



**ISOTECH**

✓ The Source for Calibration Professionals

## **User Maintenance Manual**

# **ISOTECH-BONNIER ARGON TRIPLE POINT APPARATUS MODEL 471**

# Guarantee

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This instrument has been manufactured to exacting standards and is guaranteed for twelve months against electrical break-down or mechanical failure caused through defective material or workmanship, provided the failure is not the result of misuse.

In the event of failure covered by this guarantee, the instrument must be returned, carriage paid, to the supplier for examination and will be replaced or repaired at our option.



FRAGILE CERAMIC AND/OR GLASS PARTS ARE NOT COVERED BY THIS GUARANTEE INTERFERENCE WITH OR FAILURE TO PROPERLY MAINTAIN THIS INSTRUMENT MAY INVALIDATE THIS GUARANTEE

The company is always willing to give technical advice and assistance where appropriate. Equally, because of the programme of continual development and improvement we reserve the right to amend or alter characteristics and design without prior notice. This publication is for information only.



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# 1. Unpacking And Initial Inspection

Our Packing Department uses custom designed packaging to send out your unit, but as accidents can still happen in transit, you are advised, after unpacking the unit, to inspect it for any sign of shipping damage and confirm that your delivery is in accordance with the packing check list. If you find any damage or that part of the delivery is missing notify us or our agent and the carrier immediately. If the unit is damaged you should keep the packing for possible insurance assessment.

## **2. Before You Use The Equipment**

Please read the manual and familiarise yourself with all warnings, hazards and safety information. Regularly inspect the equipment and accessories. Do not use if there is any sign of damage. Keep the equipment clean, only use a damp cloth. Do not use solvents or allow liquids to enter the case.

### **3. Cautionary Note**

The contents of the cell is under extreme pressure. The unit must not be exposed to temperatures above 50°C.

Internal Pressure at 50°C: 1640 psi

Operators of this equipment shall be adequately trained in the handling and use of cryogenic liquids and cryogenically cooled parts.

They shall be competent with the management of apparatus which may be pressurised and under extremes of temperature.

Adequate PPE shall be used at all times.

## 4. Safety Warnings

### 4.1. Do Not Modify or Disassemble

Do not use the apparatus for jobs other than those for which it was designed.

- There are no user serviceable parts inside.
- Do not dismantle or modify the apparatus.
- For repair information contact Isothermal Technology Ltd.

### 4.2. Be Careful Where It Is Used

- The equipment is not for use in hazardous areas or in an environment close to flammable materials or gases.
- The equipment must be used on a flat level surface, with adequate space around it for air flow and avoid blocking ventilation slots.
- Ensure free space above the unit to avoid risk of cold burns.
- The equipment should only be used indoors in an area where there is adequate ventilation.
- Avoid excessive heat, humidity, dust and vibration.

### 4.3. Be Careful With Extremes of Temperature

- Do not use the equipment outside its recommended temperature range.
- Wear appropriate protective clothing.

## 5. Introduction

The Argon Triple Point Apparatus is both simple in construction and in use. A video accompanying this handbook describes the apparatus and shows you how to use it, please take a look at [www.isotech.co.uk/argon](http://www.isotech.co.uk/argon).

### 5.1. Precautions

The apparatus uses liquid nitrogen. The user should be adequately trained and familiar with handling and use of liquid nitrogen.

Do this **before** you try and use the apparatus.

Ensure the room of use is well ventilated. Large volumes of gaseous nitrogen are produced as the cryogenic liquid nitrogen boils.

### 5.2. On Arrival

For transportation a solid rod of stainless steel has been placed down the re-entrant tube of the apparatus to increase the rigidity during transport, please ensure you remove this before use.

Check you have the following items:

- User maintenance manual
- The cased apparatus with three tubes exiting the top.
- A pressure safety valve (hexagonal screw-on sealing cap with over pressure disc). Note: The filling tube should have this screwed onto it for transit.
- 2 nitrogen filling funnels with long and short tubes (pipes) exiting the bottom.
- A plastic funnel.
- A bag marked "Pressure Release Valve Assembly" containing:
  - a) Two ball bearings (do not lose them).
  - b) A tube with hexagonal connector at one end (pressure release tube).
  - c) A disc with 3 holes (base plate).
  - d) A glass dome.
  - e) A selection of washer shaped weights.
  - f) A cap with 4 holes and a ledge (pressure release valve).(See Appendices)

You will need to provide:

- **Liquid nitrogen (N<sub>2</sub>):** 15 to 18 litres per day of use. (dependant on starting temperature)
- **A suitable jug/flask:** about 1 to 2 litres in volume to transfer the N<sub>2</sub> from its container to the funnels.
- **An allen key (1.5mm):** to fix the disc to the pressure release tube.
- **Tissues**
- **An adjustable spanner/wrench**
- **A hot air gun or hairdryer:** to warm the container outlet tube.
- **A barometer:** to know the ambient pressure.
- **Protective clothing:** when handling the liquid nitrogen.



## 6. Realisation procedure

**Important Note:** It is advisable before starting the filling process to check that the central re-entrant tube does not contain any condensed moisture inside. This can be done by inserting a length of absorbent pipe-cleaner repeatedly until the re-entrant tube is dry. Keep the room well ventilated as a lot of nitrogen boils off as the cell cools.

### 6.1. Inserting the filling funnels

There are two funnels which are inserted into the apparatus to guide the liquid nitrogen into the apparatus. A small funnel with a long angled outlet tube which is inserted into the central tube (the re-entrant tube) of the apparatus. The larger funnel has a shorter outlet tube which is inserted into the filling tube after removing the pressure safety valve (See Appendix 1).

You are now ready to fill (tip: Put some tissues on top of the apparatus and wrap some tissue around the exposed tubes to absorb moisture condensing from the atmosphere).

### 6.2. Filling the apparatus with liquid nitrogen (N<sub>2</sub>)

- Initially only fill the small funnel with liquid nitrogen and leave for 15 to 20 minutes, this ensures that the re-entrant tube is the coolest part of the cell.
- After the initial 15 to 20 minutes fill both funnels, this starts to cool the cell from the top.  
**Important Note: The plastic funnel when inverted fits over the large funnel to reduce splashing.**
- Refill the funnels every 15 minutes for the first hour, then every 10 minutes thereafter.
- Once the cell has cooled the larger funnel starts to empty quickly as the nitrogen fills the apparatus. The larger funnel can now be filled as soon as it empties.
- When approximately 15 litres of liquid nitrogen have gone into the apparatus liquid nitrogen will flow out of the fixed part of the pressure release tube (See Appendix 1).

The apparatus should be left to rest for 30 minutes, then add liquid nitrogen to the funnels until it flows out of the pressure release tube again, the apparatus should be left to rest for a further 15 minutes, after which the funnels can be removed.

Filling the apparatus uses typically 15 litres of liquid nitrogen, and take about 2 ½ hours. There are 39g of argon in the cell.

### 6.3. Calculating the weight for the pressure release valve assembly

- From the certificate of conformity the weight and pressure detailed on the melt graph should be noted. The ambient pressure should then be measured using an accurate barometer.
- The weight for the pressure release valve should be reduced by 1 g for every 7 mb **above** the pressure stated on the certificate of conformity.
- 1 g should be added for every 7 mb **below** the pressure stated on the certificate of conformity.
- After the 30 minutes, the apparatus is sealed by screwing on the pressure safety valve which is pre-set to 3 bar. (See Appendix 2)

**Note:** So what is happening as we fill the apparatus? Nitrogen from the small funnel fills the re-entrant tube making it the coolest part of the cell. Nitrogen from the larger funnel cools the cell from the top slowly enough so that the Argon inside the cell can condense onto the underside of the top of the cell and drip down into a well surrounding the re-entrant tube. The liquid argon eventually fills the well and freezes, cooling to the liquid nitrogen temperature.

Once the cell has cooled more nitrogen fills the apparatus to the top.

### 6.4. Inserting the SPRT

At this stage insert an SPRT that has been pre-cooled in liquid nitrogen into the re-entrant tube, this will cool the thermometer before entering the cell and also displace the liquid nitrogen in the re-entrant tube.

## 6.5. Pressure release valve assembly fitting

**Important Note: Keep the pressure release valve assembly parts dry and free of frost.**

- a. Defrost the thread and washer on the fixed section of the pressure release tube with the hot air gun until dry, then screw on the pressure release tube and base plate assembly and tighten.
- b. Add the pressure release valve (with the bearings inside) and weights (calculated in section 6.3) as shown in Appendix 1. Dry these in situ with the hot air gun to ensure frost-free operation and place the glass dome over the assembly to prevent frosting of the valve.

**Note: The main bearing supplied is critical for the correct operation of the apparatus and should be positioned so that it sits directly on top of the pressure release tube.**

## 6.6. Realising the melt plateau

Monitor the SPRT, after initial cooling the SPRT temperature will slowly rise with the apparatus temperature until the triple point is achieved, typically 2 to 3 hours.

Remove the SPRT for 5 to 10 seconds wiping down with a clean dry tissue and re-insert, this acts as a warm rod to move the cell onto the flattest part of the plateau.

The plateau will last about 3 hours during which time calibrations can be performed.

Pre-cool subsequent SPRT's for calibration in liquid N<sub>2</sub>.

Note: Pressure and temperature are related, and so by pressurising the liquid nitrogen its boiling temperature can be increased.

Approximately 170g (including the pressure release valve and bearings) is needed to raise the boiling temperature to just above the argon triple point.

The pressure release valve assembly temporarily seals the apparatus. The pressure and temperature rise inside the apparatus and the valve will open, allowing nitrogen gas to escape. At this point a faint hiss can be heard and the glass dome frosts over.

Different diameter ball bearings will need different weights. Take care when assembling or taking apart not to lose them.

## 6.7. Using the apparatus on subsequent days

To reuse the next day – check the apparatus is not still pressurised, this can be done by partially unscrewing the over pressure valve, if gas can be heard escaping, leave the valve in place until it subsides then remove the valve.

**Carefully** unscrew the pressure release valve assembly and put in a warm, dry place.

## 6.8. Timeline guide

Filling and sealing takes about 2 to 3 hours. Exact time and volumes of nitrogen will vary depending on local ambient conditions.

## 7. Adjustments

The standard way of optimising the apparatus is to change the weights on the pressure release valve. This can compensate for local and daily variations in ambient pressure.

Increasing the weight will speed the warming process, but reduce the plateau length.

Reducing the weight will slow the warming, lengthening the plateau and may even stop the plateau occurring.

Whilst it is possible to change the diameter of the ball bearings in the pressure release valve, this is not advisable. This changes the seat diameter of the ball on its seat within the pressure release valve with quite dramatic effects on the weight requirements.

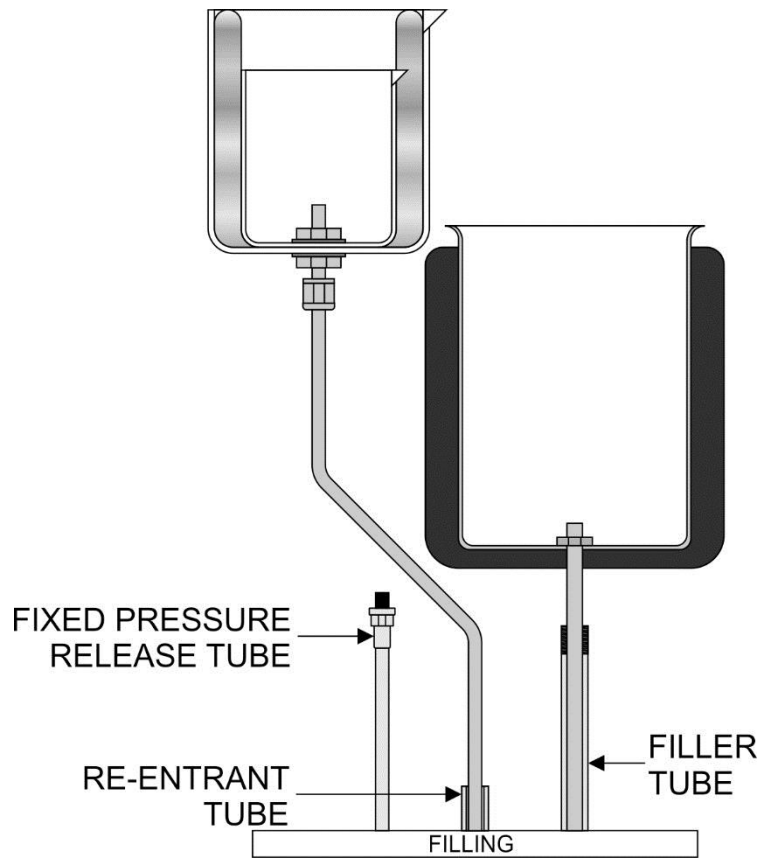
### 7.1. Some equivalents

1g change in the weight of the pressure release valve assembly is equivalent to 46 mK change in set point temperature

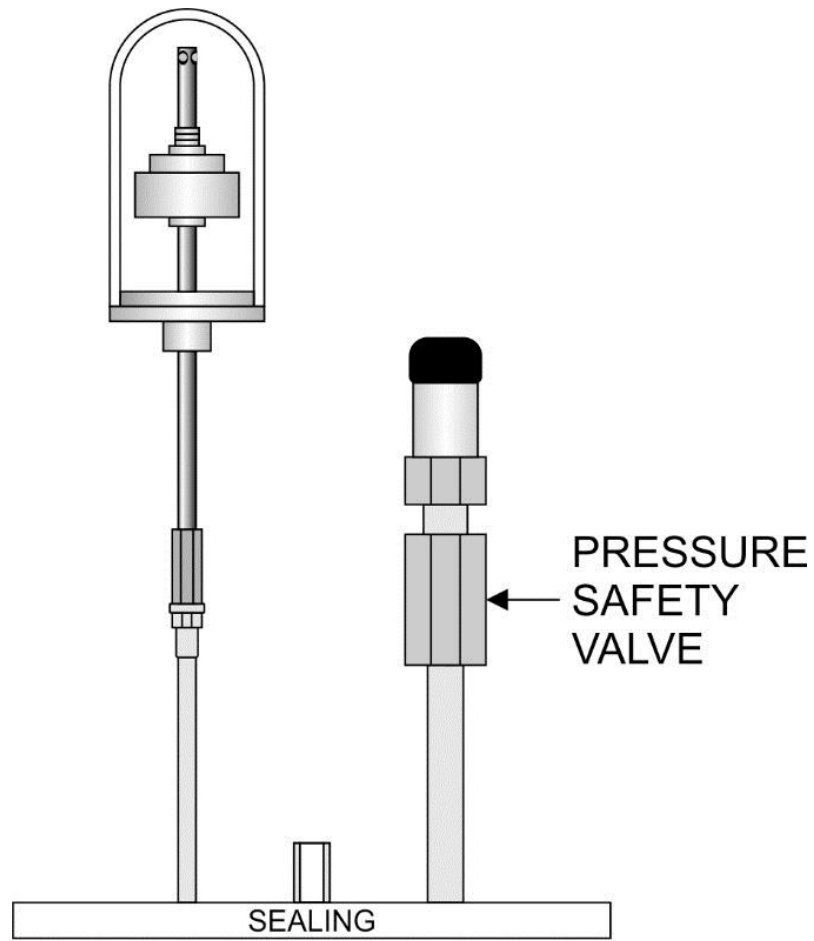
7 mb change in ambient pressure is equivalent to a 1g change. As the ambient pressure increases, reduce the weight.

## 8. Appendices

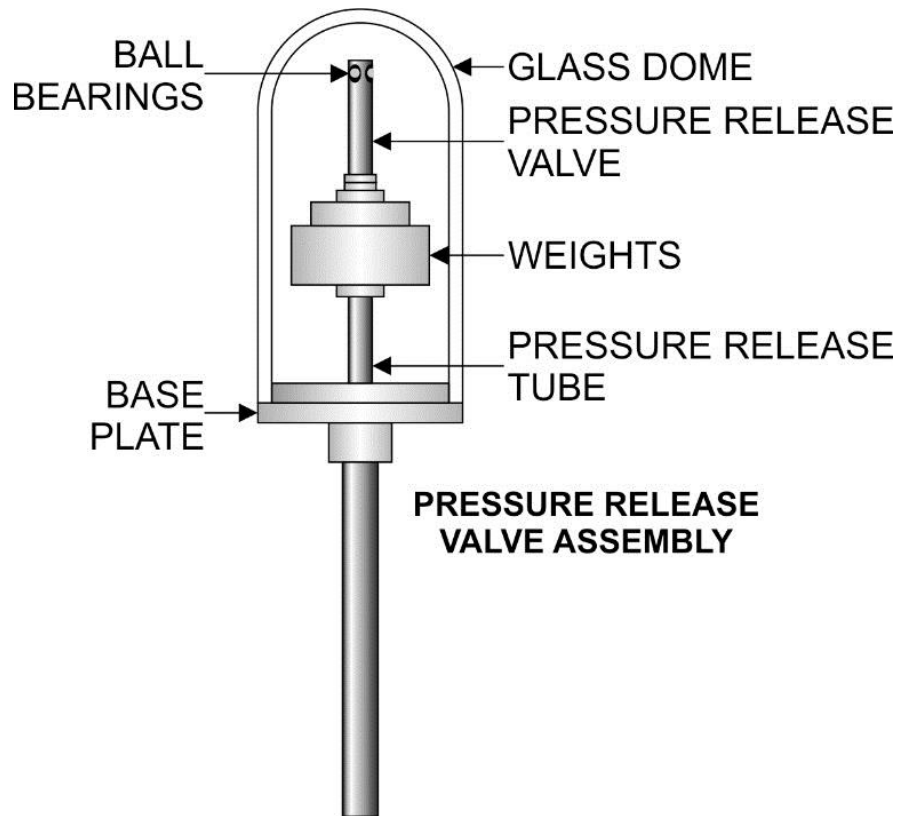
### 8.1.Appendix 1



## 8.2.Appendix 2



### 8.3.Appendix 3



## 9. Certificate of Conformity

A separate certificate of conformity is provided with unit and includes a purity analysis certificate for the Argon used in the cell and a melt curve recorded from the unit which includes the weight used for the pressure relief valve assembly and the ambient pressure.

A full inter-comparison of your apparatus to our reference cell is available at extra cost.