Introduction

For nearly 45 years carbon black manufacturers have relied on a structure test method currently known as Carbon Black-Oil Absorption Number (OAN). The industry adopted a standardized test method in 1965, ASTM D2414, citing a linear relationship to SBR compound dewater. The test method was originally named Dibutyl Phthalate Absorption Number of Carbon Black and is still known as “DBP”. Another method seriously studied at the same time was known as compressed void volume. Both methods were considered equally useful in terms of their relationship to compound properties, but the introduction of the mechanical oil absorptometer provided an automated analysis which greatly simplified an otherwise tedious and operator dependent measurement. Carbon black structure measurements found in literature includes:

• Oil Absorption Number or OAN (also known as DBP)
• Oil Absorption Number of Compressed Sample or COAN (also known as 24M4)
• Compressed Void Volume or VV
• Absorptivity index (V'/V) from transmission electron microscopy

Since the early 1960’s, industry researchers have published papers citing challenges with structure information obtained from dibutyl phthalate oil absorption. An important deficiency found in many studies is the lack of consistent relationships between carbon black oil absorption numbers and properties of filled compounds such as viscosity and modulus. This limits a compounder’s ability to consistently predict or design performance properties. The specific structure parameter cited as most important in determining relationships to compound properties is carbon black intra-aggregate void volume, often referred to as primary structure. Intra-aggregate void volume for a mass of carbon black represents the volume in which a polymer can occlude, thereby resulting in a shift in effective filler volume fraction.

When mixing carbon black with a liquid, there is no methodology available to isolate intra-aggregate void space. An oil absorption number represents the total volume between and within a mass of aggregates resulting in a dependency on the bulk density of the product. An example of the influence of bulk density on oil absorption numbers is shown in Table 1.

The example in Table 1 includes loose black and pelletized N650, a carcass or soft grade used in tires and mechanical rubber goods. These two samples were taken from the same production unit but collected at different points in the carbon black process. These samples of N650 have equivalent primary structure levels as indicated in the void volume data shown at 55 and 100 MPa applied pressure, but have very different bulk density (i.e. pour density). The OAN data for these samples suggests the loose black has a higher structure level.
than the pelletized sample, yet there is no evidence of differences in morphology. From this example we can conclude that OAN is not a specific measure of intra-aggregate void volume or primary structure, and OAN numbers are highly overestimated values of structure as compared to compressed void volumes (or typical COAN levels). This example also demonstrates that OAN is dependent on bulk density, and bulk density is clearly not an intrinsic characteristic of the carbon black. Manufacturing carbon blacks to an OAN structure specification is one reason end-users will continue to find differences in carbon black performance between suppliers.

In the 1970’s, the carbon black industry adopted an improved oil absorption test currently known as Carbon Black Oil Absorption Number of Compressed Sample or COAN (24M4). COAN is an oil absorption number on a carbon black which has been compressed in a press four times at 165 MPa or 24,000 psi. By compressing the carbon black, a higher bulk density is achieved by moving aggregates close together. Compression also causes some aggregate breakage or reduction. Both effects serve to reduce the measured oil absorption number. Although the COAN measurement is an improved estimate of primary structure compared to OAN, it has not replaced OAN as the manufacturing target for carbon black production. A number of reasons can be cited as to why COAN has not replaced OAN:

- Specific to carbon black primary structure
- Intrinsic characteristic of carbon black
- Elimination of solid-liquid interactions (no oil)
- Faster
- Lower cost
- Non-dusting
- Replacement for both OAN and COAN structure testing

### Compressed Void Volume

Due to existing history with compressed void volume and the advances in microprocessors and instrumentation, D24 encouraged instrument companies to develop a modern void volume analyzer. Jaron Technologies developed the first new generation void volume analyzer which became commercially available in 2004. This instrument was designed for single-pressure equilibrium measurements, and later adapted to step-wise multi-pressure measurements. Jaron has sold a number of these instruments for research and evaluation.

G. A. Joyce and W. M. Henry [1] used the Jaron instrument to study void volumes of 54 carbon blacks along with traditional oil absorption methods, and modeled the data with SBR and NR compound properties obtained with an RPA. Conclusions from their study indicated compressed void volumes provided significantly stronger and more consistent relationships to compound properties compared to oil absorption methods—an indication that compressed void volumes are more specific to carbon black primary structure. Compound properties studied included processability and reinforcement measurements including rheometer.
viscosity and dynamic shear modulus, $G'$. Optimum applied compression pressures were determined by modeling compound properties with all levels of applied pressure. The comparison of structure methods with an SBR compound dynamic shear modulus, $G'$ at 1% SSA, using a single-term model is shown in Figure 1.

Figure 1 is a representation of the strength of the statistical relationship between the carbon black structure and dynamic shear modulus using a single-term model or straight-line equation. OAN exhibited the weakest relationship of all the structure methods with an $R^2$ of only 0.78. COAN provided a much stronger relationship with an $R^2$ of 0.91. Compressed void volumes exhibited the strongest relationship which varied with applied pressure. The $R^2$ values ranged from 0.94 to a maximum of approximately 0.98. The maximum relationship was observed at approximately 105 MPa applied pressure. This optimum level of compression pressure produces a void volume level which relates best to the average aggregate state within the SBR compounds. Other researchers have shown that some aggregate reduction always occurs during mixing.

An important observation from this study is that the best carbon black structure measurement minimizes inter-aggregate voids and also produces aggregate reduction or fracture during the measurement process. Compression experiments from void volume analysis achieve both effects while OAN provides little of either. COAN provides some of both effects, but since the compressed sample has to be aerated for the subsequent oil absorption COAN numbers contain an un-controlled quantity of inter-aggregate void space (i.e. variable density) which leads to measurement error and inflated structure estimates.

**Dynamic Void Volume Analyzer (DVVA)**
The most recent advance in compressed void volume instrumentation is the Dynamic Void Volume Analyzer or DVVA4000 from Micromeritics Corporation. The DVVA was developed in conjunction with Columbian Chemicals R&D to produce an instrument for the carbon black industry capable of fully automated compression experiments. A comparison of the DVVA and oil absorption methods is shown in Table 2.

As indicated in Table 2, the DVVA provides the desired properties of an improved structure method previously outlined by D24. A recent study with this new instrument by G. A. Joyce, W. M. Henry and R. W. Magee [2] was presented in October 2008 at the ACS Rubber Division Meeting in Louisville, KY. In this study, dynamic

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**Table 2. Comparison of DVVA and Oil Absorption Test Methods**

<table>
<thead>
<tr>
<th></th>
<th>DVVA</th>
<th>Oil Absorption</th>
</tr>
</thead>
<tbody>
<tr>
<td>Primary Structure Measurement</td>
<td>Yes</td>
<td>No</td>
</tr>
<tr>
<td>Intrinsic CB Characteristic</td>
<td>Yes</td>
<td>No</td>
</tr>
<tr>
<td>Faster Test</td>
<td>Yes</td>
<td>No</td>
</tr>
<tr>
<td>Lowest Cost</td>
<td>Yes</td>
<td>No</td>
</tr>
<tr>
<td>Non-Dusting</td>
<td>Yes</td>
<td>No</td>
</tr>
<tr>
<td>Self-Cleaning</td>
<td>Yes</td>
<td>No</td>
</tr>
<tr>
<td>Structure Stability Measure</td>
<td>Yes</td>
<td>?</td>
</tr>
<tr>
<td>Requires Ventilation</td>
<td>No</td>
<td>Yes</td>
</tr>
<tr>
<td>Produces Oil Waste</td>
<td>No</td>
<td>Yes</td>
</tr>
<tr>
<td>Continuous calibrations required</td>
<td>No</td>
<td>Yes</td>
</tr>
</tbody>
</table>
scan experiments were investigated on carbon blacks using the dynamic void volume analyzer, DVVA 4000, from Micromeritics Corporation. The DVVA 4000 was designed for a sample mass of 1 gram to accommodate low-density powders or pelleted products. A picture and diagram of the DVVA with major components is shown in Figures 2 and 3.

**FIG. 2 - DVVA 4000**

Calculation of the measured-compressed void volume, $VV_M$, is shown in Equation 1:

$$VV_M = \left( \frac{V_a - V_t}{m} \right) 100$$

**FIG. 3 - DVVA 4000 Block Diagram**

where $V_a$ is equal to the apparent volume of the sample in cm$^3$, $V_t$ is equal to the theoretical volume of the solid sample in cm$^3$, and $m$ is the sample mass in grams.

The unit of measure for $VV_M$ is cm$^3$/100 g. The apparent volume, $V_a$, is defined by Equation 2:

$$V_a = \frac{m}{\rho_t}$$

where $h$ equals the measured sample height in cm, and $D$ is equal to the cylinder diameter in cm. The theoretical solid sample volume, $V_t$, is defined by Equation 3:

where $m$ is the sample mass in grams and $\rho_t$ is the true or skeletal density of carbon black, commonly accepted as 1.9 g per cm$^3$ for many carbon blacks. The DVVA software has several input fields including one for measured skeletal densities.

**OAN Predictive Modeling from DVVA Data**

A broad range of furnace carbon blacks from very low (~35 cm$^3$/100 g) to very high (~175 cm$^3$/100 g) OAN were included in this study. The carbon blacks included a variety of Statex®, Furnex®, and other experimental furnace carbon blacks from Columbian Chemicals Company along with Standard Reference Blacks (SRB) from ASTM International which are produced by various manufacturers. Also included in the study were low-density powder blacks collected from reactor ports or bag collectors along with corresponding pelletized products. Several hundred samples were included within the various analyses discussed in the paper. The colloidal space encompassed by the samples is shown in Figure 4, and represents a majority of commercially important rubber carbon blacks.

A predictive model for oil absorption numbers should have a standard error equal to or less than the OAN test reproducibility standard deviation (OAN SR) since this analysis is a comparison of the variation between samples, not replicates within a single sample. According to ASTM D2414, the pooled OAN SR is 1.4 cm$^3$/100 g. Initial predictive modeling of OAN made use of large sample sets containing a variety of carbon blacks. Initial studies indicated that universal OAN prediction models from void volume data did not appear feasible since the best model standard error was 6.7 cm$^3$/100 g.

Subsequent studies methodically limited the sample sets to specific groups of products for predictive modeling. This process of categorization of sample sets ultimately led to modeling specific carbon black production lines in order to obtain the lowest standard error between OAN and predicted OAN (POAN) based on dynamic void volume data. The chart in Figure 5 summarizes the progression of predictive modeling from highest to lowest standard error.

**Equation 4**

$$OAN = -23.1642 + 0.11099 \times VV^1 @ 19MPa - 0.19485 \times VV^2 @ 32MPa + 0.17652 \times VV^1 @ 102MPa$$
OAN predictive models are not limited to product physical form. Loose powder and pelleted samples were included within the modeling sample sets such that a single model is useful for analyzing product from reactor ports, bag collectors and finished-pelletized product. An example of within-grade OAN prediction of loose black and pelleted product is shown in Figure 7.

Figure 7 is an example of in-process sampling and structure analysis of two carbon black products. The POAN data in Figure 7 are from a single OAN prediction model based on DVVA analysis of loose and pelleted blacks. The first product’s pelleted samples have OAN and POAN levels near the target of 130 cm$^3$/100 g and loose black samples (from reactor ports) in the range of 137-140 cm$^3$/100 g. The second product exhibits OAN and POAN levels slightly higher than the target of 121 cm$^3$/100 g for pelleted product, while the loose black samples exhibit OAN levels of approximately 130 cm$^3$/100 g.

Figure 7 also demonstrates one of the complications from use of the OAN structure method, which is that loose black samples always exhibit higher OAN levels than corresponding pelleted samples; however, there is no evidence of reduction in primary structure due to the pelletization process, only a difference in bulk density as previously indicated in Table 1. Dynamic void volume analyses of the N650 samples from Table 1 are shown in Figure 8. These void volume pressure scans indicate the loose black sample exhibits a higher level of void volume than pelleted black at very low applied pressures, and this difference is due to lower bulk density of the loose black sample. At applied pressures of approximately 55 MPa and greater, the two pressure scans converge, indicating an equivalent level of void volume or primary structure for the loose black and pelleted samples.

Modeling samples from a single tread line using a two-term model resulted in a standard error of 0.7 cm$^3$/100 g, a level which is half the OAN SR of 1.4 cm$^3$/100 g. Observed versus predicted OAN data for the single tread line sample set are shown in Figure 9. This two-term model, shown in

Equation 5

\[ OAN = -27.3769 + 8.4943 \times VV @ 141 \, MPa - 6.5465 \times VV @ 204 \, MPa \]

Equation 5, includes 70 samples in the range of approximately 100 to 130 cm$^3$/100 g.

**Modeling SBR and NR Compound Properties**

A variety of rubber carbon black products were analyzed for in-rubber rheometer and dynamic properties using an Alpha Technologies Rubber Process Analyzer 2000 (RPA) per ASTM D6601. Test compounds included styrene-butadiene rubber (SBR) and natural rubber (NR) formulations per ASTM D3191 and D3192, respectively. The rheometer properties were obtained at 150 °C using 30 minute cures and dynamic properties at 100 °C at 1 Hz and 1, 10 and 50 percent single strain amplitude (SSA).

Compounds were also prepared in the ASTM NR D-3192 formulation for stress-strain properties. Tensile stress measurements were made with a United Testing System per ASTM D412, and reported as delta IRB-7.

The processability and vulcanize properties of SBR and NR compounds were modeled with the three structure measurements including OAN, COAN and VV. The best
single-term predictive model for the SBR compound minimum rheometer torque, \( S'_\text{min} \), was the squared term of void volume, \( VV^2 \) and is shown in Figure 10 along with OAN and COAN predictive models. The chart in Figure 10 is a plot of the model \( R^2 \) values and displayed as a function of applied pressure for the DVVA data, but is a constant for OAN and COAN. This chart indicates the void volume data exhibits a stronger and more consistent relationship to compound viscosity as compared to COAN or OAN. At low applied pressures, the DVVA data initially exhibits a similar level of \( R^2 \) as COAN, and then improves with increasing applied pressure. OAN exhibits the weakest relationship of the three structure methods.

The chart in Figure 11 is a plot of the \( VV \) and oil absorption models \( R^2 \) values for the best single-term predictive models for the SBR compound shear modulus, \( G' \) at 1% SSA. The DVVA relationship is displayed as a function of applied pressure but is a constant for OAN and COAN. Similar to Figure 10, this chart also indicates the void volume data exhibits a stronger and more consistent relationship to compound viscosity as compared to COAN or OAN.

Higher order prediction models were evaluated for each of the SBR and NR compound properties. A summary of the best prediction models for SBR compound shear modulus, \( G' \) at 1% SSA is shown in Table 3.

The best 2 and 3-term prediction models include differential void volumes. Higher order models exhibited significantly lower standard error than single-term models which demonstrates the value of dynamic scan data compared to single-pressure VV data. Surface area measurements were included in the predictive modeling, and when combined with void volume measurements, provided very low standard errors. Compound properties such as viscosity and low-strain shear modulus can be effectively described through the use of statistical models based on improved structure measurements.

---

**Table 3. Modeling Summary**

<table>
<thead>
<tr>
<th>Dependent Var. = SBR ( G' ) @ 1% SSA from RPA</th>
<th>Best Model</th>
<th>VV Predictor Vars</th>
<th>Model Std Error (kPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Best Model</td>
<td>VV2 @ 171 MPa</td>
<td>( VV^2 )</td>
<td>131</td>
</tr>
<tr>
<td>2-term</td>
<td>VV2 @ 164 MPa, VV2 @ 170 MPa</td>
<td>( VV^2 )</td>
<td>74</td>
</tr>
</tbody>
</table>

**FIG. 6 –** Measured and Predicted OAN from Single Carcass Line Model

**FIG. 7 -** Within-Grade OAN Prediction – Powder and Pellets
SUMMARY
A new dynamic void volume analyzer (DVVA) has been utilized to study predicted oil absorption numbers, and relationships to compound properties. The DVVA produced improved structure measurements compared to oil absorption methods based on significantly stronger statistical relationships to processability and reinforcement properties of SBR and NR compounds. DVVA scan data were modeled and used to predict OAN data within a production process with an accuracy equal to or better than the precision of the oil absorption testing. The advancements identified in structure measurements with the DVVA should facilitate the replacement of oil absorptometers in the carbon black industry, and ultimately replace existing product specifications based on OAN.

REFERENCES
This award recognizes Dr. Michael Warskulat’s exceptional contributions to Committee D24 on Carbon Black in the development of new analytical standards, for improvement of existing standards, and for strong leadership in developing international standards for the global market.

Dr. Warskulat received his diploma in Physical Chemistry in 1986 and his PhD in Chemistry in 1990 from the Universitaet Hannover, Germany. After graduating Michael joined Degussa AG, Zaventem, Germany as a Lab Manager. In 1991 he became the Lab Manager at Degussa’s Cologne facilities. Then in 1998 he transferred to Degussa’s Cofrablack plant in Ambes, France as the Lab Manager. In 2001 Michael became the Senior Manager Technical Services. Today he is the Director Technical Market Management Europe and NAFTA for Applied Technology Rubber Additives/Advanced Fillers and Pigments, Evonik Degussa Gmbh.

Michael initially became involved in improving D1510 Iodine Adsorption Number by studying the preparation and constituents of the iodine solution, stability of the iodine solution, and effect of shake time on the repeatability and reproducibility. Of special interest was the influence of potassium iodide concentration on the adsorption value. From his studies it was concluded that the concentration levels were critical to stabilize the iodine solution and care was required in the preparation of the iodine solution.

He was instrumental in the improvement of D5230 Automated Individual Pellet Hardness. His work included extended studies on the effect of each of the individual test parameters (crush diameter, force drop, rate of piston movement, and pellet size) on the results on the crushing of individual pellets. From these extensive studies D5230 was updated defining standard conditions.

D3493 Oil Absorption Number (previously Dibutylphathalate Number ) used DBP for years which had come under fire for being a potential carcinogen. This fact required special handling of the waste stream after testing which was an issue with both the manufactures and consumers of carbon black. Michael was instrumental in the conversion of D3493 from DBP to paraffinic oil. The use of DBP needed to be addressed using a replacement oil that was a drop-in replacement. Michael was instrumental in organizing an international crosscheck using paraffinic oil to validate its usage. The name of the standard was then changed from DBP to Oil Absorption Number to reflect this advancement.

In addition to his work in D24 Michael has carried ASTM’s flag to the European community through his work in ISO TC45/ SC3/WG3. He strongly emphasized the need to support mutually agreed on standard development. If an organization had already developed a standard for a particular property then the international committee should not develop a separate standard. Eventually the two standards would start to diverge or one would continue to advance while the other could possibly remain stagnant. In either case two standards for the same property could co-exist which was not good for the international community.

The success of ASTM D24 on Carbon Black is due to the dedicated contributions of individuals like Michael Warskulat. Michael has made significant contributions across many of D24’s standards. It is through his hard work and perseverance that ASTM is able to see continuous improvement and development of new methods for the global
ASTM International Committee D24 on Carbon Black

Prague, Czech Republic | 5-7 October 2009

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Contact

Joe Koury
ASTM International
Phone: +1-610-832-9804
jkoury@astm.org

Online registration will be available on the ASTM website in April 2009.

www.astm.org/MEETINGS/prague
www.astm.org/COMMIT/D24.htm

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**D24 Activities**

**D24.11 Carbon Black Structure**  
**Chairman: George Joyce**  
Columbian Chemicals Co.  
(770) 792-9467  
gjoyce@columbianchemicals.com

**D2414 Oil Absorption Number**  
Sid Richardson (P. Eubanks) reviewed Section 8.1.1 and recommended that the section be re-written as follows: 8.1.1 Model - Three different types of adsorptometers are in use: 1) early models based on springs and mechanical indication of torque (Type A and B), 2) second generation adsorptometers equipped with load cells and digital torque display (Type E*), and 3) current model adsorptometers which are designed with a torque measuring system that includes a micro-computer and software to continuously record torque and oil volume with time (Types H and C and modified Type E*). Types A, B, and E* are designed to stop mixing at a predetermined, fixed torque level, which is the recommended procedure for measuring hard or tread blacks (calibration procedure A). The computer controlled models (Types H and C and modified Type E*) are required for running calibration procedure B the recommended torque curve analysis for the determination of the end-point of soft or carcass blacks. The Type H and C and modified Type E* adsorptometers can also provide an end-point at a fixed or predetermined torque level such that these types of adsorptometers are well-suited for measuring OAN of both hard and soft carbon blacks. Several components influence the calibration: the dynamometer torque spring or the load cell, the torque limit switch or the indicator set point, the damper (oil damper or electronic damping), and the mixing head consisting of two counter rotating blades and a mixing bowl. It is necessary that all of these components are in good condition and are properly adjusted to achieve acceptable calibration.

*Type E adsorptometers can be modified with add-on a micro-computer system.

Cabot (M. Mongardi) suggested changes to Section 8.3.5 which currently reads “For measured values on the of the SRB’s that are consistently outside the expected variability listed in Guide D4821…” and should read “For normalized values of the of the SRB’s that are consistently outside the x-chart limits listed in Guide D4821…”

Columbian Chemical (G. Joyce) presented the results of the OAN round robin which was initiated due to questions about the SRB E8 OAN target. The results validate the existing OAN target, but also indicate that paraffin oils produced a higher mean level with E8 (+1 unit) and lower mean level with D8 (-1 unit). Differences were observed between DBP and paraffin oils with each of the SRB carcass standards (standard rubber blacks). Poor precision was also observed with paraffin oil and the F-8 (N683) standard compared to DBP oil. Therefore, D2414 will be balloted to reinforce the statement within D2414 which states that DBP oil should always be used for referee testing whenever a testing discrepancy exists. Also, the method currently indicates that paraffin oils provide equivalent results as DBP, but data has been presented to D24 which indicates differences vs DBP oil for standard grades such as E8 (N660).

Continental Carbon (J. Bailey) presented the results of the LPRS OAN and COAN data with E8. This data indicated DBP and specific paraffin oils produced statistically different results. Both Phazol 7 and PLC1 oils produced average values of 91.4 and 91.3 cc/100g, respectively vs DBP oil with an average value of 87.4 cc/100g. Users of these oils should consider discontinuing their use.

**D6086 Void Volume (VV)**  
Columbian Chemicals (G. Joyce) presented a paper titled, “Advancements in Structure Measurements of Carbon Black”. The presentation data confirmed that dynamic void volume is an improved structure measurement compared to oil absorption methods. The presentation included successful prediction of OAN and COAN testing from DVVA data - within a production unit, and indicated that universal OAN prediction models are not feasible (i.e. samples from multiple units and/or producers). The presentation also included modeling of in-rubber properties from RPA and stress-strain testing. Both processability and reinforcement properties from RPA testing were modeled using DVVA with improved accuracy as compared to OAN or COAN.

Section 10 Void Volume Calculations will ballot changes to Section 10.1 as follows:

10.1 The measured void volume \( V_m \) is calculated from the measured apparent compressed volume as follows. The apparent compressed volume of the sample is evaluated by Equation 2.

\[
V_A = h x 3.1416 D^2/4
\]

where:

\[ V_A = \text{the apparent compressed volume of the carbon black sample, cm}^3, \]
\[ h = \text{the “height” of the compressed carbon black in the cylinder, cm, and} \]
\[ D = \text{the diameter of the cylinder, cm.} \]

The Appendix will be updated to include a “Brief Description of a Dynamic Void Volume Device” (Appendix X2).

**D24.21 Carbon Black Surface Area and Related Properties**  
**Chairman: Dirk Roller**  
Evonik Degussa GmbH  
(44) 92 23 396 4615  
dirk.roller@evonik.com

**D1510 Iodine Adsorption Number**  
Wording in D1510 has been changed in Section A1.1.1 has been changed to “A1.1.1 Sufficiently dry potassium iodide…” and in Section “A1.2.3.1 Sufficiently dry KI…”

There was substantial discussion regarding test result variations when comparing recent results to the original accepted SRB 8 test results. In general it was agreed that more rigorous analyses of recent LPRS and task force data was needed to try to pinpoint specific problem areas.
carbon black industry.

D24.31 Non-Carbon Black Components of Carbon Black
Chairman: Jeffery A. Melsom
Michelin Americas Research & Development Co.
(864) 422-4079
jeff.melsom@us.michelin.com

D1514
Sieve Residue
The Sieve Task Group formed mission is to “Develop a test method for the determination of sieve residue of carbon black which is more meaningful for sensitive applications as in extruded profiles. The test result should provide an indication whether the carbon black may cause imperfections on the extrudate surface.”

A discussion was held concerning the wording of the “rub out” of the sieve residue—is some of the residue hard carbon black? The title of the subcommittee is “non-carbon black components”, yet often part of the residue is carbon black. A note will be added to state sieve residue may also contain carbon black. Also reported by Continental Carbon (J. Bailey) evidence that that wearing a glove does not change the result of the sieve residue test. Additional data will be presented at the next meeting (June 2009).

Cabot (D. Roller) presented information on a sieve residue apparatus for MRG grades. A drawing of the instrument was distributed.

D24.41 Carbon Black Nomenclature and Terminology
Chairman: Ricky Magee
Columbian Chemicals Co.
(770) 792-9472
rmagee@columbianchemicals.com

D24.45 USA Committee to ISO
Chairman: John A. Bailey, Jr.
Continental Carbon Co.
(281) 647-3851
john@okstats.com

The next meeting of ISO/TC45/SC3/WG3 will be held in Cochin, India from 26 – 30 October 2009.

D24.51 Carbon Black Pellet Properties
Chairman: Pierre Hirtt
Hitec Luxembourg
(352) 49-847-8740
pierre.hirtt@hitec.lu

D24.61 Carbon Black Sampling and Statistical Analysis
Chairman: John A. Bailey, Jr.
Continental Carbon Company
(281) 647-3851
john@okstats.com

A report on the analysis of the LPRS data for SRB 8E and the effect of the various equipment, materials, and methods compared to SRB 8A was given. Several interesting trends were observed but more data is needed to reach any firm conclusions. The report showed the importance of a similar data collection and analysis for the remaining SRB 8 materials to establish the uniformity and equipment, materials, and methods effect baseline. A multivariate analysis is needed to better understand the interrelationships of the various factors.

Laboratory Proficiency Rating System (LPRS)
A report on the analysis of the LPRS data for SRB 8D and the second SRB 8B. The data on the SRB 8D material is due by April 1st and the 8B by July 1st in order to have data analysis completed for reporting at the June and October meetings.

Due to the importance of collecting the equipment and method data for use with Minitab, the cover letter explaining the importance of providing this information for each laboratory will again be sent with the samples. The cover letter states that this information is so critical that if it is not provided, that laboratory’s data will not be used. The letter will be revised to emphasize the importance of providing all the requested information.

Standards Inventory
Continental Carbon’s uniformity data on IRB #8 was presented. An ITP will be conducted to determine the lot values. Participating laboratories are: Cabot (2 – 4 laboratories), Evonik, Sid Richardson, and Columbian. The twelve odd numbers bags will be used for D3191 testing and the twelve even numbered bags for D3192 testing. Continental Carbon’s non-rubber uniformity test data will be used in D4122 with an appropriate note on the data source.

The initial SRB 8 acceptance data is available when purchasing the materials but has not been published in any D24 standard. Reported problems achieving the control limits for some tests and materials have been validated through the LPRS programs and some differences have been shown to be statistically significant. The LPRS data will be used to revise SRB 8 target and control limit values for statistically significant differences. The SRB8 data table will be posted on the ASTM D24 page website after it has been reviewed and approved by D24. The table will also be provided to Laboratory Standards for use with shipments and e-mailed to all D24 members. Future revisions to the data must be approved during D24 meetings before the website is updated.
A ballot will be issued to harmonize rubber test conditions in D3191 Carbon Black in SBR (Styrene-Butadiene Rubber) – Recipe and Evaluation Procedures and D1392 Carbon Black Evaluation in NR (Natural Rubber)

D24.81 Carbon Black Microscopy and Morphology
Chairman: Bonnie Mcwade-Furtado
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(978) 670-7069
bonnie_mcwade@cabot-corp.com

D3849-02
Primary Aggregate Dimensions from Electron Microscope Image Analysis
Columbian Chemical (R. Magee) presented information showing a digital method to measure particle size and MVA Consultants (J. Millette) gave an update on his work with a manual measuring method. Additional data on both methods will be presented at the next meeting.

About ASTM International

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