# MRG-059-ZP: Irradiation of [Ru(bpy)3]Cl2 * 6 H2O in the ChemSpeed robot - repetition of -ZN-20 and -ZO-7

**Date:** 2025-02-03
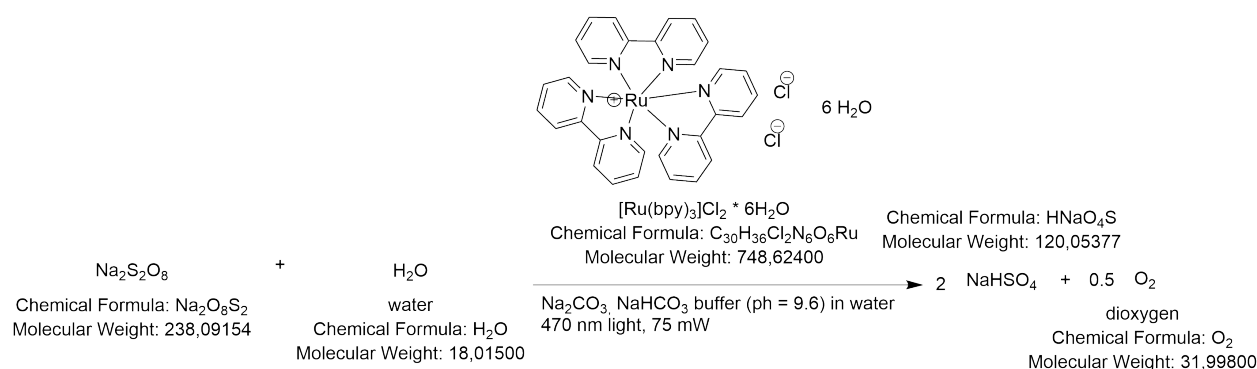**Tags:** Radiation  O2  [Ru(bpy)3]Cl2*6 H2O  AE  reference analytics  HTE  MRG
**Category:** HTE
**Status:** Done
**Created by:** Michael Ringleb

---

## Reaction scheme/sample structure



$[Ru(bpy)_3]Cl_2 * 6H_2O$
Chemical Formula: $C_{30}H_{36}Cl_2N_6O_6Ru$
Molecular Weight: 748,62400

Chemical Formula: $HNaO_4S$
Molecular Weight: 120,05377

$Na_2S_2O_8$ + $H_2O$ $\longrightarrow$ 2 $NaHSO_4$ + 0.5 $O_2$

Chemical Formula: $Na_2O_8S_2$
Molecular Weight: 238,09154

water
Chemical Formula: $H_2O$
Molecular Weight: 18,01500

$Na_2CO_3$, $NaHCO_3$ buffer (ph = 9.6) in water
470 nm light, 75 mW

dioxygen
Chemical Formula: $O_2$
Molecular Weight: 31,99800

## Literature/reference experiments

| Literature | https://doi.org/10.1021/acscatal.6b02595 |
|---|---|
| Reproduction | / |
| Related experiment | HTE - AE-434: Preparation of stock solutions for the irradiation of [Ru(bpy)3]Cl2 * 6 H2O<br>HTE - AE-404: Manufacturing, Test and calibration of HTE vial with O2 and T spot<br>HTE - AE-406: MRG-059-ZM: Irradiation of [Ru(bpy)3]Cl2 * 6 H2O in the ChemSpeed robot - reproduction of standard conditions - new vial<br>HTE - MRG-059-ZN: Irradiation of [Ru(bpy)3]Cl2 * 6 H2O in the ChemSpeed robot - screening of pH, [Ru], [Ox] and Irradiance<br>HTE - MRG-059-ZO: Irradiation of [Ru(bpy)3]Cl2 * 6 H2O in the ChemSpeed robot - repetition of experiments from -ZN with not reproducible results |

## Reagents/Parameters table

for content of the stated solutions see HTE - AE-434: Preparation of stock solutions for the irradiation of [Ru(bpy)3]Cl2 * 6 H2O

for calculations see:

| Run | Used [Ru] solution | V [Ru] [mL] | obtained conc. [µM] | Used [Ox] solution | V [Ox] [mL] | obtained conc. [mM] | Used [NaHCO3] solution | V [NaHCO3] [mL] | obtained conc. [M] | Used [Na2CO3] solution | V [Na2CO3] [mL] | obtained conc. [M] | obtained pH according to HTE - AE-413: Titration of Na2CO3 with NaHCO3 and vice versa | V [H2O] [mL] | power setting [V] | obtained power output [mW] AE-341: Power measurment with different power settings from Joy-It with 470 nm |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Ru II | 0.425 | 10 | Ox III | 0.425 | 6 | NaHCO3 II | 0.753 | 0.0886 | Na2CO3 II | 0.324 | 0.0114 | 9.2 | 6.573 | 0.18 | 75 |
| 2 | Ru II | 0.425 | 10 | Ox III | 0.425 | 6 | NaHCO3 II | 0.602 | 0.0708 | Na2CO3 II | 0.828 | 0.0292 | 9.2 | 6.221 | 0.18 | 75 |

`*final volume of all reaction solutions is 8.5 mL`

# Irradiation Parameters

Power measured using [Power Meter] 843-R-USB + 919P-020-12 unless specified otherwise.

| Oxygen sensor | Light Source Name | Wavelength [nm] | Power Setting [mW] | Analog input control voltage [V] using Equipment - Joy-it JT-RD6006 DC POWER SUPPLY |
|---|---|---|---|---|
| FireSting Fiber-Optic Oxygen Meter | Light Source - LCS-0470-50-22 | 470 | see table above | see table above |

| | |
|---|---|
| Used beam combiner [Name or None] | none |
| Irradiation distance [cm] | 9.5 |
| Thermostat temperature [°C] | / |
| Stirring speed [rpm] | 400 |
| Start time irradiation [s] | see csv/json |
| End time irradiation [s] | see csv/json |

# Procedure/observations

## Experiment was done by AE

| Date | Time | Step | Observations |
|---|---|---|---|
| 03.02 | 16:15 | The Calibration HTE - AE-404: Manufacturing, Test and calibration of HTE vial with O2 and T spot was used<br>The protocol for a fully automated workflow (Protocol - Operation of automated workflow for investigation of oxygen evolution) was utilized with a rare earth metal stir bar | |
| | - 16:35 | everything was set up according to the Protocol - Operation of automated workflow for investigation of oxygen evolution | |
| | | The python script is loaded as described in Protocol - Operation of automated workflow for investigation of oxygen evolution and the "experiment.yml" is initialized with four experiments (see reagents table for volumes of reactants) with the "run"-parameter = "true" and one at the end of the queue with "run" = "false" | |
| | | The python script is executed with "python run.py" | |
| | | AutoSuite program was started -->priming of tubings | |
| | 16:40 | **from this point on the execution was done fully automatic (except the changing of lids and refill of stock vials) according to the table with steps executed by the automated platform in the Protocol - Operation of automated workflow for investigation of oxygen evolution** | |
| | ca. 16:50 | MRG-059-ZP-1-1 was started | septum was pierced 0 times at this point |
| | ca. 18:10 | MRG-059-ZP-2-1 was started | at this point the setpum in the vial lid was pierced 9 times |
| | ca. 19:30 | MRG-059-ZP-1-2 was started | at this point the setpum in the vial lid was pierced 18 times |
| | ca. 20:50 | MRG-059-ZP-2-2 was started | at this point the setpum in the vial lid was pierced 27 times |
| | ca. 22:10 | MRG-059-ZP-1-3 was started | at this point the setpum in the vial lid was pierced 36 times |

| | approx. 23:30 | ZP-1-3 end | | at this point the setpum in the vial lid was pierced 45 times |
|---|---|---|---|---|

# Results

## Analysis

done with hte_streamlit (https://github.com/jschneidewind/hte-streamlit)

Standard conditions look good.


max rate ydiff by Experiment Group

### pH = 9.2


max rate ydiff by Experiment Group

-ZP-1-1 looks like outlier, other two are at 2.1. In comparison to -ZO-7: ok fit (-7 has rate of 2.24 and 2.33) --> should be in margin of error. -20 much higher rate (approx. 2.9) does not fit to other experiments, therefor likely double fluke or error in run.

Highlights improtance of addition of known conditions in each run.

## Linked experiments

- AE-262: Irradiation of PhPDA (AE-257), 1.5 mg/mg SDS, 2 mg/mL PhPDA after 3 d

- 1st screening of Ru-cat concentration

- MRG-059-W: Preparation of stock solutions for the irradiation of [Ru(bpy)3]Cl2 * 6 H2O screenings (MRG-059-X, -Y, -Z)

- MRG-059-X: Irradiation of [Ru(bpy)3]Cl2 * 6 H2O in the ChemSpeed robot, fully automated peripherals - 1st screening of sacrificial oxidant

- MRG-059-Y: Irradiation of [Ru(bpy)3]Cl2 * 6 H2O in the ChemSpeed robot, fully automated peripherals - 2nd screening of [Ru]

- MRG-059-Z: Irradiation of [Ru(bpy)3]Cl2 * 6 H2O in the ChemSpeed robot, fully automated peripherals - 1st screening of pH (9.2 - 10.4)

- MRG-059-ZA - Irradiation of [Ru(bpy)3]Cl2 * 6 H2O in the ChemSpeed robot - 2nd pH screening (5.8 -12)

- AE-341: Power measurment with different power settings from Joy-It with 470 nm

- AE-342: Preparation of stock solutions for the irradiation of [Ru(bpy)3]Cl2 * 6 H2O I

- MRG-059-ZB: Irradiation of [Ru(bpy)3]Cl2 * 6 H2O in the ChemSpeed robot, fully automated peripherals - screening of light intensity

HTE - MRG-059-ZI: Irradiation of [Ru(bpy)3]Cl2 * 6 H2O in the ChemSpeed robot, fully automated peripherals - screening of light intensity I

HTE - MRG-059-ZJ: Irradiation of [Ru(bpy)3]Cl2 * 6 H2O in the ChemSpeed robot, fully automated peripherals - reproduction of standard conditions - new vial (with ruthenium stock solution or directly weighed in)

HTE - AE-379: Test and calibration of O2 vial for O2 and T measurment in irrad setup

HTE - AE-383: Preparation of stock solutions for the irradiation of [Ru(bpy)3]Cl2 * 6 H2O

HTE - MRG-AE-059-ZK: Irradiation of [Ru(bpy)3]Cl2 * 6 H2O in the ChemSpeed robot, fully automated peripherals - reproduction of standard conditions - new vial, new T calibration

HTE - AE-MRG-059-ZL: Irradiation of [Ru(bpy)3]Cl2 * 6 H2O in the ChemSpeed robot - reproduction of standard conditions - new calibration

HTE - AE-404: Manufacturing, Test and calibration of HTE vial with O2 and T spot

HTE - AE-405: Preparation of stock solutions for the irradiation of [Ru(bpy)3]Cl2 * 6 H2O I

HTE - AE-406: MRG-059-ZM: Irradiation of [Ru(bpy)3]Cl2 * 6 H2O in the ChemSpeed robot - reproduction of standard conditions - new vial

PDF generated with elabftw, a free and open source lab notebook
File generated on 10-04-2025 at 10:13
6 / 7

HTE - AE-413: Titration of Na2CO3 with NaHCO3 and vice versa

HTE - AE-414: Preparation of stock solutions for the irradiation of [Ru(bpy)3]Cl2 * 6 H2O

HTE - MRG-059-ZN: Irradiation of [Ru(bpy)3]Cl2 * 6 H2O in the ChemSpeed robot - screening of pH, [Ru], [Ox] and Irradiance

HTE - AE-422: Preparation of stock solutions for the irradiation of [Ru(bpy)3]Cl2 * 6 H2O

HTE - MRG-059-ZO: Irradiation of [Ru(bpy)3]Cl2 * 6 H2O in the ChemSpeed robot - repetition of experiments from -ZN with not reproducible results

HTE - AE-434: Preparation of stock solutions for the irradiation of [Ru(bpy)3]Cl2 * 6 H2O

## Linked items

Equipment - Irradiation Set-Up

Equipment - Joy-it JT-RD6006 DC POWER SUPPLY

Light Source - UHP LED 470 nm

Protocol - Operation of automated workflow for investigation of oxygen evolution - as of 03.07.2024

## Attached files

newplot26.png
sha256: 214b00a9a03b5c3b43c1c09ba8719e8c6946ec62847728ce0a1c8985e276dcd6

newplot25.png
sha256: 36983452c79904491516ce428a658a4af036ae8e75b1b15a6e256e7f4dc371fd

MRG-059-ZP.zip
sha256: c98ff57741ba1c710b731e9085a187109fa615630e48dc0f48cc6d0911f1fd9a

AE-434-HTE.xlsx
sha256: 479edddc401588aeae7aa62c4f774069efb8adca818817665c66e8e8a9bf441f