Electronic Supplementary Information for Visible Lightinduced Alkylpyridylation of Styrenes via a Reductive Radical Three-component Coupling

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1. General Information

All chemical reagents are obtained from commercial suppliers and used without further purification. All unknown compounds are characterized by ¹H NMR, ¹³C NMR, MS, HRMS. Irradiation of visible light was performed with a 90W Kessil A360W Blue LED lamp. Analytical thin-layer chromatography are performed on glass plates precoated with silica gel impregnated with a fluorescent indicator (254 nm), and the plates are visualized by exposure to ultraviolet light.¹H NMR and ¹³C NMR spectra are recorded on an AVANCE 400 Bruker spectrometer operating at 400 MHz and 100 MHz in CDCl₃, respectively, and chemical shifts are reported in ppm. Multiplicities are indicated by s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), and br (broad). High-resolution mass spectra are taken on a Waters SYNAPT G2-Si instrument in the electrospray ionization (ESI) mode.

2. General Procedure

General procedure for visible light-induced alkylpyridylation of styrenes via a reductive radical three-component coupling: A 10 mL reaction vessel with a magnetic stirring bar was equipped with alkenes (0.2 mmol), 4-cyano pyridine (0.3 mmol), NHPI esters (0.3 mmol), HE (0.3 mmol) and MTBE (2 mL). The mixture was irradiated with a 90 W blue LED lamp (1 cm away with cooling from a fan), and stirred under Ar atmosphere at r.t. overnight. After the solvent had been removed under reduced pressure, the residue was purified by flash chromatography using PE-AcOEt (10:1-8:1, v/v) as the eluent to 1,1-diarylalkane derivatives.

3. NMR Spectra of All Products



¹H NMR Spectrum of Compound 1 (400MHz, CDCl₃)



¹³C NMR Spectrum of Compound 1 (100MHz, CDCl₃)





-10

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¹³C NMR Spectrum of Compound 4 (100MHz, CDCl₃)



¹³C NMR Spectrum of Compound 5 (100MHz, CDCl₃)





¹H NMR Spectrum of Compound 6 (400MHz, CDCl₃)







¹³C NMR Spectrum of Compound 7 (100MHz, CDCl₃)











¹H NMR Spectrum of Compound **10** (400MHz, CDCl₃)







¹³C NMR Spectrum of Compound **11** (100MHz, CDCl₃)











¹H NMR Spectrum of Compound **14** (400MHz, CDCl₃)











¹H NMR Spectrum of Compound **16** (400MHz, CDCl₃)



¹³C NMR Spectrum of Compound **16** (100MHz, CDCl₃)



¹H NMR Spectrum of Compound 17 (400MHz, CDCl₃)



¹H NMR Spectrum of Compound **18** (400MHz, CDCl₃)



¹⁹F NMR Spectrum of Compound **18** (377MHz, CDCl₃)



¹³C NMR Spectrum of Compound 18 (100MHz, CDCl₃)



¹H NMR Spectrum of Compound **19** (400MHz, CDCl₃)



¹⁹F NMR Spectrum of Compound **19** (377MHz, CDCl₃)













¹H NMR Spectrum of Compound **22** (400MHz, CDCl₃)



F80 7 - 5 5. 00 T 34 5.0 4.5 f1 (ppm) 4.0 5.5 2.0 8.0 7.5 7.0 6.5 6.0 3. 5 3.0 2.5 1.5 1.0 0.0 0. ō



9.0



¹³C NMR Spectrum of Compound **23** (100MHz, CDCl₃)



¹H NMR Spectrum of Compound 24 (400MHz, CDCl₃)



¹H NMR Spectrum of Compound **25** (400MHz, CDCl₃)







¹H NMR Spectrum of Compound **27** (400MHz, CDCl₃)



¹H NMR Spectrum of Compound **28** (400MHz, CDCl₃)







¹³C NMR Spectrum of Compound **29** (100MHz, CDCl₃)