Application Note
Energy and Chemicals

Confirmation of Coal Quality Using an Agilent Handheld FTIR Spectrometer

Quality of coal was determined based on silicate level and hydrocarbon content.

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Introduction

Coal is a fundamental commodity in the global economy. It is a complex substance with an overall composition that is locale dependent. The coalification process is affected by geological and mineralogical conditions, as is coal quality and impurity profiles. For this reason a variety of chemical and physical tests are employed in the industry to define the quality and composition of coal.

The value of coal is linked to the specific application for which it is being used, and these applications range from its simple ability to provide heat to its use as a starting material for the synthesis of chemicals. The price of coal for a specific application is directly related to its quality, and therefore verifying its composition is essential. Coal customers require assurance that the quality of coal they are receiving is as expected, and that the coal has not been intentionally adulterated with less expensive coal or other materials. For example, there have been instances in certain parts of the world where black rock or asphalt pieces have been added to coal shipments to increase the weight.

In this application note, we show how an Agilent 4100 ExoScan handheld FTIR*, equipped with diffuse reflectance sampling technology, can discriminate between coal of acceptable quality and coal that is intentionally adulterated with lower quality material. The portability of this handheld FTIR system enables these measurements to be made in real-time, in the field.

Value of handheld FTIR for the analysis of coal

Though FTIR spectroscopy has been used for years in coal research and analysis, it is only recently that this powerful technique has been made available for use in non-laboratory environments. Advantages of portable FTIR for the analysis of coal include:

- Coal can be rapidly analyzed in the field and at shipment or receiving sites.
- Adulterated, impure or otherwise out-of-specification material can be detected in real time and decisions can be made on the spot regarding acceptance or rejection.
- Identity and quality of a coal shipment can be ascertained and payment based on objective parameters can be made.
- Water content as well as inorganic and organic components of coal can be measured, giving an accurate picture of the coal quality.
- Extent of the coalification process in a locale can be ascertained providing real time metrics for exploration and/or mining activities.

Figure 1a. Agilent 4300 FTIR system in handhelp operation for at-site use. When used for at-site analysis of coal, all instrument functions are executed using the attached PDA. An automated, on-board method for the verification of coal quality is initiated by simple touch screen commands and results are clearly displayed on the PDA.

The Agilent 4300 handheld FTIR (Figures 1a and 1b) is well-suited to the analysis of coal in either lab or field applications. The battery powered system is designed for either handheld, mobile use, or as an effective bench top FTIR spectrometer for laboratory applications.

Figure 1b. Agilent 4300 FTIR system being used in the field.

Though a number of different sampling technologies are available for the 4300 instrument, the high efficiency diffuse reflectance sampling interface is the preferred technology for coal measurements. The system has the required performance and ruggedness, providing answers in demanding environments, and software that is designed for ease of use and to provide answers to non-expert users who may not be familiar with FTIR.

Experimental

Fifteen samples of coal, sourced from Eastern Europe, were measured using a diffuse reflectance sampling interface. Each of the coal samples was ground or crushed to a granular (2-3 mm) state to improve uniformity. The coal was measured neat, with each sample spectrum acquired in less than 3 minutes (330 co-added interferograms recorded at 16 cm^{-1} resolution). Some of the coal samples were known to be mixed with lower quality coal from a different locale. The objective of the work was to determine if the FTIR measurement could differentiate the higher quality, higher value coal from that mined from known low quality coal producing regions.

The 4100 ExoScan model has been discontinued. Similar results can be achieved with current Agilent handheld FTIR models.

Results and discussion

The amount of silicate minerals in the coal samples is the primary differentiator for coal acceptability. An IR absorbance band at 1216 cm^{-1} (Figure 2) originates from the feldspar silicate inclusions in the coal samples. This band is not observed in coal known to be sourced from high quality coal producing regions. Therefore, the amount of the silicate band arising from feldspar is greater in coal that is reported to be of lower quality, or sourced from poor coal producing regions.

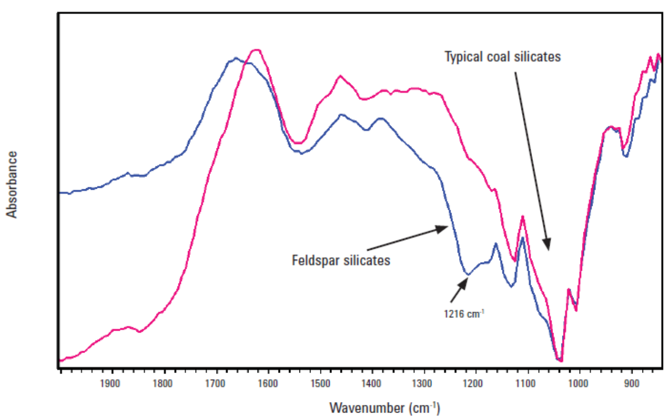

Figure 2. The IR spectrum of lower quality coal (blue) contrasted with that of a higher quality coal (red). The feldspar type silicate absorbance at 1216cm^{-1} is observed in the lower quality coal samples

A peak height ratio measures this 1216 cm^{-1} feldspar band relative to the other silicate absorbance bands in the region.

Lower quality coal (i.e., samples 12–14) exhibits substantially higher absorbance ratios (Figure 3) than the better performing coal. For these coal samples, this measurement is a primary indicator of coal quality, since it is free of false positives for the higher quality coal and false negatives for those samples described as lower quality.

In addition to the coal quality index using the feldspar ratio, the diffuse reflectance spectra of the coal samples also exhibit differences in the amounts of aliphatic CH stretch in the “hydrocarbon” region of the IR spectra (Figure 4). Hydrocarbons in coal represent the primary class of organic compounds that will actually burn and contribute to the calorific or energy content of the coal. The hydrocarbon IR region has interference from hydroxyl (OH) absorbance originating from other components in the coal and therefore is difficult to measure quantitatively with a single peak area or peak height measurement. For this reason, the hydrocarbon bands at 3000-2800 cm^{-1} are measured in peak areas and the ratio using a local total peak area from 3350-2800 cm^{-1} is calculated. This peak area ratio technique effectively eliminates the interference from the hydroxyl absorbance.

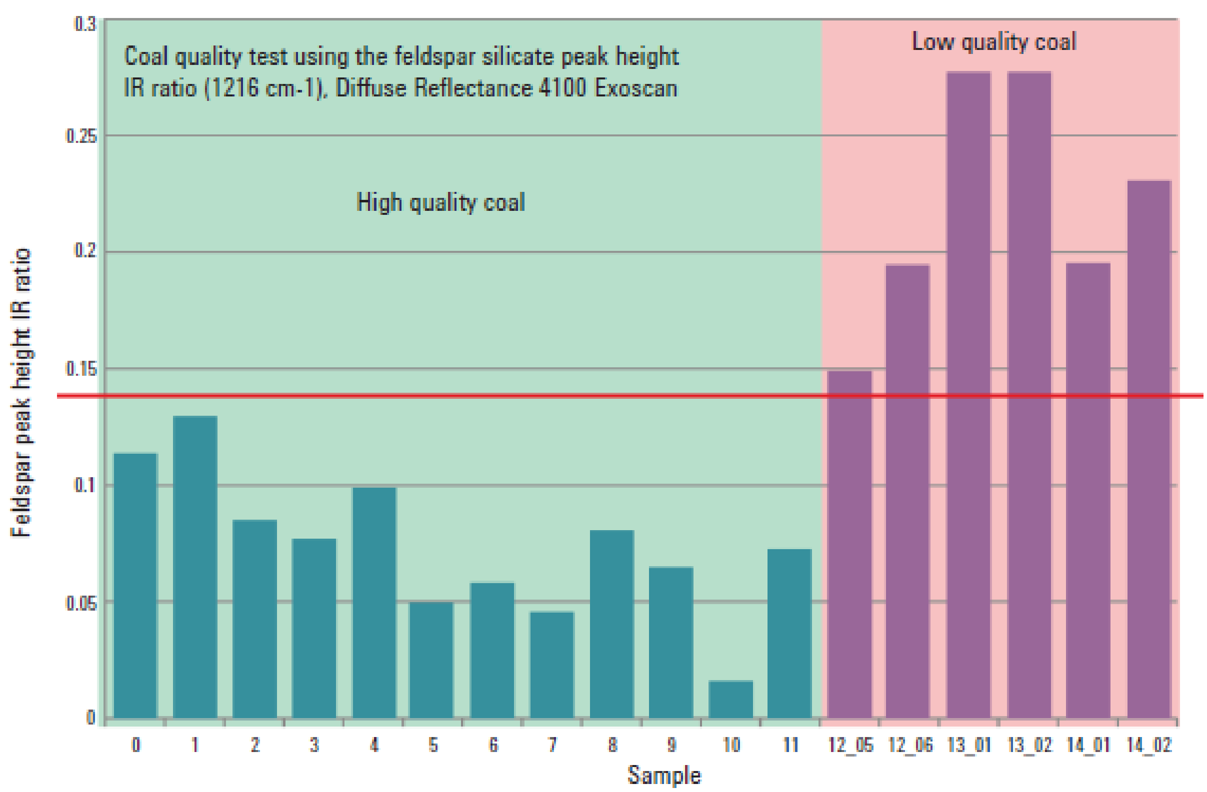

Figure 3. The IR ratio used for the “Coal Quality” measurement indicates the relative amount of additional silicates (i.e. feldspar) and quartz containing minerals in coal. Samples 12-14 have been identified as lower quality coal. The red line indicates the threshold for low coal quality, with no false positive or false negative results. Samples 0-11 are the averages of 2-3 measurements.

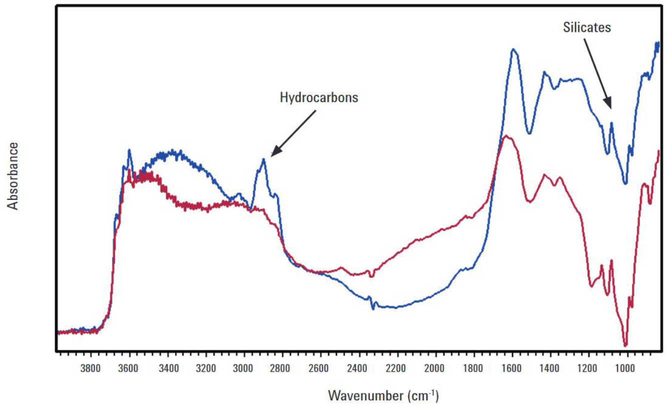

Figure 4. IR Spectra of coal samples indicating high hydrocarbon absorbance bands (blue) contrasted to a sample with lower hydrocarbon bands (red).

By setting a threshold indicative of acceptable quality coal, the peak area hydrocarbon ratio is used to determine the quality of coal samples (Figure 5, red line).

Samples 12-14 have received complaints from customers regarding the poor quality of the coal and the hydrocarbon levels are clearly below the threshold. Some samples, such as 4 and 11, are from mines known for high quality coal, but have lower hydrocarbon content. As was stated earlier, coal is heterogeneous; it is expected that samples coming from different portions of the mine will have various amounts of hydrocarbons. For example, samples 0–10 are from the main cut of the mine while sample 11 came from a side cut. This shows the usefulness of direct hydrocarbon measurement to further discriminate coal quality within a single mine. Premium quality coal could be differentiated

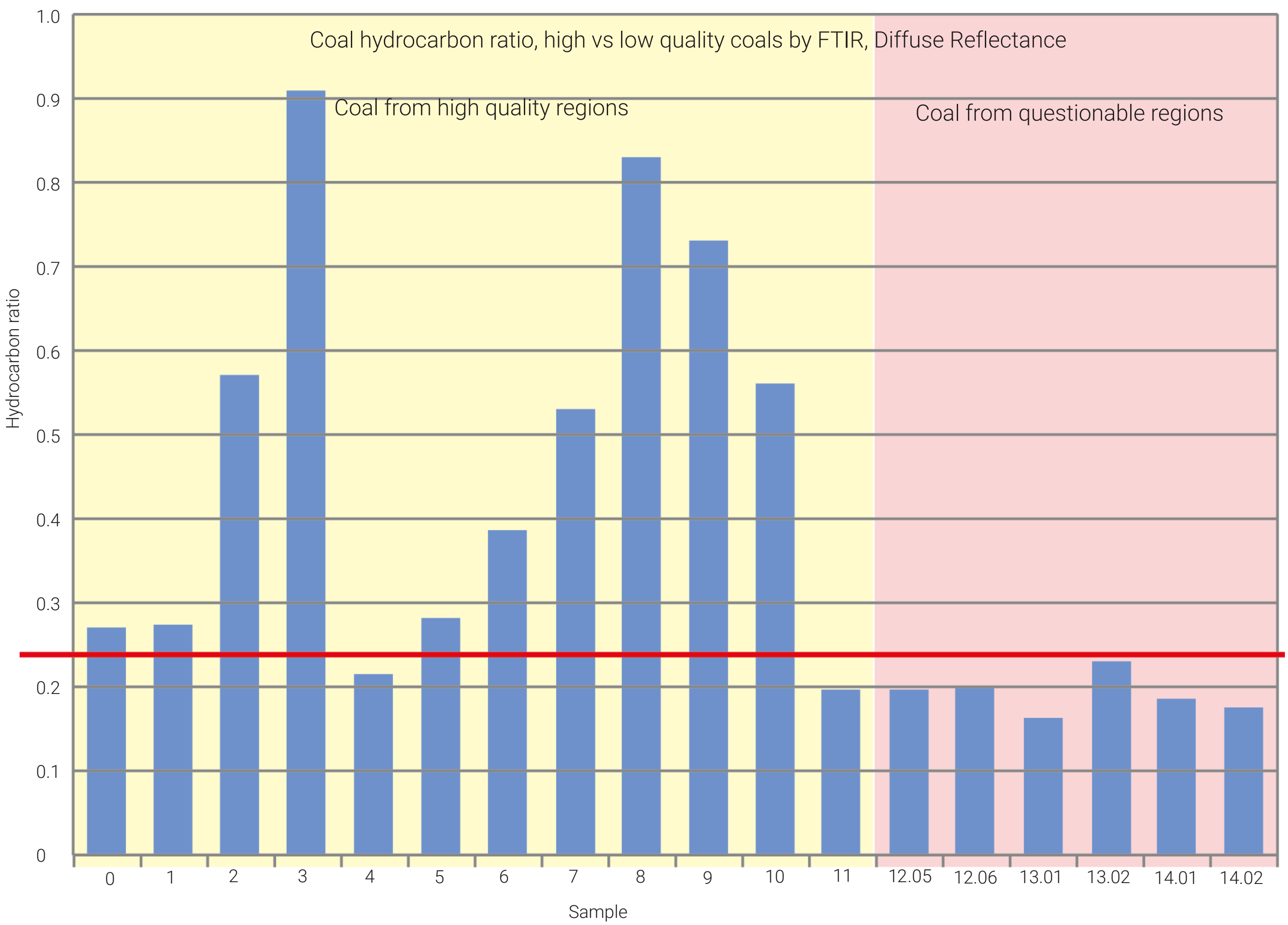

Figure 5. The IR ratio method provides the relative amount of hydrocarbons in coal. Samples 4 and 11-14 have been identified as low hydrocarbon content coal. Samples 0-11 are the averages of 2-3 measurements.

from standard coal using the hydrocarbon values from the same mine or region. Samples such as 0, 1, and 5 contain borderline low hydrocarbons.

The hydrocarbons measurement has also been modeled with a PLS classification (0–1 scale) method, which incorporates the hydrocarbon IR peak ratio. The hydrocarbon ratio measurement is included for additional comparison between coal samples, but is not the primary determination parameter. The feldspar IR ratio has also been scaled from 0–1, one indicating high amounts of feldspar contamination and zero indicating the ideal quality of coal. Thresholds are set, and colored alerts are displayed using the standard Agilent MicroLab software to indicate the quality of the sample. For example, the red alert for sample 13 (Figure 6) indicates high feldspar silicate and low hydrocarbon components. The yellow alert for sample 1 (Figure 7) indicates that the coal is of marginal quality, but is still acceptable. If the feldspar silicate component is at or below the threshold, and the hydrocarbon component is above its threshold, the coal is considered to be higher quality, as is the case for sample 10 (Figure 8).

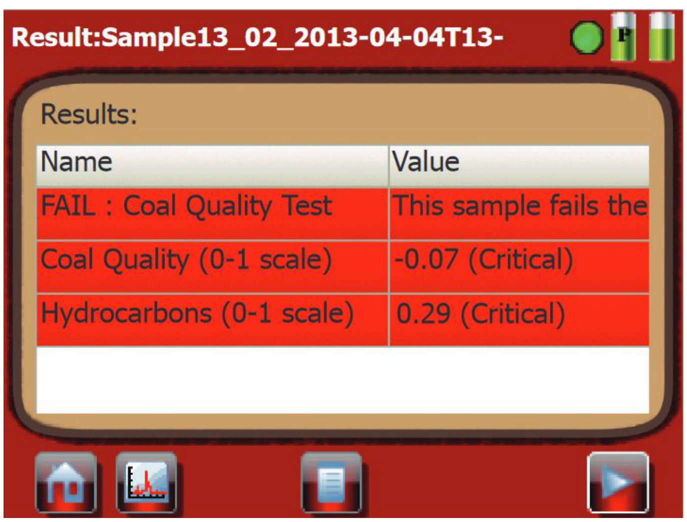

Figure 6: The coal quality method indicates the failure of sample 13 for both the feldspar ratio "Coal Quality" and aliphatic HC content tests (Red – unacceptable quality).

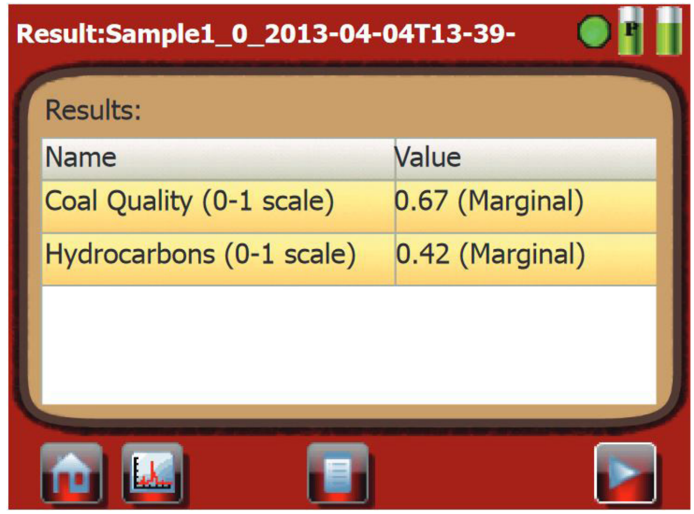

Figure 7: The coal quality method indicates that sample 1 marginally passes the feldspar ratio "Coal Quality" and hydrocarbon content tests (Yellow – marginally acceptable).

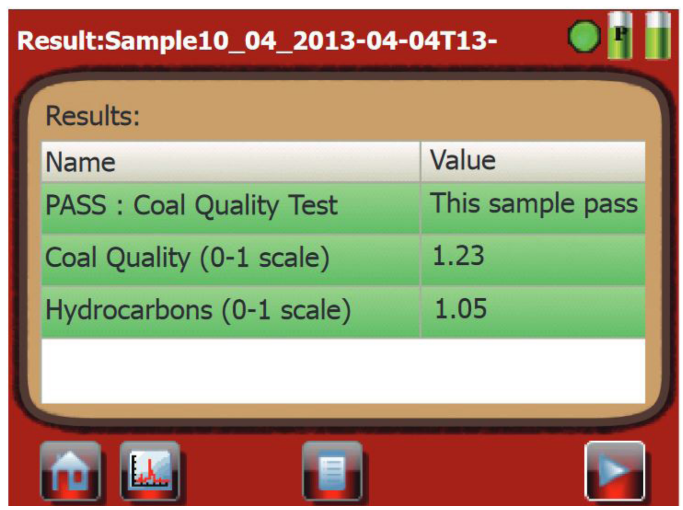

Figure 8: The coal quality method indicates that sample 10 fully passes the feldspar ratio "Coal Quality" and hydrocarbon content tests (Green – acceptable).

Conclusion

An Agilent handheld FTIR system equipped with diffuse reflectance sample interface was successfully employed to measure the quality of Eastern European coal and to determine if the coal had been adulterated or contaminated with lower quality material. A method was developed which clearly differentiates quality of this particular coal, based primarily on silicate level and secondarily on hydrocarbon content. The method provides users with colored alerts to quickly and accurately indicate the quality of the coal sample.

Additional methods can be created depending on the specific requirements of other coal producing locations. In all cases, the handheld FTIR enables quality control personnel to move the measurement from the laboratory to the field (coal production, shipment or receiving sites) and make timely and informed decisions regarding the quality of the coal. For this reason, handheld FTIR systems offer potential—and significant—economic benefit to coal producers and their customers.

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