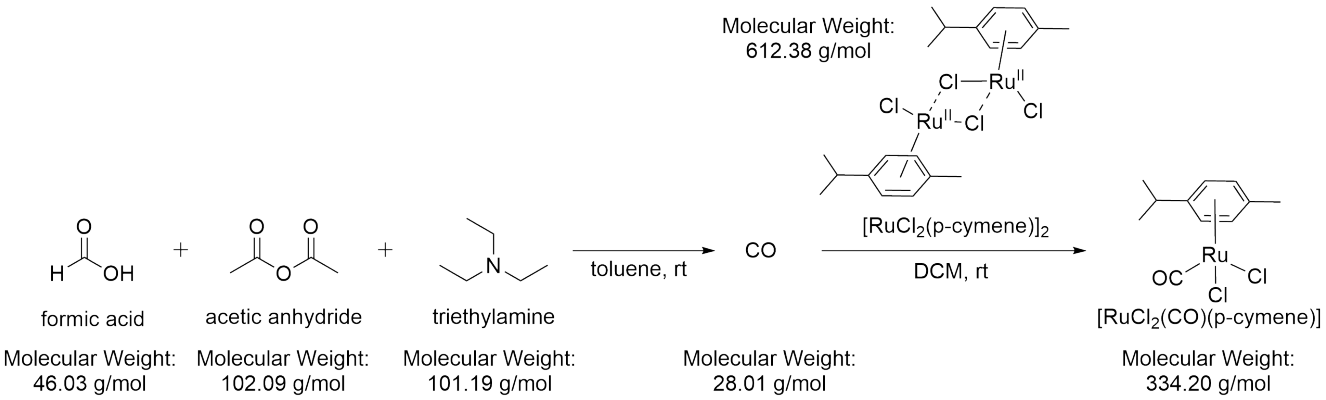
AE-248: Synthesis of [RuCl2(CO)(p-cymene)]

Date: 2024-03-25
Tags: [RuCl2(CO)(p-cymene)] NMR AE reference analytics 1H reference procedure
Status: Done
Created by: Alexander Eith

Reaction scheme/sample structure



Literature/reference experiments

Literature	https://doi.org/10.1039/D1EE01053K
Reproduction	/
Related experiment	Experiment - AE-100: Synthesis of [RuCl2(CO)(p-cymene)], Experiment - AE-221: Synthesis of [RuCl2(CO)(p-cymene)]

Reagents

Name	CAS/Experiment number	Amount [mmol]	Equivalents	$Mass_{theo}$ [mg]	$Mass_{exp}$ [mg]	Molar mass [g/mol]	$Volume_{theo}$ [mL]	$Volume_{exp}$ [mL]	Density [g/mL]
$[RuCl_2(p\text{-cymene})]_2$	52462-29-0	1.30	0.5	800	794.24	612.38	/	/	/
Formic acid	Experiment - AE-247: Degassing of formic acid	2.60	1	119.6	/	46.03	0.098	0.098	1.22
Acetic anhydride Ac_2O	108-24-7	2.60	1	265.4	/	102.1	0.246	0.246	1.08
Triethylamine NEt_3	Experiment - AEI-046: Degassing of NEt3 (Triethylamin)	5.20	2	526.24	/	101.2	0.72	0.72	0.73
DCM	Experiment - KRA-068: Degassing of DCM	/	/	/	/	/	16.0	16	/
Toluene	Experiment - AEI-016: Degassing of Toluene	/	/	/	/	/	5.2	5.0	/

Procedure/observations

All steps, unless mentioned otherwise, were carried out under argon atmosphere using standard Schlenk technique. (see Protocol [Protocol] Schlenk Technique)

Date	Time	Procedure	Observation/Comments
25.03		The H-tube was prepared	
	9:20	The $[RuCl_2(p\text{-cymene})]_2$ was weighted at air in one of the two tubes (tube A). The flask was subsequently set under argon.	The $[RuCl_2(p\text{-cymene})]_2$ is a red glittering solid.
	9:45	DCM (16 mL) was added to tube A in argon counter stream.	A dark red solution was obtained.
	9:50	To the other tube (tube B) toluene (5.0 mL) was added in argon counter flow.	
	9:53	Fromic acid (98 µL) was added using a 100 µL Eppendorf Pipette in argon counterflow. The tip was flushed with argon for approx. 10 s prior to use. The flask was closed.	
	9:54	Acetic anhydride (246 µL, not dried, not degassed) was added using a 1000 µL Eppendorf Pipette in argon counterflow. The tip was flushed with argon for approx. 10 s prior to use. The flask was closed.	
		The reaction vessel was closed against the atmosphere and the Schlenk-line.	
	9:57 - 10:02	NEt_3 (0.72 mL) was added dropwise to tube B (approx. 5 min) while being stirred (520 rpm).	Some vapor formation during adding of first approx. 0.2 mL. After approx. 2 min the formation of gas bubbles was observed. Pictures: 20240326_095846.jpg 20240326_095916.jpg 20240326_100031.jpg 20240326_100056.jpg 20240326_100342.jpg 20240326_104117.jpg
	10:02 - 11:02	The reaction was stirred for 1:00 h at 520 rpm.	A dark red solution was obtained at the end. after reaction under CO.jpg
	11:02	The solution of tube A was transferred to the new 50 mL- schlenk tube (with a stirrer bar) using a syringe and a metal cannula. The flask was tilted so that non of the liquid in tube B could get into flask A, also the stop cock of flask B was kept closed	after transfer.jpg

	11:04 - 11:20	The solvent (DCM) was removed under reduced pressure using an external cooling trap.	after removing DCM.jpg
	11:21 - 15:45	The obtained solid was dried under reduced pressure for approx. 4:20 h.	A salmon red solid was obtained. dried solid.jpg
		The soild was stored under argon in the fumehood at rt.	
27.03	10:35	The solid was transfferred into a vial and stored under argon at rt. During this transferre some matirial was transffered into a seperatly prepared 10 mL flask (approx. 5mg)	Signficant amunts of product were lost due to the highly electrostatic behaviour
	12:20	To the flask containing to approx. 5 mg DCM-d2 (0.7 mL) was added	
		An NMR sample was prepared according to Protocol - Preparation of NMR Sample	NMR sample.jpg

Analysis

Date	Time	Sample name	Analysis method	Analytik device	Solvent	Raw Data	Processed Data	Comparative Data	Interpretation
27.03	14:16	AE-28-1	NMR 1 H	ZAF 300 MHz	DCM-d2	AE-28-1.zip	AE-28-1_10.nmrium	Experiment - AE-100: Synthesis of [RuCl2(CO)(p-cymene)]	analog to AE-100, some impurties are observed

Product characterization

	mass [mg]	purity [%]	$mass_{pure}$ [mg]	amount [mmol]	Yield [%]	Desciption
AE-248-1	662.65	77 AE-248-NMR-puritiy.xlsx	510.24	1.53	58	salmon red solid

Future recommendations

Old procedure	Problem	Suggested new procedure

Linked experiments

- AEI-016: Degassing of Toluene
- AEI-032: Degassing of DCM
- AEI-046: Degassing of NEt3 (Triethylamin)
- AEI-057: Synthesis of [RuCl2(CO)(p-cymene)]
- AE-100: Synthesis of [RuCl2(CO)(p-cymene)]
- KRA-063: Degassing of formic acid (80 %)
- KRA-068: Degassing of DCM
- AE-221: Synthesis of [RuCl2(CO)(p-cymene)]
- AE-247: Degassing of formic acid

Linked resources

Protocol - Preparation of NMR Sample

Protocol - Schlenk Technique

Attached files

AEI-0013.cdxml
sha256: a5d83ef61c882538b0fa9abd8cd69e6c44ef56b9acdad689ff66f798e4301229

AE-248-NMR-puritiy.xlsx
sha256: 1367707f450e970502dae0382d71ab6d37683ef5a13685cbee1a2614b6f62299

AE-28-1_10.nmrium
sha256: 0e23716ee8b3324373f447aba9d788c18317ebc06a714944108be1360eb7e0f3

AE-28-1.zip
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20240326_100031.jpg
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NMR-sample.jpg
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after-removing-DCM.jpg
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after-transfer.jpg
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dried-solid.jpg
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after-reaction-under-CO.jpg
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20240326_100342.jpg
sha256: fac549cd195674b2c0c78b95cad33e970cabcbb02bf128a4c6b3aeb038c592c8

Comment

On 2024-10-25 09:00:40 Kristína Rabatinová wrote:
* missing the .png file and chemdraw file

Unique eLabID: 20240325-91b9b33765d7c8f75265b5c4c89c8cf7473697d2
Link: https://elab.water-splitting.org/experiments.php?mode=view&id=914