

Calo 2000

The first true non-isothermal Reaction Calorimeter

Report #1: Reproducibility & Accuracy

Report #2: Dynamic response

Summary

Calo 2100 and 2200 are the first calorimeters which allow working also without any calibration:

If you are happy with a first accuracy of about 5% to 10% you do not require any cp determination or use of calibration heater or anything else. You can speed up your investigations, also for unstable chemical products it's a big improvement! You can follow the information at the screen, you see immediately what happens and have the power and heat results on-line!

A calibration heater can be used only as a supervision, a 10 minutes pulse with also a 10 minutes pause is ok.

All the following investigation modes deliver accurate results:

- a) Reactor controlled, isothermal temperature (constant)
- b) Jacket controlled, isoperibol temperature (near constant)
- c) Reactor controlled, "isothermal" ramp temperature (constant ramp)
- d) Jacket controlled, "isoperibol" ramp temperature (near constant ramp)

The most accurate time response you get in the isoperibol mode but reaction temperature is not stable. For accurate Arrhenius equation determination it's better to use the isothermal mode with a more oscillating response due to cascade control. Oscillation has no accuracy influence to the heat integration.

First improvements can be made with only zero drift calibration; no calibration heater is necessary, only a reaction free part is required.

Further improvements of measurements can be achieved by using the calibration heater together with some stabilizing time combined with a reaction free ramp. Under such conditions you are able to adjust your experiment (off-line, after experiment) to about 1..2% accuracy.

Cp determination is no longer required, if you have a rough knowledge of cp, density and specific weight of your supply and additions you are using. The rest is calculated by Calo 2000!

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1 REPORT #1

1.1 SYSTAG's high-tech development behind 100 years old brick walls!



In front the sales, administration, accounts and other offices. In the background rooms for development, final assembly and acceptance tests.

1.2 Development and test system Calo 2100



Whole system Calo 2100

Test system with monitor very left, control cabinet with Combilab modules, main cabinet Calo 2100 with distillation at left and dosages at right.

The thermostat UNISTAT 160w (water cooled) is packed to reduce noise and right the N2 bomb to purge calorimetry cabinet.



Open system cabinet Calo 2100

1 litre glass reactor, cylindrical, triple wall. The aluminium packed unit is the fine heater.

1.3 Main purposes of investigation: reproducibility and accuracy

The goal was to determine the reproducibility and accuracy of the system, first with water based solutions and second with an organic solvent. The reason is: water has a very high cp in comparison with organic solvents, also the vapor phase is quite different with their heat conductivity, therefore some differences may occur. First we have made a system calibration with only water. It was now the goal to get knowledge of differences, time responses and also about standard deviations.

Therefore we have done the following procedures:

According to a customer wish we started immediately with dilution of H_2SO_4 as a water based system.

Thereafter we have investigated the alcoholysis of acetic anhydride in methanol as an organic system.

Each investigation was made under the 4 operation conditions:

- a) Reactor controlled, isothermal temperature (constant)
- b) Jacket controlled, isoperibol temperature (near constant)
- c) Reactor controlled, "isothermal" ramp temperature (constant ramp)
- d) Jacket controlled, "isoperibol" ramp temperature (near constant ramp)

Thereafter the evaluation was done for all investigated systems for HF (heat flow) and HB (heat balance), therefore each experiment delivered two results!

- 1) First we evaluated the raw value: this is the result we can get immediately, also on-line, because it's only based on the basic calibration of the system.
- 2) Second we have made, if it looks necessary, an adjustment for better accuracy, which can not be done on-line! We have found, that the water based experiments were enough accurate and have omitted these adjustment there. Only the organic systems was adjusted because it looks more critical.
- 3) As a result we have calculated the mean value and the standard deviation (mean square deviation) of the 8 results of one group as a quality information.

As an additional investigation we have analyzed the response behavior of an isoperibol experiment during dilution of H_2SO_4 (no accumulation) and also a calibration pulse of 10 minutes.

1.4 Investigation of dilution of H₂SO₄ in water

Remark:

Because it was a customer wish we had to use what we had:

Problem of SYSTAG's H₂SO₄:

We had no unused H₂SO₄ on stock. We have taken an old bottle with used H₂SO₄ (it was used sometimes for WE determination, up to two or three years ago during some days), the original concentration was 95..97%, the actual concentration was unknown.

Dilution of H₂SO₄ in H₂O

Basic information:

H ₂ SO ₄	98.08 g/mol	H ₂ O	18.01 g/mol
	1.834 g/ml		1.00 g/ml
	1.4192 J/g.K		4.185 J/g.K

1. Customer information about dilution experiment:

H₂SO₄, 98%, 555.5 g in 1000 g H₂O, slowly added over 2 hours: 93 kcal

93 kcal * 4.185 = 389 kJ

100% --> 100% * 389 / 98% = 397 kJ for 100% concentration (approx.)

555.5 g --> 5.663 mol 1 mol --> 70.1 kJ dilution energy in 176.6 g H₂O

2. Some information about Calo 2100 calibrations, corrections and experimental conditions:

The system (1 l cylindrical glass reactor with triple wall) was calibrated some days before with 1015 g water at 5, 25, 45, 65 and 85°C with a UNISTAT 160w and all parameters were evaluated. Therefore it's ideally suited for experiments which are using also water.

After this first calibration, the addition check was made: Supply of 350 g H₂O at 5°C with an addition of 350 g H₂O at 25°C. After this test, a small correction (fine Temp. Coeff. Corr.) as well as a fitting figure for A was adjusted.

The following experiments were never corrected, that means only the calibrated system was used without any further corrections - or in other words - it is the same as you would do the experiment without any calibration before and after experiment!!

One exception: The pulse response time test was exactly fine-tuned, HF and HB zero must be shifted by 0.15 or 0.2 W and the scale factor had also changed by about +2%.

The reactor content was stirred with a special propeller at 250 rpm. It is made of stainless steel and we were a little afraid about the behavior of this stirrer with H₂SO₄. At 25°C, nothing happens but at higher temperature....! We were not able to change it before but we will do that later. Details are described later in the ramp experiment.

1.4.1 Reactor controlled, isothermal 25°C, addition H₂SO₄

We started with 350 g H₂O at 25°C and added 200 g H₂SO₄ during 60 min. We have chosen reactor (cascade) control.

The evaluation has given: 124 kJ for HFC and 125.2 kJ for HBC. The confidence range is therefore approx. +/-0.5% !

200 g = 2.039 mol --> 142.9 kJ for 100% concentration (customer information) - 124 kJ --> 87% concentration.

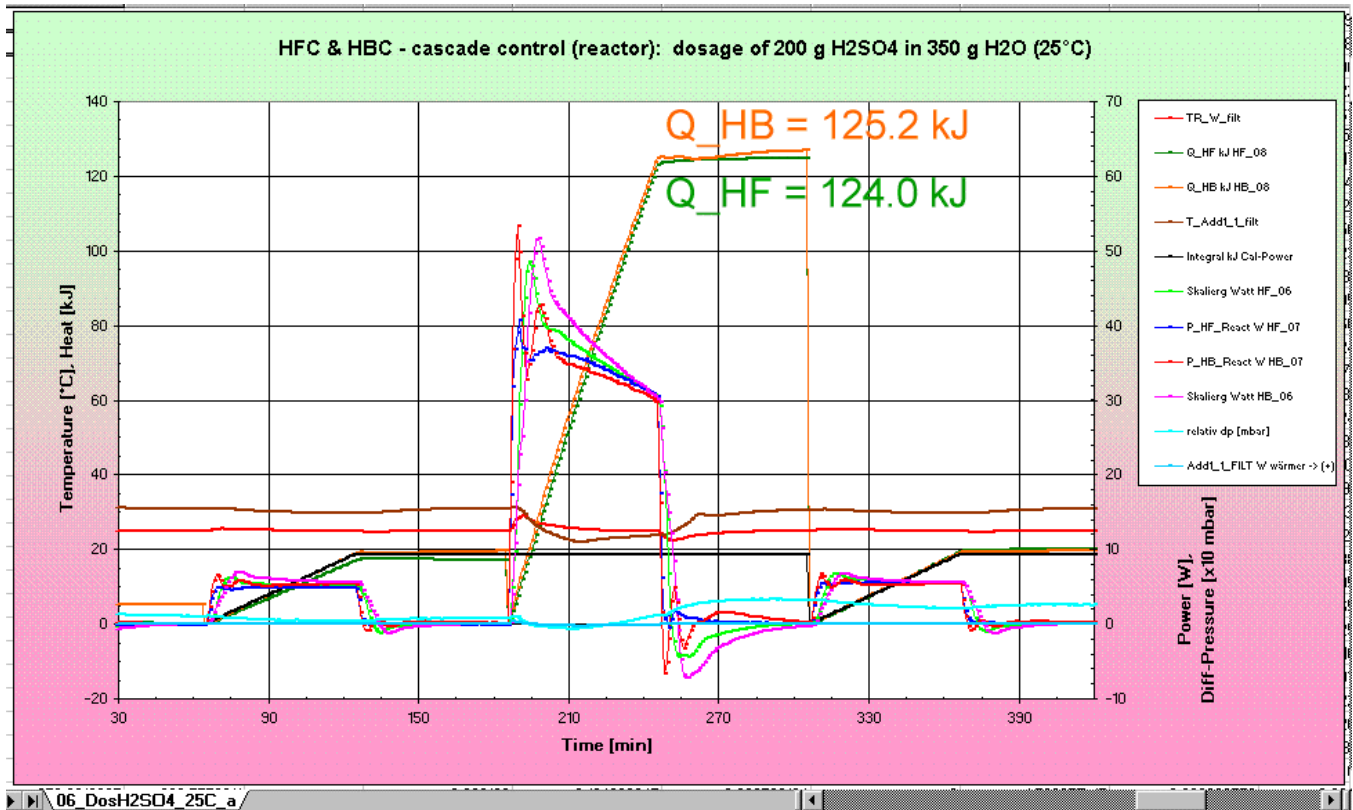


Diagram description:

The interesting processed curves are the blue (measured power [W] of reaction or calibration) and middle green (integration of the power [kJ] for total energy) for HF and the red (measured power of reaction or calibration) and orange (integration of the power for total energy) for HB.

- The light green (HF) or the pink (HB) is the transformed and pre-scaled power information, derived from the temperature measurements.
- The black shows the integration of the electrical power [W] of the calibration heater.
- The light red curve shows the temperature TR [°C] and the brown the temperature at the addition inlet [°C] (higher because of cabinet heating).
- The light blue curve is the reactor pressure difference to the atmosphere outside in 10 mbar per scale division. It indicates that the system is nearly tight. The measured pressure difference is about max. +40 mbar.

1.4.2 Reactor controlled, "isothermal" ramp 5°C to 65°C, addition H₂SO₄

The same experiment was done in a ramp to show how it is working. We have chosen reactor (cascade) control. The supply of 350 g H₂O was cooled down to 5°C, then also calibrated with 5.21 W, the ramp was started with 10 K/h, after 1 hour the addition of 200 g was started for 1 hour. Thereafter we let ramp the system up to 65°C, thereafter a second 5.21W calibration was done.

The results: HF 123.7 kJ and HB 120.2 kJ. But above 50°C the system deviates from the base line (it should be reaction free). Also we have seen at the next day (it was running by night) that the whole content was really black as well as the SS stirrer! My interpretation is: there must be a reaction which produces around 1W of exothermic energy, starting after 50°C.

A lot of vapor production and losses can be nearly excluded because the system pressure difference was risen to about +500 mbar and was not released (blue curve)!

The calibration pulses are only for checking the system, not more. Also here no fine calibration after experiment was done.

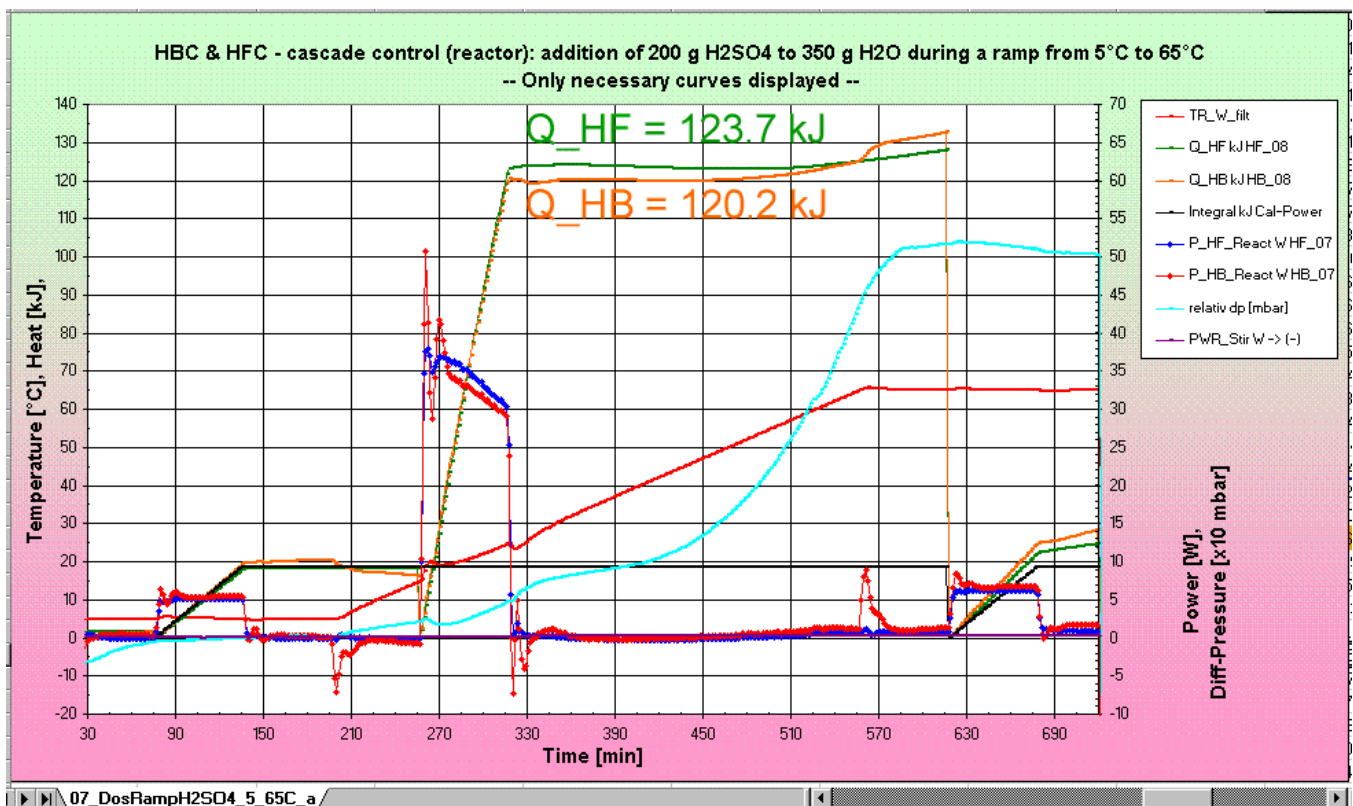
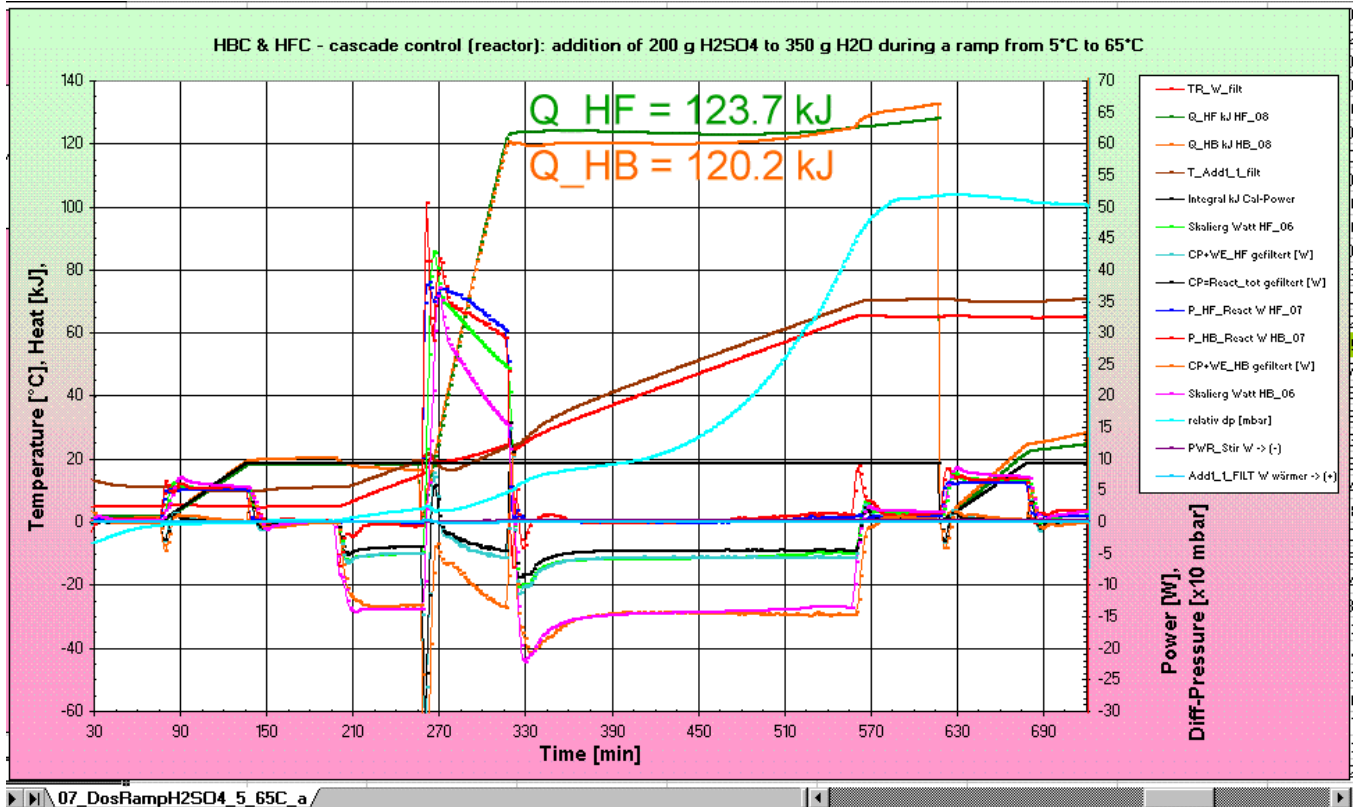


Diagram description, marked "only necessary curves displayed"

The interesting processed curves are the blue (measured power [W] of reaction or calibration) and middle green (integration of the power [kJ] for total energy) for HF and the red (measured power of reaction or calibration) and orange (integration of the power for total energy) for HB.

- The black shows the integration of the electrical power [W] of the calibration heater.
- The light red curve shows the temperature TR [°C].
- The light blue curve is the reactor pressure difference to the atmosphere outside in 10 mbar per scale division. It indicates that the system is nearly tight. The measured pressure is about max. +520 mbar.
- The violet curve is the corrected stirrer power which is always below 1 Watt.



Description of the dense diagram (with a lot of curves)

There you can recognize also the value of Cp (black), the difference from the Cp to the transformed temperature measurement and scaled in Watt: light green for HF and pink for HB. The other orange line (HB) or light blue (HF) (both = Cp+WE) is the real base line and fits nearly over the full range the pink or light green curve. Only above 50°C you can recognize that the measured lines increase for round about 1 W and stays also during the isothermal phase above the zero line. This leads to an increase in energy Q.

SUMMARY for ISOTHERMAL constant and ramp runs with CASCADE CONTROL:

All results are between 120 and 125 kJ WITHOUT ANY FINE TUNING! Which measurement is correct? I can not answer it but the confidence range is about +/-2%. That means you can get the same information also on-line with the same accuracy because it's only based on the system calibration.

1.4.3 Jacket controlled, isoperibol, constant 25°C, addition H₂SO₄

We started with 350 g H₂O at 25°C and added 200 g H₂SO₄ during 60 min.

The evaluation has given: 123.1 kJ for HFC and 125 kJ for HBC. The confidence range is therefore approx. 1% ! In experiment no. 3 we got 124 kJ for HFC and 125.2 kJ for HBC, we are therefor very, very close!

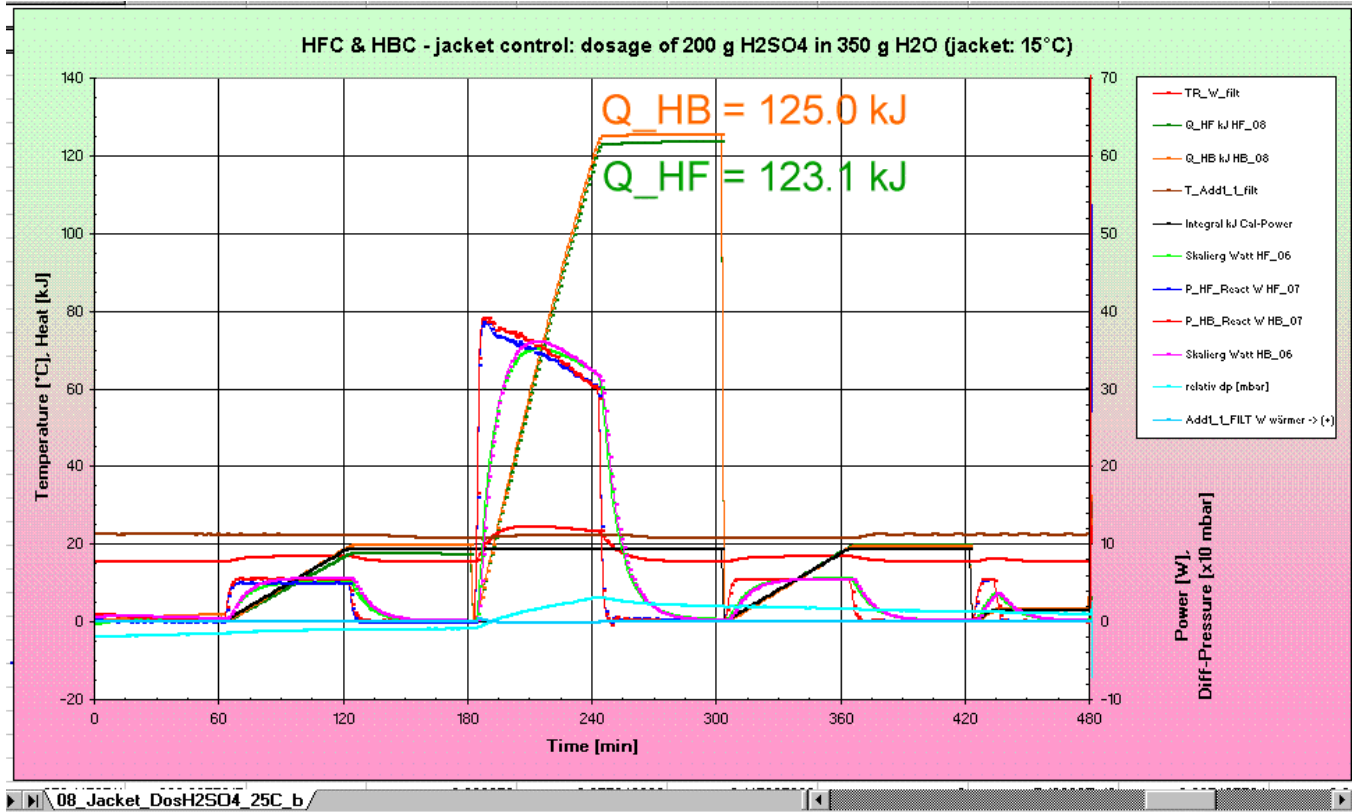


Diagram comment:

You can recognize immediately, that there is much less oscillation during changes (at start or at the end of dosage). The power behavior looks much more accurate/stable over the whole period! It shows that the jacket control delivers the better response of the process. If you compare the measured, transformed and pre-scaled light green and pink curves you can see the very slowly reaction of the temperatures (near 20 minutes). After processing our response is as fast as before!

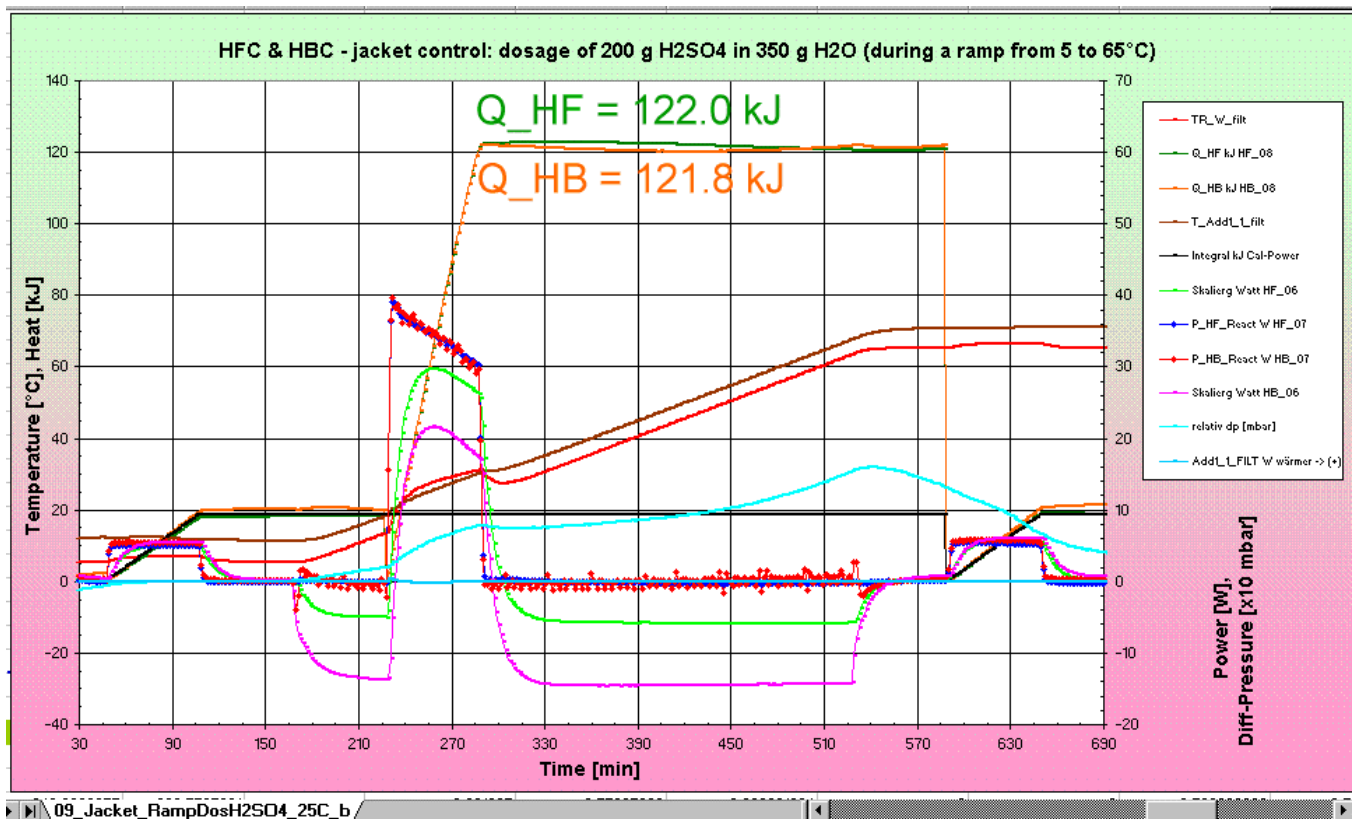
1.4.4 Jacket controlled, "isoperibol" ramp 5°C to 65°, addition H₂SO₄

The same experiment as no. 2 was done in a ramp to show how it is working under jacket control. The supply of 350 g H₂O was cooled down to 5°C jacket, then also calibrated with 5.21 W, the ramp was started with 10 K/h, after 1 hour the addition of 200 g was started for 1 hour. Thereafter we let ramp the system up to 65°C, thereafter a second 5.21W calibration was done.

The results: HF 122 kJ and HB 121.8 kJ instead of HF 123.7 kJ and HB 120.2 kJ before. Also above 50°C nothing is to see as in no. 2! We have seen that also the content stayed clear, no further reaction occurred!

The vapor production produced less pressure, I guess the system was not as tight as before: max. 160 mbar and a loss during the stable temperature at 65°C.

The calibration pulses are only for checking the system, not more. Also here no fine calibration after experiment was done or necessary.



Description of the diagram

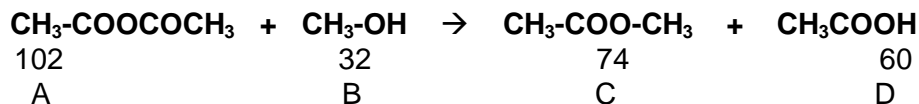
Also here nearly no oscillation but a very slowly response time of the light green and pink curves (derived from temperatures). The processed reaction power signals (red for HB and blue for HF) are also very fast and show a very good behavior. Only the noise of HB is larger than of HF or during the cascade controlled system.

The stability of the zero line from 5 to 65°C is very good without any adjustments!!!

1.5 Investigation of alcoholysis of acetic anhydride in methanol, uncorrected

First some information about theory:

Reaction:



Theory:

Product	Heat Capacity	Formula Weight	Heat of Combustion
A	185.4 J/mol.K	102 g/mol	-1807.1 kJ/mol
B	81.6 J/mol.K	32 g/mol	-726.6 kJ/mol
C		74 g/mol	-1593.4 kJ/mol
D		60 g/mol	-872.8 kJ/mol
Summary of Enthalpies:			-67.9 kJ/mol

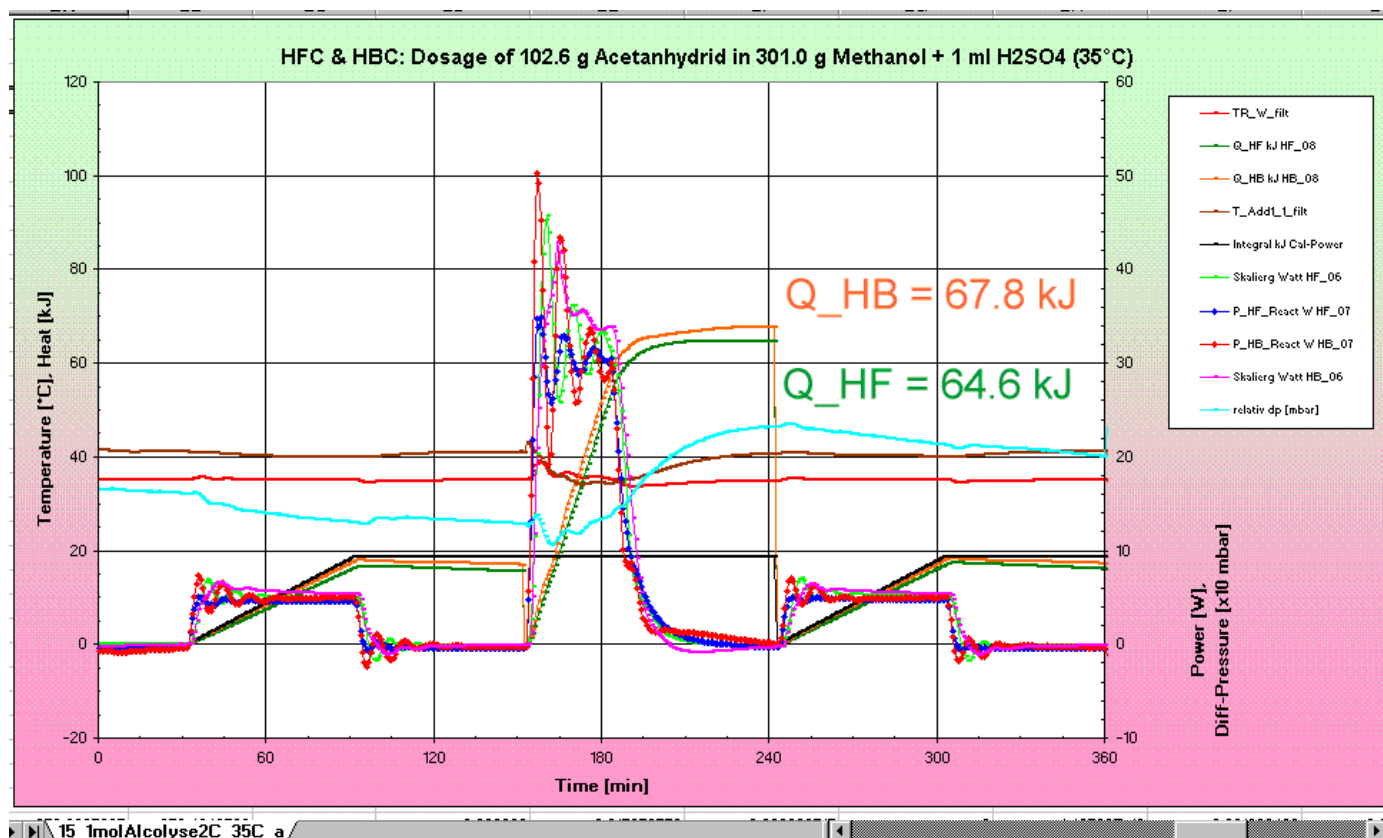
1.5.1 Reactor controlled, isothermal 35°C, addition acetic anhydride

In the following experiments, all calibration heaters were switched on during 90 minutes. This is normally never necessary but for system calibration and system information it's a must. Do not feel that all experiment require such a long calibration!! See for that chapter 2.1.1 Calibration heater response with jacket control.

All tests were made in the reactor with only a base system calibration (with 1 lit water, the same as before).

The temperature control was more sensitive and tends to oscillate a small amount (can be partly corrected later by a larger P-Band).

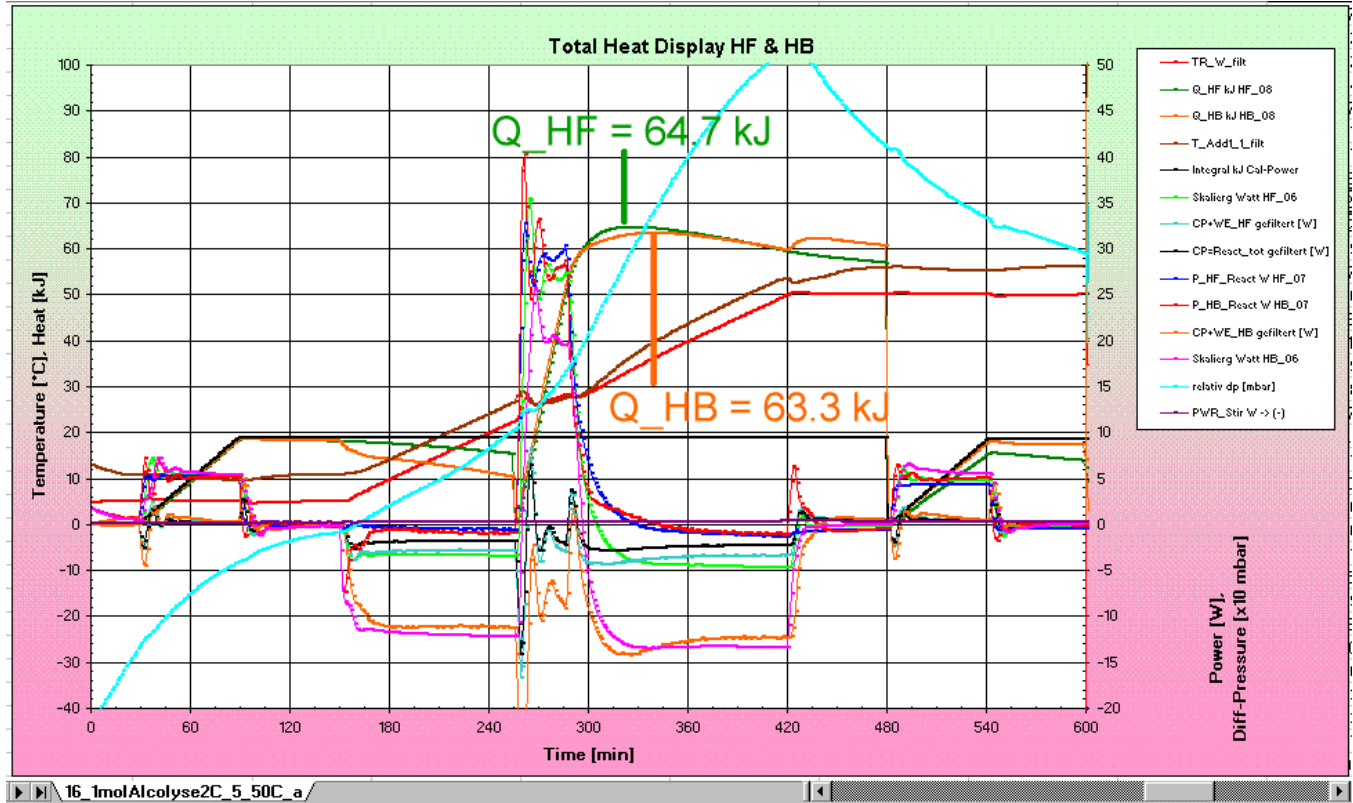
A part of accumulation can also be seen.



Results are discussed in chapter 1.7 Evaluation of mean value and standard deviation.

1.5.2 Reactor controlled, "isothermal" ramp 5°C to 50°C, addition acetic anhydride

Also 300 g methanol and addition during 30 minutes of 102.5 g acetic anhydride during the ramp.

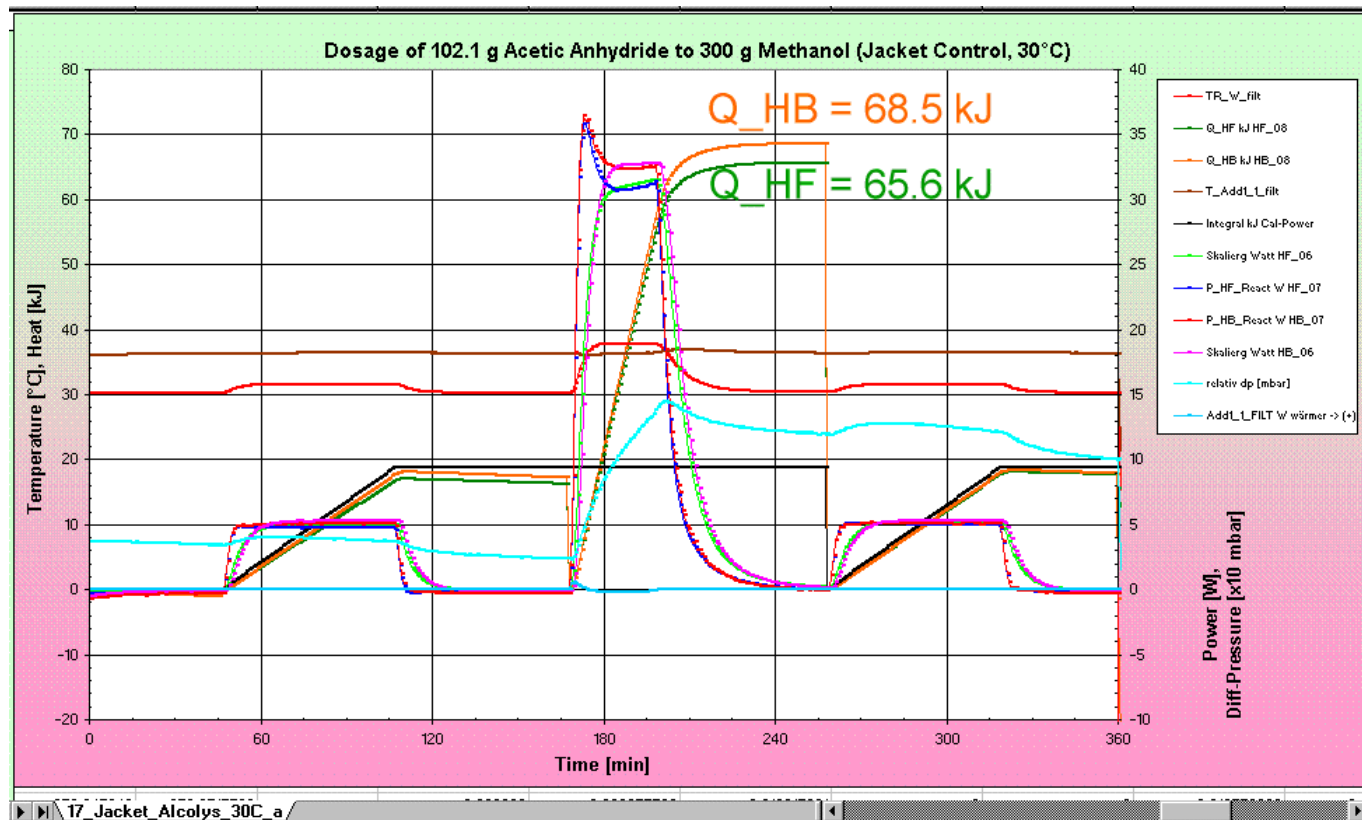


We can recognize a zero drift during real isothermal phase and a wrong water value, therefore the bend of integral reduction occurs. More about such problem zones is given in chapter 1.8 Some words about off-line corrections / adjustments.

Results are discussed in chapter 1.7 Evaluation of mean value and standard deviation.

1.5.3 Jacket controlled, isoperibol, constant 30°C, addition acetic anhydride

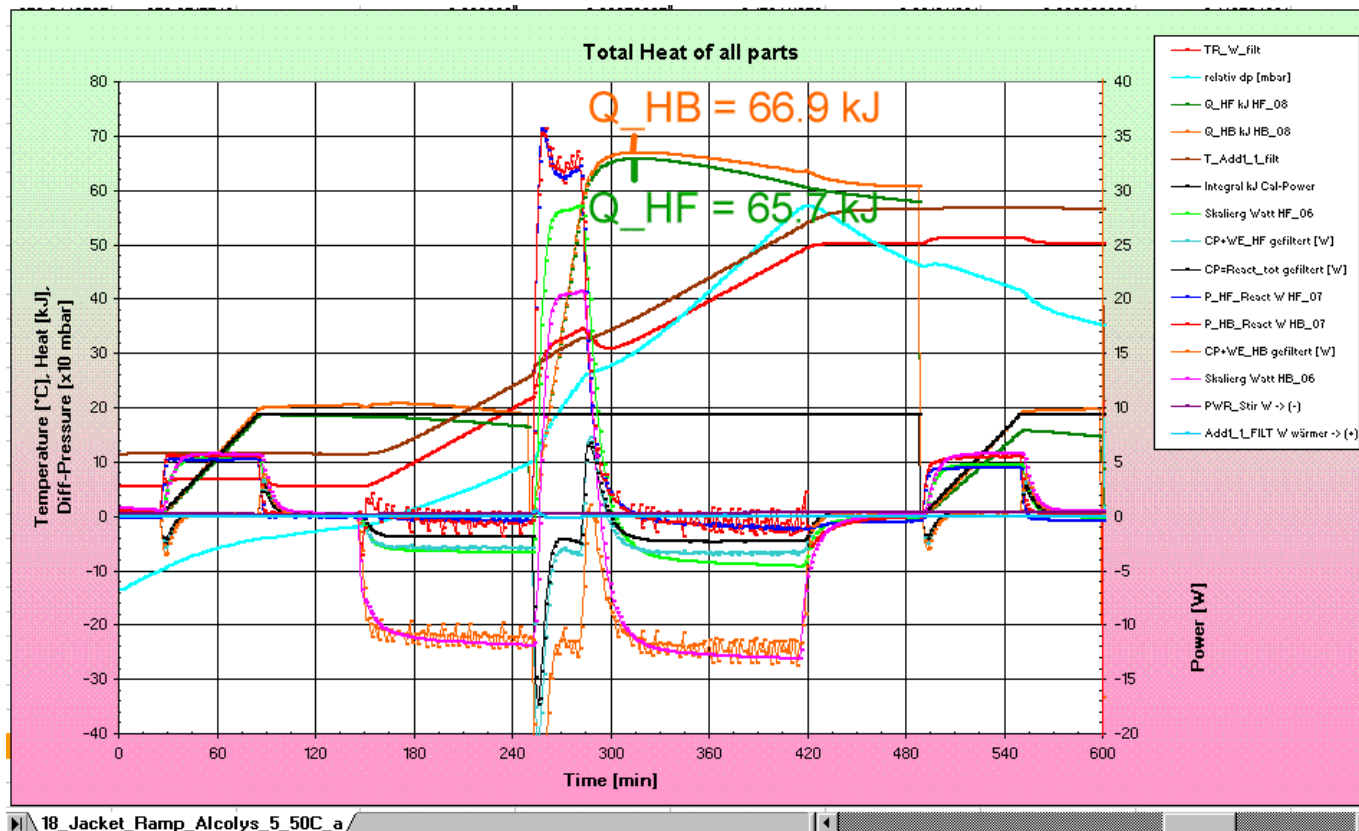
The isoperibol behavior shows an increase in reactor temperature during reaction of about 8 K. But the real power of reaction has no oscillation as with reactor or cascade control.



Results are discussed in chapter 1.7 Evaluation of mean value and standard deviation.

1.5.4 Jacket controlled, "isoperibol" ramp 5°C to 50°C, addition acetic anhydride

The same we can recognize as with reactor control: a shift of zero line and an mismatch of WE (water equivalent) which leads to a bend heat curve. Therefore we took the maximum value as result!



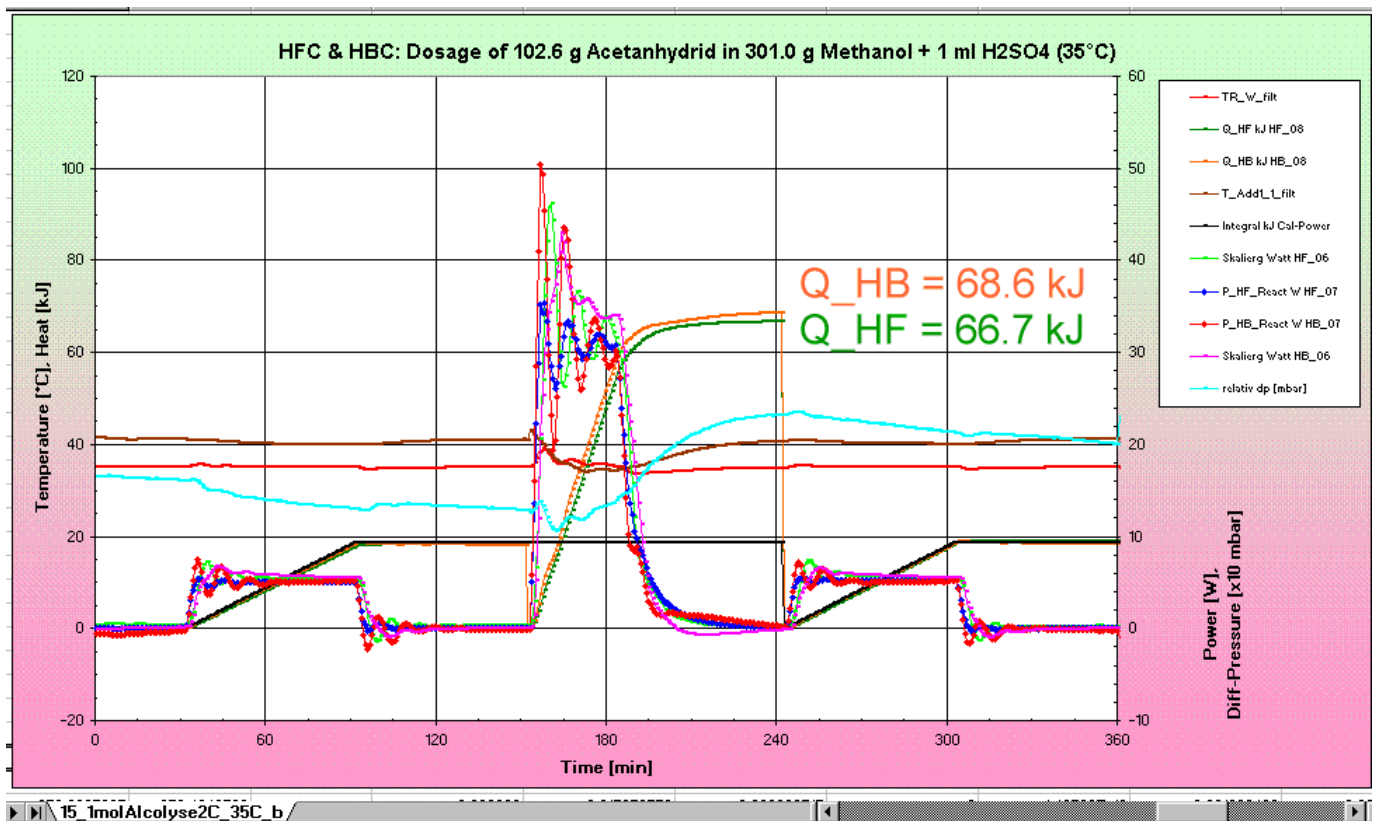
Results are discussed in chapter 1.7 Evaluation of mean value and standard deviation.

1.6 Investigation of alcoholysis of acetic anhydride in methanol, corrected and adjusted

We have used the same experiments as shown in the chapter before! We have corrected and adjusted the zero shift, if necessary the temperature coefficient of the system (only for ramp experiments possible) and in some cases also WE.

1.6.1 Reactor controlled, isothermal 35°C, addition acetic anhydride

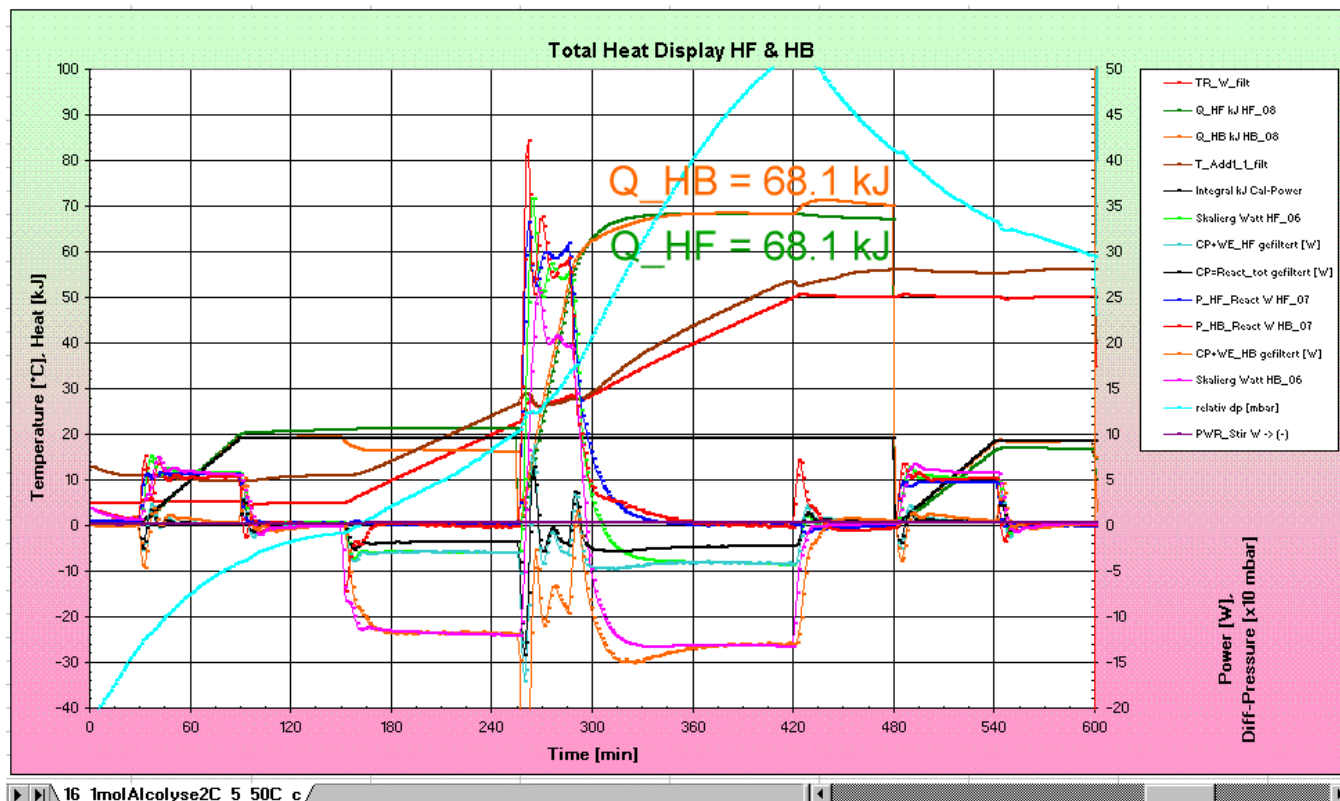
After correction of zero shift (HF +0.4 Watt and HB +0.15 Watt) the results are acceptable. No further correction was necessary. To do that, no calibration heater pulse is necessary, only a reaction free zone.



Results are discussed in chapter 1.7 Evaluation of mean value and standard deviation.

1.6.2 Reactor controlled, "isothermal" ramp 5°C to 50°C, addition acetic anhydride

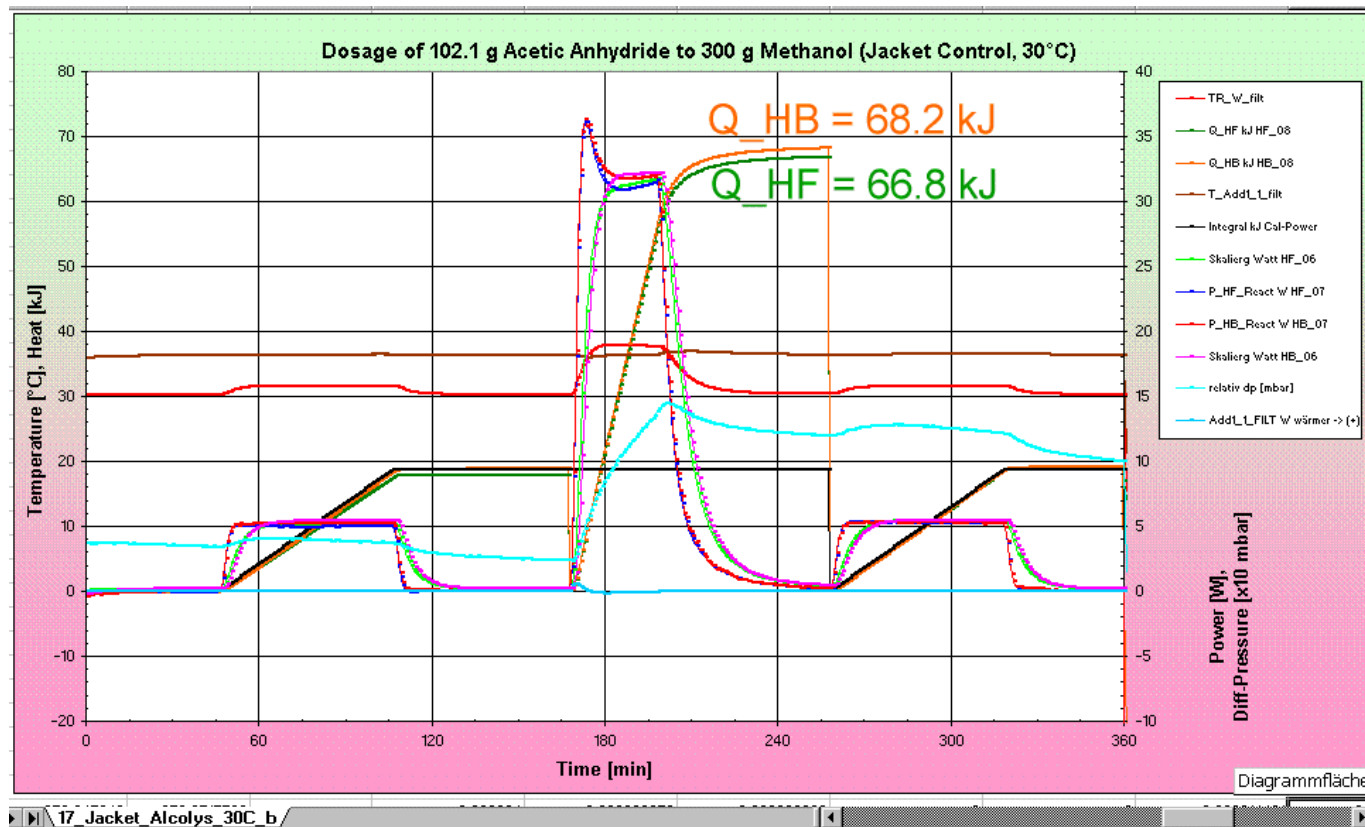
Here we had done the same zero corrections as mentioned before and additional shifted (HB) and bend (HF) the WE value. After this small corrections, the result looks quite nice. The same value is more a lucky coincidence than such a high accuracy.



Results are discussed in chapter 1.7 Evaluation of mean value and standard deviation.

1.6.3 Jacket controlled, isoperibol, constant 30°C, addition acetic anhydride

The change was also a zero shift and for HB also a small change of scaling span (+2.8%). This span scaling is only possible, if we have a calibration pulse otherwise only the zero shift can be realized.

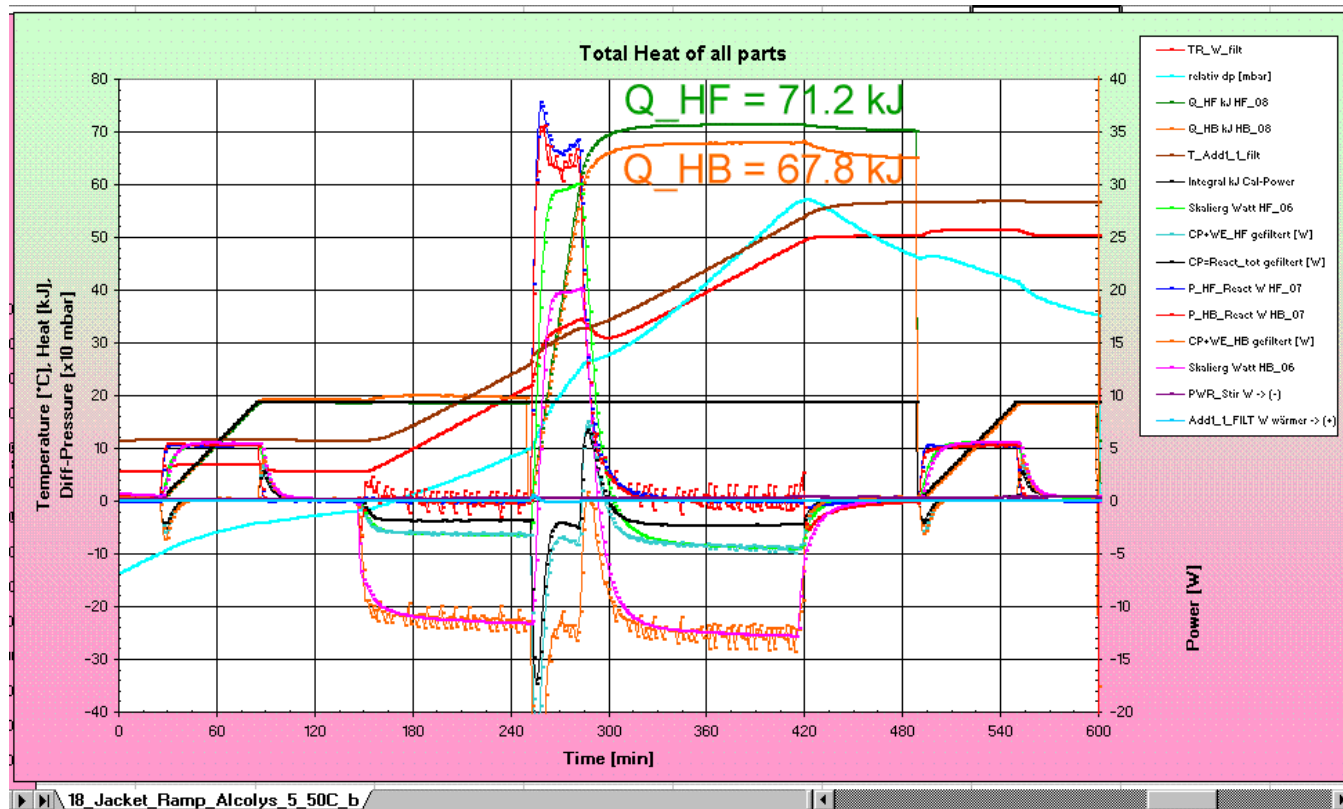


Results are discussed in chapter 1.7 Evaluation of mean value and standard deviation.

1.6.4 Jacket controlled, "isoperibol" ramp 5°C to 50°C, addition acetic anhydride

The present experiment required a small zero shift as well as a temperature coefficient correction for HF. The WE value was in both situation HF and HB to adjust.

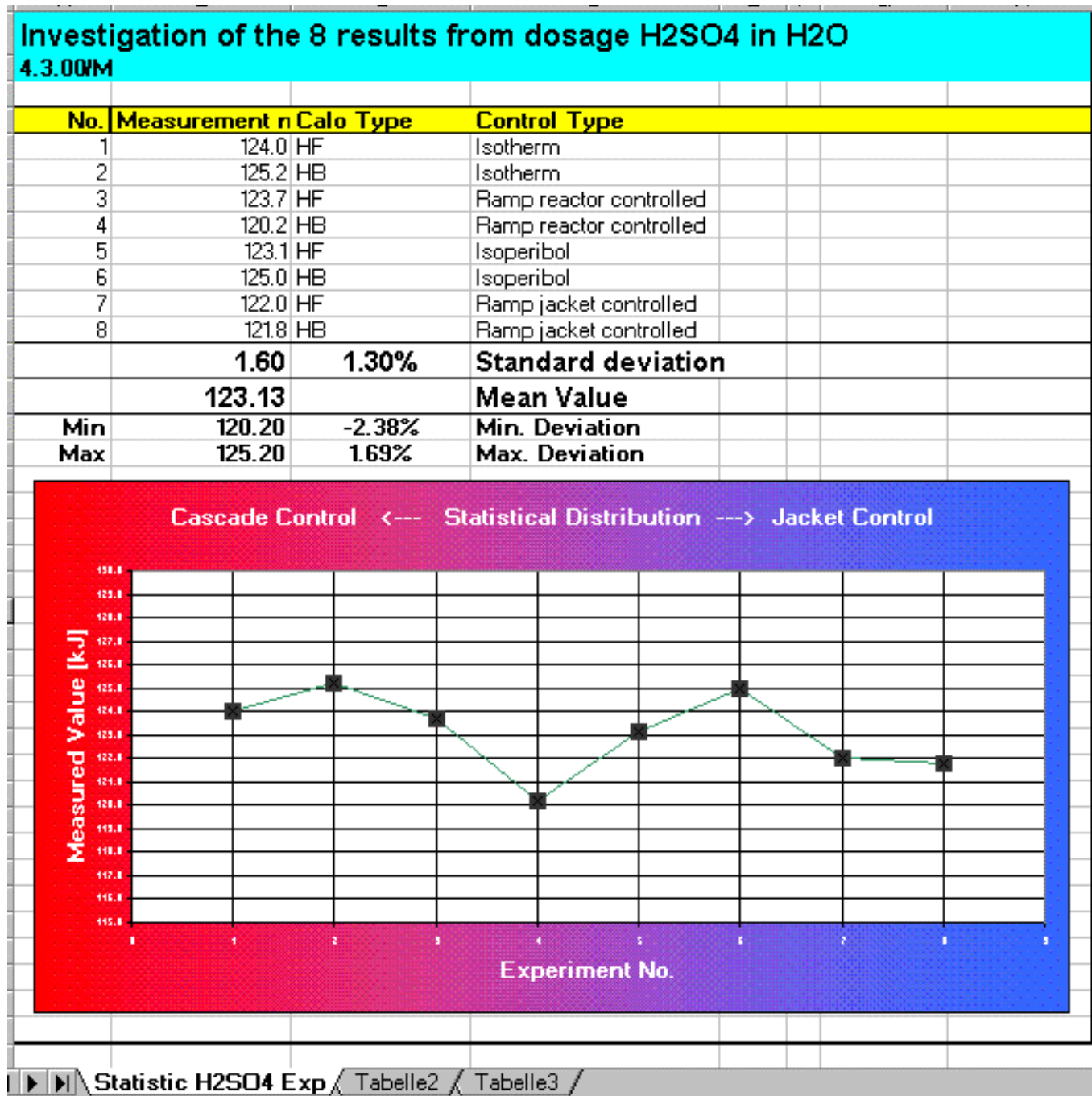
After corrections the value for HF looks too large, the HB values is more in the many times seen range.



Results are discussed in chapter 1.7 Evaluation of mean value and standard deviation.

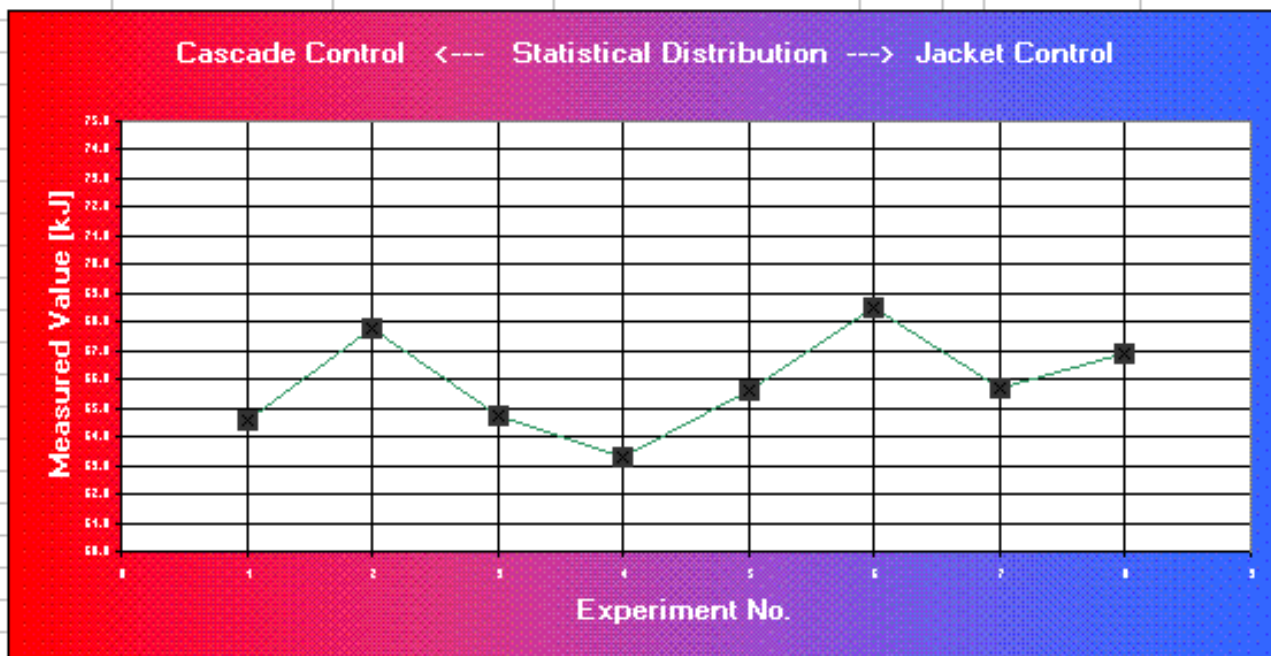
1.7 Evaluation of mean value and standard deviation

We had used the before showed experiments. 4 experiments deliver 8 results (HF and HB each).



Investigation of the 8 results from uncorrected alcoholysis
9.3.00WM

No.	Measurement	n	Calo Type	Control Type
1	64.6	HF	Isotherm	
2	67.8	HB	Isotherm	
3	64.7	HF	Ramp reactor controlled	
4	63.3	HB	Ramp reactor controlled	
5	65.6	HF	Isoperibol	
6	68.5	HB	Isoperibol	
7	65.7	HF	Ramp jacket controlled	
8	66.9	HB	Ramp jacket controlled	
		1.64	2.49%	Standard deviation
		65.88		Mean Value
Theory	67.90	-2.97%	Mean Deviation to Theory <--	
Min	63.30	-3.92%	Min. Deviation	
Max	68.50	3.97%	Max. Deviation	

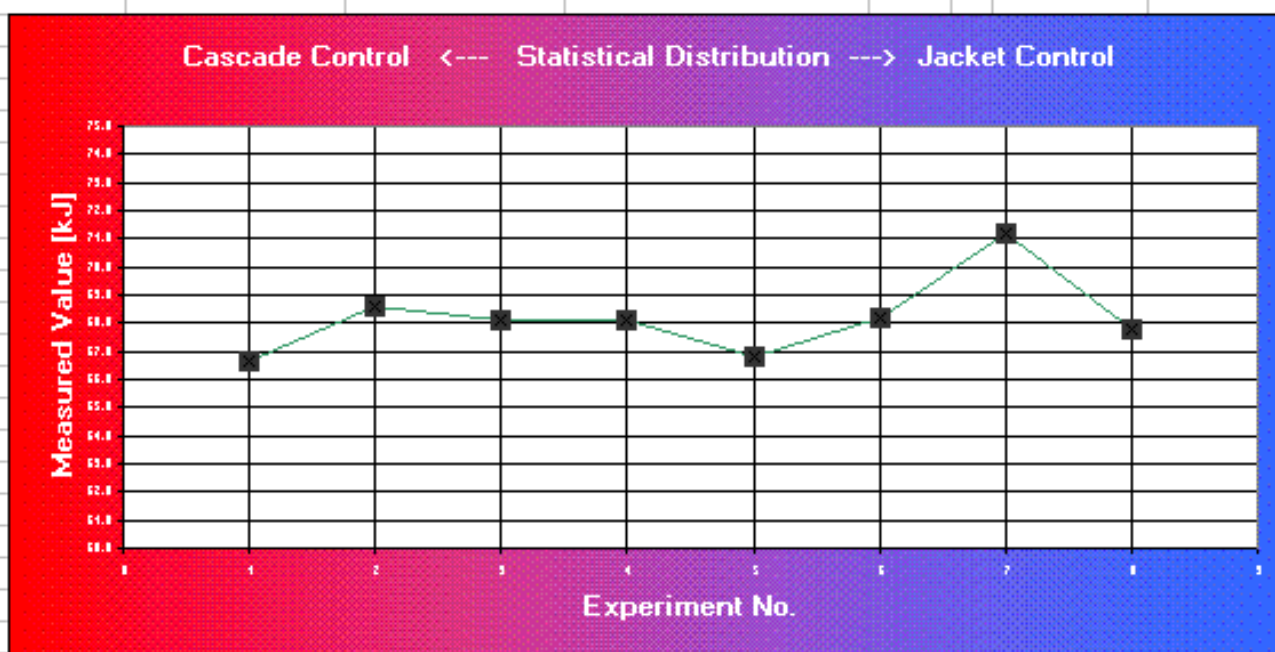


Statistic ALcholys uncorr. / Tabelle2 / Tabelle3

Investigation of the 8 results from corrected alcoholysis

9.3.00M

No.	Measurement	n	Calo Type	Control Type
1	66.7	HF	HF	Isotherm
2	68.6	HB	HB	Isotherm
3	68.1	HF	HF	Ramp reactor controlled
4	68.1	HB	HB	Ramp reactor controlled
5	66.8	HF	HF	Isoperibol
6	68.2	HB	HB	Isoperibol
7	71.2	HF	HF	Ramp jacket controlled
8	67.8	HB	HB	Ramp jacket controlled
	1.31		1.92%	Standard deviation
	68.18			Mean Value
Theory	67.90		0.41%	Mean Deviation to Theory <--
Min	66.66		-2.23%	Min. Deviation
Max	71.20		4.43%	Max. Deviation



Statistic Alcoholys corr. / Tabelle2 / Tabelle3 /

Discussion

We can see the following conclusions:

- Calibration with water and experiment also water based are very close together and require seldom a fine adjustment. The standard deviation is only 1.3%!
- Calibration with water and organic solvents for experiments looks more difficult. One reason is the lower heat capacity and another one the different heat transfer coefficient of the vapor phase which cannot be neglected.
- The most critical error is due to zero shift: the non corrected organic experiments have a mean value of 65.9 kJ and the corrected one of 68.2 kJ. This is for the uncorrected results a shift downwards of 3.3%, together with the max. deviation we found of about 4% delivers the

uncorrected experiments a max. error range of -7.3% up to +0.4% in comparison to the corrected mean value.

- The deviation of the uncorrected mean value to the theory is only -2.97%, for total uncorrected results also not too bad !
- The results of corrected experiments has not only a better mean value, also the standard deviation is decreased from 2.5% to 1.9%.

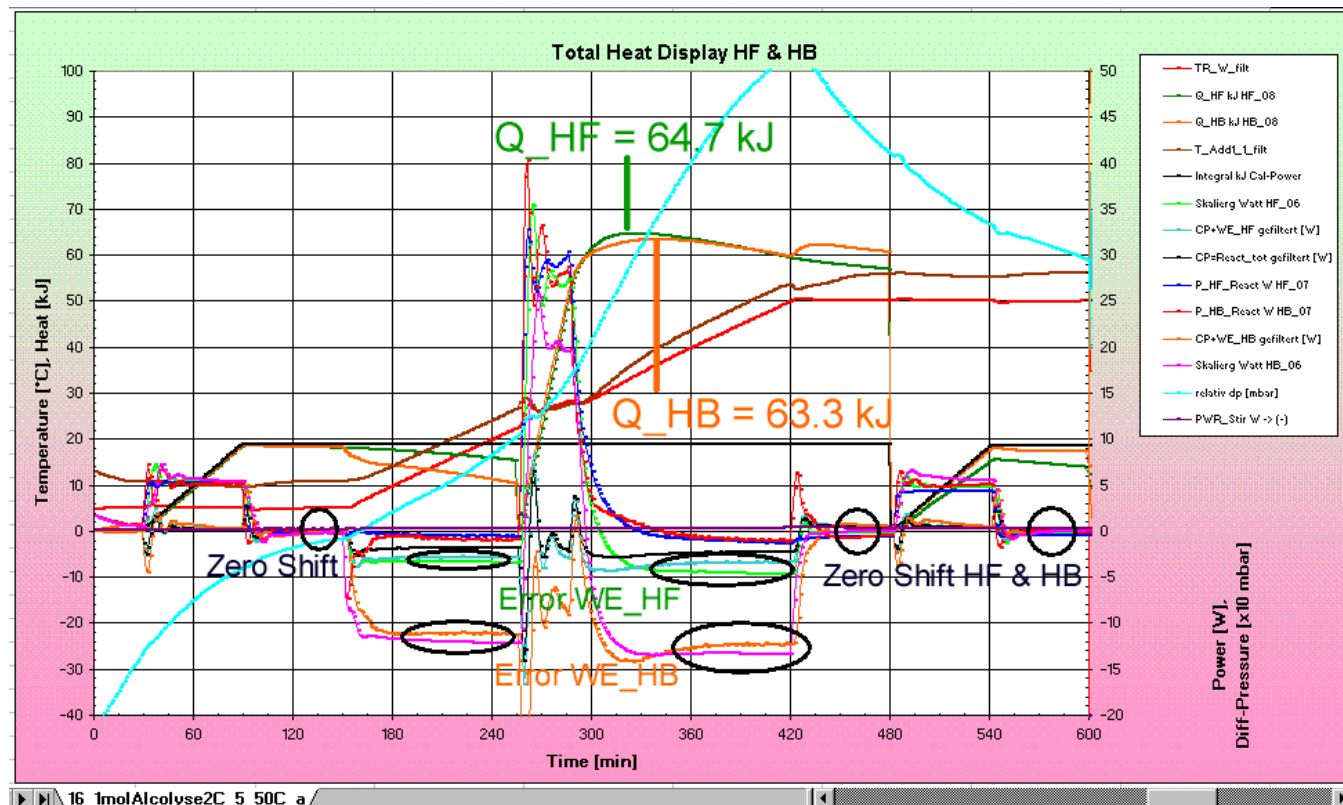
The deviation of the corrected mean value to the theory is only +0.41% ! Not bad! We have also to calculate the additional dosage of 102.5 g instead of 102.1 g (the balance control was not yet optimized!). If we calculate this error of $100 * (102.5/102.1 - 1) = 0.39\%$, we get a final accuracy of

Final mean deviation = +0.41% -0.39% → +0.02% - very excellent!

These results show that most users can work without any calibration, as long they can live with such errors. Only for high accuracy individual calibration is a must.

1.8 Some words about off-line corrections / adjustments

We take this diagram for a short adjustment information. It looks a little bit confusing because both information HF and HB are in the same diagram. But for better comparison it makes sense.



In the smaller circles you can recognize some small deviations: a zero shift. This zero shift from about 0.15 to 0.4 Watt is during integration a problem: over long time it sums up!

The larger error is coming from different WE (water equivalent) values which are oval marked. The WE_HB was too less (instead of 4.5 kg glass we have seen 4.8 kg glass!) and the WE_HF was too less bend over temperature increase. During water based experiments, these WE were near perfect. It's the difficulty of organic solvents and their vapor phase! Therefore some small corrections are necessary.

We have done this in chapter 1.6.2 Reactor controlled, "isothermal" ramp 5°C to 50°C, addition acetic anhydride.

Now you can understand for what we require some reaction free zones:

- at isothermal conditions for zero shifts
- at ramp time for perfect water equivalent values fitting

These adjustments are necessary, if you have a very low energy experiment, like crystallization. If the power of a reaction is in the range of 50 Watts, 0.5 Watt shift is only 1%! If the power is itself only 0.5 Watt, it's 100%! Also it is depending on your expected accuracy.

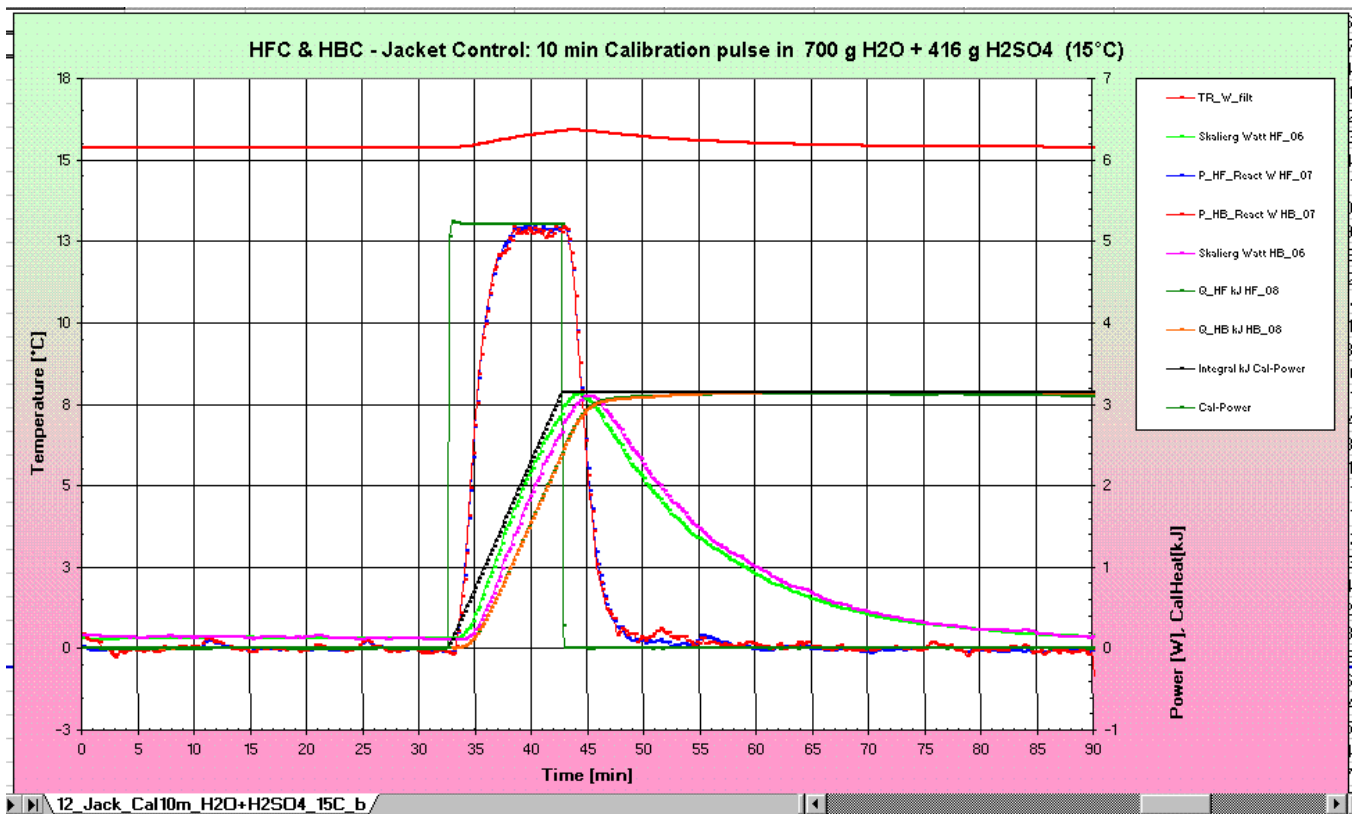
Only Calo 2000 has all these options you require!

2 REPORT #2

2.1 Dynamic response

2.1.1 Calibration heater response with jacket control

Investigation under jacket control: better response without oscillation due to no cascade control.



You can recognize the dead time which is due to the calibration heater! There is a heater part in an isolation which is put into a metal tube. This tube is put in a glass tube again. All these materials produce a time lag which can be recognized above. Not only a time lag is the result, also a time constant which is larger than the real system time constant.

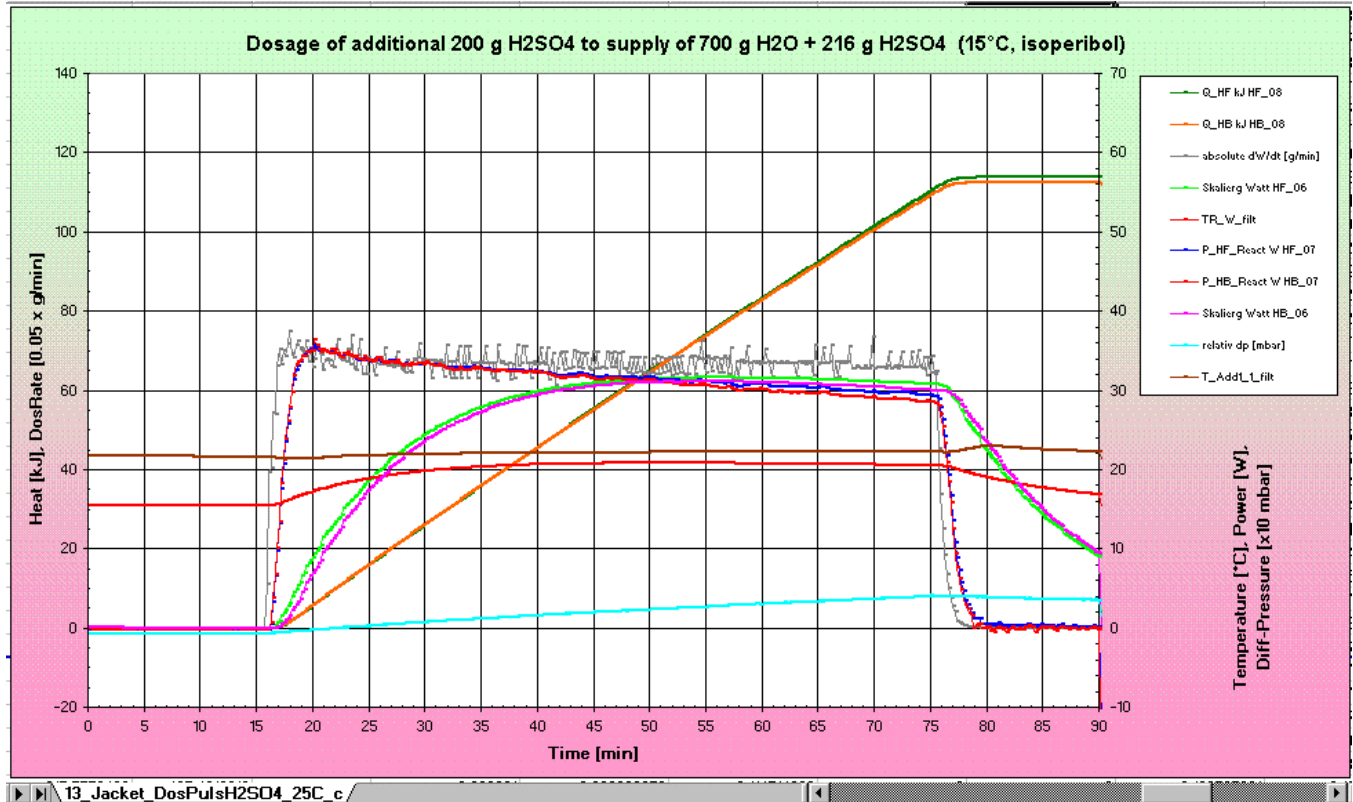
To recognize the system time constant we have used the H₂SO₄ dosage, see next chapter.

Why do we use only 5 Watt for calibration? There are two reasons: first to prevent any hot surface at the calibration heater which could lead to wrong chemical reactions and second to be able to "zoom" the curves for also an exact zero adjustment, not only span!

2.1.2 Reaction response with jacket control

We had used a full reactor (700 g H₂O and 416 g H₂SO₄ was inside).

The best response we get with jacket control. Therefore I investigated the exact behavior of the pulse response. I expected approx. a time constant of about 60 sec, that means after (3*tau) you get less than 10% or after (5*tau) <1% error. This time is coming from the very complex filtering of the different signals to get a sensitivity down to <50mW.



This curves were adjusted: HB had a zero shift of -0.15W and HF of -0.2W. Also the span had to be adjusted: HF by +2% and HB also by +2%. After that, all was very fine.

You can recognize, the gray curve: this is the dosage flow rate dW/dt (differential of balance weight). The red and blue are the corresponding reaction power in Watt, after processing of the measured data HF_raw as light green curve and HB_raw as pink curve. Also you see very nice the small overshoot of the dosage flow at start of dosage → it leads immediately also to a small increase of reaction power. During the dosage you can also see, how much the power decreases due to increasing concentration!

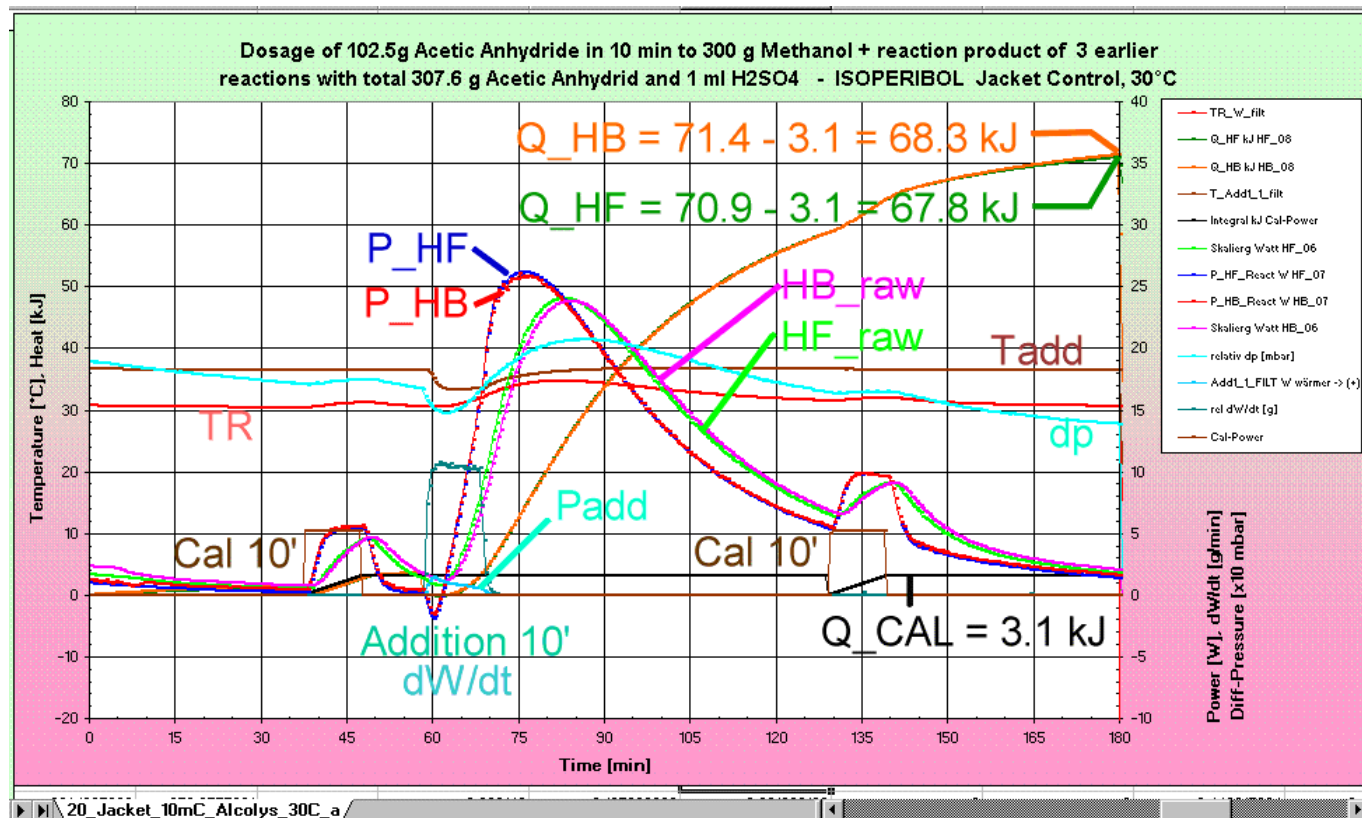
This picture demonstrates very well the excellent time response of Calo 2000!

SUMMARY:

The time response is totally independent on the content or cp of content. Also it is fully independent if cascade or jacket control is used. The time delay and the rise time is only dependent on the filtering of all signals.

Any slow reaction of the control loop does not influence the pulse response time!

2.2 Short calibration and short addition with second calibration (too early!), isoperibol 30°C, jacket control



If a calibration is needed during an experiment, a calibration of at least 10 minutes is recommended with a waiting time of another 10 minutes at least.

Here we show such an experiment. You can recognize that after 10 minutes the power (HF: blue, HB: red) is near back to zero before the addition starts (not the temperature derived signals!). During addition the power of reaction increases very fast until to the end of the addition. A large accumulation was the result. The power increases further for about 5 minutes and goes then slowly back. The idea for the second calibration was for checking the system. We have not calculated such a long accumulation time. It's therefore more a disturbance than a help for supervision!

GENERAL SUM UP:

For standard and quick investigations, any calibration can be left off - or only one for verification is necessary. Under such circumstances an accuracy in a range of 5% up to 10% is possible, as all these diagrams show.