

# Calo2310 eco - base - pro

The New Generation Reaction Calorimeter for  
iso and non-isothermal Heatflow and  
Heatbalance Calorimetry

**SYSTAG** Login

Experiment: ..hydrid\_Adiabatisch\Halbes\_Mol\_bei\_20Grad\ 05:32:39 Experiment  
Recipe: ADIA-Control\Acetanhydrid\_adiabatisch State  
Phase: 17 Auf Reaktionsende warten 00:01:47 Report

HF<sub>Raw</sub> -0.03 K TR 36.21 °C  
HB<sub>Raw</sub> -0.19 K  
T<sub>J In</sub> 36.55 °C

On-Line Evaluation

Power	Heat
HF 3.6 W	27.3 kJ
HB 6.1 W	30.0 kJ
Ref 0.0 W	

Cal 0.0 W 3.00 kJ  
mass 475.5 g cp 2.51 J/g  
A (t) 0.0352 A (t-1) 0.0350 m<sup>2</sup>  
U (t) 123.10 U (t-1) 114.01 W/m<sup>2</sup>  
cp(t) 2.54 cp(t-1) 2.47 J/g

Integration OFF Calli  
Integration

Wenn Reaktion beendet, weiter  
Continue

Δp 0.205 bar

Adiabatic Run

ΔT 0.54 K  
T Cool In 24.4 °C  
T Head 23.8 °C  
T Amb 24.3 °C  
T Cover 45.19 °C  
T J Out 36.35 °C  
HF Raw -0.03 K  
HB Raw -0.19 K  
T J In 36.55 °C

Press Vent Vac  
p Reactor 1.176 bar  
Δp 0.205 bar  
Speed 100 rpm  
Torque 18.9 Ncm  
7.0 pH pX  
36.21 °C HR  
0.0 W C  
38.5 °C

0.0 g tare  
51.1 g Dos1 tare  
0.0 g

Screen Setup Sensors Dosage Table Historical Display  
Database Fine Tuning HTD Default

Launch

AIN's DOT's 25.02.2005 14:56:00 System Quit Exit

HB Refl  
HF  
HB



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## Editorial

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Peter E. Meier, Dipl.Eng. (ETH) is the President of SYSTAG and played a decisive role in the development of the Calo 2000.

The re-development of a universal calorimeter started in 1998 by SYSTAG and reached a successful conclusion with creation of the Calo2310 family.

The goal was to develop a calorimeter that would enable the client to obtain the most realistic, synchronous image of a process being evaluated. This information, in turn enables the user to base his decisions on accurate measurements instead of risky assumptions.

Based on a model-supported principle, the new Calo2000 family will meet all requirements encountered in practice. More than a thousand experiments conducted during the development stages have proved that the instrument is extremely robust and convergent at all times.

During the past seven years, the process has been extensively refined and developed into a fully matured series that meets a variety of requirements at an affordable cost.

We like to mention in particular the excellent kinetic properties of the model-supported versions of the Calo2310 base and pro that provide a true reflection of the reaction output.

Special attention was given to creating software that is user friendly, easy to operate and complies with the requirements of FDA Guideline CFR21 Part 11. The system can be certified if required. As you can see, your investment is already well protected for future requirements.

Naturally, the reaction calorimeters of the Calo2310 series are, in their own right, automated laboratory reactors of the highest quality. They can be equipped with different accessories to progress from a simple isothermal to non-isothermal heat-flow reaction calorimeter to a combined, non-isothermal heat balance and heat-flow calorimeter.

Upgrading to a more powerful version can be performed at any stage.

Do not hesitate to contact us for mutual discussions to help you. Your problem is our challenge.

Yours  
Peter Meier

## Introduction to Reaction Calorimetry

### Historical Background

Reaction calorimetry was developed mainly in the Basle chemical industry during the 1960-1980 eras by Dr. Willy Regenass of Ciba and Dr. Ludwig Hub of Sandoz. Right from the beginning, SYSTAG supported development and acquired vast technical know-how in due course. This know-how was, after patent rights had expired, slowly but surely used for the introduction of new products and above all for development.

### Current Technical Level of Reaction Calorimetry

It is not surprising that no new basic technologies were developed. The principles used in the heat-flow calorimeter developed by Dr. Willy Regenass (later sold under the name of RC1 by Mettler-Toledo) and the combined heat-flow and heat balance calorimeter developed by Dr. Ludwig Hub (later known as Contalab) are still valid. Unfortunately, in our opinion, little use was made of currently available technological advances.

A derivative was the Output Compensation Calorimeter, (HEL, UK) which in principle is nothing more but a heat-flow calorimeter. All problems that are inherent in a heat-flow calorimeter still persist.

### Why is a New Generation of New Reaction Calorimeters Needed?

As their weak points, that have existed since the 80's were never successfully and industrially viable tackled, it was SYSTAG's goal in 1998 to develop a new generation of instruments.

### Required Capabilities

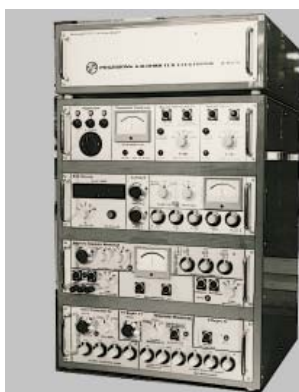
An evaluation of many investigations under practical conditions showed clearly that most users cannot work isothermally only, as the course of a reaction in a production process comprises often a combination of ramps, isothermal or isoperibolic phases and changes to reflux.

An important point to consider was the development of a method that delivers reliable measurements at varying temperatures. Of utmost importance was the ability to conduct reliable calibrations and measurements for all sizes and designs of reactors. The shape of the reactor should be as close as possible to the actual production unit to be developed. A highly accurate kinetic function (within a minutes range) should be displayed.

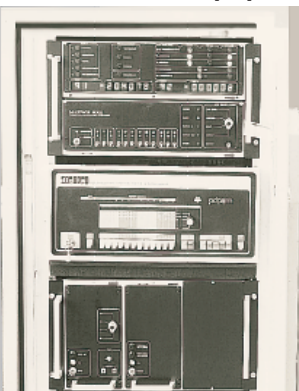
Based on the abovementioned reasons it became SYSTAG's declared goal to reduce weak points within Heat-flow Calorimetry by supplementary methods. A meticulous analysis of all methods that could fulfill these needs was conducted.

### Various generations of Ciba-Geigy/Systag calorimeter control units

1966: WFK 66



1974: WFK 74/pdp8



1975: WFK 75



1981: BSC 81



## Problems related to Heat-flow Calorimetry

### General Problems in Heat-Flow Calorimetry

Heat-flow calorimeters, with all their advantages, also possess some significant drawbacks. Most experiments had to be conducted under isothermal conditions to yield acceptable accuracy although isothermal conditions are seldom encountered in practice. Various approximation methods were used to overcome this hurdle; the success was questionable. The following limitations fall into these categories:

1. **Liquid Level Changes during Dosing:** Heat exchange surface areas changes.
2. **Viscosity Changes during Reaction:** Heat conductivity U changes.
3. **Stirrer Speed Variations:** Heat conductivity U and surface A change.
4. **Condensation on Reactor Lid:** Heat loss factor to surroundings not taken into account.
5. **Heat of Evaporation:** Heat loss and possible loss of material not taken into account.
6. **Unstable Baseline during Temperature Ramping:** Changes due to radiation, change of water value as well as density and cp of sample.
7. **Change to Reflux Phase cannot be Measured:** Extremely complex phase change including 3 additional water values (Lid, Condenser Tube and Reflux Condenser).
8. **Crystallisation on Reactor Wall:** Extreme change of heat conductivity U.

### Problems Relating to Power Compensation Calorimetry

With the exception of faster reactor control, this method offers only limitations:

- A. It is **impossible** to measure reaction output that is **greater than** the **heater input**.

- B. The **heating coils are inside the reactor**, and create **hot spots**. This can cause unwanted chemical reactions.
- C. Although the heat output is directly measured, the chemical output **cannot be** directly interpreted, as the heat transfer due to the heat flow technique principle is not constant and does not remain stable (See previous section).

For the above mentioned reasons, a method based on power-compensation calorimetry was excluded as it contained too many restrictions.

### Causes of Heat-Flow Measuring Errors

The measurement is based on the principle of a defined heat transfer. The reactor wall between the internal reactor space and the jacket for the heat exchange medium is used. The temperature difference is the measure of the heat-flow; together with the surface the total heat-flow is obtained.

As many users know from practical experience, measurements become unreliable as soon as a viscosity change takes place, crystallisation on the reactor wall occurs or a variable vortex is formed. In the first two instances a change of heat transfer „U“ takes place due to a viscosity increase of the liquid film or even solidification on the reactor wall. A varying vortex changes the transfer area „A“ which in turn will change the heat-flow volume.

This would now be corrected to a large degree by means of calibration heating, before and after an experiment. Provided all processes are linear, the results would be correct. Unfortunately this is not the case.

The cause of the errors stems from a basic problem: The probes and the heat transfer characteristics are influenced by the chemical reactions themselves. It is a fact that the measuring element forms an integral part of the system to be

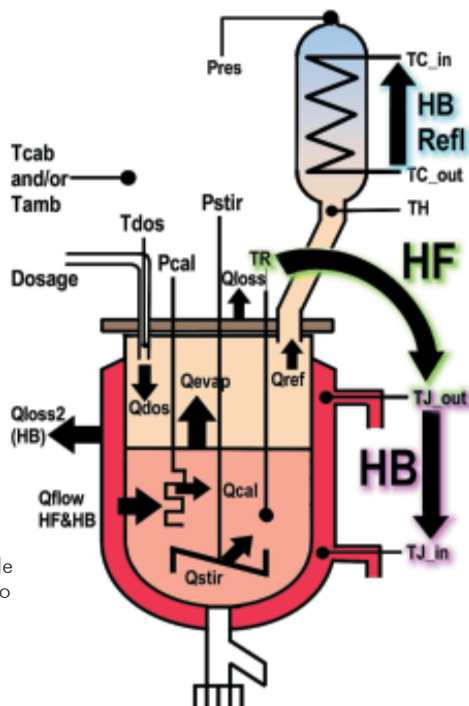
## Conclusion

measured. It is, therefore, impossible to eliminate this problem. An alternative measuring process has to be found.

The heat balance method measures only the flow and the resulting temperature difference in the outer jacket of the reactor between inlet and outlet points. Using flow, cp and the density of the heat transfer medium the output and resultant energy can be calculated.

Chemical reactions and the resulting variations do not have an influence on the measuring system as it is positioned outside the inner reactor space. Although the heat transfer liquid is not absolutely stable, properties are known and can be determined, this is not always the case when dealing with reaction products!

*Based on the results obtained from these investigations only the heat-flow and heat balance methods remained as viable options.*



**Illustration 6**  
Schematic principle of a complete Calo 2000, showing all measuring points and influencing factors.

## Considerations Regarding Scale-Up

When scaling up to a large volume, the calorimeter must not only provide accurate total energy measurements but also the current output of a reaction (e.g. the start of an exothermic reaction) to avoid uncontrolled cooling situations, during which large energies are released.

Let us examine accurately the size relations: Measurements are performed in a 1! reaction calorimeter. The obtained results are projected to a 5000 ! reactor. Assuming that the dosing rate remains proportionately the same (5000 times greater per time unit), the output will also be 5000 times greater. This output energy has to be removed. Unfortunately, the heat exchange area of a 5000! reactor does not increase in the same ratio. As an approximation we can use  $5000^{2/3} = 292$ . This means the output increases relative to the exchange surface area by a factor of 17 ( $5000/292=17$ )

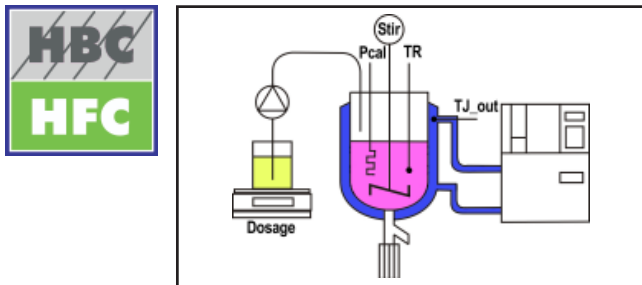
It is of great significance whether you are measuring 50 or 60 Watt, as this translates to 250 000 or even 300 000 Watt on a larger scale with corresponding cooling costs.

Likewise, in a 1! reactor 50 Watt will result in a temperature difference of for example 7K. With a reduced surface area this converts at 5000! to:  $7K \times 17 = 119K$ . This is most certainly extremely safety relevant.

These two examples demonstrate that scaling up can only be carried out reliably and optimally with accurate basic measurements. Any uncertainties will lead to excessive safety reserves, which are hardly acceptable in financial terms. This example demonstrates that the cost of a reaction calorimeter is quickly amortised if instead of an energy requirement of 200kW only 100kW have to be provided. One could obviously reduce the dosing rate by half; however, this would increase production time significantly, thereby increasing costs with regard to amortisation per production volumes/year.

## Measuring Principle

### The traditional Heat-Flow Calorimetry (HFC)



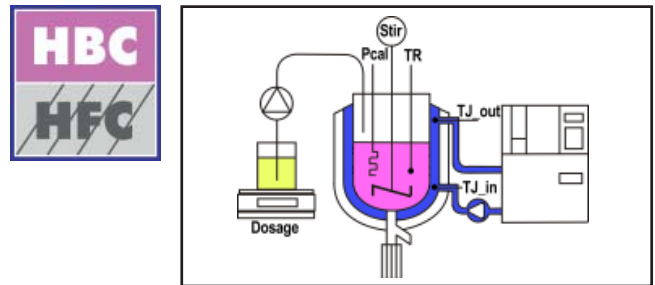
Equation:

$$HF [W] = (TR - TJ\_Out) [K] * A [m^2] * U [W/m^2.K]$$

**HF:** Heat Output  
**TR:** Reactor Temperature  
**TJ\_Out:** Jacket Temperature - Outlet  
**A:** Heat Exchange area between Test material and Reactor Jacket  
**U:** Heat Transfer Coefficient

The up to now used **Heat-flow Calorimetry (HFC)** is an extremely robust measuring method providing a very good signal. The jacket protects the measuring system to a major degree against external influences. However, measurements are extremely dependent on liquid level, viscosity and vortex in the reactor. Any accumulation of solid materials near the reactor wall causes serious consequences due to massive changes in the transfer coefficient which seldom remains linear.

### Heat Balance Calorimetry at the Reactor (HBC)



Equation:

$$HB [W] = (TJ\_Out - TJ\_In) [K] * cp [J/g.K] * r [g/ml] * F [ml/s]$$

**HB:** Heat Output  
**TJ\_In:** Jacket Temperature at Inlet  
**TJ\_Out:** Jacket Temperature at Outlet  
**cp:** Specific heat of the Heat Exchange Medium  
**r:** Density of the Heat Exchange Medium  
**F:** Flow

**Heat balance calorimetry (HBC)** has the major advantage that any changes on the transfer between reactor contents and jacket become insignificant. Looking at it from this angle it is the ideal method. We must, unfortunately mention some disadvantages: The measured signal is considerably smaller and the surrounding temperature has a greater influence on the results of the measurements. In addition, the water value for non-isothermal measurements is considerably larger than the heat-flow (HFC) method. However, both methods combined are an unbeatable concept.

### Heat Balance Calorimetry at the Reflux Condenser

Heat balance carried out at the reflux condenser is practically identical, even in relation to the water value of the reflux condenser (!), to HBC measurements in the reactor (see top right column). A major advantage is the relatively stable operating

temperature of the cooling medium, only weak isoperibolic conditions prevail, making evaluations somewhat easier. However, a change from a non-boiling phase to a boiling phase makes an accurate evaluation extremely difficult.

## Characteristics of HFC and HBC

### Differences between Calo2310 eco - base - pro

Reaction	Calo2310 eco	Calo2310 base	Calo2310 pro
<b>Events</b>	<b>Isothermal HFC</b>	<b>Non-isothermal HFC</b>	<b>Non-isothermal HBC &amp; HFC</b>
Change of surface a) during semi-batch reaction b) change in vortex	In both cases linear interpolation with two calibration heatings by KaloGraph in offline mode.	Based on model creation, surface is automatically calculated and correct-ed (also online). Average vortex can be entered into USR table.	Based on model creation, surface is automatically calculated and corrected (also online). Average vortex can be entered in USR table.
Influence of warm additions (e.g. during low temperature calorimetry)	Dosing above liquid level with high drop is more difficult to compensate than short drop dosing. Below-level dosing ideal and easily compen-sated.	Dosing above liquid level with high drop is more difficult to compensate than short drop dosing. Below-level dosing ideal and easily compen-sated.	Above-level dosing, even at high drop fully compensated by HBC.
Condensation on Reactor Lid	Results in an error. Partially compensated due to isothermal operation and two calibrations.	Results in error in non-isothermal operation despite two calibrations.	No noticeable influence due to lid heating.
Changes in Viscosity during Reaction	Linear compensated with two calibration heatings by KaloGraph. Permissible ratio approx 1:2.	Can only be corrected with two calibration heatings (as in eco). Permissible ratio approx 1:2.	During HBC no noticeable influence most suitable for polymerisations. Any configuration is acceptable.
Changes in Boiling Point during Reaction	As reaction temperature remains stable, small errors due to evapo-ration changes will be noticed. Linear compensated with two calibration heatings by KaloGraph.	Due to closed system no measurable changes of evaporation output, resulting in excellent measuring performance.	Due to closed system in HBC & HFC no measurable changes of evaporation output, resulting in excellent measuring performance.
Crystallisation during Reaction or Dissolving Crystallisation, Measurement during Ramping	Can instantly change heat transfer U. Results can be wrong by large factors. No measurements on ramps – non-isothermal.	Can instantly change heat transfer U. Results can be wrong by large factors. Limited ramping measure-ments, provided reactor walls remain free from crystallization.	In HBC mode almost no changes noticeable. Good results even during very high crystallization levels (e.g. up to 30-50% of volume) HBC ex-tremely robust. Ramping okay.
Online cp Measurement	Separate process during calibration. Despite HFC mode acceptable accuracy, due to mostly stable conditions.	Separate process during calibration. Despite HFC mode acceptable accuracy, due to mostly stable conditions.	Separate process during calibration. Despite HFC mode acceptable accuracy, due to mostly stable conditions. HBC not being used.
Influence of Ambient Temperature	HFC well screened by jacket, little interference.	HFC well screened by jacket, little interference.	HBC well screened due to vacuum jacket, which provides extreme protection also to HFC. Lid heating reduces most interfering factors from above for HBC and HFC significantly.
Dependency from Thermostats	Very independent, provided the thermostat has fast control char-acteristics (between 5 . . . 10 K/min).	Very independent, provided the thermostat has fast control char-acteristics (between 5 . . . 10K/min). Must be incorporated into model.	Very independent, provided the thermostat has fast control char-acteristics (between 5 . . . 10 K/min). Additional lid thermostat ensures excellent screening from external in-fluences even at extreme tempera-tures.

# Calo2310 eco

## Isothermal Heat Flow Calorimeter (HFC)

### Characteristics

The heat-flow calorimeter **Calo2310 eco** provides an economical solution at entry level. Simple operations combined with high functionality were made a top feature. Complex recipes can be realized using integrated recipe control for fully automatic operation.

Individual phases can be initiated or interrupted not only time but also event controlled. Apart from full automated operation by means of a recipe, experiments can be carried out manually, in step by step mode. When conducting first time experiments operation via synoptic is highly valued. When executing manually controlled experiments it is of course possible to perform a calorimetric evaluation. All recorded data are protected against unauthorised changes.

The system can be upgraded to a non-isothermal heat-flow Calo2310 base at a later stage.

### System Configuration

Reactor	1 l double jacket, cylindrical
Stirrer	Heidolph, with anchor stirrer, 40-2000 rpm with torque measuring facility
Dosing	1 x gravimetric from balance
Thermostat	Unistat Tango -40°C... +200°C
Temp. measurement	TR and TJ
Calorimetry	Heat flow calorimetry in reactor
Calibration heating	20 Watt, with supply and measurement
Temp. Control	Jacket and reactor control
Dosing Control	Direct connections for balance and pump
Reflux	Standard reflux condenser installed
Construction	Portable trolley for fixed cabinet
Control System	Optimally equipped COMBILAB System with PC for Windows XP with Office, FIX, ElemX for setup, OperX for recipe and SysGraph with Module KaloGraph for evaluation.
Software	CFR21 Part 11 compliant

### Options

Dosing	2 <sup>nd</sup> gravimetric dosing from balance measured
pH	Control from 2 <sup>nd</sup> dosing
pH	Control from 2 <sup>nd</sup> dosing
Pressure	Pressure, vacuum, venting, measured and controlled
Reflux	Heat balance calorimetry within reflux condenser
Distillation	Magnetic splitter before reflux condenser
Temp. Range	Alternative ranges and thermostats available
Software	SecureX, IQ/OQ - FAT/SAT

### Standard Configuration

1280x1024 LCD Colour Monitor  
with synoptic and graphics

Recipe Control OperX

1 l Double jacketed reactor



Unistat Tango

Combilab Control Unit



### Options

Pressure/Vacuum/Venting

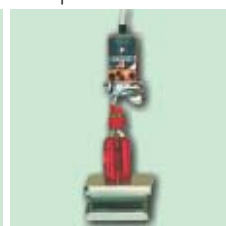
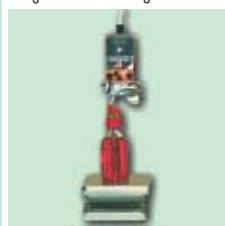
Reflux Condenser, Reflux Splitter,  
Distillate Cooler & Balance



1 x gravimetric dosing

2<sup>nd</sup> gravimetric dosing,  
also for pH

Pressure reactor



# Calo2310 eco Capabilities

## Technical Data



**NOTE:** Typical values obtained at ambient temperature (+25°C).  
Most measurements made with a 700ml volume of ethanol over a range of +5 ...+50°C.

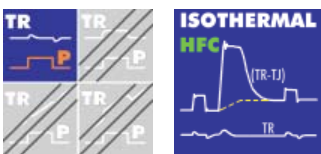
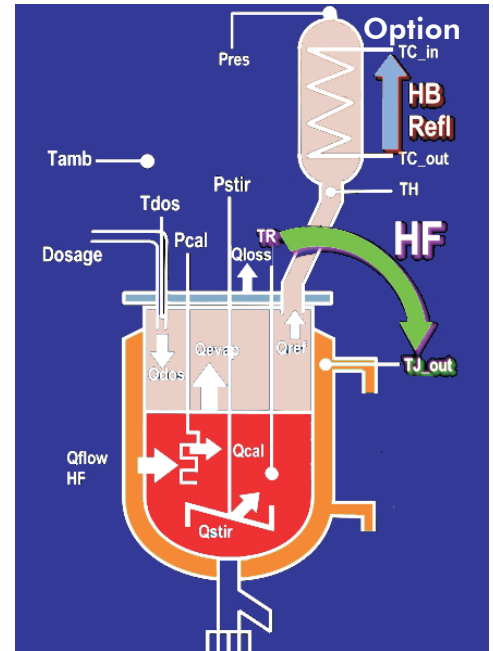
Thermostat Unistat Tango (HTF190) .....	-40°C .....	+200°C
Temperature range Jacket (TJ) .....	-30°C .....	+190°C
Temperature range Reactor (TR) .....	-20°C .....	+180°C
Typical HFC heat value – Reactor ..	approx. 0.200 K/W	
Typical HBC heat value Reflux (20 l/h H <sub>2</sub> O) .....	approx. 0.045 K/W	
Resolution of TR, TJ, dTC .....	0.007 K	
Resolution – remaining temp. values .....	0.007 K	
Sensitivity HFC, reactor, isothermal .....	>500 mW	
Sensitivity HBC, reactor, reflux, isothermal .....	>1000 mW	
Dynamic, HFC, reactor, isoperibol .....	<400 W (short term)	
Dynamic, HBC Reflux, isothermal .....	<400 W (short term)	
Reactor – Time Constant HFC, Reactor .....	ca. 900 s	
Reflux – Time Constant HBC, Reflux .....	ca. 600 s	



**Accuracy:**

HFC, Reactor, isothermal with calibration .....	4 %
HBC, Reflux, isothermal .....	10 %
cp Determination .....	automatic

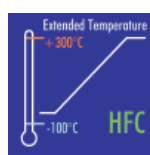
## Measuring Concept



**CFR 21 part 11**

**SecureX**  
Name:  
Keyword:

**IQ / OQ FAT/SAT**



## „Blue Window“ on-line display

On-Line Evaluation		
Power	Heat	
Cal	0,0 W	34,1 KJ
mass	635,0 g	cp 2,42 J/g+K
A (t)	0,0440	A (t-1) 0,0440 m²
U (t)	153,05	U (t-1) 122,75 W/m²
cp (t)	2,91	cp(t-1) 1,92 J/g+K
<b>Calibration ON</b>		
<b>Reset Values</b>		

Explanation of pictograms: Refer to Pages 24 & 25

# Calo2310 base

## Non-isothermal heat-flow HF Calorimeter

### Characteristics

The **Calo2310 base** is a sophisticated high performance heat-flow calorimeter of the non-isothermal type. High signal resolution of up to 100 mW (isothermal) can be achieved. The automatic „Zero Watt“ baseline (ref. Page 22) makes the selection of any type of baselines unnecessary. The instrument is ideally suited for all low viscosity reaction with little crystallisation.

The „Blue Window“ displays real-time results with an accuracy of approx. 10%. It is recommended to conduct experiments, where possible with two brief calibrations (approx. 15 min). The offline corrections can be used, if necessary to achieve high accuracy. This is very useful for the partial correction of possible viscosity changes or traces of crystallisation.

The perfect execution of complex, fully automatic recipes with manual interventions is the main strength of all instruments in the Calo2310 series. Apart from fully automatic operations, experiments can be conducted manually, step by step. This is highly valued during first time experiments. All data recorded are protected to prevent unauthorised changes. The system can be upgraded at any stage to a combined non-isothermal heat balance and heat-flow calorimeter Calo2310 pro.

### System Configuration

Reactor	1 l double jacket, cylindrical, sealed
Stirrer	Heidolph, 40 – 2000 rpm, basket stirrer (max. 200 rpm) torque measurement facility
Dosing	1 x gravimetric from balance
Thermostat	Unistat Tango -40°C... +200°C
Temp. measurement	TR and TJ
Calorimetry	Heat flow calorimetry in reactor
Calibration heating	20 Watt, with supply and measurement
Temp. Control	Jacket and reactor control
Dosing Control	Direct connections for balance and pump
Pressure	Pressure, vacuum, venting, measured and controlled
Reflux	Standard reflux condenser installed
Construction	Portable trolley for fixed cabinet
Control System	Optimally equipped COMBILAB System with PC for Windows XP with Office, FIX, ElemX for setup, OperX for recipe and SysGraph with Calo 2000 module, for evaluation.
Software	CFR21 Part 11 compliant
<b>Options</b>	
Dosing	2 <sup>nd</sup> gravimetric dosing from balance measured
pH	Control from 2 <sup>nd</sup> dosing
pH	Heat balance calorimetry with specially designed Condenser
Reflux	Magnetic splitter before reflux condenser
Distillation	Alternative ranges and thermostats available
Temp. Range	SecureX, IQ/OQ - FAT/SAT
Software	

### Standard Configuration

1280x1024 LCD Colour Monitor  
with synoptic and graphics



Recipe Control OperX



1 l Double jacketed reactor



Pressure/Vacuum/Venting



### Options

Reflux Condenser, Reflux Splitter,  
Distillate Cooler & Balance



Unistat Tango



Combilab Control Unit



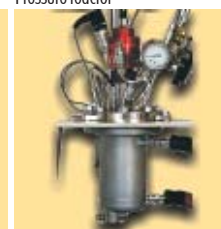
1 x gravimetric dosing



2<sup>nd</sup> gravimetric dosing,  
also for pH



Pressure reactor



# Calo2310 base Capabilities

## Technical Data



**NOTE:** Typical values obtained at ambient temperature (+25°C).  
Most measurements made with a 700ml volume of ethanol over a range of +5 ...+50°C.

Thermostat Unistat Tango (HTF190)	-40°C	+200°C
Temperature range Jacket (TJ)	-30°C	+190°C
Temperature range Reactor (TR)	-20°C	+180°C
Typical HFC heat value – Reactor	approx. 0.200 K/W	
Typical HBC heat value Reflux (20 l/h H <sub>2</sub> O)	approx. 0.045 K/W	
Resolution of TR, TJ, dTRJ, dTC	0.001 K	
Resolution – remaining temp. values	0.007 K	
Sensitivity HFC, reactor, isothermal	> 100 mW	
Sensitivity HBC, reactor, reflux, isothermal	> 500 mW	
Dynamic, HFC, reactor, isoperibol	< 1000 W (short term)	
Dynamic, HBC Reflux, isothermal	< 500 W (short term)	
Response 10%-90%, HFC, Reaktor	90 s	
Response 10%-90%, HBC, Reflux	90 s	

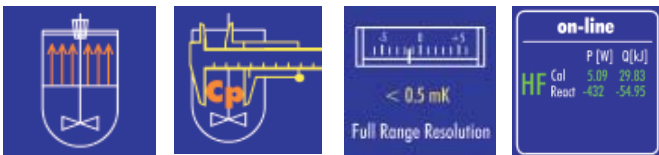
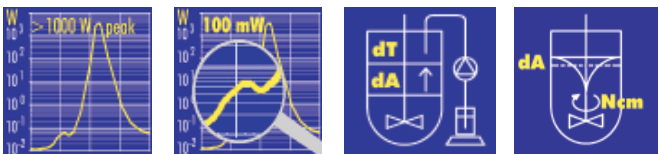
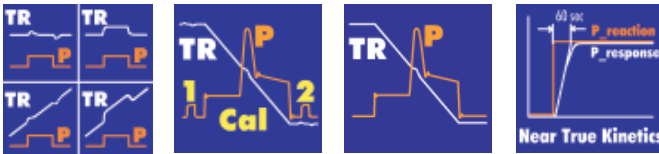
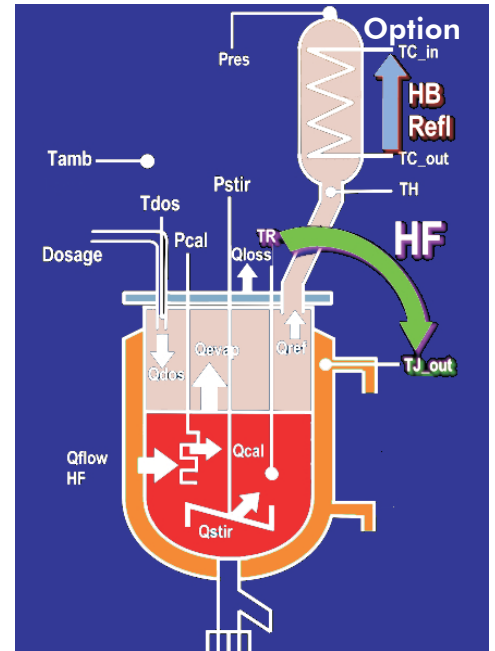
### Accuracy:

HFC, Reactor, isothermal with calibration	2 %
HBC, Reflux, isothermal	5 %
HFC, Reactor, isothermal without calibration (!)	10 %
HBC, Reflux, isothermal without calibration (!)	20 %
(!) Pre-calibrated, with pure solvent, downloaded as USR.mbd.	



Water value determination pre-calibrated, live accurately measurable  
cp Determination automatic

## Measuring Concept



**CFR 21**  
**part 11**

**SecureX**  
Name:  
Keyword:

**IQ / OQ**  
**FAT/SAT**

Extended Temperature  
+ 300°C  
-100°C  
**HFC**

## „Blue Window“ on-line display

On-Line Evaluation	
Power	Heat
<b>HF</b> -0,1 W	<b>33,1 kJ</b>
Ref 0,0 W	
Cal 0,0 W	34,1 kJ
mass 635,0 g	cp 2,42 J/g·K
A (t) 0,0440	A (t-1) 0,0440 m <sup>2</sup>
U (t) 153,05	U (t-1) 122,75 W/m <sup>2</sup>
cp(t) 2,91	cp(t-1) 1,92 J/g·K
<b>Integration ON</b>	<b>Calibration ON</b>
<b>Reset Values</b>	

Explanation of pictograms: Refer to Pages 24 & 25

# Calo2310 pro - Non-isothermal Heat Balance and Heat-flow (HB & HF) Calorimeter

## Characteristics

The **Calo2310 pro** is a sophisticated combined high performance, heat balance and heat flow calorimeter of the non-isothermal type. High signal resolution of up to 100mW (isothermal) is possible. The automatic „Zero Watt“ baseline obviates the selection of any other baseline. The problems commonly encountered with surface area variations, viscosity changes and variable vortex shapes caused by the stirrer are completely removed when using heat balance calorimetry. This instrument is the ideal solution for polymerisation and crystallisation processes.

The „Blue Window“ provides real-time results with an accuracy of approx. 10%. It is recommended to conduct experiments, where possible with two brief calibrations of approx. 15 minutes. The offline corrections provide, if required, extreme accuracy.

The optimal execution of complex, fully automatic recipes with an option for manual intervention is the major strength of all instruments in the Calo2310 series. Other than fully automatic operation, experiments can be conducted manually, step by step. This is highly valued during first time experiments. Secure data recording prevents unauthorised access.

## System Configuration

Reactor Stirrer	1 l triple jacket, conical, sealed Heidolph, 40 – 2000 rpm, basket stirrer (max. 200 rpm) torque measurement facility
Dosing	1 x gravimetric from balance
Thermostat	Unistat 380w -80°C... +200°C and CC302-3
Temp. measurement	TR, TJ_in, TJ_out, TT_in, TT_out, Tcovr
Calorimetry	Heat flow and heat balance calorimetry in reactor
Calibration heating	20 Watt, with supply and measurement
Temp. Control	Jacket and reactor control, reactor cover control
Dosing Control	Direct connections for balance and pump
Pressure	Pressure, vacuum, venting, measured and controlled
Reflux	Standard reflux condenser installed
Construction	Portable trolley for fixed cabinet
Control System	Optimally equipped COMBILAB System with PC for Windows XP with Office, FIX, ElemX for setup, OperX for recipe and SysGraph with Calo 2000 module, for evaluation.
Software	CFR21 Part 11 compliant
<b>Options</b>	
Dosing	2 <sup>nd</sup> gravimetric dosing from balance
pH	measured
pH	Control from 2 <sup>nd</sup> dosing
Reflux	Heat balance calorimetry with specially designed Condenser
Distillation	Magnetic splitter before reflux condenser
Temp. Range	Alternative ranges and thermostats available
Software	SecureX, IQ/OQ - FAT/SAT

### Standard Configuration

1280x1024 LCD Colour Monitor  
with synoptic and graphics



Recipe Control OperX



1 l Triple jacketed reactor  
(with low-temperature option)



Pressure/Vacuum/Venting



### Options

Reflux Condenser, Reflux Splitter,  
Distillate Cooler & Balance



Unistat 380w



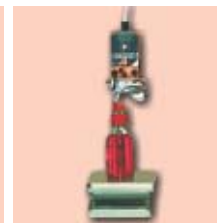
Combilab Control Unit



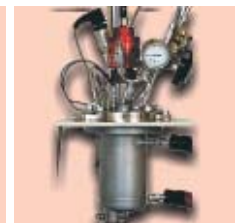
1 x gravimetric dosing



2<sup>nd</sup> gravimetric dosing,  
also for pH



Pressure reactor



# Calo2310 pro Capabilities

## Technical Data

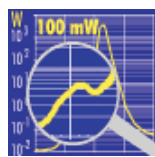
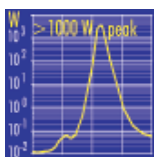
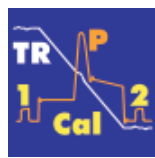
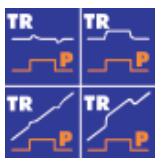
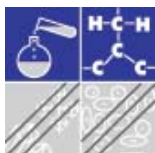


**NOTE:** Typical values obtained at ambient temperature (+25°C).  
 Most measurements made with a 700ml volume of ethanol over a range of +5 ...+50°C.

Thermostat 380w (HTF190) ..... -80°C ..... +200°C  
 Temperature range Jacket (TJ) ..... -50°C ..... +190°C  
 Temperature range Reactor (TR) ..... -40°C ..... +180°C  
 Typical HFC heat value – Reactor ..... approx. 0.200 K/W  
 Typical HBC heat value Reaktor ..... approx. 0.025 K/W  
 Typical HBC heat value Reflux (20 l/h H<sub>2</sub>O) ..... approx. 0.045 K/W  
 Resolution of TR, TJ, dTRJ, dTC ..... 0.0004 K  
 Resolution – remaining temp. values ..... 0.0070 K  
 Sensitivity HFC, reactor, isothermal ..... >100 mW  
 Sensitivity HBC, reactor, reflux, isothermal ..... >500 mW  
 Dynamic, HFC& HBC, reactor, isoperibol..... <1000 W (short term)  
 Dynamic, HBC Reflux, isothermal..... <500 W (short term)  
 Response 10%-90%, HFC & HBC, Reactor ..... 90 s  
 Response 10%-90%, HBC, Reflux ..... 90 s

### Accuracy:

HFC, Reactor, isothermal with calibration ..... 2 %  
 HBC, Reactor, isothermal with calibration (?) ..... 4 %  
 HBC, Reflux, isothermal ..... 5 %  
 HFC, Reactor, isothermal without calibration (1) ..... 10 %  
 HBC, Reactor, isothermal without calibration (1), (?) ..... 10 %  
 HBC, Reflux, isothermal without calibration (1) ..... 20 %  
 (1) Pre-calibrated, with pure solvent, downloaded as USR.mbd.  
 (?) Limited measuring range of TJ for exact HBC:  
 Heat exchange fluid HTF190 or DWTherm only from -20 < +190°C  
 Heat exchange fluid Marlotherm X only from -45 to < +160°C  
 Water value determination pre-calibrated, live accurately measurable  
 cp Determination ..... automatic

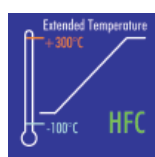


on-line		
	P [W]	Q [kJ]
HF Cal	5.09	29.83
HF React	-432	-54.95
HB Cal	5.21	31.56
HB React	-445	-56.35

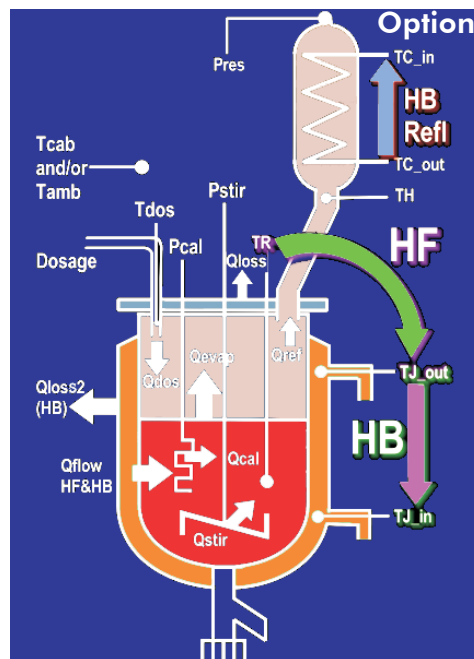
**CFR 21 part 11**

**SecureX**  
Name:  
Keyword:

**IQ / OQ FAT/SAT**



## Measuring Concept



## „Blue Window“ on-line display

On-Line Evaluation			
	Power	Heat	
<b>HF</b>	-0,1 W	33,1	kJ
<b>HB</b>	-1,1 W	27,9	kJ
<b>Ref</b>	0,0 W		
<b>Cal</b>	0,0 W	34,1	kJ
<b>mass</b>	635,0 g	2,42	J/g+K
<b>A (t)</b>	0,0440	<b>A (t-1)</b>	0,0440 m <sup>2</sup>
<b>U (t)</b>	153,05	<b>U (t-1)</b>	122,75 W/m <sup>2</sup>
<b>cp(t)</b>	2,91	<b>cp(t-1)</b>	1,92 J/g+K
<b>Integration ON</b>		<b>Calibration ON</b>	
<b>Reset Values</b>			

Explanation of pictograms: Refer to Pages 24 & 25

## Manual Operation and Recipe

### Manual Control

The manual control function is most important. A first time reaction can be carried out completely manually. The so called „Zero Recipe“ can be activated for an online display in the „Blue Window“ for the simultaneously display of Calorimetric data. An online calorimeter with a manual operation mode represents an absolute novelty.

### Partial and Full View of the Synoptic Monitor

For ease of reference only the essentials are displayed (Illustration 10), but full functionality is retained. If you, the calorimetric specialist, require more information, simply change to Full View (Illustration 11).

### Starting and Changing of Running Functions

The synoptic screen contains a number of keys, to open windows for the relevant functions by mouse-click (Examples as in Illustration 12). Relevant parameters such as Final value, Time duration can be entered here. The operation is initiated with

„Start“ held with „Hold“ and restarted with „Continue“ if a ramp is interrupted and continued. The „Off“ key will terminate the operation.

### Recipe Control

This most important function is of great significance for fully automatic operation. A recipe control consists basically of a sequence of individual Basic Operations. Up to 5 Basic Operations can be combined in parallel per step. As soon as these are completed, the next step is initiated until the final step has been reached. After completion all functions will be switched off.

When operating manually, it is always required that operations are supervised. Recipe control on the other hand allows unsupervised operation.

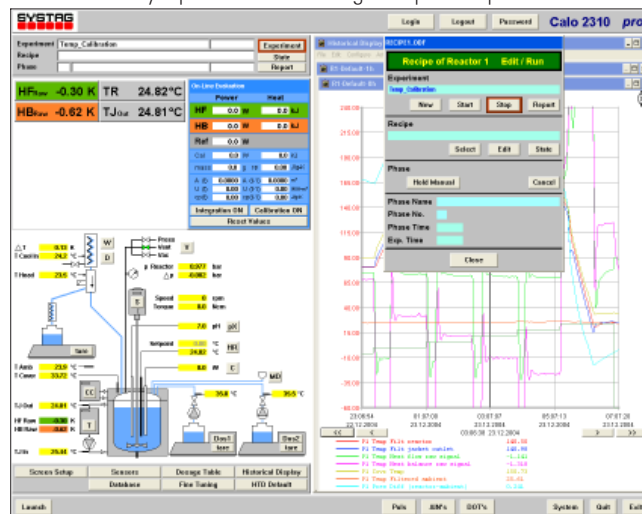
### Process Safety

As soon as limit values are exceeded, additions are stopped, stirrer and thermostat change into safety mode as defined in the recipe and the recipe is terminated. The user must define whether the stirrer continues or stops operating in an alarm situation.

Illustration 10 Partial View (Essentials only) of the synoptic screen



Illustration 11 Full View (Displaying complete details) of the synoptic screen including an open recipe window



## Basic Operations, Recipe and Data Capture

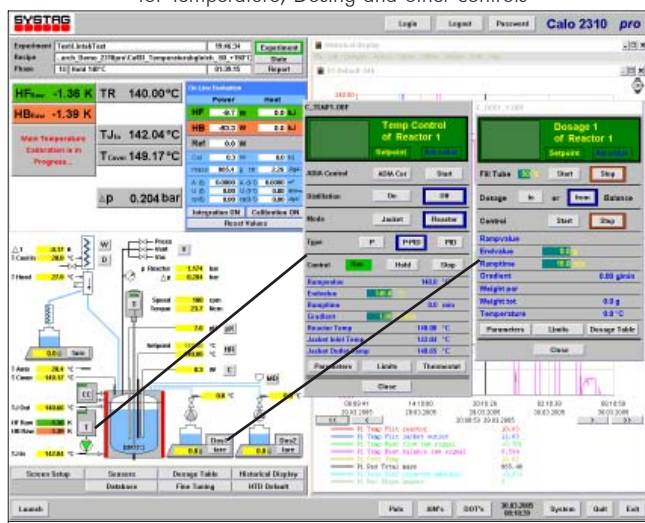
### User intervention at any stage

Whether operating in manual or recipe mode the user is always in full control and can change individual parameters or complete recipes, without terminating the recipe. This is particularly important if an experiment unexpectedly changes direction. Indications for these situations are provided by the online display. The versatile program structure and the flexible intervention options allow the use of the Calo 2310 in the very early phases, when optimizing processes, following the principle of „Faster time to market!“

### Basic Operations (Part)

- Stirrer (with definition for emergency alarm)
- Temperature control (jacket, reactor, cover) control parameters
- Calibration heating
- Heat integration and cp determination, combined
- Emergency alarms (TJ, TR, p and emergency set-point for thermostat)
- Dosing from balance 1 (and 2), dosing parameters 1 (and 2)
- pH Calibration, single sided pH control with second dosing control, as well as pH control parameters
- Automatic boiling point determination according to head temperature TH or TR
- Parameters for reflux splitter and distillation on balance 3, with parameters and termination criteria
- Pressure, vacuum and venting control, control parameters
- Digital In/Out (2 outputs)
- Timer, Wait Until, Loop as well If Then Else
- Report, Experiment Hold
- Experiment END

Illustration 12 Manual operation using windows for Temperature, Dosing and other controls



### Basic Operations in Recipe

Illustration 13 shows a recipe containing numerous phases, each Basic Operation is easily recognizable.

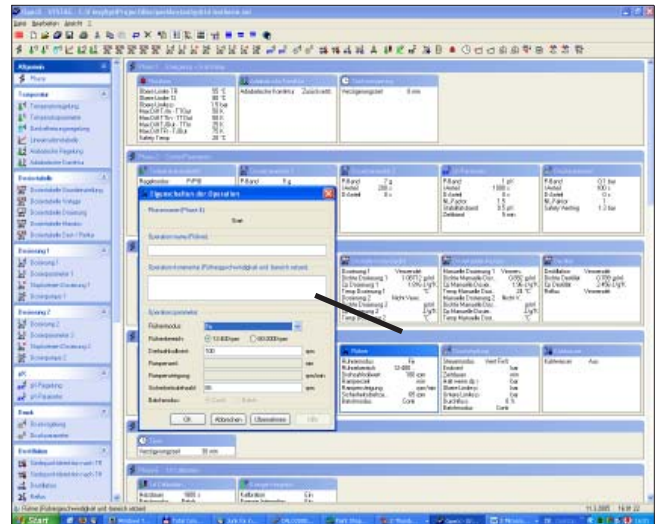


Illustration 13 OperX Recipe editor, on the left column containing Basic Operations, in the background individual BO's, one is opened for setting parameters.

### Reportfile

Manual entries (Ref. Illustration 26) and automatic messages are compiled in a report. Each message can initiate simultaneously up to 6 entries of different user selectable variables with date and time. This generates continuous reports, making a manual journal superfluous. (Ref. Page 25)

### Alarmfile

In the alarm file all interventions and recipe phases are recorded with date and time. This file can be sorted using a variety of criteria for display or printing. Every intervention can be followed up and examined. Artifacts can be identified as such, eliminating any guess-work.



## Sequences and Data Flow for off-line Evaluation

- Torque corrections
- Water value, separately for HFC and HBC each
- Reflux, cooling medium information
- Reflux, water value, cp feed and scaling factor
- Sequences and Dataflow for Offline Evaluation

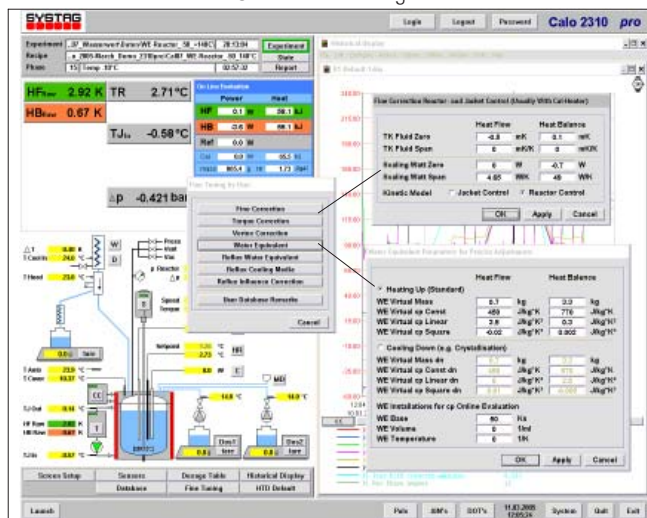
In the **Calo2310 eco**, only the dosing table can be accessed, the rest is fixed in the background. Only in the **Calo2310 base** and **Calo2310 pro** are the user-tables accessible and can be changed with a separate calibration run.

The System Table in all calorimeters cannot be changed by the user. It contains all Basic Parameters of the manufacturer's calibration.

Illustration 16 Dosing table



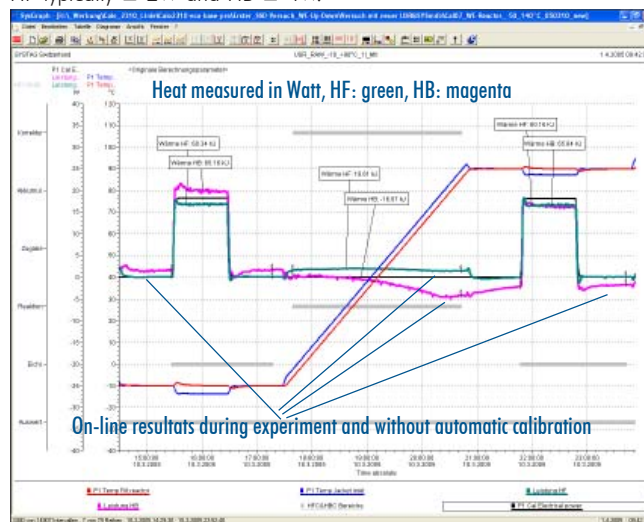
Illustration 17 User table showing some sub-windows



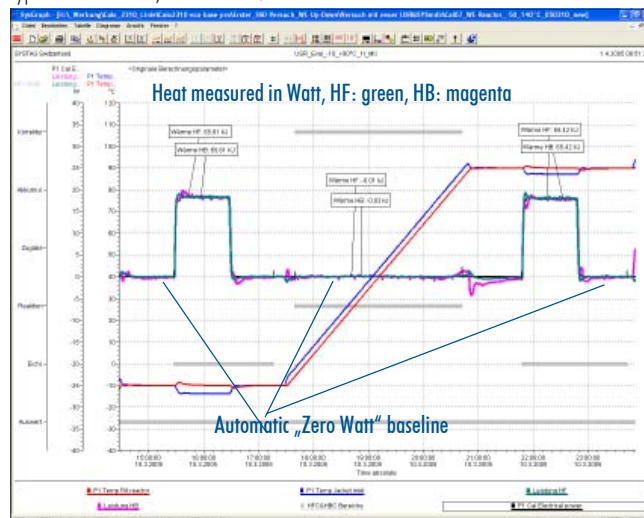
## System Calibration by the User

After an evaluation the user can store the new table with SysGraph and incorporate it as a new User Table. The system is now optimized for the specific experimental conditions. (For example: solvent only). This is a method to perform accurate operations without calibrations during an experiment.

**Illustration 18** A presentation of an evaluation with manufacturer's calibration only, without any adjustments by the user. Works calibration is optimized for -50°C to +150°C. Errors are within a few Watt; HF typically  $\pm 2$  W and HB  $\pm 4$  W.



**Illustration 19** User calibration in the range of -10°C to +90°C using a high-boiling alcohol  $\rightarrow$ ; automatic „Zero Watt“ baseline; typical HF  $\pm 1$  W, HB  $\pm 2$  W.



## SysGraph Evaluations: KaloGraph and Calo2000

### SysGraph

**SysGraph** is the universal graphics evaluation program supplied by SYSTAG. Two different modules are available:

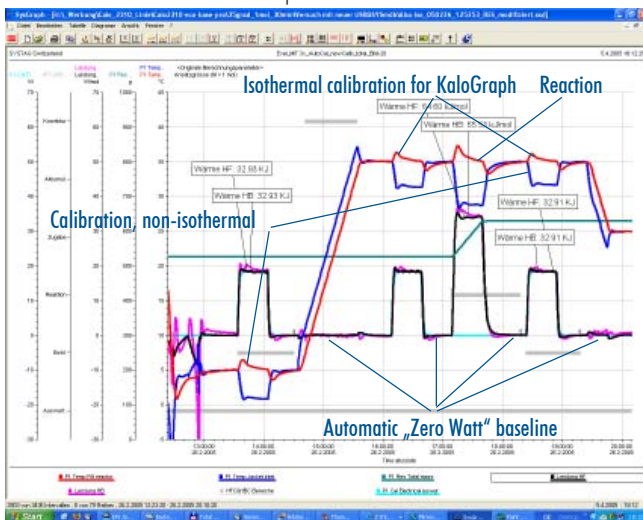
### KaloGraph for Calo2310 eco

The **KaloGraph** is used for isothermal heat flow calorimetry evaluations.

The evaluation must always be performed by means of calibration heatings; one before and one after the reaction. A manual baseline has to be determined as well.

This method is able to accommodate variations in viscosity, changes in reactor filling level, boiling point variations and changes in the cp value with reasonable accuracy. The only limitation is a linear interpolation of changes, which does not always apply. In most cases excellent results are obtained using purely isothermal heat flow calorimetry. A further limitation is presented in Illustration 21: The real-time behaviour in relation to the reactor time constant „tau“ which is approximately 10...15 minutes becomes obscured. The true kinetics are no longer accurately displayed, however, the energy content of the integral is extremely accurate.

**Illustration 20** Calibration at two temperature levels and automatic adjustment of the water value



The following example shows an alcoholysis experiment, evaluated by two methods: using KaloGraph (Illustration 21) followed by Calo 2000 (Illustration 22). The above mentioned differences are clearly highlighted.

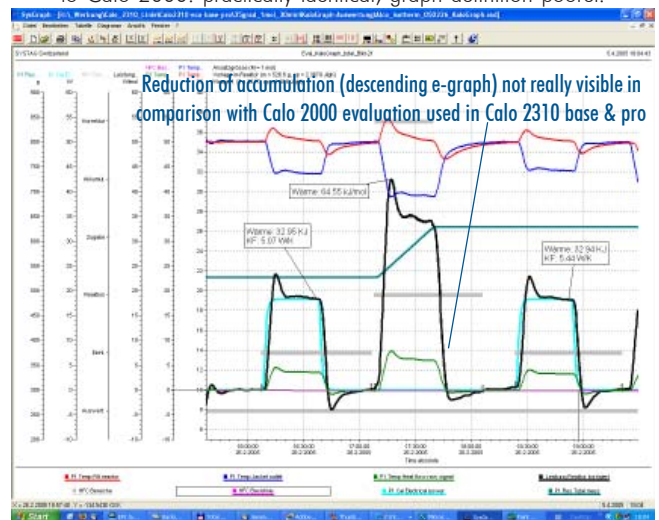
### Calo2000 for Calo2310 base and Calo2310 pro

The **Calo2000** Package is designed for non-isothermal heat-flow and heat balance calorimetry. A principle differentiation between three evaluation methods must be made:

1. Evaluation without automatic adjustment
2. Evaluation with automatic adjustment
3. Manual corrections of the user table (USR.mdb) and evaluation without automatic adjustment as an iterative process.

This can be explained as follows: As soon as a calibration by means of calibration heating is carried out prior and after the reaction, the user can calculate results using an automatic adjustment. The values are obtained from the table that was used during the experiment. An evaluation of the validity is performed. In particular, if the cp values or the water value of the instrument are not perfectly correct, an automatic adjustment is carried out and the new values are entered into the internal table. The new values

**Illustration 21** Evaluation with KaloGraph. Result compared to Calo 2000: practically identical, graph definition poorer.



can be stored for further calculations or alternatively exported with a keystroke as a new User table (Ref. Illustration 23). This table can be used for a fully calibrated operation without the need for a new calibration during the experiment.

### Example of an Evaluation

Illustration 20 shows an alcoholysis, during which the receiver was first calibrated at 10°C with 20W, followed by heating to 35°C. This step is required for the automatic water equivalent value determination but falls away when using the Calo2310 eco. The next calibration heating to 35°C is only introduced for demonstration purposes to illustrate an isothermal evaluation and is not necessary for Calo2310 base or pro. The subsequent reaction is followed by another calibration.

### Comparison of both Evaluation Methods

Illustration 21 presents the isothermal evaluation using a Calo 2310 eco; Illustration 22 displays the Calo 2310 pro evaluation. Energy levels of 64.55 kJ/mol and 64.60 kJ/mol correspond well to each other. An HB evaluation shows 65.53 kJ/mol. All values correspond well with each other with

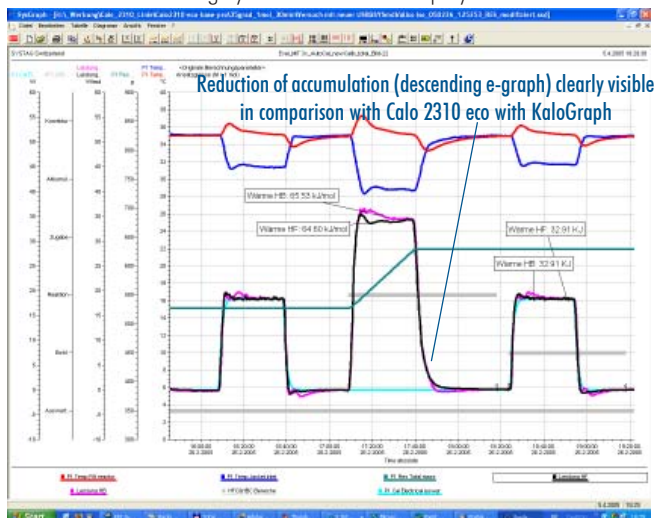
deviations of less than 1.5%. The energy content is equal, whether using isothermal and non-isothermal evaluations.

By examining the shape of the graphs it is apparent that the Calo2000 evaluation provides a more realistic reflection of the reaction course due to the elimination of the time constant of the reactor, based on model generation. With only a 90 sec pulse increment time (regardless of the level in the reactor, small or large cp value of the reactor contents) excellent values are obtained for kinetic investigations. The isothermal version of the Calo2310 eco cannot achieve this goal; the error is 3 ... 5 min, isoperibolically possibly even 7...15 mins.

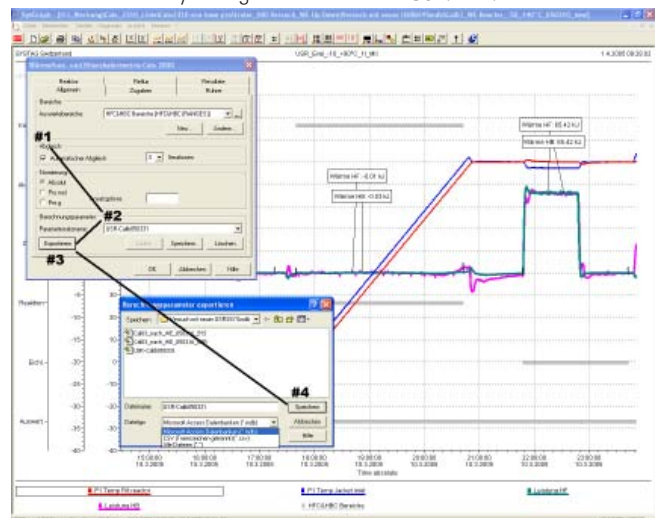
Separate y-axes allow the uncluttered scaling of up to 8 graphs (Illustration 20-23). The evaluation calculates the energy values in kJ automatically and writes them immediately into the corresponding output graphs (Illustration 20-22). Values are not only graphically but also numerically documented in a single display. Additional text can be entered to make the evaluation even more meaningful.

The numerical results are available from an access databank (SysGraph) and can be used for special purposes by the user.

**Illustration 22** Evaluation with Calo2000, highly accurate kinetic display



**Illustration 23** Procedure for user calibration followed by storage of data as new USR.mdb.



## Standard Baseline and novel, automatic „Zero Watt“ Baseline

### Non-isothermal Operation

As most technical processes contain heating and cooling cycles (or vice versa) it makes, therefore, little sense only to be able to accurately measure isothermally. There was no alternative choice due to technological reasons, no appropriate instruments were available. In other words: the techniques used during the 1970's (over 30 years ago) are still in use today. A present-day system must be capable to measure, by necessity accurately over a far wider range.

### Baseline for Relative Measurements

The limitations of the conventional Heatflow calorimeters still currently in use can be demonstrated by conducting a simple experiment:

When heating water using a ramp of for example +20K/h from +5°C to +65°C and a calibration heating before and after, the ramp should not show an output. Unfortunately this is not the case with all conventional calorimeters (including the Calo 2310 eco). This is a result of the measurement of relative temperature differences, which by using a baseline are re-functioned to an „absolute measurement“.

### „Zero Watt“ Baseline, only by Absolute Measurements

This new concept which becomes fully operational in the **Calo2310 base** and **Calo2310 pro** does not require the selection of a baseline. Due to the generation of a model, the calorimeter is directly calibrated in absolute values providing a 0 Watt measurement (without a chemical reaction) with a small error of a few Watt. Only the **Calo2310 eco** operates by establishing a manual baseline as relative measurements are used.

### Example: Calo2310 base and Calo2310 pro, respectively

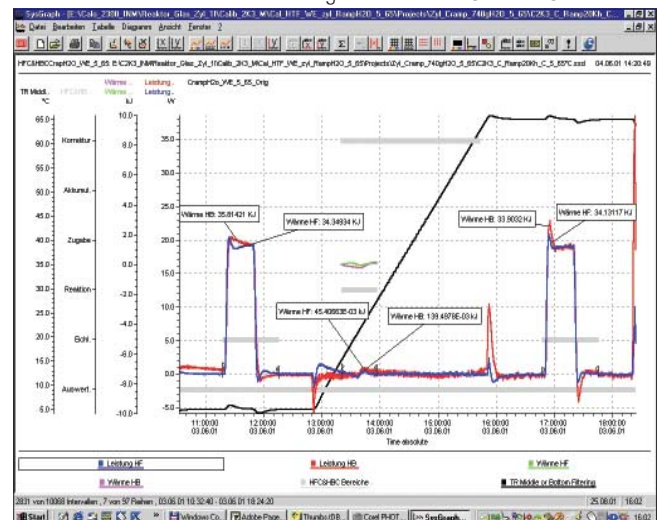
Illustration 24 presents the heating of water from 5°C to 65°C at 20K/h. Note the excellent zero line of the output. A kink is noticeable at 20...23°C due to evaporation of condensation (mid summer, high relative humidity!). This would not have happened if a Calo2100 was used as it is contained in a temperature controlled cabinet. The calorimeters of the Calo2000 series provide a correct baseline, the „**Zero Watt**“ Baseline.

### Measuring, even without Calibration

As the total system of the **Calo2310 base** and **Calo2310 pro** is calibrated in absolute terms, a reaction can be measured without calibration heating (prior and after the reaction). Obviously, there is a slight decrease in accuracy but still ideal for a quicker evaluation.

A major advantage for the user lies in the ability to calibrate the working range himself afterwards (Ref. Illustration 23) enabling him to work experiment specific with accuracy and without calibration heating. This is a tremendous advantage when working with unstable materials that do not permit waiting times required for calibration heating.

Illustration 24 Heating water from 5°C to 65°C





## Pictograms and their Meaning

### Qualitative Specifications

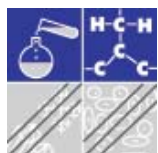
We use pictograms to describe individual instruments in simple terms, regarding their operation and capabilities

#### Application: Mostly Syntheses



The synthesis is the most commonly used application, and some other not extremely critical reactions. „Critical’ is meant in a measuring context and not in a chemical sense.

#### Application: Synthesis, Polymerisation



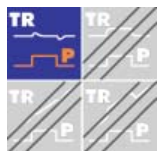
Next to a synthesis, polymerisations can be comprehensively measured, provided a certain output is generated, for example 50 Watt. This does not require major efforts with regard to prevent influences from the surrounding environment.

#### Application: Synthesis, Crystallisation



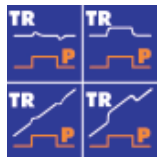
Crystallisation deserves particular attention as it can only be measured with a non-isothermal instrument based on heat balance. The only suitable instrument is the **Calo2310 pro** besides the specially designed **Calo2200** in a cabinet.

#### Operation: Only Isothermal



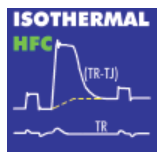
All currently used calorimeters are of isothermal design with various approximations to achieve a quasi non-isothermal operation. Results are never completely reliable, hence the eternal need for interlaboratory test.

#### Operation: All Modes



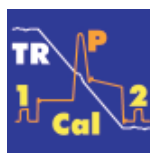
The 4 symbols are representative of all possible operational modes: isothermal (left column), isoperibolic (right column) both relating to fixed (upper line) or ramp set value (lower line). Isothermal operation is achieved by reactor control, isoperibolic operation by jacket control.

#### Conventional Isothermal HFC



The traditional method involving calibration heating and baseline adjustment for the correction of changing heat transitions is still in use for a whole range of isothermal calorimeters. Isothermal calorimetry does not require information regarding cp or water values.

#### Non-isothermal with Calibration



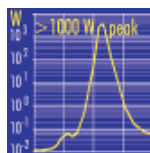
Non-isothermal operation with calibration at high and low temperature levels yields even more accurate results as the measurements can be evaluated offline a well.

#### Non-isothermal without Calibration



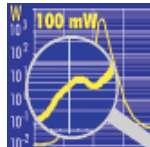
Non-isothermal operation without any calibration heating and excellent accuracy is provided the new top of the range models **Calo2310 base and pro**. Productivity is significantly improved and measurements involving chemically unstable reactions can be finally performed without undue worries.

#### Extreme Dynamic



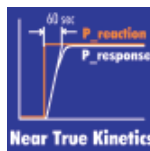
Due to an all embracing mathematical evaluation technique, which simulates the system very accurately, a performance dynamic is obtained that has never been seen before. The model is almost equal to the actual kinetics of the chemical process. Ideal is the additional isoperibolic operation mode.

#### Extreme Sensitivity



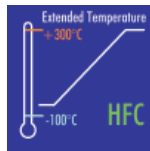
Without additional stabilisation of the surrounding temperature by means of a cabinet, excellent sensitivity can be obtained from HFC in isothermal mode due to the Calo2000 principle.

#### Outstanding Kinetics



The Calo2000 series meets demands for a real-time output projection of a reaction in all operational modes that delivers quick responses (from 10% to 90%) 90 sec. without additional errors, regardless of substance volume in the reactor, small or large cp.

#### Extremely Wide Temperature Range



As the HBC is subject to more limitations, HFC operation is ideally suited for extremely low temperature operation. The complete temperature range can be optionally covered (with at least 2 heat transfer media) and allows true non-isothermal calorimetry.

#### Automatic cp Determination



An internal routine provides an automatic online determination cp. If necessary or required, the routine can be added manually or by recipe, after the use of a calibration heating.

## Pictograms and their Meaning (Continuation)

### Vortex Correction, Stirrer Torque



During HFC, the vortex generates an additional error that can be manually measured and entered into the evaluation for correction using this entry. The torque is also measured during HFC and HBC and expressed in Ncm.

### Complete Corrections



When carrying out dosing procedures, further problems are encountered: change in the wetted surface area A, causing a change in transition coefficient U (HFC only) as well as an output variation due to uneven temperatures of the reactor contents and the dosed educt, both errors are corrected.

### Sealed System



An accurate measurement of small outputs depends on a completely sealed system as errors caused by evaporation can be significant. Using a simple method, this can be safely conducted even in a glass reactor.

### No Condensation – No „Reflux“



A particular problem is condensation on the cover of the reactor, leading to an actual „reflux“. This can cause serious measuring errors. It can be controlled by appropriate means such as lid heating, insulation.

### Extreme Resolution



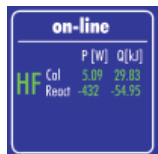
To maintain satisfactory model generation and high sensitivity, the measured values must have a resolution over the entire range of less than 1mK. Long term stability must be maintained.

### High Accuracy at Low Temperatures



Measurements at low temperatures are difficult due to ice formation from moist air or melting respectively. Using specialised insulation and purpose-built thermostats, low temperature systems can operate without difficulties at temperatures of  $-70^{\circ}\text{C}$ ... $-80^{\circ}\text{C}$ .

### Online Guide Values for HFC/HBC: P & Q



The Calo2000 series provide immediate online information. The accuracy is obviously not identical to an offline evaluation but is sufficient for an initial estimate. Ideal for higher output ranges (from approx. 5...10 Watt). Response time 10%-90%: 90 sec for both measurements.

### Online Guide Values HFC/HBC: P & Q

on-line		P [W]	Q [kJ]
HFC	Cal	5.09	29.83
	React	-432	-54.95
HBC	Cal	5.21	31.56
	React	-445	-56.35

Online information, as described above, for both HFC and HBC. The same limitations apply. Response time for 10% - 90%: 90 sec, identical for both measuring methods.

### CFR 21 Part 11 Compliant Protocol



All captured data are filed in their own secure data files that cannot be tampered with, in compliance with FDA and GMP requirements.

### Access Control



An optional access control is available, comprising various access levels, dedicated names and passwords. During extended periods of inactivity, the system is locked, but continues to run in the background. A new logon procedure has to be carried out which is recorded.

### Certification



An installation and operation certificate can be supplied. We also carry out factory and site acceptance tests.

### Method:

#### Heat Flow Only



Conventional heat flow calorimetry (HFC). The accuracy depends on filling height, viscosity and stirrer vortex. Accumulation of solid material and crystallisation near the reactor wall leads to erroneous results.

### Method:

#### Heat Balance Only



The heat balance calorimetry (HBC) method is not sensitive to any transfer changes between the reactor contents and the jacket and provides a robust measuring method. It is also used at the reflux condenser.

### Combined Method: Simultaneous HBC/HFC



The combined simultaneous use of HBC and HFC supplies two independent measuring results. The major advantage is that both methods are complimentary. The user creates his own „Interlaboratory Test“ and increases the significance of the obtained results considerably.

## Calo2310 eco

### Detailed Instrument Specifications

Reactor	Glass, 1l, double jacket (permits working with volumes from 350 ml up to 1000 ml)
Stirrer	40 – 400 and 200 – 2000 rpm, torque measured, 200/40Ncm, anchor stirrer, glass
Dosing 1	gravimetric from balance 1, to 4200 g with pump, Gamma 4, 1l/h
Thermostat	Unistat Tango (HTF190) -40°C to +200 °C. Working range for TR -20°C to +180°C
Temp. Measurement	Pt-100, Class A. TR, TJ_out; optional TH, TC_in, TC_out
Calorimetry	Heat-flow in reactor
Calibration heating	20 Watt, glass, with supply and measurement
Temp. Control	Jacket and reactor control (cascade)
Construction	Standard – on portable frame for positioning in fixed cabinet

### Options

Dosing 2	1 x gravimetric from balance 2 to 4200g with pump Gamma 4, 1l/h; can also be used for pH control (compulsory due to mass balance)
Temp. Dos 1 & 2 Measurement	Additions temperature for automatic heat correction
pH measurement	with semi-automatic calibration
pH Measuring Range	0°C . . . 100°C
pH Control	Single sided (choice of acidic or alkaline range). Dosing from balance 2, volume max 1l/h (Gamma 4). Caution: During pH control mode no further dosing from balance 2 can be carried out
Atmospheric Pressure Measurement	0...1300 mbar <sub>abs</sub>
Pressure/Inertisation Control resp.	For 0 – 1300mbar <sub>abs</sub> (0 . . . 60 bar for autoclave, optional!). PID duty cycle control
Vacuum Control	approx. 40 . . . 1000mbar <sub>abs</sub> reactor pressure. PID duty cycle control
Venting Control	Used for pressure or vacuum relief. Automatic monitoring and control for sealed operation with glass reactor. PID duty cycle control.
Reflux Balance Calorimetry	High-intensity design condenser with temperature measurement and constant flow facilitator.
Reflux Splitter	For distillations, with 24 VDC magnetic splitter
Distillation	Separate balance 3 for receiving vessel
Temperature Control	Adiabatic control

## Calo2310 base

### Detailed Instrument Specifications

Reactor	Glass, 1l, double jacket (permits working with volumes from 350 ml up to 1000 ml), sealed
Stirrer	40 – 400 and 200 – 2000 rpm, torque measured, 200/40Ncm, special basket stirrer, glass
Dosing 1	gravimetric from balance 1, to 4200 g with pump, Gamma 4, 1l/h
Thermostat	Unistat Tango (HTF190) -40°C to +200 °C. Working range for TR -20°C to +180°C
Temp. Measurement	Pt-100, Class A. TR, TJ_out, T_amb; optional TH, TC_in, TC_out
Calorimetry	Heat-flow in reactor
Calibration heating	20 Watt, glass, with supply and measurement
Temp. Control	Jacket and reactor control (cascade) and various other required controls
Atmospheric Pressure Measurement	0...1300 mbar <sub>abs</sub>
Pressure/Inertisation Control resp.	For 0 – 1300mbar <sub>abs</sub> (0 . . . 60 bar for autoclave, optional!). PID duty cycle control
Vacuum Control	approx. 40 . . . 1000mbar <sub>abs</sub> reactor pressure. PID duty cycle control
Venting Control	Used for pressure or vacuum relief. Automatic monitoring and control for sealed operation with glass reactor. PID duty cycle control.
Electronic Safety Control	max 1300mbar <sub>abs</sub> (selectable) for automatic venting
Mechanical Safety Valve	max 1500mbar <sub>abs</sub> for safety venting if electronic control fails
Construction	Standard – on portable frame for positioning in fixed cabinet

### Options

Dosing 2	1 x gravimetric from balance 2 to 4200g with pump Gamma 4, 1l/h; can also be used for pH control (compulsory due to mass balance)
Temp. Dos 1 & 2 Measurement	Additions temperature for automatic heat correction
pH measurement	with semi-automatic calibration
pH Measuring Range	0°C . . . 100°C
pH Control	Single sided (choice of acidic or alkaline range). Dosing from balance 2, volume max 1l/h (Gamma 4). Caution: During pH control mode no further dosing from balance 2 can be carried out
Reflux Balance Calorimetry	Special, high intensity condenser, vacuum insulated and mirror coated, with transparent inspection strip. Specially constructed for HBC measurements with water value correction of reflux condenser, head temp. measurement, TH and constant flow facilitator.
Reflux Splitter	For distillation, with 24 VDC magnetic splitter. Specially designed for HBC with water value correction in reflux splitter.
Distillation	Separate balance 3 for receiving vessel
Temperature Control	Adiabatic control

# Calo2310 pro

## Detailed Instrument Specifications

Reactor	Glass, 1l, triple jacket (permits working with volumes from 200 ml up to 1000 ml), sealed
Stirrer	40 – 400 and 200 – 2000 rpm, torque measured, 200/40Ncm, special basket stirrer, glass
Dosing 1	gravimetric from balance 1, to 4200 g with pump, Gamma 4, 1l/h; measurement of additions temperature for automatic heat correction.
Main Thermostat	Unistat 380w (HTF190) -80°C to +200 °C. Working range for TR from -50°C to +180°C
Lid Thermostat	Polystat (HTF190), 20°C to +200 °C, supported by 380w with supplementary heat exchanger, suitable for lower temperature operation
Temp. Measurement	Pt-100, Class A. TR, T <sub>J_in</sub> , T <sub>J_out</sub> , T <sub>cov</sub> , T <sub>ThStat_in</sub> , T <sub>ThStat_out</sub> , T <sub>dos1</sub> , T <sub>amb</sub> ; optional T <sub>dos2</sub> , TH, TC <sub>in</sub> , TC <sub>out</sub>
Calorimetry	Heat-flow and heat balance in reactor
Calibration heating	20 Watt, glass, with supply and measurement
Temp. Control	Jacket and reactor control (cascade) and various other required controls
Atmospheric Pressure Measurement	0...1300 mbar <sub>abs</sub>
Pressure/Inertisation Control resp.	For 0 – 1300mbar <sub>abs</sub> (0...60 bar for autoclave, optional!). PID duty cycle control
Vacuum Control	approx. 40...1000mbar <sub>abs</sub> reactor pressure. PID duty cycle control
Venting Control	Used for pressure or vacuum relief. Automatic monitoring and control for sealed operation with glass reactor. PID duty cycle control.
Electronic Safety Control	max 1300mbar <sub>abs</sub> (selectable) for automatic venting
Mechanical Safety Valve	max 1500mbar <sub>abs</sub> for safety venting if electronic control fails
Construction	Standard – on portable frame for positioning in fixed cabinet

## Options

Dosing 2	1 x gravimetric from balance 2 to 4200g with pump Gamma 4, 1l/h; can also be used for pH control (compulsory due to mass balance) with Temp. Dos 2 Measurement Additions temperature for automatic heat correction
pH measurement	with semi-automatic calibration
pH Measuring Range	0°C...100°C
pH Control	Single sided (choice of acidic or alkaline range). Dosing from balance 2, volume max 1l/h (Gamma 4). Caution: During pH control mode no further dosing from balance 2 can be carried out
Reflux Balance Calorimetry	Special, high intensity condenser, vacuum insulated and mirror coated, with transparent inspection strip. Specially constructed for HBC measurements with water value correction of reflux condenser, head temp. measurement, TH and constant flow facilitator.
Reflux Splitter	For distillation, with 24 VDC magnetic splitter. Specially designed for HBC with water value correction in reflux splitter.
Distillation	Separate balance 3 for receiving vessel
Temperature Control	Adiabatic control

## Control System – Configuration

### Requirements at customer site

Calo 2310 eco & base	Electricity supply: 3 x 230/400 VAC. <u>Single</u> 3 phase group with 16A each and corresponding distributors.. Cooling water for Thermostat Tango. Cooling water with pressure reducer for reflux (approx. 2...6 bar, 20l/h, if required). Vacuum with cooled condensate trap (if required). N <sub>2</sub> with pressure reducer for inertisation (if required).
Calo 2310 pro	Electricity supply: 3 x 230/400 VAC. <u>Two</u> 3 phase groups with 16A each and corresponding distributors. Cooling water for Thermostat 380w. Cooling water with pressure reducer for reflux (approx. 2...6 bar, 20l/h, if required). Vacuum with cooled condensate trap (if required). N <sub>2</sub> with pressure reducer for inertisation (if required).
PC	Must be supplied and installed by SYSTAG. Fully equipped Pentium PC with colour printer and TFT colour screen.
Software Programs	Windows XP, Office package, FIX/MMI, ElemX and OperX, release 1.2 or higher, SysGraph with module KaloGraph (Calo2310 eco) and additional module Calo2000 for Calo2310 base and pro.

### Further Calo2000 Information: [www.systag.ch](http://www.systag.ch)

#### Range of information available in English:

#### General information – preview and introduction

Internet	<a href="http://www.systag.ch/E_100_Intro.html">www.systag.ch/E_100_Intro.html</a>
	<a href="http://www.systag.ch/E_300_RCalo.html">www.systag.ch/E_300_RCalo.html</a>

#### The White Paper of reaction calorimetry

Internet	<a href="http://www.systag.ch/A4e_white_paper_f.pdf">www.systag.ch/A4e_white_paper_f.pdf</a>
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#### Calo 2000 general publications

Internet	<a href="http://www.systag.ch/E_720_REP_RC.html">www.systag.ch/E_720_REP_RC.html</a>
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#### R&D report No.3 (d-l Mandelic acid)

Investigation into the suitability of heat flow and heat balance calorimeters in non-isothermal, homogenous and inhomogenous 2 phase systems (liquid/solids)

Internet	<a href="http://www.systag.ch/A4e_MandAcid_1a32.pdf">www.systag.ch/A4e_MandAcid_1a32.pdf</a>
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#### Semi-batch reaction kinetics

From heat-up to reflux! Measurements with the new reaction calorimeter Calo 2000 (only in German).

Internet	<a href="http://www.systag.ch/KaloFreiberg2001/index.html">www.systag.ch/KaloFreiberg2001/index.html</a>
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#### R&D report No.4 : Baseline Calo 2100 vs. Calo 2300

The Criterion "Base-Line" for the Evaluation of the Usefulness of a Reaction Calorimeter.

Internet	<a href="http://www.systag.ch/A4e_C2K_Basis_1a20.pdf">www.systag.ch/A4e_C2K_Basis_1a20.pdf</a>
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Options	on request
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## Calo2310: Technical Details that lead to Success

### Important details that lead to success:

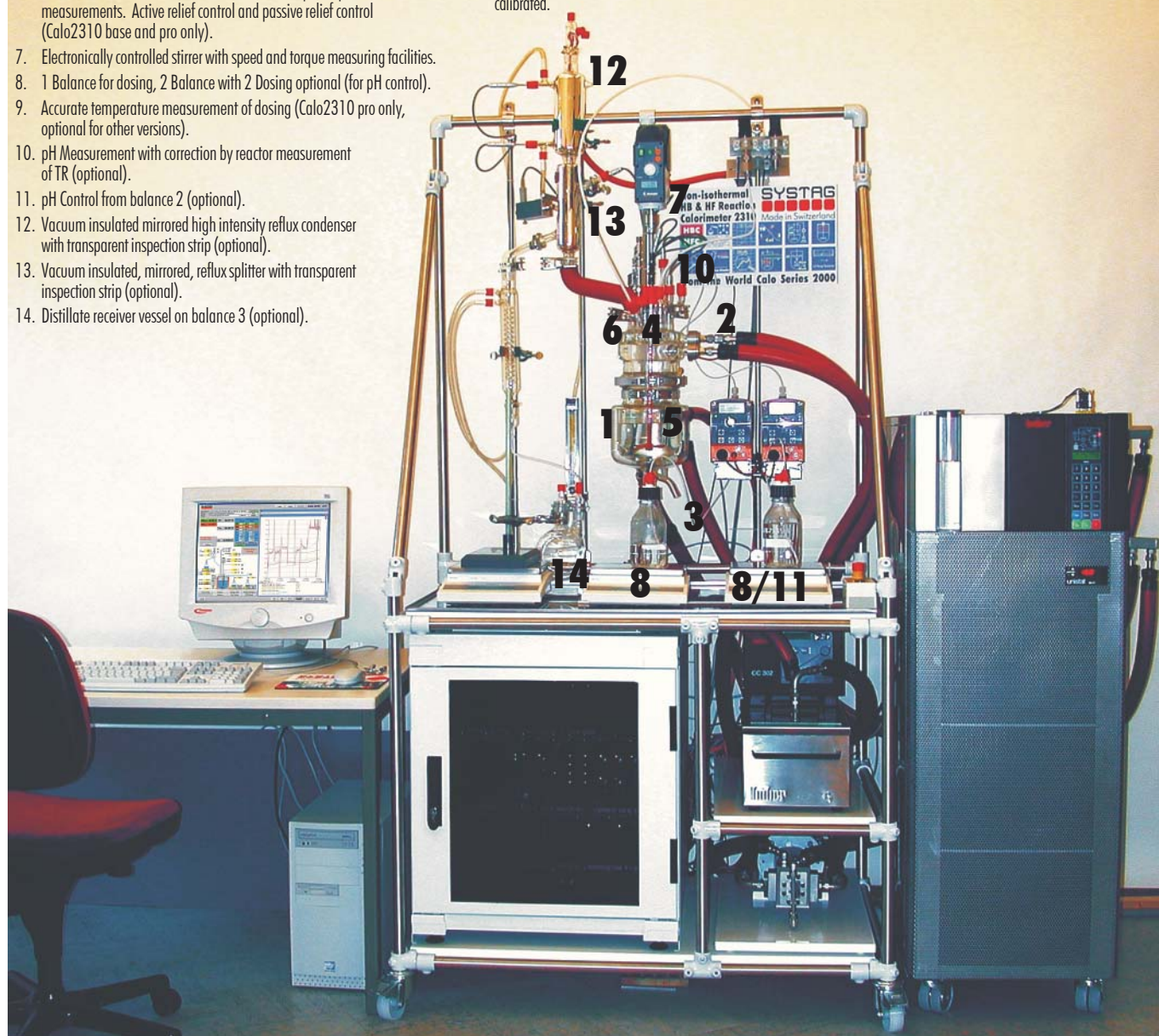
1. Vacuum insulated double jacket reactor vessel (Calo2310 pro only).
2. Heated reactor cover (Calo2310 pro only).
3. Accurately stabilized flow with constant circulation speed in jacket (Calo 2310 pro only).
4. Sealed magnetic coupling for stirrer (Calo2310 base and pro)
5. Range of stirrers (glass, steel or coated) available. Ideal for low viscosity solvents are basket and propeller stirrers. Alternatively anchor and impeller stirrers are available.
6. Sealed cover construction with reactor and atmospheric pressure measurements. Active relief control and passive relief control (Calo2310 base and pro only).
7. Electronically controlled stirrer with speed and torque measuring facilities.
8. 1 Balance for dosing, 2 Balance with 2 Dosing optional (for pH control).
9. Accurate temperature measurement of dosing (Calo2310 pro only, optional for other versions).
10. pH Measurement with correction by reactor measurement of TR (optional).
11. pH Control from balance 2 (optional).
12. Vacuum insulated mirrored high intensity reflux condenser with transparent inspection strip (optional).
13. Vacuum insulated, mirrored, reflux splitter with transparent inspection strip (optional).
14. Distillate receiver vessel on balance 3 (optional).

### System Construction

The standard version of the system is portable for placing into a cabinet at floor level. A separated version can be supplied i.e. wet component separated from control unit and thermostat. Cables of up to 5 meter lengths are acceptable without problems. Due to the design of the frame and the standardized rods, extensions can be easily made when required.

### Options

Please contact your SYSTAG agency for information on the many available options. This includes other types of reactors, steel reactor with special insulation, FTIR etc. Note: Reactors for Calo2310 base and pro must be calibrated.



Technical changes can be made at any time

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**SYSTAG, System Technik AG**

Bahnhofstrasse 76, CH-8803 Ruschlikon/ZH  
 Tel +41(0)44 704 5454 Fax +41(0)44 704 5455  
 E-mail: [infos@systag.ch](mailto:infos@systag.ch) Internet: [www.systag.ch](http://www.systag.ch)

**System Technik Deutschland GmbH**

Rodheimerstr, 63, D-61191 Rosbach/Frankfurt a.M.  
 Tel +49 (0)6003 93 50 50 Fax +49 (0)6003 93 50 522  
 E-mail: [info@systag-deutschland.de](mailto:info@systag-deutschland.de) Internet: [www.systag.ch](http://www.systag.ch)