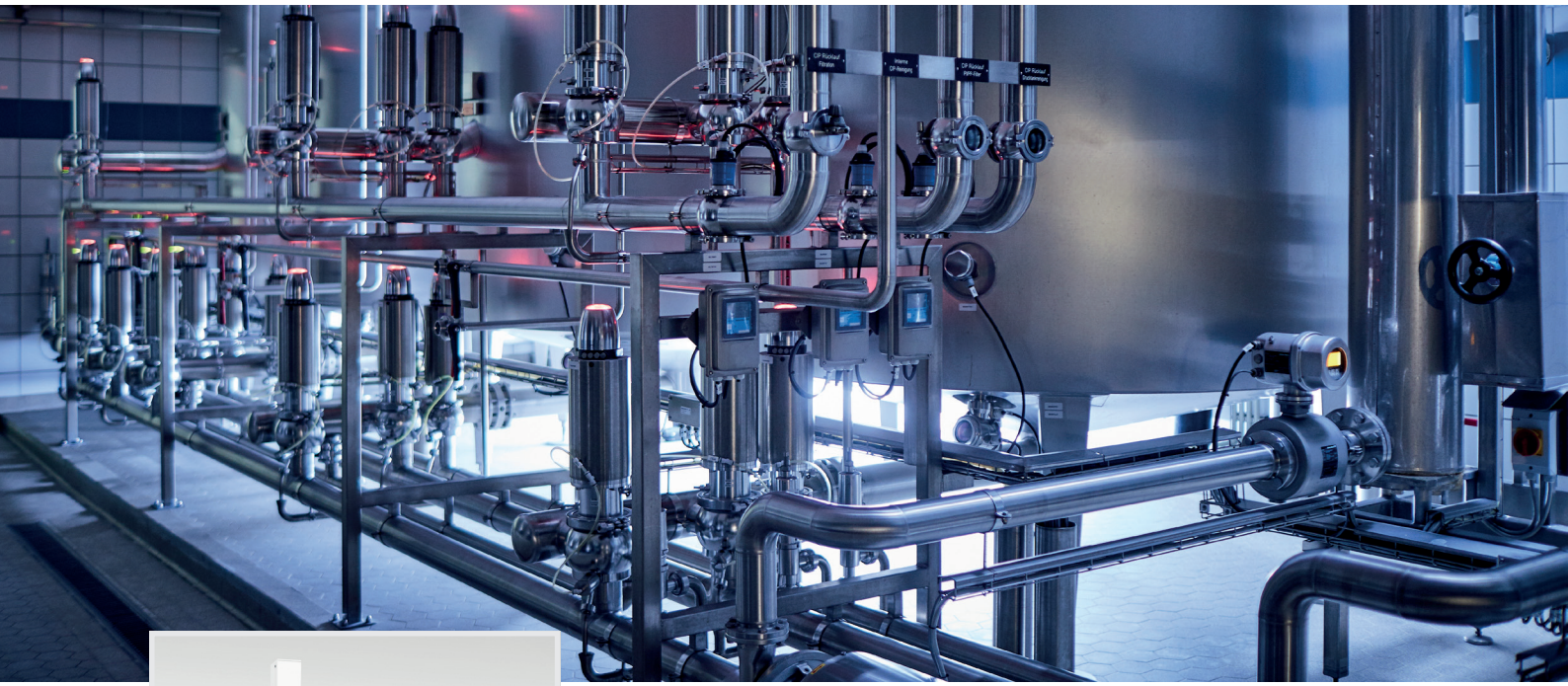


Challenge

Reproducible and precise determination of TOC and TN contents in the lower ppm range in highly concentrated H_2O_2 matrices (up to 70% H_2O_2).

Solution

Fully automated and simultaneous TOC/TN measurement using catalytic high temperature combustion. Undiluted sample measurement up to 70% H_2O_2 concentration.

Analysis of TOC/TN in Product Quality Monitoring of Hydrogen Peroxide

Introduction

Hydrogen peroxide is used in many processes. It can be used as a disinfectant in the food industry or in water purification, as an oxidation agent in chemical synthesis or as an environmentally friendly means of bleaching pulp, paper, and textiles. According to the different fields of application, products of various H_2O_2 concentrations and purity grades are available. The industrial production of hydrogen peroxide is done via the anthraquinone process, followed by cleanup steps, such as fractional distillation.

To guarantee product quality H_2O_2 is monitored for elemental impurities, for example, for heavy metals or for TOC concentration. This application note focuses on the determination of TOC and TN in undiluted H_2O_2. The TOC concentration in H_2O_2 samples can vary in a wide range from approx. 1 to 500 mg/L depending on the manner of product stabilization and grade of cleanup. TN is a parameter optionally monitored for process control but not, however, typically specified for final products. Since H_2O_2 is a strong oxidant and sensitive to decomposition, the way in which the sample is handled and the robustness of the materials used inside the TOC/TN analyzer are crucial for the overall stability of the analytical method.

The measurements were performed on a multi N/C 2100S and a multi N/C 3100. Both instruments allow simultaneous analysis of TOC and TN, enabling high sample throughput. Equipped with the Focus Radiation NDIR detector technology, the systems allow highly sensitive CO_2 detection for reliable results down to lowest concentration ranges.

analytikjena
An Endress+Hauser Company

Materials and Methods

Samples and Reagents

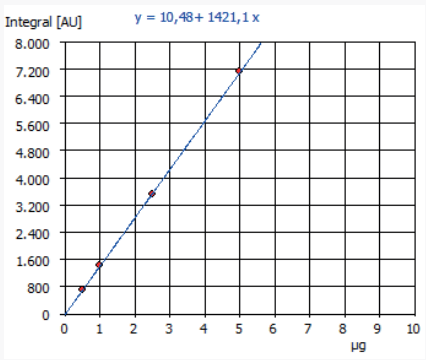
Samples from different product chains, batches and clean-up stages as well as reference materials were analyzed applying one and the same method.

Sample Preparation and Measurement

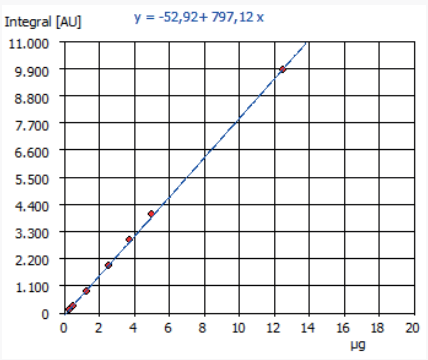
The samples had been stored in a refrigerator at 4 °C until analysis and then transferred into suitable autosampler vials. Since the production process of H_2O_2 takes place in a strongly acidic pH, the TIC (total inorganic carbon) content of the product is negligible. Hence, total carbon (TC) is set equal to total organic carbon (TOC).
For TC/TN determination representative sample aliquots were injected directly into the combustion tube. The samples were oxidized completely at 800 °C in an oxygen atmosphere using a platinum catalyst. After purification of the reaction gas, the formed CO_2 and NO were transported to the detector by means of a carrier gas. The quantification of TC was carried out by non-dispersive infrared spectrometry in the focus radiation NDIR detector. TN was detected with an electrochemical detector (ChD).

Calibration

For total carbon (TC) a wide range calibration from 1 to 200 mg/L was carried out using potassium hydrogen phthalate standard solutions prepared in ultrapure water. For TN the system was calibrated using a mix of ammonium sulphate and potassium nitrate standard solutions prepared in ultrapure water, to cover a range from 0.5–25 mg/L.



Residual SD:	27,003AU	Linearity:	OK
Method SD:	38,00µg/l	Variance homogeneity:	OK
Method VC:	0,84449%	Detection limit:	106,0µg/l
Qual. of rep.:	0,99994	Identification limit:	212,1µg/l
Correl. coeff.:	0,99997	Quantification limit:	454,1µg/l



Residual SD:	41,156AU	Linearity:	OK
Method SD:	103,3µg/l	Variance homogeneity:	OK
Method VC:	1,4036%	Detection limit:	149,7µg/l
Qual. of rep.:	0,99988	Identification limit:	299,3µg/l
Correl. coeff.:	0,99994	Quantification limit:	564,6µg/l

Within the method up to three calibration ranges can be linked to each parameter in order to cover an over-all working range of up to three magnitudes.

Figure 1 and 2: Example of TC and TN calibration and method characteristics

Method Parameters

The following method settings were used to determine the TC and TN content:

Table 1: Method settings

Parameter	multi N/C 2100S / 3100
Measurement parameter	TC/TN
Digestion	High temperature digestion at 800 °C with platinum catalyst
Number of repetitions	min. 3, max. 4
Rinse with sample before injection	3 times
Injection volume	250 µL

Results and Discussion

Roughly and completely cleaned-up hydrogen peroxide solutions of different batches and H_2O_2 concentrations were measured undiluted. The concentration values reported below are based on the averages of at least three replicate injections.

Table 2: Results

Sample name	TOC [mg/L]	TOC RSD [%]	TN [mg/L]	TN RSD [%]
Sample 1 (50% H_2O_2)	196.6	4.3	12.35	2.9
Sample 2 (70% H_2O_2)	167.5	4.1	19.01	2.7
Standard TC 100 TN 10	103.2	0.4	10.06	0.3
30% H_2O_2_LOT1	3.95	0.7	3.26	1.6
31% H_2O_2_LOT2	1.20	1.1	1.03	2.2
60% H_2O_2_LOT2	2.12	0.5	1.84	2.0
Standard TC 2.5 TN 2.5	2.58	0.9	2.45	0.6

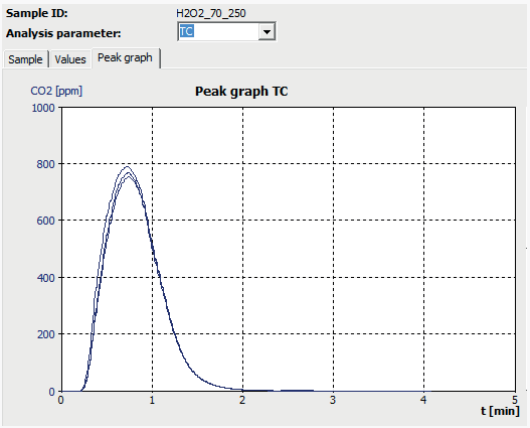


Figure 3: example for a TOC measurement curve of sample 2

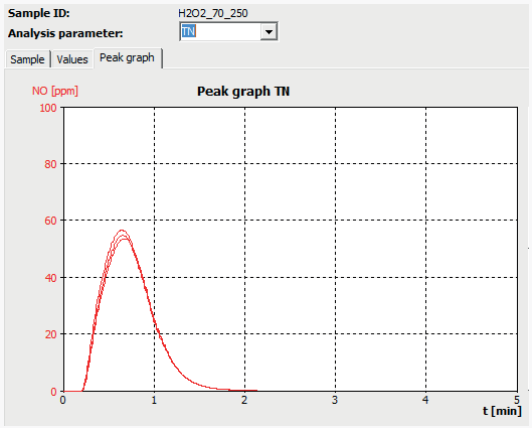


Figure 4: example for a TN measurement curve of sample 2

Conclusion

The results of the analysis show that multi N/C 2100S and multi N/C 3100 are ideally suited for the determination of the TOC and TN content in hydrogen peroxide. The flexibility of multi N/C analyzers in terms of the injection volumes used and the free choice of combustion temperatures up to 950 °C enable an optimum method setup for measuring undiluted hydrogen peroxide samples with concentrations up to 70%. Using up to three calibration ranges for each parameter allows the application of one single wide-range method to cover TOC/TN monitoring tasks for the whole hydrogen peroxide product spectrum of interest.

Thus, multi N/C analyzers offer a highly comfortable solution for monitoring intermediate and finished products for TOC/TN concentrations in hydrogen peroxide production and quality control. A high degree of automation can be achieved using AS 60 or AS vario, which ensures cost-efficient analyses and a high sample throughput in routine quality control labs.

Headquarters
Analytik Jena GmbH
Konrad-Zuse-Strasse 1
07745 Jena · Germany

Phone +49 3641 77 70
Fax +49 3641 77 9279

info@analytik-jena.com
www.analytik-jena.com

Version 1.0 · Author: BeBl
en · 12/2020