Electronic Supplementary Information for:

**Morphology stabilization strategies for small-molecule bulk heterojunction photovoltaics**

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**Supplementary Figures and Tables Mentioned in the Main Text:**

![Figure S1. UV-Vis absorption of respective neat components dissolved in Chloroform at 0.2 mg/mL (Left) and after casting into thin films (Right).](image1)

**Figure S1.** UV-Vis absorption of respective neat components dissolved in Chloroform at 0.2 mg/mL (Left) and after casting into thin films (Right).

![Figure S2. DSC thermograms of DPP(TBFu)₂ (2nd Cycle), PC₆₁BM (2nd Cycle), CP (1st & 2nd Cycle), and N₃-ISL (1st & 2nd Cycle).](image2)

**Figure S2.** DSC thermograms of DPP(TBFu)₂ (2nd Cycle), PC₆₁BM (2nd Cycle), CP (1st & 2nd Cycle), and N₃-ISL (1st & 2nd Cycle).
Figure S3. a) FTIR Spectrum of N₃-ISL at various UV-Exposure Time. The presence of azides on N₃-ISL are easily observable from its characteristic peak at 2090 cm⁻¹. As a reference, no azide peak was visible on DPP(TBFu)₂. The results clearly show that as the UV-exposure time is increased, the intensity of the azide peak gradually decreases, indicating the conversion of the azide into the reactive nitrene for the photo-linking reaction.

b) Reference neat components without UV or thermal stimuli; c) Blends of DPP(TBFu)₂:PCBM (6:4) with 0wt%, 10wt% and 50wt% of N₃-ISL with the corresponding UV (254nm for 10mins) or thermal (110°C for 60mins) stimuli. Broad peaks were observed between 9.5 min to 10.5 min elution times after UV and thermal treatment of samples with N₃-ISL indicating evidence of higher molecular weight species (arrows indicate estimated average molecular weights of the respective peaks which is within the range of the corresponding the azide linked species and PCBM dimer (A₃) (See SI Fig S5.), whereas no obvious shoulder peak was observed for the blend samples without N₃-ISL. Qualitatively, it is clear that at 50wt% of N₃-ISL, a larger fraction of higher molecular weight peaks were observed as compared to 10wt% of N₃-ISL. Additionally, UV treatment resulted in a proportionally larger amount of higher molecular weight species than the thermal treatment. Interestingly, the PCBM peak significantly decreased upon UV and thermal stimuli, which may suggest that PCBM is more reactive towards the external stimuli as compared to DPP(TBFu)₂.

However, quantification of the extent of reaction necessitates information on the molar absorptivity of the produced species, which is not available. (See experimental procedures section for details on sample preparation and measurement conditions).
Figure S4. MALDI-TOF MS analysis. Panel (a) represents the control 6:4 blend of DPP(TBFu)₂ donor and PCBM acceptor before (bottom) and after (top) 10 min of UV treatment showing the presence of the dimerized PCBM species (A3, see Figure S5). Panel (b-c) is from the control blend before UV (b) and thermal (c) treatment with 50wt% of the N₃-ISL loading and after UV exposure (b) and thermal treatment at 110°C for 1 hour with 10wt% and 50wt% of N₃-ISL (Insets are zoomed-in to the m/z range to indicate the linked species (D₁-D₄ and A₁-A₃) present in the blend. Asterisk (*) indicates the additional molecular weight of one matrix molecule or Na⁺. Peaks are labelled according to the species outlined in Figure S5. It is evident that PCBM dimer (A₃) is only formed upon UV exposure but not thermal treatment, whereas the azide linked species D₁-D₄ and A₁-A₂ are formed via both external stimuli. On a side note, A₁ is detected in the blend before the respective UV or thermal treatment, likely due to the nitrene insertion reaction occurring in the solution at 50°C in chloroform before film deposition.
Figure S5. Predicted azide-linked species present in the BHJ under either UV or thermal activation. Each species is labeled with a designator A0, A1,...,D0, D1 etc. which correspond to the peaks observed in Figure S4.
Table S1. Summary of the overall device data of the optimum (Opt), and after 50h of thermal stability test at 110°C in Argon with and without the respective additives

<table>
<thead>
<tr>
<th>Additive (%)</th>
<th>$V_{oc}$ (V)</th>
<th>$J_{sc}$ (mA cm$^{-2}$)</th>
<th>FF (%)</th>
<th>PCE (%)</th>
<th>Gain in PCE (%)</th>
<th>Loss in PCE (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Opt</td>
<td>After 50h</td>
<td>Max.</td>
<td>After 50h</td>
<td>Opt</td>
<td>After 50h</td>
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<tr>
<td>0</td>
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<td>0(UV10)</td>
<td>0.93</td>
<td>0.88</td>
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<td>6.06</td>
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<td>40.8</td>
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<td>CP</td>
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<td>43.5</td>
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<td></td>
<td>10</td>
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<td>4.23</td>
<td>7.46</td>
<td>36.7</td>
<td>45.3</td>
</tr>
<tr>
<td>N$_3$-ISL (UV10)</td>
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<td>3.54</td>
<td>36.0</td>
<td>37.5</td>
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<td>1.56</td>
<td>1.62</td>
<td>32.2</td>
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<tr>
<td>N$_3$-ISL (UV0)</td>
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<td>46.7</td>
<td>42.3</td>
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<td>6.10</td>
<td>5.43</td>
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<td>4.65</td>
<td>3.84</td>
<td>38.7</td>
<td>37.5</td>
</tr>
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</table>

*a Photovoltaics data were taken from an average of 6 batches of devices.

b The gain in PCE were calculated using the optimum PCE of the control device as reference.

c Loss in PCE was calculated from the optimum PCE of the individual device.

d Devices were exposed to 10 minutes of UV exposure (254nm) before aluminum deposition.
Figure S6. J-V curves of DPP(TBFu)$_2$:PC$_{61}$BM 6:4 with varying amount of the N3ISL linker (0, 1 and 10 wt%) under various UV exposure times (0, 10, and 30 min as the solid, dotted and dashed lines, respectively). Devices were thermally treated for 10 min at 110°C.

Table S2. Calculated endothermic enthalpy of melting of 6:4 - DPP(TBFu)$_2$:PC$_{61}$BM blend with varying additive loading

<table>
<thead>
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<th>Additive loading %</th>
<th>Enthalpy of melting (J/g)$^a$</th>
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<tbody>
<tr>
<td></td>
<td>CP</td>
</tr>
<tr>
<td>0</td>
<td>21.4</td>
</tr>
<tr>
<td>10</td>
<td>7.61</td>
</tr>
<tr>
<td>25</td>
<td>2.38</td>
</tr>
<tr>
<td>50</td>
<td>-</td>
</tr>
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</table>

$^a$enthalpy of melting was calculated based on the specific mass of DPP(TBFu)$_2$ in the blend of 6:4 - DPP(TBFu)$_2$:PC$_{61}$bm with varying loading of the additive from the DSC thermograms
Figure S7. Optical microscopy of the BHJ films after heating to 240 °C (above the melting temperature) for 5 min. The amount of the respective additive is indicated for each panel.

Figure S8. Photoluminescence emission spectra of the 6:4 blends of DPP(TBFu)2:PC61BM with the respective additives at As cast (top three panels) and after thermal treatment of 110°C for 1 h (bottoms three panels). The normalized integrated PL emission is summarized in Figure 4b of the main text.
General Experimental Procedures

Thin film preparation for morphological and electrochemical characterization: The photoactive layers of each respective material were prepared by first dissolving each stock solution separately for 24hrs in the respective solvents at 50°C in chloroform at a concentration of 20mg/ml. The blended solutions were then stirred for 2hrs, and then filtered with a 0.2µm pore diameter PTFE filter, and left to stir for another hour before deposition on glass/ITO/PEDOT:PSS substrates via spin coating at 3000rpm. Thermal annealing were performed under Argon atmosphere at 110°C at various time intervals, and were allowed to cool down to room temperature under argon atmosphere before conducting any morphological and electrochemical analysis. The film thickness were kept between 140nm-152nm as determined by a Dektak 150 profilometer.

Atomic force microscopy characterization: Surface topography of all blend films were studied by atomic force microscopy (AFM) using an Asylum Research Cypher in AC mode using Atomic Force AC240TS tips directly on measured transistors surface under ambient conditions.

Optical Microscope: Optical microscope images were obtained with a NIKON Eclipse E600.

UV-Vis and PL Characterization: Absorption spectra of the thin films were acquired with a UV–vis-NIR UV-3600 (Shimadzu) spectrophotometer, and the optical band gap ($E_g^{opt}$) was determined from the absorption edge of the thin film sample. Photoluminescence Spectra was carried out on a LabRAM HR Raman spectrometer at a laser excitation at 532 nm. An average of 15 measurements were conducted across the thin film to ensure accuracy of the data.

Thermal Characterisation: Differential scanning calorimeter (DSC) thermograms were measured with a PerkinElmer DSC8000 calibrated with indium and zinc, using a scanning rate of 10 °C/min. The respective Melting ($T_m$), and Crystallization ($T_c$) temperatures were characterized by their peak temperatures, and their endothermic and exothermic enthalpies were calculated from the surface area underneath both melting endotherms and crystallizing exotherms using the specific mass ratio in the stated blend composition of the primary components and the two additives. Samples were drop-casted evenly onto a SiO$_2$ substrate with a slow evaporation of the solvent (Chloroform) at 30 °C under argon atmosphere before transferring the powder into an aluminium pan. Samples with N$_3$-ISL were UV cured for 10 minutes after drying on the SiO$_2$ substrate.

Solar-Cell Fabrication and Testing: Solar cells were fabricated with a 35 nm layer of PEDOT:PSS (Ossilla M 121 Al 4083) deposited and annealed (110 °C) on a glass substrate patterned with 300 nm of ITO. The BHJ active layer was spin-cast at 3000 rpm from a solution of a blend of DPP(TBFu)$_2$:PC$_{61}$BM with the respective loading of the additive CP and N$_3$-ISL in chloroform at a total solid concentration of 20g/mL$^{-1}$. The active layers were determined to be ≈143nm thick using a Bruker Dektak XT profilometer. An 80 nm thick aluminium cathode was deposited (area 16 mm$^2$) by
thermal evaporation (Kurt J. Lesker Mini-SPECTROS). Electronic characterization was performed under simulated AM1.5G irradiation from a 300 W Xe arc lamp set to 100 mW cm$^{-2}$ with a calibrated Si photodiode (ThorLabs). Current–voltage curves were obtained with a Keithley 2400 source measure unit. Thermal stability measurements were performed with the device annealed under Argon atmosphere at 110°C at various time intervals, and were allowed to cool down to room temperature under argon atmosphere before measuring its photovoltaic performance. Impedance spectroscopy was carried out using a SP-200 potentiostat at frequencies ranging from 1 MHz up to 1 Hz. Each semicircle in the impedance response were fitted with a resistance in parallel to constant phase element (model shown in Figure S9). Device fabrication was performed under an argon atmosphere and testing was performed in a nitrogen filled glovebox.

**Photocrosslinking (UV) reaction:** Photo-crosslinking reaction were performed using a low intensity hand-held (8W) UV lamp, set to emit 254nm light, with the bulb positioned approximately 1cm from the substrates at the specified time intervals under argon conditions.

**FTIR:** The FTIR measurements were conducted on Nicolet 6700 from ThermoFisher Scientific. N$_3$-ISL was dissolved in chloroform at a concentration of 20mg/ml and left to stir at 50°C for 1h. Before drop casting 10µL on SiO$_2$ substrates under Argon atmosphere, and left to dry at room temperature before conducting the measurements at ambient conditions.

**GPC and Mass Spectrum analysis:** The GPC measurements were made on a Shimazu CT-20A with a detector Shimazu SPD-20A. Matrix-Assisted-Laser-Desorption/Ionization Time of Flight (MALDI-TOF) MS spectrum was recorded on a Bruker MALDI-TOF AutoFlex speed instrument using trans-2-[3-(4-tert-Butylphenyl)-2-methyl-2-propenylidene] malononitrile and 2,5-Dihydroxybenzoic acid as matrix using Tetrahydrofuran as a solvent. The samples were spin-coated onto SiO$_2$ substrates according to the solar cells thin film fabrication without deposition of PEDOT:PSS and the respective electrodes. The active layer were then treated with UV(254nm) or thermal annealing at 110°C at the respectiv e time intervals under Argon atmosphere, and subsequently dissolved in 1ml of THF to perform the respective GPC and MALDI-MS measurements.

**Synthetic Methods for CP and N$_3$-ISL**

All reagents were of commercial reagent grade (Sigma-Aldrich, Acros and Fluorochem) and were used without further purification. Toluene, Chloroform, Tetrahydrofuran (Fisher Chemical, HPLC grade) and chlorobenzene (Alfa Aesar, HPLC grade) were purified and dried on a Pure Solv-MD Solvent Purification System (Innovative Technology, Amesbury, United States) apparatus. Normal phase silica gel chromatography was performed with an Acros Organic silicon dioxide (pore size 60 Å, 40–50 µm technical grades). The (1H) and (13C) NMR spectra were recorded at room temperature using per-deuterated solvents as internal standards on a NMR Bruker Advance III-400 spectrometer (Bruker, Rheinstetten, Germany). Chemical shifts are given in parts per million (ppm) referenced to residual 1H or 13C signals in CDC$_2$(1H: 7.26, 13C: 77.16) and dichloromethane-d$_2$ (2H: 5.32, 13C:53.84). EI-MS spectrum was recorded on an EI/CI-1200L GC-MS (Varian) instrument. Atomatic-Pressure-Photoionization-Source(APPI) MS spectrum was recorded on an ESI/APCI LC-MS Autopurification System with a ZQ Mass detector (Waters, Milford, United States) instrument using a positive mode. Matrix-Assisted-Laser-Desorption/Ionization Time of Flight (MALDI-TOF) MS spectrum was recorded on a Bruker MALDI-TOF AutoFlex speed instrument using alpha-cyano-4-hydroxycinnamic acid, and 2,5-Dihydroxybenzoic acid as matrix. Final products synthesized used in
the active layers were purified using a Biotage Isolera™ Spektra Accelerated Chromatographic Isolation System™ with a Biotage ZIP® Sphere cartridges (60μm spherical silica) before device fabrication.

**Synthetic Route for CP and N3-ISL**

![Synthetic Route Diagram](image)

**Figure S10.** Overall synthetic route for CP and N3-ISL

**Synthetic Procedures**

DPP(TBFu)\(^2\) \(^{51}\) and [6,6]-phenyl-C61-butyric acid (PCBA) \(^{52}\) was synthesized according to literature procedure, while PC\(_{61}\)BM was purchased from Ossila. Synthesis of 2,5-Dihydro-3,6-di-2-thienylpyrrolo[3,4-c]pyrrole-1,4-dione (DPP(Th)\(^2\)) \(^{53}\) and subsequent mono-alkylation\(^{54}\) of the DPP(Th)\(^2\) with 3-(bromomethyl)heptane to obtain MEHDPP(Th)\(^2\) (1) and 1-azido-6-bromohexane (3) \(^{55}\) are based on literature procedures. 6-(Boc-amino)hexyl bromide (2) and 2-Benzofuranylboronic acid (6) was purchased from Sigma Aldrich.

**NBocEHDPP(Th)\(^2\) (4a) and N\(_3\)EHDPP(Th)\(^2\) (4b)**

MEHDPP(Th)\(^2\) (1) (0.492g, 1.19 mmol) and anhydrous potassium carbonate (0.329 g, 2.39 mmol) were stirred in anhydrous (DMF) (50 mL) under nitrogen at 130 °C for 1 h. 6-(Boc-amino)hexyl bromide (2) (0.401g, 1.43 mmol) was then added dropwise and the reaction mixture stirred at 130 °C for a further 20 h. The reaction mixture was allowed to cool to room temperature, 1N HCl(aq) was added and left to stir for 1hr before pouring into ice water (1 L). The suspension was then left to stir for 3 hours, and the precipitate was filtered and, dried under vacuum to give the crude product, and later separated via chromatography using an elution solvent of CHCl\(_3\):Hexane (2:1) to obtain NBocEHDPP (4a) (0.729 g, 96%) as a dark red solid; \(^1\)H NMR (400 MHz, Chloroform-d) δ 8.92 (dd, \(J = \)
PBocEHDPP(ThBr)₂ (5a) and N₂EHDPP(ThBr)₂ (5b)

N-bromosuccinimide (NBS) (0.230g, 1.29mmol) was added to a solution of NBocEHDPP(Th)₂(4a) 0.396g, 0.647mmol) in chloroform at 0°C in which the flask was wrapped in aluminium foil to exclude light. After 24 hrs, the reaction mixture was poured into Hexane and left to stir in an ice bath. The mixture was filtered and the solid was washed with methanol (2 × 200 mL) then dried under vacuum. The crude product was recrystallized from Hexane/chloroform to give NBocEHDPP(ThBr)₂ (5a) as a dark purple solid (0.320g, 64%). 1H NMR (400 MHz, Chloroform-d) δ 8.68 (dd, J = 23.0, 4.2 Hz, 2H), 7.31 (dd, J = 7.3, 4.2 Hz, 2), 4.62 (s, 1H), 4.02 (t, J = 7.8 Hz, 2H), 3.96 (t, J = 7.7 Hz, 2H), 3.11 (q, J = 6.6 Hz, 2H), 1.85 (d, J = 7.2 Hz, 1H), 1.74 (d, J = 7.5 Hz, 2H), 1.62 – 1.22 (m, 16H), 0.91 (td, J = 7.2, 2.3 Hz, 18H); δ c (101 MHz, CDCl₃) 161.42, 139.43, 135.49, 135.33, 131.73, 131.47, 131.95, 119.15, 119.11, 108.07, 107.73, 46.01, 42.09, 40.46, 39.12, 38.16, 31.25, 30.33, 30.17, 29.98, 29.95, 29.71, 28.44, 28.31, 26.51, 26.38, 23.53, 23.05, 22.99, 22.71, 14.14, 14.04, 10.48. MS (APPI): m/z [M+Na]⁺ = 792.094. For N₂EHDPP(ThBr)₂ (5b), the procedure is similar to 5a to obtain a yield of 75%. 1H NMR (400 MHz, Chloroform-d) δ 8.72 (d, J = 4.2 Hz, 1H), 8.66 (d, J = 4.2 Hz, 1H), 7.36 – 7.20 (m, 2H), 4.02 (q, J = 6.7, 5.7 Hz, 2H), 3.99 – 3.91 (m, 2H), 3.30 (t, J = 6.8 Hz, 2H), 1.85 (d, J = 6.0 Hz, 1H), 1.78 (t, J = 7.6 Hz, 2H), 1.64 (q, J = 6.9 Hz, 2H), 1.58 (s, 8H), 1.53 – 1.43 (m, 4H), 1.43 – 1.20 (m, 9H), 0.99 – 0.83 (m, 6H); MS (ESI-TOF): m/z [M⁺] = 696.05.

PBocEHDPP(TBFu)₂ (7a) and N₂-ISL (7b)

PBocEHDPP(ThBr)₂ (5a) (0.114g, 0.148mmol), Benzoferan-2-boronic acid (6) (0.059g, 0.370mmol), tris(dibenzylideneacetone)dipalladium(0)(0.1eq), and tri-tert-butylphosphonium tetrafluoroborate (0.4eq) was mixed with anhydrous toluene and 2.0 M potassium phosphate (aq) in a 50mL Schleck Tube and the resulting mixture was degassed for 10 min. The reaction mixture was then stirred and heated to 90°C under argon overnight. The reaction mixture was allowed to cool down to room temperature, after which it was poured into 300mL of methanol and then stirred for 30 min. The precipitated solid was then collected by vacuum filtration and washed with several portions of distilled water, methanol, isopropanol, and petroleum ether. The crude product was purified by flash chromatography using chloroform as eluent, and the solvent was removed in vacuo to obtain a Greenish-Gold solid of PBocEHDPP(TBFu)₂ (7a) (0.101g, 80%). 1H NMR (400 MHz, Chloroform-d) δ 9.04 (dd, J = 24.9, 4.2 Hz, 0H), 7.64 (q, J = 5.5 Hz, 1H), 7.56 (t, J = 7.9 Hz, 1H), 7.37 (t, J = 7.7 Hz, 1H), 7.29 (d, J = 1.6 Hz, 3H), 7.12 (d, J = 12.1 Hz, 0H), 4.57 (s, 0H), 4.19 (t, J = 7.9 Hz, 1H), 4.13 (t, J = 6.9 Hz, 0H), 3.16 (s, 1H), 1.98 (s, 0H), 1.86 (s, 1H), 1.51 (d, J = 48.2 Hz, 10H), 0.94 (dt, J = 13.2, 7.0 Hz, 2H); δ c (101 MHz, CDCl₃) 0.75, 10.28, 13.85, 23.10, 23.58, 26.37, 26.48, 28.12, 28.51, 29.68, 29.96, 30.28,
CP  
Firstly, NBocEHDPP(TBFu)₂ (7a) (175 mg, 0.17 mmol) was dissolved in dichloromethane (8mL), and 1.5 mL of TFA was added and stirred at room temperature. The reaction as measured using thin layer chromatography until all of the starting material has reacted, which is approximately 2 hours later. 8mL of saturated Na₂CO₃ aqueous and water were respectively used to quench the reaction. The Organic phase was then separated from the aqueous phase, collected and dried with Na₂SO₄. The organic solvent was then removed to afford NH₂EHDPP(TBFu)₂ as a dark Green Solid. This was then precipitated using a mixture of hexane:CH₂Cl₂, and used for the next step without any further purification. NH₂EHDPP(TBFu)₂ (27.0 mg, 36.29 μmol) was then dissolved in DMF (500 μl). A separate solution of PCBA (14.62mg, 16.3μmol) in CHCl₃ (900 μl), was stirred and sonicated at room temperature for 15 min, and then HATU (6.82 mg, 17.93 μmol) and DIEA (7.58mg, 10.22μl, 58.68 μmol) were sequentially added. After 10 min stirring at room temperature, this was added to the solution of NH₂EHDPP(TBFu)₂. The reaction as measured using thin layer chromatography until all of the starting material has reacted. At the end of the reaction, the resulting mixture was concentrated under reduced pressure. The residue was dissolved in CHCl₃ (2 ml), washed with HCl (5%) and NaHCO₃ (sat.), dried over Na₂SO₄, and concentrated under reduced pressure. The resulting crude was washed with methanol/hexane and purified by flash chromatography using CH₂Cl₂ as eluent giving a dark green solid of CP (56.6%, 15mg). ¹H NMR (400 MHz, Chloroform-d) δ 9.08 (d, J = 4.2 Hz, 1H), 8.96 (d, J = 4.2 Hz, 1H), 7.95 – 7.84 (m, 2H), 7.64 (dd, J = 9.5, 4.0 Hz, 4H), 7.56 (m, 2H), 7.51 (t, J = 7.4 Hz, 2H), 7.45 (br,1H), 7.37 (t, J = 7.7 Hz, 2H), 7.29 (br, 1H), 7.15 (s, 1H), 7.10 (s, 1H), 5.88 (s, 1H), 4.13 (dq, J = 16.2, 7.4 Hz, 4H), 3.33 (d, J = 6.1 Hz, 2H), 2.94 – 2.82 (m, 2H), 2.41 (t, J = 7.5 Hz, 2H), 2.30 – 2.15 (m, 2H). MS (MALDI-TOF): m/z [M]+ = 769.95.
Figure S11. $^1$H NMR of N$_3$-ISL

Figure S12. $^{13}$C NMR of N$_3$-ISL
Figure S13. MALDI-TOF Mass Spectra of N₃-ISL

Figure S14. ¹H NMR of CP.
Figure S15. $^{13}$C NMR of CP.

Figure S16. MALDI-TOF Mass Spectra of CP.
Supporting References


