high precision thermoregulation

Whitepaper

Temperature Control

for Chemical Research and Production

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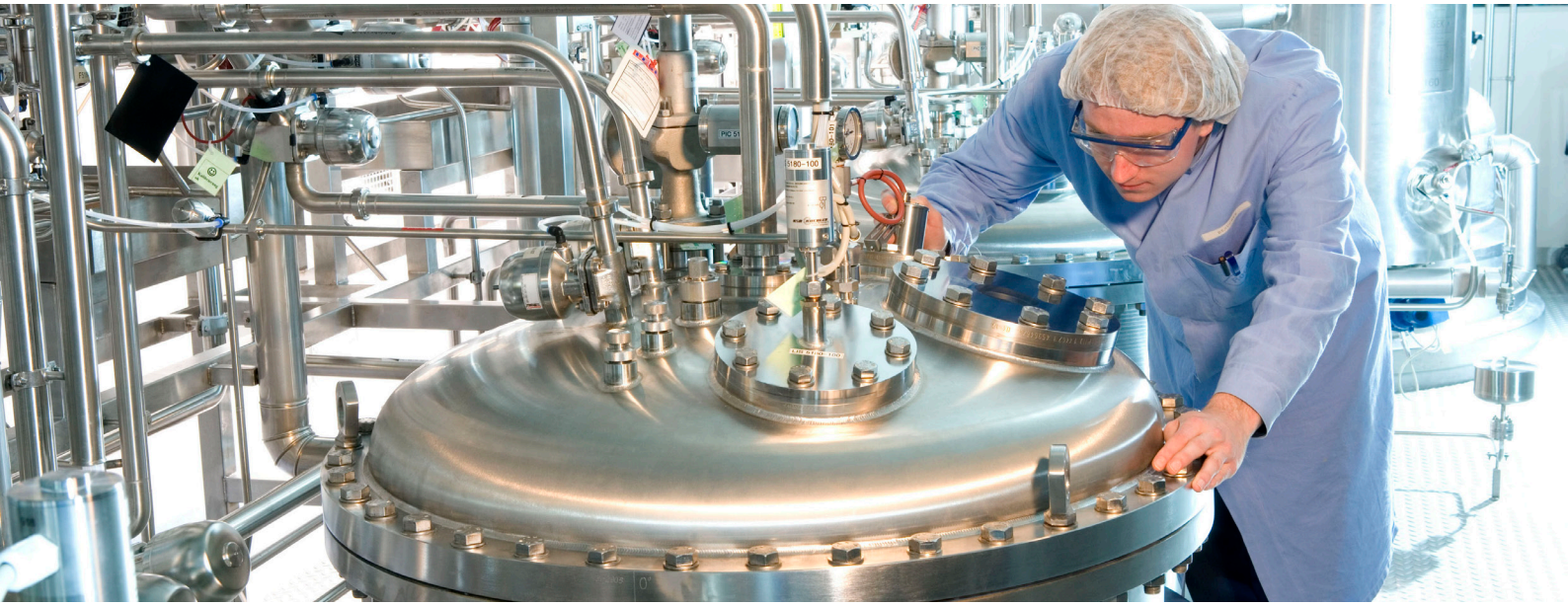


Temperature Control

for Chemical Research and Production

Precise temperature control has a significant influence on performance and quality in chemical process engineering. The Unistat range of temperature control systems ensure accurate temperatures and stable process conditions in research laboratories, pilot plants and kilolabs worldwide. The new Unistat Hybrid technology offers solutions for temperature control of large scale production reactors, enabling an affordable, partial modernisation of existing central plant cooling/heating systems.

When it comes to chemical reactions in production processes, the right temperature is a crucial factor. Therefore, chemical processes need an appropriate and reliable heating/cooling system. For process engineers it is essential to find the best possible compromise between yield, quality and productivity. Over the years and with the support of process engineers from the chemical and pharmaceutical industry, the Unistat range (Fig. 1) has been developed and refined. This collaboration has ensured that Unistats remain at the forefront of technology, making them suitable for controlling the temperature of chemical and bioreactors, autoclaves, reaction blocks & calorimeters in mini, pilot and distillation plants. As a result of their thermodynamic characteristics, Unistats offer cutting-edge performance regardless of the application and excelling even under heavily changing system conditions. To demonstrate the performance of the Unistat range over 200 case studies have been carried out. They show the Unistats unique ability to adapt to process requirements, offering precise stability and control.

Figure 1: Unistat temperature control systems allow scale-up from small reactors used in process development, to production volume.

The Unistat Principal

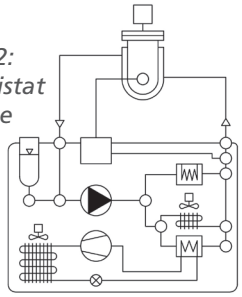
Reliable temperature control of a chemical process is critical to success and also dependent on an effective thermal transfer. With traditional "open bath" thermostats limitations occur: The flash point of thermal fluids at high temperatures is causing a relativly narrow safe operating temperature range. Degradation of thermal fluid, due to evaporation, water absorption and oxidation, dramatically reduces the thermal conductivity of the fluid. The large volume of fluid required to fill a bath, limits the performance and the dynamics of heating and cooling.

The Unistat principle (Fig. 2), unlike conventional technology, uses no internal bath. Instead a low internal volume in conjunction with an expansion vessel is used to compensate for volume change induced by heating and cooling. This principle reduces the mass of fluid and as a result dramatically increases the rate of temperature change, achieving cooling rates of several hundred degrees per hour.

Pressure & Flow

High heating and cooling capacities are worthless if pressure from the pump is too high. Key to good heat transfer is the pump performance. In order to achieve optimal circulation, high flow rates with low pump pressure are required. This allows heating and cooling energy to be transported very effectively by the Unistat so fast ramp rates are possible (Fig. 4).

Figure 2: The Unistat Principle



Many manufacturers promote the high pressure rates that are achievable in their systems, but the laws of physics tell us that high pressure is actually detrimental to flow rates and thermal transfer. Pressure reduces flow which causes restriced heat transfer - this has a dramatic effect on performance.

Most glass reactors have a maximum pressure of 0.5 bar - anything above will compromise the reactor. Pressure creates heat, therefore, the heat losses associated with high pressure have a greater impact when trying to achieve low temperatures.

For a comparison of thermal dynamics it is essential to take into account the cooling power density [W / l]. Cooling power density is particularly important for applications with a continuously changing reaction mass. Precise temperature control and process stability are crucial factors in obtaining the desired temperature result.
The better the heat transfer the quicker the temperature change. When endothermic or exothermic reactions occur, the Unistats impressive ability to react to the process, quick temperature changes and an intergrated process safety feature, allows the reaction to be kept under tight control without over or undershoot, resulting in higher purity levels (Fig. 3).

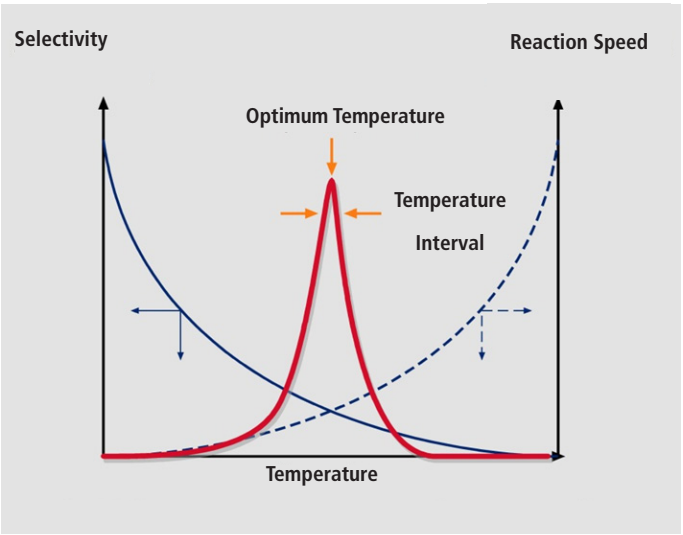


Figure 3: Temperature control of a chemical reaction has a significant influence on selectivity and reaction rate. The optimum is often in a narrow temperature interval.

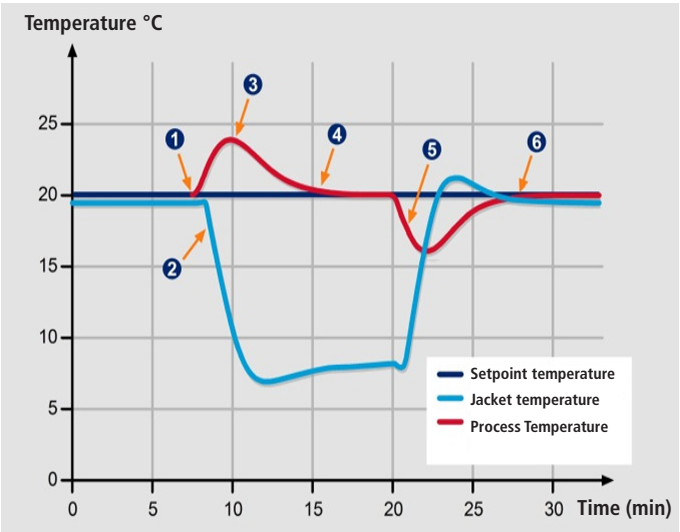


Figure 4: Regulation of an exothermic reaction in a reactor is fast and reliable and destruction of the reactor contents is avoided.

Thermal reactions are compensated quickly and reliably (Figure 4):

① Exothermic reaction starts. ② The Unistat compensates by rapid cooling. ③ Process temperature rise is brought under control.
④ The reaction is cooled to setpoint temperature. ⑤ As the process temperature drops the Unistat heats. ⑥ The process is brought back to the desired temperature.

Power according to DIN 12876

DIN – Deutsches Institut für Normung, is the national standards body for Germany.

DIN 12876 demands that the quoted cooling capacity is measured at full pump power, using water as a HTF. Reducing pump power reduces the heat entering the system, leading to more net cooling capacity which makes lower temperatures possible. Reducing the pump speed increases cooling power and in some cases 30 Watts to 50 Watts and up to 5 °C lower end temperatures can be achieved. In accordance to DIN, Huber always quote cooling power at full pump power.

Unistats have a very low filling volume and therefore a very high cooling power density [W / l]. This ratio shows how quickly a system changes temperature. A crucial parameter in terms of safety.

Figure 5: Unistats are in use in many industries, particularly in reactor temperature control.

Typical Unistat Applications:

- Reaction Systems, Autoclaves
- Pilot Plant
- Mini Plant
- Process Development
- Double jacketed vessels
- Reaction Calorimetry
- Distillation
- Plant testing
- Material Testing
- Combinatorial Chemistry
- Semiconductor Industry
- Kilolab
- Vacuum Chambers

Practical Features

In temperature control importance is placed on the primary functions of a system such as heating and cooling power, but often secondary attributes play a decisive role in performance. Huber systems offer users numerous functions and settings which make control simpler. These settings allow very fine tuning of the application.

Water Separation System

Occasionally water from previous applications, cleaning or moisture absorbed from the atmosphere during storage can build up within the heat transfer fluid circuit. Unistats offer the unique water separation system which allows residual water to be removed. If water is not removed, ice builds up during low temperatures, preventing heat transfer and in some cases resulting in costly repairs.

Optimised heat transfer

The Unistat range has improved pump connections with wide internal diameters to minimise flow losses and internal pressure. Wider bores, low pressure and low flow resistance allow higher flow rates. This has a significant influence on heat transfer and results in improved cooling and heating capacity, improved process control and faster response times. Large Unistats are equipped with M24 x 1.5 pump connections (Fig. 6). Bench top models are supplied with M16 x 1 adaptors as standard; allowing existing connections and hoses to be used without modification.

Figure 6: Large cross-sections and pump connections minimise pressure losses and provide optimum heat transfer.

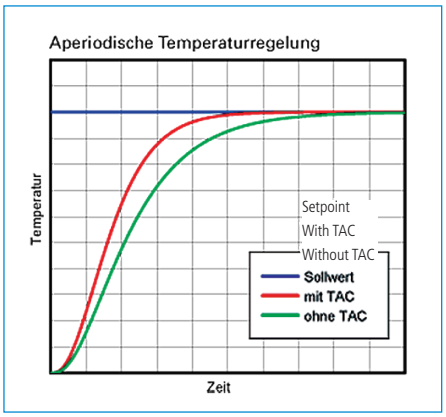
True Adaptive Control

Varying research criteria and process demands change the thermal load on the temperature control system. The solution is True Adaptive Control (TAC) which has the capability to adapt automatically to changing demands. By building a multi-dimensional model of the process, TAC is able to automatically adjust PID parameters to cope with and respond rapidly to sudden changes in the process.

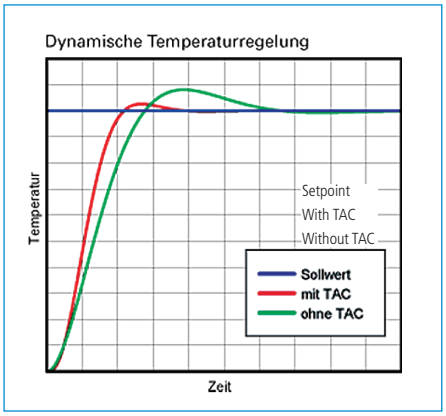
Operating in both „Jacket" and „Process", TAC provides responsive and close control with rapid changes and no overshoot to the process. User defined ramp rates allow for faster or slower response (Fig. 7). If TAC is not required, the user can manually adjust the PID parameters.

Figure 7: TAC generates a multidimensional model of the process to achieve the desired temperature in the shortest times without overshoot.

Rapid Ramping (no overshoot)



Faster Ramping (minimal overshoot)



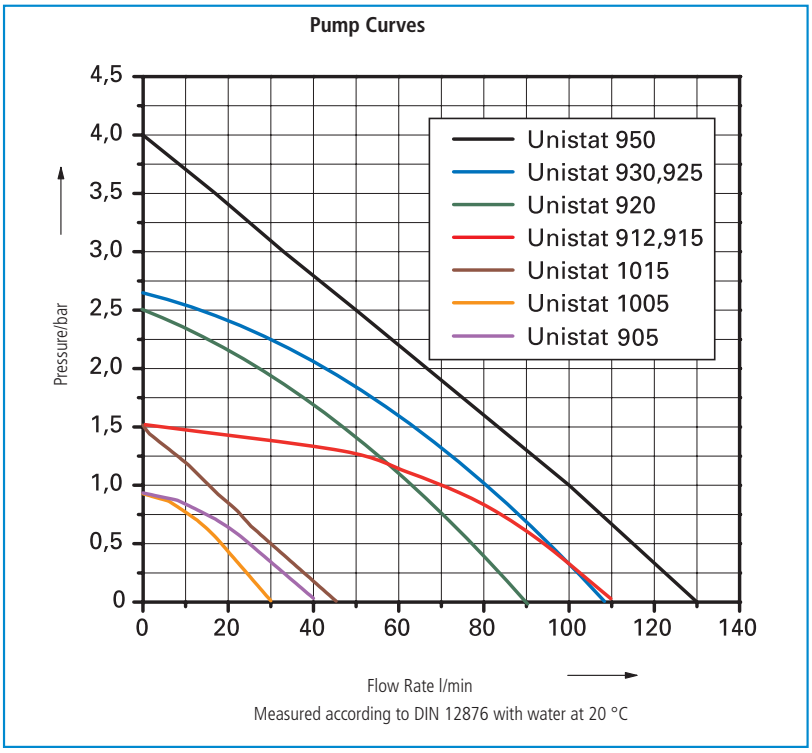


Figure 8: Pump curves show reduced flow as pressure increases.

Professional Scale-up

Over 50 models are available in the Unistat product group, offering precise temperature control from -120 °C to +425 °C. They range from very compact units to powerful models for controlling temperature of production reactors, to the Unistat Hybrid for production scale.

Petite Fleur

High heating and cooling capacities, sophisticated safety functions and an extensive list of features - all in compact housing with small footprints.

Figure 12: As the smallest of the Unistats the Petite fleur is suitable for the research laboratories.

Truly compact units occupy the smallest space whilst at the same time ensuring a high cooling power density (watts/litre). Even the Petite Fleur (Fig. 12), the smallest Unistat in the range, offers the best thermodynamic properties and full functionality despite being incredibly compact with a small footprint.
The working temperature range of the Petite Fleur is from -40°C to +200°C with cooling powers of 480 watts.

The powerful variable speed pump guarantees optimum circulation with flow and pressure rates of 33l/min and 0.9 bar. Unistats, including the Petite Fleur, offer many safety options such as TAC, VPC and Process Safety feature.

The Petite Fleur comes with a RS232/RS485 interface, a potential free contact, and connection for external control signals (ECS). Optional available is an analogue interface (0/4-20mA or 0-10V). The Petite Fleur is available in two versions for either temperature control of externally closed or externally open applications.

Figure 15: Ex-p Pressurised cabinet for use within hazardous areas in ATEX zones 1 & 2.

Models for Process Technology

The Unistat product range offers many models with the power to control a range of reactors from small scale to 1,000 litres. The product range has recently expanded with the air cooled Unistats 510 and 610.

Unistats are suitable for many typical applications in process and process engineering, e.g. temperature control of bioreactors, autoclaves and reaction blocks in Mini and Pilot Plants. The Unistat series offers cooling capacities up to 150 kW and operating temperatures from -120 °C to +425 °C.

In addition, options for weather protection and/or winter operation are available, allowing units to be located outdoors and controlled remotely with the detachable controller (Fig. 14). For Unistats sited in ATEX zones 1 & 2, Huber offers two solutions. The Unistat can either be placed in an Ex-p pressure chamber (Fig. 15) or an ATEX certified controller can be mounted in the ATEX area whilst the Unistat remains in the safe zone.

Figure 14: The detachable Pilot ONE controller offers the latest touchscreen technology.

Production Reactors of 10 m³ or more

The introduction of the Unistat Hybrid technology extends the range of Huber systems, allowing temperature control of large volume reactors of 10,000 litres or more.

The Unistat Hybrid combines the precise control of the Unistat range with the power of existing energy resources in a production facility such as steam, cooling water or liquid nitrogen (fig. 16), offering powerful process control and a partial

cost effective modernisation of existing resources.

The Unistat Hybrid system (Fig. 17) allows the Unistat to manage the interaction of the individual energy sources and to control temperature in accordance with the requirements. This results in faster heating times for large volume reactors heated by steam or respectively faster cooling times with liquid nitrogen or cooling water.

The advantages of this technique are precise control, an extended temperature range, more heating and cooling power and reliable control of thermal reactions. (Fig. 18). Linking the Unistat Hybrid into an existing centralised cooling/heating system results in better process conditions and improved production output.

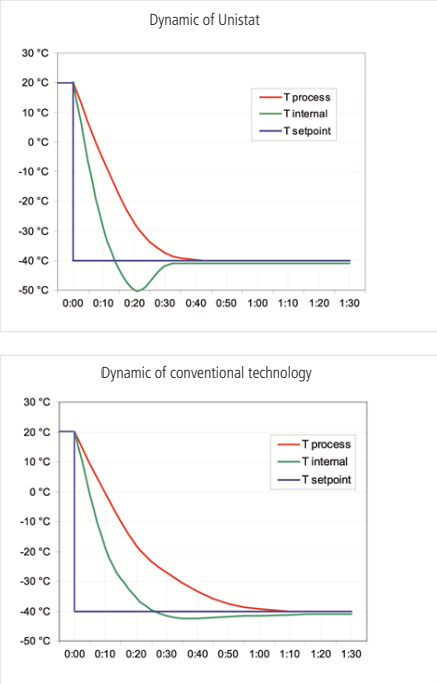


Figure 18: A comparison of the thermodynamics of Unistats and conventional technology illustrates the practical benefits. Time savings in research tasks and an improved production throughput are the main arguments.

Modernisation of Chemical Plants with the Unistat Hybrid

Chemical and pharmaceutical production plants often use central cooling and heating systems in production processes for temperature control. The Unistat Hybrid system optimises existing systems by connecting a hydraulically sealed temperature control unit. The available cooling and heating capacity is increased and the range of temperatures expanded.

Benefits

- Higher heating and cooling capacities
- Use of existing energy resources such as steam, cooling water, LN2 liquid nitrogen, etc.
- Temperature range extension in existing facilities
- Precise control of the process temperature
- Reliable compensation of thermal reactions
- Low cost modernisation of existing facilities
- Avoids costly and time consuming system renovations

Figure 17: With a Unistat Hybrid system production reactors of 10,000 liters and more can be controlled. Advantages are a high precision control and reliable compensation of thermal reactions.

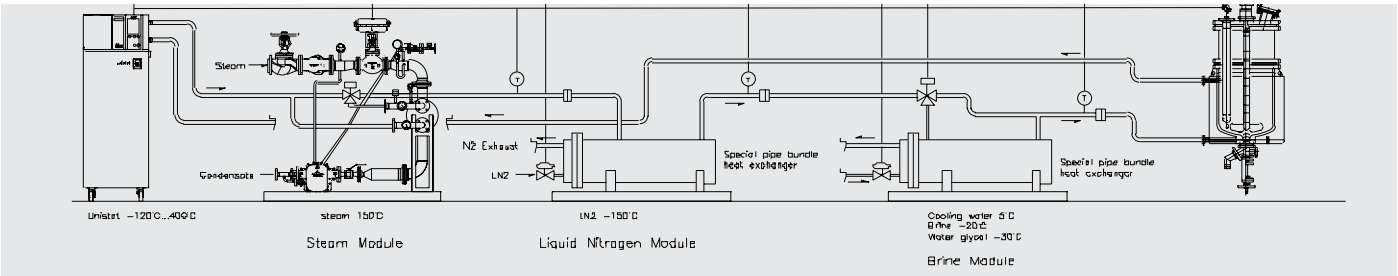


Figure 16: Complete solution: The Unistat hybrid temperature control system used in conjunction with external heat exchangers and various energy sources (steam, cooling water, nitrogen) and controls the interaction.

Low Operating Costs

Compared to conventional heating and cooling circulators, Unistats achieve greater efficiency and reduce resource consumption. The efficient energy management system keeps electrical power consumption to a minimum. Reduced operating costs and minimal use of water further saves on utility consumption. The hydraulically sealed construction of the Unistat range prevents the formation of oil vapours and oxidation, extending the lifetime of costly thermal fluids by years.

Environmental Commitment

1995 signalled the end of the use of CFC's as refrigerants. In 1991, four years ahead of the ban, all Huber systems were built without CFC's and R22 which was later banned in 2000. As a result Huber customers were the first to buy machines that reached -120°C, fully Chlorine free and environmentally friendly. Bespoke temperature control systems developed in accordance with the global K6 directive from Roche requires that chillers are HCFC, HFC & PFC free. Now the entire Huber range is offered with natural refrigerants as an option or as standard. The energy management system keeps energy consumption to a minimum.

The Unistats highly efficient energy conversion from electrical power to temperature control as well as the speed and accuracy saves resources, time and money. With a Huber chiller consumption of water can be reduced to zero and, in addition, the materials used in manufacturing are recyclable stainless steel, copper and high grade polymers.

Reducing the energy consumption of the products by using innovative construction methods and by unique systems remain a priority. The current units are a great start, leading the way to a reduced energy consumption.

Pilot ONE Controller

The Pilot ONE (Fig.19) can be removed from the Unistat and mounted remotely to allow flexible control. Further features such as: the programmer, adaptive cascade control, ramp functions, calendar start, individual user menus, sensor calibration and analogue and digital interfaces for integration in process control systems round off the Unistats functionality.

The Pilot ONE offers comprehensive safety features and continuously monitors the process. This feature allows Unistats to run unattended. In the event of an emergency, alarms are activated and, depending on the user defined settings, the system will then either turn off or the emergency over-temperature protection function will be activated; switching the machine to 100% cooling in the event of a thermal runaway.

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Pilot ONE Controller Advantages & Functions:

- 5,7" TFT touchscreen
- Graphic with 480x640 Pixel
- 11 Languages
- USB & Ethernet
- Remote control
- Convenient menu prompts
- Temperature profile represented as a real time graph curve
- Fully backwards compatible

Figure 19: Unistats are fitted with the Pilot ONE controller as standard.