Challenge
Automated solution for reliable and reproducible determination of carbon traces in process intermediates of foam glass production

Solution
Combustion elemental analysis – efficient high temperature combustion coupled with highly sensitive NDIR detection for carbon

Intended audience
Foam glass production, industrial quality control, contract labs dealing with analysis of building materials like glass

Determination of Total Carbon Content in Foam Glass and Related Samples by Combustion Elemental Analysis Coupled with NDIR Detection

Introduction

Due to its excellent thermal and acoustic insulation properties, low weight, and very high mechanical stability, foam glass, also called cellular glass, is a popular insulation material for buildings. It is obtained from recycled glass, and particulate carbon. In addition to these main components, other aggregates, including dolomite, feldspar, lime, and quartz sand, are used for its production. In the first production step, the recycled glass and mineral aggregates are used to produce a glass melt, which, after cooling, is ground into a fine powder using industrial-size ball mills. In a second step, small amounts of carbon black (soot) are added to the glass powder. This mixture is then melted again. At 1,000 °C the black carbon is converted into gaseous CO_2, which cannot escape due to the high viscosity of the molten glass. The melt thus becomes a foam which retains its porous structure even after cooling. Depending on the desired shape and utilization of the foam glass, the glass/soot mix is melted either inside casting molds to form blocks and other precisely defined shapes, or just like it is in a tunnel kiln to form foam glass granulate or rubble.
Since the carbon black content is essential for the formation of the CO_2 bubbles and thus for the properties and quality of the resulting insulating material, exact metering and precise knowledge of its concentration in the glass/carbon black mix is crucial and therefore an important test parameter for process control and quality assurance.
In addition to TOC analyzers, which are mainly used for the determination of carbon sum parameters in the field of environmental analysis of water and soil, element-specific, combustion-based elemental analyzers have become established for the determination of carbon contents. The multi EA 4000 is such an elemental analyzer, designed for rapid determination of total carbon contents (TC) over a wide concentration range in mixed inorganic/organic sample matrices, like they are needed in the production of foam glass.

Materials and Methods

A combustion elemental analyzer type multi EA 4000 was used for the determination of total carbon contents. The analyzer is based on the high-temperature combustion principle for sample digestion, using a robust ceramic combustion tube. The multi EA 4000 is an open system, the combustion furnace is arranged in horizontal orientation. Samples are fed via a simple gas lock. This allows easy operation and automation of the analysis process. For the here described measurements a solids sampler type FPG 48 was used to transfer the samples, which have been filled in ceramic boats, into the furnace and to remove these boats with residue after analysis for fully disposal.
The total carbon (TC) contents have been determined by combustion at 1,060 °C in a pure oxygen stream. Therefor approximately 300 mg of the samples and standards were weighed into ceramic boats. The standards were analyzed directly. For the samples an additional step for sample preparation was necessary. Therefor 10% H_3PO_4 was added dropwise to the sample boats until the entire surface of the samples was humidified by the acid. This was done to remove the CO_2 absorbed from the environment air. Before analysis the samples must be dried completely at temperatures up to 130 °C. Afterwards the dried samples were combusted at 1,060 °C in a pure oxygen atmosphere. The formed reaction gas is cleaned and dried sufficiently by integrated systems before it is transferred to the NDIR (non-dispersive infrared) detector for carbon determination.

Samples and reagents

- Different glass/soot mixtures
- Glass standard I - 0.273% C (performance check)
- Glass standard II - 0.317% C (calibration and performance check)
- H_3PO_4, 10% (acidification of the samples)

Sample preparation

The glass/soot mixtures contain small particles with a big surface. On this surface, gases like carbon dioxide (CO_2) from ambient air are easily adsorbed. For a correct analysis of the carbon content of the samples, this adsorbed, additional CO_2 must be removed to avoid too high false analysis results. Therefor diluted phosphoric acid (H_3PO_4, 10%) is added to the samples. To remove the excess of H_3PO_4 the samples were dried at 130 °C until complete dryness.

Calibration

The analyzer has been calibrated before analysis. The applied calibration principle is constant concentration. A solid standard material, with carbon concentrations of 0.317% C, was used for calibration, see details in table 1. To cover a wide concentration range, different quantities of this standards have been used to vary the absolute element content. The resulting calibration curve is shown in figure 1. Correctness of calibration was checked with different standards.

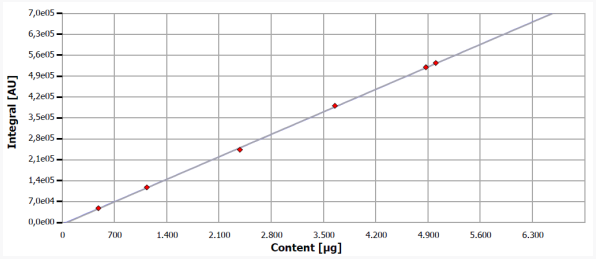

Figure 1: TC calibration with glass standard II - 0.317% C

Table 1: Calibration of the instrument

Standard	Parameter	Concentration	Weight	Calibration range
Glass standard II	TC	0.317% C	170–1,600 mg	0.54–5.01 mg C

Method parameters

The used process parameters for sample introduction and combustion are summarized in table 2.

Evaluation parameters

The used parameters for detection and evaluation are summarized in table 3.

Table 2: Process parameters multi EA 4000 and FPG 48

Parameter	Specification
Temperature	1,060 °C
Oxygen flow	2.5 L/min
FPG 48 parameter set	TC_inorg

Table 3: Parameters for carbon detection (NDIR)

Parameter	Specification C detection
Max. integration time	600 s
Start	0.12 ppm
Stop	5 ppm
Block	3

Results and Discussion

Different glass/soot samples and the certified glass standards have been analyzed for their carbon contents. For analysis quantities around 300 mg were used for both, standard and samples. The gained measurement results are summarized in table 4. They are average values of triplicate analysis.

The results are reproducible, and the standard deviations are very low. Slight deviations from the reference values are to be expected and the differences are in the familiar range. The small differences in the carbon contents of the three samples are tiny variations between different production batches.

Measurement results are available in short time. For simultaneous determination, even for higher contents, 2 to 3 minutes are sufficient for a total carbon analysis.

To depict this, exemplarily selected measuring curves for the carbon determination in sample “glass/soot mix 1” and “glass standard II 0.317% C” are shown in figures 2 and 3.

Table 4: Result of determination of total carbon (TC) in glass/soot mixes and reference materials

Sample	TC ± SD [%]
Glass/soot mix 1	0.275 ± 0.005
Glass/soot mix 2	0.281 ± 0.002
Glass/soot mix 3	0.279 ± 0.005
Glass standard I (0.273% C)	0.272 ± 0.006
Glass standard II (0.317% C)	0.320 ± 0.002

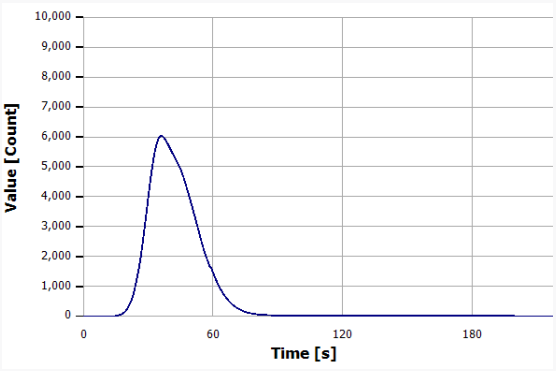

Figure 2: TC measuring curve of sample “glass/soot mix 1”

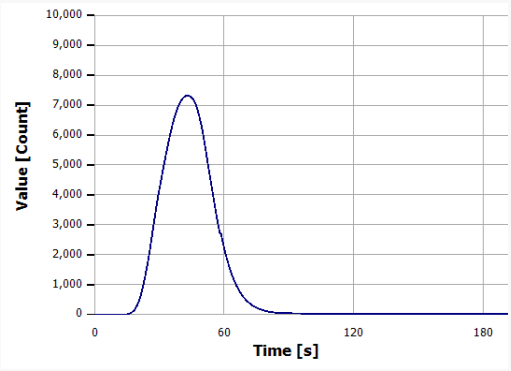

Figure 3: TC measuring curve of “glass standard II 0.317% C”

Summary

The used multi EA 4000 is well suited for the quick and precise determination of smallest carbon contents as required for the analysis of materials from foam glass production. In routine applications the solids autosampler with its 48 positions and boat deposition station enables high sample throughput at minimum operation effort. For small sample numbers manual sample supply is possible as an alternative. Another advantage of the analyzer is its matrix flexibility, which allows to measure other matrices which are used for foam glass production for their carbon contents. The system can easily be upgraded for sulfur analysis or chlorine analysis in pure organic matter.

Figure 4: multi EA 4000 C analyzer

Recommended device configuration

Table 5: Overview of devices and accessories

Article	Article number	Description
multi EA 4000 C	450-126.564	Combustion elemental analyzer for determination of carbon contents in solids
FPG 48	450-126.574	Autosampler for solids for multi EA 4000
Boat deposition station	450-889.728	Accessory for FPG 48, for the automated disposal of used sample boats

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