Advances in Structure Measurement using Compressed Void Volume

BY GEORGE JOYCE
(Columbian Chemicals Company, Marietta, GA)

During 2005 several events related to the compressed void volume structure method were noteworthy. 1) D24 revised the D6086 void volume standard for the first time in 10 years. 2) Jaron Technologies sold several void volume instruments to carbon black and tire manufacturers. 3) Hitec Luxembourg announced their joint program with Degussa to research and develop an improved void volume analyzer for the carbon black industry. 4) A paper was presented at the ACS Rubber Div Meeting on November 1-3, 2005 entitled “Modeling the Equilibrium Compressed Void Volume of Carbon Black” by G. Joyce and W. Henry of Columbian Chemicals Company.

These events and others will lead to improved structure characterization through advances in the compressed void volume test method and instrumentation to ultimately replace the oil absorption methods.

ASTM International D6086-05 Standard no longer refers to compressed volume index or CVI. This test method for Carbon Black - Void Volume (VV) describes the scope, terminology, significance and use, and relevant calculations. The test method will undergo further definition of procedures and testing parameters as these are defined within the 24.11 sub-committee.

Jaron Technologies supplied a number of single-pressure void volume instruments to carbon black and tire manufacturers during 2005. These instruments are under-going evaluation and are being used to develop test procedures and parameters. Jaron Technologies can be contacted at ronhurst@sbcglobal.net.

Hitec Luxembourg S.A. announced they will carry out an R&D project in cooperation with Degussa A.G. of Cologne in the framework of the intergovernmental initiative EUREKA (www.eureka.be/inaction/ShowProject.do?id=3597). Hitec’s R & D efforts are targeted at development...
of an instrument to measure carbon black and silica structure through compressed structured volume tests (CVST). Hitec currently produces test equipment for the carbon black and rubber industry.

Modeling the Equilibrium Compressed Void Volume of Carbon Black by G. Joyce and W. Henry of Columbian Chemicals Company, Marietta, GA. [1] A technical paper was presented at the Fall ACS Rubber Division Meeting in Pittsburgh on November 1-3, 2005 on void volume analysis of carbon black. This paper describes a large study which includes a broad range of furnace blacks (54 samples), that were evaluated for compressed void volume, in-rubber properties and typical colloidal properties. This paper describes the structure analysis by multi-point equilibrium void volume along with precision estimates and subsequent modeling of the data. One of the most useful models describing the compressed volume and the log of the applied pressure is shown below:

\[ VV = A_1 + B_1 \log_{10} P \]

Examples of multi-point void volume data using the log P model for a very high and a very low structure carbon black are shown in Figure 1:

The paper further describes the use of predictive modeling to obtain estimates of OAN and COAN, and in-rubber properties from the void volume information using Figure 2.
advanced multiple and polynomial regression models. Predictive models with standard errors approaching the COAN precision were obtained from multi-point VV model parameters indicating the COAN structure property could be modeled across a wide range of products. Conversely, the OAN structure property was found to exhibit a very poor relationship to void volume data when modeled across a wide range of carbon black products.

The most important conclusion from this current study of void volume was the observation that the void volume data was a significant improvement in a structure test compared to oil absorption methods. This finding was based on in-rubber properties from testing a large number of SRB and NR compounds using an RPA 2000, and focusing on rheometer and dynamic properties known to be strongly related to carbon black structure. Two examples of the in-rubber data are shown in Figures 2 and 3:

Figure 3 indicates void volume data is much better related to the low-strain shear modulus of these compounds than oil absorption (or effective volume fractions derived from oil absorption). The structure relationships to in-rubber properties vary with compression pressure for void volume testing, and are constant for the single-value oil absorption methods. The void volume data exhibited an optimum at approximately 105 MPa pressure indicating this level of aggregate reduction best approximates the carbon black void volume related to this reinforcement property [1].

**FOOTNOTES**

Degassing ASTM SRB B-6 Prior to Measuring Nitrogen Surface Area (NSA)

(based on a presentation by Tony Thornton, Micromeritics Instrument Corporation, Norcross, Georgia)

A question arose within a company concerning why ASTM B-6 results were always low. This company used ASTM SRB B-6 as a control to determine whether their measure was stable and in control. Although the results were within the ASTM Control Chart Limits as defined in D4821 Validation of Test Method Precision and Bias, Table 1 – SRB 6 Control Chart Limits, their results always tended to be close to the lower control limit. Upon further discussions it was determined that ASTM’s D6556 procedure was being followed with Flow Degassing at 300ºC for 30 minutes.

From the discussions Micromeretics conducted a limited experiment to try and assess why this was occurring. The reason for the low results could have come from a number of areas such as the sample, the instrument, or even the procedure. Emphasis was placed on the degassing time with the results in the table below.

### CONCLUSIONS
- NSA results do vary with cooling if the sample is degassed 60 minutes or less
- NSA results are repeatable if degassed for 120 minutes
- NSA results do not increase with extra degassing
- Recommended longer degassing time for SRB B-6

Finally, ASTM’s D6556 Total and External Surface Area by Nitrogen Adsorption states in Section 8.4 Flow Degassing (Subsections 8.4.2 and 8.4.3) states, “The minimum degassing time that gives a stable surface area (that is, a surface area that does not increase with additional degassing) may be used for degassing. Once the typical degassing times have been determined, future samples can be degassed on the basis of time alone, if desires, allowing a reasonable margin of excess time.”

### Table 1: Degassing Study varying Degassing and Cooling Time

<table>
<thead>
<tr>
<th>Tube</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
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<tr>
<td>Mass (grams)</td>
<td>0.2514</td>
<td>0.2445</td>
<td>0.2558</td>
<td>0.2558</td>
<td>0.2556</td>
<td>0.2499</td>
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<td></td>
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<tr>
<td>1st Degassing (min.)</td>
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<td>60</td>
<td>30</td>
<td>60</td>
<td>30</td>
<td>60</td>
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<tr>
<td>Cooled (min.)</td>
<td>0</td>
<td>0</td>
<td>60</td>
<td>60</td>
<td>30</td>
<td>30</td>
</tr>
<tr>
<td>NSA (m²/g)</td>
<td>109.46</td>
<td>108.95</td>
<td>107.76</td>
<td>108.09</td>
<td>107.85</td>
<td>107.49</td>
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<tr>
<td>1st Degassing (min.)</td>
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<td>60</td>
<td>60</td>
<td>60</td>
<td>60</td>
<td>60</td>
</tr>
<tr>
<td>Cooled (min.)</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>NSA (m²/g)</td>
<td>109.67</td>
<td>110.32</td>
<td>110.21</td>
<td>108.42</td>
<td>110.35</td>
<td>108.44</td>
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<tr>
<td>THIRD DEGASSING AND ANALYSIS</td>
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<td>60</td>
<td>60</td>
<td>60</td>
<td>60</td>
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</tr>
<tr>
<td>Cooled (min.)</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
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</tr>
<tr>
<td>NSA (m²/g)</td>
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<tr>
<td>3rd Degassing (min.)</td>
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<td>Overnight</td>
<td>Overnight</td>
<td>Overnight</td>
<td>Overnight</td>
<td>Overnight</td>
</tr>
<tr>
<td>Cooled (min.)</td>
<td>60</td>
<td>90</td>
<td>120</td>
<td>150</td>
<td>180</td>
<td>210</td>
</tr>
<tr>
<td>NSA (m²/g)</td>
<td>110.44</td>
<td>111.49</td>
<td>110.90</td>
<td>110.85</td>
<td>110.29</td>
<td>110.46</td>
</tr>
</tbody>
</table>

From the table the control chart limits for B-6 (N220) are:
- Lower Control Limit: 108.41
- Target Value: 110.0
- Upper Control Limit: 111.59
ASTM D-24 on Carbon Black to Celebrate
50 Years with ASTM International

ASTM International’s Technical Committee on Carbon Black will be celebrating 50 years during their December committee week in Atlanta, Georgia. A special celebration will be held in honor of this accomplishment. Details of the actual celebration will be made known as soon as they have been finalized.

During the last 50 years D-24 has successfully developed carbon black standards for the carbon black, rubber, mechanical rubber goods and tire industries. Today, ASTM International’s Technical Committee D-24 on Carbon Black standards are recognized as the standards of choice due to their technical merit and market relevancy.

The committee was initially formed in 1955 and approved at the September 20, 1955, meeting of the ASTM Board of Directors. The first organizational meeting was held in Buffalo, N.Y., on February 29, 1956, and the first official meeting of Committee D-24 was held on June 22, 1956, in Atlantic City, N.J.

In 1952 members of the eight U.S. carbon black producers formed the Carbon Black Industry Committee and began to hold meetings in Amarillo, Texas, to rationalize and develop uniform chemical and physio-chemical testing procedures. A scope was developed with the primary propose for their existence being defined. The scope (in part) stated the committee’s goal was, “...the promotion of knowledge of the properties of carbon blacks, including stimulation of research and the development of test methods, classifications, and nomenclature...” Today the scope remains essentially unchanged.

Until this time, each carbon black producer and each rubber goods or tire manufacturer had its own specifications and test methods for the carbon black it sold or purchased. The story is told by Frank Svetlik, a founding father of D-24, that once a lot of carbon black was produced, it was tested to determine if it met the specifications of any customers from whom orders had been received. If it failed to meet requirements of Company A, then it would be tested by the methods of Company B, Company C, etc., until it qualified for a particular customer. This duplication of testing was, of course, costly and time-consuming and it created a chaotic situation that grew progressively worse as the types of blacks increased in number.

In July 1956, an ASTM Bulletin was issued that contained the first publication of carbon black test methods by ASTM. This original publication included tentative test methods for pellet size distribution, solvent-extractable material, sieve residue, attrition, benzene discoloration, iodine adsorption, fines, heating loss, pour density, volatile content, pH value, ash content, and mass strength.

The committee abandoned early the notion of developing carbon black specifications and committed its efforts to the development of test methods that were useful in characterizing carbon blacks for purposes of assuring standardization and interchangeability of grades between producers with regard to their reinforcement, processing and handling. Research has been encouraged by communicating the needs of the market and the inadequacy of certain test methods and by providing a classification system for carbon black grades that is based primarily on particle size and permits input into the classification number by the petitioning party. D-24 encourages the development of new carbon black grades with unique performance properties by encouraging exclusive producer/user evaluations of non-classified carbons in order to establish the commercial value of the new grade. Committee D-24 classification of a new grade is encouraged when multiple producers or multiple users of a new grade exist and there is commercial value to the standardization of the properties of the carbon.

In 1959, D-24 first developed the idea of an Industry Reference Black (IRB) for use in standardizing various test methods and in conducting comparisons with other carbon blacks. These IRBs have been replaced as supplies have been consumed. Additionally, other Standard Reference Blacks have been developed to permit calibration of the various test methods. Other reference materials maintained by the committee include an Industry Tint Reference Black and an Industry Tint Zinc Oxide.

D-24 responded to the quality revolution of the early 1980s by facilitating the agreement among all carbon black producers of common target properties for each grade of carbon black. This most significant accomplishment permits the use of Statistical Process Control on a grade of carbon black independent of its producer and has advanced the standardization of grades on an international basis. Those target properties for each grade have been listed in D 1765 since 1985.

While the concept of carbon black surface activity or surface energy has been under study for at least 50 years, there currently is no ASTM method for its measurement. Modern research continues around the world on this property in an effort to establish whether it can augment carbon black reinforcement theory with respect to the observed effects in different polymers. The application of fractal geometry to carbon black morphology by Gerspacher and others has focused attention on the role of the carbon-carbon network in rubber reinforcement

Committee D-24 is unique in its organization in that every main committee member is also a member of every
subcommittee. The Executive Subcommittee is composed of each subcommittee chairman and secretary along with the main committee officers. This organization has provided exceptional teamwork in the development of standards and has permitted more expedient standards development by the resulting open communications and by the use of concurrent subcommittee and main committee letter ballots. Through change in the by-laws in 1990, the committee adopted the preferred use of the International System of Units (SI) which further united the international community with D-24.

Much work and method development has taken place since D-24’s inception in 1956. The continued progress in carbon black standard development and the search for better and more modern ways to characterize carbon blacks remains a primary objective.

A more detailed and updated history will be published in the next edition of The Carbon Aggregate.

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The key tenets of their scope, “...the promotion of knowledge of the properties of carbon blacks, including stimulation of research and the development of test methods, classifications, and nomenclature...” remains unchanged to this day.

A review and updated history will be published in the next edition of The Carbon Aggregate as well as details regarding the venue of the celebration.

Upcoming New Section
Carbon Black – Black-Talk

A major purpose of this newsletter is to inform the readers of the activities of D-24. Primary the information is gathered during the two committee weeks when D-24 meets (June and December). Presented are the individual activities of each of the technical committees as well as articles of interest to both producers and consumers of carbon black.

In order to further advance the carbon black testing and to generate feedback a new section (BLACK TALK) will commence in the future. The idea is to elicit best practices or tips of the trade in the handling and measuring of carbon black. This may entail anything of interest to the reader. For example how best to keep a lab clean, special cleaning supplies used to remove carbon black from the lab and instruments, tips to make testing easier. The door is open to any suggestions that may be out there which may be of interest to anyone involved in the handling and testing of carbon black.

So please send any suggestions to the editor for future consideration and publication.

Remember, Black Talk is your forum to speak out without fear of Black Lash. All submitters will remain anonymous unless you would like appropriate credit.

Special Section
Tips of the Trade!

New section to appear in the next issue.

All New!
In order to further advance the carbon black testing and to generate feedback a new section (BLACK TALK) will commence in the future.
D24 Activities

DD24.11: Carbon Black Structure
Chairman: George Joyce
Columbian Chemicals Co.
(770)792-9467
gjoyce@phelpsdodge.com

D2414 Oil Absorption Number
The committee will draft a ballot for an alternative practice of setting the TLS on absorptometers at 5000 mNm for machines dedicated to tread blacks only (D2414). In addition a proposal to include oil density data into the Annex A1 will be made and balloted.

D3493 Oil Absorption Number of Compressed Sample (COAN)
C. Derrien (Cabot) indicated their laboratories switched from COAN calibrations to using OAN calibrations for the SRB-7 round robin testing, and because good results were obtained, calibrations based on COAN will be eliminated and OAN calibrations will be used. All of Cabot and Columbian labs use this methodology for determining COAN values.

P. Eubanks (Sid Richardson) presented COAN data for calibration of Titan press and COAN calibrations vs. setting target COAN values with B-6 by varying the press pressure and using OAN normalizations. The data appeared to indicate larger variation when using the method of varying the press pressure, but the data was non-normalized. No consensus was reached on performing any further work.

Compressed Void Volume
G. Joyce (Columbian Chemicals Co.) presented a study on Compressed Void Volume which he presented at the ACS Rubber Div 168th Meeting in Nov 2005 entitled “Modeling the Equilibrium Void Volume of Carbon Black”. The data indicates that Void Volume measurements provide a significantly improved structure measure when compared to oil absorption methods. Data models and predictive models were presented indicating CB colloidal properties (VV and ln NSA) can be used to predict in-rubber properties with a high degree of accuracy. Growing consensus is that VV will replace oil absorption.

J. Thompson presented results of a multi-lab crosscheck on compressed void volume using 5 Jaron instruments within SRCC, Columbian, Cabot and Jaron. Five labs tested the SRB-6 series standards in duplicate on two days. The results indicate reasonable uniformity across the five instruments, and after normalization using target values, uniformity appears to exceed oil absorption testing.

D24.21: Carbon Black Surface Area and Related Properties
Chairman: Michael Warskulat
Degussa AG (Germany)
(49)022-3396-4603
michael.warskulat@degussa.com

D1510 Iodine Adsorption Number
George Joyce (Columbian Chemicals Co.) indicated that some concentration limits are not realistic. According to his estimation, multiple titrations would be needed in order to measure the concentrations with the requested accuracy. Furthermore, it would be desirable to refer to a certified primary standard (potassium dichromate, NIST). A proposal was made to broaden the accepted limits of concentrations and to make regressions on the raw data using the SRB HT set. Studies with solutions (iodine/KI and thiosulphate) having deviating concentrations provided satisfactory results after such regression. Discussion did not lead to a common standpoint: some members regarded this approach as pragmatic, others preferred to maintain clear requirements on concentrations of the solutions used for the test; only if problems could not be solved, such regression should be permissible (see D 4821).

D24.31: Non-Carbon Black Components
Chairman: Jeffery A. Melsom
Michelin North America, Inc.
(864)260-2973
jeff.melsom@us.michelin.com

ICBA (European organization) is looking at PAHs in rubber products. A question D24.31 may want to consider in the future is should a method be developed within this committee to measure PAHs and which ones. There doesn’t appear to be any published PAH tests within the carbon black area. The FDA has a method to address low range (500 ppb for 20 products). The committee will need to monitor the progress of this legislation (may become law in Europe in June 2006) and see how this develops globally. Additional information will be presented at the June 06 meeting.

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

D3265 Tint Strength
George Joyce (Columbian Chemicals Co.) presented results from a study where samples were prepared using a high-end coffee grinder and with the Speedmixer. SRB 6 series gave good results – the only exception being A6 (N134) which was found too low. However, it could be shown that by increasing the milling time the results were aligning closer with results obtained using a muller.

D6556 Total and External Surface Area by Nitrogen Adsorption
Tony Thornton (Micromeritics) gave a presentation on the influence of sample degassing on the NSA result. Results indicated that 60 min degassing with N2 was not sufficient for SRB B-6. Appropriate degassing time has to be evaluated by the user of the method as mentioned in sections 8.4.2, 8.4.3, and 8.5.

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D1765 Carbon Blacks used in Rubber Products

D1765 will be balloted to remove the column titled “¢ Stress at 300% Elongation, MPa (psi), ...” from Table 1 Carbon Black Properties. Table a1.1 Range of Data will also be balloted to remove the column titled “Modulus ¢ IRS #7 MPa”.

The following ISO standards have been approved:
ISO 1306:1995 Determination of Pour Density
ISO 8511:1995 Determination of Pellet Size Distribution
ISO 11234:1995 Determination of Dust Content
ISO 6810:1995 Determination of CTAB Surface Area -- There was some discussion about the apparatus being archaic, however, it was approved in the meeting with the agreement a task force will work on a new procedure to incorporate all the proposed changes.

The following ISO standards are being worked on:
ISO/FDIS 1126 Determination of Loss on Heating -- This work is being held up due to the need to provide equipment drawings in a revisable CAD format. ISO 1138/DAmd 1 Determination of Sulfur Content -- Committee decided to consider this as an amendment to the old standard versus a DIS. ISO/FDIS 1304 Determination of Iodine Adsorption Number -- Precision data was developed by ISO members and was included in the final draft standard. The standard was passed on to the Central Secretariat and is delayed due to a change in the secretary. ISO/DIS 1437 Determination -- Precision data was developed by ISO members, was included in the final draft standard, and is currently being balloted. ISO/DIS 4656.2 Determination of OAN and COAN -- New precision data from ISO members has been included. This document also requires revisable format drawings.

ISO 5435; 1994 Determination of Tinting Strength -- New precision data is being collected. ISO 15825 Aggregate Size Distribution by DCP -- The standard will use SRB B-6 as the reference black and will eliminate SRB B-7. ISO 18852 (Formerly ISO/DIS 4652.2) Determination of NSA and STSA -- Standard has been published. ISO CD 21168 Determination of Bound Rubber Content -- Draft is being balloted. ISO CD 21169 Determination of Mass Strength -- This work has been withdrawn.

New Work in Progress- Mercury Porosimetry -- A task force was formed from Germany, France and Italy to look into this work as compared to previous work done under earlier examinations. A report will be made at the next meeting.

The Japanese delegation presented work on a new Individual Pellet Crush Strength Test (ISO vs. JIS). It appears that ASTM has done quite a bit more work in this area so ISO is behind in this area.

ISO is now developing their own R/R data for inclusion in standards.

D24.45: USA Committee to ISO (ISO TC45/SG3/WG3) Chairman: Thomas Powell. Degussa Corporation (330) 665-5120 thomas.powell@degussa.com

D24.45: USA Committee to ISO (ISO TC45/SG3/WG3) Chairman: Thomas Powell. Degussa Corporation (330) 665-5120 thomas.powell@degussa.com

D24.51: Pellet Properties Chairman: Christophe Derrien Cabot Corporation (514)524-2506 christophe.derrien@cabot-corp.com

D5230 Automated Individual Pellet Hardness

A general statement will be included in section 9.1.4 allowing alternate methods for reporting pellet hardness data that is agreed upon between the producer and the customer. Wording will be similar to that presently in 7.2.4.

D24.61: Sampling & Statistics Chairman: John A. Bailey, Jr. Continental Carbon Company (281) 647-3851 jbailey@continentalcarbon.com

D3849 Morphological Characterization of Carbon Black Using Electron Microscopy

A Task Force was formed to focus on improving resolution guidelines and developing an additional method to calculate mean particle size from STSA values.

D4821 Validation of Test Method Precision and Bias

The standard will be revised clarifying the use of the HT standards for a statistical correction for the iodine test is only if a correction is required.

D24.71: Testing in Rubber Chairman: Charles D. Leonard DSM Copolymer (504) 267-3575 charlie.leonard@dsm.com

D3192 Carbon Black Evaluation in Natural Rubber

T. Powell (Degussa) reported on the task force work to find alternative rubber compounds for use in D3192. There were no labs interested in working on a synthetic NR mill mixed formula, so the removal of the D3192 data from D1765 will be balloted in D24.41.

Dxxxx (New Test Method) Carbon Black Dispersion -- Volume Resistivity

L. Nikiel (Sid Richards) gave a detailed presentation on volume resistivity (relevant to D 2663). The committee determined that this work item (WK5299) should be resubmitted as a subcommittee ballot as a new test method.

D24.81: Microscopy and Morphology Chairman: Bonnie McWade-Furtado Cabot Corporation (978)670-7096 bonnie.mcwade@cabot-corp.com

The next LPRS round will use N299 provided by DEC (300 lbs). Data on 5 minute fines and attrition testing will be collected. Samples to be distributed by Feb 15, 2006. Data to John Bailey by April 1, 2006. Results will be reported at the June meeting.