SAFETY / HAZARD CONTAINMENT

focus

Efficient Reaction Control by Temperature and Pressure Measurement in Microwave Digestion

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Microwave digestion is one of the standard sample preparation procedures for elemental determination in analytical chemistry. In closed microwave transparent pressure vessels the samples are heated with a suitable acid solution to temperatures typically between 200 and 260°C completely dissolving and destroying the sample.

Whilst heating samples and acid solutions in a microwave field brings the advantage of speed to the process, only in exceptional circumstances will two samples behave in the same way, with heating depending on sample constitution and weight. Therefore uneven evolution of temperature and pressure inside different digestion vessels during the process, coupled with the occasionally induced spontaneous reactions are always a severe safety concern. To overcome this safety problem various sensor systems have been developed to measure the main reaction parameters of pressure and temperature, allowing active control of the microwave power by these data.

In principle, temperature is the only parameter which could be influenced actively by the microwave and pressure is only a by-product of the reaction at elevated temperatures. Nevertheless, pressure is the critical parameter in view of safety. Furthermore some special requirements must be fulfilled by an optimal sensor system for microwave digestion systems:

The function of the sensor used should not to be influenced by a strong microwave field. Shielded sensors are unwieldy and rarely absolutely free from interference.

The sensor should bear no risk of contamination to the sample and all components inside the microwave oven should be absolutely corrosion resistant. Coated sensors and tubular systems cannot fulfil this requirement.

Reaction parameters should be determined in each digestion vessel individually, directly and without time delay in order to get an efficient and safe control of the whole process.

Sensors sheathed with fluoropolymers are unsuitable because of the good heat insulating properties of all fluoropolymeric materials. With temperature measurement by conventional broadband IR-technologies, as used frequently in combination with a sheathed temperature probe in a reference vessel, only the surface temperature of the digestion vessel is detected and therefore this technology is unsuitable for sample temperature measurement.

TEMPERATURE MEASUREMENT

Infrared temperature measurement is based on the physical fact, that each solid body emits heat radiation which depends only on its temperature and emission coefficient. At temperatures above 500°C the maximum of this irradiation is in the infrared frequency range. The exact formula for this phenomena was developed by Boltzmann:

$$S = a\,T^4$$

As most bodies also absorb infrared irradiation it is generally only possible to detect the temperature by this principle on unhindered views of the bodies surface; ie, there should be no other infrared-absorbing object between the sensor and the body. This is why, by using conventional broadband measuring sensors only the vessel surface temperature can be detected and not the sample temperature itself *(see figure 1)*.

Therefore useful methods can only be based on the detection of heat radiation in a frequency range where

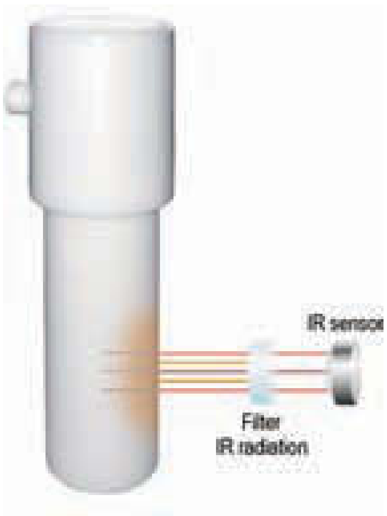

Figure 1. Principle of Measurement for monitoring the sample temperature

the vessel material is transparent. This enables the real sample temperature to be determined easily and directly in real time. The accuracy of this measurement is further improved by filtering out the irradiation emitted by the surface of the digestion vessels. From the detected infrared-radiation and the Boltzmann equation, modified for this frequency range, the sample temperature is calculated in real-time.

It is possible with this technology to detect the sample temperature in Teflon-vessels and their quartz inserts inside a microwave oven in a measurement range from 100 to 300°C.

The precision is relatively high (+/- 1° C at 200° C). It is only through a precise knowledge of all sample temperatures inside the microwave oven that the microwave power can be regulated in an optimal way. The speed of the temperature sensor used in one experiment is illustrated in *figure 2*, where a typical, fast and exothermic reaction was detected in the heating-up period and corrected by the control mechanism. By reducing the microwave power the temperature of the sample was regulated until the requested temperature was reached.

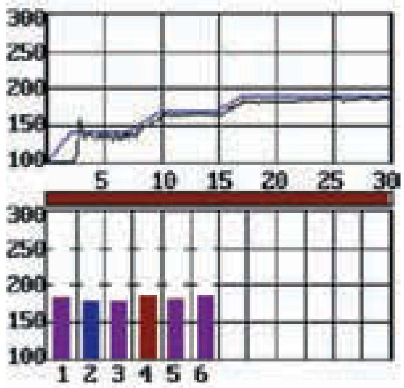

Figure 2. Control of fast exothermic reactions.

Triggering of the rupture disc is detected by the sudden drop of the sample temperature as the solution is vaporised and blown-off suddenly. In this case microwave power is switched off at once.

This is depicted in *figure 3*. After approx. 8.5 min. a sudden drop of the temperature was detected in vessel 12 and interpreted as burst of a rupture disc and the speedwave shut itself down automatically for safety reasons. Both examples illustrate impressively the safe process control by the described temperature measurement technology.

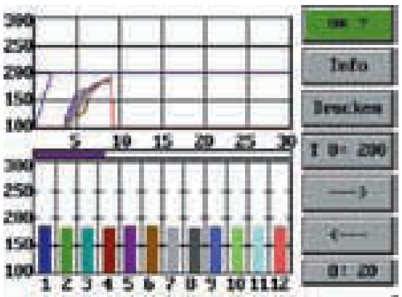

Figure 3. Detection of a rupture-disc burst.

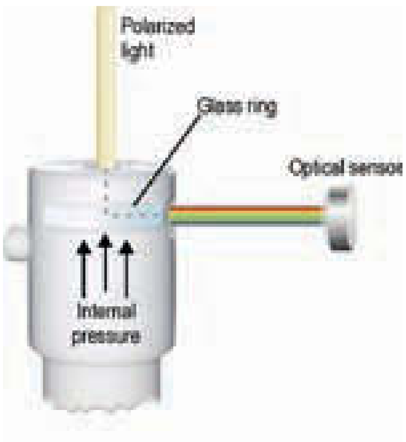

Figure 4. Principle of Measurement for monitoring pressure

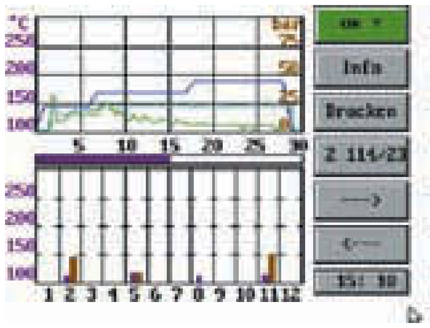

Figure 6. Example of temperature and pressure control.

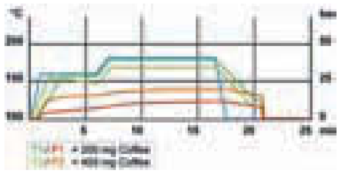

Figure 7. Gain on information due to pressure monitoring

PRESSURE MEASUREMENT

A development from the house of BERGHOF is a non-contact, optical pressure measurement in all digestion vessels specifically developed for microwave digestion systems. The method is based on the measurement of the change of the optical properties of a glass ring, which is permanently integrated in the pressure vessel lid as sensor element. If illuminated by polarised light this ring causes a change in colour of the transmitted light, which is proportional to the internal pressure of the vessel. The internal pressure is transferred directly to the glass ring by the TFM lid with its sliding lip seal *(see figure 4)*.

The glass ring in each lid is mounted in such a way that it does not need to be reinstalled for every new digestion and therefore the ring has absolutely no effect on vessel handling. No additional time and labour is necessary for the connection of sensors.

At every full rotation of the turntable, i.e. every ten seconds each digestion vessel passes through an optical system comprising a polarised light emitter and a light receiver. During this time the pressure of all vessels in the microwave is determined simultaneously to the above described temperature measurement.

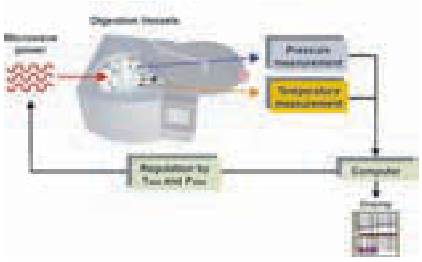

Figure 5. Principle of the microwave control mechanism with temperature and pressure control.

The pressure curve is displayed in real-time on the controller and memorised for all vessels. The precision of this method is ± 5bar (± 72psi) over the complete operating range from 0 to 120bar (0 – 1740psi) and therefore more than sufficient for the safe control of digestion processes.

The speedwave is continuously controlled by both measured temperature and pressure data *(see figure 5)*. The microwave is controlled solely by the temperature as long as the internal pressure of the vessel is far less than the pre-set maximum pressure.

For this pressure limit any values from 0 to the maximum pressure of the vessel type are allowed and could be defined by the operator.

If the internal pressure of the vessel approaches the maximum pressure limit of the vessel the microwave power is adjusted accordingly and thereby the burst of rupture discs prevented effectively.

Figure 6 illustrates the pressure determined control mechanism of the microwave. Following a rapid temperature and pressure rise caused by an exothermic reaction in the initial phase the pressure curve approaches slowly the pre-set maximum pressure of 25 bar.

Then the microwave power is reduced accordingly and the exceeding of the pressure limit is prevented. As the decomposition of the sample leads to a carbon dioxide production and therefore a increase of pressure the temperature is reduced slowly in order to keep the internal pressure constant.

This example clearly shows the interaction of temperature and pressure measurement for microwave control and how this is used for a efficient and safe reaction control.

Optimisation and maximisation of the sample weigh-in is shown in *figure 7*. Digestion of up to 500mg coffee could be performed easily in 40bar standard digestion vessels.

This sample weight is generally sufficient for detection of trace elements in food etc. Higher sample weights can be digested in high-pressure vessels.

SUMMARY

The described technologies for measuring temperature and pressure enable detection of both parameters in real-time simultaneously during microwave digestion. All sample and temperature curves are memorised. The given temperature profile takes first priority for the control of the microwave power. If the maximum pressure limit of the digestion vessel is approached microwave power is reduced accordingly thus preventing triggering of the rupture disc.