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Application News

Total Organic Carbon Analyzer TOC-L
On-line TOC Analyzer TOC-4200

Detection of Phenol Leakage into Wastewater Using TOC Measurement

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User Benefits

- Phenol leakage into wastewater can be detected using a TOC analyzer without pretreatment.
- The TOC-L laboratory TOC analyzer enables single-instrument monitoring of wastewater, process water, and cleaning water.
- Rapid detection of phenol leakage in wastewater is achieved through continuous monitoring using the TOC-4200 on-line TOC analyzer.

■ Introduction

Phenol (Fig. 1) is a widely used industrial chemical employed in the production of plastics, pharmaceuticals, and other products. Because phenol is toxic and has a strong odor, its presence in process water, cleaning water, and wastewater must be carefully controlled, and leaks must be detected at an early stage. The conventional 4-aminoantipyrine colorimetric method (4-AAP method) is specific to phenol; however, it requires extraction, pH adjustment, and other pretreatment steps, which make the analysis time-consuming.

Total organic carbon (TOC) analysis, by contrast, oxidizes organic compounds in the sample and measures the resulting CO_2. This allows rapid evaluation of organic contamination without pretreatment, regardless of the type of organic compound present. The Shimadzu TOC-L total organic carbon analyzer can be used not only for wastewater management but also for quality control of process water and cleaning processes. The TOC-4200 on-line TOC analyzer enables rapid detection of phenol leakage through continuous monitoring.

This article presents an example of phenol leakage detection in wastewater using the Shimadzu TOC-L total organic carbon analyzer, which employs the combustion catalytic oxidation method.

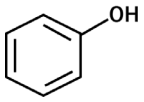


Fig. 1 Structural Formula of Phenol

■ Sample and Analysis Conditions

Wastewater collected from a Shimadzu facility was used as the base matrix for the analysis. Phenol was added to achieve concentrations of 3, 5, 10, 15, 25, and 50 mg/L, and seven samples (Samples 1-7) were prepared, as summarized in Table 1. TOC measurements of these samples were conducted under the conditions shown in Table 2.

Table 1 Samples

Sample	Added Phenol Concentrations (mg/L)
1	0 (Wastewater, used as the TOC blank)
2	3
3	5
4	10
5	15
6	25
7	50

Table 2 Measurement Conditions

Analyzer:	TOC-L total organic carbon analyzer
Oxidation Method:	680 °C combustion catalytic oxidation
Catalyst:	Standard catalyst
Measurement Item:	NPOC (non-purgeable organic carbon; TOC by acidification and sparging)
Calibration Curve:	2-point calibration curve using 0 to 50 mg/L potassium hydrogen phthalate (aq.)
Injection Amount:	50 µL
Sample:	Wastewater from Shimadzu facility
TOC Dopant:	Phenol (Special grade)

■ Measurement Results

Table 3 shows the results of measurements for wastewater with no phenol added and for samples prepared by adding phenol. Satisfactory results, close to 100 %, were obtained for TOC recovery in all samples. Fig. 2 shows the correlation graph of the added concentration and the measured concentration. A strong correlation is observed (R coefficient of 0.9999). Fig. 3 shows the calibration curve data for the measurements, and Figs. 4 (a) to (g) show the measurement data for each sample.

These results confirm that the TOC-L can rapidly measure phenol-containing samples without pretreatment. When many samples must be analyzed, the ASI-L autosampler can be used to automatically measure multiple samples, improving laboratory efficiency.

Although this study used the TOC-L as a laboratory analyzer, the TOC-4200 is suitable for continuous wastewater monitoring. Both the TOC-L and TOC-4200 share the same basic measurement principle and many common components, so the TOC-4200 can provide results consistent with the TOC-L. However, because TOC measures the total organic carbon, the measured value reflects not only phenol but also other organic substances present in the sample. When phenol-specific quantification is required, TOC monitoring should be combined with a confirmation method, such as the 4-AAP method.

Table 3 Summary of Measurement Results

Sample	TOC Measurement Value (mg/L)	TOC Recovery Rate (%)
1	1.691	-
2	4.017	101.2
3	5.551	100.8
4	9.444	101.2
5	13.21	100.3
6	20.98	100.8
7	40.58	101.6

$$\text{TOC recovery rate (\%)} = \frac{\text{TOC value after subtracting the wastewater-only TOC}}{\text{Theoretical TOC concentration of added phenol (Phenol concentration} \times 0.7657^{*1})}$$

*1 Carbon mass ratio (content) in phenol

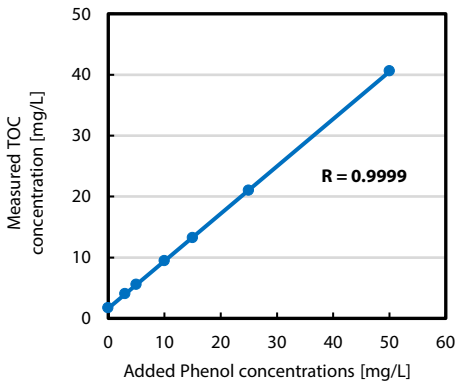


Fig. 2 Correlation Graph of Added Concentration and Measured Concentration

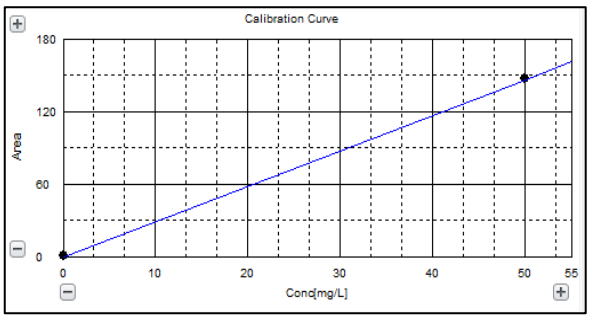
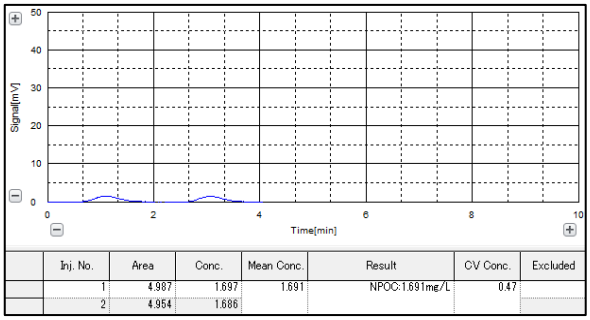
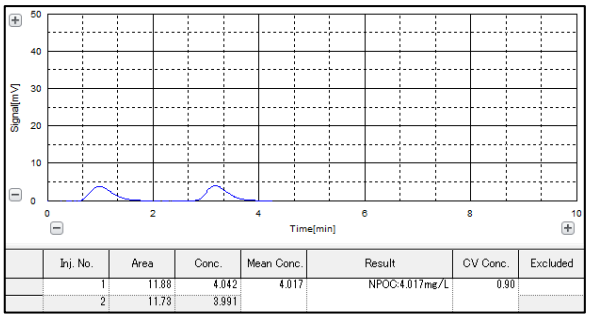


Fig. 3 Calibration Curve Data



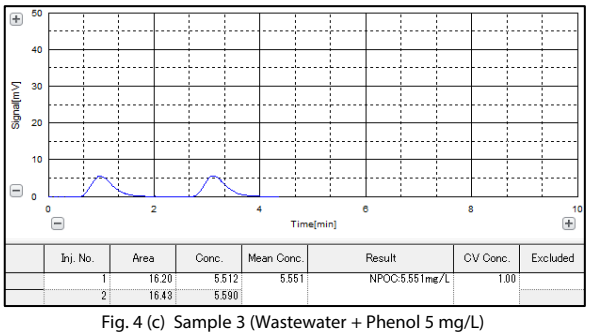
Inj. No.	Area	Conc.	Mean Conc.	Result	CV Conc.	Excluded
1	4.987	1.697	1.691	NPOC:1.691mg/L	0.47	
2	4.954	1.686				

Fig. 4 (a) Sample 1 (Additive-Free Wastewater)



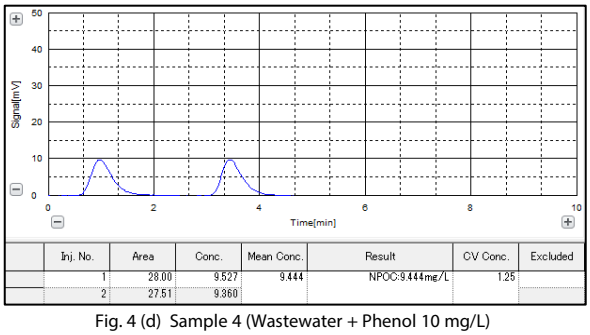
Inj. No.	Area	Conc.	Mean Conc.	Result	CV Conc.	Excluded
1	11.88	4.042	4.017	NPOC:4.017mg/L	0.90	
2	11.73	3.991				

Fig. 4 (b) Sample 2 (Wastewater + Phenol 3 mg/L)



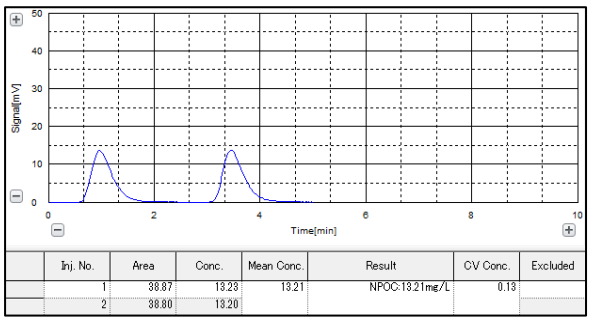
Inj. No.	Area	Conc.	Mean Conc.	Result	CV Conc.	Excluded
1	16.20	5.512	5.551	NPOC:5.551mg/L	1.00	
2	16.43	5.590				

Fig. 4 (c) Sample 3 (Wastewater + Phenol 5 mg/L)



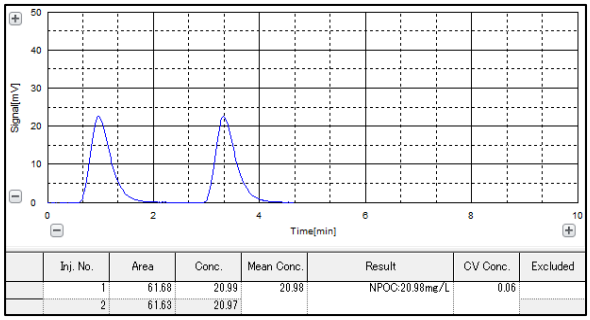
Inj. No.	Area	Conc.	Mean Conc.	Result	CV Conc.	Excluded
1	28.00	9.527	9.444	NPOC:9.444mg/L	1.25	
2	27.51	9.360				

Fig. 4 (d) Sample 4 (Wastewater + Phenol 10 mg/L)



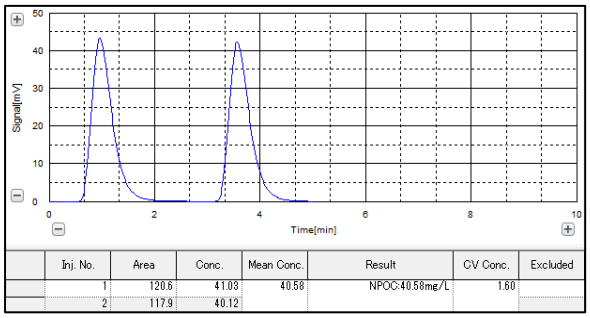
Inj. No.	Area	Conc.	Mean Conc.	Result	CV Conc.	Excluded
1	38.87	13.23	13.21	NPOC:13.21mg/L	0.13	
2	38.80	13.20				

Fig. 4 (e) Sample 5 (Wastewater + Phenol 15 mg/L)



Inj. No.	Area	Conc.	Mean Conc.	Result	CV Conc.	Excluded
1	61.68	20.99	20.98	NPOC:20.98mg/L	0.06	
2	61.63	20.97				

Fig. 4 (f) Sample 6 (Wastewater + Phenol 25 mg/L)



Inj. No.	Area	Conc.	Mean Conc.	Result	CV Conc.	Excluded
1	120.6	41.03	40.58	NPOC:40.58mg/L	1.60	
2	117.9	40.12				

Fig. 4 (g) Sample 7 (Wastewater + Phenol 50 mg/L)

■ Conclusion

Shimadzu TOC analyzers enable rapid measurement of phenol-containing samples without pretreatment. The TOC-L laboratory TOC analyzer is useful for wastewater management and for quality control of process water and cleaning processes. When handling multiple samples, the ASI-L autosampler supports fully automatic, high-throughput measurement.

In addition, the TOC-4200 on-line TOC analyzer enables continuous measurement of samples at a cycle time of less than 10 minutes, allowing continuous monitoring of wastewater.

TOC analyzers can measure the total amount of carbon in samples and can also be used to manage all types of organic compounds, not limited to phenol. Just one TOC analyzer is sufficient to conduct water-quality management with confidence, including wastewater management.

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