Experimental Details:

General Methods. $^1$H, $^{13}$C, NMR spectra were recorded on a Bruker 400 instrument. Chemical shifts ($\delta$) are reported in ppm relative to residual solvent signals for $^1$H and $^{13}$C NMR ($^1$H NMR: 7.26 ppm for CDCl$_3$, 3.34 ppm for CD$_3$OD; $^{13}$C NMR: 77.0 ppm for CDCl$_3$, 49.00 ppm for CD$_3$OD. $^{13}$C NMR spectra were acquired with $^1$H broad band decoupled mode. Mass spectra were recorded on a Micro mass LCT spectrometer using electrospray (ES) ionization techniques. Optical rotations were measured on a Perkin-Elmer 241 polarimeter. The enantiomeric excess (ee) of the products was determined by chiral stationary phase CSP-HPLC (Daicel Chiralpak AD and Chiralcel OD columns), using a UV detector operating at 254 nm. Infrared (IR) spectra were recorded as thin films between NaCl plates. Absorption maximum ($\nu_{\text{max}}$) was reported in wavenumbers (cm$^{-1}$) and only selected peaks are reported.

Materials. Analytical grade solvents and commercially available reagents were used as received, unless otherwise stated. Reactions were checked for completion by TLC (EM Science, silica gel 60 F254). Flash chromatography was performed using silica gel 60 (0.040-0.063 mm, 230-400 mesh). Racemic samples were prepared using tetrabutylammonium bromide as a catalyst at room temperature overnight.
Experimental procedure for the preparation of catalyst \(N\)-(2-methoxy) benzyl quinidinium bromide (4b)

To a stirred suspension of quinidine (3.47 g, 10.7 mmol) in THF (42.0 mL), 2-(methoxy) benzyl bromide (2.80 g, 13.9 mmol) was added. The resulting mixture was heated at 60 °C and stirred for 48 h at the same temperature. After cooling to r.t., the solvent was removed in vacuo. The residue was purified by flash chromatography on silica gel eluting with chloroform:methanol 9:1 affording the title compound as a purple solid (6.94 g, 95% yield). (O. Marianacci, G. Micheletti, L. Bernardi, F. Fini, M. Fochi, D. Pettersen, V. Sgarzani, A. Ricci Chemistry-A. European Journal, 2007, 13, 29, 8338)

\([\alpha]_{D}^{20} = +400 \text{ (c = 0.50 in CH}_{2}\text{Cl})\); \(^1\)H-NMR (400 MHz, CDCl\(_3\)): 8.77 (d, \(J = 4.4\) Hz, 1H), 8.12 (d, \(J = 9.6\) Hz, 1H), 7.96 (dd, \(J = 7.6\) Hz, 1.6 Hz, 1H), 7.91 (d, \(J = 4.4\) Hz, 1H), 7.49 (dt, \(J_\text{d} = 8.6\) Hz, \(J_\text{t} = 1.4\), 1H), 7.40 (dd, \(J = 9.2\) Hz, 2.4 Hz, 1H), 7.18 (d, \(J = 2.4\) Hz, 1H), 7.13 (dt, \(J_\text{d} = 6.5\) Hz, \(J_\text{t} = 1.4\), 1H), 7.01 (d, \(J = 8.4\) Hz, 1H), 6.96 (d, \(J = 6.4\) Hz, 1H), 6.73 (d, \(J = 6.8\) Hz, 1H), 6.27 (d, \(J = 12.0\) Hz, 1H), 6.13-6.06 (m, 1H), 5.28-5.22 (m, 2H), 4.75 (d, \(J = 2.8\) Hz, 1H), 4.73-4.69 (m, 1H), 4.01 (s, 3H), 3.93 (s, 3H), 3.74-3.67 (m, 2H), 3.61 (t, \(J = 12.4\) Hz, 1H), 2.94-2.86 (m, 1H), 2.66-2.61 (m, 1H), 2.50-2.43 (m, 1H), 1.95-1.77 (m, 3H), 1.11-1.04 (m, 1H); \(^{13}\)C-NMR (100.6 MHz): 158.6, 158.2, 148.0, 144.3, 143.3, 136.4, 135.8, 132.7,
132.0, 125.6, 122.0, 121.6, 120.6, 118.6, 115.5, 111.4, 100.6, 69.7, 64.1, 58.6, 56.0, 55.6, 53.9, 53.6, 38.6, 24.1, 24.5, 21.3; HRMS: m/z found [M-Br]+ 445.2482, C_{28}H_{33}N_{2}O_{3} requires 445.2491.
General procedure for the catalytic asymmetric addition of dimethyl bromomalonate to styrylisoxazole (Table 2)

To a test tube equipped with a magnetic stirring bar, were sequentially added the relevant styrylisoxazole 1a-k (0.10 mmol, 1.0 equiv), toluene (5.0 mL), catalyst 4b (2.63 mg, 0.005 mmol, 0.050 equiv) and dimethyl bromomalonate 2 (27 µL, 0.20 mmol, 2.0 equiv). The test tube was placed at the stated temperature (-37° C) then K₃PO₄ 50% w/w was added in one portion (0.140 mL, 0.50 mmol, 5.0 equiv). The mixture was then stirred vigorously and after the stated time the reaction was quenched with NH₄Cl sat. sol. (4 mL) and the product extracted with toluene (3 x 1 mL). The combined organic phases were evaporated in vacuo and the residue was purified by flash chromatography on silica gel eluting with petroleum ether:EtOAc mixtures.

(+)-Dimethyl -2-(3-methyl-4-nitroisoxazol-5-yl)-3-phenylcyclopropane-1,1-dicarboxylate (7a)

Prepared following general procedure using 3-methyl-4-nitro-5-styrylisoxazole 1a (23 mg, 0.10 mmol). Reaction time: 24 h. Column chromatography (Solvents petroleum ether:EtOAc 9:1) afforded 7a as a yellow oil (33.5 mg, 93% yield).

The ee of the product was determined by CSP-HPLC using a Chiralpak AD column (n-hexane/i-PrOH 90:10, flow rate 1.0 mL/min, tₘₐₓ = 14.95 min, tₘᵢₙ = 13.86 min, 89% ee); [α]D²⁰ = +20 (c = 0.50 in CHCl₃); ¹H-NMR (400 MHz, CDCl₃): 7.34-7.30 (m, 5H), 4.26 (d, J = 8.4 Hz, 1H), 3.96 (d, J = 8.4 Hz, 1H), 3.74 (s,
(+)-Dimethyl -2-(4-methoxyphenyl)-3-(3-methyl-4-nitroisoxazol-5-yl)cyclopropane-1,1-dicarboxylate (7b)

\[
\begin{array}{c}
\text{Me} \\
\text{N} \quad \text{NO}_2 \\
\text{COCMe} \\
\text{COCMe} \\
\text{MeO} \\
\end{array}
\]

Prepared following general procedure using 5-(4-methoxystyryl)-3-methyl-4-nitroisoxazole 1b (26 mg, 0.10 mmol). Reaction time: 42 h. Column chromatography (Solvents petroleum ether:EtOAc 9:1) afforded \(7b\) as a yellow oil (38.7 mg, 99% yield).

The ee of the product was determined by CSP-HPLC using a Chiralpak AD column (\(n\)-hexane/iPrOH 80:20, flow rate 1.0 mL/min, \(t_{maj} = 18.06\) min, \(t_{min} = 14.31\) min, 90% ee); \([\alpha]_D^{20} = +23\) (c = 0.55 in CHCl₃); \(^1\)H-NMR (400 MHz, CDCl₃): 7.24-7.22 (m, 2H), 6.86-6.84 (m, 2H), 4.20 (d, \(J = 8.4\) Hz, 1H), 3.90 (d, \(J = 8.4\) Hz, 1H), 3.79 (s, 3H), 3.73 (s, 3H), 3.53 (s, 3H), 2.57 (s, 3H); \(^{13}\)C-NMR (100.6 MHz): 168.9, 166.5, 165.2, 159.6, 156.1, 129.8, 124.0, 114.1, 55.4, 53.6, 53.2, 45.0, 36.1, 29.8, 26.6, 11.8; IR (NaCl) / cm⁻¹: 1742 s, 1548 s; HRMS: \(m/z\) found [M+H]^+ 391.1135, \(C_{18}H_{19}N_2O_8\) requires 391.1141.

(+)-Dimethyl -2-(2,3-dichlorophenyl)-3-(3-methyl-4-nitroisoxazol-5-yl)cyclopropane-1,1-dicarboxylate (7c)
Prepared following general procedure using 5-(2,3-dichlorostyryl)-3-methyl-4-nitroisoxazole 1c (30 mg, 0.10 mmol). Reaction time: 42 h. Column chromatography (Solvents petroleum ether:EtOAc 9:1) afforded 7c as a yellow oil (42.4 mg, 99% yield).

The ee of the product was determined by CSP-HPLC using a Chiralpak AD column (n-hexane/iPrOH 80:20, flow rate 1.0 mL/min, t\text{maj} = 10.98 min, t\text{min} = 8.98 min, 91% ee); [\alpha]_D^{20} = +20 (c = 1.00 in CHCl_3); \textsuperscript{1}H-NMR (400 MHz, CDCl_3): 7.46-7.43 (m, 1H), 7.22-7.21 (m, 2H), 4.29 (d, J = 8.4 Hz, 1H), 4.00 (d, J = 8.4 Hz, 1H), 3.76 (s, 3H), 3.58 (s, 3H), 2.58 (s, 3H); \textsuperscript{13}C-NMR (100.6 MHz): 168.1, 165.8, 165.2, 156.2, 134.1, 133.6, 132.9, 130.6, 128.2, 127.3, 53.7, 53.5, 44.4, 35.8, 29.8, 27.2, 11.7; IR (NaCl) / cm\textsuperscript{-1}: 1754 s, 1518 s; HRMS: m/z found [M+H\textsuperscript{+}] \textsuperscript{429.0266, C_17H_{15}N_2O_7Cl_2 requires 429.0256.

(+)-Dimethyl -2-(2-chlorophenyl)-3-(3-methyl-4-nitroisoxazol-5-yl)cyclopropane-1,1-dicarboxylate (7d)

Prepared following general procedure using 5-(2-chlorostyryl)-3-methyl-4-nitroisoxazole 1d (27 mg, 0.10 mmol). Reaction time: 48 h. Column chromatography (Solvents petroleum ether:EtOAc 9:1) afforded 7d as a yellow oil (35.8 mg, 91% yield).
The ee of the product was determined by CSP-HPLC using a Chiralpak AD column (n-hexane/iPrOH 90:10, flow rate 1.0 mL/min, t\textsubscript{maj} = 14.50 min, t\textsubscript{min} = 12.97 min, 92% ee); [\alpha\textsubscript{D}\textsuperscript{20}] = +50 (c = 0.65 in CHCl\textsubscript{3}); \textsuperscript{1}H-NMR (400 MHz, CDCl\textsubscript{3}): 7.41-7.39 (m, 1H), 7.31-7.27 (m, 3H), 4.31 (d, J = 8.0 Hz, 1H), 4.01 (d, J = 8.4, 1H), 3.76 (s, 3H), 3.55 (s, 3H), 2.58 (s, 3H); \textsuperscript{13}C-NMR (100.6 MHz): 168.5, 166.0, 165.3, 156.2, 135.8, 130.6, 129.9, 129.8, 129.7, 126.9, 53.6, 53.3, 44.4, 35.4, 29.8, 26.9, 11.8; IR (NaCl) / cm\textsuperscript{-1}: 1730 s, 1533 s; HRMS: m/z found [M+H]\textsuperscript{+} 395.0636, C\textsubscript{17}H\textsubscript{16}N\textsubscript{2}O\textsubscript{7}Cl requires 395.0646.

(+)-Dimethyl 2-(4-fluorophenyl)-3-(3-methyl-4-nitroisoxazol-5-yl)cyclopropane-1,1-dicarboxylate (7e)

Prepared following general procedure using 5-(4-fluorostyryl)-3-methyl-4-nitroisoxazole 1e (25 mg, 0.10 mmol). Reaction time: 24 h. Column chromatography (Solvents petroleum ether:EtOAc 9:1) afforded 7e as a yellow oil (37.4 mg, 99% yield).

The ee of the product was determined by CSP-HPLC using a Chiralpak AD column (n-hexane/iPrOH 80:20, flow rate 1.0 mL/min, t\textsubscript{maj} = 12.32 min, t\textsubscript{min} = 10.46 min, 87% ee); [\alpha\textsubscript{D}\textsuperscript{20}] = +10 (c = 0.72 in CHCl\textsubscript{3}); \textsuperscript{1}H-NMR (400 MHz, CDCl\textsubscript{3}): 7.32-7.29 (m, 2H), 7.05-7.01 (m, 2H), 4.22 (d, J = 8.4 Hz, 1H), 3.91 (d, J = 8.4 Hz, 1H), 3.74 (s, 3H), 3.53 (s, 3H), 2.57 (s, 3H); \textsuperscript{13}C-NMR (100.6 MHz): 168.5, 166.3, 165.0, 162.7 (d, J = 247 Hz), 156.2, 130.4 (d, J = 8.0 Hz), 128.0 (d, J = 3.0 Hz), 115.8 (d, J = 21 Hz), 53.7, 53.3, 44.9, 35.7, 31.7, 29.8, 11.7; IR
(+)-Dimethyl 2-(3-methyl-4-nitroisoxazol-5-yl)-3-(thiophen-2-yl)cyclopropane-1,1-dicarboxylate (7f)

Prepared following general procedure using 3-methyl-4-nitro-5-(2-(thiophen-2-yl)vinyl)isoxazole 1f (24 mg, 0.10 mmol). Reaction time: 42 h. Column chromatography (Solvents petroleum ether:EtOAc 9:1) afforded 7f as a yellow oil (35.8 mg, 98% yield).

The ee of the product was determined by CSP-HPLC using a Chiralpak AD column (n-hexane/iPrOH 90:10, flow rate 1.0 mL/min, t\textsubscript{maj} = 16.38 min, t\textsubscript{min} = 14.81 min, 87% ee); [\(\alpha\)]\textsubscript{D}\textsuperscript{20} = +13 (c = 0.58 in CHCl\textsubscript{3}); \(^1\)H-NMR (400 MHz, CDCl\textsubscript{3}): 7.24 (d, J = 0.8 Hz, 1H), 7.01-7.00 (m, 1H), 6.97-6.95 (m, 1H), 4.18 (d, J = 8.0 Hz, 1H), 3.99 (d, J = 8.0 Hz, 1H), 3.73 (s, 3H), 3.61 (s, 3H), 2.57 (s, 3H); \(^{13}\)C-NMR (100.6 MHz): 168.0, 166.1, 164.8, 156.1, 134.8, 127.5, 127.1, 126.3, 53.7, 53.4, 45.3, 31.3, 29.8, 28.2, 11.7; IR (NaCl) / cm\textsuperscript{-1}: 1734 s, 1549 s; HRMS: m/z found [M+H]\textsuperscript{+} 367.0582, C\textsubscript{15}H\textsubscript{13}N\textsubscript{2}O\textsubscript{7}S requires 367.0600.
(+)-Dimethyl 2-(furan-2-yl)-3-(3-methyl-4-nitroisoxazol-5-yl)cyclopropane-1,1-dicarboxylate (7g)

Prepared following general procedure using 5-(2-(furan-2-yl)vinyl)-3-methyl-4-nitroisoxazole 1g (22 mg, 0.10 mmol). Reaction time: 42 h. Column chromatography (Solvents petroleum ether:EtOAc 9:1) afforded 7g as a yellow oil (34.8 mg, 99% yield).

The ee of the product was determined by CSP-HPLC using a Chiralcel OD column (n-hexane/iPrOH 90:10, flow rate 1.0 mL/min, t_maj = 23.55 min, t_min = 15.32 min, 86% ee); [α]_D^{20} = +24 (c = 0.53 in CHCl_3); \(^1\)H-NMR (400 MHz, CDCl_3): 7.35-7.34 (m, 1H), 6.35-6.33 (m, 2H), 4.11 (d, \(J = 8.0\) Hz, 1H), 3.84 (d, \(J = 7.6\) Hz, 1H), 3.71 (s, 3H), 3.68 (s, 3H), 2.57 (s, 3H); \(^{13}\)C-NMR (100.6 MHz): 167.8, 166.0, 165.0, 156.1, 146.6, 143.0, 111.0, 109.5, 53.7, 53.5, 43.7, 29.8, 29.3, 26.5, 11.7; IR (NaCl) / cm\(^{-1}\): 1734 s, 1518 s; HRMS: m/z found [M+H]^+ 351.0820, C_{15}H_{15}N_{2}O_{8} requires 351.0828.

(+)-Dimethyl 2-(2-methoxyphenyl)-3-(3-methyl-4-nitroisoxazol-5-yl)cyclopropane-1,1-dicarboxylate (7h)

Prepared following general procedure using 5-(2-methoxystyryl)-3-methyl-4-nitroisoxazole 1h (26 mg, 0.10 mmol).
Reaction time: 72 h. Column chromatography (Solvents petroleum ether:EtOAc 9:1) afforded 7h as a yellow oil (37.8 mg, 97% yield).

The ee of the product was determined by CSP-HPLC using a Chiralcel OD column (n-hexane/i-PrOH 95:5, flow rate 0.50 mL/min, t\text{maj} = 39.46 min, t\text{min} = 29.21 min, 96% ee); [α]_D^{20} = +5.0 (c = 0.31 in CHCl_3); \text{^1}H-NMR (400 MHz, CDCl_3): 7.31-7.27 (m, 1H), 7.18-7.16 (m, 1H), 6.94-6.90 (m, 1H), 6.87 (d, J = 8.4 Hz, 1H), 4.23 (d, J = 8.4 Hz, 1H), 3.94 (d, J = 8.4 Hz, 1H), 3.83 (s, 3H), 3.76 (s, 3H), 3.51 (s, 3H), 2.58 (s, 3H); \text{^13}C-NMR (100.6 MHz): 169.3, 166.6, 165.6, 158.7, 156.1, 129.7, 129.2, 129.0, 128.4, 120.9, 110.5, 55.7, 53.5, 53.0, 44.4, 32.9, 26.7, 11.8; IR (NaCl) / cm\(^{-1}\): 1734 s, 1518 s; HRMS: m/z found [M-H]^- 389.0966, C_{18}H_{17}N_2O_8 requires 389.0985.

(+)-Dimethyl 2-(2-bromophenyl)-3-(3-methyl-4-nitroisoxazol-5-yl)cyclopropane-1,1-dicarboxylate (7i)

\[
\begin{align*}
\text{Me} & \\
\text{NO}_2 & \\
\text{COOMe} & \\
\text{COOMe} & \\
\text{Br} & \\
\end{align*}
\]

Prepared following general procedure using 5-(2-bromostyryl)-3-methyl-4-nitroisoxazole 1i (31 mg, 0.10 mmol). Reaction time: 45 h. Column chromatography (Solvents petroleum ether:EtOAc 9:1) afforded 7i as a yellow oil (43.4 mg, 99% yield).

The ee of the product was determined by CSP-HPLC using a Chiralpak AD column (n-hexane/i-PrOH 90:10, flow rate 1.0 mL/min, t\text{maj} = 16.78 min, t\text{min} = 15.22 min, 93% ee); [α]_D^{20} = +1.0 (c = 0.62 in CHCl_3); \text{^1}H-NMR (400 MHz, CDCl_3): 7.60-7.58 (m, 1H), 7.33-7.26 (m, 2H), 7.23-7.18 (m, 1H), 4.31 (d, J = 8.4 Hz, 1H), 3.99 (d, J = 8.6 Hz, 1H), 3.76 (s, 3H), 3.55 (s,
(+)-Dimethyl 2-(2,4-dimethoxyphenyl)-3-(3-methyl-4-nitroisoxazol-5-yl)cyclopropane-1,1-dicarboxylate (7j)

Prepared following general procedure using 5-(2,4-dimethoxystyryl)-3-methyl-4-nitroisoxazole 1j (29 mg, 0.10 mmol). Reaction time: 72 h. Column chromatography (Solvents petroleum ether:EtOAc 85:15) afforded 7j as a yellow oil (39.1 mg, 93% yield).

The ee of the product was determined by CSP-HPLC using a Chiralcel OD column (n-hexane/i-PrOH 80:20, flow rate 0.75 mL/min, t_maj = 23.57 min, t_min = 20.01 min, 94% ee); $[\alpha]_D^{20} = +4.0$ (c = 0.36 in CHCl$_3$); $^1$H-NMR (400 MHz, CDCl$_3$): 7.08-7.05 (m, 1H), 6.44-6.42 (m, 2H), 4.18 (d, $J = 8.4$ Hz, 1H), 3.87 (d, $J = 8.4$ Hz, 1H), 3.80 (s, 3H), 3.79 (s, 3H), 3.72 (s, 3H), 3.54 (s, 3H), 2.57 (s, 3H); $^{13}$C-NMR (100.6 MHz): 169.4, 166.7, 165.6, 161.1, 159.7, 156.1, 129.5, 113.2, 104.0, 98.6, 55.7, 55.5, 53.4, 53.1, 44.4, 32.7, 29.8, 26.7, 11.8; IR (NaCl) / cm$^{-1}$: 1734 s, 1510 s; HRMS: m/z found [M-H]$^- 419.1109$, C$_{19}$H$_{19}$N$_2$O$_9$ requires 419.1091.

(+)-Dimethyl 2-hexyl-3-(3-methyl-4-nitroisoxazol-5-yl)cyclopropane-1,1-dicarboxylate (7k)
Prepared following general procedure using 3-methyl-4-nitro-5-(oct-1-en-1-yl)isoxazole 1k (23 mg, 0.10 mmol). Reaction time: 60 h. Column chromatography (Solvents petroleum ether:EtOAc 95:5) afforded 7k as a colourless oil (36.4 mg, 99% yield).

The ee of the product was determined by CSP-HPLC using a Chiralpak AD column (n-hexane/i-PrOH 95:5, flow rate 1.0 mL/min, t_{maj} = 6.26 min, t_{min} = 7.49 min, 84% ee); [α]_D^{20} = +7.0 (c = 0.32 in CHCl_3); ^1H-NMR (400 MHz, CDCl_3): 3.83-3.82 (m, 4H), 3.65 (s, 3H), 3.55 (d, J = 7.6 Hz, 1H), 2.53 (s, 3H), 1.64-1.58 (m, 2H), 1.49-1.40 (m, 2H), 1.34-1.24 (m, 6H), 0.89-0.85 (m, 3H); ^13C-NMR (100.6 MHz): 169.4, 166.8, 166.5, 165.1, 156.0, 54.1, 53.4, 43.2, 41.7, 33.1, 31.7, 28.8, 28.1, 27.2, 22.6, 14.1, 11.7; IR (NaCl) / cm⁻¹: 1750 s, 1526 s; HRMS: m/z found [M-H]⁻ 367.1512, C_{17}H_{23}N_{2}O_{7} requires 367.1505.

(+)-Dimethyl 2-(2,3-dihydrobenzo[b][1,4]dioxin-6-yl)-3-(3-methyl-4-nitroisoxazol-5-yl)cyclopropane-1,1-dicarboxylate (7l)

Prepared following general procedure using 5-(2-(2,3-dihydrobenzo[b][1,4]dioxin-5-yl)vinyl)-3-methyl-4-nitroisoxazole 1l (29 mg, 0.10 mmol). Reaction time: 48 h. Column chromatography (Solvents petroleum ether:EtOAc 8:2) afforded 7l as a yellow oil (39.8 mg, 95% yield).
The ee of the product was determined by CSP-HPLC using a Chiralpak AD column (n-hexane/iPrOH 80:20, flow rate 1.0 mL/min, t_maj = 18.58 min, t_min = 23.14 min, 90% ee); [α]_D^{20} = -23 (c = 0.88 in CHCl3); ¹H-NMR (400 MHz, CDCl₃): 6.82-6.75 (m, 3H), 4.23 (s, 4H), 4.15 (d, J = 8.0 Hz, 1H), 3.84 (d, J = 8.0 Hz, 1H), 3.72 (s, 3H), 3.58 (s, 3H), 2.56 (s, 3H); ¹³C-NMR (100.6 MHz): 168.8, 166.4, 165.2, 156.1, 143.7, 143.6, 125.4, 125.2, 121.5, 117.6, 117.5, 64.4, 64.4, 53.6, 53.3, 44.9, 36.0, 26.7, 11.7; IR (NaCl) / cm⁻¹: 1742 s, 1522 s; HRMS: m/z found [M+H]^⁺ 419.1078, C₁₉H₁₉N₂O₉ requires 419.1091.
Electronic Supplementary Material (ESI) for Chemical Communications
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General procedure for the oxidation and successive methylation with trimethylsilyl diazomethane of cyclopropanes 7a and 7b.

To a solution of cyclopropane 7 (a, b) (0.1 mmol) in THF (1.0 mL) was added dropwise a solution of KMnO₄ in H₂O : Acetone (3.5 :1 ). The reaction mixture was stirred for 1 h at room temperature and then a Na₂SO₃ saturated solution (10 mL) was added to destroy the excess of KMnO₄: the formation of a brown precipitate was observed (MnO₂). The mixture was then acidified with HCl 6 M until pH = 3. At this point it was noted that the solution became clear. The mixture was then extracted with DCM (3 x 1mL) and the combined organic phases were evaporated. The crude (assumed 0.1 mmol) was then dissolved in diethyl ether : MeOH (9 : 1, 1 mL) and treated with trimethylsilyl diazomethane (0.6 mmol, 95 μl) added drop-wise until the formation of bubbles ceased. The mixture was then filtered through a small silica plug end evaporated. The product was then purified by chromatography on silica gel using dichloromethane as the eluent.

(+)-Trimethyl 3-phenylcyclopropane-1,1,2-tricarboxylate 8a.

Following the general procedure compound 8a was obtained as a pale yellow oil (18 mg, 60% for two steps). The ee of the product was determined by HPLC using a Chiralpak AD column (n-hexane/iPrOH 80:20, flow rate 1.0 mL/min, tₘₐₖ = 8.76 min, tₘᵢₙ = 5.77 min, 85% ee); ¹H-NMR (400 MHz, CDC13): 7.18 (m, 5H), 3.71 (s, 3H), 3.66 (s, 3H), 3.54 (d, J = 7.6 Hz, 1H), 3.38 (s, 3H), 3.15 (d, J = 7.6 Hz, 1H); ¹³C-NMR (100.6 MHz): 169.68, 166.58, 165.75, 132.92, 128.67, 128.49, 127.93, 53.32, 53.02,
52.75, 44.18, 36.22, 31.05; m/z found [M+H]$^+$ 293.112, C$_{15}$H$_{17}$O$_6$ requires 293.1025.

(+)-trimethyl 3-(4-methoxyphenyl)cyclopropane-1,1,2-tricarboxylate 8b.

Following the general procedure compound 8b was obtained as a pale yellow oil (20 mg, 62% for two steps). The ee of the product was determined by HPLC using a Chiralpak AD column (n-hexane/iPrOH 80:20, flow rate 1.0 mL/min, $t_{maj}$ = 20.64 min, $t_{min}$ = 8.66 min, 87% ee); $^1$H-NMR (400 MHz, CDC$_3$): 7.17 (d, $J$ = 8.4 Hz, 2H), 6.8 (d, $J$ = 8.4 Hz, 2H), 3.81 (s, 3H), 3.78 (s, 3H), 3.75 (s, 3H), 3.58 (d, $J$ = 7.6 Hz, 1H), 3.50 (s, 3H), 3.21 (d, $J$ = 7.6 Hz, 1H); $^{13}$C-NMR (100.6 MHz): 169.74, 166.66, 165.82, 159.21, 129.77, 124.77, 113.86, 55.34, 53.28, 53.05, 52.72, 44.21, 35.74, 31.20; m/z found [M+H]$^+$ 323.1102, C$_{16}$H$_{19}$O$_7$ requires 323.1131.

(+)-3-Phenyl-cyclopropane-1,1,2-tricarboxylic acid dimethyl ester 10.

$^1$H-NMR (400 MHz, CDC$_3$): 7.33-7.25 (m, 5H), 3.81 (s, 3H), 3.66 (d, $J$ = 7.2, 1H), 3.50 (s, 3H), 3.25 (d, $J$ = 7.2, 1H), $^{13}$C-NMR (100.6 MHz): 174.9, 166.3, 165.5, 132.6, 128.7, 128.6, 128.1, 53.4, 53.1, 44.7, 36.6, 30.8; m/z found [M+H]$^+$ 279.0894, C$_{14}$H$_{15}$O$_6$ requires 279.0869.
Electronic Supplementary Material (ESI) for Chemical Communications
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Electronic Supplementary Material (ESI) for Chemical Communications
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Electronic Supplementary Material (ESI) for Chemical Communications
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**Missing Component Report**
Component Expected Retention (Calibration File)

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Sample Number : A
AutoSampler : SER200
Instrument Name : PerkinElmer LC
Instrument Serial # : None
Delay Time : 0.00 min
Sampling Rate : 2.2727 pts/s
Sample Volume : 1.000000 ul
Sample Amount : 1.0000
Data Acquisition Time : 17/12/2010 14:58:23

Date : 14/04/2011 16:56:56
Sample Name : CD65 Racemate
Study : 
Rack/Vial : 0/12
Channel : B
A/D mV Range : 1000
End Time : 32.35 min
Area Reject : 0.000000
Dilution Factor : 1.00
Cycle : 4

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Missing Component Report
Component Expected Retention (Calibration File)

All components were found
Electronic Supplementary Material (ESI) for Chemical Communications
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Missing Component Report:
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All components were found.
Electronic Supplementary Material (ESI) for Chemical Communications
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Component Expected Retention (Calibration File)

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