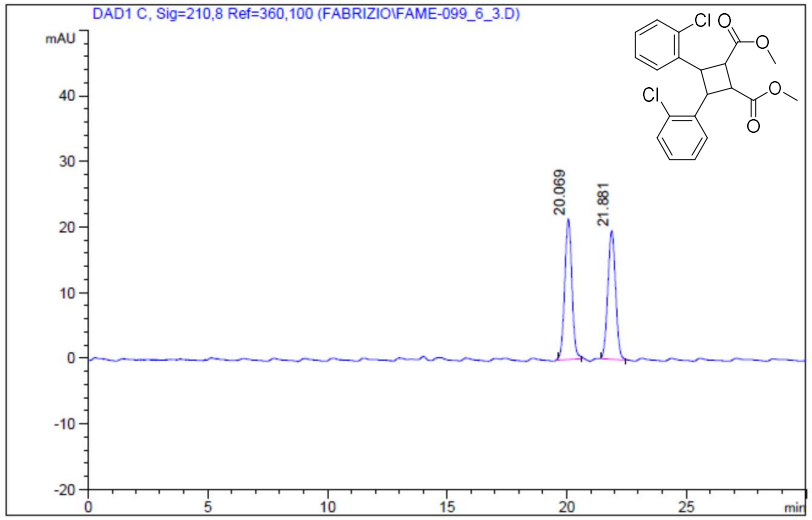


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area	Area %	Name
1	21.552	VB	0.543	27433.533	97.814	
2	35.061	BB	0.750	613.239	2.186	

- ***Dimethyl 3,4-bis(2-chlorophenyl)cyclobutane-1,2-dicarboxylate, 22***

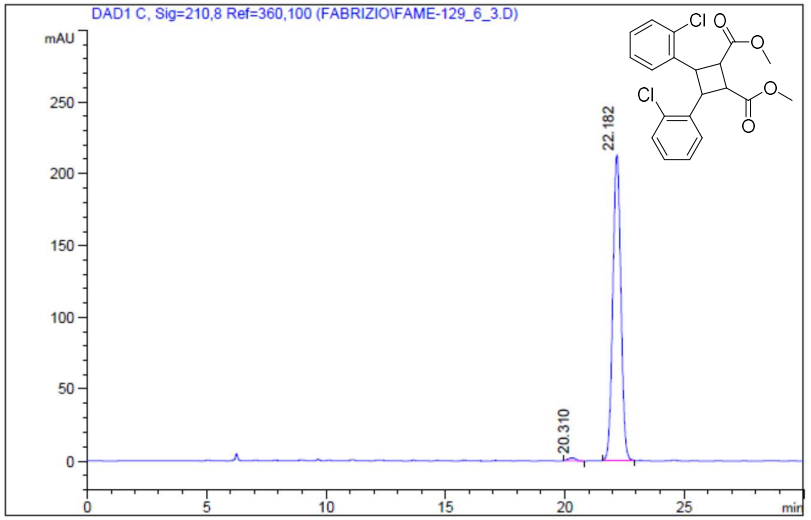
Lux 3u amyl.-1, 98:2 Hex/IPA, 0.8 mL/min, P=66 bar



Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area	Area %	Name
1	20.069	BB	0.326	448.727	50.456	
2	21.881	BB	0.349	440.614	49.544	

Lux 3u amyl.-1, 98:2 Hex/IPA, 0.8 mL/min, P=66 bar

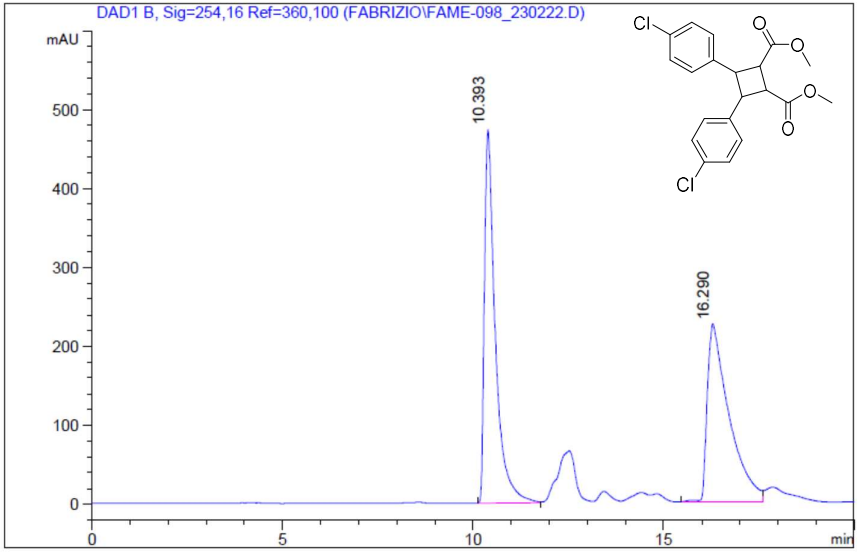


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area	Area %	Name
1	20.310	BB	0.291	46.351	0.920	
2	22.182	BB	0.368	4991.650	99.080	

- ***Dimethyl 3,4-bis(4-chlorophenyl)cyclobutane-1,2-dicarboxylate, 23***

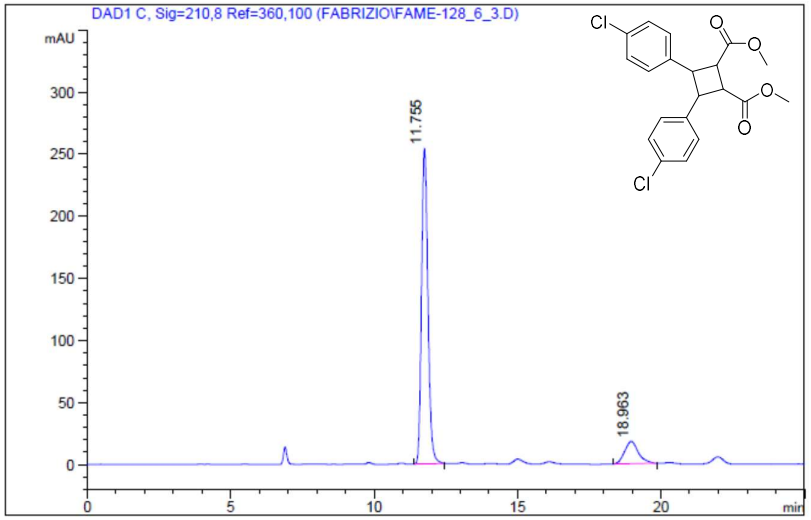
Lux 3u cell-1, 98:2 Hex/Ipa, 0.8 mL/min, P=53 bar



Signal 1: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area	Area %	Name
1	10.393	BB	0.289	9388.033	50.928	
2	16.290	BV	0.566	9045.977	49.072	

Lux 3u cell.-1, 98:2 Hex/IPA, 0.8 mL/min, P=51 bar

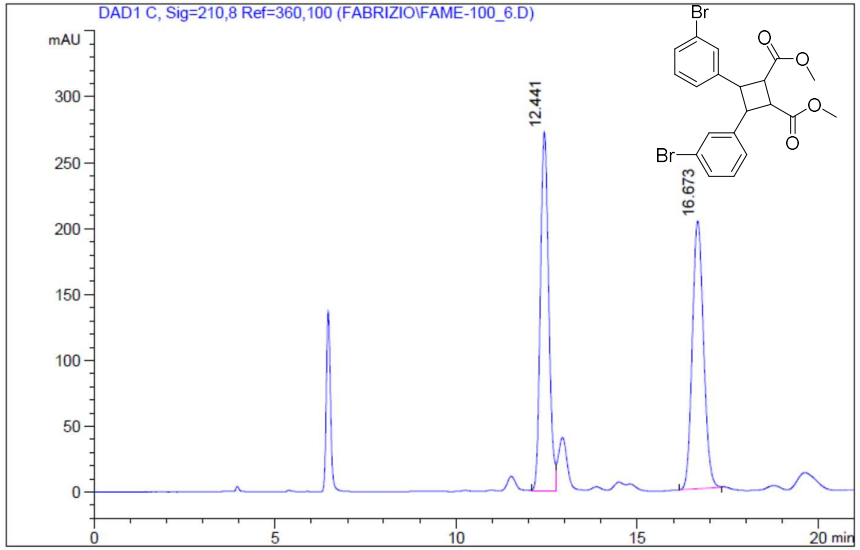


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area	Area %	Name
1	11.755	BB	0.238	3926.003	86.729	
2	18.963	BB	0.492	600.764	13.271	

- ***Dimethyl 3,4-bis(3-bromophenyl)cyclobutane-1,2-dicarboxylate, 24***

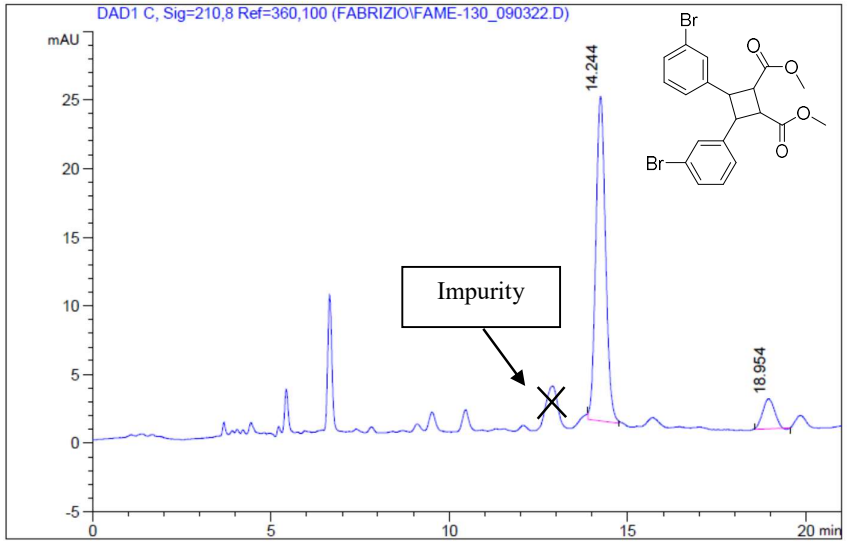
Lux 3u cellulose-1, Hex:IPA 98:2, 0.8 mL/min, p=54 bar



Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area	Area %	Name
1	12.441	BV	0.249	4356.873	49.844	
2	16.673	BB	0.335	4384.192	50.156	

Lux 3u cell.-1, Hex/IPA 98:2, 0.8 mL/min, P=57 bar

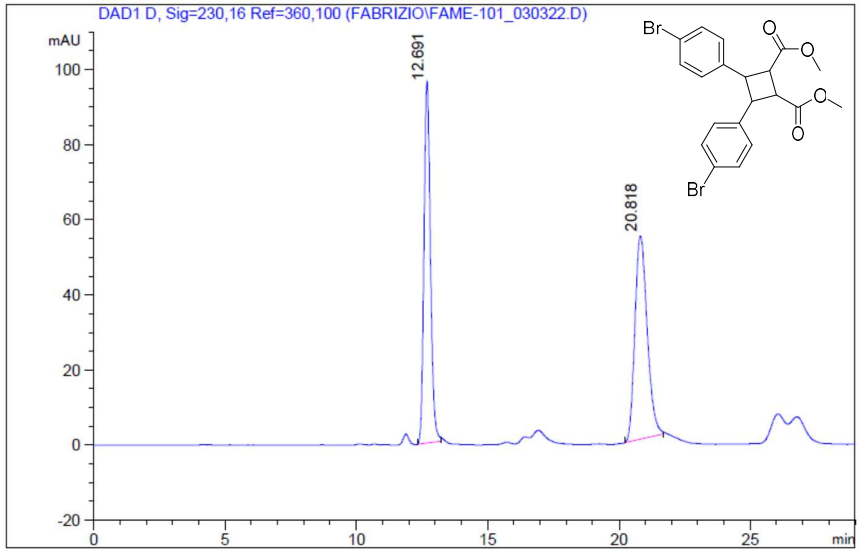


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area	Area %	Name
1	14.244	BB	0.292	445.935	89.822	
2	18.954	BB	0.317	50.531	10.178	

- ***Dimethyl 3,4-bis(4-bromophenyl)cyclobutane-1,2-dicarboxylate, 25***

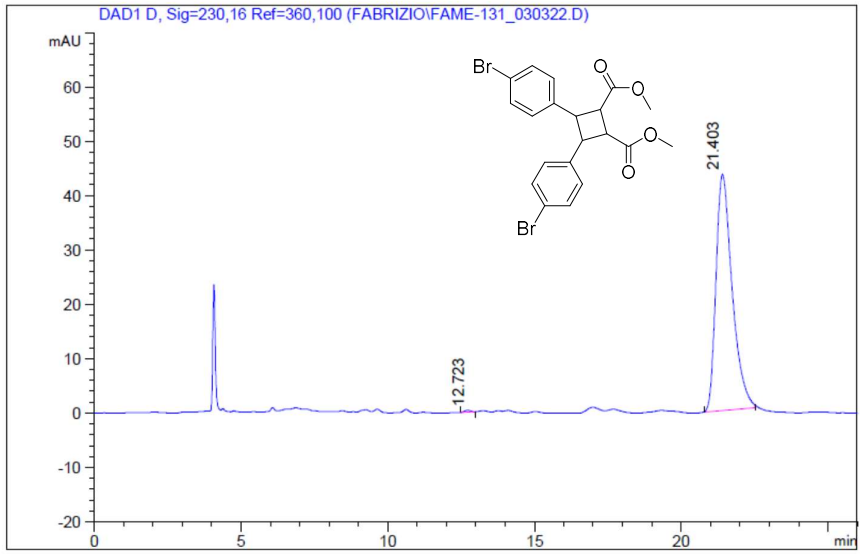
lux 3u cell.-1, 98:2 Hex/Ipa, 0.8 mL/min, P=55 bar



Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area	Area %	Name
1	12.691	BB	0.263	1646.960	48.315	
2	20.818	BB	0.503	1761.837	51.685	

lux 3u cell.-1, 98:2 Hex/Ipa, 0.8 mL/min, P=55 bar

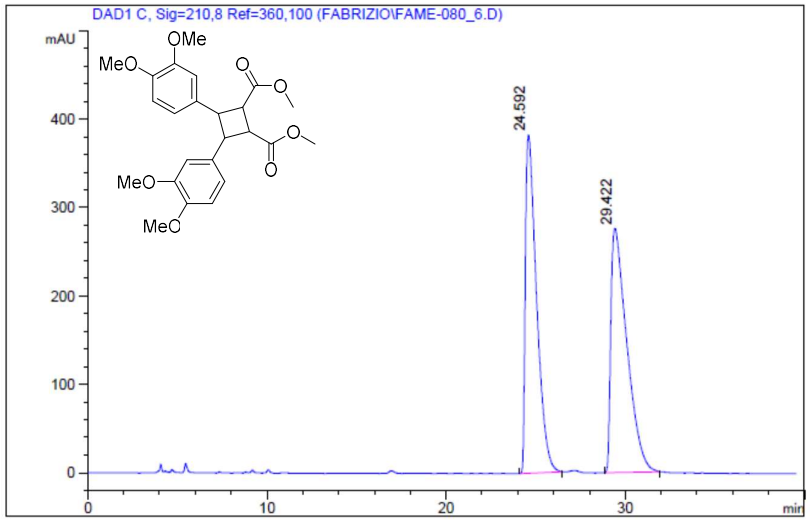


Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area	Area %	Name
1	12.723	MM	0.229	5.224	0.313	
2	21.403	BB	0.577	1666.410	99.687	

- ***Dimethyl 3,4-bis(3,4-dimethoxyphenyl)cyclobutane-1,2-dicarboxylate, 26***

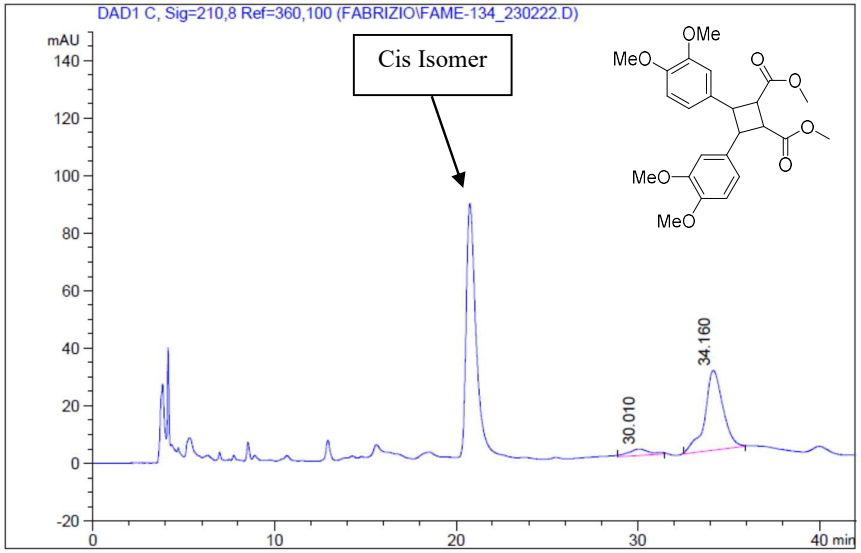
Lux 3u amylose-2, Hex:IPA 98:2, 0,8 ml/min, p=53 bar



Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area	Area %	Name
1	24.592	BB	0.653	16635.693	49.142	
2	29.422	BB	0.899	17216.623	50.858	

Lux 3u amyl.-2, 98:2 Hex/Ipa, 0.8 mL/min, P=2552 bar

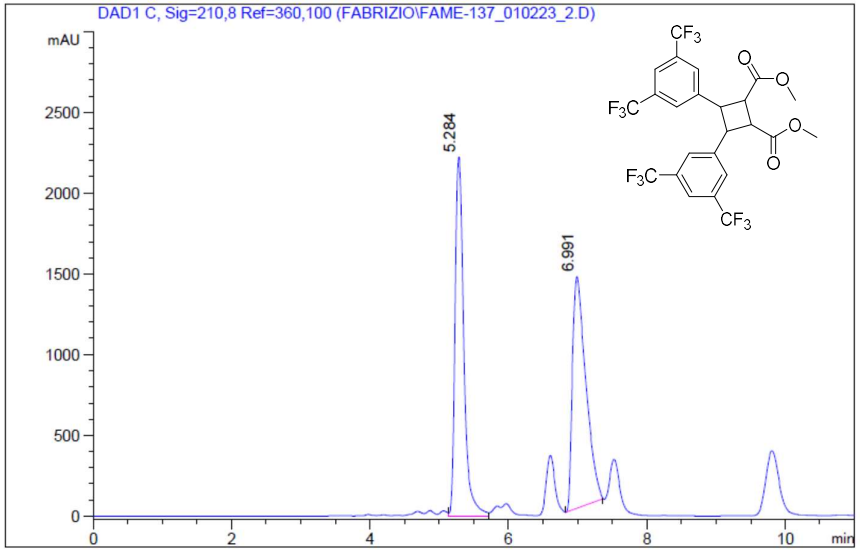


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area	Area %	Name
1	30.010	MM	1.368	186.297	8.837	
2	34.160	BB	0.918	1921.867	91.163	

- ***Dimethyl 3,4-bis(3,5-bis(trifluoromethyl)phenyl)cyclobutane-1,2-dicarboxylate, 27***

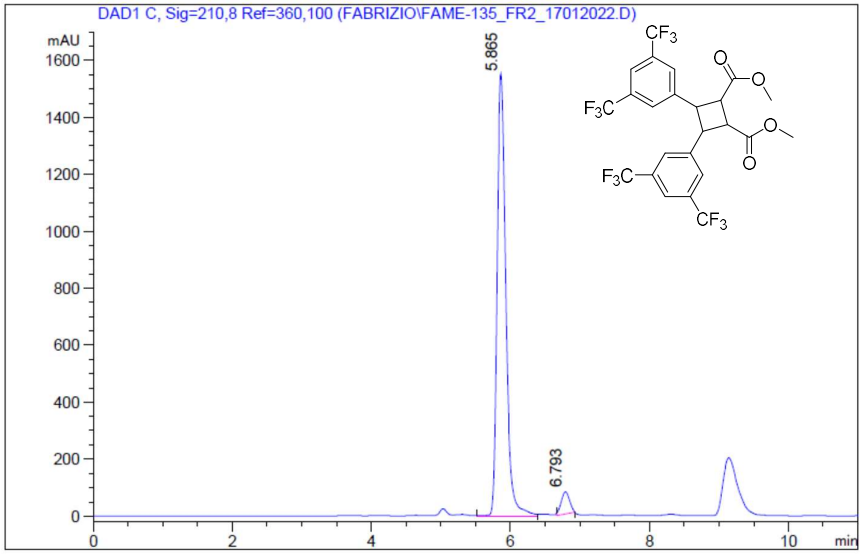
lux 3u cell.-1, 98:2 Hex/IPA, 0.8 mL/min P 50 bar



Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area	Area %	Name
1	5.284	VV	0.137	19535.533	50.334	
2	6.991	MM	0.224	19275.936	49.666	

Lux 3u cell-1, Hex:IPA 98:2, 0.8 ml/min, p=58 bar

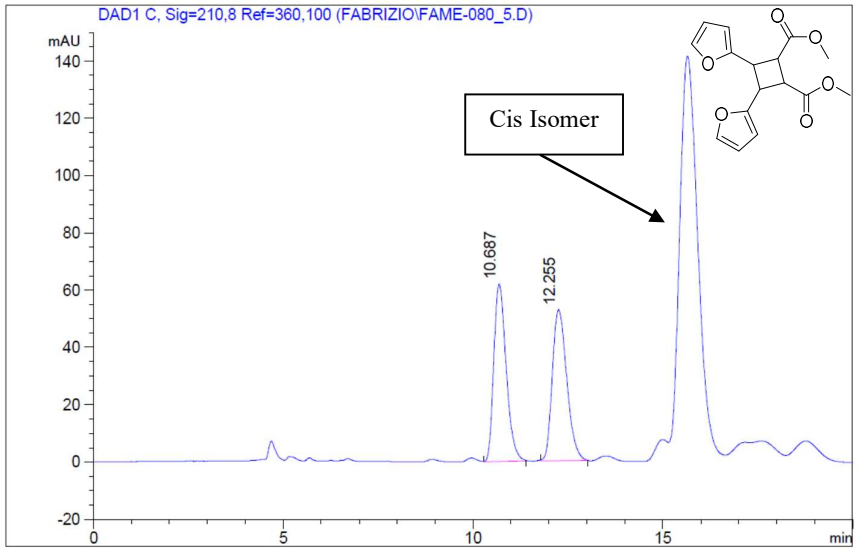


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area	Area %	Name
1	5.865	VV	0.134	13522.963	95.414	
2	6.793	MM	0.139	649.914	4.586	

- ***Dimethyl 3,4-di(furan-2-yl)cyclobutane-1,2-dicarboxylate, 28***

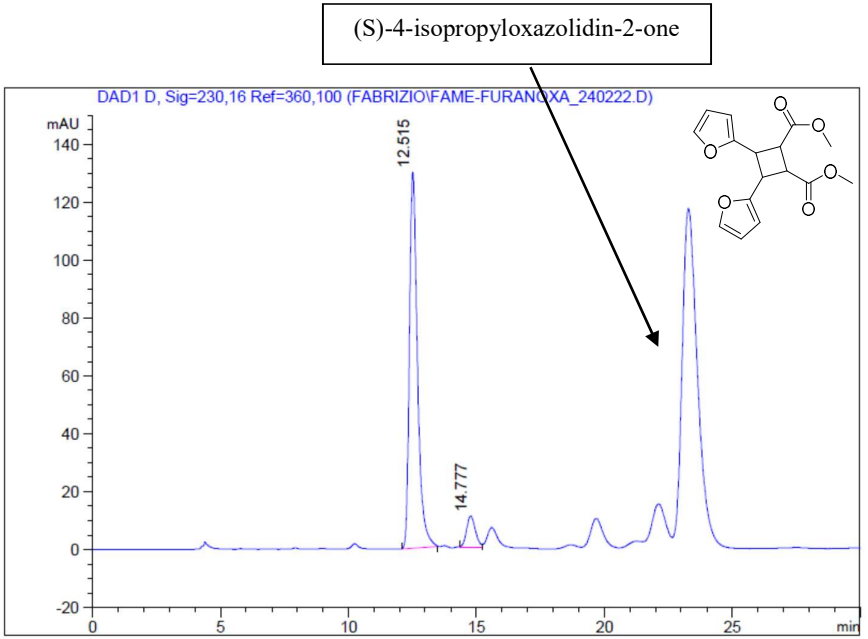
CHIRALCELL OD-H, Hex:IPA 98:2, 0,8 ml/min, p=35 bar



Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area	Area %	Name
1	10.687	VB	0.357	1407.603	50.123	
2	12.255	BB	0.416	1400.680	49.877	

chiralpack OD-H, 98:2 Hex/Ipa, 0.8 mL/min, P=31 bar

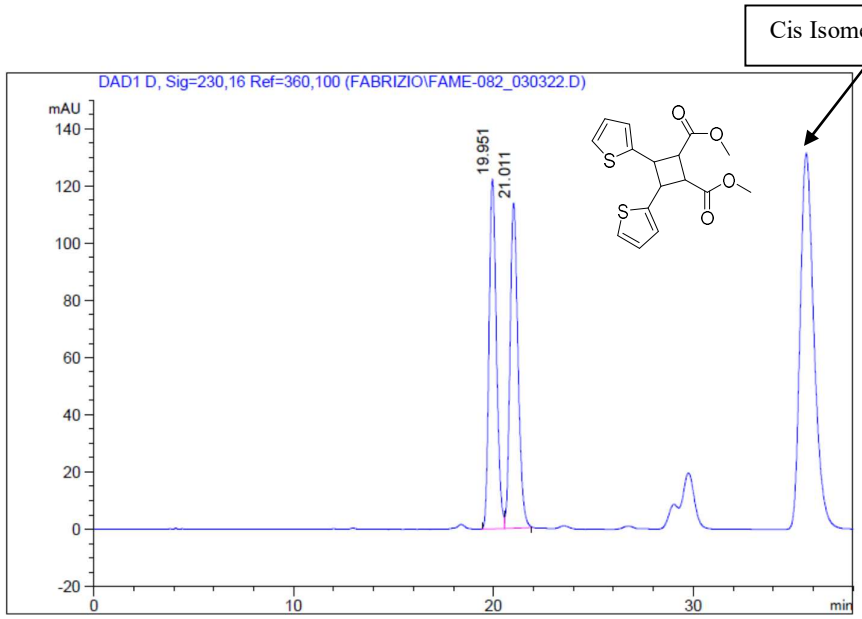


Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area	Area %	Name
1	12.515	BB	0.328	2836.081	91.703	
2	14.777	BV	0.371	256.612	8.297	

- ***Dimethyl 3,4-di(thiophen-2-yl)cyclobutane-1,2-dicarboxylate, 29***

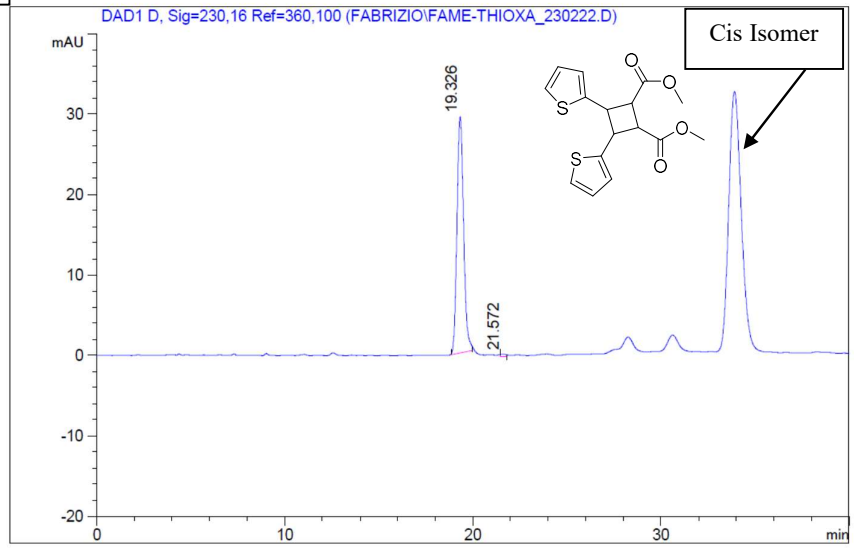
lux 3u cell.-1, 98:2 Hex/Ipa, 0.8 mL/min, P=55 bar



Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area	Area %	Name
1	19.951	BV	0.397	3151.989	49.879	
2	21.011	VB	0.429	3167.222	50.121	

Lux 3u cell-1, 98:2 Hex/Ipa, 0.8 mL/min, P=53 bar

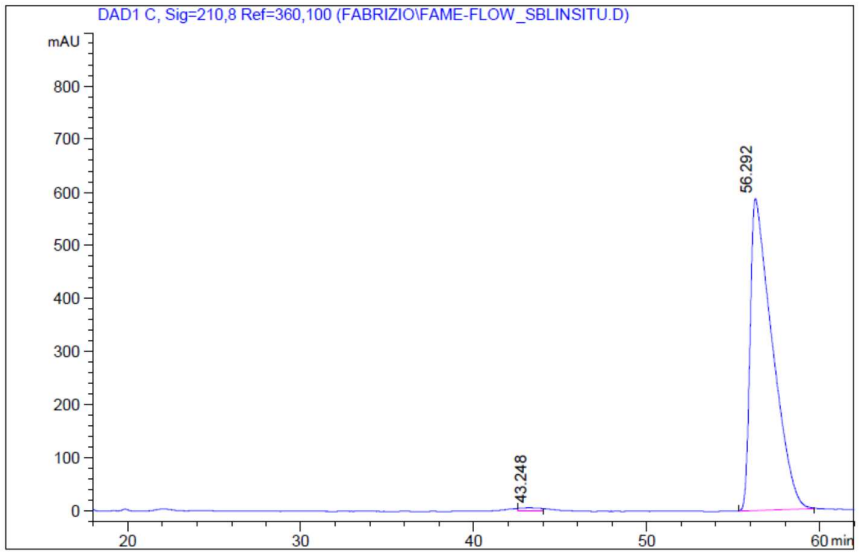


Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area	Area %	Name
1	19.326	BB	0.380	718.116	99.158	
2	21.572	MM	0.324	6.097	0.842	

- ***Dimethyl 3,4-diphenylcyclobutane-1,2-dicarboxylate 4 from (S)-3-cinnamoyl-4-isopropyloxazolidin-2-one 2d***
  In-flow cyclisation – optimised conditions for chiral auxiliary removal

Lux 5u cell.-3, 98:2 Hex/IPA, 0.8 mL/min, P=28 bar

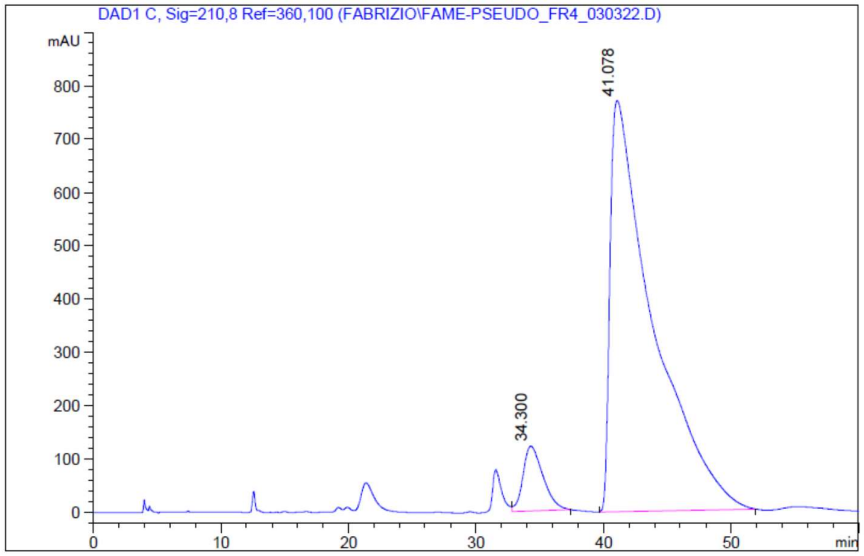


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RT [min]	Type	Width [min]	Area	Area %	Name
1	43.248	MM	1.400	525.096	0.997	
2	56.292	BB	1.201	52133.574	99.003	

- ***Dimethyl 3,4-diphenylcyclobutane-1,2-dicarboxylate 4 from (S)-3-cinnamoyl-4-isopropyloxazolidin-2-one 2d***
  Two step Flow/Batch reaction

lux 5u cell.-3, 98:2 Hex/Ipa, 0.8 mL/min, P=31 bar

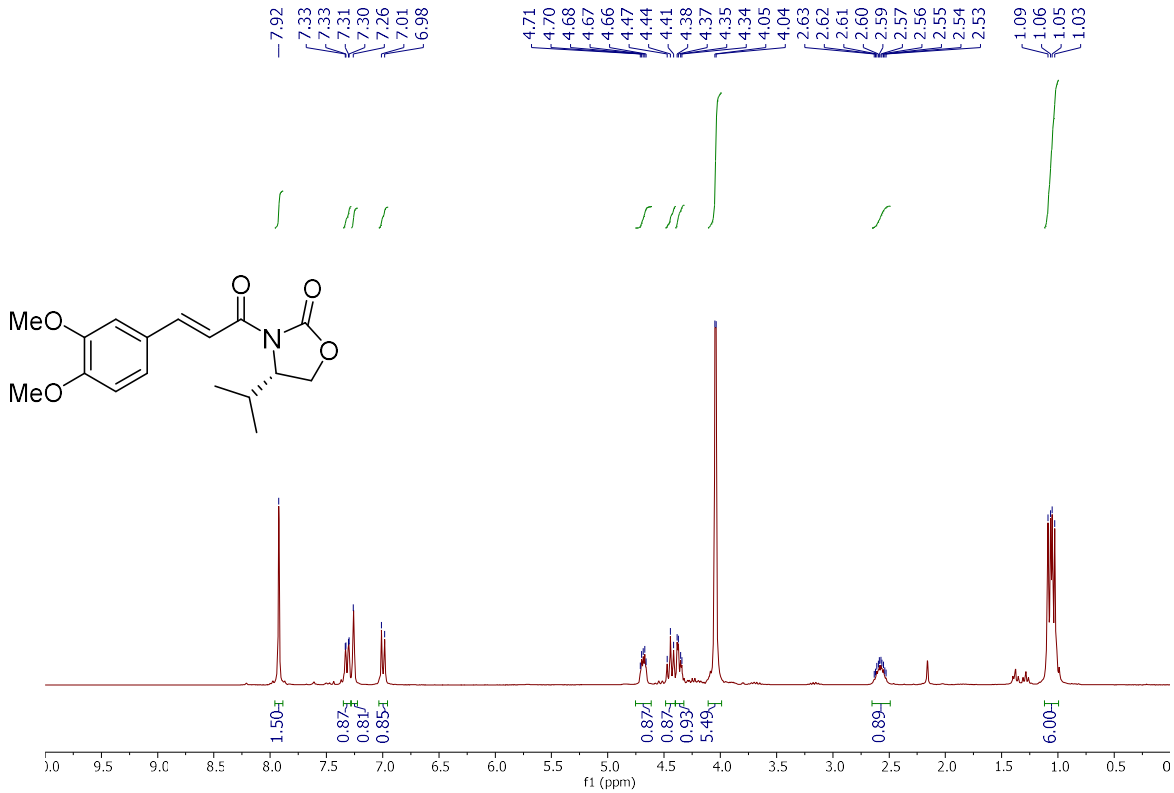


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

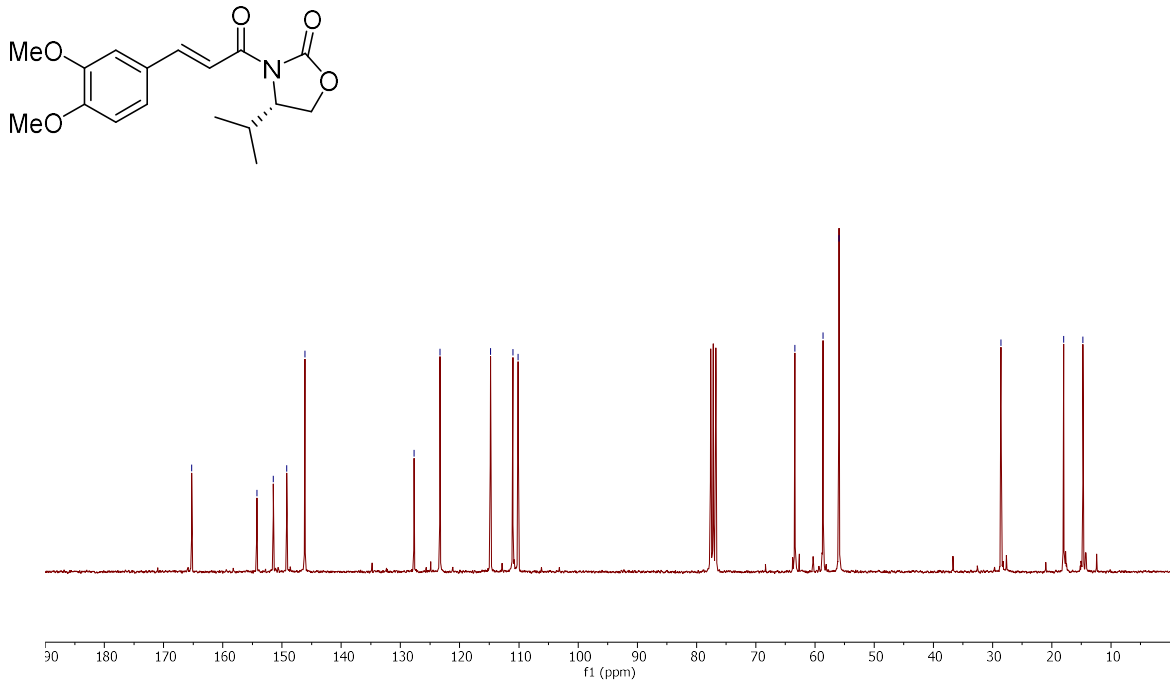
Peak #	RT [min]	Type	Width [min]	Area	Area %	Name
1	34.300	BB	1.465	13376.093	6.918	
2	41.078	BB	2.937	179985.938	93.082	

NMR SPECTRA

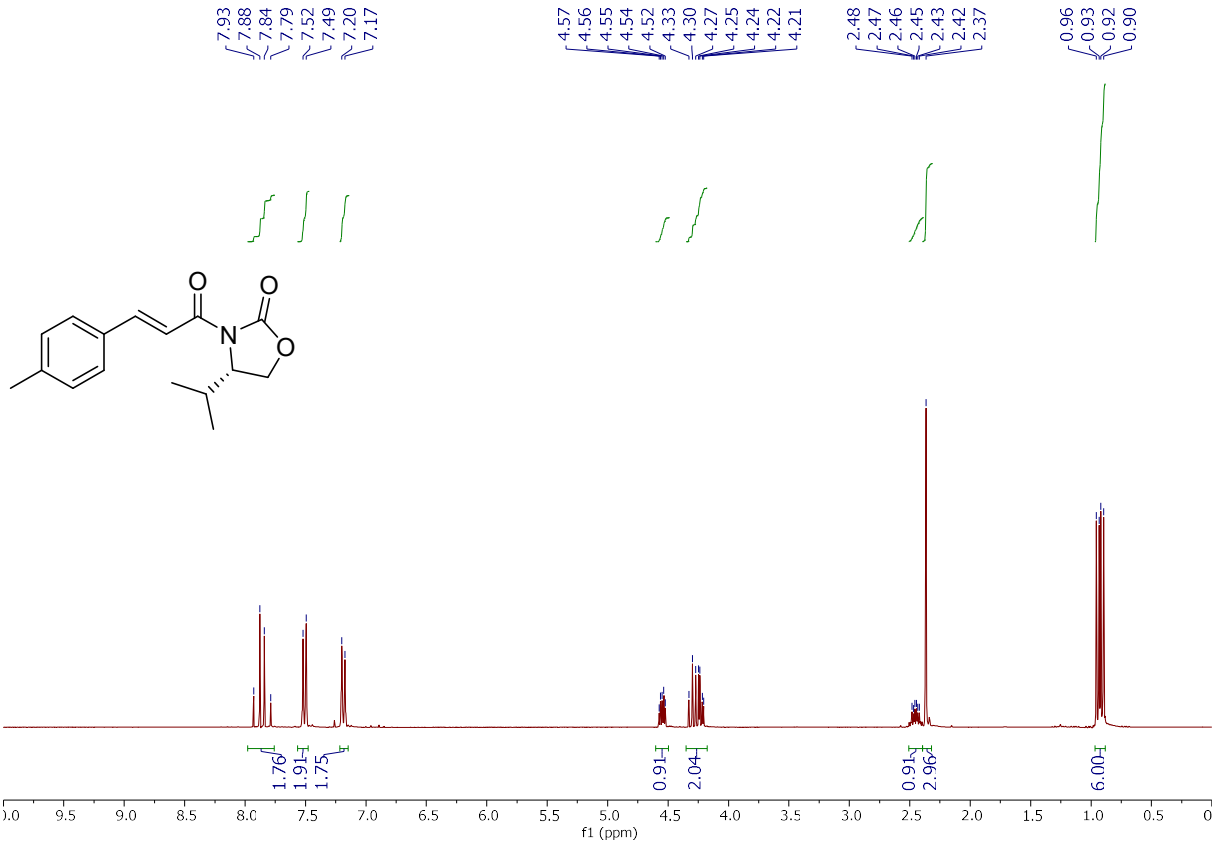
^{1}HNMR, 300 MHz, $CDCl_3$, **6**



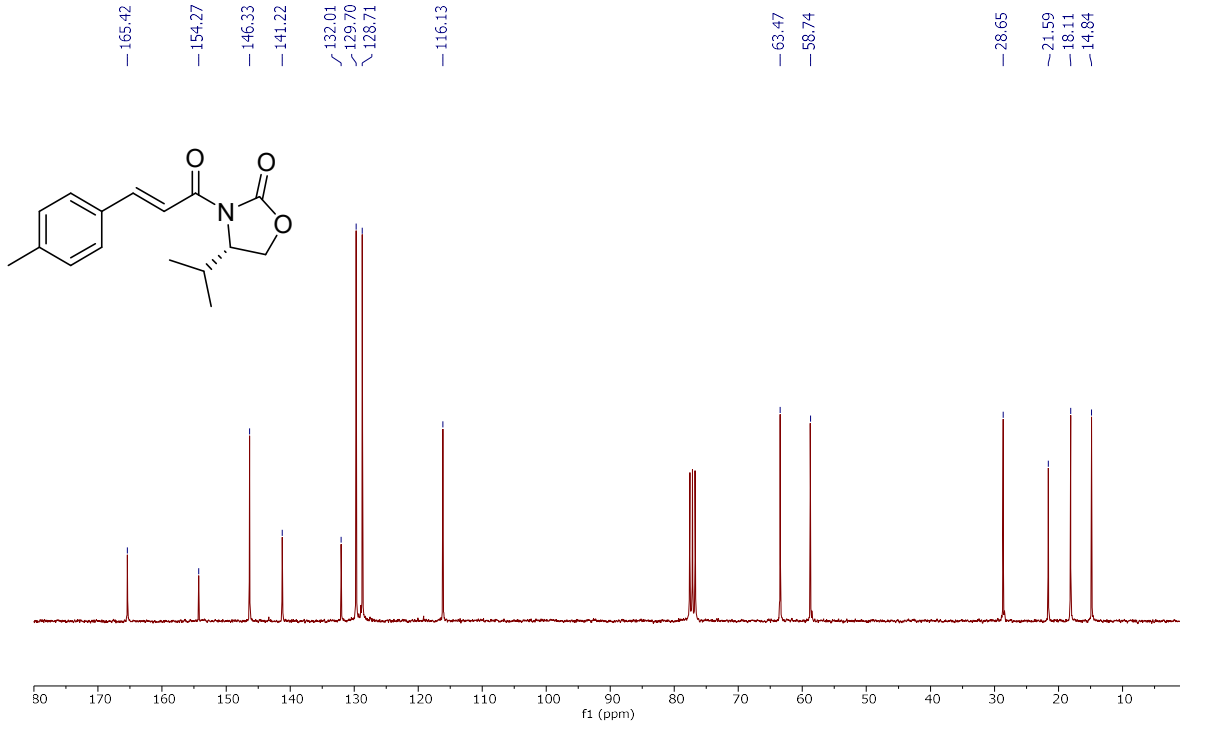
^{13}CNMR, 75 MHz, $CDCl_3$, **6**



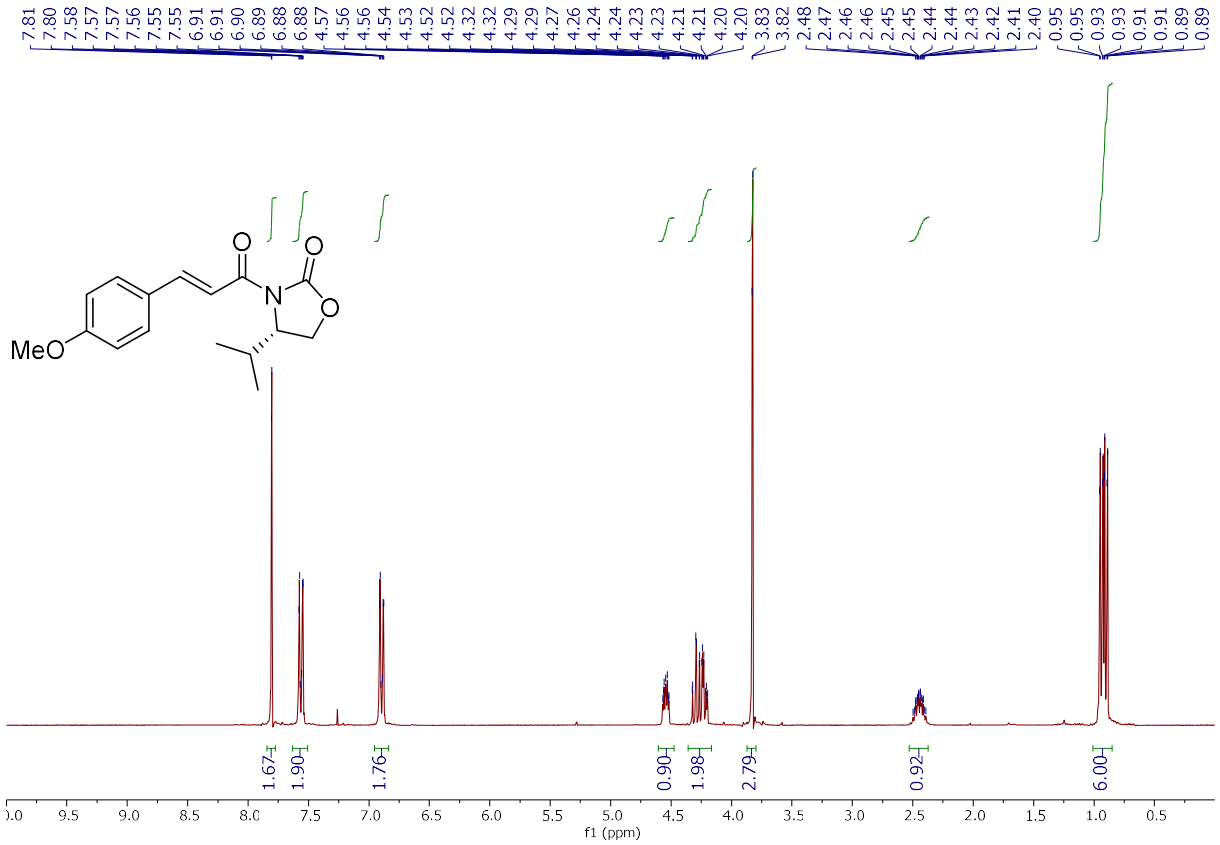
^{1}HNMR, 300 MHz, $CDCl_3$, **7**



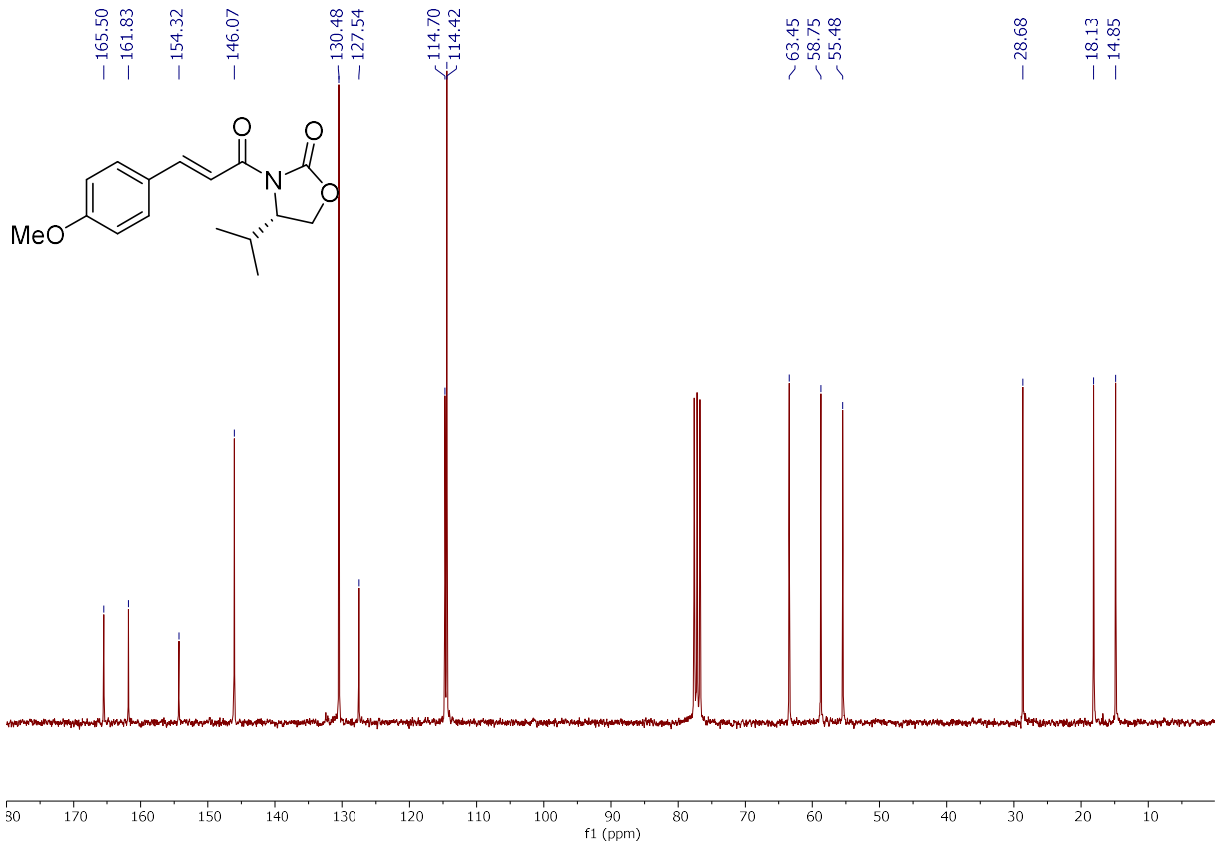
^{13}CNMR, 75 MHz, $CDCl_3$, **7**



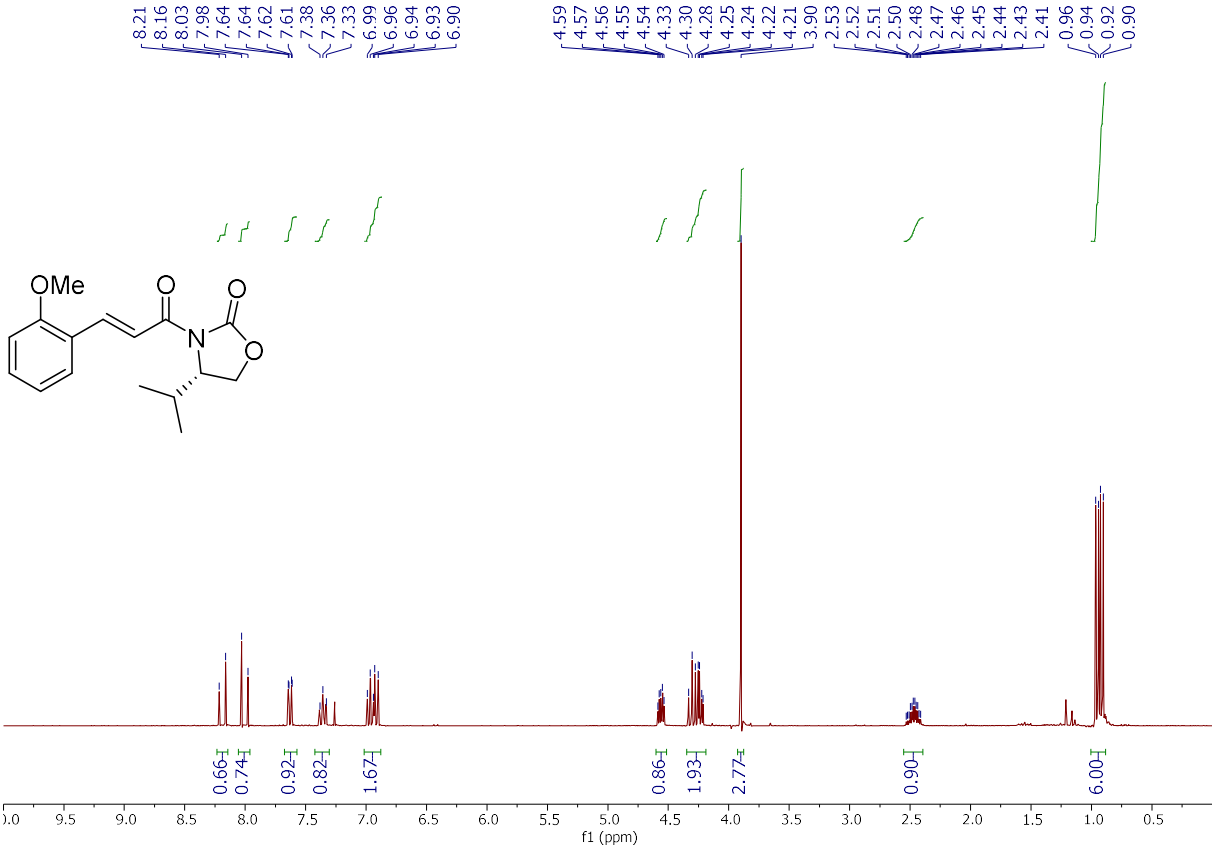
[1]HNMR, 300 MHz, $CDCl_3$, **8**



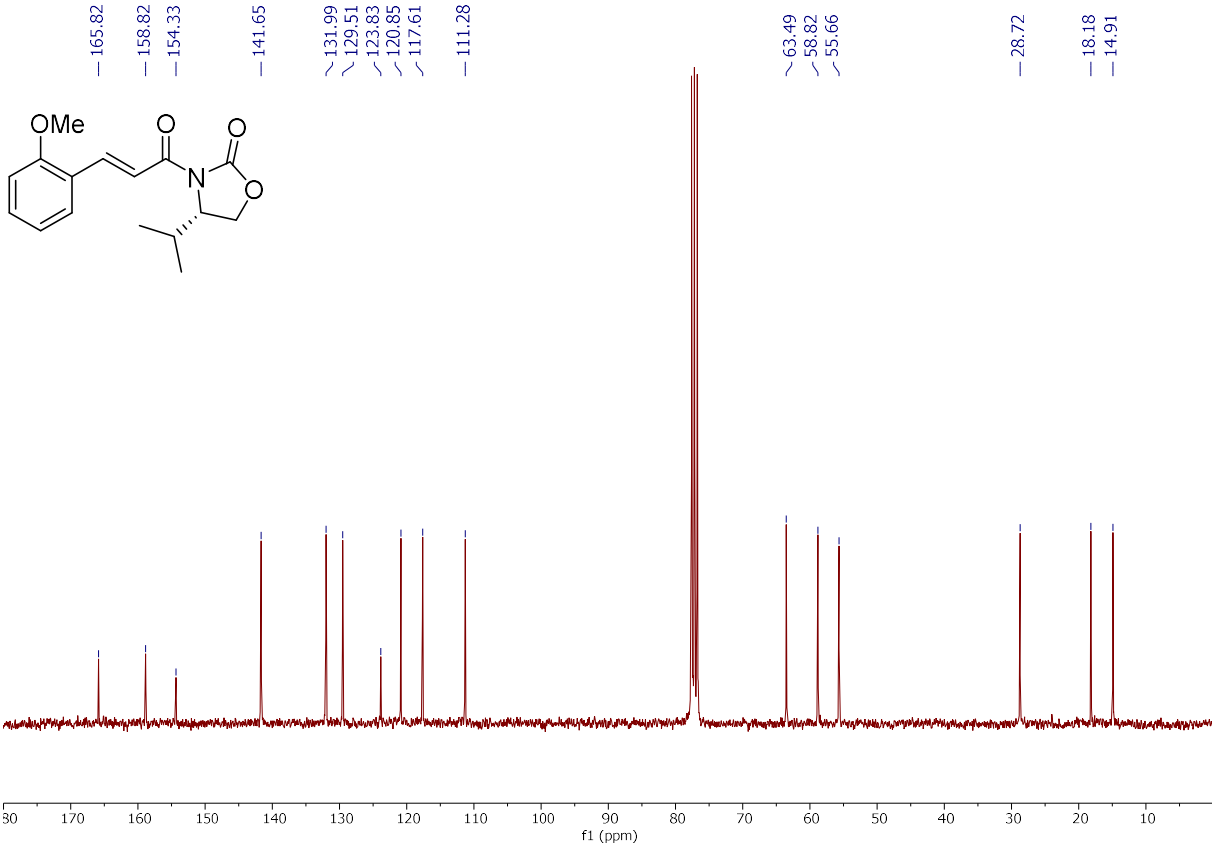
[13]CNMR, 75 MHz, $CDCl_3$, **8**



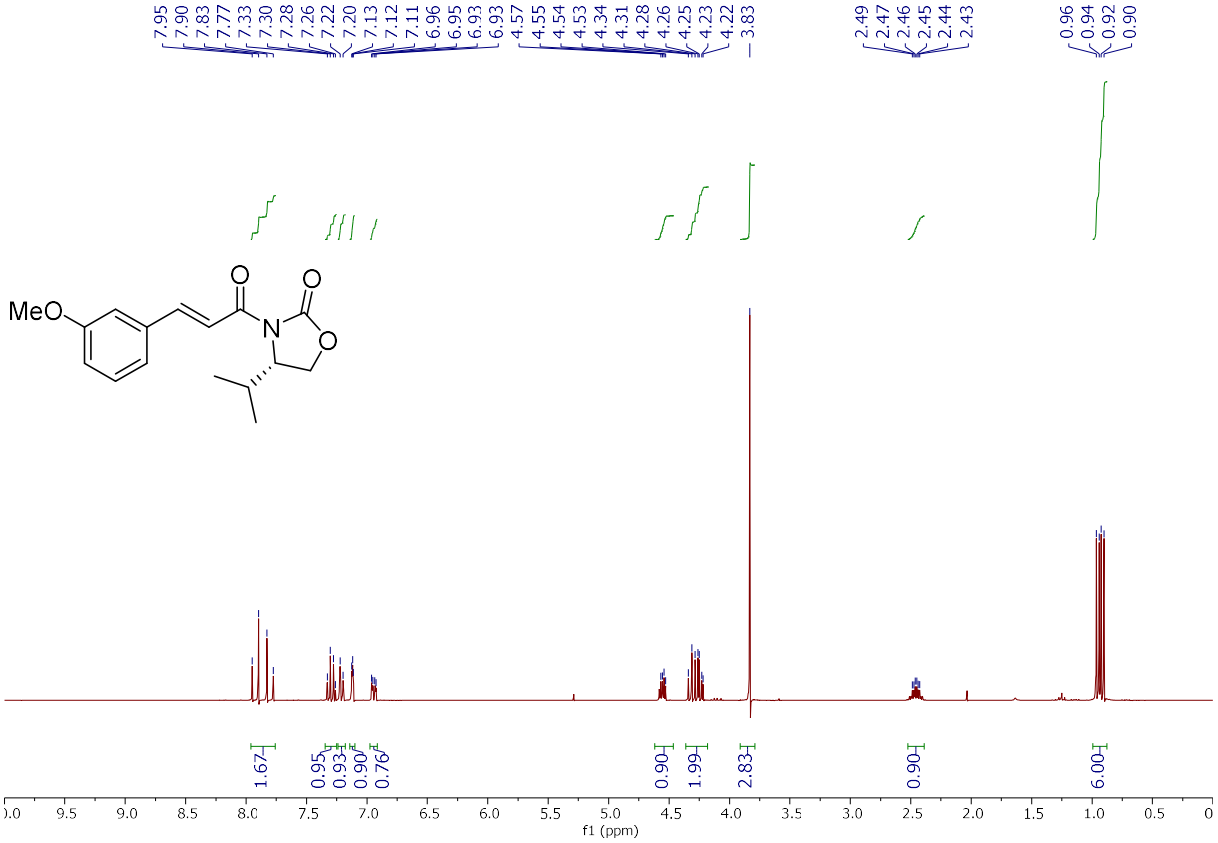
^{1}HNMR, 300 MHz, $CDCl_3$, **9**



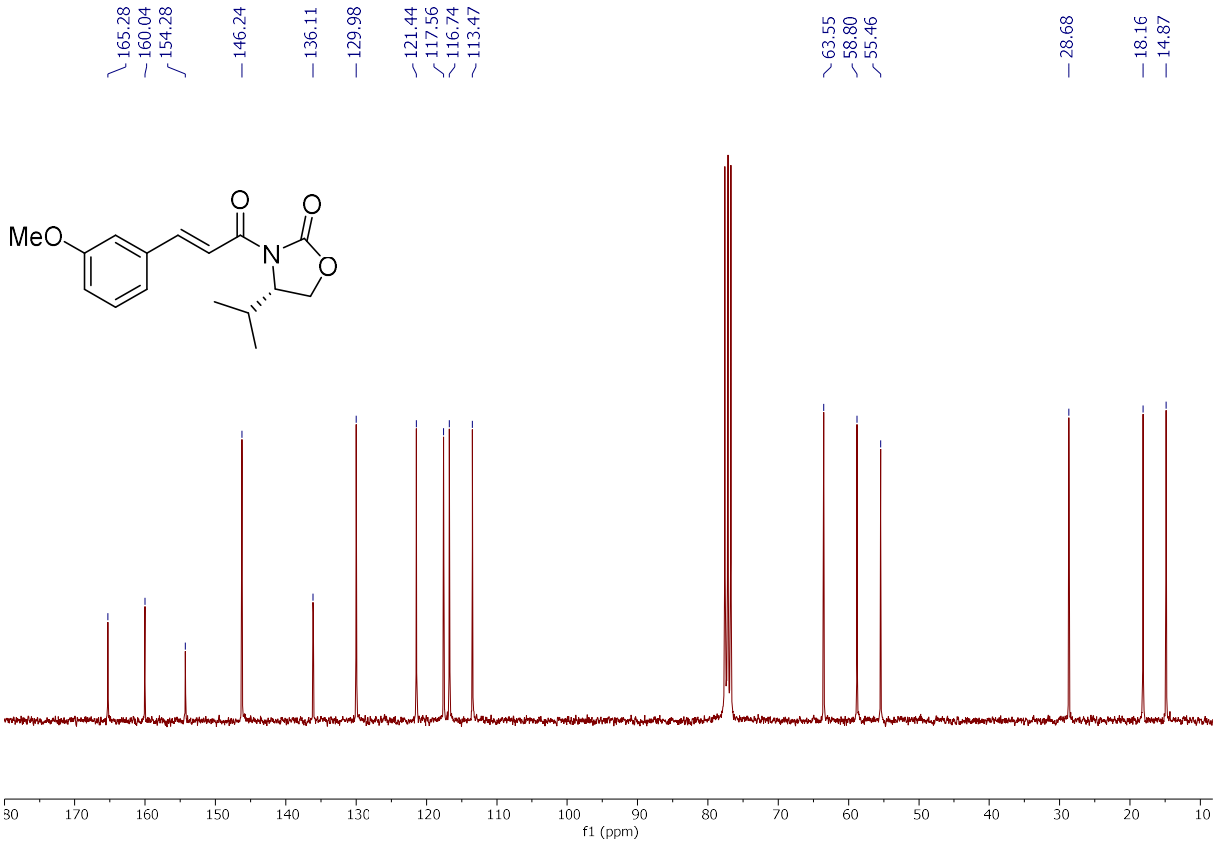
^{13}CNMR, 75 MHz, $CDCl_3$, **9**



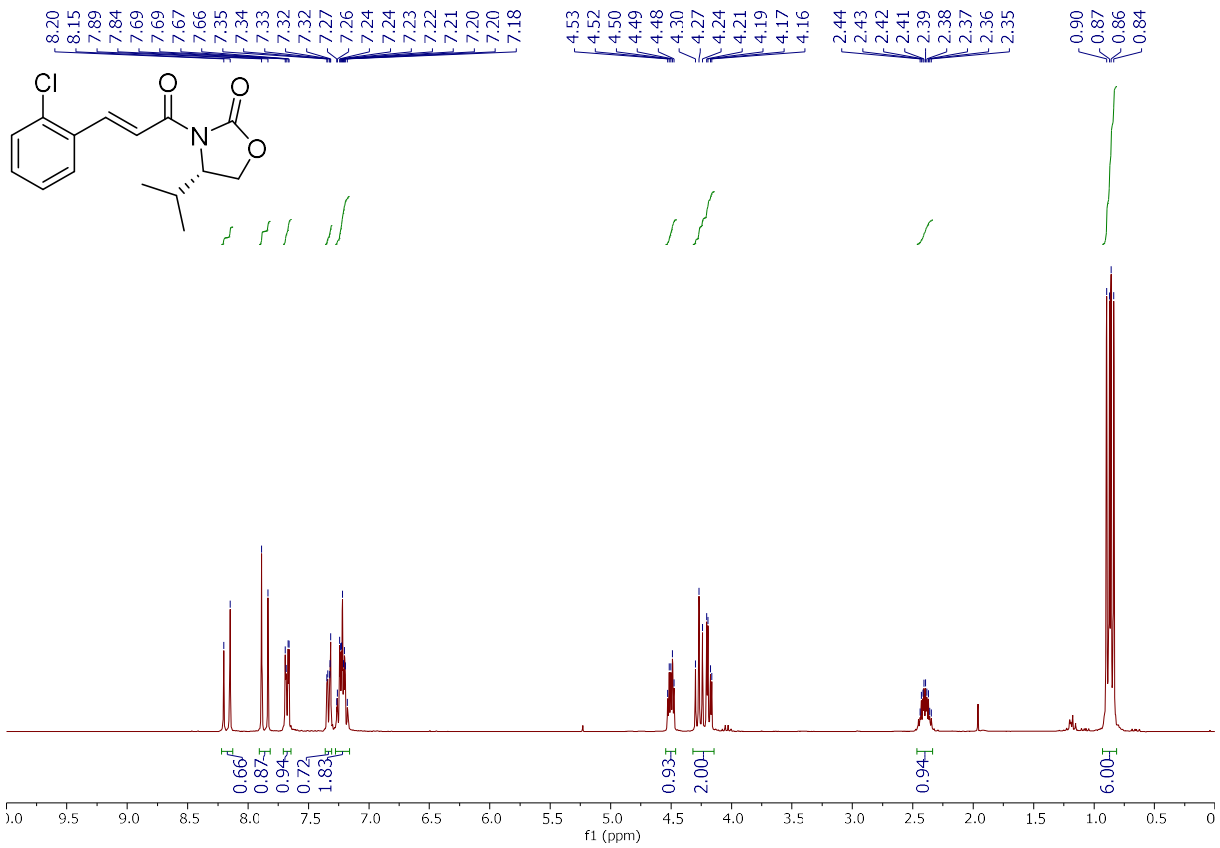
^{1}HNMR, 300 MHz, $CDCl_3$, **10**



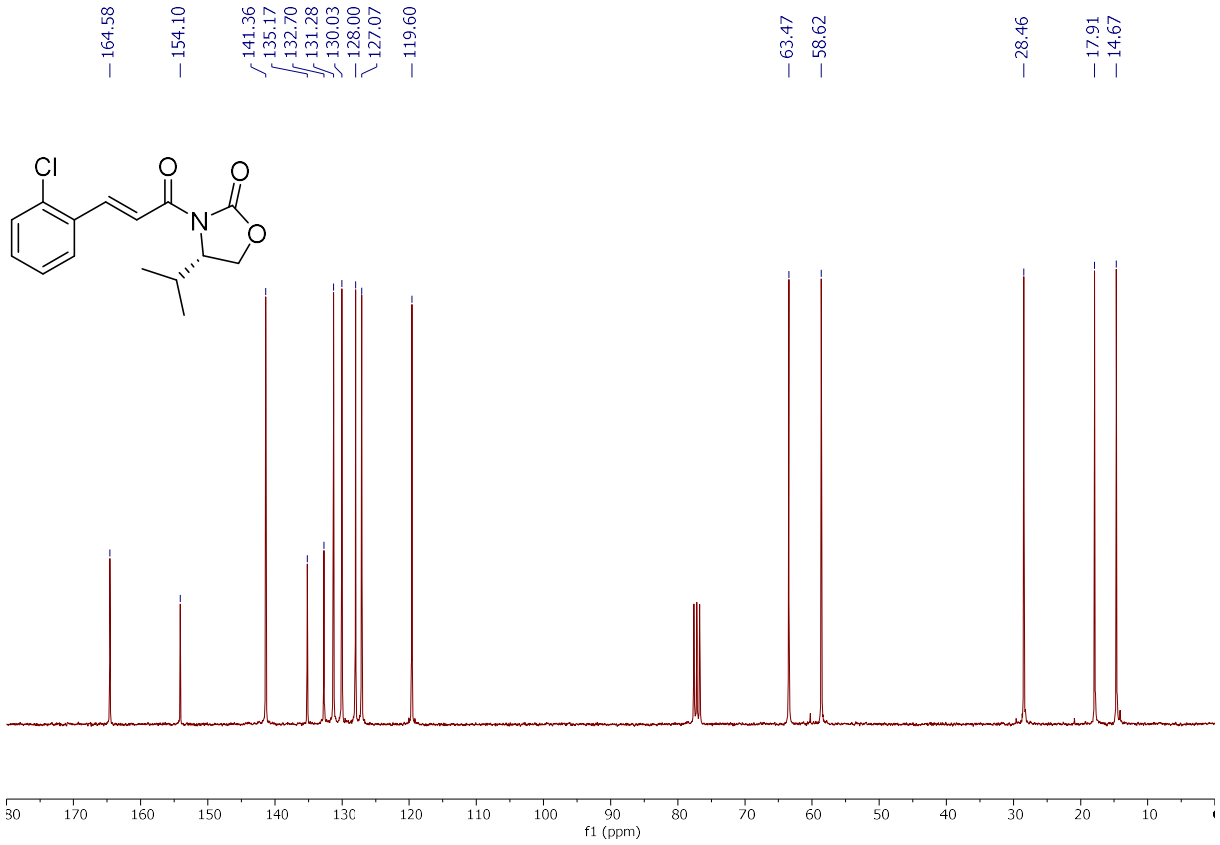
^{13}CNMR, 75 MHz, $CDCl_3$, **10**



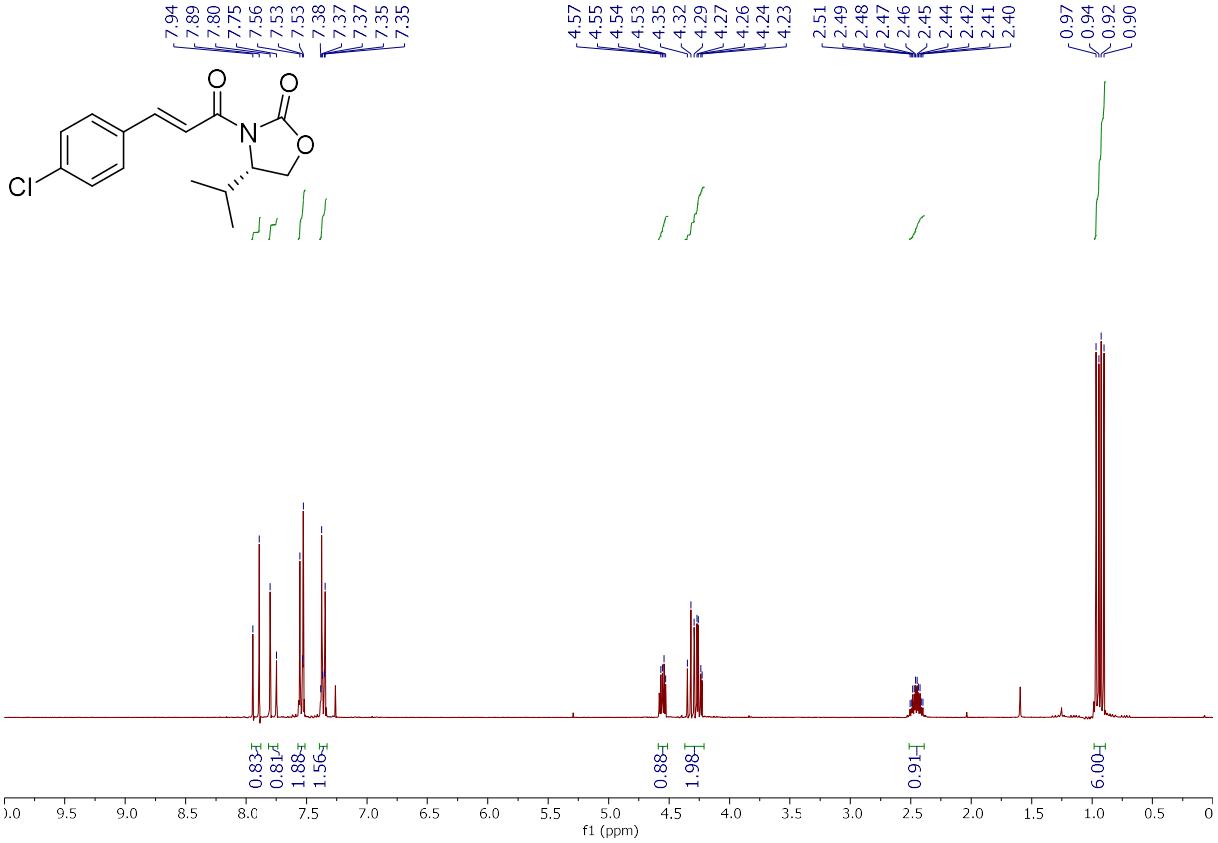
[1]HNMR, 300 MHz, $CDCl_3$, **11**



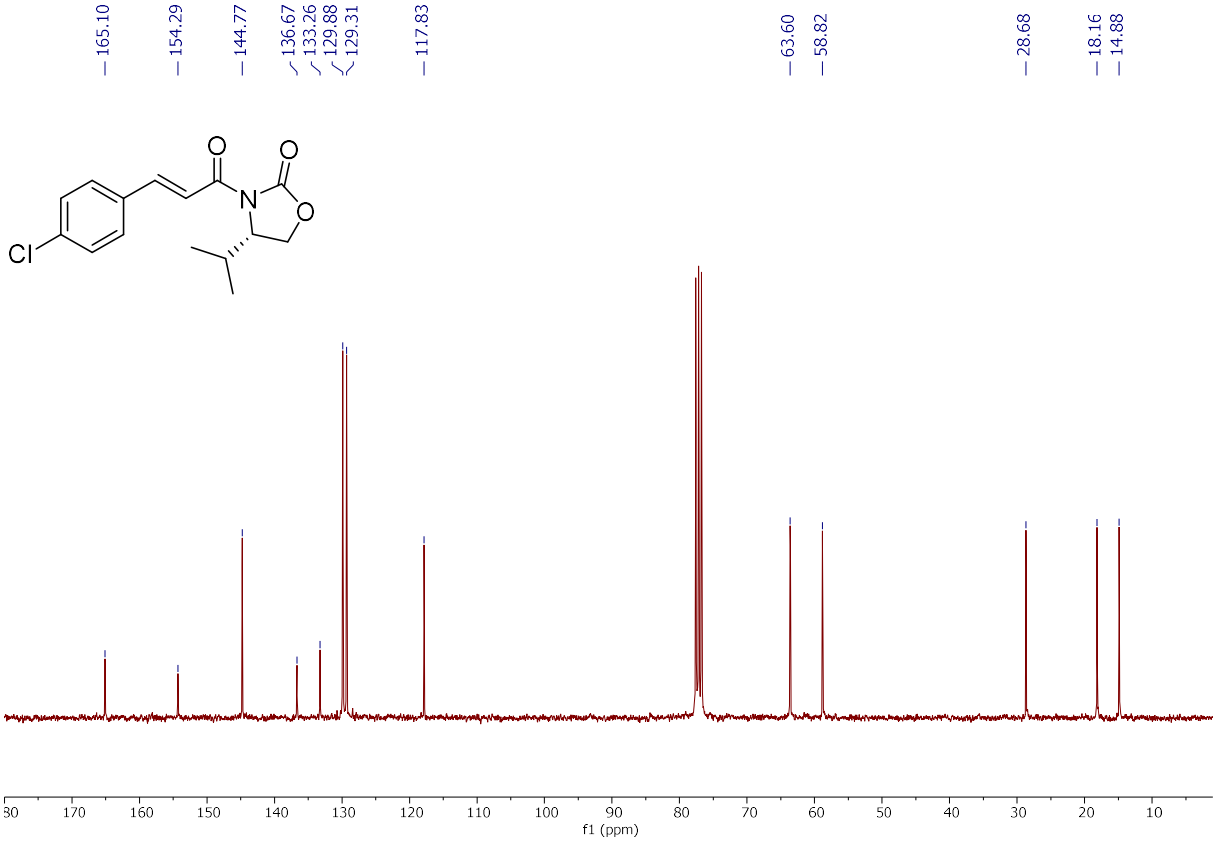
[13]CNMR, 75 MHz, $CDCl_3$, **11**



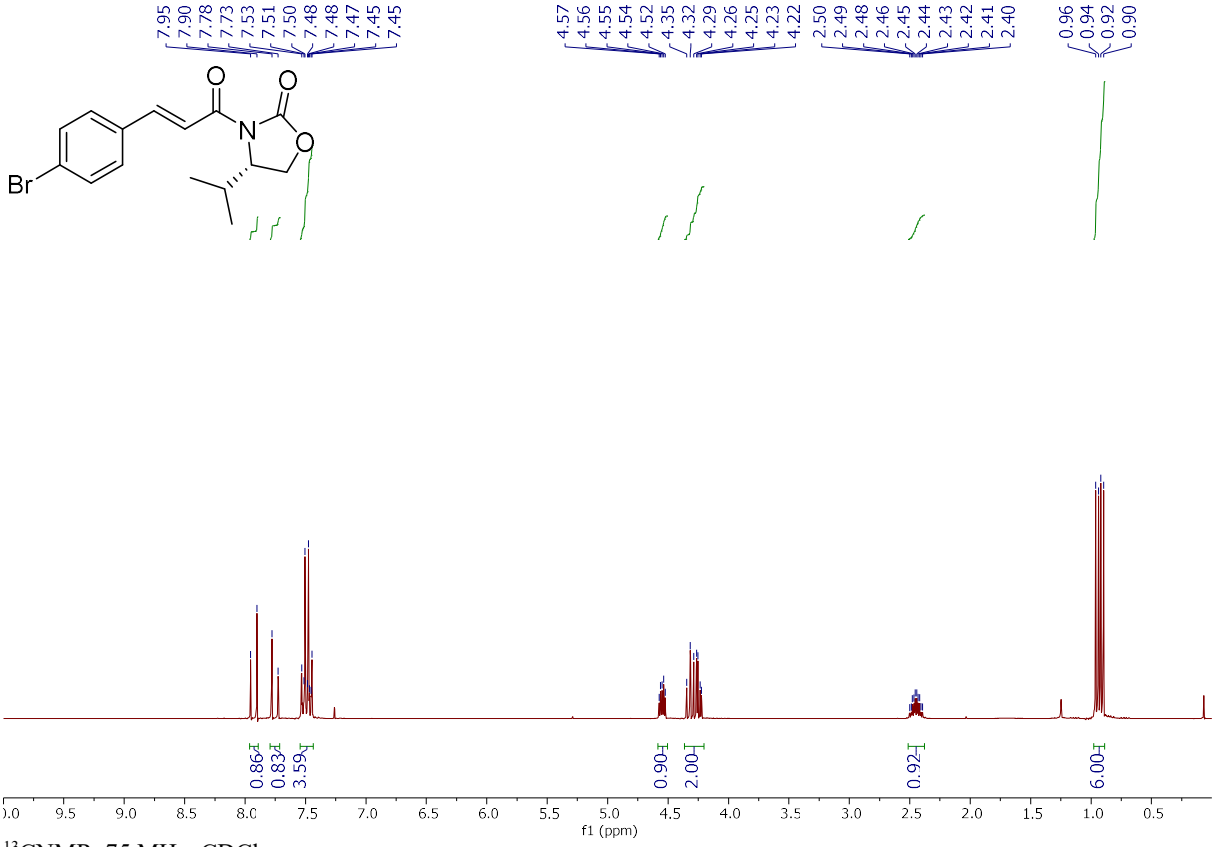
[1]HNMR, 300 MHz, $CDCl_3$, **12**



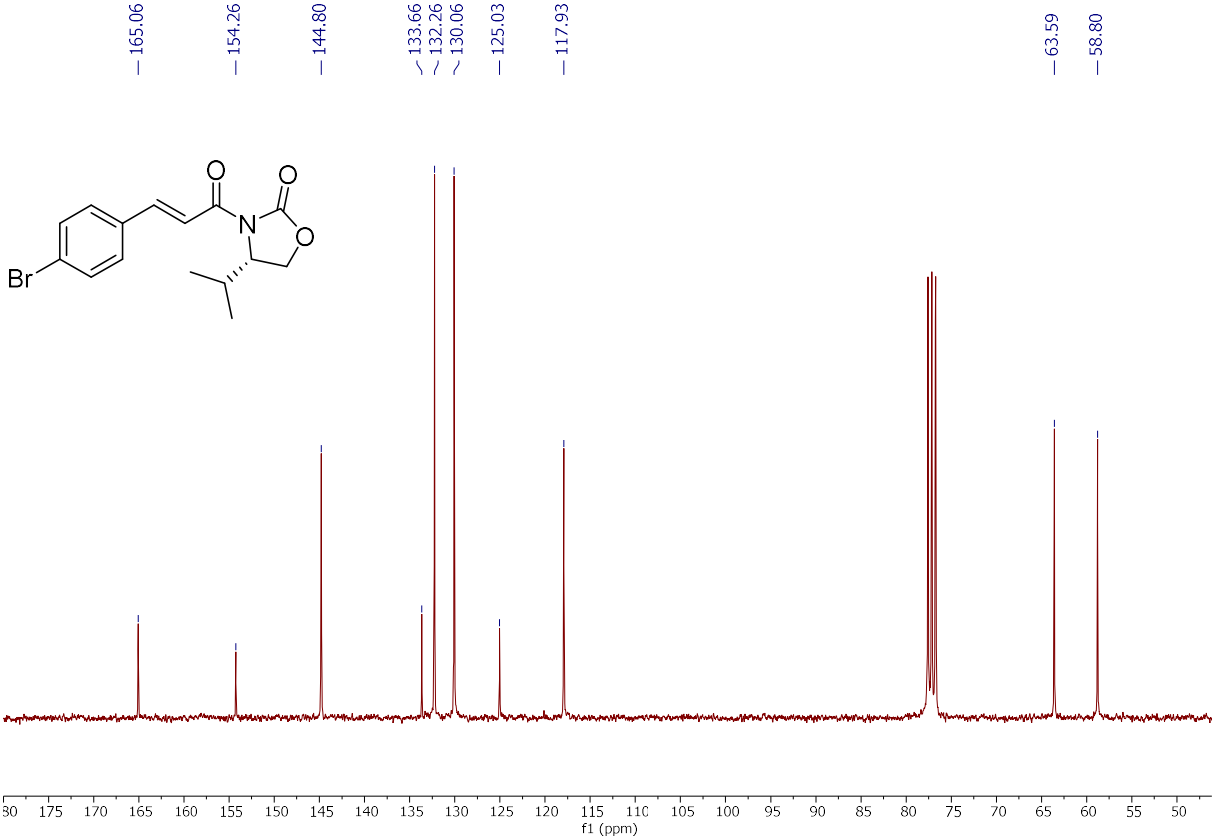
[13]CNMR, 75 MHz, $CDCl_3$, **12**



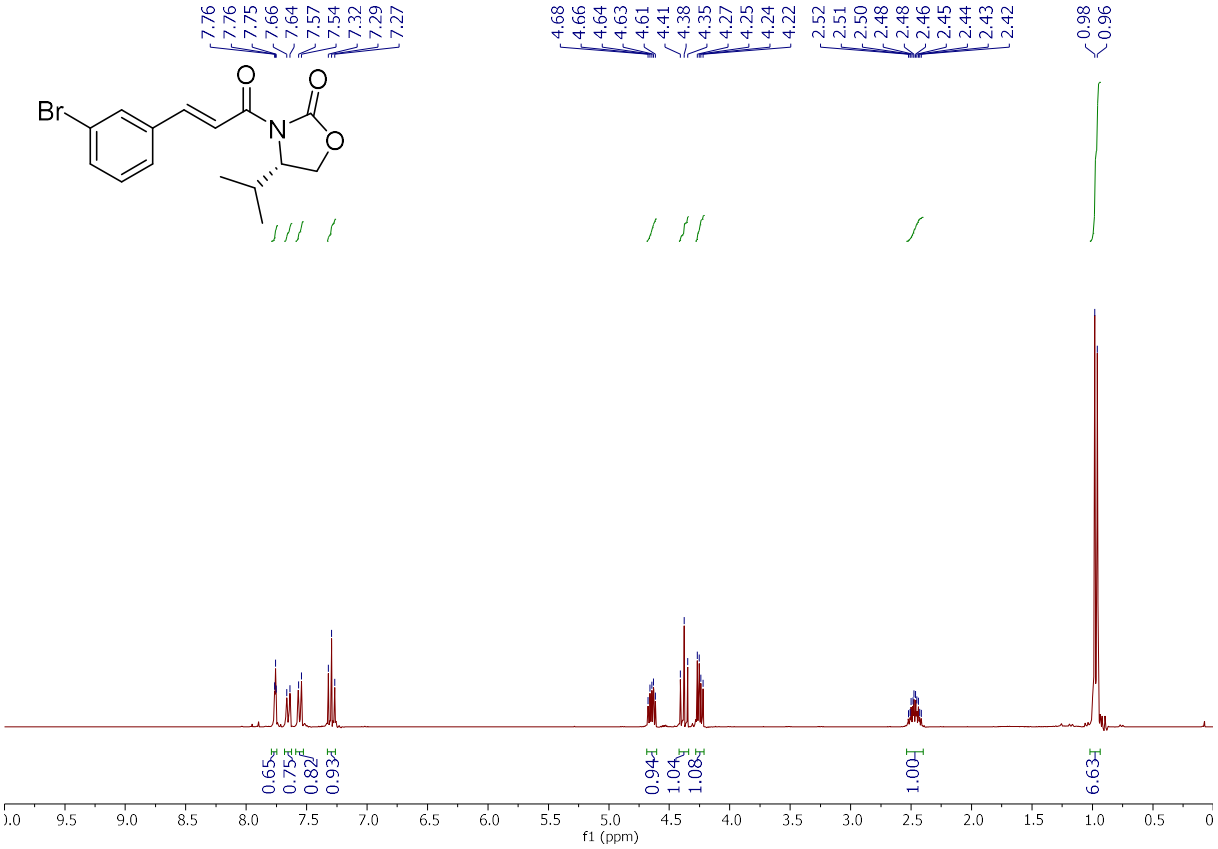
^{1}HNMR, 300 MHz, $CDCl_3$, **13**



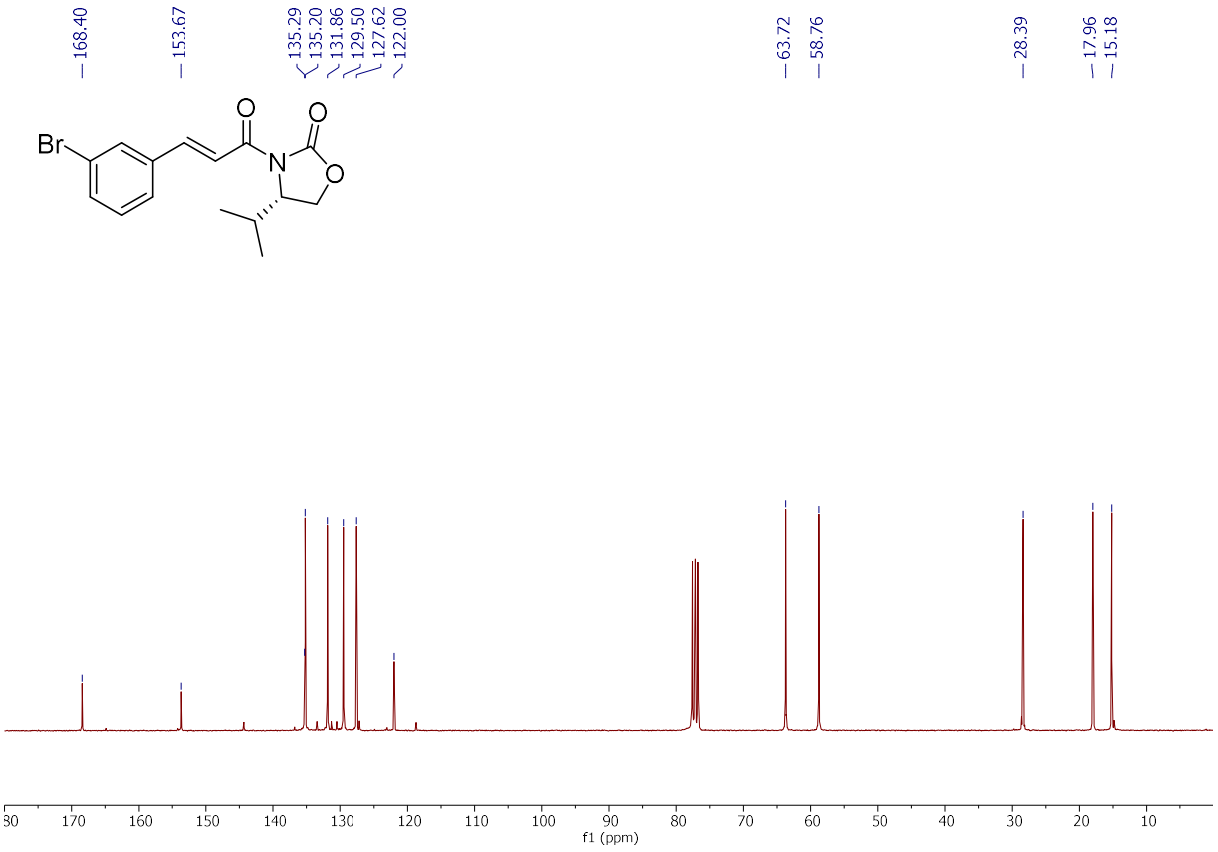
^{13}CNMR, 75 MHz, $CDCl_3$



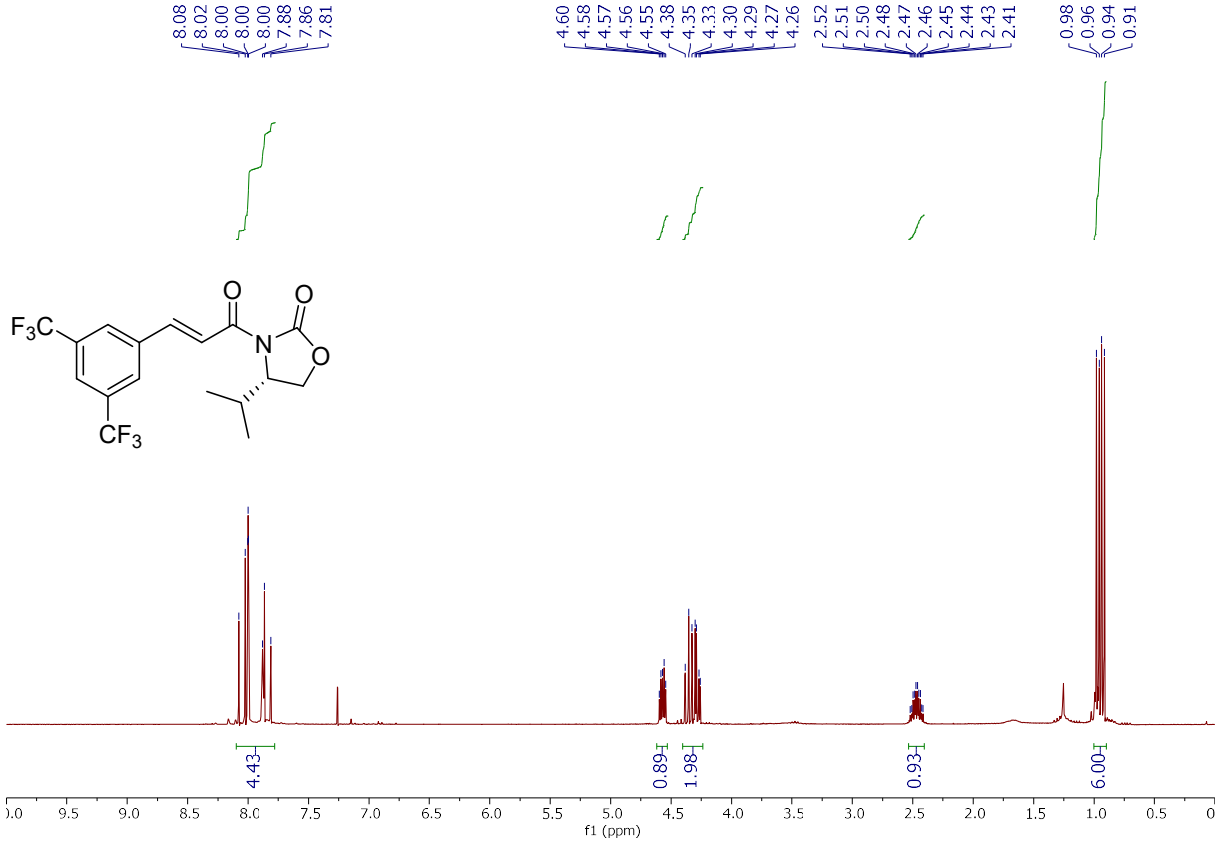
^{1}HNMR, 300 MHz, $CDCl_3$, **14**



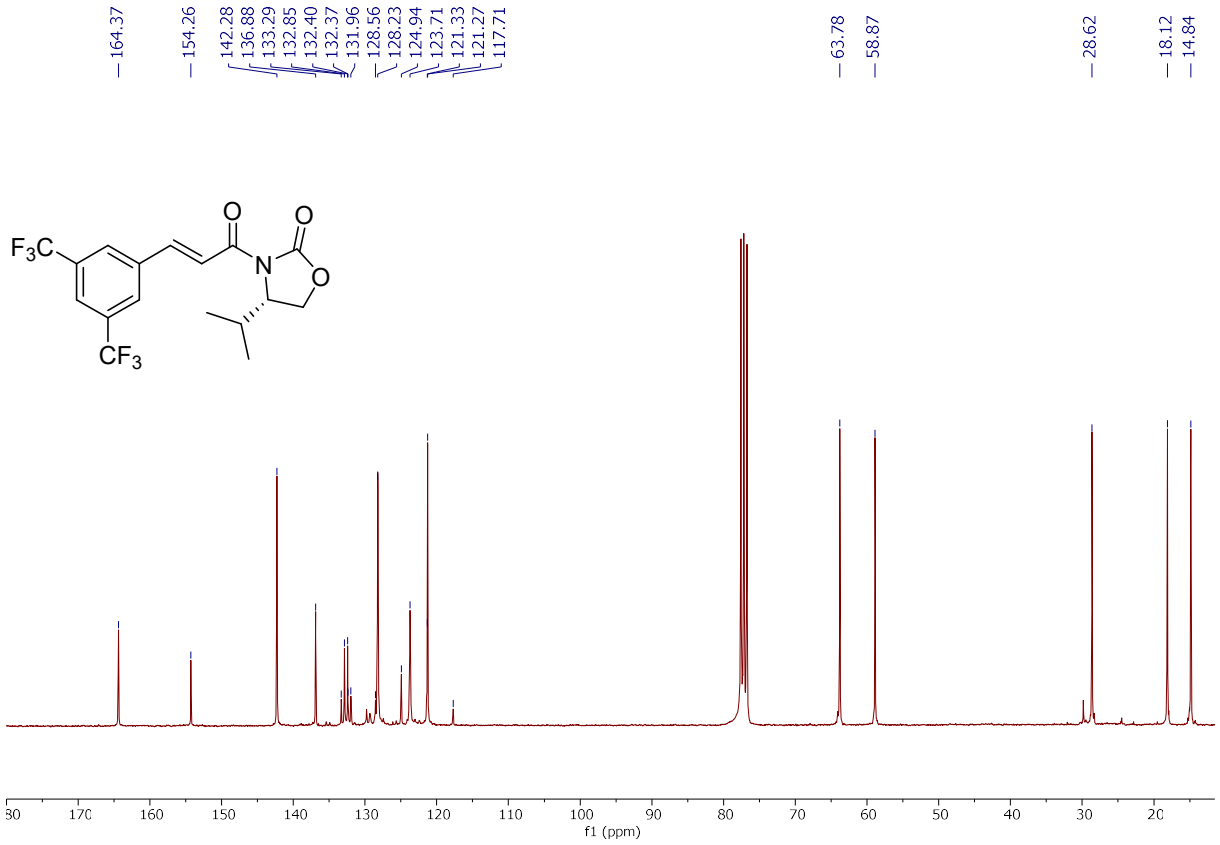
^{13}CNMR, 75 MHz, $CDCl_3$, **14**



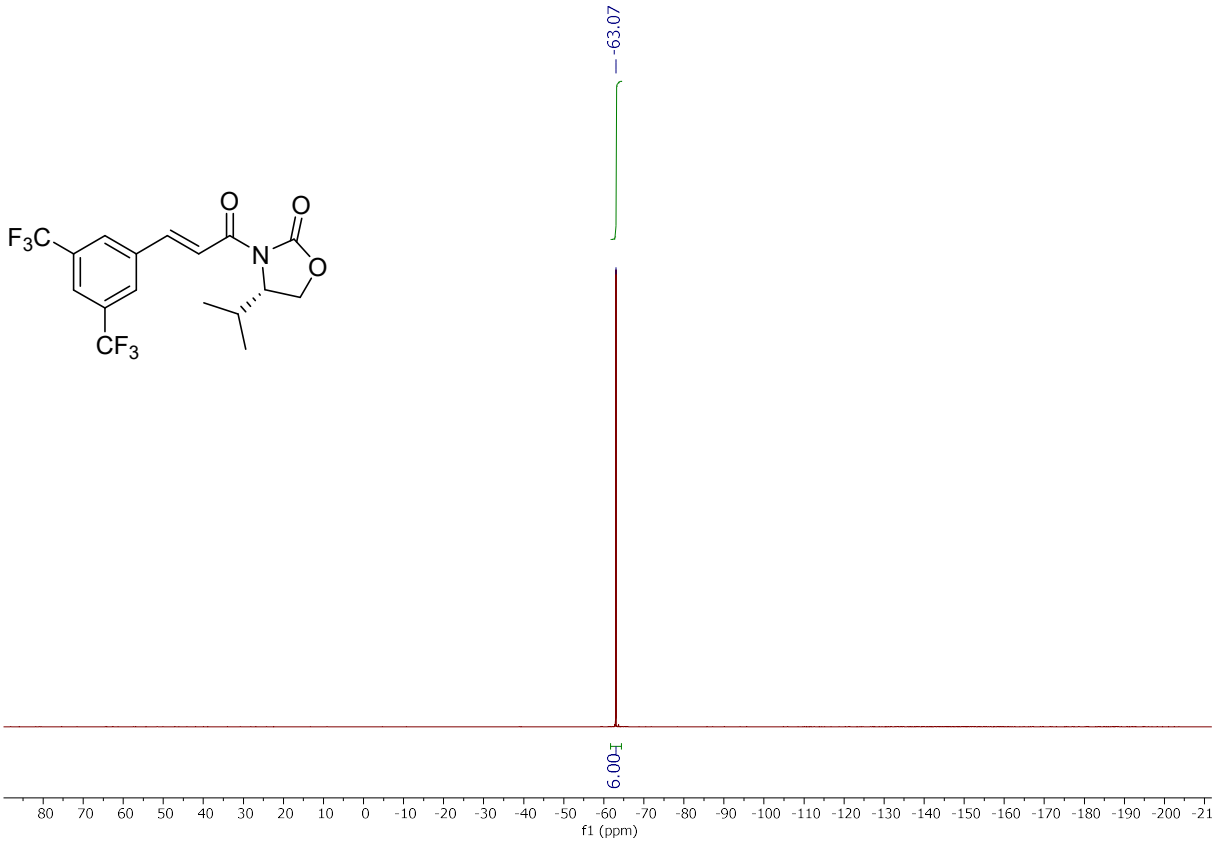
^{1}HNMR, 300 MHz, $CDCl_3$, **15**



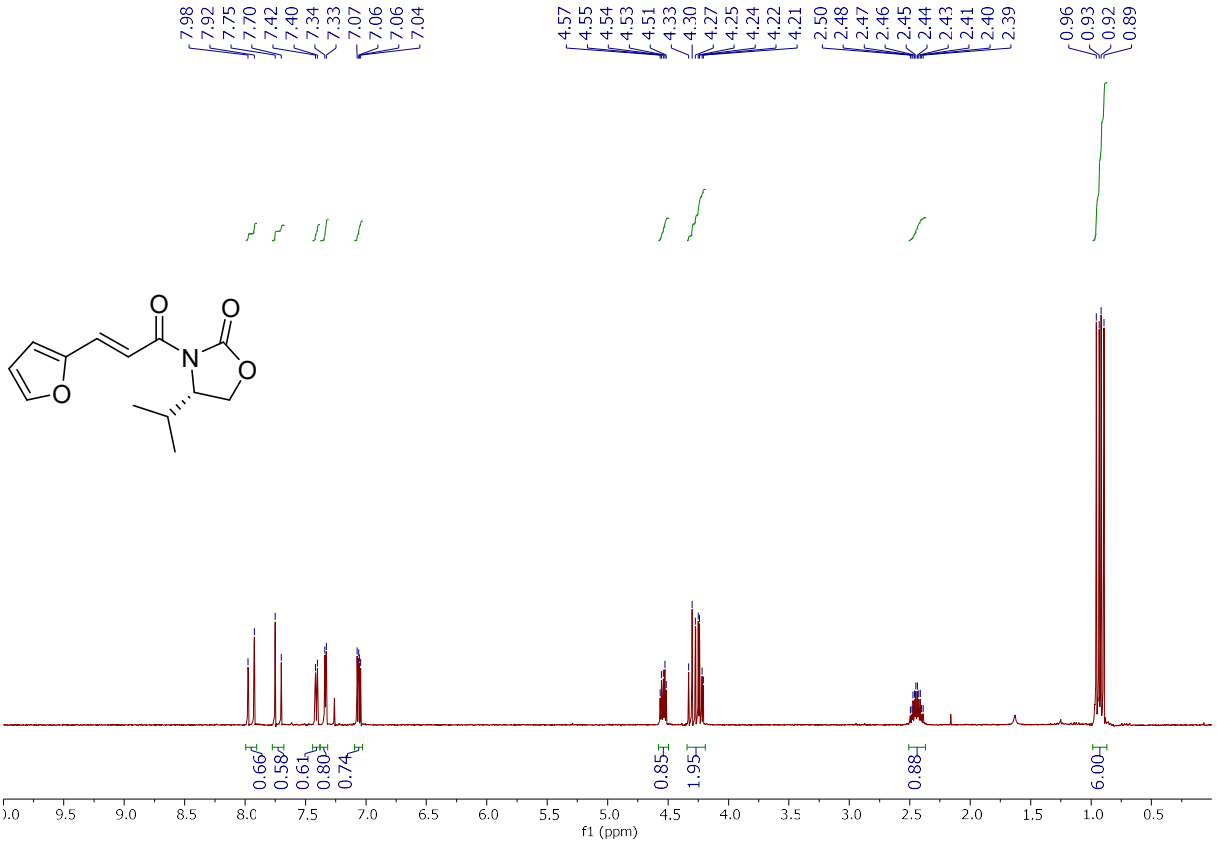
^{13}CNMR, 75 MHz, $CDCl_3$, **15**



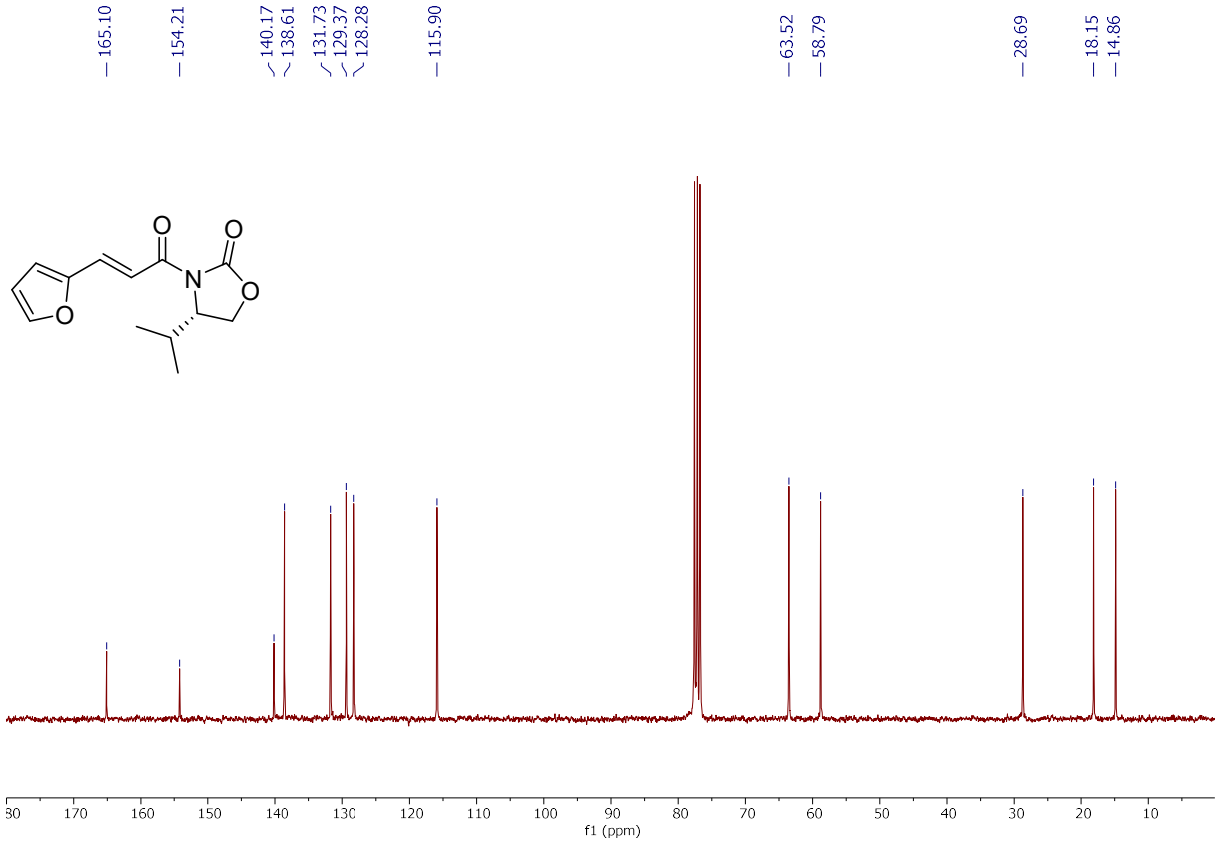
^{19}FNMR, 282 MHz, $CDCl_3$, **15**



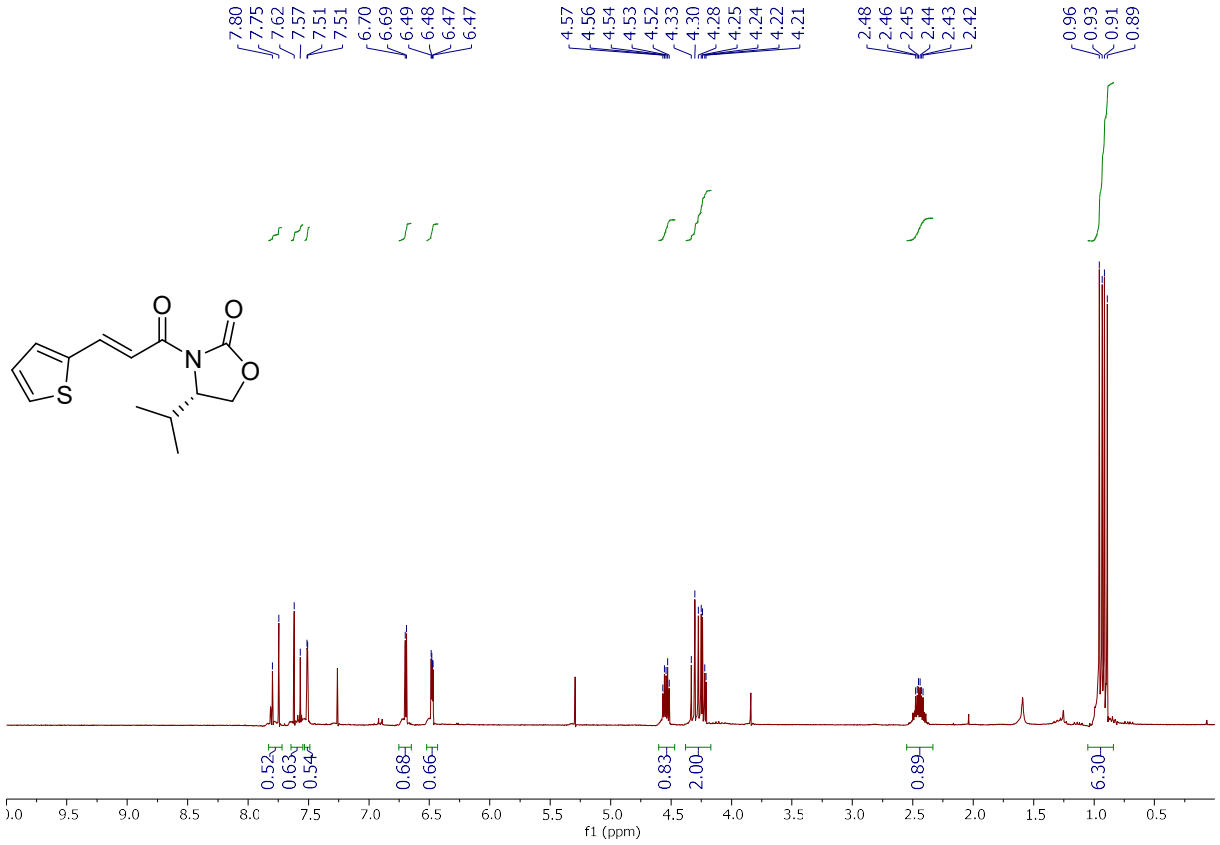
^{1}HNMR, 300 MHz, $CDCl_3$, **16**



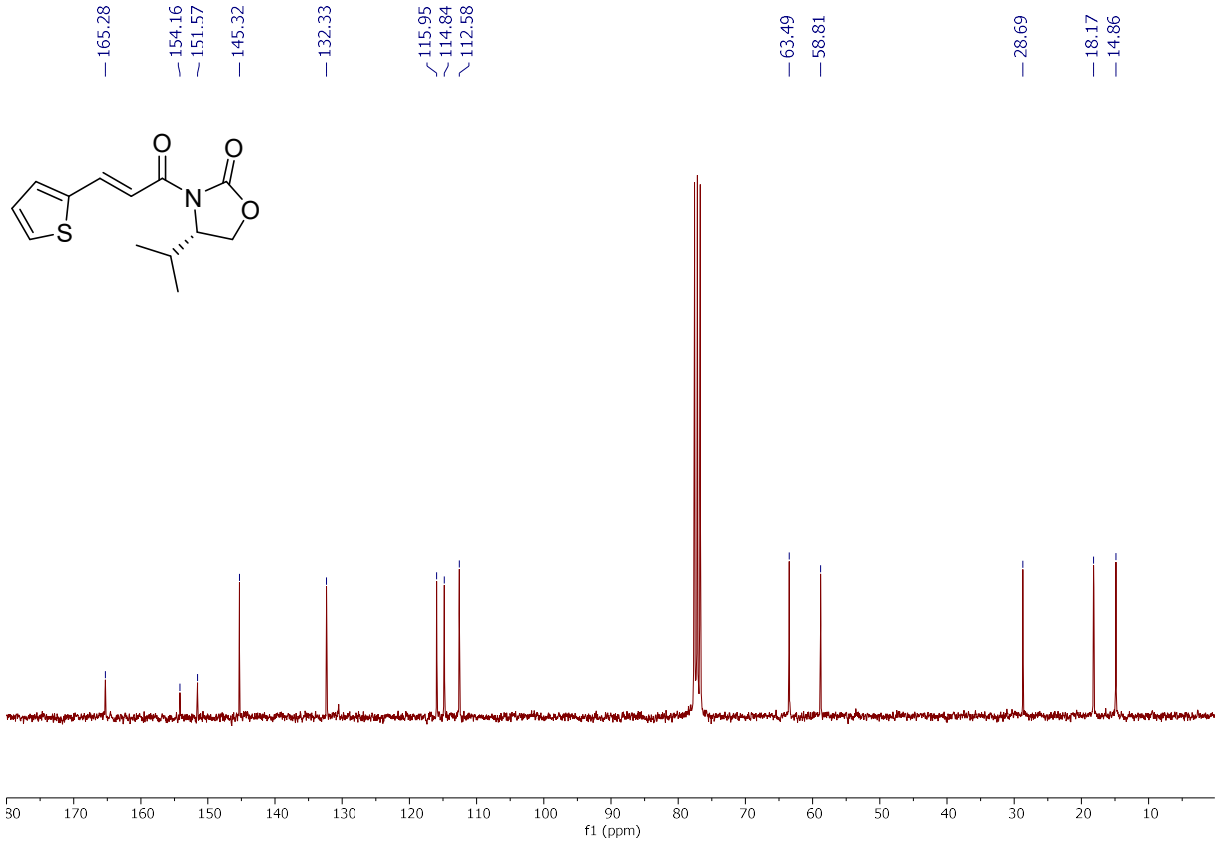
^{13}CNMR, 75 MHz, $CDCl_3$, **16**



^{1}HNMR, 300 MHz, $CDCl_3$, **17**

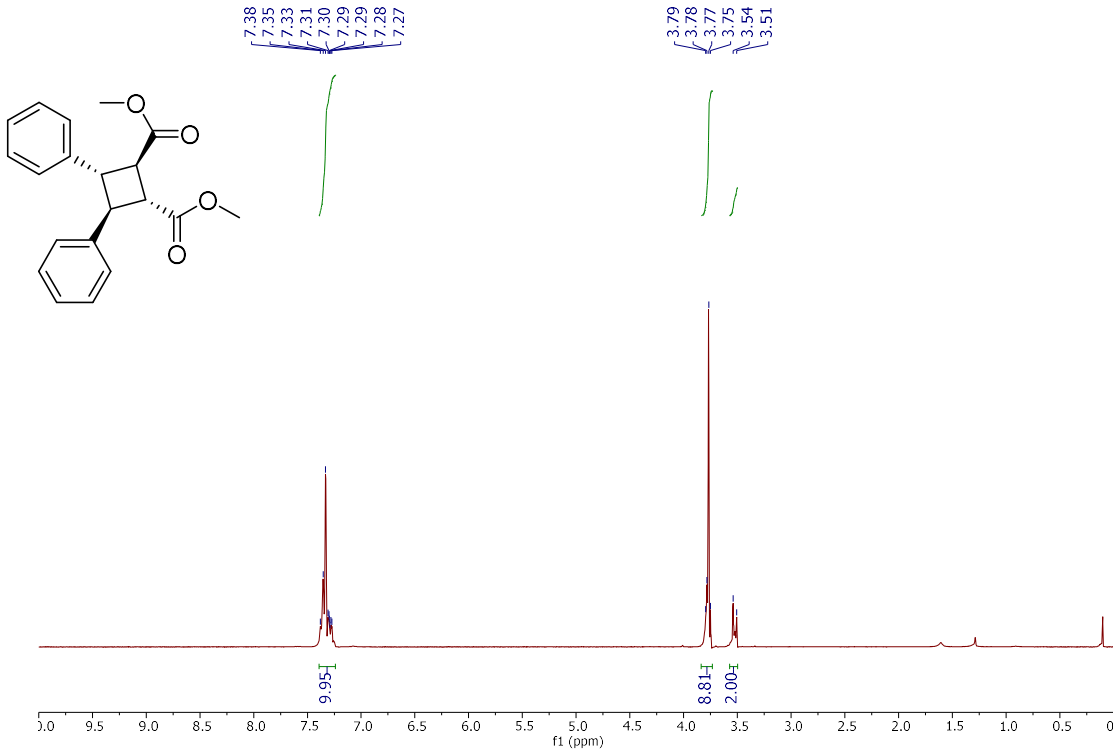


^{13}CNMR, 75 MHz, $CDCl_3$, **17**

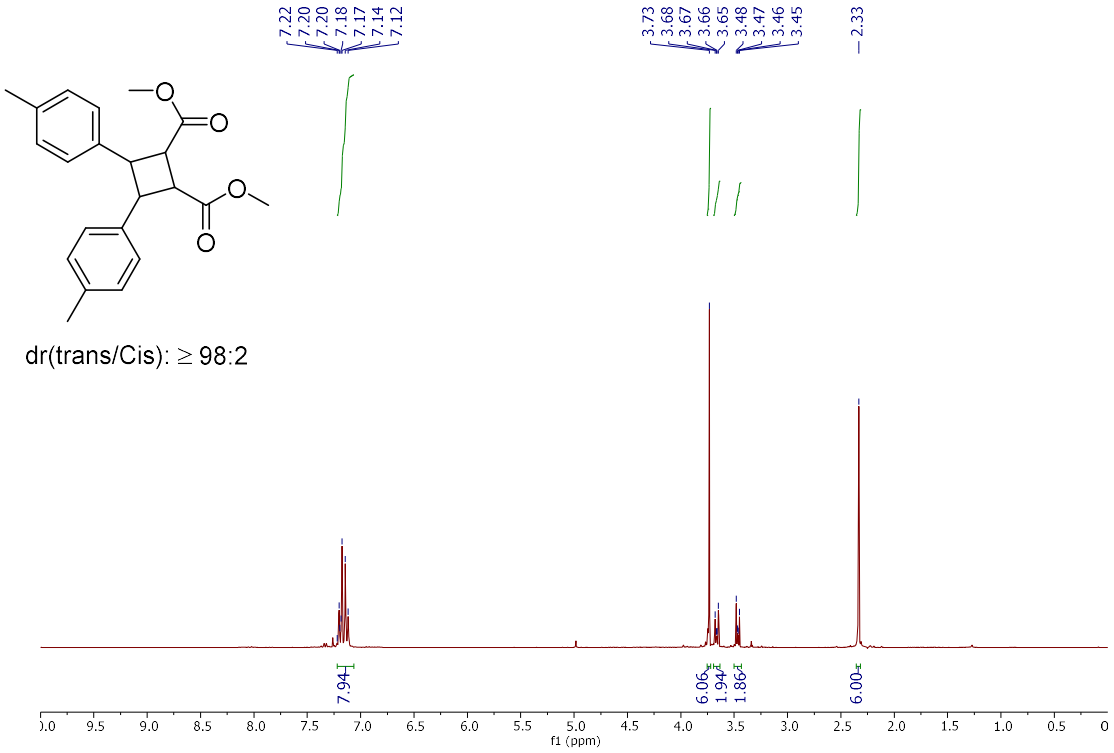


NMR SPECTRA OF ENANTIOENRICHED CYCLOBUTANES

^{1}HNMR, 300 MHz, $CDCl_3$, **4**

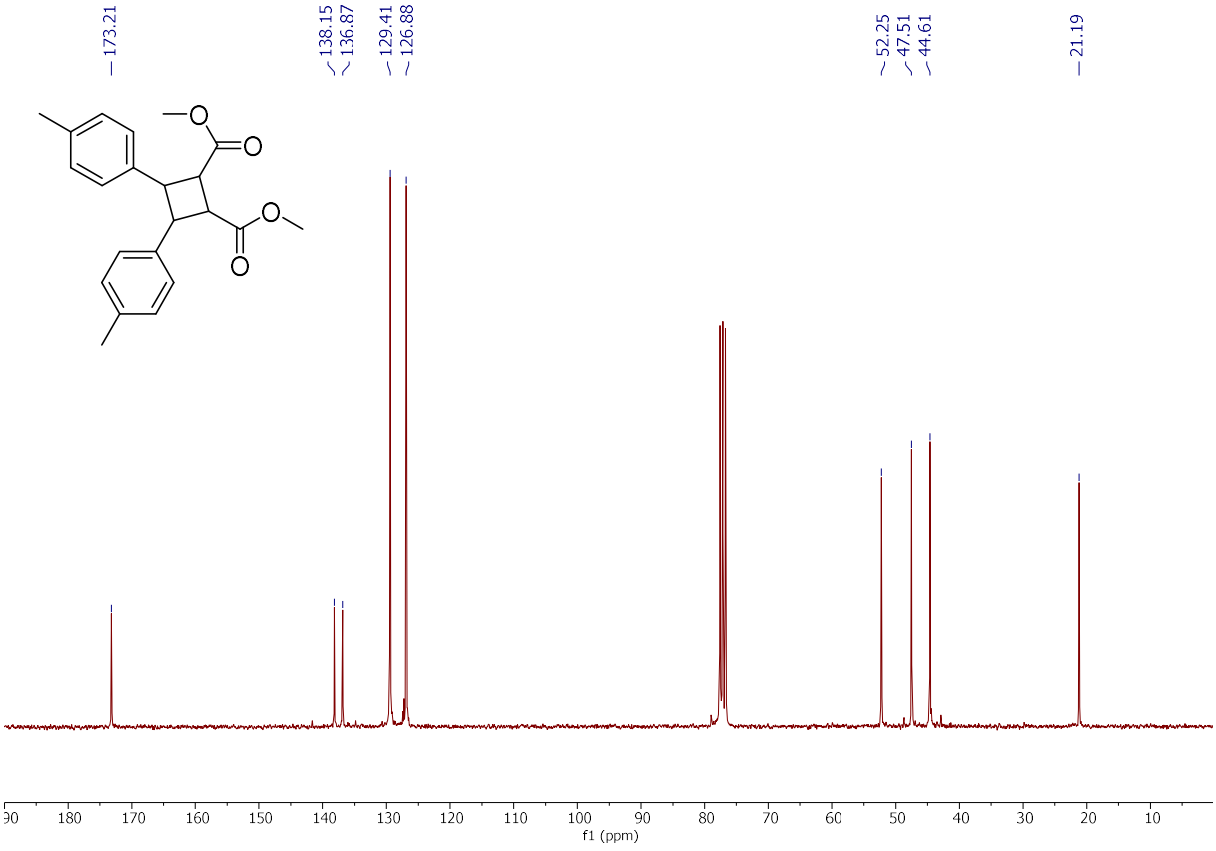


^{1}HNMR, 300 MHz, $CDCl_3$, **18**

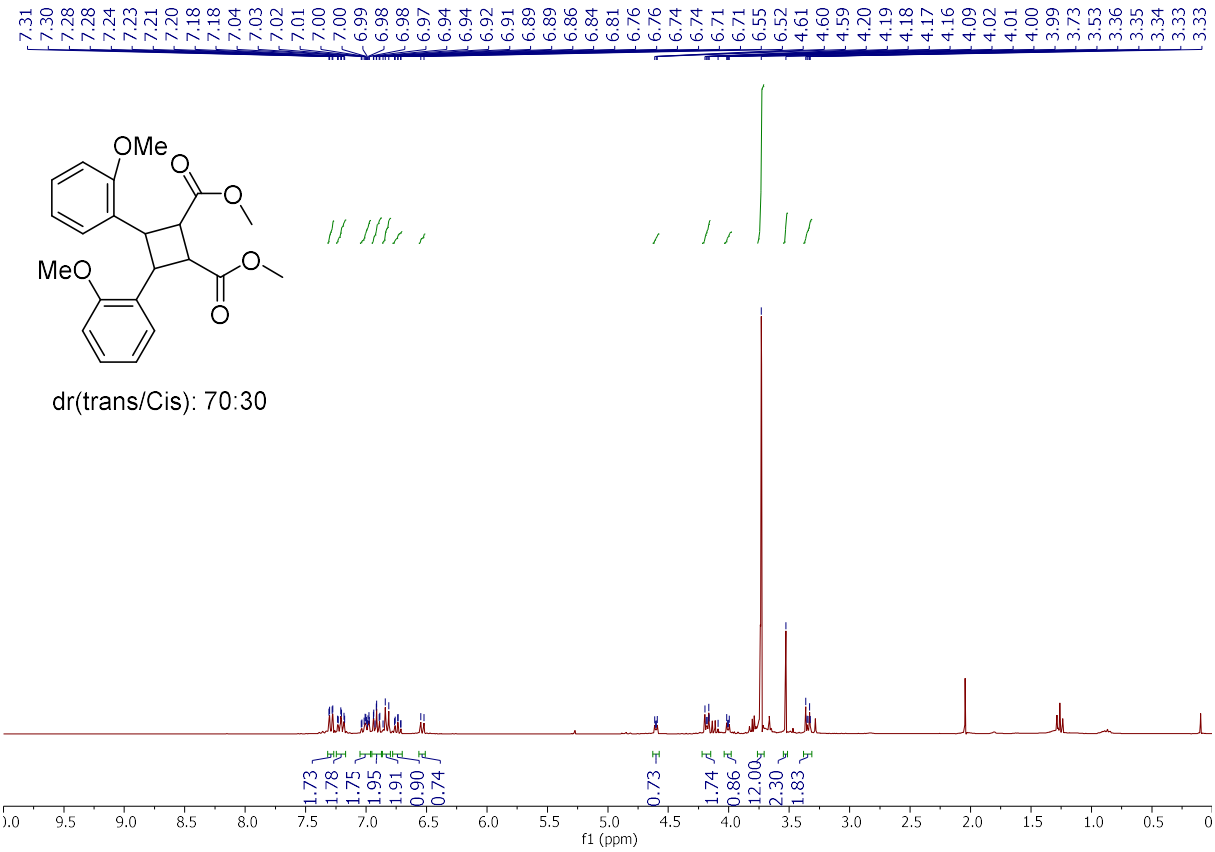


dr(trans/Cis): ≥ 98:2

^{13}CNMR, 75 MHz, $CDCl_3$, **18**

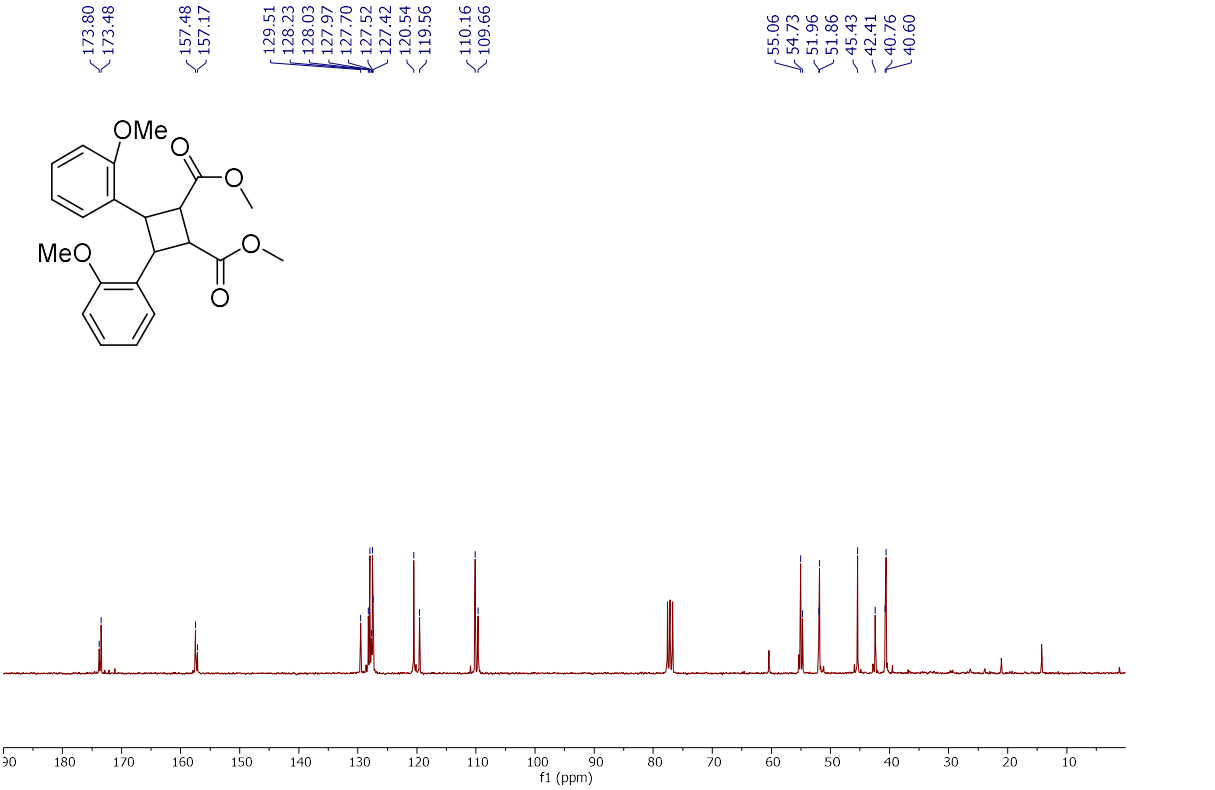


^{1}HNMR, 300 MHz, $CDCl_3$, **19**

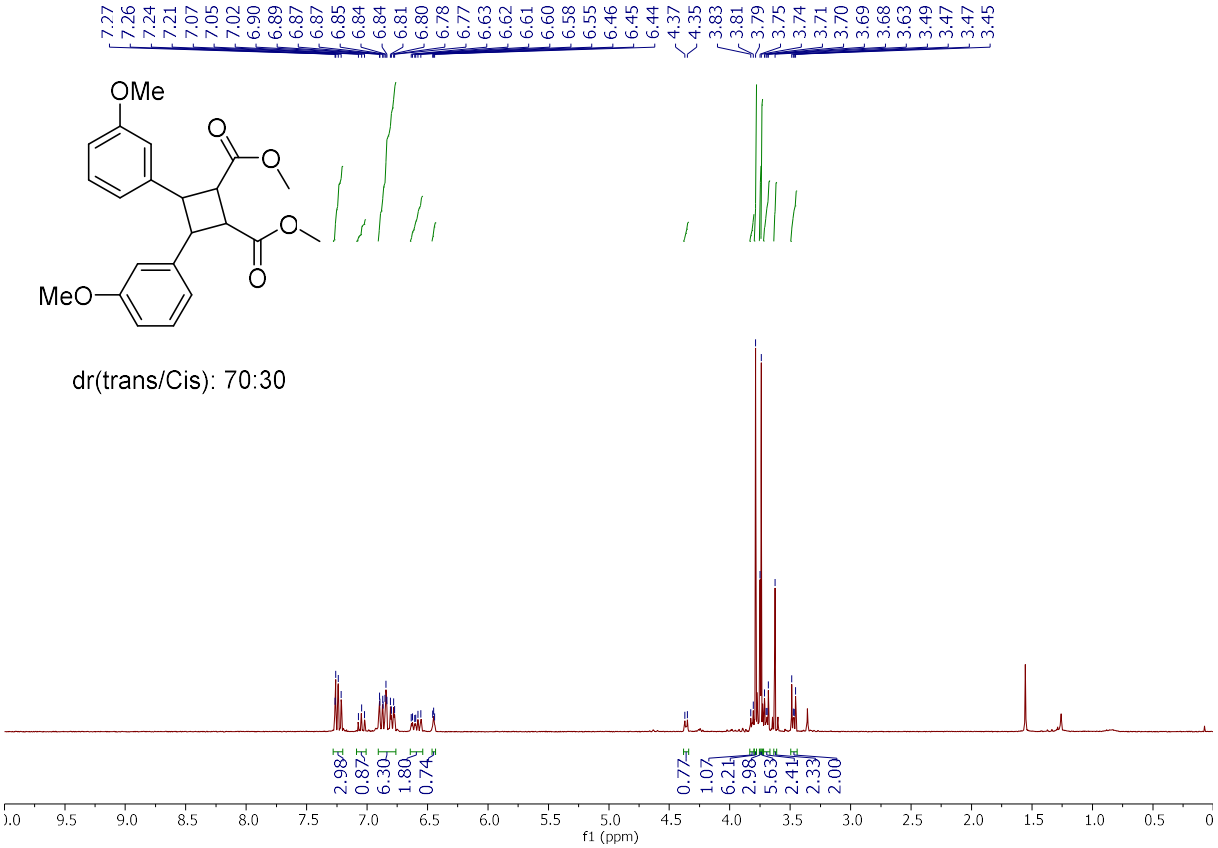


dr(trans/Cis): 70:30

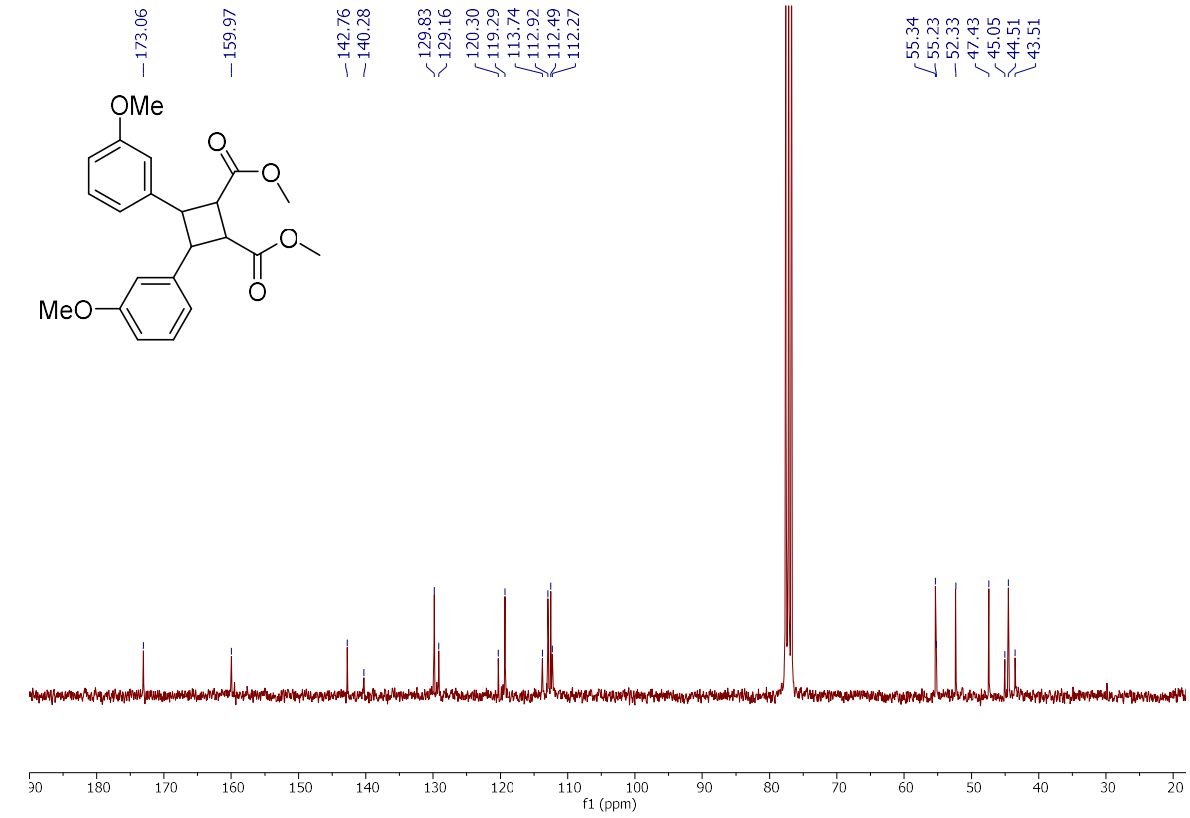
^{13}CNMR, 75 MHz, $CDCl_3$, **19**



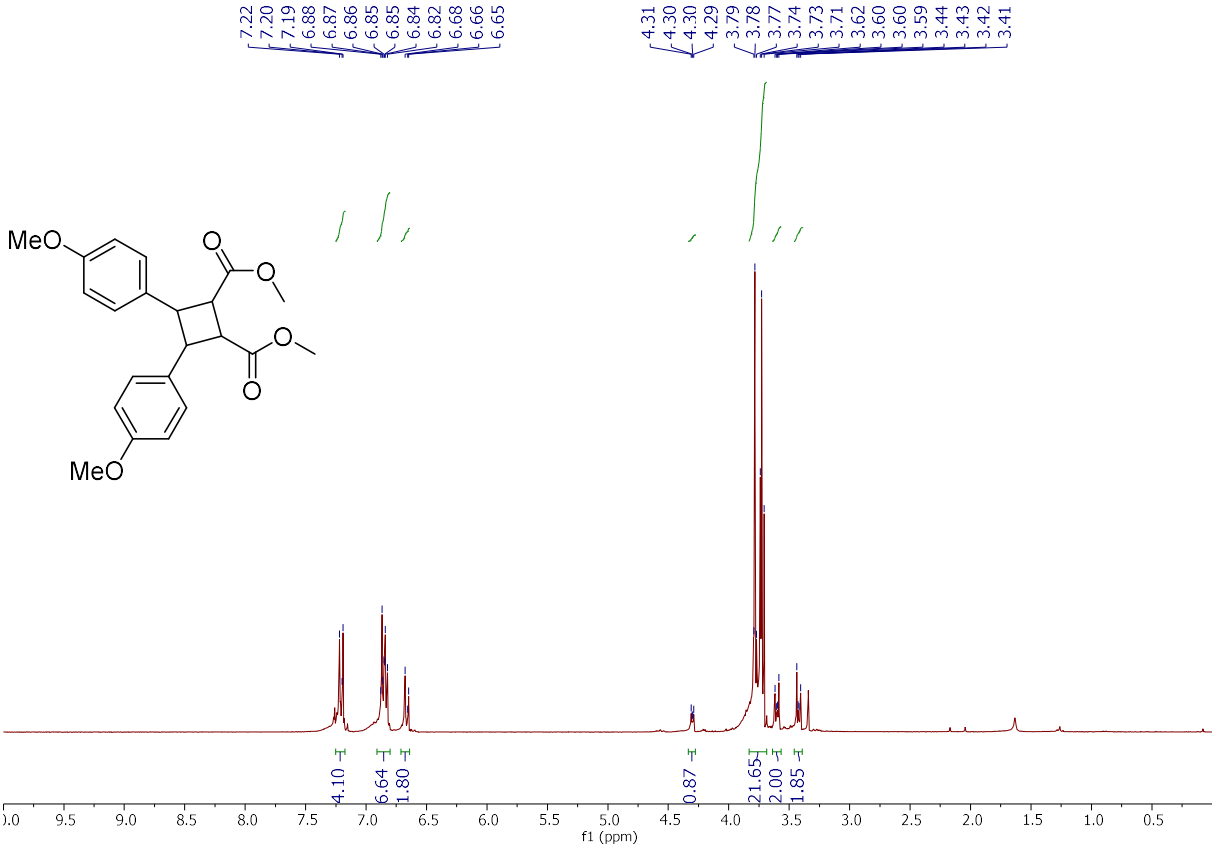
[1]HNMR, 300 MHz, $CDCl_3$, **20**



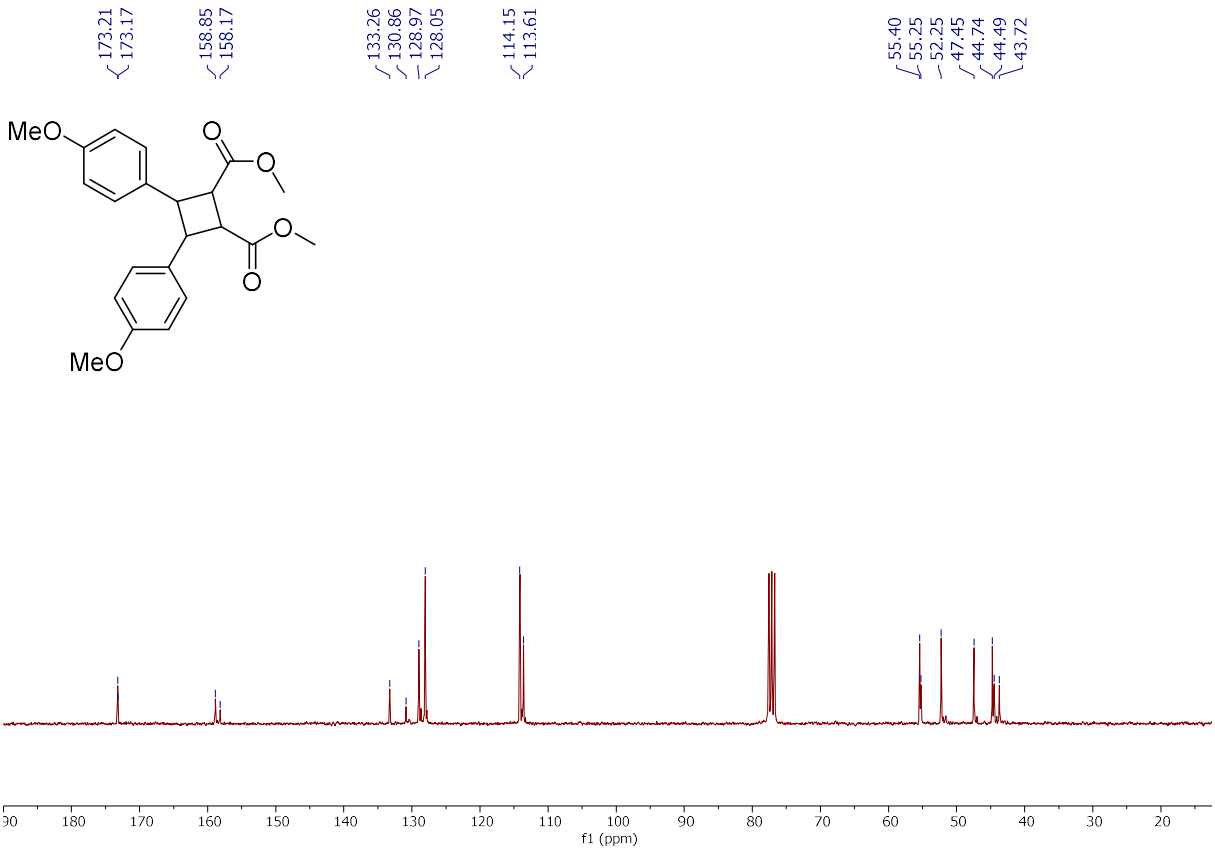
[13]CNMR, 75 MHz, $CDCl_3$, **20**



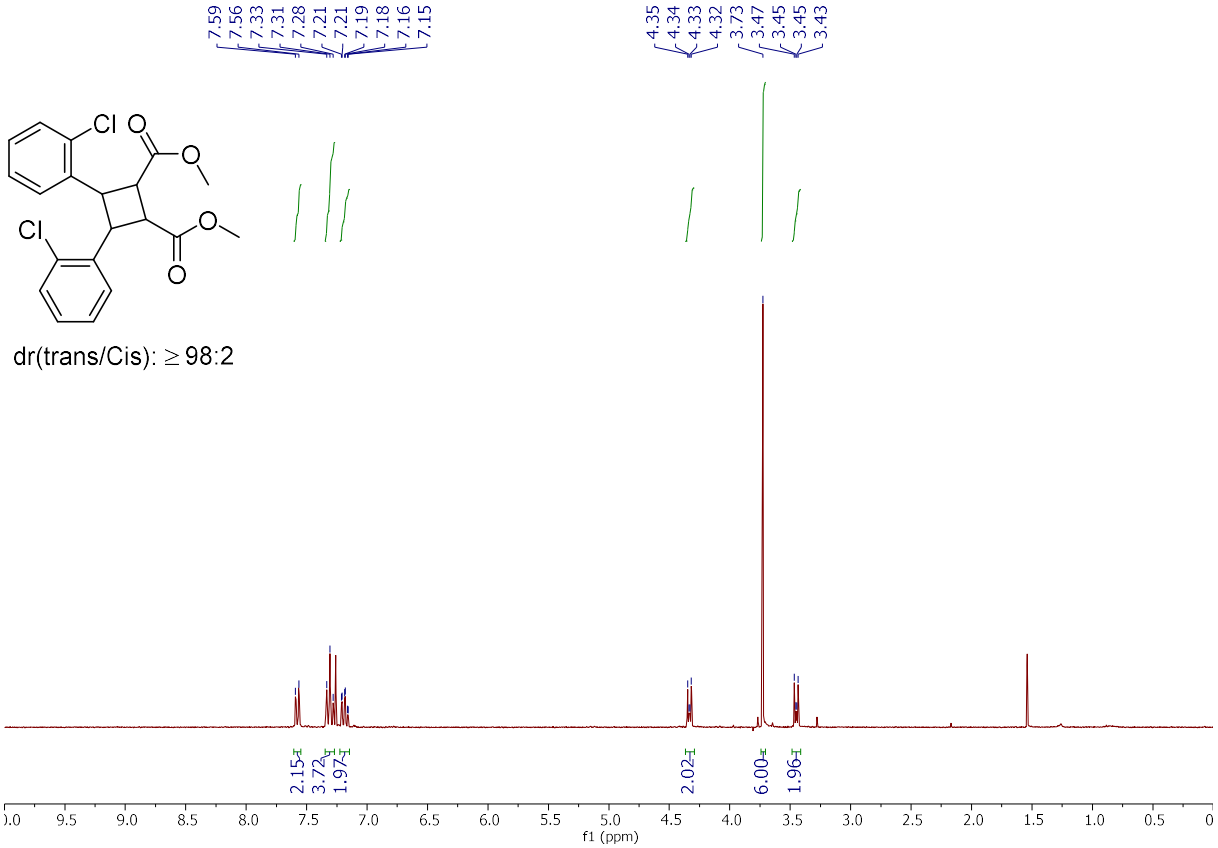
^{1}HNMR, 300 MHz, $CDCl_3$, **21**



^{13}CNMR, 75 MHz, $CDCl_3$, **21**

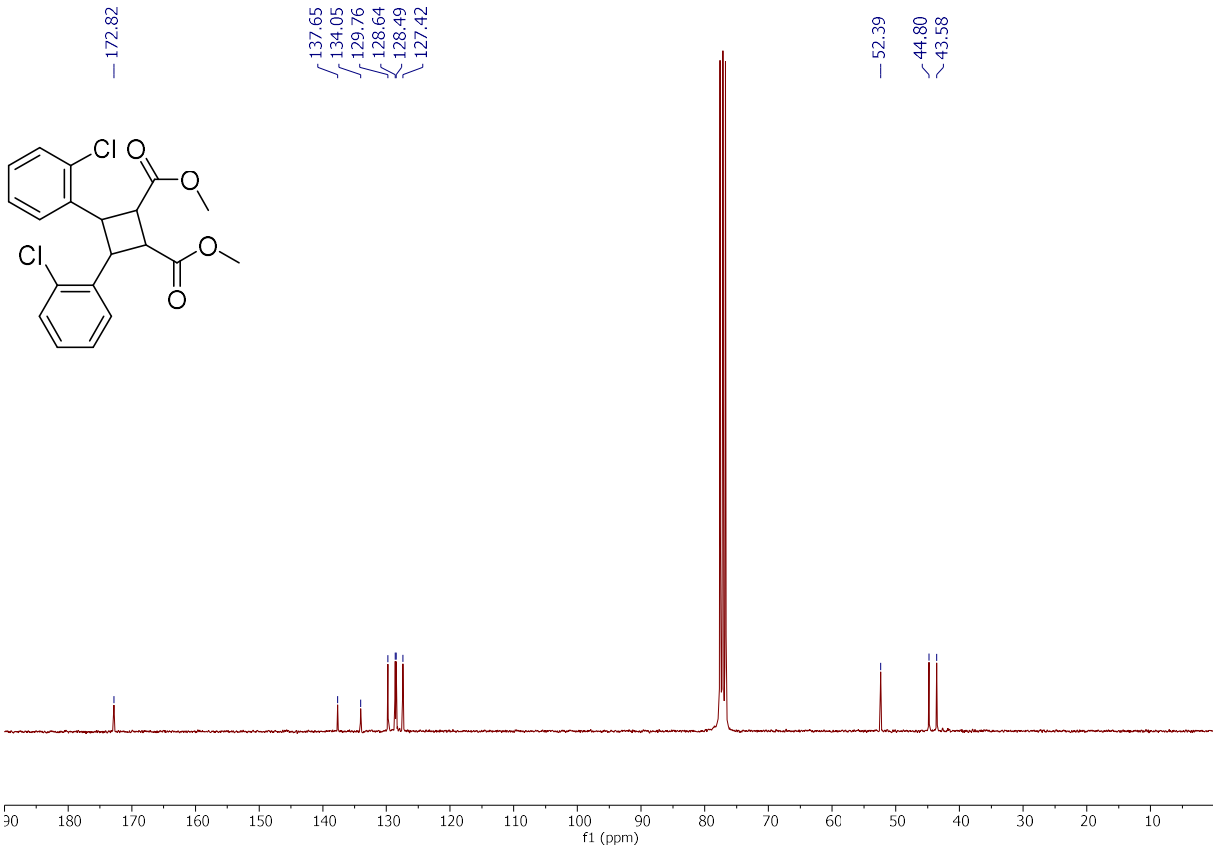


[1]HNMR, 300 MHz, $CDCl_3$, **22**

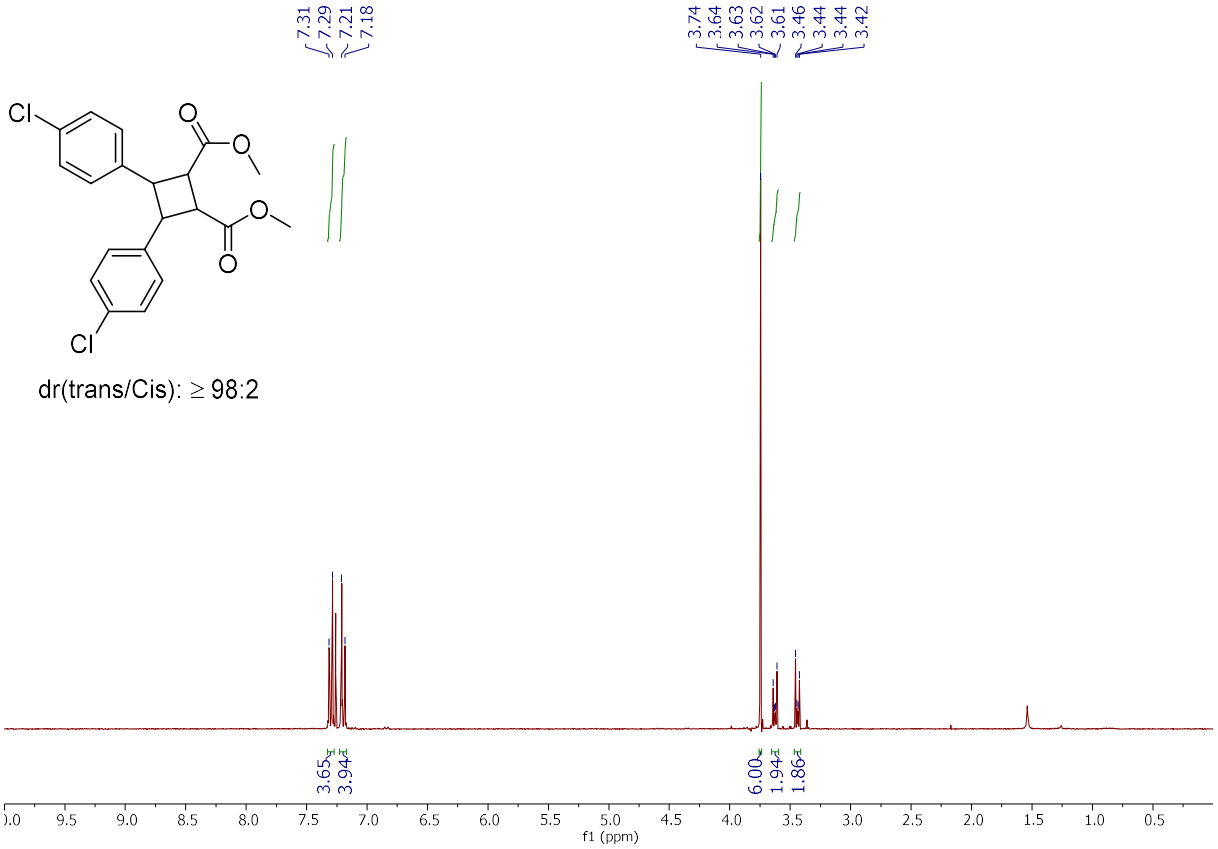


dr(trans/Cis): ≥ 98:2

[13]CNMR, 75 MHz, $CDCl_3$, **22**

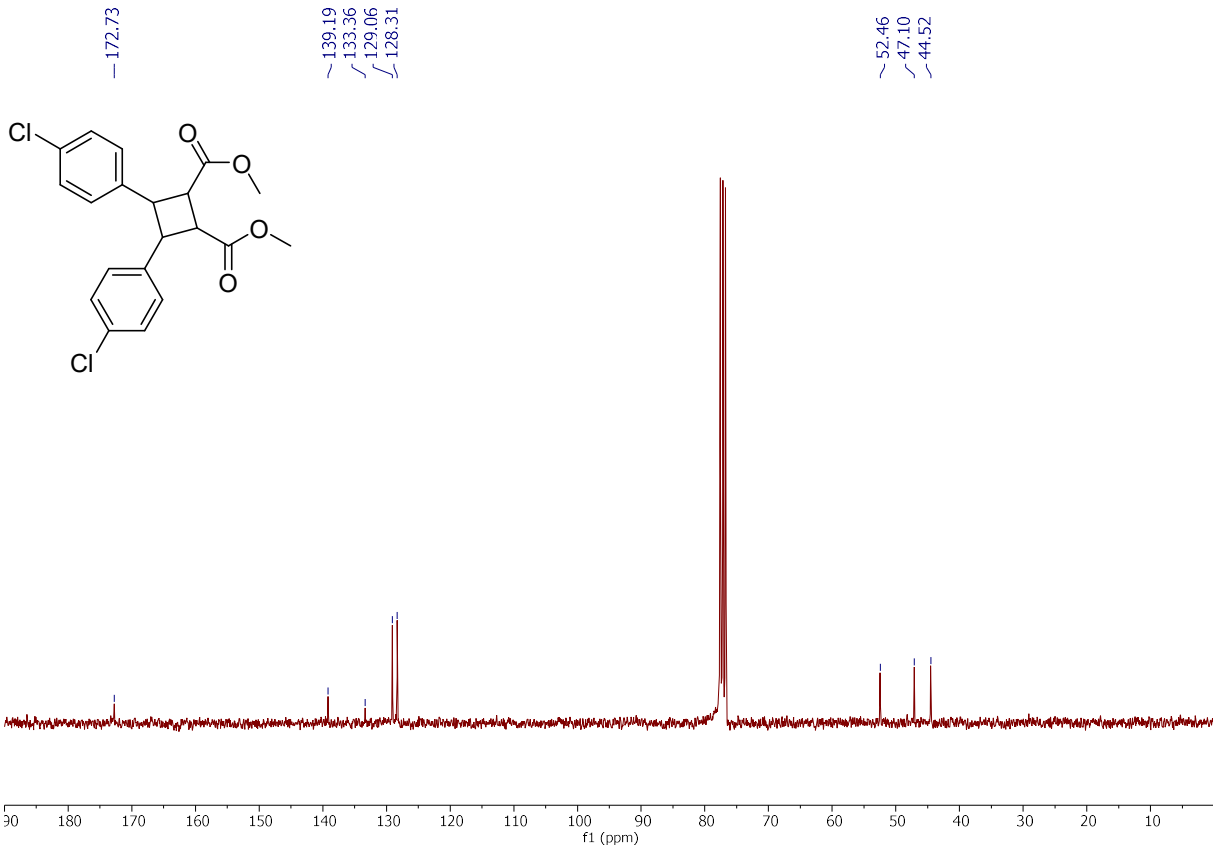


^{1}HNMR, 300 MHz, $CDCl_3$, **23**

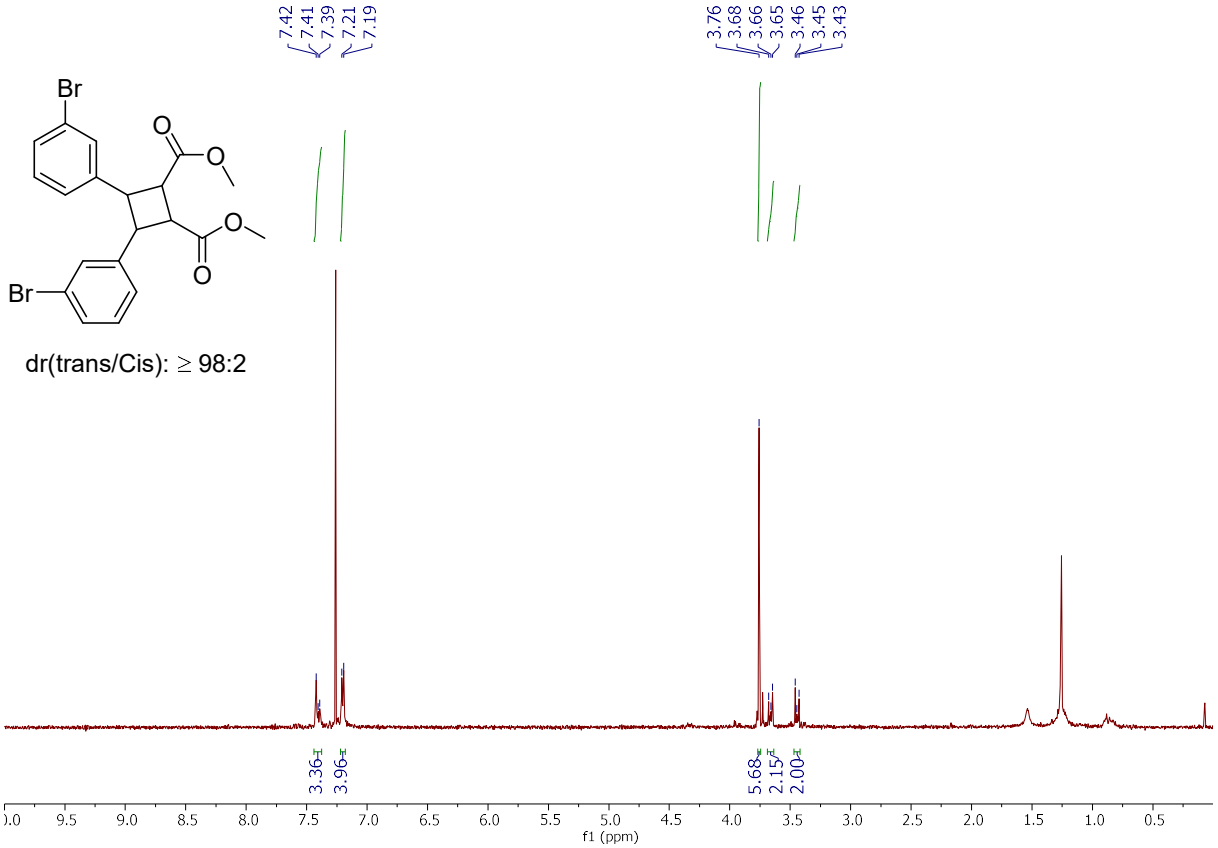


dr(trans/Cis): ≥ 98:2

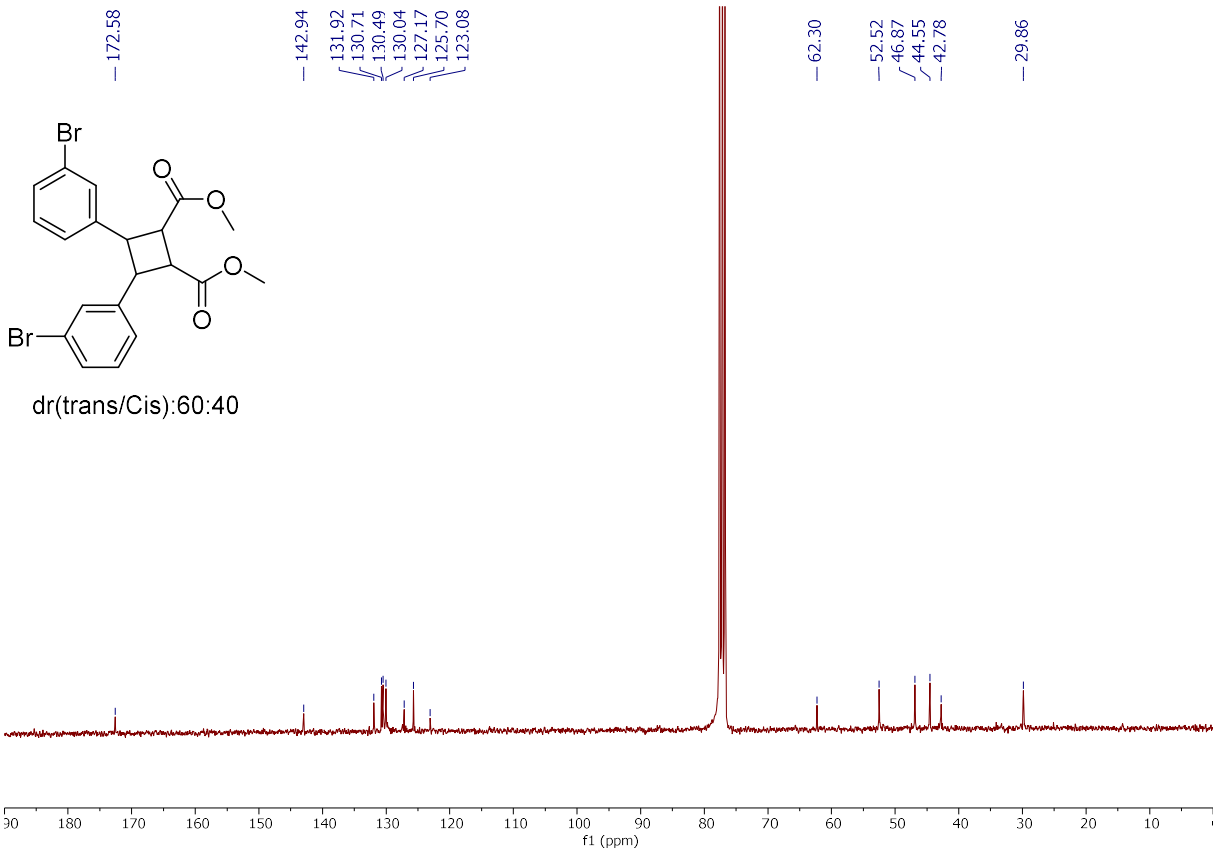
^{13}CNMR, 75 MHz, $CDCl_3$, **23**



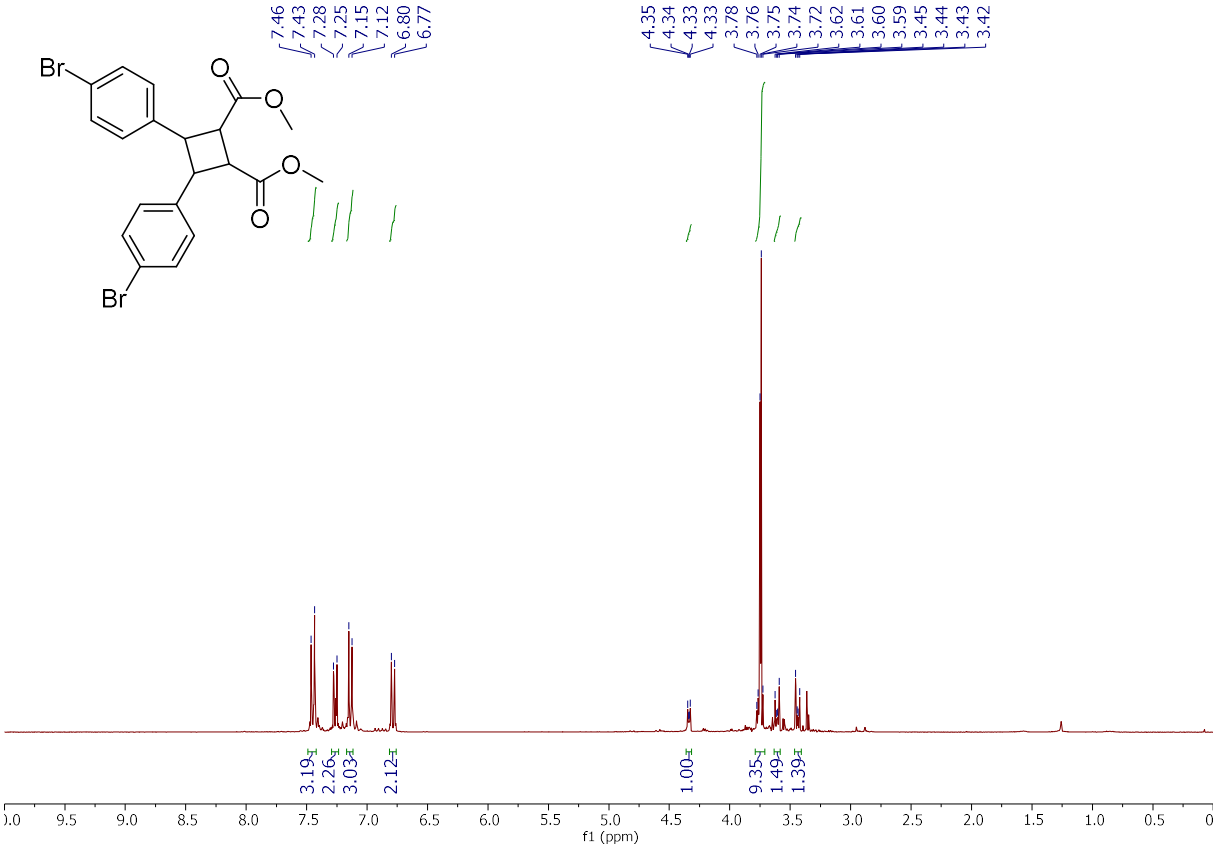
^{1}HNMR, 300 MHz, $CDCl_3$, **24**



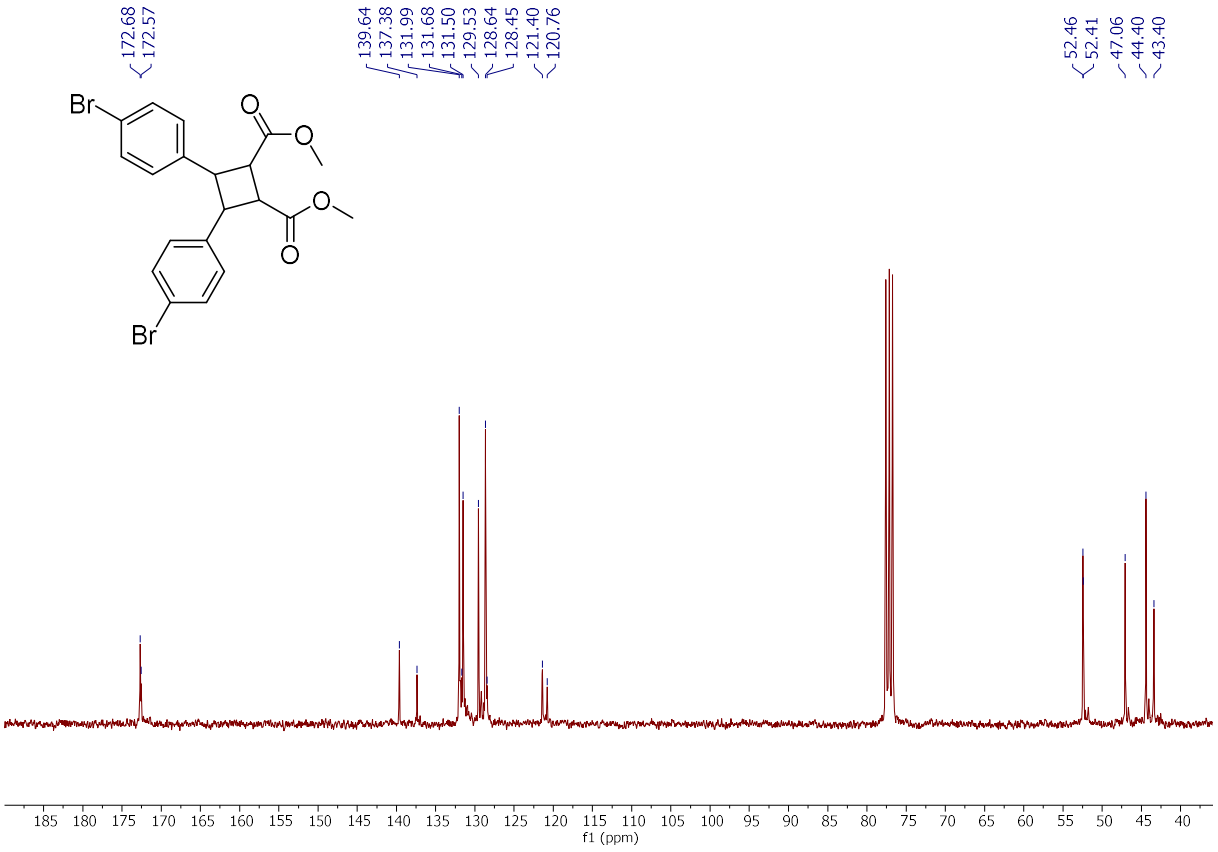
^{13}CNMR, 75 MHz, $CDCl_3$, **24**



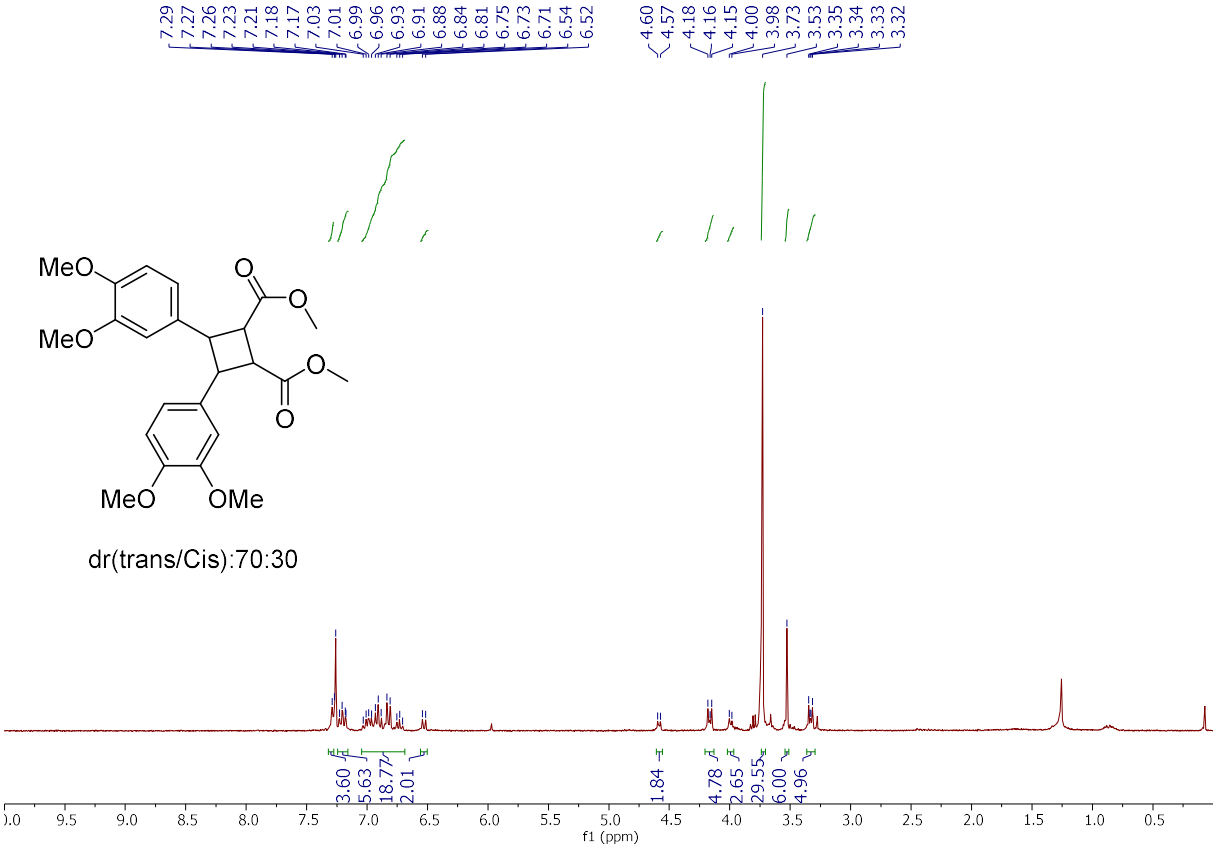
^{1}HNMR, 300 MHz, $CDCl_3$, **25**



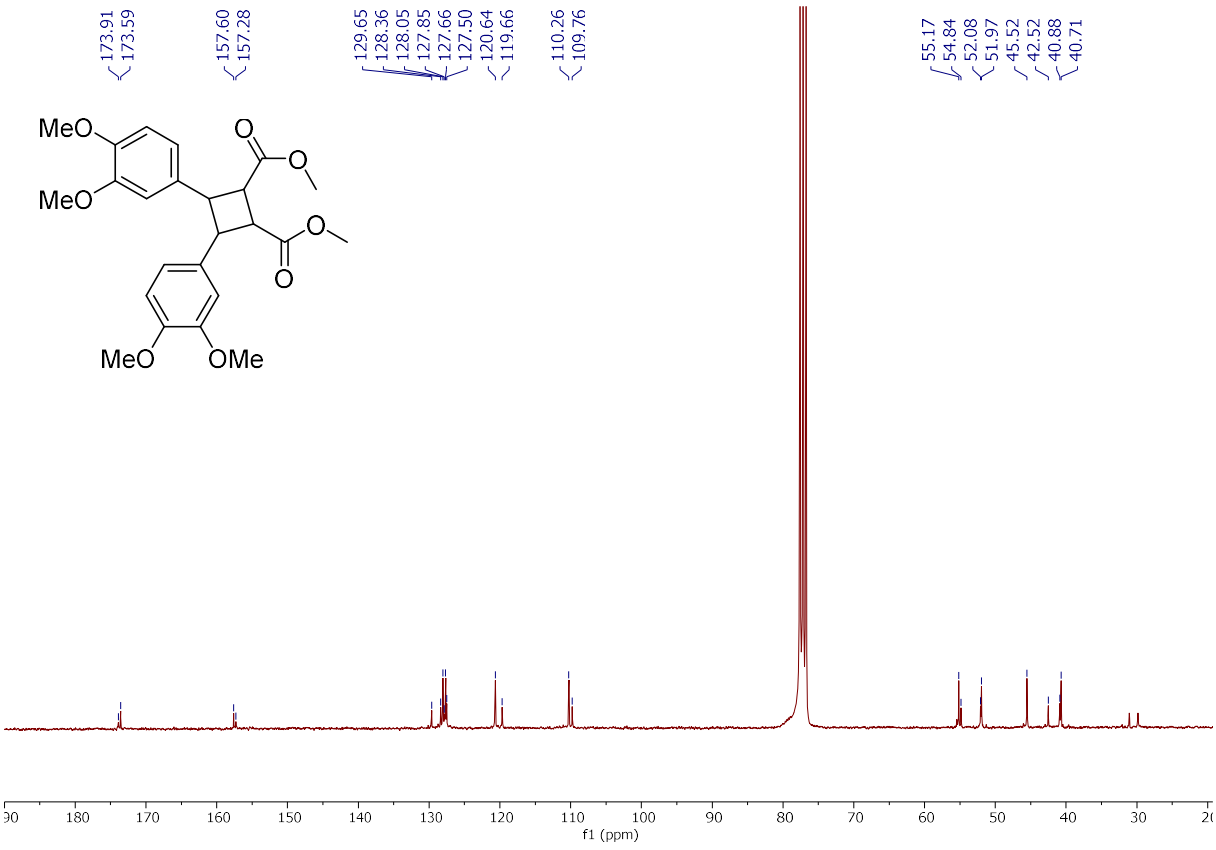
^{13}CNMR, 75 MHz, $CDCl_3$, **25**



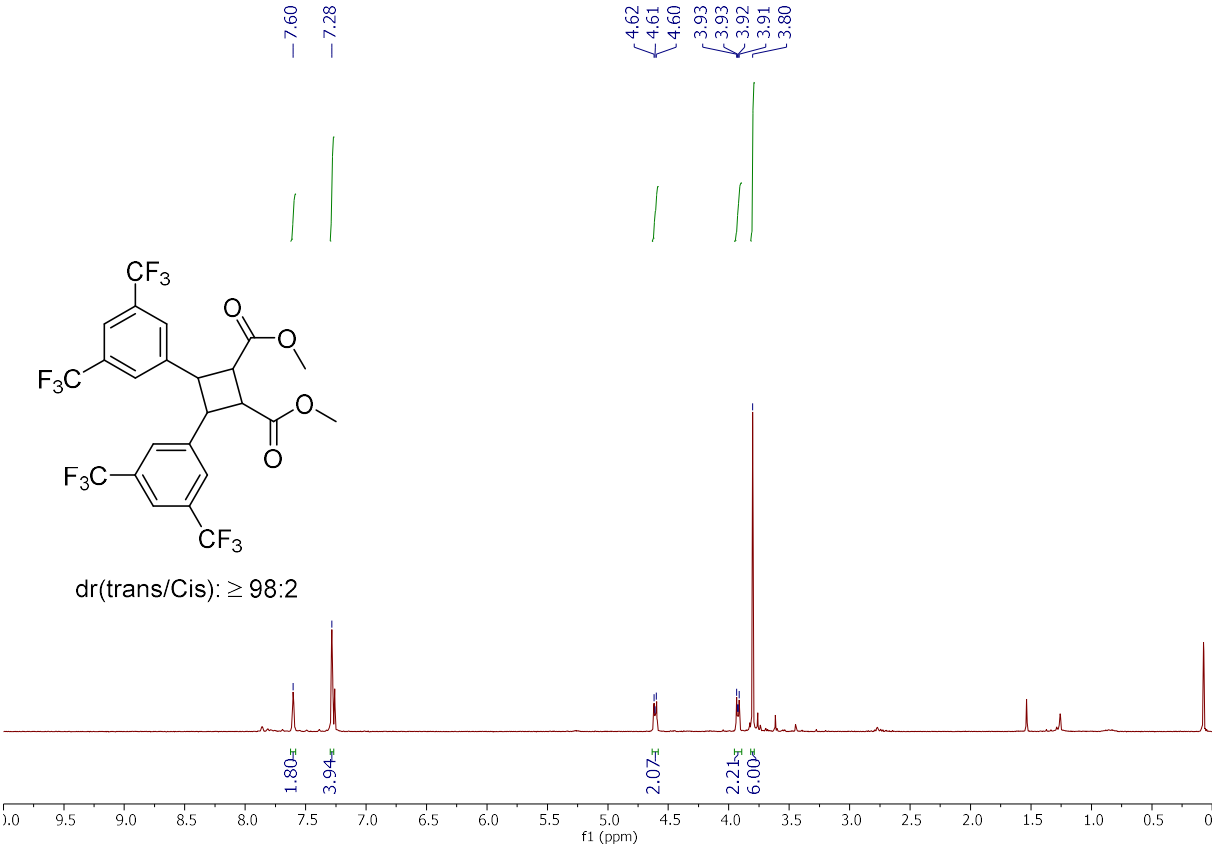
[1]HNMR, 300 MHz, $CDCl_3$, **26**



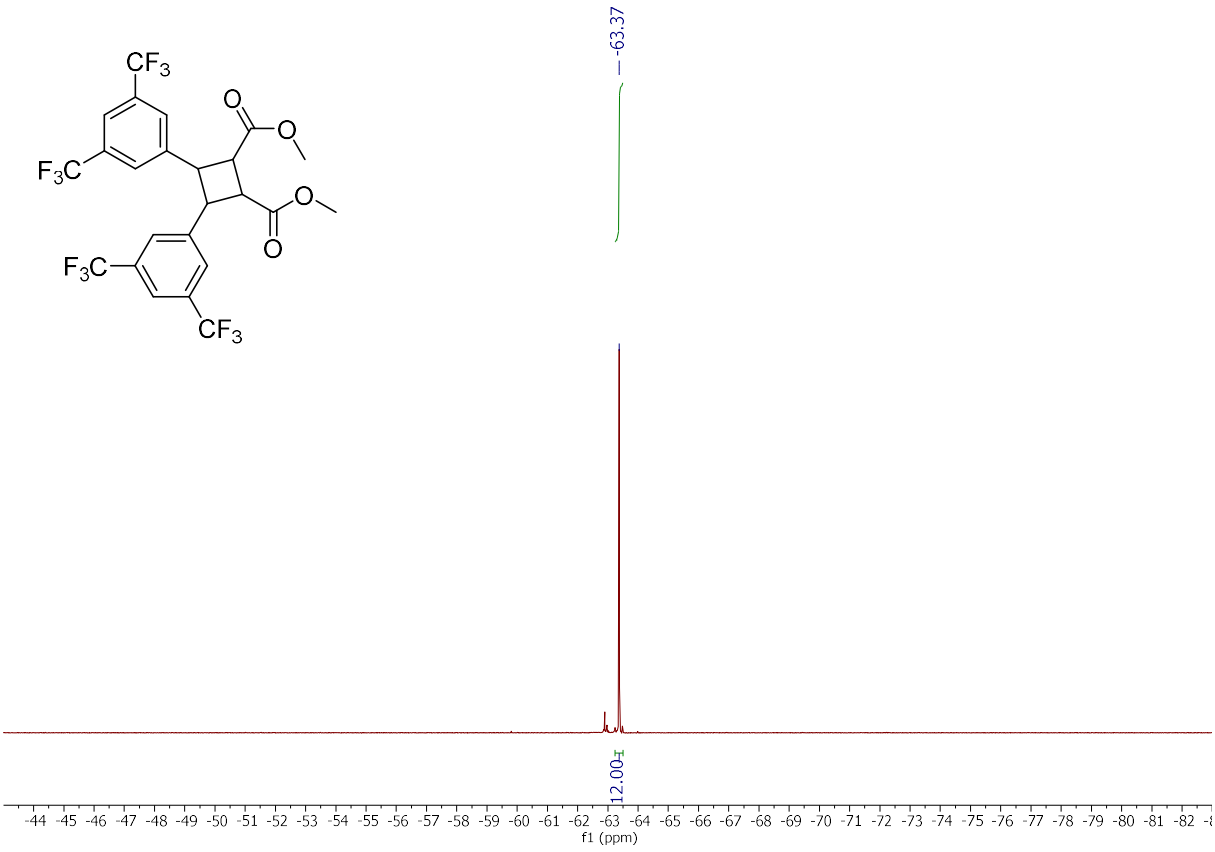
[13]CNMR, 75 MHz, $CDCl_3$, **26**



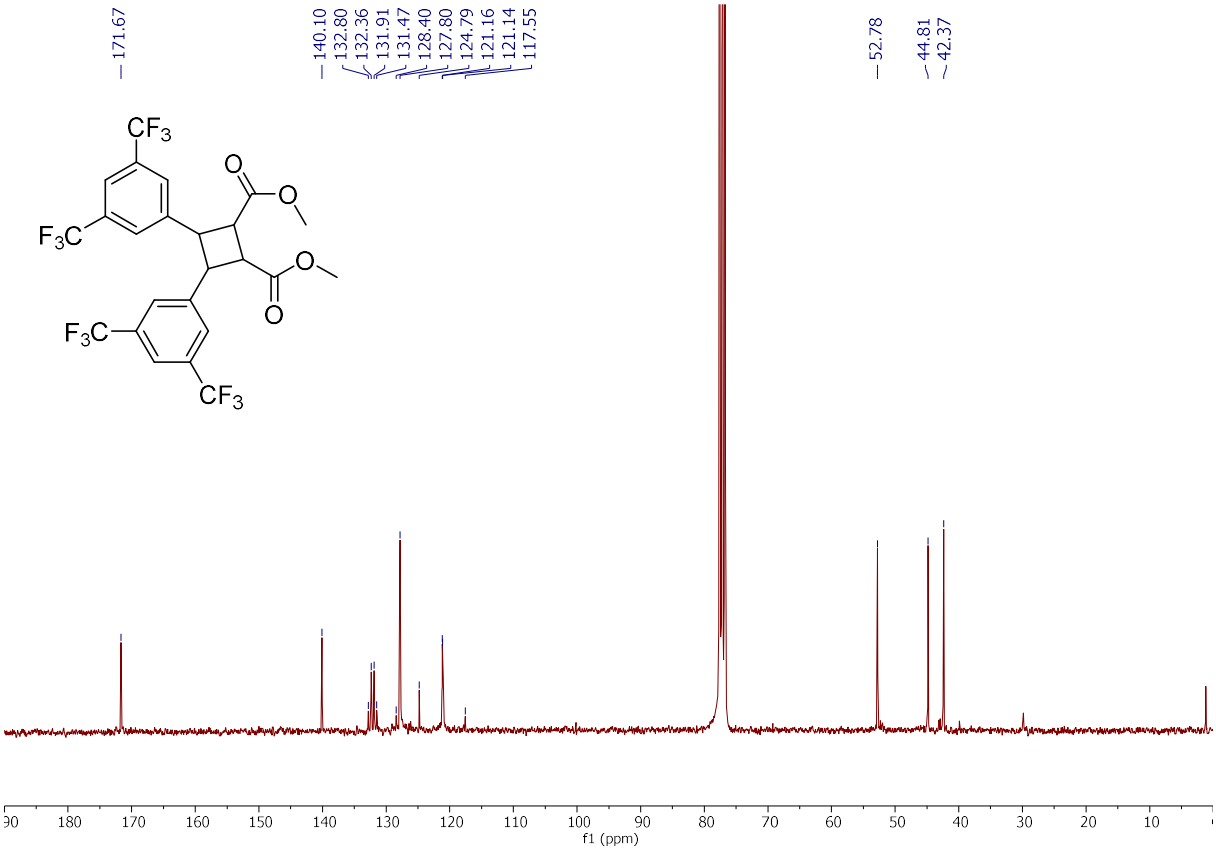
[1]HNMR, 300 MHz, $CDCl_3$, **27**



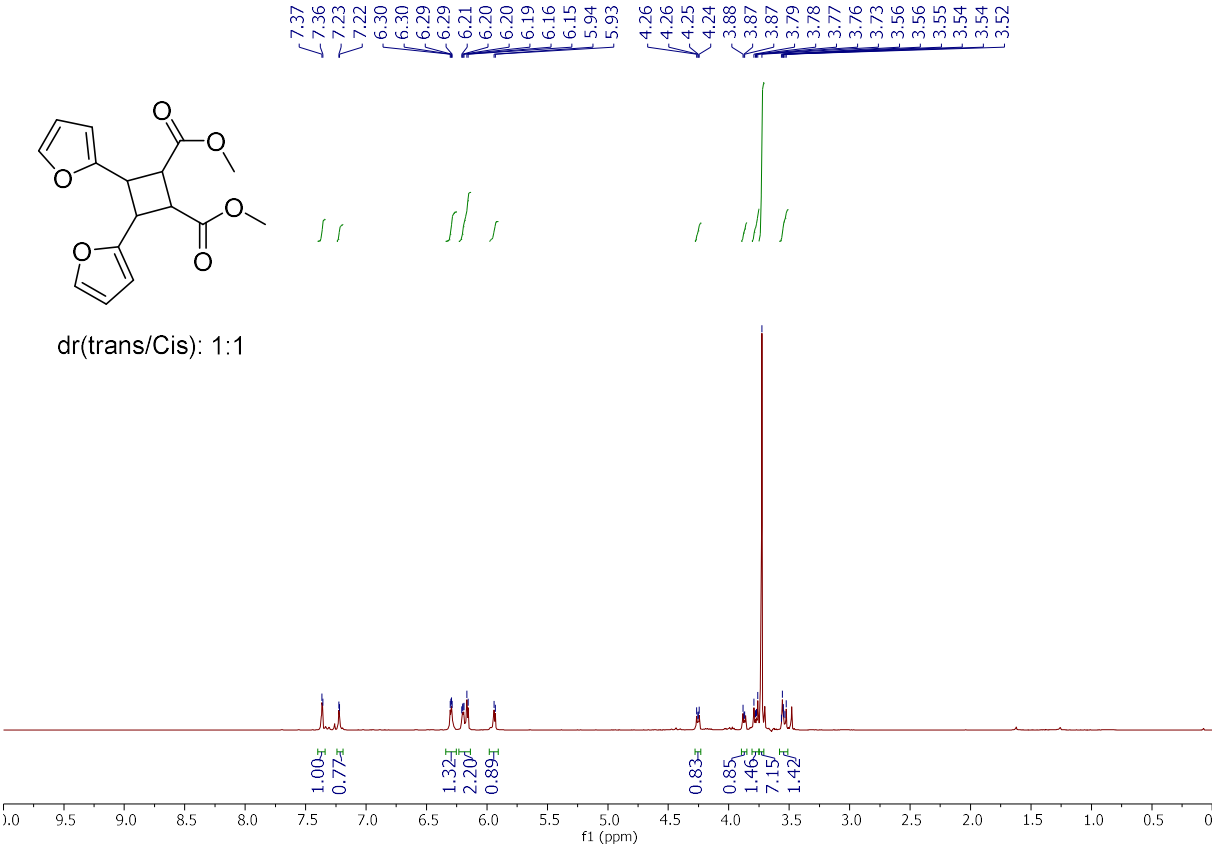
[19]FNMR, 282 MHz, $CDCl_3$, **27**



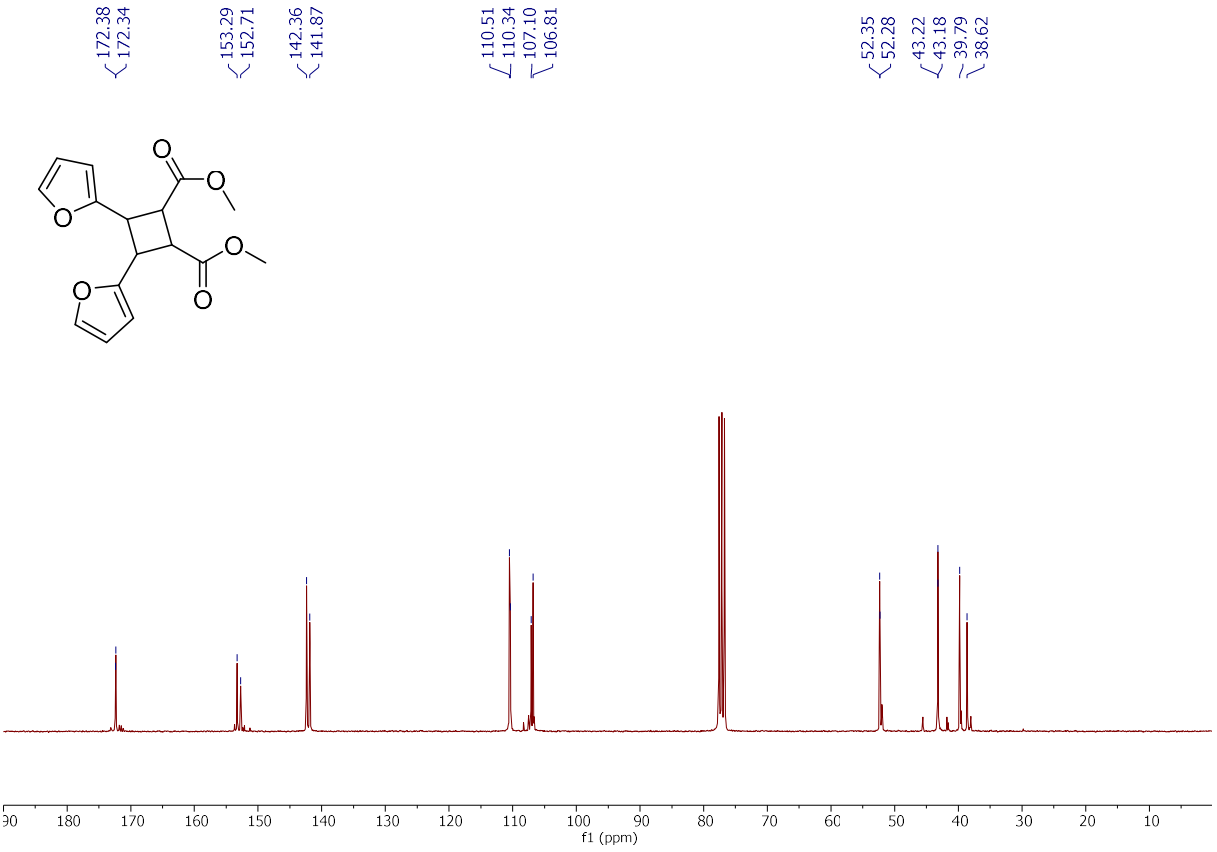
^{13}CNMR, 75 MHz, $CDCl_3$, **27**



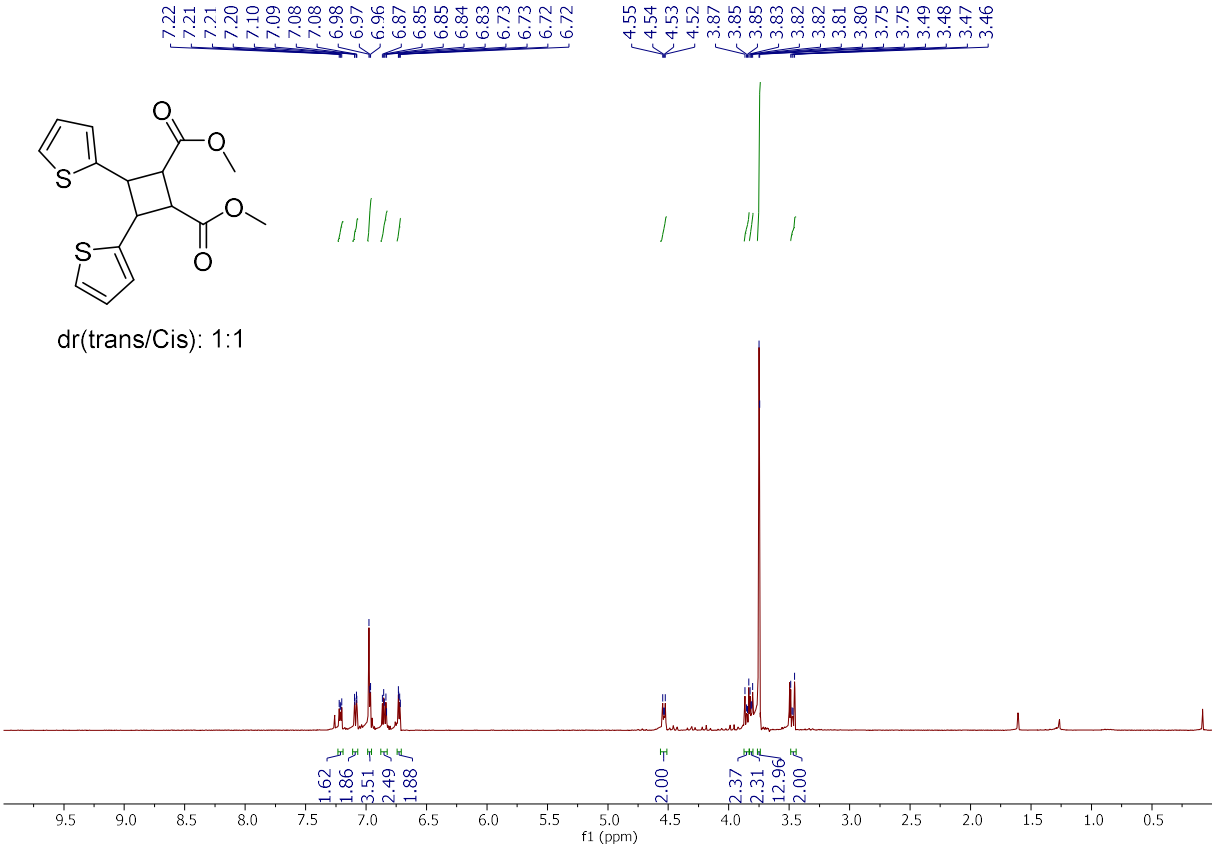
^{1}HNMR, 300 MHz, $CDCl_3$, **28**



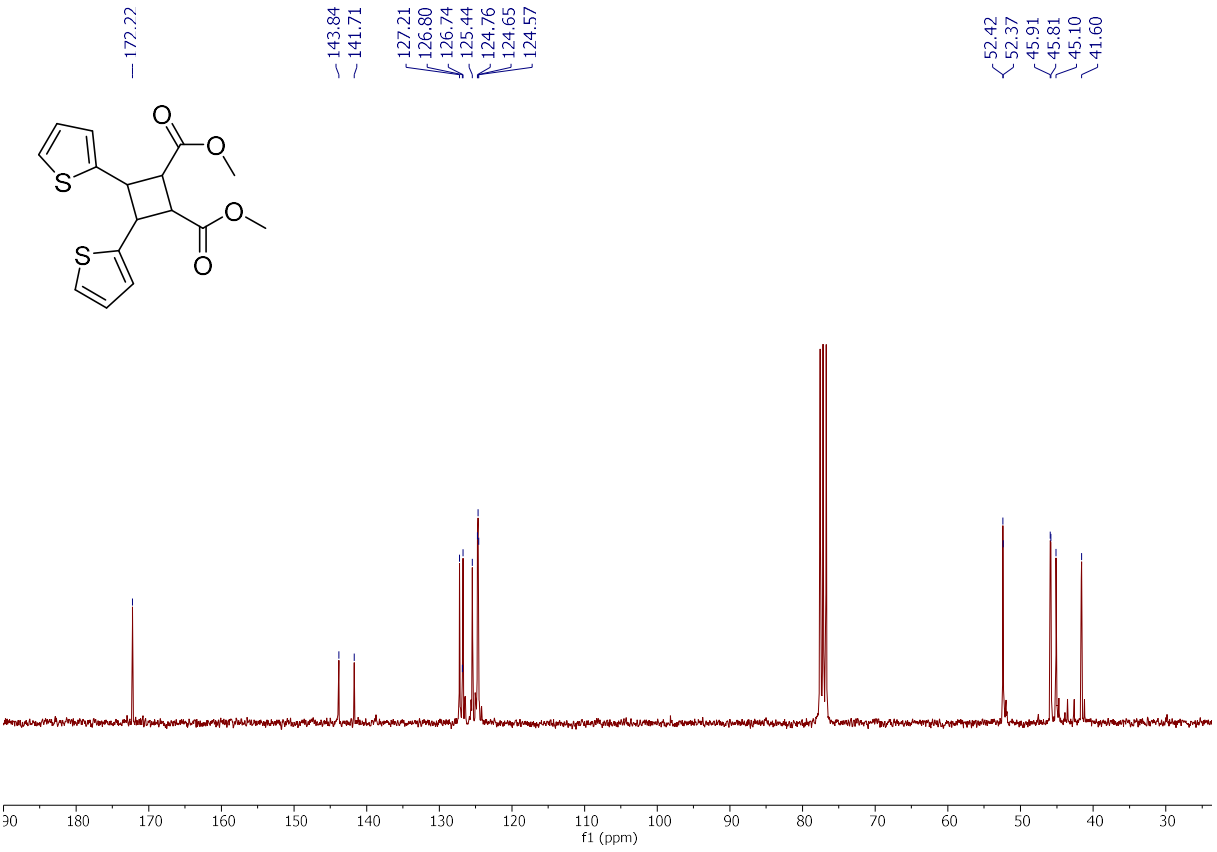
^{13}CNMR, 75 MHz, $CDCl_3$, **28**



[1]HNMR, 300 MHz, $CDCl_3$, **29**



[13]CNMR, 75 MHz, $CDCl_3$, **29**



Mass Spectra

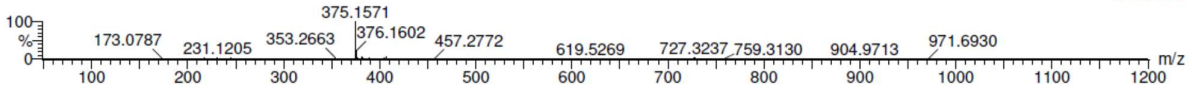
- ***Dimethyl 3,4-di-p-tolylcyclobutane-1,2-dicarboxylate, 18***

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -2.5, max = 200.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions
1 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 22-22 H: 24-24 O: 4-4 Na: 0-1

FAME-102 42 (0.845) AM2 (Ar,40000.0,0.00,0.00); Cm (10:50) 1: TOF MS ES+ 1.75e+007



Minimum:				-2.5				
Maximum:		5.0	5.0	200.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
375.1571	375.1572	-0.1	-0.3	10.5	2339.5	n/a	n/a	C22 H24 O4 Na

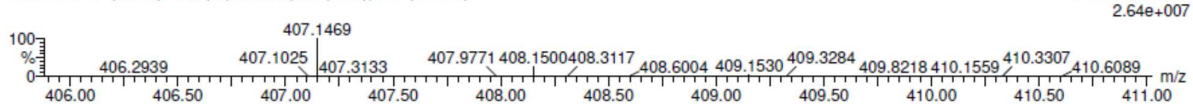
- ***Dimethyl 3,4-bis(2-methoxyphenyl)cyclobutane-1,2-dicarboxylate, 19***

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -2.5, max = 200.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
1 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 22-22 H: 24-24 O: 6-6 Na: 0-2

FAME-126 17 (0.364) AM2 (Ar,40000.0,0.00,0.00); Cm (10:103) 1: TOF MS ES+ 2.64e+007



Minimum:				-2.5				
Maximum:		5.0	5.0	200.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
407.1469	407.1471	-0.2	-0.5	10.5	1383.5	n/a	n/a	C22 H24 O6 Na

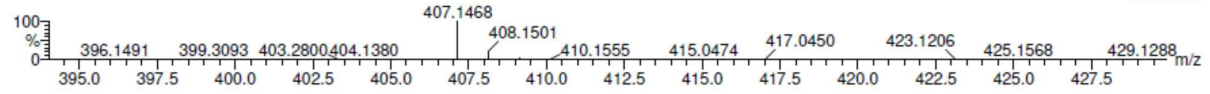
- ***Dimethyl 3,4-bis(3-methoxyphenyl)cyclobutane-1,2-dicarboxylate, 20***

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -2.5, max = 200.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
4 formula(e) evaluated with 1 results within limits (up to 10 closest results for each mass)
Elements Used:
C: 22-22 H: 24-24 O: 6-6 Na: 0-5

FAME-078 49 (0.984) AM2 (Ar,40000.0,0.00,0.00); Cm (10:50) 1: TOF MS ES+ 1.40e+007



Minimum:				-2.5				
Maximum:		5.0	5.0	200.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
407.1468	407.1471	-0.3	-0.7	10.5	1162.9	n/a	n/a	C22 H24 O6 Na

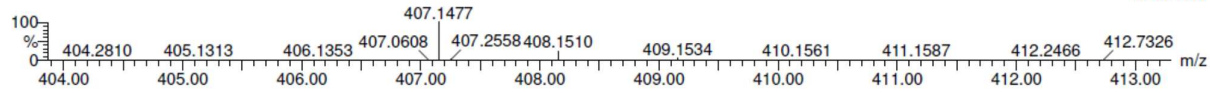
- ***Dimethyl 3,4-bis(4-methoxyphenyl)cyclobutane-1,2-dicarboxylate, 21***

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -2.5, max = 200.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions
1 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 22-22 H: 24-25 O: 6-6 Na: 0-2

FAME-077 1 (0.053) AM2 (Ar,40000.0,0.00,0.00); Cm (1:103) 1: TOF MS ES+ 4.85e+007



Minimum:				-2.5				
Maximum:		5.0	5.0	200.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
407.1477	407.1471	0.6	1.5	10.5	2603.5	n/a	n/a	C22 H24 O6 Na

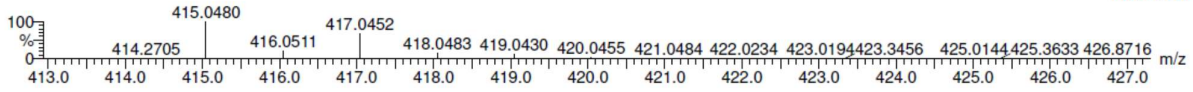
- ***Dimethyl 3,4-bis(2-chlorophenyl)cyclobutane-1,2-dicarboxylate, 22***

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -2.5, max = 200.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
4 formula(e) evaluated with 1 results within limits (up to 10 closest results for each mass)
Elements Used:
C: 20-20 H: 18-18 O: 4-4 Na: 0-5 Cl: 2-2
FAME-099 17 (0.364) AM2 (Ar,40000.0,0.00,0.00); Cm (10:50)

1: TOF MS ES+
5.07e+006



Minimum:				-2.5				
Maximum:		5.0	5.0	200.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
415.0480	415.0480	0.0	0.0	10.5	1115.5	n/a	n/a	C20 H18 O4 Na Cl2

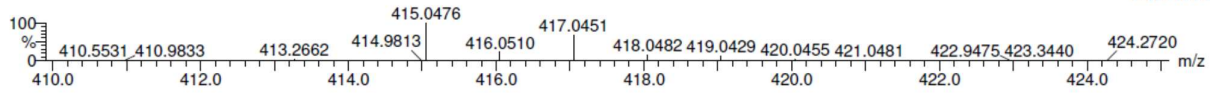
- ***Dimethyl 3,4-bis(4-chlorophenyl)cyclobutane-1,2-dicarboxylate, 23***

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -2.5, max = 200.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions
2 formula(e) evaluated with 1 results within limits (up to 10 closest results for each mass)
Elements Used:
C: 20-20 H: 18-19 O: 4-4 Na: 0-3 Cl: 2-2
FAME-098bis 20 (0.394) AM2 (Ar,40000.0,0.00,0.00); ABS; Cm (20:50)

1: TOF MS ES+
7.68e+006



Minimum:				-2.5				
Maximum:		5.0	5.0	200.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
415.0476	415.0480	-0.4	-1.0	10.5	2024.0	n/a	n/a	C20 H18 O4 Na Cl2

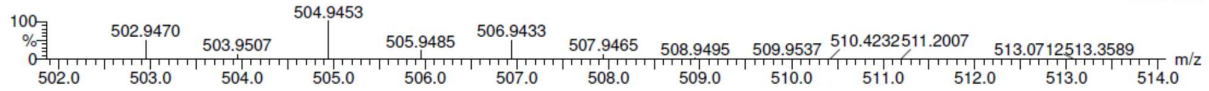
- ***Dimethyl 3,4-bis(3-bromophenyl)cyclobutane-1,2-dicarboxylate, 24***

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 50.0 PPM / DBE: min = -2.5, max = 200.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions
2 formula(e) evaluated with 1 results within limits (up to 10 closest results for each mass)
Elements Used:
C: 20-20 H: 18-19 O: 4-4 Na: 0-3 Br: 2-2

FAME-100_ 20 (0.394) AM2 (Ar,40000.0,0.00,0.00); ABS; Cm (20:50) 1: TOF MS ES+ 5.35e+006



Minimum:				-2.5				
Maximum:		5.0	50.0	200.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
502.9470	502.9470	0.0	0.0	10.5	1734.2	n/a	n/a	C20 H18 O4 Na Br2

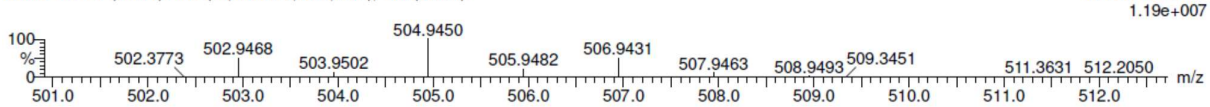
- ***Dimethyl 3,4-bis(4-bromophenyl)cyclobutane-1,2-dicarboxylate, 25***

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -2.5, max = 200.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions
1 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 20-20 H: 18-19 O: 4-4 Na: 0-2 Br: 2-2

FAME-101 20 (0.414) AM2 (Ar,40000.0,0.00,0.00); Cm (10:50) 1: TOF MS ES+ 1.19e+007



Minimum:				-2.5				
Maximum:		5.0	5.0	200.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
502.9468	502.9470	-0.2	-0.4	10.5	1952.6	n/a	n/a	C20 H18 O4 Na Br2

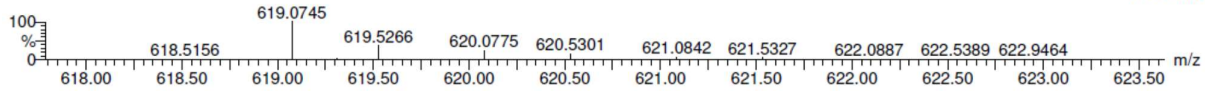
- ***Dimethyl 3,4-bis(3,5-bis(trifluoromethyl)phenyl)cyclobutane-1,2-dicarboxylate, 27***

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -2.5, max = 200.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions
1 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 24-24 H: 16-16 O: 4-4 Na: 0-1 F: 12-12

FAME-137 1 (0.053) AM2 (Ar,40000.0,0.00,0.00); Cm (1:5) 1: TOF MS ES+ 1.79e+006



Minimum:				-2.5				
Maximum:		5.0	5.0	200.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
619.0745	619.0755	-1.0	-1.6	10.5	1200.7	n/a	n/a	C24 H16 O4 Na F12

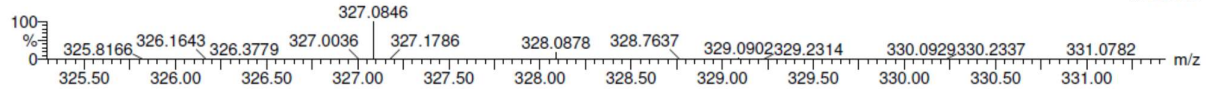
- ***Dimethyl 3,4-di(furan-2-yl)cyclobutane-1,2-dicarboxylate, 28***

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -2.5, max = 200.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions
1 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 16-16 H: 16-16 O: 6-6 Na: 0-1

FAME-081 41 (0.829) AM2 (Ar,40000.0,0.00,0.00); Cm (10:50)

1: TOF MS ES+
1.16e+007



Minimum:				-2.5				
Maximum:		5.0	5.0	200.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
327.0846	327.0845	0.1	0.3	8.5	2175.0	n/a	n/a	C16 H16 O6 Na

- ***Dimethyl 3,4-di(thiophen-2-yl)cyclobutane-1,2-dicarboxylate, 29***

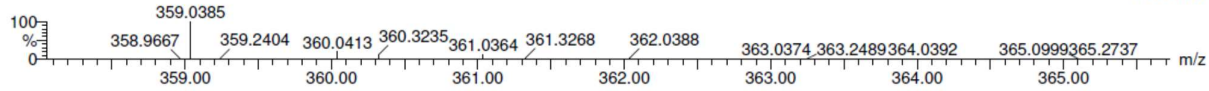
Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -2.5, max = 200.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions
2 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 16-16 H: 16-16 O: 4-4 Na: 0-2 S: 2-2

FAME-133 13 (0.276) AM2 (Ar,40000.0,0.00,0.00); Cm (10:103)

1: TOF MS ES+
2.59e+007



Minimum:				-2.5				
Maximum:		5.0	5.0	200.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
359.0385	359.0388	-0.3	-0.8	8.5	2498.8	n/a	n/a	C16 H16 O4 Na S2

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