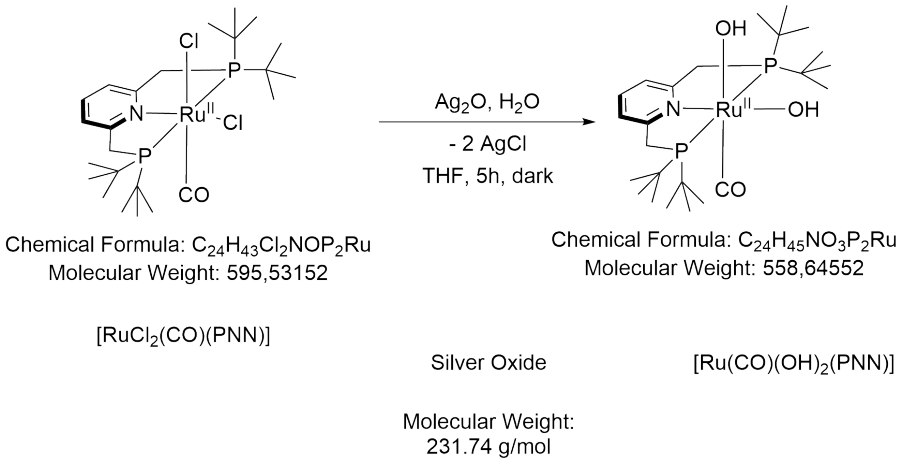
AE-456: Synthesis of [Ru(CO)(OH)2(PNPtBu)] using Ag2O, 5 h

Date: 2025-03-04
Tags: Dihydroxo Ag2O PNP P(tBu)N(py)P(tBu) [Ru(CO)(OH)2(PNP)] NMR ATR-IR AE Future reference analytics 1H 31P IR Elemental Analysis
Category: Two-photon
Status: Done
Created by: Alexander Eith

Reaction scheme/sample structure



Chemical Formula: $C_{24}H_{43}Cl_2NOP_2Ru$
Molecular Weight: 595,53152

$[RuCl_2(CO)(PNN)]$

Silver Oxide

Molecular Weight: 231.74 g/mol

Chemical Formula: $C_{24}H_{45}NO_3P_2Ru$
Molecular Weight: 558,64552

$[Ru(CO)(OH)_2(PNN)]$

Literature/reference experiments

Literature	https://doi.org/10.1039/D1EE01053K
Reproduction	/
Related experiments	Experiment - AE-KRA-204: Synthesis of [Ru(CO)(OH)2(PNPtBu)] using Ag2O, 5 h Experiment - AE-238: Synthesis of [Ru(CO)(OH)2(PNPtBu)] using Ag2O, 5 h Experiment - AE-252: Synthesis of [Ru(CO)(OH)2(PNPtBu)] using Ag2O, 5 h AE-310: Synthesis of [Ru(CO)(OH)2(PNPtBu)] using Ag2O, 5 h Two-photon - AE-382: Synthesis of [Ru(CO)(OH)2(PNPtBu)] using Ag2O, 5 h

Reagents

Name	CAS-Number/Expermient-Number	Amount [mmol]	Equivalents	$Mass_{theo}$ [mg]	$Mass_{exp}$ [mg]	Molar mass [g/mol]	$Volume_{theo}$ [ml]	$Volume_{exp}$ [ml]	Density [g/mL]
[RuCl2(CO)(PNP{tBu})]	Two-photon - KRA-DS-02: Synthesis of [RuCl2(CO)(PNP)] with 2,6-Bis((tert-butyl)phosphinomethyl)pyridine via [RuCl2(CO)(p-cymene)] I	0.42	1.00	250.00	248.84	595.53	/	/	/
Ag_2O	20667-12-3	2.58	6.14	597.32	596.73	231.74	/	/	/
H2O	Prep work - AE-442: Degassing of Milli-Q water	98.23	234	1770.13	/	18.02	1.774	1.8	0.998
THF	Prep work - AE-396: Degassing of THF	/	/	/	/	/	12.5	13	/

Work-up Reagents

Name	CAS Number / Expermient-Number	Inventory number	$Mass_{exp}$ [g]	Volume [ml]	Concentration [M]
THF	Prep work - AE-396: Degassing of THF	/	/	13.5	/
n-heptane	AE-315: Degassing of n-Heptane	/	/	20 + 4.5	/

Procedure/observations

All steps, unless mentioned otherwise, were carried out under argon using standard Schlenk technique or in an argon filled glovebox. (see Protocol Protocol - Schlenk Technique)

Due to the instability of Ag_2O and Dihydroxo to the light, the procedure was done mostly in the dark, by either wrapping the flasks containing either or both in Aluminium foil or by tuning off the light in the lab and fumehood

Date	Time	Step	Photos	Observations
04.03		A 25 mL brown glas Schlenk flask was prepared, by heating under vacuum		
	9:35	$[RuCl_2(CO)(PNP)]$ and Ag2O was weighted in air into the reaction schlenk flask and the flask was set under argon.		
		The light in the fumehood was turned out		
	10:04	THF (13 mL) was added to the reaction schlenk flask.		
	10:05	Water (1.8 mL) was added to the reaction schlenk flask		
	10:05 - 15:05	The reaction mixture was stirred for 5 h at 550 rpm. During this the flask was wrapped with aluminium foil		
	15:05 - 15:07	The reaction mixture was allowed to settle		
	15:10 - 15:15	The mixture was filtered through a pore 3 frit according to Protocol - Filtration with frit technique into a 100 mL-Schlenk	1st dilter residue.jpg 1st filtrate.jpg	Significant amounts of the solid went through the filter frit, dark solid, purple/black filterate
		A new 100 mL flask was prepared		
	15:35 - 15:42	The obtained suspension was filtered through a PTFE syringe filter (pore diameter: 0.2 µM) in the new 100 mL flask	after filtration through PTFE filter.jpg	dark violet, clear solution

	15:47 - 15:55	The solvents of the filtrate were removed under reduced pressure.		approx. 2.5 mL remained in the flask
		The water bath was removed		
	15:55 - 16:08	The remaining solvents were removed without water bath, trying to freeze the solution.	after removing water bath _ bad quality.jpg	Did not work, remained liquid. Bubbeled up during drying, still the sovlents were removed und a solid was obtained Purple suspension
	16:08 - 16:15	The obtained solid was dried under reduced pressure	after removing solvents.jpg	Pruble brownhish film
		The solid was stored in the dark at rt		
05.03	11:00 - 11:15	The obtained solid was dried under reduced pressure		
	11:17	To the solid THF (13.5 mL) was added und the obtained mixture was shaken strongly for approx. 2 min and than stirred for approx. 8 min at 665 rpm	in THF.jpg	purple brwonish suspension
	11:27	n-heptane (20 mL) was added to the mixture	after addition of heptane.jpg	purple solution, brownish solid
	11:30 - 15:00	The mixture was filtered through a pore 4 frit according to Protocol - Filtration with frit technique into a 100 mL-Schlenk		very slow filtration, but still working.
	15:00 - 15:07	The solid was washed with n-heptane (3 * 1.5 mL)	2nd filtrate.jpg	purple solution
	15:10 - 17:30	The solid was dried under reduced pressure	product.jpg	
		The solid was stored in Equipment - Freezer Lab E004 CEEC II at -20 °C in the dark under Argon		
11.03	9:00	The solid was transfferred into a 2 mL vial in a 10 mL Schlenk flask		Some material was lossed, since it sticked stongly to any surface
		During this an aliqoute (approx. 2 mg) was transfferred into another 10 mL Schlenk flask		
	9:15	Both flask were set under argon		

	13:35	From the aliquote n NMR sample was prepared in MeCN-d3 (0.7 mL) according to Protocol - Preparation of NMR Sample	NMR sample.jpg	**AE-456-1** yellowish solution
01.04		5 mL NS 14 schlenk flask was prepared		
	10:10	To the flask AE-456-1 (approx. 15 mg) was added		
		The flask was set under argon		
	10:33	To the flask MeCN-d3 (0.7 mL) was added		
	10:34	The flask was stirred at 500 rpm	dissolved.jpg	
	10:38	The NMR was prepared according to Protocol - Preparation of NMR Sample	in NMR.jpg	**AE-456-1-1**
02.04	15:20	**AE-456-1-1** was submitted	before NMR.jpg	
04.06	09:40	IR spectra where mesured according to Protocol - IR measurements, Lab E007, CEEC I using the Equipment - Fourier Transform Infrared Spectrophotometer Shimadzu IRSpirit Several conditions for the measurment where tried: * AE-456-1-IR-bkg: background measurment * AE-456-1-IR-1: resolution: 4 cm^-1, Absorbtion mode, 30 scans * AE-456-1-IR-2: resolution: 4 cm^-1, Absorbtion mode, 30 scans * AE-456-1-IR-3: resolution: 8 cm^-1, Absorbtion mode, 30 scans * AE-456-1-IR-4: resolution: 8 cm^-1, Absorbtion mode, 30 scans * AE-456-1-IR-5: resolution: 4 cm^-1, Absorbtion mode, 30 scans * AE-456-1-IR-6: resolution: 16 cm^-1, Absorbtion mode, 30 scans		
21.07	11:20	Two flask (flask 1 and 2) were prepared equipped each with a 2 mL screw cap vial		
	11:40	To flask 1 AE-456-1 (3.5 mg) was added		**AE-456-EA**
	11:45	To flask 2 AE-456-1 (approx. 4 mg) was added		**AE-456-IR-a**
	13:20	From **AE-456-IR-a** IR spectrums were meausred in air by Akuila Edwards (AG Schmitty)		
	14:00	**AE-456-EA** was submitted for elemental analysis		
22.07	/	The elemental analysis of **AE-456-EA** was measured by Sandra Köhn		

Analysis

Date	Time	Sample name	Analysis method	Used device	Solvent	Raw Data	Processed Data	Comparative Data	Interpretation
12.03	11:12	AE-456-1	NMR 1H, 31P{1H}quant	IAAC 400 MHz I	MeCN-d3	AE-456-1.zip	AE-456-1_10.nmrium	Two-photon - AE-382: Synthesis of [Ru(CO)(OH)2(PNPtBu)] using Ag2O, 5 h (**AE-382-1**)	As expected. Purer than AE-382, very few impurities, also in 1H

02.04	15:26	AE-456-1-1	NMR 1H, 13C{1H}, 31P{1H}, 13C{1H}-APT, 1H/1H-COSY, 1H/13C{1H}-HMBC, 1H/13C{1H}-HSQC, 1H/31P{1H}-HMBC	IOMC 500 MHz	MeCN-d3	AE-456-1-1-1H.zip AE-456-1-1-13C.zip AE-456-1-1-31P.zip AE-456-1-1-31P-HMBC.zip AE-456-1-1-HMBC.zip AE-456-1-1-HSQC.zip AE-456-1-1-COSY.zip AE-456-1-1-13C-APT.zip	AE-456-1-1-1H-13C-31P-APT.nmrium AE-456-1-1-31P-HMBC.nmrium AE-456-1-1-COSY.nmrium AE--456-1-1-HMBC-HSQC.nmrium	AE-456-1	Full evaluation pending. 1H and 31P fit with previous 13C: 2 weak tripletts for CH3 groups, 3 tripletts for Cq(CH3) and CH2, 4 signals in aromatic area, 3 expected, 1 signal for CO (very weak) 31P HMBC: as expected, no other signals observed 13C HSQC: CH3 and Ch2 groups observed, aromatic as well 13C HMBC: CH3/CH2 coupling observed, CH2/Cq(CH3) ?, CH2/Cq(Ar) observed, no ther coupling apparent in Aromatic are at first glance COSY: Aromatics with each other, CH2 with each other, CH3 with itself
04.06	09:40	AE-456-1-IR	ATR-IR	Protocol - IR measurements, Lab E007, CEEC I	/	AE-456-1-IR-bkg.txt AE-456-1-IR-1.txt AE-456-1-IR-2.txt AE-456-1-IR-3.txt AE-456-1-IR-4.txt AE-456-1-IR-6.txt AE-456-1-IR-2.ispd AE-456-1-IR-3.ispd AE-456-1-IR-4.ispd AE-456-1-IR-5.ispd AE-456-1-IR-6.ispd	Used for plotting: plot_ir_spectra.py AE-456-1-IR-all.png AE-456-1-IR-transmission.png AE-456-1-IR-good_spectra.png Used for plotting and evaluation: IR_single_spectra.py AE-456-1-IR-3.png AE-456-1-IR-3_peaks.csv AE-456-1-IR-4.png AE-456-1-IR-good_spectra.png	/	In absorbance mode no good spectra was obtained In transmission mode good spectra were obtained only with a resolution of 8 cm^-1, with higher and lower resolution artefacts (like positive peaks where observed) -3 and -4 are very similar (also same experimental conditions). For them full evalutaion was done. CO band can be clearly seen, in water area some distinct peaks. In gneral ok signal strenght (down to 65 % transmission).
21.07	13:20	AE-456-IR-a (Name in file: Eith_a)	ATR-IR with diamond	IR from AG Schmitt (measured by akuila)	/	Eith_a.CSV	Used for plotting: IR_new.py Eith_a_peaks.csv AE-456-IR_Eith_a.png	AE-456-1-IR Used background: Background-Mon-Jul-21-13-17-01-2025-GMT02-00.CSV	Better signal to noise ratio, still quite weak signals In general very similar to AE-456-1-IR
21.07	13:20	AE-456-IR-a (Name in file: Eith_a1_200scans)	ATR-IR with diamond	IR from AG Schmitt (measured by akuila)	/	Eith_a1_200scans.CSV	Used for plotting: IR_new.py AE-456-IR_Eith_a1_200scans.png Eith_a1_200scans_peaks.csv	AE-456-1-IR Used background: Background-Mon-Jul-21-13-17-01-2025-GMT02-00.CSV	Better signal to noise ratio, still quite weak signals Best quaility IR so far In general very similar to AE-456-1-IR
22.07	/	AE-456-EA (Name in file: AE-456EA)	elemental analysis	elemental analysis from AG Schubert (measured by Sandra Köhn)	/	AE-456-EA.jpg	C: 50.48 % H: 7.13 % N: 2.68 %	C: 51.6 % H: 8.12 % N: 2.51 % With additional 1 H2O: C: 49.99 % H: 8.22 % N: 2.43 With additional CO2 - H2O C: 51.36 H: 7.41 N: 2.40 Further possible molecules: AE-456-EA-calc.xlsx Old data: Two-photon - AE-382: Synthesis of [Ru(CO)(OH)2(PNPtBu)] using Ag2O, 5 h	Significant error compared to theoretical composition, espacially H has to large deviation. Can also not be explained by addition of not carbon containing molecules to lower H content in theoretical values. But at least significant better than AE-382-EA Other impurities cannot help, free ligand and coordinated/crystalized MeCN, THF, n-heptane all would yield increased C and H content. Inorganic compounds (like Ag2O) do help, with 2.5 % we get C: 50.31 %, H: 7.92 % and N: 2.45 %, C and N good, but H still to high, more Ag2O: unrealistic and yields to low C and N No realistic impurity found to explain deviations in elemental analysis. Maybe error in measurment, but unlikely Errors up to 1 % can realistic appear for pure, simple organic compounds: https://doi.org/10.1021/acscentsci.2c00325, just by random error, will be larger if sample is not 100 % pure and more deficil like here

Product characterization and results

Sample	Mass [mg]	Purity	$Mass_{pure}$ [mg]	Amount [µmol]	Yield [%]	Description	Storage location
AE-456-1	91.32	> 97 %	/	0.163	39	greenish grayish solid, product.jpg	Equipment - Freezer Lab E004 CEEC II A

Future recommendations

Old procedure	Problem	Suggested new procedure
1st filt through pore 3	not all solids removed	use pore 4 or 5

Linked experiments

- AEI-010: Synthesis of [Ru(CO)(OH)2(PNN)] using Ag2O

- JSC-KS-04: Degassing of n-Heptane
- AEI-024: Synthesis of [Ru(CO)(OH)2(NNN)] with 2,6-bis(N,N-diethylaminomethyl) using Ag2O
- AEI-026: Capillary production
- AEI-048: Synthesis of [Ru(CO)(OH)2(NNN)] with 2,6-bis(N,N-diisopropylaminomethyl) using Ag2O
- AEI-064: Synthesis of [Ru(CO)(OH)2(PNP)] with 2,6-bis(P,P-di(tertbutyl)phosphinomethyl) using Ag2O
- AEI-073: Synthesis of [Ru(CO)(OH)2(PNP)] with 2,6-bis(P,P-di(tertbutyl)phosphinomethyl) using Ag2O
- AE-101: Synthesis of [RuCl2(CO)(PNP)] with 2,6-Bis((tert-butyl)phosphinomethyl)pyridine via [RuCl2(CO)(p-cymene)]
- AE-102: Degassing of THF
- AE-105: Synthesis of [Ru(CO)(OH)2(PNP)] with 2,6-bis(P,P-di(tertbutyl)phosphinomethyl) using Ag2O
- AE-108: Synthesis of [RuCl2(CO)(PNP)] with 2,6-Bis((tert-butyl)phosphinomethyl)pyridine via [RuCl2(CO)(p-cymene)]
- AE-114: Synthesis of [Ru(CO)(OH)2(PNP)] with 2,6-bis(P,P-di(tertbutyl)phosphinomethyl) using Ag2O
- AE-121: Degassing of n-Heptane
- AE-145: Synthesis of [Ru(CO)(OH)2(PNP)] with 2,6-bis(P,P-di(tertbutyl)phosphinomethyl) using Ag2O
- AE-152: Degassing of MeCN
- AE-157: Synthesis of [RuCl2(CO)(PNP)] with 2,6-Bis((tert-butyl)phosphinomethyl)pyridine via [RuCl2(CO)(p-cymene)]
- AE-167: Irradiation of PhPDA (365 nm + white LED, gas phase measurement), AE-151, 1.5 mg/mg SDS, 2 mg/mL PhPDA
- AE-198: Synthesis of [Ru(CO)(OH)2(PNP{tBu})] with Ag2O with different reaction times (1, 2, 5, 24 h)
- AE-KRA-204: Synthesis of [Ru(CO)(OH)2(PNPtBu)] using Ag2O, 5 h
- AE-208: Degassing of Milli-Q water
- KRA-080: Degassing of THF
- KRA-104: Degassing of Milli-Q water
- AE-230: Synthesis of [RuCl2(CO)(PNP)] with 2,6-Bis((tert-butyl)phosphinomethyl)pyridine via

[RuCl2(CO)(p-cymene)]

- AE-232: Synthesis of [RuCl2(CO)(PNP)] with 2,6-Bis((tert-butyl)phosphinomethyl)pyridine via [RuCl2(CO)(p-cymene)]

- AE-238: Synthesis of [Ru(CO)(OH)2(PNPtBu)] using Ag2O, 5 h

- AE-246: Radiation of [Ru(CO)(OH)2(PNP{tBu})] (AE-238, 97 % pure) 365 nm and white

- AE-252: Synthesis of [Ru(CO)(OH)2(PNPtBu)] using Ag2O, 5 h

- AE-261: Synthesis of [RuCl2(CO)(PNP)] with 2,6-Bis((tert-butyl)phosphinomethyl)pyridine via [RuCl2(CO)(p-cymene)]

- AE-281: Stability test of [Ru(CO)(OH)2(PNP{tBu})] (AE-252) under air and afterwards irradiation with 365 nm and white

- AE-310: Synthesis of [Ru(CO)(OH)2(PNPtBu)] using Ag2O, 5 h

- AE-315: Degassing of n-Heptane

Prep work - AE-396: Degassing of THF

Prep work - AE-442: Degassing of Milli-Q water

Two-photon - AE-382: Synthesis of [Ru(CO)(OH)2(PNPtBu)] using Ag2O, 5 h

Two-photon - KRA-DS-02: Synthesis of [RuCl2(CO)(PNP)] with 2,6-Bis((tert-butyl)phosphinomethyl)pyridine via [RuCl2(CO)(p-cymene)] I

Linked resources

Equipment - Fourier Transform Infrared Spectrophotometer Shimadzu IRSpirit

Equipment - Freezer Lab E004 CEEC II

Protocol - Preparation of NMR Sample

Protocol - Filtration with frit technique

Protocol - Schlenk Technique

Protocol - Preparation of capillary for H2O-NMR

Protocol - IR measurements, Lab E007, CEEC I

Attached files

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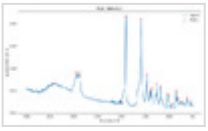

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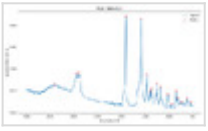

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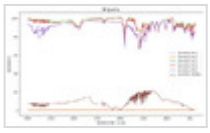

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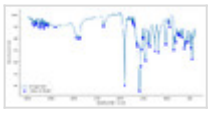

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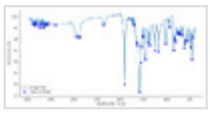

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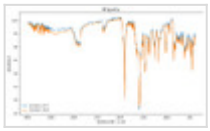

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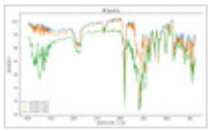

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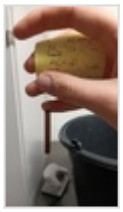

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after-filtration-through-PTFE-filter.jpg
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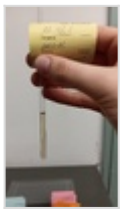

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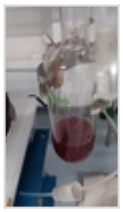

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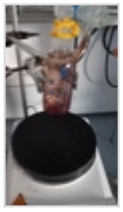

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