Access to distal meta-C–H functionalization of arylmethanesulfonic acid derivatives

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Supporting Information
Supporting Information

Experimental procedure and spectral data for all new compounds  
\textsuperscript{1}H- and \textsuperscript{13}C-NMR spectra for all new compounds  
X-ray crystals data and references

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S50-S113  
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Experimental:

General: IR spectra were recorded on a Bruker Tensor 37 (FTIR) spectrophotometer. \(^1\)H NMR spectra were recorded on Bruker Avance 400 (400 MHz) spectrometer at 295 K in CDCl\(_3\); chemical shifts (\(\delta\) ppm) and coupling constants (Hz) are reported in standard fashion concerning either internal standard tetramethylsilane (TMS) (\(\delta_H = 0.00\) ppm) or CDCl\(_3\) (\(\delta_H = 7.25\) ppm). \(^{13}\)C\\{\(^1\)H\}\ NMR spectra were recorded on Bruker Advance 400 (100 MHz) spectrometer at RT in CDCl\(_3\). Chemical shifts (\(\delta\) ppm) are reported relative to CDCl\(_3\) [\(\delta_C = 77.00\) ppm (central line of the triplet)]. In \(^1\)H-NMR, the following abbreviations were used throughout: s = singlet, d = doublet, t = triplet, q = quartet, qui = quintet, sept = septet, dd = doublet of doublet, m = multiplet and br. s = broad singlet. In \(^{13}\)C\\{\(^1\)H\}\ NMR, the nature of carbons (C, CH, CH\(_2\), and CH\(_3\)) was determined by recording the DEPT-135 spectra. The assignment of signals was confirmed by \(^1\)H, \(^{13}\)C\\{\(^1\)H\}\ CPD, and DEPT spectra. High-resolution mass spectra (HR-MS) were recorded on an Agilent 6538 UHD Q-TOF electron spray ionization (ESI) mode and atmospheric pressure chemical ionization (APCI) modes. Melting points are recorded using Tempo and Mettler FP1 melting point apparatus in capillary tubes and are uncorrected. A single crystal of 3jb was selected and mounted on an Oxford SuperNova, Dual, Cu at zero, Eos diffractometer. The crystal was kept at 298 K during data collection. Using Olex2, the structure was solved with the olex2.solve structure solution program using direct methods, and refined with the olex2. refinement package using Gauss–Newton minimization. All small-scale reactions were carried out by using a Schlenk tube. Reactions were monitored by TLC on silica gel using a combination of hexane and ethyl acetate as eluents. Solvents were distilled before use; petroleum ether the boiling range of 60–80 °C was used. Pd(OAc)\(_2\), Ac-Gly-OH, AgOAc and 1,1,1,3,3,3-hexafluoro-2-propanol (HFIP) were purchased from Sigma-Aldrich and used as received. Olefin coupling partners, NCS, DMF, DCM, ACN, K\(_2\)CO\(_3\), NaOH and NEt\(_3\) were purchased from Sigma-Aldrich/TCI/local sources and used as received. Acme’s silica gel (60–120 mesh) was used for column chromatography (approximately 20 g per one gram of crude material).
Table S1. Optimization conditions for meta-C–H olefination of arylmethanesulfonate 1a.

![Chemical structure](image)

<table>
<thead>
<tr>
<th>Entry</th>
<th>Oxidant</th>
<th>Ligand</th>
<th>Time (min)</th>
<th>Irradiation temp (°C)</th>
<th>Yield of 3ab (m:o)</th>
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<tr>
<td>1</td>
<td>Ag₂CO₃</td>
<td>Ac-Gly-OH</td>
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<tr>
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<tr>
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<tr>
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<td>N-Boc-L-valine</td>
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<td>90</td>
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</table>

Reaction conditions: ᵃYields of isolated product 3ab. Regioselectivity was determined by ¹H-NMR analysis. ᵇ1.5 equiv of 2b was used. ᶜBis-olefination product was observed. ᵈTFE as the solvent. ᵉAcetic acid as the solvent. ᵇAg₂CO₃ was used as the catalyst. ⁷PdCl₂(PPh₃)₂ was used as the catalyst. ᵈPd(PPh₃)₄ was used as the catalyst. ⁹Only 37% of the product 3ab was observed when the reaction was carried out for 15 minutes at 60 °C by using Ag₂CO₃ as the oxidant (Entry 1, Table S1). Notably, when the oxidant was changed from Ag₂CO₃ to AgOAc, the product 3ab yield was improved to 59% with “meta:others = 99:01”. When AgO was used as the oxidant, the yield decreased drastically to 9% (Entry 3, Table S1). Unfortunately, only a trace amount of the product was observed when Cu(OAc)₂ was used as an oxidant. Later, the ligands were changed, such as N-Boc-Gly-OH (Entry 7, Table S1) and N-Boc-L-valine (Entry 8, Table S1) failed to provide the product 3ab. Notably, the yield of 3ab was improved to 67% when the time was increased to 20 min (Entry 9, Table S1). Later, the time is increased to 25 min, and the temperature rose to 75 °C. Then, it was observed that yield of 3ab improved to 75% (Entry 10, Table S1). Significantly, it was observed that the increase in the concentration of olefin (1.5 equiv) resulted in improving the mono-olefination product 3ab in 83% of the yield with exclusive meta-selectivity along with trace amount of bis-olefination product (Entry 11, table S1). Encouraged by the above observation, we intended to increase the time further to 35 minutes, but it observed that the product 3ab had been started converting to the bis-olefination product was formed in just 10% yield (entry 12, Table S1). Unfortunately, the reaction not shown any
progress when the reaction was carried out in TFE and AcOH as the solvent (Entry 13, Table S1). In contrast, the reactions did not furnish any product using other palladium catalysts, such as with PdCl₂, PdCl₂(PPh₃)₂, and Pd(PPh₃)₄ (Entry 14, Table S1).

The required template bearing synthetic precursor 1 have been synthesized in two steps, as depicted in Scheme S1.

Scheme S1. Synthetic sequence for the preparation of arylmethanesulfonate ester template 1.

Table S2. Reported sulfonyl chloride starting materials.¹
General Procedure-1 (GP-1) Preparation of arylmethanesulfonyl chlorides (6):
To an oven dried 50 mL round bottom flask charged with a magnetic stirring bar, were added bezyl halide 7 (1 equiv) and thiourea (1 equiv) were refluxed together in EtOH (5 mL) at 70 °C for 3 h. The reaction progress was monitored by TLC, then EtOH was removed under reduced pressure, the obtained solid or sticky oil was slowly added to a mixture of N-chlorosuccinimide (5 equiv), 2 M HCl (2.5 mL), and MeCN (8 mL) in a 10 °C water bath to maintain the internal temperature between 10 and 20 °C and allowed the mixture to stir for 1 h. After completion of the reaction, the mixture was quenched by the addition of an aqueous NH₄Cl solution and extracted with ethyl acetate (3×20 mL). The organic phase was separated, dried (Na₂SO₄), and concentrated under reduced pressure and purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate) furnished the arylmethanesulfonyl chlorides 6 in quantitative yield, as a white or brown solid/liquid.

General Procedure-2 (GP-2) for the Preparation of Arylmethanesulfonate Esters (1):
To an oven dried 25 mL round bottom flask charged with a magnetic stirring bar, were added arylmethanesulfonyl chloride 6 (1 equiv), nitrile alcohol template 7 (1 equiv), in Dichloromethane (15 mL) as solvent then triethyl amine (1.5 equiv) was added dropwise to the reaction mixture. The reaction mixture was stirred at room temperature for 4 h. The conversion was monitored by TLC. Then, the mixture was quenched by the addition of an aqueous NH₄Cl solution and extracted with dichloromethane (3×15 mL). The organic layers were washed with saturated NaCl solution, dried (Na₂SO₄), and filtered. Evaporation of the solvent under reduced pressure and purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate) furnished the arylmethanesulfonate esters 1 in quantitative yields.

General Procedure-3 (GP-3) for Microwave Assisted meta-Selective C–H Olefination (3):
An oven-dried 10 mL glass vial equipped with a magnetic stirring bar, was charged with arylmethanesulfonate ester 1 (0.2 mmol), olefin 2 (0.3 mmol), Pd(OAc)₂ (5 mg, 10 mol%), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (67 mg, 0.4 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL). The resulting reaction mixture was subjected to microwave irradiation 90 °C for 25 minutes. The reaction mixture was cooled to room temperature. The progress of the reaction was monitored by TLC. The reaction mixture diluted with ethyl acetate (15 mL) and
filtered through a short pad of celite with an additional amount of ethyl acetate (15 mL). Evaporation of the solvent(s) under reduced pressure and purification of the residue by silica gel column chromatography using petroleum ether/ethyl acetate as the eluent furnished the desired meta-olefinated product 3 in 52% to 83% yields, as viscous colourless/pale-yellow/brown liquid or solid.

**General Procedure-4 (GP-4) for Microwave Assisted One-pot Homo-Bis-meta-Selective C–H Olefination (4):**

An oven-dried 10 mL glass vial equipped with a magnetic stirring bar, was charged with arylmethanesulfonate ester 1 (0.2 mmol), olefin 2 (0.72 mmol), Pd(OAc)$_2$ (5 mg, 10 mol%), N-Ac-Gly-OH (10 mg, 40 mol%), AgOAc (134 mg, 0.8 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL). The resulting reaction mixture was subjected to microwave irradiation at 100 °C for 35 minutes. The reaction mixture was cooled to room temperature. The progress of the reaction was monitored by TLC. The reaction mixture diluted with ethyl acetate (15 mL) and filtered through a short pad of celite with an additional amount of ethyl acetate (15 mL). Evaporation of the solvent(s) under reduced pressure and purification of the residue by silica gel column chromatography using petroleum ether/ethyl acetate as the eluent furnished the desired homo-bis-meta-olefinated product 4a-e in 60% to 70% yields, as viscous colourless/pale-yellow/brown liquid or solid.

**Plausible catalytic cycle for the formation of mono-meta olefination product 3:**

The plausible catalytic cycle for the coupling has been proposed based on the previous reports$^{2,3}$ as described in Scheme S2. Essentially, the weak coordination of nitrile group with palladium leads the metal-center to the close vicinity of the meta-C–H bond and to facilitate to form 11 membered palladacycle II by cleaving the meta-C–H bond. Then olefin binding with Pd-centre of II takes place resulted in forming intermediate III. Subsequently, 1,2-migratory insertion leads to the formation of IV followed by β-hydride elimination occasioned the meta-olefination product 3. Sequentially, the reductive elimination of acetate and hydrogen (as AcOH) from H-Pd-OAc, resulted in forming zero oxidation state palladium. The reoxidation of the Pd(0) to Pd (II) can be done by the silver(I) salt.
Scheme S2. Proposed mechanistic cycle.

Removal of the directing template:
Thus, base hydrolysis of arylmethanesulfonate ester-derived olefin product 3ab using potassium hydroxide in methanol at room temperature afforded the potassium salt product 5 in 95% yield along with 92% of recovery of nitrile alcohol template 7, which has been reused further (Scheme S3).

Scheme S3. Removal of the directing template.
2-Cyano-2-isobutyl-4-methylpentyl phenylmethanesulfonate (1a): GP-2 was carried out with arylmethanesulfonyl chloride 6a (114 mg, 0.6 mmol), triethyl amine (90 mg, 0.9 mmol), nitrile alcohol (110 mg, 0.6 mmol) in dichloromethane (DCM) (15 mL) solvent at room temperature for 4 h. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 95/5), furnished the product 1a (186 mg, 92%) as a colourless liquid. [TLC (petroleum ether/ethyl acetate 95:5, $R_f(6a) = 0.50$, $R_f(1a) = 0.40$, UV detection]. IR (MIR-ATR, 4000–600 cm$^{-1}$): 2959, 2355, 1741, 1461, 980, 653 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.53 – 7.32 (m, 5H), 4.45 (s, 2H), 3.96 (s, 2H), 1.76 (dt, $J = 13.0$, 6.5 Hz, 2H), 1.51 – 1.38 (m, 4H), 0.99 (d, $J = 6.6$ Hz, 6H), 0.96 (d, $J = 6.6$ Hz, 6H) ppm. $^{13}$C {$^1$H} NMR (100 MHz, CDCl$_3$) $\delta$ 130.7, 129.3, 129.0, 127.2, 121.2, 71.1, 57.2, 43.0 (2C), 39.3, 24.6 (2C), 23.8 (2C), 23.7 (2C) ppm. HRMS (ESI) m/z: [M+NH$_4$]$^+$ Calcd for C$_{18}$H$_{31}$N$_2$O$_3$S$^+$ 355.2050; Found 355.2063.

2-Cyano-2-isobutyl-4-methylpentyl (2-chloro-5-methoxyphenyl)methanesulfonate (1b): GP-2 was carried out with arylmethanesulfonyl chloride 6b (153 mg, 0.6 mmol), triethyl amine (90 mg, 0.9 mmol), nitrile alcohol (110 mg, 0.6 mmol) in dichloromethane (DCM) (15 mL) solvent at room temperature for 4 h. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/10), furnished the product 1b (214 mg, 89%) as a colourless solid. [TLC (petroleum ether/ethyl acetate 90:10, $R_f(6b) = 0.60$, $R_f(1b) = 0.50$, UV detection]. IR (MIR-ATR, 4000–600 cm$^{-1}$): 2958, 2353, 1595, 1471, 1175, 754 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.35 (d, $J = 8.9$ Hz, 1H), 7.09 (d, $J = 3.0$ Hz, 1H), 6.90 (dd, $J = 8.9$, 3.0 Hz, 1H), 4.63 (s, 2H), 4.12 (s, 2H), 3.82 (s, 3H), 1.79 (dd, $J = 13.1$, 6.5 Hz, 2H), 1.50 – 1.45 (m, 4H), 1.0 (d, $J = 6.6$ Hz, 6H), 0.96 (d, $J = 6.6$ Hz, 6H) ppm. $^{13}$C {$^1$H} NMR (151 MHz, CDCl$_3$) $\delta$ 158.4, 130.6, 126.3, 126.2, 121.3, 117.6, 116.8, 70.5, 55.7,
53.9, 43.0 (2C), 39.2, 24.7 (2C), 23.8 (2C), 23.7 (2C) ppm. HRMS (ESI) m/z: [(M+K)]⁺
Calcd for C₁₉H₂₈ClKNO₄S⁺ 440.1059; Found 440.1075.

2-Cyano-2-isobutyl-4-methylpentyl (3-(trifluoromethyl)phenyl)methanesulfonate (1c):
GP-2 was carried out with arylmethanesulfonyl chloride 6c (154 mg, 0.6 mmol), triethyl amine (90 mg, 0.9 mmol), nitrile alcohol (110 mg, 0.6 mmol) in dichloromethane (DCM) (15 mL) solvent at room temperature for 4 h. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 93/07), furnished the product 1c (194 mg, 80%) as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, Rf(6c) = 0.60, Rf(1b) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): 2961, 2239, 1460, 1329, 1128, 745 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ 7.76 – 7.63 (m, 3H), 7.57 (t, J = 7.7 Hz, 1H), 4.51 (s, 2H), 4.07 (s, 2H), 1.77 (dp, J = 13.0, 6.5 Hz, 2H), 1.51 – 1.40 (m, 4H), 0.99 (d, J = 6.6 Hz, 6H), 0.96 (d, J = 6.6 Hz, 6H) ppm. ¹³C (¹H) NMR (100 MHz, CDCl₃) δ 134.2, 131.6, 131.3, 129.6, 128.4, 127.4, 126.1, 126.1, 126.1, 124.9, 121.2, 71.0, 56.5, 43.0 (2C), 39.4, 24.7 (2C), 23.7 (2C), 23.7 (2C) ppm. HRMS (ESI) m/z: [(M+K)]⁺ Calcd for C₁₉H₂₆F₃KNO₄S⁺ 444.1217; Found 444.1238.

2-Cyano-2-isobutyl-4-methylpentyl (2-chlorophenyl)methanesulfonate (1d): GP-2 was carried out with arylmethanesulfonyl chloride 6d (133 mg, 0.6 mmol), triethyl amine (90 mg, 0.9 mmol), nitrile alcohol (110 mg, 0.6 mmol) in dichloromethane (DCM) (15 mL) solvent at room temperature for 4 h. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/10), furnished the product 1d (193 mg, 87%) as a colourless solid. [TLC (petroleum ether/ethyl acetate 90:10, Rf(6d) = 0.70, Rf(1d) = 0.60, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): 2959, 1461, 1331, 1170, 1130, 738 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ 7.57 (dd, J = 7.0, 2.3 Hz, 1H), 7.47 (dd, J = 7.5, 1.7 Hz, 1H), 7.39 – 7.30 (m, 2H), 4.68 (s, 2H), 4.10 (s, 2H), 1.79 (dt, J = 13.0, 6.5 Hz, 2H), 1.48 (d, J
= 6.3 Hz, 4H), 1.0 (d, J = 6.6 Hz, 6H), 0.95 (d, J = 6.6 Hz, 6H) ppm. 13C {1H} NMR (100 MHz, CDCl3) δ 135.0, 132.7, 130.7, 130.0, 127.3, 125.6, 121.1, 70.4, 53.7, 43.0 (2C), 39.2, 24.6 (2C), 23.8 (2C), 23.7 (2C) ppm. HRMS (ESI) m/z: [(M+Na)]+ Calcd for C18H26ClINaO3S+ 394.1214; Found 394.1206.

2-Cyano-2-isobutyl-4-methylpentyl (3-chlorophenyl)methanesulfonate (1e): GP-2 was carried out with arylmethanesulfonyl chloride 6e (133 mg, 0.6 mmol), triethyl amine (90 mg, 0.9 mmol), nitrile alcohol (110 mg, 0.6 mmol) in dichloromethane (DCM) (15 mL) solvent at room temperature for 4 h. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/10), furnished the product 1e (200 mg, 90%) as a colourless solid. [TLC (petroleum ether/ethyl acetate 90:10, \( R_f^6 \) = 0.70, \( R_f^1 \) = 0.60, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): 2958, 1467, 1358, 1209, 980, 740 cm⁻¹. 1H NMR (600 MHz, CDCl3) δ 7.45 (s, 1H), 7.39 (ddd, J = 13.5, 6.6, 4.7 Hz, 1H), 7.37 – 7.30 (m, 2H), 4.42 (s, 2H), 4.05 (s, 2H), 1.78 (dt, J = 13.1, 6.5 Hz, 2H), 1.52 – 1.41 (m, 4H), 1.01 (d, J = 6.7 Hz, 6H), 0.98 (d, J = 6.6 Hz, 6H) ppm. 13C {1H} NMR (101 MHz, CDCl3) δ 134.8, 130.7, 130.2, 129.5, 129.1, 129.0, 121.2, 71.0, 56.4, 43.0 (2C), 39.3, 24.7 (2C), 23.8 (2C), 23.8 (2C) ppm. HRMS (ESI) m/z: [(M+H)]+ Calcd for C18H27ClNO3S+ 372.1395; Found 372.1395.

2-Cyano-2-isobutyl-4-methylpentyl (4-chlorophenyl)methanesulfonate (1f): GP-2 was carried out with arylmethanesulfonyl chloride 6f (133 mg, 0.6 mmol), triethyl amine (90 mg, 0.9 mmol), nitrile alcohol (110 mg, 0.6 mmol) in dichloromethane (DCM) (15 mL) solvent at room temperature for 4 h. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/10), furnished the product 1f (204 mg, 92%) as a colourless solid. [TLC (petroleum ether/ethyl acetate 90:10, \( R_f^6 \) = 0.70, \( R_f^1 \) = 0.60, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): 2958, 1467, 1358, 1209, 980, 740 cm⁻¹. 1H NMR (600 MHz, CDCl3) δ 7.45 (s, 1H), 7.39 (ddd, J = 13.5, 6.6, 4.7 Hz, 1H), 7.37 – 7.30 (m, 2H), 4.42 (s, 2H), 4.05 (s, 2H), 1.78 (dt, J = 13.1, 6.5 Hz, 2H), 1.52 – 1.41 (m, 4H), 1.01 (d, J = 6.7 Hz, 6H), 0.98 (d, J = 6.6 Hz, 6H) ppm. 13C {1H} NMR (101 MHz, CDCl3) δ 134.8, 130.7, 130.2, 129.5, 129.1, 129.0, 121.2, 71.0, 56.4, 43.0 (2C), 39.3, 24.7 (2C), 23.8 (2C), 23.8 (2C) ppm. HRMS (ESI) m/z: [(M+H)]+ Calcd for C18H27ClNO3S+ 372.1395; Found 372.1395.
0.60, UV detection). IR (MIR-ATR, 4000–600 cm⁻¹): 2956, 1464, 1360, 1169, 978, 750 cm⁻¹.

¹H NMR (400 MHz, CDCl₃) δ 7.40 (s, 4H), 4.42 (s, 2H), 4.02 (s, 2H), 1.77 (dt, J = 13.0, 6.5 Hz, 2H), 1.46 (m, J = 6.2, 4.5 Hz, 4H), 1.0 (d, J = 6.6 Hz, 6H), 0.97 (d, J = 6.6 Hz, 6H) ppm.

¹³C {¹H} NMR (100 MHz, CDCl₃) δ 135.7, 132.2, 129.3, 125.8, 121.3, 71.3, 56.6, 43.2 (2C), 39.5, 24.8 (2C), 23.9 (2C), 23.9 (2C) ppm. HRMS (ESI) m/z: [(M+H)]⁺ Calcd for C₁₈H₂₇ClNO₃S⁺ 372.1395; Found 372.1401.

[S12]

**2-Cyano-2-isobutyl-4-methylpentyl (2-bromophenyl)methanesulfonate (1g):** GP-2 was carried out with arylmethanesulfonyl chloride 6g (269 mg, 0.6 mmol), triethyl amine (90 mg, 0.9 mmol), nitrile alcohol (110 mg, 0.6 mmol) in dichloromethane (DCM) (15 mL) solvent at room temperature for 4 h. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/10), furnished the product 1g (202 mg, 81%) as a colourless solid. [TLC (petroleum ether/ethyl acetate 90:10, Rf(6g) = 0.70, Rf(1g) = 0.60, UV detection). IR (MIR-ATR, 4000–600 cm⁻¹): 2956, 1465, 1360, 1175, 981, 754 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ 7.65 (dd, J = 8.0, 1.2 Hz, 1H), 7.59 (dd, J = 7.7, 1.7 Hz, 1H), 7.38 (td, J = 7.6, 1.3 Hz, 1H), 7.27 (ddd, J = 7.7, 6.3, 1.7 Hz, 1H), 4.72 (s, 2H), 4.10 (s, 2H), 1.78 (dt, J = 13.0, 6.5 Hz, 2H), 1.48 (m, J = 6.2 Hz, 4H), 1.0 (d, J = 6.6 Hz, 6H), 0.96 (d, J = 6.6 Hz, 6H) ppm. ¹³C {¹H} NMR (151 MHz, CDCl₃) δ 133.5, 132.8, 131.0, 128.1, 127.6, 125.6, 121.2, 70.6, 56.4, 43.0 (2C), 39.3, 24.0 (2C), 23.9 (2C), 23.8 (2C) ppm. HRMS (ESI) m/z: [(M+H)]⁺ Calcd for C₁₈H₂₇⁷⁹BrNO₃S⁺ 416.0890; Found 416.0895: Calcd for C₂₆H₂₃⁸¹BrNO₃S⁺ 418.0869; Found 418.0880.

[S12]

**2-Cyano-2-isobutyl-4-methylpentyl (3-bromophenyl)methanesulfonate (1h):** GP-2 was carried out with arylmethanesulfonyl chloride 6h (269 mg, 0.6 mmol), triethyl amine (90 mg, 0.9 mmol), nitrile alcohol (110 mg, 0.6 mmol) in dichloromethane (DCM) (15 mL) solvent at room temperature for 4 h. Purification of crude product using silica gel column
chromatography (hexane/ethyl acetate, 100/0 to 90/10), furnished the product 1h (199 mg, 80%) as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, $R_f(6h) = 0.70$, $R_f(1h) = 0.60$, UV detection]. IR (MIR-ATR, 4000–600 cm$^{-1}$): 2958, 1466, 1357, 1171, 979, 818, 688 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) δ 7.60 (t, $J = 1.7$ Hz, 1H), 7.58 – 7.52 (m, 1H), 7.40 (d, $J = 7.9$ Hz, 1H), 7.30 (t, $J = 7.8$ Hz, 1H), 4.41 (s, 2H), 4.05 (s, 2H), 1.78 (dt, $J = 13.0$, 6.5 Hz, 2H), 1.52 – 1.41 (m, 4H), 1.01 (d, $J = 6.6$ Hz, 6H), 0.98 (d, $J = 6.6$ Hz, 6H) ppm. $^{13}$C $^1{}$H NMR (151 MHz, CDCl$_3$) δ 133.6, 132.5, 130.5, 129.4, 129.4, 122.9, 121.2, 71.1, 56.5, 43.1 (2C), 39.4, 24.8 (2C), 23.9 (2C), 23.8 (2C) ppm. HRMS (ESI) m/z: [(M+H)$^+$] Calcd for C$_{18}$H$_{27}$BrNO$_3$S$^+$ 416.0890; Found 416.0896: Calcd for C$_{18}$H$_{27}$BrNO$_3$S$^+$ 418.0869; Found 418.0877.

2-Cyano-2-isobutyl-4-methylpentyl (4-bromophenyl)methanesulfonate (1i): GP-2 was carried out with arylmethanesulfonyl chloride 6i (269 mg, 0.6 mmol), triethyl amine (90 mg, 0.9 mmol), nitrile alcohol (110 mg, 0.6 mmol) in dichloromethane (DCM) (15 mL) solvent at room temperature for 4 h. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/10), furnished the product 1i (207 mg, 83%) as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, $R_f(6i) = 0.70$, $R_f(1i) = 0.60$, UV detection]. IR (MIR-ATR, 4000–600 cm$^{-1}$): 2959, 1473, 1358, 979, 644 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) δ 7.54 (d, $J = 8.0$ Hz, 2H), 7.32 (d, $J = 8.4$ Hz, 2H), 4.39 (s, 2H), 4.00 (s, 2H), 1.75 (dt, $J = 13.0$, 6.5 Hz, 2H), 1.52 – 1.37 (m, 4H), 0.99 (d, $J = 6.6$ Hz, 6H), 0.96 (d, $J = 6.6$ Hz, 6H) ppm. $^{13}$C $^1{}$H NMR (100 MHz, CDCl$_3$) δ 132.3, 132.1, 126.2, 123.7, 121.2, 71.2, 56.4, 43.0 (2C), 39.3, 24.7 (2C), 23.8 (2C), 23.7 (2C) ppm. HRMS (ESI) m/z: [(M+NH$_4$)$^+$] Calcd for C$_{18}$H$_{30}$BrNO$_3$S$^+$ 433.1155; Found 433.1164: Calcd for C$_{18}$H$_{30}$BrNO$_3$S$^+$ 435.1135; Found 435.1143.
2-Cyano-2-isobutyl-4-methylpentyl (2,3-dichlorophenyl)methanesulfonate (1j): GP-2 was carried out with arylmethanesulfonyl chloride 6j (154 mg, 0.6 mmol), triethyl amine (90 mg, 0.9 mmol), nitrile alcohol (110 mg, 0.6 mmol) in dichloromethane (DCM) (15 mL) solvent at room temperature for 4 h. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/10), furnished the product 1j (190 mg, 78%) as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, $R_f(6j) = 0.60, R_f(1j) = 0.50$, UV detection]. IR (MIR-ATR, 4000–600 cm$^{-1}$): 2958, 1458, 1362, 1177, 980, 752 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.52 (ddd, $J = 13.3, 7.9, 1.5$ Hz, 2H), 7.29 (t, $J = 7.9$ Hz, 1H), 4.73 (s, 2H), 4.13 (s, 2H), 1.80 (dt, $J = 13.1, 6.5$ Hz, 2H), 1.48 (m, $J = 6.2, 2.7$ Hz, 4H), 1.01 (d, $J = 6.6$ Hz, 6H), 0.97 (d, $J = 6.6$ Hz, 6H) ppm. $^{13}$C $^1$H NMR (151 MHz, CDCl$_3$) $\delta$ 134.1, 133.7, 131.6, 130.9, 128.0, 127.7, 121.1, 70.7, 54.5, 43.1 (2C), 39.3, 24.8 (2C), 23.9 (2C), 23.8 (2C) ppm. HRMS (ESI) m/z: [(M+NH$_4$)$^+$. Calcd for C$_{18}$H$_{29}$Cl$_2$N$_2$O$_3$S $^+$ 423.1270; Found 423.1280.

2-Cyano-2-isobutyl-4-methylpentyl (3,4-dichlorophenyl)methanesulfonate (1k): GP-2 was carried out with arylmethanesulfonyl chloride 6k (154 mg, 0.6 mmol), triethyl amine (90 mg, 0.9 mmol), nitrile alcohol (110 mg, 0.6 mmol) in dichloromethane (DCM) (15 mL) solvent at room temperature for 4 h. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/10), furnished the product 1k (192 mg, 79%) as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, $R_f(6k) = 0.60, R_f(1k) = 0.50$, UV detection]. IR (MIR-ATR, 4000–600 cm$^{-1}$): 2958, 1563, 1362, 1173, 979, 821 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.56 (d, $J = 2.1$ Hz, 1H), 7.51 (d, $J = 8.3$ Hz, 1H), 7.34 – 7.29 (m, 1H), 4.40 (s, 2H), 4.09 (s, 2H), 1.91 – 1.69 (m, 2H), 1.48 (t, $J = 6.0$ Hz, 4H), 1.01 (d, $J = 6.6$ Hz, 6H), 0.98 (d, $J = 6.6$ Hz, 6H) ppm. $^{13}$C $^1$H NMR (151 MHz, CDCl$_3$) $\delta$ 134.0, 133.3, 132.7, 132.6, 131.2, 131.0, 130.1, 130.0, 127.4, 121.3, 71.3, 56.0, 43.2 (2C), 39.5, 24.8 (2C), 23.9 (2C), 22.7 (2C) ppm. HRMS (ESI) m/z: [(M+K)$^+$. Calcd for C$_{18}$H$_{25}$Cl$_2$KNO$_3$S $^+$ 444.0564; Found 444.0585.
2-Cyano-2-isobutyl-4-methylpentyl (2,4-dichlorophenyl)methanesulfonate (II): GP-2 was carried out with arylmethanesulfonyl chloride 6i (154 mg, 0.6 mmol), triethyl amine (90 mg, 0.9 mmol), nitrile alcohol (110 mg, 0.6 mmol) in dichloromethane (DCM) (15 mL) solvent at room temperature for 4 h. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/10), furnished the product II (211 mg, 87%) as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, $R_f(6i) = 0.60$, $R_f(II) = 0.50$, UV detection]. IR (MIR-ATR, 4000–600 cm$^{-1}$): 2958, 1468, 1359, 1170, 977, 825, 741 cm$^{-1}$. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 7.51 (d, $J = 8.3$ Hz, 1H), 7.48 (d, $J = 2.1$ Hz, 1H), 7.32 (dd, $J = 8.3$, 2.1 Hz, 1H), 4.63 (s, 2H), 4.12 (s, 2H), 1.79 (dt, $J = 13.1$, 6.5 Hz, 2H), 1.52 – 1.41 (m, 4H), 1.01 (d, $J = 6.7$ Hz, 6H), 0.97 (d, $J = 6.7$ Hz, 6H) ppm. $^{13}$C $^{1}$H NMR (100 MHz, CDCl$_3$) $\delta$ 136.2, 135.8, 133.5, 129.8, 127.8, 124.3, 121.1, 70.6, 53.1, 43.0 (2C), 39.2, 24.7 (2C), 23.8 (2C), 23.7 (2C) ppm. HRMS (ESI) m/z: [M]$^+$ Calcd for C$_{18}$H$_{25}$Cl$_2$NO$_3$S$^+$ 405.0932; Found 405.0936.

2-cyano-2-isobutyl-4-methylpentyl o-tolylmethanesulfonate (1m): GP-2 was carried out with arylmethanesulfonyl chloride 6m (122 mg, 0.6 mmol), triethyl amine (90 mg, 0.9 mmol), nitrile alcohol (110 mg, 0.6 mmol) in dichloromethane (DCM) (15 mL) solvent at room temperature for 4 h. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/10), furnished the product 1m (170 mg, 81%) as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, $R_f(6m) = 0.60$, $R_f(1m) = 0.50$, UV detection]. IR (MIR-ATR, 4000–600 cm$^{-1}$): 2958, 2036, 1361, 1172, 984, 821, 738 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.34 (d, $J = 7.9$ Hz, 1H), 7.25 – 7.22 (m, 1H), 7.19 (d, $J = 8.4$ Hz, 2H), 4.45 (s, 2H), 3.91 (s, 2H), 2.41 (s, 3H), 1.71 (dt, $J = 13.0$, 6.5 Hz, 2H), 1.42 – 1.37 (m, 4H), 0.94 (d, $J = 6.6$ Hz, 6H), 0.91 (d, $J = 6.6$ Hz, 6H). $^{13}$C $^{1}$H NMR (101 MHz, CDCl$_3$) $\delta$ 138.3, 131.9, 131.1, 129.6, 126.5, 125.7, 121.3, 71.0, 54.7, 43.1 (2C), 24.7 (2C), 23.8 (2C), 23.7 (2C) ppm.
2-cyano-2-isobutyl-4-methylpentyl (2-fluorophenyl) methanesulfonate (1n): GP-2 was carried out with arylmethanesulfonyl chloride 6n (124 mg, 0.6 mmol), triethyl amine (90 mg, 0.9 mmol), nitrile alcohol (110 mg, 0.6 mmol) in dichloromethane (DCM) (15 mL) solvent at room temperature for 4 h. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/10), furnished the product 1n (166 mg, 78%) as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, Rf(6n) = 0.60, Rf(1n) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): 2958, 2036, 1361, 1172, 984, 821, 738 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ 7.49 (td, J = 7.5, 1.6 Hz, 1H), 7.40 (d, J = 12.1 Hz, 1H), 7.20 (dd, J = 11.0, 4.2 Hz, 1H), 7.13 (t, J = 9.1 Hz, 1H), 4.51 (s, 2H), 4.10 (s, 2H), 1.89 – 1.67 (m, 2H), 1.47 (dd, J = 6.2, 1.1 Hz, 4H), 1.00 (d, J = 6.6 Hz, 6H), 0.96 (d, J = 6.6 Hz, 6H). ¹³C {¹H} NMR (101 MHz, CDCl₃) δ 161.0 (d, J_C-F = 249.9 Hz), 132.6 (d, J_C-F = 2.4 Hz), 131.4 (d, J_C-F = 8.4 Hz), 124.7 (d, J_C-F = 3.8 Hz), 121.0, 115.8 (d, J_C-F = 21.5 Hz), 114.9 (d, J_C-F = 14.5 Hz), 70.6 , 49.8 (d, J_C-F = 3.4 Hz), 42.9 (2C), 39.2, 24.6 (2C), 23.8 (2C), 23.7 (2C). HRMS (ESI) m/z: [(M+NH₄)⁺ Calcd for C₁₉H₃₃N₂O₃S⁺ 369.2206; Found 369.2207.

Methyl (E)-3-(3-((((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methyl)phenyl)acrylate (3ab): GP-3 was carried out by using arylmethanesulfonate ester 1a (67 mg, 0.2 mmol), olefin 2b (26 mg, 0.3 mmol), Pd(OAc)₂ (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate,
100/0 to 90/15), furnished the product 3ab (70 mg, 83%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, \(R_f(1a) = 0.60\), \(R_f(3ab) = 0.40\), UV detection]. IR (MIR-ATR, 4000–600 cm\(^{-1}\)): 2918, 2856, 1717, 1457, 754 cm\(^{-1}\). \(1^H\) NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.68 (d, \(J = 16.1\) Hz, 1H), 7.62 – 7.53 (m, 2H), 7.50 – 7.40 (m, 2H), 6.48 (d, \(J = 16.1\) Hz, 1H), 4.46 (s, 2H), 4.02 (s, 2H), 3.81 (s, 3H), 1.75 (td, \(J = 13.0, 6.5\) Hz, 2H), 1.50 – 1.39 (m, 4H), 0.98 (d, \(J = 6.6\) Hz, 6H), 0.95 (d, \(J = 6.6\) Hz, 6H) ppm. \(1^C\) \(\{^1^H\}\) NMR (151 MHz, CDCl\(_3\)) \(\delta\) 167.0, 143.5, 135.3, 132.4, 129.6, 128.7, 128.2, 121.2, 119.2, 71.2, 56.9, 51.8, 43.1 (2C), 39.4, 24.7 (2C), 23.8 (2C), 23.8 (2C) ppm. HRMS (ESI) m/z: [(M+K)]\(^{+}\) Calcd for C\(_{22}\)H\(_{31}\)KNO\(_5\)S\(^{+}\) 460.1555; Found 460.1551.

**Ethyl (E)-3-(2-chloro-3-(((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methyl)-5-methoxyphenyl)acrylate (3ba):** GP-3 was carried out by using arylmethanesulfonate ester 1b (80 mg, 0.2 mmol), olefin 2a (30 mg, 0.3 mmol), Pd(OAc)\(_2\) (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 3ba (71 mg, 71%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, \(R_f(1b) = 0.60\), \(R_f(3ba) = 0.40\), UV detection]. IR (MIR-ATR, 4000–600 cm\(^{-1}\)): 2958, 1714, 1456, 1174, 980 cm\(^{-1}\). \(1^H\) NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.06 (d, \(J = 15.9\) Hz, 1H), 7.26 (s, 1H), 7.15 (dd, \(J = 10.1, 3.0\) Hz, 2H), 6.39 (d, \(J = 15.9\) Hz, 1H), 4.69 (s, 2H), 4.29 (q, \(J = 7.1\) Hz, 2H), 4.14 (s, 2H), 3.85 (s, 3H), 1.79 (dt, \(J = 13.1, 6.5\) Hz, 2H), 1.48 (dd, \(J = 6.2, 2.1\) Hz, 4H), 1.35 (t, \(J = 7.1\) Hz, 3H), 1.0 (d, \(J = 6.6\) Hz, 6H), 0.96 (d, \(J = 6.6\) Hz, 6H) ppm. \(1^C\) \(\{^1^H\}\) NMR (100 MHz, CDCl\(_3\)) \(\delta\) 166.1, 158.0, 140.3, 135.0, 127.7, 126.9, 122.3, 121.1, 119.6, 114.0, 70.6, 60.9, 55.8, 54.2, 43.1 (2C), 39.3, 24.8 (2C), 23.8 (2C), 23.8 (2C), 14.3 ppm. HRMS (ESI) m/z: [(M+K)]\(^{+}\) Calcd for C\(_{24}\)H\(_{34}\)ClKNO\(_6\)S\(^{+}\) 538.1427; Found 538.1447.
Ethyl (E)-3-(3-((((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methyl)-5-(trifluoromethyl)phenyl)acrylate (3ca): GP-3 was carried out by using arylmethanesulfonate ester 1c (81 mg, 0.2 mmol), olefin 2a (30 mg, 0.3 mmol), Pd(OAc)$_2$ (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 ºC for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 3ca (75 mg, 75%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, $R_f$(1c) = 0.60, $R_f$(3ca) = 0.40, UV detection]. IR (MIR-ATR, 4000–600 cm$^{-1}$): 2962, 1712, 1358, 1169, 1134, 979, 821 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.80 (d, $J = 7.1$ Hz, 2H), 7.73 – 7.63 (m, 2H), 6.55 (d, $J = 16.1$ Hz, 1H), 4.52 (s, 2H), 4.28 (q, $J = 7.1$ Hz, 2H), 4.12 (s, 2H), 1.77 (dq, $J = 13.0$, 6.5 Hz, 2H), 1.52 – 1.42 (m, 4H), 1.34 (t, $J = 7.1$ Hz, 3H), 1.0 (d, $J = 6.6$ Hz, 6H), 0.97 (d, $J = 6.6$ Hz, 6H) ppm. $^{13}$C {$^1$H} NMR (151 MHz, CDCl$_3$) $\delta$ 166.0, 141.5, 136.4, 133.3, 132.3 (d, $J_{C-F} = 33$ Hz), 129.4, 128.5 (d, $J_{C-F} = 33$ Hz), 125.2 (d, $J_{C-F} = 3$ Hz), 123.2 (d, $J_{C-P} = 271$ Hz), 121.6, 121.2, 71.2, 60.9, 56.4, 43.1 (2C), 39.5, 24.8 (2C), 23.8 (2C), 23.8 (2C), 14.2 ppm. HRMS (ESI) m/z: [(M+H)]$^+$ Calcd for C$_{24}$H$_{33}$F$_3$NO$_5$S$^+$ 504.2026; Found 504.2050.

Ethyl (E)-3-((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonylethyl)-4-methylphenyl)acrylate (3ma): GP-3 was carried out by using arylmethanesulfonate ester 1m (70 mg, 0.2 mmol), olefin 2a (30 mg, 0.3 mmol), Pd(OAc)$_2$ (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 ºC for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 3ma (68 mg, 76%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, $R_f$(1m) = 0.60, $R_f$(3ma) = 0.40, UV detection]. IR (MIR-ATR, 4000–600 cm$^{-1}$): 2948, 2252,
1711, 1267, 1171, 985, 732 cm\(^{-1}\). \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.64 (d, \(J = 16.0\) Hz, 1H), 7.54 (d, \(J = 1.6\) Hz, 1H), 7.46 (dd, \(J = 8.0, 1.7\) Hz, 1H), 7.28 (d, \(J = 8.0\) Hz, 1H), 6.43 (d, \(J = 16.0\) Hz, 1H), 4.51 (s, 2H), 4.25 (q, \(J = 7.1\) Hz, 2H), 2.48 (s, 3H), 1.77 (dt, \(J = 13.0, 6.5\) Hz, 2H), 1.45 (dd, \(J = 6.2, 4.3\) Hz, 4H), 1.32 (t, \(J = 7.1\) Hz, 3H), 0.99 (d, \(J = 6.6\) Hz, 6H), 0.96 (d, \(J = 6.6\) Hz, 6H). \(^{13}\)C \{\(^1\)H\} NMR (100 MHz, CDCl\(_3\)) \(\delta\) 166.7, 143.2, 140.6, 132.9, 131.7, 131.4, 128.7, 126.5, 121.2, 118.6, 70.9, 60.5, 54.4, 43.1 (2C), 39.4, 24.7 (2C), 23.82 (2C), 23.77 (2C), 19.7, 14.2. HRMS (ESI) m/z: [(M+H)]\(^+\) Calcd for C\(_{24}\)H\(_{36}\)NO\(_5\)S\(^+\) 449.2236; Found 450.2311.

**Ethyl \((E)-3-(4-chloro-3-((((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methyl)phenyl)acrylate (3da):** GP-3 was carried out by using arylmethanesulphonate ester 1d (74 mg, 0.2 mmol), olefin 2a (30 mg, 0.3 mmol), Pd(OAc)\(_2\) (5 mg, 10 mol%), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 3da (73 mg, 78%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, \(R_f(1d) = 0.60, R_f(3da) = 0.40, \) UV detection]. IR (MIR-ATR, 4000–600 cm\(^{-1}\)): 2958, 2237, 1711, 1360, 1171, 978, 819 cm\(^{-1}\). \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.70 (d, \(J = 1.6\) Hz, 1H), 7.63 (d, \(J = 16.0\) Hz, 1H), 7.53 – 7.46 (m, 2H), 6.46 (d, \(J = 16.1\) Hz, 1H), 1.79 (dt, \(J = 13.0, 6.5\) Hz, 2H), 1.48 (dd, \(J = 6.2, 2.1\) Hz, 4H), 1.33 (t, \(J = 7.1\) Hz, 3H), 0.96 (d, \(J = 6.6\) Hz, 6H) ppm. HRMS (ESI) m/z: [(M+Na)]\(^+\) Calcd for C\(_{23}\)H\(_{32}\)ClNNaO\(_5\)S\(^+\) 492.1582; Found 492.1593.
(E)-3-(2-chloro-3-(((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methyl)phenyl)acrylate (3da’): GP-3 was carried out by using arylmethanesulfonate ester 1d (74 mg, 0.2 mmol), olefin 2a (30 mg, 0.3 mmol), Pd(OAc)$_2$ (5 mg, 10 mol%), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 3da’ (73 mg, 78%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, $R_f$(1d) = 0.60, $R_f$(3da’) = 0.40, UV detection). IR (MIR-ATR, 4000–600 cm$^{-1}$): 2959, 2245, 1712, 1365, 1176, 982 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.11 (d, $J$ = 16.0 Hz, 1H), 7.71 – 7.66 (m, 1H), 7.61 (dd, $J$ = 7.7, 1.5 Hz, 1H), 7.37 (t, $J$ = 7.8 Hz, 1H), 6.42 (d, $J$ = 15.9 Hz, 1H), 4.74 (s, 2H), 4.29 (q, $J$ = 7.1 Hz, 2H), 4.13 (s, 2H), 1.79 (dt, $J$ = 13.1, 6.5 Hz, 2H), 1.48 (dd, $J$ = 6.2, 2.0 Hz, 4H), 1.35 (t, $J$ = 7.1 Hz, 3H), 1.0 (d, $J$ = 6.6 Hz, 6H), 0.96 (d, $J$ = 6.6 Hz, 6H) ppm. $^{13}$C {$^1$H} NMR (100 MHz, CDCl$_3$) $\delta$ 166.1, 140.1, 135.4, 134.4, 133.9, 128.7, 127.3, 127.0, 122.3, 121.1, 70.5, 60.8, 54.1, 43.1 (2C), 39.4, 24.7 (2C), 23.9 (2C), 23.8 (2C), 14.2 ppm. HRMS (ESI) m/z: [(M+K)]$^+$ Calcd for C$_{23}$H$_{32}$ClKNO$_5$S$^+$ 508.1321; Found 508.1337.

(E)-3-((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methyl-4-fluorophenyl)acrylate (3na) & Ethyl (E)-3-(((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methyl)-2-fluorophenyl)acrylate (3na’): GP-3 was carried out by using arylmethanesulfonate ester 1n (71 mg, 0.2 mmol), olefin 2a (30 mg, 0.3 mmol), Pd(OAc)$_2$ (5 mg, 10 mol%), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the isomeric mixture of products 3na+3na’ (62 mg, 69%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, $R_f$(1n) = 0.60,
$R_{f}(3\text{ga}+3\text{ga}') = 0.40$, UV detection]. IR (MIR-ATR, 4000–600 cm$^{-1}$): 2923, 1714, 1461, 1175, 1034, 984, 817 cm$^{-1}$. $^{1}H$ NMR (400 MHz, CDCl$_3$) $\delta$ 7.80 (d, $J = 16.2$ Hz, 1H), 7.67 – 7.07 (m, 1H), 6.89 – 6.23 (m, 1H), 4.52 (d, $J = 8.7$ Hz, 2H), 4.32 – 4.21 (m, 2H), 4.14 (d, $J = 2.7$ Hz, 2H), 1.85 – 1.74 (m, 2H), 1.47 (dd, $J = 6.2$, 2.4 Hz, 4H), 1.33 (t, $J = 7.1$ Hz, 3H), 1.00 (dd, $J = 6.6$, 1.2 Hz, 6H), 0.98 – 0.95 (m, 6H). $^{13}C$ NMR (101 MHz, CDCl$_3$) $\delta$ 166.3, 164.6, 160.7, 142.1, 136.0, 136.0, 134.0, 134.0, 132.2, 132.2, 130.9, 130.8, 130.2, 124.8, 123.2, 121.9, 121.0, 119.4, 116.7, 116.5, 116.1, 115.9, 70.7, 60.7, 60.6, 49.8, 43.0, 39.3, 24.7, 23.8, 23.7, 14.2. HRMS (ESI) m/z: [(M+H)$^+$] Calcd for $C_{23}H_{33}FNO_5S^+$ 454.2058; Found 454.2064.

Ethyl (E)-3-(4-bromo-3-((((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methyl)phenyl)acrylate (3ga) & Ethyl (E)-3-(2-bromo-3-((((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methyl)phenyl)acrylate (3ga'): GP-3 was carried out by using arylmethanesulfonate ester 1g (83 mg, 0.2 mmol), olefin 2a (30 mg, 0.3 mmol), Pd(OAc)$_2$ (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 $^\circ$C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the isomeric mixture of products 3ga+3ga' (72 mg, 71%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, $R_{f}(1g) = 0.60$, $R_{f}(3ga+3ga') = 0.40$, UV detection]. $^{1}H$ NMR (400 MHz, CDCl$_3$) $\delta$ 8.07 (d, $J = 15.9$ Hz, 1H), 7.73 – 7.55 (m, 3H), 7.40 (dd, $J = 8.2$, 2.5 Hz, 1H), 6.35 (d, $J = 15.8$ Hz, 1H), 4.75 (d, $J = 33.3$ Hz, 3H), 4.35 – 4.19 (m, 3H), 4.12 (d, $J = 7.1$ Hz, 3H), 1.78 (dd, $J = 15.3$, 7.7, 5.4 Hz, 4H), 1.47 (dd, $J = 6.2$, 1.2 Hz, 6H), 1.33 (dd, $J = 10.1$, 4.1 Hz, 6H), 1.02 – 0.92 (m, 17H). $^{13}C$ NMR (101 MHz, CDCl$_3$) $\delta$ 166.3, 166.0, 143.1, 142.0, 136.0, 134.6, 134.0, 133.7, 132.0, 129.6, 128.9, 128.9, 128.3, 128.0, 127.9, 127.0, 123.3, 122.4, 121.1, 120.3, 70.6, 70.5, 66.4, 60.8, 60.7, 56.8, 56.1, 43.0, 42.9, 39.3, 24.8, 24.7, 24.0, 24.0, 23.8, 23.7, 14.2 ppm. HRMS (ESI) m/z: [(M+Na)$^+$] Calcd for $C_{23}H_{32}^{79}BrNaNO_5S^+$ 536.1077; Found 536.1061. Calcd for $C_{23}H_{32}^{81}BrNNaO_5S^+$ 538.1056; Found 536.1057.
**Ethyl (E)-3-(3-chloro-5-(((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methyl)phenyl)acrylate (3ea):** GP-3 was carried out by using arylmethanesulfonate ester 1e (74 mg, 0.2 mmol), olefin 2a (30 mg, 0.3 mmol), Pd(OAc)$_2$ (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 3ea (77 mg, 82%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, $R_f$(1e) = 0.60, $R_f$(3ea) = 0.40, UV detection). IR (MIR-ATR, 4000–600 cm$^{-1}$): 2961, 1710, 1360, 978, 853 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) \(\delta\) 7.60 (d, $J$ = 16.0 Hz, 1H), 7.55 (t, $J$ = 1.5 Hz, 1H), 7.46 (dd, $J$ = 6.9, 5.2 Hz, 2H), 6.48 (d, $J$ = 16.0 Hz, 1H), 4.43 (s, 2H), 4.27 (q, $J$ = 7.1 Hz, 2H), 4.10 (s, 2H), 1.78 (dt, $J$ = 13.1, 6.5 Hz, 2H), 1.52 – 1.44 (m, 4H), 1.33 (t, $J$ = 7.1 Hz, 3H), 1.0 (d, $J$ = 6.6 Hz, 6H), 0.98 (d, $J$ = 6.6 Hz, 6H) ppm. $^{13}$C \(\{^1\text{H}\}\) NMR (100 MHz, CDCl$_3$) \(\delta\) 166.2, 141.7, 137.0, 135.5, 131.9, 129.8, 128.5, 128.5, 121.2, 121.1, 71.2, 60.8, 56.3, 43.1 (2C), 39.5, 24.8 (2C), 23.8 (2C), 23.8 (2C), 14.2 ppm. HRMS (ESI) m/z: [(M+H)$^+$] Calcd for C$_{23}$H$_{33}$ClNO$_5$S$^+$ 470.1762; Found 470.1774.

**Ethyl (E)-3-(2-chloro-5-(((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methyl)phenyl)acrylate (3fa):** GP-3 was carried out by using arylmethanesulfonate ester 1f (74 mg, 0.2 mmol), olefin 2a (30 mg, 0.3 mmol), Pd(OAc)$_2$ (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 3fa (73 mg, 78%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, $R_f$(1d) = 0.60, $R_f$(3fa) = 0.40, UV detection). IR (MIR-ATR, 4000–600 cm$^{-1}$): 2956, 1715, 1365, 1176, 980, 754 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) \(\delta\)
8.05 (d, J = 16.1 Hz, 1H), 7.70 (d, J = 2.1 Hz, 1H), 7.48 (d, J = 8.3 Hz, 1H), 7.46 – 7.35 (m, 1H), 6.50 (d, J = 16.0 Hz, 1H), 4.44 (s, 2H), 4.29 (q, J = 7.1 Hz, 2H), 4.08 (s, 2H), 1.77 (dt, J = 13.0, 6.5 Hz, 2H), 1.47 (t, J = 6.0 Hz, 4H), 1.35 (t, J = 7.1 Hz, 3H), 1.0 (d, J = 6.6 Hz, 6H), 0.97 (d, J = 6.6 Hz, 6H) ppm. 

\( ^{13} \text{C} \{^{1} \text{H} \} \text{ NMR (100 MHz, CDCl}_3] \delta 166.2, 139.2, 136.0, 133.6, 133.0, 130.9, 129.9, 126.6, 122.2, 121.3, 71.3, 60.8, 56.4, 43.2 (2C), 39.5, 24.8 (2C), 23.9 (2C), 23.8 (2C), 14.3 ppm. \)

HRMS (ESI) m/z: [(M+K)]\(^+\) Calcd for C\(_{23}\)H\(_{32}\)ClKNO\(_5\)S\(^+\) 508.1321; Found 508.1342.

Methyl (E)-3-(2-chloro-5-(((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methyl)phenyl)acrylate (3fb): GP-3 was carried out by using arylmethanesulfonate ester 1f (74 mg, 0.2 mmol), olefin 2b (26 mg, 0.3 mmol), Pd(OAc)\(_2\) (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 \(\text{°C}\) for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 3fb (73 mg, 80%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, \(R_f(1f) = 0.60, R_f(3fb) = 0.40, \text{UV detection})\]. IR (MIR-ATR, 4000–600 cm\(^{-1}\)): 2956, 1717, 1361, 1175, 978, 754 cm\(^{-1}\). \(^{1}\text{H} \text{ NMR (400 MHz, CDCl}_3] \delta 7.99 (d, J = 16.1 Hz, 1H), 7.63 (d, J = 2.1 Hz, 1H), 7.42 (d, J = 8.3 Hz, 1H), 7.35 (dd, J = 8.3, 2.2 Hz, 1H), 6.42 (t, J = 13.0 Hz, 1H), 4.38 (s, 2H), 4.00 (s, 2H), 3.76 (s, 3H), 1.70 (dt, J = 13.1, 6.5 Hz, 2H), 1.40 (t, J = 6.1 Hz, 4H), 0.99 (d, J = 6.6 Hz, 6H), 0.96 (d, J = 6.6 Hz, 6H) ppm. \( ^{13} \text{C} \{^{1} \text{H} \} \text{ NMR (100 MHz, CDCl}_3] \delta 166.6, 139.5, 136.1, 133.5, 133.0, 130.9, 129.9, 126.6, 125.7, 121.8, 121.3, 71.4, 56.4, 52.0, 43.2 (2C), 39.6, 29.7, 24.8 (2C), 23.9 (2C), 23.9 (2C) ppm. HRMS (ESI) m/z: [(M+Na)]\(^+\) Calcd for C\(_{22}\)H\(_{30}\)ClNNaO\(_5\)S\(^+\) 478.1425; Found 478.1411.
Ethyl (E)-3-(3-bromo-5-((((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methyl)phenyl)acrylate (3ha): GP-3 was carried out by using arylmethanesulfonate ester 1h (83 mg, 0.2 mmol), olefin 2a (30 mg, 0.3 mmol), Pd(OAc)_2 (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 3ha (81 mg, 79%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, \(R_f^{(1h)}\) = 0.60, \(R_f^{(3ha)}\) = 0.40, UV detection]. IR (MIR-ATR, 4000–600 cm\(^{-1}\)): 2961, 1710, 1360, 1172, 734 cm\(^{-1}\). \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.70 (s, 1H), 7.61 – 7.55 (m, 2H), 7.52 (s, 1H), 6.46 (d, \(J = 16.0\) Hz, 1H), 4.42 (s, 2H), 4.29 – 4.23 (m, 2H), 4.09 (d, \(J = 3.6\) Hz, 2H), 1.77 (dd, \(J = 13.1, 6.5\) Hz, 2H), 1.48 – 1.44 (m, 4H), 1.32 (t, \(J = 7.1\) Hz, 3H), 1.0 (d, \(J = 6.6\) Hz, 6H), 0.97 (d, \(J = 6.6\) Hz, 6H) ppm. \(^{13}\)C \(^{1}\)H NMR (151 MHz, CDCl\(_3\)) \(\delta\) 166.1, 141.6, 137.2, 134.7, 131.4, 130.0, 129.0, 123.3, 121.2, 121.0, 71.2, 60.8, 56.2, 43.1 (2C), 39.4, 24.7 (2C), 23.8 (2C), 23.8 (2C), 14.2 ppm. HRMS (ESI) m/z: \([\text{M+K}]^+\) Caled for C\(_{23}\)H\(_{32}\)\(^{79}\)BrKNO\(_2\)S\(^+\) 552.0816; Found 552.0817: Caled for C\(_{23}\)H\(_{32}\)\(^{81}\)BrKNO\(_2\)S\(^+\) 554.0796; Found 554.0800.

Ethyl (E)-3-(2-bromo-5-((((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methyl)phenyl)acrylate (3ia): GP-3 was carried out by using arylmethanesulfonate ester 1i (83 mg, 0.2 mmol), olefin 2a (30 mg, 0.3 mmol), Pd(OAc)$_2$ (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 3ia (81 mg, 79%), as a pale yellow solid. [TLC (petroleum ether/ethyl acetate 90:10, \(R_f^{(1i)}\) = 0.60, \(R_f^{(3ia)}\) = 0.40, UV detection]. Melting
point: 72-74 °C. IR (MIR-ATR, 4000–600 cm⁻¹): 2960, 1710, 1359, 1172, 977, 827 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ 7.99 (d, J = 16.0 Hz, 1H), 7.66 (dd, J = 5.2, 3.0 Hz, 2H), 7.32 (dd, J = 8.3, 2.2 Hz, 1H), 6.45 (d, J = 15.9 Hz, 1H), 4.42 (s, 2H), 4.27 (t, J = 7.1 Hz, 2H), 4.06 (s, 2H), 1.76 (dt, J = 13.0, 6.5 Hz, 1H), 1.47 (dd, J = 13.1, 7.1 Hz, 4H), 1.34 (t, J = 7.1 Hz, 3H), 0.99 (d, J = 6.6 Hz, 6H), 0.96 (d, J = 6.6 Hz, 6H) ppm. ¹³C {¹H} NMR (100 MHz, CDCl₃) δ 166.0, 141.7, 135.4, 134.1, 133.0, 129.9, 127.2, 126.3, 121.3, 71.3, 60.8, 56.3, 43.1 (2C), 39.5, 24.8 (2C), 23.8 (2C), 23.8 (2C), 14.2 ppm. HRMS (ESI) m/z: [(M+H)]⁺ Calcd for C₂₃H₃₇BrNO₅S⁺ 514.1257; Found 514.1261: Calcd for C₂₃H₃₉BrNO₅S⁺ 516.1237; Found 516.1247.

Ethyl (E)-3-(3,4-dichloro-5-((((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methyl)phenyl)acrylate (3ja): GP-3 was carried out by using arylmethanesulfonate ester 1j (81 mg, 0.2 mmol), olefin 2a (30 mg, 0.3 mmol), Pd(OAc)₂ (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 3ja (77 mg, 76%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, Rf(1j) = 0.60, Rf(3ja) = 0.40, UV detection)]. IR (MIR-ATR, 4000-600 cm⁻¹): 2956, 1721, 1362, 1177, 980, 754 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ 7.68 (d, J = 2.0 Hz, 1H), 7.62 (d, J = 2.0 Hz, 1H), 7.57 (d, J = 16.0 Hz, 1H), 6.48 (d, J = 16.0 Hz, 1H), 4.73 (s, 2H), 4.27 (q, J = 7.1 Hz, 2H), 4.17 (s, 2H), 1.79 (td, J = 13.0, 6.5 Hz, 2H), 1.49 (dd, J = 6.2, 3.8 Hz, 4H), 1.34 (t, J = 7.1 Hz, 3H), 1.01 (d, J = 6.6 Hz, 6H), 0.98 (d, J = 6.6 Hz, 6H) ppm. ¹³C {¹H} NMR (100 MHz, CDCl₃) δ 166.0, 140.8, 134.9, 134.8, 134.5, 130.2, 130.1, 128.6, 121.5, 121.1, 70.7, 60.9, 54.4, 43.1 (2C), 39.4, 24.8 (2C), 23.9 (2C), 23.8 (2C), 14.3 ppm. HRMS (ESI) m/z: [(M+H)]⁺ Calcd for C₂₃H₃₂Cl₂NO₅S⁺ 504.1373; Found 504.1358.
Methyl (E)-3-(3,4-dichloro-5-(((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methyl)phenyl)acrylate (3jb): GP-3 was carried out by using arylmethanesulfonyl ester 1j (81 mg, 0.2 mmol), olefin 2b (26 mg, 0.3 mmol), Pd(OAc)_2 (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 3jb (78 mg, 77%), as a colourless crystalline solid. [TLC (petroleum ether/ethyl acetate 90:10, R_f(1j) = 0.60, R_f(3jb) = 0.40, UV detection]. Melting point: 95-97 °C. IR (MIR-ATR, 4000–600 cm⁻¹): 2924, 1716, 1456, 1266, 1175, 751 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ 7.61 (d, J = 2.0 Hz, 1H), 7.55 (d, J = 2.0 Hz, 1H), 7.51 (d, J = 16.0 Hz, 1H), 6.41 (d, J = 16.0 Hz, 1H), 4.66 (s, 2H), 4.10 (s, 2H), 3.75 (s, 3H), 1.73 (dt, J = 13.1, 6.5 Hz, 2H), 1.42 (dd, J = 6.2, 3.9 Hz, 4H), 1.01 (d, J = 6.6 Hz, 6H), 0.97 (d, J = 6.6 Hz, 6H) ppm. ¹³C ⁰¹H NMR (101 MHz, CDCl₃) δ 166.5, 141.1, 135.0, 134.8, 134.4, 130.2, 130.1, 128.6, 121.1, 121.0, 70.8, 54.4, 52.1, 43.1 (2C), 39.4, 24.8 (2C), 23.9 (2C), 23.8 (2C) ppm. HRMS (ESI) m/z: [(M+H)]⁺ Calcd for C₂₂H₃₀Cl₂NO₅S⁺ 490.1216; Found 490.1211.

Ethyl (E)-3-(2,3-dichloro-5-(((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methyl)phenyl)acrylate (3ka): GP-3 was carried out by using arylmethanesulfonyl ester 1k (81 mg, 0.2 mmol), olefin 2a (30 mg, 0.3 mmol), Pd(OAc)₂ (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 3ka (72 mg, 72%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, R_f(1k) = 0.60, R_f(3ka) = 0.40, UV detection]. IR (MIR-
ATR, 4000–600 cm⁻¹: 2957, 1714, 1464, 1176, 981, 755 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ 8.04 (d, J = 16.0 Hz, 1H), 7.27 (s, 1H), 6.48 (d, J = 16.0 Hz, 1H), 4.42 (s, 2H), 4.29 (d, J = 7.1 Hz, 2H), 4.14 (s, 2H), 1.78 (dd, J = 13.1, 6.5 Hz, 2H), 1.49 (t, J = 6.5 Hz, 4H), 1.35 (t, J = 7.1 Hz, 3H), 1.01 (d, J = 6.6 Hz, 6H), 0.98 (d, J = 6.6 Hz, 6H) ppm. ¹³C NMR (100 MHz, CDCl₃) δ 166.0, 139.4, 135.8, 134.7, 134.2, 133.2, 128.1, 127.4, 123.5, 121.4, 71.5, 61.1, 56.0, 43.3 (2C), 39.7, 24.9 (2C), 23.9 (4C), 14.4 ppm.

HRMS (ESI) m/z: [(M+K)⁺] Calcd for C₂₃H₃₁Cl₂KNO₅S⁺ 542.0932; Found 542.0949.

Ethyl (E)-3-(2,4-dichloro-5-(((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methyl)phenylacrylate (3la): GP-3 was carried out by using arylmethanesulphonate ester 1l (81 mg, 0.2 mmol), olefin 2a (30 mg, 0.3 mmol), Pd(OAc)₂ (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 3la (75 mg, 74%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, Rf(1l) = 0.60, Rf(3la) = 0.40, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): 2959, 1714, 1465, 1365, 1176, 979, 754 cm⁻¹.¹H NMR (400 MHz, CDCl₃) δ 7.96 (d, J = 16.0 Hz, 1H), 7.80 (s, 1H), 7.57 (s, 1H), 6.49 (d, J = 16.0 Hz, 1H), 4.64 (s, 2H), 4.31 – 4.25 (m, 2H), 4.17 (s, 2H), 1.83 – 1.77 (m, 2H), 1.49 (dd, J = 6.2, 3.3 Hz, 4H), 1.34 (t, J = 7.1 Hz, 3H), 1.01 (d, J = 6.6 Hz, 6H), 0.97 (d, J = 6.6 Hz, 6H) ppm. ¹³C {¹H} NMR (100 MHz, CDCl₃) δ 165.9, 138.1, 136.6, 136.3, 132.4, 131.2, 131.2, 125.1, 122.7, 121.1, 70.8, 60.9, 53.1, 43.1 (2C), 39.4, 24.8 (2C), 23.8 (2C), 23.8 (2C), 14.2 ppm. HRMS (ESI) m/z: [(M+H)⁺] Calcd for C₂₃H₃₂Cl₂NO₅S⁺ 504.1373; Found 504.1380.
Ethyl (E)-3-(3-(((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methyl)phenyl)acrylate (3aa): GP-3 was carried out by using arylmethanesulfonate ester 1a (67 mg, 0.2 mmol), olefin 2a (30 mg, 0.3 mmol), Pd(OAc)$_2$ (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 3aa (70 mg, 81%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, $R_f$(1a) = 0.60, $R_f$(3aa) = 0.40, UV detection]. IR (MIR-ATR, 4000–600 cm$^{-1}$): 2958, 1693, 1635, 1357, 1169, 979, 752 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.67 (d, $J$ = 16.1 Hz, 1H), 7.62 – 7.54 (m, 2H), 7.50 – 7.39 (m, 2H), 6.48 (d, $J$ = 16.0 Hz, 1H), 4.46 (s, 2H), 4.27 (q, $J$ = 7.1 Hz, 2H), 4.02 (s, 2H), 1.76 (dp, $J$ = 13.0, 6.5 Hz, 2H), 1.52 – 1.39 (m, 4H), 1.34 (t, $J$ = 7.1 Hz, 3H), 0.99 (d, $J$ = 6.6 Hz, 6H), 0.96 (d, $J$ = 6.6 Hz, 6H) ppm. $^{13}$C {$^1$H} NMR (100 MHz, CDCl$_3$) $\delta$ 166.6, 143.2, 135.4, 132.3, 130.2, 129.6, 128.7, 128.1, 121.2, 119.6, 71.1, 60.6, 56.9, 43.1 (2C), 39.4, 24.7 (2C), 23.8 (2C), 14.2 ppm. HRMS (ESI) m/z: [(M+K)$^+$] Calcd for C$_{23}$H$_{33}$KNO$_5$S$^+$ 474.1711; Found 474.1721.

diagram

tert-Butyl (E)-3-(((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methyl)phenyl)acrylate (3ac): GP-3 was carried out by using arylmethanesulfonate ester 1a (67 mg, 0.2 mmol), olefin 2c (38 mg, 0.3 mmol), Pd(OAc)$_2$ (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 3ac (69 mg, 75%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, $R_f$(1a) = 0.60, $R_f$(3ac) = 0.45, UV detection]. IR (MIR-ATR, 4000–600 cm$^{-1}$): 2960, 1705, 1391, 1150, 983, 822 cm$^{-1}$. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 7.61 – 7.51 (m, 3H), 7.47 – 7.39 (m, 3H), 6.41 (d, $J$ = 16.0 Hz, 1H), 4.45 (s, 2H), 4.02 (s, 2H), 1.78 – 1.73 (m, 2H), 1.53 (s, 9H), 1.47 – 1.44 (m, 4H), 0.99 (d, $J$ = 6.6 Hz, 6H), 0.96 (d, $J$ = 6.6 Hz, 6H) ppm. $^{13}$C {$^1$H} NMR (151 MHz, CDCl$_3$) $\delta$ 165.9, 142.1, 135.6, 132.3, 130.2, 129.6, 128.7, 128.1, 121.6, 80.8, 71.2, 57.0, 43.1 (2C), 39.4, 28.1 (3C), 24.8 (2C), 23.9
(2C), 23.8 (2C) ppm. HRMS (ESI) m/z: [(M+K)]$^+$ Calcd for C$_{25}$H$_{37}$KNO$_5$S$^+$ 502.2043; Found 502.2043.

Phenyl (E)-3-(3-(((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methyl)phenyl)acrylate (3ad): GP-3 was carried out by using arylmethanesulfonate ester 1a (67 mg, 0.2 mmol), olefin 2d (44 mg, 0.3 mmol), Pd(OAc)$_2$ (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 $^\circ$C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 3ad (70 mg, 72%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, $R_f$(1a) = 0.60, $R_f$(3ad) = 0.40, UV detection)]. IR (MIR-ATR, 4000–600 cm$^{-1}$): 2958, 1728, 1358, 1137, 979, 748 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.87 (d, $J$ = 16.0 Hz, 1H), 7.66 (dd, $J$ = 13.3, 3.6 Hz, 2H), 7.60 – 7.45 (m, 2H), 7.41 (dd, $J$ = 8.3, 7.6 Hz, 2H), 7.26 (tt, $J$ = 8.5, 1.1 Hz, 1H), 7.22 – 7.13 (m, 2H), 6.69 (d, $J$ = 16.0 Hz, 1H), 4.49 (s, 2H), 4.05 (s, 2H), 1.78 (dt, $J$ = 13.1, 6.5 Hz, 2H), 1.47 (dd, $J$ = 6.2, 3.5 Hz, 4H), 1.0 (d, $J$ = 6.6 Hz, 6H), 0.97 (d, $J$ = 6.6 Hz, 6H) ppm. $^{13}$C {$^1$H} NMR (151 MHz, CDCl$_3$) $\delta$ 165.1, 150.7, 145.2, 135.1, 132.8, 129.8, 129.5 (2C), 129.0, 128.4, 126.0, 121.6 (2C), 121.4, 118.7, 71.3, 56.9, 43.2 (2C), 39.5, 24.8 (2C), 23.9 (2C), 23.9 (2C) ppm. HRMS (ESI) m/z: [(M+K)]$^+$ Calcd for C$_{27}$H$_{33}$KNO$_5$S$^+$ 522.1711; Found 522.1718.

Naphthalen-2-yl (E)-3-(3-(((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methyl)phenyl)acrylate (3ae): GP-3 was carried out by using arylmethanesulfonate ester 1a (67 mg, 0.2 mmol), olefin 2e (59 mg, 0.3 mmol), Pd(OAc)$_2$ (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and
hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 3ae (75 mg, 70%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, \( R_f(1\alpha) = 0.60, R_f(3ae) = 0.40 \), UV detection]. IR (MIR-ATR, 4000–600 cm\(^{-1}\)): 2926, 1732, 1363, 1137, 982, 759 cm\(^{-1}\). \(^1\)H NMR (400 MHz, CDCl\(_3\)) \( \delta 7.99 \) (d, \( J = 16.0 \) Hz, 1H), 7.89 (d, \( J = 9.4 \) Hz, 2H), 7.78 (d, \( J = 8.3 \) Hz, 1H), 7.73 (s, 1H), 7.68 (s, 1H), 7.55 – 7.48 (m, 5H), 7.33 (dd, \( J = 7.5, 0.9 \) Hz, 1H), 6.86 (d, \( J = 16.0 \) Hz, 1H), 4.51 (s, 2H), 4.07 (s, 2H), 1.79 (dt, \( J = 13.0, 6.5 \) Hz, 2H), 1.48 (dd, \( J = 6.2, 3.4 \) Hz, 4H), 1.01 (d, \( J = 6.6 \) Hz, 6H), 0.98 (d, \( J = 6.6 \) Hz, 6H) ppm. \(^13\)C \{\(^1\)H\} NMR (151 MHz, CDCl\(_3\)) \( \delta 165.2, 146.6, 145.6, 135.1, 134.7, 133.0, 130.6, 129.8, 129.1, 128.4, 128.1, 126.9, 126.6, 126.1, 125.6, 121.4, 121.3, 118.5, 118.1, 71.3, 56.9, 43.1 (2C), 39.5, 24.8 (2C), 23.9 (2C) ppm. HRMS (ESI) m/z: [(M+K)]\(^+\) Calcd for C\(_{31}\)H\(_{35}\)KNO\(_5\)S\(^+\) 572.1868; Found 572.1873.

\((E)-2-((3-(3-(((2-Cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methyl)phenyl)acryloyloxy)ethyl benzoate (3af):\) GP-3 was carried out by using arylmethanesulphonate ester 1a (67 mg, 0.2 mmol), olefin 2f (66 mg, 0.3 mmol), Pd(OAc)\(_2\) (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 3af (81 mg, 73%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, \( R_f(1\alpha) = 0.60, R_f(3af) = 0.30 \), UV detection]. IR (MIR-ATR, 4000–600 cm\(^{-1}\)): 2961, 1716, 1312, 1169, 981, 729 cm\(^{-1}\). \(^1\)H NMR (400 MHz, CDCl\(_3\)) \( \delta 8.09 – 8.02 \) (m, 2H), 7.71 (d, \( J = 16.1 \) Hz, 1H), 7.59 (s, 1H), 7.58 – 7.53 (m, 2H), 7.49 – 7.41 (m, 4H), 6.51 (d, \( J = 16.0 \) Hz, 1H), 4.57 (dqd, \( J = 7.7, 4.0, 1.6 \) Hz, 4H), 4.45 (s, 2H), 4.02 (s, 2H), 1.74 (dt, \( J = 13.0, 6.5 \) Hz, 2H), 1.48 – 1.39 (m, 4H), 0.97 (d, \( J = 6.6 \) Hz, 6H), 0.94 (d, \( J = 6.6 \) Hz, 6H) ppm. \(^13\)C \{\(^1\)H\} NMR (151 MHz, CDCl\(_3\)) \( \delta 166.4, 166.4, 144.2, 135.2, 133.2, 132.6, 130.3, 129.8, 129.8, 129.7 (2C), 128.9, 128.4, 128.3
ppm. HRMS (ESI) m/z: [(M+NH₄)⁺] Calcd for C₃₀H₄₁N₂O₇S⁺ 573.2629; Found 573.2646.

2-Cyano-2-isobutyl-4-methylpentyl (E)-(3-(3-oxobut-1-en-1-yl)phenyl)methanesulfonate (3ag): GP-3 was carried out by using arylmethanesulfonate ester 1a (67 mg, 0.2 mmol), olefin 2g (21 mg, 0.3 mmol), Pd(OAc)₂ (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 3ag (64 mg, 79%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, Rᶠ(1a) = 0.60, Rᶠ(3ag) = 0.40, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): 2922, 1671, 1614, 1357, 1172, 979, 818, 696 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ 7.64 (s, 1H), 7.52 (dt, J = 16.4 Hz, 1H), 7.47 (dd, J = 12.0, 4.6 Hz, 2H), 6.76 (d, J = 16.3 Hz, 1H), 4.48 (s, 2H), 4.02 (s, 2H), 2.39 (s, 3H), 1.76 (dt, J = 13.0, 6.5 Hz, 2H), 1.50 – 1.41 (m, 4H), 0.99 (d, J = 6.6 Hz, 6H), 0.96 (d, J = 6.6 Hz, 6H) ppm. ¹³C-{¹H} NMR (100 MHz, CDCl₃) δ 198.1, 142.0, 135.4, 132.6, 130.6, 129.7, 128.8, 128.2, 128.1, 121.3, 71.3, 56.9, 43.1 (2C), 39.4, 27.6, 24.7 (2C), 23.8 (2C), 23.8 (2C) ppm. HRMS (ESI) m/z: [(M+K)⁺] Calcd for C₂₂H₃₁KNO₄S⁺ 444.1605; Found 444.1633.

Diethyl 2-((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methylphenyl)maleate (3ah): GP-3 was carried out by using arylmethanesulfonate ester 1a (67 mg, 0.2 mmol), olefin 2h (52 mg, 0.3 mmol), Pd(OAc)₂ (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave
irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 3ah (74 mg, 73%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, \(R_f(1a) = 0.60, R_f(3ah) = 0.20\), UV detection]. IR (MIR-ATR, 4000–600 cm\(^{-1}\)): 2962, 1720, 1356, 1169, 980, 817 cm\(^{-1}\). 

\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta 7.57 (d, J = 1.5\) Hz, 1H), 7.54 – 7.50 (m, 2H), 7.46 (dd, \(J = 8.8, 6.5\) Hz, 1H), 6.33 (s, 1H), 4.46 (s, 2H), 4.42 (d, \(J = 7.1\) Hz, 2H), 4.24 (q, \(J = 7.1\) Hz, 2H), 4.04 (s, 2H), 1.77 (dt, \(J = 13.0, 6.5\) Hz, 2H), 1.46 (dd, \(J = 6.2, 3.4\) Hz, 4H), 1.38 (d, \(J = 7.1\) Hz, 3H), 1.31 (s, 2H), 0.99 (d, \(J = 6.6\) Hz, 6H), 0.96 (d, \(J = 6.6\) Hz, 6H) ppm. 

\(^{13}\)C \(\{^1\)H\} NMR (151 MHz, CDCl\(_3\)) \(\delta 167.4, 164.6, 147.4, 134.4, 132.6, 129.7, 128.8, 128.3, 127.7, 121.2, 118.8, 71.2, 62.0, 61.1, 56.8, 43.1 (2C), 39.4, 24.7 (2C), 23.8 (2C), 23.8 (2C), 14.1, 14.0 ppm. HRMS (ESI) m/z: [(M+H)]\(^+\) Calcd for C\(_{26}\)H\(_{38}\)NO\(_7\)S\(^+\) 508.2363; Found 508.2372.

**Ethyl (E)-3-((((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methyl)phenyl)but-2-enoate (3ai):** GP-3 was carried out by using arylmethanesulfonate ester 1a (67 mg, 0.2 mmol), olefin 2i (34 mg, 0.3 mmol), Pd(OAc)\(_2\) (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 3ai (63 mg, 70%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, \(R_f(1a) = 0.60, R_f(3ai) = 0.40\), UV detection]. IR (MIR-ATR, 4000–600 cm\(^{-1}\)): 2960, 1709, 1359, 1167, 984, 755 cm\(^{-1}\). 

\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta 7.55 – 7.49\) (m, 2H), 7.44 (dd, \(J = 5.4, 4.2\) Hz, 2H), 6.14 (dd, \(J = 2.4, 1.1\) Hz, 1H), 4.47 (s, 2H), 4.22 (q, \(J = 7.1\) Hz, 2H), 4.03 (s, 2H), 2.58 (d, \(J = 1.3\) Hz, 3H), 1.77 (dt, \(J = 13.0, 6.5\) Hz, 2H), 1.46 (dd, \(J = 6.2, 3.4\) Hz, 4H), 1.32 (t, \(J = 7.1\) Hz, 3H), 0.99 (d, \(J = 6.6\) Hz, 6H), 0.96 (d, \(J = 6.6\) Hz, 6H) ppm. 

\(^{13}\)C \(\{^1\)H\} NMR (100 MHz, CDCl\(_3\)) \(\delta 166.5, 154.1, 143.1, 131.2, 129.2, 128.6, 127.6, 127.2, 121.2, 118.1, 71.1, 60.0, 57.1, 43.1 (2C), 39.4, 24.7 (2C), 23.9 (2C), 23.8 (2C), 17.9, 14.3 ppm. HRMS (ESI) m/z: [(M+H)]\(^+\) Calcd for C\(_{24}\)H\(_{36}\)NO\(_7\)S\(^+\) 450.2309; Found 450.2315.
2-Cyano-2-isobutyl-4-methylpentyl \((E)-(3-(2-(methylsulfonyl)vinyl)phenyl)methanesulfonate (3aj):\) GP-3 was carried out by using arylmethanesulfonate ester 1a (67 mg, 0.2 mmol), olefin 2j (32 mg, 0.3 mmol), Pd(OAc)_2 (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 65/35), furnished the product 3aj (67 mg, 76%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, \(R_f(1a) = 0.60, R_f(3aj) = 0.10\), UV detection]. IR (MIR-ATR, 4000–600 cm\(^{-1}\)): 2921, 2855, 1734, 1458, 1184, 900 cm\(^{-1}\). \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.65 (s, 1H), 7.60 (d, \(J = 15.5\) Hz, 1H), 7.56 – 7.51 (m, 2H), 7.48 (dd, \(J = 8.8, 6.3\) Hz, 1H), 7.03 (d, \(J = 15.5\) Hz, 1H), 4.48 (s, 2H), 4.04 (s, 2H), 3.02 (s, 3H), 1.74 (dd, \(J = 13.0, 6.5\) Hz, 2H), 1.50 – 1.41 (m, 4H), 0.98 (d, \(J = 6.6\) Hz, 6H), 0.95 (d, \(J = 6.6\) Hz, 6H) ppm. \(^{13}\)C \{\(^1\)H\} NMR (151 MHz, CDCl\(_3\)) \(\delta\) 142.4, 133.5, 133.1, 130.3, 129.9, 129.7, 128.6, 127.8, 121.4, 71.4, 56.7, 43.2, 43.1 (2C), 39.5, 24.8 (2C), 23.8 (4C) ppm. HRMS (ESI) m/z: [(M+K)]\(^+\) Calcd for C\(_{21}\)H\(_{31}\)KNO\(_5\)S\(_2^+\) 480.1275; Found 480.1271.

2-Cyano-2-isobutyl-4-methylpentyl \((E)-(3-(2-(phenylsulfonyl)vinyl)phenyl)methanesulfonate (3ak):\) GP-3 was carried out by using arylmethanesulfonate ester 1a (67 mg, 0.2 mmol), olefin 2k (50 mg, 0.3 mmol), Pd(OAc)_2 (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 65/35), furnished the product 3ak (77 mg, 77%), as a white solid. [TLC (petroleum ether/ethyl acetate 90:10, \(R_f(1a) = 0.60, R_f(3ak) = 0.10\), UV detection]. Melting point: 85-87
°C. IR (MIR-ATR, 4000–600 cm⁻¹): 2959, 1363, 1306, 1021, 770 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ 7.94 (dd, J = 5.3, 3.4 Hz, 2H), 7.70 – 7.60 (m, 2H), 7.59 – 7.42 (m, 6H), 6.95 (d, J = 15.4 Hz, 1H), 4.45 (s, 2H), 4.03 (s, 2H), 1.74 (dt, J = 13.0, 6.5 Hz, 2H), 1.48 – 1.39 (m, 4H), 0.96 (d, J = 6.6 Hz, 6H), 0.93 (d, J = 6.6 Hz, 6H) ppm. ¹³C ¹H NMR (151 MHz, CDCl₃) δ 141.0, 140.3, 133.5, 133.3, 130.6, 129.8, 129.4, 129.3 (2C), 128.7, 128.4, 127.7 (2C), 121.3, 71.2, 56.7, 43.0 (2C), 39.4, 24.7 (2C), 23.8 (2C), 23.8 (2C) ppm. HRMS (ESI) m/z: [(M+Na)]⁺ Calcd for C₂₆H₃₃NNaO₅S²⁺ 526.1692; Found 526.1700.

2-Cyano-2-isobutyl-4-methylpentyl (E)-(3-(2- (diethoxyphosphoryl)vinyl)phenyl)methanesulfonate (3al): GP-3 was carried out by using arylmethanesulfonate ester 1a (67 mg, 0.2 mmol), olefin 2l (49 mg, 0.3 mmol), Pd(OAc)₂ (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 3al (63 mg, 63%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, Rf(1a) = 0.60, Rf(3al) = 0.20, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): 2963, 1465, 1359, 1171, 1026, 970, 736 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ 7.58 (s, 1H), 7.55 – 7.52 (m, 1H), 7.49 (d, J = 4.9 Hz, 1H), 7.46 (d, J = 2.0 Hz, 1H), 7.45 – 7.40 (m, 1H), 6.32 (t, J = 17.3 Hz, 1H), 4.46 (s, 2H), 4.18 – 4.08 (m, 4H), 4.02 (s, 2H), 1.78 – 1.73 (m, 2H), 1.45 (dd, J = 6.2, 4.1 Hz, 4H), 1.35 (t, J = 7.1 Hz, 6H), 0.98 (d, J = 6.6 Hz, 6H), 0.95 (d, J = 6.6 Hz, 6H) ppm. ¹³C ¹H NMR (151 MHz, CDCl₃) δ 147.2, 147.2, 135.7 (d, J_C-P = 24 Hz), 132.3, 129.7, 129.6, 128.6, 128.0, 121.2, 115.7 (d, J_C-P = 24 Hz), 71.2, 61.9, 61.9, 56.9, 43.1 (2C), 39.4, 24.7 (2C), 23.8 (2C), 23.8 (2C), 16.4, 16.3 ppm. HRMS (ESI) m/z: [(M+H)]⁺ Calcd for C₂₄H₃₉NO₆PS⁺ 500.2230; Found 500.2248.
2-Cyano-2-isobutyl-4-methylpentyl (E)-(3-(3-(dimethylamino)-3-oxoprop-1-en-1-yl)phenyl)methanesulfonate (3am): GP-3 was carried out by using arylmethanesulfonate ester 1a (67 mg, 0.2 mmol), olefin 2m (30 mg, 0.3 mmol), Pd(OAc)$_2$ (5 mg, 10 mol%), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 65/35), furnished the product 3am (64 mg, 74%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, $R_f(1a) = 0.60$, $R_f(3am) = 0.10$, UV detection]. IR (MIR-ATR, 4000–600 cm$^{-1}$): 2956, 1723, 1649, 1606, 1297, 971, 619 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.65 (d, $J = 15.3$ Hz, 2H), 7.56 – 7.50 (m, 1H), 7.46 – 7.39 (m, 2H), 6.96 (d, $J = 15.5$ Hz, 1H), 4.47 (s, 2H), 3.99 (s, 2H), 3.18 (s, 3H), 3.07 (s, 3H), 1.73 (dd, $J = 13.1$, 6.5 Hz, 2H), 1.48 – 1.38 (m, 4H), 0.97 (d, $J = 6.6$ Hz, 6H), 0.94 (d, $J = 6.6$ Hz, 6H) ppm. $^{13}$C $\{^1$H$\}$ NMR (100 MHz, CDCl$_3$) $\delta$ 166.3, 141.0, 136.3, 131.6, 129.5, 129.4, 129.2, 127.9, 121.3, 118.7, 71.3, 57.0, 43.1 (2C), 39.4, 37.4, 36.0, 29.6, 24.7 (2C), 23.8 (2C), 23.8 (2C) ppm. HRMS (ESI) m/z: [(M+H)]$^+$ Calcd for C$_{23}$H$_{35}$N$_2$O$_4$S$^+$ 435.2312; Found 435.2321.

2-cyano-2-isobutyl-4-methylpentyl (E)-(3-(2-nitrostyryl)phenyl)methanesulfonate (3ax): GP-3 was carried out by using arylmethanesulfonate ester 1a (67 mg, 0.2 mmol), olefin 2x (44 mg, 0.3 mmol), Pd(OAc)$_2$ (5 mg, 10 mol%), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 65/35), furnished the product 3ax (75 mg, 78%), as a yellow coloured liquid. [TLC (petroleum ether/ethyl acetate 90:10, $R_f(1a) = 0.60$, $R_f(3ax) = 0.40$, UV detection]. IR (MIR-ATR, 4000–600 cm$^{-1}$): 2957, 1521, 1172, 978, 824, 741, 697 cm$^{-1}$. 


1H NMR (400 MHz, CDCl₃) δ 7.98 (dd, J = 8.2, 1.1 Hz, 1H), 7.75 (dd, J = 7.9, 1.0 Hz, 1H), 7.62 (ddd, J = 12.6, 4.5, 2.5 Hz, 3H), 7.58 (s, 1H), 7.47 – 7.38 (m, 3H), 7.08 (d, J = 16.2 Hz, 1H), 4.48 (s, 2H), 4.01 (s, 2H), 1.76 (dt, J = 13.0, 6.5 Hz, 2H), 1.46 (dd, J = 6.2, 2.0 Hz, 4H), 0.98 (d, J = 6.6 Hz, 6H), 0.95 (d, J = 6.6 Hz, 6H). 13C NMR (100 MHz, CDCl₃) δ 147.9, 137.4, 133.2, 132.6, 132.6, 130.7, 129.8, 129.5, 128.3, 128.2, 127.9, 127.3, 124.8, 124.8, 121.3, 71.2, 57.0, 43.1 (2C), 39.4, 24.7 (2C), 23.8 (2C), 23.8 (2C). HRMS (ESI) m/z: [(M+NH₄)⁺] Calcd for C₂₆H₃₆N₃O₅S⁺ 502.2370; Found 502.2373.

2-cyano-2-isobutyl-4-methylpentyl (E)-(3-(4-nitrostyryl)phenyl)methanesulfonate (3ay):
GP-3 was carried out by using arylmethanesulfonate ester 1a (67 mg, 0.2 mmol), olefin 2y (44 mg, 0.3 mmol), Pd(OAc)₂ (5 mg, 10 mol%), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 ºC for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 65/35), furnished the product 3ay (73 mg, 75%), as a yellow coloured liquid. [TLC (petroleum ether/ethyl acetate 90:10, Rf(1a) = 0.60, Rf(3ay) = 0.40, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): 2957, 1592, 1338, 1174, 1108, 979, 747 cm⁻¹. 1H NMR (400 MHz, CDCl₃) δ 8.23 (d, J = 8.8 Hz, 2H), 7.68 – 7.62 (m, 3H), 7.59 (dd, J = 8.3, 2.6 Hz, 1H), 7.46 (t, J = 7.6 Hz, 1H), 7.41 (dd, J = 6.2, 1.5 Hz, 1H), 7.31 – 7.18 (m, 2H), 4.50 (s, 2H), 4.02 (s, 2H), 1.76 (dt, J = 13.0, 6.5 Hz, 2H), 1.46 (dd, J = 6.2, 3.2 Hz, 4H), 0.98 (d, J = 6.6 Hz, 6H), 0.95 (d, J = 6.6 Hz, 6H). 13C NMR (101 MHz, CDCl₃) δ 146.9, 143.3, 137.1, 132.0, 130.9, 129.6, 129.2, 128.0, 127.8, 127.5, 127.0 (2C), 124.2 (2C), 121.4, 71.4, 57.1, 43.1 (2C), 39.5, 24.7 (2C), 23.8 (2C), 23.8 (2C). HRMS (ESI) m/z: [(M+NH₄)⁺] Calcd for C₂₆H₃₆N₃O₅S⁺ 502.2370; Found 502.2373.
2-((2-(6-Methoxynaphthalen-2-yl)propanoyl)oxy)ethyl (E)-3-(3-(((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methyl)phenyl)acrylate (3an): GP-3 was carried out by using arylmethanesulphonate ester 1a (67 mg, 0.2 mmol), olefin 2n (98 mg, 0.3 mmol), Pd(OAc)₂ (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 65/35), furnished the product 3an (100 mg, 76%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, RF(1a) = 0.60, RF(3an) = 0.20, UV detection). IR (MIR-ATR, 4000–600 cm⁻¹): 2958, 1720, 1637, 1359, 1160, 981, 816 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ 7.70 – 7.64 (m, 3H), 7.53 (d, J = 16.1 Hz, 1H), 7.50 – 7.46 (m, 2H), 7.44 (q, J = 3.5 Hz, 2H), 7.41 (dd, J = 8.6, 1.8 Hz, 1H), 7.08 (dd, J = 8.9, 2.5 Hz, 1H), 7.03 (d, J = 2.5 Hz, 1H), 6.30 (d, J = 16.0 Hz, 1H), 4.46 (s, 2H), 4.43 – 4.30 (m, 4H), 4.04 (s, 2H), 3.93 – 3.84 (m, 4H), 1.77 (dt, J = 13.0, 6.5 Hz, 2H), 1.59 (d, J = 7.2 Hz, 3H), 1.46 (dd, J = 6.2, 3.5 Hz, 4H), 0.99 (d, J = 6.6 Hz, 6H), 0.96 (d, J = 6.6 Hz, 6H) ppm. ¹³C {¹H} NMR (100 MHz, CDCl₃) δ 174.4, 166.1, 157.5, 143.8, 135.4, 135.1, 133.7, 132.4, 130.3, 129.6, 129.2, 128.8, 128.1, 127.1, 126.2, 125.9, 121.3, 118.9, 118.7, 105.5, 71.2, 62.3, 62.2, 56.8, 55.2, 45.3, 43.1 (2C), 39.4, 24.8 (2C), 23.9 (2C), 23.8 (2C), 18.4 ppm. HRMS (ESI) m/z: [(M+K)⁺] Calcd for C₃₇H₄₅KNO₈S⁺ 702.2497; Found 702.2515.

2-((2-(4-Isobutylphenyl)propanoyl)oxy)ethyl (E)-3-(3-(((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methyl)phenyl)acrylate (3ao): GP-3 was carried out by using arylmethanesulphonate ester 1a (67 mg, 0.2 mmol), olefin 2o (91 mg, 0.3 mmol), Pd(OAc)₂ (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and
hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 80/20), furnished the product 3ao (84 mg, 66%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, Rf(1a) = 0.60, Rf(3ao) = 0.30, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): 2954, 1726, 1639, 1365, 1167, 985, 754 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ 7.66 (d, J = 16.0 Hz, 1H), 7.60 (s, 1H), 7.59 – 7.54 (m, 1H), 7.51 – 7.43 (m, 2H), 7.20 (d, J = 8.1 Hz, 2H), 7.06 (d, J = 8.1 Hz, 2H), 6.45 (d, J = 16.1 Hz, 1H), 4.47 (s, 2H), 4.41 – 4.29 (m, 4H), 4.05 (s, 2H), 3.74 (d, J = 7.1 Hz, 1H), 2.39 (d, J = 7.2 Hz, 2H), 1.50 (d, J = 7.2 Hz, 4H), 1.46 (dd, J = 6.2, 3.4 Hz, 4H), 0.99 (d, J = 6.6 Hz, 6H), 0.96 (d, J = 6.6 Hz, 6H), 0.86 (d, J = 6.6 Hz, 6H) ppm. ¹³C {¹H} NMR (151 MHz, CDCl₃) δ 174.5, 166.2, 143.9, 140.5, 137.4, 135.2, 132.5, 130.3, 129.6, 129.4, 129.3, 128.8 (2C), 128.2, 127.1, 127.0 (2C), 121.2, 118.8, 71.2, 62.3, 56.8, 44.9, 44.9, 43.1 (2C), 39.4, 30.1, 24.7 (2C), 23.8 (2C), 23.8 (2C), 22.3 (3C), 18.4 ppm. HRMS (ESI) m/z: [(M+K)]⁺ Calcd for C₃₆H₄₉KNO₇S⁺ 678.2861; Found 678.2879.

4-Acetamidophenyl (E)-3-(3-(((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methyl)phenyl)acrylate (3ap): GP-3 was carried out by using arylmethanesulfonate ester 1a (67 mg, 0.2 mmol), olefin 2p (61 mg, 0.3 mmol), Pd(OAc)₂ (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 65/35), furnished the product 3ap (70 mg, 65%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, Rf(1a) = 0.60, Rf(3ap) = 0.10, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): 2922, 2859, 1726, 1678, 1510, 1142, 982 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ 7.85 (d, J = 16.0 Hz, 1H), 7.68 – 7.59 (m, 2H), 7.58 – 7.47 (m, 5H), 7.10 (d, J = 8.8 Hz, 2H), 6.66 (d, J = 16.0 Hz, 1H), 4.48 (s, 2H), 4.05 (s, 2H), 2.15 (s, 3H), 1.77 (dt, J = 13.0, 6.5 Hz, 3H), 1.47 (dd, J = 6.2, 3.7 Hz, 4H), 1.0 (d, J = 6.6 Hz, 6H), 0.97 (d, J = 6.6 Hz, 6H) ppm. ¹³C {¹H} NMR (151 MHz, CDCl₃) δ 168.4, 165.2, 146.8, 145.3, 135.7, 135.0, 132.8, 130.5, 129.7, 129.0, 128.3, 121.9, 121.3, 120.8, 118.5, 71.2, 56.8, 43.1 (2C), 39.5, 24.8
2-((2-(2-Fluoro-[1,1'-biphenyl]-4-yl)propanoyl)oxy)ethyl (E)-3-(3-(((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methyl)phenyl)acrylate (3aq): GP-3 was carried out by using arylmethanesulphonate ester 1a (67 mg, 0.2 mmol), olefin 2q (102 mg, 0.3 mmol), Pd(OAc)$_2$ (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 80/20), furnished the product 3aq (73 mg, 54%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, $R_f$(1a) = 0.60, $R_f$(3aq) = 0.30, UV detection). IR (MIR-ATR, 4000–600 cm$^{-1}$): 2958, 1713, 1640, 1358, 1169, 980, 817, 734 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.63 (d, $J$ = 16.1 Hz, 1H), 7.54 – 7.43 (m, 5H), 7.43 – 7.30 (m, 5H), 7.15 (ddd, $J$ = 8.7, 5.4, 1.7 Hz, 2H), 6.42 (d, $J$ = 16.0 Hz, 1H), 4.39 (s, 6H), 4.02 (s, 2H), 3.83 – 3.78 (m, 1H), 1.75 (dd, $J$ = 13.1, 6.5 Hz, 2H), 1.55 (d, $J$ = 7.2 Hz, 3H), 1.45 (dd, $J$ = 6.2, 3.9 Hz, 4H), 0.98 (d, $J$ = 6.6 Hz, 6H), 0.95 (d, $J$ = 6.6 Hz, 6H) ppm. $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 173.7, 166.1, 144.0, 141.0 ($J_{C,F}$ = 95 Hz), 135.4, 135.1, 132.5, 130.8 ($J_{C,F}$ = 4 Hz), 130.3, 129.6, 128.9, 128.9, 128.8, 128.4, 128.1, 127.6, 123.6 ($J_{C,F}$ = 3 Hz), 121.2, 118.7, 115.3, 115.1, 71.1, 62.6, 62.1, 56.8, 44.9, 43.1 (2C), 39.4, 24.7 (2C), 23.8 (2C), 23.8 (2C), 18.2 ppm. HRMS (ESI) m/z: [(M+NH$_4$)$_3$]$^+$ Calcd for C$_{38}$H$_{48}$FN$_2$O$_7$S$^+$ 695.3161; Found 695.3141.
2-Cyano-2-isobutyl-4-methylpentyl (2',5'-dioxo-2',5'-dihydro-[1,1'-biphenyl]-3-yl)methanesulfonate (3ar): GP-3 was carried out by using arylmethanesulfonate ester 1a (67 mg, 0.2 mmol), cyclicolefin 2r (32 mg, 0.3 mmol), Pd(OAc)$_2$ (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 3ar (57 mg, 65%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate, 100/0 to 90/15), $R_f$(1a) = 0.60, $R_f$(3ar) = 0.30, UV detection]. IR (MIR-ATR, 4000–600 cm$^{-1}$): 2957, 1657, 1593, 1358, 1274, 1174, 981, 828 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.54 (dddd, $J$ = 13.4, 11.9, 6.7, 4.4 Hz, 4H), 6.90 (d, $J$ = 2.4 Hz, 1H), 6.89 – 6.82 (m, 2H), 4.51 (s, 2H), 4.05 (s, 2H), 1.77 (dp, $J$ = 13.0, 6.5 Hz, 2H), 1.48 (dd, $J$ = 6.2, 1.4 Hz, 4H), 0.98 (d, $J$ = 6.6 Hz, 6H), 0.95 (d, $J$ = 6.6 Hz, 6H) ppm. $^{13}$C {$^1$H} NMR (151 MHz, CDCl$_3$) $\delta$ 187.3, 186.2, 144.9, 137.0, 136.3, 133.5, 133.1, 132.3, 131.7, 130.0, 129.3, 127.7, 121.3, 71.4, 56.9, 43.1 (2C), 39.4, 24.7 (2C), 23.8 (2C), 23.8 (2C) ppm. HRMS (ESI) m/z: [(M+K)$^+$] Calcd for C$_{24}$H$_{29}$KNO$_5$S$^+$ 482.1398; Found 482.1406.

2-Cyano-2-isobutyl-4-methylpentyl (6-bromo-2',5'-dioxo-2',5'-dihydro-[1,1'-biphenyl]-3-yl)methanesulfonate (3ir): GP-3 was carried out by using arylmethanesulfonate ester 1i (83 mg, 0.2 mmol), cyclicolefin 2r (32 mg, 0.3 mmol), Pd(OAc)$_2$ (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 3ir (63 mg, 61%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, $R_f$(1i) = 0.60, $R_f$(3ir) = 0.30, UV detection]. IR (MIR-ATR, 4000–600 cm$^{-1}$): 2957, 2927, 1660, 1463, 1358, 1174, 981, 828, 754 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.71 (d, $J$ = 8.3 Hz, 1H), 7.40 (dd, $J$ = 8.3, 2.2 Hz, 1H), 7.32 (d, $J$ = 2.2 Hz, 1H), 6.88 (d, $J$ = 2.3 Hz, 2H), 6.81 (d, $J$ = 1.6 Hz, 1H), 4.43 (s, 2H), 4.05 (s, 2H), 1.77 (dd, $J$ = 13.0, 6.5 Hz, 2H), 1.48 (dd, $J$ = 6.2, 1.9 Hz, 4H), 1.0 (d, $J$ = 6.6 Hz, 6H), 0.98 (d, $J$ = 6.6 Hz, 6H) ppm. $^{13}$C {$^1$H} NMR (100 MHz, CDCl$_3$) $\delta$ 187.0, 184.8, 146.4, 136.7, 136.6, 135.3, 135.2, 133.7, 133.0, 132.7,
2-Cyano-2-isobutyl-4-methylpentyl (2',5'-dioxo-5-(trifluoromethyl)-2',5'-dihydro-[1,1'-biphenyl]-3-yl)methanesulfonate (3cr): GP-3 was carried out by using arylmethanesulfonate ester 1c (81 mg, 0.2 mmol), cyclicolefin 2r (32 mg, 0.3 mmol), Pd(OAc)$_2$ (5 mg, 10 mol%), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 3cr (61 mg, 60%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, $R_f(1c) = 0.60$, $R_f(3cr) = 0.30$, UV detection]. IR (MIR-ATR, 4000–600 cm$^{-1}$): 2959, 1659, 1601, 1358, 1170, 1130, 980, 754 cm$^{-1}$.$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.79 (s, 3H), 6.94 (d, $J = 2.1$ Hz, 1H), 6.93 – 6.85 (m, 2H), 4.56 (s, 2H), 4.13 (s, 2H), 1.78 (dt, $J = 13.0$, 6.5 Hz, 2H), 1.48 (dd, $J = 6.2$, 4.0 Hz, 4H), 0.99 (d, $J = 6.6$ Hz, 6H), 0.96 (d, $J = 6.6$ Hz, 6H) ppm. $^{13}$C ($^1$H) NMR (151 MHz, CDCl$_3$) $\delta$ 186.8, 185.6, 143.6, 136.9, 136.5, 134.9, 134.4, 133.8, 132.5 (q, $J_{C-F} = 32$ Hz), 129.0, 128.7 (q, $J_{C-F} = 4$ Hz), 126.8 (q, $J_{C-F} = 4$ Hz), 123.2 (q, $J_{C-F} = 271$ Hz), 71.4, 56.4, 43.1 (2C), 39.5, 24.7(2C), 23.8(2C), 23.8(2C) ppm. HRMS (ESI) m/z: [M+K]$^+$ Calcd for C$_{25}$H$_{29}$F$_3$KNO$_5$S$^+$ 550.1272; Found 550.1275.

2-Cyano-2-isobutyl-4-methylpentyl (3-(1,4-dioxo-1,4-dihyronaphthalen-2-yl)phenyl)methanesulfonate (3at): GP-3 was carried out by using arylmethanesulfonate ester 1a (81 mg, 0.2 mmol), cyclicolefin 2r (32 mg, 0.3 mmol), Pd(OAc)$_2$ (5 mg, 10 mol%), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 3at (61 mg, 60%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, $R_f(1a) = 0.60$, $R_f(3at) = 0.30$, UV detection]. IR (MIR-ATR, 4000–600 cm$^{-1}$): 2959, 1659, 1601, 1358, 1170, 1130, 980, 754 cm$^{-1}$.$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.79 (s, 3H), 6.94 (d, $J = 2.1$ Hz, 1H), 6.93 – 6.85 (m, 2H), 4.56 (s, 2H), 4.13 (s, 2H), 1.78 (dt, $J = 13.0$, 6.5 Hz, 2H), 1.48 (dd, $J = 6.2$, 4.0 Hz, 4H), 0.99 (d, $J = 6.6$ Hz, 6H), 0.96 (d, $J = 6.6$ Hz, 6H) ppm. $^{13}$C ($^1$H) NMR (151 MHz, CDCl$_3$) $\delta$ 186.8, 185.6, 143.6, 136.9, 136.5, 134.9, 134.4, 133.8, 132.5 (q, $J_{C-F} = 32$ Hz), 129.0, 128.7 (q, $J_{C-F} = 4$ Hz), 126.8 (q, $J_{C-F} = 4$ Hz), 123.2 (q, $J_{C-F} = 271$ Hz), 71.4, 56.4, 43.1 (2C), 39.5, 24.7(2C), 23.8(2C), 23.8(2C) ppm. HRMS (ESI) m/z: [M+K]$^+$ Calcd for C$_{25}$H$_{29}$F$_3$KNO$_5$S$^+$ 550.1272; Found 550.1275.
ester 1a (67 mg, 0.2 mmol), cyclicolefin 2t (47 mg, 0.3 mmol), Pd(OAc)$_2$ (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol %), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 3at (55 mg, 56%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, $R_f$(1a) = 0.60, $R_f$(3at) = 0.30, UV detection]. IR (MIR-ATR, 4000–600 cm$^{-1}$): 2958, 1661, 1594, 1356, 1255, 980, 821 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.17 (dd, $J = 6.0$, 3.0 Hz, 1H), 8.12 (dd, $J = 6.1$, 2.9 Hz, 1H), 7.82 – 7.75 (m, 2H), 7.68 (d, $J = 1.5$ Hz, 1H), 7.64 – 7.50 (m, 3H), 7.16 – 7.01 (m, 3H), 4.52 (s, 2H), 4.08 (s, 2H), 2.26 (s, 3H), 1.78 (dt, $J = 13.0$, 6.5 Hz, 2H), 1.48 (d, $J = 6.2$ Hz, 4H), 0.97 0.99 (d, $J = 6.6$ Hz, 6H), 0.96 (d, $J = 6.6$ Hz, 6H) ppm. $^{13}$C {$^1$H} NMR (100 MHz, CDCl$_3$) $\delta$ 184.8, 184.0, 147.0, 136.4, 135.6, 134.2, 134.0, 134.0, 132.2, 132.0, 131.9, 130.1, 129.5, 129.21, 127.6, 127.0, 126.0, 125.7, 121.3, 71.4, 57.0, 43.1 (2C), 39.4, 24.7 (2C), 23.9 (2C), 23.8 (2C), 19.68 ppm. HRMS (ESI) m/z: [(M+H)$^+$] Calcd for C$_{28}$H$_{32}$NO$_5$S$^+$ 494.1996; Found 494.1999.

2-Cyano-2-isobutyl-4-methylpentyl (3-(1-ethyl-2,5-dioxo-2,5-dihydro-1H-pyrrolo-3-yl)phenyl)methanesulfonate (3au): GP-3 was carried out by using arylmethanesulfonate ester 1a (67 mg, 0.2 mmol), cyclicolefin 2u (37 mg, 0.3 mmol), Pd(OAc)$_2$ (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol %), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 3au (65 mg, 71%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, $R_f$(1a) = 0.60, $R_f$(3au) = 0.20, UV detection]. IR (MIR-ATR, 4000–600 cm$^{-1}$): 2957, 1703, 1354, 1173, 982, 818 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.03 (d, $J = 1.5$ Hz, 1H), 7.95 (dt, $J = 7.6$, 1.5 Hz, 1H), 7.57 (dt, $J = 7.7$, 1.4 Hz, 1H), 7.52 (t, $J = 7.7$ Hz, 1H), 6.79 (s, 1H), 4.50 (s, 2H), 4.06 (s, 2H), 3.64 (q, $J = 7.2$ Hz, 2H), 1.77 (dt, $J = 13.0$, 6.5 Hz, 2H), 1.52 – 1.43 (m, 4H), 1.23 (t, $J = 7.2$ Hz, 3H), 0.99 (d, $J = 6.6$ Hz, 6H), 0.96 (d, $J = 6.6$ Hz, 6H).
Hz, 6H) ppm. 13C {1H} NMR (100 MHz, CDCl3) δ 170.2, 169.9, 142.6, 133.1, 130.9, 129.7, 129.6, 129.3, 128.3, 125.0, 121.3, 71.3, 56.8, 43.1 (2C), 39.4, 33.0, 24.8 (2C), 23.9 (2C), 23.8 (2C), 13.9 ppm. HRMS (ESI) m/z: [(M+Na)]+ Calcd for C24H32N2NaO5S+ 483.1924; Found 483.1935.

2-Cyano-2-isobutyl-4-methylpentyl (3-(1-ethyl-2,5-dioxo-2,5-dihydro-1H-pyrrol-3-yl)-5-(trifluoromethyl)phenyl)methanesulfonate (3cu): GP-3 was carried out by using arylmethanesulfonate ester 1c (81 mg, 0.2 mmol), cyclicolefin 2u (37 mg, 0.3 mmol), Pd(OAc)2 (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 3cu (71 mg, 67%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, Rf(1c) = 0.60, Rf(3cu) = 0.20, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): 2925, 1703, 1354, 1171, 1130, 978, 821 cm⁻¹. 1H NMR (400 MHz, CDCl3) δ 8.22 (s, 2H), 7.80 (s, 1H), 6.90 (s, 1H), 4.56 (s, 2H), 4.14 (s, 2H), 3.65 (q, J = 7.2 Hz, 2H), 1.78 (dp, J = 13.0, 6.5 Hz, 2H), 1.53 – 1.43 (m, 4H), 1.24 (t, J = 7.2 Hz, 3H), 0.99 (d, J = 6.6 Hz, 6H), 0.97 (d, J = 6.6 Hz, 6H) ppm. 13C {1H} NMR (151 MHz, CDCl3) δ 169.8, 169.3, 141.2, 134.0, 132.3 (q, J_{C-F} = 36 Hz), 130.5, 129.5, 129.3 (q, J_{C-F} = 4 Hz), 126.4, 126.1 (q, J_{C-F} = 4 Hz), 123.1 (q, J_{C-F} = 270 Hz), 121.2, 71.4, 56.3, 43.0 (2C), 39.5, 33.2, 24.7 (2C), 23.8 (2C), 23.8 (2C), 13.8 ppm. HRMS (ESI) m/z: [(M+NH4)]+ Calcd for C25H35F3N3O5S+ 546.2244; Found 546.2258.

2-Cyano-2-isobutyl-4-methylpentyl (3-(2,5-dioxo-1-phenyl-2,5-dihydro-1H-pyrrol-3-yl)phenyl)methanesulfonate (3av): GP-3 was carried out by using arylmethanesulfonate
ester 1a (67 mg, 0.2 mmol), cyclicolefin 2v (52 mg, 0.3 mmol), Pd(OAc)$_2$ (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 3av (66 mg, 65%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, R$f$(1a) = 0.60, R$f$(3av) = 0.20, UV detection]. IR (MIR-ATR, 4000–600 cm$^{-1}$): 2957, 1713, 1494, 1367, 1170, 978, 816, 736 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) δ 8.09 (s, 1H), 8.01 (d, $J$ = 7.6 Hz, 1H), 7.58 (dt, $J$ = 15.4, 7.7 Hz, 2H), 7.50 (dd, $J$ = 10.8, 4.7 Hz, 2H), 7.44 – 7.33 (m, 3H), 6.96 (s, 1H), 4.52 (s, 2H), 4.08 (s, 2H), 1.78 (dp, $J$ = 13.0, 6.5 Hz, 2H), 1.53 – 1.44 (m, 4H), 0.99 (d, $J$ = 6.6 Hz, 6H), 0.96 (d, $J$ = 6.6 Hz, 6H) ppm. $^{13}$C {$^1$H} NMR (151 MHz, CDCl$_3$) δ 169.2, 168.8, 142.6, 133.4, 131.3, 129.7, 129.5, 129.4, 129.1, 128.4, 128.0, 126.2, 125.1, 121.3, 71.3, 56.8, 43.1 (2C), 39.4, 24.7 (2C), 23.8 (2C) ppm. HRMS (ESI) m/z: [(M+K)$^+$] Calcd for C$_{28}$H$_{32}$KN$_2$O$_5$S$^+$ 547.1664; Found 547.1677.

2-Cyano-2-isobutyl-4-methylpentyl (3-(1-benzyl-2,5-dioxo-2,5-dihydro-1H-pyrrol-3-yl)phenyl)methanesulfonate (3aw): GP-3 was carried out by using arylmethanesulfonate ester 1a (67 mg, 0.2 mmol), cyclicolefin 2w (56 mg, 0.3 mmol), Pd(OAc)$_2$ (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (100 mg, 0.6 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 90 °C for 25 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 3aw (62 mg, 60%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, R$f$(1a) = 0.60, R$f$(3aw) = 0.20, UV detection]. IR (MIR-ATR, 4000–600 cm$^{-1}$): 2958, 1710, 1355, 1173, 982, 703 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) δ 8.02 (d, $J$ = 1.5 Hz, 1H), 7.94 (dt, $J$ = 7.7, 1.4 Hz, 1H), 7.59 – 7.55 (m, 1H), 7.51 (t, $J$ = 7.7 Hz, 1H), 7.39 (dd, $J$ = 8.1, 1.5 Hz, 2H), 7.32 (ddd, $J$ = 9.6, 4.4, 2.8 Hz, 3H), 6.82 (s, 1H), 4.74 (s, 2H), 4.49 (s, 2H), 4.06 (s, 2H), 1.77 (dd, $J$ = 13.1, 6.5 Hz, 2H), 1.46 (dd, $J$ = 6.2, 1.9 Hz, 4H), 0.97 (d, $J$ = 6.6 Hz, 6H), 0.95 (d, $J$ = 6.6 Hz, 6H) ppm. $^{13}$C {$^1$H} NMR (151 MHz, CDCl$_3$) δ 170.1, 169.6, 142.7, 136.2, 133.2, 130.9, 129.7, 129.6, 129.3, 128.7 (2C), 128.5 (2C), 128.3, 127.9, 125.0,
Dimethyl 3,3'-(((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methyl)-1,3-phenylene)(2E,2'E)-diacrylate (4a): GP-4 was carried out by using arylmethanesulfonate ester 1a (67 mg, 0.2 mmol), olefin 2b (21 mg, 0.48 mmol), Pd(OAc)$_2$ (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (133 mg, 0.8 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 100 °C for 35 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 4a (67 mg, 67%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, $R_f$(1a) = 0.60, $R_f$(4a) = 0.20, UV detection). IR (MIR-ATR, 4000–600 cm$^{-1}$): 2954, 1713, 1639, 1445, 1276, 981, 821 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.72 – 7.58 (m, 5H), 6.51 (d, $J$ = 16.1 Hz, 2H), 4.48 (s, 2H), 4.07 (s, 2H), 3.81 (s, 6H), 1.75 (dd, $J$ = 13.0, 6.5 Hz, 2H), 1.45 (dt, $J$ = 13.5, 6.7 Hz, 4H), 0.99 (d, $J$ = 6.6 Hz, 6H), 0.96 (d, $J$ = 6.6 Hz, 6H) ppm. $^{13}$C {$^1$H} NMR (151 MHz, CDCl$_3$) $\delta$ 166.7 (2C), 142.6 (2C), 136.0 (2C), 131.3 (2C), 129.1, 128.1, 121.2, 120.1 (2C), 71.3, 56.6, 51.9, 43.1 (2C), 39.5, 24.7 (2C), 23.8 (2C), 23.8 (2C) ppm. HRMS (ESI) m/z: [(M+H)$^+$] Calcd for C$_{26}$H$_{36}$NO$_7$S$^+$ 506.2207; Found 506.2218.

Tetraethyl 2,2'-(((((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methyl)-1,3-phenylene)dimaleate (4b): GP-4 was carried out by using arylmethanesulfonate ester 1a (67 mg, 0.2 mmol), olefin 2h (83 mg, 0.48 mmol), Pd(OAc)$_2$ (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (133 mg, 0.8 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 100 °C for 35 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 75/25), furnished the product 4b (81 mg, 60%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 85:15,
$R_f(1a) = 0.70$, $R_f(4b) = 0.20$, UV detection. IR (MIR-ATR, 4000–600 cm$^{-1}$): 2964, 1722, 1361, 1173, 981, 753 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) δ 7.62 (s, 3H), 6.33 (s, 2H), 4.46 (s, 2H), 4.41 (q, $J = 7.2$ Hz, 4H), 4.25 (d, $J = 7.1$ Hz, 4H), 4.12 (s, 2H), 1.81 – 1.77 (m, 2H), 1.48 (dd, $J = 6.2$, 3.9 Hz, 4H), 1.37 (t, $J = 7.2$ Hz, 6H), 1.33 (d, $J = 7.1$ Hz, 6H), 1.0 (d, $J = 6.6$ Hz, 6H), 0.97 (d, $J = 6.6$ Hz, 6H)) ppm. $^{13}$C {$^1$H} NMR (100 MHz, CDCl$_3$) δ 167.0 (2C), 164.4 (2C), 146.4, 135.4, 130.3, 129.3, 125.9, 121.2, 120.2, 71.3, 62.2, 61.2, 56.5, 43.1, 39.5, 24.7 (2C), 23.9 (2C), 23.8 (2C), 14.1 (2C), 13.9 (2C) ppm. HRMS (ESI) m/z: [(M+K)$^+$] Calcd for C$_{34}$H$_{47}$KNO$_7$S$^+$ 716.2501; Found 716.2508.

Diethyl 3,3'-(4-chloro-5-(((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methyl)-1,3-phenylene)(2E,2'E)-diacrylate (4c): GP-4 was carried out by using arylmethanesulphonate ester 1d (74 mg, 0.2 mmol), olefin 2a (48 mg, 0.48 mmol), Pd(OAc)$_2$ (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (133 mg, 0.8 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 100 °C for 35 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 4c (79 mg, 70%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, $R_f(1d) = 0.60$, $R_f(4c) = 0.20$, UV detection]. IR (MIR-ATR, 4000–600 cm$^{-1}$): 2961, 1710, 1638, 1362, 1268, 1173, 1041, 976, 734 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) δ 8.07 (d, $J = 16.0$ Hz, 1H), 7.75 (dd, $J = 16.7$, 2.0 Hz, 2H), 7.63 (d, $J = 16.0$ Hz, 1H), 6.48 (dd, $J = 21.0$, 16.0 Hz, 2H), 4.74 (s, 2H), 4.28 (dq, $J = 8.6$, 7.1 Hz, 4H), 4.16 (s, 2H), 1.79 (dt, $J = 13.0$, 6.5 Hz, 2H), 1.48 (dd, $J = 6.2$, 3.0 Hz, 4H), 1.34 (td, $J = 7.1$, 5.6 Hz, 6H), 1.0 (d, $J = 6.6$ Hz, 6H), 0.96 (d, $J = 6.6$ Hz, 6H) ppm. $^{13}$C {$^1$H} NMR (151 MHz, CDCl$_3$) δ 166.1, 165.8, 141.4, 139.6, 136.5, 135.0, 133.8, 132.6, 127.8, 127.7, 123.0, 121.1, 121.0, 70.7, 60.9, 60.8, 54.0, 43.1 (2C), 39.4, 24.7 (2C), 23.8 (2C), 23.8 (2C), 14.2 (2C) ppm. HRMS (ESI) m/z: [(M+H)$^+$] Calcd for C$_{28}$H$_{39}$ClNO$_7$S$^+$ 568.2130; Found 568.2134.
Diethyl 3,3’-(2-chloro-5-(((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methyl)-1,3-phenylene)(2E,2’E)-diacrylate (4d): GP-4 was carried out by using arylmethanesulfonate ester 1f (74 mg, 0.2 mmol), olefin 2a (48 mg, 0.48 mmol), Pd(OAc)$_2$ (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (133 mg, 0.8 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 100 °C for 35 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 4d (74 mg, 65%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, $R_f$ (1f) = 0.60, $R_f$ (4d) = 0.20, UV detection]. IR (MIR-ATR, 4000–600 cm$^{-1}$): 2929, 1716, 1637, 1366, 1263, 1176, 980, 823 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.10 (d, $J = 16.0$ Hz, 2H), 7.70 (s, 2H), 6.48 (d, $J = 16.0$ Hz, 2H), 4.46 (s, 2H), 4.13 (s, 2H), 1.77 (dd, $J = 13.0$, 6.5 Hz, 2H), 1.48 (t, $J = 6.5$ Hz, 4H), 1.35 (t, $J = 7.1$ Hz, 6H), 1.0 (d, $J = 6.6$ Hz, 6H), 0.97 (d, $J = 6.6$ Hz, 6H) ppm. $^{13}$C ($^1$H) NMR (100 MHz, CDCl$_3$) $\delta$ 165.9 (2C), 139.3 (2C), 135.8, 135.0, 130.7 (2C), 126.6, 123.0 (2C), 121.3, 71.4, 60.9 (2C), 56.2, 43.1 (2C), 39.5, 24.8 (2C), 23.8 (4C), 14.2, 14.1. HRMS (ESI) m/z: [(M+K)$^+$ Calcd for C$_{28}$H$_{38}$ClNO$_7$S$^-$ 606.1689; Found 606.1703.

Diethyl 3,3’-(4-bromo-5-(((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methyl)-1,3-phenylene)(2E,2’E)-diacrylate (4e): GP-4 was carried out by using arylmethanesulfonate ester 1g (83 mg, 0.2 mmol), olefin 2a (48 mg, 0.48 mmol), Pd(OAc)$_2$ (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (133 mg, 0.8 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 100 °C for 35 minutes. Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 4e (84 mg, 69%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, $R_f$ (1g) = 0.60, $R_f$ (4e) = 0.20, UV detection]. IR (MIR-ATR, 4000–600 cm$^{-1}$): 2960, 1710,
1637, 1267, 1033, 977 cm\(^{-1}\). \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.06 (d, \(J = 15.9\) Hz, 1H), 7.72 (d, \(J = 1.5\) Hz, 2H), 7.63 (d, \(J = 16.0\) Hz, 1H), 6.53 (d, \(J = 16.0\) Hz, 1H), 6.40 (d, \(J = 15.8\) Hz, 1H), 4.81 (s, 2H), 4.29 (dq, \(J = 9.1, 7.1\) Hz, 4H), 4.17 (s, 2H), 1.80 (dt, \(J = 13.0, 6.5\) Hz, 2H), 1.49 (dd, \(J = 6.2, 2.3\) Hz, 4H), 1.35 (td, \(J = 7.1, 6.0\) Hz, 6H), 1.01 (d, \(J = 6.6\) Hz, 6H), 0.97 (d, \(J = 6.6\) Hz, 6H) ppm. \(^{13}\)C \{\(^1\)H\} NMR (151 MHz, CDCl\(_3\)) \(\delta\) 166.2, 165.8, 142.6, 141.4, 137.3, 134.5, 132.4, 129.7, 129.2, 127.8, 123.2, 121.2, 70.8, 61.0, 60.9, 56.7, 43.2 (2C), 39.4, 36.6, 24.8 (2C), 23.9 (2C), 23.8 (2C), 14.3 (2C) ppm. HRMS (ESI) m/z: [(M+K)]\(^+\) Calcd for C\(_{28}\)H\(_{38}\)BrKNO\(_7\)S\(^+\) 650.1184; Found 650.1200: Calcd for C\(_{23}\)H\(_{33}\)BrKNO\(_7\)S\(^+\) 652.1163; Found 652.1181.

**Ethyl (E)-3-(3-(((2-cyano-2-isobutyl-4-methylpentyl)oxy)sulfonyl)methyl)-5-((E)-3-methoxy-3-oxoprop-1-en-1-yl)phenyl)acrylate (4f):**

An oven-dried 10 mL glass vial equipped with a magnetic stirring bar, was charged with arylmethanesulfonate ester 3ab (84 mg, 0.2 mmol), olefin 2a (48 mg, 0.48 mmol), Pd(OAc)\(_2\) (5 mg, 10 mol %), Ac-Gly-OH (10 mg, 40 mol%), AgOAc (133 mg, 0.8 mmol), and hexafluoroisopropanol (HFIP) (1.5 mL) under microwave irradiation at 100 °C for 35 minutes. The reaction mixture was cooled to room temperature. The progress of the reaction was monitored by TLC. The reaction mixture diluted with ethyl acetate (15 mL) and filtered through a short pad of celite with an additional amount of ethyl acetate (15 mL). Purification of crude product using silica gel column chromatography (hexane/ethyl acetate, 100/0 to 90/15), furnished the product 4f (74 mg, 71%), as a colourless liquid. [TLC (petroleum ether/ethyl acetate 90:10, \(R(f(3ab)) = 0.60, R(f(4f)) = 0.20,\) UV detection]. IR (MIR-ATR, 4000–600 cm\(^{-1}\)): 2959, 2873, 2235, 1576, 1363, 898, 749 cm\(^{-1}\). \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.73 – 7.63 (m, 3H), 7.61 (s, 2H), 6.51 (dd, \(J = 16.1, 2.2\) Hz, 2H), 4.48 (s, 2H), 4.27 (q, \(J = 7.1\) Hz, 2H), 4.08 (s, 2H), 3.82 (s, 3H), 1.77 (dt, \(J = 13.0, 6.5\) Hz, 2H), 1.50 – 1.42 (m, 4H), 1.34 (t, \(J = 7.1\) Hz, 3H), 0.99 (d, \(J = 6.6\) Hz, 6H), 0.96 (d, \(J = 6.6\) Hz, 6H) ppm. \(^{13}\)C \{\(^1\)H\} NMR (100 MHz, CDCl\(_3\)) \(\delta\) 166.7, 166.3, 142.7, 142.3, 136.1, 136.0, 131.3, 131.3, 129.0, 128.1, 121.2, 120.6, 120.0, 71.2, 60.7, 56.6, 51.9, 43.1 (2C), 39.5, 24.7 (2C), 23.8 (2C), 14.2 ppm. HRMS (ESI) m/z: [(M+H)]\(^+\) Calcd for C\(_{28}\)H\(_{38}\)NO\(_7\)S\(^+\) 520.2363; Found 520.2355.
Potassium (E)-3-(3-(sulfonatomethyl)phenyl)acrylate (5): To an oven dried 25 mL round bottom flask charged with a magnetic stirring bar, were added substrate 3ab (84 mg, 0.2 mmol) and MeOH (5 mL) followed by potassium hydroxide (2 equiv) at room temperature for 3 h. The reaction progress was monitored by TLC, then MeOH was removed under reduced pressure, resulted mixture was washed with ethyl acetate, the residue product 5 was obtained as white solid (60 mg, 95%). $^1$H NMR (400 MHz, DMSO) $\delta$ 7.35 (s, 1H), 7.28 (d, $J$ = 6.7 Hz, 1H), 7.19 (q, $J$ = 7.3 Hz, 2H), 7.02 (d, $J$ = 15.9 Hz, 1H), 6.31 (d, $J$ = 15.9 Hz, 1H), 3.69 (s, 2H). HRMS (ESI) m/z: [(M+H)]$^+$ Calcd for C$_{10}$H$_8$K$_2$O$_5$S$^+$ 318.9439; Found 318.9434. $^{13}$C \{$^1$H\} NMR (101 MHz, DMSO) $\delta$ 170.63, 136.54, 136.14, 135.75, 130.71, 130.35, 129.24, 128.25, 125.30, 57.83, 39.90.
$^1$H NMR (400 MHz) spectrum of 1a in CDCl$_3$

$^{13}$C $^1$H NMR (100 MHz) spectrum of 1a in CDCl$_3$
$^1$H NMR (400 MHz) spectrum of 1b in CDCl$_3$

$^{13}$C $^1$H NMR (151 MHz) spectrum of 1b in CDCl$_3$
$^1$H NMR (400 MHz) spectrum of 1c in CDCl$_3$

$^{13}$C {${^1}$H} NMR (100 MHz) spectrum of 1c in CDCl$_3$
H NMR (400 MHz) spectrum of 1d in CDCl₃

13C {¹H} NMR (100 MHz) spectrum of 1d in CDCl₃
\(^1\)H NMR (600 MHz) spectrum of 1e in CDCl\(_3\)

\(^{13}\)C \(^1\)H\) NMR (100 MHz) spectrum of 1e in CDCl\(_3\)
$^1$H NMR (400 MHz) spectrum of If in CDCl$_3$.

$^{13}$C $^1$H NMR (100 MHz) spectrum of If in CDCl$_3$. 
$^1$H NMR (400 MHz) spectrum of $1g$ in CDCl$_3$

$^{13}$C ($^1$H) NMR (151 MHz) spectrum of $1g$ in CDCl$_3$
$^1$H NMR (400 MHz) spectrum of $1h$ in CDCl$_3$

$^{13}$C {$^1$H} NMR (151 MHz) spectrum of $1h$ in CDCl$_3$
$^1$H NMR (400 MHz) spectrum of $1i$ in CDCl$_3$

$^{13}$C {$^1$H} NMR (100 MHz) spectrum of $1i$ in CDCl$_3$
$^1$H NMR (400 MHz) spectrum of $Ij$ in CDCl$_3$

$^{13}$C $^1$H NMR (151 MHz) spectrum of $Ij$ in CDCl$_3$
$^1$H NMR (400 MHz) spectrum of $^1k$ in CDCl$_3$  

$^{13}$C $^1$H NMR (151 MHz) spectrum of $^1k$ in CDCl$_3$
$^1$H NMR (600 MHz) spectrum of II in CDCl$_3$

$^{13}$C {$^1$H} NMR (100 MHz) spectrum of II in CDCl$_3$
$^1$H NMR (400 MHz) spectrum of $1m$ in CDCl$_3$

$^{13}$C ($^1$H) NMR (100 MHz) spectrum of $1m$ in CDCl$_3$
$^1$H NMR (400 MHz) spectrum of 3ab in CDCl$_3$

$^{13}$C {$^1$H} NMR (151 MHz) spectrum of 3ab in CDCl$_3$
$^1$H NMR (400 MHz) spectrum of 3ba in CDCl$_3$

$^{13}$C $^1$H NMR (100 MHz) spectrum of 3ba in CDCl$_3$
$^1$H NMR (400 MHz) spectrum of 3ca in CDCl$_3$

$^{13}$C {$^1$H} NMR (151 MHz) spectrum of 3ca in CDCl$_3$
$^1$H NMR (400 MHz) spectrum of 3ma in CDCl$_3$

$^{13}$C $^1$H NMR (100 MHz) spectrum of 3ma in CDCl$_3$
$^1$H NMR (400 MHz) spectrum of mixture of 3da and 3da'$^+$ in CDCl$_3$

$^1$H NMR (400 MHz) spectrum of 3da in CDCl$_3$
$^1$H NMR (400 MHz) spectrum of 3da’ in CDCl$_3$

$^{13}$C {$^1$H} NMR (100 MHz) spectrum of 3da’ in CDCl$_3$
$^1$H NMR (400 MHz) spectrum of mixture of 3na and 3na' in CDCl$_3$

$^{13}$C NMR (100 MHz) spectrum of mixture of 3na and 3na' in CDCl$_3$
$^1$H NMR (400 MHz) spectrum of mixture of 3ga and 3ga’ in CDCl$_3$.

$^{13}$C NMR (100 MHz) spectrum of mixture of 3ga and 3ga’ in CDCl$_3$.
$^1$H NMR (400 MHz) spectrum of 3ea in CDCl$_3$

$^{13}$C {$^1$H} NMR (100 MHz) spectrum of 3ea in CDCl$_3$
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\textsuperscript{13}C \{\textsuperscript{1}H\} NMR (151 MHz) spectrum of 3ad in CDCl$_3$
$^1$H NMR (400 MHz) spectrum of 3ae in CDCl$_3$

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$^{13}$C {$^1$H} NMR (100 MHz) spectrum of 3ag in CDCl$_3$. 
$^1$H NMR (400 MHz) spectrum of 3ah in CDCl$_3$

$^1$H NMR (400 MHz) spectrum vs NOE spectrum of 3ah in CDCl$_3$
\(^{13}\)C \(^{1}H\) NMR (151 MHz) spectrum of 3\textit{ah} in CDCl\textsubscript{3}

\(^{1}\)H NMR (400 MHz) spectrum of 3\textit{ai} in CDCl\textsubscript{3}
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\(^{13}\)C \(^{1}\)H NMR (151 MHz) spectrum of 4a in CDCl\(_3\)
$^1$H NMR (400 MHz) spectrum of 4b in CDCl$_3$

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$^{13}$C $^1$H NMR (151 MHz) spectrum of 4c in CDCl$_3$
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$^{13}$C $^1$H NMR (151 MHz) spectrum of $4e$ in CDCl$_3$
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$^{13}$C $\{^1$H$\}$ NMR (100 MHz) spectrum of $4f$ in CDCl$_3$
\(^1\)H NMR (400 MHz) spectrum of 5 in DMSO-\(d_6\)

\[^{13}\)C \(^1\)H NMR (100 MHz) spectrum of 5 in DMSO-\(d_6\)
X-ray Diffraction Analysis of Compound 3jb:
Crystal of compound 3jb was obtained by dissolving product in mixture of Acetonitrile and hexane in 3:1 ratio, allowing the solvent to slowly evaporate at room temperature. The crystal structure information for this compound has been deposited at the Cambridge Crystallographic Data Centre. **CCDC No: 2255976** contains the crystal structure information of this compound and can be obtained free of charge via [http://www.ccdc.cam.ac.uk](http://www.ccdc.cam.ac.uk)

![3jb](image)

**Figure S1.** X-ray structure of the product 3jb with the ellipsoids drawn at the 50% probability level.

**Table 1 Crystal data and structure refinement for mo_GS_DS_1_KM_340_3_0m.**

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Largest diff. peak/hole / e Å⁻³ 0.29/-0.28

References