

# Jet - Freeze Device

JFD 030



**BAL-TEC**

EM-Technology and Application



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Inspect shipment for possible damage and notify shipper if necessary.

Compare shipment with shipping papers and notify supplier of possibly missing pieces.

Save packing material. If possible, device should be shipped in the original packing for inspection or repair.

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## 1. INSTALLATION OF DEVICE

### 1.1. Safety

The JFD 030 jet-freeze device was developed in accordance with VDE 0411 regulations (regulations for electronic measuring devices and controls). A gastight design was used for the relays necessary for the operation of the device.

As a safety precaution, refill of propane into the condensation chamber as well as release of "liquid propane charges" through the jet system is not possible when the Plexiglass protective hood is open.

The instructions in Sections 1.2. and 1.3., as well as in the maintenance chapter must be observed for the prevention of accidents during the installation and connection of the device, as well as during operation.

Safety goggles must be worn  
when working with liquid gas.

### 1.2. Installation of device

The device is installed into an acid hood with proper exhaust or into a flow-box. Due to the higher density of the propane compared to air the fan should be rather built into the bottom of the hood than into its top. The hood must draw at least 30 m<sup>3</sup>/h (approx. 20 cubic feet per minute) to dilute the propane quickly enough to prevent an explosive gas-air-mixture from forming in the hood. Since a propane content of 2.1 to 9.5 volume % in air creates a highly explosive gas-air mixture, measures should be taken to prevent any concentration of gas in the exhaust line system. In addition, due to safety reasons, any open fires (smoking, bunsen burners etc.) must be prohibited in the laboratory as long as the JFD 030 jet-freeze device is in operation.

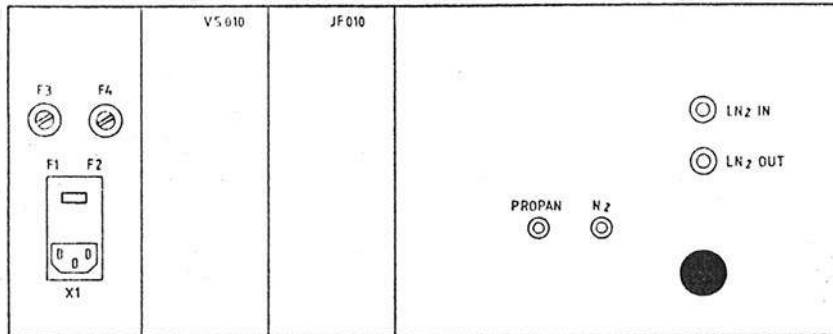
Prevent propane gas leakage  
into the workroom.

The installation and operation of the device, as well as the storage of propane gas containers in basement areas is prohibited, due to the high density of propane (0.002 gcm<sup>-3</sup>) compared to air (0.0013 gcm<sup>-3</sup>) and the related danger of explosion due to a possible concentration of the gas-air-mixture.

### 1.3. Connection of device

All of the parts required for the connection of the device to the media supply are part of the device-accessory set.

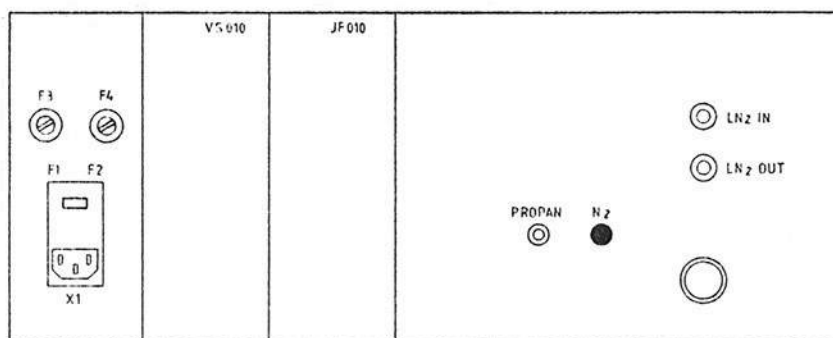
#### Connection of the exhaust gas hose



- Slide PVC hose  $\varnothing$  25/34 mm, 2 m long, onto exhaust sleeve and secure with hose clamp, if the device is not installed in a flow-box.
- Guide the other end of the exhaust hose into the proper escape and secure with hose clamp (see Section 1.2.)
- In the absence of an appropriate escape, vent hose to the outside. Secure exhaust hose.

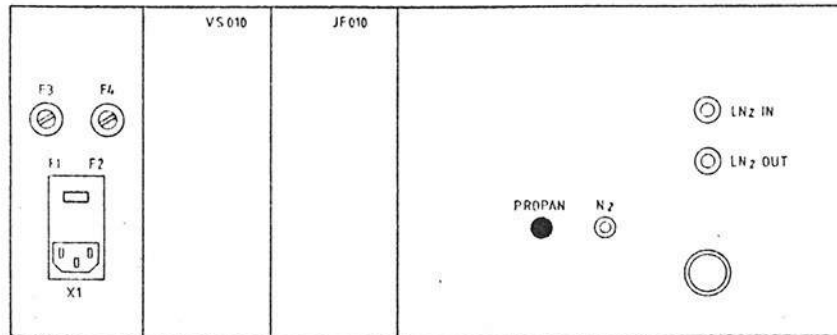
#### Connection of nitrogen gas bottle

The nitrogen gas bottle must be safeguarded against falling to prevent accidents.



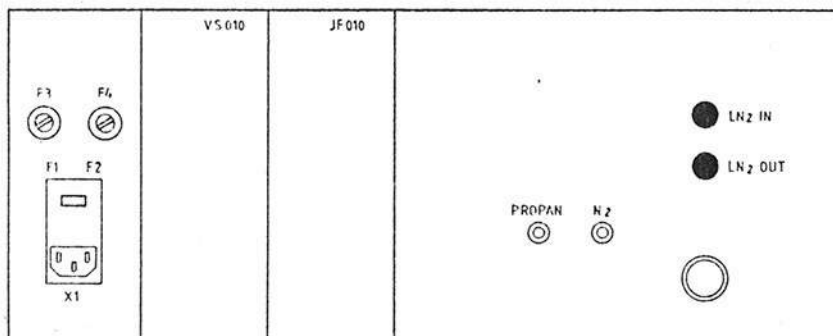
- Screw premounted hose connection  $\varnothing$  6 mm onto connection  $N_2$
- Slide hose nozzle onto PVC hose 6/12, 1.5 m long, and secure with hose clamp
- Connect the other end of the connecting hose to the nitrogen gas bottle

## Connection of propane gas container



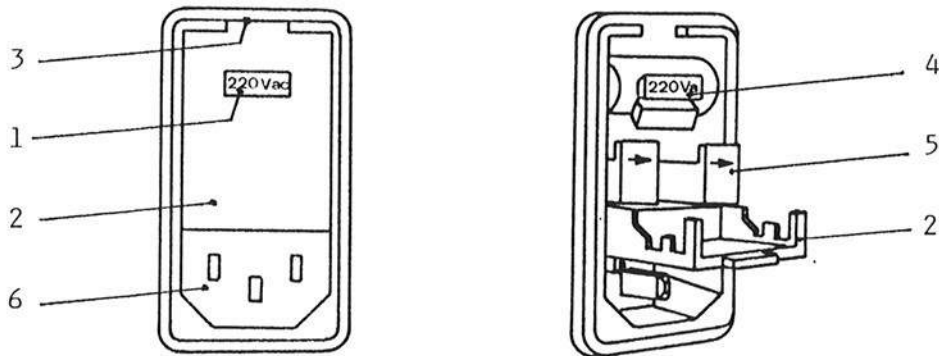
- Screw premounted hose connection  $\varnothing$  6 mm onto PROPANE connection
- Slide the propane liquid gas hose  $\varnothing$  6/14 mm, 1.5 m long, onto hose nozzle and secure with hose clamp
- Connect other end of the connection hose on the propane container.

## Connection of liquid-nitrogen supply

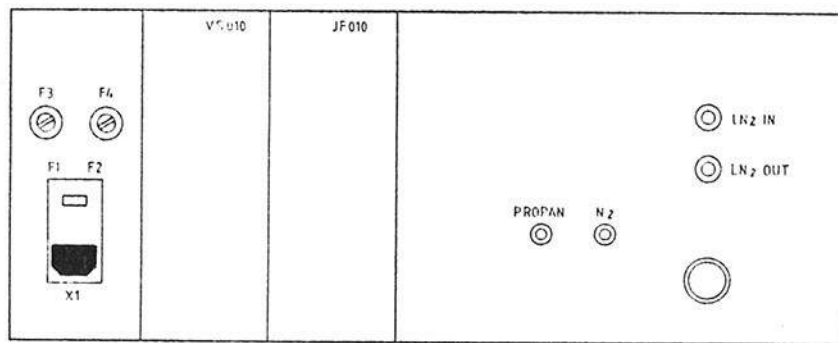


- Screw premounted hose connection  $\varnothing$  10 mm onto LN<sub>2</sub> OUT connection
- Slide Armaflex hose  $\varnothing$  10/36 mm, 1.5 m long, onto hose nozzle and secure with hose clamp
- Guide other end of the Armaflex hose into Dewar container to catch liquid nitrogen that accumulates during the cooling phase
- Screw connecting line with Armaflex insulation onto connection LN<sub>2</sub> IN
- Connect the other end of the connecting line with refill device of the nitrogen storage container.

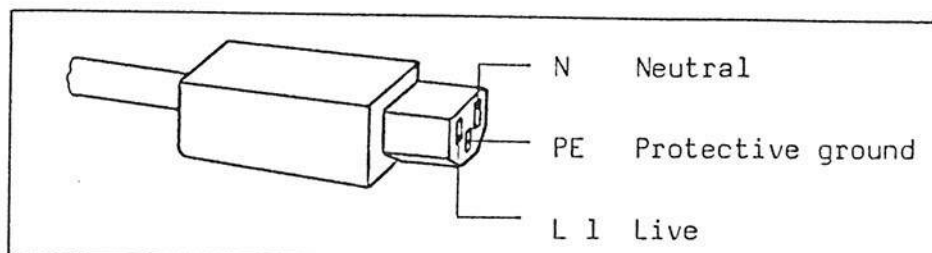
## Electrical connection



- Check connection voltage specified for the device at the inspection window (1) of the power input.
- In the event the voltage does not meet requirements, convert device for the required connecting voltage as follows:
- Press cover (2) open by sliding screwdriver into the upper slit (3)
- Remove voltage selector (4) and reinsert so that the desired voltage shows in the window (1).
- Pull out fuse holder (5) and insert fuses corresponding to the connecting voltage: fuse rating for 220 V = 1.6 AT, for 115 V = 3.15 AT
- Insert fuse holder (5) and close cover (2).



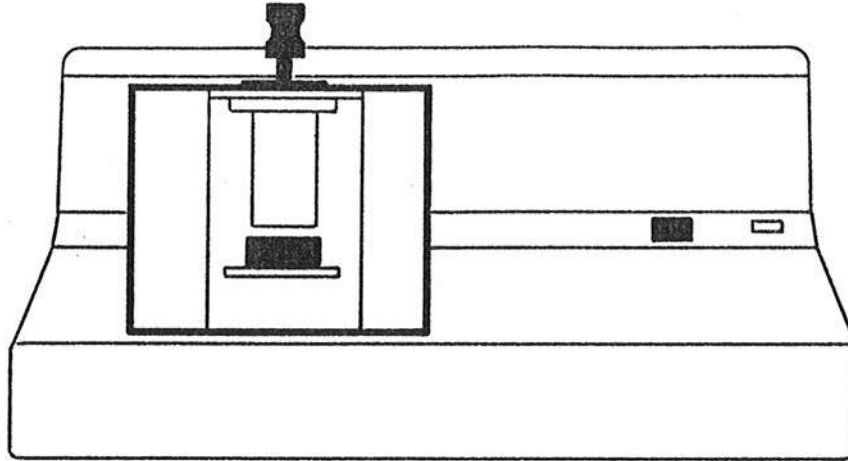
- Connector socket of power line to device mains plug X 1
- Mains plug (L1 + N + PE) of connecting cable must meet local standards (see below drawing)
- Connect connecting cable to builder outlet.





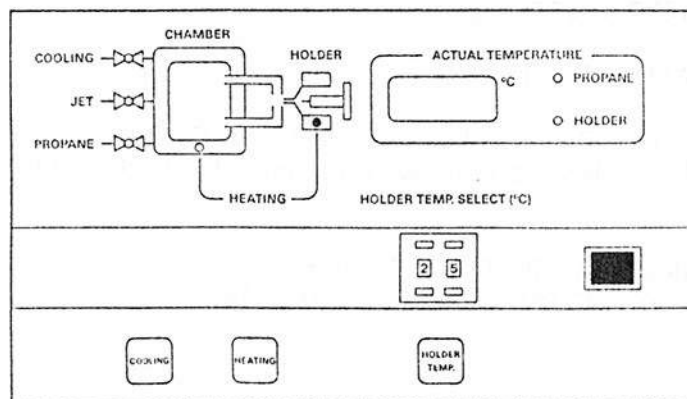
## 2. OPERATION OF DEVICE

### 2.1. Preparation of device



- Place sample storing and transfer container below the jet system on the cooling plate in working chamber
- Close Plexiglass protective cover of working chamber
- Insert empty sample holder in sample holder fixture (do not push in!)
- Set desired temperature for sample holder fixture with temperature selector switch HOLDER TEMP.SELECT

### 2.2. Switch on of device

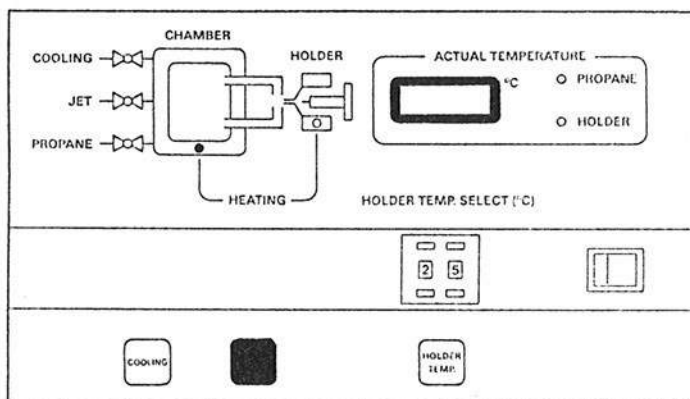


**Turn main switch to ON:**

- Switch key light lights
- All LED of the mimic diagram light briefly
- Heater of sample holder fixture turns on and heats sample holder to temperature selected with HOLDER TEMP.SELECT
- LED of HOLDER HEATING of mimic diagram lights

### 2.3. Bake-out of device

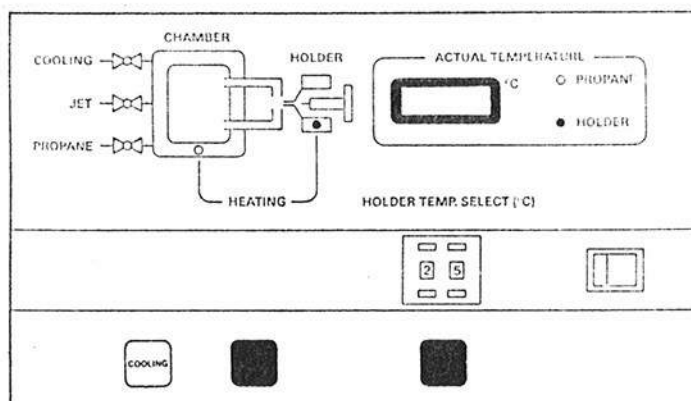
Heat cooling chamber, jet system and valves prior to freezing of specimens so that possible humidity present in the system does not impair the freezing process



#### Press HEATING key

- Heater of sample holder fixture turns off:  
LED of HOLDER HEATING goes out
- LED of CHAMBER HEATING lighths
- Set nitrogen gas pressure with pressure reducing valve of gas bottle to approx. 0.5 bar and blow nitrogen intermittently through the system during the warm up procedure
- Heat system to at least 60° C:  
Temperature is displayed on ACTUAL TEMPERATURE display

## 2.4. Switch off of bake-out process



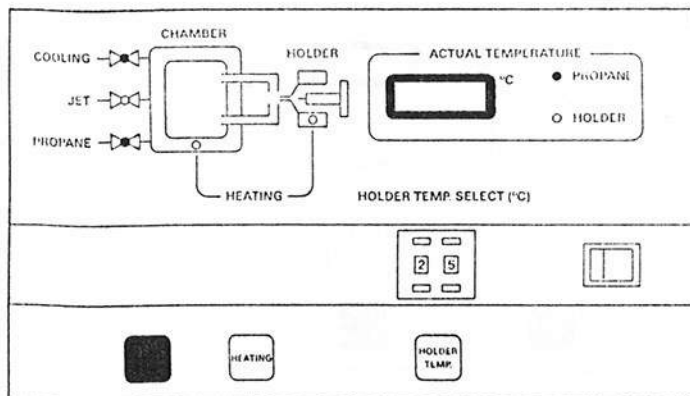
**Press HEATING key:**

- Heater of device turns off:  
LED of CHAMBER HEATING goes out
- Heater for sample holder fixture HOLDER HEATING turns on and preheats sample holder to temperature preselected with HOLDER TEMP.SELECT according to Section 2.1.
- LED of HOLDER HEATING lights
- Check temperature of sample holder by pressing HOLDER TEMP. key:  
LED of ACTUAL TEMPERATURE HOLDER lights; display shows sample holder temperature as long as key is pressed

## 2.5. Preparation of media supply

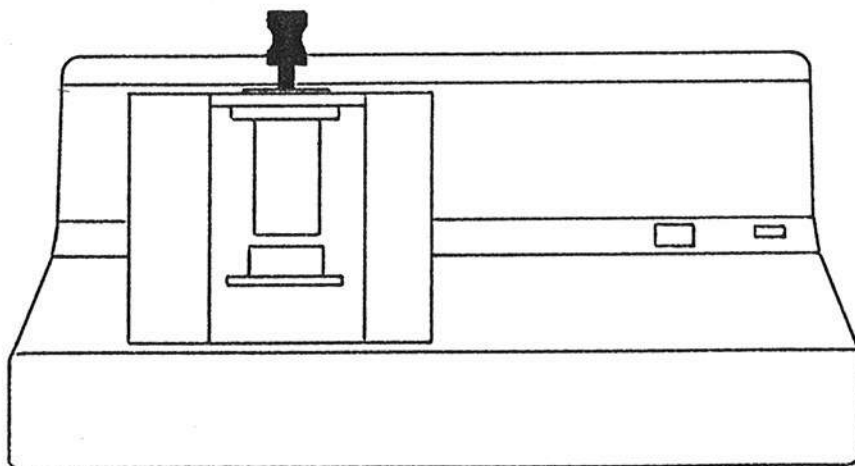
- Set nitrogen gas supply to  $5.5 \pm 0.5$  bar with pressure reduction valve of gas bottle
- Set propane gas supply to  $1.5 \pm 0.2$  bar with pressure reduction valve of gas container
- Set liquid nitrogen supply with nitrogen refill device for the cooling of the freeze system (Section 2.6.) to  $0.8 \pm 0.1$  bar, thereupon reduce pressure to 0.4 - 0.5 bar to save liquid nitrogen.

## 2.6. Cooling of freeze system



### Press COOLING key:

- Inlet valve for liquid nitrogen opens:  
LED of COOLING valve lights
- Temperature of freeze system is continually displayed on ACTUAL TEMPERATURE display
- LED of ACTUAL TEMPERATURE PROPANE lights
- Propane gas valve opens upon reaching a temperature of approx.  $-150^{\circ}\text{C}$ :  
LED of PROPANE valve lights
- Propane gas flows into condensation chamber and cools as liquid propane to approx.  $-180^{\circ}\text{C}$



### Press sample holder:

- Propane gas valve closes: LED of PROPANE valve goes out
- Nitrogen gas valve JET opens briefly: liquid propane flows to condition the jet system through both jets
- LED of JET valve lights as long as nitrogen gas valve is open ( 1 sec)

### **Pull up sample holder to starting position:**

- Propane gas bypass valve opens: LED of PROPANE valve lights. Open for about 20 seconds
- Propane gas flows into condensation chamber and fills it as liquid propane. Propane temperature increases: shown on ACTUAL TEMPERATURE display
- Condensation chamber is vented during filling process by way of the nitrogen three-way valve
- Liquid propane is cooled to approx.  $-180^{\circ}$  C after approx. 1 1/2 minutes

Condition jet system for specimen freeze process prior to freezing of specimen by releasing at least five "empty shots"

### **2.7. Preparation of specimen sandwich**

#### **Required material**

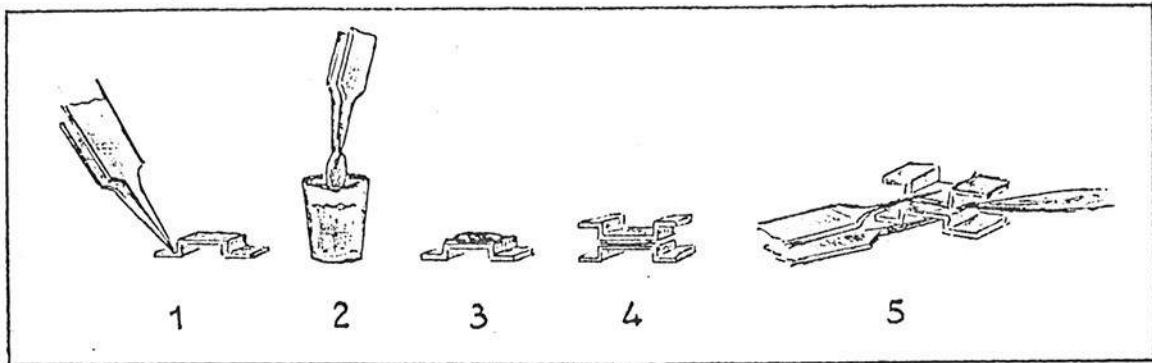
- Specimen carrier, copper, rough surface, 0.1 mm sheet thickness, dimension: 4.5 x 3 x 0.6 mm; 100 pieces per pack Order No: BU 012 056-T
- Specimen carrier grids, gold, 3 mm  $\varnothing$ , 400 mesh; 25 pieces per pack Order No: B 8010 010 19

#### **Cleaning of specimen carriers**

Specimen carriers and carrier grids are to be cleaned prior to use as follows:

- Treat specimen carrier with ultrasound in thinned  $H_2SO_4$  (approx. 1 %) for 1 minute. Wash with  $H_2O$  followed by a five minute ultrasound treatment in acetone and careful drying
- Treat specimen carrier grids in acetone for five minutes with ultrasound (degrease) and dry carefully

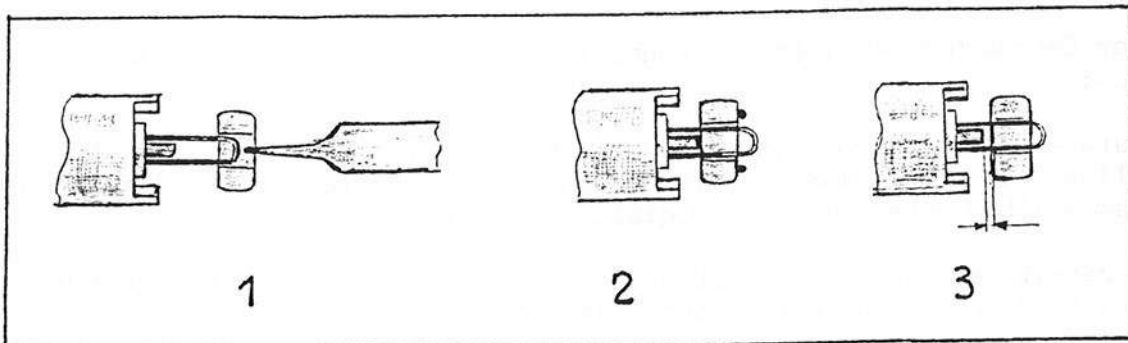
### Manufacture of specimen sandwich



- Hold specimen carrier to base with tweezers (1)
- Immerse carrier grid in medium to be frozen to fill meshes of grid (2)
- Place wetted carrier grid on specimen carrier (3)
- Complete specimen sandwich with second specimen carrier (4)
- Remove excess medium between specimen carriers with filter paper (5)

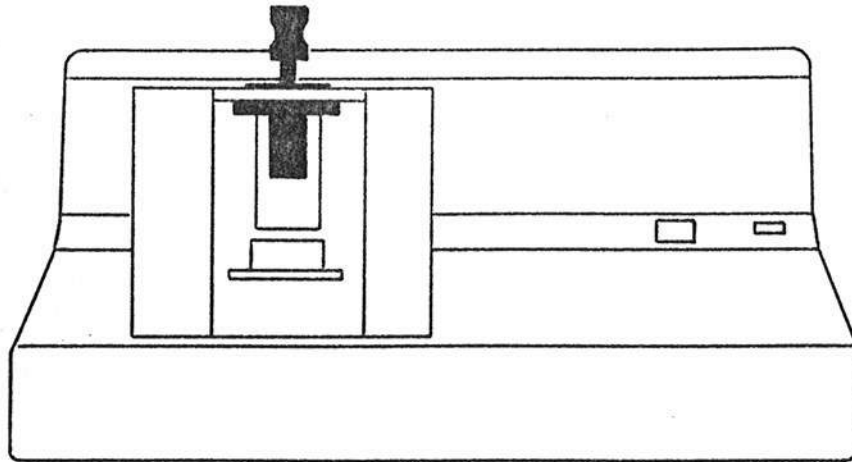
### Insert specimen sandwich in sample holder

Specimen sandwich must be inserted quickly due to the change in temperature of the sample holder after removal from the sample holder fixture



- Introduce specimen sandwich between tension springs of sample holder with the use of tweezers (1)
- Push sandwich against eject pin with tip of tweezers and align specimen carriers (2)
- Advance sandwich by approx. 0.5 mm with eject pin by pressing on the center push button of the sample holder in order to avoid contact between the specimen sandwich and the warm sample holder during freezing (3)

## 2.8. Freezing of specimen



### **Insert sample holder in holder fixture**

- Insert sample holder with specimen sandwich into sample holder fixture
- Watch for guide pin on the right of the holder fixture when inserting holder

Release a stream of liquid propane immediately after inserting sample holder as described below to prevent drying out of the specimen

### **Press sample holder**

- Holder is held by spring in pressed in position
- Propane gas valve JET opens briefly: liquid propane charges through the jets from both sides onto the specimen sandwich and freezes the specimen
- LED of JET valve lights as long as nitrogen gas valve is open ( 1 sec.)

### **Press eject button of sample holder**

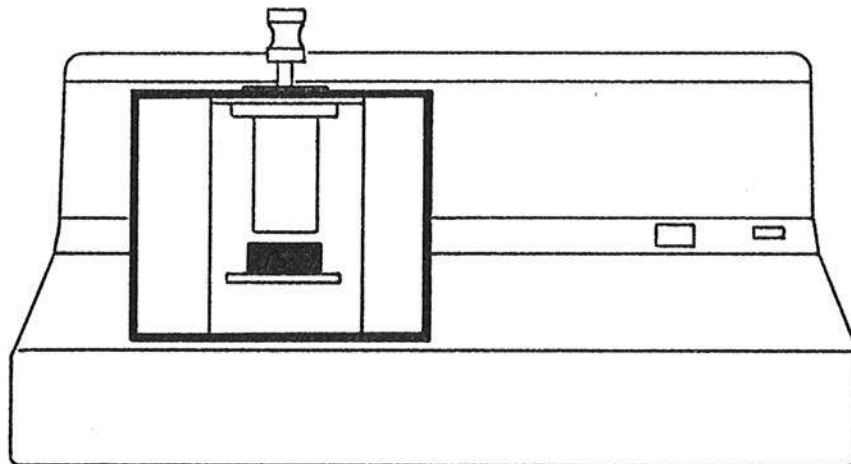
Eject specimen immediately after freezing by pressing the button in the center of the sample holder grip.

- Specimen sandwich falls into the sample storing and transfer container on the cooling plate under the jet system as described in Section 2.1.

### **Pull sample holder back to starting position**

- Propane gas by-pass valve opens: LED of propane valve lights. Open for about 20 seconds.
- Propane gas flows into condensation chamber and fills it as liquid propane. Propane temperature increases: displays on ACTUAL TEMPERATURE display
- Condensation chamber is vented during filling process by way of the nitrogen three-way valve
- Liquid propane cools to approx.  $-180^{\circ}\text{C}$  after about 1 1/2 minutes and sample holder fixture cools to preselected temperature according to Section 2.1. Device is now ready to freeze next specimen.

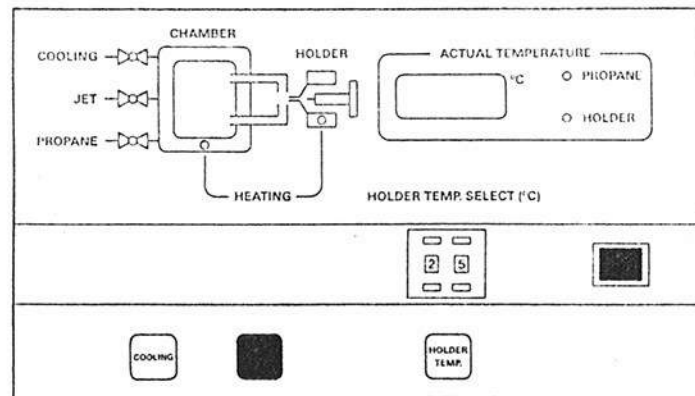
### **2.9. Removal of frozen specimen**



- Open Plexiglass protective cover of the working chamber
- Pull sample storing and transfer container as far forward on cooling plate as possible for easier removal
- Remove specimen sandwich from container with precooled tweezers, shake off liquid propane, rapidly transfer it into liquid nitrogen and insert it under liquid nitrogen into precooled double replica specimen table.



## 2.10. Switching-off device



- Close liquid nitrogen valve by pressing COOLING key
- Place storing and transfer container in acid hood or flow-box on metal block to evaporate liquid propane
- Close Plexiglass protective cover of the JFD 030 working chamber
- Close check valve of propane container
- Press HEATING key: liquid propane chamber is blown-out with nitrogen gas
- Close check valve of nitrogen gas bottle after about 5 seconds
- Close check valve of liquid nitrogen refill device
- Turn device off with main switch.



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