

**Standard Operating Procedure: Vapor deposition of
Chlorotrimethylsilane**

Synonyms : TMCS
Trimethylchlorosilane
Trimethylsilyl chloride

Formula : C₃H₉ClSi

Molecular weight : 108.64 g/mol

CAS-No. : 75-77-4

EC-No. : 200-900-5

Material is flammable, toxic, and reacts with water vapor. It may only be used in the vapor silanation rig inside a fume hood.

Date of last revision to SOP: 15 July 2020

1. Chlorotrimethylsilane

C₃H₉ClSi Safety Data Sheet

2. Approval Required

Users must undergo training before using this chemical.

3. Personal Protective Equipment

- **Protective eyewear** such as approved safety glasses, goggles required.
- **Lab coat** required
- **Rubber, neoprene, or nitrile gloves** required.

4. Engineering/Ventilation Controls

- Only use in **fume hood**. Use on the open bench is prohibited.
- Only open in nitrogen atmosphere. Reacts with water vapor in the air.

5. Chemical Handling, Storage, Cleanup and Disposal Requirements

- Conditions to avoid: Combustible; avoid contact with heat, sparks or open flame.
- Incompatibility: Reacts with water and moisture in air, liberating hydrogen chloride. Avoid contact with alcohols, acids, and oxidizers.
- Hazardous decomposition products: Organic acid vapors, hydrogen chloride, and fluorinated organics.
- The vial must be stored in a completely sealed container. Visually examine for leak before opening the outer container.
- In case of spill: Cover spill with absorbent material. Transfer to a suitable container for disposal. Report to EH&S for assistance.
- Disposal must be done through EH&S. No chemical wastes can go into the sewer system, trash or be allowed to freely evaporate.

This protocol involves a dangerous chemical, refer to the SOP and MSDS prior to handling it

Do not perform this protocol if you have not been trained by an authorized user

This protocol is intended for gas-phase monolayer deposition of a silane, to serve as a counter-adhesion agent, for glass or silicon substrates. The chemical “tridecafluoro-1,1,2,2-tetrahydrooctyl-1-trichlorosilane” will be referred to as silane for the purposes of this protocol. It can be deposited onto silicon and glass substrates. It will etch metal oxides (e.g. alumina).

***The silane reacts with water vapor in air to create hazardous vapor. Do not handle outside the fumehood. ***

PROCESS DESCRIPTION

This process utilizes a custom-fabricated vapor deposition chamber shown below in figure 1.

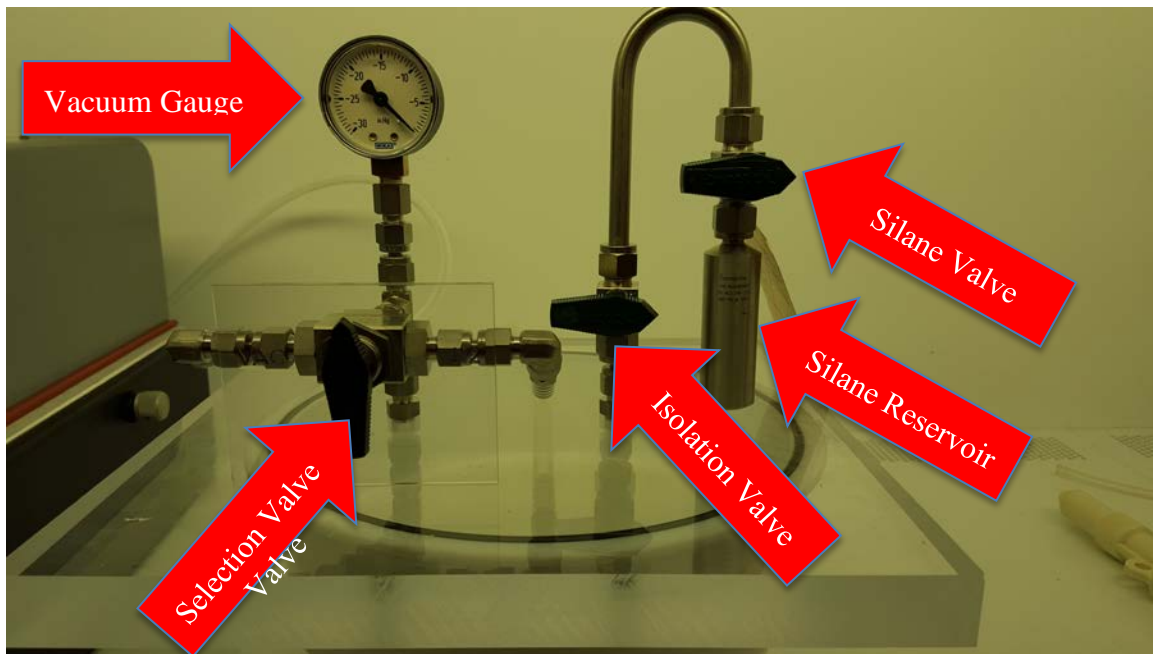


Figure 1- Silane vapor deposition chamber

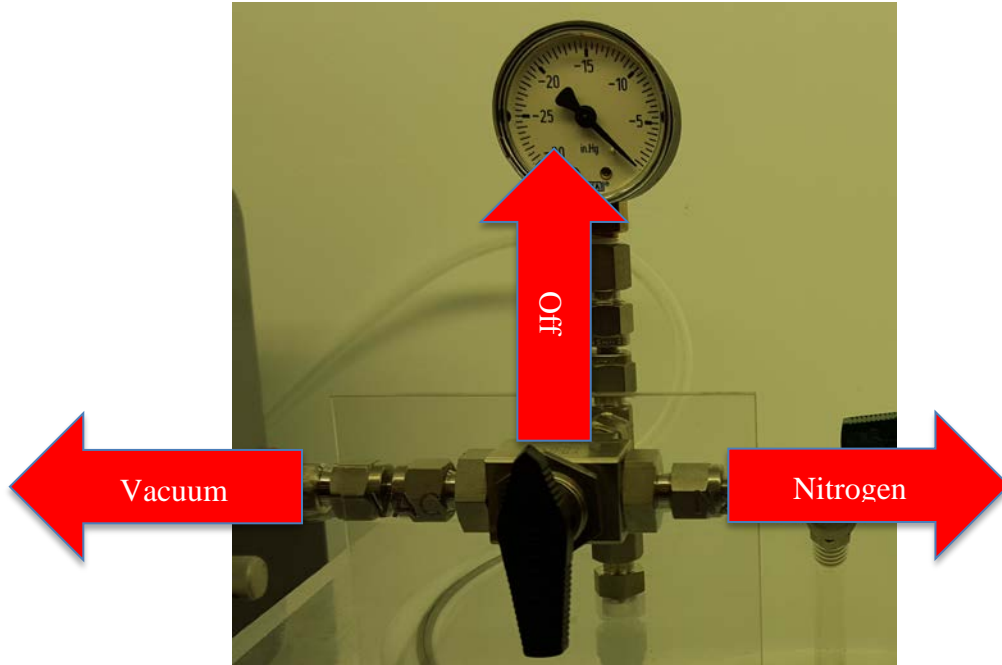


Figure 2- Selector valve settings

The chamber is connected to the fume hood's vacuum and Nitrogen gas fittings shown in figure 2.

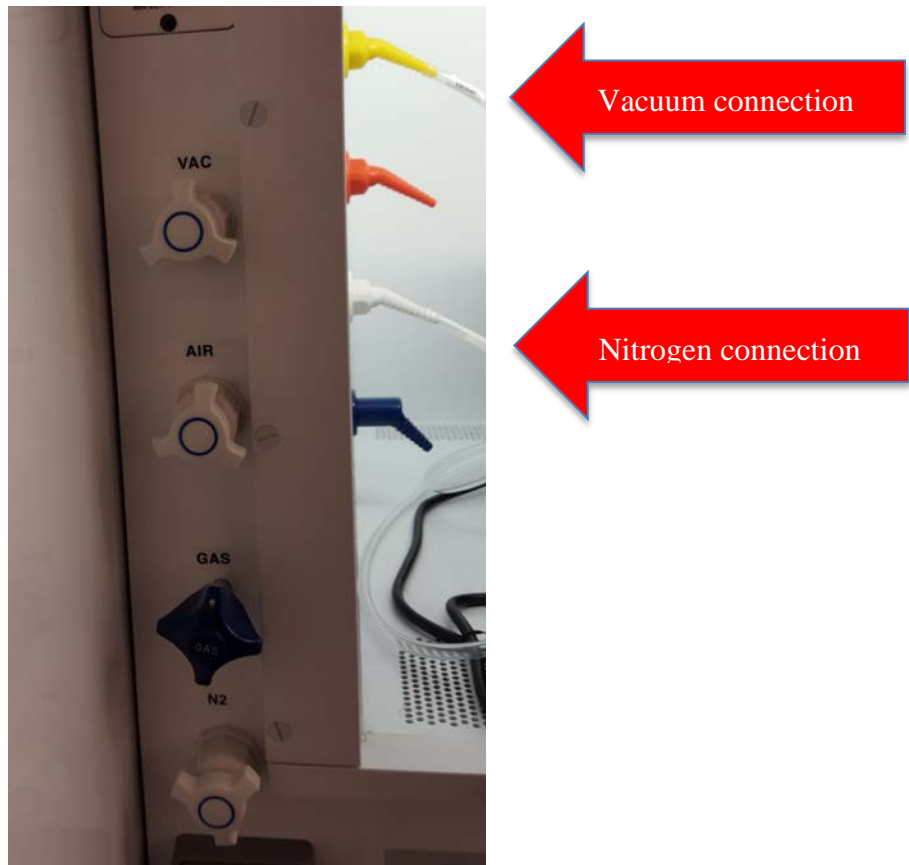


Figure 3- Gas valves and fittings

OPERATING PROCEDURE:

SETUP

1. Connect Nitrogen hose to the hose barb. The hoses are labeled.
2. Verify that the vacuum line is connected to the vacuum pump and turn the pump on.
3. Turn the selector valve to the **OFF** position (pointed up).
4. Make sure that the isolation and silane valves are **OFF** (pointed horizontally).
5. Open (CCW) the fume hood Nitrogen valve 1-2 turns.
6. Open (CCW) the fume hood Vacuum valve all the way.
7. Place samples in the vacuum chamber.
8. Check the O-ring seal, wipe with Kimwipe if dirty.

TEST VACUUM

9. Open the isolation valve
10. **Turn the selector valve to VACUUM** for ~ 30 seconds, then to **OFF**.
11. Note vacuum gauge reading.
12. Wait 1-2 minutes – vacuum should not change.

PURGE CHAMBER

13. **Verify that the isolation valve is open** (pointed vertical).
14. Turn selector valve to **NITROGEN** until gauge pressure reads zero (atm.).
15. Turn selector valve to **VACUUM** until gauge stops moving (25-30 in. Hg typ.).
16. Repeat filling the chamber with Nitrogen and evacuating 5 times.
17. Leave the selector valve in the **VACUUM** position.

DEPOSIT SILANE

18. **Open the Silane valve** exposing the Silane to the chamber.
19. **Move the selector valve to OFF.**
20. Wait 30 – 60 minutes, but not longer than 60 minutes.
21. Leave a note on the deposition rig with your name, phone number and the time the run will be finished.

PURGE CHAMBER

22. **Cycle the selector valve between Nitrogen and Vacuum 5x** to evacuate the Silane from the chamber.

SHUTDOWN

23. Move all valves to OFF position (selector valve pointing up, isolation and silane valves pointing sideways. (as shown in figure 1).
24. Close the fume hood vacuum and Nitrogen valves.
25. Remove the note from the chamber.