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STANDARDIZATION OF A CALCINED COKE BULK DENSITY TEST

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Abstract

One of the major factors affecting Hall cell anode performance is the porosity of the calcined coke used in its manufacture. Relatively simple, rapid, and inexpensive bulk density tests can give a good indication of coke porosity. However, without an industry-wide standard test, comparison of coke bulk density results among various suppliers and users is meaningless. A standard test is being developed under the sponsorship of ASTM Committee D-2 on Petroleum Products and Lubricants. This paper describes some of the bulk density tests currently used and their shortcomings. Effects of crushing equipment and strategy are shown. In most coke bulk density tests, mechanical action such as vibration is used to increase sample compaction. Selection and calibration of a vibrator and ancillary equipment are discussed. Efforts to reduce test result differences due to operator variability are described also.

Introduction

Porosity is a major factor in determining suitability of a calcined coke for Hall cell anode use. Porosity can be measured by a number of techniques: absorption of mercury, water, or other liquids, or penetration of inert gases. These techniques require expensive equipment, are limited to a small sample size, and/or are time-consuming.

A simpler indicator of coke porosity is bulk density. This involves measuring the volume of a weighed, sized coke sample, usually after vibration to improve compaction. Recent work (1) has indicated that porosity of a certain pore diameter range (1-10 μm) is particularly significant to carbon anode consumption efficiency. However, quite satisfactory correlations have been made (2, 3, 4) between important anode properties and simple vibrated bulk density tests, which give an indication of total porosity.

A bulk density value for a calcined coke is not absolute. It depends on the specific conditions selected. A number of bulk density tests are presently in use by coke suppliers and carbon and graphite electrode manufacturers. They differ in particle size range of the test sample, method of compaction, and other critical aspects. Values reported for a given coke lot vary according to the method used. Often the method used is not reported at all in suppliers' data sheets; i.e., bulk density is treated as an absolute number. These factors tend to make density comparisons for various coke sources relatively meaningless.

To eliminate ambiguity in calcined coke bulk density measurement, Alcoa requested in 1978 that a standard ASTM calcined coke bulk density test relevant for the aluminum industry be developed. This is being done under the sponsorship of ASTM Committee D-2 on Petroleum Products and Lubricants. More specifically, a test is being developed in Research and Development Division V on Physical Analysis of Fuels and Light Distillates, Section D - Petroleum. In short form, this is expressed as ASTM D02.RDDV.D. The author is chairman of the bulk density study group of this section. This group has consisted of about ten people representing aluminum producers, carbon and graphite suppliers, and coke producers.

Background on Coke Bulk Density Tests

Although a calcined coke bulk density test is simple, a basic understanding of the principles involved is essential to the development of a useful test. It must be understood that the test result is influenced by particle size and shape as well as porosity and inherent density (real density) of the material being tested. If particle size is not closely controlled, ill-defined values will be obtained. For example, fine particles contained in interstices among coarse particles will add weight without increasing volume. Hence, a bulk density value for a given coke can be increased greatly by changing from a closely sized coarse particle network to a broad particle size distribution.

Moroever, it has been shown (5) with closely sized fractions, bulk densities of calcined petroleum cokes tend to increase with decreasing particle size (Figure 1). This is because such cokes usually contain pores and fissures of a variety of sizes. As large pores are annihilated on reduction to smaller particle sizes, density increases.

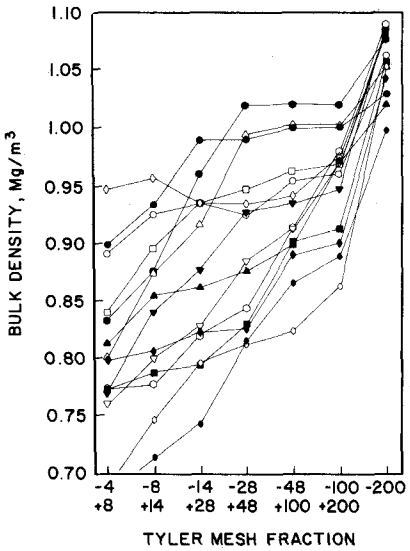

Figure 1. Vibrated Bulk Density vs. Particle Size for a Number of Calcined Cokes That Have Been Used in Anodes for Aluminum Smelting.

Another factor influencing bulk density is particle shape. Spherical particles will pack tighter than platelike or needlelike particles. They will give a higher bulk density even when inherent density and particle porosity are identical. Variation in test results due to particle shape is a disadvantage of a bulk density test. However, if the method of size reduction is controlled, particle shape will be reasonably similar for cokes typically used in anodes for aluminum manufacture. Admittedly, there could be a bulk density bias between such cokes and the needlelike cokes used in graphite manufacture.

Even with a well controlled particle size and shape, bulk density values vary according to the amount of agitation of the particles during and after pouring into the measuring container. Several tests do not require mechanical agitation, but most bulk density tests specify agitation, such as vibration. Obviously, there is a case to be made for non-agitation methods with respect to simplicity, reduction of equipment calibration, and lower equipment cost.

However, the disadvantages are outweighed by factors favoring mechanical agitation. First is the observation that with a properly calibrated mechanical agitator, variations due to operator technique in filling the measuring container are reduced; i.e., the test tends to be more reproducible. Secondly, density values are higher with mechanical agitation. Forming of pitch-bonded carbon artifacts by pressing, extruding, or vibrating tends to densify the aggregate network. Therefore, the higher bulk density values produced by mechanical agitaton are more representative of the density the aggregate would have in a pitch-bonded artifact.

Alcoa's Bulk Density Tests

The effect of coke porosity on carbon anode performance has been recognized at Alcoa smelters for many years. Early on, a commonly used bulk density test was the "Cubic Foot Weight of Coke" [ANSI/ASTM-D-292-29 (Reapproved 1978)]. In this test, a run-of-kiln coke sample is placed in a cubic foot box, the top of the sample is leveled, and the box weighed. With increased sophistication in anode manufacture and use over the years, it became obvious that this test did not satisfactorily characterize coke shipments. Because particle size is not specified (except for a 5 in. maximum), this test gives only a qualitative or, at best, semi-quantitative indication of coke density or porosity. For this reason, improved tests were sought.

Improved procedures for bulk density determinations evolved within Alcoa over a number of years. Much of the basis for particle size selection was given in a paper presented at the 103rd AIME Annual Meeting in 1974. Logically, a dry blended aggregate having the particle size distribution to be used in anode manufacture would seem to be the ideal sample for determining quality of a coke based on bulk density. However, this requires considerable crushing, sizing, and blending. Moreover, a single setting on a vibrator dial does not necessarily compact different blends to the same extent. With blended aggregates, a rather lengthy procedure involving several changes in vibrator dial setting resulted in greatest compaction.

For these reasons, an attempt was made to find simpler, close-cut particle size ranges that would correlate well with dry blended aggregate densities. For the cokes shown in Figure 1, best correlation was with the -28+48 mesh fraction. Correlation with coarser fractions was undoubtedly poorer because many of the cokes contained large pores or fissures that were eliminated during moderate size reduction. Typically, relatively few large coke particles are used in a prebaked anode aggregate since crushed butt additions are used to furnish most of the coarse particles. Correlation is poor with fine particle bulk densities because these don't reflect the density contribution of the large pores in large and intermediate size particles.

A typical Soderberg anode aggregate is considerably coarser than a prebaked anode aggregate, and butts are not available to replace coarse coke particles. For this reason, it might be expected that a coarser fraction than -28+48 mesh would be a better indicator of overall coke quality for this purpose. A correlation similar to that reported in 1974 (5) was later made using a coarse Soderberg sizing and the close-cut fractions shown in Figure 1. In this case, the bulk density of the -8+14 mesh fraction correlated best with dry blended aggregate density (Figure 2). Since Alcoa's smelting cells are predominantly of the prebaked anode type, the -28+48 mesh bulk density is standard.

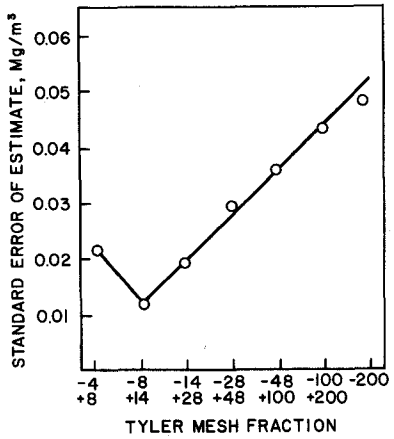

Figure 2. Standard Errors of Estimate Derived from Correlating Close-Cut Fraction Vibrated Bulk Densities of a Number of Calcined Cokes (cf Figure 1) with Vibrated Bulk Densities of Dry Blended Coke Aggregates Having a Typical Soderberg Anode Sizing: 32 wt % +8 Mesh, 12% -8+28 Mesh, 10% -28+100 Mesh, 46% -100 Mesh.

A second consideration concerning bulk density testing was method of compaction. Several commercial vibrators or joggers are available. A Syntron Model J-IA was selected for its availability and reasonably low cost. In recent years, the Model J-IA jogger has been superseded by Model J-IB. These vibrators operate at the frequency of the input alternating current (60 Hz in the U.S.A.) with an amplitude varied by a dial setting. Figure 3 gives an example of the effect of dial setting on the vibrated bulk density of a coke sample. With a Model J-IB jogger, a

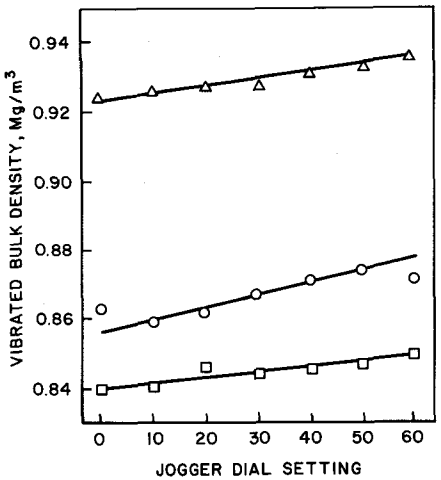

Figure 3. Effect of Jogger Dial Setting (Syntron Model J-IB) on Vibrated Bulk Density Values for Three Cokes.

setting of "35" was selected as standard ("5" with a Model J-1A jogger). This did not result in the highest possible density, but the action of the jogger is excessive at higher dial settings. Higher densities resulted when the graduated cylinder used in determining the volume of the coke sample was not clamped to the jogger table but was allowed to bounce freely on the table. A cork ring is used to prevent the graduate from "walking" off the table.

With other Syntron Model J-IA and J-IB joggers, the same dial setting on different joggers resulted in different bulk density values for a given coke sample. This was quantified in terms of vibration amplitude with an IRD Model 403 Non-Contact Measuring system. This is a proximity transducer that targets on a disc of SAE-4140 steel that is attached to the jogger table. A number of procedures were tried to make the joggers vibrate consistently. The most satisfactory solution was removal of the jogger table, setting the magnetic core to touch the armature (with the aid of an ohmmeter for detection), and winding out the core a fixed number of turns. This produced amplitudes in a reasonably narrow range for a given dial setting. Figure 4 gives an example of vibration amplitude versus dial setting for four Syntron J-IA joggers with three core settings. Since this bulk density test had been carried out in the laboratory with the original jogger for some time prior to discovery of the variation in as-received joggers, a core adjustment (1-3/8 turns out) that gave the new joggers the same characteristics as the original jogger was arbitrarily selected.

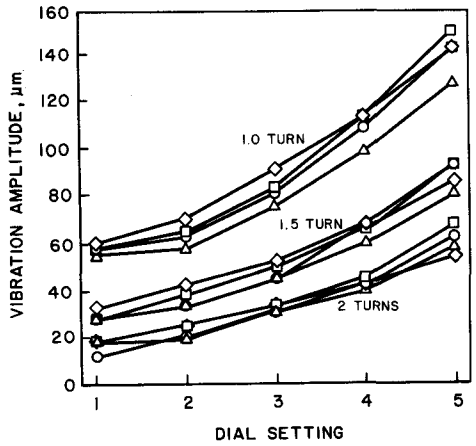

Figure 4. Static Adjustment of Syntron Model J-IA Joggers. (Core Turned in Until Touching Armature, Then Backed Off 1.0, 1.5, and 2.0 Turns.)

Some of the reported vibrated bulk density tests require vibration of the measuring cylinder as the coke sample is added. However, we opted for the procedure of pouring the coke into a graduated cylinder without vibration, then vibrating for a fixed time. For the same jogger dial setting, both methods gave nearly equivalent results, which were considerably higher than without vibration (Figure 5).

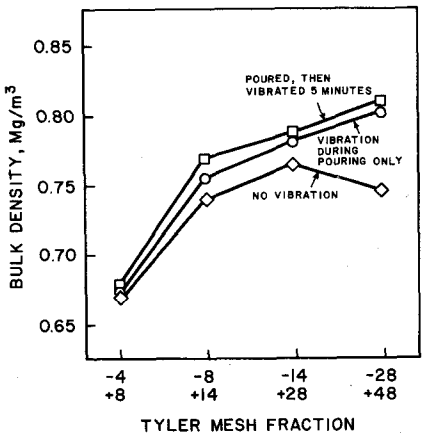

Figure 5. Effect of Vibration on Bulk Densities of Coke Fractions (Syntron Model J-IA Jogger, Dial Setting "5").

It was found, subsequently, that the rate of pouring the coke sample into the graduate effects the bulk density value, even after vibration. For example, Table I shows results, after vibration, of "dumping" coke samples into the graduate and pouring slowly in about 90 seconds. Vibrated bulk densities averaged 0.03 Mg/m^3 higher with the slow pour. (Before vibration the differences averaged 0.05 Mg/m^3, so vibration reduced this technique variable.)

Table I. Effect of Rate of Pour of Coke into Graduated Cylinder on Vibrated Bulk Density

	Vibrated Bulk Density, Mg/m^3	
	Fast Pour	Slow Pour
Coke 1	0.993	1.028
Coke 2	0.898	0.927
Coke 3	0.835	0.867
Coke 4	0.810	0.843

Note: Fast Pour -- Coke "dumped" into graduate
Slow Pour -- Coke uniformly poured into graduate in about 90 seconds

Because of this pouring technique effect, "self-feeding" through a funnel having a small orifice is being considered at Alcoa as an optional method for determining bulk density of the -28+48 mesh fraction. For this fraction, a standard filtration funnel (75 mm top ID, 58° angle, 150 mm long stem, 4.5 mm stem ID) affixed to the top of the graduate through a rubber stopper is used to self-feed the coke with the jogger activated. If the jogger is stopped a few seconds after the entire coke sample is in the graduate, the density value is close to that with slow manual pouring. (Additional vibration with the entire coke sample in the graduate results in higher density values.)

Other significant factors involving the compacting and measuring aspects of vibrated bulk density testing are graduated cylinder volume calibration and weight. Some graduated cylinders are as much as 2 mL in error at typical measuring levels. Calibration with water is essential for new graduates. More subtly, weight on the jogger table affects vibration characteristics. Hence, cutting off the tops of graduates to produce a target weight is a standard practice.

The discussion of Alcoa's bulk density testing has, to this point, been limited to test variables starting with a crushed and sized coke sample. Sampling, crushing, and sieving variations can influence test results significantly. At this time, the procedures do not include sampling specifications. As a guideline, it is recommended that ANSI/ASTM D346-78 be consulted in preparing a representative sample suitable for particle size reduction. ASTM D02.RDDV.D is currently considering the matter of calcined petroleum coke sampling for chemical and physical analyses.

In many lots of "run-of-kiln" coke there are significant quantities of "natural" -28+48 mesh particles. However, screening these particles from the run-of-kiln sample is not a satisfactory method for preparing a vibrated bulk density test sample. Table II exemplifies the importance of sample preparation on vibrated bulk density results. It shows densities of -28+48 mesh fractions prepared in different ways from a single sample of run-of-kiln coke. The first value is for a "natural" -28+48 mesh fraction. Density of this fraction was low, presumably because soft, highly porous particles are most likely to break into small pieces during coke handling.* The next value is for material that was lightly crushed; i.e., most of the coke was not crushed as finely as -28+48 mesh. The next density is for a sample crushed so that about as much

Table II. Effect of Method of Obtaining -28+48 Mesh Particles on Vibrated Bulk Density of a Coke Sample

Method of Obtaining -28+48 Mesh Fraction	Vibrated Bulk Density, Mg/m^3
"Natural" particles screened from run-of-kiln sample	0.799
Crushed so that most coke particles remained coarser than -28+48 mesh	0.872
Crushed so that nearly equal amounts of particles were coarser and finer than -28+48 mesh	0.880
Crushed so that most coke was finer than -28+48 mesh	0.893

* This result of a lower vibrated bulk density for "natural" particles relative to particles prepared by crushing is reportedly not universally true. Others experienced in this field have reported to the author that higher values are found for natural particles for some coke lots.

coke coarser and finer than -28+48 mesh was produced. The last value is for a sample crushed so that most of the product was finer than -28+48 mesh. Density increased as more harder, less porous coke was included in the -28+48 mesh fraction. To minimize this variable, a crushing procedure designed to give about equal amounts of coke coarser and finer than -28+48 mesh was devised.

A jaw crusher and roll crusher were selected as standard for preparing calcined coke fractions for vibrated bulk density testing. After preliminary jaw crushing, a high on-size product yield with minimal trial-and-error is produced using a "feeler gauge" to set the roll gap. Generally, a setting equal to the openings in the coarser of the two screens defining the bulk density fraction (28 mesh, in this case) gives a relatively high on-size yield and roughly equal amounts of coarser and finer particles. Figure 6 shows examples of yields with different roll gap settings.

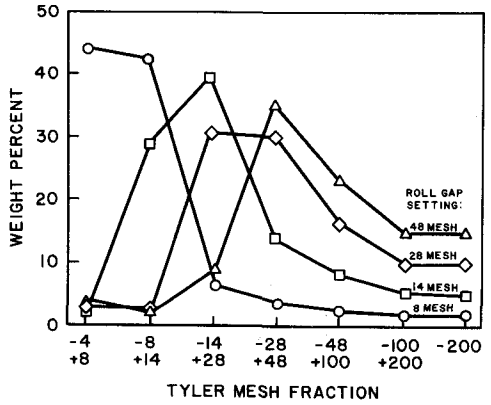

Figure 6. Yields of Coke Fractions Obtained Using Various Roll Gap Distances (Expressed as Equivalent Screen Openings).

Different crushing or grinding machines can produce particles resulting in different vibrated bulk densities from those of particles produced with a roll crusher, probably due to differences in particle shapes. For example, vibrated bulk densities of cokes reduced to -28+48 mesh with a Braun pulverizer were 0.03 Mg/m^3 higher, on the average, than densities of roll crushed -28+48 mesh fractions.

Other Calcined Coke Bulk Density Tests

As part of the ASTM calcined coke bulk density test development, other parties were solicited for bulk density test procedures. Table III summarizes tests received by the study group. ASTM D292-29 was described earlier. Since it includes no particle size range, it is, at best, a semi-quantitative indicator of coke porosity.

Table III. Summary of Methods for Determination of Bulk Density

	ANSI/ASTM D292-29	Alcoa	Kaiser	Collier	Great Lakes (1970)	Great Lakes No. 144A	ASTM B527-70*	DIN 53 194**
Coke particle size	5 in. max.	-28+48 mesh	-8+14 mesh	-20+35 mesh	-4+6 mesh	-20+48 mesh	not specified*	not specified**
Method of compaction	none	Syntron Model J-1A Jogger (60 cycles/s)	Syntron J-1A or Cleveland Electric Table	Syntron Model T-1A Jogger	none (special pouring funnel and technique used)	Syntron Model J-1A	Tap-Pak Volumeter (~4.5 taps/s)	Tap-Pak Volumeter (~4.5 taps/s)
Amplitude	----	~95 μm (graduate not clamped to jogger table)	~635 μm?? (probably 318 μm)	"low vibration intensity"	----	not specified (action controlled with ammeter)	~1600 μm	~1600 μm
Sample preparation	no size reduction	roll crusher; Ro-Tap	not specified	roll crusher; Ro-Tap	no crushing	roll crusher; Ro-Tap	not specified*	not specified**
Sample size	one cubic foot	100 g (after crushing and screening a +500 g sample)	100 g	50 cm^3	325 g (or 250 g)	100 g	50 g (or 20 g)	200 cm^3 (weighed)
Possible objections	particle size not controlled	particle size	particle size; vibration amplitude may cause particle degradation	particle size; high degree of operator skill seems necessary; vibration amplitude not specified	particle size; uncompacted density lower than compacted density; fair degree of operator skill seems necessary	particle size; vibration amplitude not specified	no particle size specified; tapping probably results in lower density than vibrating; Tap-Pak ~10 times cost of Syntron J-1A Jogger	same as ASTM B527-70

* Test is for "refractory metal powders and compounds"
** Test is for "pigments and other powdered or granulated materials"

The Kaiser test uses a relatively coarse particle size, -8+14 mesh. As mentioned earlier, this fraction characterizes cokes well for Soderberg anode application. A vibrator similar to that used at Alcoa is specified; vibrator amplitude is reported in the furnished test procedure to be ~635 μm. Even at half this amplitude, by our measurement, vibrator action was judged to be unnecessarily severe. Another shortcoming is lack of specification of a sample preparation procedure.

The Collier test (via Alcan) specifies a fraction similar to that used by Alcoa. A shortcoming of the test is lack of specification of a precise crushing procedure. Also, coke is added to the measuring cylinder with a vibratory spatula; it appears from the procedure that a considerable amount of operator skill is required for consistency. Finally, the vibration amplitude is not precisely specified.

A Great Lakes (1970) procedure involves a -4+6 particle size range and no mechanical agitation. As described earlier, it has been shown that with a particle sizing this coarse, results can be misleading. Also, without mechanical agitation operator pouring technique has a greater effect on test results than with agitation.

Great Lakes Method No. 144A is similar to the Alcoa test, although a broader fraction, -20+48 mesh, is used. Also, the proper jogger setting is selected with the aid of an ammeter; we believe the core setting procedure is better.

Two tests, ASTM B527-70 and DIN 53-194 (which are not intended for calcined cokes, but were submitted to the study group), utilize a Tap-Pak Volumeter (distributed in the U.S.A. by Shandon Scientific Company, Inc.). This is a low frequency, high amplitude device in which motion is produced by a revolving cam. It is likely that motion is more uniform from one machine to another, compared with the coil and armature arrangement of the Syntron joggers or other vibrators. However, with proper calibration of the joggers, this disadvantage should be minimal. Furthermore, at the time of development of the Alcoa test, cost of the Tap-Pak Volumeter was approximately ten times that of a Syntron Model J-IA jogger. We felt that the potential gain in machine uniformity did not warrant the cost. Apart from the method of compaction, these two tests do not specify particle size, which would make them only semi-quantitative for coke bulk density measurements. Nor is crushing equipment or procedure specified. In the case of test ASTM B527-70, sample weight must be changed, depending on the density of the material. The other test uses a sample size based on the volume of uncompacted material, rather than on weight. Both of these methods of sample size selection seem overly complicated.

Selection of a Test Method for Interlaboratory Testing

The key variable in the tentative ASTM test for calcined coke bulk density was judged to be particle size. After considerable discussion, the section could not agree upon a particle size range. Data show that density of a mid-size fraction such as -28+48 mesh is most appropriate for evaluating coke for prebaked anode manufacture. The Alcoa, Great Lakes (Method No. 144A), and Collier methods use fractions of this sort. However, for coarser aggregates more typical of Soderberg anodes a coarser fraction such as -8+14 is more appropriate; therefore, this type of size range had some support.

In addition, graphite-grade coke producers and graphite manufacturers were keenly interested in a new standard bulk density test. Although no supporting data were presented, opinions were expressed that a relatively coarse fraction is most suitable for characterizing coke for this application.

In view of this lack of consensus, it was proposed that the test procedure include any fraction that is retained between screens having openings differing by no more than $2\sqrt{2}$ (preferably, by no more than $\sqrt{2}$) and particle size range must be specified when reporting results. An additional constraint is that maximum size of the coarsest particles that can be tested is -4 mesh and minimum size of the finest particles is +28 mesh.

It is intended that the final version of the test procedure will include the statement that the standard fraction for coke to be used in prebaked anode manufacture for aluminum production is -28+48 mesh. This is the preponderant application for electrode grade coke in the U.S.A. Without this proviso, confusion in bulk density results for cokes to be used in prebaked anodes will continue.

Regarding other test conditions, the Alcoa procedure was accepted with little opposition. As mentioned earlier, there are advantages to self-feeding the coke test sample into the measuring cylinder through a funnel. However, the decision to generalize the particle size range would necessitate the use of a number of funnels of different sizes. We were unable to locate suppliers of funnels having a suitable gradation in stem ID's. Hence, the procedure of manual pouring of coke into the measuring cylinder, followed by vibration, was tentatively adopted.

Interlaboratory Test Results

An interlaboratory test series, starting with already crushed and sized samples furnished by Great Lakes Research Corporation, was completed in mid-1980. Fourteen samples (seven cokes, each at two particle size ranges: -4+6 and -28+65 mesh) were tested at each of seven laboratories. Results are given in Tables IV and V. Repeatabilities were excellent. Reproducibilities were higher, but were considered acceptable by the section members.

A second test series was designed to determine the effect of the crushing and sizing procedures on test reproducibility. For eight cokes, both run-of-kiln samples and precrushed and sized (-20+48 mesh) samples were tested. A total of ten laboratories participated in this test series. Four laboratories tested the run-of-kiln samples only, three laboratories tested precrushed and sized -20+48 mesh samples only, and three laboratories tested both (coded, so that the corresponding pair for a given coke was unknown).

Results are given in Tables VI and VII. For the precrushed and sized samples, reproducibility was 0.042 Mg/m^3, quite similar to that for the first test series using a similar fraction (-28+65 mesh). With inclusion of the crushing and sizing steps, reproducibility was higher, 0.066 Mg/m^3. However, if the results for one sample, ST-299, are excluded, reproducibility improves to 0.050 Mg/m^3. Hence, the crushing and sizing steps added relatively little variability.

Table IV. Results of Coke Bulk Density Interlaboratory Test Series Completed in Mid-1980 (-4+6 Mesh)

Coded Laboratory Designation	VIBRATED BULK DENSITY, Mg/m^3 Sample No.: YT-40	DT-71	OT-361	JT-11	ST-98	OT-328	PT-120
A	0.734	0.738	0.544	0.762	0.643	0.581	0.740
	0.728	0.742	0.549	0.764	0.647	0.585	0.736
B	0.725	0.725	0.532	0.752	0.629	0.581	0.720
	0.725	0.720	0.538	0.758	0.633	0.581	0.725
C	0.734	0.730	0.544	0.753	0.647	0.582	0.741
	0.733	0.735	0.546	0.756	0.643	0.575	0.739
D	0.718	0.716	0.520	0.740	0.649	0.568	0.731
	0.717	0.713	0.524	0.736	0.645	0.576	0.728
E	0.734	0.742	0.547	0.764	0.661	0.623	0.743
	0.730	0.742	0.532	0.762	0.655	0.624	0.743
F	0.723	0.723	0.542	0.754	0.644	0.597	0.733
	0.723	0.726	0.550	0.754	0.646	0.598	0.732
G	0.728	0.733	0.537	0.761	0.647	0.593	0.729
	0.731	0.735	0.546	0.746	0.646	0.590	0.740
AVERAGE	0.727	0.730	0.539	0.754	0.645	0.590	0.734

REPEATABILITY* = 0.010 Mg/m^3
REPRODUCIBILITY** = 0.030 Mg/m^3

Table V. Results of Coke Bulk Density Interlaboratory Test Series Completed in Mid-1980 (-28+65 Mesh)

Coded Laboratory Designation	VIBRATED BULK DENSITY, Mg/m^3 Sample No.: YT-40	DT-71	OT-361	JT-11	ST-98	OT-328	PT-120
A	0.906	0.885	0.744	0.898	0.823	0.788	0.872
	0.903	0.893	0.756	0.901	0.821	0.783	0.866
B	0.885	0.848	0.725	0.862	0.782	0.752	0.848
	0.885	0.862	0.725	0.862	0.788	0.758	0.855
C	0.893	0.877	0.738	0.873	0.801	0.775	0.858
	0.895	0.877	0.738	0.874	0.811	0.779	0.858
D	0.877	0.855	0.710	0.863	0.787	0.751	0.828
	0.876	0.857	0.714	0.866	0.789	0.749	0.831
E	0.892	0.883	0.742	0.885	0.815	0.779	0.857
	0.892	0.880	0.749	0.881	0.821	0.774	0.864
F	0.885	0.869	0.725	0.869	0.795	0.763	0.848
	0.878	0.869	0.725	0.870	0.795	0.763	0.847
G	0.893	0.875	0.714	0.879	0.794	0.771	0.848
	0.894	0.876	0.719	0.876	0.799	0.768	0.852
AVERAGE	0.890	0.872	0.730	0.876	0.802	0.768	0.852

REPEATABILITY = 0.009 Mg/m^3
REPRODUCIBILITY = 0.043 Mg/m^3

*Repeatability = $\sqrt{\text{Replicates Mean Square}}$ x Probability Multiplier

**Reproducibility = $\sqrt{\text{Repeatability Variance + Lab Variance + Sample Variance}}$ x Probability Multiplier

as in Manual on Determining Precision Data for ASTM Methods on Petroleum Products and Lubricants (RRD-2-1007)

Table VII. Results of Coke Bulk Density Interlaboratory Test Series Completed in Mid-1981 (-20+48 Mesh, Crushed and Sized By Participants)

Coded Laboratory Designation	VIBRATED BULK DENSITY, Mg/m^3 Sample No.: ST-585	ST-258	ST-395	ST-298	ST-360	ST-586	ST-299	ST-396
A	0.853	0.811	0.801	0.900	0.917	0.846	0.903	0.789
	0.859	0.811	0.801	0.898	0.919	0.844	0.909	0.789
B	0.820	0.781	0.758	0.870	0.885	0.820	0.943	0.769
	0.833	0.775	0.746	0.870	0.885	0.813	0.926	0.763
C	0.833	0.777	0.769	0.886	0.897	0.833	0.896	0.774
	0.834	0.777	0.769	0.887	0.895	0.833	0.896	0.774
D	0.849	0.796	0.786	0.860	0.891	0.792	0.894	0.777
	0.828	0.808	0.786	0.875	0.901	0.789	0.895	0.784
F	0.825	0.774	0.769	0.862	0.895	0.810	0.861	0.765
	0.823	0.778	0.769	0.863	0.890	0.811	0.862	0.756
I	0.836	0.852	0.795	0.878	0.883	0.811	0.814	0.801
	0.838	0.843	0.788	0.880	0.882	0.815	0.809	0.789
J	0.853	0.824	0.800	0.911	0.927	0.845	0.921	0.795
	0.848	0.812	0.795	0.915	0.916	0.853	0.923	0.794
AVERAGE	0.838	0.802	0.781	0.883	0.899	0.823	0.890	0.780

REPEATABILITY = 0.014 Mg/m^3
REPRODUCIBILITY = 0.066 Mg/m^3 (0.050 Mg/m^3, if Sample ST-299 is excluded)

Table VI. Results of Coke Bulk Density Interlaboratory Test Series Completed in Mid-1981 (-20+48 Mesh, Precrushed, and Sized)

Coded Laboratory Designation	VIBRATED BULK DENSITY, Mg/m^3 Sample No.: ASTM-268	ASTM-398	ASTM-397	ASTM-685	ASTM-896	ASTM-588	ASTM-860	ASTM-895
A	0.805	0.870	0.903	0.823	0.745	0.819	0.917	0.795
	0.812	0.874	0.895	0.821	0.744	0.824	0.921	0.804
B	0.781	0.862	0.893	0.833	0.775	0.813	0.885	0.758
	0.775	0.870	0.893	0.833	0.781	0.820	0.893	0.769
C	0.786	0.877	0.887	0.836	0.760	0.826	0.910	0.752
	0.787	0.878	0.887	0.835	0.760	0.826	0.911	0.752
E	0.778	0.859	0.876	0.815	0.729	0.812	0.884	0.770
	0.778	0.861	0.871	0.816	0.732	0.821	0.883	0.765
G	0.787	0.866	0.887	0.820	0.773	0.822	0.909	0.806
	0.785	0.865	0.886	0.826	0.756	0.813	0.914	0.796
H	0.762	0.855	0.869	0.808	0.749	0.796	0.876	0.756
	0.770	0.861	0.893	0.798	0.747	0.797	0.876	0.755
AVERAGE	0.784	0.867	0.887	0.822	0.754	0.816	0.899	0.774

REPEATABILITY = 0.019 Mg/m^3
REPRODUCIBILITY = 0.042 Mg/m^3

Although this reproducibility was probably adequate for the test to pass an acceptance vote by ASTM D02.RDDV.D, we felt that it could be improved. This was based on an increased awareness of the importance of the pouring step in determining bulk density values (cf Table I). This increased awareness came about as the result of an in-house Alcoa bulk density seminar and training session held after the test series described above had been run. The session resulted in improvement in bulk density test reproducibility. The major factor in producing low density values was a rapid pour rate. Although this factor had been recognized to some extent earlier and mentioned in the tentative ASTM vibrated bulk density test procedure, the importance was probably inadequately stressed. As supporting evidence, in the interlaboratory test series the Alcoa values ("Laboratory A" in Tables IV-VII) were the highest or among the highest reported, probably because the Alcoa operator consistently used a slow pour rate.

For this reason, a test series has been initiated with a revised procedure emphasizing the importance of a slow pour rate in attaining high, consistent vibrated bulk densities. The results of this test series were not available when this paper was written, but will be presented at the 1982 AIME Annual Meeting.

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References

1. S.S. Jones, R.D. Hildebrandt, and M.C. Hedlund, "Variation of Anode Performance with Coke Quality," Paper No. A77-97, presented at the 106th AIME Annual Meeting, Atlanta, GA, 1977.

2. P. Rhedey, "A Review of Factors Affecting Carbon Anode Consumption in the Electrolytic Production of Aluminum," Light Metals 1971, TMS-AIME, New York, NY (1971) pp. 385-408.

3. D. Belitskus, "Optimization of Raw Materials and Formulations for Hall-Heroult Cell Electrodes," Third Yugoslav Symposium on Aluminum, (Preprints) Edited by Prof. Andrej Paulin, University of Ljubjana (1978).

4. R.E. Gehlbach, E.E. Hardin, L.I. Grindstaff, and M.P. Whittaker, "Coke Density Determination and Its Relationship to Anode Quality," Paper presented at the 109th AIME Annual Meeting, Las Vegas, NV, 1980.

5. D. Belitskus, "Evaluating Calcined Coke for Aluminum Smelting by Bulk Density," Light Metals 1974, TMS-AIME, New York, NY (1974), pp. 863-878.