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TOC·TN APPLICATION NEWS No. 3

TOC Measuring of Etching Solutions for Etching Process

By using the TOC-5000A

Etching solution is reused for precision processing of CRT shadow masks and the etching process of patterns of printed circuit boards. However, etching solution deteriorates if organic matter concentration increases due to impurities and chemicals (surface-active agent etc.), and etching speed also slows down. In a situation like this, Shimadzu's total organic carbon analyzer TOC-5000A monitors the concentration of organic matter in the etching solution, so that changes can be made to restore operation efficiency.

Purpose

To check organic impurity level in collected etching soliltion.

Problems of Conventional Method

Conventionally, NC analyzers and gas chromatography were used for analysis, but the high concentration of hydrochloric acid and iron chloride in etching solution effected the durability of such devices, and also the maintenance of consumables such as absorbents was a burden

Measuring Conditions

Before measuring the etching solution first conduct an recovery test using a etching solution for recovery test to confirm whether or not measuring of organic matter concentration is possible without effecting the hydrochloric acid and iron chloride which are coexisting components in etching solution.

<Recovery test>

Sample (1) : Prepare and adjust etching solution for recovery test, and measure TOC.

Sample (2) : Add an organic matter (potassium hydrogen phthalate) to sample(1) to measure the TOC. Calculation is made to obtain the organic matter concentration as thc difference between sample (2) and (1).

Analyzer: TOC-5000A

Analytes: TC, IC, TOC (TC-IC)

Measuring Method: Dilute samples (1) and (2) 20 times, and measure TC-IC.

<Etching Solution Measuring>

TOC is measured before and after the etching process.

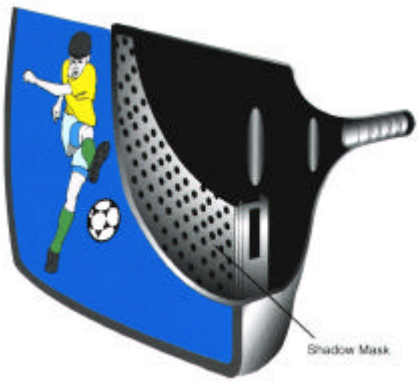
Sample I: Before etching process

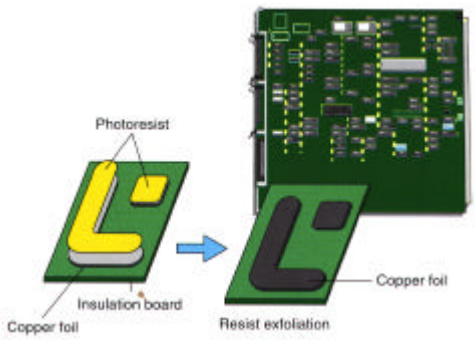
Sample II: After etching process

Analyzer: TOC-5000A

Analytes: TC, IC, TOC (TC-TC)

Measuring Method: Dilute samples I and II 20 times, and measure TC-IC.





Results

Measurement results of the recovery test are shown in Table 1.

Samples	TC value (ppmC)	IC value (ppmC)	TOC (TC-IC) value (ppmC)
(1): 54% iron chloride ($FeCl_2$,FCl_3) + 5% hydrochloric acid	32.04	1.284	30.75
(2): sample I + 100 ppmC potassium Hydrogen phthalate	136.9	1.308	135.5

Table 1: Recovery test

recovery ratio (%) = (solution (2) TOC value - solution (1) TOC value [ppmcj) / added TOC value [ppmc]

= (135.5 - 30.75)/100

= 105%

Since approximately 100% of the additive is recovered, there is no effect on the hydrochloric acid and iron chloride which are coexisting components in the etching solution, so the organic matter concentration can be accurately measured.

Next, the measuring results of the etching solution collected in a etching process are shown in Table 2.

Samples	TC value (PPMc)	Ic VALUE ((ppmC)	TOC (TC-IC) value (ppmC)
I: before etching process	38.7	0.20	38.5
II: after etching process	53.1	0.44	52.7

Note: The table values are shown after blank and diluting compensation to results shown Fig. 5 to Fig 8.

Table 2: Measured values of the etching solution

In this way the change in organic matter concentration before and after the etching process can be accurately obtained.

Benefits

The following benefits are obtained with the TOC-5000A.

1. In organic matter control in etching solutions, all organic matter, including unexpected ones, is quickly and accurately measured.
2. Operation is easy due to simple measuring principle and setup.
3. Fast measurement allows a multitude of sample measurement.
4. Maintenance is easy.
5. Consumables such as absorbents are not needed.

Complaints such as "UP to now there was no easy way to control this work", or "control was time consuming and expensive" are fixed by thc TOC analyzer, which makes the control easy.

Application in Other Fields

Here are some TOC-5000A application examples.

1. TOC measuring of electroplating solutions.
2. TOC measuring of rinse water.
3. Measurement of organic components in glass fiber for printed circuit boards.
4. TOC measuring of oil remained on the electrical components

Shimadzu would be glad to give further advice on the suitable application of this device.

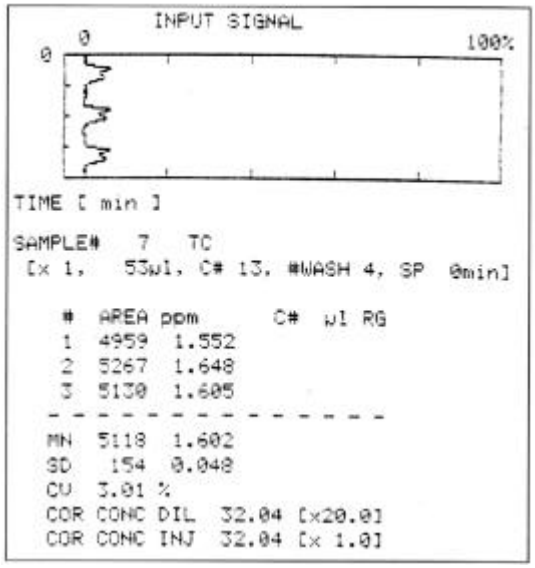


Fig. 1: TC Measurement for Sample(1)

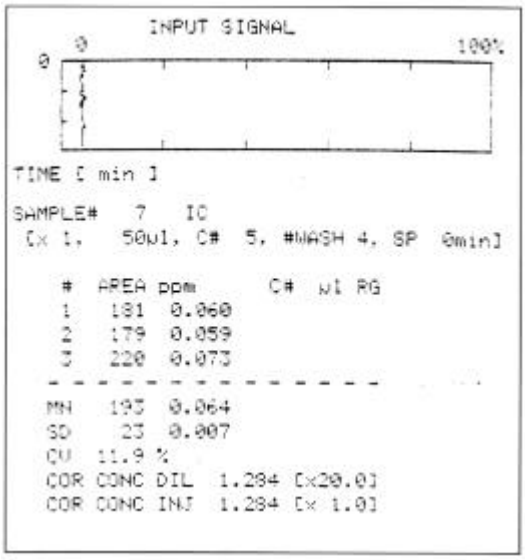


Fig. 2: IC Measurement for Sample(1)

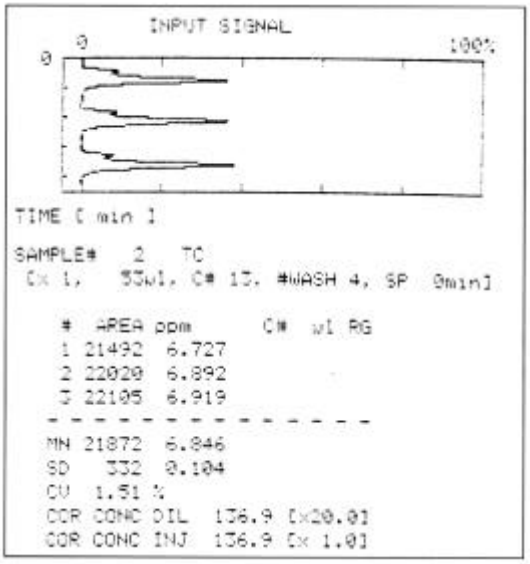


Fig. 3: TC Measurement for Sample(2)

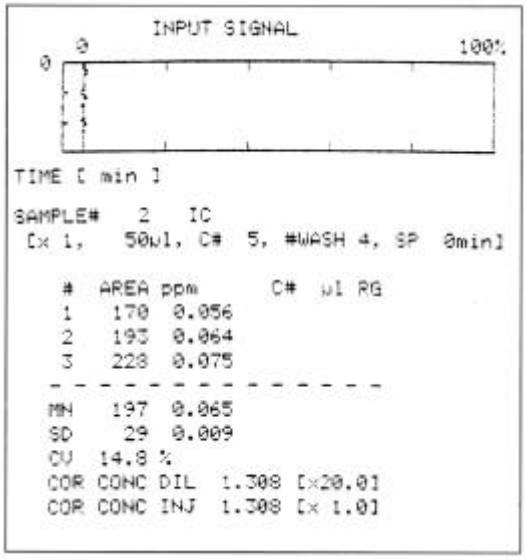


Fig. 4: IC Measurement for Sample(2)

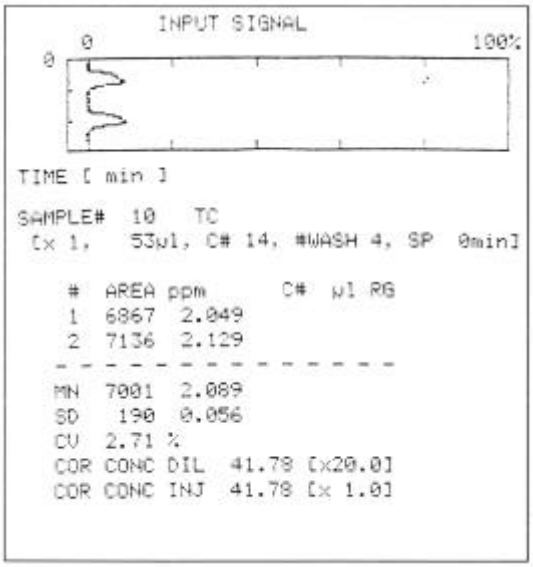


Fig. 5: TC Measurement fot Sample I

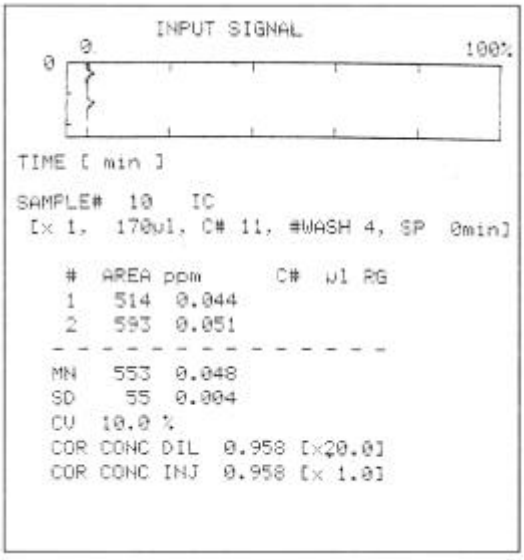


Fig. 6: IC Measurement for Sample I

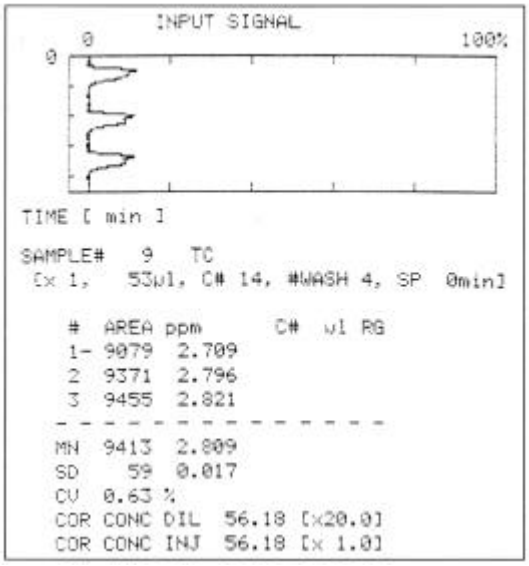


Fig. 7: TC Measurement for Sample II

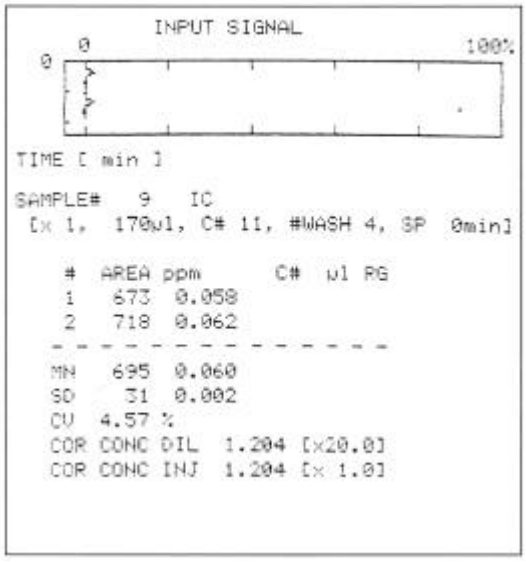
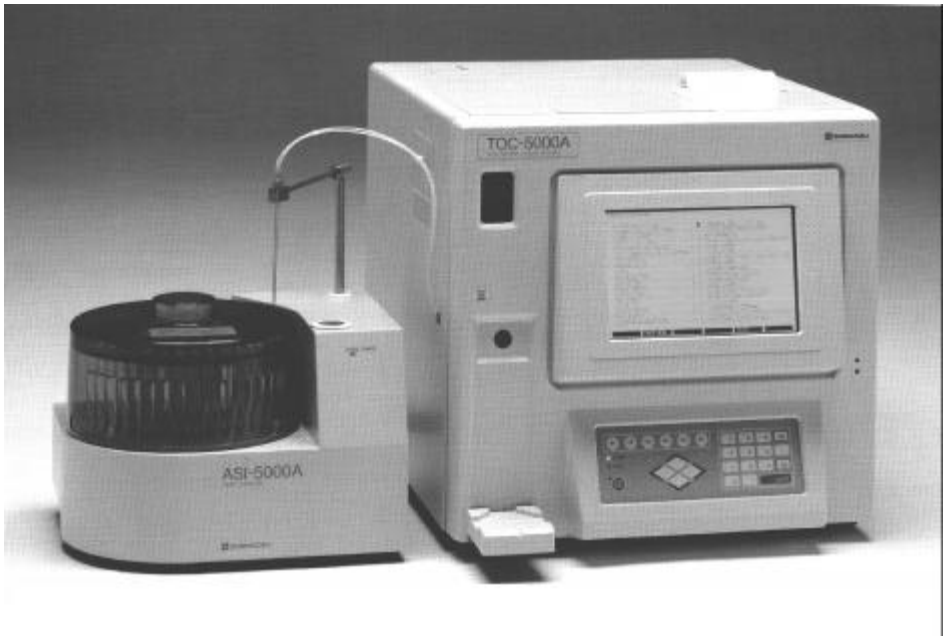


Fig. 8: IC Measurement for Sample II



Shimadsu's Total Organic Carbon Analyzer

TOC-5000A Fully Automatic Measuring System