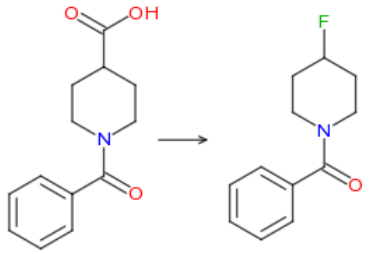
Application Note: DrySyn Illumin8

Parallel Photoreactor

DrySyn Illumin8: 450nm blue LED's

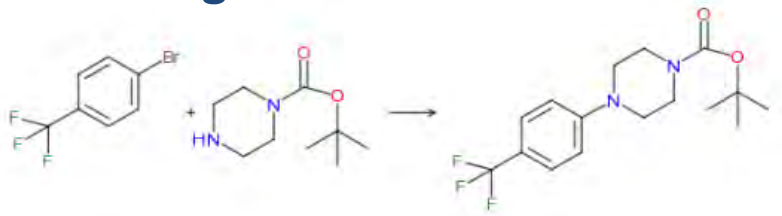
These reactions focus on % yield

Decarboxylative Fluorination



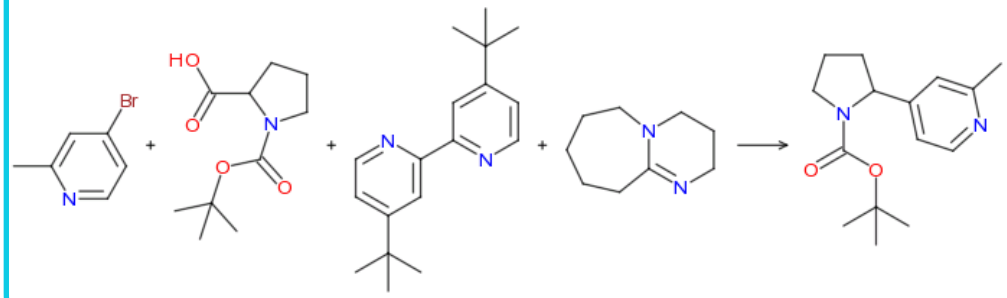
DrySyn Illumin8 = 74% yield

Aryl Amination - 1st generation Buchwald



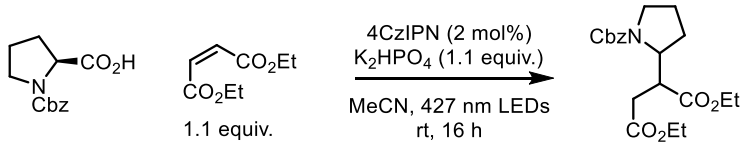
DrySyn Illumin8 = 65% yield

Decarboxylative coupling



DrySyn Illumin8 = 52% yield

Decarboxylative Radical Additions

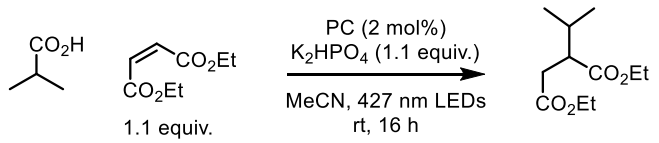


DrySyn Illumin8: 90% yield

DrySyn Illumin8: 450nm blue LED's

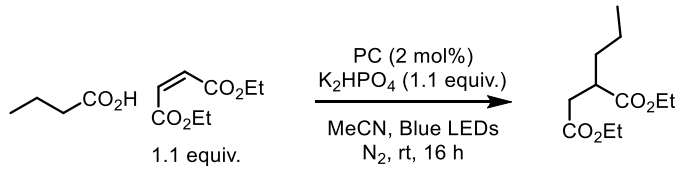
Focus on % of starting material decarboxylated

Decarboxylative Radical Additions



DrySyn Illumin8 = 98% decarboxylated

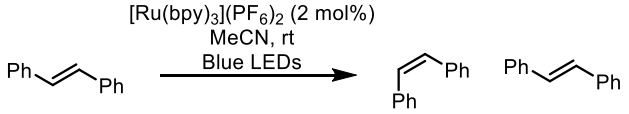
Decarboxylative Radical Additions



DrySyn Illumin8 = 70 - 85% decarboxylated

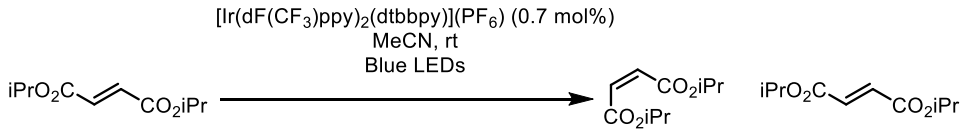
Focus on selectivity

Stilbene Isomerization



DrySyn Illumin8 = 94:6 cis:trans

Fumarate Isomerization

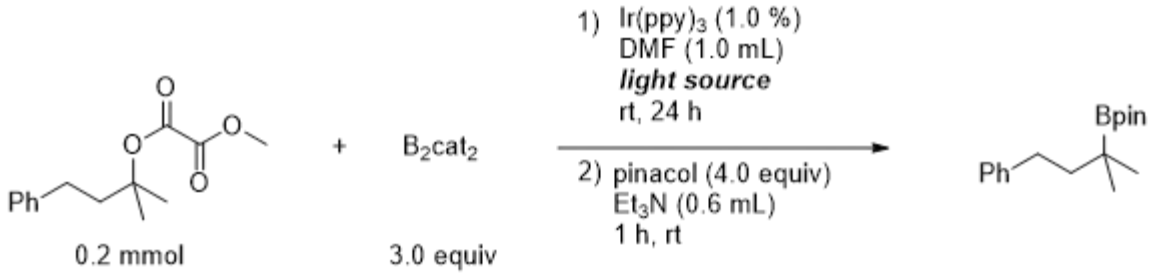


DrySyn Illumin8 = 95:5 cis:trans

DrySyn Illumin8: 450nm blue LED's

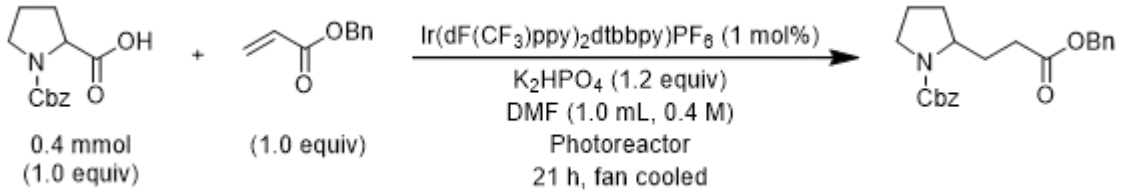
Comparison to other devices

From Angew. Chem. Int. Ed. 2019, 58,9561



Light Source	GC Yield
A	6%
B	70%
Illumin8	77%

From J. AM. Chem. Soc. 2014, 136, 10886-10889



Light Source	GC Yield
A	45%
Illumin8	40%

Note: reactions degassed by bubbling nitrogen through reaction mixture before adding to the reactor

DrySyn Illumin8: 365nm UV LED's

Evaluation of Illumin8 for UV polymerisation & Comparison vs an already in use commercially available parallel UV chemistry screening tool

Illumin8

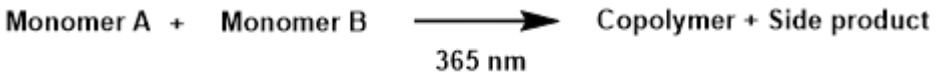
- ✓ Very small footprint instrument
- ✓ Simple set up
- ✓ Easy degas/remove of O_2
- ✓ 8 positions allowing simple screening
- ✓ Cooling fan allowing close to room temperature reaction
- ✓ Permits stirring

Alternative tool

- ✓ Timer option
- ✓ Easy to see if lamps are on/off thanks to the shielded window

DrySyn Illumin8: 365nm UV LED's

For polymerisation



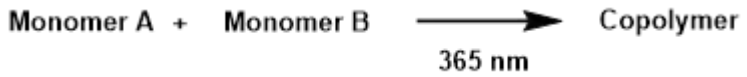
System	Reaction	Polymerisation (%)
Illumin8	Polymer conversion	43
	Side reaction	0
Commercially available alternative	Polymer conversion	56
	Side reaction	13

← **No unwanted products**

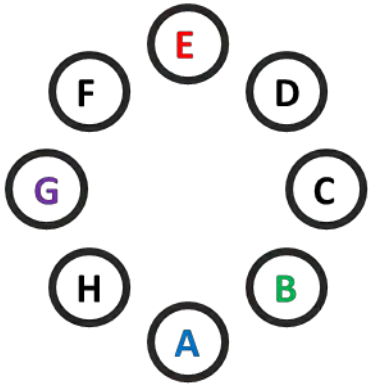
With Illumin8 the temperature of the solution after irradiation was 28 °C while with the UV chamber and no cooling system was ≈ 40 °C. The higher temperature can explain the degradation of the allyl double bond which is unwanted and yields side products.

DrySyn Illumin8: 365nm UV LED's

Reproducible parallel reaction screening



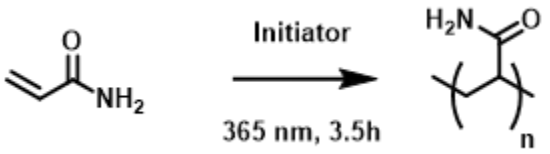
System	Position	Polymerisation (%)
Illumin8	A (4 mL)	30
	B (4 mL)	35
	E (4 mL)	33
	G (8 mL)	30



All positions in the Illumin8 reactor gave similar yield and conversion.
Also different volumes gave similar results.
On the UV chamber the positioning of the sample is critical for the yield.

DrySyn Illumin8: 365nm UV LED's

Effective light transmission



System	Expected MW (kDa)	Degree of conversion(%)
Illumin8	< 100	38
	> 100	23
Commercially available alternative	< 100	42
	> 100	0

← Sought after product achieved

With Illumin8 high MW polymer can be obtained (conversion based on NMR data, need confirmation by GPC). Using the UVP chamber no conversion was ever obtained for MW > 100 kDa.

Even after 3.5 hours of irradiation the temperature was 28 °C with Illumin8.

DrySyn Illumin8: 450nm blue LED's
DrySyn Illumin8: 365nm UV LED's

Any questions?

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