

Spotlight Feature: Environmental Control & Water Monitoring

The Right Handling of Samples

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

Susanne Gollor, WTW GmbH

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 CONVINCED WITH A WIDE RANGE IN FUNCTIONALITIES AND CONVENIENT HANDLING

Author Details:

Susanne Gollor
Product Management Photometry and Turbidity
WTW GmbH
Dr.-Karl-Slevogt-Strasse 1
82362 Weilheim, Germany
Phone +49-(0)881-183-0
Fax +49-(0)881-183-420
e-mail: info@wtw.com
www.wtw.com

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 CO₂. Afterwards the sample and cuvette reagent with permeable membrane are brought together and digested in a thermo-reactor. The arising CO₂ 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

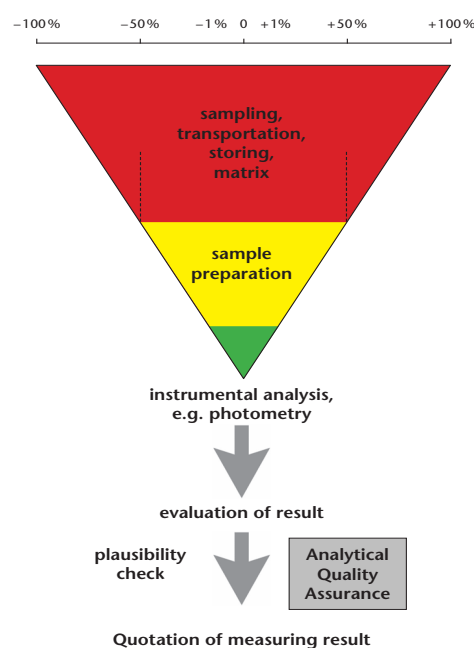


Figure 1. Failure frequency from taking the sample until retrieving the measurement result



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 CO₂ 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 CO₂. 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: CO₂ 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 CO₂. 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 CO₂ 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 (CO₂, HCO₃) 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.

Portable Meters for the Standard Parameters of Water: pH, D.O. Conductivity

WTW is a leading manufacturer of measuring instruments for the standard parameters of water and offers a solution for every application. If you need precision, portability, compact size, ruggedness and low power consumption for field use, then our 3xi family of portable instruments should be your choice. These meters are available in four different grades:

1. The **315i series** is for standard applications that require no automatic documentation and no enhanced sensor options. The series includes meters for the three most important standard parameters of water: pH/mV, D.O. and conductivity.
 2. If you expect to use special sensors or additional calibration procedures (e.g. for D.O.), the **330i series** is an ideal solution.
 3. If you require flexibility and easy and reliable data transmission, the **340i series** should be your choice. Within this series, there are available single parameter instruments for pH, D.O. and conductivity, as well as combination (pH/Cond, pH/D.O.) and multiparameter (pH/D.O./Cond) instruments.
 4. The **Multi 350i** is a class of its own. Fitted with special sensors, this exceptional multiparameter instrument can measure, display and store up to 3 parameters plus temperature simultaneously.
- Another different line of portable instruments is the **VARIO** line. These are quality pocket instruments for random sampling in process control, fast lab and service applications and training scopes. The VARIO is available for pH/mV and conductivity measurement.

Circle no. 76



Portable Water Laboratories: pHOtoFlex and Turb 430 in a Handy Field Case



With the pHOtoFlex and Turb 430 Series, WTW offers a new and innovative instrument line for optical measurements in the field. The **pHOtoFlex Series** are multi-purpose 6-wavelengths photometers, covering not only almost any chemical parameter in water analysis but also turbidity (photoFlex Turb) and pH measurement with automatic temperature compensation. If you only want to measure turbidity, the **Turb 430 Series** comprises an infrared or tungsten version of these new turbidimeters.

To make the instruments even more suitable for field use, they are now available as complete sets in a practical field case with a particularly user-friendly and innovative small table tray serving as onsite bench. Depending on the instrument model, the sets come with different accessories: For the pHOtoFlex models: adjustable pipette with 5 ml volume, pH electrode SenTix® 41, stand for pH electrode, buffer solutions with pH 4.01 and 7.00, beaker, calibration kit with turbidity standards (pHOtoFlex Turb). For the Turb 430 models: calibration kit with turbidity standards.

All sets are completed by empty cuvettes, PC connecting cable, waterproof quick instruction, cleaning tissues etc. Together with the LabStation and its new software package LSdata the sets offer a complete solution for field and laboratory tasks.

Circle no. 75

VIS and UV/VIS Spectrophotometers

WTW

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spectroFlex 6100 and spectroFlex 6600

From water analysis to research labs

- 150 test kits and spectral analysis
- Enhanced AQA functions
- USB interface

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