PHYWE Systeme GmbH & Co. KG
Robert-Bosch-Breite 10
D-37079 Göttingen

Telefon +49 (0) 551 604-0
Fax +49 (0) 551 604-107
E-mail info@phywe.de

Operating instructions

Fig. 1: 04364-10 Function and operating elements of the Critical point apparatus

TABLE OF CONTENTS

1 SAEFTY PRECAUTIONS

Attention!

- Carefully read these operating instructions before operating this instrument. This is necessary to avoid damage to it, as well as for user-safety.
- Do not start up this instrument in case of visible signs of damage to it.
- Only use the instrument for the purpose for which it was designed.
- Do not exceed the stated maximum operating conditions, 5 MPa or 55 °C. Working at temperatures below 0 °C is also not permissible.
- Do not expose measuring capillaries to external mechanical loads. Do not use them if they are scratched or have any other defects, as they could then burst under the high operating pressure. Avoid exposing them to thermal shock, e.g. do not fill them with hot and then cold water in direct succession.
- The water container must always be filled when the apparatus is in use. After completion of the experiment, the water must be removed from the container again; otherwise damage may occur to the capillary, among other things.
- Always ensure that the piston is at the lower stop before starting evacuation.
- Fit a Woulfe bottle between pump and system prior to evacuation.
- Set up the critical point apparatus in a room in which the temperature remains constant during the experiment.

- As the exhaust air from the pump could possibly contain mercury vapour, lead it out to the open air.
- Only open valves when the piston has been turned to the down position. Inadvertent opening of valves can be avoided by taking off the valve knob.
- After use, always turn the piston to the down position.
- We strongly recommend that you place the apparatus in a seamless mercury tray (see "List of equipment") when using it for practical work.

Attention!

To prevent damage to the device, the capillary must not be immersed into water for a longer period! Otherwise the adhesive can dissolve, eventually. This will cause the capillary to slide out of the fitting!

After the experiment, empty the transparent acrylic glass cylinder and dry the adhesive surface around the capillary with a paper cloth.

Attention!

The apparatus contains approx. 360 g of mercury.
Take care when handling mercury. Observe the pertinent safety regulations.

Signal word
DANGER

Hazard statement(s)

H360	May damage fertility or the unborn child.
H330	Fatal if inhaled.
H372	Causes damage to organs through prolonged or repeated exposure.
H410	Very toxic to aquatic life with long lasting effects.

Precautionary statement(s)

P201	Obtain special instructions before use.
P273	Avoid release to the environment.
P309 + P310	IF exposed or if you feel unwell: Call a POISON CENTER or doctor/physician.
P304 + P340	IF INHALED: Remove victim to fresh air and keep at rest in a position comfortable for breathing.
P501	Dispose of contents/ container to an approved waste disposal plant.

2 PURPOSE AND DESCRIPTION

The critical point apparatus enables the most important phenomena which occur with real gases to be treated quantitatively and demonstratively. It serves in particular for the practical determination of critical data for various gases, as well as for the measurement of vapour pressure curves, of Van der Waals coefficients and of molar heats of vaporization, and also for the determination of molar masses.

The various changes in state and different forms of representation (Clapeyron or Amagat charts) make the differentiation between ideal and real gases particularly concise. The transparent measurement arrangement allows phase transitions between liquid and gas to be visually followed. The easily exchanged gas to be examined is compressed in a high-pressure resistant glass capillary, one end of which has been closed by melting it together, by hydraulic pressure from a pressure chamber which is filled with mercury.
The basic thermodynamic quantities pressure p, temperature T and volume V can be set over a wide and physically interesting range.

3 FUNCTIONAL AND OPERATING ELEMENTS

The apparatus stands on a tripod with non-slipping feet and consists of the following components (see Fig. 1):

1 The compression and measuring capillary, made from an extremely resistant special glass. Each individual capillary is tested at 7.5 MPa and 60 °C prior to release. A scale on the capillary allows the determination of volumes. When in use, the lower meniscus of the mercury column shows the position to be read for the determination.

2 The transparent acrylic glass cylinder serves as jacket for connection to a circulating thermostat for holding constant temperature. It can be tightly closed with the lid with O-ring supplied with it. An opening in the lid allows a thermometer to be inserted for monitoring the temperature. To avoid the lid from being burst off by increased pressure, it must be additionally secured by fixing it to the upper hose nipple with string. When doing this, ensure that the lid can be lifted up just enough to relieve excess pressure, but not so loose that it could still spring out sufficiently to break the thermometer.

Warning! When pressure equalization with the surroundings cannot occur, the apparatus could be subject to damage!

3 The cylinder has two hose nipples for connection to the circulating bath. The thermostated water must flow in through the lower hose nipple. For connection, only use hoses which can be tightly clamped on with the tubing clamps supplied. Adjust the pinchcock so that the inflow of water is such, that the water pressure is completely relieved via the upper hose nipple connection. Should too much water flow in, then the lid could burst off and the cylinder overflow.

4 The stainless steel pressure chamber, filled with mercury. This is sealed to the measuring capillary to form a pressure tight system.

5 The handwheel operates a spindle drive for movement of the piston in the pressure chamber. A clockwise turn of the handwheel causes a piston stroke, and a corresponding reduction in the volume (max. 16 turns for the complete piston stroke).

6 The manometer, which displays the gas pressure in the capillary.

7 Inlet valve

8 Connecting thread, for fitting on a compressed gas container.

9 Outlet valve, with hose barb for connection to a vacuum pump.

Fig. 2: Experimental set-up Thermal equation of state and critical point P2320400

4 HANDLING

4.1 Preparing for use

For reasons of transport safety, the measuring capillary is not mounted in the apparatus as supplied. The pressure chamber is filled with mercury, but is closed with a rubber stopper and secured by a metal plate which is screwed in position. First check that the pressure piston is in the fully down position, then undo the screws and remove the metal plate. Before mounting the measuring capillary, check that the pressure sealing parts are free of dust and grease. Clean them with an alcohol-wetted cloth if necessary. To mount the measuring capillary, place the accompanying O-ring in the groove of the metal flange, then position the capillary over the opening of the pressure chamber so that the scale can be read from the front direction and fix it on firmly with the three screws. Now put the acrylic glass cylinder on, easing its attachment over the flange of the capillary, whereby the fitting should be lightly greased.

4.2 Leak test

The air confined in the measuring space is used for this test. Check that the inlet and outlet gas valves are closed. Close the lower hose nipple of the acrylic glass cylinder (fit on a short piece of tubing closed with a tubing clamp) and fill the cylinder with water. Turn the handwheel clockwise to cause the piston to move upwards and so slowly increase the pressure in the capillary up to the maximum pressure of 5 MPa. After temperature equalization, the pressure should remain constant for 1−2 minutes. On completion of the test—as after every experiment—slowly return the piston to the down position.

Attention!

Do not continue to turn the handwheel after the clearly perceptible lower stop for the piston has been reached.

4.3 Filling with gas

The following points must be carefully followed before a new gas is filled in:

- Carefully turn the handwheel anti-clockwise down to the lower stop, so that the mercury surface is with certainty below the inner opening of the valves.
- Meticulously remove all residues of gas from the pressure chamber. To achieve this, evacuate the chamber for at least 30 minutes, so that even any gas which has dissolved in the mercury is removed. To be certain that no mercury can pass into the pump, insert a Woulfe bottle in front of the pump, holding it firmly in position with a stand and universal clamp (see Fig. 2).
- When screwing on a compressed gas container, be sure not to cant it, as this could result in damage to the inner container valve which is made of plastic. When a container is screwed on, the inner valve is put out of operation until the container is screwed off, when it again takes over the closing of the container. The screwing on and screwing off of containers should therefore be carried out rapidly to avoid unnecessary outflow of gas.

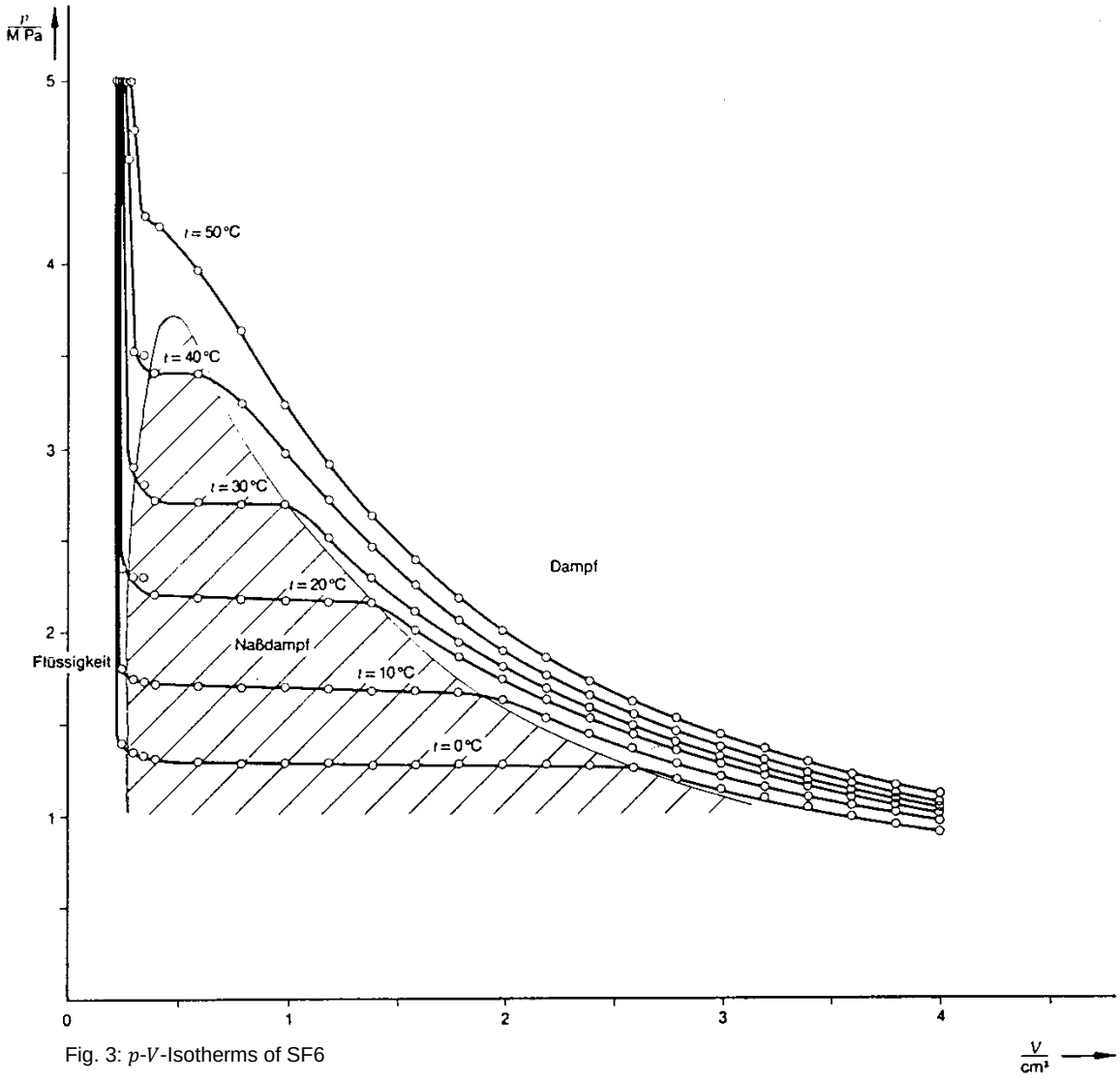

Fig. 3: p-V-Isotherms of SF6

- When connecting the gas container, it is necessary to prevent any potentially disturbing remainder of air from being introduced into the chamber. After having connected the gas container, open the inlet valve a little to rinse out any remaining air. Close the inlet valve and again evacuate the pressure chamber. After pumping for about 2 minutes, close the outlet valve and open the inlet valve to allow gas to flow into the chamber. Close the inlet valve. Remove the gas container and vacuum pump. The apparatus is now ready for use.

4.4 Use of a circulating thermostat

A circulating thermostat can be connected so that quantitative measurements can be made at various temperatures. The lower hose nipple then serves as water inlet, the return flow to the thermostat is via the upper hose nipple. Ensure that the cylinder lid is secured in position as described under 3.2, and that there is no hindrance to the flow back to the thermostat. In particular, check that the return hose connected to the upper hose nipple is not sharply bent.

First use the pinchcock to completely close off the water inflow, then switch the circulating pump on. Open the pinchcock until the level of the water in the cylinder rises about 1–2 cm per second. This flow rate is sufficient for the target temperature to be attained, and also ensures that not too much pressure is exerted on the acrylic glass cylinder.

Attention!

Too high a water pressure could press the lid off and cause flooding!

5 EXPERIMENTS

The following gases are suitable for practical work:

- Sulphur hexaflouride: p_c = 3.77 MPa; t_c = 45.54 °C
- Ethane: p_c = 4.88 MPa; t_c = 32.3 °C
- Carbon dioxide: p_c = 7.38 MPa; t_c = 31.0 °C *

(p_c = critical pressure; t_c = critical temperature; the critical pressure of carbon dioxide is far above the maximum permissible pressure for this apparatus).

Fig. 2 shows a complete experimental set-up. For qualitative experiments, it is sufficient to fill the water container with hot or cold water.

For quantitative experiments, a circulating thermostat must be used. After a change in temperature, wait a few minutes before reading the temperature, to allow temperature equalization between the water bath and the gas to take place. Turn the handwheel to compress the gas until the mercury

column is visible in the measuring capillary. With suitable gases, and on increasing compression, an increasing liquefaction of the gas can be seen above the mercury meniscus. The corresponding pressure values can be determined more accurately by lightly tapping the manometer to reduce frictional effects.

Fig. 3 shows an example of an experimentally determined group of isotherms (Clapeyron chart) for sulphur hexafluoride (SF6).

_____

*CRC Handbook of Chemistry and Physics 76th edition
David R. Lide (Editor)
1995 Boca Raton New York
S. **6**-54 ff

6 TECHNICAL DATA

(typical values for 25 °C)
Operating temperature range: 5–40°C
Rel. humidity < 80 %

Operating pressure	0… max. 5 MPa
Operating temperature	0... max. 55 °C
Readable volume range	0... 4 ml
Volume scale divisions	0.05 ml
Manometer scale divisions	0.1 Mpa
Connecting thread for compressed gas	R 1/8”
Mercury filling	approx. 360 g
Mass of the apparatus	10.5 kg

7 LITERATURE

TESS expert Handbook Laboratory Experiments Physics 13502-32

8 LIST OF EQUIPMENT

Excerpt from the equipment list:

Critical point apparatus	04364-10
Immersion thermostat	08493-93
Bath for thermostat, 6 l	08487-02
Rotary valve vacuum pump, one stage	02740-95
Mercury tray	02085-00
Compressed gas, sulphur hexafluoride, 74 g	41772-21

9 WARRANTY

We guarantee the instrument supplied by us for a period of 24 months within the EU, or for 12 months outside of the EU. Excepted from the guarantee are damages that result from disregarding the Operating Instructions, from improper handling of the instrument or from natural wear.
The manufacturer can only be held responsible for the function and technical safety characteristics of the instrument, when maintenance, repairs and alterations to the instrument are only carried out by the manufacturer or by personnel who have been explicitly authorized by him to do so.

10 WASTE DISPOSAL

The packaging consists predominately of environmentally compatible materials that can be passed on for disposal by the local recycling service.

Should you no longer require this product, do not dispose of it with the household refuse.
Please return it to the address below for proper waste disposal.

PHYWE Systeme GmbH & Co. KG
Customer Service
Robert-Bosch-Breite 10
D–37079 Göttingen
Germany

Phone +49 (0) 551 604-274
Fax +49 (0) 551 604-246