

Autoclaves for study of Gas Hydrate Formation

Salient features

- For examination / study of gas hydrate formation
- Pressures upto 350 bar & temperatures upto 100°C
- 100 ml to 100 ltr net filling volume
- Analysis of effectiveness & efficiency of thermo dynamic and kinetic gas hydrate inhibitors
- MOC: SS316 / Hasteloy C etc.
- Glass window with camera for visual observation of gas hydrate formation
- Long term experiments of upto 30 days can be performed

Autoclaves for observation of gas hydrate formation

Gas hydrates are inclusion compounds of gases in a lattice of water molecules. Huge amounts of methane are stored around the world under the sea floor in the form of solid methane hydrates. Methane hydrates, represent a new and completely untapped reservoir of fossil fuel, because they contain, immense amounts of methane, which is the main component of natural gas. Methane hydrates belong to a group of substances called Clathrates - substances in which one molecule type forms a crystal-like cage structure and encloses another type of molecule. If the cage-forming molecule is water, it is called a hydrate. If the molecule trapped in the water cage is a gas, it is a gas hydrate, like methane hydrate. Methane hydrate also poses problems during transportation of natural gas. Temperature and pressure conditions in pipelines especially in cold areas allow the formation of hydrates. These hydrates form agglomerates and tend to clog valves, pumps, pipelines and other parts. It is desirable to avoid the formation of hydrates rather than removal of existing hydrate due to economical and safety reasons. The production as well as study of artificial gas hydrates are done in special autoclaves like the gas hydrate autoclave System, under specific pressure and temperature conditions. At room temperature and normal atmospheric pressure, methane hydrate is unstable dissociating into water and gas. Pipeline conditions can be simulated in the gas hydrate autoclaves to check the effectiveness & optimization of hydrate-inhibitors. Pressure-resistant borosilicate / quartz / sapphire-glass windows in the gas hydrate autoclaves allows the use of one or multiple boroscope-cameras for observing or recording the processes of gas hydrate formation inside the autoclave. The autoclaves can also be provided with magnetic stirrer to simulate turbulent mixing conditions. Overhead stirrer can be connected to a torque sensor to perform torque measurements to study viscosity changes. Refer page 42 for 25 ltr gas hydrate system.



High pressure vessel system for hydrogen induced disbonding tests

Amar manufactures & supplies systems for Hydrogen Induced Disbonding (HID) tests as per ASTM G146. These tests are used to simulate & study the effects of hydrogen environment under very high pressures from 150 - 250 bar & temperatures from 400 - 500°C on bimetallic plates that are to be used under similar conditions in refineries. The results indicate the resistance of bimetallic steels & its alloy to hydrogen induced disbonding. Such tests can be used to decide the material metallurgy, its heat treatment, manufacturing & fabrication technology for use in refineries in similar environments. The system can be manually operated or completely automated.

Initially the test samples are put inside the vessel, pressurized with hydrogen to very high pressure upto 150 - 200 bar & then heated to desired temperature of around 400 - 500°C for a period of around 48 hrs. After the test is over, the vessel is cooled at a pre-defined rate of around 150°C/hr till the temperature reaches 200°C. The vessel pressure is then released completely & cooled further to remove the test samples.



Pressure vessels for soaking of diamonds & precious stones

- High pressure high temperature pressure vessels are often used to purify & improve the finish of diamonds & precious stones by subjecting them to high pressure & temperature conditions.

Pressure vessel for gas / liquid storage

- It can be used as a gas/ liquid storage pressure pot to transfer liquid/ gas in the autoclave/ other pressure vessel at higher pressure.



Note: For standard autoclave models refer page 2 to 11 & for non-stirred pressure vessels models refer page 28 to 31.



Optional Accessories

Optional accessories are offered to increase the versatility of the equipment, to add value & feature to the standard product & to provide complete range of instrument/ equipment required for a particular application. Most of the optional accessories are common for stirred, non-stirred, glass, shaker & fabricated reactors unless specified. The standard optional accessories can be enquired by simply mentioning their code & required specifications.

Complete mounting of all the accessories shall be done on autoclave stand / trolley.

All the indicators / controllers are mounted on a common SS panel.

Gas Pressure Regulator (FPR)

To manually charge different gases at desired pressures upto 140 bar / 2000 psi or higher into the reactor from gas cylinder. Nitrogen, Oxygen & Hydrogen can be charged through same regulator (with special adaptor). The regulator is made from SS316 & comes with inlet - outlet pressure gauges & flexible SS braided Teflon PTFE high pressure hose pipe (4m long) with non return valve.

- Optional:**
- 1) Regulators upto 350 bar, automated regulator
 - 2) Regulator for other gases like NH₃, CO₂ etc. & MOC of Hastelloy C
 - 3) Pressure reducing station with auto changeover



Gas Booster (GB)

Gas boosters are useful when the cylinder pressures are much lower than the autoclave rated pressures. In such case the booster takes gas at lower pressure from cylinder & compresses the same to deliver at higher pressures. They are generally pneumatically operated. Special gas booster pumps are available for liquid CO₂ used in supercritical fluid extraction system. The booster systems are supplied with air filter regulator, pressure relief valves, inlet-outlet pressure gauges, valves & flexible hose pipe.

- Optional:**
- Boosters upto 700 bar pressure



Gas Booster



SS mounting with all fittings & valves

Thermal Gas Mass Flow Meter (MFM) / Controller (MFC)

MFM can be used to measure accurate mass flow rate of gas (in gm/hr or LPH) & totalized quantity of mass / volume (in gms/ltr) charged in the autoclave at any point. Mass flow controller (MFC) is used to charge the set flow rate of gas into the autoclave at high pressures up to 100 bar or it can be used in pressure control mode to indicate the gas flow & total gas uptake to maintain desired set pressure inside the autoclave (ideal for hydrogenation). The same MFM / MFC can be used for multiple gases by just entering the conversion factor for different gas densities provided the gases are inert to each other. The MFM/MFC comes with high pressure flexible hose, inlet filter with digital gas flow indicator cum totalizer & additional pressure PID controller with pressure sensor if the MFC is used in pressure control mode. User has to specify the maximum flow rate range, pressure, gas & mode (flow control or pressure control) for ordering MFM/MFC.

- Optional:**
- 1) Ex-proof MFM / MFC can be offered on request.
 - 2) MFM/MFC upto 300 bar pressure



MFC



Flow Indicator & Totaliser



Ex-proof MFC

Coriolis Gas - Liquid Mass Flow Meter/Controller (CFM/CFC)

These are used for higher & accurate gas or liquid flow rate indication or control in cases where thermal mass flow meters are not suitable. A common meter can be used for different gases & liquids for a particular range of flow.

- Optional:**
- Ex-proof CFM/CFC can be offered on request



Coriolis Mass Flow Meter



Coriolis Mass Flow Controller

Digital Pressure Indicator / Controller (DPI)

It consists of SS316 pressure sensor (transmitter) & digital pressure indicator/ controller (mounted on common control panel) with pressure alarm & optionally heater cut off for safety. Digital pressure indicator has pressure reading in bar & psi, where as controller reads any one of the units. The controller is normally used with mass flow controllers (MFCs) or solenoid/flow control valves to maintain constant pressure inside the autoclave. With MFC, gas flow indicator & totalizer are also provided on the same panel. The pressure sensor has temperature limitation upto 100°C & hence the same is provided with water cooling jacket.

- Optional:**
- 1) Intrinsically safe pressure sensor.
 - 2) Pressure sensors with Hastelloy C / Inconel wetted parts can be offered on request.



Solenoid Valve



Pressure Sensor

Optional Accessories

Liquid / Slurry / Gas Charging High Pressure Pot (HPP)

It is used to transfer liquids, slurries or gases in to the autoclave under pressure. It consists of high pressure SS-316 pot designed for working pressure 100 bar or higher with port for nitrogen gas (N₂), liquid / slurry inlet with valve & funnel, outlet valve, pressure gauge, pressure safety valve, high pressure hose & NRV. The liquid to be charged is fed into the pot from top funnel & pressurized with N₂ gas until its pressure is higher than the autoclave pressure & then under pressure the liquid is charged into the autoclave. The quantity & rate of flow of liquid charged is not known precisely in this system. However a level indicator or sight glass or weighing balance or flow meter can be provided optionally to measure the liquid charged or its flow rate. Pots of different pressure, M.O.C. & sizes can be offered.

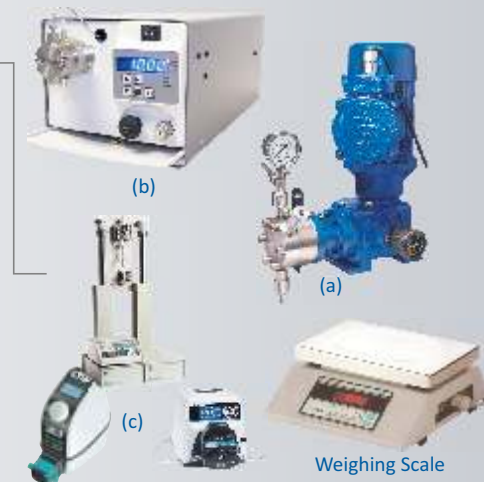
These pots can also be used for storage of gases when gas cylinders are located at a far away place. With this option approximate quantity or flow of gas consumed can also be determined by measuring the pressure drop.

Volume: 250 ml, ½ ltr, 1 ltr, 2 ltr, 5 ltr, 10 ltr

Optional: a) Forward pressure regulators can be provided at the outlet of (FPR) pot if they are used as gas charging.

b) Pots upto 200 bar / 350 bar

c) Pots in MOC Hastelloy C, Inconel, Monel, Titanium



Ethylene EO / Polypylene PO Oxide Pot (EPP)

It consists of SS 316 (EO/PO) horizontal pot with inlet, outlet valves, gas inlet with dip tube, thermowell, pressure gauge & high pressure hose pipe for 10 bar working pressure.

It is used for ethoxylation / propoxylation.

Volume: 1 ltr, 2 ltr & 5 ltr

Optional: Ex-proof weighing balance to measure the quantity of EO/PO charged.

Liquid Metering Pump System (LMP)

This system is used to charge liquid at a desired rate from as low as 1 ml/hr to 100 ltr/hr, when the autoclave is under pressurized condition. The system comes with a metering pump, flow indicator, controller, liquid sump, pressure gauge, strainer & high pressure hose.

Pressure safety valve, flow totalizer can be offered on request. Types of pumps offered are:

a) Diaphragm metering pumps for pressures upto 100 bar & minimum flow range of 60-600 ml/hr to maximum 10-100 lit/hr. The flow rates are varied by varying the motor speed with variable frequency drive. Materials: SS316, option: Hastelloy C, Titanium, PTFE

b) High pressure more accurate HPLC type low flow metering pumps for high pressures upto 350 bar & flow range from 0.01 upto 100ml/min.

These pumps can be used along with precision weighing scales to measure the total liquid charged at any point of time. Materials: SS316, option: Hastelloy C, Titanium

c) High pressure syringe, PTFE diaphragm, gear, peristaltic pumps can be offered for pumping corrosive chemicals at low / high pressures for specific application & flow rate condition.

In line flow meters can be connected to measure & control the flow of the liquids.

Auto Cooling System (ACV / ACP)

This system is useful to control temperature overshoots for highly exothermic / out of control / runaway reactions as well as for faster cooling upon completion of reaction.

The control panel of the autoclave gives 230V, 1Ø output to connect external auto-cooling system. Options available are:

a) Solenoid valve (ACV) for autoclaves upto 25 ltr or

b) Pneumatically actuated ball valves (ACV) for autoclaves above 25 ltr connected at the inlet of the internal cooling coil with external source of water supply.

This is standard for 50 ml - 250 ml autoclaves, however for bigger autoclaves if the cold water line pressure is less than 2 bar then it is better to opt for external pump & tank water cooling system. Normal tap water cooling is not effective at higher temperatures due to steam back pressure whereas pump & tank auto cooling system gives positive pressures & faster cooling.



Optional Accessories

c) Pump & Water Cooling Tank (ACP): It consists of a SS 304 water tank & pump to circulate water in the internal cooling coil of reactors. Cooling starts automatically if the rate of heating rises suddenly. It comes with monoblock pump, tank, pipeline & flexible hose pipes with quick release coupling & is mounted on the autoclave trolley itself. Ice can be put in the water or the hot water can be discharged & fresh water replenished on continuous basis for more effective and faster cooling.

Optional: Ex-proof group IIC pump



(c)

Magnetic drive & pressure Sensor Cooling System (MCS)

It is a simple 10 ltr SS 304 tank with submersible pump to circulate water in the magnetic drive & pressure sensor jacket to prevent temperature rise beyond 80°C. It is useful if tap water connection is not near the autoclave & when water needs to be condensed.

Open Bath Heating Circulator (HB)

It is used to heat jacketed autoclave from ambient to 150°C by circulating thermic fluid inside the jacket. It consist of bath, pump, level switch, heater & internal cooling coil for cooling from high temperature to ambient temperature using chilled water or brine solution.

It is suitable for reactors up to 5 liter volume

Optional: Ex-proof heating bath

Closed Loop Heating Circulator (CLH)

It is used to heat jacketed autoclave from ambient to 350°C by circulating thermic fluid. It consist of a closed pressure withstanding tank, magnetically coupled pump, heater, level indicator & internal cooling coil for cooling from high temperature to ambient temperature using chilled water or brine solution.

Suitable for reactors up to 100 ltr volume

Optional: Ex-proof circulator

Low temperature open bath circulator (CB)

It is used to cool or control reactor temperature to -50°C by circulating thermic fluid cooled to up to -75°C inside the jacket or internal coil. It consists of a bath, pump, compressor, condenser & level switch.

Suitable for reactors up to 100 ltr volume

Heating Cooling open bath circulator (HCB)

It is used to heat, cool & control reactor temperature by circulating thermic fluid in the autoclave jacket. It consists of a bath, pump, heater, compressor, condenser & level switch.

Temperature range: -25°C to 175°C.

Suitable for reactors up to 5 ltr volume

Closed loop heating cooling circulator (CLS)

It is used to heat, cool & control reactor temperature by circulating thermic fluid in the autoclave jacket. It consists of a bath, pump, heater, compressor, condenser & level switch.

Temperature range: -35°C to 200°C / -80°C to 180°C.

Suitable for reactors up to 1000 ltr volume.

Flow Control Valves (FOV / FCV)

These valves can be connected at jacket inlet for controlling flow of steam/ hot oil/ water for temperature control from PID or at the inlet or outlet of the autoclaves for control of pressure.

Two options are available:

- (FOV) On/off pneumatic ball valve actuated by 230V output from panel to solenoid valve inline of air supply
- (FCV) Pneumatic proportionate flow control valves with I to P convertor (for accurate temperature / pressure control).



FOV



FCV

Reflux Condenser (RC)

It is a jacketed single tube SS-316 heat exchanger with packing material inside used to reflux the condensate back into the reactor & vent off the uncondensed vapours. It is connected directly on the autoclave lid & works under full autoclave pressure. A receiver pot can be connected at the bottom of the reflux condenser with valve for reflux take off. It is available in 0.01, 0.02 or 0.05 m² area.

Optional: Reflux condensers can be offered in different materials & area.



Optional Accessories

Condenser (CN)

It is a SS-316 shell & tube reverse flow heat exchanger for distillation/condensing vent vapours from the autoclave upto 10 bar pressure. It is offered in different surface areas such as 0.1, 0.2, 0.5, 1 & 2 m². The condensate can be collected separately in a receiver or optionally refluxed back into the reactor.

- Optional:**
- Higher area & pressures upto 350 bar
 - Different materials
 - Corrugated tubes heat exchangers for better efficiency / compact size.



Receiver Pot (RP)

SS 316 receiver pot can be connected at the outlet of the shell & tube or reflux condensor to collect the condensate separately. It is also provided with a port to apply vacuum & offered in 250 ml, 500 ml, 1 ltr, 2 ltr & 5 ltr volumes. Higher volumes & other materials can be offered on special request. It can be optionally provided with level indication.

Back Pressure Regulator (BPR)

It is SS 316 regulator mounted on the vent line of the autoclave & is used for maintaining constant pressure inside the autoclave upto 350 bar. The pressure is maintained by releasing the excess pressure into the atmosphere or through a hose to safe area. The pressure can be set initially on the gauge, by manually varying the knob until the gas comes out. Once the set pressure is exceeded the excess pressure is released until the autoclave pressure becomes equal to or below the set pressure. The pressure release is slow & gradual & the set pressure can be varied at any point.

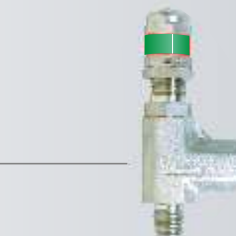
- Optional:**
- Electronic actuated digital pneumatic back pressure regulator, where the pressure is set digitally & can be released at preset rate of pressure release (6 bar air supply is required).
 - Pneumatically actuated pilot operated back pressure regulator (air / N₂ gas supply for rated pressure is required to activate the same)
 - Electronic control unit & forward pressure regulator with 4 mtr. hose for activating (b) above.
 - Materials: Hastelloy C, PTFE etc.



Pressure Safety Valve (PSV)

High pressure autoclaves are provided with built in safety rupture disc, however additional safety can be offered by providing pressure safety valve for which release pressure can be set manually by increasing or decreasing the spring tension or by simply changing the spring. The set pressure should normally be around the design pressure. Safety relief valves are standard supply for glass autoclave & shaker hydrogentor where rupture discs are not available for low pressures. These valves come with PTFE / Kalrez O-rings.

Optional: Materials: Hastelloy C, inconel



Chain Pulley / Hydraulic Head Lifting System (PL/EL/HL)

For autoclave volumes from 10 ltr to 250 ltr & removable head design, the head & vessel are too heavy to lift manually. Hence chain pulley block mounted on same trolley with SS Stand can be offered for lifting the head with minimum efforts.

- Optional:**
- Electric (EL) chain pulley lift
 - Hydraulic lid (HL) lifting arrangement can be offered on request for any reactor volume. Refer page 8 for picture of the same.



SCADA Software for remote operation & recording (SCD)

SCADA is a supervisory control & data acquisition software with all controllers / indicators having RS 485 modbus communication port or PLC & HMI / touch panel, for online display, set point changes & data logging of various parameters like pressure, temperature, motor RPM, motor current / torque, liquid / gas flow rate with totaliser, heater temperature, level, pH, ORP, turbidity, IR etc. remotely from PC as well as locally from panel. It gives continuous online data logging at predefined (variable) time interval, online graphical representation as well as historical data & graphs on PC for single or multiple autoclaves. RS 485-232 convertor & cable upto 50 m or higher is also supplied.

Optional: Wireless data communication from PC to panel or mobile alerts can be supplied on request.



Optional Accessories

Liners (LN)

Removable PTFE / Metal / Glass liners can be offered for autoclaves from 100 ml - 25 ltr. These liners can be used for reactions that are corrosive, to prevent the autoclave body from corrosion. PTFE & glass liners can be used upto 200°C & metal upto 500°C. The heat transfer is poor with PTFE & glass liners. Glass liners cannot be fabricated accurately. Hence, Amar recommends the use of completely corrosion resistant metal autoclaves made from special alloys like Hastelloy, Inconel, Titanium etc. over glass/teflon liners. Liners should be used only if cost is the constraint or corrosive chemicals are to be used sparingly. Metal liners can be offered in Hastelloy C , Inconel , Monel , Titanium etc.



Catalyst Basket (CBS/CBD)

It is provided to improve the efficiency of the catalysts & for holding the catalyst so that it will not be destroyed or changed by the stirring action of impeller. The baskets are made from SS316 wire mesh & connected to the stirrer so that they rotate with the stirrer. It is available for 450 ml-100 ltr autoclaves. The catalyst basket can be static which is stationary (CBS) or dynamic (CBD) which rotates with the impeller.



Catalyst Filters (CF)

These are small 7 microns SS 316 sintered cup filters which are threaded to bottom of the sampling dip tube so that the catalyst does not come out while sampling liquid. It is very useful when the catalyst is expensive or pyrophoric. These filters may reduce the rate/ flow of the sampling liquid due to the resistance offered by the fine mesh, hence they need regular cleaning to prevent choking. Filters are available for 500 ml - 100 ltr autoclaves.



Optional: a) Different micron sizes
b) Materials: Hastelloy C

Catalyst Addition Device (CAD)

It is used for one time catalyst charging under pressure during the reaction. It consists of a 3 ml to 150 ml (depending on Autoclave size) small container with airtight cap, which is openable. The powder is filled in the container which is then threaded to the autoclave head from below to a separate port with needle valve on the top. Under atmospheric & pressurized conditions the powder remains inside the container as the cap remains closed.

One has to apply gas pressure greater than autoclave pressure from the needle valve on that port so that the cap opens & releases the catalyst inside the autoclave under pressure.

Depending on the optional accessories selected, the catalyst addition device may not be possible due to space constraint on the head. Available for autoclave sizes 100 ml - 100 ltr & upto 250°C.



Catalyst Filtration & Recycling System (CFR)

It consists of vertical SS sintered filter cartridges in a SS housing. After the batch is over the reactor liquid is transferred to the catalyst filter under reactor/nitrogen pressure. After filtration is over, catalyst is taken back in to the reactor by back washing, thus recycling the same & making it available for the next batch. Thus catalyst is never exposed to atmosphere & is reused. Generally this system is suitable & available for 2 ltr to 2000 ltr reactor volume. These filters are available with zero hold-up volume.



Catalyst Slurry Charging System (CSC)

It is a pressure reactor with magnetic drive stirring, inlet & outlet valves, pressure gauge & nitrogen/vent. Solvent & catalyst are charged in the vessel & the slurry formed by mixing is transferred under nitrogen pressure into the reactor. The pressure rating & volume of this system is designed depending on the quantity of catalyst, pressure & temperature rating of the autoclave. Specially designed control system developed by Amar can be offered to charge the catalyst slurry in continuous mode at a pre-defined flow rate under pressure. This is very useful for CSTR, where no suitable pumps are available for slurry. This system is available for any reactor volume, pressures upto 350 bar & for a wide range materials.



Optional Accessories

Catch Pot (CP)

It is used to collect the vent vapours / gases from the autoclave vent / rupture disc / safety valve port, when the vapour / gas is highly hazardous & cannot be released directly into the atmosphere. It is a pressure vessel, designed depending on volume of autoclave & its design pressure. It is provided with inlet, outlet, vent, dip tubes, pressure gauge, safety valve & optionally a flame / flash back arrester. It is normally filled with water. The gases / vapours collected are safely released through the flame / flash back arrester to avoid any hazard. It is suitable & available for any autoclave volume.



Vacuum Pump (VP)

Suitable rotary vane or diaphragm oil free vacuum pumps can be supplied for vacuum from 100 mbar upto 0.001 mbar in the reactor. It is used either before starting the batch or for high vacuum distillation. Suitable analogue or digital vacuum indicator with controller can be offered on request. The reactor fittings would change for very high vacuum & this may limit its pressure rating

View Windows / Light & Sight Glass (SG)

These are quartz / sapphire view glass windows / light & sight glass of small diameter or along the length of the vessel with special cameras & software for continuous online viewing / recording in jacketed vessels to see the reaction. Suitable for high pressures upto 200 bar & any autoclave / reactor volume.



Multiple view windows



Round view window



Sight glass along the vessel length

In Situ FTIR Spectroscopy (IR)

In situ high temperature, high pressure infrared (IR) probe is offered for real time chemical reaction monitoring. It provides specific information about reaction initiation, conversion, intermediates & end point. Suitable for 1 ltr to 100 ltr reactors upto 100 bar & 200°C.

Other Accessories / Options

- Level transmitter (LT) / switch (LS) with indicator to measure or maintain level inside the reactor under high pressure. Used mainly in CSTR.
- pH/ turbidity (TB) / DO / ORP sensors with indicators & controllers for high pressure & temperature application. pH can be controlled automatically by variable speed acid & base metering pumps.
- Sampling pot with condenser for cooling / condensing & collecting the sample taken at high pressure & temperature.
- Torque sensor for accurate measurement of the motor torque, where change in torque indicates change in viscosity of the reaction.
- High pressure ultrasound transducer for high frequency mixing.
- Ex-proof certified gas purge panels for electrical accessories / utilities like heating cooling circulators etc.



(a)



(b)



(c)



(d)



(e)



(f)