# Strem Kit Manual

## 96-7560: EvoluChem™ Photocatalytic Alkylation Kit

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

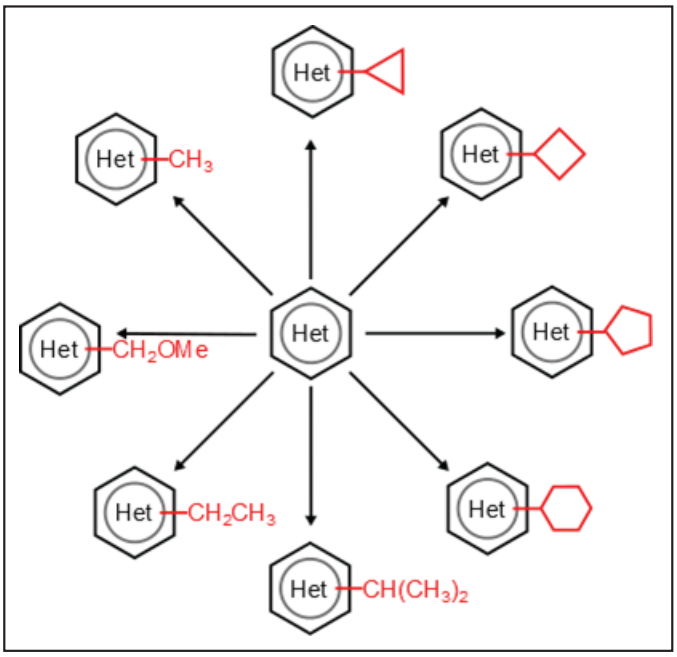
*Sold in collaboration with HepatoChem*

## 96-7560 EvoluChem™ Photocatalytic Alkylation Kit

1 kit

### Product overview:

The trifluoroborate alkylation reaction (Minisci reaction)[1] is a powerful late stage functionalization tool. Our kit allows convenient, one-step production of eight different analogues of a lead compound in mg quantities. Each reaction vial contains 75 µmol of trifluoroborate alkylation reagent (pre-weighed) and a stirring bar to react with 50 µmol of substrate. C-H functionalization will primarily occur on electron-deficient heteroarenes at one or several positions.




### 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

### Material required, but not supplied

- Customer supplied substrate
- Customer supplied reaction solvent(s)
- Trifluoracetic acid
- EvoluChem™ PhotoRedOx Box
- EvoluChem™ Light Source 18W-450 nm
- EvoluChem™ Vial Holder *(98-7650)*
- Nitrogen or Argon line for sparging solvents with two needles
- DMSO
- Stirring plate
- 500 µL - 1.0 ml syringe

### 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.

**Kit Contents**

| Description | Label | Quantity | Amount |
|---|---|---|---|
| Potassium cyclopropyltrifluoroborate / Potassium persulfate | cyclopropyl-$BF_3K$/ $K_2S_2O_8$ | 2 x vials | 75 µmol / 100 µmol |
| Potassium cyclobutyltrifluoroborate / Potassium persulfate | cyclobutyl-$BF_3K$/ $K_2S_2O_8$ | 2 x vials | 75 µmol / 100 µmol |
| Potassium cyclopentyltrifluoroborate / Potassium persulfate | cyclopentyl-$BF_3K$/ $K_2S_2O_8$ | 2 x vials | 75 µmol / 100 µmol |
| Potassium cyclohexyltrifluoroborate / Potassium persulfate | cyclohexyl-$BF_3K$/ $K_2S_2O_8$ | 2 x vials | 75 µmol / 100 µmol |
| Potassium ethyltrifluoroborate / Potassium persulfate | ethyl-$BF_3K$/ $K_2S_2O_8$ | 2 x vials | 75 µmol / 100 µmol |
| Potassium isopropyltrifluoroborate / Potassium persulfate | isopropyl-$BF_3K$/ $K_2S_2O_8$ | 2 x vials | 75 µmol / 100 µmol |
| Potassium methoxymethyltrifluoroborate / Potassium persulfate | MOM-$BF_3K$/ $K_2S_2O_8$ | 2 x vials | 75 µmol / 100 µmol |
| *tert*-butyl peracetate | TBA | 2 x vials | 250 µmol |
| Ir[dF($CF_3$)ppy]$_2$(dtbbpy][$PF_6$] ***(Strem# 77-0425)*** / | Ir-cat-1 | 2 x vials | 8 µmol |



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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 |
| --- | Potassium cyclopropyltrifluoroborate | 1065010-87-8 | 147.98 |
| --- | Potassium cyclobutyltrifluoroborate | 1065010-88-9 | 162.00 |
| --- | Potassium cyclopentyltrifluoroborate | 1040745-70-7 | 176.03 |
| --- | Potassium ethyltrifluoroborate | 44248-07-9 | 135.97 |
| --- | Potassium isopropyltrifluoroborate | 1041642-13-0 | 149.99 |
| --- | Potassium methoxymethyltrifluoroborate | 910251-11-5 | 151.97 |
| --- | *tert*-butyl peracetate solution TBPA (50% mineral spirits | 107-71-1 | 132.16 |
| --- | Potassium persulfate | 7727-21-1 | 270.32 |

## Typical Protocol

- The typical protocol is performed at 0.1 mol/l of substrate prepared as solution in DMSO or $H_2O$/ACN with 5 equiv. of trifluoroacetic acid. Each sealed reaction vial contains either 75 µmol of $BF_3K$ reagent with 100 µmol of $K_2S_2O_8$ or 250 µmol of tert-butyl peracetate.
- Typically, the reaction is performed with 6200K white LED or blue LED (450 nm).
- The alkylation production kit contains 2 sets of vials allowing the screening of two different substrates or 1 substrate in two solvents.
- Sparging reaction solvents with nitrogen or argon while transferring reagents is important to achieve highest conversions of product. See protocol diagram for instructions.

## Protocol at 500 µl volume reaction condition

1. In the vial provided containing the Ir catalyst, prepare 4.0 ml of substrate solution at 0.1 mol/L of substrate (400 µmol substrate needed) in either DMSO or $H_2O$/ACN with 153 µL of trifluoroacetic acid (5 equiv.). Mix thoroughly.
2. Degas the substrate/catalyst solution with subsurface sparging via $N_2$ or Ar line with exit needle for 5 minutes.
3. Using a clean and dry syringe, add 500 µL of the substrate solution to each reaction vial (8 reactions).
4. Place samples in vial holder **98-7650**.
5. Turn on lamp (6200K white LED or blue LED 450 nm) and stir vials for 2 to 4 hours (or longer if necessary and there is substrate remaining). Be sure to plug in fan to maintain RT.
6. Upon completion of reaction, prepare analytical sample for each reaction condition with 5 µl sample diluted into 200 µl in either DMSO or water/acetonitrile 50/50.
7. Analyze resulting analytical samples by LC/MS.



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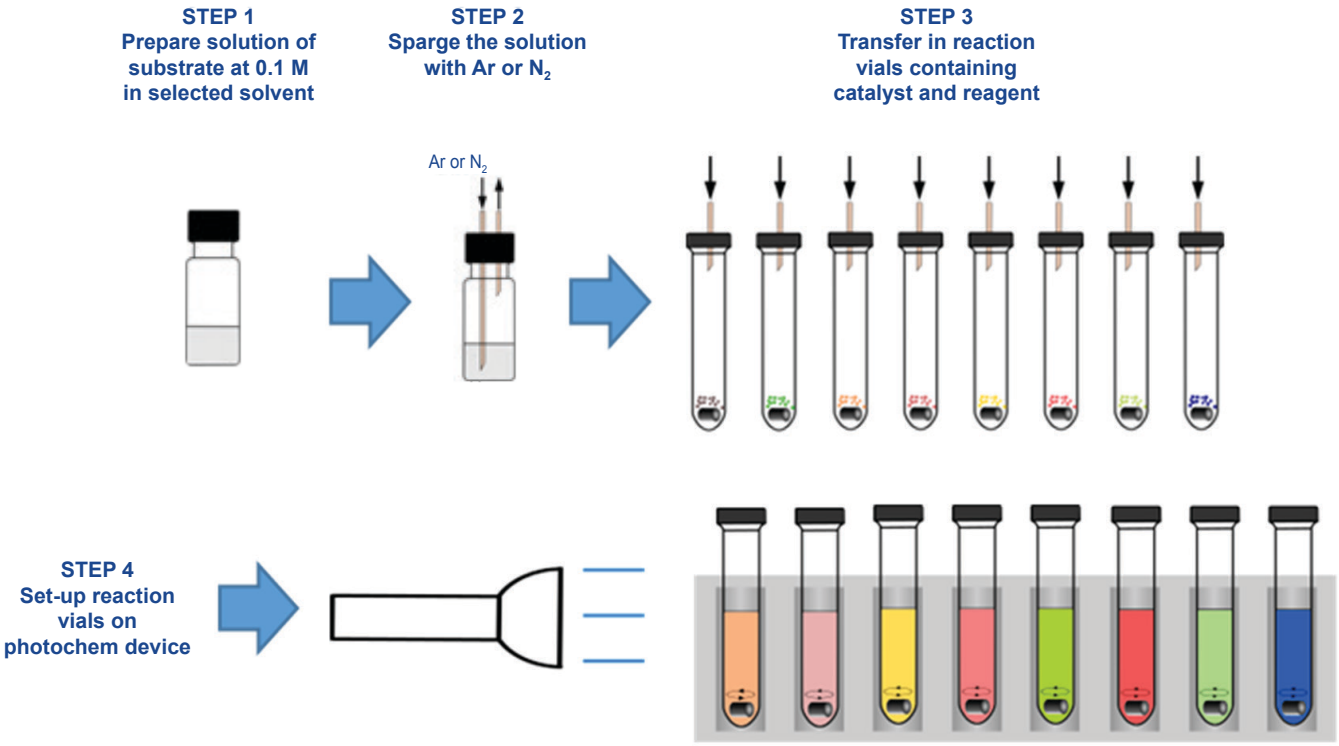
## Protocol Diagram



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