

# Strem Kit Manual

## 96-7520: Iridium/Nickel PhotoRedOx Base and Solvent Screening Kit 1

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

Sold in collaboration with HepatoChem

### 96-7520 EvoluChem™ Iridium/Nickel PhotoRedOx Base and Solvent Screening Kit 1 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.

#### 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

#### Storage and Stability

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

Kit Contents

Description	Label	Quantity	Amount
Ir[dF(CF <sub>3</sub> )ppy] <sub>2</sub> (dtbbpy)[PF <sub>6</sub> ] (Strem# 77-0425) / NiCl <sub>2</sub> -dme / dtbbpy / Cs <sub>2</sub> CO <sub>3</sub>	Ir/Ni-dtbbpy / Cs <sub>2</sub> CO <sub>3</sub>	2 x vials	0.1 μmol / 0.5 μmol / 0.5 μmol / 15 μmol
Ir[dF(CF <sub>3</sub> )ppy] <sub>2</sub> (dtbbpy)[PF <sub>6</sub> ] (Strem# 77-0425) / NiCl <sub>2</sub> -dme / dtbbpy / K <sub>3</sub> PO <sub>4</sub>	Ir/Ni-dtbbpy / K <sub>3</sub> PO <sub>4</sub>	2 x vials	0.1 μmol / 0.5 μmol / 0.5 μmol / 15 μmol
Ir[dF(CF <sub>3</sub> )ppy] <sub>2</sub> (dtbbpy)[PF <sub>6</sub> ] (Strem# 77-0425) / NiCl <sub>2</sub> -dme / dtbbpy / K <sub>2</sub> HPO <sub>4</sub>	Ir/Ni-dtbbpy / K <sub>2</sub> HPO <sub>4</sub>	2 x vials	0.1 μmol / 0.5 μmol / 0.5 μmol / 15 μmol
Ir[dF(CF <sub>3</sub> )ppy] <sub>2</sub> (dtbbpy)[PF <sub>6</sub> ] (Strem# 77-0425) / NiCl <sub>2</sub> -dme / dtbbpy / KOH	Ir/Ni-dtbbpy / KOH	2 x vials	0.1 μmol / 0.5 μmol / 0.5 μmol / 15 μmol
Ir[dF(CF <sub>3</sub> )ppy] <sub>2</sub> (dtbbpy)[PF <sub>6</sub> ] (Strem# 77-0425) / NiCl <sub>2</sub> -dme / dtbbpy / Li <sub>2</sub> CO <sub>3</sub>	Ir/Ni-dtbbpy / Li <sub>2</sub> CO <sub>3</sub>	2 x vials	0.1 μmol / 0.5 μmol / 0.5 μmol / 15 μmol
Ir[dF(CF <sub>3</sub> )ppy] <sub>2</sub> (dtbbpy)[PF <sub>6</sub> ] (Strem# 77-0425) / NiCl <sub>2</sub> -dme / dtbbpy / K <sub>2</sub> CO <sub>3</sub>	Ir/Ni-dtbbpy / K <sub>2</sub> CO <sub>3</sub>	2 x vials	0.1 μmol / 0.5 μmol / 0.5 μmol / 15 μmol
Ir[dF(CF <sub>3</sub> )ppy] <sub>2</sub> (dtbbpy)[PF <sub>6</sub> ] (Strem# 77-0425) / NiCl <sub>2</sub> -dme / dtbbpy / DABCO	Ir/Ni-dtbbpy / DABCO	2 x vials	0.1 μmol / 0.5 μmol / 0.5 μmol / 15 μmol
Ir[dF(CF <sub>3</sub> )ppy] <sub>2</sub> (dtbbpy)[PF <sub>6</sub> ] (Strem# 77-0425) / NiCl <sub>2</sub> -dme / dtbbpy / DBU*	Ir/Ni-dtbbpy / DBU	2 x vials	0.1 μmol / 0.5 μmol / 0.5 μmol / ---
DBU	DBU	2 x vials	15 μmol

\*DBU is added later. See protocol.

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

**Strem Chemicals, Inc.**  
7 Mulliken Way  
Newburyport, MA 01950  
U.S.A.  
Tel: 978.499.1600  
Fax: 978.465.3104  
Email: info@strem.com

**Strem Chemicals, Inc.**  
15, rue de l'Atome  
Zone Industrielle  
67800 BISCHHEIM France  
Tel: (33) 03 88 62 52 60  
Fax: (33) 03 88 62 26 81  
Email: info.europe@strem.com

**Strem Chemicals, Inc.**  
Postfach 1215  
77672 KEHL  
Germany  
Tel: 0 78 51/ 7 58 79  
Email: info.europe@strem.com

**Strem Chemicals UK Ltd.**  
An Independent Distributor of Strem Chemicals Products  
Newton Hall, Town Street  
Newton, Cambridge  
England CB22 7ZE  
Tel: +44 (0)1223 873 028  
Fax: +44 (0)1223 870207  
Email: enquiries@strem.co.uk

Reagent Information			
Strem Item#	Vial	CAS	MW
77-0425	Ir[dF(CF <sub>3</sub> )ppy] <sub>2</sub> (dtbbpy)][PF <sub>6</sub> ]	870987-63-6	1121.91
93-2801	NiCl <sub>2</sub> -dme	29046-78-4	219.72
07-0273	4,4'-Bis(di-t-butyl)-2,2'-bipyridine(dtbbpy)	72914-19-3	268.40
93-5514	Cesium carbonate	534-17-8	325.82
19-3800	Potassium phosphate tribasic	7778-53-2	212.27
93-1940	Potassium phosphate dibasic	7758-11-4	174.20
19-3150	Potassium hydroxide	1310-58-3	56.11
93-0311	Lithium carbonate	554-13-2	73.89
93-1912	Potassium carbonate	584-08-7	138.20
N/A	1,4-diazabicyclo[2.2.2]octane (DABCO)	280-57-9	112.17
N/A	1,8-Diazabicyclo[5.4.0]undec-7-ene (DBU)	6674-22-2	152.24

## Typical Protocol

- The typical protocol is performed at 0.05 mol/l using a solution containing two coupling components. Each sealed reaction vial contains 0.1 μmol of photocatalyst, 0.5 μmol Ni catalyst, 0.5 μmol ligand and 15 of μmol base (for all bases except 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. (μl)	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

- Should solubility of substrate be an issue, lower concentrations can be used although longer reaction times may be required.
- The Ir/Ni photoredox kit contains 2 sets of vials allowing the screening of two different solvents. Recommended solvents include but are not limited to acetonitrile, dimethylformamide, dimethylacetamide, dimethylsulfoxide.
- 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 μl volume reaction condition

- Prepare the required volume of substrate solution at 0.05 mol/L containing both coupling substrates. For example, 900 μl solution for 8 reaction conditions (100 μl extra to compensate potential evaporation).
- Degas substrate solution with subsurface sparging via N<sub>2</sub> or Ar line with exit needle for 5 minutes.
- Using a clean and dry syringe, add 100 μl of the substrate solution to each reaction vial (excluding DBU for now).
- Add 100 ul of substrate solution to vial containing DBU base vial. Repeat degas of solution.
- Add full contents to remaining DBU reaction vial.
- Repeat steps 2 and 5 for each substrate solvent mixture.
- Stir the reaction vials for 5 minutes prior to turning on the light to allow catalysts to fully dissolve (some bases will remain insoluble). The card containing the vials can be placed directly on the opening of the photochemistry device.
- Turn on lamp and stir vials for 18 to 24 hours (or longer if necessary). Be sure to plug in fan to maintain RT.
- Upon completion of reaction, 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.

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15, rue de l'Atome  
Zone Industrielle  
67800 BISCHHEIM France  
Tel: (33) 03 88 62 52 60  
Fax: (33) 03 88 62 26 81  
Email: [info.europe@strem.com](mailto:info.europe@strem.com)

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Germany  
Tel: 0 78 51/ 7 58 79  
Email: [info.europe@strem.com](mailto:info.europe@strem.com)

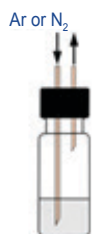
**Strem Chemicals UK Ltd.**  
An Independent Distributor of Strem Chemicals Products  
Newton Hall, Town Street  
Newton, Cambridge  
England CB22 7ZE  
Tel: +44 (0)1223 873 028  
Fax: +44 (0)1223 870207  
Email: [enquiries@strem.co.uk](mailto:enquiries@strem.co.uk)

## Protocol Diagram

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



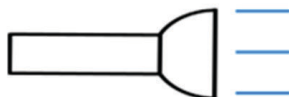
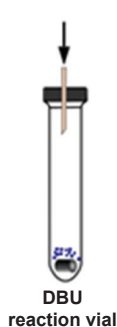
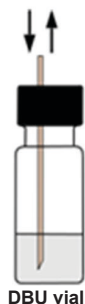
**STEP 2**  
Sparge the solution with Ar or N<sub>2</sub>



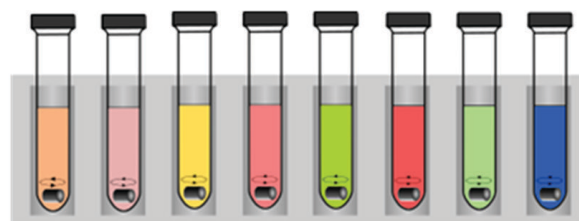
**STEP 3**  
Transfer in reaction vials containing catalyst and reagent (except DBU reaction vial)



**STEP 4-5**  
Mix substrate solution (100µl) with DBU and transfer into DBU reaction vial



**STEP 6**  
Set-up reaction vials on photochem device



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