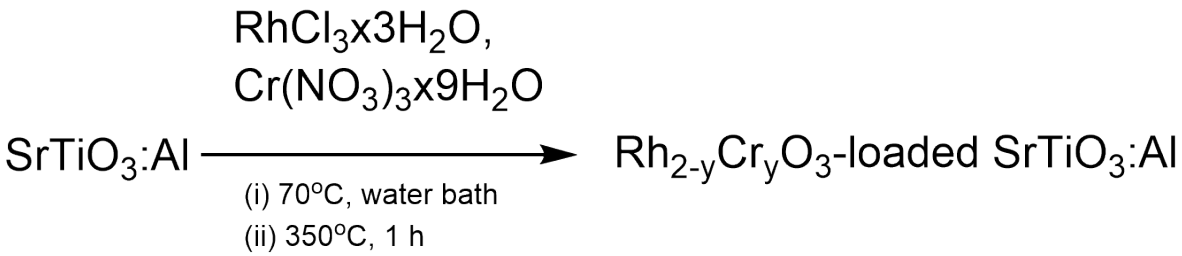
NB-342: Modification of EA-354 (SrTiO3:Al, upscaled batch) with Rh, Cr oxide cocatalyst (0.2 wt%), fresh solutions of RhCl3*3H2O and Cr(NO3)3*9H2O

Date: 2025-11-11
Tags: NB | synthesis | Furnace | Muffle Furnace | SrTiO3 | RhCl3 | Osterloh | RhxCryO3 | Al:SrTiO3 | RhCrO3:Al:SrTiO3 | Cr(NO3)3
Category: SrTiO3
Status: Done
Created by: Nadzeya Brezhneva

Reaction scheme/sample structure



ChemDraw File (linked): NB-189-RhCr-oxide-loaded-Al-SrTiO3.cdxml

Literature/reference experiments

Literature	https://doi.org/10.1039/C9EE00310J
Reproduction	/
Similar experiments	SrTiO3 - NB-323: Modification of EA-354 (SrTiO3:Al, upscaled batch) with Rh, Cr oxide cocatalyst (0.2 wt%)

Reagents

Name	CAS Number / Experiment Number	Inventory number	Amount	Equivalents	$Mass_{theo}$ [mg]	$Mass_{exp}$ [mg]	Molar mass [g/mol]	Density [g/ml]	Volume [ul]
Al:$SrTiO_3$ (EA-354)	SrTiO3 - EA-354: Preparation of Al:SrTiO3 (using EA-352-SrTiO3-molten-salt and SrCl2 as flux) at 1000°C, 10 h, Osterloh route , upscaling x15, filtration with PVDF	/	0.83 mmol	1	150	152.05	183.49	/	/
$RhCl_3$ solution (9.87 mM in H2O)	Prep work - NB-KRA-340: Preparation of RhCl3 and Cr(NO3)3 stock solutions	/	2.92 umol	0.2 wt% Rh[a]	/	295	209.26	/	295
$Cr(NO_3)_3$ solution (28.81 mM in H2O)	Prep work - NB-KRA-340: Preparation of RhCl3 and Cr(NO3)3 stock solutions	/	5.76 umol	0.2 wt% Cr[b]	/	200	238.011	/	200
milli-Q water	/	/	0.19 mol	/	/	/	18	0.998	3505[c]

[a] - Rh content in the final product

[b] - Cr content in the final product

[c] - for suspension preparation

Excel sheet for reagent calculation

NB-342-calculations.xlsx

Furnace Parameters

Equipment - Muffelofen L3/11/P320, Nabertherm GmbH, Lab 106, CEEC I, (Matilda)

Temperature/time parameters

Used zone or charge sensor	Zone
Used delayed start	/
Used automatic/manual/extended holdback	Automatic
The temperature band entered for manual/extended holdback (°C)	/
End time [min], relative to start of program	/

Segments

	Target Temperature (°C)	**Duration (h)**	**Rate (°C/h)**	**Temperature band (°C)**	**Description of the segment**	**Observations**
First segment	350	0:33	600	/	Increase	/
Second segment	350	1:00	/	/	Hold	/
End segment	0	/	/	/	Natural cooling down	/

Procedure/observations

For transfer of precise liquid amount, Eppendorf pipettes were used (for 10 - 100 ul: 100 ul Eppendorf, above 100 ul: 1000 ul Eppendorf).

Date	Time	Step	Observations	Pictures
11.11.2025	17:35	Weighing **EA-354**, transfer to a 25 mL Schott glass beaker.	Slightly creamy solid.	/
	17:40	Placing the glass stirring bar (8 mm) inside the beaker.	/	/
	17:50	Addition of 3505 uL of H_2O (3x1000 + 505) to the beaker.	Creamy, slightly pinkish suspension.	/
	17:51	Placing the beaker with the suspension into the water bath (currently, at room T).	/	/
	17:52	Switching the stirring (400 rpm).	/	/
	17:53	Drop-by-drop addition of 295 uL of 9.87 mM $RhCl_3$ solution to a beaker under stirring.	/	/
	17:54	Drop-by-drop addition of 200 uL of 28.9 mM $Cr(NO_3)_3$ solution to a beaker under stirring.	/	/
	17:55	Swtching the heating mode on (70 °C, precise mode).	/	/
	17:55-19:30	Evaporating of water under constant stirring at 70 °C.	/	20251111_175528-start of evaporation of water.jpg

	19:30	Stop stirring, since all water has been evaporated, removing the stirring bar from the beaker.	In general solid seems quite homogeneous	20251111_193204-after evaporation of water.jpg
	19:40	Transfer of the solid to the quartz crucible using Smartspatula, covering the crucible with lid and Al foil before further use.	/	20251111_195700-crucibles with dried samples-top 0.2 wt% loading bottom 0.05 wt% loading.jpg
	20:00	Loading the crucible with the dried sample (together with the crucible with the sample SrTiO3 - NB-341: Modification of EA-354 (SrTiO3:Al, upscaled batch) with Rh, Cr oxide cocatalyst (0.05 wt%), fresh solutions of RhCl3 and Cr(NO3)3) in a muffle furnace Equipment - Muffelofen L3/11/P320, Nabertherm GmbH, Lab 106, CEEC I, (Matilda).	/	20251111_200317-crucibles inside the furnace.jpg
	20:03	Start of the program (350 °C, 1 h, 10 °/min).	/	/
12.11.2025	0:30	Removal of the samples from the furnace.	T = 163 °C when opening the lid Grey solid (more intense grey colour in comparison with SrTiO3 - NB-321: Modification of EA-354 (SrTiO3:Al, upscaled batch) with Rh, Cr oxide cocatalyst (0.05 wt%)).	/
	15:50	Collecting the sample from the beaker, transfer to a 5 mL vial with a snap cap, weighing. m = 142.57 mg	**NB-342-0.2 wt% Rh,Cr-oxide loaded Al:$SrTiO_3$** Creamy greyish solid.	20251112_160416-final product.jpg

Product characterization

Sample	Mass [mg]	Purity	$Mass_{pure}$[mg]	Amount [mol]	Yield [%]	Description	Image	Storage location
NB-342-0.2 wt% Rh,Cr-oxide loaded Al:$SrTiO_3$	142.57	/	/	/	93.76	Creamy greyish solid.	20251112_160416-final product.jpg	Lab E004 (CEEC II), SSC cabinet

Results

Modification of Al:$SrTiO_3$ sample (**EA-354**, upscaled batch) with Rh, Cr oxide cocatalyst (0.2 wt% Rh and Cr in final product) was performed. The final product (**NB-342-0.2 wt% Rh,Cr-oxide loaded Al:$SrTiO_3$**) is

represented with grey solid, m = 142.57 mg, yield 93.76 %.

Linked experiments

- Prep work - NB-340: Preparation of RhCl3*3H2O and Cr(NO3)3*9H2O stock solutions
- SrTiO3 - NB-162: Preparation of SrTiO3:Al with RhyCr2-yO3 (Osterloh route), I attempt, 1000 C 10h
- SrTiO3 - NB-175: Preparation of SrTiO3:Al (1100 C, 3 h, IAAC furnace) (Osterloh route)
- SrTiO3 - NB-285: Preparation of SrTiO3:Al (from self-made SrTiO3, NB-283, batch V, Osterloh (no Al2O3), upscaling x3), 1000 C, 10 h, filtration with PVDF
- SrTiO3 - NB-289: Modification of NB-285 (SrTiO3:Al (from self-made SrTiO3, Osterloh, no Al2O3, PVDF filter) 1000 C, 10 h) with Rh, Cr oxide cocatalyst
- SrTiO3 - NB-301: Modification of NB-285 (SrTiO3:Al (from self-made SrTiO3, Osterloh, no Al2O3, PVDF filter) 1000 C, 10 h) with Rh, Cr oxide cocatalyst I
- SrTiO3 - EA-354: Preparation of Al:SrTiO3 (using EA-352-SrTiO3-molten-salt and SrCl2 as flux) at 1000°C, 10 h, Osterloh route , upscaling x15, filtration with PVDF
- SrTiO3 - EA-359: Modification of Al:SrTiO3 (EA-354) via deposition of Rh, Cr oxide co-catalyst, 350°C, 1h
- SrTiO3 - NB-321: Modification of EA-354 (SrTiO3:Al, upscaled batch) with Rh, Cr oxide cocatalyst (0.05 wt%)
- SrTiO3 - NB-323: Modification of EA-354 (SrTiO3:Al, upscaled batch) with Rh, Cr oxide cocatalyst (0.2 wt%)
- SrTiO3 - NB-341: Modification of EA-354 (SrTiO3:Al, upscaled batch) with Rh, Cr oxide cocatalyst (0.05 wt%), fresh solutions of RhCl3*3H2O and Cr(NO3)3*9H2O

Linked resources

- Equipment - Muffelofen L3/11/P320, Nabertherm GmbH, Lab 106, CEEC I, (Matilda)
- Equipment - Muffle furnace Nabertherm LT 15/11/P330 (AWZ 304 lab)
- Protocol - Heat treatment using Muffelofen L3/11/P320, Nabertherm GmbH, Lab 106, CEEC I
- Protocol - Muffle furnace Nabertherm GmbH LT 15/11/P330 (Lab AWZ 304)

Attached files

NB-342-calculations.xlsx
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20251112_160416-final product.jpg
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20251111_175528-start of evaporation of water.jpg
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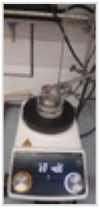

20251111_193204-after evaporation of water.jpg
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20251111_200317-crucibles inside the furnace.jpg
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20251111_195700-crucibles with dried samples before calcination-top 0.2 wt% loading bottom 0.05 wt% loading.jpg
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Link: https://elab.water-splitting.org/experiments.php?mode=view&id=3422