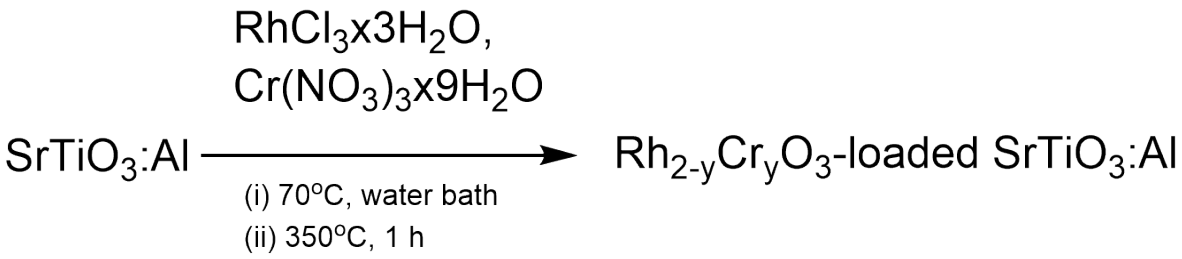
NB-321: Modification of EA-354 (SrTiO3:Al, upscaled batch) with Rh, Cr oxide cocatalyst (0.05 wt%)

Date: 2025-10-27
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 - EA-358: Modification of Al:SrTiO3 (EA-354) via deposition of Rh, Cr oxide co-catalyst, 350°C, 1h, Upscaling (3.33x) SrTiO3 - EA-359: Modification of Al:SrTiO3 (EA-354) via deposition of Rh, Cr oxide co-catalyst, 350°C, 1h

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	/	0.81 mmol	1	150	151.03	183.49	/	/
9.87 mM $RhCl_3$ solution (**EA-357**)	#EA-357	/	0.73 umol	0.05 wt% Rh[a]	/	75	209.26	/	75
28.8 mM $Cr(NO_3)_3$ solution (**EA-357**)	#EA-357	/	1.44 umol	0.05 wt% Cr[b]	/	50	238.011	/	50
milli-Q water	/	/	0.19 mol	/	/	/	18	0.998	3875[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-321-calculations.xlsx

Furnace Parameters

Equipment - Muffle furnace Nabertherm LT 15/11/P330 (AWZ 304 lab)

Protocol - Muffle furnace Nabertherm GmbH LT 15/11/P330 (Lab AWZ 304)

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/Files
27.10.2025	18:40	Weighing **EA-354** in a 25 mL Schott glass beaker.	Slightly creamy solid.	/
	ca. 18:45	Placing the glass stirring bar (8 mm) inside the beaker.	/	/
	18:46	Addition of 3875 uL of H_2O (3x1000 + 875) to the beaker.	Creamy, slightly pinkish suspension.	20251027_184656-after addition of water.jpg
	ca. 18:48	Placing the beaker with the suspension into the water bath (currently, at room T).	/	/
	18:49	Switching the stirring (400 rpm).	/	/
	18:50	Fast addition of 75 uL of 9.87 mM $RhCl_3$ solution to a beaker under stirring.	/	/
	18:52	Fast addition of 50 uL of 28.9 mM $Cr(NO_3)_3$ solution to a beaker under stirring.	/	/
	18:53	Swtching the heating mode on (70 °C, precise mode).	/	20251027_185519-start of heating.jpg
	18:53-20:30	Evaporating of water under constant stirring at 70 °C.	/	/

	20:30	Stop stirring, since all water has been evaporated, removing the stirring bar from the beaker.	In general solid seems quite homogeneous	20251027_203107-after evaporation.jpg
	ca. 20:35	Transfer of the solid to the quartz crucible using Smartspatula, covering the crucible with lid and Al foil before further use.	/	20251027_203754-solid transferred to crucible before calcination.jpg 20251028_004550-crucibles covered with Al foil.jpg
28.10.2025	ca. 10:35	Loading the crucible with the dried sample in a muffle furnace Equipment - Muffle furnace Nabertherm LT 15/11/P330 (AWZ 304 lab).	/	20251028_103723-crucibles before calcination.jpg 20251028_103806-crucibles inside the furnace.jpg
	10:40	Start of the program (350 °C, 1 h, 10 °/min).	/	20251028_104132-start of calcination.jpg
	19:20	Removal of the sample from the furnace.	T = 163 °C when opening the lid Slightly grey solid.	/
	20:25	Collecting the sample from the beaker, transfer to a 4 mL vial with a screw cap, weighing. m = 148.40 mg	**NB-321-0.05 wt% Rh,Cr-oxide loaded Al:$SrTiO_3$** Slightly grey solid.	20251028_203010-final product.jpg

Product characterization

Sample	Mass [mg]	Purity	$Mass_{pure}$[mg]	Amount [mol]	Yield [%]	Description	Image	Storage location
NB-321-0.05 wt% Rh,Cr-oxide loaded Al:$SrTiO_3$	148.40	/	/	/	98.26	Slightly grey solid.	20251028_203010-final product.jpg	Lab E004 (CEEC II), SSC cabinet (SrTiO3-6 container)

Results

Modification of Al:$SrTiO_3$ sample (**EA-354**, upscaled batch) with Rh, Cr oxide cocatalyst (0.05 wt% Rh and Cr in final product) was performed. The final product (**NB-321-0.05 wt% Rh,Cr-oxide loaded Al:$SrTiO_3$**) is represented with slightly grey solid, m = 148.40 mg, yield 98.26 %.

Linked resources

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

Protocol - Heat treatment using Muffelofen L3/11/P320, Nabertherm GmbH, Lab 106, CEEC I

Attached files

NB-321-calculations.xlsx
sha256: 950437204a986eb1b69ef05c2814c496b5f0d00f75d0a5fabe98ef62040c1d53

20251028_203010-final product.jpg
sha256: 9e79828cce4643361b3b56bb20ba24b72c643bf0be3f5b6749858b231c5c7d81

20251027_184656-after addition of water.jpg
sha256: 94281b634889230df3cc7c66c0d7d7af6fd7441b30cc58fad1dbdaf10248bd4c

20251027_203754-solid transferred to crucible before calcination.jpg
sha256: 91ebbc36a9407f2818b688ee16c7301496092527b6b94e77b8f3d77126840251

20251027_203107-after evaporation.jpg
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20251027_185519-start of heating.jpg
sha256: 2aba07e13bc87192e6de9d8f34d5d67373eb5cefe64d15008588493d1b44487f

20251028_103723-crucibles before calcination.jpg
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20251028_004550-crucibles covered with Al foil.jpg
sha256: ba59eb481be8c977056f254473977ff960308f6a708c69ce38ecbc2a09f21bb8

20251028_104132-start of calcination.jpg
sha256: e0d01050067f72de0367930d55e595be053c63016e421032e8b04b13483824a7

20251028_103806-crucibles inside the furnace.jpg
sha256: dc978cb64016b945fe874a679a8b1572618ee806ca3c1d5e65e300ab39836e8a

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Link: https://elab.water-splitting.org/experiments.php?mode=view&id=3263