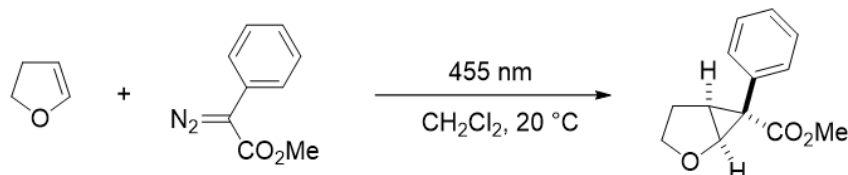


Visible Light-promoted Metal-free Cyclopropanation

Summary



Visible light promoted cyclopropanation of dihydrofuranes with Aryldiazoacetates.

Upscaling:

4.5 mmol methyl 2 phenyl 2 diazoacetate, 13.5 mmol 2,3-dihydrofuran, 20 ml dichloromethane:
 Reaction time 16 h with "normal" LED (Oslo SSL80@700 mA), 5.5 h with Cree XPG3@2000 mA (approx. 2.75-fold light intensity)

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 |
|----------|---|
| 3,0 mmol | 2,3-Dihydrofuran CAS 1191-99-7 |
| 1,0 mmol | Methyl diazophenylacetate CAS 22979-35-71 |
| 10 ml | Dichlormethane |

Methode

Visible light induced cyclopropanation at 20 °C under magnetic stirring (1300 rpm).

Add 3.0 mmol 2,3-dihydrofuran, 10 ml dichloromethan and 1.0 mmol methyl 2 phenyl 2 diazoacetate to a 30 ml vessel. Degas by bubbling nitrogen through the solution for 5 min and then switch on the light source (455 nm). After 4 h the yellow color of the diazoester has faded.

Evaporate all volatiles and purify the residue on silica gel column chromatography using a mixture of EtOAc-hexanes (1:9) as eluent to afford the pure product.

References:

S. Budde, F. Goerdeler, J. Floß, P. Kreitmeier, E.F. Hicks, O. Moscovitz, P. H. Seeberger, H. M. L Davies, O. Reiser Visible-light mediated oxidative ring expansion of annellated cyclopropanes to fused endoperoxides with antimalarial activity. *Org. Chem. Front.* 2020, 7, 1789-1795, DOI: 10.1039/DOQ000168F.
 I.D. Jurberg, H.M.L. Davies Blue light-promoted photolysis of aryldiazoacetates. *Chem. Sci.*, 2018, 9, 5112-5118, DOI: 10.1039/c8sc01165f

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