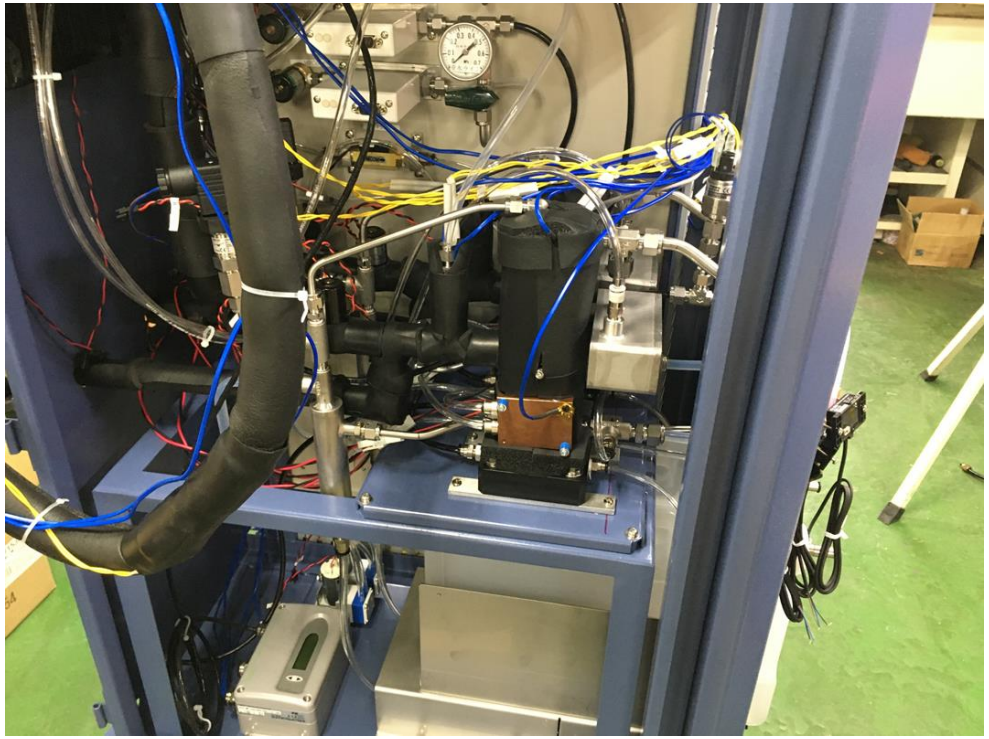
H_2O-D_2O Mixing Gas Humidity Generator



Overview

This humidity generator controls the relative humidity (RH) based on the two-temperature method. The device consists of four mass flow controllers and two water tanks which are saturators for H_2O and D_2O, respectively. Each tank is a precise temperature controlled by a Peltier cooling/heating system. Continuous contrast variation (H_2O/D_2O) measurement is possible by changing the flow rate of each humid gas line.

Table 1 Spec of the mixing gas humidity generator

Me-240dp (Micro Equipment Inc, Tokyo, Japan)

Relative humidity	
Dew point	-15 °C to 81 °C 7 %RH@25 °C to 85 %RH@85 °C Dry gas mixing below 6 °Cdp
Accuracy	±0.1 °C (approximately ±0.5 %RH)
Flow rate	0.05 L/min. to 3 L/min (max. 3 L/min. at the outlet)
Accuracy in gas mixing	< ±1 %
Pressure	1 atm
Device Remote control	Mitsubishi MC Protocol via Ethernet
Heating tube length	10 m

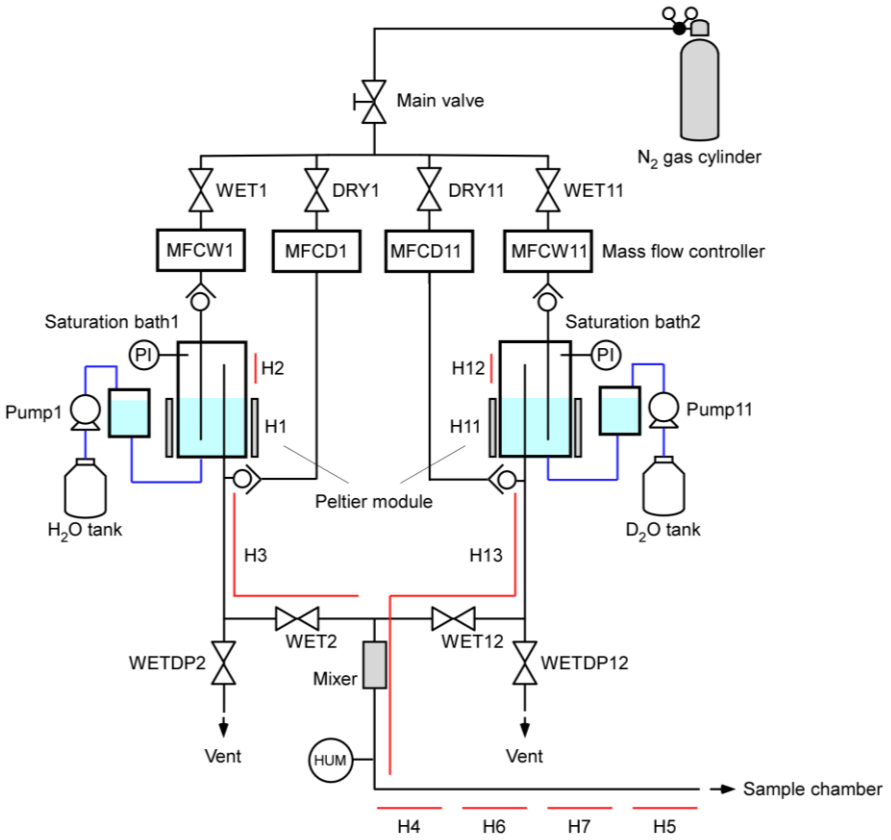


Fig. 1 Schematic of the system

Comparison of contrast-variation humidity-control techniques

Table 2 Comparison of our device to conventional humidity control techniques.

	Present Study	Saturated salt solutions	Temperature controlled water bath	Gas flow
Humidity stability	0.5 %		0.01%?	1%
RH change	Water bath temp	Salt concentration	Water bath temp	Mass flow
Equilibration time	minutes	hours	hours	minutes
H_2O/D_2O change	Mass flow	Change of reservoir	Change of reservoir	Change of reservoir

- In conventional humidity generators, the water in the reservoir tank has to be changed to carry out the contrast variation method. The water change takes several hours, including the time needed to adjust the temperature. This is a major problem in neutron experiments where beam time is limited.
- Our equipment is characterized by two independent saturation chambers and precise dew point control using a two-temperature method. It is possible to automatically generate humidity gases with quick humidity changes and continuous H_2O-D_2O composition.

Humidity control by the two-temperature method

Approach to generate precise humidity:

1. The dry air from the gas cylinder/compressor passes through the water in the tank.
2. The gas becomes pre-saturated vapor with a dew point almost equals to the water's temperature(T_1).
3. The tank headspace is set to a temperature of a few degrees higher than T_1, so the pre-saturated vapor contains excess water compared to the dew point of T_1.
4. Then, the vapor through the outlet line cools down to T_1. This process forms condensate water and provides the precise dew point of T_1.
5. RH can be calculated from the saturated vapor pressure (P_{ws}) at the dew point(T_1) and the chamber temperature ($T_{chamber}$) .

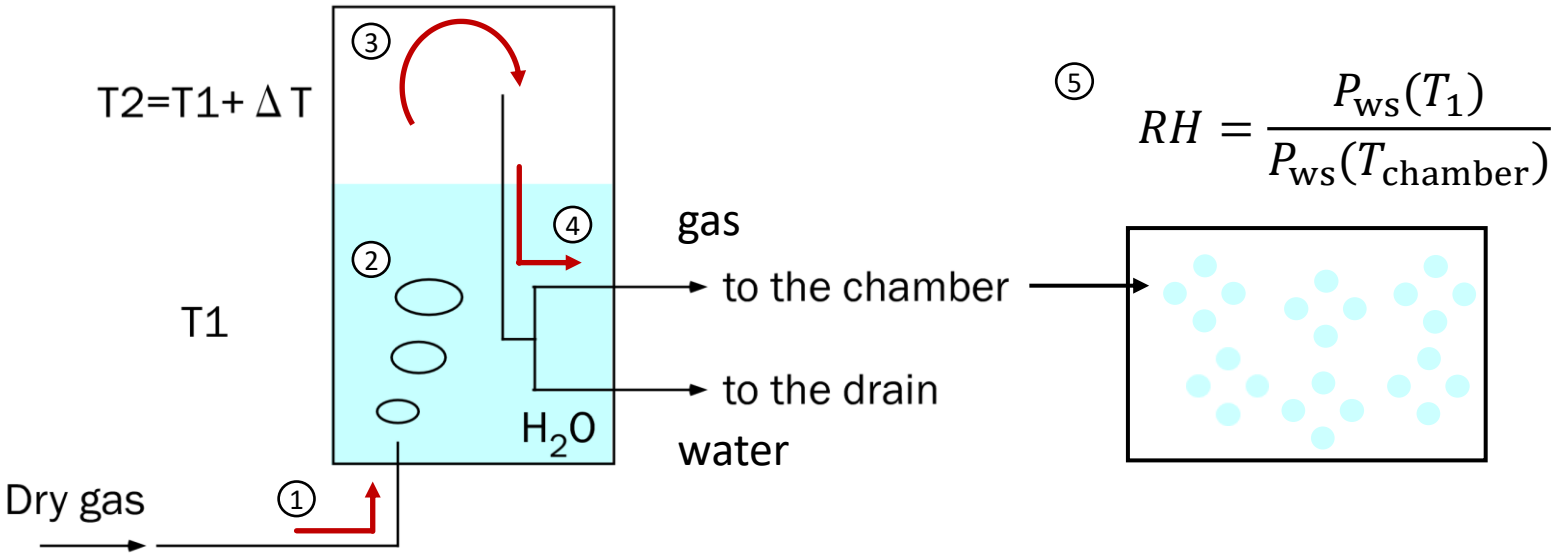


Fig. 2 Schematic of the saturator.

Performance of humidity control (1)

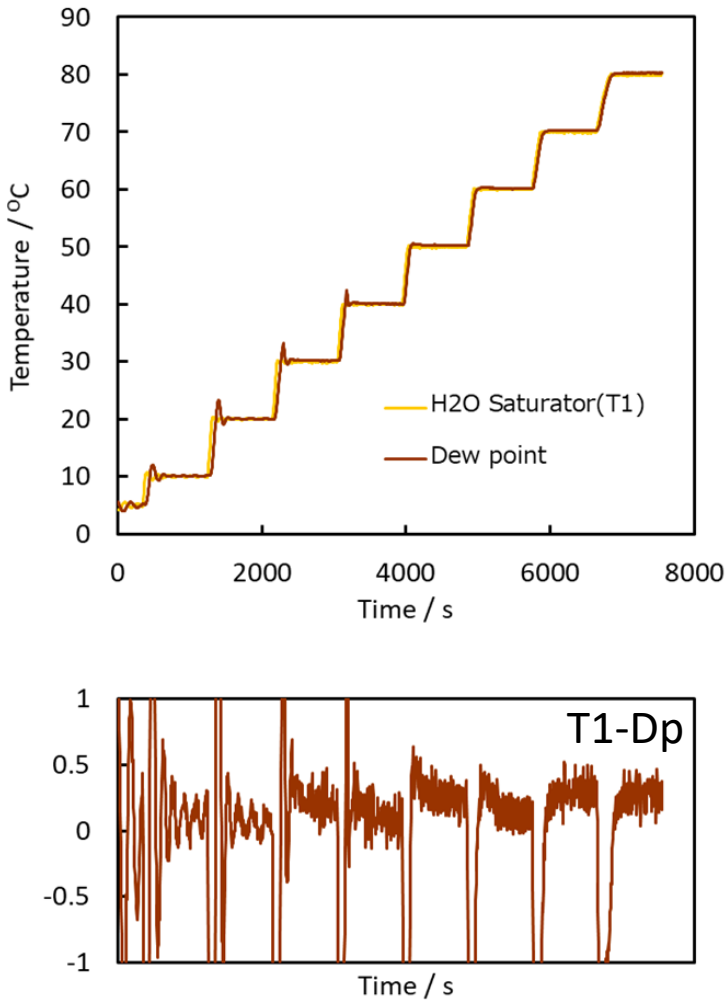


Fig. 3 Evolution of the Dp at the end of the transfer heating tube (not in the sample chamber)

- The relationship between the dew point of the humid gas and the temperature of the saturator (T1) was examined using a mirror dew point hygrometer, Dew Star S-1 (Shinyei technology Co., Kobe, Japan).
- The hygrometer was attached to the end of the heating tube.
- The dew point and saturator temperatures were almost the same, confirming that the performance was as designed in the previous page.

Performance of humidity control (2)

The stability of the relative humidity was investigated by a humidity sensor (SHT35, Sesirion), and the results are shown in Figure 4. Depending on the generated dew point, the relative humidity in the test chamber becomes stable within a few minutes.

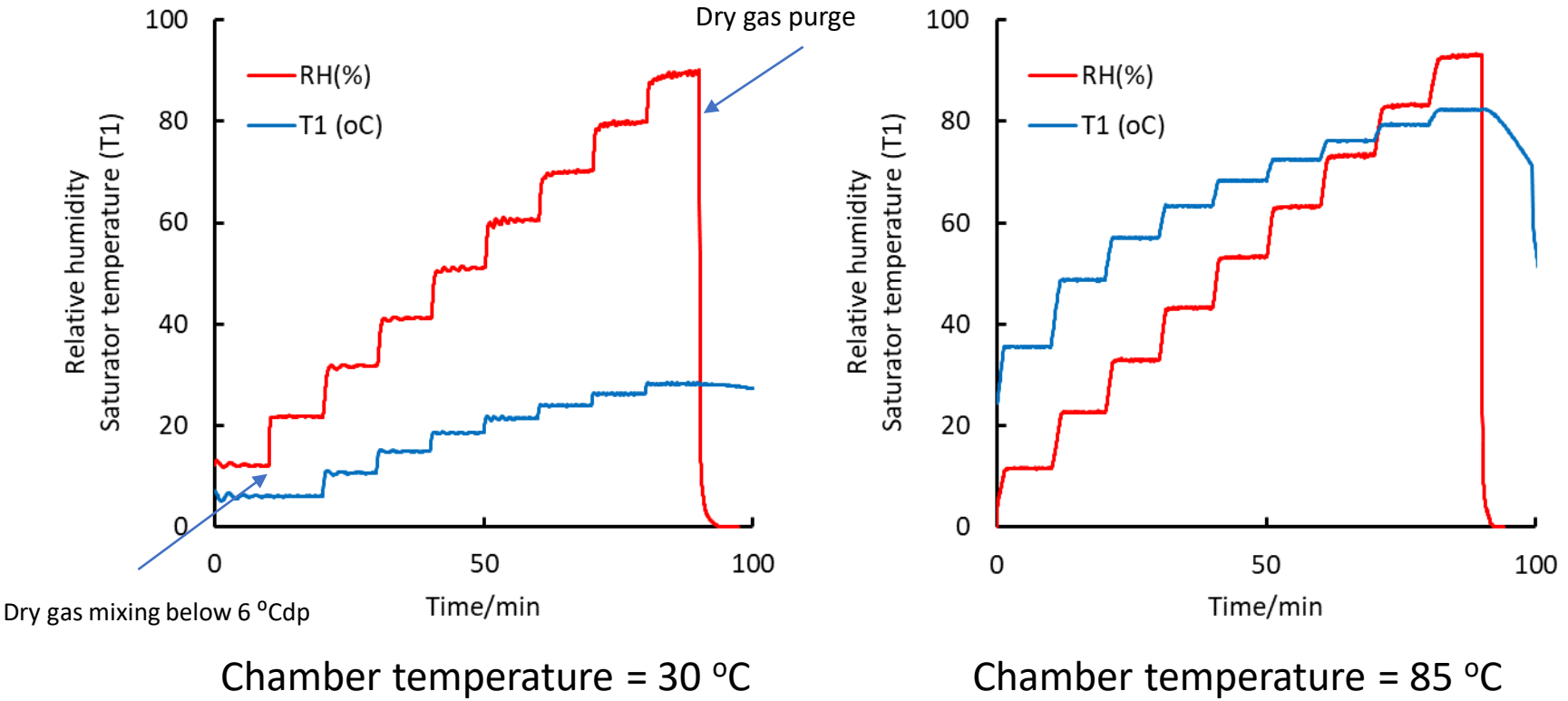


Fig. 4 Evolution of the RH in the test chamber at 30 oC and 85 oC.

Composition of mixing gas

- Heavy water and light water have different saturation vapor pressures. Therefore, even if the dew point is the unity, the absolute humidity (water content per volume) is different.
- In this device, the composition of the humid gas that comes out when the dew point is 80 ^{o}Cdp with a flow rate of 1 L/min and H/D ratio of 0.50 is as shown in the Table (sample temperature is 85 ^{o}C).
- See the following paper for the calculation method.
  "In situ vapor sorption apparatus for small-angle neutron scattering and its application", Review of Scientific Instruments 76, 113904 (2005); https://doi.org/10.1063/1.2134151, Man-Ho Kim et al.

Table 3 Composition of 80%RH-85^{o}C H_2O-D_2O mixing gas with both flow rate (inlet dry gas) of 0.5 L/min.

	Flow rate at outlet L/min	Absolute humidity mol/m^3	Partial pressure P/Pa	Composition of mixing gas (in mol)
H_2O humid gas	1.23	15.92	47416	0.53
D_2O humid gas	1.16	14.86	44239	0.47

Performance of H2O-D2O mixing gas

- The molar composition of the gas mixture was evaluated by FTIR measurement of the condensate water. Condensate was obtained by blowing 80 C dew point of gas onto a copper plate at 20° C.
- The composition of the gas mixture agrees with the value calculated from the saturation vapor pressure and equation of state of H2O and D2O.

Flow rate [L/min.]			Composition		
H_2O line	D_2O line	x	H_2O	D_2O	Total
0.0	1.1	0.000	0.007(4)	0.993(3)	1.000
0.1	1.0	0.091	0.120(4)	0.878(3)	0.998
0.2	0.9	0.182	0.219(4)	0.786(2)	1.005
0.3	0.8	0.273	0.315(4)	0.684(2)	0.999
0.4	0.7	0.364	0.413(4)	0.588(2)	1.001
0.5	0.6	0.455	0.503(4)	0.507(2)	1.010
0.6	0.5	0.545	0.586(4)	0.422(2)	1.008
0.7	0.4	0.636	0.665(4)	0.330(2)	0.995
0.8	0.3	0.727	0.753(4)	0.243(2)	0.996
0.9	0.2	0.818	0.839(4)	0.164(2)	1.002
1.0	0.0	1.000	0.993(4)	0.001(2)	0.994

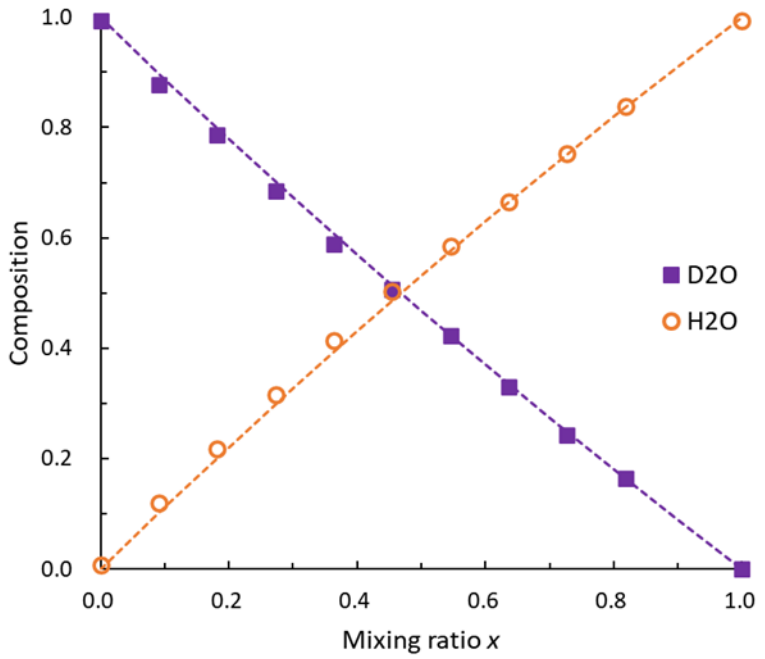


Fig. 5 Composition of gas mixture

Quick operation guide

Startup:

1. Purge the system for 10-20 minutes at room temperature until the hygrometer (HUM1 DP) at the outlet of the device indicates -80 °Cdp.
2. For the first water supply, set the "間欠OFF" value of the "飽和槽給水制御" in the system settings to zero, and run the pump continuously. The water supply stops automatically.
3. Check the current temperature of the saturation tanks (T1 and T11). If the saturation tank temperature is higher than the line temperature, wait until the tanks cool down.

Start operation:

- Touch panel / Software; Set the humid gas parameters and press "調湿運転開始"(Run) key.
- IROHA; SampleEnv --> me240

Sample change:

- Switch the operation mode to “パージ運転開始”(Purge) and wait until the dew point of the chamber falls below room temperature.

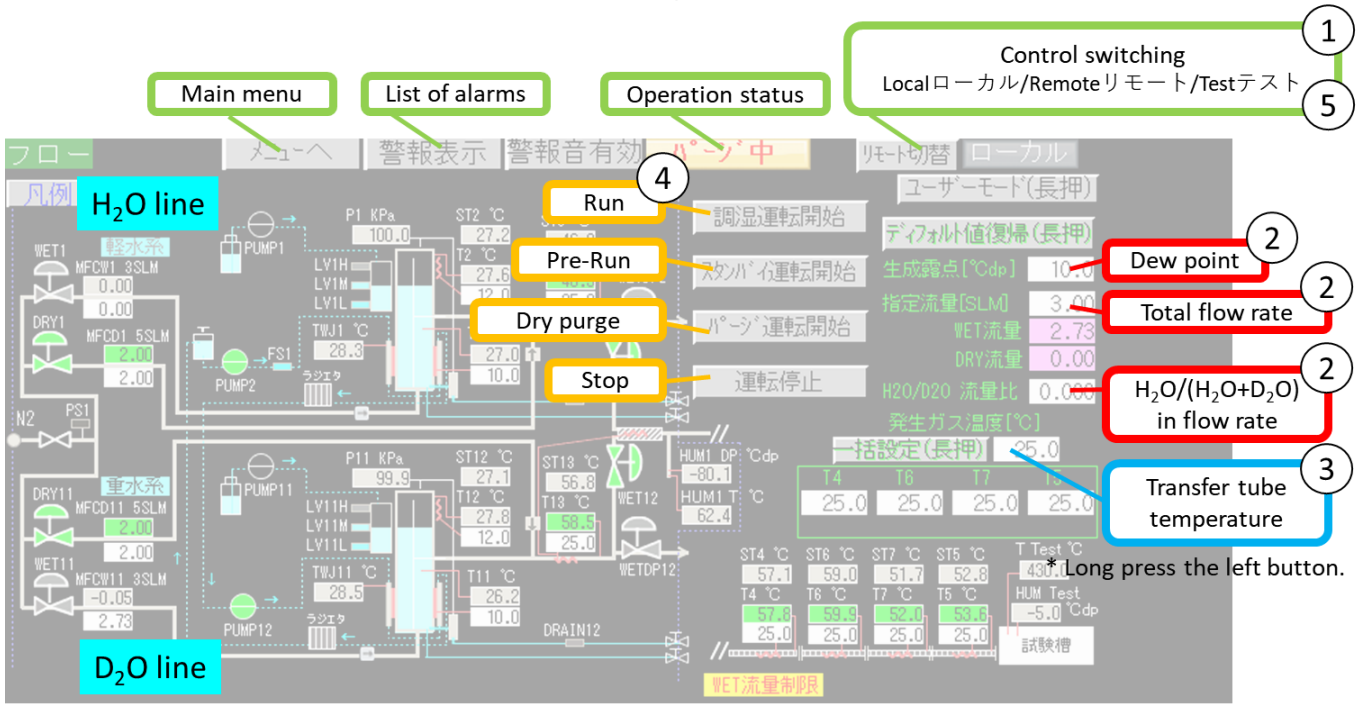
Stop operation:

1. Set SV of dew point to 10 °Cdp and perform "調湿運転開始"(Run) .
   Wait until the temperature of the saturation tanks fall below 40 °C.
2. Purge for 30 minutes.
3. Finally, press the “運転停止“(stop) key.

How to use the windows software

Step for generating humidity gas to the chamber:

1. Switch to Local mode, when any other control mode is selected.
2. Edit the humidity gas parameters (dew point, flow rate, and H/(H+D) composition).
3. Set the heating tube temperature. (dew point + 15 ^{o}C is recommended.)
4. Click on the “Run” (The details of operation modes are described in the next slide).
5. Switch to Remote mode in the case of using IROHA control system.
   In Local mode, commands from IROHA will be ignored.



PV (control on)
Set point

PV (control off)
Set point

Open

Close

Commands for IROHA interface

- prmSetOperation
  0. Stop: Cut off the power to the heaters and close all valves.
  1. Run: Introduce the humidity gas with the set dew point into the chamber.
  2. Dry Purge: Purge the chamber with N_2 flow rate of 2 L/min.
  3. Pre-Run: Just warm up/cool down the water tank to the set dew point.

Parameter		Valve				Heater power	
		Humid line	Dry line	Chamber	Vent	Tank	Line
0	Stop	Close	Close	Close	Close	Off	Off
1	Run	Open	Close	Open	Close	On	On
2	Dry purge	Close	Open	Open	Close	Off	On
3	Pre-run	Open	Close	Close	Open	On	On

- prmSetDewPoint Set point of T1 and T11 (=dew point) Unit: ^{o}C Range:-15 to 81
- prmSetFlowRate Set point of total flow rate for inlet gas Unit: L/min. Range:0.05 to 3
- prmSetMixingRatio Mixing parameter *x*; $x H_2O/(xH_2O+(1-x)D_2O)$ Range: 0.00-1.00
- prmSetTempTube4, 5, 6, 7 Set point of heating tubes Unit: : ^{o}C Range: 0-120

Alarms

T1上限	90.0	Upper limit of T1 temperature
T1下限	4.0	Lower limit of T1 temperature
TWJ1上限	60.0	Upper limit of TWJ1 temperature
P1上限	300.0	Upper limit of H_2O tank pressure
T2上限	98.0	Upper limit of T2 temperature
ST2ズレ	10.0	T2-ST2 temperature differential value
T3上限	120.0	Upper limit of T3 temperature
ST3ズレ	10.0	T3-ST3 temperature differential value
T11上限	90.0	Upper limit of T11 temperature
T11下限	4.0	Lower limit of T11 temperature
TWJ11上限	60.0	Upper limit of TWJ11 temperature
P11上限	300.0	Upper limit of D_2O tank pressure
T12上限	98.0	Upper limit of T12 temperature
ST12ズレ	10.0	T12-ST12 temperature differential value
T13上限	110.0	Upper limit of T13 temperature
ST13ズレ	10.0	T13-ST13 temperature differential value
T4上限	110.0	Upper limit of T4 temperature
ST4ズレ	10.0	T4-ST4 temperature differential value
T5上限	110.0	Upper limit of T5 temperature
ST5ズレ	10.0	T5-ST5 temperature differential value
T6上限	110.0	Upper limit of T6 temperature
ST6ズレ	10.0	T6-ST6 temperature differential value
T7上限	110.0	Upper limit of T7 temperature
ST7ズレ	10.0	T7-ST7 temperature differential value

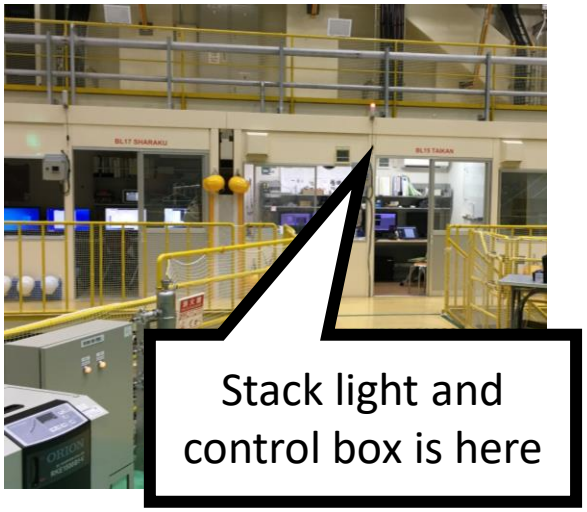
LV1H高	Upper limit of H_2O tank water level
LV11H高	Upper limit of D_2O tank water level
LV1L低	Lower limit of H_2O tank water level
LV11L低	Lower limit of D_2O tank water level
FS1循環水停止	Lack of cooling water
WLEAK漏水検知	Water leakage in the device
PS1圧力低下	Low-supply pressure
EMR1非常停止	Emergency stop button
EMR2非常停止	Emergency stop button
EMR3非常停止	Emergency stop button
SRZ通信異常	Temperature controller communication error
DRAIN2 軽水凝縮水検出	Upper limit of H_2O drain
DRAIN12 軽水凝縮水検出	Upper limit of D_2O drain

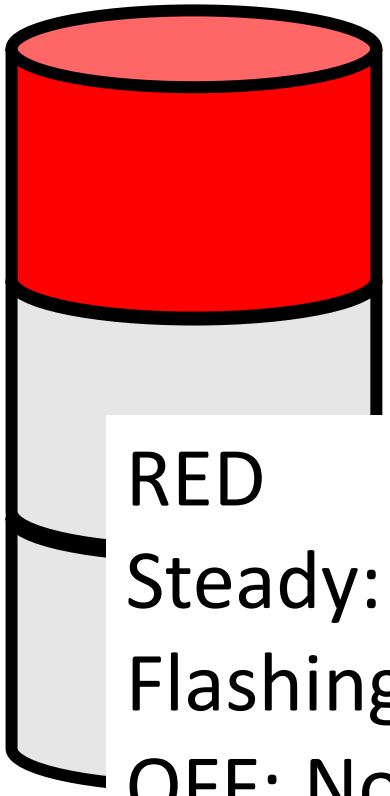
Thermocouple disconnection

T1	☑
T2	☑
ST2	☑
T3	☑
ST3	☑
TWJ1	☑
T11	☑
T12	☑
ST12	☑
T13	☑
ST13	☑
TWJ11	☑
T4	☑
ST4	☑
T5	☑
ST5	☑
T6	☑
ST6	☑
T7	☑
ST7	☑

Warning light

If the interlock is activated, the operation will automatically stop, and an alarm will sound.
Press the "buzzer-off" key in the control box on the wall of the operation room, and then contact the beamline staff.





RED
Steady: Normal operation
Flashing: Emergency Stop
OFF: No operation

Color coding for stack lights in MLF.

Refilling water / Release the condensate

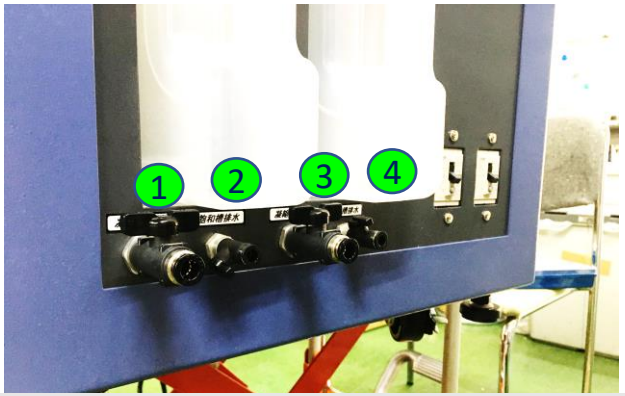
- Each reservoir tank has a capacity of 3 litter each. Refill with water as needed.
- The amount of water consumed depends on the humidity generated (See the below table).

Do not open the front cap.
Just release the quick connect.

Table Water consumption per 24 hours (unit: L)

RH(%)	Temperature(℃)		
	30	60	85
30	0.01	0.07	0.24
60	0.03	0.15	0.60
85	0.04	0.23	1.09

- Condensate in the saturators should release manually.
- Open and release the drain valve of condensate once every two days.
- However, this should not be done during measurements (the dew point will fluctuate).



1&3: Drain of condensate.
2&4: Do not open (Drain of water Tanks.)