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Producing Clean Coal Samples from Western Canadian Coalfields Using the Water-Based Roben Jig Process: Application to an Industrial Setting

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Table of Contents

1. Introduction

This project is part 3 of a three year investigation on the use of the Roben Jig (also known as Boner Jig) for washing coal. Summary reports have been completed for the first two years. Because of the continued necessity to introduce the background outlining the importance of this research, some parts of this report have been borrowed from the previous reports.

There are a number of coalfields in British Columbia (BC): several thermal coalfields and two major metallurgical coalfields, the Kootenay and Peace River (Figure 1). Metallurgical coals are carbonized in blends in commercial coke ovens to produce coke for use in ironmaking blast furnaces in steelworks.
One of the main challenges after finding and identifying coal seams is the evaluation of the quality of the coal resource during the exploration stage. Understanding coal quality can be a complex process and is key to conducting a sound economic evaluation of the resource. During the exploration phase of coal mine development, evaluation of metallurgical coal quality is often done using samples collected from small drill cores, since the bulk of the coal deposit is generally deep underground.

Coal samples collected during exploration are first prepared by screening and then performing lab scale or pilot scale washing that simulates the coal behaviour in commercial coking coal wash plants. The coarser coal is processed using mixtures of organic liquids and the finer fraction is cleaned by a process called froth flotation. The quality of the coal produced by these smaller scale washing methods is critical in understanding the market potential of the coal. For that reason these processes must endeavour to produce the same quality coal as a commercial plant.

On a lab scale, the float-and-sink procedure (Figure 2) is used to separate coal from dirt, rock and mineral matter using density separation. The lower density solutions tend to float mainly the coal. During the float-and-sink process, the coal sample is separated at relative densities (SG) between roughly 1.40 SG and 1.80 SG using tanks of organic mixtures made from white spirit (1.40 SG), perchloroethylene (PCE; 1.60 SG) and methylene bromide (1.80 SG) in accordance with ASTM D4371-06 (2019)e1). This produces clean coal at the target ash, sulphur and calorific content that is typical of what would be produced in a commercial coal washing plant.

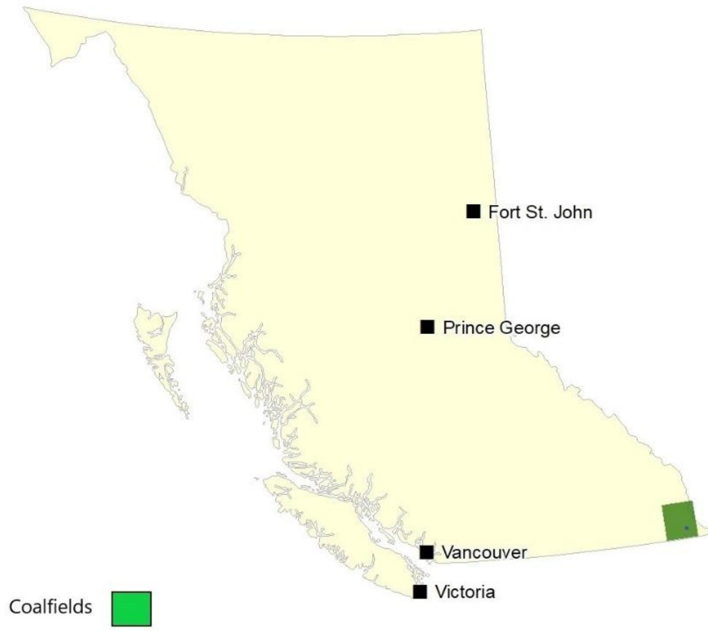

Figure 1. Location of coalfields in southeastern British Columbia from which the coal samples used in this study originated.

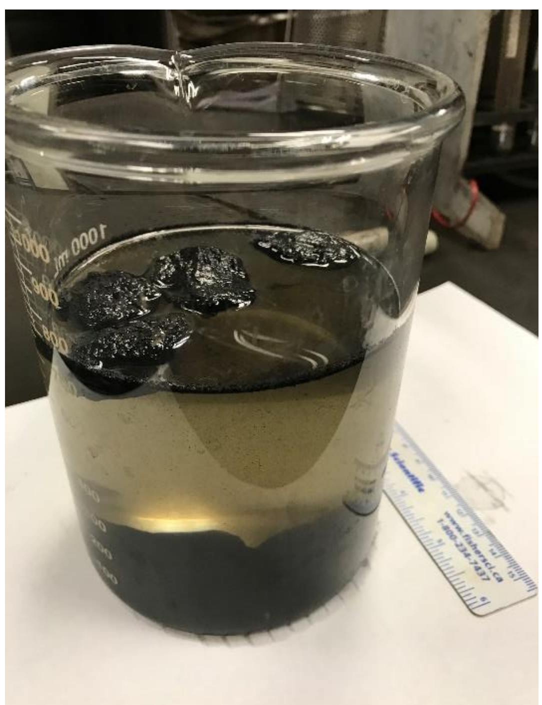

Figure 2. Coal particles floating in perchloroethylene (PCE).

Commercial plants separate the coal into size fractions that are then processed in equipment that separates the coal from waste (rock, dirt and minerals) using differences in density—coal being less dense than the waste. The equipment uses water-magnetite mixtures of controlled density in cyclones and baths, centrifugal force for coal-water mixtures in cyclones, and relative settling rates of the coal particles of differing densities in water to isolate/separate the 'clean' coal in jigs and settling tanks. The mid size coal is cleaned by water-based density separations (spirals, hindered bed separators) while the finest sizes are treated by water-based froth flotation, which can 'float' the fine coal particles from the waste. Exploration samples are treated/cleaned in a similar fashion.

Project economics are based on the results of the float-and sink and froth flotation testing procedure, which gives information on the yield of clean coal and on the quality of the clean coal as well as that of the resulting coke. The coking characteristics of a metallurgical coal deposit are critical in evaluating project economics, in particular the expected price for the clean coal. It is important to ensure that coal/coking properties are correctly assessed from drill core samples in order to properly evaluate project economics.

2. Background

Historically, the major concern in the handling and use of organic liquids such as perchloroethylene (PCE) has been the safety risks associated with human exposure, since PCE is a known carcinogen and poses a safety hazard for laboratory operators. Figure 3 shows a laboratory technician working in a specially designed fume hood, wearing personal protective equipment, including a respirator mask.

In addition to the health issues, there have been increasing concerns about the impact these liquids have on the quality of coking coal. Based on the authors' and their colleagues' experience going back several decades, cleaned drill core coal samples often had lower caking/coking properties than bulk or production coal samples. A number of investigations have examined how PCE and other organic liquids may impact the coking quality of coal samples, including American and Australian work (DuBroff et al., 1985; Campbell, 2010; Iveson and Galvin, 2010, 2012). These studies found that there were different impacts depending on the quality characteristics of the coal being assessed. Coals similar to the western Canadian coking coals (higher inert, lower thermal rheology) appeared to have been negatively impacted.

Based on these observations, the Canadian Carbonization Research Association (CCRA) undertook a preliminary program to investigate the impact of organic liquids used in float-and-sink procedures on the coal and coke properties of a higher inert, low fluidity, western Canadian coal sample (Holuszko et al., 2017). This study looked at the effects of PCE on coal rheology and coke quality. It was found that an 80%

decrease (relative to the control sample) in Gieseler maximum fluidity occurred in the perchloroethylene-treated coal immediately following treatment. The coke resulting from the treated sample showed a 16-point decrease (absolute) in coke strength after reaction (CSR) (decrease from 74 to 58) relative to the control sample. These two coal and coke quality parameters are key when evaluating metallurgical coal resources and reserves. The ramifications of using the wrong numbers for these parameters when determining the characteristics of product for sale, are severe and could result in unwarranted project abandonment or false over valuation of the property.

After the initial study outlined above, the CCRA completed an exploratory study that examined an alternative to organic liquids by washing coal samples in a jig. A lab scale Roben Jig (Figures 4, 5) was used to clean several coals using only water, and the resulting quality characteristics of the clean coal and its coke were compared to those of coal that was processed using the traditional process of washing with organic chemicals. That investigation found that it was possible to produce a clean coal product with quality properties very similar to those obtained using the organic liquids (Mackay et al., 2019). The Roben Jig–cleaned coals had similar results for most coal quality parameters, with better results for coal rheology parameters. These findings are important because they demonstrate that the water-based Roben Jig can be used to produce clean-coal composites similar to those obtained from traditional float-and-sink methods.

Figure 3. Operator working with organic liquids in a specially designed fume hood.

Figure 4. Roben Jig equipment used in this study.

Figure 5. Inverted Roben Jig with coal slice to be removed.

3. Objectives

The studies completed on the Roben Jig to date have verified the jig as a tool to assist in evaluating coal deposits with respect to coal and coke quality at the exploration phase. They have shown that traditional organic liquids (perchloroethylene, white spirits and methylene bromide) can negatively affect coal rheology and coke strength, resulting in an undervaluation of exploration samples (Mackay et al., 2019). Tasks completed include (1) the development of a jig methodology (2) a comparison of coal and coke quality when using the jig versus organic liquids (3) the identification, characterization and mitigation of misplaced particles and (4) a comparison of jig-washed coal to coal from an industrial process plant (using the same raw coal). Three clean coal samples were prepared for carbonization from three different washing processes: Organic Liquids (bulk wash) and Roben Jig (bulk jigging), Industrial Wash Plant.

The yardstick for proving the effectiveness of the Roben Jig in cleaning coal is to compare it to an industrial setting. This work was initiated in 2018 when clean coal washed through the jig was compared with coal washed through an industrial processing plant (Mackay et al., 2019). Subsequent work has focused on creating clean coal composites for charging in a pilot-scale coke oven (340 kg capacity) and determining the resulting coke quality. The previous work used only a small carbonization oven—the sole-heated oven (12 kg capacity). This small oven produces only sufficient coke to measure the coke reactivity index (CRI) and the coke strength after reaction (CSR). The larger size pilot scale carbonization oven yields enough coke to determine four additional indices, aimed at determining the coke strength at ambient temperature. These are a series of drum indices (ASTM, JIS, Micum/IRSID).

The research conducted aimed to answer the following questions:

1) Since misplaced particles occur throughout the segregated coal column, how do the high ash particles (fragments of minerals and rock) affect the coke-strength drum indices measured on coke produced in a pilot scale coke oven?
2) How does the coke that is made from clean coal derived from a) organic liquids washability, b) the Roben Jig and c) industrial processing plant, compare with respect to all relevant coke characteristics?
3) What is the best methodology and expected cost to do ‘bulk jigging’—the process where the Roben Jig is used to create 400 kg of clean coal for charging in a pilot scale coke oven?

Another objective of the research group is to draft a standard operating procedure for the Roben Jig for the purpose of producing small-mass clean coal samples in British Columbia.

The success of this project is beneficial to the coal industry for the following reasons:

- It eliminates the potential negative effects of perchloroethylene and other organic liquids on small-mass exploration coal and coke quality parameters.
- It reduces the exposure of lab technicians/operators to carcinogenic organic liquids.

4. Experimental Washing Methodology

A coal bulk sample of low inert Western Canadian metallurgical coal of high volatile A bituminous rank (RoMax 1.02) originating from a mine site in Southeastern British Columbia was split into three subsamples for laboratory and pilot scale oven testing. The subsamples were respectively washed in an industrial plant, in water-based Roben Jig and in organic liquids following float-and-sink gravity separation (1.40-1.80 SG). The raw feed coal from the Industrial plant was sampled and sent to Birtley for washing in the Roben Jig and Organic Liquids. The clean coal from the same coal run was also collected and sent to Birtley for clean coal analysis. The Roben Jig and Organic Liquids washed coals were stored in a refrigerator in sealed containers when not being worked on. The clean product from the Industrial plant was stored in a freezer until it was time to analyse for clean coal quality characteristics on all three clean samples (Roben Jig, Organic Liquids, Industrial Plant.

The research group devised two Roben Jig methodologies that could yield products with lower ash content while minimizing misplaced coal and rock particles. These methodologies were compared to the original coal washing methodologies from Phase 1 research (Mackay et al., 2018). The clean coals from all processes were then compared to the product from an industrial coal washing plant. The method for the industrial coal washing plant is detailed in Mackay et al. (2019).

4.1 Coarse Coal Washing

The coarse coal particles (greater than 0.50 mm) from each coal sample were washed in several different ways:

- Raw coal was segregated into a single coarse fraction (12.5 × 0.5 mm) and washed in organic liquids using the float-and-sink method as described in Mackay et al., 2018(Phase 1 Method: Float and Sink, One Coarse Fraction) and targeting a specific ash percentage. Following this,
- one specific gravity was chosen to ‘bulk wash’ the remainder of the raw coal to create a clean coal composite. The bulk washing in organic liquids is described below.
- Raw coal was segregated into one coarse fraction (12.5 × 0.5 mm) and washed in the Roben Jig (Mackay et al., 2018, Phase 1 Method: Roben Jig, One Coarse Fraction).

- A new method called ‘Bulk Jigging’ was developed for this phase of research and is explained in detail below.
- Raw coal was washed in an industrial coal washing plant.

4.2 Fine Coal Washing

Common to all methodologies, the fine coal (particle sizes less than 0.5 mm) was washed using froth flotation (ASTM D5114-90(2018)e1). The froth-flotation was completed on a timed basis where clean froths were collected at 30 and 60 second time intervals and then continuous clean froth was collected until no froths were available. The bulk frothing method included collecting froth until “completion” which means that it was collected until no other coal particles were frothing. The clean coal resulting from this method was recombined with the coarser coal (greater than 0.5 mm) when creating clean coal composite samples.

4.3 Bulk Washing in Organic Liquids

The results of ‘Phase 1 Method: Float-and-Sink, One Coarse Fraction’ (Mackay et al., 2018) were reviewed, a target ash % was chosen for the clean sample, and a ‘cut point’ (the specific gravity at which all clean coal that is floating, is combined with the coal that floats at all lower specific gravities, to create the clean coal composite) was then selected. As an example, in a washability table that listed the mass and ash value for the coal that floated at specific gravities of 1.30, 1.40, 1.50 and 1.60, one may choose a cut-point of 1.50 SG, depending on the target ash percentage. The remainder of the raw coal would then be floated in a large bath of 1.50 SG liquid instead of undergoing a series of density separations at specific gravities of 1.30 to 1.80.

4.4 Bulk Jigging Using Roben Jig

The intent of bulk jigging is similar to that of bulk washing in organic liquids, in which, the process of cleaning the coal can be sped up by eliminating some of the detail in the washing process (i.e. finding a ‘cut point’ in the jig column).

First, a trial is carried out by jigging 15 kg of raw coal and removing 12–18 slices from the jig column. The ashes are determined for each slice and reviewed with the corresponding apparent relative densities (ARD). Apparent relative density was measured using the Australian Standard AS 1038.26. The obvious rock (>1.90 ARD) and clean coal (<1.35 ARD) zones are identified and measured. For instance, once the column is inverted, the rock would be located in approximately the top 15 cm of the jig column and the clean coal would be in the bottom 30 cm. Next, the higher ash coal zone is identified—this zone is always the area between the obvious rock and cleanest coal. More batches of raw coal are jigged and the thickness of the slices is increased because the boundaries between obvious clean coal, higher ash coal and rock are roughly known. For each batch, slices with similar ARD values are grouped together into buckets. Once the bulk jigging is complete, the ash % of each bucket is analyzed. A specific ash % is targeted and the buckets with the requisite ash % are added to the clean coal sample.

5. Analysis

5.1 Clean Coal Analysis

Clean coal composites were created for the Roben Jig and Organic liquids wash processes by recombining the clean coarse fractions with the clean fine fractions based on the original size fraction proportions for the coal sample.

Each clean coal composite was analyzed at GWIL Industries–Birtley Coal & Minerals Testing Division (Calgary, AB) for yield (%), proximate analysis, free swelling index (FSI), specific gravity (SG), total sulphur, Hardgrove Grindability Index (HGI), calorific value (kcal/kg), mercury, ultimate analysis, mineral analyses of the ash, phosphorus in coal (calculated, %), Gieseler maximum fluidity, Ruhr dilatation, ash fusion (oxidizing and reducing), chlorine, fluorine, alkali extraction–light transmittance test, Sapozhnikov X and Y indices, and caking index (G). Petrographic analysis of the coal was carried out at David E. Pearson & Associates (Victoria, BC) whereas coke petrography was done at CanmetENERGY (Ottawa, ON).

5.2 Carbonization

Clean coal samples from this study were sent to CanmetENERGY in Ottawa. Upon reception, coals were air dried in open air in the laboratory for 12 hours for the 12 kg capacity sole heated oven charges and 24 hours in the case of the larger samples that were to be coked in the 340 kg capacity Carbolite oven. The coal samples were then homogenized prior to preparing charges for.

A description of the features and operating conditions for carbonization of coal in the sole-heated oven (ASTM D2014-20), is provided in Mackay et al. (2019), as is a description of the Carbolite pilot oven. Those descriptions include the procedure for the preparation of coke samples from coals in this project for the measurement of coke reactivity (CRI) and coke strength after reaction (CSR) by means of the sole-heated oven, following a procedure developed at CanmetENERGY (MacPhee et al., 2013).

6. Results

6.1 Experimental Washing

Details of the methods of removing rock from coal (washability) are included in the methodology section. Three clean samples were made for carbonization: Industrial Wash Plant clean product, Organic Liquids clean product and the Roben Jig clean product. The Industrial Wash Plant clean product was produced at a mine site in Southeast BC. The raw coal from the plant run was washed at GWIL Industries–Birtley Coal & Minerals Testing Division for this research product.

6.1.1 Mass Weight vs. Specific Gravity

The graphs below (Figures 6, 7), which track the mass % vs specific gravity (or in the case of the Roben Jig, apparent relative density), show the percent of the entire raw coal sample that floated at each specific gravity during the float-and-sink process.

The float-and-sink process is perfect in organic liquid baths because it is a fixed method (when laboratory standards such as ASTM are followed) – the liquids stay at the specified densities and the coal is allowed to float to the top of each bath. The graph for the Organic Liquids washability shows that approximately 65% of the coal floats at 1.80 SG or less. The graph for the Roben Jig float-and-sink shows the same trend. The end of the curve on the Roben Jig graph is less steep than that for the Organic Liquids due to the fact there are several more data points to plot on the graph – with apparent relative density values increasing beyond 2.00 SG. This is a function of the methodology of slice removal while using the Jig; slices are incrementally removed from the inverted product cylinder, which enables the user to see more detail in coal particle apparent relative density.

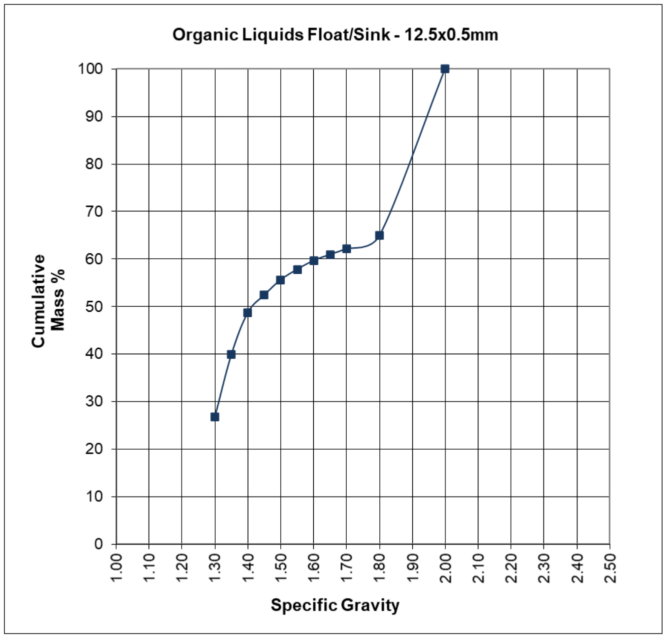

Figure 6. Percent of the entire raw coal sample that floated at each specific gravity in the Organic Liquids (float-and-sink) method.

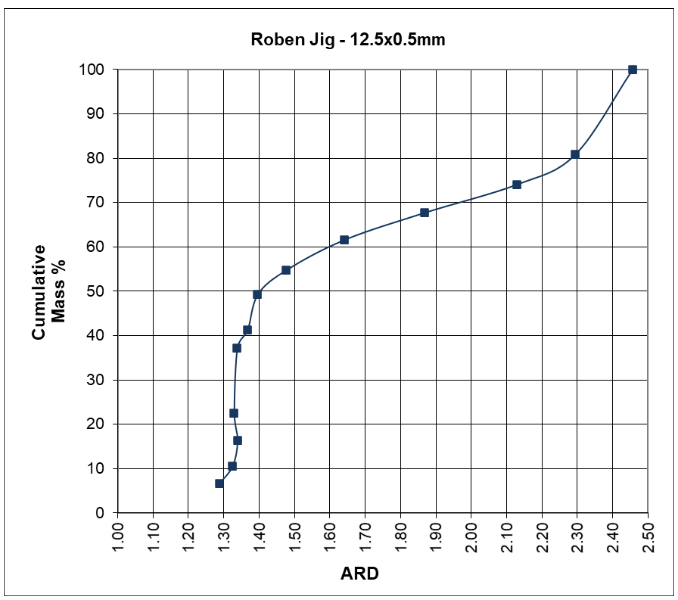

Figure 7. Percent of the entire raw coal sample that floated at each apparent relative density using the Roben Jig method.

6.1.2 Mass Weight vs. Ash Content

Figures 8 and 9 show curves representing the cumulative weight % vs. the cumulative ash %. Both graphs show that approximately 60–65% of the coal has an ash content of equal to or less than 12% ash.

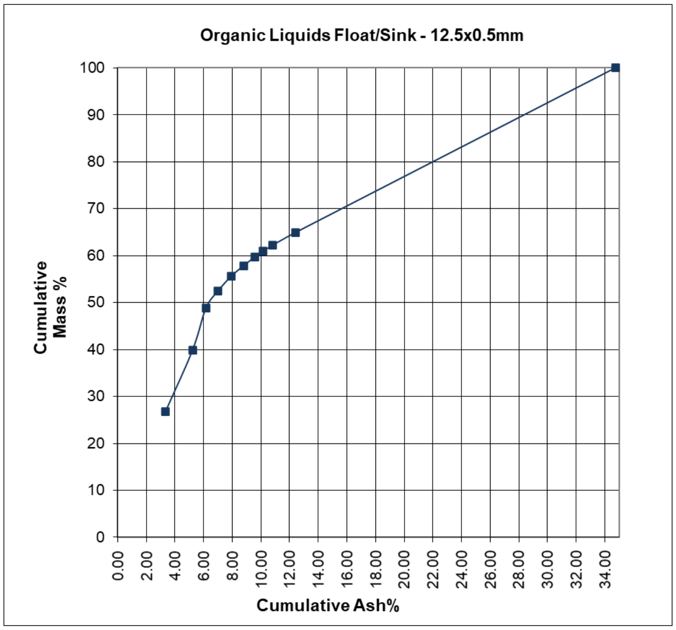

Figure 8. Cumulative mass % versus cumulative ash % in the Organic Liquids (float-and-sink) method.

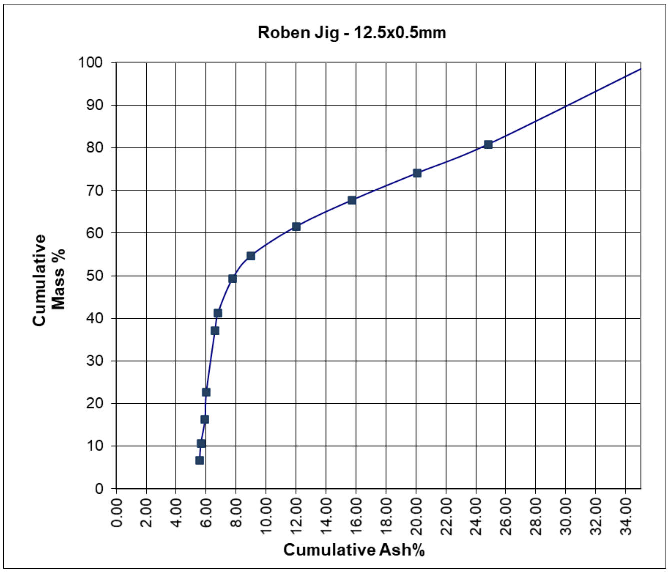

Figure 9. Cumulative mass % versus cumulative ash % in the Roben Jig method.

6.1.3 Ash Content vs. Specific Gravity

Figures 10 and 11 display the curves ash % vs. specific gravity (or in the case of the Roben Jig, apparent relative density). Both graphs show the appropriate trend of increasing fractional ash % with increasing density. The curves are similar in shape except that the Organic Liquids curve is steeper. This is partly because the Roben Jig curve has more data points to graph beyond 2.00 SG. The individual ash % corresponding to the specific gravities are similar in both cases – for instance the ash % for the coal floating at 1.80 SG in organic liquids is close to 50%, while the ash % for coal in the 1.80 apparent relative density slice, according to the graph, would also be close to 50%.

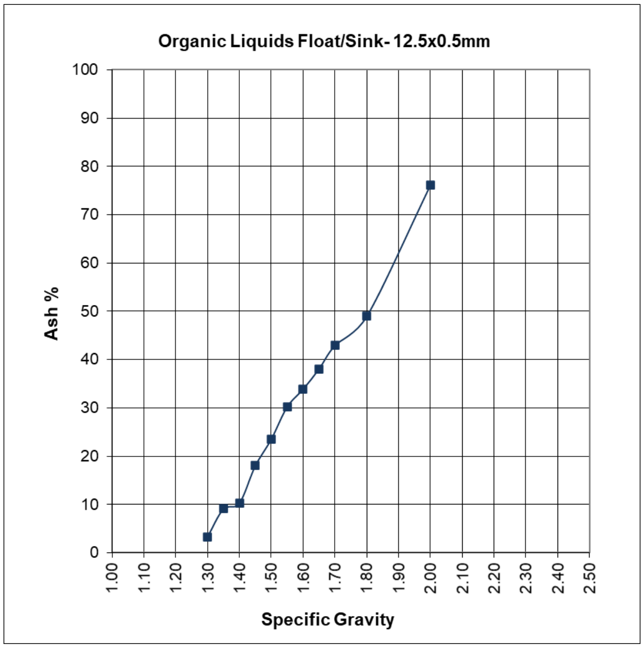

Figure 10. Fractional Ash % vs specific gravity for the Organic Liquids (float-and-sink) method.

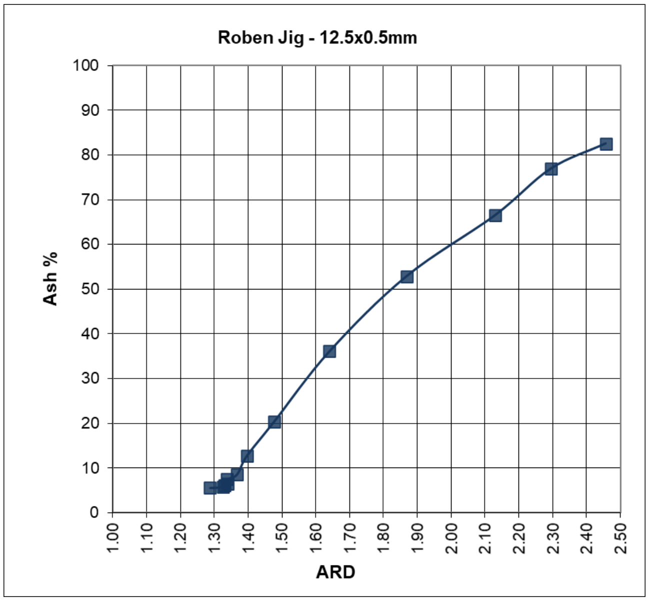

Figure 11. Fractional Ash % vs apparent relative density for the Roben Jig method.

The Roben Jig research work from 2018 found that particles can be misplaced in slices of different density (Mackay et. Al, 2019). A small amount of high-density particles contaminate the lower apparent relative density slices and a small amount of low-density particles contaminate the higher apparent relative density slices. It is also recognized that industrial wash plants do not perfectly segregate the particles and that 'misplaced' particles occur. The best way to test what particles make up the clean coal product is to complete a float-and-sink test using organic liquids. First the wash plant clean coal was sieved to remove the -0.5mm. The resulting sample was named "Simulated Industrial Wash Plant Clean" to differentiate from the whole of the sample.

Table 1 and Figure 12 illustrate the densities and ashes that comprise the Industrial Wash Plant clean product. Table 1 shows that the bulk of the material – approximately 80% of the coal-floated in the 1.35 SG bath. The expected ash of this material is 5.45% (cumulative average of the 1.30 SG and 1.35 SG floated coal). A total of 0.23% of the material was comprised of particles of approximately 68% ash and of densities above 1.80 SG. Figure 12 also illustrates this trend.

Table 1. Simulated clean product data from the Industrial Wash Plant.

Simulated Industrial Wash Plant Clean 50x0.5mm					
				CUMULATIVE	
SG	WT(g)	WT%	ASH %	WT%	ASH %
1.30 FLT	42545	59.93	3.97	59.93	3.97
1.30-1.35	13626	19.19	10.09	79.12	5.45
1.35-1.40	5795	8.16	15.49	87.28	6.39
1.40-1.45	3803	5.36	19.80	92.64	7.17
1.45-1.50	2618	3.69	24.88	96.33	7.85
1.50-1.55	1423	2.00	31.03	98.33	8.32
1.55-1.60	663	0.93	36.30	99.26	8.58
1.60-1.65	151	0.21	38.86	99.48	8.65
1.65-1.70	103	0.15	42.83	99.62	8.70
1.70-1.80	104	0.15	47.25	99.77	8.75
1.80 SNK	164	0.23	67.85	100.00	8.89

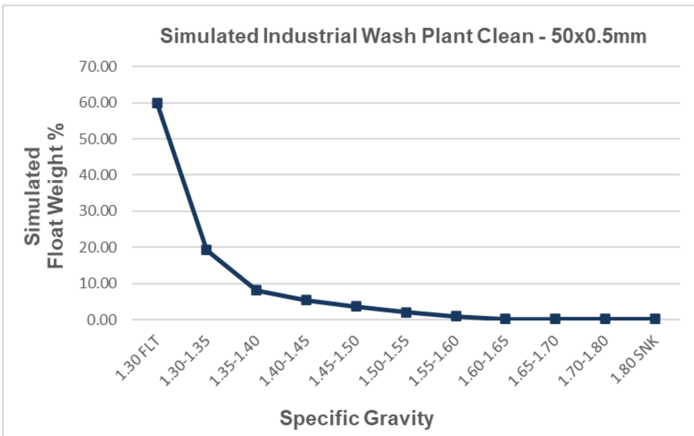

Figure 12. Weight % of Industrial Wash Plant simulated clean product at various densities.

Table 2 and Figure 13 illustrate the particle ash % and specific gravities of the particles that comprise the Roben Jig clean product. Like the Industrial Wash Plant clean product, approximately 80% of the coal floated in the 1.35 SG bath. A portion of the particles (1.55%) had a specific gravity of 1.80 SG or higher. This is slightly more than that of the Industrial Wash Plant clean product, but still a very small amount.

The biggest concern with the occurrence of misplaced particles in a clean coal product is the potential effect on coal or coke quality. This point will be addressed in the following sections.

Table 2. Simulated clean product data from the Roben Jig

Simulated Roben Jig Clean at 1.42 ARD					
				CUMULATIVE	
SG	WT(g)	WT%	ASH %	WT%	ASH %
1.30 FLT	6362	62.13	3.60	62.13	3.60
1.30-1.35	1908	18.63	9.69	80.76	5.01
1.35-1.40	1077	10.52	15.34	91.28	6.20
1.40-1.45	178	1.74	20.84	93.02	6.47
1.45-1.50	207	2.02	25.84	95.04	6.88
1.50-1.55	168	1.64	31.54	96.68	7.30
1.55-1.60	74	0.72	35.36	97.40	7.51
1.60-1.65	38	0.37	40.65	97.77	7.63
1.65-1.70	32	0.31	45.21	98.09	7.75
1.70-1.80	37	0.36	49.74	98.45	7.91
1.80 SNK	159	1.55	73.90	100.00	8.93

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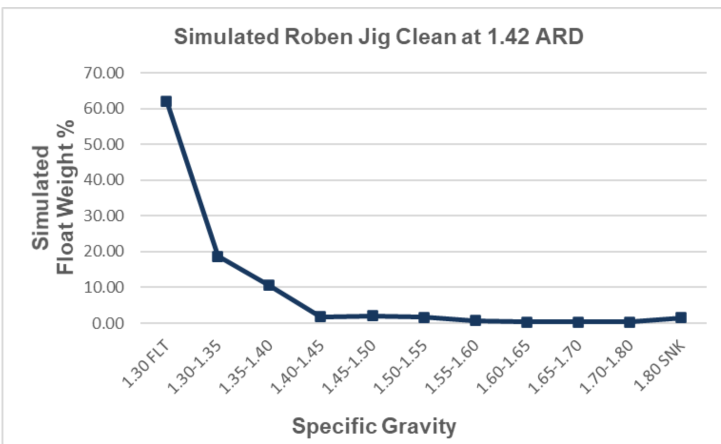

Figure 13. Weight % of Roben Jig simulated clean product at various densities.

6.2 Clean Coal Analysis

During this phase of the project, three clean coal products were compared: the Industrial Wash Plant product, the Organic Liquids clean products and the Roben Jig clean products. Table 3 compares several clean coal parameters for each coal product.

Table 3. Clean coal analyses for each clean coal product.

Air-dried Basis	**Industrial Wash Plant**	**Roben Jig**	**Org Liq**
Moist%	1.00	1.05	1.08
Ash%	8.65	8.99	8.79
VM%	28.40	28.66	28.59
FC%	61.95	61.30	61.54
%S	0.73	0.72	0.73
Calorific Value (kcal/kg)	7766	7690	7700
Chlorine ppm	98	201	2931
Fluorine ppm	232	180	223
HGI	83	80	79
SG	1.31	1.32	1.31
FSI	8.0	8.5	8.0
LT%	93	93	92
%P in coal (db)	0.08	0.05	0.04
Utlimate Analysis			
Moist%	1.00	1.05	1.08
%C	79.83	78.02	78.72
%H	3.97	3.97	3.98
%N	1.59	1.60	1.61
%S	0.73	0.72	0.73
Ash%	8.65	8.99	8.79
%O b/d	4.23	5.65	5.09

For most of the analyses the results were very similar and within laboratory repeatability. The Roben Jig was able to produce a clean coal product very similar to that from an industrial wash plant. The chlorine in the coal washed by the Organic Liquids method was higher due to residue of perchloroethylene being present on the surface and within pore spaces in the coal.

Tables 4 and 5 display the fluidity and dilatation for each clean coal product. The fluidity of the Roben Jig product was higher than that of the Organic Liquids clean product. This is showing how mixtures of perchloroethylene, white spirits and methylene bromide reduce the rheology and namely the fluidity of some coals. The fluidity of the plant product was much higher. This was due to a difference in "aging" of the coals. The plant product coal was kept frozen during the time when the jigging and washing in organic liquids was occurring. This frozen state kept the coal from adsorbing oxygen and prevented it from oxidizing and thus retaining its original fluidity – much like freezing meat stops it from decomposing. The raw coal that was being washed in the Roben Jig and Organic Liquids methods was first thawed from a frozen state, tested in the laboratory and then stored in a refrigerator at 4°C. In order to simulate equivalent aging conditions, the Industrial Wash Plant clean product was stored in a refrigerator at 4°C for the same number of days as the other samples. On April 3, 2020, the fluidity of the plant product was retested (Plant

Rev in Table 4) and the result was 461 ddpm, which was higher than that of the Organic Liquids clean product and lower than that from the Roben Jig product.

Similar differences in dilatation can be observed in Table 5. The Roben Jig clean product dilatation was higher than the organic liquids treated coal, while the plant product had the highest dilation value. Differences between the dilatation values were not as significant as in the case of the fluidity. The dilatation of the plant product was also retested on April 3, 2020 (Plant Rev in Table 5) with a result (180%), that was higher than that of both the Organic Liquids clean coal product and the Roben Jig clean coal product.

Table 4. Gieseler Fluidity results for each clean coal product.

GIESELER FLUIDITY				
Temperatures °C	**Industrial Wash Plant**	**Industrial Wash Plant Rev**	**Roben Jig**	**Org Liq**
Initial Soft (1ddpm)	403	404	412	411
Max Fluidity	448	448	453	450
Solidification	486	483	490	484
Range	83	79	78	73
Max. ddpm	1975	461	768	401

Table 5. Ruhr Dilatation results for each clean coal product.

RUHR DILATATION				
Temperatures °C	**Industrial Wash Plant**	**Industrial Wash Plant Rev**	**Roben Jig**	**Org Liq**
SOFT TEMP	382	378	373	382
TMCONT.	428	436	461	430
TMDIL.	476	472	478	464
%CONT.	27	28	25	25
%DIL	185	152	148	113
% TOTAL DIL	212	180	173	138
%SD 2.5	187	158	153	120

Table 6 summarizes the mineral analysis of ash for each of the clean coal products. Results for the three products are very similar. The slight differences that are shown are not significant. It is known that there will likely be misplaced particles in the clean coal product from jigging. The ash chemistry results are an indication that these misplaced particles do not change the chemical makeup of the clean coal resulting from the Jig.

Table 6. Mineral analyses of ash for each clean coal product.

MINERAL ANALYSIS OF ASH			
	Industrial Wash Plant	**Roben Jig**	**Org Liq**
SiO_2	61.73	63.40	63.49
Al_2O_3	27.97	23.72	24.26
TiO_2	1.40	1.36	1.42
CaO	1.36	1.32	1.46
BaO	0.66	0.51	0.63
SrO	0.26	0.28	0.31
Fe_20_3	1.93	2.92	2.52
MgO	0.51	0.61	0.51
Na_2O	0.04	0.01	0.07
K_20	1.23	1.46	1.06
P_20_5	1.97	1.71	1.89
SO_3	0.42	0.40	0.42
Undet.	0.52	2.3	1.96

Table 7 displays identical ash fusion temperatures (under reducing and oxidizing conditions) for all three clean coal products.

Table 7. Ash fusion temperatures for each clean coal product.

Clean	**REDUCING**				**OXIDIZING**			
Products	**IDT**	**ST**	**HT**	**FT**	**IDT**	**ST**	**HT**	**FT**
Industrial Wash Plant	+1500	+1500	+1500	+1500	+1500	+1500	+1500	+1500
Roben Jig	+1500	+1500	+1500	+1500	+1500	+1500	+1500	+1500
Org Liq	+1500	+1500	+1500	+1500	+1500	+1500	+1500	+1500

Table 8 summarizes the petrographic analyses for the three clean coal products. The results are very similar, the rank (RoMax) ranged from 0.99 to 1.02. Total Reactives for the three coals range from 85.0% to 87.1%, with total inerts ranging from 12.9% to 15.0%.

Table 8. Petrographic analyses of each clean coal product.

Sample Identifier	**Industrial Wash Plant**	**Roben Jig**	**Org Liq**
Mean Maximum Reflectance (RoMax)	1.02	0.99	1.00
V-8		1.0	2.0
V-9	34.0	59.0	47.0
V-10	56.0	39.0	49.0
V-11	10.0	1.0	2.0
Reactive Components			
Vitrinite	78.0	80.7	79.8
Liptinite	1.3	1.1	0.9
Reactive Semifusinite	5.7	5.3	5.5
Total Reactives	**85.0**	**87.1**	**86.2**
Inert Components			
Inert Semifusinite	5.7	5.3	5.5
Fusinite	4.0	1.9	2.8
Inertodetrinite	0.4	0.6	0.4
Mineral Matter	4.9	5.1	5.1
Total Inerts	**15.0**	**12.9**	**13.8**

Summarizing, the analytical testing of the three clean coal products produced by the Industrial Wash Plant, the Roben Jig and the Organic Liquids, shows that when using the same raw coal, all three washing methods produce a very similar clean coal product. The exception is that the Roben Jig and Industrial Wash Plant products have higher rheology (fluidity and dilatation) and lower chlorine compared to the Organic Liquids product. Our conclusion is that rheology and chlorine values from the Roben Jig and Industrial Wash Plant clean products better represent the coal characteristics, since these procedures are not inhibited by the presence of perchloroethylene and other organic liquids.

6.3 Carbonization

Clean coal products from the three washing processes (as described above) were carbonized. These subsamples (~625 kg of plant clean and ~460 kg each from Roben Jig and organic liquid float-and-sink washing) were received at CanmetENERGY in Ottawa on December 30, 2019 from GWIL Industries–Birtley Coal & Minerals Testing Division (Calgary, AB) and are listed in Table 9.

Table 9. Coal samples carbonized at CanmetENERGY.

Industrial Plant	**Roben Jig**	**Organic Liquids**
100% CCRA	100% CCRA	100% CCRA
#197666 2019-1	#195013 JIG-CCC	#195013 F/S CCC

Upon reception, the samples were air dried in open air in the laboratory for 12 hours (24 hours in the case of the larger sample of 100% Industrial Wash Plant clean products) and homogenized prior to preparing charges for coking in CanmetENERGY's 12 kg capacity sole-heated oven and its 340 kg capacity Carbolite pilot coke oven.

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6.3.1 Sole-Heated Oven (ASTM D2014-20)

A 12 kg sample of coal (70–80% –3.35 mm or –6 mesh) was divided equally and each half charged into one chamber (approximately 280 mm in width, length and depth) of a double-chambered oven. A weighted piston applied a constant force corresponding to a pressure of 15.2 kPa (2.2 psi) to the top of the coal bed (thickness in the 76–90 mm range), which was heated from below according to a prescribed temperature program. The sole temperature was raised from 554°C to 950°C at a heating rate of 0.9–1°C/min. The movement of the load was continuously monitored during the test, which was complete when the temperature at the top of the coal bed reached 500°C (normally reached after a period of 6–7 hours). The measured expansion or contraction of the sample was converted to a reference base of 833 kg/m^3 (52 lbs./cu. ft.) and 2% moisture.

After carbonization, the semi-coke was removed from the sole-heated oven and reheated in a stainless steel holding box (229 mm wide, 292 mm long and 305 mm deep) that is hermetically sealed on top with a 3 mm thick section of stainless steel and lined with a 3 mm thick layer of ceramic-fibre insulation. The steel has an exit hole 1 cm in diameter in the centre for venting the hot coke gases. Also, the holding box is fitted on the bottom with a stainless steel inlet tube (150 mm long and 6 mm inside diameter) connected to a cylinder of nitrogen gas, which allows for continuous flushing of the coke with the gas (5–10 l/min flow rate) to prevent its combustion. This treatment heated the semi-coke to 1100°C to complete the annealing of the coke.

A schematic of a sole-heated oven is presented in Figure 14 and a photo of the sole-heated oven used in this project is shown in Figure 15.

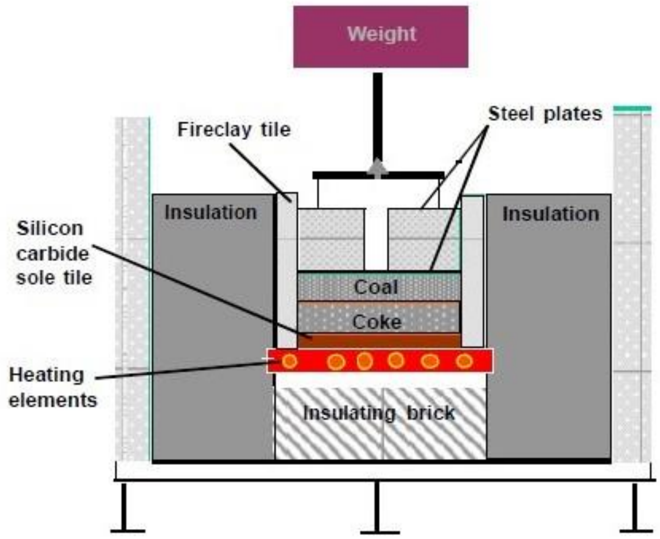

Figure 14. Schematic diagram of the CanmetENERGY sole-heated oven.

Figure 15. CanmetENERGY sole-heated oven (12 kg capacity) used in this study.

Cokes from the sole-heated oven were assessed for apparent specific gravity (ASG) and hot strength properties, including CSR and CRI (following the ASTM D5341-19 standard), and were analyzed for proximate (moisture, ash, volatile matter and fixed carbon), sulphur and carbon forms/textures using an optical microscope.

The ASG of coke is defined as the ratio of the mass of a volume of dry coke to the mass of an equal volume of water. Coke ASG varies with the rank and ash content of the coal carbonized, the bulk density of the coal charge in the oven, the carbonization temperature and the coking time (Price and Gransden, 1987). In this project, the ASG of cokes was determined following a method developed at CanmetENERGY and related to the ASTM D167-12 and ISO 1014:1985 standards.

According to ASTM D5341-19, the CRI is the percentage weight loss of the coke sample after reaction in CO_2 at 1100°C for 2 hours. The cooled, reacted coke is then tumbled in an I-drum for 600 revolutions at 20 rpm. The cumulative percentage of +9.5 mm coke after tumbling is denoted as the CSR.

6.3.2 Carbolite Pilot Oven

Specifications of the Carbolite pilot scale movable wall coke oven (Carbolite Gero Ltd., Sheffield, United Kingdom) are listed in Table 10 and the oven is shown in Figure 16.

Table 10. Specifications of the CanmetENERGY Carbolite pilot movable-wall coke oven.

Coke Oven Specifications	Carbolite Pilot Movable Wall Coke Oven
Chamber Width, mm	460
Chamber Volume, m^3	0.405
Charge Weight, kg	~340-350
Coal Size, % passing 3.35 mm	80-85
Charge Density in Oven (dry), kg/m^3	809-825
ASTM bulk density (wet), kg/m^3	773-783
Charge Moisture, %	~2.5-3.2
Heating Control (Flue Temperature), °C	875°C start increase 15°C/h to 1130°C
Pushing Time, hrs	3 hrs after CT=950°C (usually around 18 h)
Quench	Water (wet) normally; N_2 gas (Dry) is also possible
Coke Treatment (Conditioning)	Client specified (usually 1 or 3 drops from 3 m height)

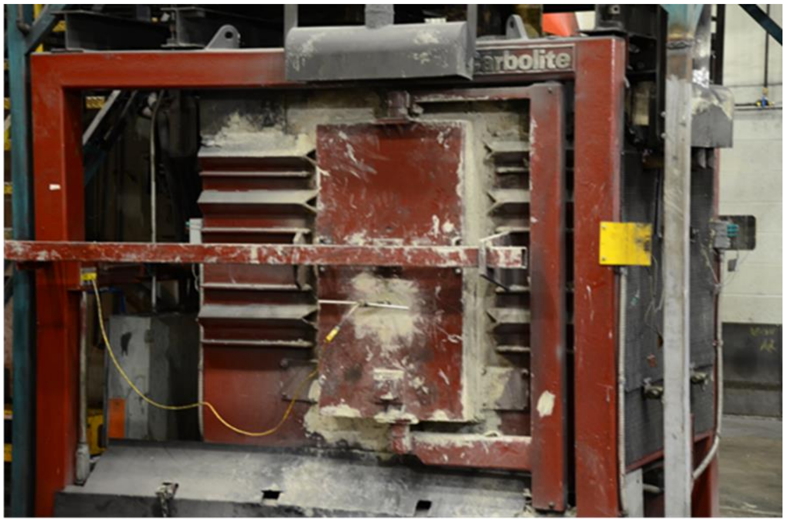

Figure 16. CanmetENERGY Carbolite pilot-scale coke oven (340 kg capacity) used in this study.

To simulate industrial coking, the temperature of the oven is kept low (875°C) at the beginning of the carbonization cycle, to limit the heat input to the coal, and then gradually raised (15°C/h) until the flue temperature reaches 1130°C. The oven is normally charged with coal of which 85 ±5% is less than 3 mm, and the coal moisture is adjusted so as to achieve a dry-coal bulk density in the oven in the range 810–825 kg/m^3. The oven is discharged 3 hours after the centre temperature of the coke reaches 950°C. The coke is water quenched and dropped 3 m onto a concrete floor in order to condition or stabilize it. This process is carried out in preparation for the drum testing, followed by measurement of the resultant coke properties.

The coke discharged from the Carbolite oven is assessed for size distribution, proximate analysis, sulphur, coke stability and hardness using the ASTM tumbler method, the cold strength drum index (DI) test of the Japanese Industrial Standard (JIS), CSR/CRI, ASG and texture.

6.3.3 Carbonization Results and Discussion

Table 11 presents analysis data for cokes produced in CanmetENERGY's sole-heated oven and Carbolite pilot coke oven from the three coal samples.

Table 11. Coke quality obtained from sole-heated oven and Carbolite pilot oven tests.

	Date Received		DEC/30/19	DEC/30/19	DEC/30/19	DEC/30/19	DEC/30/19	DEC/30/19
	Weight Received		4-DRUMS	4-DRUMS	3-DRUMS	3-DRUMS	3-DRUMS	3-DRUMS
	Project		GBC-PH2B	GBC-PH2B	GBC-PH2B	GBC-PH2B	GBC-PH2B	GBC-PH2B
	Coal Index		27662	27663	27664	27665	27666	27667
	Description		100% CCRA #197666 2019-1 SHO,ASG,CSR	100% CCRA #197666 2019-1 C-2898	100% CCRA #195013 JIG-CCC SHO,ASG,CSR	100% CCRA #195013 JIG-CCC C-2899	100% CCRA #195013 F/SCCC SHO,ASG,CSR	100% CCRA #195013 F/SCCC C-2900
Sole-Heated Oven Test	Expansion/Contraction	%	-7.5		-6.4		-9.8	
Coal Sieve Analysis, cumulative	6.30 mm	%		1.51		1.25		2.01
	3.35 mm	%		14.76		16.06		17.57
	1.70 mm	%		31.81		35.35		36.18
	0.85 mm	%		48.93		55.07		54.18
	0.50 mm	%		59.89		67.65		65.52
	passing 3.35 mm	%		85.24		83.94		82.43
Carbonization Results	Oven Test Number			C-2898		C-2899		C-2900
	Test Date			JAN/14/20		JAN/15/20		JAN/16/20
	Moisture in Charge	%		2.9		2.6		2.8
	Net dry charge weight	kg		324.6		324.7		325.2
	ASTM BD	kg/m^3		773.7		775.3		781.7
	Oven dry BD	kg/m^3		816.8		817.0		818.4
	Coking time	h:min		19:02		18:50		18:45
	Final Center Temp	°C		1080		1078		NA
	Time to 900 °C	h:min		15:43		15:30		15:26
	Time to 950 °C	h:min		16:02		15:50		15:45
	Time to 1000 °C	h:min		16:29		16:17		16:10
	Time to Max Wall Pressure	h:min		2:00		2:00		1:38
	Max wall pressure	kPa		4.6		4.8		4.9
	Max gas pressure	kPa		7.8		6.1		4.6
	Coke Yield	%		74.4		74.0		74.2
Coke Proximate	Moisture		0.15	<0.1	TBD	<0.1	TBD	0.12
	Ash		11.65	11.38	TBD	11.37	TBD	11.55
	Volatile Matter		1.44	0.73	TBD	0.82	TBD	1.12
	Fixed Carbon		86.91	87.89	TBD	87.81	TBD	87.32
	Sulphur		0.49	0.55	TBD	0.55	TBD	0.53
Sieve Analysis of Coke, cumulative	100 mm sieve	%		0.0		0.5		1.3
	75 mm sieve	%		9.0		15.0		12.9
	50 mm sieve	%		53.7		55.6		51.3
	37.5 mm sieve	%		84.6		85.0		82.6
	25.0 mm sieve	%		94.6		94.2		92.7
	19.0 mm sieve	%		95.9		95.7		94.9
	12.5 mm sieve	%		96.8		96.6		96.0
	Passing 12.5 mm sieve	%		3.2		3.4		4.0
	Mean coke size	mm		53.9		55.9		54.2
ASTM Coke Tumbler Test	Stability			57.3		53.2		51.7
	Hardness			65.5		64.9		67.1
IRSID Coke Tumbler Test	I10			21.2		23.0		21.8
	I20			77.0		75.1		75.3
	I40			47.4		43.7		40.2
MICUM Coke Tumbler Test	M40			77.0		72.9		70.0
	M10			8.0		8.9		10.0
JIS Coke Tumbler Test	50 mm sieve 30 rev			32.2		33.9		23.4
	25 mm sieve 30 rev			90.6		88.5		87.6
	15 mm sieve 30 rev			94.0		92.5		92.5
	50 mm sieve 150 rev			13.8		4.2		1.6
	25 mm sieve 150 rev			74.8		70.7		67.7
	15 mm sieve 150 rev			81.9		79.3		79.1
Coke Density	ASG		0.983	0.913	0.986	0.910	1.008	0.918
CSR	CSR		67.0	69.2	70.8	66.7	69.9	67.5
CRI	CRI		17.7	16.0	16.0	16.4	17.7	17.7

The sole-heated oven contraction level for the three coal samples carbonised at CanmetENERGY (Table 9) was, on average, -7.9 ± 1.7% (Table 11). The type of washing media, namely float-and-sink (organic liquids) and Roben Jig (water), had a minor effect on the level of contraction, with float-and-sink washing leading to slightly better contraction.

The size distribution of the three coal samples charged and carbonized in CanmetENERGY-Ottawa Carbolite pilot oven was similar, 82-85% < 3.35 mm, but the Industrial Wash Plant clean product (#197666

2019-1) included a higher proportion of coal fines <0.5 mm, 40%, than the Roben Jig (#195013 JIG-CCC) and float-and-sink (#195013 F/S-CCC) washed samples, 32-33%.

The three coal samples listed in Table 9 were carbonised in the Carbolite pilot coke oven on consecutive days, between January 14 and 16, 2020. The oven bulk density (dry) for the tests was very similar, 816.9 ± 0.1 along with coking times, 18 hrs 56 min ± 9 min, maximum oven wall pressures, 4.7 ± 0.1, and maximum internal gas pressures, 7.0 ± 1.2 kPa. Figure 17 presents Carbolite pilot oven wall, internal gas pressure profiles and oven centre temperature profiles against coking time.

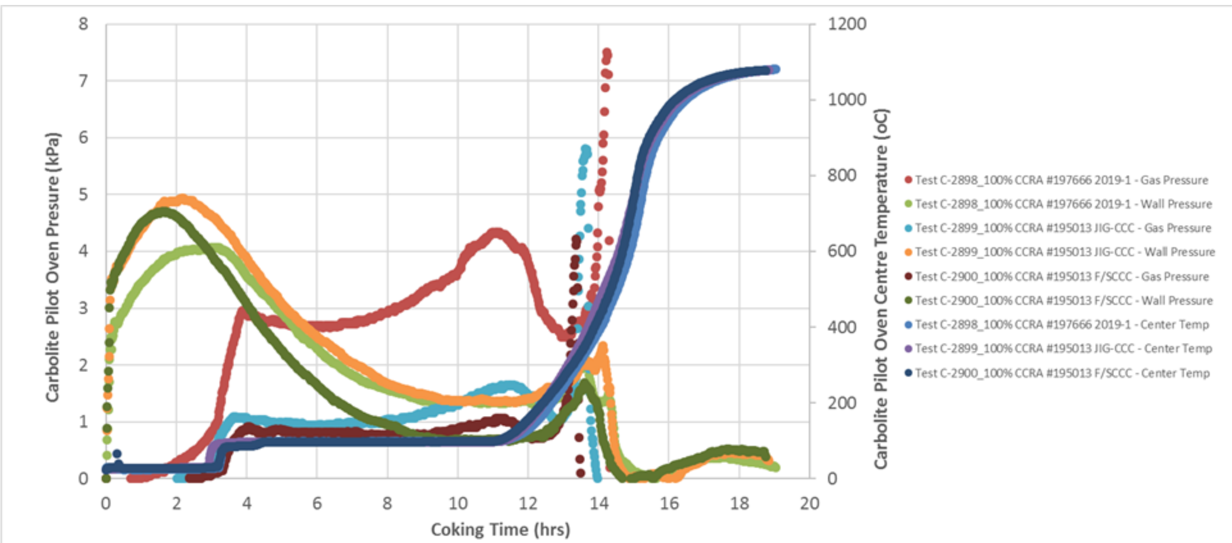

Figure 17. Carbolite Pilot Coke Oven Wall, Gas, and Centre Temperature Profiles.

The low volatile matter (VM) content remaining in the cokes produced in both the small sole-heated oven and large Carbolite pilot oven (< 1.5% VM), is an indication that the coals were essentially fully carbonised during the tests.

An examination of the quality of the cokes produced in the Carbolite pilot oven from the three coal samples indicates that the single seam western Canadian coal selected for this work (High volatile – A Bituminous, RoMax 1.02, inert level 15%, Gieseler maximum fluidity 1,975 ddpm, revised to 461 ddpm) yields good quality coke irrespective of the washing method used. This statement is supported by the comparable coke tumbler indices at ambient temperature (ASTM Stability/Hardness, Micum M40/M10, IRSID I40/I10 and JIS 150/15) for the samples. The coke hot strength, as determined from CSR and CRI, is also similar for the three samples washed in the different media. Moreover, the Apparent Specific Gravity (ASG) of coke from the Carbolite pilot oven are found to be almost identical for the three samples, at 0.91. The higher coke ASG for the cokes issued from the sole-heated oven, 0.99 on average, is attributed to the higher load

(15 kPa) put on the coal during coking in the smaller oven when compared to pressure on coal in the more voluminous Carbolite pilot oven (4-8 kPa). Despite the higher density of cokes formed in the sole-heated oven, their average CSR of 69 and CRI of 17 are comparable to CSR and CRI of cokes from the Carbolite pilot oven, which averages 68 and 17, respectively.

The coke quality for the lower inert, high fluidity coal selected in this study was essentially found to be unaffected by the washing media, particularly when treated with organic liquids. This is quite different from the significant decrease in coke quality (ambient and hot properties) observed in the earlier work when washing a higher inert, low fluidity medium volatile bituminous (mvb) western Canadian metallurgical coal (RoMax 1.22, inert level 32%, Gieseler maximum fluidity 27 ddpm) with organic liquids (Holuszko et al., 2017).

The observation in the present work that washing with Organic Liquids has a negligible effect on the coke quality of a good coking coal of specific rank and rheology, is supported by an earlier investigation conducted in Australia which found that organic liquids, and in particular perchloroethylene (PCE), has a negligible effect on coals with relatively good initial coking properties (Iveson and Galvin, 2010 & 2012). In that work, one of the coals treated with PCE, namely a low rank (RoMax 0.93), low inert (12.5%) and high fluidity (2,352 ddpm) coal, was found to essentially retain the CSR and CRI levels of the untreated coal. Of note is that this particular Australian coal was of similar rank, inert level and Gieseler maximum fluidity as the western Canadian coal that was studied in the current work (RoMax 1.02, inerts 15%, and Gieseler maximum fluidity 1975 ddpm, revised to 461 ddpm).

7. Conclusions

From this work, the following conclusions are drawn:

1) The washability characteristics for the Organic Liquids (float-and-sink) process was similar to that of the Roben Jig.
2) The clean coal quality characteristics for the three clean coal products originating from the Industrial Wash Plant, Organic Liquids and the Roben Jig methods were very similar, although rheological characteristics were better for the Industrial Wash Plant and Roben Jig samples. Chlorine values were lower for the Roben Jig and Industrial Wash Plant products than for the Organic Liquids washed coal, due to the use of perchloroethylene in the latter.
3) The misplaced particles occurring in the Industrial Wash Plant clean product and the Roben Jig clean product were similar in ash content and specific/apparent gravity. The misplaced particles in the Roben Jig clean coal product were found to behave similarly to that of an industrial plant.

4) The three samples (Industrial Wash Plant, Roben Jig (water) and Organic Liquids) were carbonized on consecutive days in January 2020 in a small scale sole-heated oven (12 kg) and a pilot scale Carbolite coke oven (340 kg) at CanmetENERGY-Ottawa Met Fuels Laboratory, in order to determine coke quality.
5) Sole-heated oven contraction level for the three coal samples were, on average, -7.9 ± 1.7. The float-and-sink (Organic Liquids) washing reported slightly better contraction than the Roben Jig (water) washing procedure.
6) The coal seam selected for this work (RoMax 1.02, inert level 15%, Gieseler maximum fluidity 1,975 ddpm, revised to ddpm) yielded good quality coke irrespective of the washing method and medium.
7) The ambient and hot coke quality results for the three samples were comparable and independent of the washing media. This confirms that the misplaced particles in the Industrial Wash Plan product and Roben Jig product did not affect the quality of the coke that was produced.
8) The sole-heated oven cokes and Carbolite pilot oven cokes, despite having different densities, produced coke with similar CSR and CRI values.
9) A western Canadian coal possessing good Gieseler maximum fluidity (~2,000 ddpm), low inerts (~15%) and of low rank (RoMax 1.02) should yield good quality coke irrespective of the washing method used, particularly when washed with organic liquids. This is contrary to what was found during GBC Phase 1 work (2015), when a western Canadian coal with low Gieseler maximum fluidity (<20 ddpm) and high inert levels, produced a poorer quality coke when washed with organic liquids.
10) The Geoscience BC sponsored work during 2015-2019 on washing metallurgical coals from western Canada without compromising their fundamental coking properties/characteristics and leading to good quality coke, has shown that the H_2O-based Roben Jig is definitely the better method and is more suitable than the traditional coal washing with heavy organic liquids, which (i) pose serious safety hazards/risks for laboratory operators and (ii) may also negatively impact the coking quality results of the clean coal samples.

8. References

Campbell, M. (2010): Options for sink/float analysis – economic and technical review; *in* Proceedings of 37th Symposium on the Geology of the Sydney Basin, Pokolbin, Australia, May 2010.

DuBroff, W., Kaegi, D.D., Knoerzer, J.J. and Spearin, E.Y. (1985): Solvent pretreatment of coal to improve coke strength; United States Patent and Trademark Office, patent number 4528069, URL <http://patft.uspto.gov/netacgi/nph-Parser?Sect1=PTO2&Sect2=HITOFF&p=1&u=%2Fnetahtml%2FPTO%2Fsearch-bool.html&r=3&f=G&l=50&co1=AND&d=PTXT&s1=4528069&OS=4528069&RS=4528069> [November 2017].

Holuszko, M.E., Leeder, R., Mackay, M., Giroux, L., MacPhee, J.A., Ng, K.W. and Dexter, H. (2017): Effects of organic liquids on coking properties of a higher-inert western Canadian coal; Fuel Processing Technology, v. 155, p. 225–231, URL <https://doi.org/10.1016/j.fuproc.2016.06.021> [November 2019].

Iveson, S. and Galvin, K.P. (2010): Influence of organic liquids on coal carbonisation properties; Australian Coal Association Research Program (ACARP), Brisbane, Australia, Project C17051.

Iveson, S. and Galvin, K.P. (2012): The effect of perchloroethylene on coking properties; Fuel, v. 95, p. 504–513, URL <https://doi.org/10.1016/j.fuel.2011.10.009> [November 2012].

Leeder, W.R., Todoschuk T., Howey, C., Giroux, L., Ng, K.W., MacPhee, T. (2015): Predictive Model for Blending Coking Coals to produce High Strength Coke; METEC & 2nd ESTAD, Düsseldorf, Germany, June 2015, pp 1-9.

Mackay, M.L., Giroux, L., Leeder, R.L., Dexter, H., Halko, J., Holuszko, M., and Thomas, D. (2019): Producing clean coal from western Canadian coal fields using the water-based Roben Jig process; *in* Geoscience BC Summary of Activities 2018: Minerals and Mining, Geoscience BC, Report 2019-01, p. 87-100, URL <http://cdn.geosciencebc.com/pdf/SummaryofActivities2018/MM/2017-013_SoA2018_MM_Mackay.pdf> .

Mackay, M.L., Leeder, R.L., Giroux, L., Dexter, H., Holuszko, M., Halko, J., Howey, C. and Thomas, D. (2018): Producing clean coal from western Canadian coal fields using the water-based Roben Jig process; *in* Geoscience BC Summary of Activities 2017, Geoscience BC, Report 2018-01, p. 69–86, URL <http://cdn.geosciencebc.com/pdf/SummaryofActivities2017/MM/SoA2017_MM_Mackay.pdf>

MacPhee, J.A., Giroux, L., Ng, K.W., Todoschuk, T., Conejeros, M. and Kolijn, C. (2013): Small-scale determination of metallurgical coke; Fuel, v. 114, p. 229–234, URL <https://doi.org/10.1016/j.fuel.2012.08.036> [November 2019].

Price, J.T. and Gransden, J.F. (1987): Metallurgical coals in Canada: resources, research, and utilization; Energy, Mines and Resources Canada, CANMET Report 87-2E.

9. ASTM Standard Methods

ASTM D2014-20: Standard test method for expansion or contraction of coal by the sole-heated oven; ASTM International, West Conshohocken, PA, 2010.

ASTM D4371-19: Standard test method for determining the washability characteristics of coal; ASTM International, West Conshohocken, PA, 2012, URL <http://www.astm.org/cgi-bin/resolver.cgi?D4371-06(2012)> [November 2019].

ASTM D5114-18: Standard test method for laboratory froth flotation of coal in a mechanical cell; ASTM International, West Conshohocken, PA, 2010.

10. Australian Standard Methods

AS 1038.26-2005 (R2016). Coal and coke – Analysis and testing Higher rank coal and coke – Guide for the determination of apparent relative density. Standards Australia. <https://www.standards.org.au/standards-catalogue/sa-snz/mining/mn-001/as--1038-dot-26-2005> [June 2005, reconfirmed 2016]

Appendix

Summary tables from report and analyzes. Also available as .xlsx file.

Table 1. Simulated clean product data from the Industrial Wash Plant.

Simulated Industrial Wash Plant Clean 50x0.5mm					
				CUMULATIVE	
SG	WT(g)	WT%	ASH %	WT%	ASH %
1.30 FLT	42545	59.93	3.97	59.93	3.97
1.30-1.35	13626	19.19	10.09	79.12	5.45
1.35-1.40	5795	8.16	15.49	87.28	6.39
1.40-1.45	3803	5.36	19.80	92.64	7.17
1.45-1.50	2618	3.69	24.88	96.33	7.85
1.50-1.55	1423	2.00	31.03	98.33	8.32
1.55-1.60	663	0.93	36.30	99.26	8.58
1.60-1.65	151	0.21	38.86	99.48	8.65
1.65-1.70	103	0.15	42.83	99.62	8.70
1.70-1.80	104	0.15	47.25	99.77	8.75
1.80 SNK	164	0.23	67.85	100.00	8.89

Table 2. Simulated clean product data from the Roben Jig

Simulated Roben Jig Clean at 1.42 ARD					
				CUMULATIVE	
SG	WT(g)	WT%	ASH %	WT%	ASH %
1.30 FLT	6362	62.13	3.60	62.13	3.60
1.30-1.35	1908	18.63	9.69	80.76	5.01
1.35-1.40	1077	10.52	15.34	91.28	6.20
1.40-1.45	178	1.74	20.84	93.02	6.47
1.45-1.50	207	2.02	25.84	95.04	6.88
1.50-1.55	168	1.64	31.54	96.68	7.30
1.55-1.60	74	0.72	35.36	97.40	7.51
1.60-1.65	38	0.37	40.65	97.77	7.63
1.65-1.70	32	0.31	45.21	98.09	7.75
1.70-1.80	37	0.36	49.74	98.45	7.91
1.80 SNK	159	1.55	73.90	100.00	8.93

Table 3. Clean coal analyses for each clean coal product.

Air-dried Basis	**Industrial Wash Plant**	**Roben Jig**	**Org Liq**
Moist%	1.00	1.05	1.08
Ash%	8.65	8.99	8.79
VM%	28.40	28.66	28.59
FC%	61.95	61.30	61.54
%S	0.73	0.72	0.73
Calorific Value (kcal/kg)	7766	7690	7700
Chlorine ppm	98	201	2931
Fluorine ppm	232	180	223
HGI	83	80	79
SG	1.31	1.32	1.31
FSI	8.0	8.5	8.0
LT%	93	93	92
%P in coal (db)	0.08	0.05	0.04
Utlimate Analysis			
Moist%	1.00	1.05	1.08
%C	79.83	78.02	78.72
%H	3.97	3.97	3.98
%N	1.59	1.60	1.61
%S	0.73	0.72	0.73
Ash%	8.65	8.99	8.79
%O b/d	4.23	5.65	5.09

Table 4. Gieseler Fluidity results for each clean coal product.

GIESELER FLUIDITY				
Temperatures °C	**Industrial Wash Plant**	**Industrial Wash Plant Rev**	**Roben Jig**	**Org Liq**
Initial Soft (1ddpm)	403	404	412	411
Max Fluidity	448	448	453	450
Solidification	486	483	490	484
Range	83	79	78	73
Max. ddpm	1975	461	768	401

RUHR DILATATION				
Temperatures °C	**Industrial Wash Plant**	**Industrial Wash Plant Rev**	**Roben Jig**	**Org Liq**
SOFT TEMP	382	378	373	382
TMCONT.	428	436	461	430
TMDIL.	476	472	478	464
%CONT.	27	28	25	25
%DIL	185	152	148	113
% TOTAL DIL	212	180	173	138
%SD 2.5	187	158	153	120

Table 6. Mineral analyses of ash for each clean coal product.

MINERAL ANALYSIS OF ASH			
	Industrial Wash Plant	**Roben Jig**	**Org Liq**
SiO_2	61.73	63.40	63.49
Al_2O_3	27.97	23.72	24.26
TiO_2	1.40	1.36	1.42
CaO	1.36	1.32	1.46
BaO	0.66	0.51	0.63
SrO	0.26	0.28	0.31
Fe_20_3	1.93	2.92	2.52
MgO	0.51	0.61	0.51
Na_2O	0.04	0.01	0.07
K_20	1.23	1.46	1.06
P_20_5	1.97	1.71	1.89
SO_3	0.42	0.40	0.42
Undet.	0.52	2.3	1.96

Table 7. Ash fusion temperatures for each clean coal product.

Clean	**REDUCING**				**OXIDIZING**			
Products	**IDT**	**ST**	**HT**	**FT**	**IDT**	**ST**	**HT**	**FT**
Industrial Wash Plant	+1500	+1500	+1500	+1500	+1500	+1500	+1500	+1500
Roben Jig	+1500	+1500	+1500	+1500	+1500	+1500	+1500	+1500
Org Liq	+1500	+1500	+1500	+1500	+1500	+1500	+1500	+1500

Table 8. Petrographic analyses of each clean coal product.

Sample Identifier	Industrial Wash Plant	Roben Jig	Org Liq
Mean Maximum Reflectance (RoMax)	1.02	0.99	1.00
V-8		1.0	2.0
V-9	34.0	59.0	47.0
V-10	56.0	39.0	49.0
V-11	10.0	1.0	2.0
Reactive Components			
Vitrinite	78.0	80.7	79.8
Liptinite	1.3	1.1	0.9
Reactive Semifusinite	5.7	5.3	5.5
Total Reactives	**85.0**	**87.1**	**86.2**
Inert Components			
Inert Semifusinite	5.7	5.3	5.5
Fusinite	4.0	1.9	2.8
Inertodetrinite	0.4	0.6	0.4
Mineral Matter	4.9	5.1	5.1
Total Inerts	**15.0**	**12.9**	**13.8**

Table 9. Coal samples carbonized at CanmetENERGY.

Industrial Plant	Roben Jig	Organic Liquids
100% CCRA	100% CCRA	100% CCRA
#197666 2019-1	#195013 JIG-CCC	#195013 F/S CCC

Table 10. Specifications of the CanmetENERGY Carbolite pilot movable-wall coke oven.

Coke Oven Specifications	Carbolite Pilot Movable Wall Coke Oven
Chamber Width, mm	460
Chamber Volume, m^3	0.405
Charge Weight, kg	~340-350
Coal Size, % passing 3.35 mm	80-85
Charge Density in Oven (dry), kg/m^3	809-825
ASTM bulk density (wet), kg/m^3	773-783
Charge Moisture, %	~2.5-3.2
Heating Control (Flue Temperature), °C	875°C start increase 15°C/h to 1130°C
Pushing Time, hrs	3 hrs after CT=950°C (usually around 18 h)
Quench	Water (wet) normally; N_2 gas (Dry) is also possible
Coke Treatment (Conditioning)	Client specified (usually 1 or 3 drops from 3 m height)

Table 11. Coke quality obtained from sole-heated oven and Carbolite pilot oven tests.

	Date Received		DEC/30/19	DEC/30/19	DEC/30/19	DEC/30/19	DEC/30/19	DEC/30/19
	Weight Received		4-DRUMS	4-DRUMS	3-DRUMS	3-DRUMS	3-DRUMS	3-DRUMS
	Project		GBC-PH2B	GBC-PH2B	GBC-PH2B	GBC-PH2B	GBC-PH2B	GBC-PH2B
	Coal Index		27662	27663	27664	27665	27666	27667
	Description		100% CCRA #197666 2019-1 SHO,ASG,CSR	100% CCRA #197666 2019-1 C-2898	100% CCRA #195013 JIG-CCC SHO,ASG,CSR	100% CCRA #195013 JIG-CCC C-2899	100% CCRA #195013 F/SCCC SHO,ASG,CSR	100% CCRA #195013 F/SCCC C-2900
Sole-Heated Oven Test	Expansion/Contraction	%	-7.5		-6.4		-9.8	
Coal Sieve Analysis, cumulative	6.30 mm	%		1.51		1.25		2.01
	3.35 mm	%		14.76		16.06		17.57
	1.70 mm	%		31.81		35.35		36.18
	0.85 mm	%		48.93		55.07		54.18
	0.50 mm	%		59.89		67.65		65.52
	passing 3.35 mm	%		85.24		83.94		82.43
Carbonization Results	Oven Test Number			C-2898		C-2899		C-2900
	Test Date			JAN/14/20		JAN/15/20		JAN/16/20
	Moisture in Charge	%		2.9		2.6		2.8
	Net dry charge weight	kg		324.6		324.7		325.2
	ASTM BD	kg/m^3		773.7		775.3		781.7
	Oven dry BD	kg/m^3		816.8		817.0		818.4
	Coking time	h:min		19:02		18:50		18:45
	Final Center Temp	°C		1080		1078		NA
	Time to 900 °C	h:min		15:43		15:30		15:26
	Time to 950 °C	h:min		16:02		15:50		15:45
	Time to 1000 °C	h:min		16:29		16:17		16:10
	Time to Max Wall Pressure	h:min		2:00		2:00		1:38
	Max wall pressure	kPa		4.6		4.8		4.9
	Max gas pressure	kPa		7.8		6.1		4.6
	Coke Yield	%		74.4		74.0		74.2
Coke Proximate	Moisture		0.15	<0.1	TBD	<0.1	TBD	0.12
	Ash		11.65	11.38	TBD	11.37	TBD	11.55
	Volatile Matter		1.44	0.73	TBD	0.82	TBD	1.12
	Fixed Carbon		86.91	87.89	TBD	87.81	TBD	87.32
	Sulphur		0.49	0.55	TBD	0.55	TBD	0.53
Sieve Analysis of Coke, cumulative	100 mm sieve	%		0.0		0.5		1.3
	75 mm sieve	%		9.0		15.0		12.9
	50 mm sieve	%		53.7		55.6		51.3
	37.5 mm sieve	%		84.6		85.0		82.6
	25.0 mm sieve	%		94.6		94.2		92.7
	19.0 mm sieve	%		95.9		95.7		94.9
	12.5 mm sieve	%		96.8		96.6		96.0
	Passing 12.5 mm sieve	%		3.2		3.4		4.0
	Mean coke size	mm		53.9		55.9		54.2
ASTM Coke Tumbler Test	Stability			57.3		53.2		51.7
	Hardness			65.5		64.9		67.1
IRSID Coke Tumbler Test	I10			21.2		23.0		21.8
	I20			77.0		75.1		75.3
	I40			47.4		43.7		40.2
MICUM Coke Tumbler Test	M40			77.0		72.9		70.0
	M10			8.0		8.9		10.0
JIS Coke Tumbler Test	50 mm sieve 30 rev			32.2		33.9		23.4
	25 mm sieve 30 rev			90.6		88.5		87.6
	15 mm sieve 30 rev			94.0		92.5		92.5
	50 mm sieve 150 rev			13.8		4.2		1.6
	25 mm sieve 150 rev			74.8		70.7		67.7
	15 mm sieve 150 rev			81.9		79.3		79.1
Coke Density	ASG		0.983	0.913	0.986	0.910	1.008	0.918
CSR	CSR		67.0	69.2	70.8	66.7	69.9	67.5
CRI	CRI		17.7	16.0	16.0	16.4	17.7	17.7