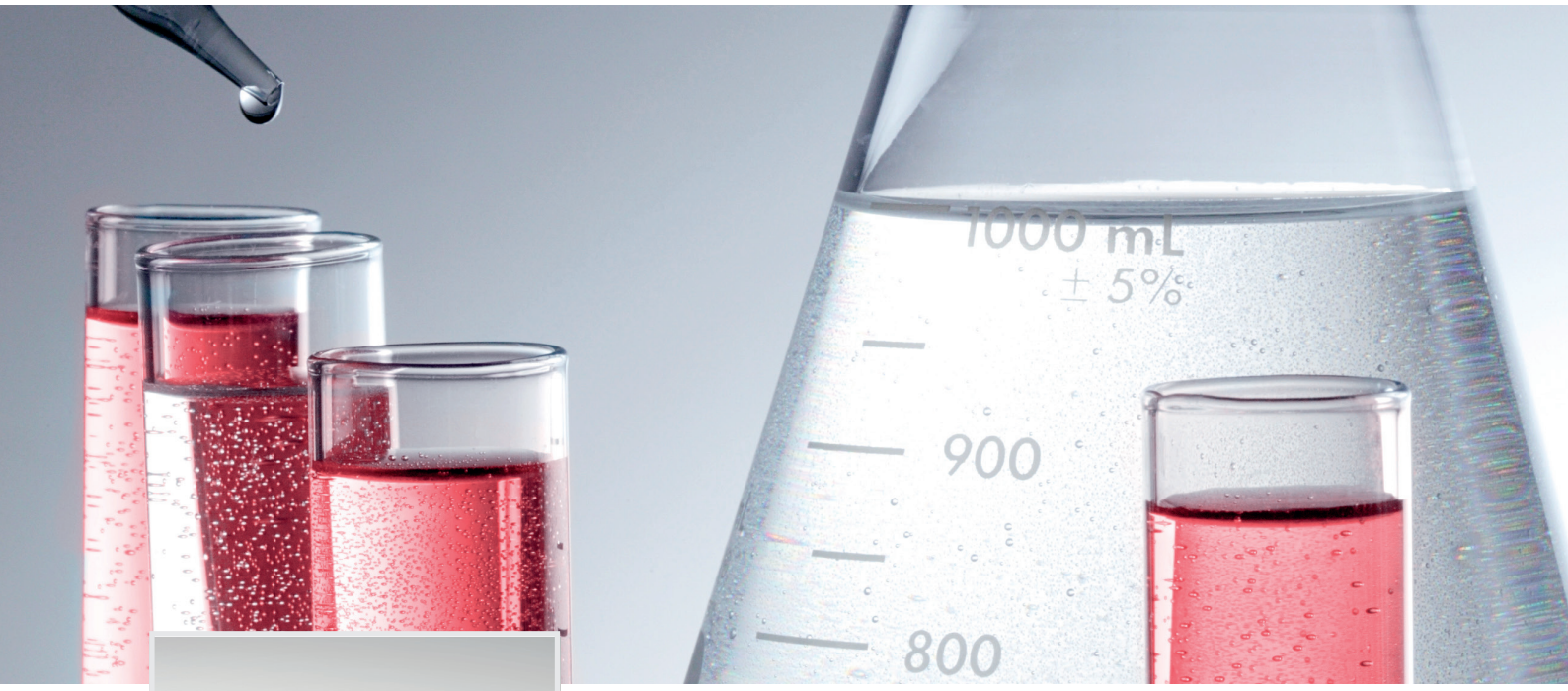

Challenge

Reliable determination of TOC in samples with an aggressive matrix.

Solution

Fully automated product quality control of H_3PO_4 for TOC impurities by robust UV persulfate oxidation and NDIR detection.

TOC Determination in Phosphoric Acid by UV Digestion

Introduction

Phosphoric acid is used in many industries, for example, in the production of fertilizers and detergents or in food industries as a preservative in beverages or as an antioxidant in meat products. The monitoring of impurities in phosphoric acid is crucial for these kinds of applications. In addition to other parameters, the TOC is used as a sum parameter in quality control that addresses the whole range of contamination by organic impurities.

There are two very common approaches that can be applied for TOC determination in liquid aqueous matrices: catalytic high temperature combustion and wet chemical digestion promoted by UV light and additional chemical oxidants.

Wet chemical UV digestion is the principle of choice for very aggressive matrices, such as concentrated phosphoric acid or other mineral acids (except hydrochloric acid). While the phosphoric acid may quickly damage the combustion tube and catalyst in catalytic combustion TOC methods, the big advantage of UV digestion is a minimum of maintenance and wear of the TOC system. The oxidation power of the multi N/C UV HS that was used for these measurements is very high due to its high-power, long-life UV reactor. Hence, a complete oxidation of the organic compounds inside the sample is ensured.

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Materials and Methods

A TOC analyzer multi N/C UV HS based on the UV/wet chemical digestion principle was used for the measurements of phosphoric acid samples. Sample introduction was done using the automatic sampling system AS vario.

Samples and Reagents

- Three different samples of concentrated phosphoric acid were analyzed.
- Phosphoric acid 10% (for optional TIC control measurement)
- Sulfuric acid 2 M for automatic acidification in NPOC mode and preparation of oxidation reagent
- Sodium peroxodisulphate solution 80 g/L as oxidation reagent

Sample Preparation

All samples were diluted manually before measurement, due to the high viscosity of the original sample. The dilution ratios are shown in table 1.
No further preparation steps were applied. Sample acidification can be omitted for NPOC testing in concentrated acids.

Calibration

Calibration standards of different concentration levels from 0.5 – 100 mg/L were prepared from a 1000 mg/L TOC stock solution of potassium hydrogen phthalate in water and acidified with sulfuric acid.

A calibration run was started, and a linear calibration curve was obtained. An example is shown below.

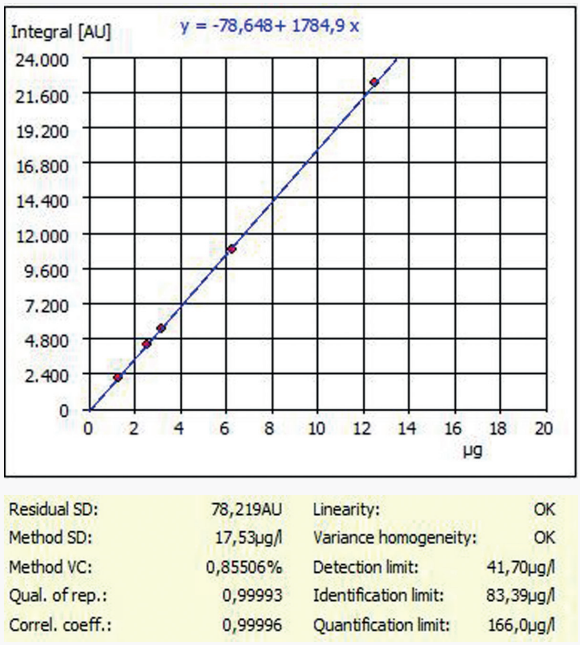

Figure 1: liner regression and method characteristics for TOC calibration

Within the method up to 3 calibration ranges can be linked to each parameter in order to cover an over-all working range of up to 3 magnitudes. Detection limits and limits of quantification are depending on the selected working range and can be derived from the method characteristics given above.

Instrumentation

The measurements were performed in the NPOC mode with method settings shown in the below table.

Parameter	Method Settings
Method / Parameters	NPOC + oxidation reagent
Digestion	UV / wet chemical oxidation
Number of replicates	min. 2 – max. 3 injections
Rinsing cycles	3 times with sample
Sample purge time.	180 sec
Injection volume	5000 µl
Dilution ratios	1 in 10

Results and Discussion

The following table shows the mean values of the NPOC measurements calculated from at least duplicate injections as well as the relative standard deviation. A typical NPOC measurement curve is shown in the picture below.

Table 1: Results TOC

Sample ID	Sample Dilution	Mean Value TC [mg/L]	RSD [%]
Sample 1	1 in 10	65.7	0.7
Sample 2	1 in 10	192	0.8
Sample 3	1 in 10	84.8	0.5
Control Standard 10 mg/L TOC	-	10.1	0.9

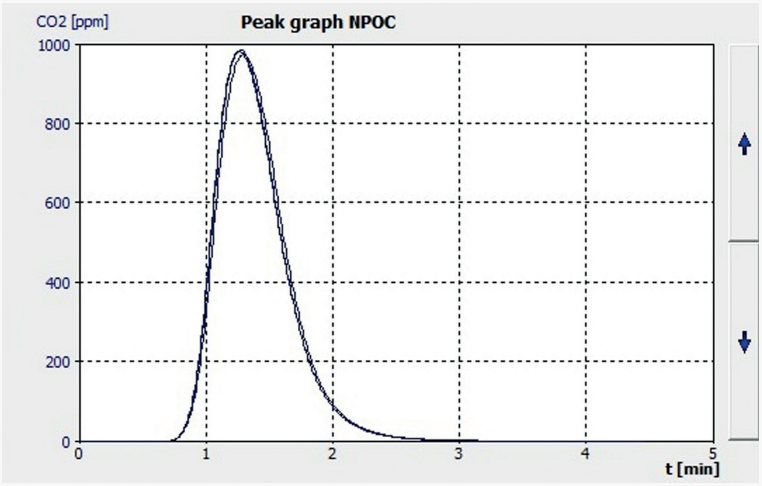

Figure 2: example of a measurement signal for sample 2

Conclusion

The results show very good reproducibility. The instrument is very well suited for the determination of TOC in concentrated phosphoric acid and similar samples with aggressive matrices.

By applying the high throughput autosampler AS vario the parallel purge and analyses feature of multi N/C analyzers assure short sequence runtimes.

A high degree of automation combined with the well-proven Self Check System for trouble free unattended system operation makes TOC analyses by multi N/C systems the most comfortable parameter in your lab.

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