

Strem Kit Manual

96-7510: EvoluChem™ Photochemical Methylation Array Kit



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Sold in collaboration with HepatoChem

96-7510 EvoluChem™ Photochemical Methylation Array Kit

1 kit

Product overview:

The EvoluChem™ photochemical kits are ideal tools for the investigation of reaction conditions. This enables you to conveniently screen multiple reaction conditions simultaneously using pre-weighed catalysts and reagents. We offer pre-selected arrays of reagents, catalysts and/or salts or custom arrays depending on your needs.

Benefits

- Facilitates screen of photochemical reaction conditions
- Enables up to 32 reaction conditions simultaneously
- Save substrate using low scale reaction conditions
- Save time on optimization

Recommendations

- Safety personal protection such as gloves, safety glasses and lab coat should be worn at all times.
- Always use a clean and dry syringe to add and transfer solution.

Storage and Stability

- Store at 2-8°C in dark.
- Stable for 12 months.

Material required, but not supplied

- Customer supplied substrate
- Customer supplied reaction solvent(s)
- EvoluChem™ PhotoRedOx Box
- EvoluChem™ Light Source 18W-450 nm
- Nitrogen or Argon line for sparging solvents with two needles
- DMSO
- Stirring plate
- Syringe, decapper and reaction block

Kit Contents			
Description	Label	Quantity	Amount
Ir[dF(CF ₃)ppy] ₂ (dtbbpy)][PF ₆] (Strem# 77-0425) / <i>tert</i> -butyl peracetate	Ir dF(CF ₃) tBPA	8 vials	0.1 μmol/12.5 μmol
Ir[(ppy) ₂ (dtbbpy)][PF ₆] (Strem# 77-0410) / <i>tert</i> -butyl peracetate	Ir ppy tBPA	8 vials	0.1 μmol/12.5 μmol
50/50 Acetonitrile/ trifluoroacetic acid	ACN/TFA 1/1	1 vial	1 ml
Acetonitrile (10 equiv. trifluoroacetic acid*)	ACN/TFA 10 eq.	1 vial	1 ml
Acetic acid (10 equiv. trifluoroacetic acid*)	Acetic Ac. TFA 10 eq.	1 vial	1 ml
Acetic acid/water (10 equiv. trifluoroacetic acid*)	Acetic Ac. water TFA 10 eq.	1 vial	1 ml
Substrate stock vial 1	Substrate stock 1	1 vial	--
Substrate stock vial 2	Substrate stock 2	1 vial	--
Substrate stock vial 3	Substrate stock 3	1 vial	--
Substrate stock vial 4	Substrate stock 4	1 vial	--

*based on 0.05 M substrate solution

Visit www.strem.com for new product information and a searchable catalog.

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Reagent Information			
Strem Item#	Vial	CAS	MW
77-0425	$\text{Ir}[\text{dF}(\text{CF}_3)\text{ppy}]_2(\text{dtbbpy})][\text{PF}_6]$	870987-63-6	1121.91
77-0410	$\text{Ir}[(\text{ppy})_2(\text{dtbbpy})][\text{PF}_6]$	676525-77-2	913.95
N/A	tert-butyl peracetate (50% mineral spirits)	107-71-1	132.16

Typical Protocol

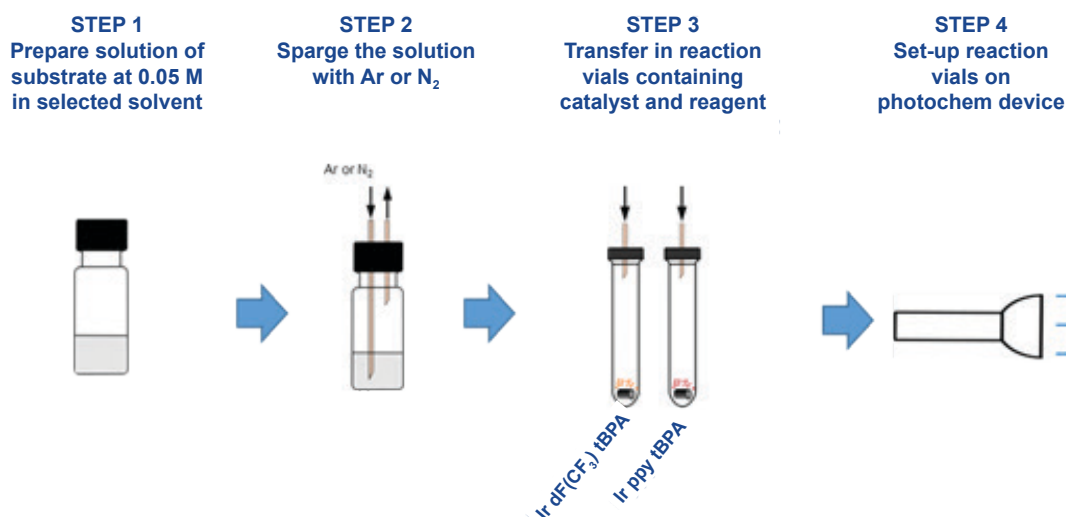
- The typical protocol is performed at 0.05 mol/l concentration reaction condition using a solution of substrate in 4 different solvents. Each sealed reaction vial contains 0.1 μmol of photocatalyst and 12.5 μmol of *tert*-butyl peracetate.
- Based on the concentration of the substrate stock solution and the volume added, the following reaction stoichiometry can be achieved with the standard photomethylation kit. See table below.
- Should solubility of substrate be an issue, lower concentrations can be used although longer reaction times may be required.
- The photomethylation kit contains four solvent mixtures. If the user prefers, alternate solvents can be screened. It is recommended that alternative solutions be prepared at 0.05 M or 0.1M with 10 equiv. of trifluoroacetic acid based on substrate.
- Sparging reaction solvents with nitrogen or argon while transferring reagents is important to achieve highest conversions of product. See protocol diagram for instructions.

Conc. [M]	Vol. (μl)	Equiv. Cat.	Equiv. TBPA
0.05	50	0.04	5.00
0.10	50	0.02	2.50
0.05	100	0.02	2.50
0.10	100	0.01	1.25

Protocol at 50 μl volume reaction condition

- Prepare the required volume of substrate solution at 0.05 mol/L in each solvent using the empty stock solution vials for each solvent. For example, 150 μl solution for 2 reaction conditions (50 μl extra to compensate potential evaporation).
- Degas first substrate solution with subsurface sparging via N_2 or Ar line with exit needle for 5 minutes.
- Using a clean and dry syringe, add 50 μl of the substrate solution to each reaction vial.
- Repeat steps 2 and 3 for each substrate solvent mixture.
- Stir the reaction vials in the photochemical device for 18 to 24 hours.
- Remove the vial caps using a decapper.
- Prepare analytical sample for each reaction condition with 5 μl sample diluted into 200 μl in either DMSO or water/acetonitrile 50/50. Alternatively, reaction solvent can be evaporated *in vacuo* and crude mixture diluted in water/acetonitrile prior to preparation of analytical sample.
- Analyze resulting analytical samples by LC/MS.

Protocol Diagram



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