LABTEX®
White Paper

A Guide to High Performance Temperature Control

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In this Labtex White Paper:

the Science of Success

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Introduction to Temperature Control

Precise temperature control has a significant influence on performance and quality in chemical process engineering. The Unistat range of temperature control systems from Labtex 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 critical and so accordingly, is the need for an appropriate heating/cooling solution. For process engineers it is essential to find a 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 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 and calorimeters in mini, pilot and distillation plants. As a result of their thermodynamic characteristics, Unistats offer cutting edge performance regardless of the application, excelling even under heavily changing system conditions.

To demonstrate the performance of the Unistat range over 200 case studies have been created to show the Unistats unique ability to adapt to process requirements, offering precise stability and control.

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

The Unistat Principle

Reliable temperature control of a chemical process, is critical to success and, is dependent on effective thermal transfer. With traditional "Open bath" thermostats, limitations occur with; the flash points of thermal fluids at high temperatures, causing a narrow safe operating temperature range. Degridation of thermal fluid due to evaporation, water absorption and oxidation which dramatically reduces the thermal conductivity of the fluid. The large volume of fluid required to fill the bath puts a constraint on performance and severely limit the dynamics of heating and cooling.

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.

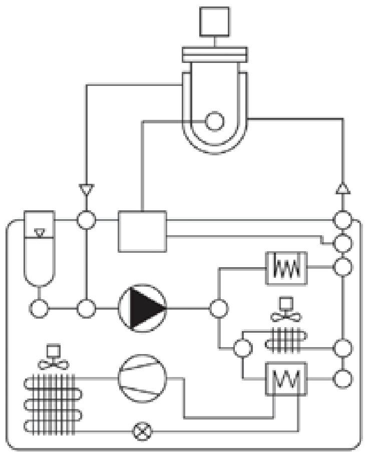


Figure 2: The Unistat principle combines powerful thermodynamics and modern microelectronics.

Pressure & Flow

Key to good heat transfer is 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). 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.

Most glass reactors have a maximum pressure of 0.5 Bar – anything above this 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 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 and 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 intergrated process safety feature allows the reaction to be kept under tight control without over or undershoot, resulting in higher purity levels, and better efficiency (Fig. 3).

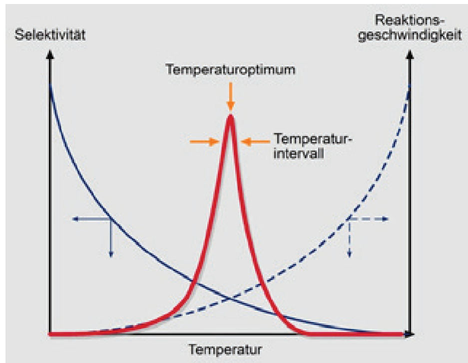


Figure 3: Temperature control of a chemical reaction has a significant influence on selectivity and reaction rate. The optimum is often in a narrow temperature interval.

Pressure & Flow continued

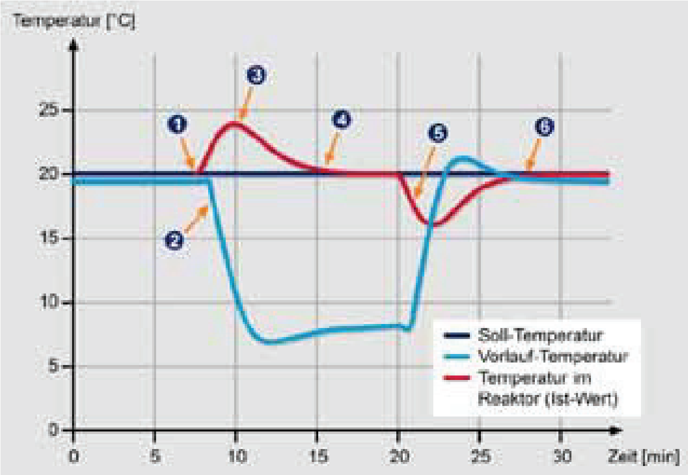


Figure 4: Regulation of an exothermic reaction in a reactor is fast and reliable and destruction of the reactor contents is avoided.

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

- **Exothermic reaction starts**
- **The Unistat compensates by rapid cooling**
- **Process temperature rise is brought under control**
- **The reaction is cooled to setpoint temperature**
- **As the process temperature drops the Unistat heats**
- **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 to be 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 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.

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**

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

True Adaptive Control

Varying research criteria and process demands change the thermal load on the temperature control system; what does not change is the requirement for good control.

The solution is True Adaptive Control (TAC) which has the capability to automatically change with those 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 – automatically and under all conditions.

User defined ramp rates allow for faster or slower response (Fig. 6). If TAC is not required, the user can manually adjust the PID parameters.

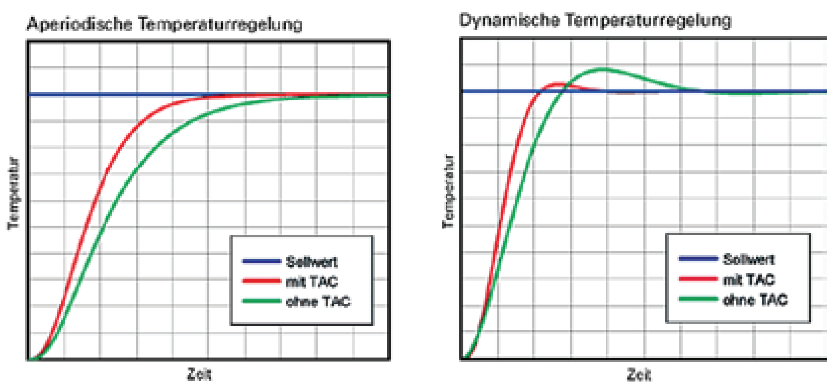


Figure 6: TAC generates a multidimensional model of the process to achieve the desired temperature in the shortest times without overshoot

Figure 7: Large cross-sections and pump connections minimise pressure losses and provide optimum heat transfer

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. 7). Bench top models are supplied with M16 x 1 adaptors as standard; allowing existing connections and hoses be used without modification.

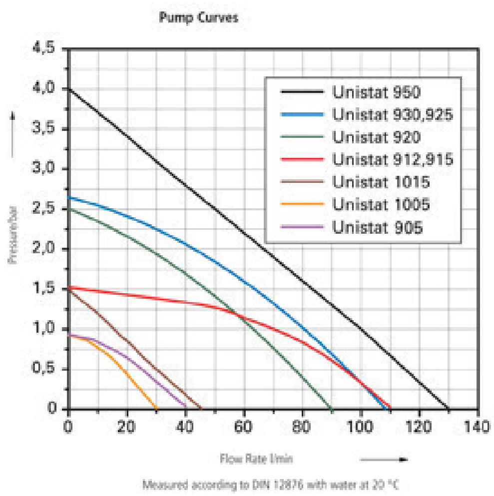


Figure 8: Pump curves show reduced flow as pressure increases

Practical features

In temperature control importance is placed on the primary function of a system such as heating and cooling power, but often secondary attributes play a decisive role in performance. High heating and cooling capacities are worthless if pressure from the pump is too high. Pressure reduces flow which causes heat transfer to be restricted and this has a dramatic effect on performance. With low pressure and high flow – smaller, less expensive units are required.

Huber systems from Labtex offer users numerous functions and settings which make control simpler. These settings allow very fine tuning of the application; for example, True Adaptive Control (TAC) intelligent temperature control system, continuously analyses the system, learning with the application so the more you use it the better control. VPC – Variable Pressure Control, with soft start, protecting delicate glassware against breakage and compensating for pressure changes in the thermal fluid. Intelligent temperature control techniques ensure minimum pressure with maximum flow for optimum heat transfer.

Figure 9: The Unistat range offers over 50 standard models for temperature control applications in research, pilot plant and production from - 120 to +425° C

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.

Pilot ONE Controller

Unistats offer full functionality in the Pilot ONE Controller. This 5.7 inch colour touch screen controller can be customised to clearly display all the important parameters for the application such as setpoints actual values and limits. Temperature gradients can be displayed in a real time graphic format and the unit can be switched from Celsius to Fahrenheit.

In addition the Pilot ONE Controller offers interfaces in 6 language options, temperature control programs can be written and stored and a range of safety features allow Unistats to run unattended. The Pilot ONE Controller 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.

Figure 10: Unistat were developed with process engineers in chemical and pharmaceutical companies

Analogue and Digital Interfaces

Numerous data interfaces allow Huber systems to integrate into equipment or process control systems. Standard features include an RS232/RS485 interface and various analogue interfaces complying to NAMUR standards are available. The optional Web.G@te module (Fig. 11) provides additional ports for Ethernet and USB, enabling the exchange of data over corporate networks, the Internet or the storage of data and profiles on USB sticks.

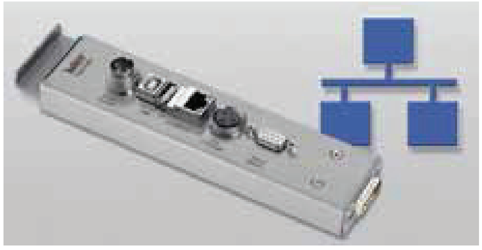


Figure 11: WebG.@te enables instrument control and data transfer via network or Internet

Unistats stand out in safety and economy! 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.

The Unistat Pilot offers Comprehensive safety features and continuously monitors the process allowing Unistats to run remotely. 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 overtemperature protection function will be activated; switching the machine to 100% cooling in the event of a thermal runaway.

Performance in the smallest space

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

Truly compact units occupy the smallest space whilst at the same time ensuring a high cooling power density (watts/litre). Even the Petite Fleur, the smallest Unistat in the range, (Fig. 12) 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.

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

The powerful variable speed pump guarantees optimum circulation with flow and pressure rates of 33 l/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 the Com.G@ te interface which offers RS232/ RS485, a potential free contact, analogue interface (0/4-20mA or 0-10V) and connection for external control signals (ECS). The Petite Fleur is available in two versions for either temperature control of externally closed or externally open applications.

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 or more depending on the application. The product range has recently expanded with the addition of the new Unistats 905, 905w, 912w and 915w (Fig. 13). These new models 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. All models cover a temperature range of -90 to +250°C and offer a heating capacity of 6 kW. Cooling capacities range from 4.5 to 11 kW depending on the model. The variable speed circulation pump achieves flow rates up to 110 l/min.

The Unistat-Pilot enables further automation of work processes with features which include a programmer, ramp function, calendar, start, customisable user menus, sensor calibration and analogue and digital interfaces for connection to process control systems. 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 13: The new Unistats 905, 905w 912w and 915w are suitable for applications in process and process technologies, such as reactors, autoclaves, mini plant, pilot plants, reaction blocks, distillation columns, etc.

Figure 14: The removable Unistat Pilot can be used as a remote controller

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

Production reactors of 10 m^3 or more

The introduction of the new 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.

Using a Unistat Hybrid system (Fig. 17) in this way, allows the Unistat to manage the interaction of the individual energy sources and control temperature accordingly. 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. Linking the Unistat Hybrid into an existing centralised cooling/heating system results in better process conditions and improved production output.

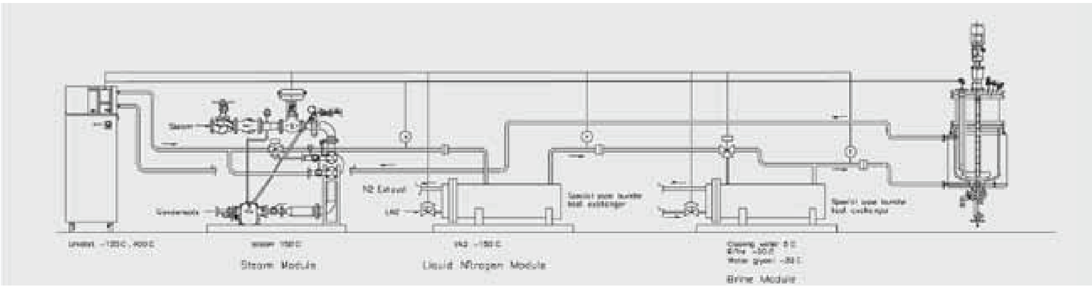


Figure 16: Complete solution: The Unistat hybrid temperature control system used in conjunction with external heat exchangers and various energy sources (steam, cooling water, nitrogen) and controls the interaction.

Low operating costs

Compared to conventional heating and cooling circulators, Unistats achieve greater efficiency and reduce resource consumption. Operating costs (electricity, cooling water, thermal fluids, etc.) are less than other systems.

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

For Huber, the eradication of CFC's is good but not sufficient, the current target is to phaze out all FC's, well before the deadline in 2015. Reducing energy consumption in the build and operation of systems remains a priority and the current units are a great start, leading the way in reduced energy consumption.

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.

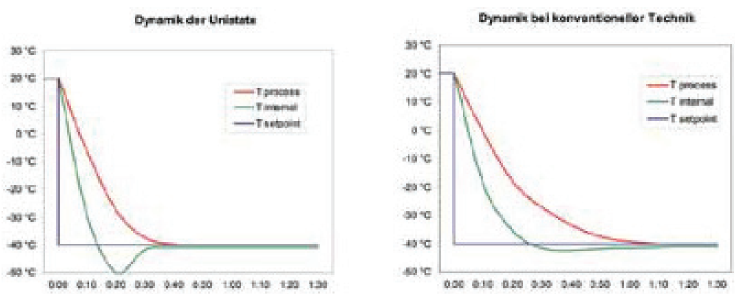


Figure 18: A comparison of the thermodynamics of Unistats and conventional technology illustrates the practical benefits. Time savings in research tasks and an improved production throughout are the main arguments.

Modernisation of Chemical Plants with the Unistat Hybrid

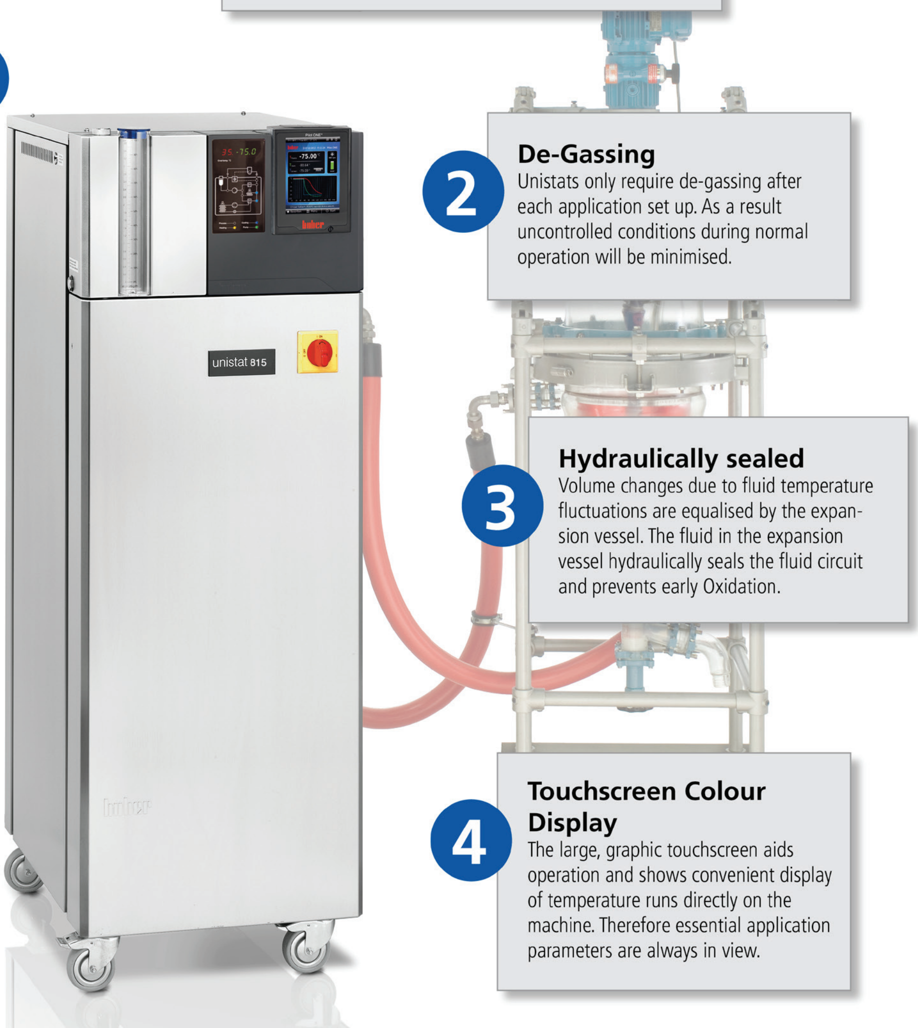
Chemical and pharmaceutical production plants, often use central cooling and heating systems in production processes for temperature control. The Unistat Hybrid system optimises existing systems by connecting a hydraulically sealed temperature control unit. The available cooling and heating capacity 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

Practical Advantages

Advantages:



1

Process Safety
Unistats provide an option to allow the circulation pump and compressor to continue to work despite an over temperature trip. This allows controlled heat removal and protects your thermally controlled products from being destroyed.

2

De-Gassing
Unistats only require de-gassing after each application set up. As a result uncontrolled conditions during normal operation will be minimised.

3

Hydraulically sealed
Volume changes due to fluid temperature fluctuations are equalised by the expansion vessel. The fluid in the expansion vessel hydraulically seals the fluid circuit and prevents early Oxidation.

4

Touchscreen Colour Display
The large, graphic touchscreen aids operation and shows convenient display of temperature runs directly on the machine. Therefore essential application parameters are always in view.

5

Data-Communication
Unistats offer numerous possibilites for data communication. RS232, Ethernet and USB interfaces are fitted as standard, as well as various analogue interfaces.

6

Temperature Control
The intelligent temperature control TAC analyses the controlled fluid circuit continuously, and adjusts the control parameters automatically. The result is the best control results even with difficult applications.

7

Pressure Control
The pressure control VPC continuously monitors the pressure in the connected application and therefore protects the sensitive glass reactor from breakage.

8

Power / Volume
Unistats have a high power to volume ratio (Watts/Litre). In practice, Unistats offer a very high speed of temperature change in the region of several hundreds of Kelvin per hour.

9

Space saving design
Unistats have a compact design requiring little space. The power to volume ratio (Watts/cm^3) according to DIN 12876 documents the extremely small space requirement of the Unistats.

10

Heat Transfer
Powerful circulation pumps and a large hose cross section ensure maximum flow rates and optimum heat transfer.

Case Study: Unistat® 615w

Heating & cooling a Buchi 250 litre glass lined stainless steel reactor through 60K

Requirement

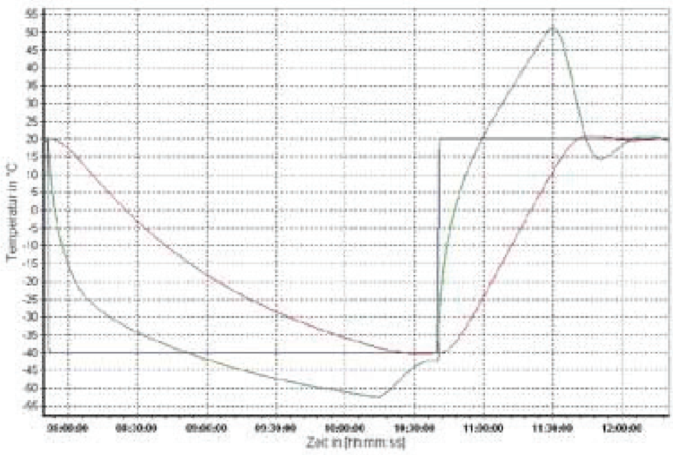
This case study shows the remarkable power transfer capabilities of the Unistat range in using a Unistat 615w to heat and cool a 250-litre Buchi Glas Uster GLSS reactor.

Method

The Unistat was connected to the reactor using two 2 metre insulated metal hoses. The reactor was filled with 200 litre of Ethanol.

Results

The Unistat cools the process from 20 °C to -40 °C (60 K) in approximately 150 minutes. It can be seen from the jacket temperature that the system is "comfortable" with this load. The heat up time back to 20 °C takes approximately 60 minutes.

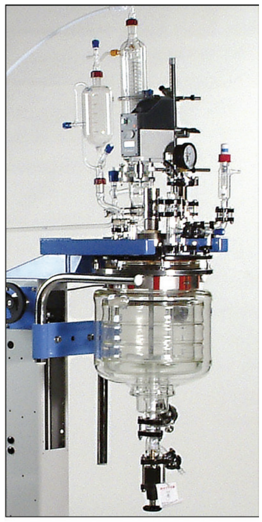


Setup details

Temperature range:	–60...200°C
Cooling power:	9.5 kW @ 200…0°C 8.0 kW @ -20°C 4.8 kW @ -40°C 1.2 kW @ -60°C
Heating power:	12 kW
Hoses:	M38x1,5; 2x2 m
HTF:	DW-Therm (#6479)
Reactor:	Buchi Glas Uster CR252 250 litre glass lined (enameled) steel reactor
Reactor content:	200 litre Ethanol
Reactor stirrer speed:	90 rpm
Control:	process

Case Study: Unistat® 1005w

Cooling an Asahi 10 litre triple wall reactor to -110 °C



Requirement

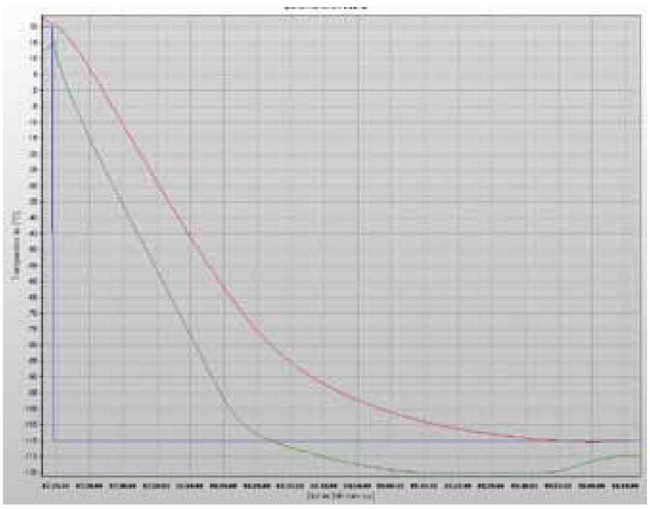
Temperatures required to carry out chemistry in specialised cryogenic research have become lower and lower. This case study demonstrates that the process temperature inside an Asahi vacuum insulated glass reactor can be comfortably cooled and controlled at -110°C by using a Unistat 1005w.

Method

The Asahi reactor was connected to the Unistat 1005w using two 1.5 metre insulated metal hoses. Under process control, a process set-point of -110°C was entered and the results recorded using the Huber "SpyControl".

Results

The jacket temperature cools quickly in a linear fashion to -100°C in approximately 55 minutes ramp rate of 2 K/min) before slow.



Setup details

Temperature range:	-120...100°C
Cooling power:	1.5 kW @ 100…-40°C 1.4 kW @ -60… -80°C 1.0 kW @ -100°C
Heating power:	2.0 kW
Hoses:	2 x1.5 m; M30x1.5 (#6386)
HTF:	Kryothermal S
Reactor:	10 litre insulated jacketed glass reactor
Reactor content:	10 litre M90.055.03
Stirrer speed:	~ 200 rpm
Control:	process

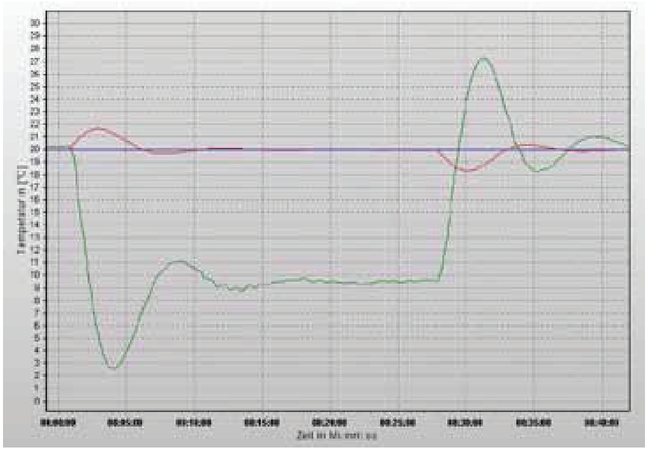
Case Study: Unistat® 510w

Controlling a simulated 300 W (258 kcal / hr) exothermic reaction in a 15 litre Buchi Glas Uster reactor.

Requirement
This case study shows the temperature profile of a specific test while undergoing a simulated exothermic reaction.

Method
A Unistat 510w has been selected to control the process temperature inside a 5 litre glass lined (enameled) steel reactor which is 2/3 filled with M20.235.20.

Results
The 300 W exothermic reaction increases the process temperature by approx. 1.7 K and the Unistat compensates the temperature difference in 9 minutes. After a while the heater is removed out of the reactor and the process temperature goes down to approx. 18.3°C. The Unistat takes 12 minutes to bring back the process temperature to its set-point.



Setup details
Unistat® 510w & Buchi Glas Uster reactor

Temperature range:	-50...250°C
Cooling power:	5.3 kW @ 0…250°C 2.8 kW @ -20°C 0.9 kW @ -40°C
Heating power:	6.0 kW
Hoses:	2x1.5 m; M30x1.5 (#6386)
HTF:	DW-Therm (#6479)
Reactor:	15 litre glass lined (enameled) steel reactor
Reactor content:	10 litre M20.235.20 (#6162)
Stirrer speed:	80 rpm
Control:	process

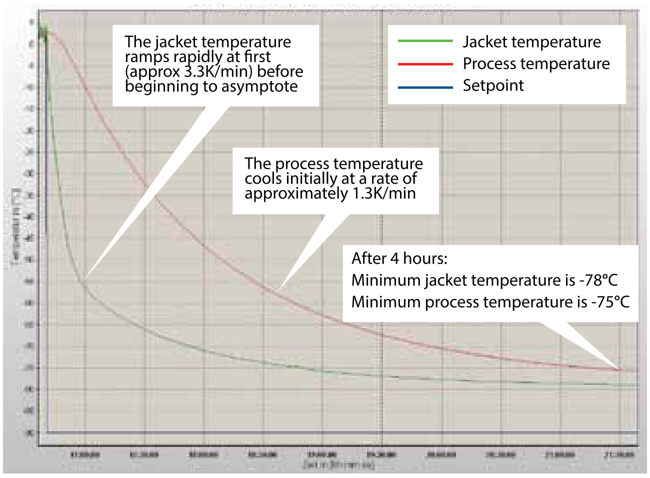
Case Study: Unistat® 910w

Cooling a Diehm 100 litre jacketed glass reactor to Tmin

Requirement
This case study looks at the minimum process temperature that a Diehm 100 litre reactor can reach when connected to a Unistat 910w.

Method
The Unistat and reactor are connected using two 1.5 meter insulated metal hoses. The reactor is filled with 75 litre of "M90.055.03", a Huber supplied silicon based HTF.

Results
A 100 litre reactor represents a large thermal load for the Unistat 910w which is designed to operate on reactors of up to 50 litres however, over time the Unistat 910w can still bring the process temperature to -75°C.



Setup details
Unistat® 910w & Diehm 100 litre reactor

Temperature range:	-90…250°C
Cooling power:	5.2 kW from 250°C to -20°C 4.7 kW @ -40°C 3.1 kW @ -60°C 0.9 kW @ -80°C
Heating power:	6.0 kW
Hoses:	M38x1.5; 1x 2m #6657; 1x1m # 6655, VPC Bypass installed
HTF:	M90.055.03 (#6259)
Reactor:	100 litre Diehm un insulated jacketed glass reactor
Reactor content:	75 litre M90.055.03
Stirrer speed:	410 rpm
Control:	process

Contacts

Labtex is a specialist laboratory and process chemistry equipment solutions provider. A dynamic team of professionals with expertise in research and process development across a wide range of industries and disciplines.

You will find that talking to Labtex is a breath of fresh air. No complicated telephone system, just real people ready to react professionally to your enquiry. We pride ourselves on our customer service based on quick response, accurate informed advice and attention to detail.

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