

# Strem Kit Manual

## 96-7550: Iridium/Nickel PhotoRedOx Base and Iridium Catalyst Screening Kit



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

Sold in collaboration with HepatoChem

### 96-7550 EvoluChem™ Iridium/Nickel PhotoRedOx Base and Iridium Catalyst Screening 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

#### Reagent Information

Strem Item#	Vial	Label ID	CAS	MW
77-0425	$\text{Ir}[\text{dF}(\text{CF}_3)\text{ppy}]_2(\text{dtbbpy})[\text{PF}_6]$	Ir cat 1	870987-63-6	1121.91
77-0410	$\text{Ir}(\text{dtbbpy})(\text{ppy})_2\text{PF}_6$	Ir cat 2	676525-77-2	913.95
77-0453	$\text{Ir}(\text{dF}-\text{CF}_3\text{-ppy})_2(\text{bpy})\text{PF}_6$	Ir cat 3	1092775-62-6	1009.70
77-7030	$\text{Ir}(\text{dF-ppy})_3$	Ir cat 4	387859-70-3	762.72
77-0218	$\text{Ir}(\text{dmpy})_2(\text{dtbbpy})\text{PF}_6$	Ir cat 5	1607469-49-7	970.06
77-0330	$\text{Ir}(\text{dF}-\text{CH}_3\text{-ppy})_2(\text{dtbbpy})\text{PF}_6$	Ir cat 6	1335047-34-1	1013.96
93-2801	$\text{NiCl}_2\text{-dme}$	Ni	29046-78-4	219.72
07-0273	4,4'-Bis(di-t-butyl)-2,2'-bipyridine(dtbbpy)	dtbbpy	72914-19-3	268.40
93-5514	Cesium carbonate	$\text{Cs}_2\text{CO}_3$	534-17-8	325.82
N/A	Cesium fluoride	CsF	13400-13-0	151.90
N/A	1,8-Diazabicyclo[5.4.0]undec-7-ene (DBU)	DBU	6674-22-2	152.24

Visit [www.strem.com](http://www.strem.com) for new product information and a searchable catalog.

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Kit Contents		
Description	Quantity	Amount
$\text{Ir}[\text{dF}(\text{CF}_3)\text{ppy}]_2(\text{dtbbpy})][\text{PF}_6]$ <b>Ir cat-1 (Strem# 77-0425)</b> / Ni-dtbbpy $\text{Cs}_2\text{CO}_3$	2 x vials	0.1 $\mu\text{mol}$ / 0.5 $\mu\text{mol}$ / 15 $\mu\text{mol}$
$\text{Ir}(\text{dtbbpy})(\text{ppy})_2\text{PF}_6$ <b>Ir cat-2 (Strem# 77-0410)</b> / Ni-dtbbpy $\text{Cs}_2\text{CO}_3$	2 x vials	0.1 $\mu\text{mol}$ / 0.5 $\mu\text{mol}$ / 15 $\mu\text{mol}$
$\text{Ir}(\text{dF}-\text{CF}_3\text{-ppy})_2(\text{bpy})\text{PF}_6$ <b>Ir cat-3 (Strem# 77-0453)</b> / Ni-dtbbpy $\text{Cs}_2\text{CO}_3$	2 x vials	0.1 $\mu\text{mol}$ / 0.5 $\mu\text{mol}$ / 15 $\mu\text{mol}$
$\text{Ir}(\text{dF-ppy})_3$ <b>Ir cat-4 (Strem# 77-7030)</b> / Ni-dtbbpy $\text{Cs}_2\text{CO}_3$	2 x vials	0.1 $\mu\text{mol}$ / 0.5 $\mu\text{mol}$ / 15 $\mu\text{mol}$
$\text{Ir}(\text{dmppy})_2(\text{dtbbpy})\text{PF}_6$ <b>Ir cat-5 (Strem# 77-0218)</b> / Ni-dtbbpy $\text{Cs}_2\text{CO}_3$	2 x vials	0.1 $\mu\text{mol}$ / 0.5 $\mu\text{mol}$ / 15 $\mu\text{mol}$
$\text{Ir}(\text{dF}-\text{CH}_3\text{-ppy})_2(\text{dtbbpy})\text{PF}_6$ <b>Ir cat-6 (Strem# 77-0330)</b> / Ni-dtbbpy $\text{Cs}_2\text{CO}_3$	2 x vials	0.1 $\mu\text{mol}$ / 0.5 $\mu\text{mol}$ / 15 $\mu\text{mol}$
$\text{Ir}[\text{dF}(\text{CF}_3)\text{ppy}]_2(\text{dtbbpy})][\text{PF}_6]$ <b>Ir cat-1 (Strem# 77-0425)</b> / Ni-dtbbpy $\text{CsF}$	2 x vials	0.1 $\mu\text{mol}$ / 0.5 $\mu\text{mol}$ / 15 $\mu\text{mol}$
$\text{Ir}(\text{dtbbpy})(\text{ppy})_2\text{PF}_6$ <b>Ir cat-2 (Strem# 77-0410)</b> / Ni-dtbbpy $\text{CsF}$	2 x vials	0.1 $\mu\text{mol}$ / 0.5 $\mu\text{mol}$ / 15 $\mu\text{mol}$
$\text{Ir}(\text{dF}-\text{CF}_3\text{-ppy})_2(\text{bpy})\text{PF}_6$ <b>Ir cat-3 (Strem# 77-0453)</b> / Ni-dtbbpy $\text{CsF}$	2 x vials	0.1 $\mu\text{mol}$ / 0.5 $\mu\text{mol}$ / 15 $\mu\text{mol}$
$\text{Ir}(\text{dF-ppy})_3$ <b>Ir cat-4 (Strem# 77-7030)</b> / Ni-dtbbpy $\text{CsF}$	2 x vials	0.1 $\mu\text{mol}$ / 0.5 $\mu\text{mol}$ / 15 $\mu\text{mol}$
$\text{Ir}(\text{dmppy})_2(\text{dtbbpy})\text{PF}_6$ <b>Ir cat-5 (Strem# 77-0218)</b> / Ni-dtbbpy $\text{CsF}$	2 x vials	0.1 $\mu\text{mol}$ / 0.5 $\mu\text{mol}$ / 15 $\mu\text{mol}$
$\text{Ir}(\text{dF}-\text{CH}_3\text{-ppy})_2(\text{dtbbpy})\text{PF}_6$ <b>Ir cat-6 (Strem# 77-0330)</b> / Ni-dtbbpy $\text{CsF}$	2 x vials	0.1 $\mu\text{mol}$ / 0.5 $\mu\text{mol}$ / 15 $\mu\text{mol}$
$\text{Ir}[\text{dF}(\text{CF}_3)\text{ppy}]_2(\text{dtbbpy})][\text{PF}_6]$ <b>Ir cat-1 (Strem# 77-0425)</b> / Ni-dtbbpy $\text{DBU}^*$	2 x vials	0.1 $\mu\text{mol}$ / 0.5 $\mu\text{mol}$ / ---
$\text{Ir}(\text{dtbbpy})(\text{ppy})_2\text{PF}_6$ <b>Ir cat-2 (Strem# 77-0410)</b> / Ni-dtbbpy $\text{DBU}^*$	2 x vials	0.1 $\mu\text{mol}$ / 0.5 $\mu\text{mol}$ / ---
$\text{Ir}(\text{dF}-\text{CF}_3\text{-ppy})_2(\text{bpy})\text{PF}_6$ <b>Ir cat-3 (Strem# 77-0453)</b> / Ni-dtbbpy $\text{DBU}^*$	2 x vials	0.1 $\mu\text{mol}$ / 0.5 $\mu\text{mol}$ / ---
$\text{Ir}(\text{dF-ppy})_3$ <b>Ir cat-4 (Strem# 77-7030)</b> / Ni-dtbbpy $\text{DBU}^*$	2 x vials	0.1 $\mu\text{mol}$ / 0.5 $\mu\text{mol}$ / ---
$\text{Ir}(\text{dmppy})_2(\text{dtbbpy})\text{PF}_6$ <b>Ir cat-5 (Strem# 77-0218)</b> / Ni-dtbbpy $\text{DBU}^*$	2 x vials	0.1 $\mu\text{mol}$ / 0.5 $\mu\text{mol}$ / ---
$\text{Ir}(\text{dF}-\text{CH}_3\text{-ppy})_2(\text{dtbbpy})\text{PF}_6$ <b>Ir cat-6 (Strem# 77-0330)</b> / Ni-dtbbpy $\text{DBU}^*$	2 x vials	0.1 $\mu\text{mol}$ / 0.5 $\mu\text{mol}$ / ---
$\text{DBU}$	2 x vials	120 $\mu\text{mol}$

\*DBU is added later. See protocol.

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## Typical Protocol

- The typical protocol is performed at 0.05 mol/L substrates using a solution containing two coupling components. Each sealed reaction vial contains 0.1  $\mu\text{mol}$  of photocatalyst, 0.5  $\mu\text{mol}$  Ni catalyst, 0.5  $\mu\text{mol}$  ligand and 15  $\mu\text{mol}$  of base (for  $\text{Cs}_2\text{CO}_3$  and CsF only - for DBU conditions, base is added separately and shipped in a separate vial).
- Based on the concentration of the substrates stock solution and the volume added, the following reaction stoichiometry can be achieved with the standard Ir/Ni photoredox kit. See table below.

Conc. [M]	Vol. ( $\mu\text{l}$ )	Equiv. Ir Cat.	Equiv. Ni Cat	Equiv. base
0.050	100	0.02	0.10	3.0
0.050	200	0.01	0.05	1.5
0.025	200	0.02	0.10	3.0
0.025	100	0.04	0.20	6.0

- If low solubility of substrate is an issue, lower concentrations can be used; however, longer reaction times may be required.
- The Ir/Ni photoredox kit contains 2 sets of reaction vials allowing the screening of two different solvents. Recommended solvents include: MeCN, DMF, DME, dioxane and DMSO.
- 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 100 $\mu\text{l}$ volume reaction condition

- Prepare the required volume of substrate solution at 0.05 mol/L containing both coupling substrates. For example, 2.0 ml solution for 18 reaction conditions (200  $\mu\text{l}$  extra to compensate potential evaporation).
- Degas substrate solution with subsurface sparging via  $\text{N}_2$  or Ar line with exit needle for 5 minutes.
- Using a clean and dry syringe, add 100  $\mu\text{l}$  of the substrate solution to each reaction vial containing  $\text{Cs}_2\text{CO}_3$  and CsF (excluding DBU vials for now).\*\*\*
- Add remaining 800  $\mu\text{l}$  of substrate solution to vial containing DBU. Stir and degas for 5 minutes.
- Transfer 100  $\mu\text{l}$  substrate/DBU solution to each DBU reaction vial.
- 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  $\mu\text{l}$  sample diluted into 200  $\mu\text{l}$  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.

\*\*\*Alternatively, 2.24  $\mu\text{l}$  of DBU can be added to each of the 6 DBU reactions separately using 10  $\mu\text{l}$  Hamilton Syringe (not provided) instead of premixing substrate solutions and DBU base. In this case, first add 100  $\mu\text{l}$  substrate solution to DBU reaction vials, followed by addition of 2.24  $\mu\text{l}$  DBU and proceed to step 6.

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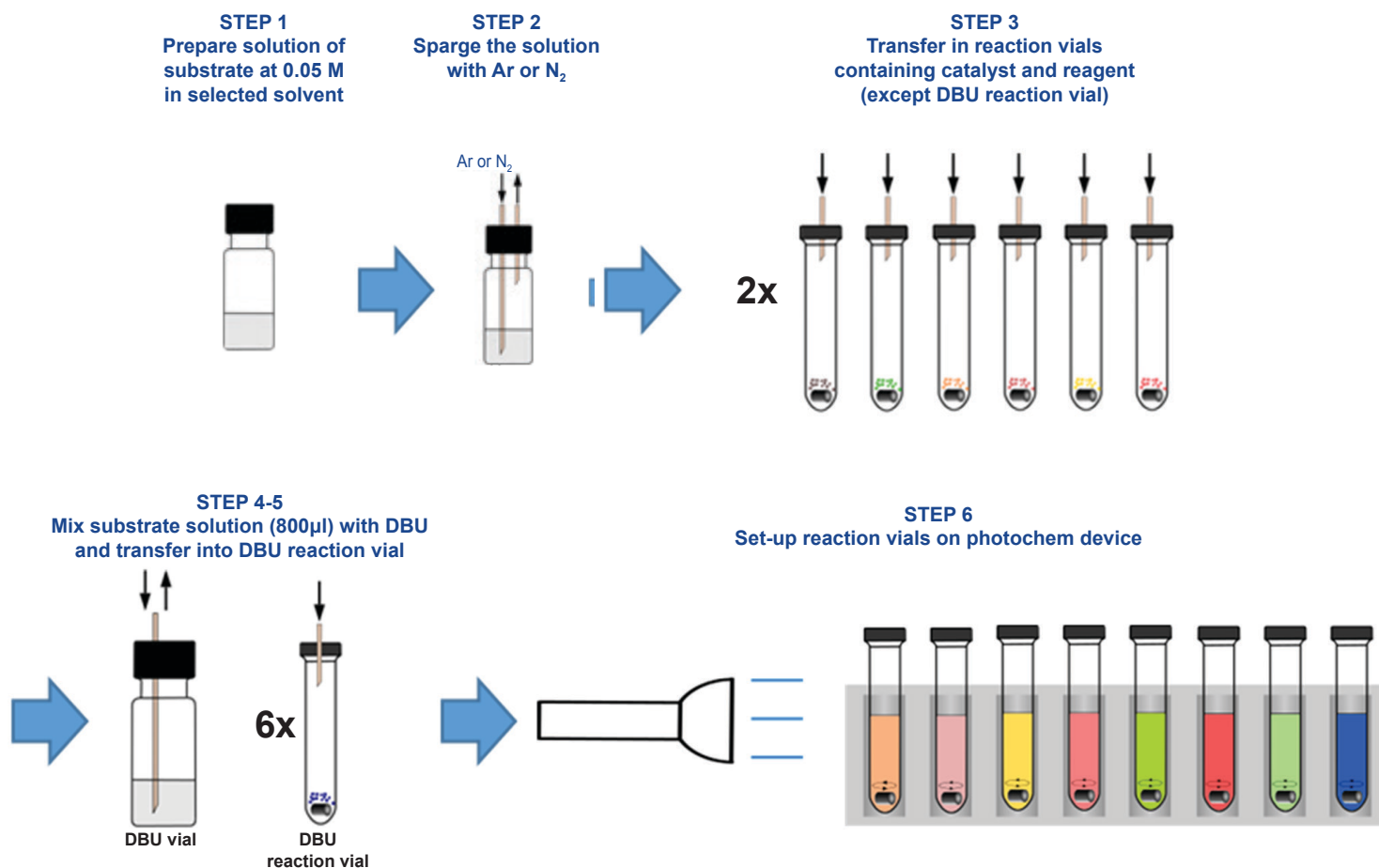
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## Protocol Diagram



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