High-Pressure Autoclaves for laboratory and research





The Company

The ESTANIT GmbH, based 1956, is busy with the production and selling of high-grade steel castings, greycast iron, stealing and metal casting, forgings, transmissions and gear wheels as well as all kinds of industrial products.

ESTANIT Company owned the complete and long based technical knowledge and experience of ERNST HAAGE Company. Starting in June 2003, ESTANIT Company overtook the complete Paper Test Equipment and in February 2005 the High

Pressure Eqipment with the range of Valves and Fittings, as well as Autoclaves and Pilot Plants.

In the area of analysis technique ESTANIT expand the programm by distillation analyzers.

During the years and up to now the company name and the products became a well-known name in the national and international industries. The products have been sold successfully in some hundreds worldwide.

... the results of more than 60 years experience in developing High Pressure Equipment!



HAAGE Autoclave, 1953



HAAGE Autoclave, 2013

TÜV NORD CERT GMBH TÜV NORD CERT GMBH Solvallige Zertifizento

Quality – ISO 9001

The ISO quality control program is recognized world-wide as a very high standard of excellence.

ESTANIT achieved this prestigious award several years ago, and continues efforts to assure that each product leaving our factory is thoroughly tested and certified to be faultless.

Each component of a product as well as the final product itself runs through various quality controls.

All of our suppliers are continuously monitored by our quality control system for their reliablity and for the right quality of their products.

HAAGE high-pressure autoclaves

are versatile laboratory autoclaves, useful in a wide range:

Many applications

- heterogeneous and homogeneous catalytic reactions
- gas-/ liquid reactions, i.e. carbonylations, ethoxydations, halogenatios, hydrogenations, hydro-cracking, oxydations, polymerisations
- liquid-/liquid reactions
- exothermic and endothermic reactions
- components testing under high-temperature and high-pressure, i.e. stability tests and corrosion tests
- development of fine chemicals
- extraction-process development
- processes with super-critical CO², other gases or super-critical water
- catalyst evaluation and development

Many accessories

A wide range of accessories is available, i.e. stirrer systems, internal cooling coils, valves, measuring and controlling systems

Batch-wise and continuous-flow operation

For continuous-flow operation of the mostly batch-wise operated autoclaves several accessories are available such as: high-pressure feed pumps and gas compressors, storage and receiving vessels, heat exchangers, i.e. gas and liquid pre-heaters and pre-coolers, gas-/liquid separators, gas- and liquid-sampling during reaction by reflux condensors and pressure coolers, mass flow measurement of the actual and total gas consumption, high-pressure controllers for the gas-feeding to realize pressure-constant operation

Standard design and custom-made design

This brochure describes and shows pictures of our product line: "High-pressure autoclaves for laboratory and research".

These autoclaves and their basic accessories are products with standardized desgin and manufacturing within short delivery periods.

Apart from this standard program, for decades we have been designing and manufacturing "Custom-made high-pressure autoclaves" in other sizes, other operational parameters, with special accessories and made of special materials.

To clarify your needs and requirements please use our detailed "Technical Questionnaire" - available with ESTANIT or with our agents.



Volume	ins. dia.	ins.depth	max. perm. pressure in bar up to 350°C				350 bar	500 bar	1000 bar
litres	mm	mm	50	100	200	350	500°C	500°C	500°C
0,15	40	130							Δ
0,3	55	170							Δ
0,6	60	230							Δ
I	80	240							Δ
2	100	285							Δ
5	125	440							Δ
10	175	470							_
20	230	560					♦■		_
50	300	800			\Diamond	♦■	♦■		_
100	395	940			\Diamond	♦■	♦■		_

- made in welded design
- made of forged material
- with massive line
- △ made of forged high quality warm fimaterial

Interchangeable vessels

The same autoclave cover with stirrer and mounting frame may be combined with interchangeable autoclave-vessels of lower volumes on request.

Internal parts like stirrer shaft, thermometer tubes, dip tube, cooling coil and the electrical heater need to be adapted.

Slim vessels for better heating and stirring

The autoclave vessels are generally designed in a slim shape (= ratio of int. diameter : int. depth) to achieve the following advantages compared with vessels with nearly square shape:

- more outside vessel surface available for entering more heating energy
- better balanced temperature profile
- better stirring efficiency
- tests using smaller quantities of liquid products even in bigger sized vessels can be realized

Material

All inner wetted parts are constructed out of high quality stainless steel, German material no. 1.4571 equivalent to SS 316 Ti.

Other construction materials are available on request, such as: Hastelloy B or C, Inconel 600/625, Incoloy, Tantalum, Titanium

Heating

Electrical Heating

All autoclaves are equipped with a powerful electrical heating system with a programmable PID-controller with ramp control and cascade-control using 2 thermocouples for measurement of the inside- and the wall- temperature of the autoclave for a more precise and faster heating control, minimizing temperature overshoots.

Heating/Cooling Jacket

As an alternative, an optional heating/ cooling jacket installed on the autoclave vessel for connection to an oil-heating system with heat-exchanger for heating and cooling of the autoclave is available.

Measuring and Controlling

Three packages are available from the basic version with an electrical control-box and many additional options, up to a PC based system with a sophisticated process-control-software, complete with visualization and data collection.



Mounting Frames



Three different frames are available depending on the volume of the autoclave and customers' requirement.





Mounting Frames

Autoclave Lift

This is the most comfortable alternative of our mounting frames for the high-pressure autoclaves.

Autoclave lifts are available for manual operation (handwheel or hydraulic pump) or equipped with an electrical pump or drive.

The autoclave lift is made of aluminium frames.

The cover of the autoclave with the stirrer system is fixed on top of the autoclave lift together with:

- all tubes for liquid and gas supply
- electrical device connection cables

The great advantage of an autoclave lift, compared with traditional mounting frames, is that, in case of opening the autoclave all tube connections and electrical cable connections to devices on the cover of the autoclave can remain fixed.

It is not necessary to dismount and remove these connections for opening the autoclave.

Thus manual handling of heavy and hot autoclave vessels is no longer necessary. On demand the autoclave vessel can manually torn out in the down position for easy emptying and cleaning.

Traditional activities before opening the autoclave like dismounting incoming and outgoing tubes and wires as well as the lifting and transport of the autoclave cover including the stirrer and valves by means of crane, are no longer necessary.

Thus it is possible to achieve high convenience, easy handling and save time with regard to opening, charging / discharging, closing and internal inspection of the autoclave.









Stirrer Systems



Magnetic Stirrers

For all autoclaves with volumes from 0,15 ltrs. up to 1.000 ltrs. glandless magnetically coupled rotary stirrer systems are available. Their operation parameters are calculated in accordance with the parameters of the corresponding autoclave, maximum up to 1.000 bar, 650°C, 3.000 rpm.

Stirrer elements and stirrer shafts are both exchangeable.

Autoclaves without stirrer system may be upgraded lateron. This is easy to realize, because each autoclave cover out of the standard program is supplied with a central-threaded hole for connection of the stirrer. This hole is closed with a blind plug, should the stirrer system not be included within the initial order.

High-Torque Stirrers

For high-viscosity applications high-torque stirrers with a direct-driven stirrer shaft with a self-sealing system are available as an alternative to the stirrer systems with magnetic coupling.

The maximum range of operation is: 200 bar, 500°C, 200 rpm

Control Features

Digital speed indication (standard), digital torque indication (option) and remoteadjustment of the stirring speed from the control box are available.

Magnetic Stirrers

The autoclaves can be equipped with glandless high-pressure rotary stirrers with integrated magnetic couplings. They come in 3 different standard sizes for the transfer of a maximum torque of 60/120 or 180 Ncm. Bigger stirrers with magnet couplings for up to 8.000 Ncm are available on request.

Stirrer Elements and Catalyst Baskets

Several stirrer elements for various applications are available (see table). They are screwed onto the stirrer shaft and therefore exchangeable.

Catalyst Baskets for holding supported catalysts can be screwed onto the stirrer shaft instead of a stirrer element.

Stirrer drive

The drive of the stirrer is adjustable between 0 and 3.000 rpm. The stirring speed of the DC motors is remote-adjusted from the electrical control box.

Speed Indication

Display of the stirrer shaft speed is achieved by a digital instrument mounted on the electrical control box.

Stirrer shaft speed is measured by an inductive sensor which takes its measurement by an indication magnet mounted on the stirrer shaft.

Inertisation Gas

Stirrer systems can be equipped, as an option, with a valve for the connection to an inert gas supply to enable cleaning or inertisation of the stirrer and the autoclave with gas.

Construction

The high-pressure stirrers with magnetic coupling consists of two systems: the Inner and the Outer one.

The Inner System

is mounted inside a pressure-tight housing screwed into the cover of the autoclave, tightened by a static sealing. Within this housing, the stirrer shaft is supported by ball bearings, easy to exchange. The inner magnetic system of the stirrer's magnetic coupling is fixed to the top of the stirrer shaft.

The Outer System

is enclosed into an aluminium housing. It includes the outer magnetic system of the magnetic coupling, mechanically connected with the stirrer motor for driving the inner magnetic

HighTorque Stirrers

For

high-viscosity applications, stirrer systems are available with

direct driven stirrer shaft with self-sealing system for sealing against the operation pressure of the autoclave.

The maximum range of operation for the High-Torque Stirrers is: 200 bar, 500°C, 200 rpm

Stirrer Elements





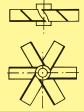
Mixing Gyro

stirring function: suspending, dispersing liquid/liquid, solid/liquid, gasing viscosity range: 20 Pa s stream range: 3,0 primary stream direction: radial-tangential



Anchor Stirrer

stirring function: heat exchange, mixing viscosity range: 2–10 Pa s stream range:passing-turbulent performance characteristics Ne: 0,2–2,0 primary stream direction: tangential



Turbine Stirrer

stirring function: homogenizing, dispersing liquid/liquid, solid/liquid, suspending viscosity range: 10 Pa s stream range: turbulent performance characteristics Ne: 1,5 primary stream direction: axial, radial



Propeller Stirrer

stirring function: homogenizing, dispersing liquid/liquid, solid/liquid, gasing viscosity range: 10 Pa s stream range: turbulent performance characteristics Ne: 0,35–0,85 primary stream direction: axial-radial

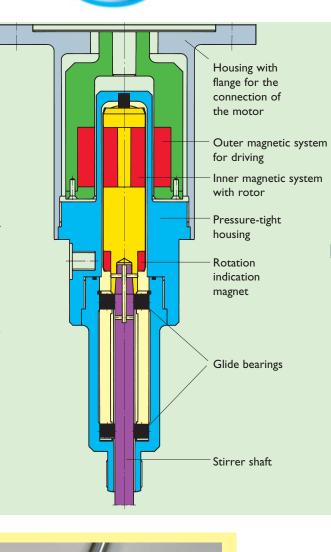
Stirrer Systems

system. The power of the motor drive is transferred contactless by the outer magnetic system to the inner magnetic system.

This principle of a glandless construction does not need to seal any rotating parts against the pressure inside the autoclave by dynamic sealings. Therefore, magnetic stirrers can be used even at high operation pressure (up to 1.000 bar) and high stirring speed (up to 3.000 rpm).

The size (maximum transferrable torque) of this magnetic coupling is chosen depending on the volume of the autoclave and the viscosity of the medium to be stirred.

Max. operation pressure and the material of construction for all wetted parts of the stirrer including the stirrer shaft and the stirrer element are chosen in accordance with the operation pressure and the material of the autoclave.









Standard Accessories

Each autoclave is equipped with the following standard accessories:

- Gas inlet valve with dip tube
- Gas outlet valve
- Pressure gauge
- Thermocouple tube for the measurement of inner temperature
- Connection for a second thermocouple for measurement of the autoclave wall temperature
- Rupture disc device with spare discs
- Central connection hole with thread for a stirrer system

Optional Accessories

Autoclave Vessel

- Interchangeable vessels in different sizes (volume) for use with the same autoclave cover, stirrer and Measuring & Controlling System
- Inside polish of the autoclave vessel
- Digital display of the actual operation pressure, measured by a pressure transducer installed on the autoclave cover
- Cooling coil inside the autoclave
- Remote-control of cooling coil, gas-inlet valve and gas-outlet valve, operated from the control box or from a PC with process control software

Heating

- Heating/cooling jacket installed on the autoclave vessel instead of electrical heating system
- Oil heating system for connection to the heating/cooling jacket with electronic cascade-control and heat exchanger for heating and cooling
- Independently adjustable safety temperature cut-off system, including an additional thermocouple mounted in the wall of the autoclave
- "EMERGENCY-OFF" button for fast cutoff of the electrical heating and cut-off of gas feeding (if option "gas-inlet valve with remote control" has been ordered)

Three packages

for measuring and controlling of an autoclave system are available:

M&C package I:

Electrical control box

M&C package 2

Electrical control box plus PC-system with hard- and software for

- data collection
- programming a temperature set-point

M&C package 3:

Electrical control box plus PC-system with process-control hard- and software

for:

- visualisation
- programming of set-points
- data collection and graphics
- operation of the autoclave system by means of the PC
- automatisation of test runs

M&C package I

Electrical control-box

For operation of the autoclave system and for digital indication of the actually measured data.

Standard features include a PID-controller for the operation temperature, using two





Stirrers

- Glandless rotary stirrers with magnetic couplings for maximum torques of 60/120/180 Ncm and optionally more
- Controller for remote-adjustment of the stirring speed and digital instrument for speed indication installed in the electrical control box
- High-Torque Stirrers for high viscosity applications with direct driven stirrer shaft and self-sealing system
- Torque measuring system for the stirrer with digital indication of the actual torque
- Gas inertisation valve for magnetic stirrers
- Stirrer elements for different stirring tasks (exchangeable)
- Catalyst baskets mounted on the stirrer shaft (exchangeable)

Valves

- Bottom drain valve (not available for bench top autoclaves), removable for easy maintenance
- Safety valve instead of rupture disc device
- Non-return valve for gas inlet
- Reflux condensor for gas sampling during reaction
- Pressure cooler with dip tube for liquid sampling during reaction
- 2 sight glasses (windows) installed opposite to the autoclave wall only on special demand under consideration of pressure and temperature

Continuous-flow operation

- Pressure controllers for gas-feeding to realize pressure-constant continuousflow operation
- Mass-flow measurement with digital indication of the actual and the total gas consumption
- Take-off condensers with receiving vessel for removing of a volatile liquid phase during reaction
- Storage- and buffer-vessels
- Dosing pumps and compressors for liquid- and gas-feeding
- High-pressure heat exchangers such as gas and liquid pre-heaters and -coolers
- High-pressure separators for separating the gas phase and the liquid phase

separate thermocouples for measuring the inside temperature and the wall temperature of the autoclave. Additional options are available (see table 1).



Features - control box (table I) √ ■ Mainswitch with power visual indication **Heating System** ■ ON/OFF-buttons: Heating PID-temperature controller Temperature control: Cascade control or Ramp function S Measurement of inner temperature and wall temperature of the autoclave complete with parallel working digital temperature indications ■ Self-optimization of the temperature controller for calculation of PID-parameters Optical and acoustical alarm at over-temperature ■ Button: Alarm Acknowledge **Stirrer System** √ ■ ON/OFF buttons: stirrer motor Digital indicator for the stirring speed Remote control for adjustment of the stirring speed via control box Torque-measurement system for the stirrer motor, with digital indication of the actual torque Option **Pressure Indication** Pressure transducer with digital indication of the operation pressure, optical and acoustical alarm at over-pressure Option **Additional remote-controlled Features** ■ Button "Cooling ON" with visual indication remote control of the cooling coil by a magnet valve Option Button "Cooling OFF" Button "Gas Inlet OPEN" with visual indication remote control for Button "Gas Inlet CLOSED" Option gas inlet and gas outlet by pneumaticaly driven ■ Button "Gas Outlet OPEN" with visual indication high-pressure valves ■ Button "Gas Outlet CLOSED" Option **Additional Safety Features** Emergency STOP-Button installed directly on the autoclave-frame for fast CUT-OFF of the electrical heating system and gas feeding Option Safety-Temperature-Limiting system (independently working from the PID temperature controller) with separate, additional thermocouple installed in the autoclave wall complete with adjustable electronic temperature-limiting device for heating CUT-OFF Option

Measuring & Controlling



M&C package 2

Electrical control box plus PC with data collection system



Electrical control box

The electrical control box can be equipped with all options according to the table "Features" (Table 1).

A process and programm controller with up to eight channels and setup program is include for configuration from PC.

The Display is a 5" color screen.

PC with colour printer and soft/hardware for data collection and graphical display of:

- inner temperature
- wall temperatur
- operation pressure
 (if option "pressure transducer with digital indication of the operation pressure" is ordered together with the electrical control box)
- stirring speed

set-point programming of:

inner temperature or operation pressure

Additional Features

- Pressure controller for continuous-flow operation
- Mass flow controller for consumption gas or liquid
- Control of the lifting functions to rasing and lowering the autoclave vessel

M&C package 3

Electrical control box with process-control system based on an SPS-system

Electrical control box

The electrical control box can be equipped with all options according to the table "Features" (table 1).

The operation of the autoclave system via the control box or alternatively by means of the PC with process-control system can be selected via 2 additional buttons installed on the electrical control box.

PC with colour printer and process-control system

for visualization of:

- inner temperature (actual value)
- inner temperature (set point)
- wall temperature (actual value)
- over-temperature (alarm)
- operation pressure (actual value)
 (if option "pressure transducer with digital
 indication of the operation pressure" is ordered together with the electrical control box)
- stirring speed (actual value)

- torque of the stirrer (actual value) (if option "torque-measurement system for the stirrer motor" was ordered for the electrical control box)
- status of all remote-controlled autoclave-components
- main data of a test run (name of the test run, name of the user, start/stop of data collection)

for set-point programming of:

- inner temperature
- stirring speed

for data collection with graphical display of:

- inner temperature
- wall temperature
- operation pressure (if option has been ordered for the electrical control box)
- stirring speed

- actual torque of the stirrer (if option has been ordered for the electrical control box)
- alarms
- main data of the test run (name of the test run, name of the user, start/stop of data collection)

for operation of the autoclave system by means of the PC:

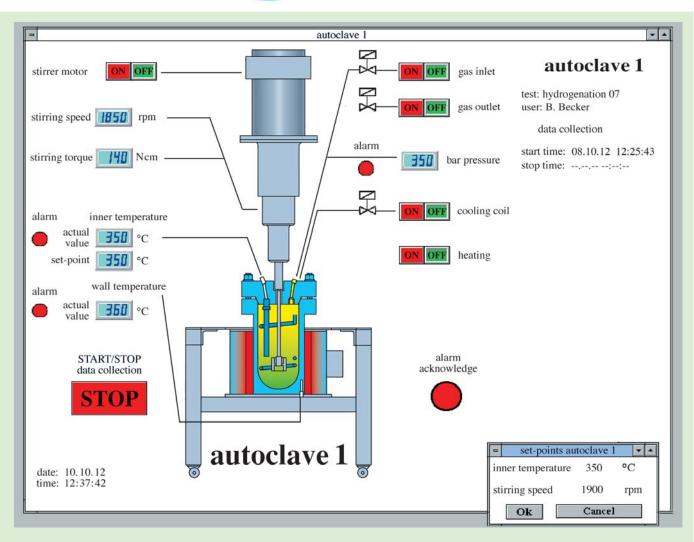
- stirrer motor: ON/OFF
- heating: ON/OFF
- cooling ON/OFF
- (if the relevant options have been
- gas inlet ON/OFF
- ordered for the
- gas outlet ON/OFF J electrical control box)
- alarm acknowledge: buzzer OFF

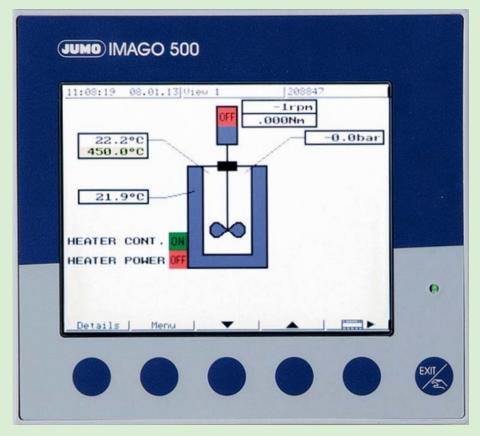
data collection: START/STOP

- for automatization of test runs:set-points can be pre-programmed and changed via time control
- switching ON/OFF of remote-controlled autoclave-components can be preprogrammed by time-control

The process control-system can be upgraded for the parallel operation of several autoclave systems.

Measuring & Controlling

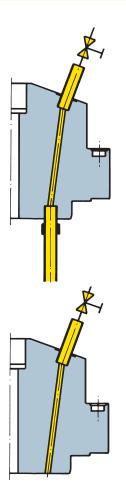






Valves

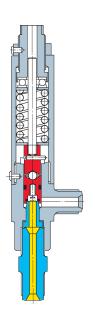














Gas inlet valve with dip tube

A gas inlet valve, complete with dip tube, is used to fill the autoclave with gas for the chemical reaction.

Optionally it can be equipped with a nonreturn-valve to protect the gas supply. If remote control of the gas inlet is required this high-pressure gas inlet valve is equipped with a pneumatical drive (optional).

Gas outlet valve

A gas outlet valve is used for decompression of the autoclave system, after the reaction has been finished.

It may also be used for inertisation of the autoclave prior to filling with reaction gas. If remote control of the gas outlet is required this high-pressure gas outlet valve is equipped with a pneumatical drive (optional).

Rupture disc

The rupture disc device protects the autoclave system against over-pressure.

The rupture disc breaks in the case of over-pressure (= more than the max. working pressure of the autoclave).

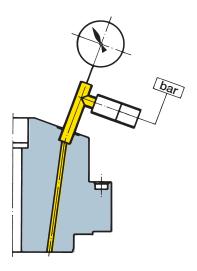
The rupture disc device is delivered together with spare discs.

Safety valve

The safety valve protects the autoclave against over-pressure (= more than the max. allowed pressure of the autoclave). In case of over-pressure the safety valve opens.

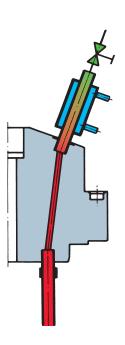
It closes automatically if the sealing area has not been damaged during blowing-off by e.g. an abrasive medium like a catalyst.











Pressure gauge The pressure gauge indicates the actual operation pressure directly on top of the autoclave. The range is 0-400 bar. Pressure transducer The pressure transducer measures the

The pressure transducer measures the operation pressure inside of the autoclave and transfers this value into an analogue signal of 4-20 mA, which is transmitted to a digital indicator installed on the electrical control box.

The actual operation pressure is now indicated not only by the pressure gauge but also digitally indicated on the control box.

Reflux condenser for gas sampling

The reflux condenser is used for gas sampling during the reaction.

A condenser with a cooling jacket is equipped with a fine-regulation valve to collect a small quantity of a gas-sample during the reaction. The liquid phase within the sample is recondensed by cooling water flowing through the jacket and flows back into the autoclave leaving the gas sample.

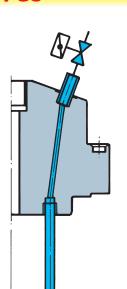


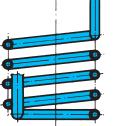
Pressure cooler with dip tube for liquid sampling

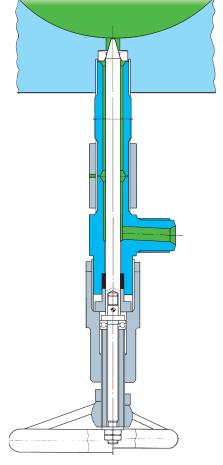
The pressure cooler is used for the collection of liquid samples during the reaction. A condenser with a cooling jacket is equipped with a fine-regulation valve and connected to a dip tube, which reaches to the bottom of the autoclave. The cooling jacket is required to cool down the liquid phase, thereby preventing vapour at the outlet of the sampling valve.

The pressure cooler with dip tube could be used additionally to empty the autoclave after the reaction has been finished. This can be achieved by giving a small gas-pressure on the liquid phase.

Valves













Cooling coil

A cooling coil inside the autoclave allows:

- the control of an exothermal reaction
- to cool down the medium inside the autoclave after reaction has been finished
- to heat-up the medium within the autoclave by use of a heat transfer medium e.g. an oil-heating/cooling system with temperature controller.

Together with an autoclave ordered with a cooling coil, two blind plugs are delivered separately. Therefore enabling the autoclave to be operated without the cooling coil. A magnet valve is connected to the cooling coil on top of the autoclave if the option "remote control for cooling coil" has been ordered. Thereby cooling water can be switched ON and OFF via push buttons mounted on the electrical control box and automatic cooling can be programmed by setting a limit temperature value at the temperature controller of the electrical heating system.

Bottom drain valve

A bottom drain valve is used to empty the autoclave after the reaction has been finished.

Autoclaves with floor stand frame or with autoclave lift can be equipped with a bottom drain valve.

Nearly no dead-space has been realized by the special design of the valve.

The valve can be screwed out of the autoclave for easy repair and maintenance in case e.g. of a damaged sealing surface. It is equipped with an easy-to-exchange stem tip and valve seat.

Inside polish

The inside surface of the autoclave vessel (without autoclave cover and inside components) can be polished as an option to improve the corrosion resistance and to prevent sticking of chemicals on the autoclave wall.



HAAGE

high-pressure autoclaves

are – beside the autoclaves described in this brochure – also available in custom-made design:

- Volumes of upto 1.000 ltrs.
- Special alloys for all wetted parts:
 - Hastelloy B or C
 - Titanium
 - Inconel 600, 625
 - Incoloy
 - Tantalum
- Heating/cooling jacket instead of an electrical heating system for connection e.g. to an oil-heating/cooling system with temperature controller





high-pressure pilot plant



HAAGE pilot plants

are designed as turn-key pilot plants with all measuring and controlling systems for continuous operation of the pilot plant according to the special process requirements of the customer.



Technology for Research and Industry

Since nearly 60 years we develop and manufacture a wide range of technical products and laboratory equipment. Our major customers are the chemical industry, gas industry, refineries, paper industry, and research institutes. Applications for our products are in the field of research and production. Sales and service facilities in European and overseas countries are covered by independent agents.

Our technical expertise enables our staff to offer flexibility for customers specific requirements. Our experience in engineering, machining of stainless steel and special alloys together with the application of microprocessor technology and measuring and controlling systems provides an excellent base for product development.

We apply an extensive quality system according to DIN ISO 9001/EN 29001, which has been certified by the German TÜV-CERT.









Manufacturing **Program**

autoclaves

automatic distillation apparatus for boiling analysis of fuels and solvents (DIN 51751, ASTM D 86, ASTM D 850, ASTM D 1078, ISO 3405 a. o.)

bellows valves buffer vessels fittings fixed bed reactors fluidized bed reactors high pressure autoclaves high pressure components high pressure pilot plants high pressure valves Intensive Gasing Plants paper test equipment paper-sheet forming apparatus pilot plants sample bottles stirred autoclaves valves

please ask for

Service

Besides manufacturing new equipment in accordance to your requirements we also offer repair works and modifications of third party products in our facility.





