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Useful Peripheral Equipment for Calorimetry

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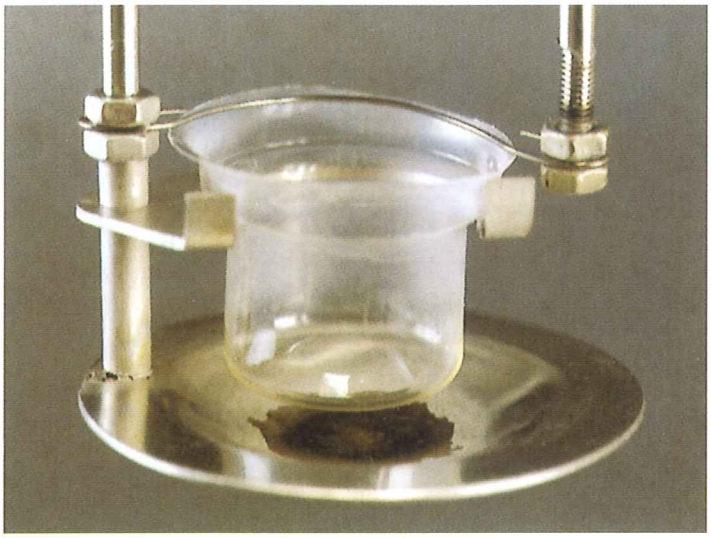

Fig. 1:
The sample and disposable crucible burn with no residue. Direct contact with the ignition wire is ensured.

Various accessories have been developed for the C5000 Calorimeter System, offering the user even more possibilities for simpler, timely analysis.

From the point of view of the analyst, determining the gross or net calorific value of solid and liquid samples has always been and still is a very demanding task. Of course the analysis itself, i.e. the routine within the procedure, is largely taken care of by automated device technology, but this is no replacement for the user's expertise when it comes to preparing the samples.

Even when it was introduced to the market two years ago, the C5000 Calorimeter System offered among other features a high level of automation, modular design and free choice of the working mode (adiabatic, isoperibolic or dynamic). In spite of this, the C5000 and its associated peripheral equipment are constantly being expanded and further developed.

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Example: preparing a sample

When preparing a sample for combustion, particular attention is devoted to the igniters. The practice has long been to use an ignition wire that combines two electrodes and which first incandesces and is then brought to the point of combustion by applying an electrical voltage. Direct contact with the combustion sample likewise ignites the sample. But frequently the sample is not ignited by direct contact with the ignition wire, but rather by means of an additional cotton thread that serves as a connecting element between the ignition wire and the sample. Because of the pure oxygen atmosphere in the decomposition vessel at the time of the ignition, the igniters undergo combustion resembling an explosion, thus ensuring that the sample will be certain to ignite.

Simple handling

To further simplify this handling, the "fixed ignition wire" was developed some time ago. A defined, reproducible electrical voltage causes the wire to incandesce but not to actually burn. This saves the time and expense of fastening a new wire and removing the unburned residue of the old wire from the fastening points of the electrodes after every combustion.

One manufacturer took this as a starting point and developed the disposable crucible (Fig. 1). By this we mean a crucible made of combustible material that burns together with the sample, leaving no residue behind. The disposable crucible is suspended in the crucible container of the decomposition vessel just like a traditional high-quality steel or quartz crucible. Direct contact with the ignition wire is ensured by the specially designed shape of the upper surface of the crucible. This simplifies the loading of the decomposition vessel and reduces preparation time to a minimum.

The combination of sample container and igniter provides additional advantages: At the moment of ignition, the disposable crucible begins to burn along its entire surface. An unbroken wall of flame rushes in on the sample from all sides. The main result of this phenomenon is improved oxygen supply, which means optimized combustion. Because of the disposable crucible, it is now more difficult for materials that always yield poor results because of their tendency to spark to distribute their unburned particles evenly over the surface of the decomposition vessel and thus to cause a reduction in the amount of energy determined in the results of the sample.

Because the disposable crucible looses its connection to the crucible suspension at the moment when combustion begins, a plate-shaped support prevents the crucible from falling together with the sample onto the bottom of the decomposition vessel or even into the absorption fluid. Contamination of the sample with sulfur or halogens is excluded because of the purity of the raw materials used in manufacturing the crucible. This is especially crucial for subsequent analysis when the crucible burns simultaneously with the sample.

Older decomposition vessels, including the standard series C5010 or the catalytically active decomposition vessels of the C5012 series can be retrofitted with this special support with no problem.

Combustible crucible and software

When combined with the C5000 calorimeter system, the disposable crucible becomes a factor in reducing costs. The software in the device offers various possibilities for automatically recording the extraneous energy portion of the disposable crucible during weighing in or for taking it into account during the subsequent calculation of calorimetric values. Completely independently of whether the sample was weighed first and then the igniters, or whether the sequence was reversed, the system records the values and assigns them accordingly in the correct order. This

function can also be used with all traditional igniters. It is required, however, that the known gross calorific value of the igniter be entered in the appropriate dialog box in the software.

In addition to determining gross calorific value, decomposition is also possible

In addition to simply determining gross calorific value, the C5000 calorimeter system is also ideal for quantitative decomposition of a wide variety of sample materials. The actual procedure is based on the "bomb decomposition" procedure as specified in DIN 51577. In the simplest terms this means the destruction of the mechanical structure of the sample as a result of pressure and temperature.

The manufacturer has also come up with something special in regard to this topic as well: A special decomposition vessel of the C5012 series with catalytically active inner surfaces that even regenerate themselves. This means no wear, or reprocessing after a certain usage period.

In comparison to traditional systems, this system offers, in addition to simultaneously reducing the potential for hazard and drastically cutting the use of consumable material, quantitative decomposition of the entire range of halogens and sulfur. With a simple decomposition time of less than three minutes, accompanying materials such as water, solids and other interference-producing materials have no effect on the complete decomposition.

Analysis of gasses generated by combustion as well

If the gasses generated by combustion are also to be subjected to analysis, the special C5030 degassing station is available for this purpose. With this separate station, it is possible to reduce the pressure of combustion gasses slowly and in a controlled manner through a second, external absorption section. The decomposition option in the setup of the calorimeter makes it possible to deactivate automatic degassing by the system after the experiment.

This procedure may be beneficial from two completely different points of view.

■ First: for a relatively high total halogen content, ⩾10%. It may happen in this case that the oxygen partial pressure is high enough in the decomposition vessel after combustion to guarantee complete solution behavior of the halogens in the gaseous phase. In this case the C5030 degassing station can make it possible to avoid the losses of reduced pressure.

■ Second: when working in the area of traces. Special control standards are available for monitoring the decomposition system and the peripheral detection equipment for aqueous decomposition. Combinations for chlorine and sulfur as well as for fluorine and bromine are available, based on a mineral oil basis. In principle, the spectrum of detection methods for follow-up analysis based on the decomposition method described above extends from titration, ISA and ion chromatography through ICP, AAS and IR spectroscopy to photometry. The spectrum of samples that can be processed is by no means any less extensive. It ranges from plastics to natural products and mineral oils, from sludges to various types of special wastes.

The system has proven especially valuable in routine analysis in the area of waste oil regulation (Appendix 1, sample taking of and experiments dealing with waste oil). The same applies to production and quality control in the refinery area.

Fig 2: The C5000 Control calorimeter system

What to do with biomasses?

More and more, biomasses as energy carriers are the center of attention in the area of renewable energies. Because of the sample matrix and the energy level, which is often relatively low, it is necessary in this case to weigh in a significantly larger quantity of sample material than normally. The standard decomposition vessels C5010 and C5012 can be fitted with a special crucible holder for larger crucibles made of quartz and high-grade steel for this purpose. This has made it possible to open up a new area of application by means of a small but extremely useful accessory. This is another area where there will be innovations for further basic applications.

Increased automation

The following components for the C5000 calorimeter system make daily work in the lab much more pleasant and simultaneously increase the level of automation:

Sample rack

The C5020 sample rack is used to reliably record and manage individual fuel samples as well as samples consisting of larger quantities (up to 12 compartments for prepared crucibles).

The recording and processing of the samples can thus take place with a time delay. Special sealing elements prevent volatile components from seeping out and prevent the sample from absorbing moisture. A direct connection with the control computer definitively excludes the possibility of mixing up samples.

Control and evaluation software

Automation and local data recording are not at all foreign concepts when it comes to thermal analysis.

A control and evaluation software package based on the technology of the latest generation has been developed by IKA with CalWin for all calorimeters. CalWin is used to prepare, control, evaluate and manage measurements with the C4000, C5000 control (see Fig. 2) C5000 duo-control and C7000 calorimeters of IKA. The software requires a PC capable of running under the Windows 95 or Windows NT operating system with at least one free serial interface. The requirements for the software are no more stringent than those for the operating system.

A standard commercially available interfaces cable (included with delivery) is used to connect the previously installed calorimeter with a PC onto which CalWin has been installed, via a standardized serial interface.

The system is also network-capable and LIMS-compatible – but it still leaves enough free space so that it doesn't interfere with creativity in the laboratory.

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