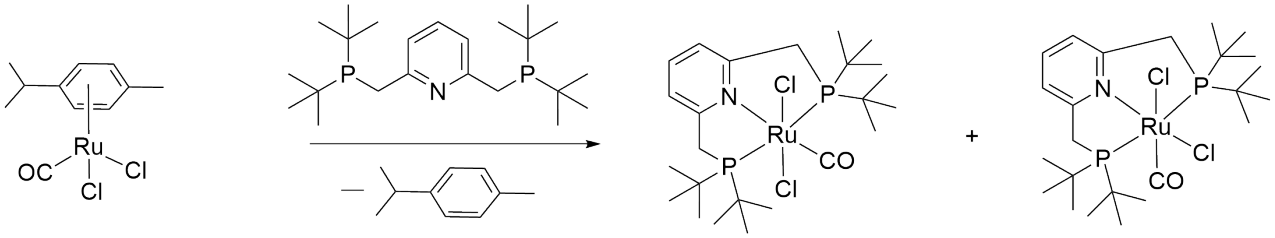
# AE-261: Synthesis of [RuCl2(CO)(PNP)] with 2,6-Bis((tert-butyl)phosphinomethyl)pyridine via [RuCl2(CO)(p-cymene)]

**Date:** 2024-04-18
**Tags:** [RuCl2(CO)(p-cymene)] Dichloro PNP [RuCl2(CO)(PNP)] P(tBu)N(py)P(tBu) NMR AE 1H 31P
**Status:** Done
**Created by:** Alexander Eith

## Reaction scheme/sample structure




[$RuCl_2(CO)(p\text{-cymene})$]

Molecular Weight: 334.20 g/mol

2,6-bis((di-*tert*-butylphosphaneyl)methyl)pyridine
Chemical Formula: $C_{23}H_{43}NP_2$
Molecular Weight: 395,55152

$[RuCl_2(CO)(P^{tBu}N^{py}P^{tBu})]$

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

## Literature/reference experiments

| | |
|---|---|
| **Literature** | https://doi.org/10.1039/D1EE01053K |
| **Reproduction** | / |
| **Related experiments** | Experiment - AE-157: Synthesis of [RuCl2(CO)(PNP)] with 2,6-Bis((tert-butyl)phosphinomethyl)pyridine via [RuCl2(CO)(p-cymene)]<br>Experiment - AE-230: Synthesis of [RuCl2(CO)(PNP)] with 2,6-Bis((tert-butyl)phosphinomethyl)pyridine via [RuCl2(CO)(p-cymene)]<br>Experiment - AE-232: Synthesis of [RuCl2(CO)(PNP)] with 2,6-Bis((tert-butyl)phosphinomethyl)pyridine via [RuCl2(CO)(p-cymene)] |

## Reagents

| Name | Amount [mmol] | Equivalents | Purity | $Mass_{theo,pure}$ [mg] | $Mass_{theo}$ [mg] | $Mass_{exp}$ [mg] | Molar mass [g/mol] | Volume [ml] |
|---|---|---|---|---|---|---|---|---|
| [$RuCl_2(CO)(p\text{-cymene})$] ( Experiment - AE-248: Synthesis of [RuCl2(CO)(p-cymene)] | 1.53 | 1.00 | 0.77 | 505 | 657 | / | 334.20 | / |
| PNP, 2,6-Bis(N,N-(tert-butyl)phosphinomethyl)pyridine ( Experiment - AE-253: Synthesis of 2,6-Bis(P,P-di(tertbutyl)phosphinomethyl)pyridine | 1.91 | 1.25 | 0.90 | 756 | 840 | 840 | 395.55 | / |
| THF ( Experiment - KRA-080: Degassing of THF | / | / | / | / | / | / | / | 70 |
| n-heptane ( Experiment - AE-120: Degassing of n-Heptane | / | / | / | / | / | / | / | 75 |

## 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)

| Date | Time | Step | Observations |
|---|---|---|---|
| 18.04 | | A 150 mL Schlenk flask was prepared | |
| | 14:40 | The air cooler was dried by setting under vacuum | approx. 1:30 h |
| | 15:50 | PNP(tBu) (840 mg) was weighted in the Glovebox K004 into the 150 mL Flask | white solid, PNPtBu.jpg |
| | 16:15 | The whole product from AE-248: Synthesis of [RuCl2(CO)(p-cymene)] was used for the reaction | Ru.jpg |
| | 16:20 | The flask which contained the [Ru] was washed with THF (2 * apprx. 10 mL) and the obtained solutions were transferred into the 150 mL flask | in-THF.jpg |
| | - 16:30 | To the reaction flask THF (50 mL) was added | |
| | 16:35 | The reaction mixture was stirred at rt and the flask was conected to the cooler in argon counterflow. | A red solution is obtained |
| | 16:40 - 17:10 | The reaction micture was heated to reflux | |
| | 17:10 - | The reaction mixture was heated to reflux for approx. 15:20 h at 350 rpm. | at-reflux.jpg<br>after-reaction-at-reflux.jpg |
| 19.04 | - 8:30 | The reaction mixture was cooled down to r.t. | at-rt.jpg |
| | 10:15 - 10:35 | The volume of the remaining reaction mixture was reduced under reduced pressure to approx. 35 mL | reduced-volume.jpg |
| | 11:15 - 11:30 | To the solution n-Heptane (60 mL) was added under stirring. | after-adding-n-hept.jpg |
| | 13:10 - 13:45 | The obtained mixture was filtered according to Filtration with frit technique and the residue was washed with n-Heptane (3 * 5 mL) | start-of-filtration.jpg |
| | 13:47 - 13:55 | The obtained solid was dried under reduced pressure. | after first drying.jpg |

| | | | |
|---|---|---|---|
| 22.04 | 10:15 - 10:55 | The obtained solid was dried under reduced pressure. | on fritjpg |
| | 11:30 | A NMR-sample was prepared from AE-261-1 according to [Protocol] Preparation of NMR Sample | NMR.jpg |
| 26.04 | 10:30 | The solid was transffered into a 10 mL Flask | **AE-261-1** |

## Analysis

| Date | Time | Sample name | Analysis method | Used device | Solvent | Raw Data | Processed Data | Comparative data | Interpretation |
|---|---|---|---|---|---|---|---|---|---|
| 22.04 | 22:26 | AE-261-1 | 1H, 31P (quant) | IAAC 400 MHz I | DCM-d2 | AE-261-1_10.zip | AE-261-1_10.nmrium | Experiment - AE-230: Synthesis of [RuCl2(CO)(PNP)] with 2,6-Bis((tert-butyl)phosphinomethyl)pyridine via [RuCl2(CO)(p-cymene)] -2 | Desired product at 65 ppm. Purity according to 31P of 94 % |

## Product characterization

| | mass [mg] | purity [%] | $mass_{pure}$ [mg] | amount [mmol] | Yield [%] | |
|---|---|---|---|---|---|---|
| AE-261-1 | 825.60 | 94 | 776.06 | 1.30 | 86 | greenish solid, prod.jpg |

## Linked experiments

- JSC-KS-04: Degassing of n-Heptane
- AEI-047: Synthesis of 2,6-Bis(P,P-di(tertbutyl)phosphinomethyl)pyridine
- AEI-057: Synthesis of [RuCl2(CO)(p-cymene)]
- AEI-060: Synthesis of [RuCl2(CO)(PNP)] with 2,6-Bis((tert-butyl)phosphinomethyl)pyridine via [RuCl2(CO)(p-cymene)]
- AEI-072: Synthesis of [RuCl2(CO)(PNP)] with 2,6-Bis((tert-butyl)phosphinomethyl)pyridine via [RuCl2(CO)(p-cymene)]
- AE-100: Synthesis of [RuCl2(CO)(p-cymene)]
- 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-108: Synthesis of [RuCl2(CO)(PNP)] with 2,6-Bis((tert-butyl)phosphinomethyl)pyridine via [RuCl2(CO)(p-cymene)]
- AE-120: Degassing of n-Heptane
- AE-157: Synthesis of [RuCl2(CO)(PNP)] with 2,6-Bis((tert-butyl)phosphinomethyl)pyridine via [RuCl2(CO)(p-cymene)]
- KRA-080: Degassing of THF
- AE-221: Synthesis of [RuCl2(CO)(p-cymene)]
- 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-248: Synthesis of [RuCl2(CO)(p-cymene)]
- AE-253: Synthesis of 2,6-Bis(P,P-di(tertbutyl)phosphinomethyl)pyridine

## Linked resources

Protocol - Preparation of NMR Sample

Protocol - Filtration with frit technique

Protocol - Schlenk Technique

## Attached files

prod.jpg
sha256: d3b1e5ae30bd99a40fd678a2a35bbed02c35328809789b81629e873c95169215

NMR.jpg
sha256: a6e73b1966723ba79ebade8d7be02eb8a5626c144540e1f0edb495891eaeeb1c

on-fritjpg
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AE-261-1_10.nmrium
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AE-261-1_10.zip
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Ru.jpg
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PNPtBu.jpg
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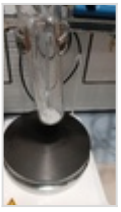

after-first-drying.jpg
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at-rt.jpg
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reduced-volume.jpg
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in-THF.jpg
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after-reaction-at-reflux.jpg
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after-adding-n-hept.jpg
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start-of-filtration.jpg
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at-reflux.jpg
sha256: 67812c9659cf049e3e40c00060cef8f800994ef4eed8b7d2579b6dec1b6eeba9

## Comment

On 2025-02-19 15:08:13 Kristína Rabatinová wrote:
Note: specify that whole ru storage schlenk was used

Unique eLabID: 20240418-95059e9a21c50d0a979af71db3d34e36ff920a23
Link: https://elab.water-splitting.org/experiments.php?mode=view&id=959