

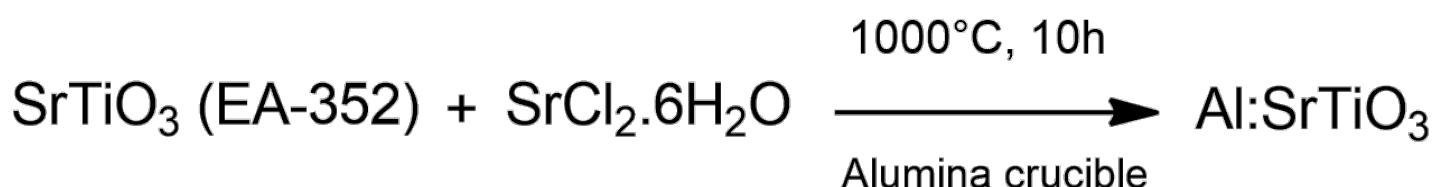
EA-354: Preparation of Al:SrTiO₃ (using EA-352-SrTiO₃-molten-salt and SrCl₂ as flux) at 1000°C, 10 h, Osterloh route , upscaling x15, filtration with PVDF

Date: 2025-10-09
Tags: EA SrTiO₃ Osterloh Al:SrTiO₃
Upscaling Filtration SrCl₂ molten salt
Category: SrTiO₃
Status: Done
Created by: Ebrahim Abedini

Objective

Doping of Al on the SrTiO₃ (EA-352) via the Osterloh route (no addition of Al₂O₃-getting the Al from the alumina crucible). 15 times upscaling.

Reaction scheme/sample structure



ChemDraw File (linked): [EA-354.cdx](#)

Literature/reference experiments

Literature	https://doi.org/10.1039/C9EE00310J
Reproduction	/
Similar experiments	SrTiO ₃ - NB-285: Preparation of SrTiO ₃ :Al (from self-made SrTiO ₃ , NB-283, batch V, Osterloh (no Al ₂ O ₃), upscaling x3), 1000 C, 10 h, filtration with PVDF

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]
SrTiO ₃ -molten-salt (EA-352)	SrTiO ₃ - EA-352: Synthesis of SrTiO ₃ from SrCO ₃ and TiO ₂ via molten-salt approach, 1000°C, 10 h, Upscaling (2x)	/	30.246	1	15 × 0.37 = 5.55	15 × 0.37	183.49	/	/	/
SrCl ₂ ·6H ₂ O, 99 % (ACS), Strem Chemicals	10025-70-4		299.864	10	15 × 5.33 = 79.95	15 × 5.33	266.62	/	/	/

Work-up and Analytical Reagents

Name	CAS Number / Experiment Number	Inventory number	Mass _{exp} [g]	Volume [ml]	Concentration [M]
milli-Q water	/	/	/	150ml for removing the sample from the crucible Washing the sample after thermal treatment to remove the SrCl ₂ flux ~ 10000ml 100ml for making the slurry	/
0.1 M AgNO ₃ solution	Prep work - ASD-040: Preparation of 0.1 M AgNO ₃ solution	/	/	ca. 0.1-0.2 (no precise values, just few drops for each Cl ⁻ test)	0.1

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	2050

Segments

Program 7	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: 79°C
Second segment	1000	10:00	/	/	Hold	/
End segment	/	/	/	/	Natural cooling	End temperature (before opening): 104°C

Procedure/observations

Date	Time	Step	Observations	Pictures
01.10.2025	00:54	SrCl ₂ ·6H ₂ O was weighed in a weighing bowl.	White crystals	20251001_0054-Weighing SrCl2.6H2O.jpg
	01:08	SrTiO ₃ (EA-352) was weighed in a weighing bowl.	Creamy white fine clumps	20251001_0108-weighing SrTiO3.jpg
	01:10	All of the weighed materials were transferred into an agate mortar (10cm).	Mixture of crystals and creamy white powders	20251001_0110-the components before mortaring.jpg
	01:24	The components were ground and mixed in the mortar.	Creamy white fine powder	20251001_0124-the components after mortaring.jpg
	01:28	The mortared powder was transferred into 3*150ml alumina crucibles.	Creamy white fine powder	20251001_0128-transferring the mortared powder into the alumina crucibles.jpg
	01:28	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	20251001_0128-pressing the powder in the crucible.jpg
	01:32	The crucibles with lid were transferred to AWZ 304, CEEC II, Equipment - Muffle furnace Nabertherm LT 15/11/P330 (AWZ 304 lab) .	3 alumina crucibles	20251001_0132-the crucibles inside the furnace.jpg
	01:33	The heating program was designed according to Protocol - Muffle furnace Nabertherm GmbH LT 15/11/P330 (Lab AWZ 304) , and saved on P=7. The heating program was started.	Initial temperature: 79°C	20251001_0133-start of the heating program.jpg
	18:15	The temperature of the furnace was checked during the cooling phase.	T: 515°C	20251001_1815-checking the temperature of the furnace in cooling down phase.jpg
02.10.2025	11:43 - 11:44	After cooling down, the furnace was turned off and opened. The crucibles were removed from the furnace.	End temperature: 104°C	20251002_1143-end of the heating program.jpg 20251002_1144-the crucibles after heat treatment in the furnace.jpg
		Work-up procedure		

06.10.2025	12:17	The crucible was opened.	Hard, rock-shaped, grey/creamy solid Material was formed on the upper outer side of the crucibles	20251006_1217-the formed material after heat treatment.jpg 20251006_1217-the formation of the material on the upper-outer side of the crucibles.jpg
	12:19	25 mL H ₂ O was added to each crucible with the calcined sample using graduated cylinder. Using smart spatula, the suspension was mixed and the material was scratched to be dissolved. The suspension was left for 1 h for better dissolution of SrCl ₂ and its further transfer from the crucible.	Grey/creamy suspension	20251006_1219-addition of water to dissolve SrCl2-before mixing.jpg 20251006_1219-addition of water to dissolve SrCl2-after mixing.jpg
	13:08 - 13:11	The crucibles were transferred to the Equipment - Ultrasound Bath Fisherbrand select 300 (E004) . The crucibles were sonicated in the UltraSonication bath for 15 s ("Eco mode").	Grey/creamy suspension	20251006_1308-sonication of the crucibles-1.1.jpg 20251006_1311-sonication of the crucibles-1.2.jpg
	13:24	Transfer of the sonicated suspension from the crucible to a 600 mL glass beaker.	Grey/creamy suspension	20251006_1324-the suspensions were transferred to the beaker.jpg
	13:40	2nd round of addition of water/sonication was done.	Grey/creamy suspension	20251006_1340-2nd round of addition of water/sonication.jpg
	13:58	3rd round of addition of water/sonication was done.	Grey/creamy suspension	20251006_1358-3rd round of addition of water/sonication.jpg
	14:25	4th round of addition of water/sonication was done.	Grey/creamy suspension	20251006_1425-4th round of addition of water/sonication.jpg
	14:33	5th round of addition of water/sonication was done.	Grey/creamy suspension	20251006_1433-5th round of addition of water/sonication.jpg
	14:40	6th round of addition of water/sonication was done.	Grey/creamy suspension	20251006_1440-6th round of addition of water/sonication.jpg
	14:40	All of the suspension was collected in a 600ml beaker.	Grey/creamy suspension	20251006_1440-collecting the suspension in a beaker.jpg
	14:57	Set-up filtration with PVDF filter (0.22µm), solvent trap and pump.	/	20251006_1457-filtration setup.jpg
	15:32	Start of filtration, transfer of the suspension from the glass beaker.	/	20251006_1532-start filtration through PVDF filter.jpg 20251006_1533-start filtration.jpg
	15:37	1: Test of the presence of Cl⁻ anions in the supernatant using 0.1 M AgNO₃. Ca. 1.5-2 mL of supernatant was transferred to a 5 mL vial. Several drops of 0.1 M AgNO ₃ solution were added to the vial using another glass pipette. The vial was swirled for several seconds	Turbid solution, positive result.	20251006_1537-Cl- test-1.jpg
	15:40 - 15:45	Pre heated warm water 70 °C was gradually added from the beaker to the filter (250 ml), while careful scratching of the solid with Smartspatula for better washing of the solid.	Grey/creamy suspension	20251006_1540-addition of warm water.jpg 20251006_1541-mixing the suspension with spatula on the filter.jpg 20251006_1545-after first round of washing.jpg
	15:46	2: Test of the presence of Cl⁻ anions in the supernatant using 0.1 M AgNO₃. Ca. 1.5-2 mL of supernatant was transferred to a 5 mL vial. Several drops of 0.1 M AgNO ₃ solution were added to the vial using another glass pipette. The vial was swirled for several seconds	Turbid solution, positive result.	20251006_1546-Cl- test-2.jpg
07.10.2025	16:17 - 01:44	Pre heated warm water 70 °C was gradually added from the beaker to the filter (3*250ml), while careful scratching of the solid with Smartspatula for better washing of the solid. Then test of the presence of Cl⁻ anions in the supernatant using 0.1 M AgNO₃ was done (Ca. 1.5-2 mL of supernatant was transferred to a 5 mL vial. Several drops of 0.1 M AgNO ₃ solution were added to the vial using another glass pipette. The vial was swirled for several seconds). This procedure was repeated till negative result of Cl ⁻ test.	Tests 3 - 14: Turbid solution, positive result. Test 15 - 16: Transparent solution, negative result. Total amount of warm water used for washing till this point: 10000ml	20251006_1617-Cl- test-3.jpg 20251006_1643-Cl- test-4.jpg 20251006_1728-Cl- test-5.jpg 20251006_1816-Cl- test-6.jpg 20251006_1844-Cl- test-7.jpg 20251006_1916-Cl- test-8.jpg 20251006_2000-Cl- test-9.jpg 20251006_2038-Cl- test-10.jpg 20251006_2112-Cl- test-11.jpg 20251006_2159-Cl- test-12.jpg 20251006_2245-Cl- test-13.jpg 20251006_2330-Cl- test-14.jpg 20251007_0019-Cl- test-15.jpg 20251007_0138-last round of washing.jpg 20251007_0144-Cl- test-16 next to water.jpg
	01:50	By addition of 100ml water, the slurry was made on the top of the filter.	Grey/creamy suspension	/

01:59 - 02:03	The slurry was transferred into a petri dish (10cm). The dish was covered with Al foil with holes on it. The dish was transferred to the Equipment - Drying oven Binder FD 56 (E004, CEEC II) for further drying at 100°C, overnight.	Grey/creamy suspension	0251007_0159-transferring the slurry into the dish for drying.jpg 20251007_0203-covering the dish with Al foil.jpg
14:18	The dried sample was removed from the oven.	Grey/creamy solid	20251007_1418-the dried sample.jpg
14:20	The dried sample was collected from the dish with a smart spatula and weighed on the weing paper.	Grey/creamy solid	/
14:32	The sample was transferred into a 10ml snap cap vial and covered with Al foil. Named: EA-354-Al:SrTiO₃-Osterloh-15times-scaleup	Grey/creamy powder EA-354-Al:SrTiO₃-Osterloh-15times-scaleup	20251007_1432-EA-354-Al:SrTiO₃-Osterloh-15times-scaleup.jpg

Product characterization

Sample	Mass [mg]	Purity	Mass _{pure} [mg]	Amount [μmol]	Yield [%]	Description	Image	Storage location
EA-354-Al:SrTiO₃-Osterloh-15times-scaleup	5222.61	/	/	/	94.10	Grey/creamy powder	20251007_1432-EA-354-Al:SrTiO₃-Osterloh-15times-scaleup.jpg	Equipment - Safety storage cabinet - CEEC II Lab E004, Shelf A, Ebi container

Results

Al:SrTiO₃ sample using SrTiO₃-Osterloh-route without addition of Al₂O₃, annealed at 1000°C, 10 h, washed via filtration (PVDF) procedure was obtained. The yield of the final product was 94.10%.

Linked experiment

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

Linked resources

Equipment - [Ultrasound Bath Fisherbrand select 300 \(E004\)](#)

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-354.png
sha256: caffeb786d8310c112d8d2221233a51ef63e7c31bb83be1ede91486539da1569

EA-354.cdx

sha256: d072d0a147cb0e1589f747f95c6db9894d5285bd5621a629a519cb27dd0cba7d

20251001_0054-Weighing SrCl₂·6H₂O.jpg

sha256: 6932355ea95befe57bdf2fcb682fedf52d1e0a9f45e8f0878c14e7e470a6cf53



20251001_0108-weighing SrTiO₃.jpg

sha256: ebf13afaef7476e41e2899e7dbb1cc0ed220cf0dcf7883b89bcc1183d6436c5a



20251001_0110-the components before mortaring.jpg

sha256: 4efce383a16be89607cf32179ac69a8067a7e6efc82330795d7eb58c4eb05195



20251001_0124-the components after mortaring.jpg

sha256: fca19ba4b59b35deb275ecdd39f19e376612e7a1aa99bcbfbce80a4955b3aa2d



20251001_0128-transferring the mortared powder into the alumina crucibles.jpg

sha256: b2e665bd9cda4317edcea98f78480cd99bf4d2cc77c5359c8b73917ece51a424



20251001_0128-pressing the powder in the crucible.jpg

sha256: e9c83f50391a339c36fc042811c7e95fb842b9f1b088f4d0758678478ce46776



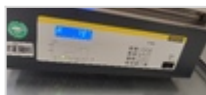
20251001_0132-the crucibles inside the furnace.jpg

sha256: 2776d0d9292cbccc7c90a1ff3beb1129bf6de965e46c90e9522b65868b7a986e



20251001_0133-start of the heating program.jpg

sha256: b22d6845a44c9f2295a2cd2f2365bab8f436bb97a257725888c53f64a7f24638



20251001_1815-checking the temperature of the furnace in cooling down phase.jpg

sha256: 94ae0184b05fc8ca131324e3f3d391cc244e2ab8e7d6d9766ec0ebfc4dd18079



20251006_1217-the formed material after heat treatment.jpg

sha256: 0b2ba227d8cebee5c467c88a54c979f92539fd0bb9cd1474f143ce377e179969



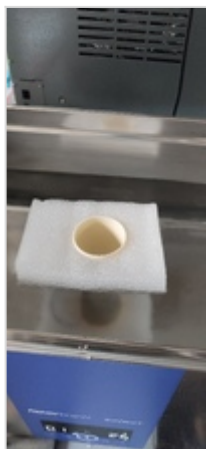
20251006_1308-sonication of the crucibles-1.1.jpg

sha256: 9eb277ffa37403c34167fcbe27b3eae4fd63b4f27c1929030274bc1aaa766d46



20251006_1311-spination of the crucibles-1.2.jpg

sha256: 17723af1204de3f56944b917ace6c9b662438612032ed5cada60e64c453b3b62



20251006_1217-the formation of the material on the upper-outer side of the crucibles.jpg

sha256: 0a0fb2bf73079d70bc03bd1f6f5134a5374e45949af13fec58a998287bd60df8



20251006_1219-addition of water to dissolve SrCl₂-after mixing.jpg

sha256: e5f77edafcab169a1be2e6d5238c541f989d3c2965069aee1f594c57afa499d1



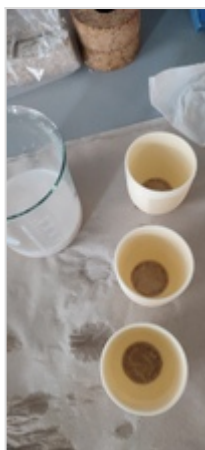
20251006_1219-addition of water to dissolve SrCl₂-1.jpg

sha256: d780c6c36092e47dda1e81e846afbbdc8f9eb70f3b322d1553677624daa2423c



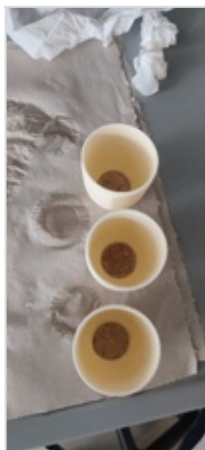
20251006_1324-the suspensions were transferred to the beaker.jpg

sha256: 677725f239a854a08849b1156c81a99e05e3092b1921127c8b46b1fc2108446a



20251006_1340-2nd round of addition of water/sonication.jpg

sha256: 6db8839ffdae874ae222580435d0ca9e6ad3e80d7d8d96b1a08c9f44ee5f9e1f



20251006_1358-3rd round of addition of water/sonication.jpg

sha256: d54de9119d5c8da7f3d06d66a486f23f7eff72eb2d806caa8ebd099d8d5ff82c



20251006_1533-start filtration.jpg

sha256: 4103435776c07a6be43ccf72bb9ff284df89fd0f270e3424bfde61f111443cc8



20251006_1457-filtration setup.jpg

sha256: 79fff66bb7270020aa293dcd6724548515b6453d5675ce1dd6639d59884586ea



20251006_1440-collecting the suspension in a beaker.jpg

sha256: 0e1b69ad4a1cccff64ff417dc404b5d481f53d84ecf184cd856e50beb61bce62



20251006_1440-6th round of addition of water/sonication.jpg

sha256: e7363748e8df81fef1c95cb5a9abdce5d8df097941193120d1bcc8ce0ac44f4f



20251006_1532-start filtration through PVDF filter.jpg

sha256: 8191261027eabb7dfa73eb2f97e59f7708b48adbb0fa787aa25ce711b55cbaf8



20251006_1433-5th round of addition of water/sonication.jpg

sha256: d06b7705a94e99ba88a76e9acb4770282ccdf4c6ec3251a8699bbf4a1bea478a



20251006_1425-4th round of addition of water/sonication.jpg

sha256: aace53f633e7d44b499db82cbf83590b53f6a742633ea0e0d39043694f3ce968



20251006_1537-Cl- test-1.jpg

sha256: 4c4087f4cae057166155a0b8f4dcfe469a91d4409b4f70328cfbe3a9f935ae91



20251006_1540-addition of warm water.jpg

sha256: 8246c50c31c4551f7039a27a5daacabdcf7f362621e05e20d669155b362a6f3e



20251006_1541-mixing the suspension with spatula on the filter.jpg

sha256: 2e6a43a4bb12e97453b99e77463a4d544d6ed583b5f28e8d3fb1d37f5d614d5a



20251006_1545-after first round of washing.jpg

sha256: 9c63664c113db754e7fed460af8894323d095b6fdc578c65bb83a4c989c16dc0



20251006_1546-Cl- test-2.jpg

sha256: 0125c56f0d44db65ffcbff445e5dab290fb241df83b3f18aa0907503d5fa35db



20251006_1617-Cl- test-3.jpg

sha256: 14f71e2c0f11615bca784fb98d8bdda71ede256a2bc3cbb504b3b8e330783c28



20251006_1643-Cl- test-4.jpg

sha256: 23bae8a971c5bd2f58328587fa1b1b351fd67630d009a77f2dbab46f127e953c



20251006_1728-Cl- test-5.jpg

sha256: 7adfe51d0d4dbf94e1eb50ef909476b9fc4b0334c21c17edb1b834b9ba454fd1



20251006_1816-Cl- test-6.jpg

sha256: 8e4d702e4b8de29a4f728eaaa52fe852967deb331f19ee5b7782bb645b728283



20251006_2112-Cl- test-11.jpg

sha256: d33cee424bd159653080d7fe8d03e9641bc0c17ad3c6ae5863d9446252fed5da



20251006_1916-Cl- test-8.jpg

sha256: 054a01c3749f9549bd29d3d5c1e7f8ad53d0deac6a2a97dd5ddadd8c267c7a4e



20251006_2000-Cl- test-9.jpg

sha256: bfc477fe3d5d57846795bf2bf942f14f751969468d11cafc0ebd5bab4367aed6



20251006_1844-Cl- test-7.jpg

sha256: c00dafa7599e843eed0622d9c744e68c1cf800c7b9a468f10d49fbe2fa34629f



20251006_2038-Cl- test-10.jpg

sha256: e790d837d082df9c88c5c19b2a7bb127be0c66d8cf30a7bc38592f9d4cc4768d



20251006_2159-Cl- test-12.jpg

sha256: 45fe169f2ba4b71a8bb2807478ea0e205729013da88b592a047bbc429a76f63a



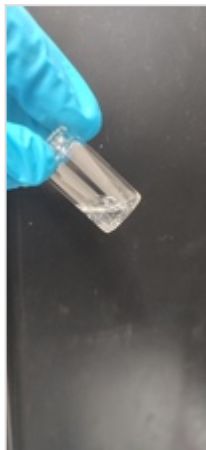
20251006_2245-Cl- test-13.jpg

sha256: 2b9f3e26ba5dcdd542c6f208260e27fcb815194d347718dbf11e4c07de781ace



20251006_2330-Cl- test-14.jpg

sha256: 6a09c880ecc9d0664001bc87214183749f873e1ed34f42c3fe378b3a3e063363



20251007_0019-Cl- test-15.jpg

sha256: 2f35e7831f46aef5f1b4d5dc9b990bcce00c78414aaa3a8d84192e60585844a8



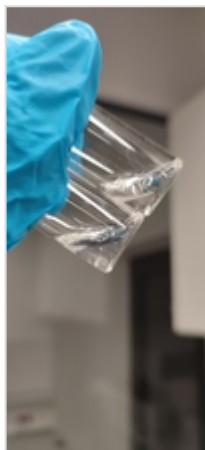
20251007_0138-last round of washing.jpg

sha256: 2cffdeab82c0d47b0297f65666046aff9d5927808e90e7b456b8c16b099b663b



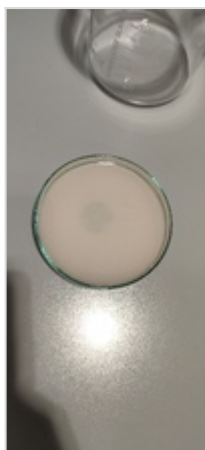
20251007_0144-Cl- test-16 next to water.jpg

sha256: ed8edae5e28fb485da6bd763131e1363cf8121a0a11f487484c77b744c702e72



20251007_0159-transferring the slurry into the dish for drying.jpg

sha256: 2b10bfac7409b45efde6f5b949839fd0dce671b5ccc4abef66c693482dc38ac7



20251007_0203-covering the dish with Al foil.jpg

sha256: 9d1dc6444bdd43f19611e4dae92432c1d46ac74427a47ee4419af97b1f2d9b61



20251007_1418-the dried sample.jpg

sha256: 9f292af8f6c56a157558623e017fce747850dfad561eb995c0203ce3626ede3



20251007_1432-EA-354-Al:SrTiO3-Osterloh-15times-scaleup.jpg

sha256: 0cd8fbfe4894019fdbf23cec2791a16223be1317e48ecfa125239e341b5f48fc



20251002_1143-end of the heating program.jpg

sha256: dc94d6c3b2b69aed71056a6cdf6406df99d48cb0e35e0f867c7c84d4af0b8bd1



20251002_1144-the crucibles after heat treatment in the furnace.jpg

sha256: 825de76985e13006c57f082f811bc8dfa2b1342aa4019a8ad76f569c6e6af4b3



Comments

On 2025-11-25 13:33:04 Nadzeya Brezhneva wrote:

01.10.2025 01.33: Please adjust the link (protocol for using furnace) to a Nabertherm muffle furnace in AWZ 304, the samples were calcined there, not in 1400 °C Nabertherm furnace

On 2025-11-25 19:06:30 Ebrahim Abedini wrote:

Ebrahim: Done.



Unique eLabID: 20251009-b3dd99a4a87e97ad845f6f011e6dd41cfc0b622a

Link: <https://elab.water-splitting.org/experiments.php?mode=view&id=3153>