Strem Kit Manual

96-7510: EvoluChem™ Photochemical Methylation Array Kit

METALS • INORGANICS • ORGANOMETALLICS • CATALYSTS • LIGANDS • NANOMATERIALS • CUSTOM SYNTHESIS • cGMP FACILITIES

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)*** / *tert*-butyl peracetate	Ir dF(CF_3) tBPA	8 vials	0.1µmol/12.5 µmol
Ir[(ppy)$_2$(dtbbpy)][PF_6] ***(Strem# 77-0410)*** / *tert*-butyl peracetate	Ir ppy tBPA	8 vials	0.1µmol/12.5 µmol
50/50 Acetonitrile/ trifluoroacetic acid	ACN/TFA 1/1	1 vial	1 ml
Acetonitrile (10 equiv. trifluoracetic acid*)	ACN/TFA 10 eq.	1 vial	1 ml
Acetic acid (10 equiv. trifluoracetic acid*)	Acetic Ac. TFA 10 eq.	1 vial	1 ml
Acetic acid/water (10 equiv. trifluoracetic 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*

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Reagent Information			
Strem Item#	**Vial**	**CAS**	**MW**
77-0425	$Ir[dF(CF_3)ppy]_2(dtbbpy)][PF_6]$	870987-63-6	1121.91
77-0410	$Ir[(ppy)_2(dtbbpy)[PF_6]$	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

1. 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).
2. Degas first substrate solution with subsurface sparging via N_2 or Ar line with exit needle for 5 minutes.
3. Using a clean and dry syringe, add 50 µl of the substrate solution to each reaction vial.
4. Repeat steps 2 and 3 for each substrate solvent mixture.
5. Stir the reaction vials in the photochemical device for 18 to 24 hours.
6. Remove the vial caps using a decapper.
7. 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.
8. Analyze resulting analytical samples by LC/MS.

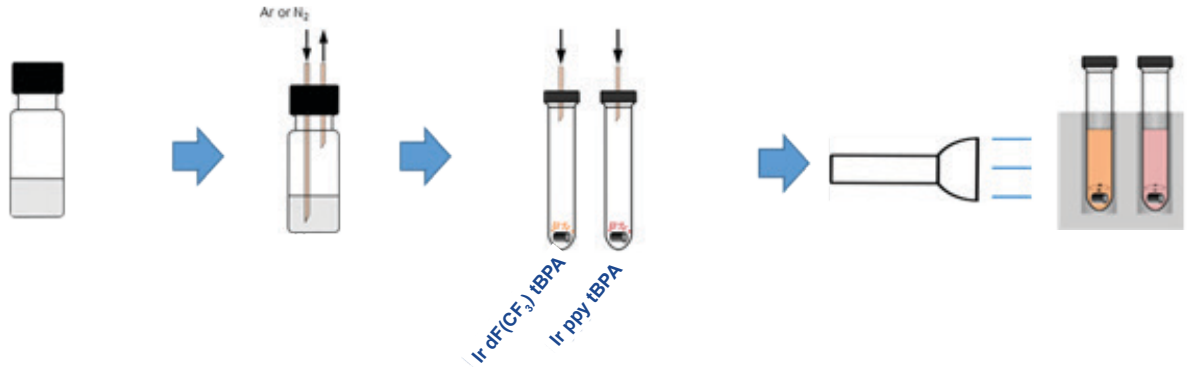
Protocol Diagram

STEP 1
Prepare solution of substrate at 0.05 M in selected solvent

STEP 2
Sparge the solution with Ar or N_2

STEP 3
Transfer in reaction vials containing catalyst and reagent

STEP 4
Set-up reaction vials on photochem device



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