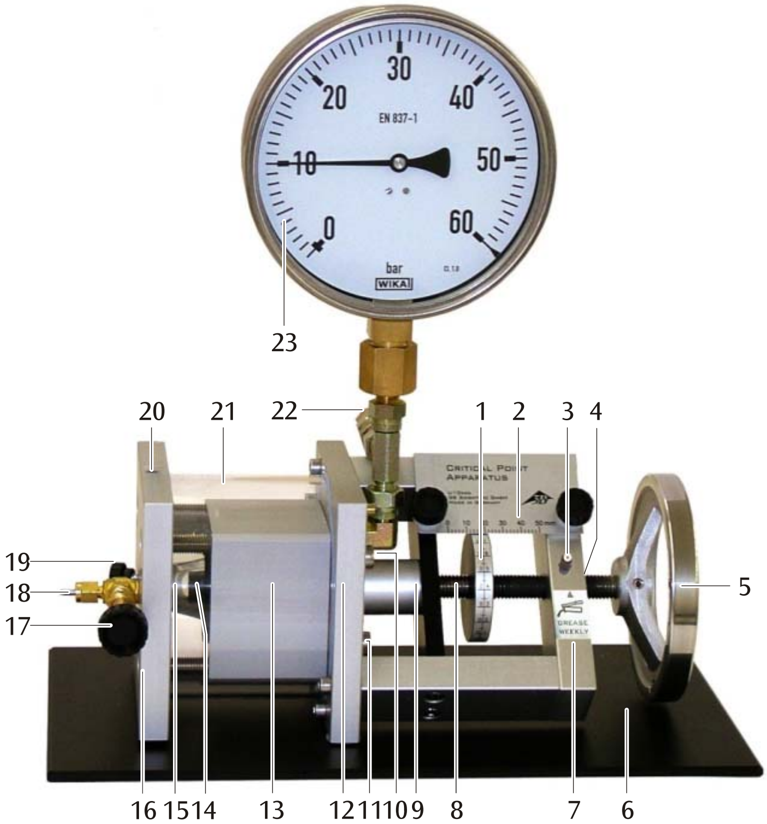
3B SCIENTIFIC® PHYSICS

...going one step further

Critical Point Apparatus 1002670

Instruction sheet

02/13 MH/JS



1 Vernier scale
2 Fixed scale
3 Grease nipple
4 Threaded bush
5 Handwheel
6 Base
7 Frame
8 Threaded axle with piston
9 Piston cover
10 Outlet for thermal medium
11 Inlet for thermal medium
12 End plate
13 Cylinder
14 Conical seal
15 Measuring cell
16 Valve plate
17 Regulating valve
18 Gas connection fittings 1/8" (for Minican® gas container)
19 Flush valve
20 Hole for thermocouple
21 Heat casing
22 Safety valve
23 Manometer (excess pressure indicator)

1. Contents of instruction manual

On delivery, the critical point apparatus is filled with hydraulic fluid. The test gas is not included.

Before filling with the test gas, carry out a volume calibration, as described in chapter 6, using air as an approximation of an ideal gas.

Filling with the test gas is described in chapter 7.

Experimental investigations are described in chapter 8.

Important notes on storage of the test gas and equipment (if not in use for a long period) are stated in chapter 9.

Owing to the inevitable diffusion of the test gas through the conical seal, it is necessary to degas the hydraulic fluid in the equipment, as described in chapter 10. This must be done before the equipment is put away for storage (after removing the test gas) or if it has been in use for a long time.

The threaded bush in the frame must be lubricated regularly and also inspected at lengthier intervals. Refer to section 11 for instructions.

Maintenance work as described in chapter 12 is only required if the rubber components get worn out and their functionality is adversely affected.

2. Safety instructions

When used properly, the operation of the critical point apparatus is not dangerous, since both the experimenter and the equipment are protected by a safety valve. However, it is extremely important to observe a few precautionary measures:

- Read the instruction sheet thoroughly and follow the instructions therein.
- Do not exceed the maximum permissible values for pressure and temperature (60 bar and 10-60°C).
- Do not operate the equipment without qualified supervision.
- Always wear safety goggles.

Only increase the temperature at low pressure with pure gas phase in the measuring cell.

- Before increasing the temperature, wind the handwheel outwards so that maximum volume is attained in the measuring cell.

When conducting adjustments, make sure that the safety valve does not point in the direction of people who could be injured or objects that could be damaged if the valve cover shoots out. When conducting experiments, pay special attention too to the alignment of the safety valve.

- When setting up the apparatus, make sure that the safety valve does not point in the direction of people who could be injured or objects that could be damaged.
- When adjusting the safety valve, wrap your arms around the apparatus to reach the valve at the back.

If the conical seal is overtaxed, it could get damaged or even destroyed.

- Never set a pressure above 5 bar if the regulating valve or the flush valve is open, i.e. if there is no back pressure from the gas in the measuring cell.
- Never create underpressure by turning the hand wheel inwards when the valves are shut.

In the frame there is a threaded bush, which is to be regarded as a safety-related feature (see section 9).

- Lubricate the threaded bush every 100 cycles.
- Inspect the threaded bush annually.

To prevent damage by corrosion inside the instrument,

- use a 2:1 mixture of water and anti-freeze fluid as the thermal medium.

Only as real gas for SF_6 and nitrogen as ideal gas.

3. Description

The critical point apparatus allows us to investigate the compressibility and liquefaction of a gas. Measurements allow determination of the critical point for the gas as well as the recording of isotherms for an adiabatic *p-V* diagram (Clapeyron diagram). The gas used for testing is sulphur hexafluoride (SF_6). SF_6 has a critical temperature of 318.6°K (45.5°C) and a critical pressure of 3.76 MPa (37.6 bar) which makes for a simple experiment set-up.

The critical point apparatus consists of a transparent measuring cell of particularly well sealed, pressure-resistant design. The volume of the measuring cell can be modified by turning a fine-adjustment wheel and can be read by means of a fixed scale and a rotating vernier scale to an accuracy of one thousandth of the maximum volume. The pressure is applied via a hydraulic system using castor oil approved for medicinal use. The measuring cell and hydraulic system are isolated from one another by a conical seal which rolls up when there is an increase in pressure. This design means that any pressure difference between the measuring cell and the oil reservoir is negligible in practical terms. A manometer measures not the pressure of the actual gas but that of the oil, thus eliminating any need for a space within the measuring cell. When observing transitions from gas to liquid or vice versa, the lack of such a dead space means that the development of the very first drop of liquid as well as the disappearance of the last bubble of gas can be observed. The measuring cell is surrounded by a transparent chamber of water. A circulating thermostat arrangement (water bath) means that a constant temperature can be maintained during the experiment with a high degree of accuracy. The temperature can be read and monitored using a thermometer.

The fact that volume, pressure and temperature can all be read with a high degree of accuracy means that accurate *p-V* diagrams or *pV-p* diagrams can be recorded without much difficulty. Pressure and temperature-dependent volume correction enable us to achieve accurate quantitative results which are well in agreement with published values.

4. Contents

1 Critical point apparatus, filled with hydraulic fluid (castor oil). With attached gas connection fittings for MINICAN® gas container and protection for gas supply connections. Test gas (SF_6) not included.

1 Oil filling device

1 Allen key, 1.3 mm (for grub screw on the vernier scale)

1 Plastic tubing, 3 mm diameter

1 1/8" tube fitting (wrench width 11 mm)

1 Grease gun

5. Technical data

Sulphur hexafluoride:

Critical temperature:	318.6 K (45.5°C)
Critical pressure:	3.76 MPa (37.6 bar)
Critical volume:	197.4 cm³/mol
Critical density:	0.74 g/mol

Maximum values:

Temperature range:	10-60°C
Maximum pressure:	6.0 MPa (60 bar)
Threshold value for safety valve:	6.3 MPa (63 bar)
Theoretical long-term pressure:	7.0 MPa (70 bar)
Theoretical rupture pressure:	>20.0 MPa (200 bar)

Materials:

Test gas:	Sulphur hexafluoride (SF_6)
Hydraulic fluid:	Castor oil
Measuring cell:	Transparent acrylic
Temperature coating:	Transparent acrylic
Recommended thermal medium:	mixture of water and anti-freeze in the ratio 2:1

Determination of volume:

Piston diameter:	20.0 mm
Piston surface:	3.14 cm²
Displaced volume:	3.14 cm² × displacement
Maximum volume:	15.7 cm³
Scale division for displacement:	0.05 mm
Maximum displacement:	50 mm

Determination of pressure:

Manometer:	Class 1.0 (max. 1% deviation from full scale value)
Measured quantity:	Excess pressure
Indicator:	60 bar max.
Manometer diameter:	160 mm

Connections:

Hole for temperature sensor:	6 mm dia.
Connections for thermal medium:	7 mm dia.
Connection for regulating valve:	1/8” dia.
Gas connection:	1/8” (3.17 mm) dia. (as supplied)

General specifications:

Dimensions:	380 x 200 x 400 mm³
Weight:	7 kg approx.

6. Volume calibration

6.1 Preliminary notes:

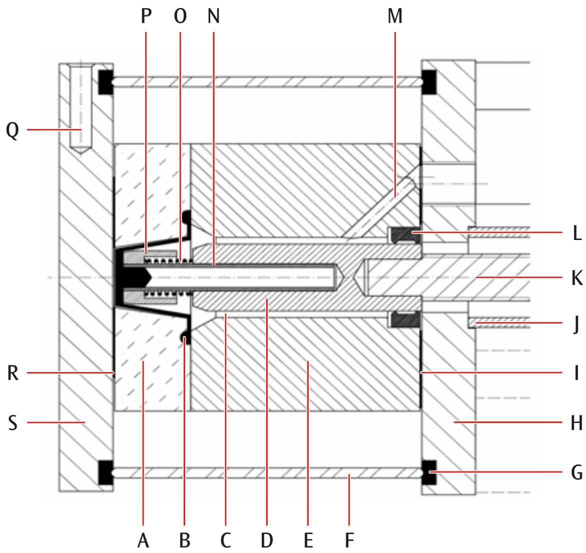


Fig. 1: Cross-section of apparatus with measuring cell (A), conical seal (B), oil chamber (C), piston (D), cylinder (E), heat casing (F), silicone seal (G), end plate (H), square grommet (I), piston cover (J), threaded axle (K), gasket (L), manometer connection (M), guide tube (N), spring (O), sleeve (P), hole for temperature sensor (Q), circular grommet (R) and valve plate (S)

One turn of the handwheel winds the piston into/out of the cylinder by means of a threaded axle. This leads to a change of volume in the oil chamber (see Fig. 1). Since oil is practically incompressible and all the other components other than the conical seal are almost rigid, a change in volume in the oil chamber causes the conical seal to deform, thereby creating an almost equal change in volume ΔV_G in the measuring cell. As a first approximation for ΔV_G, we can assume:

$$\Delta V_G = A \cdot \Delta s \qquad (1)$$

where $A = 3.14\ \text{cm}^2$ and Δs = displacement of piston.

The piston displacement is shown in divisions of 2 mm on the fixed scale. Intermediate values are read on the vernier scale in divisions of 0.05 mm.

The fixed scale can be moved by loosening the two knurled screws. The vernier scale can be repositioned and turned around the threaded axle on loosening the grub screw (between scale positions 0 9 and 1 0).

6.2 Zero point calibration:

The zero point for the volume scale must be determined by conducting a calibration.

For this, we take advantage of the fact that in a pressure range of 1-50 bar and in a temperature range of 270-340 K, air acts as a near-ideal gas (the real gas factor has a deviation of less than 1% from 1). Therefore, at a constant temperature (e.g. room temperature) for two piston displacements s_0 and s_1 and for the corresponding pressures p_0 and p_1 of the trapped air, we get:

$$p_0 \cdot s_0 = p_1 \cdot s_1 \quad (2)$$

Substituting $s_0 = s_1 + \Delta s$ and rearranging gives:

$$s_1 = \frac{p_0}{p_1 - p_0} \cdot \Delta s \quad (3)$$

Rough calibration of scales:

- Open the regulating valve wide.
- Loosen the grub screw for the vernier scale by half a turn (it is now possible to turn the scale easily on the threaded axle without moving the handwheel, although a counterpressure acts against this independent movement).
- Wind the handwheel out till you detect a noticeable resistance.
- Without turning the handwheel, turn the vernier scale on the threaded axle till the 0.0 mark is on the top and the fixed scale shows approx. 48 mm.
- Loosen the knurled screws of the fixed scale and shift the scale to the side till the 48-mm bar is exactly above the centre line of the vernier scale (see Fig. 2).
- Tighten the knurled screws again. In doing so, make sure that the fixed scale does not press against the vernier scale.

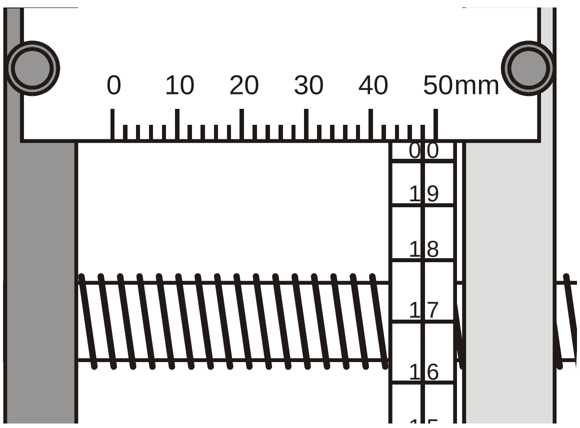


Fig. 2: Piston position reading at 48.0 mm

Zero correction:

- Shut the regulating valve (the pressure in the measuring cell now corresponds to the ambient pressure p_0 = 1 bar. To within the accuracy of the measurement, the manometer should display an excess pressure of 0 bar).
- Wind the handwheel in till an excess pressure of 15 bar has been reached (absolute pressure p_1 = 16 bar).
- Read the piston position s_1 and calculate the displacement $\Delta s = s_0 - s_1$.
- Calculate the zero corrected piston position $s_{1,\,corr}$ using Equation 3.
- Adjust the vernier scale to the corrected value and, if necessary, move the scale again.
- If required, wind the handwheel out a little and secure the vernier scale with the grub screw.

Measurement example:

p_0 = 1 bar, p_1 = 16 bar, $p_1 - p_0$ = 15 bar
s_0 = 48.0 mm, s_1 = 3.5 mm, Δs = 44.5 mm
Therefore, $s_{1,\,corr}$ = 2.97 mm.

The vernier scale must therefore be adjusted so that now only 2.97 mm are shown instead of 3.50 mm.

Note:

After calibrating the zero point, it is possible to obtain qualitatively accurate measured values. With regard to temperature T and pressure p, it is also possible to obtain quantitatively accurate measurements of the isotherms in range around to the critical point where the two phases exist simultaneously. However, especially in the liquid phase, the measured isotherms are rather too widely separated.

6.3 Detailed calibration:

The exact relation between the volume V_G in the measuring cell and the scale reading s is dependent on the volume of oil in the oil chamber. The oil chamber also expands marginally in proportion to the pressure as a result of the spring in the manometer tube. Additionally, when the temperature is increased, the castor oil expands to a greater extent than the rest of the equipment. This means that the pressure rises at a slightly greater rate at higher temperatures. All of these phenomena can be calculated if appropriate calibration has been effected using air as an ideal gas.

The ideal gas equation would thus be:

$$\frac{p \cdot V}{T} = n \cdot R \quad (4)$$

with $R = 8.314 \frac{\text{J}}{\text{K mol}}$

After taking the overpressure reading p_e, the absolute pressure can be calculated from:

$$p = p_e + 1 \text{ bar} \quad (6)$$

The absolute temperature is given by:

$$T = \vartheta + \vartheta_0 \text{ where } \vartheta_0 = 273.15°\text{C} \quad (7)$$

The volume is given by:

$$V_G = A \cdot s \quad (8)$$

where $A = 3{,}14 \text{ cm}^2$ and s is the "effective" piston displacement.

From the measured displacement s_e, it is possible to calculate the effective piston displacement as follows:

$$s = s_e + s_0 + C_p \cdot p - C_\vartheta \cdot \vartheta \quad (9)$$

By substituting in equation 4, we get:

$$\frac{p \cdot (s_e + s_0 + \beta_p \cdot p - \beta_\vartheta \cdot \vartheta) \cdot A}{\vartheta + \vartheta_0} - n \cdot R = 0 \quad (10)$$

If we take several readings at various temperatures and pressures, we can calculate the term:

$$Q = \sum_{i=1}^{n} \left(\frac{p_i \cdot (s_i + s_0 + \beta_p \cdot p_i - \beta_\vartheta \cdot \vartheta_i) \cdot A}{\vartheta + \vartheta_0} - n \cdot R \right)^2 \quad (11)$$

The free parameters s_0, β_P, β_ϑ and n should be appropriately selected so that the value of Q is reduced to a minimum.

Additionally required (see also chapter 8):

1 Compressor or bicycle pump and valve
1 Bath/circulating thermostat 1008653/1008654
1 Dig. quick-response pocket thermometer 1002803
1 Type K NiCr-Ni immersion sensor, -65°C-550°C 1002804
2 Silicone tubes, 1 m 1002622
1 l Anti-freeze fluid with corrosion-inhibiting additive for aluminium engines (e.g., Glysantin® G30 manufactured by BASF)

Conducting the calibration:

- Connect the circulation thermostat as described in chapter 8 and fill it with the water/anti-freeze mixture.
- Connect the plastic tube (3-mm internal diameter) to the 1/8" gas connection fittings.
- Open the regulating valve.
- Wind the handwheel outwards, making the piston move till it reaches say the 46.0 mm position.
- Use a compressor or a bicycle pump to create an excess air pressure of approx. 3-8 bar in the measuring cell.
- Shut the regulating valve.
- To record measurements, vary the volume in the measuring cell or the temperature of the thermostat and wait till a stationary equilibrium has been attained. Then take a pressure reading.
- Use appropriate adjustment software to set the s_0, β_P, β_ϑ and n parameters so that the quadratic equation for the errors Q is reduced to a minimum (see equation 11).
- If you like, you can adjust the vernier scale around s_0 so that this correction is not necessary.

With the set parameters, it is possible to calculate the "effective" piston displacement s from the measured displacement s_e using Equation 9 and then to calculate the calibrated measuring cell volume using Equation 8.

Sample measurements:

Table 1: Measured values for calibration

i	s_e / mm	ϑ	p / bar
1	40.0	20.0°C	6.6
2	20.0	20.0°C	12.4
3	10.0	20.0°C	23.3
4	5.0	20.0°C	41.8
5	3.5	20.0°C	53.9
6	5.0	20.0°C	41.8
7	5.0	10.0°C	38.9
8	5.0	30.0°C	45.3
9	5.0	40.0°C	49.0
10	5.0	50.0°C	53.5

The following parameter values are obtained:

s_0 = 0.19 mm, $\beta_P = 0.023 \frac{\text{mm}}{\text{bar}}$, $\beta_\vartheta = 0.034 \frac{\text{mm}}{\text{grd}}$ and n = 0.00288 mol.

7. Filling with test gas

7.1 Handling of sulphur hexafluoride:

Sulphur hexafluoride (SF_6) is a non-toxic gas and is absolutely safe for humans. The MAC value for danger of suffocation on account of oxygen deprivation is 1000 ppm. That is equivalent to 6 filled measuring cells per 1 m^3 of air.

However, SF_6 is extremely harmful to the environment and can give rise to a greenhouse effect 24,000 times stronger than CO_2. Therefore, do not allow large quantities to be released into the environment.

7.2 Gas connection via fixed pipes:

Additionally required:

1 SF_6 gas cylinder with manufacturer's/supplier's recommended gas fittings/valves, e.g. SH ILB gas cylinder and Y11 L215DLB180 regulating valve from Airgas (www.airgas.com).

1 Pipes with outer diameter of 1/8" and, if necessary, adapters, e.g. from Swagelok (www.swagelok.com).

1 open-end spanner (13 mm), 1 open-end spanner (11 mm)

According to the principles of "good laboratory practice", it is recommended to utilise a gas supply via fixed pipes, especially if the equipment is regularly in operation.

Filling begins with several flush cycles in which the air is flushed out of the pipe. The number of cycles required to flush out the air depends on the length of the pipe (more precisely, on the ratio of the pipe length to the volume of the measuring cell). In the process, care should be taken that the quantity of the greenhouse gas SF_6 released in the environment is reduced to a minimum.

Connecting a fixed pipe:

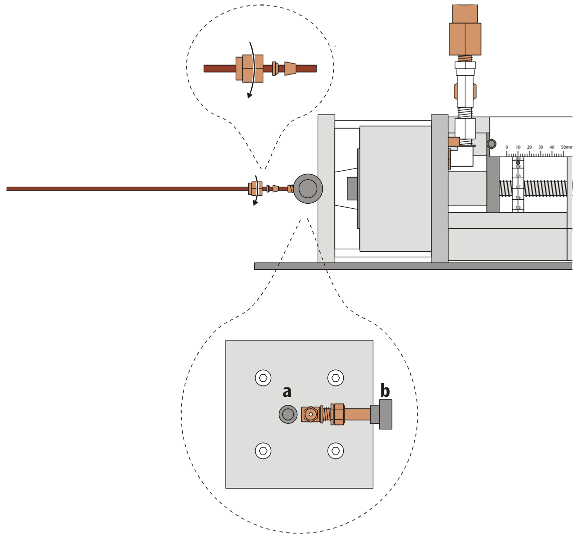


Fig. 3: Connecting a fixed pipe
(a) flush valve, (b) regulating valve

- If necessary, pull out the protection for the gas connection and loosen the valve nut (11 mm) to remove the 1/8" gas connection fittings.
- Connect the pipe (if necessary with adapters) to the gas fitting.
- Beginning with the valve nut, slide the supplied screw joints onto the tubing. (See Fig. 3: follow the sequence and alignment specified along with the cable binder)
- Insert the pipe into the regulating valve and tighten the valve nut till the point is reached where it is no longer possible to move the pipe any further using only your fingers.
- Hold the regulating valve still with an open-end spanner (13 mm) and tighten the valve nut by a further 270°.

Now, the connection is gas-tight. When loosening the valve nut afterwards, the regulating valve also needs to be held still with a spanner.

Flushing out air:

- Use the handwheel to set the piston position to 10 mm.
- Slowly open the regulating valve and let in the SF_6 till a pressure of approx. 10 bar has been attained.
- Shut the regulating valve.
- Open the flush valve slightly till the pressure has dropped to almost 0 bar.
- Shut the flush valve.

Filling with test gas:

- After at least four flush cycles, open the regulating valve till the pressure attained is once again 10 bar.
- Shut the regulating valve.
- Turn the handwheel in the reverse direction till the piston reaches a position of say 46 mm.
- Slowly open the regulating valve and shut it again when a pressure of 10 bar has been attained.

7.3 Filling with gas from a MINICAN®:

Additionally required:

1 MINICAN® gas container with SF_6, e.g. from the company Westfalen (www.westfalen-ag.de

If the equipment is used only occasionally, it is more practical to draw the test gas from a MINICAN® gas container. The gas connection of a MINICAN® container is similar in design to a commercial spray can, i.e. it opens when the MINICAN® container is pressed directly onto the gas connection fittings.

Here too, filling begins with several rinsing cycles for flushing out the air.

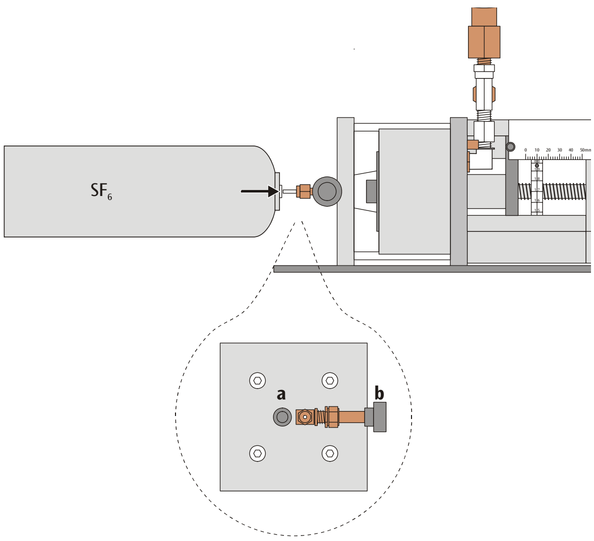


Fig. 4: Filling with test gas from a MINICAN® gas container
(a) flush valve, (b) regulating valve

Flushing out air:

- If necessary, pull off the protection for the gas connection.
- Use the handwheel to set the piston position to 10 mm.
- After removing the protective cap, position the MINICAN® container with SF_6 onto the gas connection fittings.
- Press the MINICAN® container onto the gas connection fittings, slowly open regulating valve (b) and let in SF_6 till a pressure of approx. 10 bar has been attained.
- Shut the regulating valve.

- Open the flush valve slightly till the pressure has dropped to almost 0 bar.
- Shut the flush valve.

Filling with test gas:

- After at least four flush cycles, press the MINICAN® gas container against the gas connection fittings. Slowly open the regulating valve and let in SF_6 till a pressure of approx. 10 bar has been attained.
- Shut the regulating valve.
- Wind the handwheel in the opposite direction till the piston reaches a position of say 46 mm.
- Press the MINICAN® gas container against the gas connection fittings, slowly open the regulating valve and shut it again when a pressure of 10 bar has been attained.

7.4 Recommendation for storage lasting for short periods of time:

One gas filling can remain in the measuring cell for several days.

If no experiments are being conducted, wind the handwheel back till the piston is in a position where it is subjected to the lowest possible pressure – say, for instance, 46 mm.

If possible the apparatus should always be kept filled with the thermal medium.

8. Experiments

8.1 Experiment set-up:

Additionally required:

1	Bath/circulating thermostat	1008653/1008654
1	Dig. quick-response pocket thermometer	1002803
1	Type K NiCr-Ni immersion sensor, -65°C-550°C	1002804
2	Silicone tubes, 1 m	1002622
1 l	Anti-freeze fluid with corrosion-inhibiting additive for aluminium engines (e.g., Glysantin® G30 manufactured by BASF)	

- Place the equipment at a suitable height so that it is convenient to observe the measuring cell. Position it so that the safety valve does not point in the direction of any people who could be injured or objects that could be damaged.
- Connect the silicone tubing from the outlet of the circulation thermostat to the inlet of the heat casing and from the outlet of the heat casing to the inlet of the circulation thermostat.
- Prepare the thermal medium consisting of 2 parts water to 1 part anti-freeze by volume.
- Fill the circulated thermostat bath.

8.2 Qualitative observations:

Liquid and gaseous states, dynamic state during phase transformation, transition points occurring at different temperatures.

- Vary the volume by turning the handwheel and the temperature by means of the thermostat. Observe the safety instructions while doing so.
- Carefully shake the set-up to conduct simple observations on the boundary between liquid and gas.

In the vicinity of the critical point, it is also possible to observe the critical opalescence. Owing to the constant changing of state between liquid and gaseous states in small regions of the measuring cell, a kind of “mist” develops and the sulphur hexafluoride appears to be turbid.

8.3 Measuring isotherms in a *p*-*V* diagram:

- At maximum volume, set the desired temperature on the circulation thermostat.
- Gradually reduce the volume in the measuring cell (in steps down to a position of 10 mm). Wait till a stationary equilibrium has been attained before taking pressure readings.
- Then, beginning with the minimum volume, gradually increase the volume till the piston position is once again at 10 mm. Wait till a stationary equilibrium has been attained before taking pressure readings.
- Convert the excess pressure readings into absolute pressure and the piston positions into volume, as described in chapter 6.

In the low-volume region, stationary equilibrium is attained more quickly during transition from higher to lower pressure – i.e. from a lower volume to a greater volume – since the phase boundary layer for the phase transition from liquid to gas is created by vapour bubbles present throughout the liquid. Stationary equilibrium then takes around 1 to 5 minutes to attain, whereby the measurements on the fringe of the region where both phases exist take longest.

The recommended threshold value of 10 mm refers to a filling pressure of 10 bar. Above this value, there will certainly be no occurrence of a liquid phase in the permissible temperature range. The threshold value shifts to the “right” if the filling pressure is higher.

8.4 Measuring isochores in a *p*-*T* diagram:

- Set the desired initial temperature. Subsequently set the desired volume.
- Gradually allow the temperature to decrease.
- Wait till a stationary equilibrium has been attained then take the pressure reading.

Measurements where both phases are present can be plotted to generate a vapour-pressure curve.

Attainment of equilibrium takes up to 20 minutes after each change of temperature due to the fact that the water bath and the measuring cell must attain the desired temperature first.

8.5 Determining the mass of gas:

Blow the gas out of the measuring cell into a gas-tight plastic bag and then weigh it:

- If necessary, remove the gas supply pipe and attach gas connection fittings.
- Wind out the handwheel, say to 46 mm.
- Open the regulating valve a little and release the gas through the gas connection fittings into the plastic bag.
- Shut the regulating valve.
- Determine the mass of the released gas. In doing this, take into consideration the empty weight of the bag and the buoyancy of air.
- Reduce the volume of the measuring cell till the pressure in the measuring cell has reached its original value.
- Calculate the original mass of gas from the volume difference before and after emptying the measuring cell and the volume which is still present in the measuring cell.

Comparison with quoted values:

Using tabulated values, e.g. Clegg et al. [4], it is alternatively possible to calculate the mass of gas in the measuring cell from the measurements of ϑ, p, and V.

8.6 Evaluation:

We can clearly see from Fig. 5 that, despite the relatively simple equipment, it is possible to achieve measurements which match closely to the reference values plotted on the graph.

8.7 Bibliography:

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[6] Vargaftik, N.B.: Handbook of Physical Properties of Liquids and Gases, 2nd ed., Hemisphere Publishing Corporation, Washington, 1983

[7] Nelder, J. and Mead, R.: Comp. J., Vol. 7, p. 308, 1965

9. Storage for long periods without use

If no experiments are to be conducted over a long period, the test gas should be released and the piston should be turned to its rest position where the conical seal is only very slightly curled and does not press against the walls of the measuring cell.

- If necessary, allow the equipment to cool. Wind the handwheel back till the lowest possible pressure is present.
- Release the test gas through the flush valve.
- Turn the handwheel to move the piston to its “rest position”, at approx. 5 mm.
- Shut the flush valve again.

- Before storing away the equipment, the hydraulic fluid needs to be degassed (as described in chapter 10) if the equipment has been in use over a long period of time.

- Store the equipment in a safe place where it is not exposed to direct sunlight.

- The thermal medium should be kept in the apparatus during storage, as the additives inhibit corrosion and efflorescence caused by electrochemical potentials between the different materials. Alternatively, the apparatus can be flushed with deionised water and then dried using compressed air (oil-free, max. 1.1 bar).

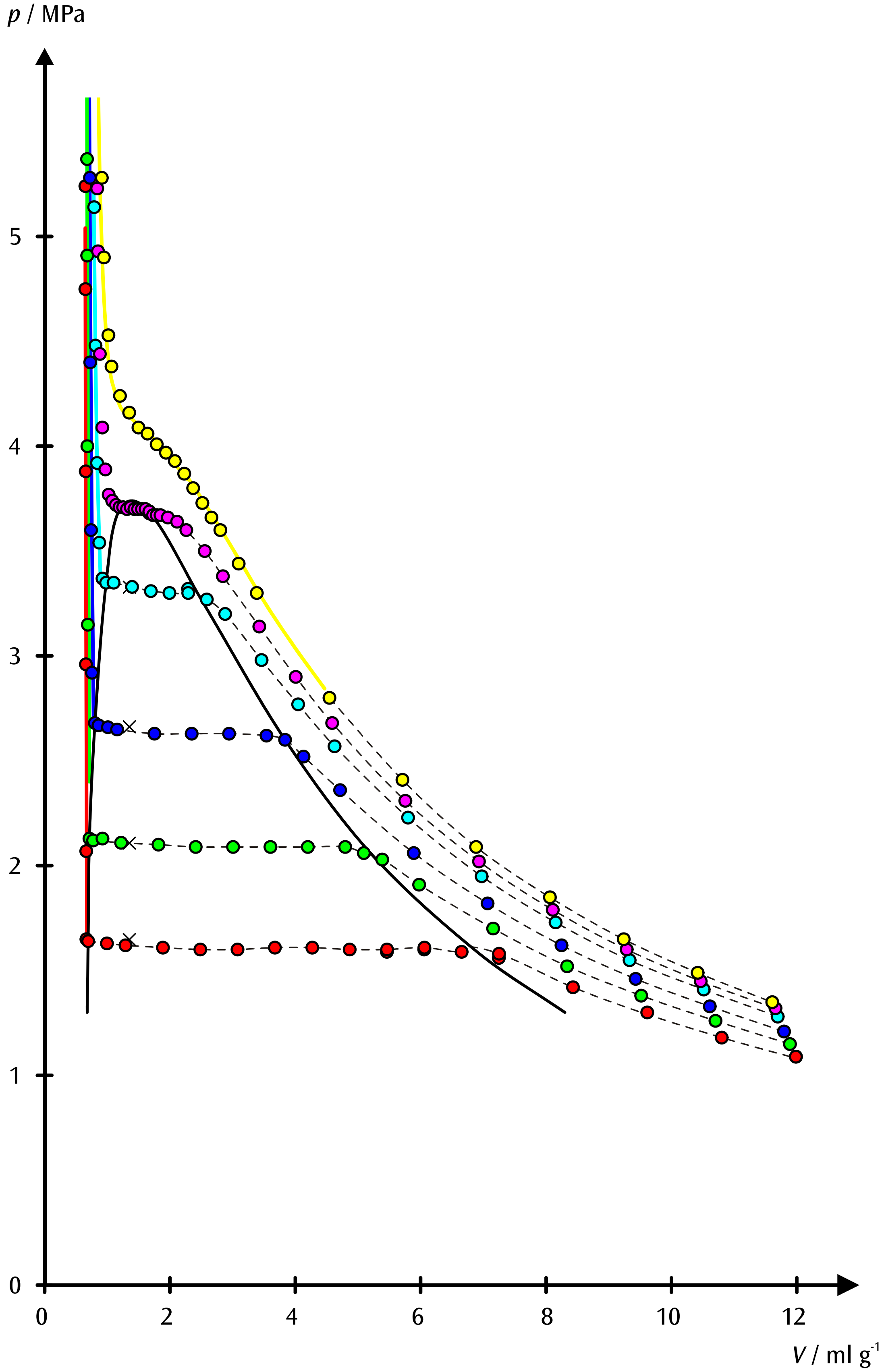

Fig. 5: *p-V* diagram of SF_6, measured with the critical point apparatus:
Readings taken at 10°C (red), 20°C (green), 30°C (blue), 40°C (cyan), 45°C (magenta) and 50°C (yellow),
(black line) threshold value of liquid-gas mixture, (×) Reference values from [1] for vapour pressure,
Reference values from [2] for pressure of liquid at 10°C (red line), 20°C (green line), 30°C (blue line), 40°C (cyan line)
and 50°C (yellow line)

10. Degassing the hydraulic fluid

Owing to the inevitable diffusion of the test gas through the conical seal, the pressure in the measuring cell slowly decreases over a long period. The gas diffusing through the conical seal first dissolves in the hydraulic fluid but does not have any significant influence on the measurements.

However, if the test gas is removed from the equipment (for storage of the equipment) and the pressure of the hydraulic fluid consequently falls to the ambient pressure, then the test gas will escape from the hydraulic fluid due to Henry's law. This leads to a gradual increase in pressure in the oil chamber which must be avoided at all costs as there is no back pressure in the measuring cell. On account of this, it is necessary to cleanse the hydraulic fluid of all gas before storing the equipment.

To degas the hydraulic fluid, the oil is made to boil in a vacuum. Since the pressure difference on both sides of the conical seal should not exceed a particular limit, it is necessary to maintain, as best as possible, the existing underpressure constant on the gas side.

Additionally required:

1 Castor oil approved for medicinal use e.g. 1002671

1 Vacuum tube, 6 mm internal diameter
1 Stopcock (or variable-leak valve)
1 Vane-type rotary pump

1 Open-end spanner (14 mm), 1 pair of tweezers
Absorbent paper, cardboard box

Storage of the equipment:

- If necessary, allow the equipment to cool. Wind the handwheel back till the lowest possible is present.
- Release the test gas through the flush valve and shut the flush valve thereafter.
- If necessary, remove the gas supply pipe and attach the gas connection fittings.
- Unscrew the vernier scale.
- Open the regulating valve.
- Wind the handwheel so that piston moves in till an excess pressure of 1 bar has been attained.
- Shut the regulating valve.
- Wind the handwheel back by two turns.
- Place the equipment with the manometer facing downwards towards the ground). The manometer should rest on a support approx. 6-cm-thick (see Fig. 6).

Caution: the piston should never be wound out to more than 25 mm, since the guide tube may slip out during subsequent operations.

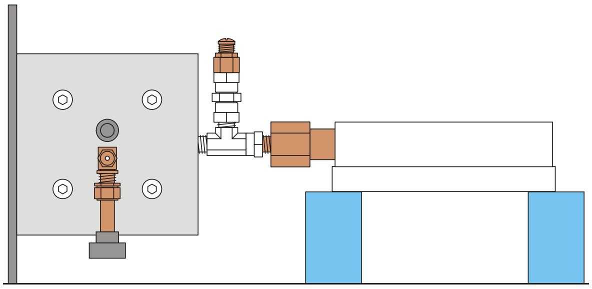

Fig. 6: Storage of the equipment for oil filling

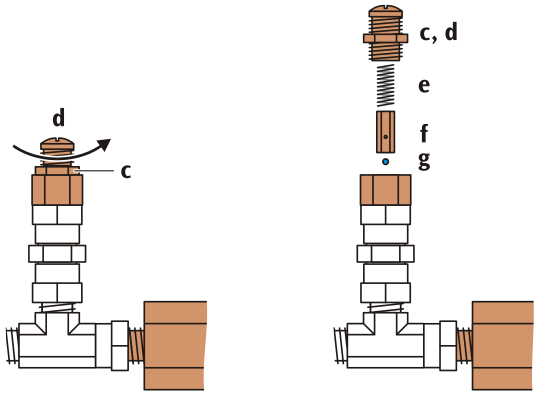

Fig. 7: Dismantling the safety valve
(c) counter nut, (d) valve cap, (e) compression spring, (f) hexagonal piston, (g) steel ball bearing

Dismantling the safety valve:

- Loosen the counter nut (14 mm) and use a screwdriver to remove the valve cap (see Fig. 7).
- Remove the compression spring, the hexagonal piston and the steel ball bearing in succession with a pair of tweezers and store them in a safe place, for instance in a cardboard box.

Assembly of the oil filling device:

- Loosen the valve nut of the oil filling device, remove the cover and place the valve nut above the safety valve (see Fig. 8).
- Do not screw the oil filling device on too tight (the gasket ring should not be squeezed out).
- Open the regulating valve.
- Wind the handwheel inwards to its end position up to the frame (if necessary, loosen the vernier scale). Subsequently wind the handwheel out by 3 turns.
- Place absorbent paper underneath and fill the oil container with castor oil to no more than half way.
- Screw on the cover of the oil filling device with the valve nut.

Connection of vacuum pump:

- Connect a plastic hose with 3 mm internal diameter to the gas connection fittings of the equipment and the smaller connector of the oil filling device.

- In order to connect the vacuum pump, take a vacuum hose with 6 mm internal diameter and connect it via a stopcock or preferably via a three-way valve to the larger connector of the oil filling device.

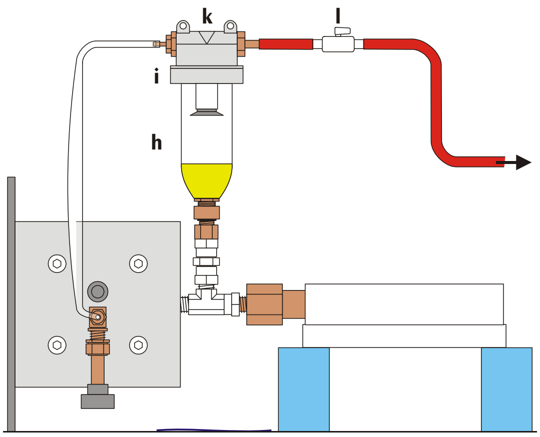

Fig. 8: Assembly of the oil filling device and connection of vacuum pump (h) oil container, (i) valve nut, (k) cover, (l) stopcock (or variable-leak valve)

Degassing:

- Check whether the regulating valve is open and the flush valve is shut.
- Switch on the vacuum pump. Open the stopcock a little and observe the formation of bubbles in the castor oil.

Close the stopcock to interrupt the evacuation process if the formation of bubbles is so strong that they can reach the filter that is mounted on the cover. The stopcock may be opened only after the bubbling has subsided.

After several minutes (depending on the suction capacity of the connected vacuum pump), the vaporising pressure of the castor oil is attained and the oil begins to boil. This can be noticed when vapour bubbles begin to form "out of the blue" and rapidly become larger in size as they move through the oil.

The oil is now is sufficiently degassed.

- Shut the regulating valve and the stopcock.

Dismantling:

- Pull out the vacuum hose from the stopcock (the hose fitting with the stopcock continues to remain on the oil filling device).
- To avoid any surges, slowly open the stopcock and wait for the pressure to even out.
- Pull out the hoses from both of the connectors on the oil filling device.
- Unscrew the container from the safety valve.

Since castor oil is relatively viscous, it trickles out of the container very slowly. Thus, this step can be conducted easily. A cleaning cloth (or kitchen paper) which is held below the container immediately after unscrewing it prevents any drops forming.

- With a cleaning cloth, remove excess oil from the safety valve and subsequently wind the handwheel inwards very slightly till the oil level in the valve is exactly at the same level as the edge where the steel ball bearing sits.
- Insert the steel ball bearing, position the hexagonal piston with the short bore onto the ball bearing (use tweezers for this) and insert the compression spring into the longer bore.
- Carefully screw the valve cap on in its end position (not too tight) and loosen it by two turns.

Positioning the safety valve:

- Set-up the equipment and place it in a way that the safety valve does not point in the direction of people who could get injured or objects which could get damaged.
- Open the regulating valve. Wind the handwheel fully out and shut the regulating valve again.
- Turn the handwheel in till an excess pressure of approx. 65 bar has been attained.
- From the front, wrap your arms around the apparatus to reach the safety valve located at the back. Slowly unscrew the valve cap of the safety valve till the pressure drops to approx. 63 bar.
- Tighten the counter nut (14 mm).

Rest position:

- Wind the handwheel back till the pressure has dropped to max. 10 bar.
- Open the regulating valve and turn the handwheel to its "rest position" at approx. 5 mm.
- Shut the regulating valve.

After completing these steps, the equipment can either be stored or refilled with test gas.

11. Upkeep and maintenance of threaded bush

11.1 Lubricating the threaded bush

To minimise wear, the threaded bush in the frame should be lubricated approximately every 100 cycles (one cycle = a pressure increase from 10 to 60 bar and the subsequent reduction to 10 bar), or once weekly. Lubrication only takes about 1 min and extends the service life of the bush significantly. For lubrication, a light-coloured multi-purpose grease with no graphite or similar additives is recommended.

Procedure:

- Inject one full stroke of lubricant from a conventional grease gun into the threaded bush through the nipple at the frame.
- Wipe up any surplus lubricant emerging from the bush.

When it emerges, the lubricant will also pick up any traces of plastic that might have worn off during operation, so that will be flushed out too.

11.2 Examine threaded bush.

The threaded bush in the frame is subject to slow but constant wear, and therefore the axial play must be checked once a year:

- Release the pressure from the measuring cell and adjust the piston to the 10 mm position.
- Using a vernier caliper, determine the minimum and maximum distance between the handwheel flange and frame; to do so, first wind in the handwheel and then wind it out.

If the two distances differ by more than 0.3 mm, then the bush needs to be replaced.

11.3 Replacing the threaded bush

Additionally required:

1 Threaded bush from set of seals (1002672)

The threaded bush is to be replaced no later than every ten years even if the limit of wear has not been reached (tests on a rig failed to produce any measurable wear [<0.05 mm] after 1000 cycles), because reliable data on the long-term stability of the plastic used (POM-C) are not yet available.

- Depressurise the measuring cell.
- Unscrew the fixed scale.
- Undo the grub screw of the handwheel flange and remove handwheel.
- Loosen the four screws in the cross piece of the frame and remove it along with the threaded bush by winding it down the axle.
- Unscrew the lubricating nipple (size SW 7) and use a 3-mm Allen key to loosen the threaded pin screwed in across the threaded bush by 4 turns.
- Knock the threaded bush out from the side of the handwheel using a suitable mandrel. Alternatively insert an M14 screw loosely into the bush and force the bush by hitting the head of the screw.
- Fit the new bush such that the cross piece is aligned with the lubrication nipple.
- Clamp the bush in a vice (with flat jaws or suitable insert).
- Screw back in the threaded pin (min. 6.0 mm countersunk) and the lubricating nipple.

Bush material: POM-C = Polyoxymethylene copolymer Oversize (press fit): 0.05 – 0.1 mm.

12. Changing the seals

Additionally required:

1 Allen key (6 mm)

1 Set of seals for critical point apparatus 1002672
consisting of
1 Conical seal,
1 Circular grommet,
1 Grommet 78x78 mm^2 ,
4 Copper gasket washers
1 Threaded bush

After a certain period of time, it may be necessary to replace the conical seal or other seals, especially if the equipment has been exposed to direct sunlight.

12.1 Dismantling the equipment:

- If necessary, allow the equipment to cool and wind the handwheel back till the lowest possible pressure is present.
- Release the test gas through the flush valve and shut the flush valve.
- If necessary, dismantle the tubing.
- Open the regulating valve.
- Wind the handwheel back till it has come to a position of 25 mm.
- Tilt the equipment to the right and place it in an upright position on a suitable surface resting on the handwheel and the edge of the equipment base.
- Use the Allen key (6 mm) to uniformly loosen each of the four screws in the valve plate by 1/8 of a turn till the tension has been reduced.
- Unscrew and remove the screws.
- Also remove the copper gasket washers.
- With increasing force, twist the valve plate to the left and right till the seals have been loosened. Do not twist the regulating valve.
- Remove the valve plate (the measuring cell might still be sticking to the plate).
- Twisting the equipment some more to loosen the remaining seals between the measuring cell and the cylinder and between the measuring cell and the valve plate.
- Twist the guide tube to remove it from the conical seal.

12.2 Cleaning the dismantled equipment:

Castor oil can be removed quite easily by using white spirit. However, white spirit attacks the acrylic of the casing and measuring cell. Use a (mild) washing-up liquid solution to remove greasy finger marks and other impurities. New seals too should be cleaned with white spirit and a washing-up liquid.

12.3 Assembling the equipment:

In case castor oil had been removed from the oil chamber:

- Pour a fresh quantity of castor oil in up to about 5 mm below the upper edge of the cylinder (at the beginning of the depression).
- Insert both of the silicone seals.
- Turn the conical seal inside out and dampen the stud with some castor oil then screw it into the guide tube.
- Unfold the conical seal back to its original shape, position the spring on the piston and insert the guide tube into the piston.
- Mount the measuring cell and position it flush along the edges of the cylinder.
- Place the heat casing at the centre of the lower silicone seal.
- Fit the circular grommet and, with the help of a ruler placed on the heat casing, position it parallel to the cylinder (see Fig. 9, the semicircular holes should then be below the valve openings).

Fig. 9: Positioning the circular grommet

- Place the valve plate at the centre and position it parallel to the end plate.
- Fit the M8×40 screws with new copper gasket washers and loosely screw them in.
- Tighten the screws. Take care to ensure that there is uniform pressure on the circular grommet (if the pressure is too high, the grommet makes a greyish mark on the transparent acrylic, whereas if the pressure is lower the surface looks milky).

12.4 Recommissioning:

- Degas the hydraulic fluid and pour the oil into the equipment (see chapter 10).
- Position the safety valve (see chapter 10).
- Conduct a fresh volume calibration (see chapter 6).