Coke-making and coke test-work using pilot scale and small scale ovens are commonly used, and are useful for quality control of production samples, exploration samples, blending programs, international export grading and for targeted research.

ALS Coal’s Coking Research Centre located has internationally recognised expertise and has provided small scale coke making, pilot scale coke making, coke testing and laboratory analysis facilities to Australian and International coal producers for over thirty years. The Centre has conducted in excess of 3000 pilot scale coke tests on a large number and range of coals from Australia and overseas. Test work is able to be undertaken on coal samples supplied from bore cores, operating mines, coal preparation plants and superintending samples.

Coking Coal Evaluation Programs

Comprehensive coal evaluation programs have been formulated to address most aspects of identifying and assessing a coal’s coking properties and expected utilisation behaviour. The program includes performing a comprehensive set of laboratory analyses, coke-making exercises and coke tests on the sample coal as well as a technical evaluation report to provide an interpretation of the results.

Laboratory Testwork

Laboratory analyses of the coal and coke are critical to understanding the underlying quality of the test coal. It is recommended for several of the tests that the laboratory tests are performed on both the coal and coke (proximate analysis, sulphur, and ash elemental analysis). This allows further validation of the quality of the coke product and allows further investigation of the results in the Technical Evaluation Report.

A typical laboratory analysis of a coking test coal would include determination of the following analyses:

- Proximate Analysis
- Ultimate Analysis
- Total Sulphur
- Chlorine
- Ash Elemental Analysis
- Phosphorus
- Gieseler Plastometry
- Audibert-Arnu Dilatometry
- Crucible Swelling Number
- Caking Index (G-Index)
- Sapozhnikov Plastometry
- Gray King Coke Type
- Petrographic Analysis

Coke-making

Coke-making is performed in duplicate in two small scale coke ovens or singularly in one of the two available pilot scale coke ovens. Pilot scale coke-making performed at the Coking Research Centre is completed according to Australian Standards (AS 2267-1997).

Small Scale Coke-making

The small scale coke oven is a bench scale coke oven test that is commonly used when there is not enough sample mass to perform a pilot scale test. The general procedure for small scale coke-making is:

1. The coal is crushed to pass a specific target.
2. The moisture of the coal is adjusted to a predetermined level.
3. The coal is packed to the target bulk density in a box.
4. The box is pushed into the small scale coke oven set at a predetermined temperature.
5. The charge is pushed 30 minutes after the centre of the charge has reached the set temperature.
6. The coke is quenched with water and dried in a fan forced oven.
7. Composite samples are prepared for further testing.

Small scale coke making is commonly used by clients as a quality check for superintending purposes to ensure the product quality does not deviate from the norm and in applications where sample mass is limited, for example, exploration programs. Small scale coke making can also be used to investigate bulk density and blending options. Small scale coke-making requires approximately 18kg of coal for duplicate analysis. From this quantity, modified Micum drum testing and NSC reactivity (CRI/CSR) testing can be performed.
**Pilot Scale Gravity Fed Coke-making**

Pilot scale coke-making is designed to simulate the coal to coke transformation as measured in a commercial byproduct battery oven. Both pilot scale ovens have a moveable wall that is fitted with a load cell to measure the force exerted on the wall by a coal during its transformation into coke. These measurements are used to determine if a coal is likely to have a damaging wall pressure. Measurement of the external gas pressure is also possible through the use of pressure probes that are inserted into the centre of the charge (along with thermocouples used to measure centre temperature). The procedure for pilot scale coke-making from a gravity fed coal is:

1. The coal is crushed to 80-85 percent passing a set size.
2. The moisture of the coal is adjusted to target.
3. The coal is gravity fed into a pilot-scale coke oven via roof charging port.
4. The temperature of the oven walls starts at the resting temperature.
5. The wall temperatures of the coke oven are ramped at a set rate.
6. Once the centre of the charge reaches the target temperature, the charge is soaked for one hour.
7. The charge is pushed and discharged into a quenching cart.

After the removal of coke from the oven, the coke undergoes several treatment processes. The first treatment stage is for the coke to be cooled by water quenching, followed by air drying. Stabilisation is then performed by dropping the coke from a predetermined height to simulate the breakage that occurs at a coke works. The coke is then sized and prepared for physical and chemical analysis, including mechanical strength (drum indices), reactivity (CRI), post-reaction strength (CSR) and laboratory analyses.

Pilot scale testwork is the preferred scale because a comprehensive set of coke tests (eg reactivity testing and all drum tests) can be performed. Pilot scale testwork is regularly performed by clients to monitor coke quality but usually not as frequently as small scale testing. Research programs frequently use pilot scale coke making and can be used to assess blending options, additives, investigate coking parameters such as bulk density, charge sizing and coking temperature profile. Pilot scale coke-making requires approximately 450kg of coal for analysis.

**ALTERNATIVES TO STANDARD PROGRAMS**

- Blending of multiple coal samples
- Blending of coals with additives
- Variations to charge top size
- Selective crushing of blend components
- Coal moisture content
- Variation of bulk density (box charge required)
- Stamp charging (box charge required)
- Variation to heating ramp rates
- Variation to soak times

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**Pilot Scale Box Charge Coke-making**

Pilot scale box charging is commonly used when there is insufficient sample to perform a gravity fed coke-making charge or when the bulk density must be controlled accurately. Box charging requires 100kg less coal than pilot scale coke-making (350kg of coal is required as compared to 450kg for gravity), which can be beneficial in exploration programs and blending programs where sample mass is scarce. Unlike small scale coke-making, box charge cokemaking can produce enough coke to perform a majority of the pilot scale drum tests including reactivity testing (CRI/CSR).

For box charges, the test coal is packed at a set bulk density into a prepared box made of cardboard and plywood. The box is then pushed into the pilot scale coke oven with a ram via a door. Wall pressure measurements and internal pressure measurements are reported for pilot scale box cokemaking, but due to the box interfering with direct coal to wall contact, wall pressure measurements can only be classified as an indicative value in reports.

**Pilot Scale Stamp Charge Coke-making**

Stamp charge coke-making is commonly used in density variation programs and blending programs where higher densities of coal are required. This type of coke-making is applicable to the Indian coking coal market, where stamp charging in industrial coke ovens is widespread. The stamp charging densities available range from 815 kg/m3 through to 1000 kg/m3. Stamp charging and blend stamp charging programs are commonly used to increase the buying potential of the coal for international markets as this procedure simulates commercial stamp charging cokemaking.

For stamp charging, the amount of coal required for the predetermined bulk density is packed into a prepared box made of cardboard and plywood. The box is then pushed into the pilot scale coke oven via the door (rather than fed via the roof). Like box-charge coke-making, wall pressure measurements are also indicative for stamp charging.

**Pilot Scale Twin-Box Coke-making**

Twin-box pilot scale charging is commonly used when there is insufficient sample to perform a single box or gravity fed coke charge. Twin box charges require 200kg per coal sample and is suitable for test programs with restrictions in sample mass, for example, exploration programs.

Twin-box coke-making is performed with the use of two samples being coked in one coke-making test. Twin-box coke-making uses similar techniques to those of the boxcharge coke-making method, where a predetermined bulk density (generally 815 kg/m3) is packed into a prepared box made of cardboard and plywood. For twin-box coking, the box is divided into two adjacent sections into which two separate samples are packed. The twin-sample box is then pushed into the pilot scale coke oven via the door similar to a single-box coke charge. Twin-box pilot scale coke making has the same temperature profiles and sample preparation methods as those of the single-box charge method.

Coke is discharged from the oven using a different method from a gravity fed coke charge. A specially designed discharge cage is used to hold the coke, pushed from the oven. The coke is quenched within the cage. The use of this cage, rather than the quenching tray, prevents sample cross-contamination and allows correct identification of the individual samples.

For both samples, internal pressure measurements are available and recorded independently. Wall pressure measurement profiles are recorded, but due to the box interfering with direct coal to wall contact, wall pressure measurements can only be classified as an indicative value. It is also known that one sample can create wall force thus creating a pressure reading; whereas the other sample may not. This creates a false reading of wall pressure for the second sample.

Like single box-charge coke-making, twin-box coke-making produces less coke than gravity fed coke-making. Twin-box coke charging generally only produces enough coke to perform reactivity testing (CRI/CSR) and 1-2 drum indices tests per sample.
Coke Testing

After coke-making the coke quality must be evaluated. Drum tests measure volume breakage and abrasion resistance. Drum testing is a common analysis used to determine and understand the cold strength of a coke sample and the influence that mechanical action has on the coke. NSC reactivity testing is designed to determine the Coke Reactivity Index (CRI) and Coke Strength after Reaction (CSR). The method simulates the changes in coke strength resulting from exposure of coke to a blast furnace environment. Coke test-work performed at the Coking Research Centre is completed according to Australian Standards (AS 1038.13 - 1990).

NSC Reactivity testing

When coke descends into the blast furnace, the coke is subjected to abrasion (coke rubbing together and against the walls) and a reaction with counter-current blast furnace gases containing carbon dioxide. This process physically alters and chemically reacts with the coke, creating fines due to weakening coke. The production of fines decreases burden permeability, resulting in higher required coke rates and reduced blast furnace hot metal productivity. NSC reactivity testing is designed to simulate the reactions that occur as the coke descends through the blast furnace and measures the cokes resistance to degradation. The method simulates the changes in coke strength resulting from exposure of coke to a blast furnace environment.

Coke Reactivity Index (CRI) represents the percentage of coke that reacts with carbon dioxide under specified conditions. Coke Strength after Reaction (CSR) represents the drum strength of coke that is weakened during the process.

For NSC Reactivity testing, -21mm +19mm coke is subjected to an atmosphere of carbon dioxide at 1100°C for 2 hours. After cooling, the coke is weighed, where the comparison of the sample weight before and after the reaction determines the Coke Reactivity Index (CRI). The coke sample is then tumbled (600 revolutions/30mins) and sized at 10mm. The Coke Strength after Reaction (CSR) value is determined by calculating the +10mm coke material, where the proportion of +10mm coke represents the CSR value.

Drum Index testing

Drum Indices performed by the Coking Research Centre are listed and described below:

ASTM TUMBLER TEST

ASTM drum testing was developed by the American Society for Testing and Materials D3402 (carried out according to AS1038.13) and is designed to determine a relative measure of the resistance of degradation of coke due to abrasion and impact. This procedure uses -75 + 50mm coke that undergoes 1400 revolutions. The coke is then sized (square aperture) at +25mm and +6.3mm to yield the stability factor and hardness factor respectively.

BS MICUM INDICES

BS Micum drum testing is from the British Standard BS1016, which is used to determine the strength of coke by mechanical action. Coke fractions of +60mm are used for this procedure, which undergoes 100 revolutions and is sized using round-hole screens. Coke is sized at +40mm denoted as $M_{40}$, and coke passing -10mm represents $M_{10}$.

IRSID INDICES

After completion of the determination of AS Micum indices, all coke fractions are returned to the drum for determination of IRSID indices. The IRSID drum test procedure is performed according to Australian Standard AS1038, and is used to determine the strength and impact resistance of coke by mechanical action. IRSID drum testing involves AS Micum tested coke fractions to be returned to the drum, where a further 400 revolutions is applied (giving the required total of 500 revolutions). The IRSID test requires the sizing process to be repeated using round-hole screens. Coke is sized at +40mm, 20mm and -10mm, each fraction representing $I_{40}$, $I_{20}$ and $I_{10}$ respectively.

JIS INDICES

JIS drum testing is performed according to Japanese Standards Association JIS K 2151 and AS1038.13. The test is used to determine abrasion resistance and assess the physical strength of the coke. JIS drum indices require the use of +50mm coke undergoing 30 revolutions and sized at +15mm using square-hole screen. The coke remaining on top of the screen (+15mm) represents $D_{150}$. The coke is then returned to the drum and undergoes a further 120 revolutions (150 revolutions in total) and sized at +15mm. The coke remaining on top of the screen (+15mm) represents $D_{150}^{150}$.

MODIFIED MICUM DRUM INDICES

Modified Micum drum testing determines the strength and impact resistance of coke by mechanical action for small scale coke oven samples and carried out according to an inhouse method. Coke sized at +40mm is used for this procedure, which undergoes 100 revolutions in a specially designed drum and sized using round-hole screens. Coke is sized at +30mm representing $M_{30}$, and coke passing -10mm represents $M_{10}$. Modified Micum drum testing is the only drum testing available for samples coked in the small scale oven.
Report

The Coking Research Centre currently offers two types of report for documenting coke testing results, a standard report or a Technical Evaluation Report.

Standard Report

A standard report includes all relevant properties and coking outcomes. No interpretation of results is given in a standard report. A standard report includes:

- Relevant laboratory analyses, if requested.
- Coke charge preparations including coal charge crush sizing, moisture content, oil additions.
- Coking conditions including charge conditions, temperature profile, coking rate, throughput and productivity, oven dimensions, internal pressure and wall pressure measurements.
- Coal properties including all tested laboratory analyses.
- Coke chemical and physical results including yield, coke size analysis, drum indices and NSC reactivity (CRI/CSR) results.

Technical Evaluation Report

For an in-depth and comprehensive understanding of a coal’s coking properties, technical evaluation reports are available. Technical evaluation reports are an interpretative assessment of coal and coke quality parameters by an experienced coal utilisation consultant. The report can either focus on a set of results for one or more coals or focus on an interpretive analysis of a research program. The objectives of a Technical Evaluation Report are to:

- Examine the utilisation behaviour of the coking coal in an industrial coke oven.
- Examine the potential performance of the produced coke in a blast furnace.
- Benchmark the performance of the coal against that of a wide range of competitor coals previously tested by the Coking Research Centre.
- Highlight positive and negative aspects of the coal’s behaviour.
- Assess where the coal fits into the market.
- Explore potential blending partners.
- Provide recommendations for future testing requirements.

Case Study: Mineral Catalysis Index (MCI)

The rank and reactive maceral content account for the majority of the variation seen in coke quality, but for coals of similar rank and reactive maceral content it has been found that the composition of the mineral matter can have a significant impact on coke quality, especially coke reactivity. Zhang et al. found that the mineral composition of coal (represented by mineral catalysis index (MCI)) is a key factor affecting coke quality besides the coal rank and the plastic properties of the coal. MCI has been found by ALS Coal to be a useful index to clarify the variation in CRI of products from a single mine.

Depending on the extensiveness of the report and the quantity of coking coals evaluated, the cost for technical evaluation reports will be confirmed upon completion of the coke test-work.