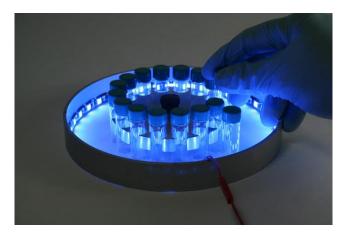
#### Lamps in Kanai laboratory







White LED (visible light) 株式会社ヴァロール製 http://www.valore.jp/led/vbl\_s.html



Aldrich LED (435 or 400 nm) http://www.sigmaaldrich.com/japan/labware /micro-photochemical-reactor.html

LED of specific wavelength (e.g. purple or green) 株式会社ヴァロール製 http://www.valore.jp/led/vbl\_s.html



UV LED (365 nm) 大興製作所製 http://www.daico.co.jp/products /products.php?id=21

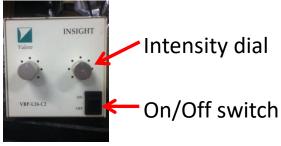
### Setup of photoreactions (visible light LED)



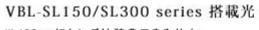
Reaction vessel

LED lamp

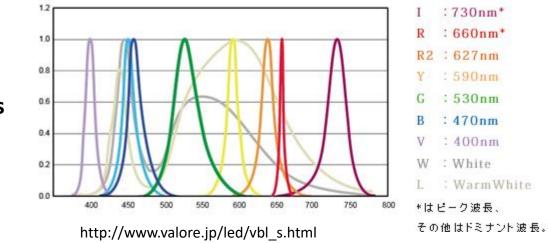
- Put the reaction vessel ca. 2 cm from the lamp.
- White LED is stored in a box behind the old glove box (4F refresh room).
- Light intensity can be modulated with 2 dials.



Controller of Valore LEDs



※ 400 nm にレンズは装着できません。



Spectra of Valore LEDs

#### Setup of photoreactions (UV)



Run reactions in the UV-cut box. (It is in the measurement room of 4<sup>th</sup> floor.)

[CAUTION!] UV is harmful. Never use UV lamps out of the box.

#### **<u>37-degree experiment</u>**

500 nm LEDs are used in Sohma group for biological study. They are stored in

- Incubator at 4F
- Eisai 1F

## <u>Tips</u>

[In general]

- Photoreactions should be run in a test tube or a screw vial. Flasks do not give good results.
- Large scale reactions usually result in poor yield.

## [Reaction in a screw vial]

- Vials can be put in a fraction collector (large), then in front of the lamp. (See appendix 3)
- Aldrich LED is suitable for a 4 mL screw vial.

#### [For visible light]

- White LED is recommended for initial trials.
- Light is too shiny for people around you. Put cartons or aluminum foils around the reaction.

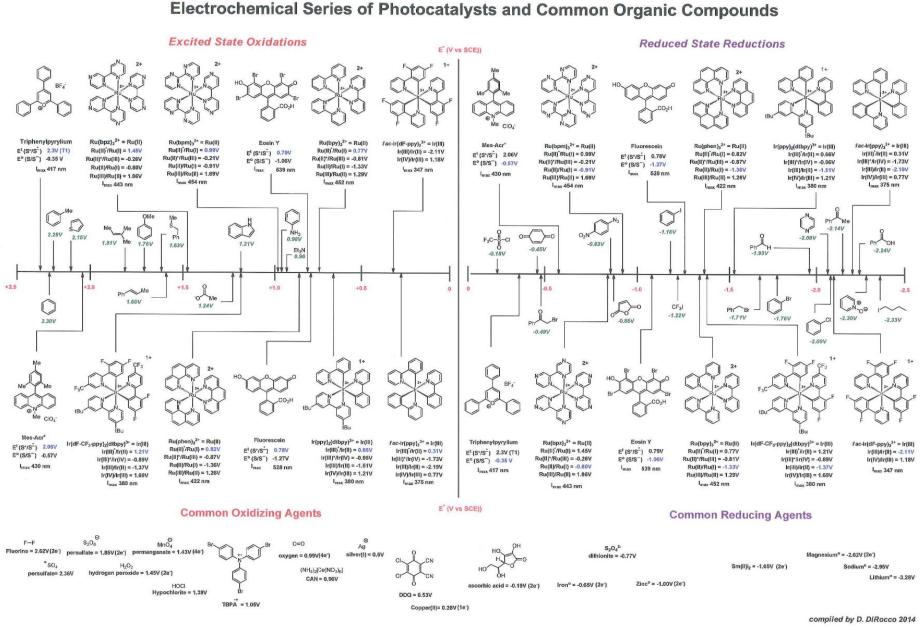
# [For UV]

- UV light is harmful, so be sure to use it inside the UV-cut box.
- Turn on the fan attached on the UV-cut box (in order to release the heat inside the box).

[Recommended reviews of photoredox catalysis]

- 有機合成化学協会誌 2014, 72, 538.
- *Chem. Rev.* **2013**, *113*, 5322. (with  $\lambda_{max}$  and  $E_{1/2}$  of representative photoredox catalysts)

# Appendix 1: Redox potential of common photocatalysts



http://brsmblog.com/wp-content/uploads/2014/09/Electrochemical-Series.pdf



#### Appendix 2: Absorption and excited state lifetime of common photocatalysts

entry	photo catalyst	excited-state lifetime, $\tau$ (ns)	$\begin{array}{c} \operatorname{excitation} \lambda_{\max} \\ (nm) \end{array}$	$\frac{\text{emission } \lambda_{\text{max}}}{(\text{nm})}$	ref
1	Ru(bpm)32+	131 <sup>b</sup>	454	639 <sup>b</sup>	161
2	Ru(bpz)32+	740	443	591	55
3	Ru(bpy)32+	1100	452	615	1, 3
4	Ru(phen)32+	500	422	610 <sup>c</sup>	1, 129
5	Ir[dF(CF <sub>3</sub> ) ppy] <sub>2</sub> (dtbbpy) <sup>+</sup>	2300	380	470	77
6	Ir(ppy)2(dtbbpy)*	557		581	58, 77
7	Cu(dap)2*	270		670 <sup>d</sup>	33
8	fac-Ir(ppy)3	1900	375	494 <sup>e</sup>	38

Modified from *Chem. Rev.* **2013**, *113*, 5322.

# Appendix 3: Photoreactions with many vials



-3 vials can be piled up in one fraction collector.