AE-450: Synthesis of Ag2(17)O from AgNO3 and H2(17)O

Date: 2025-02-18
Tags: Ag2O | AE | reference procedure | AGNO3 | 17O
Category: Two-photon
Status: Done
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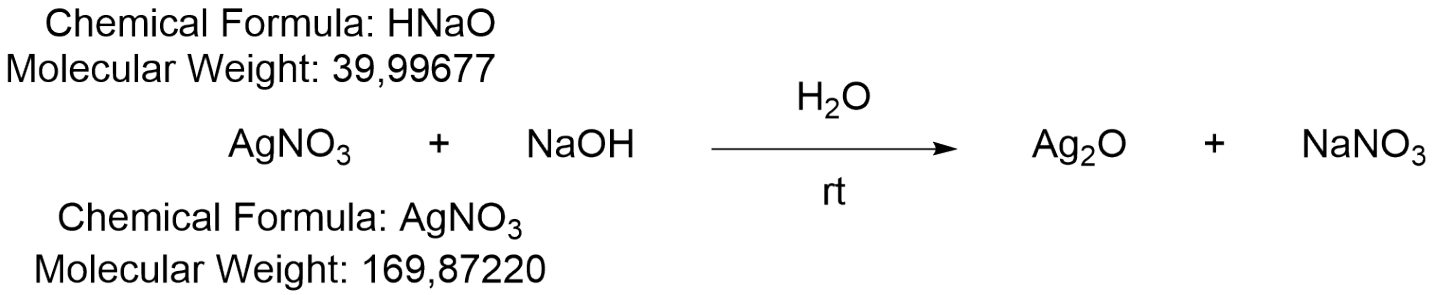
Reaction scheme/sample structure



Literature/reference experiments

Literature	Preperative_Inorganic_Chemistry_Volume_2_Brauer, Link to book, p.1037 and 10.15227/orgsyn.036.0046
Reproduction	/
Similar experiments	Two-photon - AE-443: Synthesis of Ag2O from AgNO3

Reagents

Ag217O-syn.xlsx for calcualtion

Name	CAS Number / Experiment Number	Inventory number	Amount [mmol]	Equivalents	$Mass_{theo}$ [mg]	$Mass_{exp}$ [mg]	Molar mass [g/mol]	Density (g/ml)	Volume [µl]
Silvernitarate	7761-88-8	/	2.26	1	384	384.15	169.872	/	/
Sodium hydroxide	1310-73-2	/	2.26	1	90.04	90.07	39.9971	/	/
H2(17)O 10 % in H2(16)O	/	/	/	/	/	/	18.20	0.998	155 + 95 + 5

Work-up and Analytical Reagents

Name	CAS Number / Experiment Number	Inventory number	$Mass_{exp}$ [g]	Volume [ml]	Concentration [M]
Milli-Q water	/	/	/	approx. 70	/

Procedure/observations

Date	Time	Step	Pictures	Observations
18.02	13:35	In a GC vials with screw caps AgNO3 (flask 1) was weighed		both white solids
		In a GC vials with 300 µL micro inserts with screw caps NaOH (flask 2) was weighed	left NaOH right AgNO3.jpg	
	13:50	To the flask 1H2(17)O 10 % in H2(16)O (155 µL) and to the flask 2 H2(17)O 10 % in H2(16)O (95 µL) was added using a 100 µL Eppendorf pipette		
	13:54	flask 2 was vortexed (Equipment - VWR® VV3, Vortex Mixer, stage 5/6, than 6/6) for approx. 3 min and than approx. 4 min		some solids remain at the bottom
	13:55	flask 1 was vortexed (Equipment - VWR® VV3, Vortex Mixer, stage 5/6) for approx. 1 min	left NaOH right AgNO3 after addition of water.jpg	clear solution
	14:05	to flask 2 H2(17)O 10 % in H2(16)O (5 µL) was added using a 10 µL Eppendorf pipette		
	14:04	flask 2 was vortexed (Equipment - VWR® VV3, Vortex Mixer, than 6/6) for approx. 1 min	after vortexing left NaOH right AgNO3.jpg	some solids remain at the bottom
	14:06 - 14:09	The mixture in flask 2 was taken up in a 100 µL Eppendorf pipette for approx. 30 times to yield dissolution of remaining solids	NaOH after Eppendorf.jpg	slightly cloudy solid
	14:09	The solution of flask 2 was added to flask 1 using a 100 µL eppendorf pipette	After addition of NaOH.jpg	after addition brown solid forms at layer
	14:12	The obtained mixture was vortexed (Equipment - VWR® VV3, Vortex Mixer, stage 2/6) for approx. 20 and than at stage 4/6 for approx. 2 min. In between the sample was shaken (approx. 3 times for 5 s)	after vortexing.jpg	after vortexing more brwon solid forms
		The vial was stored at rt in the dark	after reaction.jpg	bit darker, more blackish, mixture solidified
	15:35	To the mixture Milli-Q water (approx. 1 mL) was added using a squirt bottle		
		The mixtrue was vortexed (Equipment - VWR® VV3, Vortex Mixer, stage 4/6) for 10 s		dark suspension

		The suspension was transferred into a 14 mL centrifuge tube using a glas pipette		
		This was repeated with the 1 mL and 1.5 mL of water		
	- 15:40	The centrifuge tube was filled to 10 mL with Milli-Q water		
	15:45 -	The mixture was centrifuged (Equipment - Centrifuge Hettich EBA 20, 2 min, 6000 rpm) and the supernatent liquid was taken off using a glas pipette	after first centrifugation.jpg	clear liquid, dark solid
	- 16:15	The obtained solid was washed with Milli-Q water (6 * 10 mL) by centrifuged (Equipment - Centrifuge Hettich EBA 20, 2 min, 6000 rpm) and taken off the supernatent liquid	combined liquid phases.jpg	no changes in appreance
	16:20	After the last centifugation water (approx. 1 mL) was added in the tube and the suspension was transffered into a 5 mL snap on cap vial using a glas pipette. To get a good suspension the liquid was taken up into the pipette several times		
		To the tube water (0.3 and 0.1 mL) was added and the obtianed suspension was transffered into the vial		
		The supernatant soultion in the vial was taken off into the tube and shortly centrifuged.		
		Ther supernantant soultion in the tube was decanted and the remaining suspension was transffered into the vial	solids.jpg	balck slurry
	16:35	The top of the vial was covered with puntucered aluminium foil and placed in an drying oven over night at 95 °C		
19.02	07:50	The vial was removed from the oven capped and stored at room temerpature in the dark.		
20.02	09:00 - 12:40	The solid was dried under reduced pressure (Schlenk line) at rt		
		The solid was stored in the dark at rt		

Analysis

Product characterization

Sample	Mass [mg]	Purity	$Mass_{pure}$[mg]	Amount [mmol]	Yield [%]	Description	Image	Storage location
AE-450	225.02	/	/	0.97	86	dark dray, black solid	product.jpg	E004 SSC

Results

Product obtained in good yield

Linked experiment

Two-photon - AE-443: Synthesis of Ag2O from AgNO3

Linked resources

Equipment - VWR® VV3, Vortex Mixer

Equipment - Centrifuge Hettich EBA 20

Attached files

AE-443.cdxml
sha256: 9fd79b07a2c2244119f5827b96960f021b248a9d4b21eaea37527e0d6b109da6

Ag217O-syn.xlsx
sha256: 3c7ec664399b84e1dc5b5efc5507483fa3eeb8d60b01513db206cc764f0ea286

after-reaction.jpg
sha256: df77c0f0ac46fa7a634326b79d5115e39a9bd70914232bf5ace4b6ec69765617

after-first-centrifugation.jpg
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solids.jpg
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product.jpg
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combined-liquid-phases.jpg
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after-vortexing.jpg
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left-NaOH-right-AgNO3.jpg
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left-NaOH-right-AgNO3-after-addition-of-water.jpg
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NaOH-after-Eppendorf.jpg
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after-vortexing-left-NaOH-right-AgNO3.jpg
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After-addition-of-NaOH.jpg
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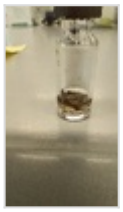

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Link: https://elab.water-splitting.org/experiments.php?mode=view&id=1771