Sample preparation for coal analysis

Dr Tanja Hanke, Retsch GmbH, Germany, and Dr Andreas Theisen, Eltra GmbH, Germany discuss the influence that sample preparation has on elemental analysis results when testing the quality of coal.

The most common types of coal (lignite, bituminous and anthracite) are distinguished by their different chemical and physical properties. The calorific value of coal can be determined by analysing its carbon content, for example by using combustion analysers. In addition, efficient management of the desulfurisation plant requires control of the sulfur content. Compared to the large amount of coal a typical laboratory sample consists of – a 10 l bucket or even more – the sample volume required for analysis is fairly low, only approximately 1 g. The German industry standard (DIN 51701) defines the sample amount to be tested, as this relates to the particle size: the bigger the particles are, the more sample material is required.

However, a small amount needs to represent a large quantity. Therefore, an important step to take before analysing coal is sample preparation with laboratory crushers and mills to obtain a representative sample. For more inhomogeneous coal samples, such as brown coal, the sample preparation step becomes even more important in ensuring reproducible analysis results. The fineness of the sample can influence the standard deviation of the analysis results, as will be shown in this article using brown coal as an example. In general, using a sample divider to produce a representative part sample and a final grind size of the sample of 100 μm help to obtain results with high reproducibility.

Table 1. Standard deviations of measurement results		
Particle size	Measured carbon content* (%)	Measured sulfur content* (%)
8 mm	63.77 +/- 1.21	0.0478 +/- 0.047
100 µm	64.61 +/- 0.50	0.0450 +/- 0.004
* 10 measurements of each sample were carried out in an ELTRA CS-580 analyser.		

Original sample of brown coal

The sample crushed in a jaw crusher has a particle size of approximately 8 mm..

After the crushing process, the coal sample was pulverised in the ultra-centrifugal mill ZM 200 to a fineness of 100 µm.

Jaw crusher (BB 300).

Sample preparation of coal

Pre-crushing with jaw crushers

The first step in the sample preparation process is pre-crushing the coal with jaw crushers. Retsch´s jaw crushers are available in four different sizes, accepting feed sizes from 35 mm – 130 mm, as well as a variety of materials. Depending on the jaw crusher model, the sample material and the chosen gap width, a final fineness of 0.5 – 5 mm can be obtained. The BB 300 jaw crusher can be used to prepare batches of sample material or run continuously if larger sample amounts are processed.

In a trial, brown coal was pre-crushed and ground before elemental analysis to demonstrate the influence of sample preparation on the analytical results. 4 kg of brown coal (pieces of approximately 130 mm) were pre-crushed with the BB 300. Crushing obtained particle sizes of <8 mm, which were suitable for elemental analysis using for example, Eltra's CS-580 combustion analyser.

Fine grinding

If the particle size of an inhomogeneous material is too large, the analytical results may show considerable standard deviations. These can be significantly reduced by pulverising the sample to a particle size below 100 µm – for example with Retsch's high speed rotor mill ZM 200. This mill processes samples very quickly thanks to a rotor speed of up to 18,000 rpm. The sample only remains in the grinding chamber for a short time, which means that the characteristic properties of the sample are not altered. It is possible to process sample volumes up to 5 l.

The brown coal sample, obtained by pre-crushing with the BB 300, is divided into representative fractions using the Retsch sample divider PT 100. After that, one fraction is used for fine grinding in the ZM 200, which is equipped with a 12-teeth rotor and a 0.2 mm ring sieve. The 100 g sample is milled to a final fineness of 100 µm (d90 value) in 30 sec. This 100 µm sample is analysed using the Eltra elemental analyser CS-580 and the results are compared to those of the 8 mm sample with regard to the standard deviations (Table 1).

From the results obtained, it can be clearly seen that fine size reduction significantly lowers the statistical error of the results, because the homogeneity of the sample is improved. Deviations in carbon content were reduced from ±1.21% to only ±0.5% and from ±0.047% to ±0.004%, regarding sulfur content.

Summary

The quality control of coal involves a variety of applications, such as producing a representative sample by crushing, dividing and fine grinding before elemental analysis. Correct preparation and homogenisation of the sample is an indispensible prerequisite in obtaining reproducible and reliable analysis results with a minimum standard deviation.

Analysis of carbon and sulfur

Following correct sample preparation, elemental analysers can determine the carbon and sulfur content of materials. They are also able to determine the hydrogen content for coals with lower calorific value. Combustion analysers, such as the CS-580, can be used to reliably analyse carbon, hydrogen and sulfur concentrations in solid materials. A variety of instruments are available to analyse any possible combination of elements.

For carbon and sulfur analysis, a coal sample can be weighed in a sample carrier (e.g. a ceramic boat). This carrier, loaded with the sample can then be introduced into a hot furnace, which operates at around 1350°C, in an oxygen stream. The released combustion gases (CO_2, SO_2) are measured with sensitive infrared cells. It is possible to adapt the infrared cells to the user's individual requirements in regards to the required measuring range. WC