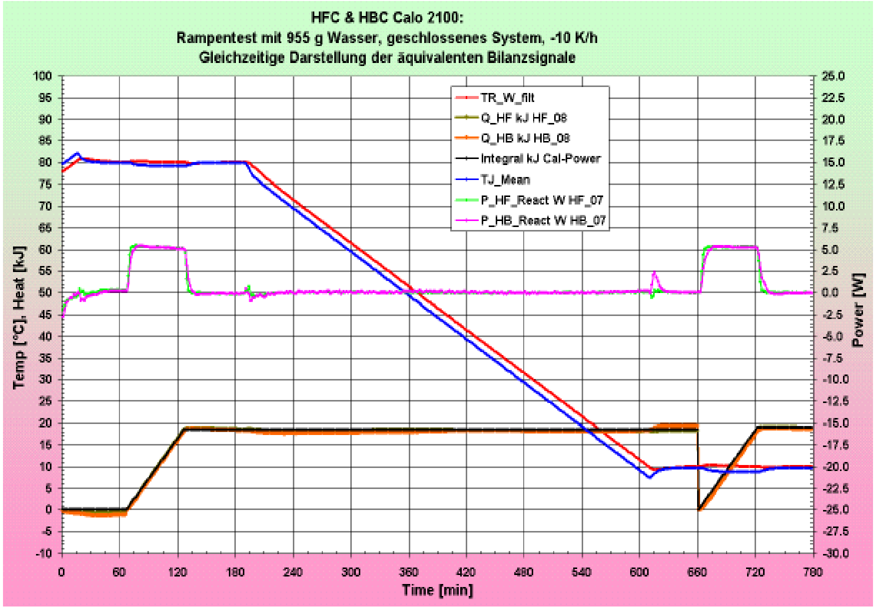
INFORMATION

Reaction Calorimetry

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

Peter E. Meier, dipl. Ing. ETH, SYSTAG



SYSTAG
automatically better

Table of Contents

Reaction-Calorimetry

Definitions

HFC	Heat Flow Calorimetry
HBC	Heat Balance Calorimetry

SYSTAG
automatically better

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

1 Introduction

Sometimes I am being asked:

"What is the difference between the Calo 2000 principle and an RC1, an HEL-Simular, or even our own calorimeters SC1 ... SC4?"

The answer is:

"The Calo 2000 principle is the first reaction calorimeter commercially available, which allows for the recording of chemical power in absolute Watt and which exhibits practically no time error - except the filter time constant of 60 sec. for the evaluation of the measurement."

To achieve this the baseline has to be set to 0 W exactly. Due to various interferences (temperature ramp, dosing etc.) this is not always possible .

Therefore the real behaviour of the baseline is a criterion of quality.

To understand this issue in full detail it is necessary to understand some prerequisites. A detailed description can be found in ref. [1]. I recommend to carefully study this publication in order to be able to understand the correlation between water equivalent and cp and the various interferences.

In this paper I want to elucidate the role of the baseline in non-isothermal calorimetry and how you can assess the possibilities of your current system with a very simple experiment.

2 Definition of "Non-Isothermal Calorimetry"

A priori every calorimetric measurement is non-isothermal due to the fact that every reaction generates heat (or consumes heat) and influences the temperature of the investigated system. But every change in temperature leads to a consumption or generation of energy which cannot be measured from the outside. The heating of the sample can for instance be described by the equation Qs = cps * ms * DTs (s for sample). This heat is consumed inside and cannot be measured as heat flow from the outside. Therefore this part has to be accounted for in the energy balance. Only this makes it possible to measure Q (energy) or P (power).

Therefore in most calorimeters an attempt is made to keep the temperature constant either in the jacket (isoperibol) or even better directly in the reactor (isothermal). This has the following consequences: first energy enters the sample and therefore cannot be measured at this time. Then due to temperature adjustment energy leaves the sample. It is only now that it is possible to measure the heat flow. With this method which is now essentially equal to the isothermal method it is possible to accurately determine the energy Q. The major disadvantage is that the chemical power P cannot be measured because P is needed for the temperature change of the sample and the water equivalent as well [1] and it is not possible to directly measure this by the determination of the temperature difference. Only when we use a model and simulate the same behaviour within this model it becomes indirectly possible to assess the chemical power of the reaction. A method like this enables us to realise the measurement of power with accurate time resolution which can be used for kinetic evaluations.

But as soon as you carry out a batch reaction you are dealing with a real non-isothermal calorimetry because the temperature at the beginning is different from the final temperature. In this case a balance over the entire system has to be carried out taking into account all possible interferences [1]. Common calorimeters make a simple balance which is only accurate for small differences in temperature but models which allow for the evaluation of the absolute power of the chemical reaction are lacking.

At this point the Calo 2000 principle comes in and for the first time allows for an absolute measurement of chemical power over a wide temperature range.

Definition of the Base-Line

This range is approximately 100 K wide between the beginning and end of the measurement.

As soon as a reasonable model is at hand there is no longer the need for isothermal or isoperibol operation (reactor or jacket temperature control respectively). The quality of the results of the measurement is equal no matter whether reactor control or the simple jacket control was used. It even turned out that due to the simpler jacket control the dynamics of a chemical reaction can be recorded more precisely because without master controller less interferences superimpose the results (tendency for oscillations).

3 Criterion"Base-Line"

3.1 What is the baseline?

In an experiment the power is measured only indirectly. This indirect way of measurement is influenced by a lot of interferences. The model calculation should take these interferences into account as good as possible and should try to eliminate them. Due to the fact that a reactor is a very complex system it is obvious that this cannot be achieved perfectly. To completely describe the whole system it would be necessary to install an infinite number of sensors in the sample and the reactor system (wall, jacket, stirring, etc.). But usually there is only one sensor for one part of the system (for instance TR, TJ, TH, Tamb etc.). Therefore each sensor represents only one single point of the respective part. It is obvious that this single measuring point does not represent the integral behaviour of the system. Especially with pronounced changes in temperature (jumps and kinks) there are clear differences within the system due to the high heat flow. In this case one sensor is not enough to fully describe the whole system. In this situation all methods fail and errors are introduced. The objective is to identify those possible errors to be able to assess the quality of the calorimetric system.

The baseline therefore is the line of reference for 0 W of chemical reaction power. This reference line is a calculated value and can deviate from the ideal and therefore the power connected to this reference does not necessarily have to exist in reality. Therefore it is imperative to determine the baseline in different calorimeters and under various conditions.

3.2 Dependencies of the Base-Line

The baseline is simply represented by the term **dQreact/dt = Preact** in an experiment **without** chemical reaction. For illustration here the scheme of the system:

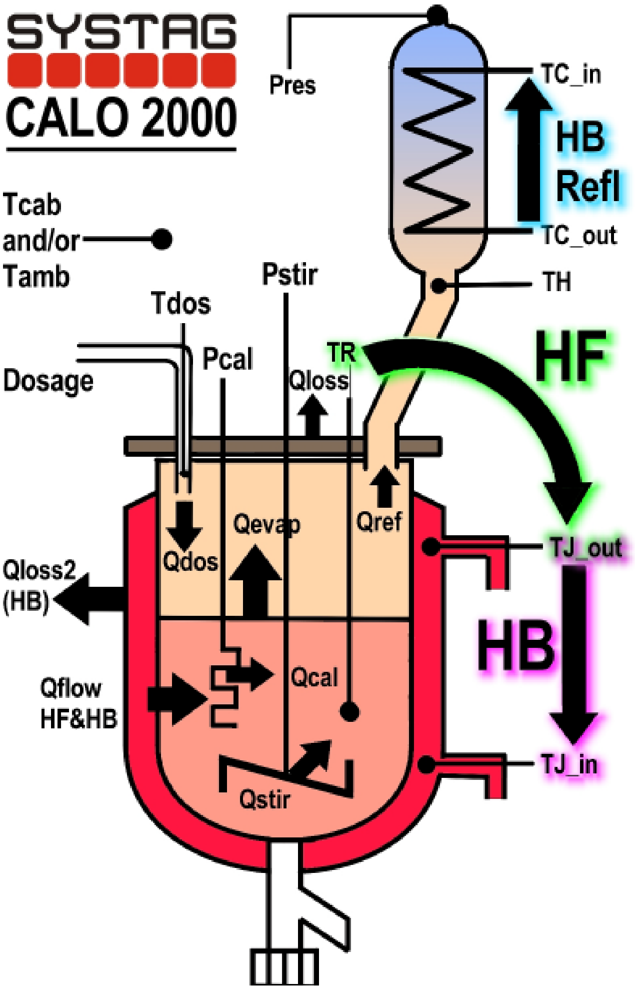


Figure 1 Summary of the most important interferences

Definitions

Qflow	heat flow introduced through the walls and measured by HF or HB
Qreact	energy of the chemical reaction
Qwe	energy of the water equivalent
Qloss	loss to the environment (HFC and HBC)
Qloss2	additional loss of jacket at HBC
Qcal	heating energy for calibration
Qstir	stirring energy
Qevap	energy of evaporation
Qdos	energy due to different temperature of dosing in relation to TR
Qref	energy from the reflux cooler
HF	measurement of heat flow
HB	measurement of heat balance
HB Refl	measurement of heat balance in reflux cooler

The sum of all energies must be 0:

$$Qflow + (Qreact + Qwe) + Qcal + Qstir + Qdos + Qevap + Qloss + Qref = 0$$

Qflow	is measured by the heat flow HF or the heat balance HB
(Qreact + Qwe)	represent the energy of the reaction and the water equivalent (we = water equivalent)
Qcal	is measured electrically
Qstir	is calculated from the revolution and the torque
Qdos	is calculated according to cp * m * (TR – Tdos)
Qevap	it is almost impossible to measure but it must not be neglected
Qloss	can be evaluated by a system calibration as a function F{TJ, TR, Tcab and/or Tamb}
Qref	is equivalent to the measurement of HB at the reflux condenser

Therefore Qreact results from:

$$-Qreact = \Sigma Qn$$

with n representing all other subscripts

With heat flow calorimeters a special focus has to be devoted to the exchange area **A** and the overall coefficient of heat transfer **U** because these two factors directly influence the calculation! An uncontrolled change directly results into an error in the baseline!

These considerations make it clear that an open reactor without proper insolation in an environment with changing temperature results into problems of the baseline. This is the reason why for the calorimeter with the highest sensitivity, the Calo 2100 or Calo 2200, a controlled environment around the reactor was realised.

4 How do you check the baseline?

Though it is very difficult to determine a good baseline it is very easy to check the quality of the baseline! To achieve this you have to fill the calorimeter with a chemically inert substance and to subject this to a heating ramp. That is all! For calibration of the system it is expedient to heat the system for calibration before and after such a ramp.

In the following we show the results for 1 l water, cooled from 80°C with -10 K/h to 10°C. In addition we show some intermediate steps of the measurement and calculation signals, to enable you to understand the evaluation of the results.

Furthermore we present a very fast ramp from 10°C (5°C) with +60 K/h (1 K/min) to 80°C (75°C), which naturally show stronger deviations from the model. This ramp could be applied to a batch process for instance.

This enables you to compare and to estimate the errors which can be expected and to base the decision for the apparatus necessary for your applications on logical reasoning.

5 Practical Examples

1 l of water was subjected to cooling and heating experiments in a cylindrical triple jacketed glass reactor (with vacuum insolation). Results and comments follow on the next pages.

Raw Signals in the Reaction Calorimeter

Figure 2
Raw signals for Calo 2300 or Calo 2904

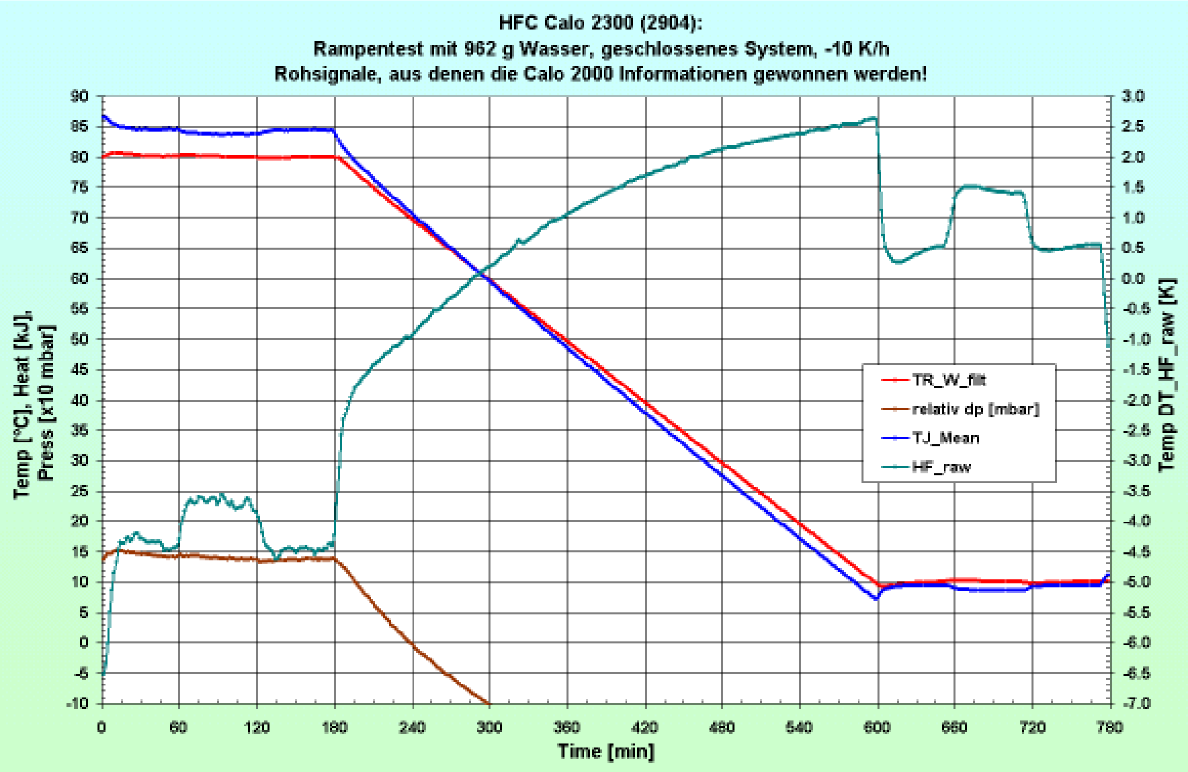
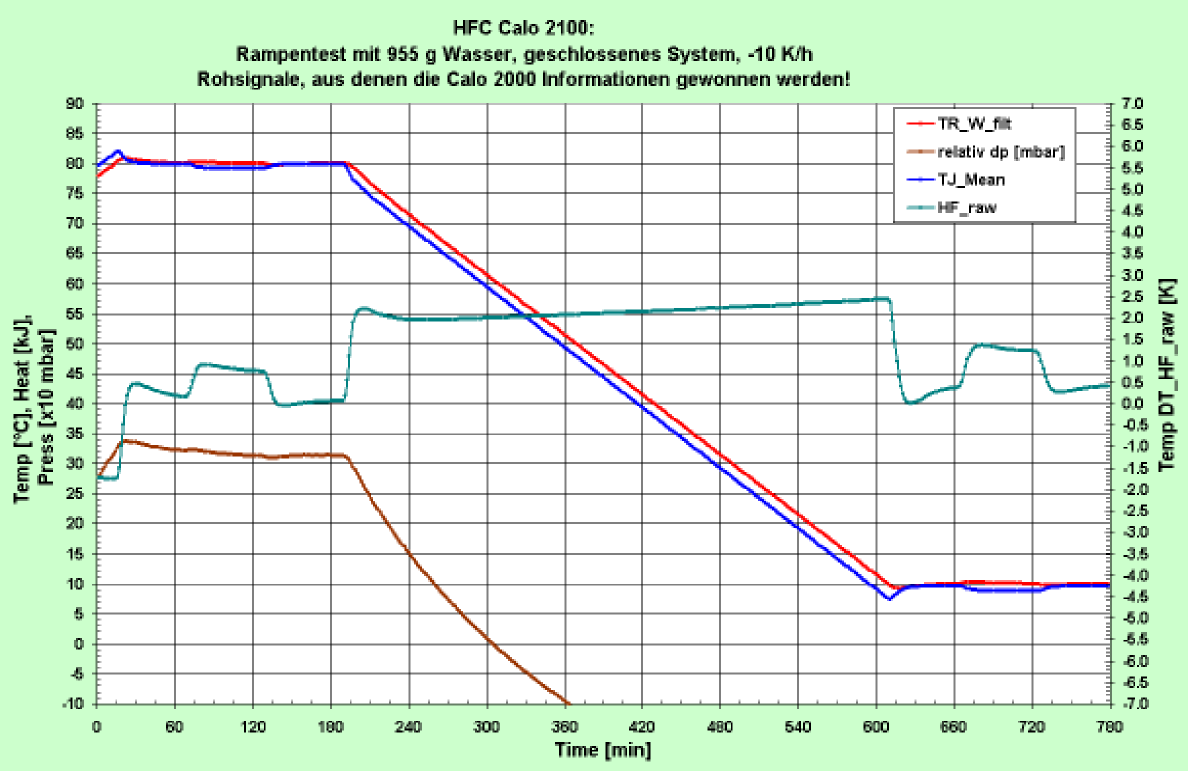


Figure 3
Raw signal for Calo 2100 or Calo 2200



Comments

Signals Calo 2300, Calo 2904, Both Without Box

The proof for the fact that the system was closed and not leaking was achieved by measuring the difference in pressure of +150 mbar at 80°C. With cooling the pressure decreased; for air according to the general gas equation of state and for the vapour according to the vapour pressure line.

The course of the temperature of the jacket TJ_Mean is equal to the temperature of a system in an open environment: at higher reactor temperature the temperature of the jacket - despite cooling - has to be higher to make up for the loss due to cooling by radiation.

The temperature of the reactor behaves like it has to with a reactor feedback control. With turning on the calibration heating the temperature rises slightly and falls again to the set value (compare the zoomed figures 8 and 9). This leads to a down regulation of the jacket temperature (to remove the calibration power).

The temperature difference HF_raw between the reactor and the jacket shows a pronounced increase of -4.5 K at 80°C to +0.5 K at 10°C. This increase is strongly curved. The jump of about 2 K from isothermal conditions to the ramp is very well discernible. This difference is approximately equal to the sum of Cp of the sample and Cp of the water equivalent WE. Because this does not represent a chemical reaction this part has to be neutralized in the evaluation.

A further indication to a system change is the amplitude of this temperature difference at the same calibration power: at high temperature (80°C) this amplitude is ca. 0.75 K and at low temperature (10°C) 0.9 K. This is equal to a change of approximately 15%. The reasons for this are a variation of the water equivalent, thermal transmittance U, and the thickness of the film at the reactor wall (at both, the inner side with water and the side of the jacket with silicon oil).

The next indication can be found in the noise behaviour of the temperature difference at 80°C and 10°C. At 80°C we find a noise of ca. 0.3 K (peak to peak). The explanation is simple but unpleasant: from a certain temperature on (ca. 45°C) we find a higher degree of evaporation of water, which condenses on the lid of the system and as soon as there is enough is flowing or dripping back. This represents a kind of refluxing below the boiling point which is very disturbing.

Signal Calo 2100 and Calo 2200 - Temperature Controlled Box

In this case as well, we find no leaks at more than 300 mbar excess pressure and 80°C. Contrary to the system without box the two temperatures TJ and TR are parallel. This is represented by the temperature difference as well. The difference from 80°C to 10°C for HF_raw is only ca. 0.4 K and not 5 K as before. The level differences of the calibration heating are comparable to above because we are dealing with the same physical conditions. But the noise completely disappears at higher temperatures, which results in the disappearance of the errors introduced by evaporation and refluxing. This could be achieved by keeping the temperature of the inside box at somewhat higher temperatures than TR.

The curves generated by the Calo 2100/2200 look much more trustworthy than any curves from other systems but this increase in quality has to be realised with an accordingly higher effort.

Pre-Processed Signal - Still on the "Kelvin-Signal-Level"

Figure 4

Pre-processed Signal in watt but simultaneous behaviour of the temp.-diff. for Calo 2300 or Calo 2904

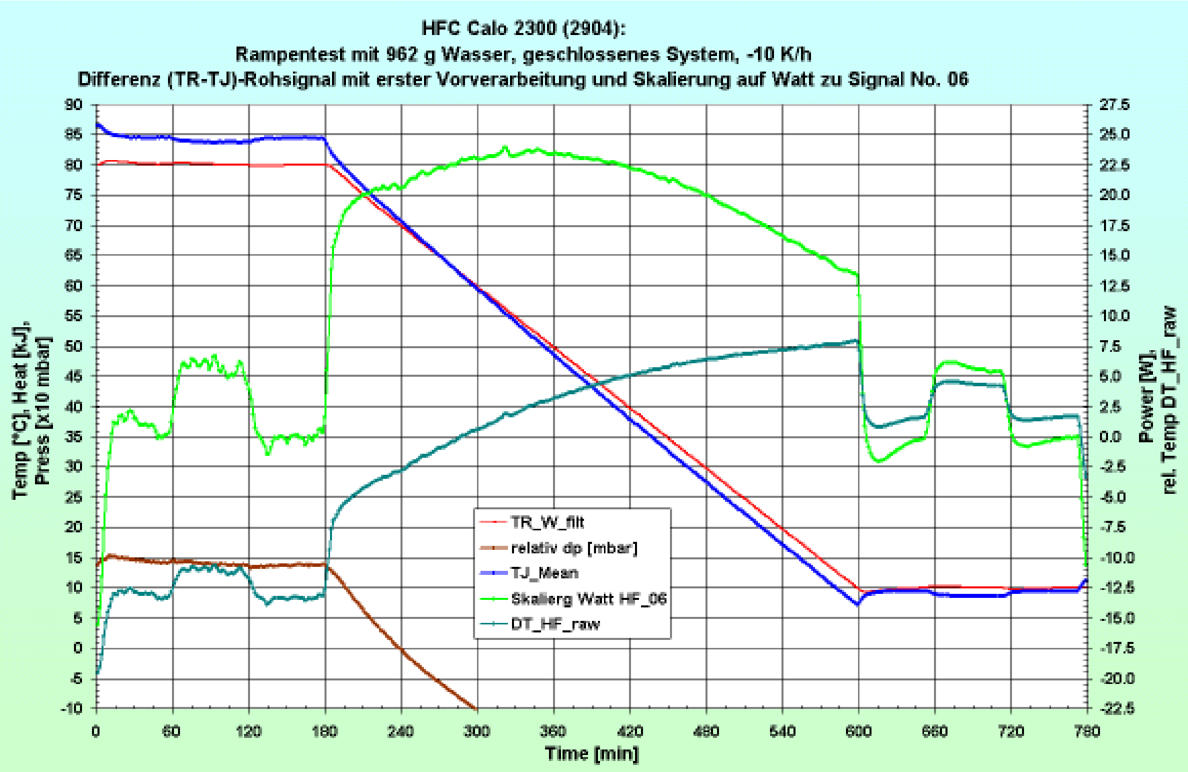
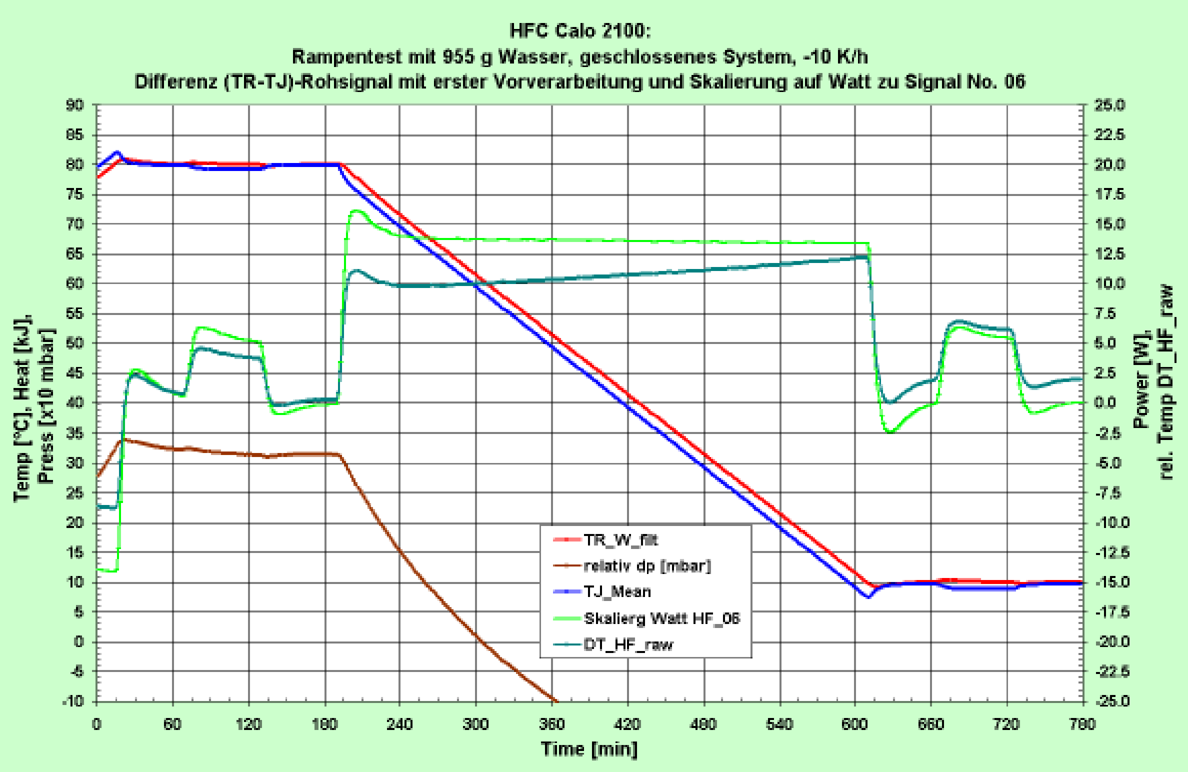


Figure 5

Pre-processed Signal in watt but simultaneous behaviour of the temp.-diff. for Calo 2100 or Calo 2200



Comments

Signals Calo 2300, Calo 2904, Both Without Box

We are dealing with some transformations of the Calo 2000:

At first we process the signals of the temperature difference (from here on only relative scaling will be used) with all necessary corrections to compensate for all the interferences which the system is exposed to in reality. These are model corrections which directly work on the temperature difference signal and therefore the term "Kelvin-signal-level".

Subsequently the signal is transformed into a proportional watt-signal with corresponding normalisation. From this point on we are only working on the "Watt-signal-level". Only now the rest of the model calculations is carried out which lead to the effective chemical reaction power.

All known common calorimeters perform a small part of activities on the "Kelvin-signal-level" compared to the Calo 2000 with subsequent re-scaling to watt without any further model calculations except a fixed value for the water equivalent in the "Watt-signal-level". This is the reason why no real baseline can be realised.

Looking at the light green curve HF_06 (in W), we find that both zero lines at 80°C and at 10°C lie at 0 W and the level corresponds to approximately 5..6 W. At the kink of the ramp at 80°C the curve HF_06 is strongly superelevated compared to the kink at 10°C. This is mainly due to asymmetries and to partly not well compensated interferences. Therefore pronounced corrections have to be carried out later which cannot be done entirely without error.

Due to this normalisation the noise caused by condensation is more pronounced at 80°C; this contributes approximately 2 W (peak to peak).

Signals Calo 2100 and Calo 2200 - Temperature Controlled Box

The same light green curve HF_06 in the Calo 2100 and 2200 looks rather straight. Especially nice is the long ramp: after the kink at 80°C to the ramp the light green curve HF_06 is almost horizontal, i.e. external influences are very constant and do not change!

This statement allows the conclusion that with this information a model will yield results of much higher accuracy.

IMPORTANT:

In both experiments the light green curve HF_06 shows temporal synchronous behaviour to the raw temperature difference DT_HF_raw (TR-TJ). This implies that all retardations in the system are also completely included. This is the case with most other calorimeters as well! In the test with water this is especially pronounced because the cp of water is 4.18 J/g.K but most organic solvents have values in the region of 2 J/g.K.

From this it can be deduced that this raw "Watt"-curve DT_HF_raw can never represent the chemical reaction power (in this example the calibration power). Therefore kinetic deductions are not possible! Only after the following calculations it is possible to make a statement about the kinetics.

Calculation of the Chemical Reaction Power Applying a Model on the “Watt-Signal-Level“

Figure 6
Chem. power for Calo 2300 or Calo 2904

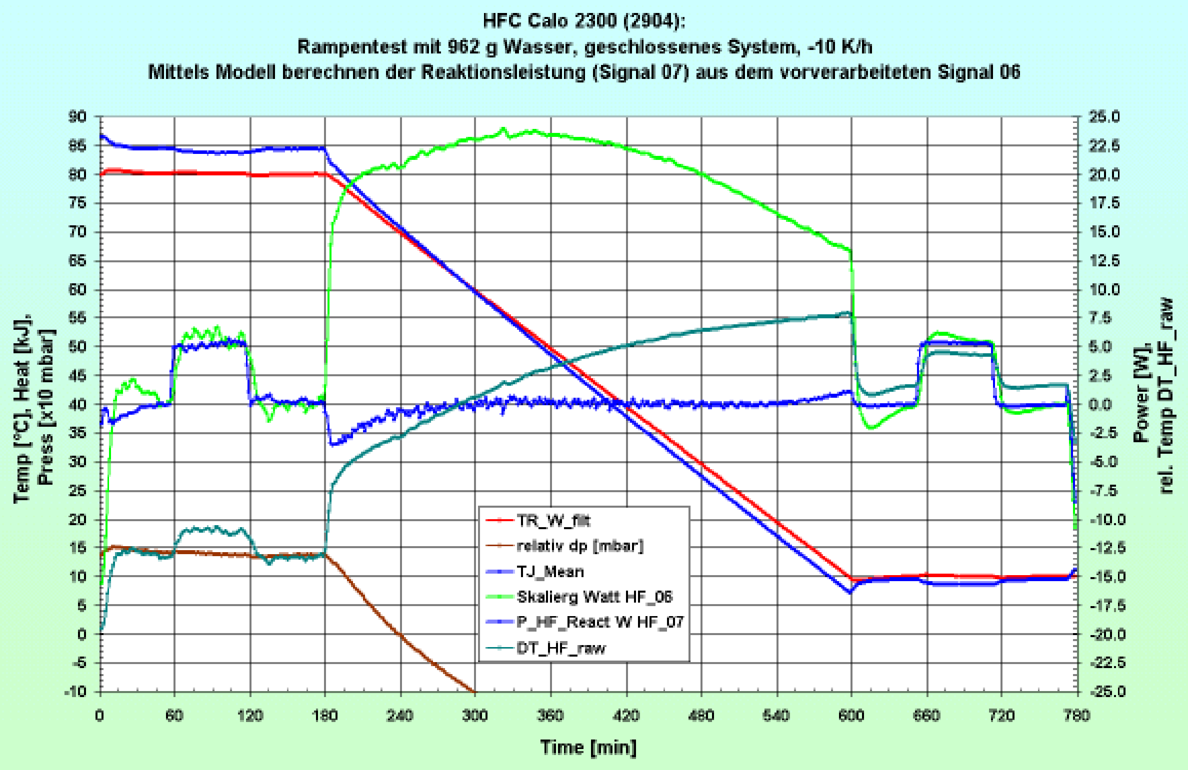
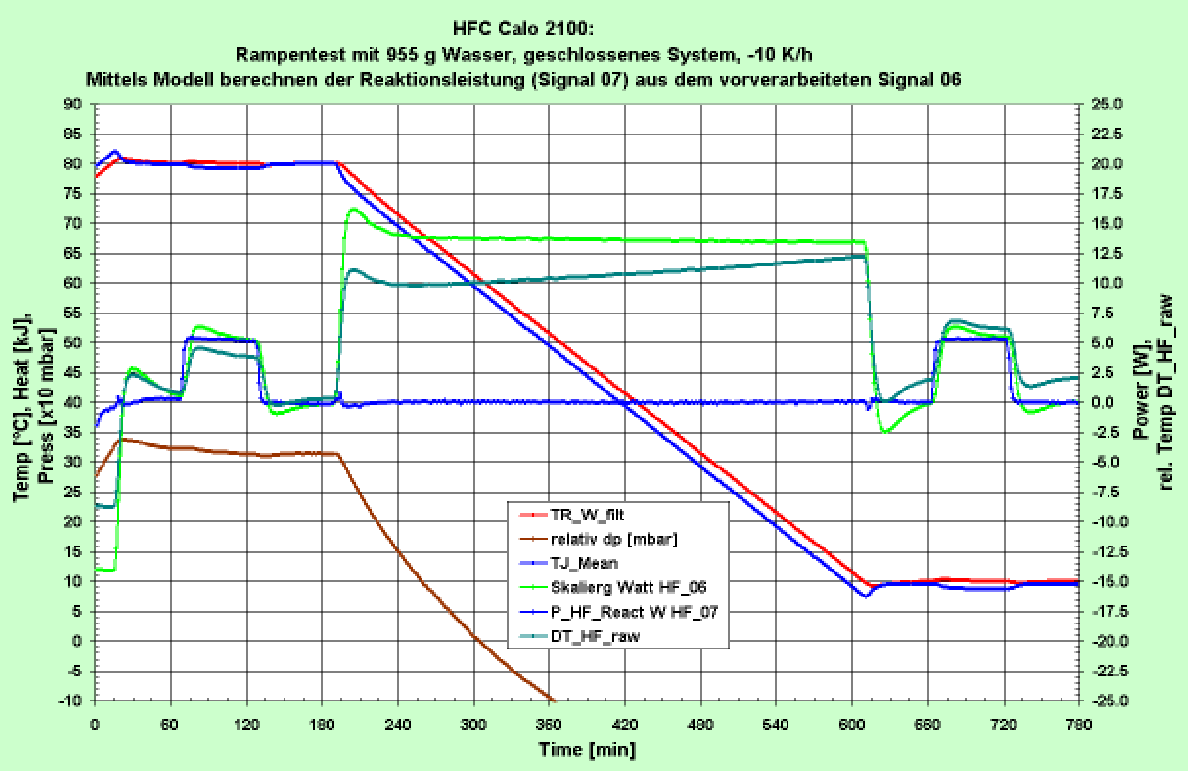


Figure 7
Chem. power for Calo 2100 or Calo 2200



SYSTAG
automatically better

Comments

Signals Calo 2300, Calo 2904, Both Without Box

We are working within the “watt-signal-level”. After carrying out the model calculations we get the effective chemical reaction power - with more or less accuracy! This shows the dark blue curve P_HF_React or abbreviated the signal HF_07.

Two points instantly raise our attention: a) the impulses of the calibration heating have a rectangular effect and b) the ramp has a disturbance near the kinks. Inbetween we get a reasonable baseline of 0 watt over a broad window.

Remarkable is the reduction of the condensate-refluxing power compared to the former figure 4. In figure 4 we were dealing with about 4 W now we see that due to the evaluation the value is between 0.5 and 1 W. This illustrates how the simple temperature difference sometimes is misleading and that it is not sensible to put too much trust in this!

Signals Calo 2100 and Calo 2200 - Temperature Controlled Box

The statement of the last page that HF_06 seems to be more reliable is proved here by the dark blue curve P_HF_React (HF_07)! The baseline (i.e. reaction power = 0 W) of the ramp from 80°C to 10°C is like in dreams! Only a slightly higher degree of noise is noticeable compared to isothermal conditions at 80°C or 10°C. At the kink only a very small disturbance of approximately 1 W from isothermal to ramp and back can be noticed.

It is obvious that with this kind of calorimeter it is possible to accurately measure cristallizations* or even a very weak chemical reaction and that this is possible with a high degree of accuracy over a broad temperature range. This is not possible with any other calorimeter available today! Mind you that at 80°C we are only 20 K below the boiling point and still no error is noticeable!

Please compare the raw curve DT_HF_raw (relative scaling), as well as the Watt-curve HF_06 to the results of the calculation of the real chemical reaction power HF_07: these are worlds apart!

*** Restriction:**
With HFC only being measurable , as long as there are no inhomogeneities or changes in viscosity. In this case HBC has to be applied!

Magnification of the Transition from Ramp to Isothermal and Subsequent 5.2 W Calibration

Figure 8
Magnification of figure 6 with time markers for Calo 2300 or Calo 2904

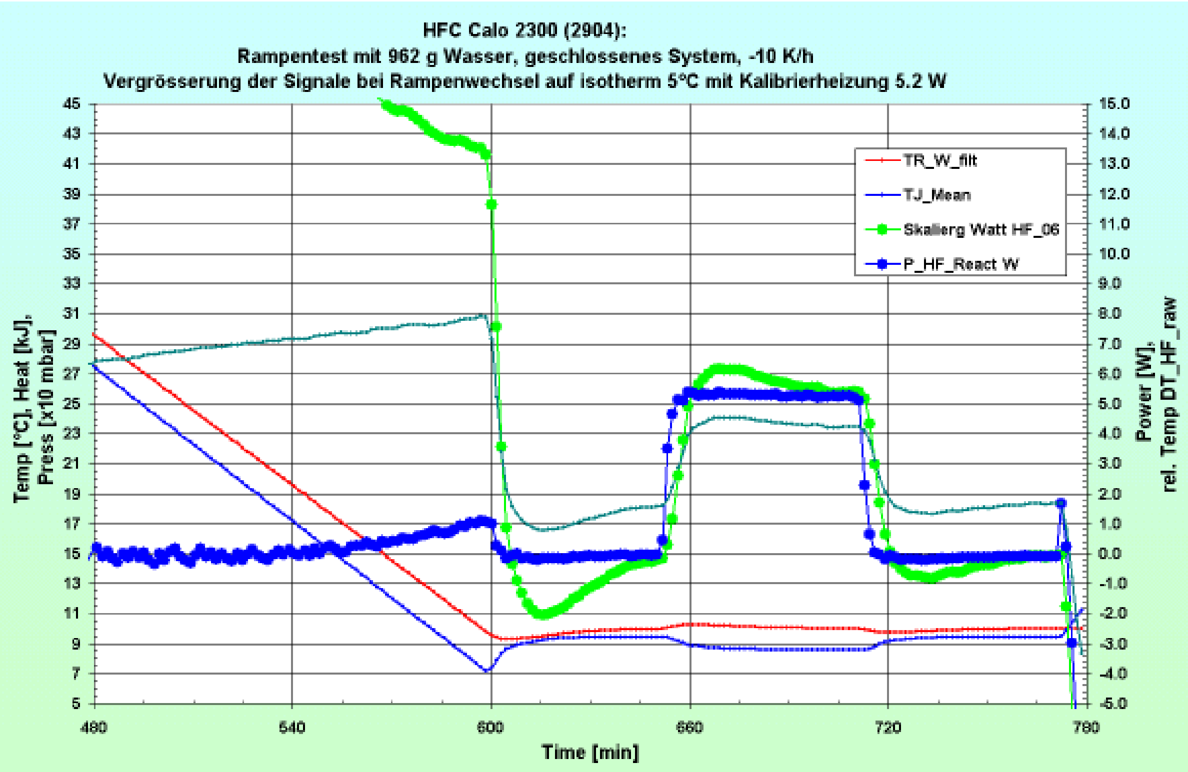
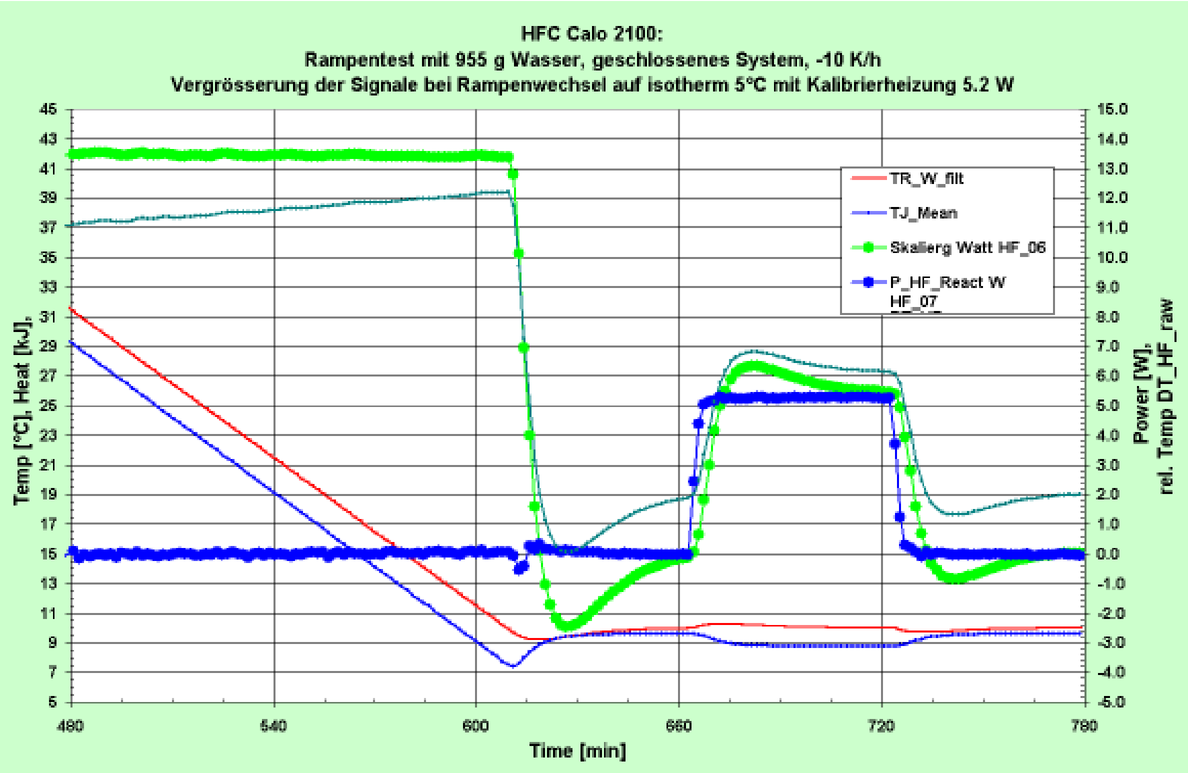


Figure 9
Magnification of figure 7 with time markers for Calo 2100 or Calo 2200



SYSTAG
automatically better

Comments

Signals of the Calo 2300, Calo 2904, Both Without Box

This magnification shows both Watt-curves with special time markers. The whole range of 780 minutes is separated into 500 points. Therefore the spacing between two time markers is 94 sec (93.6 s). With a time constant of 60 sec for the whole measuring system, the increase of a rectangular jump from 0 to 100% takes exactly 60 sec to reach 63%.

But our calibration heating has an additional time constant (metal shell in a glass tube, with air gap, without heat transfer medium). 63% from 0 to 5.2 W is equal to 3.28 W or of a jump from 5.2 W to 0 W 63% is 1.92 Watt (= 37% of 5.2 W).

From the dark blue curve P_HF_React it can be seen that it takes ca. 1.2 ....1.5 time intervals 94 sec which leads to an overall time constant of ca. 120..150 sec. The calibration heating therefore has a time constant of ca. 60..90 sec.

Investigations showed that the calibration time should not be below 10 min to yield results with sufficient accuracy (the longer the more accurate).

The signal HF_06 which is deduced from the temperature needs 3 time intervals to reach the same level, i.e. ca. 280 sec or ca. 2 ... 2.5 longer! This nicely shows that the signal which is proportional to temperature does not represent the correct kinetics!

Furthermore we can determine the noise level of the ramp: ca. 0.5 W (peak to peak).

Signal Calo 2100 and Calo 2200 - Temperature Controlled Box

The situation of the signals in this case is almost equal to the one above but the noise of the ramp is lower and is approximately 0.2 W (peak to peak). This peak value is equal to an average of below 100 mW! Using isothermal conditions this value is even lower.

The good model leads only to a short error at the ramp kink from -10 K/h to 0 K/h of ca. 0.8 W for ca. 4 time intervals (i.e. 6 min).

The stability of the zero-line = baseline is extraordinary good.

Power Integration of the Electrical Calibration and the Chemical Power

Figure 10
Integration of the chem. power curve and the calibration heating (set 0 just before calibration) for Calo 2300 or Calo 2904

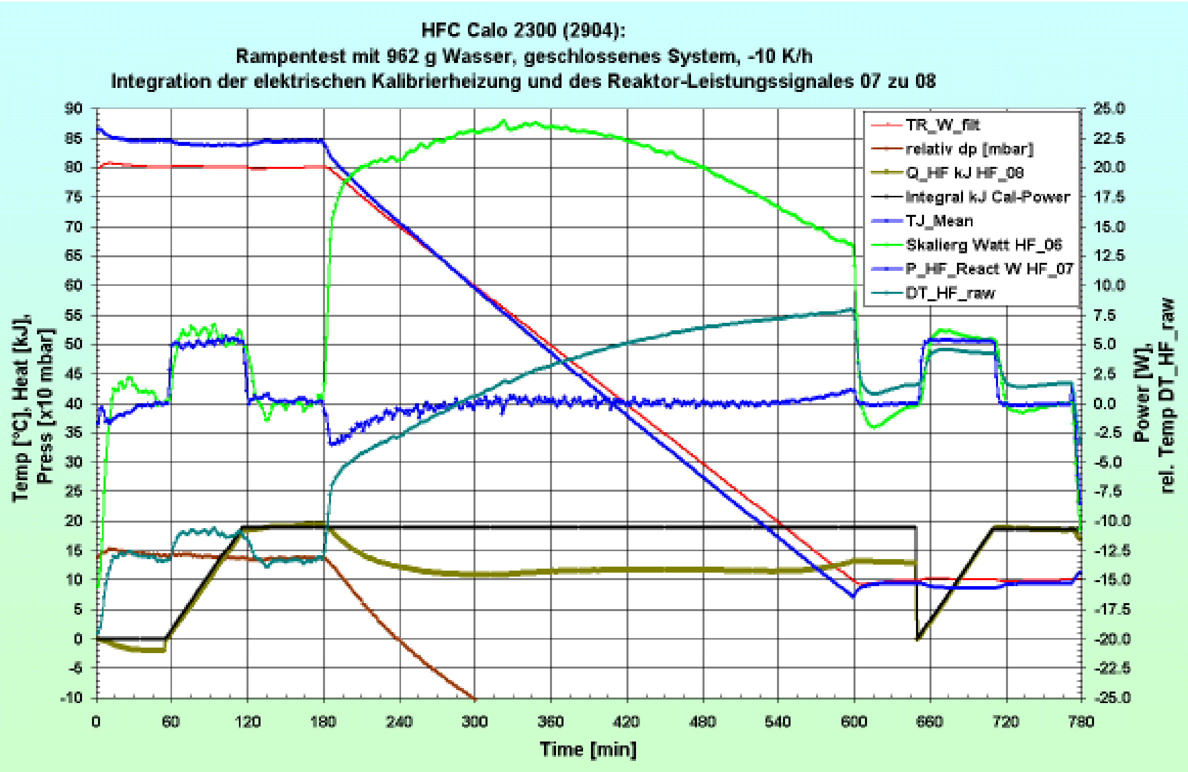
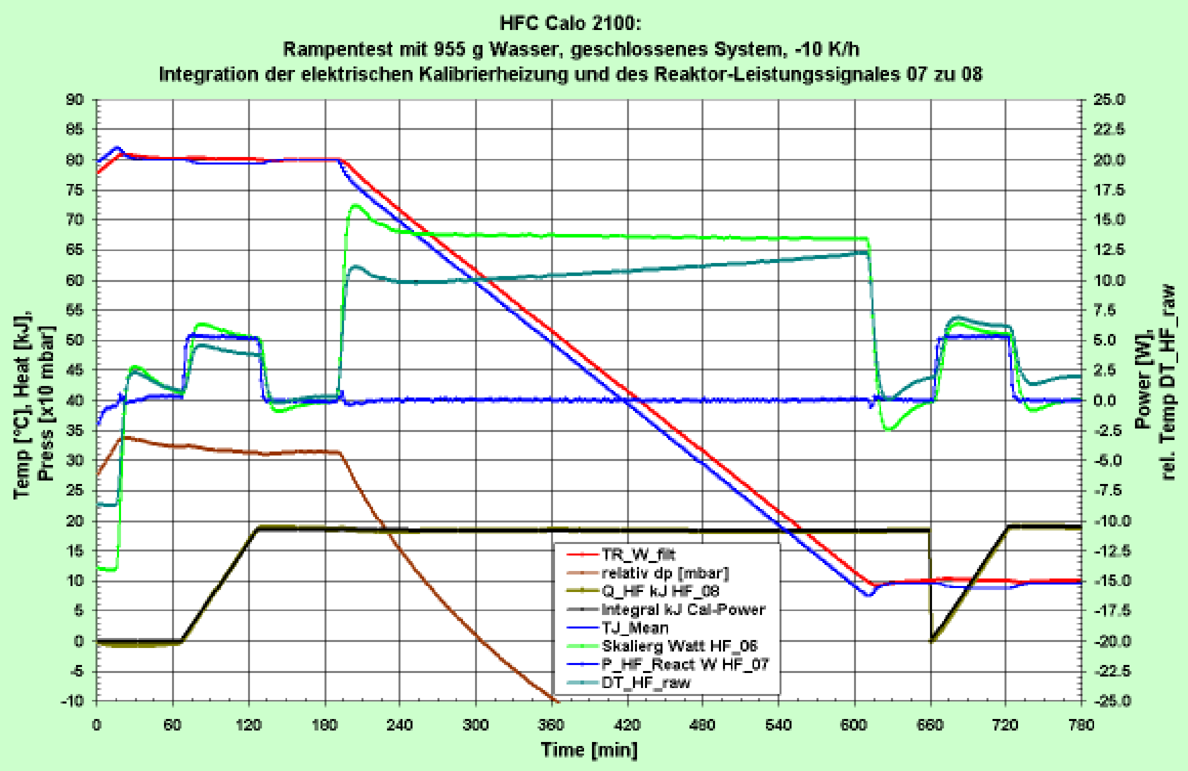


Figure 11
Integration of the chem. power curve and the calibration heating (set 0 just before calibration) for Calo 2100 or Calo 2200



Comments

Signals Calo 2300, Calo 2904, Both Without Box

All curves discussed up to now are also included in the figures presented here. Two integrals have been added: the integral of the electrical calibration heating (black) and the integral of the chemical reaction power (yellow-green). Both integrators were set to 0 immediately before switching on the calibration heating.

The yellow-green energy curve Q_HF, signal HF_08 in [kJ] gives the measured overall energy from the start of the first calibration heating at 80°C to the start of the second calibration at 10°C. What could have been expected from the beginning turned out to be true: the error at the transition from isothermal to the ramp is very pronounced with ca. 9 kJ. But the integral is rather stable from ca. 70°C on (changes smaller than +/- 2 kJ).

The conclusion from this: at transitions errors always have to be expected and this error is dependent on the slope of the ramp. If the chemical power is in the order of 100...200 kJ, then this error is of minor importance. If the reaction energy is small this error has to be avoided or the kink has to be put outside the zone of evaluation.

Signals Calo 2100 and Calo 2200 - Temperature Controlled Box

The integral from 80°C up to 10°C is extraordinarily stable and does not leave room for improvement. The smallest energies can be measured even over kinks.

As a consequence this is the system of choice for measuring smallest reaction energies like they occur in crystallizations in organic solvents. Example of a pharma product: ca. 50..100 J/g with for example only 80 g substance in 1 l solvent results in ca. 4 to 8 kJ. Due to the good kinetics it is possible to distinguish what is happening when and whether there are recrystallizations occurring and, and, and...

Comparison of Power and Energy of HFC & HBC of the Same Experiment

Figure 12
Comparison of HFC & HBC of the same experiment for Calo 2300 or Calo 2904

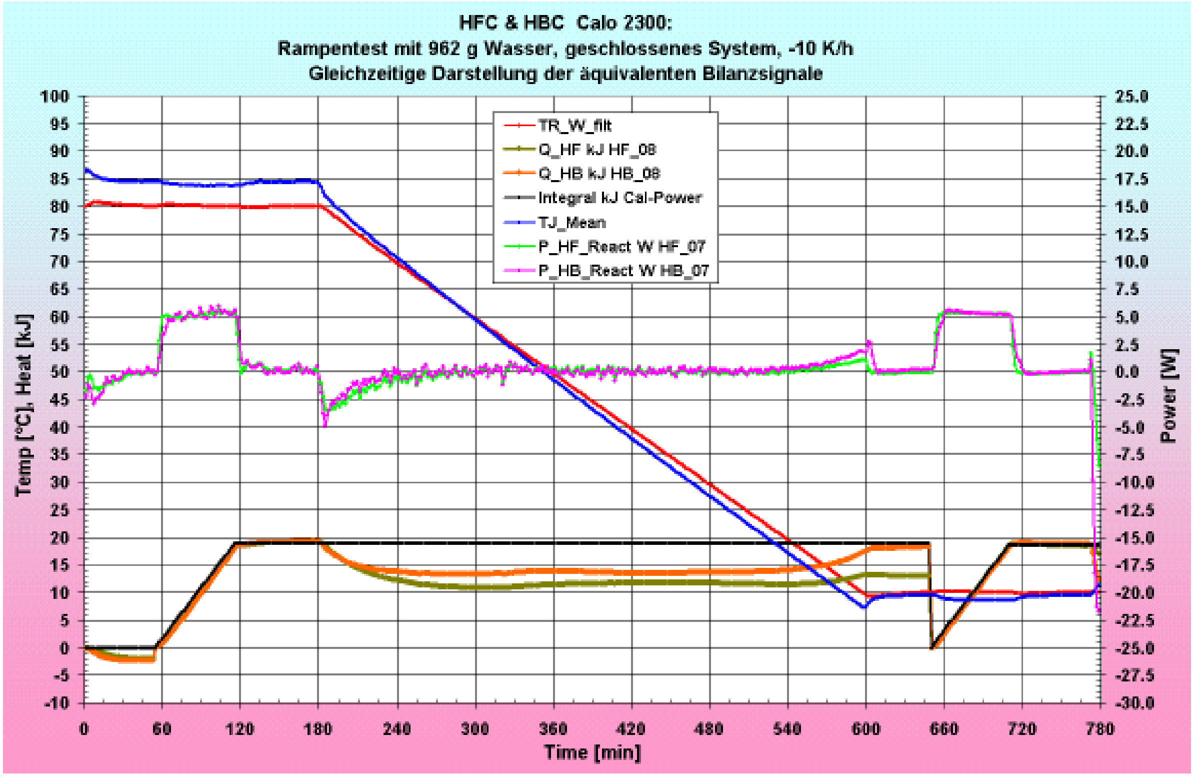
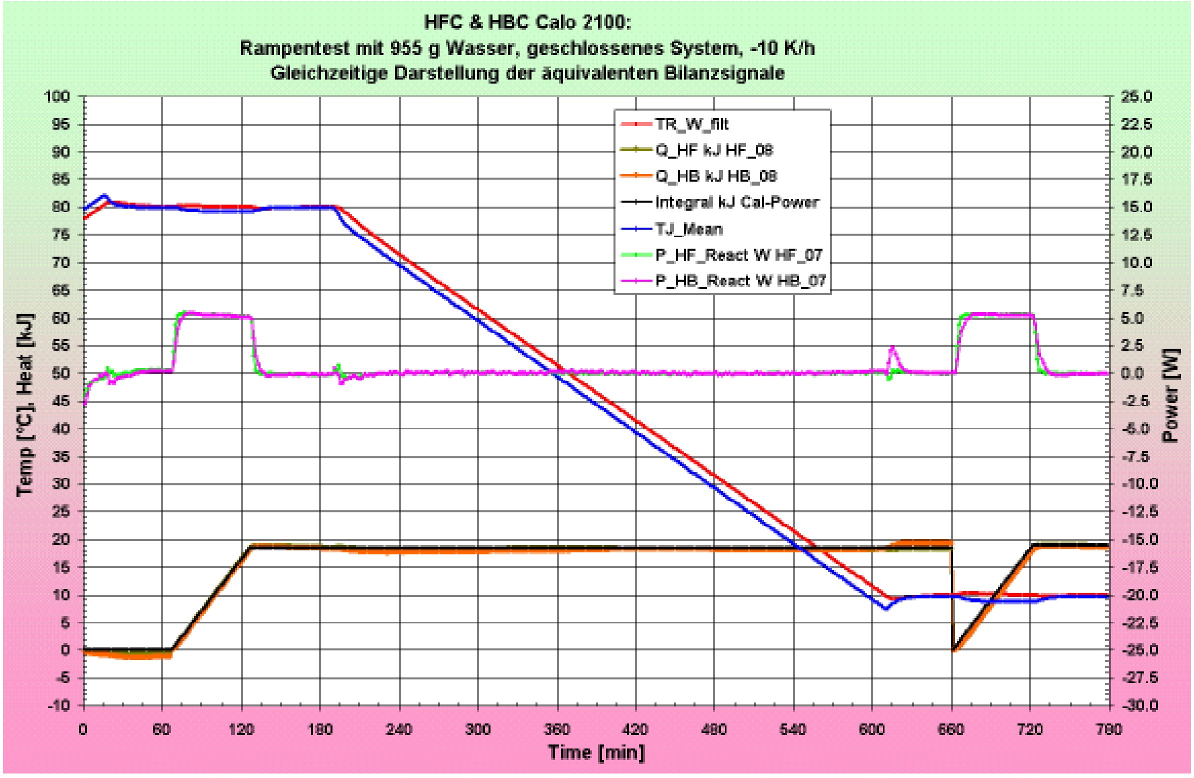


Figure 13
Comparison of HFC & HBC of the same experiment for Calo 2100 or Calo 2200



Comments

Signals Calo 2300, Calo 2904, Both Without Box

Up to now we have only shown signals of the heat flow measurements. This should enable you to compare with the conventional calorimeter used nowadays. The measurement of the heat balance is very similar therefore it is not necessary to show this a second time. Here we are going to show the direct comparison of the two independent methods.

Mind You:

Both methods of measurement are simultaneously applied in the Calo 2100, the Calo 2200, and the Calo 2300. Therefore you are always provided with two independent measurements at the same time; it is almost like your own interlaboratory study! In all calorimeters of the type Calo 290X there is only one heat-flow calorimetry therefore there is no heat balance measurement in the reactor. This can lead to problems with inhomogeneous samples and samples which change in the course of reaction (compare ref. [1]).

The evaluation shows in this experiment an almost identical behaviour of the heat balance measurement and the heat flow measurement. Only the noise and the interference at the kinks is somewhat higher. This is generally true for measuring the balance because this is more critical and yields less raw signal. But in various different applications this method proved to be more robust and therefore the reliability of deductions from results obtained by this method is very high.

The integration of the balance measurement is extraordinary as well. This yields a signal with very little deviation; only -6 kJ in comparison to the heat flow measurement with -9 kJ.

This agreement is only true as long as no inhomogeneities or changes in viscosity occur. In ref. [1] your are provided with more information on this subject.

Signals Calo 2100 and Calo 2200 - Temperature Controlled Box

The integration of both signals P_HF and P_HB is very convincing. Q_HB shows a small oscillation in comparison to Q_HF. This is caused by the slightly higher noise at the transitions from isothermal to ramp conditions. The measurement of Q_HB is stable over the whole range within +/-1 kJ.

Information:

The power used to heat the reactor and the sample during the ramp was with the Calo 2300 (2904) ca. 25 W (radiation and evaporation) and with the Calo 2100 (2200) ca. 15 W (here less power is necessary due to the support by the thermostated box, no losses, but low additional radiation). A rest error is therefore equal to, for instance, 1 W (0.5 W) and this is equal to ca. 4 % (3 %) of the used power.

HFC & HBC at +60 K/h (Batch-Simulation)

Figure 14
Fast heating ramp for Calo 2300 or Calo 2904

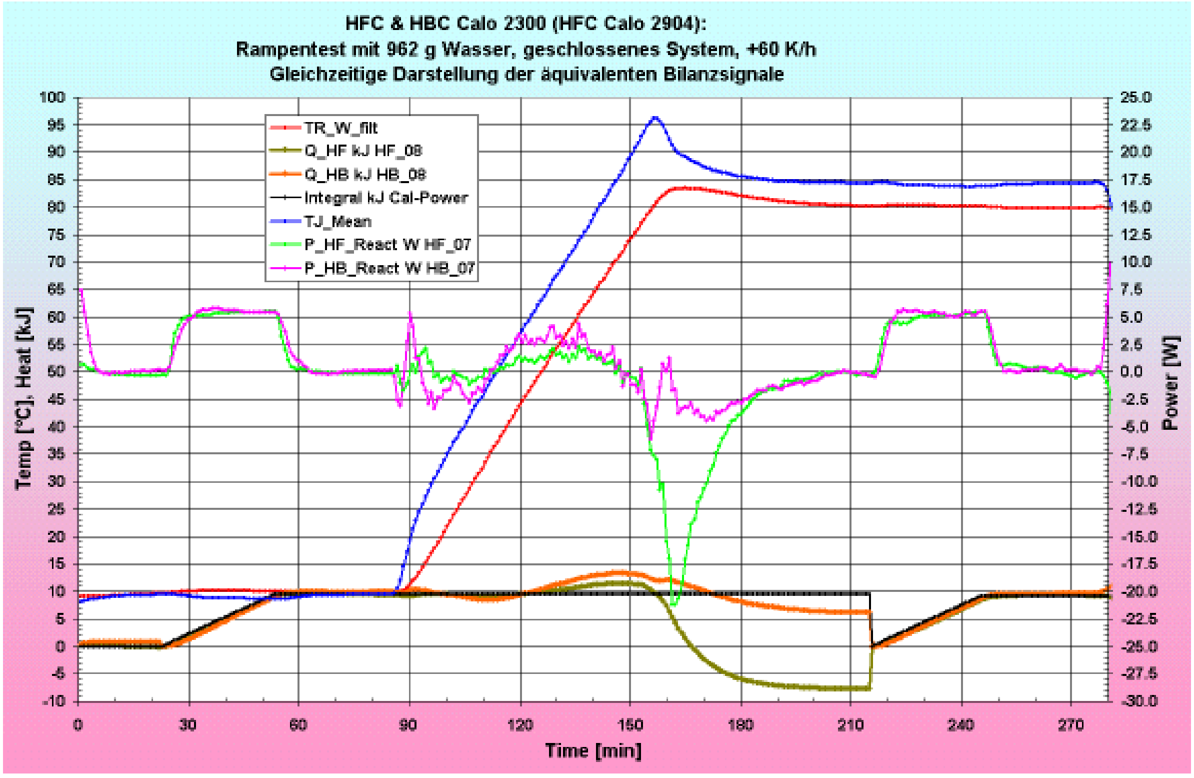
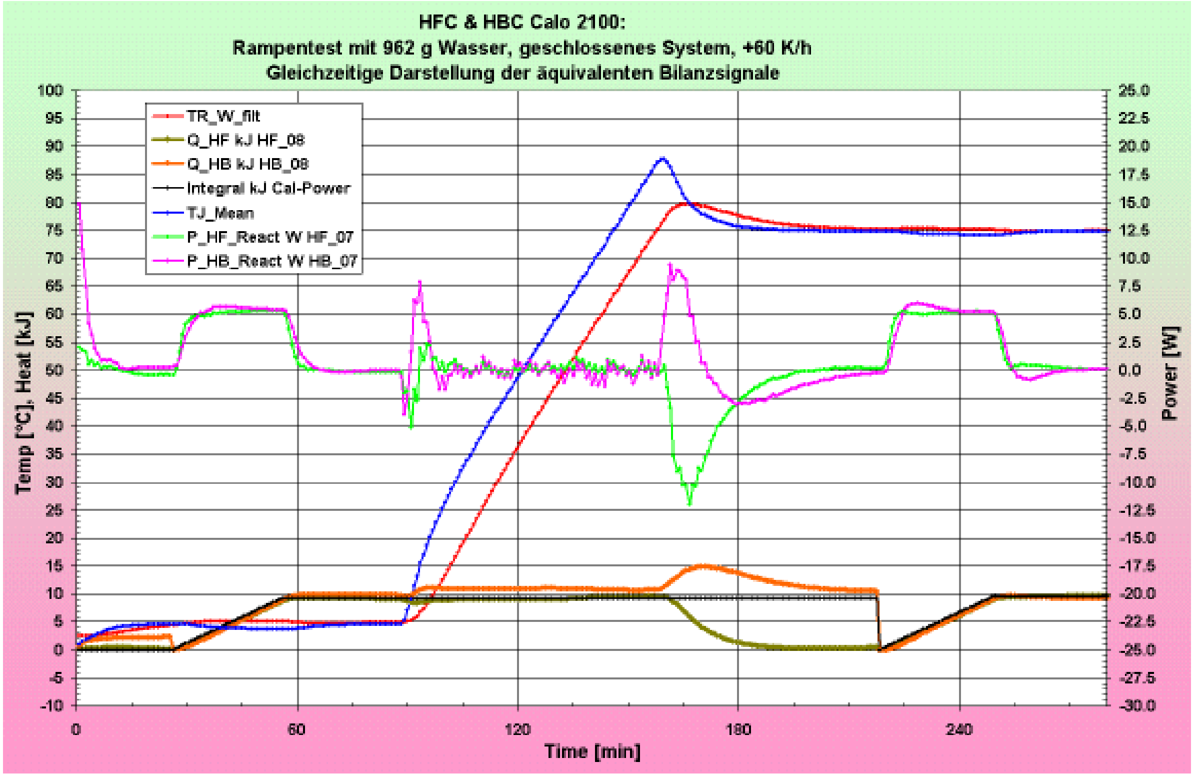


Figure 15
Fast heating ramp for Calo 2100 or Calo 2200



Comments

Signals Calo 2300, Calo 2904, Both Without Box

Up to now we have been dealing with the slow, negative ramp. This ramp is of great interest for sensitive crystallizations and exhibits fundamental properties of the whole system during slow ramp conditions. In practical use fast positive ramps are necessary as well (preparation of the reaction mixture; heating to start the reaction, possibly up to refluxing to finish the reaction).

The following is apparent: The interferences are remarkably stronger and especially the transitions from isothermal to ramp conditions leads to a strong disturbance of the system. This is due to the model assumption which only takes into account a single reference point per subsystem and does not consider a distributed system. Especially when looking at the overshoot of the jacket temperature when the final value of 80°C is reached, it becomes apparent that there are strong thermal flows within the whole reactor system including the walls (water equivalent) which lead to inhomogeneities not only in the sample but in the whole reactor system. This especially influences the measurement of the heat flow. The error of this measurement becomes apparent in the integration. The integration of the heat balance gives a moderate error of +/-4 kJ compared to the error of the heat flow measurement of ca. -18 kJ. This once again demonstrate the robust behaviour of the heat balance measurement.

All in all an uncertainty of ca. +/-4 W (peak) in the ramp has to be anticipated for both measurement methods. With high reaction powers the measurement uncertainty is not strongly influenced.

Signals Calo 2100 and Calo 2200 - Temperature Controlled Box

This experiment was carried out as one of the first from 5°C to 75°C. Due to the danger of freezing in the reactor during the cooling down phase to 5°C, the range was changed to 10°C to 80°C.

In comparison to the Calo 2300 the ramp phase is much more stable and shows an error in the order of +/-1.5 W, i.e. ca. 2.5 x better than before. The integration of the power shows this even more impressive: both curves, the Q_HF and the Q_HB curve, are horizontal in the region of the ramp.

Information:

The power to heat the reactor and the sample during the ramp was in both cases ca. 100 W. A residual error of 1.5 ... 4 W is therefore ca. 1.5 ... 4% of the applied power.

Conclusion

6 Requirements Influencing the Choice of the Calorimeter

The representation of the kinetics in a non-isothermal system calls for the application of a comprehensive model of the whole system. Only this enables us to eliminate most of the interferences - or there influence is at least diminished to a reasonable extent - and to make reliable assertions on the absolute values of the power and of their sequence in time.

The investigation and the determination of the energy in non-isothermal behaviour requires high performance equipment exceeding the common standards available on the market. The choice of a certain calorimeter is therefore dependent on the requirements which have to be fulfilled.

For most users confronted with calorimetry for the first time calorimetry is an enigmatic issue. Therefore for many people it is often very hard to find the right and essential specifications for a calorimeter.

This paper is aimed at shedding some light on the topic "reaction-calorimetry". In combination with ref. [1] the reader should be able to determine the necessary specifications of a calorimeter for a certain application.

7 Training and Test Measurements

Periodically we offer training workshops. Please contact the nearest representative for further information. For test measurements do not hesitate to contact our representatives as well.

Appendix

8 Experimental

960 g deionised water was used as sample. The Experiment was carried out in a cylindrical 1 l glass reactor with vacuum insolation (reduction of **Qloss**).

All examples were carried out in a closed sealed reactor to minimize evaporation (**Qevap**).

In the **Calo 2100** the reactor was equipped with a temperature controlled box and an additional heating for to fine adjust of the jacket temperature.

With the **Calo 2300** (with the **Calo 2904**, only HFC) the reactor was situated in ordinary room atmosphere and the jacket temperature was only controlled by the thermostat without fine adjustment as with the Calo 2100.

9 References

[1! Peter E. Meier, 'Reaktionskalorimetrie - Untersuchung über Tauglichkeit von Wärmefluss- und Wärmebilanzkalorimetern bei nicht-isothermen, homogenen und inhomogenen 2-Phasen Systemen (fest-flüssig)', ***SYSTAG***, Juli 2000, ***Symposium PhandTA*** *in Basel*, 27.9.2000.

The author can be contacted under: peter_meier@systag.ch.