EA-352: Synthesis of SrTiO3 from SrCO3 and TiO2, 1000°C, 10 h, Osterloh route, Upscaling (2x)

Date: 2025-10-01
Tags: Future synthesis EA Furnace Muffle Furnace SrTiO3 Osterloh TiO2 Upscaling
Category: SrTiO3
Status: Done
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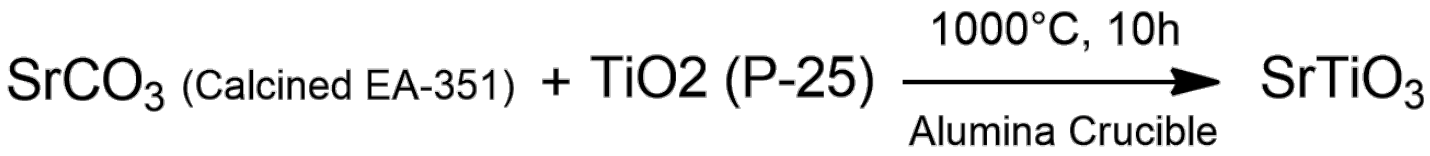
Objective

Synthesis of SrTiO3 from SrCO3 and TiO2 (P-25) via solid state approach at 1000°C,10h. 2 times upscaling.

Reaction scheme/sample structure



ChemDraw File (linked): EA-352.cdx

Literature/reference experiments

Literature	https://doi.org/10.1039/C9EE00310J
Reproduction	/
Similar experiments	SrTiO3 - NB-283: Preparation of SrTiO3 from SrCO3 and TiO2, 1000 C, 10 h, batch V

Reagents

Name	CAS Number / Experiment Number	Inventory number	Amount [mmol]	Equivalents	$Mass_{theo}$ [g]	$Mass_{exp}$ [g]	Molar mass [g/mol]	Density (g/ml)	Volume [ml]	Concentration [mM]
Calcined $SrCO_3$ (EA-351)	SrTiO3 - EA-351: Calcination of SrCO3, 300 C, 1 h, Upscaling (3x)	/	41.99	1	2 × 3.10 = 6.20	6.21	147.63	3.5	/	/
TiO_2, Aeroxide® P25, Thermo Scientific	13463-67-7	C137952	42.06	1	2 × 1.68 = 3.36	3.37	79.87	/	/	/

Work-up and Analytical Reagents

Name	CAS Number / Experiment Number	Inventory number	$Mass_{exp}$ [g]	Volume [ml]	Concentration [M]

Ethanol	64-17-5	/	/	1	/

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	861

Segments

Program 2	**Target Temperature (°C)**	**Duration (h)**	**Rate (°C/h)**	**Temperature band (°C)**	**Description of the segment**	**Observations**
First segment	1000	01:38	600	/	Increase	Initial temperature: 20°C
Second segment	1000	10:00	/	/	Holding	/
End segment	/	/	/	/	Natural cooling	End temperature (before opening): 217°C

Procedure/observations

Date	**Time**	**Step**	**Observations**	**Pictures**
29.09.2025	21:55	Weighing 6.21 g of calcined $SrCO_3$ (EA-351) in a weighing bowl.	White clumpy powder	20250929_2155-weighing SrCO3.jpg

	22:03	Weighing 3.37 g of TiO_2 (P-25) in a weighing bowl.	White very fine powder	20250929_2203-weighing TiO2.jpg
	22:06	The weighed samples were transferred into an agate mortar (d: 10cm).	White powders	20250929_2206-transferring the weighed samples into one agate mortar.jpg
	22:12 - 22:20	The weighed SrCO3 and TiO2 in the mortar were mixed while mortaring. During the mixing time, 250µl of disstiled EtOH (from the washing bottle) was added to the mixture then got mixed with the powder with a smart spatula. This procedure was repeated 4 times. In total 1ml of EtOH was used.	Addition of EtOH helped the mixture to be more homogenized. After mixing/mortaring the mixture turned to be more fine and less puffy.	20250929_2212-addition of 250µl EtOH to the mixture-1.jpg 20250929_2214-addition of 250µl EtOH to the mixture-2.jpg 20250929_2216-addition of 250µl EtOH to the mixture-3.jpg 20250929_2217-addition of 250µl EtOH to the mixture-4.jpg 20250929_2220-the mixture before transferring to crucible.jpg
	22:22	The mixture was transferred into a 150ml alumina crucible.	White powder	20250929_2222-transferring the mixture into the alumina crucible.jpg
	22:24	The mixture inside the crucible was pressed with the agate pestle (without applying force and just with help of weight of the pestle).	Pressed and evened white powder	20250929_2224-pressing the mixture in crucible with pestle.jpg
	22:31	The crucible with lid was transferred to AWZ lab 304 Equipment - Muffle furnace Nabertherm LT 15/11/P330 (AWZ 304 lab). The heating program was designed according to Protocol - Muffle furnace Nabertherm GmbH LT 15/11/P330 (Lab AWZ 304), and saved on P=2. The heating program (EA-352-P=2) was started.	Initial temperature: 20°C	20250929_2231-crucible inside the furnace.jpg
30.09.2025	00:20	The temperature of the furnace was checked during the heating phase. Note: the furnace can not be heated with 600 (°C/h).	T: 887°C Because of the size of the furnace (15L), the heating takes more time than 10C°.min^{-1}	20250930_0020-checking the temperature of the program during heating phase.jpg
	12:39	The temperature of the furnace was checked during the cooling phase.	T: 721°C	20250930_1239-checking the temperature of the furnace in cooling down phase.jpg
	23:52	After cooling down the furnace, turned off and opened.	End temperature (before opening): 217°C	20250930_2352-End of heating program.jpg

	00:38 - 00:39	The crucible was opened and the formed $SrTiO_3$ was checked.	White big clump with the shape of bottom of the crucible	20251001_0038-formed SrTiO3 in crucible after heating program-1.jpg 20251001_0039-formed SrTiO3 in crucible after heating program-2.jpg
	00:40	The formed $SrTiO_3$ was weighed in a weighing bowl.	White big clump	20251001_0040-Weighing SrTiO3.jpg
	00:42	With the help of a smart spatula, the big clump was broken into smaller pieces.	White soft clumps	20251001_0042-breaking the big clump into smaller pieces.jpg
	00:47	The weighed $SrTiO_3$ was collected after breaking the big clump into a 20ml snap-cap vial and covered with Al foil. Named: **EA-352-$SrTiO_3$**	White soft clumps **EA-352-$SrTiO_3$**	20251001_0047-SrTiO3.jpg
	00:47	The formed SrTiO3 of this batch was compared with SrTiO3 - NB-283: Preparation of SrTiO3 from SrCO3 and TiO2, 1000 C, 10 h, batch V to see the color differences between two batches.	This batch: White NB-283: more creamy/grey	20251001_0047-comparison of this batch with NB-283-1.jpg 20251001_0047-comparison of this batch with NB-283-2.jpg

Product characterization

Sample	Mass [g]	Purity	$Mass_{pure}$[g]	Amount [µmol]	Yield [%]	Description	Image	Storage location
EA-352-$SrTiO_3$	7.64	/	/	/	98.98	White soft clumps	20251001_0047-SrTiO3.jpg	

Results

Synthesis of SrTiO3 using SrCO3 and TiO2 (P-25) was done. The colour of the product was whiter in comparison with previous batches. The mass yield: 79.7%. The theoretical yield: 98.98%.

Future recommendations

Old procedure	Problem	Suggested new procedure
Using Equipment - Muffle furnace Nabertherm LT 15/11/P330 (AWZ 304 lab)	Long time of cooling down	Using Equipment - Muffelofen L3/11/P320, Nabertherm GmbH, Lab 106, CEEC I, (Matilda) instead.

Linked experiment

SrTiO3 - EA-351: Calcination of SrCO3, 300 C, 1 h, Upscaling (3x)

Linked resources

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

Equipment - Advanced irradiation chamber V1.0 I

Equipment - Advanced power measurment chamber V1.0 I

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

Equipment - Manual irradiation setup

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

Attached files

EA-352.cdx
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EA-352.png
sha256: 37c623ae90cc4a81ffcaa54744deaf1113d664e53b315a91cc830df0f0140ad5

20250929_2155-weighing SrCO3.jpg
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20250929_2206-transferring the weighed samples into one agate mortar.jpg
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20250929_2220-the mixture before transferring to crucible.jpg
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20250929_2224-pressing the mixture in crucible with pestle.jpg
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20250929_2222-transferring the mixture into the alumina crucible.jpg
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20250929_2231-crucible inside the furnace.jpg
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20250930_0020-checking the temperature of the program during heating phase.jpg
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20250930_1239-checking the temperature of the furnace in cooling down phase.jpg
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20250930_2352-End of heating program.jpg
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20251001_0038-formed SrTiO3 in crucible after heating program-1.jpg
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20251001_0039-formed SrTiO3 in crucible after heating program-2.jpg
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20251001_0040-Weighing SrTiO3.jpg
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20251001_0042-breaking the big clump into smaller pieces.jpg
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20251001_0047-comparison of this batch with NB-283-2.jpg
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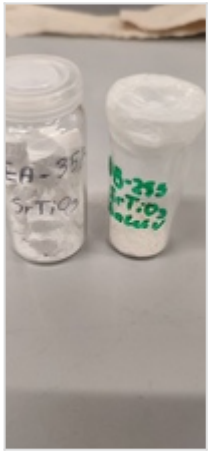

20251001_0047-comparison of this batch with NB-283-1.jpg

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20251001_0047-SrTiO3-molten-salt.jpg
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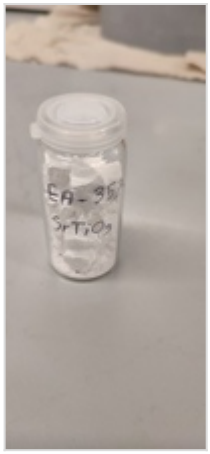

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