



## **SOP for laboratory practice**

Example: General protocol for mesoporous SiO<sub>2</sub> synthesis

**Supplementary item No 6**



## Imprint

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The responsibility for the contents of this publication lies with the authors.

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Title of Procedure: <b>General protocol for mesoporous SiO<sub>2</sub> synthesis</b>	Revision Date: 2011-08-12
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<b>Scope and applicability</b>	
	Department, Lab or Center: NanologicaAB
	Research Group:
	Lab Bldg., Room(s): Drottning Kristinas Väg 45, 11428 Stockholm
	Operation/Experiment: Batch Protocol
	Material(s): <b>Mesoporous silica prepared by sol-gel route.</b>

<b>Special PPE Required:</b>				
<b>x</b>	Goggles	Type: according to EN 166-168, 170		
<b>x</b>	Face masks	Type: according to EN 149/2001	Filter: FFP3	
<b>x</b>	Protective Clothing			
<b>x</b>	Laboratory coat	Material: 65 % polyester, 35 % cotton (minimum)	Type:	
<b>x</b>	Gloves			
<b>x</b>	Material: nitrile	Type: according to EN 374	Thickness: 0.22 mm	Other
<b>x</b>	Material: para-aramide (PPTA)	Type: according to EN 374	Thickness: 0.22 mm	Other: Thermo-resistant
	Respirator (If yes, contact EHS Office for additional assistance)			

<b>Special precautions</b>	
	Permits: No
	Mgmt. Approval: No
	Training: No
	Medical Surveillance: No
	Other: No

Special emergency procedures	
Fire / Evacuation:	Use a fire extinguisher and active the fire alarm.
Chemical Spill:	Use plenty of water to wash it.
Medical Emergency:	If chemicals contact with any part of the body, wash the affected zone with plenty of water.
Personal Exposure:	No concerns if production is carried out in well-ventilated area or inside fume hood.

Steps-Operation	Equipment/Glasware
Preparation and dissolution of template agent (TA) and Reaction Vessel	PVC bottle (thermal resistant)
Synthesis, reaction	Mechanical stirrer
	Glass reservoir for mixing silica sources
Hydrothermal treatment	Heating jacket, Oven
Filtration	Porcelain Funnel (24 cm diameter) Flask (5 l) Filter paper (24 cm diameter)
Extraction	Heating-Stirring mantle Round-flask (1 l) Condenser
Calcination	Porcelain crucible (1 l)
	Calcination furnace
Packaging	Sealable Plastic bags or drums

Procedure		
Task	Hazards	Precautions
<b>1. Preparation of the solvent</b>	Mild effects in eyes and respiratory system	Use gloves, face shield and goggles.
<b>2. Weigh of TA</b>	Mild effects in eyes and respiratory system	Work inside the fume hood. Use gloves, face shield and goggles.
<b>3. Preparation of TA solution (Micelles). Heat and stir.</b>	Mild effects in hands	Work inside the fume hood. Use heat-resistant gloves, handle with care.
<b>4. Weigh of silica source</b>	Exposure to inhaled toxic vapors	Work inside the fume hood. Use gloves and goggles.
<b>5. Addition of the silica source</b>	Mild Effects in eyes Corrosion by spillage	Use gloves and goggles. Handle with care, clean with plenty of water in case of spillage. The use of a funnel is permitted in order to facilitate addition.
<b>6. Reaction (stirring)</b>	-	-
<b>7. Hydrothermal treatment (HT): (oven)</b>	Increased Pressure of Reaction Vessel	Use heat-resistant gloves, handle with care.

<b>8. Filter the synthesis mixture.</b>	Over-pressure in bottle Allow to cool properly.	Work inside the fume hood. Use heat-resistant gloves, handle with care. Open the reaction vessel very slowly.
<b>9. Extraction of the TA</b>	Acute Effects in eyes if in contact with solvents Corrosion by spillage	Work inside the fume hood. Use gloves and goggles, and face protection. Handle with care, clean with plenty of water in case of spillage. Ensure that condenser is securely attached.
<b>10. Filter the “extracted” silica</b>	Acute Effects in eyes if in contact with solvent. Corrosion by spillage	Work inside the fume hood. Use gloves and goggles, and face protection. Handle with care, clean with plenty of water in case of spillage. Ensure that condenser is securely attached.
<b>11. Drying of the “extracted” silica (oven)</b>	Risk of inhalation of silica particles from the drying cake.	Use gloves, face protection (mask) and goggles. Keep area well ventilated and the remaining cake covered with filter paper as to prevent airborne particles from escaping.
<b>12. Calcination</b>	Mild effects in hands and respiratory system if exposed to exhausts.	Use gloves, face shield and goggles. Keep area well ventilated and cover the crucible with filter paper as to prevent airborne particles from escaping.
<b>13. Packaging</b>	Danger to respiratory system if particles are inhaled in large concentrations.	Work inside the fume hood. Use gloves, face mask and goggles and work in well ventilated areas. Keep solid exposed to air for as short time as possible to limit concentration of airborne particles.