Strem Kit Manual

96-7570: Iridium/Nickel PhotoRedOx Base and Solvent Screening Kit 2 (C-O coupling)

HepatoChem
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Sold in collaboration with HepatoChem

96-7570 **EvoluChem™ Iridium/Nickel PhotoRedOx Base and Solvent Screening Kit 2 (C-O coupling)** 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
$Ir[dF(CF_3)ppy]_2(dtbbpy)[PF_6]$ ***(Strem# 77-0425)*** / $NiCl_2$-dme / dtbbpy Cs_2CO_3	Ir/Ni-dtbbpy/ Cs_2CO_3	2 x vials	0.1µmol / 0.5 µmol / 0.5 µmol / 15 µmol
$Ir[dF(CF_3)ppy]_2(dtbbpy)[PF_6]$ ***(Strem# 77-0425)*** / $NiCl_2$-dme / dtbbpy K_3PO_4	Ir/Ni-dtbbpy/ K_3PO_4	2 x vials	0.1µmol / 0.5 µmol / 0.5 µmol / 15 µmol
$Ir[dF(CF_3)ppy]_2(dtbbpy)[PF_6]$ ***(Strem# 77-0425)*** / 5 mol% $NiCl_2$-dme* / dtbbpy K_2CO_3	Ir/ 5 mol% Ni-dtbbpy/ K_2CO_3	2 x vials	0.1µmol / 0.5 µmol / 0.5 µmol / 15 µmol
$Ir[dF(CF_3)ppy]_2(dtbbpy)[PF_6]$ ***(Strem# 77-0425)*** / 2.5 mol% $NiCl_2$-dme* / dtbbpy K_2CO_3	Ir/ 2.5 mol% Ni-dtbbpy/ K_2CO_3	2 x vials	0.1µmol / 0.25 µmol / 0.25 µmol / 15 µmol
$Ir[dF(CF_3)ppy]_2(dtbbpy)[PF_6]$ ***(Strem# 77-0425)*** / 1.25 mol% $NiCl_2$-dme* / dtbbpy K_2CO_3	Ir/ 1.25 mol% Ni-dtbbpy/ K_2CO_3	2 x vials	0.1µmol / 0.125 µmol / 0.125 µmol / 15 µmol
$Ir[dF(CF_3)ppy]_2(dtbbpy)[PF_6]$ ***(Strem# 77-0425)*** / $NiCl_2$-dme / dtbbpy DABCO	Ir/Ni-dtbbpy/ DABCO	2 x vials	0.1µmol / 0.5 µmol / 0.5 µmol / 15 µmol
$Ir[dF(CF_3)ppy]_2(dtbbpy)[PF_6]$ ***(Strem# 77-0425)*** / $NiCl_2$-dme / dtbbpy Quinuclidine	Ir/Ni-dtbbpy/ Quin.	2 x vials	0.1µmol / 0.5 µmol / 0.5 µmol / 15 µmol
Control	Control	2 x vials	---
Quinuclidine	Quinuclidine	2 x vials	10 µmol

**based on 0.1 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	$Ir[dF(CF_3)ppy]_2(dtbbpy)][PF_6]$	870987-63-6	1121.91
93-2801	$NiCl_2$-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
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	Quinuclidine	100-76-5	111.18

Typical Protocol

- The typical protocol is performed at 0.1 mol/l of bromide substrate with an excess of alcohol, typically 3 equivalents prepared as a solution containing two coupling components and 10 µmol of quinuclidine. Acetonitrile is the suggested solvent, although acetone and ethyl acetate are also suitable. Each sealed reaction vial contains 0.1 µmol of photocatalyst, 0.5 µmol Ni catalyst, 0.5 µmol ligand and 15 of µmol base. 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. Quin.	Equiv. base
0.100	100	0.01	0.05	0.10	1.5
0.200	50	0.01	0.05	0.10	1.5
0.050	100	0.02	0.10	0.20	3.0
0.025	100	0.04	0.20	0.40	6.0

- The Ir/Ni photoredox kit contains 2 sets of vials allowing the screening of two different substrate combinations or 1 combination and 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 100 µl volume reaction condition

1. In the 4mL vial containing the quinuclidine, prepare 1.0 ml of substrate solution at 0.1 mol/L of bromide and excess alcohol (typically 3 equivalents, although less alcohol can be used). If different concentration is used, adjust volume accordingly. For example, 1.0 ml solution for 8 reaction conditions (extra to compensate potential evaporation). Acetonitrile is the recommended solvent, although acetone and ethyl acetate can be used.
2. Degas 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 100 µl (or desired volume based on substrate concentration) of the substrate solution to each reaction vial.
4. Repeat steps 2 and 3 for each substrate solvent mixture.
5. Place samples in vial holder **98-7600**. Stir the reaction vials for 5 minutes prior to turning on the light to allow catalysts to fully dissolve (some bases will remain insoluble).
6. Turn on lamp and stir vials for 12 to 24 hours (or longer if necessary). Be sure to plug in fan to maintain RT.
7. Upon completion of reaction, remove the vial caps using a decapper.
8. 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.
9. Analyze resulting analytical samples by LC/MS.

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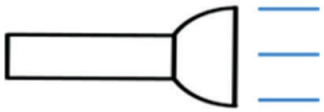
Protocol Diagram

STEP 1
Prepare solution of Bromide at 0.1 mol/l alcohol (3eq) in Quinuclidine

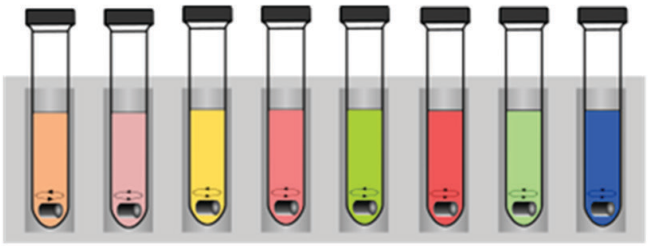
STEP 2
Sparge the solution with Ar or N_2

STEP 3
Transfer in reaction vials

STEP 4
Repeat steps 2 & 3



STEP 5
Set-up reaction vials on photochem device



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