



# Determination of Calcium Carbonate in Secondary Cement Products using the TOC-5000A Solid Sample TOC Analysis System

Cement, whose best known representative is concrete, is a material essential for today's modern buildings with many kinds and types. It is anticipated that it will be further diversified in the future. It is well known that because cement is alkaline and contains large amounts of calcium, it slowly reacts with the carbon dioxide in the air to produce calcium carbonate, causing its characteristics to deteriorate (neutralization of concrete). Since the formation of calcium carbonate is an important factor affecting the durability of cement products, accurate determination of the amount of calcium carbonate is necessary in researches to improve the quality of cement products (e.g. development of surface treatment methods preventing carbonation). Here we present an example of the Shimadzu TOC-5000A Solid Sample TOC Analysis System

# **Purpose**

Using the Shimadzu TOC-5000A Solid Sample TOC Analysis System to measure the amount of calcium carbonate in secondary cement products.

## Comparison with Conventional Methods

being put to use for this purpose.

The evaluation of the amount of calcium carbonate present in secondary cement products is generally carried out by the methods described below, but the following problems are associated with these procedures:

- Dissolving in hydrogen chloride / Titration (JIS method)
   Produces accurate results, but time consuming and laborious.
- X-ray analysis of calcium carbonate
   Not a very quantitative method. Sample preparation is time consuming and laborious.
- Thermal analysis
   Measurement is time consuming and results are not very
   quantitative. Only small amount of samples can be analyzed
   and the results can be influenced by uneven distribution of
   calcium carbonate.
- Ignition loss method
   Same problems as associated with thermal analysis.

# Measuring Conditions

Sample: Broken up cement type board

Used analyzer: TOC-5000A Solid Sample TOC Analysis System

(TOC-5000A + SSM-5000A)

Items to be measured: IC (inorganic carbon) and TC (total carbon).

TOC (total organic carbon), which is TC - IC,

can also be obtained.



## Results

The measurement results are shown in Table 1, and peak data in Fig. 1.

Table 1

	IC value [ppmC]	TC value [%C]
Sample 1	2428	3.303
Sample 2	9215	4.035

### Benefits

The amount of calcium carbonate in the cement was determined simply and accurately.

- Rapid analysis
  - Measurements were performed at approximately 5 6 minutes per sample\*.
- Large amount of sample can be measured.
  - Analysis is possible for up to 1g of sample, and 30mg of carbon content\*, minimizing the effect of unevenly distributed calcium carbonate within the sample.
- Highly quantitative measurement
  - Amounts of carbon within the range of 0.1  $30 \text{mg}^*$  can be accurately measured.
- Easy operation
  - The sample is simply placed on the sample boat and inserted into the device. Unlike manual analyses, deviations between measurement data obtained by different people are minimized.
- Consistency with JIS method
  - The IC measurement of the Shimadzu TOC-5000A Solid Sample TOC Analysis System, which acidifies the sample and determine the amount of carbon dioxide produced, is based on the same principle with the JIS method\*\*, which involves dissolving in hydrochloric acid and titration.

Note: \* Varies according to the characteristics and shape of the sample

\*\* Method whereby sample is acidified, and generated carbon dioxide is absorbed into absorber liquid and measured by titration.

## Application in other Fields

- Determination of calcium carbonate in concrete used for buildings and bridges. (Evaluation of concrete neutralization.)
- Determination of carbon in inorganic compounds such as ceramics.

# Features of the Solid Sample Analysis System

TOC measurements of many types of solid sample, such as soil, sludge and sediment, as well as aqueous samples, are possible. Carbon measurement in adhered residues by swab sampling method for cleaning validation under the revised GMP is also possible.

# **■Sample 1** INPUT SIGNAL 199% IC measurement SAMPLE# 1 IC [X30, C# 1, DENSITY 1.0mg/pl] # AREA ppm AMNT(mg) 1 21452 2495 31.20 2 20758 2372 30.60 3 22010 2507 30.70 MN 21406 2428 30.83 SD 70.47 CU 2.90% INPUT SIGNAL **■Sample 1** 100% TC measurement TIME [ min ] PLE# 1 TC 1, C# 13, DENSITY 1.0mg/bl] # AREA % ANNT(mg) 1 21525 3.299 30.20 2 21269 3.292 93.90 3 21854 3.316 30.50 MN 21549 3.303 30.20 SD 0.012 CU 0.37% **■Sample 2** 100% IC measurement 9215 7.133 161.6 1.75% **■Sample 2** TC measurement E# 5 TC , C# 13, DENSITY 1.0mg/ul) AREA % ANNT(mg) 26283 4.015 30.30 26220 4.072 29.80 26124 4.017 30.10 26209 4.035 30.06

Fig. 1

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