

## Measurement of TOC in Copper (II) Sulfate Solution and Nickel Sulfamate Solution using TOC-V<sub>CSH</sub>

Copper sulfate solution and nickel sulfamate solution are generally used as plating solutions for the copper and nickel plating processes in the manufacture of printed circuit boards. Organic additives are added to these plating solutions, but since the additive itself affects plating quality, it is important to control its concentration to ensure the quality of the printed circuit board. In this respect, the TOC analyzer can be used to manage the concentration of the organic additive.

As the salt concentrations in the copper sulfate solution and nickel sulfamate solution can amount to several percent or more, this salt can be expected to interfere with direct analysis with the combustion type TOC analyzer. Accordingly, dilution of the solution is

required to prevent this interference. When conducting analysis of plating solution using an combustion type TOC analyzer, the solution must be diluted by a factor of more than 25 in consideration of the salt concentration. However, since the Shimadzu combustion type TOC analyzer incorporates an automatic dilution capability, TOC analysis can be conducted by utilizing this function to dilute high-salt concentration samples automatically in the TOC analyzer.

Examples of the analysis of copper sulfate and nickel sulfamate simulated plating solutions using the Shimadzu TOC-V<sub>CSH</sub> Oxidative Combustion Type TOC Analyzer is presented here.

### ■ Measurement of TOC in Copper ( II ) Sulfate Solution

For the simulated sample plating solution, a 160 g/L copper sulfate ( II ) pentahydrate + 140 g/L sulfuric acid solution was prepared, and 50 mg C/L (carbon concentration of 50 mg/L) potassium hydrogen phthalate was added as the TOC constituent. This was diluted by a factor of 25 using the automatic dilution function, and the results are shown in Fig.1. In addition, the instrument was calibrated using a 50 mg C/L potassium hydrogen phthalate standard solution that was automatically diluted by a factor of 25, and a calibration curve was generated. The measurement results are shown in Table 1, clearly indicating that the added TOC constituent was accurately measured.

#### <Measurement Conditions>

Analyzer : Shimadzu TOC-V<sub>CSH</sub> Combustion Type TOC Analyzer  
 Measurement item : TOC (TOC by acidification and sparging method)  
 Dilution Factor : 25  
 Sample : (1) 160 g/L copper sulfate ( II ) pentahydrate + 140 g/L sulfuric acid solution  
 (2) Solution consisting of the solution (1) above with 50 mg C/L potassium hydrogen phthalate added

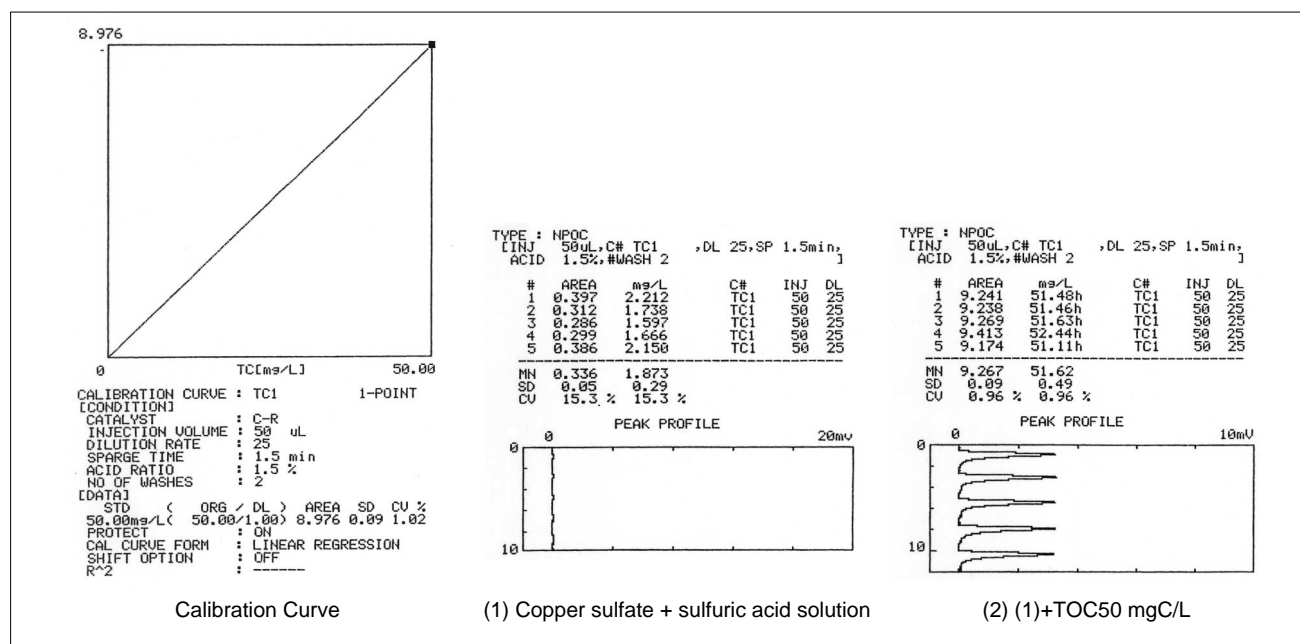


Fig.1 Measurement of TOC in Copper ( II ) Sulfate solution

Table 1 Measurement of TOC in Copper( II ) Sulfate solution

Sample Name	TOC Value [mg C/L]
(1) 160 g/L copper sulfate ( II ) pentahydrate + 140 g/L sulfuric acid solution	1.87
(2) Solution (1) above + 50 mg C/L potassium hydrogen phthalate	51.6

### ■ Measurement of TOC in Nickel Sulfamate Solution

As the simulated sample plating solution, a 200 g/L nickel sulfamate solution was prepared, and 50 mg C/L (carbon concentration of 50 mg/L) potassium hydrogen phthalate was added as the TOC constituent. This was diluted by a factor of 25 using the automatic dilution function, and the results are shown in Fig.2.

The measurement results are shown in Table 2, clearly indicating that the added TOC constituent was accurately measured.

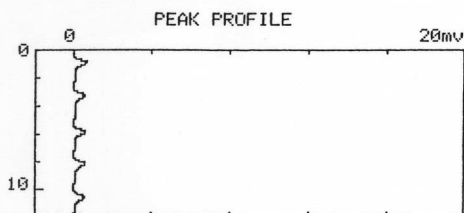
#### <Measurement Conditions>

Analyzer : Shimadzu TOC-V<sub>CSH</sub> Combustion  
Type TOC Analyzer  
Measurement item : TOC (TOC by acidification and sparging method)  
Dilution Factor : 25  
Sample : (1) 200 g/L nickel sulfamate solution (Nickel Amidosulfate ( II ) Tetrahydrate) solution  
(2) Solution consisting of the solution (1) above with 50 mg C/L potassium hydrogen phthalate added

```
TYPE : NPOC
[INJ 150uL,C# TC3 ,DL 25,SP 1.5min,
ACID 1.5%,#WASH 2 ]

# AREA mg/L C# INJ DL
1 2.066 3.684 TC3 150 25
2 1.991 3.551 TC3 150 25
3 1.937 3.454 TC3 150 25
4 1.941 3.462 TC3 150 25
5 1.775 3.166 TC3 150 25

MN 1.942 3.463
SD 0.11 0.19
CV 5.50 % 5.50 %
```

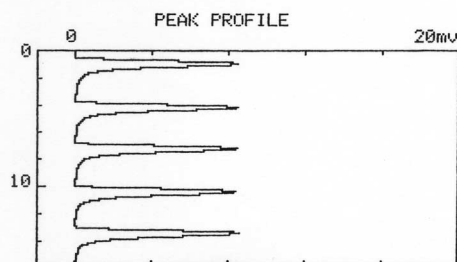


(1) Nickel sulfamate solution

```
TYPE : NPOC
[INJ 150uL,C# TC3 ,DL 25,SP 1.5min,
ACID 1.5%,#WASH 2 ]

# AREA mg/L C# INJ DL
1 30.34 54.11h TC3 150 25
2 30.61 54.59h TC3 150 25
3 30.42 54.25h TC3 150 25
4 30.21 53.88h TC3 150 25
5 30.16 53.79h TC3 150 25

MN 30.35 54.12
SD 0.18 0.32
CV 0.59 % 0.59 %
```



(2) (1)+TOC50 mgC/L

Fig.2 Measurement of TOC in Nickel Sulfamate solution

Table 2 Measurement of TOC in Nickel Sulfamate solution

Sample Name	TOC Value [mgC/L]
(1) 200 g/L nickel sulfamate solution	3.46
(2) Solution (1) above + 50 mgC/L potassium hydrogen phthalate	54.1

#### NOTES:

\*This Application News has been produced and edited using information that was available when the data was acquired for each article. This Application News is subject to revision without prior notice.



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