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ACARP MATTERS

TOP: Retort used in the CSR Test.
BOTTOM: Ken Riley being congratulated on his Standards Award by Barry Isherwood (Chairman, Standards Australia Committee MN/1).

Barry Isherwood, Manager, Coal Technology, Xstrata Coal and industry monitor for these ACARP research projects said: "The carbon content of coal will be of ever increasing significance into the future, particularly with the introduction of carbon emission trading to address climate change issues".

"So the need for accurate and precise analysis of carbon in coal is most crucial," he said.

"The three ACARP-funded projects have inputted in a very significant way, into the development of viable ISO Standards, relevant not only to international coal trade, but also important environmental concerns"

Research project leader Ken Riley from the CSIRO said there had been a worldwide use of inappropriate analytical methods to measure trace elements and many of these methods lacked the required accuracy and sensitivities to demonstrate the advantages that Australian export coals possessed.

COAL TESTING DOES MATTER!!! TRACE ELEMENTS IN COAL – INTERNATIONAL STANDARD

International coal trade has always been negotiated around certain quality parameters including Total Moisture, Ash, Volatile Matter, Total Sulfur and Calorific Value (thermal coals) to name but a few; these parameters are mostly determined by laboratory tests defined by the International Standards Organization (ISO). More recently, parameters such as trace elements and carbon content have been recognised as increasingly important in the utilisation of thermal coals for power generation.

The Australian Coal Association via ACARP, has successfully funded a number of research projects, relating to the determination of carbon in coal, trace elements and coke quality, the results of which support Australia's arguments for either the need for new standard procedures or for relevant changes to existing standards.

Australian representatives at ISO meetings have consequently been able to present research data generated from these projects and thus make compelling cases for inclusion or change.

TRACE ELEMENTS IN COAL – INTERNATIONAL STANDARD

Trace elements are present in all coals at very low levels certainly less than 0.1 percent and some at levels of less than 0.0001 percent. Some of these trace elements are environmentally-sensitive and may be released into the environment during the utilisation of coal. These include elements such as arsenic, selenium and mercury. Australian coals generally contain very low levels of these trace elements.

Initial work by CSIRO and ACARP in the area was very well received within Australia and had lead to the development of the existing Australian Standard.

"We decided to use this existing Australian Standard (AS), make appropriate changes to it and develop it internationally into an ISO standard that was robust and readily accepted by coal suppliers, consumers and laboratories worldwide," Mr Riley added.

As a result of this research project, a final draft international standard in the form of a guide has now been forwarded to ISO for consideration and for voting internationally, with publication expected next year. The guide does not prescribe the exact procedures that must be followed, but rather offers guidance to the selection of analytical methods.

INSTRUMENTAL DETERMINATION OF CARBON IN COAL

Traditionally, classical methods developed more than 150 years ago were used to analyse coal for its carbon, hydrogen and nitrogen contents. These methods originated from work conducted by the German chemist, Justus Liebig, who was not very impressed with the procedures and reputedly stated: "Ich glaube nur, dass sie unter den schlechten die am wenigsten schlechte ist (I only believe that among the bad choices, it is the least bad)."

With the development of new instrumental techniques, these time-consuming and labour intensive methods were eventually replaced. Unfortunately, the use of these rapid instrumental methods resulted in an apparent and unacceptable decrease in reproducibility (agreement of results between laboratories).

Research project leader on this study, Ken Riley said standard methods in Australia and other countries had lagged far behind industry practice in this area.

"To measure carbon, hydrogen and nitrogen (CHN) in coal, analytical chemists had started using modern instruments but there had been no corresponding development of appropriate standardised methods."

"With the need to now calculate greenhouse gas emissions and the possible introduction of carbon taxes, carbon content in coal is a particularly important parameter. Therefore accuracy and precision are required in its determination."

Part of the ACARP Project included a worldwide survey on the use of CHN instrumental analysers. The

“The aim of this ACARP-funded project therefore was to expedite the development and international acceptance of accurate and sensitive methods for the determination of trace elements in coal,” he said.

Adrian Reifenstein, Combustion and Carbonisation Manager, ACIRL and research project leader for this ACARP project said it was then decided that a common international standard was required for the Coke Reactivity Test.

“One of the major controllers of the price Australian producers receive for their coking coal is how well it performs as a coke and the Coke Reactivity Test happens to be the best measure of coke performance in a blast furnace,” Mr Reifenstein explained.

“Producers in some countries were getting better results than others for their coal, not because their coal was inherently better but because the parameters they were using were giving their coal a positive bias.”

researchers concluded that there was a strong need to “standardise” the approach taken to calibrate these instrumental analysers. There were a diversity of calibration or reference materials used and also differences in the manner in which they were being used.

A major outcome of the research was the development of the Australian Standard, AS 1038.6.4-2005, Higher rank coal and coke—Ultimate analysis—Carbon, hydrogen and nitrogen—Instrumental method.

This method was the first standard in the world, to require the use of “pure” organic compounds to calibrate CHN analysers. This approach has now been successfully adopted by ASTM (the American Society for Testing and Materials) in the United States and is the basis of the new ISO Standard.

Ken Riley, a pleased and recent recipient of the “Meritorious Contribution Award” from Standards Australia, for his activities in standardisation of analytical methods as well as for this ACARP-funded research work completed by CSIRO, said the procedure was expected to become an ISO standard by end of this year.

COKE REACTIVITY TEST; CRITICAL PARAMETERS

The Coke Reactivity Test was developed by Japanese steelmaker, Nippon Steel Corp in the 1970's in an attempt to get an indication of coke performance in a blast furnace. This test has two components- the CRI and the CSR component. The CRI component measures the reactivity of coke to Carbon Dioxide (CO_2) and the CSR component measures the strength of the coke. This test was recognised as a good indicator of blast furnace productivity and quickly became popular around the world.

However, a problem arose when American steelmakers deviated from the original test by using inches as a form of measurement instead of millimeters. Europeans followed their lead by adopting a hybrid version of testing. Eventually four or five different standards for the Coke Reactivity Test (CRI) came into existence, digressing slightly from the original Japanese test.

To bring some clarity to the test, there was a German initiative to develop an ISO Coke Reactivity Test. However, Australian producers were concerned that the new test would attempt to encompass all of the various national methods, creating potential to produce a broad range of outcomes for any given sample. A proposal was put to the ISO technical committee for coke to examine a set of variables that were possible sources of variation within the Coke Reactivity Test.

The aim of the research study was to check how these variables impacted the Coke Reactivity Test and thereby coke performance.

The variables included the following:

- The diameter of the retort,
- The time taken for the sample temperature to recover to 1100ºC after placement of a cold retort into the test furnace,
- The time interval between the retort re-attaining 1100ºC and change over to Carbon Dioxide (CO_2) gas flow (or soaking time),
- The temperature at which the retort is held; and,
- Screen sizes.
- The results of the study indicated that the diameter of the retort, furnace recovery response time and reaction temperature were the definite variables that impacted the Coke Reactivity Test.

However, no significant change was identified in the Coke Reactivity Test results as an effect of screen size changes. It was also noted that the change in soaking time influenced the CSR results but not the CRI results.

“We managed to develop a standard that controlled operating parameters that were important to control and allowed flexibility in those parameters which did not influence the test results,” Adrian Reifenstein explained.

“The repeatability and reproducibility of the test was proved and this went on to become a part of the ISO 18894 standard, published in April 2006. This test hasn’t gained universal acceptance yet, but we are certain that it is a step in the right direction.”

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