

ROTH high-pressure laboratory autoclave, Model II 200 ml, 250 ml, 300 ml working volume

2098.1ff. / 2094.1ff. / 2108.1ff.

Operator's manual

Please read carefully before use!

1. Description

Model II laboratory autoclaves are made of V4A stainless steel and can be used in experimental laboratories for all reactions where gas formation and a rapid increase in pressure can be expected. The 200 ml and 300 ml cylinders are designed for max. 100 bar working pressure, the 250 ml cylinder for max. 200 bar working pressure and max. 300 °C. The autoclave consists of a cylinder and a screw-on head. The top of the head enables attachment of the bursting disc holder and at the side there are three G1/4" threaded openings allowing following accessories to be fitted: valve, reducer screw joint for the manometer, screw-threaded stopper type A for plugging a possible opening, temperature sensor or bursting disc locking device II when using magnetic stirrer head MRK 10. The model II series is extremely versatile. It is possible to measure the inner temperature of the autoclave cylinder with a temperature sensor. Gas is distributed via the valve. Gaseous or liquid samples are fed or withdrawn through an adapter with immersion rod.

The autoclaves are sealed with flat gaskets made of PTFE (for max. +180 °C working temperature) or fine silver (for max. +300 °C working temperature).

2. Unpacking

Please unpack the unit carefully and check for any possible damage. It is essential that any signs of transport damage are recognized upon unpacking or, if applicable, the details of any damage are recorded by the carrier (post, forwarding agent or railway).

Please make sure that the threads are not damaged or soiled.



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3. Assembly and Operation

3.1 Assembly Instructions

Attention: Use **PTFE-spray** (Art. No. K007.1) before screwing.
Do not use PTFE-thread tape!

The autoclave head should be screwed together first in the following order.

Attention: When using the adapter with immersion rod, mount the adapter before completing steps a-e (see 3.1f).
When using the temperature sensor, mount the sensor before completing steps a-c (see 3.1 e).
When using the magnetic stirrer head, mount the stirrer head before completing steps a-c (see 3.1g)
In this case a temperature sensor cannot be fitted.

- a. **Bursting disc**
First, place the fine silver gasket, No 40 in the opening, then place the bursting disc with the camber facing outwards. Take care that it is lying in the middle of the opening. Then screw in the locking screw and tighten it as firmly as possible with screw wrenches No 50 and No 55 (for the head). Please make sure that the bursting disc does not slip.
- b. **Manometer**
Second, place gasket No 12 in one of the three side openings, screw the locating and reducing adapters (with appropriate gaskets, No 12) for the manometer and tighten slightly with screw-wrench No 19 and No 27. Then place gasket No 18/20 in the reducing adapter, screw in the manometer and tighten it slightly with screw-wrench No 22.
- c. **Valve**
Third, place gasket No 12 in one of the side openings, screw in the threaded adapter and the valve. After the SWAGELOK screw connection has been fitted, tighten with screw-wrench No 14.

Assembly instructions for SWAGELOK screw connection

1. Insert the tube section (6mm) of the thread adapter completely into the SWAGELOK screwed rod fitting.
 2. Tighten the screw fitting hand-tight.
 3. Final positioning is obtained by turning the screw nut $1\frac{1}{4}$ times clockwise.
- d. **Screw-threaded stopper A**, for plugging the third side opening.
Fourth place gasket No 12 in the third side opening, screw in the stopper and tighten it slightly with screw-wrench No 19.



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e. Temperature sensor

Caution: Loosen the locking screw on the sensor before inserting the temperature sensor!

If the sensor is to be used, screw-threaded stopper A is not required for plugging the third opening. First place fine silver gasket No 12 in the third opening and then push the sensor through the side opening. By gently bending the sensor to the bottom, it is possible to push in the whole sensor. Do not bend too hard as the integral FeCuNi-thermocouple may otherwise break. Screw in the sensor and tighten it with screw-wrench No 17. Because of the relatively hard silver gasket the sensor must be screwed tightly to be fully sealed. Finally the side locking screw on the sensor should be tightened again.

f. Adapter with immersion rod

Before the autoclave head can be assembled as described in a to e, the adapter with immersion rod must be fitted into the head as follows: first place gasket No 40/42 in the top head opening, then screw the adapter and tighten it with screw-wrench No 55. Then proceed as stated above in a to e. Finally, place gasket No 12 in the side connecting opening of the adapter, the second valve (see 3.1c) is screwed in/attached and tightened with screw-wrench No 14.

g. Magnetic stirrer head MRK 10

Before the autoclave head can be assembled, the magnetic stirrer must be fitted into the head as follows: first place gasket No 40/42 in the top head opening, then screw in the adapter for the magnetic stirrer head (adapter piece for stirrers) and screw tightly with screw-wrench No 50. Before installing the stirrer head, the gassing stirrer must be screwed onto the stirrer head with the provided Allen key. Now the magnetic stirrer can be screwed in together with the available gasket and tightened with screw-wrench No 29.

h. Bursting disc locking device II

As the top opening is no longer available, because of the stirrer head, for mounting the large bursting disc, a bursting disc locking device must be mounted on the side of the head as follows: first place fine silver gasket No 15 in the opening of bursting disc locking device II, then bursting disc 8.100 with the camber facing outwards. Take care that it is lying in the middle of the opening. Then screw in the locking screw and tighten it as firmly as possible with screw-wrenches No 19 and No 27. Please make sure that the bursting disc does not slip.

Place gasket No 12 in one of the three side openings and screw in bursting disc locking device II, then tighten it with screw-wrench No 27. Finally screw the bursting disc locking device safety cage over the bursting disc holder.

Now screw the manometer and the valve as described in b and c above tightly to the autoclave head.

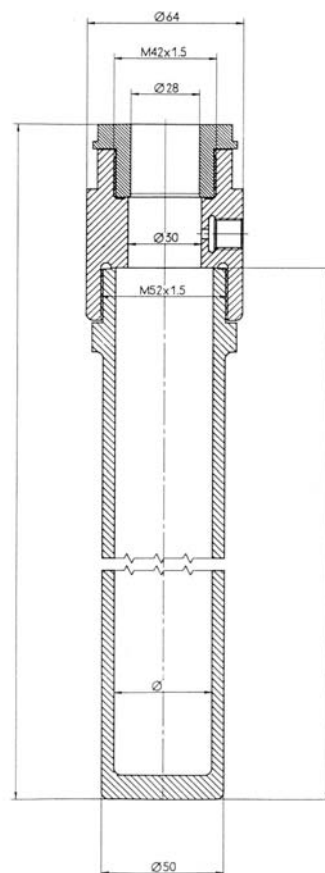
Screw-threaded stopper A is not required when installing the magnetic stirrer head as bursting disc locking device II is screwed in the third opening of the head.



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Finally place the large gasket No 50/51 into the autoclave lid and then screw the head with the assembly to the autoclave cylinder and tighten with screw-wrench No 55.

The diagram shows the assembly of the autoclave with the correct position of the gaskets and bursting disc. The three G1/4" side openings on the head are for connecting the valve, the reducing screw joint for the manometer, screw-threaded stopper or the temperature sensor or for connecting the bursting disc locking device II when using magnetic stirrer head MRK 10.



3.2 Screw joints

a. Bursting disc

A fine silver gasket should always be placed under the bursting disc for safety reasons. Because the silver gasket is relatively hard the locking screw for the bursting disc must be screwed tightly to prevent the bursting disc from being pressed out when under pressure. The bursting disc is naturally very sensitive and can no longer be used even after being only slightly damaged. Please make sure during assembly that the camber points are positioned in the direction of the pressure.

Attention: The bursting disc must be replaced after applying full mechanical strain.

b. Reducing screw joint

For manometer, valve, screw-threaded stopper, temperature sensor, adapter with immersion rod, adapter for magnetic stirrer head, bursting disc locking device II, autoclave cylinder and head.

Attention: Use **PTFE-spray** (Art. No. K007.1) before screwing.

When attaching the screw-on parts, care should be taken that the thread is not screwed on slantwise, which can cause the threads to seize (cold-welding). The parts should be screwed in and using PTFE-gaskets slightly tightened with the appropriate screw-wrench. We do not recommend screwing too tightly when using PTFE-gaskets at the beginning, otherwise deep indentations from the pressure will form and the gaskets can no longer be used after a short time. However, when using fine silver gaskets the parts must, because of the relatively hard fine silver, be screwed as tightly as possible to obtain a perfect seal.

Damaged gasket rims and threads may no be used. They should also always be cleaned carefully when dirty.

c. Gas distribution

Gas is distributed in the autoclaves directly via the valve/adaptor. The PTFE-high-pressure tube is connected to the valve on one side and to the gas bottle through the reducing screw joint on the other.



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3.3 Sealing

The autoclave and all screw-in accessories such as bursting disc, manometer, valve, temperature sensor, screw-threaded stopper, adapter with immersion rod, adapter for magnetic stirrer head, bursting disc locking device II, autoclave cylinder and head are sealed with flat gaskets made of PTFE (max. +180 °C working temperature) or of fine silver (max. 300 °C working temperature).

For safety reasons the bursting disc must be sealed with a fine silver gasket.

The threads are subjected to high mechanical stress, therefore to protect and lubricate (stainless steel on stainless steel is hard running) the individual parts when screwing, PTFE-spray must be used. The **PTFE-spray** (Art. No. K007.1) can be applied to max. +260 °C.

When using the relatively soft PTFE-flat gaskets, it is sufficient to slightly tighten the screw-on parts with the appropriate screw-wrench. However, when using fine silver gaskets, it is necessary to screw the parts as tightly as possible because of the relatively hard fine silver to obtain a perfect seal.

Damaged gasket rims and threads may not be used. They should also always be cleaned carefully when dirty.

If you are unable to seal the laboratory autoclave, please localize the untight spot, then check the position of the gasket and its condition. If necessary, retighten with a screw-wrench.

Advice when working with silver gaskets

When working with silver gaskets it is essential to tighten the gasket very firmly in order to achieve the required tightness. It is recommended to anneal the silver gasket beforehand at approx. +850 °C. By doing this, the gasket will become softer even when cold, and will seal better.

3.4 Safety

The high-pressure laboratory autoclave is manufactured in accordance with the safety regulations for pressure chambers. It is tested and coded accordingly. Laboratory autoclaves 200 ml and 300 ml undergo compression tests at 300 bar and the 250 ml autoclave at 400 bar at 20 °C. In accordance with engineering standards, the autoclaves are to be operated with a bursting protection, a valve and a pressure measuring device. The named safety devices are to be mounted directly into the autoclave head (bursting disc 28.100/28.200) or into one of the G1/4" side connections. The autoclave is heated with the heating mantle 20 S or 30 S. To obtain optimal heat distribution and utilization, the heating mantles have been adapted exactly to the dimensions of the autoclave.

The laboratory autoclave, because of its construction, the rigid test conditions and the large bursting disc inside the autoclave head, offers a great degree of safety. Nevertheless, working at high pressure remains dangerous. The laboratory autoclave must, therefore, be set up in such a way that no-one can be injured through decomposition or overpressure caused by overheating or due to unexpected reactions. We also advise setting up the operating elements of electrical attachments in such a way that they can be always switched off.



3.5 Operation

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High-pressure laboratory autoclaves are to be operated in accordance with the safety regulations for pressure chambers. Our laboratory autoclaves are experimental autoclaves in accordance with Annex 5, § 17 Section 19 (1) of the German Health and Safety at Work Regulations (BetrSichV).

Experimental autoclaves must be checked by a specialist after each application.

Experimental autoclaves must be set up in special chambers or behind protective walls designed in such a way that the autoclaves are protected against outside influences and that all employees or third parties are protected. Safety and measuring equipment must be controlled and operated from a safe place. Pressure and temperature must be continuously controlled and recorded when operating experimental autoclaves in accordance with safety requirements.

For safety reasons, the autoclave should be subjected to a compression test regularly depending on the mechanical strain.

4. Heating and Stirring

High-pressure laboratory autoclaves are heated with a heating mantle specially constructed for the autoclave. The heating mantle has been adapted according to the dimensions of the autoclave to obtain optimal heat distribution and utilization. The heating mantle has a temperature control of max. +300 °C.

This model allows, via a temperature sensor, the inner temperature in the autoclave cylinder in combination with thermostat WRX 2000 to be displayed, recorded and controlled on an LCD-display.

Stirring in the autoclave cylinder is performed by a magnetic stirrer built-in into the heating mantle. This stirrer will activate a PTFE-coated stirring magnet inserted in the autoclave via a rotary magnetic field and will produce infinitely adjustable stirring without direct drive in the closed autoclave.

A separate stirrer head with permanent magnetic clutch and gassing stirrer is available for special tasks. Suitable for 60.000 cst viscosities.

Notice:

Carl Roth GmbH + Co KG cannot accept responsibility for any accidents, damage or breakdown that result from using heating sources other than the designated autoclave heating mantles (Art. No. 2088.1, 0042.1, 2092.1 and 2158.1).

5. Maintenance and Cleaning

High-pressure laboratory autoclaves must be checked by a specialist after each application. Autoclave cylinder and head should be unscrewed and taken apart for storing as the material in combination with the thread can lead to cold-welding of the parts. V4A-steel laboratory autoclaves can, depending on the soiling and substances used, be cleaned with a suitable solvent or with Roth RBS-Neutral special cleansing agent (Art. No. 0180.1). The RBS-Neutral concentrate has an excellent cleaning effect and should be added to water at a concentration of 4-6%.



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6. Technical Specifications

	200 ml cylinder	300 ml cylinder	250 ml cylinder
Inner volume:	265 ml	350 ml	320 ml
Working volume:	200 ml	300 ml	250 ml
Height:	220 ml	300 ml	300 ml
Weight:	1400 g	1900 g	2250 g
Material: V4A-stainless steel	1.4571	1.4571	1.4571
Working temperature:	+ 300 °C	+ 300 °C	+ 300 °C
Working pressure:	max. 100 bar	max. 100 bar	max. 200 bar
Test pressure:	300 bar	300 bar	400 bar
Outer diameter of cylinder:	50 mm	50 mm	50 mm
Inner diameter of cylinder:	40 mm	40 mm	37 mm
Outer diameter of heat:	64 mm	64 mm	64 mm
Base form: outside and inside flat	flat	flat	flat

Technical Specifications, Autoclave Head II

Weight:	ca. 1250 g
Material: V4A-stainless steel	1.4571
Working temperature:	+ 300 °C
Working pressure:	max. 200 bar
Outer diameter:	64 mm
Connection openings on head:	Upper head opening for bursting disc 28.100 / 28.200 or adapter with immersion pipe or adapter for magnetic stirrer head MRK 10. Three openings on the side with G ¼ " thread.

7. Warranty

The high-pressure laboratory autoclave has been manufactured with extreme care and has been subjected to a pressure test.

The guarantee period covers 6 months according to the terms below:

We shall eliminate all defects arising from material and/or manufacturing errors within the guarantee period either by repairing or exchanging the parts or by replacing the unit. Replaced parts or units will fall under our ownership. The warranty does not cover damages which have occurred as a result of incorrect handling and use. Faults which only have an insignificant effect on the value of the unit or its efficiency during use are also not covered.

The warranty expires if the machine has been tampered with by non-authorized persons, or if other than original spare parts and accessories have been used.

The warranty period is not affected by the performance of any guarantee services. The warranty for replaced parts ends with the expiration date of the guarantee period for this laboratory autoclave. Should you have a warranty claim, please send us the laboratory autoclave or the faulty part with an exact description of the complaint together with the invoice number and date. We will not accept liability for any other defects or claims for damages unless liability on our part is mandatory.



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