DETERMINATION OF SURFACE CARBON

Figure 1: Model CM190 Total/Organic Surface Carbon Analyzer

PRINCIPLES OF OPERATION AND TYPICAL USE

The amount of surface carbon on steel has been shown to have a direct effect on how well the steel will accept diverse types of coatings. Also, the measurement of surface carbon on several types of materials is used to determine cleanliness. This procedure is used to measure the amount of surface carbon on a variety of materials. The instrument and method described allows for the differentiation of organic and inorganic surface carbon. This method has been performed successfully on coupons of cold-rolled steel, zinc plated steel, stainless steel, aluminum, titanium, and surface mount components.

The system is composed of a CM5380 Two Zone Horizontal Combustion Furnace and a CM5017 Carbon Analyzer. The sample is first placed in the cool zone of the combustion tube. Then, the system is given time to purge itself of any atmospheric CO_2 that may have entered when inserting the sample. After purging, the sample is moved into the first zone of the furnace with a manipulator rod or hook ladle. This zone is set at a temperature of 460°C and all organic surface carbon on the sample is oxidized to CO_2. The gases then go through the second zone heated to 590°C where any incomplete combustion products are "burned" and converted to CO_2. After this, potentially interfering gases are removed by a series of post-scrubbers. The carrier gas is then introduced into the Carbon Analyzer where the carbon dioxide is detected, measured quantitatively, and displayed as µg of carbon.

Once all the organic carbon is liberated the sample can then be analyzed for inorganic surface carbon if desired. This is achieved by moving the sample into the second heat zone. Here, the inorganic surface carbon is oxidized to CO_2. The gases then go through the same series of post-scrubbers as before. Again, the CO_2 is detected and measured quantitatively by the Carbon Analyzer.

PROCEDURE

ASSEMBLY

Assemble the Carbon Analyzer and Dual Zone Furnace as instructed in each of their respective manuals (see Figure 1 for an example of the finished set up).

NOTES: The system is designed to accommodate either 15 mm or 25 mm combustion tubes. The 15-mm tube allows for coupons of up to 0.4-inch width to be introduced into the furnace. Generally, the coupons used are 4 inches long but coupons up to 8 inches long have been used successfully. The 25-mm tube allows for coupons of up to 0.75 inches wide to be introduced into the furnace. The same length limitations of the smaller width coupon are true for the larger coupons.

Most samples are introduced using either a manipulator rod or hook ladle, both equipped with a steel basket at the end. Hook ladle introduction only allows for the measurement of organic or total surface carbon. There are a variety of sample introduction methods for analyzing surface carbon. For technical assistance contact UIC Europe.

ANALYSIS

Sample Preparation and Handling

Metal coupons are usually cut into appropriate sizes with either a die (ideally) or with shears. No matter what type of samples are being analyzed it is recommended that they be stored in plain manila style envelopes prior to analysis. If possible, the samples can also be stored in a clean, closed glass container such as a desiccator. **Plastic bags are not recommended** for storage as they can cause contamination and high results.

Samples should be always handled with clean forceps to avoid contamination. Also, it is recommended that samples be analyzed as soon as possible after cutting or cleaning. Every time the samples are exposed to the air, they can become contaminated with amounts of carbon measurable by this technique.

Blank Determination

Once the system is assembled and the combustion tube conditioned, sample analyses can begin. Set up the Carbon Analyzer's coulometric cell as instructed in the manual. Connect the Furnace to the Analyzer. The flow rate is usually set to 100 ml / minute (up to 200 ml / minute has been used successfully). Insert the manipulator rod into the breech block or the ladle into the "cool zone" of the combustion tube and seal the system. Allow the system to purge itself of any atmospheric carbon dioxide that may have entered when the system was opened. The purge time will depend on the size of combustion tube being used and the carrier gas flow rate. It is usually 1 to 5 minutes. Determine the background rate of the system by entering "blank" as the sample name in the Sample Entry screen of the CM5017 Coulometer. Press "Begin Analysis" and then insert the sample introduction device being used into the first heating zone (at this point, there are no samples in the furnace). The CM5017 will automatically determine and store the blank value according to the user selectable settings saved within the system's memory (if inorganic surface carbon is going to be analyzed, slide the manipulator rod into the second heating zone half way through the blank determination). The instrument will use the saved blank value in calculating the end result values.

Calibration

Although calibration is not required, the instrument's performance can be checked using a sucrose standard (benzoic acid and dioctyl sebacate have also been used). If solid sucrose is to be used, first determine the background rate of the system using a scoop ladle and a small platinum boat. After determining the background rate with this type of sample introduction, weigh an appropriate amount of sucrose into the platinum boat and analyze.

A more ideal instrument performance check would be to take a sample that has been "burned in" and spike the sample with a known quantity of sucrose solution ("Burned in" means that the sample was previously analyzed and then kept clean). Then analyze this sample and calculate recovery. The user should establish acceptance criteria for whichever type of performance check is used.

Sample Analyses

At the end of the blank run remove the rod or ladle from the furnace. If a ladle is being used slide it into the cool zone of the furnace and leave it there for a few moments to cool. Then, remove it from the combustion tube and place it on a clean heat-resistant surface (a glass plate or beaker is often used). If a manipulator rod is being used, unscrew the end of the breech block holding the rod and pull the rod out of the combustion tube without sliding it through the Teflon ferrules that seal around it. Sit this assembly on the lab bench to cool in such a way that it will not become contaminated. Often, it is hung from the edge of the lab bench by the breech block plug using a ring stand and clamp.

Once cool, place a sample on the sampling device and insert the sample into the cool zone of the combustion tube. Seal the system and allow for the purge time. Start the Carbon Analyzer and insert the sample into the first zone of the furnace. The analysis endpoint will be automatically determined by the CM5017 according to the user selectable settings saved within the instrument. Individual coulometer readings and end results are saved on the USB Flash Drive and/or printed to an attached printer, at the end of the analysis session.

If the determination of inorganic surface carbon is desired, start the Carbon Analyzer again and move the sample into the second zone of the furnace. Again, the CM5017 will automatically determine the endpoint and save/print the results.

NOTE: It is important that each time a sample is analyzed it is placed into each heating zone at the same place. This helps insure consistent combustion temperatures.

RESULTS

The sensitivity of the Carbon Analyzer is better than 1 µg C. Overall accuracy is limited by the reproducibility of successive background measurements. Using standard materials, deviations are typically better than 0.15% relative or ± 1 to 2 µg of C (whichever is greater). Sample homogeneity normally limits the accuracy of the analysis rather than the instrument's capability.

NOTE: This technique is appropriate only for sample materials which do not contain compositional carbon that will react at the analysis temperature.

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