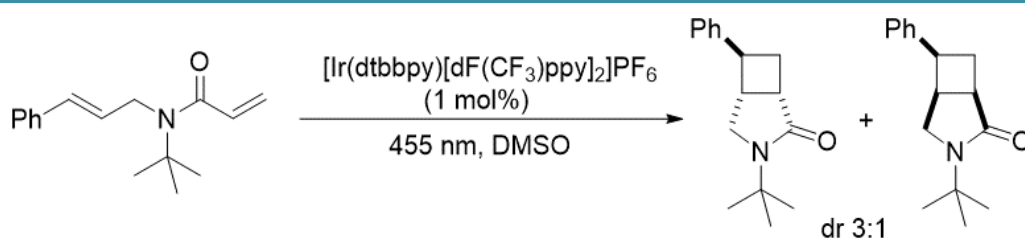


Visible Light-Mediated [2+2] Photocycloaddition of Amide-linked Dienes



Summary

Visible Light-Mediated [2+2] Photocycloaddition of Amide-linked Dienes to 3-azabicyclo[3.2.0]heptan-2-ones.

Equipment

Item #	Description
8053 000 100	XELSIUS Basic Unit, Software Version: 2.44
8053 000 201	Reflux Condenser Module
8053 000 202	HV Vials Starter Kit, 1 - 30 ml
8053 000 401	Photochemistry Research Kit

Chemicals

	Description
0,8 mmol	Styrene CAS: 100-42-N-(tert-Butyl)-N-cinnamylacrylamide
8,0 μmol	[Ir(dtbbpy)[dF(CF ₃)ppy] ₂]PF ₆ CAS: 870987-63-6
0,5 mmol	Tosylamid CAS: 70-55-3
20 ml	DMSO

Methode

Visible light mediated [2+2] cycloaddition at 40 °C under magnetic stirring (1300 rpm).

Add a solution of 0.8 mmol *N*-(*tert*-Butyl)-*N*-cinnamylacrylamide and 8.0 μmol [Ir(dtbbpy)[dF(CF₃)ppy]₂]PF₆ 20 ml DMSO to a 30 ml vessel. Dissolve in and degas by bubbling nitrogen through the solution for 10 min. Heat to 40 °C, and then switch on the light source (455 nm) for 3 – 4 h.

Pour the mixture into 20 ml water and extract with four 30 ml portions of *tert*-butylmethylether. Wash the combined organic phases with 10 ml water and brine, dry on MgSO₄, and evaporate all volatiles. Purify the yellow residue on silica gel column chromatography using a mixture of EtOAc-hexanes (1:3) as eluent to afford both diastereomers.

References:

S. Kerres, E. Plut, S. Malcherek, J. Rehbein, O. Reiser Visible Light-Mediated Synthesis of Enantiopure γ -Cyclobutane Amino and 3-(Aminomethyl)-5-phenylpentanoic Acids. *Adv. Synth. Catal.* 2019, 361, 1400-1407.

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