

TECHNICAL NOTE

Autodilution with the vario TOC cube

Introduction

Although the vario TOC cube is calibrated over a large concentration range, liquid samples with unknown origin or affected by unexpected contamination can contain higher TOC concentrations than covered by the current calibration.

The CO₂ signal detected by the IR detector is typically linearly correlated over several decades with the carbon concentration in the sample. To ensure reliable results, the measured concentration should always be within the calibration range.

To avoid double measurements and manual selection of overranged samples, the vario TOC cube provides a tool which automatically reduces the CO₂ concentration in the measuring gas to achieve a final concentration within the calibration range.

AUTODILUTION

Automatic adaptation of the injection volume ensures high quality organic carbon analysis.



Autodilution

By using the precise methods of our vario TOC cube, the concentration of the analyte (CO₂) in the measuring gas is automatically reduced. This is achieved by an automated calculation of a suitable injection volume which leads to a final CO₂ concentration in the middle of the current calibration. The calculation of the injection volume is based on the pre-analysis of a very small sample aliquot. The sample pre-analysis is neither massively affecting the cycling time nor resulting in other maintenance actions than usual.

With this method, the total carbon amount, which is combusted, is reduced for overranged samples and a CO₂ dilution, which meets the calibration, is processed by the IR detector.

To operate the vario TOC cube with the automated dilution function for unknown samples, the following steps have to be conducted:

- Do not enter an injection volume for the unknown sample and use a "precise" method (Figure 1).
- In this case you are asked to confirm that the sample volume should be calculated automatically (Figure 2) and an aliquot of the sample is injected and analyzed.
- From the results of this sample aliquot analysis, a suitable injection volume is calculated for this sample to meet the calibration range (Figure 3).
- The adapted injection volume is combusted and all carbon in the sample is transferred into CO₂.
- Consequently, the concentration of final analyte (CO₂), which is detected, is reduced in the measuring gas to meet the calibration range.

Example

In the example below, a 50 mg/l NPOC sample was first analyzed with 1 ml injection volume (Sample - not diluted). The resulting CO₂ concentration lies above the current calibration range of the instrument, resulting in slightly overestimated NPOC concentrations. Using the automatically calculated injection volume (Sample-autodiluted), the analyses meet the calibration range and the NPOC concentration could be analyzed with a high precision and accuracy.

| Hole Pos. | Name | Method | NPOC vol. [ml] | NPOC Area | NPOC [mg/l] |
|-----------|----------------------|----------------|----------------|-----------|-------------|
| 1 | Sample - not diluted | NPOC - precise | 1.000 | 0 | 0.000 |
| 1 | Sample - not diluted | NPOC - precise | 1.000 | 0 | 0.000 |
| 1 | Sample - not diluted | NPOC - precise | 1.000 | 0 | 0.000 |
| 2 | Sample - autodiluted | NPOC - precise | 0.000 | 0 | 0.000 |
| 2 | Sample - autodiluted | NPOC - precise | 0.000 | 0 | 0.000 |
| 2 | Sample - autodiluted | NPOC - precise | 0.000 | 0 | 0.000 |

Figure 1. To use the autodilution function, the injection volume has to be set to zero.

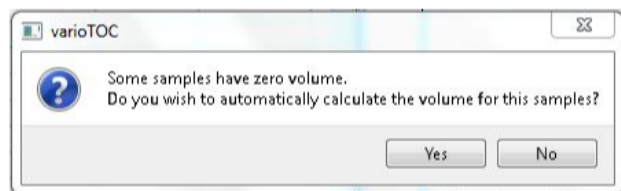


Figure 2. A pop-up window asks to confirm the automatic injection volume calculation.

| Hole Pos. | Name | Method | NPOC vol. [ml] | NPOC Area | NPOC [mg/l] |
|-----------|----------------------|----------------|----------------|-----------|-------------|
| 1 | Sample - not diluted | NPOC - precise | 1.000 | 118 199 | 51.614 |
| 1 | Sample - not diluted | NPOC - precise | 1.000 | 119 432 | 52.153 |
| 1 | Sample - not diluted | NPOC - precise | 1.000 | 119 743 | 52.289 |
| 2 | Sample - autodiluted | NPOC - precise | 0.100 | 11 545 | 50.086 |
| 2 | Sample - autodiluted | NPOC - precise | 0.100 | 11 747 | 50.971 |
| 2 | Sample - autodiluted | NPOC - precise | 0.100 | 11 788 | 51.149 |

Figure 3. Results of the analysis of a 50 mg/l sample using autodilution.

Benefits

The vario TOC cube can work with the procedure described above because the sensitive, wide-range IR detectors measures concentrations from the very low to high carbon range. In addition, the injection and flow control concepts also support the analysis of particle containing samples with high particle diameters up to 0.8 mm and injection volumes from 0.1 ml up to 2 ml.

The approach for meeting the calibration range with unknown samples of the vario TOC cube has been chosen for several reasons:

- An alternative would be the dilution of the liquid sample with water, but then the results can be affected by the water quality and interferences. In addition, the water consumption is higher and a potential water container has to be observed, maintained and exchanged frequently.
- If transferring the liquid sample to another vessel for dilution, this vessel could potentially have a different blank or be contaminated and therefore affect the final result.
- The quality of the results with the approach of the vario TOC cube is ensured, because the mixture of highly pure gases are adapted according to the underlying calibration. There is no influence of interferences, e.g. resulting from a different sample container where the samples is transferred to.
- The injection volume is always displayed and the use of an additional dilution factor obsolete.
- No pre-defined dilution factors are necessary, the appropriate amount of sample needed for the injection is calculated automatically.
- The vario TOC cube can still support automatic acidification in NPOC mode, because the sample is not transferred to another vial.

Conclusions

The vario TOC cube delivers a valid concept for meeting the calibration range in case of unknown samples. No cumbersome double-measurements, additional calibrations or manual dilutions are necessary. Consequently, this method saves time and resources, especially when analyzing many samples with unknown concentrations.

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