

## TOC and TN Evaluation of Hydrogen Peroxide Solution

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### User Benefits

- ◆ Simultaneous and rapid determination of total organic carbon (TOC) and total nitrogen (TN) in hydrogen peroxide solution can be realized.
- ◆ High-concentration hydrogen peroxide solution can be analyzed without dilution.
- ◆ Automatic analysis of multiple samples is possible, with the use of the ASI-L auto-sampler.

### Introduction

Due to the characteristic of decomposing into water and oxygen, hydrogen peroxide has gained considerable attention as an environmentally friendly chemical. It finds versatile applications across various concentrations, ranging from common disinfectants and household bleach to industrial oxidants and cleaning agents in semiconductor manufacturing processes.

In the field of semiconductors, as devices are becoming increasingly miniaturized and densely integrated, the demand for utmost purity in the chemicals used for cleaning processes has become increasingly stringent. Consequently, meticulous quality control may be necessary for cleaning agents such as hydrogen peroxide solution.

By using the Shimadzu TOC-L Total Organic Carbon Analyzer, it is possible to measure the total organic carbon (TOC) in high-concentration hydrogen peroxide solution without dilution. Furthermore, by incorporating the TNM-L Total Nitrogen Unit, simultaneous measurement of total nitrogen (TN) concentration in hydrogen peroxide solution is achievable. This capability makes it a valuable tool for managing contamination in hydrogen peroxide solution.

This article describes an example of TOC and TN measurements for 30 % hydrogen peroxide solution by using the Shimadzu TOC-L analyzer.

### Sample Preparation

In this experiment, samples were prepared by adding specific amounts of TOC and TN to 30 % hydrogen peroxide solution of special reagent grade.

More specifically, potassium hydrogen phthalate was added to the 30 % hydrogen peroxide solution to obtain TOC concentrations of 1 mgC/L, 5 mgC/L, and 10 mgC/L, respectively. Additionally, potassium nitrate was added to achieve TN concentrations of 1 mgN/L, 5 mgN/L, and 10 mgN/L, respectively. Table 1 summarizes the sample preparation.

Table 1 Sample Preparation

Samples	TOC [mgC/L]	TN [mgN/L]
Hydrogen peroxide solution	0	0
Hydrogen peroxide solution + 1 mg/L	1	1
Hydrogen peroxide solution + 5 mg/L	5	5
Hydrogen peroxide solution + 10 mg/L	10	10

### Analysis Method

A system consisting of the TOC-L Total Organic Carbon Analyzer with the addition of the TNM-L was employed for the analysis (Fig. 1). The measurement conditions are outlined in Table 2.

For TOC measurement, the non-purgeable organic carbon (NPOC) method was applied. In the conventional NPOC method, the sample is initially acidified and then subjected to sparging to remove inorganic carbon (IC), after which the total carbon (TC) is measured as TOC. However, in this experiment, as the 30 % hydrogen peroxide solution used was already acidic, no additional acid was required.

Calibration of the instrument was performed using 0 and 20 mgC/L potassium hydrogen phthalate aqueous solutions for TC measurements, and 0 and 20 mgN/L potassium nitrate aqueous solutions for TN measurements. The calibration curves were corrected by performing an origin shift to account for the TOC or TN contained in the pure water used in preparing the standard solution.



Fig. 1 System Consisting of the TOC-L Total Organic Carbon Analyzer and the TNM-L Total Nitrogen Unit  
(A) ASI-L Auto-Sampler, (B) TNM-L Total Nitrogen Unit, (C) TOC-L Total Organic Carbon Analyzer

Table 2 Measurement Conditions

Instrument	TOC-L <sub>CPH</sub> + TNM-L
Catalyst	TOC/TN catalyst
Measurement Items	NPOC (= TOC using acidification and sparging) TN
Injection Volume	50 µL
Calibration Curves	TC: 2-point calibration curve with 0 and 20 mgC/L of potassium hydrogen phthalate aqueous solution TN: 2-point calibration curve with 0 and 20 mgN/L of potassium nitrate aqueous solution

## Measurement Results

Table 3 Measurement Results of Hydrogen Peroxide Solution

Samples	TOC [mgC/L]	Recovery Rate [%]	TN [mgN/L]	Recovery Rate [%]
Hydrogen peroxide solution	5.39	-	0.34	-
Hydrogen peroxide solution + 1 mg/L	6.40	100	1.33	98.9
Hydrogen peroxide solution + 5 mg/L	10.2	96.5	5.26	98.5
Hydrogen peroxide solution + 10 mg/L	15.1	97.0	10.1	97.6

The results of measuring TOC and TN in the hydrogen peroxide solution are shown in Table 3. It is noteworthy that all samples exhibited a recovery rate of 96 % or higher.

Fig. 2 illustrates the correlation between the added concentrations and the measured concentrations, revealing a strong correlation with a correlation coefficient of 1.000. This indicates that the added TOC and TN were accurately measured. Examples of detailed peak data from the TOC-L analyzer are provided in Figs. 3 and 4.

The obtained findings confirm that the TOC-L Total Organic Carbon Analyzer in conjunction with the TNM-L, enables the measurement of TOC in hydrogen peroxide solution at concentrations of approximately 1 mgC/L and TN at concentrations of approximately 1 mgN/L.

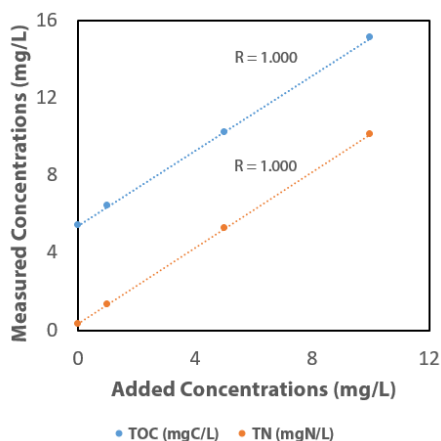
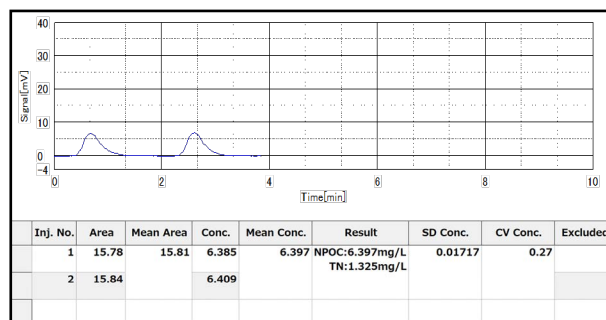


Fig. 2 Correlation between the Added Concentrations and the Measured Concentrations

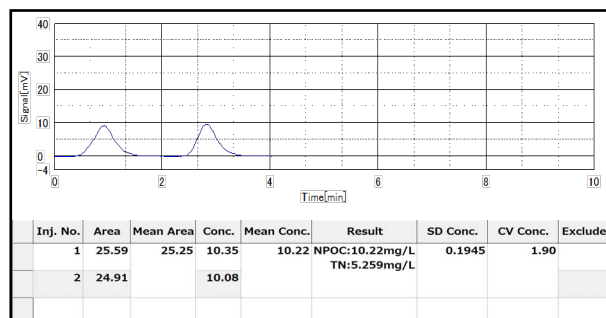
## Conclusion

The TOC-L and TNM-L system allows for simultaneous and rapid measurement of TOC and TN, even in high-concentration hydrogen peroxide solution without the need for dilution. This combined system proves to be a valuable tool for effective impurity management in hydrogen peroxide solution.

What's more, the incorporation of the ASI-L auto-sampler facilitates the automated measurement of multiple samples, thereby enhancing analysis efficiency.

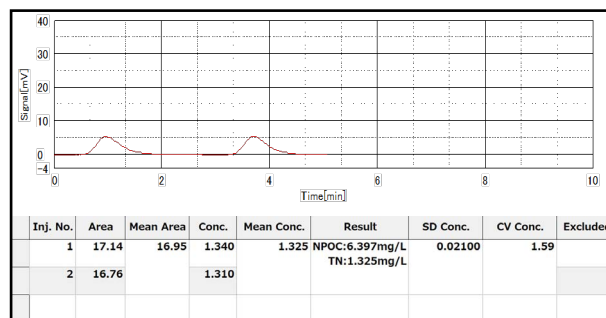


(a) Hydrogen Peroxide Solution + TOC 1 mgC/L

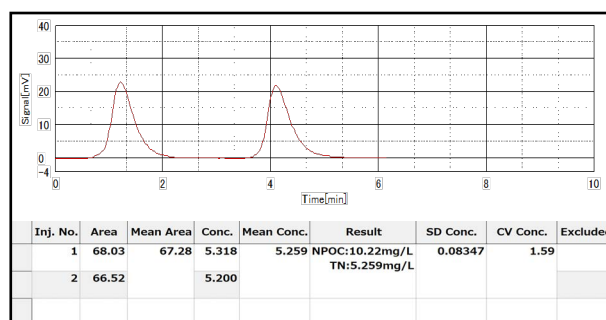


(b) Hydrogen Peroxide Solution + TOC 5 mgC/L

Fig. 3 Results of Hydrogen Peroxide Solution TOC Measurement



(a) Hydrogen Peroxide Solution + TN 1 mgN/L



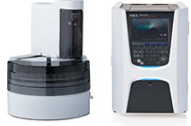
(b) Hydrogen Peroxide Solution + TN 5 mgN/L

Fig. 4 Results of Hydrogen Peroxide Solution TN Measurement

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