Light Metals 2013 *Edited by: Barry Sadler*
TMS (The Minerals, Metals & Materials Society), 2013

Calcined Coke Round Robin 19 and the Precision of Bulk Density Tests

Marvin Lubin[1], Les Edwards[1], Lorentz Petter Lossius[2]
[1]Rain CII Carbon, 2627 Chestnut Ridge Rd, Kingwood, TX, 77345, USA
[2]Norsk Hydro ASA, P.O. Box 303, Øvre Årdal NO-6882, Norway

Keywords: Calcined Coke, Anode, Bulk Density

Abstract

Round robins (RR's) are useful for laboratories to benchmark performance against other labs. RR19 was a collaboration between Rain CII, Hydro Aluminium and R&D Carbon and was organized after the special session on coke bulk density arranged by TMS and ASTM at the 2011 TMS Annual Meeting. Five calcined coke samples representing a range of chemical and physical properties were prepared and sent to 28 laboratories around the world. A key objective was to compare the repeatability and reproducibility of different bulk density and apparent density methods. The paper discusses the organization of RR19 and presents a statistical analysis of the following quality parameters: S, V, Ni, Fe, Ca, Si, Na, P, real density, Lc, VBD, TBD and Hg apparent density. In a companion paper, the properties of bench scale and pilot scale anodes produced with the cokes are presented along with correlations to coke properties.

Introduction

This worldwide ASTM round robin (RR) was a collaborative effort between Rain CII Carbon, Hydro Aluminium, and R&D Carbon organized after the 2011 TMS special session on coke bulk density testing. The primary focus of the RR was to examine the repeatability and reproducibility of different bulk and apparent density tests currently used in the industry. Real density, L_c, sulfur, and trace metals were added to the RR to generate additional precision data on these properties. It was the 19th round robin organized by Rain CII and is hereafter referred to as RR19.

A companion paper published in these proceedings [1] reports on the relationship between calcined coke properties and pilot scale anodes produced with the five cokes from RR19. A particular focus is on bulk density correlations and the paper provides some additional background information and references from the 2011 TMS bulk density session [2]. A key problem addressed by this work is the lack of a universally accepted test method for measuring coke bulk density which provides both good repeatability and good reproducibility, and some level of predictability with respect to anode quality. As a result, at least three different coke bulk density tests and specifications are in common use making it very difficult to compare results between laboratories and anode plants.

Round Robin Plan

A wide range of laboratories were invited to participate in RR19 and Table 1 shows the list of 28 labs that participated. There was no cost for participants and RR19 ran from October 2011 to January 2012. A total of five calcined petroleum coke samples with a wide range of properties were sent to each lab.

The coke samples originated from 300 kg lots that were homogenized and divided using rotary sample dividers as shown in Figure 1. A large 150 kg lot was sent to R&D Carbon for bench scale and pilot anode preparation and testing.

Table 1: List of Participating Labs

AJ Edmond - Long Beach	USA
AJ Edmond - Mead	USA
Alcoa Aluminerie Deschambault	Canada
Alcoa- Europe Spain	Spain
Alcoa Lake Charles	USA
Aluminerie Alouette	Canada
BHP Billiton Hillside	South Africa
BHP Billiton Mozal	Mozambique
Boyne Smelters Limited (RTA)	Australia
BP Cherry Point Refinery	USA
BP Europa SE	Germany
BP Wilmington	USA
Dubai Aluminium Company	UAE
Emirates Aluminum Company	UAE
Hydro Aluminium, Ardal	Norway
Hydro Aluminium, Porsgrunn	Norway
Hydro Aluminium, Sunndal	Norway
New Zealand Aluminium Smelters Ltd	New Zealand
Petrocoque	Brasil
R&D Carbon	Switzerland
Rain CII Carbon Lake Charles	USA
Rain CII Carbon Moundsville	USA
Rain CII Carbon Vizag	India
RTA Arvida Reseacrh and Development Center	Canada
RTA Centere-Analytique Vandreuil	Canada
RTA LRF	France
Statoil	Norway
Tomago Aluminium Company	Australia

Figure 1: Rotary splitter used for sample preparation

Each lab was asked to split their coke sample into two and perform the preparation and sample analyses for each split in duplicate. Laboratories were asked to undertake all analyses that they were capable of running using industry accepted standards from ASTM and ISO.

Sample Preparation

Rain CII supplied 3 coke samples, all produced with a rotary kiln:

- Coke A – 100% straight run, low sulfur coke.
- Coke B – 100% straight run high sulfur coke with a highly isotropic structure.
- Coke C – blended coke.

Hydro supplied 2 calcined coke samples:

- Coke S – 100% straight run, low sulfur coke.
- Coke HB – blended coke.

The two blended coke samples were prepared differently. Coke C contained five different green cokes blended prior to calcination. The blend is used routinely by several smelters in North America. The HB blend was generated by Hydro by blending a low sulfur, straight run calcined coke with a US Gulf Coast coke blend. It is typical of the quality used at two Hydro smelters in Norway.

Analytical methods

The tests performed for the RR are summarized below.

Vibrated Bulk Density (VBD)

Traditional VBD equipment requires the use of a vibrating feeder, graduated cylinder and a vibrating table and usually measures a particular size fraction. Multiple crushing and screening steps are required to prepare samples for the VBD tests examined in RR19.

- ASTM D4292-10 – "Standard Test Method for Determination of Vibrated Bulk Density of Calcined Petroleum Coke". Samples were prepared to 28x48 mesh (0.3-0.6mm).

- ASTM D7454-08 – "Standard Test Method for Determination of Vibrated Bulk Density of the 1.17 - 4.7 mm Calcined Petroleum Coke Fraction Crushed to 0.42 - 0.83 mm, using a Semi-Automated Apparatus".

Tapped Bulk Density (TBD)

This test is similar in principal to a VBD test but uses tapping equipment instead of a vibrating table. The method is based on ISO 10236 (1995) utilizing naturally occurring fractions. There is no sample preparation involved other than screening at sizes of:

- 0.25-0.5 mm, 0.5-1 mm, 1-2 mm, 2-4 mm and 4-8 mm. The last four were tested in RR19

GeoPyc - Trans Axial Pressure Mode (TAP)

The GeoPyc method measures bulk density by controlling the force and measuring the displacement of a teflon plunger used to compact the bed of coke, Figure 2. The method has been described previously [3] and an ASTM standard is currently under development. It is important to note that no standard was available for RR19 so each lab selected their own instrument measurement parameters. The GeoPyc equipment can be used for measurement of bulk density using any of the preparation methods in common use. Seven labs with GeoPyc equipment participated in RR19 and reported results for the following preparation methods:

- ASTM D4292 (28x48 mesh), ASTM D7454 (20x35 mesh) and ISO 10236 (0.5-1mm, 1-2mm, 2-4mm and 4-8mm)

Mercury Apparent Density (Hg AD)

Based on the Pechiney Hg AD method [4].

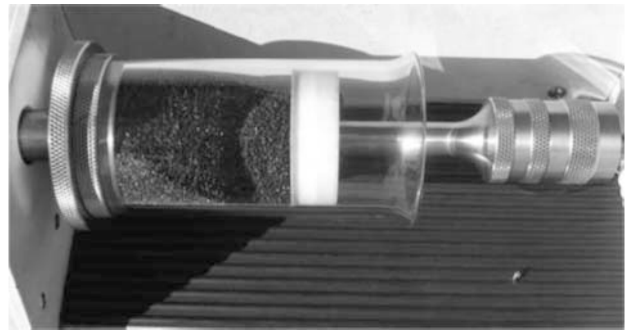

Figure 2: GeoPyc Bulk Density Measurement

Sulfur and Trace Metal Impurities

Sulfur and impurity elements including Na, Si, P, Ca, V, Ti, Mn, Fe, and Ni were analyzed by several methods as follows:

- XRF - ASTM D6376-06 (2010) and ISO 12980 (2000)
- ICP - ASTM D5600-04 (2009) and ISO 14435 (2005)
- AA - ASTM D5056-02 (2007) and ISO 8658 (1997)

Lc: Average Crystallite Height by XRD

- ASTM D5187-91 (2010) and ISO 20203 (2005)

Real Density

Real density was analyzed by xylene, helium pycnometry, or calculated from Lc using the following standards/procedures:

- Helium - ASTM D2638-10 and ISO 21687 (2006)
- Xylene - ISO 8004 (2010)
- Lc – real density calculated from the Lc result using an algorithm based on many comparative analyses.

Round Robin Results

RR19 was a proficiency RR where all participants' results are shown as returned. A set of statistical tools designed for evaluating consistency was used to determine possible outliers. The outliers were removed from the between-lab averages and the standard deviations to make the averages and the standard deviations representative of normal analysis levels and ranges. All precision calculations are according to ASTM E691.

This paper is a summary of the much larger and complete RR reports issued to the participating labs involved [5,6] The RR reports will be submitted as official ASTM reports so anyone will be able to request a copy in the future.

Vibrated Bulk Density - Standard and GeoPyc Comparison

The determination of the bulk density of calcined petroleum coke is an important property because it is an indirect measure of coke porosity which influences anode pitch demand and density.

ASTM D4292

The ASTM D4292 method requires the sample to be crushed and prepared to 28x48 Tyler mesh (0.6-0.3mm) using a specific and rather time consuming procedure. The prepared material is transferred using a vibrating spoon into a graduated cylinder which sits on a vibrating table. There is no industry standard sample to calibrate or check the equipment but the VBD set-up must be in accordance with the D4292 procedure.

A total of 12 labs measured ASTM D4292 VBD's and it is the most widely used test in the industry for coke bulk specifications.

Seven labs that routinely measured VBD's by the D4292 method also had the GeoPyc equipment. All these labs agreed to analyze their "prepared" 28x48 mesh VBD samples on both the standard vibrating table equipment and the GeoPyc equipment. Results are shown in Table II. "Count" = number of labs that provided results but the number varies because outlier results are excluded.

Table II: D4292 Averages for VBD and GeoPyc (g/cm³)

Coke	Methods	Count	Mean	Std. Dev.	Range
A	D4292	12	0.834	0.027	0.080
	D4292 GeoPyc	7	0.831	0.029	0.090
C	D4292	12	0.860	0.025	0.082
	D4292 GeoPyc	7	0.858	0.018	0.053
HB	D4292	11	0.878	0.035	0.108
	D4292 GeoPyc	6	0.875	0.020	0.056
S	D4292	11	0.903	0.044	0.145
	D4292 GeoPyc	6	0.893	0.024	0.067
B	D4292	12	0.993	0.024	0.089
	D4292 GeoPyc	7	0.981	0.022	0.064

Results are shown in Figure 3 for an easy comparison. On average, the GeoPyc results are lower and more repeatable than the D4292 VBD results.

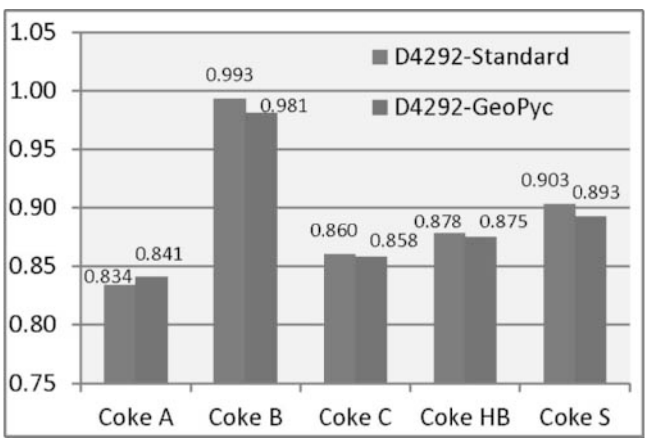

Figure 3: Lab average [g/cm³] D4292 on standard equipment vs. GeoPyc shown with 3 decimals

ASTM D7454

The ASTM D7454 method requires samples to be prepared and crushed to 20x35 Tyler mesh (0.85–0.60mm) and measured using the STAS semi-automated VBD equipment. The sample preparation method is time consuming just like the D4292 test. The STAS equipment uses a photo-electric sensor to detect the coke bed once it reaches the 50-mL mark in a graduated cylinder. This eliminates operator parallax errors reading heights in the graduated cylinder.

Six labs measured VBD by the D7454 method and three of these had the GeoPyc equipment for comparison. All results are shown in Table III. The STAS equipment must be calibrated using coke standards whereas the GeoPyc requires no coke calibration standards.

Although the repeatability is better using the GeoPyc equipment, just as it was for D4292 test, the results for the GeoPyc were slightly higher than the STAS VBD results.

Table III: D7454 Averages for VBD and GeoPyc (g/cm³)

Coke	Methods	Count	Mean	Std. Dev.	Range
A	D7454	6	0.787	0.017	0.044
	D7454 GeoPyc	3	0.794	0.003	0.006
C	D7454	6	0.818	0.015	0.037
	D7454 GeoPyc	3	0.834	0.011	0.022
HB	D7454	6	0.838	0.016	0.040
	D7454 GeoPyc	2	0.848	0.003	0.004
S	D7454	6	0.856	0.020	0.058
	D7454 GeoPyc	2	0.867	0.002	0.002
B	D7454	6	0.949	0.006	0.016
	D7454 GeoPyc	3	0.955	0.003	0.006

ISO 10236

The ISO 10236 TBD method uses a very simple preparation method which requires no crushing and separates the coke into different naturally occurring size fractions by screening. The bulk density of each size fraction is measured by feeding a graduated cylinder connected to a tapping device. Size fractions of 8x4mm, 4x2mm, 2x1mm, and 1x0.5mm were measured in RR19.

A total of seven labs used this method and six labs had the GeoPyc equipment. The TBD results reported in Table IV are the average of all five RR samples for each fraction. The agreement between the TBD and GeoPyc averages was generally excellent.

Table IV: ISO 10236 TBD for Tap and GeoPyc (g/cm³)

Measurement Type	Count	Mean	Std. Dev.	Average Range
8x4 mm TBD	7	0.721	0.015	0.038
8x4 mm GeoPyc	4-6	0.718	0.009	0.023
4x2 mm TBD	7	0.767	0.015	0.038
4x2mm GeoPyc	4-6	0.758	0.011	0.026
2x1 mm TBD	7	0.828	0.018	0.048
2x1 mm GeoPyc	4-6	0.823	0.007	0.016
1x0.5 mm TBD	7	0.873	0.019	0.055
1x0.5 mm GeoPyc	4-6	0.871	0.014	0.037

Apparent Density Using Mercury (Hg AD)

The Pechiney Hg AD test requires samples to be prepared to 10x20 mesh (1.7-0.85mm). Samples are then placed in a pycnometer and subjected to vacuum before Hg is added. This test has now been largely phased out for occupational health and safety reasons and few labs are able to run the test today. Hg AD results for the five RR samples are shown in Table V. It is worth noting that the difference in Hg AD results for the five cokes was only ~2.6% vs 20-35% for the other VBD/TBD methods.

Table V: RR Lab Averages for Hg AD [g/cm³]

Coke	Mean	Std. Dev.	Range
A	1.718	0.021	0.070
B	1.763	0.012	0.035
C	1.724	0.021	0.075
HB	1.723	0.023	0.065
S	1.738	0.009	0.025

VBD, TBD, Hg AD: Repeatability and Reproducibility

Improving the overall precision of bulk density measurements has been a focal point over the last few years. Precision data calculated for the various tests as a result of RR19 are shown below. Repeatability refers to precision within the same lab and reproducibility refers to the precision or agreement between labs.

D4292 - ASTM [g/cm³]

	Documented Precision	RR-VBD Precision	GeoPyc Precision
Repeatability (r)	0.014	0.015	0.014
Reproducibility (R)	0.046	0.087	0.070

The stated reproducibility of ASTM D4292 is poor at 0.046 g/cc, and it was found to be even worse in RR19 (0.087). Anode producers need better certainty of bulk density than ±0.05 g/cm³ when comparing potential coke supplies and evaluating conformance to coke VBD specifications. It was anticipated that there would be improved reproducibility for the D4292 test as a result of revisions made in 2010 but this was not observed. The test is poor for comparing results between different laboratories.

D7454 - ASTM [g/cm³]

	Documented Precision	RR-VBD Precision	RR-GeoPyc Precision
Repeatability (r)	0.0036	0.013	0.014
Reproducibility (R)	n/a	0.043	0.019

Alcan (now RTA) developed this method with a more automated measurement method compared to D4292. The within-lab repeatability is quite good, similar to the D4292 test. The reproducibility is better than the D4292 method but still quite poor overall. The GeoPyc results are much better but the results represent only three labs and are not valid according to E691.

ISO 10236 (1-2mm) [g/cm³]

	Documented Precision	RR-TBD Precision	RR-GeoPyc Precision
Repeatability (r)	0.01	0.015	0.019
Reproducibility (R)	0.02 0.03 (2012)	0.052	0.026

For the most commonly specified 1-2 mm fraction the observed reproducibility was 0.052 g/cm³. In October 2012, ISO revised the reproducibility to 0.03 g/cm³ which is more in line with the observed precision in RR19. For other size fractions, R varied between 0.035-0.054.

The repeatability for the GeoPyc equipment was comparable with the TBD equipment in RR19. The reproducibility was better and should be further improved after development of a standard procedure for setting instrument parameters such as compaction force and number of consolidation cycles that all labs follow.

Apparent Density by Mercury (Hg AD) [g/cm³]

	Stated Precision	RR Precision
Repeatability (r)	0.006	0.023
Reproducibility (R)	0.011	0.051

Similar to the D4292 VBD and D7454 VBD, Hg AD is an analysis that is useful within the same lab but has a larger uncertainty between labs. This makes it difficult to undertake reliable comparisons of cokes when using data from different labs and coke supplies. The spread in Hg AD for these cokes was low making it difficult to distinguish between the five cokes in RR19.

Correlation between VBD/TBD and Hg AD

The five samples used in RR19 represent a wide range of low/high density cokes. Results for all the bulk density methods are summarized in Table VI. Coke C and HB both have the same Hg AD results but a significantly different bulk density using the D4292 and D7454 test methods. Coke A has a similar Hg AD to Coke C and HB but the VBD/TBD methods all show a significantly lower density.

Table VI: Various Bulk Density Results for RR19 Samples

Method [g/cm³]	A	C	HB	S	B
D4292	0.831	0.860	0.878	0.903	0.993
D7454	0.787	0.818	0.838	0.856	0.949
ISO 8x4mm	0.629	0.700	0.702	0.720	0.851
ISO 4x2mm	0.655	0.725	0.762	0.780	0.911
ISO 2x1mm	0.713	0.791	0.829	0.850	0.960
ISO 1x0.5mm	0.770	0.830	0.867	0.898	0.998
Hg AD	1.718	1.724	1.724	1.738	1.763

The Hg AD test was correlated with the various bulk density tests. Correlations were generally good and a correlation matrix for all tests is shown in Table VII. The best correlation between the Hg AD and VBD tests was the ASTMD4292 test with an R^2 of 0.98.

Table VII: Correlation Matrix of Various Density Methods

	D4292	D7454	ISO 8x4 mm	ISO 4x2 mm	ISO 2x1 mm	ISO 1x.5 mm
D7454	1.00	-	-	-	-	-
ISO 8x4mm	0.97	0.98	-	-	-	-
ISO 4x2mm	0.97	0.98	0.97	-	-	-
ISO 2x1mm	0.95	0.96	0.96	0.99	-	-
ISO 1x.5mm	0.97	0.98	0.96	1.00	1.00	-
Hg AD	0.98	0.96	0.93	0.90	0.89	0.91

Sulfur and Trace Metal Results

Sulfur analyses were performed by the following methods: 16 XRF, 2 AA, and 5 unreported. Sulfur is a very critical parameter for both quality and environmental reasons.

Results for the sulfur content are summarized in Table VIII. The average of the reported values ranges from 1.21 to 4.46%, which reflects the wide range of cokes included in RR19. The variability increases significantly as the sulfur content increases, which suggests that some labs have fewer reliable standards for high sulfur cokes since these fall outside the normal range that smelters typically analyze.

Table VIII: Sulfur Content [%]

Coke	Unit	Mean	Std. Dev.	Range
A	%	1.50	0.07	0.35
B	%	4.46	0.24	0.99
C	%	3.07	0.12	0.45
HB	%	1.21	0.09	0.33
S	%	2.13	0.07	0.31

Trace metals analyses were performed by the following methods: 16 XRF, 5 ICP, 1 AA, and 2 unreported. The majority of the RR19 participants analyzed the trace metals in conjunction with sulfur content by XRF. The results are summarized in Table IX.

Table IX: RR Lab Averages for Trace Metals [ppm]

Element	Unit	A	B	C	S	HB
Vanadium	ppm	97	593	392	147	235
Nickel	ppm	181	268	206	68	176
Iron	ppm	176	443	302	75	212
Silicon	ppm	69	155	157	44	255
Calcium	ppm	77	132	99	20	141
Sodium	ppm	49	102	47	35	63
Titanium	ppm	2	15	8	2	5
Manganese	ppm	2	4	3	1	3

An example from the RR19 report [6] for vanadium is shown in Figure 4. It shows how each lab performed on an average basis for all five cokes. The graph highlights the consistency of the results with only three labs being clearly outside one standard deviation.

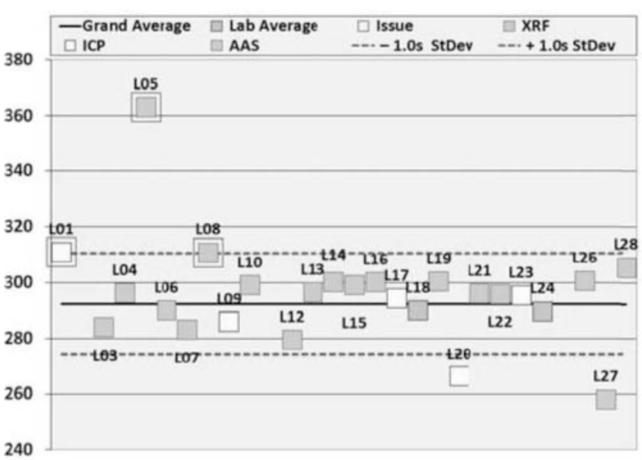

Figure 4: RR Vanadium Averages (ppm) by Lab; 25 labs, "Issue" means less than 5 cokes or 5 duplicates measured

Similar to sulfur, the variability of the trace metals (V, Ni, Ca, Si, Fe) increased significantly at higher concentrations and labs should consider acquiring a more extensive set of calibration standards.

Real Density

Real density is used as a measure of calcination level and results are shown in Table X. Real density is measured on a sample ground to -200 mesh (-75 µm) and the density is measured using helium or xylene as the displacement media. The overall reproducibility between the 25 labs was acceptable, but there were some issues between labs with Coke B which is a relatively hard and highly isotropic, low RD coke.

Table X: Lab Averages for Real Density [g/cm³]

Coke	Count	Mean	Std. Dev.	Range
A	25	2.078	0.008	0.029
B	25	1.995	0.012	0.116
C	25	2.065	0.007	0.029
HB	24	2.067	0.009	0.034
S	24	2.065	0.007	0.029

The greater variability and range of coke B is shown in Figure 5. Three labs calculated RD's based on an Lc-RD algorithm and these are the high results shown with green markers. This method is clearly not suitable for isotropic cokes with significantly lower RD's. These lab results and the Lab 14 results are excluded from the average and standard deviation data for coke B in Table X.

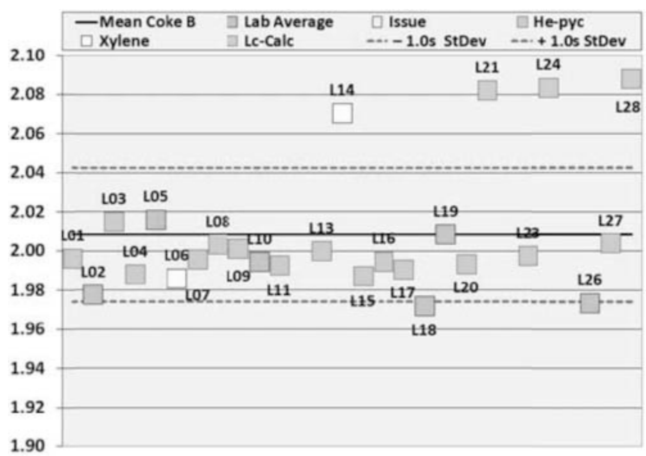

Figure 5: RD lab averages for Coke B (Blue = not known); 25 labs, "Issue" means less than 5 cokes or 5 duplicates measured

Lc, average crystallite height

Lc is a measure of the average crystallite size and is directly proportional to the heat treatment the coke receives during calcination. All Lc results were performed using an XRD method and are shown in Table XI.

Table XI: RR average result for L_c [Å]

Coke	Mean	Std. Dev.	Range
A	30.4	0.5	1.6
B	30.8	1.0	3.2
C	27.7	0.7	2.2
HB	29.3	0.7	3.8
S	27.4	0.5	2.1

For sponge cokes, there is usually a good correlation between RD and Lc. The correlation for the five cokes in RR19 was poor however with an R^2 value of 0.25. Data are shown in Table XII. When the isotropic coke was removed (coke B) the R^2 value increased to 0.77.

Regarding precision, the r&R observed in RR19 for real density by He-pycnometry was 0.007 and 0.017 g/cm³, fairly close to the standard's r&R of 0.005 and 0.013 g/cm³. For Lc, the r&R

observed in RR19 was 0.9 and 2.2 Å, somewhat higher than the standard's r&R of, 0.5 and 1.9 Å.

Table XII: RD and L_c relationship

Coke	RD Average	L_c Average
A	2.078	30.4
B	1.995	30.8
C	2.065	27.7
HB	2.067	29.3
S	2.065	27.4

Discussion and Conclusions

Overall, the results for the five samples in RR19 showed reasonable consistency for tests other than bulk density. The isotropic nature and greater hardness of coke B drove some additional variation in RD results. The agreement between labs for sulfur and vanadium also deteriorated at higher S and V levels and this is believed to be due to a lack of suitable calibration standards. The detailed RR19 report [6] shows a greater spread in results for trace metals like Si, Ca, Fe and Ni at higher concentrations and this is also believed to be due to a lack of calibration standards by some labs at higher concentration levels.

RR19 participants were able to analyze both the blended and single source coke samples with the same level of precision. Sulfur analysis by four different methods showed good precision among all labs, with only two labs being significantly outside of one standard deviation.

Most labs are set up to measure bulk density using one method, and only two labs were able to run all bulk density tests. The VBD/TBD precision statements in the current standard procedures seem optimistic based on the RR19 results but that is likely because some labs are not following procedures exactly as written including the use of non-standard equipment.

The above is not a new finding but it highlights the difficulty of running bulk density tests with complicated sample preparation procedures. For most tests, the within lab repeatability was much better than the between lab reproducibility. Sample preparation differences are clearly driving most of the variation between labs.

The ISO TBD test eliminates the difficult sample preparation steps involved with the two ASTM VBD tests and it offers significantly better precision than the ASTM D4292 test and about the same level of inter-lab precision as the semi-automated ASTM D7454 method – at least in the RR19 study. The GeoPyc equipment improves the within-lab precision for most of the bulk density tests.

The within-lab repeatability for measuring the bulk density on naturally occurring size fractions with the GeoPyc equipment showed the best overall repeatability in RR19. This is perhaps not surprising given the fully automated nature of the equipment. The sample can be poured into the measuring chamber without the need for special vibrating feeders and the measurement is fully automated after this.

Based on this, the ASTM committee has requested that a new bulk density procedure be developed using naturally occurring size fractions and the GeoPyc equipment. The procedure will be multi-faceted and will allow measurement of naturally occurring size fractions like the ISO 10236 test or samples prepared using the ASTM D4292 or D7454 preparation methods. The results presented in RR19 show that the equipment is versatile enough to use with most sample preparation methods.

In the companion paper to this one [1], none of the bulk density tests or the Hg AD test stood out as being any better than the other for predicting baked anode densities. On the other hand, all the tests showed a strong correlation with optimum pitch level. Based on this, it seems reasonable for the industry to settle on bulk density tests with the best overall precision – particularly for cross-lab comparisons.

When looking at the other tests in RR19, the precision for sulfur, trace element impurities, Lc and RD are all acceptable, notwithstanding previous comments about high S and trace metals levels and calibration standards. The only caveat to this is the calculation of RD from Lc results. This is not a recommended practice for any cokes or coke blends containing isotropic structures. Labs also need to take care with crushing and grinding harder cokes like coke B. This can contribute to additional iron contamination if non-tungsten carbide grinding equipment is used. Care also needs to be taken to ensure that real density samples are ground to 95% passing 200 mesh (75μm). Grinding times may need to be adjusted for harder cokes.

Recommendations

Once the ASTM committee tentatively approves a new bulk density procedure as outlined above, a new RR will be initiated using prepared samples for most of the preparation methods, including 28x48, 20x35 and screened, naturally occurring fractions so that they can all be included in the ASTM precision statement.

More labs are encouraged to participate in RR studies like this in the future so that consistency can be improved throughout the industry. ASTM started a new Proficiency Test Program in 2012 known as the ILS program. They will conduct industry wide round robins every 6 months with two calcined and two green coke samples as long as enough interest remains.

References

1. M. Lubin, L. P. Lossius, L. Edwards and J.Wyss, "Relationships Between Coke Properties and Anode Properties – Round Robin 19," *Light Metals*, 2013

2. Petroleum Coke VBD Special Session, *Light Metals,* 2011, 925-963

3. M. Lubin, L. Edwards and J. Marino, "Improving the Repeatability of Coke Bulk Density Testing," *Light Metals*, 2011

4. R. Barral, "Coke Apparent Density by Mercury Pycnometry," Aluminium Pechiney Standard Procedure, 1999, A.07.11.V06

5. Lorentz Petter Lossius and Marvin Lubin, "ASTM-RCII-RDC-Hydro 2011 - Petroleum Coke Round Robin RR19; Bulk Density and Hg Apparent Density", Report distributed to RR19 participants June 21st 2012

6. Lorentz Petter Lossius and Marvin Lubin, "ASTM-RCII-RDC-Hydro 2011 - Review of Petroleum Coke Properties Measured in RCII RR19; RD, Lc, S and Metals", Report distributed to RR19 participants March 27th 2012