LABORATORY EQUIPMENT

Reaction control
Control of pressure and temperature

Full reaction control
Modern sensor technology for full reaction control during microwave digestion

Today, microwave digestion is a standard procedure of sample preparation for element determination in analytical chemistry. Since samples only behave the same way in rare cases, it is necessary to monitor the reaction parameters of pressure and temperature for safety reasons.

During microwave digestion process samples are heated in microwave transparent pressure vessels in the presence of an acid mixture to 200-260°C. During this process samples are transferred into solution by complete digestion. The advantage of microwave digestion lies in the fact of a direct and fast heating. However, the actual heating in the microwave field is dependent on the type of sample, the sample quantity, etc. Only in rare instances will two samples exhibit exactly the same behavior.

For reasons of safety and to minimize the risk of unequal heating or spontaneous induced exothermic reactions monitoring of reaction parameters pressure and temperature is of special importance. Modern sensor devices are not only relevant to measure temperature and pressure data but also deliver important information to control microwave power.

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General requirements

The parameter actively influenced by the microwave is temperature. Pressure is only a by-product, which also presents a critical parameter in terms of safety. In general, sensor systems have the following requirements:

- → No absorption
  Even a strong microwave field must not be able to influence the sensor. Shielded sensors have the disadvantage of not working absolutely flawlessly and being not easy to handle.
- → Quick reaction time
  To be able to effectively monitor spontaneous exothermic reactions, the measurement speed of the sensors must be high and instantaneous.
- → No contamination
  The sensor must not contaminate the sample.
- → No corrosion
  All components in the oven must be fully corrosion-resistant. Coated sensors and tube systems have their own disadvantages.
- → Direct measurement
  The reaction parameters must be determined as quickly as possible in each vessel in order to regulate the output efficiently and safely.

Temperature measurement

Temperature can be measured in digestion vessels in different ways.

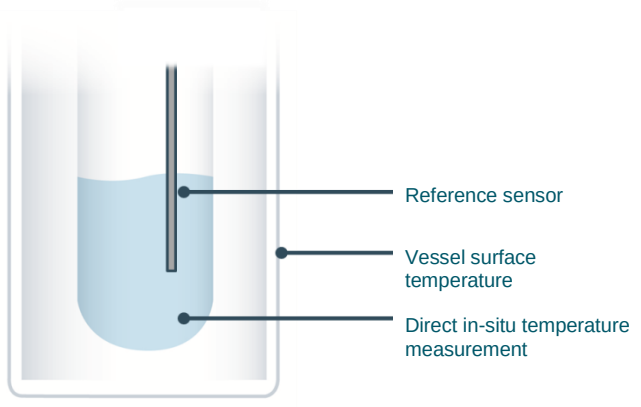

Figure 1: Types of temperature measurement

In the past few years, the use of sensors in reference vessels has become more common. Thermosensors coated with fluoroplastics are dipped directly into the sample solution. The reaction control is limited to this single vessel only, partly for cost reasons. In the case of highly inhomogeneous samples or samples with differing reactivity, temperature is not sufficiently monitored. In addition, the time of the reaction, which often involves multiple protective sleeves around the sensors, is limited by the transition of heat through the coating.

This results in delayed detection, with it being hard to monitor spontaneous exothermic reactions. This is why additional infrared sensors, which measure the external temperature of the digestion vessel, are often used. This does not result in any information being obtained about the current actual temperature of the sample inside the vessel.

With the modern in situ temperature method, the sample temperature is measured directly and this method has the following advantages.

- → Quick reaction time
  From a physical perspective, the speed of the measurement is only limited by the speed at which the infrared radiation from the sample reaches the detector. Since there are no materials between the sample and the sensor, the temperature is measured instantaneously.
- → No contamination
  The sensor lies outside of the digestion vessel and the microwave field.
- → Easy handling
  The sensor is not tricky to set up.

Contact-less mid IR-temperature measurement

Infrared temperature measurement is based on the physical fact, that above absolute zero each solid body emits radiation depending on his temperature and emission coefficient. At temperatures less than 500°C the maximum of this irradiation is in the infrared frequency range. The exact formula for this phenomenon was developed by 'Stefan Boltzmann'.
As most bodies absorb also infrared irradiation it is generally only possible to detect the temperature by this principle of bodies on which surface the view is unhindered. This means that there is no other infrared-absorbing object between the sensor and the body.

A feasible solution bases on a detection of the heat radiation in a spectral range where the vessel material is transparent. By this, the real sample temperature is 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.

Figure 2: In situ measuring principle to determine sample temperature

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 relative high (+/- 1 °C at 200 °C). Only through the precise knowledge of all sample temperatures inside the microwave oven the microwave power could be regulated in an optimal way.

Microwave control with exothermic samples

Spontaneously reacting samples show how quickly thermometers need to work. Typically, rapid exothermic reactions occur in the heating phase. This can be clearly seen in the example of the digestion of polymer granulates. The acid attack and the start of the melting of the particles lead to a rapid rise in temperature. Through the continuous determination of temperature data, the microwave power is regulated such that careful venting can be guaranteed.

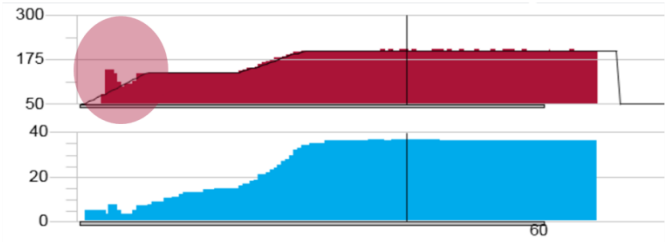

Figure 3: Temperature and pressure development during exothermic reaction

In addition, the rise in temperature goes hand in hand with an increase in pressure (blue curve figure 3). Here, the total pressure in the vessel is made up of the total vapour pressure of the acid mixture at the relevant temperature and the partial pressure of gaseous substances formed. In the example mentioned, the pressure is built up through the decomposition of the polymer granulate and the formation of CO_2 as a decomposition product. Monitoring pressure is also an important parameter, which is required for regulating microwave power and safe reaction control during digestion.

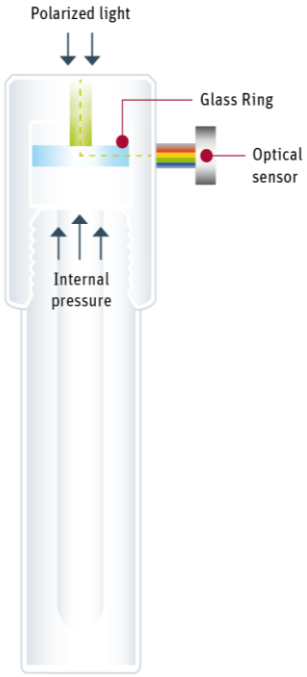

Figure 4: Contact-less measurement of pressure

Pressure measurement

Similar to temperature also pressure can be controlled via external sensors in reference vessels. But, main disadvantages are risk of contamination and leakage. Therefore, contact-less pressure measurement should be the method of choice.

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. Pressurization of a glass-sensor element turns the polarization plane of an irradiated light beam which is proportional to the pressure inside the vessel. This turn is measured by an analyzer calculating the pressure herefrom. 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 labor 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 polarized 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. The pressure curve is displayed in real-time on the controller and memorized for all vessels. The precision of this method is ± 5 bar (± 72 psi) over the complete operating range from 0 to 120 bar (0 – 1740 psi) and therefore more than sufficient for the safe control of digestion processes.