

Strem Kit Manual

96-7530: Iridium/Nickel PhotoRedOx Base and Ligand Screening Kit 1

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

Sold in collaboration with HepatoChem

96-7530 EvoluChem™ Iridium/Nickel PhotoRedOx Base and Ligand 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.

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) / NiCl ₂ -dme / dtbbpy / Cs ₂ CO ₃	Ir/Ni-dtbbpy / Cs ₂ CO ₃	2 x vials	0.1 μmol / 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 ₃ PO ₄	Ir/Ni-dtbbpy / K ₃ PO ₄	2 x vials	0.1 μmol / 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 ₂ HPO ₄	Ir/Ni-dtbbpy / K ₂ HPO ₄	2 x vials	0.1 μmol / 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 ₂ CO ₃	Ir/Ni-dtbbpy / K ₂ CO ₃	2 x vials	0.1 μmol / 0.5 μmol / 0.5 μmol / 15 μmol
Ir[dF(CF ₃)ppy] ₂ (dtbbpy)[PF ₆] (Strem# 77-0425) / NiCl ₂ -dme / bphen / Cs ₂ CO ₃	Ir/Ni-bphen / Cs ₂ CO ₃	2 x vials	0.1 μmol / 0.5 μmol / 0.5 μmol / 15 μmol
Ir[dF(CF ₃)ppy] ₂ (dtbbpy)[PF ₆] (Strem# 77-0425) / NiCl ₂ -dme / bphen / K ₃ PO ₄	Ir/Ni-bphen / K ₃ PO ₄	2 x vials	0.1 μmol / 0.5 μmol / 0.5 μmol / 15 μmol
Ir[dF(CF ₃)ppy] ₂ (dtbbpy)[PF ₆] (Strem# 77-0425) / NiCl ₂ -dme / bphen / K ₂ HPO ₄	Ir/Ni-bphen / K ₂ HPO ₄	2 x vials	0.1 μmol / 0.5 μmol / 0.5 μmol / 15 μmol
Ir[dF(CF ₃)ppy] ₂ (dtbbpy)[PF ₆] (Strem# 77-0425) / NiCl ₂ -dme / bphen / K ₂ CO ₃	Ir/Ni-bphen / K ₂ CO ₃	2 x vials	0.1 μmol / 0.5 μmol / 0.5 μmol / 15 μmol

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Kit Contents (continued)

Description	Label	Quantity	Amount
Ir[dF(CF ₃)ppy] ₂ (dtbbpy)[PF ₆] (Strem# 77-0425) / NiCl ₂ -dme / (MeO) ₂ bpy / Cs ₂ CO ₃	Ir/Ni-(MeO) ₂ bpy/ Cs ₂ CO ₃	2 x vials	0.1 μmol / 0.5 μmol / 0.5 μmol / 15 μmol
Ir[dF(CF ₃)ppy] ₂ (dtbbpy)[PF ₆] (Strem# 77-0425) / NiCl ₂ -dme / (MeO) ₂ bpy / K ₃ PO ₄	Ir/Ni-(MeO) ₂ bpy/ K ₃ PO ₄	2 x vials	0.1 μmol / 0.5 μmol / 0.5 μmol / 15 μmol
Ir[dF(CF ₃)ppy] ₂ (dtbbpy)[PF ₆] (Strem# 77-0425) / NiCl ₂ -dme / (MeO) ₂ bpy / K ₂ HPO ₄	Ir/Ni-(MeO) ₂ bpy/ K ₂ HPO ₄	2 x vials	0.1 μmol / 0.5 μmol / 0.5 μmol / 15 μmol
Ir[dF(CF ₃)ppy] ₂ (dtbbpy)[PF ₆] (Strem# 77-0425) / NiCl ₂ -dme / (MeO) ₂ bpy / K ₂ CO ₃	Ir/Ni-(MeO) ₂ bpy/ K ₂ CO ₃	2 x vials	0.1 μmol / 0.5 μmol / 0.5 μmol / 15 μmol
Ir[dF(CF ₃)ppy] ₂ (dtbbpy)[PF ₆] (Strem# 77-0425) / NiCl ₂ -dme / biox / Cs ₂ CO ₃	Ir/Ni-biox/ Cs ₂ CO ₃	2 x vials	0.1 μmol / 0.5 μmol / 0.5 μmol / 15 μmol
Ir[dF(CF ₃)ppy] ₂ (dtbbpy)[PF ₆] (Strem# 77-0425) / NiCl ₂ -dme / biox / K ₃ PO ₄	Ir/Ni-biox/ K ₃ PO ₄	2 x vials	0.1 μmol / 0.5 μmol / 0.5 μmol / 15 μmol
Ir[dF(CF ₃)ppy] ₂ (dtbbpy)[PF ₆] (Strem# 77-0425) / NiCl ₂ -dme / biox / K ₂ HPO ₄	Ir/Ni-biox/ K ₂ HPO ₄	2 x vials	0.1 μmol / 0.5 μmol / 0.5 μmol / 15 μmol
Ir[dF(CF ₃)ppy] ₂ (dtbbpy)[PF ₆] (Strem# 77-0425) / NiCl ₂ -dme / biox / K ₂ CO ₃	Ir/Ni-biox/ K ₂ CO ₃	2 x vials	0.1 μmol / 0.5 μmol / 0.5 μmol / 15 μmol

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
07-0472	Bathophenanthroline (bphen)	1662-01-7	332.40
N/A	4-4'-Dimethoxy-2-2'-bipyridine ((MeO) ₂ bpy)	17217-57-1	216.24
N/A	2,2'-bis[(4S)-4-benzyl-2-oxazoline (biox)]	133463-88-4	320.39
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

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. 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 on next page.

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Typical Protocol (continued)

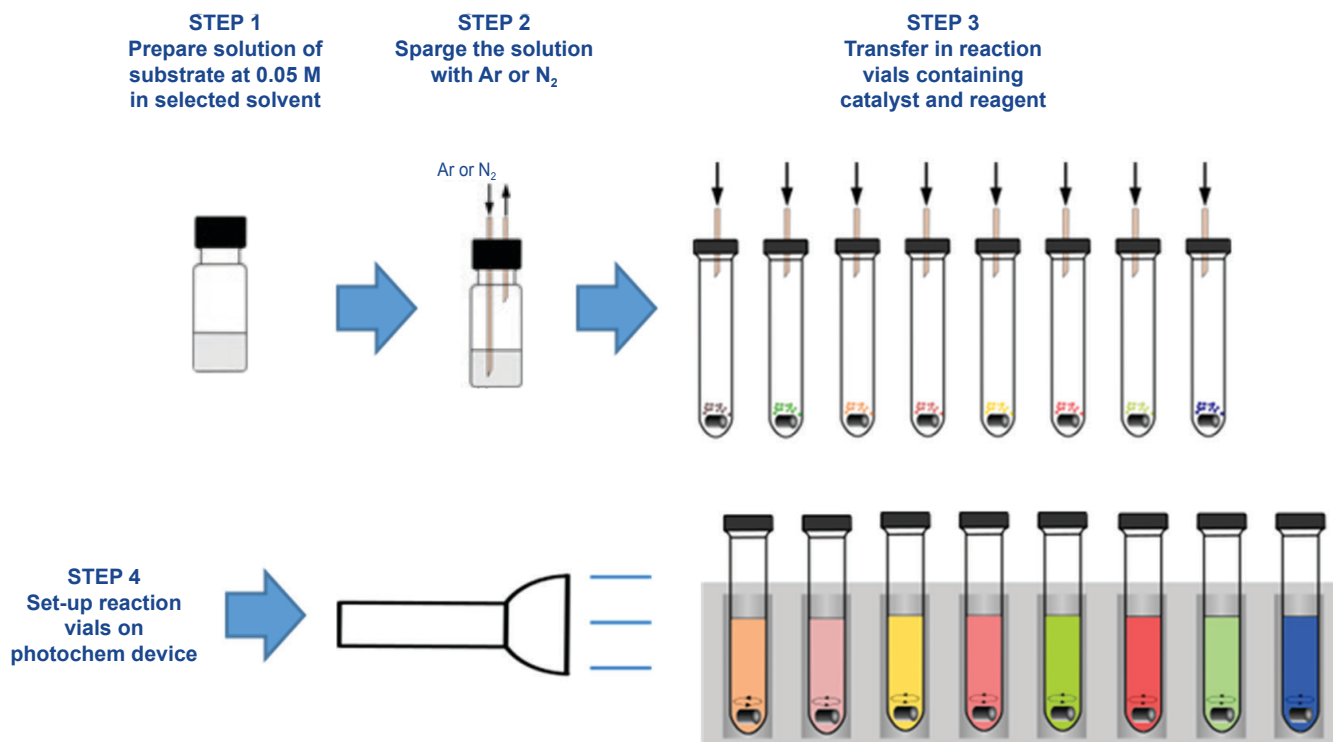
Conc. [M]	Vol. (μl)	Equiv. Ir Cat.	Equiv. Ni Cat	Equiv. base
0.050	100	0.02	0.10	3.0
0.050	200	0.01	0.05	1.5
0.025	200	0.02	0.10	3.0
0.025	100	0.04	0.20	6.0

- 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.
- Sparging reaction solvents with nitrogen or argon while transferring reagents is important to achieve highest conversions of product. See protocol diagram for instructions.
- The vial holder shipped with the kit can be placed directly on the photochemistry device.

Protocol at 100 μl volume reaction condition

1. Prepare the required volume of substrate solution at 0.05 mol/L containing both coupling substrates. For example, 1800 μl solution for 16 reaction conditions (200 μl extra to compensate for evaporation).
2. Degas substrate solution with subsurface sparging via N₂ or Ar line with exit needle for 5 minutes.
3. Using a clean and dry syringe, add 100 μl of the substrate solution to each reaction vial.
4. Stir the reaction vials for 5 minutes prior to turning on the light to allow catalysts to fully dissolve (some bases will remain insoluble).
5. Turn on lamp and stir vials for 18 to 24 hours (or longer if necessary). Be sure to plug in fan to maintain RT.
6. Upon completion of reaction, 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



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