Tech Note
multi N/C x300 Series – TOC Calibration Strategies

Comparison of TOC calibration strategies with multi N/C x300 series

Introduction

Non-dispersive infrared spectroscopy (NDIR) is the most widely used detection method employed by commercially available TOC analyzers in the laboratory. The optical detector must be calibrated for the determination of TOC concentrations. There are different approaches to calibrate the analyzer.

The most widely used procedure is calibration by varying TOC concentrations and constant injection volume, the so-called "Classic" calibration approach.

The first alternative option is to calibrate by varying injection volumes and constant TOC concentration, enabled by the VITA flow management system. This calibration strategy is called "Easy Cal".

The second alternative calibration option is offered by the multi N/C 3300 in combination with e.g. AS vario autosampler. Thanks to an automated dilution function, it is possible to prepare different calibration levels from one stock solution by the autosampler, which is called "Automatic Dilution" approach.

Your Benefits

- Free choice of calibration strategy, whatever suits best
- Fully automated calibration
- Labor and time savings to produce calibration standards
- One calibration for the entire working range

Application

All calibration measurements were performed by multi N/C 3300 with AS vario, rack 72 pos. and 40 mL vials, using an NPOC method. Further possible configurations of multi N/C 3300, also supporting the "Automatic Dilution" calibration approach would be AS vario, rack 100 pos. and 20 mL vials (in this case max. dilution ratio is limited to 1:400), AS 21hp or AS 10e with dilution rack with 50 mL vials (max. 10 calibration standard pos. for "Automatic Dilution" available). The method and instrument parameters are listed in Table 1. The standards used were prepared from a 1000 mg/L potassium hydrogen phthalate stock solution.

Table 1: Method parameters multi N/C 3300

Parameter	Values
Method	NPOC
Furnace temperature	750 °C
Number of repetitions	4 out of 4
Injection volume	500 µL (for constant volumes, "Classic", "Automatic Dilution") variable (for constant concentration, "Easy Cal")

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To make the three different calibration methods comparable, the same procedure was used for this study. All calibration runs consist of the same five calibration points. The calibration range was defined by the maximum possible range covered by Easy Cal, which is 1:20, using the maximum dosing volume range from 50 – 1000 µL. The other two calibration strategies, however, can cover significantly wider concentration ranges. Table 2 lists the prepared standards and the injection volumes as well as the dilution factors of the calibrations.

Table 2: Concentrations of calibration standards and injection volumes

Calibration point	Calibration "Classic"		Calibration "Easy Cal"		Calibration "Automatic Dilution".		
	c(TOC) [mg/L]	V_{INJ} [µL]	c(TOC) [mg/L]	V_{INJ} [µL]	c(TOC) [mg/L]	Dilution factor	V_{INJ} [µL]
1	0.5	500	5	50	50	1:100	500
2	1.0	500	5	100	50	1:50	500
3	2.5	500	5	250	50	1:25	500
4	5.0	500	5	500	50	1:10	500
5	10.0	500	5	1000	50	1:5	500

All standards used were filled into 40 mL sample vials and covered with aluminum foil before analysis. For the "Classic" calibration, the five standards (Table 2) were filled into 5 different sample vials. For the "Easy Cal" calibration, only one full sample vial was positioned on the autosampler. For the "Automatic dilution" calibration, five sample vials were filled with the standard and additional five empty vials were positioned on the autosampler for dilution preparation with system water of the multi N/C 3300.

Results

The generated calibration parameters of the three different calibration approaches are shown in Table 3.

Table 3: Calibration parameters

Calibration strategy	k0	k1	R^2	Limit of Detection [mg/L]	Limit of Quantification [mg/L]	Range boarders [µg]
"Classic"	-16.85	1491.97	0.99972	0.15	0.57	0.25 – 5.00
"Easy Cal"	10.86	1411.18	0.99981	0.04	0.17	0.25 – 5.00
"Automatic Dilution"	-16.62	1459.36	0.99969	0.16	0.64	0.25 – 5.00

All three calibration results show very comparable key figures. Both, the slope (k1) and the coefficient of determination (R^2) are almost identical for the three calibration curves. The "Easy Cal" calibration shows slightly lower detection (LOD) and quantification limits (LOQ). The calibration curves are shown in the

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following Figures 1-3. It is also clear from these figures that there are no significant differences between the calibrations.

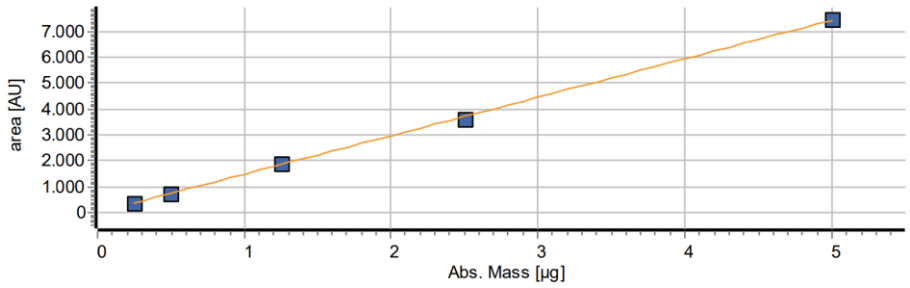

Figure 1: "Classic" calibration curve

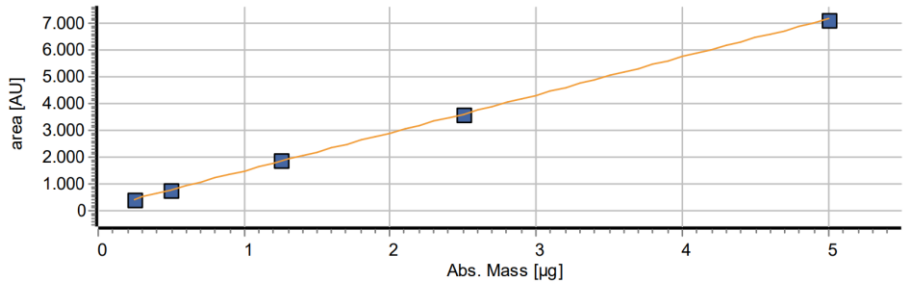

Figure 2: "Easy Cal" calibration curve

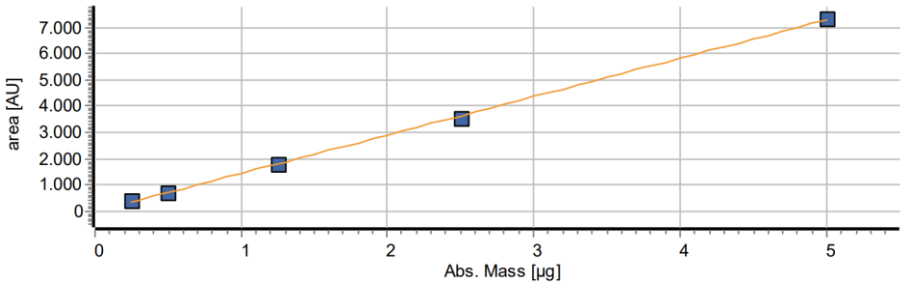

Figure 3: "Automatic Dilution" calibration curve

Recovery

The quality of each calibration was checked using a quality control standard (QC). The QC standard was analyzed once and then evaluated with the specific calibration parameters as listed in Table 3. The measurement results are shown in Table 4.

Table 4: Measurement results of QC standard (4.0 mg/L TOC, injection volume 500 µL)

Meas. Parameter	Avg. integral net [AU]	Avg. concentration [mg/L]	RSD [%]	Recovery [%]	k0	k1
NPOC	2929	3.95	0.67	98.75	-16.85	1491.97
NPOC	2929	4.14	0.67	103.5	10.86	1411.18
NPOC	2929	4.04	0.67	101.0	-16.62	1459.36

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All three calibration methods gave a comparable TOC recovery rates of the QC standard tested. The recovery rates range from 98.75 % to 103.5 %.

Conclusion

In this study the three available calibration strategies "Classic", Easy Cal" and "Automatic Dilution" were carried out with a comparable approach. All 3 strategies produced equally reliable results, meaning the choice of calibration method has no influence on the quality of the results.

The "Classic" calibration approach by varying TOC concentrations and constant injection volume is standardized in many norms (e.g. DIN EN ISO 20236), most widely used and forms the cornerstone of calibrations in routine analysis.

"Easy Cal" as the first alternative calibration option uses varying injection volumes while keeping the TOC concentration constant is enabled by the VITA flow management system in the multi N/C x300 analyzers. By the "Easy Cal" approach a concentration range within a factor of 20 can be calibrated by default. In contrast to the "Classic" calibration, only one calibration stock standard solution is required here, which can save most of the labor and time required for standard preparation. By utilizing more than one stock solutions in the "Easy Cal" approach, the calibration boundaries can easily be pushed to bigger concentration ranges.

The "Automatic Dilution" approach as the second alternative calibration strategy combines the advantages of the first two calibration strategies. On the one hand, labor and time can be saved to produce many calibration standards and on the other hand no restriction of the calibration range is encountered. For larger calibration ranges, "Automatic Dilution" offers considerable time savings in the preparation of standards compared to 'Classic' calibration. According to DIN EN ISO 20236, at least 5-10 calibration solutions must be prepared as evenly as possible over the calibration range. Automatic dilution in a 50 mL vial makes it possible to cover a calibration range over a factor of 1000 (e.g. 1 -1000 mg/L) with just one stock standard solution.

All three calibration methods were compared using an NPOC method. However, the two "concentration-dependent" methods ("Classic" and "Automatic Dilution") are also suitable for calibration of the parameter total bound nitrogen (TN_b) or combined NPOC/TN methods.

Reference: TechNote_multiNC_0003_TOC_Calibration Strategies_en.docx

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