

# Application News

## No. 069

### Total Organic Carbon Analysis

## Cleaning Validation of Food Production Equipment by TOC and TN Measurement

Cleaning validation is the process of checking for residues of the previous product after cleaning production equipment and verifying that the amount does not exceed the allowable limit. Factories producing food products attach high priority to cleaning validation from the viewpoints of quality control and safety.

In addition to organic matter, food products also contain proteins, amino acids, etc. However, it is possible to evaluate the concentrations of organic matter by total organic carbon (TOC) and proteins and amino acids, which are nitrogen compounds, by total nitrogen (TN).

Therefore, proper cleaning of production equipment can be confirmed by measuring the TOC and TN contained in the rinse water after washing the equipment.

In general, the Kjeldahl method is widely used to measure proteins in food products, but time-consuming manual work is necessary, as analysis, distillation, etc. of individual samples is performed by using multiple reagents and implements.

However, if measurements are performed with a system consisting of a thermal decomposition-chemiluminescence TNM-L total nitrogen unit added to a Shimadzu TOC Analyzer TOC-L, simultaneous measurement of TOC and TN is possible quickly in a single operation requiring only about 5 minutes. Moreover, automatic measurement of multiple samples is also possible by using an autosampler.

This application news introduces an example in which the effect of cleaning was confirmed by measuring the TOC/TN of the rinse water when cleaning a container in which vinegar had been introduced, using Shimadzu TOC-L<sub>CPH</sub> and TNM-L.

M. Tanaka

### Sample Preparation

Using the round stainless steel container (capacity: approximately 700 mL) shown in Fig. 1, vinegar was deposited on the entire inner surface of the container by introducing approximately 5 mL of rice vinegar or brown-rice black vinegar. The container was cleaned by the following procedure, and the rinse water recovered after cleaning was used samples for TOC/TN measurement.

<Cleaning procedure>

- ① Discard the vinegar in the container.
- ② Introduce purified water up to the top of the container, stir, and then remove the used rinse water from the container. This rinse water is called 1<sup>st</sup> rinse water.
- ③ Washing again as in ②. This water is called 2<sup>nd</sup> rinse water.
- ④ Washing again in the same manner. This water is called 3<sup>rd</sup> rinse water.



**Fig. 1 Stainless Steel Container**

### Analysis Method

The TOC and TN of the purified water and the 1<sup>st</sup> to 3<sup>rd</sup> rinse water used in cleaning were measured simultaneously. The TOC calibration curve was prepared by calibration with 250 mgC/L of a potassium hydrogen phthalate aqueous solution. The TN calibration curve was prepared by calibration with 0 and 5 mgN/L of a potassium nitrate aqueous solution.



**Fig. 2 Typical Food Production Equipment**

**Table 1 Measurement Conditions**

Analyzer	: TOC-L <sub>CPH</sub> + TNM-L total nitrogen unit
Catalyst	: TOC/TN catalyst
Measurement items	: Simultaneous measurement of TOC (= TOC using acidification and sparging) and TN
Calibration curves	: TOC : Single point calibration curve using 250 mgC/L of potassium hydrogen phthalate aqueous solution TN : Two-point calibration curve using 0 and 5 mgN/L of potassium nitrate aqueous solution

### Measurement Results

Table 2 shows the results of measuring the purified water used in cleaning and the rinse water after cleaning the containers in which rice vinegar or brown-rice black vinegar was introduced. The concentrations of TOC and TN became progressively lower with repeated cleaning, and the concentrations of TOC and TN in the 3<sup>rd</sup> rinse water were on approximately the same level as the purified water used in cleaning.

We were able to confirm the container cleaning effect by evaluating the concentrations of TOC and TN in the rinse water in this method.

**Table 2 Measurement Results**

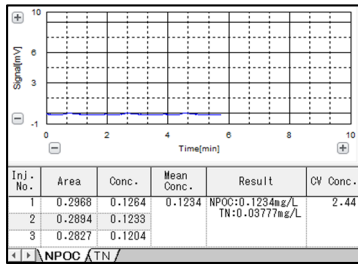
Sample	Measured TOC value (mgC/L)	Measured TN value (mgN/L)
Purified water used in cleaning	0.123	0.038

Sample	Measured TOC value (mgC/L)	Measured TN value (mgN/L)
Rice vinegar rinse water (1 <sup>st</sup> )	196.2	1.62
Rice vinegar rinse water (2 <sup>nd</sup> )	3.36	0.076
Rice vinegar rinse water (3 <sup>rd</sup> )	0.127	0.046

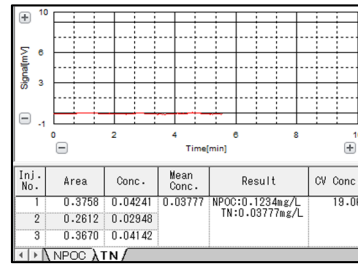
Sample	Measured TOC value (mgC/L)	Measured TN value (mgN/L)
Brown-rice black vinegar rinse water (1 <sup>st</sup> )	129.4	3.63
Brown-rice black vinegar rinse water (2 <sup>nd</sup> )	0.535	0.062
Brown-rice black vinegar rinse water (3 <sup>rd</sup> )	0.116	0.040

■ Measurement Data

Purified water  
used in cleaning

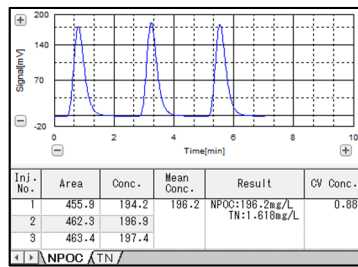


TOC measurement

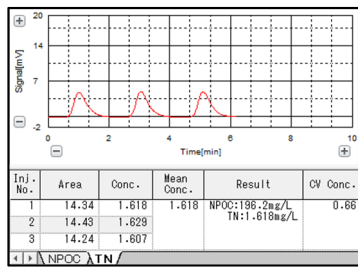


TN measurement

Rice vinegar  
rinse water (1<sup>st</sup>)

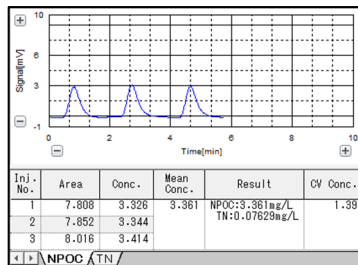


TOC measurement

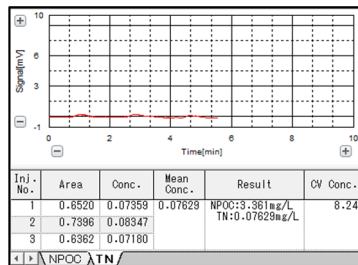


TN measurement

Rice vinegar  
rinse water  
(2<sup>nd</sup>)

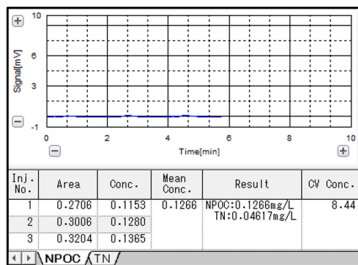


TOC measurement

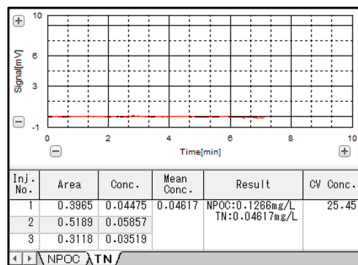


TN measurement

Rice vinegar  
rinse water  
(3<sup>rd</sup>)



TOC measurement



TN measurement

Fig. 3 Measurement Data of Rice Vinegar Rinse Water



Fig. 4 TOC-L Total Organic Carbon Analyzer +TNM-L Total Nitrogen Unit



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