Usage Policies Notebook for 5” Nano Multi-Purpose Furnace

Revision date

December 2014

Emergency Plan for 5” Nano Multi-Purpose Furnace

Standard Operating Procedures for Emergencies

Contact information

Person	Phone number
Lab Manager	Jake Hes, 949-824-8239 (day), 562-522-8328 (alternate)
Director	G.P. Li: 949-824-4194 (day), 949-824-2047 (alternate)
Staff	Mo Kebaili: 949-824-8239 (day), 949-494-5892 (alternate)
Super User	Carlos Ruiz (818) 527-6349 (Anytime, voicemail or text only)

Hazardous chemicals, gases, and conditions

Hazard name	Description of hazard
High voltage	Electrical shock, ignition source
CH_4 (Methane)	Flammable, Non-corrosive
H_2 (Hydrogen)	Flammable, Non-corrosive
N_2 (nitrogen) gas	Asphyxiant Non-flammable,
Ar (Argon)	Non-flammable, non-toxic

Alarms or indications of danger

Alarm type	Condition and response
Gas Detector (Enmet)	
H_2 and CH_4	
O_2 flow Alarm	Problem with process state. Halt process and correct problem or notify the staff or the lab manager before continuing.
Pungent or foul smell	Gas leak. Shutdown the tool at once and evacuate the area. Contact the staff and the lab manager.

Emergency shutdown plan #1

In the event of an emergency, when there is very little time, *press the large red emergency shut-off button at the entrance of the room l*, this will shutdown the gas system, and will stop the gas flow. Leave the facility at once, and then contact the lab manager or the staff.

Emergency shutdown plan #2

In the event of an emergency, when there are a few minutes available, *turn off the gas switches on the gas panel, the control power and the main power*. This will stop gas flow into the system. If there is no fire, and no smell of gases, enter the room and close off all gas cylinders by turning them fully clockwise. Check the oxygen tank in room w2349, feel the door for possible fire, and if safe, close the oxygen tank by turning fully clockwise. Leave the facility at once, then, contact the staff and the lab manager.

IMPORTANT INFORMATION

The 5" nano furnace is an engineering prototype that was designed and assembled in the INRF cleanroom for R&D applications. Since the furnace is not a commercially manufactured tool, it hasn't been completely validated and tested the way a commercial tool is validated during development and design cycle hence the 5" nano furnace doesn't have all the fail-safe mechanisms, which are normally standard in commercial tools. The INRF staff has done preliminary validation and testing, and has identified issues that have been addressed to make sure that the 5" nano furnace is safe to operate. Not all potential issues have been identified or addressed, so the validation and testing is an on-going process. When new issues with the 5" nano furnace are identified, corrective actions will be implemented or recommended.

Hydrogen and methane combustible gases are used at atmospheric pressure in the 5" nano furnace to grow carbon nanotubes. To avoid any potential fire or explosion, it is important to understand the lower and upper explosive limits of these flammable gases.

A combustible gas will ignite only if the following three conditions are met:

- There is enough combustible gas to start a fire
- There is enough oxygen to oxidize the combustible gas
- There is a source of energy to ignite the combustible gas

If all of the above three conditions are not present at the same time, then the combustible gas will not ignite and sustain a fire.

To have enough combustible gas to start a fire means that the concentration of the combustible gas present has to be between its lower explosive limit (LEL), or lower flammable limit (LFL) and its upper explosive limit (UEL), or upper flammable limit (UFL).

For hydrogen:

- 100% LEL is equal to 4% hydrogen by volume in air
- The UEL is equal to 75% hydrogen by volume in air

For methane:

- 100% LEL is equal to 5% methane by volume in air
- The UEL is equal to 16% methane by volume in air

Hydrogen and methane will auto ignite in the presence of oxygen at a temperature around 500°C.

If the combustible gas concentration is 100% LEL or higher up to the UEL, then there is enough combustible gas present that could ignite and sustain a fire.

When the 5" nano furnace is used to grow carbon nanotubes, we already meet two of the three conditions to have a potential fire or explosion:

Inside the quartz tube:

- We have enough combustible gas (hydrogen and methane)
- We have the energy source (furnace temperature at 900°C)
- The missing condition is the presence of oxygen

It is important to not allow oxygen to get inside the quartz tube when the carbon nanotubes process is being run. Oxygen has the possibility to get inside the quartz tube from the gas injector inlet port, or from the end cap.

Outside the quartz tube:

- We have enough oxygen
- We have the energy source (furnace temperature at 900°C)
- The missing condition is the presence of combustible gas

The quartz tube end cap is connected to stainless tubing via Teflon tubing. This allows the exhausted combustible gases to cool down before they enter the scavenger box.

The INRF staff has already established that once the combustible gases enter the scavenger box, they are diluted to below their LEL levels, and this is only true if the exhaust blowers are active.

To prevent a potential fire or explosion, we need to keep the oxygen from getting inside the quartz tube through the gas injector port, and we need to keep the combustible gases from getting outside the quart tube through the end cap.

Usage Policies for 5” Nano Multi-Purpose Furnace

Standard policies for usage

The 5″ nano furnace can accept up to 5″ substrate to grow carbon nanotubes. Mass flow controllers digitally control H_2, CH_4, N_2, and Ar gas flows. The programmable temperature range is up to 1000°C.

Contact information

The INRF staff or the lab manager can be reached at (949) 824-8239 or (949) 824-9831.

Authorized users

Only INRF registered users who have completed the training and passed the certification can use this equipment. Users can only use the portion of the system for which they have been trained.

Training

Users must have received direct training from the staff in order to use this equipment. Users are expected to understand the nature of the system, as well as the proper control and use of the gases. Training varies slightly, depending on the process to be performed. Contact the staff for details and to arrange for a training session.

Usage log sheets

Users are required to log all activities in the log sheets provided. All users must log in when they used the furnace (date and time), which gases they used, and when they completed their process in the user log sheets. If users notice anything unusual, they should record it in the user log sheets, and add details in the main comments area on the log sheet. Any maintenance to the tool will be logged in the maintenance log sheet (maintenance staff only).

Safety equipment

As safety equipment for use on this equipment, cleanroom gloves and tweezers should be used when handling pieces in the tube. Care should be taken to avoid burns when working near the furnace tube. The users need to use the gas leak detector to make sure that no gas is leaking from the furnace tube end cap.

Standard equipment and materials

The laboratory provides the following gases: N_2, H_2, CH_4, and Ar. Other gases must be cleared with the lab manager.

User maintenance *

To keep the furnace contaminant free, keep the end cap on the tube when it is not in use to keep a positive pressure within the furnace tube under N_2 flow.

Waste disposal

Dispose of alcohol soaked wipes in a waste container marked for flammable solid waste.

Scheduling

Reservation can be done online, and the system can be used on a first come, first served usage if no reservation was made.

Other issues

Users should remain physically present in the cleanroom facility during the entire use of the 5" nano furnace.

At no time should a user adjust a pressure regulator on a gas line. Gas control should be "on" or "off" only, using only the appropriate valves. For most gases, this is usually the valve at the cylinder head.

Non-standard use

Users may not modify any hardware on this equipment. For use of non-standard processes, gases or materials, contact the staff or the lab manager.

Usage Notes for 5” Nano Multi-Purpose Furnace

Guide for using the 5” Nano Multi-Purpose Furnace

Gas Cylinders

All gas cylinders should be turned on or off at the cylinder valves. At no time should a user adjust a pressure regulator. Clockwise for all valves, means CLOSED. The standard off configuration for the system is to close the cylinders valves, but leave all other valves alone.

System Setup

The 5” nano furnace can accept up to 5” substrate to grow carbon nanotubes. Mass flow controllers digitally control H_2, CH_4, N_2, and Ar gas flows. The programmable temperature range is up to 1000°C.

When the 5” nano furnace is in the idle mode, 2.5 L/min of nitrogen gas flows in the quartz tube. This nitrogen flow maintains a positive pressure in the quartz tube to keep it clean from particulates contamination.

Mass-Flow Controllers Setting

There are four mass flow controllers dedicated for H_2, CH_4, N_2, and Ar used by the 5” nano furnace. Each mass flow controller can be digitally set to maintain a preset gas flow.

Use the table below to preset the gas flow:

GAS CHANNEL	GAS SETTING DISPLAY	ACTUAL GAS FLOW
N_2 -- NITROGEN	**(Desired Value) / 100**	**Desired Value in sccm**
Ar -- ARGON	**(Desired Value) / 100**	**Desired Value in sccm**
CH_4 -- METHANE	**(Desired Value) / 70**	**Desired Value in sccm**
H_2 -- HYDROGEN	**(Desired Value) / 50**	**Desired Value in sccm**

Example:

GAS CHANNEL	GAS SETTING DISPLAY	ACTUAL GAS FLOW
N_2 -- NITROGEN	**(2500) / 100**	**2500 sccm**
Ar -- ARGON	**(1000) / 100**	**1000 sccm**
CH_4 -- METHANE	**(3500) / 70**	**3500 sccm**
H_2 -- HYDROGEN	**(500) / 50**	**500 sccm**

Safety Check

The 5″ nano furnace is monitored by a set of combustible gas leak detectors. There is a combustible detector installed in the back of the furnace where the mass flow controllers are located. There is a second combustible gas detector installed above the wafer loading area of the furnace front.

The users should verify that the exhaust scavenger has a negative flow.

The users should check for any potential leak of combustible gases with the handheld leak detector, at the injector gas inlet port and at the front-loading area of the 5″ nano furnace.

In case of a potential combustible gas leak, the users should abort their process, they should stop flowing combustible gases inside the 5″ nano furnace, by setting the hydrogen and methane mass flow controllers to zero. The users should immediately contact the INRF staff or the lab manager.