# QRYSTAL-CVD-1100
# Chemical Vapour Deposition System

Specification sheet

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New Delhi, India

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# 1 Multi-zone 1100 °C furnace

## 1.1 QRY-820 multi-zone 1100 °C stationary furnace system

### 1.1.1 QRY-911 : 2" diameter, 1200 mm long process tube

**Process tube specification**

- Material: Transparent quartz
- Dimension: OD = 48 mm x ID = 45 mm x L = 1200 mm
- Maximum temperature
  - Transient: 1200 °C
  - Continuous: 1100 °C

### 1.1.2 EM-126, -128, -12C : Furnace with 1, 2 or 3 independently heated zone

**EM-126: 1 zone (6 subzones) 1100 °C, split tubular furnace**

- Number of independently controlled subzones: 6
- Length of the zone: Configurable and computer controlled.
- Max hot zone length: 450 mm
- Heating element: Kanthal-A1
- Temperature sensor: N-type Thermocouple

**EM-128: 2 zone (8 subzones) 1100 °C, split tubular furnace**

- Number of independently controlled subzone: 8
- Length of each zone: Configurable and computer controlled.
- Max hot zone length: 600 mm
- Heating element: Kanthal-A1
- Temperature sensor: N-type Thermocouple

**EM-12C: 3 zone (12 subzones) 1100 °C, split tubular furnace**

- Number of independently controlled subzone: 12
- Length of each zone: Configurable and computer controlled.
- Max hot zone length: 900 mm
- Heating element: Kanthal-A1
- Temperature sensor: N-type Thermocouple

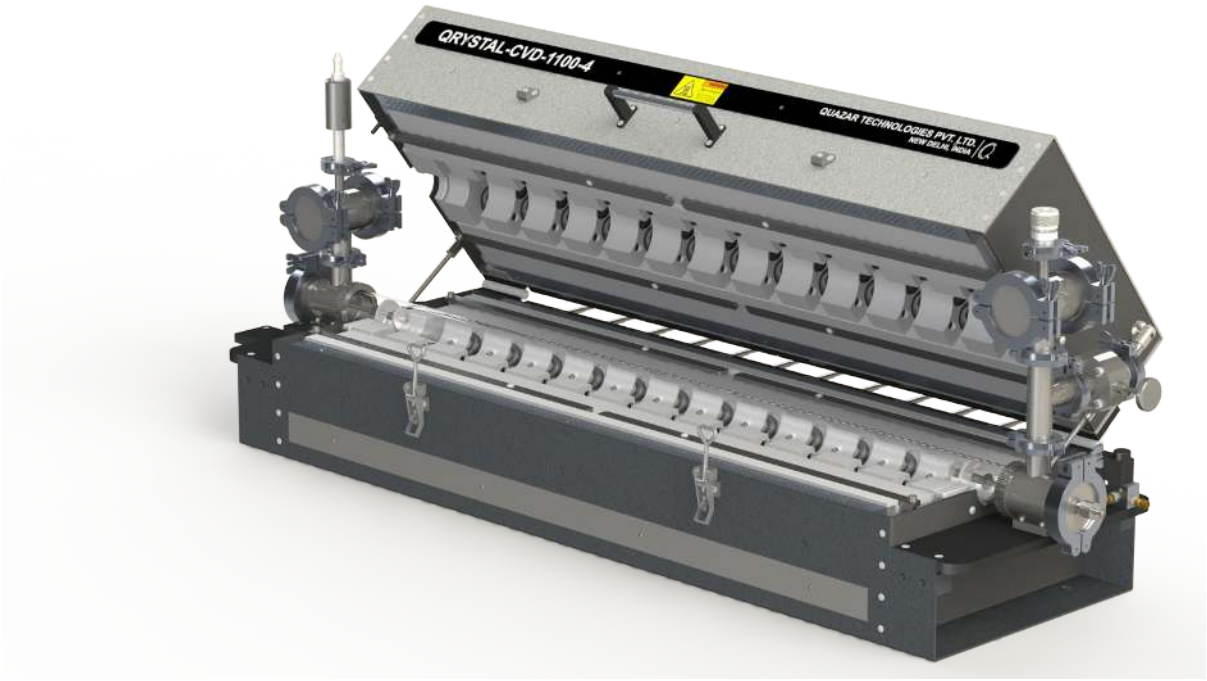



Figure 1: QRY-12C:3-zone, 1100 °C, split tubular furnace with gas and vacuum input-output ports

**Furnace skin temperature:** Less than 20 °C above ambient
**Note:** In order to achieve temperature uniformity within each independently controlled heating zone, they are further divided into 75mm long subzones, each having its own temperature sensor and heater. *The user can set the temperature of each subzones independently to get the desired heating zone length and temperature profile/gradients*

### 1.1.3 EE-102 12-channel temperature controller

- Temperature setpoint resolution: 0.1 °C
- Isothermal temperature stability: Better than 0.1 °C
- Maximum temperature
  - Transient: 1200 °C
  - Continuous: 1100 °C
- Number of temperature sense channels: 15
- Number of temperature controller PID loops: 12
- Number of temperature ramps and dwells/holds: Unlimited

- Maximum ramp-rates at full-power.
  - Better than 280 °C/min. at 30 °C for a single subzone
  - Better than 100 °C/min. up to 300 °C
  - Better than 50 °C/min. up to 600 °C
  - Better than 10 °C/min. up to 900 °C
  - Better than 5 °C/min. at maximum temperature
  - Negative temperature ramps better than -5 °C/min above 500 °C.
  - Negative temperature ramps better than -1 °C/min below 500 °C. The controlled negative ramps are limited by the rate of natural cooling.

## 1.2 Furnace temperature profile

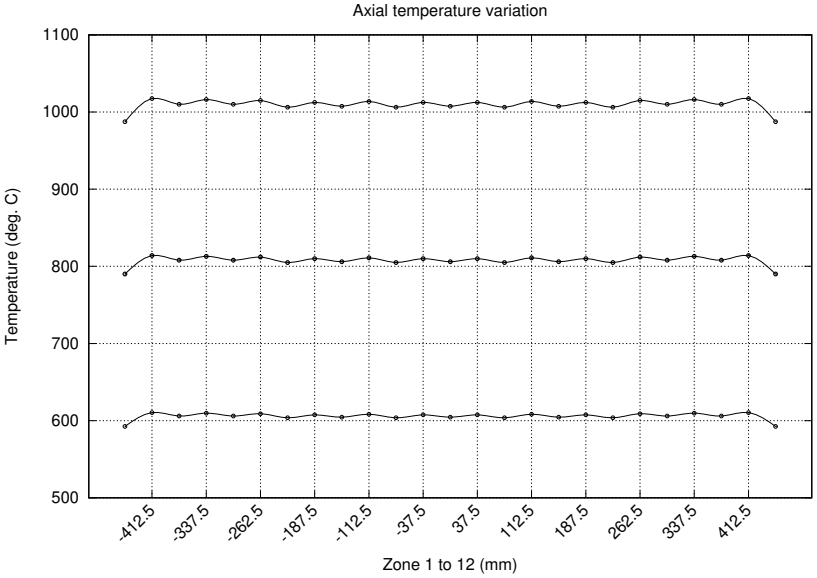
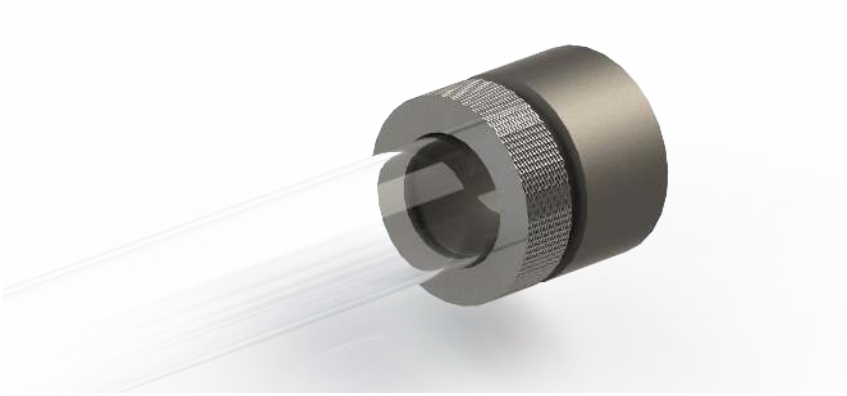



Figure 2: Axial temperature variation at different temperature (12 zone furnace)

## 1.3 Adapter for smaller diameter tube (optional)

| Description | Value |
|---|---|
| Tube OD | Up to 30 mm |
| Tube length | 1160 mm |
| Adapter OD | 48 mm |
| Construction | SS 316 |
| O-ring material | Viton |

(a)



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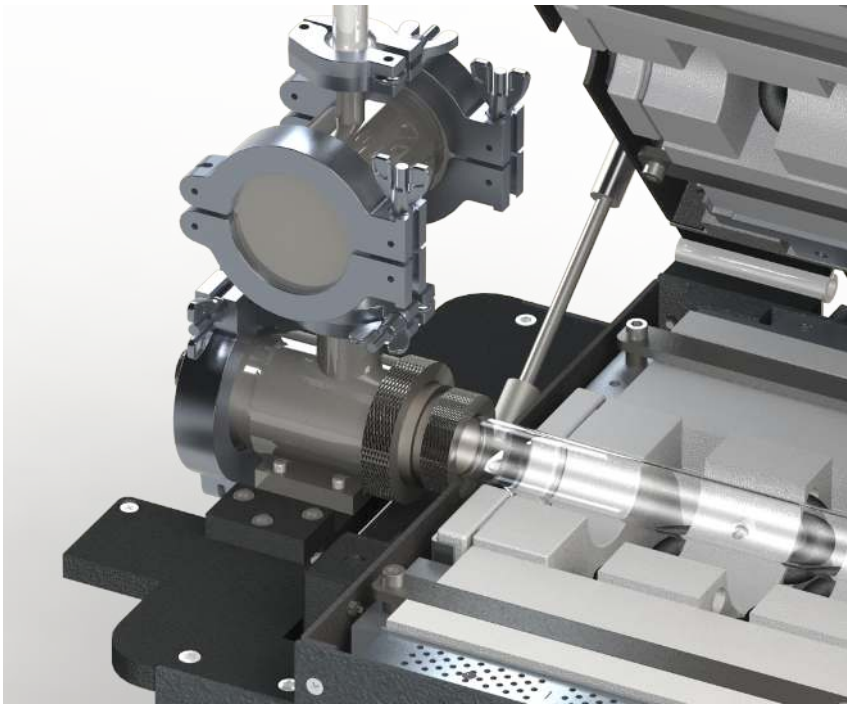


Figure 3: Adapter for smaller diameter tube. **(a)** Tube mount, **(b)** Mounted on the furnace

# 2 CVD framework

## 2.1 QRY-810 CVD framework for stationary furnace

The framework provides the foundation structure of the CVD system. All modules and subsystems are attached to the framework. The framework consists of the following items

- QRY-110 CVD chassis: It consists of two 19" instrumentation racks to hold all electronic modules. It also acts as the base for the furnace.
- EE-902 Supply controller: Ensures optimal distribution of power to the different parts of the system. It has built-in miniature circuit breakers (mcb) and contactors to implement safety interlocks. The supply controller can handle upto 64A of current sufficient for the all models of the QRYSTAL system.
- EE-903 Chassis controller: The chasis controller consists of a data acquisition system to record various process parameter for eg. Vacuum level. It also controls vacuum pump and purge valve.
- EE-904 System controller with monitor, keyboard, mouse: It provides synchronization and control of the various consituent modules of the CVD system through user friendly graphical interface. It also enables automation of the synthesis process by incorporating preset and user defined recipes.

## 2.2 QRY-811 CVD framework for movable furnace

- QRY-111 CVD chassis for movable furnace
- EE-902 Supply controller
- EE-903 Chassis controller
- EE-904 System controller with monitor, keyboard, mouse

# 3 QRY-830 : $10^{-3}$ mbar CVD vacuum system

## 3.1 QRY-141 vacuum components

- Process tube vacuum coupling with replaceable metal filters for pump protection
- Vacuum level sensor (mounted at the end of the process tube):
  - Pirani gauge ($10^{-3}$ to 0.5 mbar)
  - Linear sensor: 0.1 mbar to 1000 mbar
- Chamber isolation: Butterfly valve
- Release valve
- Purge valve

## 3.2 STD-VP-103-250 : Double stage rotary vane vacuum pump

- Pump type: Two stage rotary pump
- Pump throughput: 15 $m^3$/hour
- Ultimate vacuum: Better than $10^{-2}$ mbar

# 4 QRY-840 : MFC system, expandable up to 4 channels

Supports upto 4 MFC channels which can be chosen as per user requirements from the MFC's listed below:

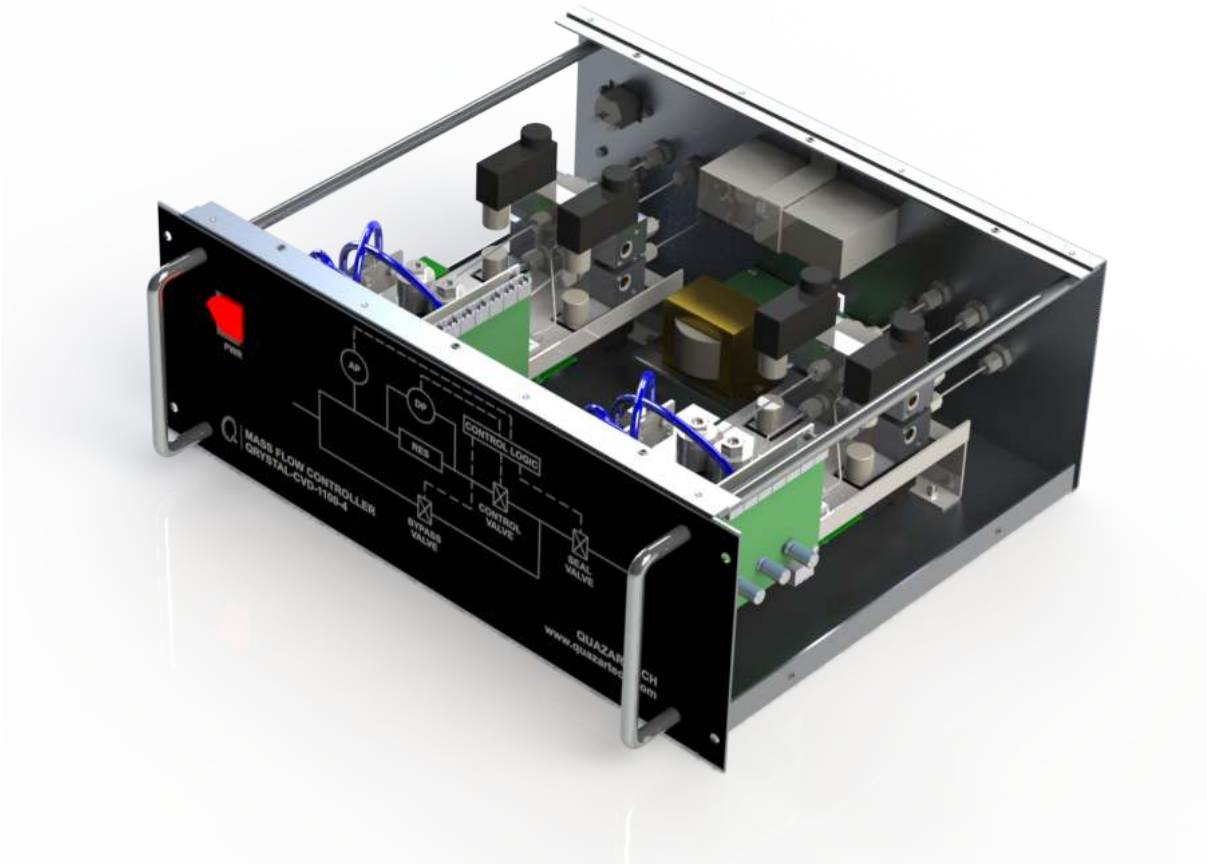



Figure 4: 4-channel mass flow controller module

## 4.1 EM-201 : 0 - 10 sccm flow

- Supported gases: Nitrogen, oxygen, hydrogen, helium, argon, air, and methane.
- Flow: 0 – 10 sccm
- Setpoint resolution: Better than 0.05% of the full-scale
- Stability: Better than 0.1% of the full-scale
- For flushing, a flow of 5000 sccm or more is provided with an on/off valve, installed in parallel to the MFC.

- Perfect shut-off is ensured with another on/off valve installed in series with the MFC and flush valve.
- The tube outlet can be purged through an on/off valve

## 4.2 EM-202 : 0 - 100 sccm flow

- Supported gases: Nitrogen, oxygen, hydrogen, helium, argon, air, and methane.
- Flow: 0 – 100 sccm
- Setpoint resolution: Better than 0.05% of the full-scale
- Stability: Better than 0.1% of the full-scale
- For flushing, a flow of 5000 sccm or more is provided with an on/off valve, installed in parallel to the MFC.
- Perfect shut-off is ensured with another on/off valve installed in series with the MFC and flush valve.
- The tube outlet can be purged through an on/off valve

## 4.3 EM-203 : 0 - 1000 sccm flow

- Supported gases: Nitrogen, oxygen, hydrogen, helium, argon, air, and methane.
- Flow: 0 – 1000 sccm
- Setpoint resolution: Better than 0.05% of the full-scale
- Stability: Better than 0.1% of the full-scale
- For flushing, a flow of 5000 sccm or more is provided with an on/off valve, installed in parallel to the MFC.
- Perfect shut-off is ensured with another on/off valve installed in series with the MFC and flush valve.
- The tube outlet can be purged through an on/off valve

# 5 Liquid precursor vaporizer integrated with the CVD: LPV-01

| Description | Value |
|---|---|
| Container | 22mL Quartz tube |
| Elastomers | Teflon and viton |
| Maximum operating temperature | Better than 150 °C |
| Temperature setpoint resolution | 1 °C |
| Temperature stability | Better than 0.1 °C |
| MFC flow-rate | User configurable |
| Flow modes | Bypass, bubbling, head-space sampling |
| Transfer line | Heated |
| Isolation | An on/off valve ensures LPV is completely cut-off when not in use |
| Control | Fully computer controlled |

Figure 5: LPV-01, liquid precursor vaporizer

# 6 User interface and recipe editor

Figure 6: User interface of Qrystal

- The system has an embedded control software, named Qrystal, to monitor and control the following parameters of the system. All the monitored parameters are recorded.
  - Zone temperature as pseudo-colored bar graph
  - Heater Power
  - Process tube pressure (i.e. Pirani and gauge pressure)
  - MFC inlet pressure
  - MFC flow setpoint and actual flow
  - Real-time plots
  - Flush and flow shut-off status
  - Pump monitor/control
  - Purge monitor/control
  - Recipe load, edit, save, and run control
  - LPV temperature
  - LPV transfer line temperature
- User can program their recipe using Python scripts which gives the user the capability to sequentially control the zone temperatures, ramps, flow rates, flushing of gases, and the

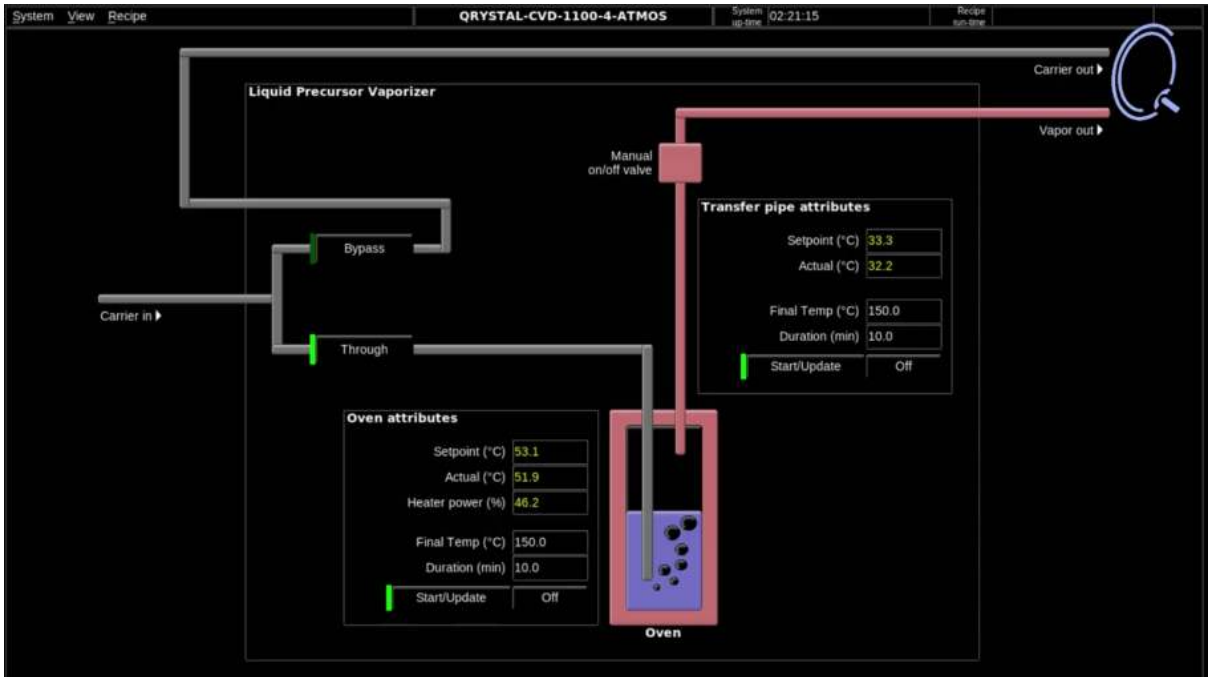



Figure 7: User interface of the integrated LPV

turning on/off of the purge & vacuum pump, LPV parameters, etc. at user defined times. This allows the CVD to be operated without the presence of the user. In case the CVD encounters a problem, the user will be notified through an integrated alarm system.

- Advanced user can use SDK to develop custom applications for remote monitoring and control using Modbus TCP/IP over LAN.

Listing 1: Example of a recipe

```
'''
A typical baking recipe.

        * Ramp up to 500 deg.C in half an hour
        * Hold there for 30 minutes
        * The tube is evacuated continuously

'''

from qrystal import Recipe

recipe = Recipe()

ev1 = recipe.system_initialize (begin_time="00:00:00")
```

```
ev2 = recipe.tcon_initialize_ramps (begin_time="00:00:00")

ev3 = recipe.tcon_mains_switch_on (begin_time="00:00:00")

ev4 = recipe.chss_ctrl_pump_on (begin_time="00:00:00")

zones = [0, 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11]

ev5 = recipe.tcon_begin_linear_ramps (
        begin_time="00:00:00", zone_idx_list = zones,
        end_temp_deg_C=500, duration="00:30:00")

ev6 = recipe.tcon_begin_linear_ramps (
        begin_time=ev5.end_time(), zone_idx_list = zones,
        end_temp_deg_C=500, duration="00:30:00")

ev7 = recipe.chss_ctrl_pump_off (begin_time=ev6.end_time())

ev8 = recipe.tcon_mains_switch_off (begin_time=ev6.end_time())

ev9 = recipe.system_initialize (begin_time=ev8.end_time())

recipe.run()
```

# 7 Accessories

- QRY-910 : 2” diameter, 1160mm long process tube
- QRY-920 : Replaceable downstream filters
- QRY-930 : Extra radiation shield
- QRY-940 : Toolkit
- QRY-950 : Center ring and O-ring
  - QRY-951 : KF-10 center ring with o-ring
  - QRY-952 : KF-25 center ring with o-ring
  - QRY-953 : KF-50 center ring with o-ring
  - QRY-954 : O-ring 48-54 for quartz tube
- QRY-960 : External thermocouple for calibration

# 8 Safety and interlock specification

1. As soon as the furnace lid is opened, mains supply to the heater is disengaged.
2. Heater mains can be engaged only if furnace temperature is within normal range, viz. 0 °C to 1200 °C.
3. An MFC can be activated
   (a) If process tube is not over-pressurized, viz. tube pressure <5 psig
   (b) If its inlet pressure stays above certain threshold, viz. 30 psig
4. Pump can be switched on only when purge is closed.
5. Purge can be opened if
   (a) The pump is off
   (b) Tube pressure is greater than certain threshold, viz. 0 mbar
6. During purge operation, tube is always maintained under positive pressure, viz. between 0 mbar and 100 mbar, to prevent back flow of oxygen from atmosphere.

# 9 Deliverable

- Oven assembly
  - Quartz tube
  - 12-zone Furnace
  - Gas supply and vacuum manifold
- Controller assembly
  - System controller
  - Supply controller
  - Chassis controller
  - Multi-channel temperature controller
  - Multi-channel mass flow controller
- Liquid Precursor Vapourizer
- Vacuum pump
- Qrystal software suite

**Accessories**

- Quartz tube
- Alumina boat
- Exhaust pipe
- Gas connection pipe

## 10 Installation requirement

- Floor area (W × D × H) : 1800 mm x 800 mm x 2000 mm
- Power supply
  - The system needs single phase, 40 Amp, 220 VAC. The supply wire must have at least 10 $mm^2$ cross section area (copper).
  - The system is to be permanently connected to a dedicated MCB, located within 5 meter distance from the installation area.
  - Grounding: Neutral voltage should not be higher than 5 V w.r.t ground.
- Gas supply
  - Cylinders, along with pressure regulators, may be placed as per user's convenience. Please note that these items are not supplied with the product.
  - Gas supply ports should be accessible to within 5 meter distance from the installation area.
  - Gas supply port should be compatible with 1/8" OD SS tubing, as shown in Figure 8.
- An exhaust system or access to an open area should be made accessible within 10 m of the CVD installation location. The system comes with a 10 m long and 19 mm wide PVC exhaust pipe to carry the gases to either your existing exhaust system or an open area.

(a) (b)

Figure 8: Gas supply essentials. (a) A double stage pressure regulator. (b) A standard 1/8" Swagelok adaptor for 1/8" OD SS pipe. This port should be available either directly on the cylinder pressure regulator, or at the access point in the gas distribution network.

# 11 Recipes

## 11.1 Molybdenum Disulphide ($MoS_2$)

### 11.1.1 Characterization

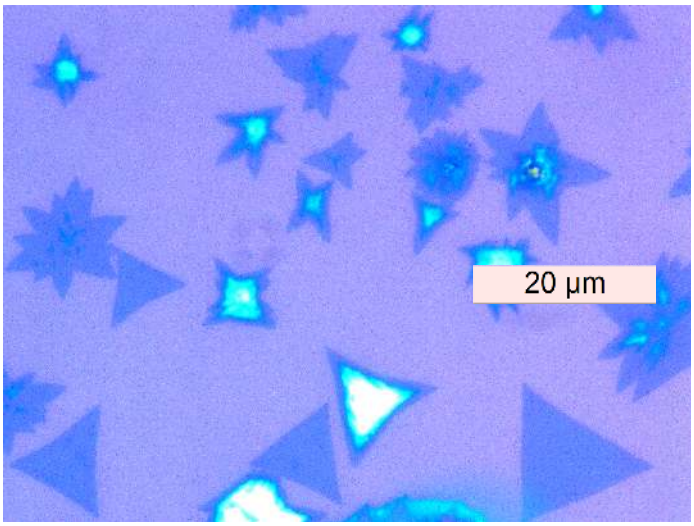



Figure 9: Triangles of CVD deposited $MoS_2$ on SiO2/Si waffer as seen under a top illuminated optical microscope. The image was taken at Prof. Pintu Das's lab, IIT Delhi.

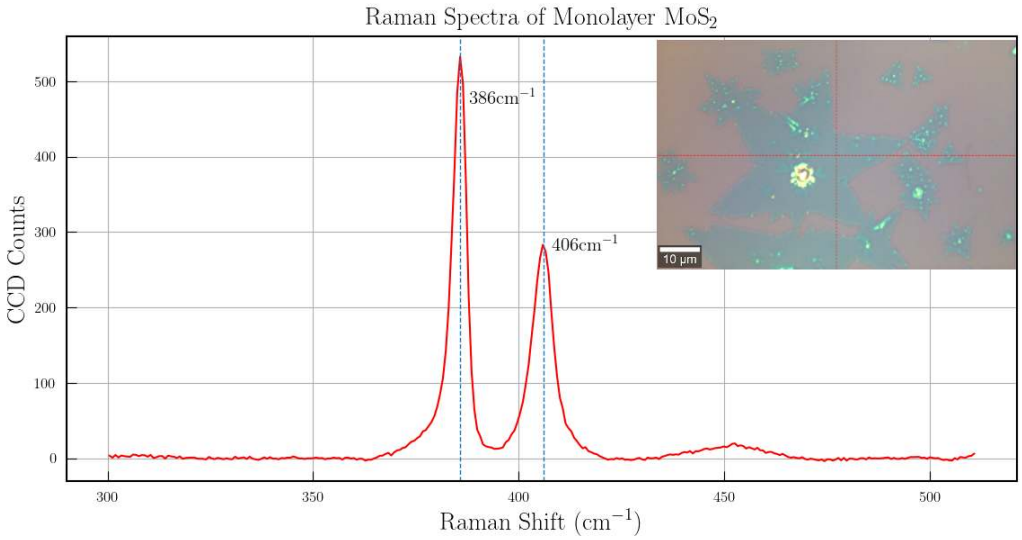



Figure 10: Raman spectra of a Monolayer $MoS_2$ deposited on SiO2/Si with Qrystal-CVD. The inset image shows the location on the crystal where the spectra was obtained. The Raman spectra was taken by our expert collaborator, Mr. Saroj Jha, at AIRF, JNU.

### 11.1.2 Pre-requisites

The following will be available with Quazar personnel visiting for demonstration.

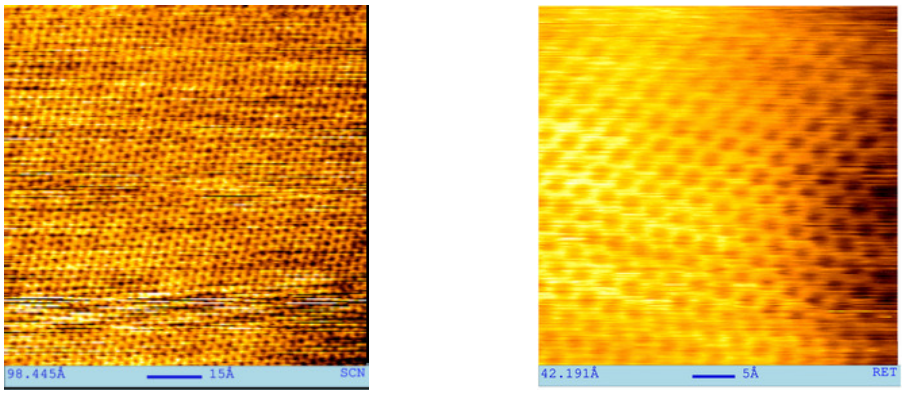
- Precursors: $MoO_3$, sulfur
- Substrate: $Si/SiO_2$ wafer with 300 nm oxide thickness for optical contrast
- Labware: Alumina boats, 10 mm × 50 mm × 25 mm

The following are to be arranged by the user.

- Isopropyl alchohol (99.9% pure), acetone, deionized water, hydrogen peroxide, sulfuric acid, glass pipette (5ml & 10ml), rubber gloves, diamond scribe (for cutting wafers), tweezers, spatula, beakers, petri dish, conical flasks, butter paper (for sample weighing and storage), cotton rolls (for quartz tube cleaning), lint free tissue papers, desiccator with silica gel, scissors, small plastic boxes for storing samples, hot air gun, and air blow gun.
- Weighing machine with a resolution of at least 0.1 mg
- Gas supply: Argon (Highest Grade) and Oxygen (Highest Grade)
- Characterization facility: 1000-1500x (5x-150x objective and 10-15x eye-piece) top-illuminated optical microscope for visual inspection (Eg. Leica DM750M, Zeiss Axioscope 5), and RAMAN spectrometer (Eg. HORIBA LabRAM HR Evolution Confocal Raman Microscope).

## 11.2 Graphene

### 11.2.1 Characterization




(a) (b)

Figure 11: Images showing the honeycomb structure of Qrystal CVD grown Graphene on copper at two magnifications. The above images was obtained on nanoREV$^{TM}$ Ambient Air STM at Quazar Tech.

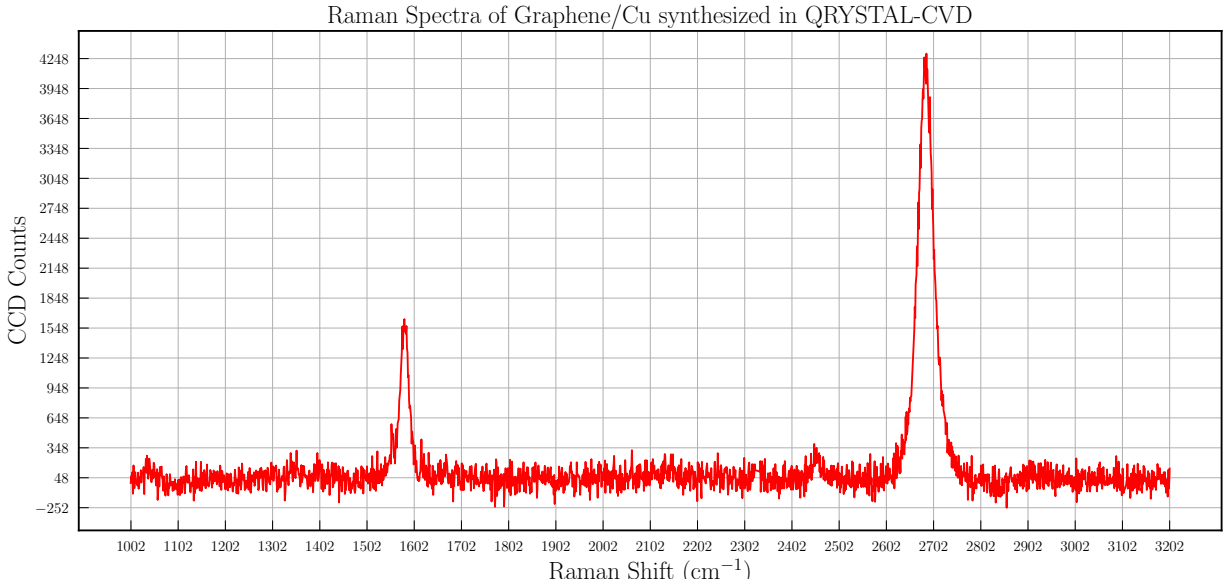



Figure 12: Raman spectra of Qrystal CVD grown monolayer Graphene on a copper substrate. The above data was obtained with Renishaw Invia series Raman spectrometer by our expert collaborator, Prof. Pranav Shirhatti, at TIFR-Hyderabad.

### 11.2.2 Pre-requisites

The following will be available with Quazar personnel visiting for demonstration.

- Substrate: Copper Foil 99.8% purity with 0.025mm thickness
- Labware: 12$mm$ diameter quartz tube sample holder

The following are to be arranged by the user.

- Isopropyl alchohol (99% pure), glacial acetic acid, acetone, deionized water, rubber gloves, tweezers, beakers, petri dish, conical flasks, cotton rolls (for quartz tube cleaning), lint free tissue papers, desiccator with self indicating silica gel, scissors, small plastic boxes for storing samples, and hot air gun.
- Gas supply: Hydrogen (Highest Purity), Methane (Highest Purity), Argon (Highest Purity)
- Characterization facility: 1000-1500x (5x-150x objective and 10-15x eye-piece) top-illuminated optical microscope for visual inspection (Eg. Leica DM750M, Zeiss Axioscope 5), and RAMAN (Eg. HORIBA LabRAM HR Evolution Confocal Raman Microscope).

## 11.3 Molybdenum Diselenide (Not included in demonstration)

### 11.3.1 Characterization

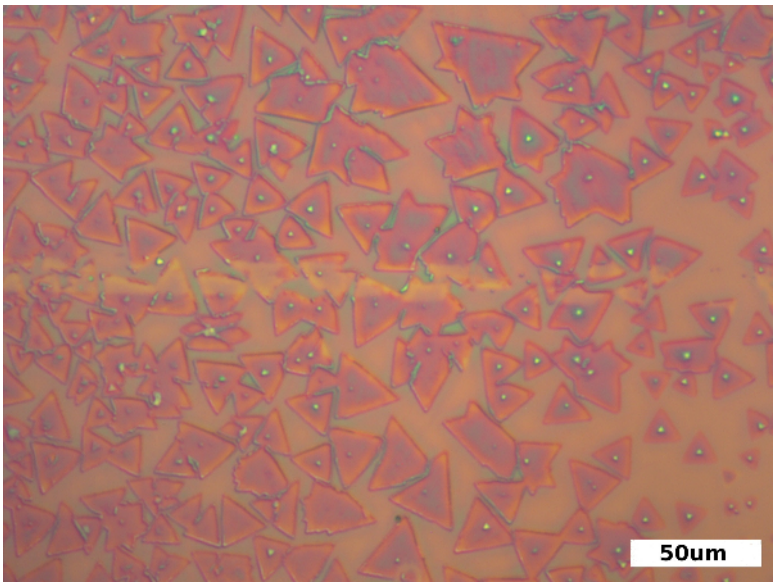



Figure 13: Triangles of CVD deposited $MoSe_2$ on SiO2/Si waffer as seen under a top illuminated optical microscope. The image was taken with a the TIM at Prof Subhasis Ghosh's lab at JNU.

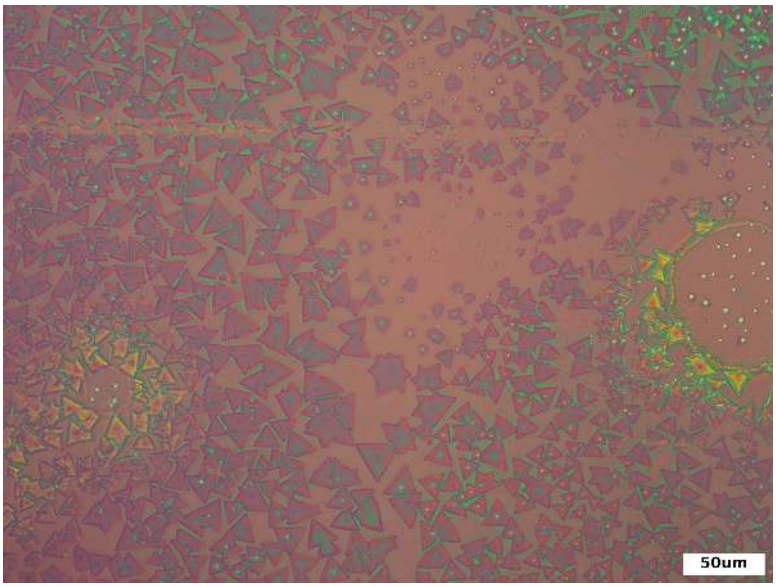



Figure 14: Triangles of CVD deposited $MoSe_2$ on SiO2/Si waffer as seen under a top illuminated optical microscope. The image was taken with a the TIM at Prof Subhasis Ghosh's lab at JNU.