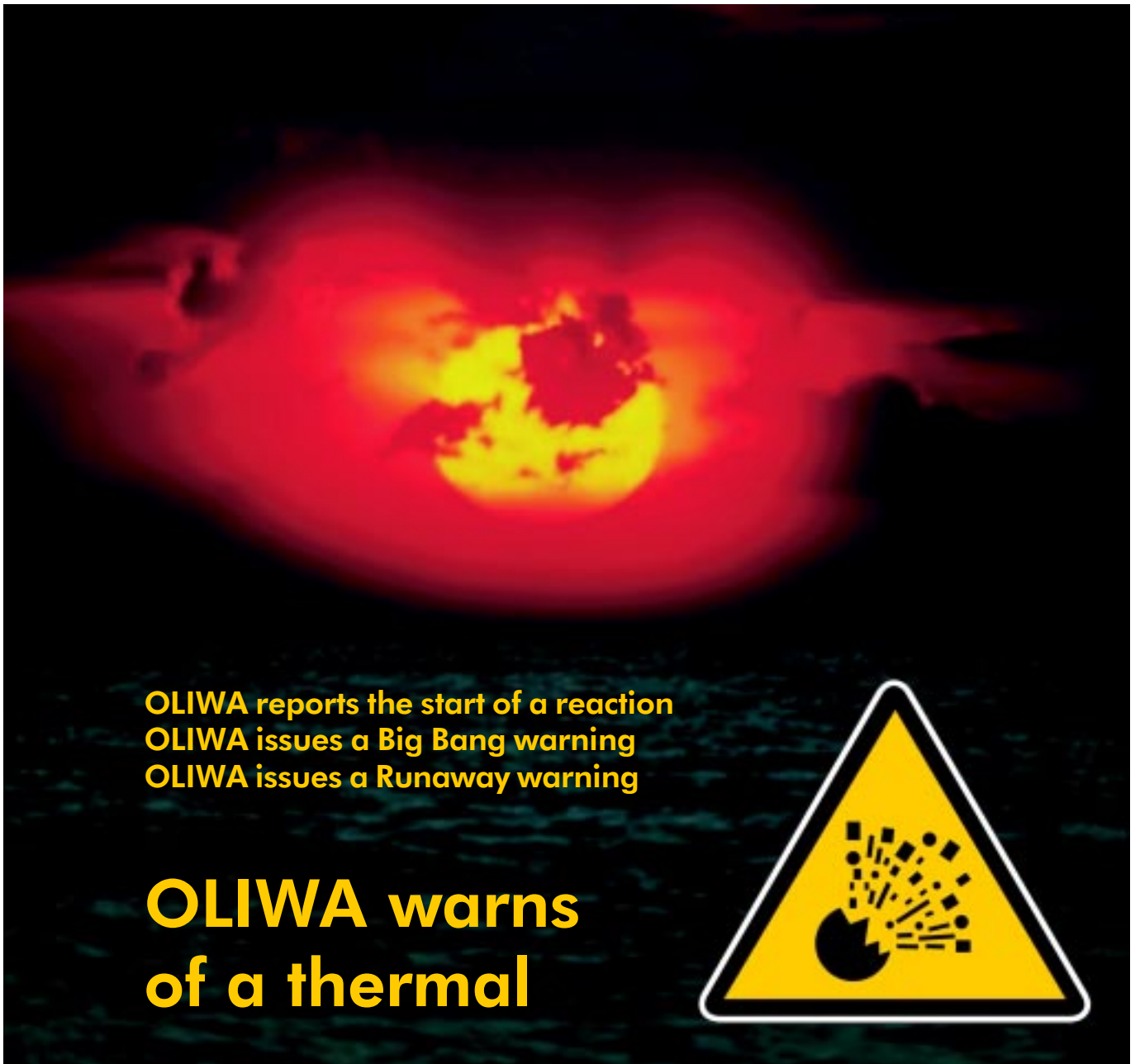




# Runaway alarm in a laboratory, pilotplant and production



OLIWA reports the start of a reaction  
OLIWA issues a Big Bang warning  
OLIWA issues a Runaway warning

**OLIWA warns  
of a thermal**



## The OLIWA system for early thermic danger recognition

### Danger recognition and reporting

OLIWA is an on-line warning system which has the capability to recognize the danger in a thermal explosion (runaway) in advance and to report this by setting off an alarm. The theories on which this recognition principle is based have already been publicized several times and are here only sketched very briefly.

A thermal explosion (runaway) is characterized by a progressive increase in heat development. The reacting substance heats itself up, first slowly but then increasingly quickly until finally such large quantities of energy are freed in split seconds that the installations are destroyed, often with devastating consequences. (A typical temperature run for example can be seen in Figure 1). It is remarkable that the run of such a self heating process is very similar in the most varied chemical substances and reaction types. This is based on laws of nature (Arrhenius), which means that an explosion, for example at certain temperatures or at certain reaction speeds, can no longer be avoided. Alternatively this compliance with laws offers the opportunity to recognize such dangerous procedures early on.

OLIWA is a monitoring system for recognizing dangerous self heating processes early on. Generally the danger can already be identified at an early stage, so that effective counter measures can be introduced. See also Figure 11 on Page 8.

### How does OLIWA work?

The basic principle of OLIWA danger recognition is simple: the reaction is dangerous when the heat development increases progressively.

$$d^2Q/dt^2 > 0 \quad Q: \text{Heat} \quad [1]$$

With the help of a simple heat balance

$$dQ/dt = G \cdot c \cdot dT_R/dt + KF \cdot (T_R - T_J) \quad [2]$$

(G = mass, c = specific heat, K = heat flow coefficient, F = area,  $T_R$ ,  $T_J$  = temperatur in reactor or heat-/cooling jacket)

The term [1] can be described:

$$d^2TR/dt^2 > 0 ; \quad d(T_R - T_J)/dt > 0 \quad [3]$$

Please note that in term [3] no parameters specific to the substance or installation occur.

Consequently OLIWA needs no set-up, adjustment or priori knowledge.

The principles of many, even very complicated set-ups, are often very simple. Even rocket power is based on a simple principle. The performance capability of the set-up is determined by the maturity of the realization.

The OLIWA principle, from the basic idea to the current system, was developed over approx. 20 years. The complex computer programme evaluates many additional criteria, checks a series of logical conditions and carries out automatic checks etc. Consequently, OLIWA has characteristics which far exceed the basic principle.

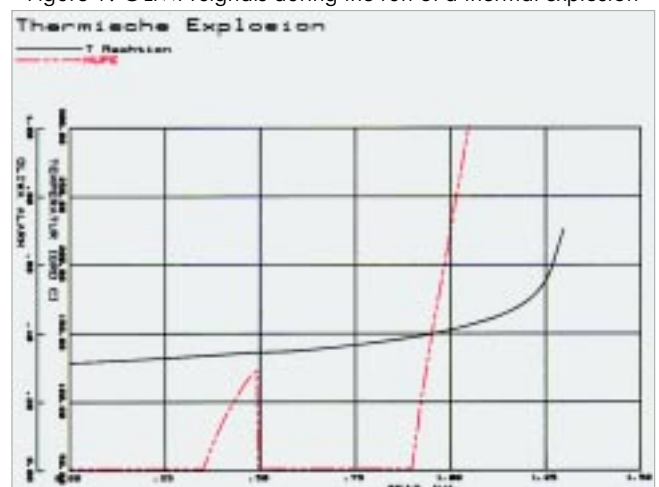
The next pages will give you some examples.

### The first basic ability of OLIWA

Reliable, early recognition of dangers in all situations

Even the implementation of basic principles (term [3]) gives some practical problems. It is known that measurement signals are afflicted by noise in an industrial environment, which are reinforced by the first derivative. These noisy signals can be filtered. This however immediately leads to the next problem: for the early recognition of a slow process the filter must be designed differently than for a quick warning of an unexpected reaction, caused for example by a wrong addition of a reactive component.

Figure 1: OLIWA signals during the run of a thermal explosion



## The basic abilities of OLIWA-Solo

Figure 1 gives an example for the first situation. A slow, insidious process is initiated and must be identified as early as possible. For this purpose a filter with a long time constant is suitable, which suppresses fast signal fluctuations caused by noise and disturbance. It must indicate a high sensitivity to slow accelerations. As can be seen from Figure 1, the danger will actually be signalled by OLIWA at a point where a curve can barely be observed with the naked eye.

Vice versa, the alarm signal goes back to zero as soon as the danger passes. In Figure 1 for example a short pause in the temperature rise occurs, probably caused by evaporation of the remaining solvent. The OLIWA signal goes to zero, but goes up again, when the thermal explosion is initiated again.

In contrast to this Figure 2 shows a sudden reaction, which is recognized by OLIWA and which is signalled by an alarm within approx. 20 seconds.

A classic filter, designed for slow processes, will suppress the dangerous situation in Figure 2 as 'noise'. Vice versa a fast reacting, highly sensitive filter procedure continuously gives false alarm signals due to harmless signal fluctuations.

OLIWA is equipped with a mature, complex mathematical procedure, which has the capability to carry out a reliable identification from slow and from fast acceleration processes in an optimum way.

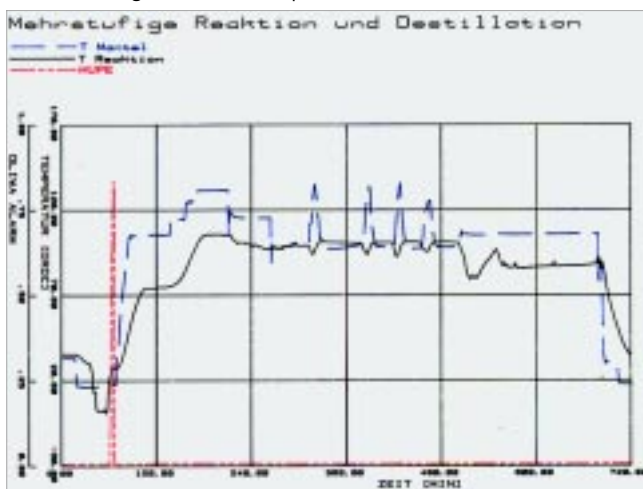
### Second basic ability of OLIWA

**Reliable distinction between dangerous and harmless situations: no false alarms.**

In daily practice a wide range of different, also harmless chemical and physical processes are running in a reactor. Many of these can indicate similarities with dangerous runaway reactions in certain time zones, even though in reality they are harmless. Figure 2 gives an example out of the 'life' of a reactor during a 12 hours period.

First water and ice are present. Subsequently a mixture in accordance with a successful Friedel-Crafts reaction is added from another reactor. At the point when the ice is probably used up and a further mixture (containing aluminium chloride) is added, a dangerous situation may possibly result. This process is displayed in 'slow-motion' in the second diagram. OLIWA reacts within 20 seconds.

Figure 2: Multi step reaction and distillation



Section from Figure 2: 'slow-motion' display



## Additional capabilities of OLIWA system

Subsequently the mixture is heated up, further components are added and a vacuum distillation follows. During all these operations diverse temperature runs result and after a while the deviations take on large positive values. During the distillation a pump disturbance occurs, the vacuum deteriorates and the reactor temperature (T reaction) begins to increase. This situation might be falsely interpreted as a dangerous exothermy through a simple evaluation of the derivatives. It needs a logical integration of several criterias in order to distinguish the individual potentially dangerous situation from all other harmless situations in a day run such as is displayed in Figure 2.

The OLIWA alarm evaluation uses a highly developed system of logical integrations, which guarantees a high sensitivity to distinctions between dangerous and harmless situations.

### Additional capabilities of OLIWA

Apart from the danger evaluation described so far the OLIWA computer programme also carries out a series of further evaluations. Some amongst them increase the intelligence of the situation evaluation, others go beyond the scope of an alarm system and give additional information about the condition of the process.

In the category of intelligent situation evaluation for example fall taking into account of the function efficiency of the control loop or of the relief energies.

Figure 3 displays a situation in which the controller controls, after the first addition of components reacting in an exotherm way, correctly to a complete cooling. The cooling however is limited by the lowest temperature of the available cooling medium. In similar situations it can also occur during a failed control loop. A runaway reaction under such conditions must be classified as dangerous, as if the controller might be able to perform its function. For comparison therefore in Figure 4 a run is displayed from the same reaction (time controlled), this time carried out in a low temperature reactor. The variable heat development is caught each time through an appropriate cooler. This process is more secure.

As mentioned earlier, OLIWA can also deliver additional valuable information about the condition of the process in harmless situations. As example the optical signal is selected with which the equipment shows a harmless exothermy. In Figure 3 for example the optical signal (punctuated line) appears after the addition of the first portions, because the added component reacts exotherm. The temperature profile however shows clearly that this reaction is controlled. The light signal can be seen here as a sign, that the reaction is running. For example OLIWA can be used successfully for the identification of the 'start' of a Grignard-reaction.

Figure 3: Manually controlled reaction

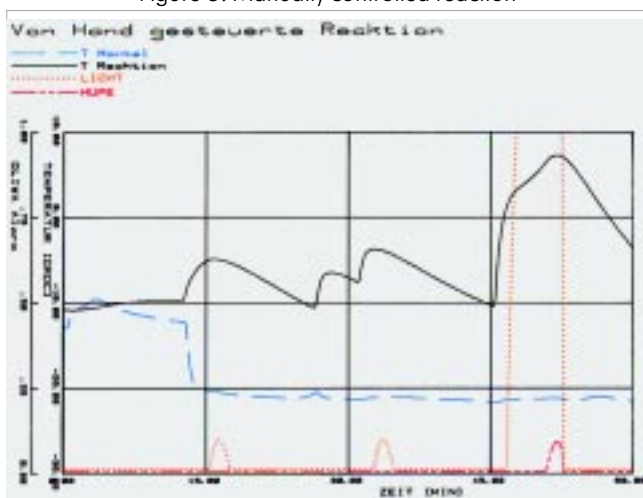


Figure 4: Manually controlled reaction in a low temperature reactor, time-lapsed



## The most important OLIWA characteristics

Apart from all this, Figure 3 is a further example of the reliable distinction between harmless and dangerous conditions by OLIWA.

Because the first additions have not led to any danger, only the light signal 'exothermy' will appear. The last portion however has carelessly become too large. For a short time the temperature run takes on a dangerous form. A decomposition process is started. Luckily the added component decomposes here, which is quickly reacted out and the temperature quickly starts to drop again. The dangerous situation was recognized by OLIWA and was signalled with the sound alarm.

The OLIWA programme judges the functional efficiency of the control loop and takes this information into account in the danger evaluation.

- Quantification of danger, expressed through varying length of light and sound (sound only with danger)
- Additional information about the process condition transmitted through a separate signal (light)
- Function efficiency of the temperature controller accounted for
- Reliable day and night monitoring, including self monitoring and reporting of disruptions
- Flexible installation options
- Intrinsic safety including zone 0 possible
- Automatic triggering of counter measures possible, including specific custom made emergency off programmes

### List of the most important characteristics of the OLIWA system

- Early recognition of the start of a thermic explosion (runaway).
- Reliable distinction between dangerous and harmless conditions.
- Optimal identification of slow and fast processes through a capable filter procedure
- Independent of knowledge of the process and the installation

### Investments in a higher degree of safety

Chemical processes are becoming more and more complex and the process demands more and more difficult. Nevertheless, one can state, that the total degree of safety in today's installations is higher than in the past. This is possible due to intensive use of known safety tests. In many operations one is already in the flat part of the curve, where a further improvement is very costly. The use of OLIWA will be particularly economic here.

Figure 5: Principle of an installation protected with OLIWA

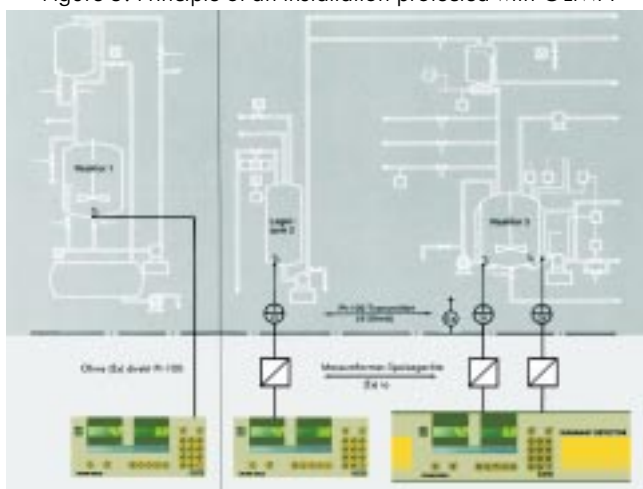
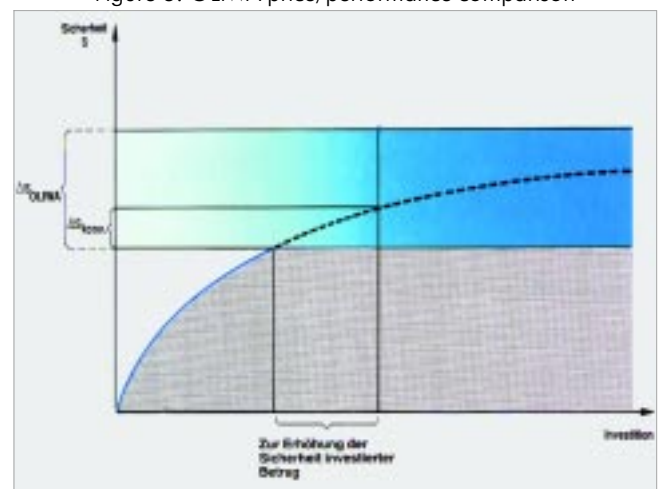


Figure 6: OLIWA price/performance comparison



## OLIWA for laboratory

### Use for process development

The OLIWA-Solo is a unit of the MIDILAB family in the widest possible sense. When used for development, this results in large synergies. OLIWA-Solo allows the detection of the start of a reaction. Consequently, the developer receives important information much earlier than with conventional monitoring. In addition this information can be logged and can even be called up as trigger for further measures. As a result the process control field is extended by the factor of the 1<sup>st</sup> and 2<sup>nd</sup> derivative of TR with respect to time.

Apart from that two further temperatures can be recorded and logged. Also the integration with previously generated temperatures TR and TJ (with MIDILAB temperature controller) is possible, so that two new sensors are not necessary. Slave controls (on/off) or other signal recordings are also possible. For example the data recording of the 1<sup>st</sup> and the 2<sup>nd</sup> derivative and of alarm recorder can be executed with MIDIVIEW or SYBAREX 3.

### Safety examination with new documentation options

Thermal safety examinations in reaction calorimetry can now be documented with the OLIWA reaction (alarm value) and in addition gives information where dosage rates need to be reduced and also where

they need to be increased! It is not therefore just an aid for increased safety, but also for the optimization of the production.

### Increased work safety

With the OLIWA Solo you obtain warning equipment which, during real danger warns you insistently, not only optically but also acoustically. This gives you the time to take measures and to make yourself safe.

Additionally, when you plan to use controller devices which supply an inhibitor, drain off the reaction mass or whatever is the best protection option you can really work with more peace of mind. Even glass reactors can be inclined to burst during strong temperature changes. The danger potential can be even higher with autoclaves.

However it should not just be the danger which justifies the use of OLIWA. The avoidance alone of an extreme polymerisation can save you a lot of cleaning up and loss of material.

Figure 7: OLIWA-Solo for a laboratory



Figure 8: Back of OLIWA-Solo for a laboratory



## OLIWA for production

### OLIWA-Solo in pilot plant

Many experiments are already carried out in a pilot plant, even though all safety clarifications have not yet been determined completely. In these situations OLIWA is precisely the alarm equipment which helps you against nasty surprises.

### OLIWA-Solo in small quantity production

For the clinical test phase products must be produced in small quantities, before the definitive production installation is planned and prepared, with all safety tests in place. With OLIWA you can therefore produce with increased safety, even though all safety tests have not been fully completed.

### OLIWA-Solo in bulk production

In general these installations are equipped with all possible safety set-ups. Also limiting values can be set very tightly, so that usually a sufficiently good protection is available.

OLIWA can above all be used as a redundant system for the bulk production, which can also be used when the primary system goes slightly wrong.

### Automatic protection measures

Depending on the alarm signal, OLIWA allows the triggering of up to two different actions. As a result an inhibitor can be added, the reactor can be flooded or even drained off.

In addition the sophisticated alarm system with light and sound gives a good feel for the danger situation. As a result life and limb as well as the actual installation can be protected, because sufficient advance warning time was given.

### Connection to a process control system and OLIWA protocol

With the RS-485 bus (Modbus protocol) a process control system can call up measurement and alarm values at any time. Consequently this information is also available for a higher system.

When no external protocol options are planned, the internal one can be used. This internal memory operates as a tachograph in HGV. The measurement and alarm values from the last couple of hours can be reconstructed at any time. The read-out can be carried out with MIDIVIEW.

Figure 9: OLIWA-Solo for production



Figure 10: Connection of OLIWA-Solo for production



## Technical data for OLIWA-Solo

### Inputs

Reactor Temp.	Pt-100 or AIN1 for 4-20 mA, supply of a two-wire measurement converter available
Jacket Temp.	Pt-100 or AIN2 for 4-20 mA, supply of a two-wire measurement converter available
OLIWA Run	Potential free contact or 24VAC or DC
Quitting (Alarm/Error)	Potential free contact or 24 VAC or DC for Suppression of alarm, up to 12 pulse for a max. of 60 minutes or as error quitting
Free inputs	Analogue inputs AIN1 and AIN2, providing they are free, can amongst others be used for recording and on/off control of other signals

### Arithmetic algorithmus

Response threshold for very slow 'creeping' processes (in steps of 10 minutes and more): acceleration of inner temperature  $2 \cdot 10^{-7} \text{ } ^\circ\text{K/sec}^2$ . For relative fast processes (in steps of approx. 1 min and more): acceleration of inner temperature  $10^{-6} \text{ } ^\circ\text{K/sec}^2$ . Outputs with variable duty-cycle ratio: the danger measurement is shown in 2 x 16 discrete duty-cycle ratio steps. The first 16 steps are for light alarms and the second 16 for light and sound alarms combined. Start threshold  $2 \cdot 10^{-7}$  or  $10^{-6} \text{ } ^\circ\text{K/sec}^2$ ; change to 100% duty-cycle ratio (sound and light combined) at  $10^{-4} \text{ } ^\circ\text{K/sec}^2$ .

### Outputs

Alarm	One each for light and sound
Error	One each for light and sound
General Outputs	4 DOT with 24VDC can be allocated freely, in the same way for a) alarm thresholds

Free outputs	b) Init phase (approx. 5 minutes) c) Run Digital outputs DOT1 to DOT4 and analogue AOT1 and AOT2, providing they are free, can amongst others be used for recording and on/off applications of other signals.
Load capacity	The sum of all 24VDC currents must remain smaller than 0.9 Amp.

### Supply

Mains	230 or 115 VAC (can not be switched)
Frequency	50...60 Hz
Power	50 VA
HGV version	24 VDC (on request)

### Communication

Interface	RS-232 or RS-485, can be switched
Baudrate	4800 or 9600 Baud
Protocol	Modbus MB1 and MIDVIEW V3

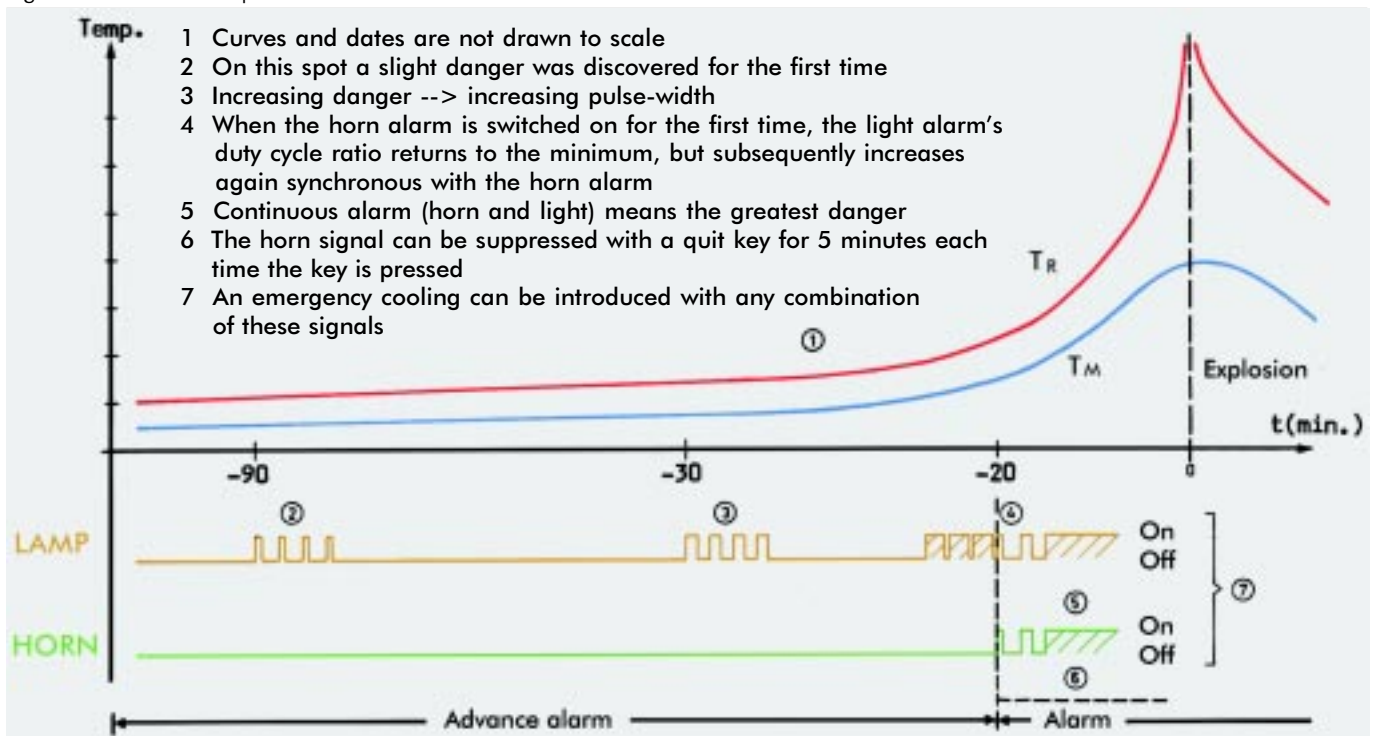
### Specially for laboratory version

Temp. inputs	Can be selected as Pt-100 or AIN1/2 with 4-20mA, also possible as a mix Temperature Taux1 and Taux2 (Pt-100) for general recording and monitoring purposes
2 x T-inputs	

### Specially for production version

Temp. inputs	Only via AIN1/2 with 4-20mA
Separation relays	It is essential that on site separation relays are switched in all 24VDC leads in the same cabinet (EMC)

Figure 11: Alarm concept



Technical changes reserved

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