The Right Handling of Samples

Improve the Quality of Measuring Results by Providing the Right Sample Treatment

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The usage of cuvette and reagent tests is nowadays standard for the photometric identification of parameters, e.g. for self-check.

High value is set especially on fast and easy obtainable measuring results; regarding accuracy being comparable with the time-consuming extensive standard methods. Faulty measuring results are annoying and moreover cause higher expenses.

To ensure a correct measurement result at the end of a long process, the preparation and the handling of the sampling procedure should be appropriate.

> ***"THE NEW WTW SPECTRAL PHOTOMETERS BELONGING TO THE SPECTRO-FLEX SERIES FOR THE VIS AND UV/VIS AREA CONVINCE WITH A WIDE RANGE IN FUNCTIONALITIES AND CONVENIENT HANDLING"***

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A lot of time is invested into the development of commercial test kits in order to achieve precise photometric measuring results in an easy and fast way, using reagents, cuvette or powder tests. When results are faulty in most cases the according measuring instrument or the test kits are made responsible. However, when taking a closer look, the failure is usually not caused by the instrument or the chemistry! The analytical quality assurance (AQA) enhances the failure search. The photometers provide extensive functionalities, such as automatic system check and AQA routines for standard solutions with acceptable tolerance range.

The new spectral photometers belonging to the spectro-Flex series for the VIS and UV/VIS area convince with a wider range in functionalities and convenient handling. Most failures occur in general whilst drawing the sample, during conservation or transport of the sample from the original site to the analysing location and finally during preparation of the sample. In addition failures are made with dilution and pipetting. Research studies have proven that almost 98-99% of failures happen prior to the actual measurement.

You should also bear in mind that due to the complexity of the material several commercial test kits cannot be as simple as preferably desired: Especially the TOC cuvette test kits show that is worthwhile to carry out pretests and "practice" with personal samples and that adequate handling of the samples is essential for good results. In general pretests serve for an improved and familiar handling and optimise the corresponding test in its special environment, considering that the water hardness, the pre-handling of the water and mainly the consistency of the sample may differ distinctly depending on the origin.

When processing a TOC cuvette test, the inorganic part of the carbon (TIC) must be extracted in the form of CO_2. Afterwards the sample and cuvette reagent with permeable membrane are brought together and digested in a thermo-reactor. The arising CO_2 diffuses into the indicator liquid. Factors with extreme impact on the quality of the measuring results are:

- conservation of samples • pH value
- stirring times • digestion and standing time
- disruption ions • dilution

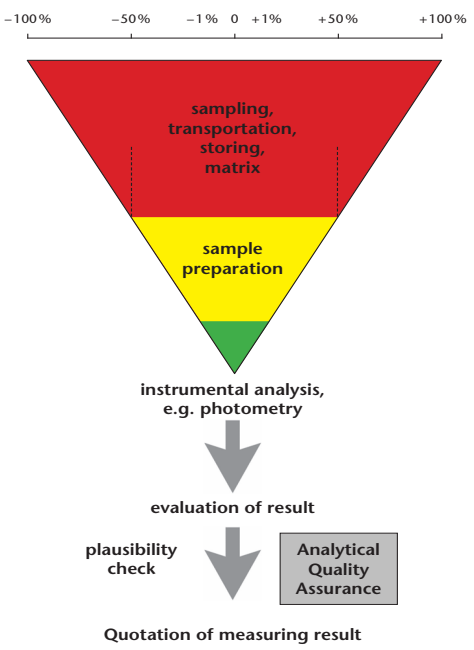


Figure 1. Failure frequency from taking the sample until retrieving the measurement result

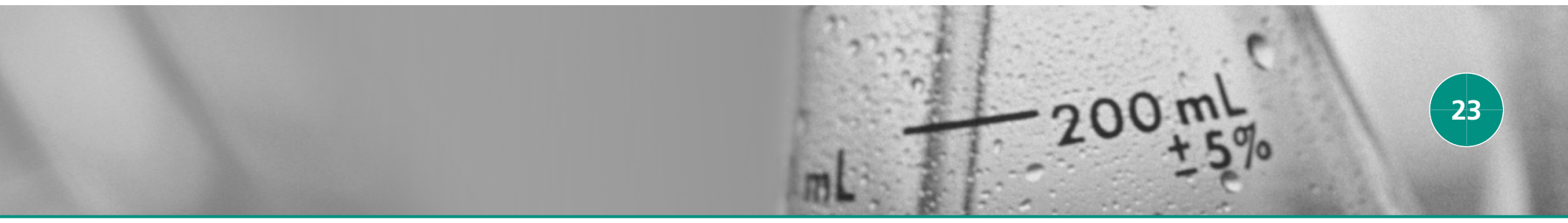
THE RIGHT CONSERVATION OF SAMPLES

Hydrochloric acid (HCl) is especially used for the conservation of samples, leading to a high chloride value in the sample and disturbing the test's colour indicator for the definition of the concentration. This disturbance falsifies the measuring value. For TOC samples sulfur or phosphoric acid should be used for conservation – if at all.

Besides the previously mentioned negative influence on colour indicators for the desired reaction, complex accumulation or masking can also cause faulty results. Especially chloride in high concentration is very often a disturbing ion in cuvette tests. A major trap thereby is the distribution of salt in the winter! The acidification of samples through HCl is also noticeable. The instruction leaflets give essential information regarding the permissible concentration of disturbing ions and usage of cleaning agents on sample beakers/containers, which must be followed. Matrix effects and other influences can be traced by means of AQA measures with standard solutions. The new spectral photometers spectro-Flex for VIS and UV/VIS range give additional potential for quality control using the function of matrix check via spiking.

pH VALUE

The pH value plays a major role when determining the TOC concentration. The organic carbon is transferred into CO_2 through acidification and can then be subsequently excluded. Therefore the pH value must be kept and should state a pH of 2.0, or even 1.8. When setting and checking the pH value it must be paid attention that the pH value is not displaced by buffer impacts! For checking in smaller sample containers for example the pH electrode SenTix Mic is very suitable. With tests using pH paper very often the wrong range is anticipated. The water hardness degree is very important. Where a readjustment of the sample is necessary an acid of 5% is usually sufficient, however a slightly higher concentration may also be used where applicable.



STIRRING TIMES

The stirring time can or - when required - must be prolonged, in order to withdraw the complete inorganic carbon in the form of CO_2. An approximate duration of 30 minutes has meanwhile become a solid average value. An ultrasonic bath is also recommendable. The pretreatment of the sample and the water hardness contribute to varying testing conditions, which can be corrected through preliminary tests using TOC cuvette tests:

Using several samples and different stirring times (and perhaps slight variations regarding pH value) it is more likely to reach a higher sensitivity for the right procedure, thereby avoiding implausible measurement values and reducing usage of test kits. To state a standard stirring time for all sample environments is almost impossible.

DIGESTION AND STANDING TIME

The TOC cuvette test requires an exact observance of times: CO_2 is generated when digesting the organic components and diffuses through a membrane into the indication fluid. Some systems may require applying the screw cap directly after inserting the sample and the digesting reagent in order to avoid the loss of CO_2. Then again other systems could involve screwing several cuvette components (prepared sample and indication part). In this case it is mandatory that the membrane remains dry and does not cause reaction failures. The digestion is processed in the preheated reactor for two hours by 120°C headfirst (gas diffusion) and cools down for one hour. As it is possible that further CO_2 is released during the cooling procedure, it is most essential to keep all same times for comparability of values in one sample environment.

DILUTION

An unwanted carbon source is very often assumed clean "ion exchanger water". Experience has unfortunately shown that in many cases this water bears a considerable strain of carbon (CO_2, HCO_3) and CSB. Therefore it is suggested to dilute TOC samples with water for analysing purposes.

SAVING COSTS

The list of potential failures is incredibly long, when considering that pipette failures are also likely to happen. The "empirical" preparatory work with complicated tests has proven value, when satisfying and precise results are achieved with only minor wastage of cuvettes, resulting of course in reduced expenses. The TOC cuvette test, as here introduced by WTW/Merck, is an absolutely eco-friendly test excluding heavy metal. It uses a membrane, that only lets carbon dioxide pass and is handled same as a conventional cuvette with screw top (here a multi-usable aluminum cap) therefore very user-friendly. After testing in a trial

environment with according preparations and is consequently proficient in handling the test, will receive satisfying and reliable results. Regardless of which manufacturer the results comprise a higher spreading/ tolerance than typically with other parameters. Each user should take this into consideration. The TOC test has for these reasons eventually until today not succeeded to replace the CSB test. However, the results for each cuvette or reagent test – regardless for which parameter – always mirror the diligence applied at sampling, sample preparation and test handling.