Welcome to the Fall edition of your CAD News newsletter published by Jamie Przybylski. As I sit here writing this message, the leaves are changing colors, there is a noticeable crispness in the air, the World Series is on TV, and it is a reminder that summer has ended. It is also the time of year when we get together in some city conduct our annual CAD RETEC® technical conference. This year was quite special, because we celebrated our 50th anniversary, and that is quite special.

Congratulations to Sandra Davis and her committee for organizing this very special RETEC® in Louisville, KY. The conference was a tremendous success with approximately 480 attendees, which makes it one of the best-attended conferences in our 50 year history. The conference was anchored by a strong technical program, an interesting panel discussion, and it featured over 60 tabletop exhibitors and plenty of professional and social interactions. The conference organizers read the surveys that you fill out and submit, and they try and listen to you, and this is evident in the cities that are chosen, the length and format of the conference, and some of both the traditional and new features of the RETEC®. Finally, I want to say thank you to everyone who attended and financially supported the conference. The proceeds from our RETEC® support scholarships and continuing education for the professionals in our industry. We are insuring the future success of our industry by developing the people who will lead the next generation of color scientists.

Our next technical forum will be at the ANTEC® in Cincinnati, Ohio on April 22-24. Scott Heitzman is chairing this event, and he is busy putting together a full palette of technical papers for you. Please visit specad.org for more information, and you can find a link to register to attend as well.

Our group continues to grow and evolve, and there are a couple of changes I’d like to share with you. First, George Rangos has taken over the Endowment Committee from Johnny Suthers. George has served on the board for quite a long time, and he has served in virtually every capacity including the five year rotation throughout the chair positions. He is very capable of managing this very important function of our Division. We would like to thank Johnny for his years of leadership, during which time he not only managed the funds but he worked directly
Chairman’s Message - continued

with the students that CAD was nurturing. Secondly, I’d like to welcome Mark Freshwater as the newest member to your Board of Directors. Mark received the highest number of votes in the spring election, and he will fill an opening that was created recently. We look forward to working with Mark, and we are confident that he will bring new energy and ideas to the CAD.

Please check the website often, and look for emails from SPE about ongoing events in general. I look forward to seeing everyone in Cincinnati in April.

Best Regards,

Jim Figaniak
CAD Chairperson

Annual Board Elections of Color & Appearance Division

Deadline for Nomination Submissions:
December 15, 2012

The Color & Appearance Division of the SPE will be conducting its annual board elections in February 2013. The election is open to SPE members with CAD as their primary division. Time commitment is four meetings per year including those held at ANTEC® and RETEC®, and participation in CAD activities and initiatives. If you are interested or would like to nominate a colleague, please contact Ann Smeltzer at the noted email address or phone before December 15th.

Nominate a Colleague by emailing Ann Smeltzer at ann.smeltzer@clariant.com, or calling 412-298-4373

Submit your nomination now!

Mission Statement

The Color and Appearance Division of SPE strives to educate, train, inform and to provide professional interaction opportunities to the global community involved in visual performance and aesthetics of plastics.

Disclaimer:
The information submitted in this publication is based on current knowledge and experience. In view of the many factors that may affect processibility and application, this data/information does not relieve processors from the responsibility of carrying out their own tests and experiments; neither do they imply any legally binding assurance of certain properties or of suitability for a specific purpose. It is the responsibility of those to whom this information is supplied to ensure that any proprietary rights and existing laws and legislation are observed.
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The Color and Appearance Division (CAD) commits to the publishing of at least three newsletters a year (four, if there is sufficient material to justify the extra issue). Each newsletter is electronically distributed to our membership of nearly 1,000. Each sponsor’s art directly links to the company’s website.

In addition, we print one of these newsletters on 80# gloss coated stock. All electronic versions are also posted on the SPE website where it available for anyone to download.

For the small donation of $300 per year, we offer a business card sized (2 x 3.5 inches) mention in our newsletter,

We currently have the following slots available for sponsorship:
(2) 2 x 3.5 inch or (1) 4 x 3.5 inch

If interested in learning more, please contact:
SHARYL REID Phone: 864-968.2426
Email: Sharyl_Reid@us.aschulman.com
COUNCILOR’S REPORT

The Fall Council meeting was held on September 15, 2012 in Dearborn, MI. The day before the Council meeting, there were various committee meetings, including Divisions Committee and Conference Oversight Committee. The Fall meeting is the election meeting at which time the next leaders of SPE are selected by the Councilors.

MEMBERSHIP: While membership has shown slow growth over the past couple years, in the first half of 2012, there has been 2% decrease in membership. This has been attributed to reduced retention rates and the scheduling of ANTEC®. It is felt that the retention rates have been negatively impacted due to the difficulties associated with membership renewal using the website which are directly related to the management software being used by the organization. The scheduling of ANTEC® adversely impacted membership numbers because there were less than 12 months between ANTEC® 2011 and ANTEC® 2012, therefore members who joined in order to attend ANTEC® 2011 at the member rate were able to register for ANTEC® 2012 as a member without renewing their membership.

FINANCIAL: Despite budgeting more tightly for 2012 than 2011, the mid-year financial review has shown a $52,000 shortfall. The second half is typically lower in revenue for the organization so there is little expectation of making up this shortfall. Additionally, the impact of ANTEC® Mumbai is unknown. The worst case scenario has a year-end shortfall of $350,000 for the organization. A revised budget is being prepared to minimize the shortfall.

ELECTION: Vijay Boolani was elected by the Councilors as the President-Elect. Additionally, Raed Al-Zubi was elected as Senior Vice-President and Dick Cameron was elected Vice-President.

Sincerely,

Sandra Davis
CAD Councilor

EDITOR’S NOTE

Hopefully many of you had a chance to attend the past CAD RETEC® in Louisville this fall. It was a historic event: the 50th anniversary conference conducted by the Color and Appearance Division of the SPE. Sandra Davis and her team should be commended for making it successful and memorable. One thing that made it memorable for me was the attendance of one special person, Robert Charvat. Not only has Bob been a good friend and mentor to many of us on the board, he has the distinction of being one of the founding members of the CAD and not only attended the very first conference, but also helped finance it. Bob was recognized at the awards reception and given a plaque listing all of the conferences to commemorate his involvement throughout the years. I have included a picture of Bob and his plaque. Bob is no longer a board member, but he stays involved and is always in our hearts. One other thing I would like to mention is that I am the professor in Terra State Community College’s Coloring of Plastics program, which Bob was also instrumental in starting and nurturing. For those of you that might have not seen it in Plastics News in August, there was a nice article about our program. As always, I ask you to spread the word about Terra to your colleagues and contacts that may be in the need of color training. If you have not had a chance to see the article, click here to the link. Have a save and happy Holiday season!

Jamie Przybylski
CAD Editor
COLOR MISCHIEF #7
CAN STORAGE OF SAMPLES OR STANDARDS BE A PROBLEM?

The answer to the title question is "Oh my, yes it can!"

Many organizations store their standards and samples in more ways than anyone wants to take the time to even think about. And! Many of them are just plain wrong.

However, there is one storage method at the top of the list that defies any reasonable technical justification. What is that awful procedure? Storing the standards, or samples, measurement data in a computer memory! Then, believe it or not, using that canned data for subsequent virtual judgments for things like quality control of production batches and other vital issues. In all coloring of plastics adventures, one must try to reduce or eliminate as many variables as are known. Or at least reduce them to manageable values that can be compensated for. It’s the unknown variables that doom projects to failure.

Now, let’s address the chief subject of this article. The total objective of standard and sample storage is to protect the pieces from any environment that might, repeat might, alter their visual and or measurement stability. There are a number of cases where this problem occurs and is totally unknown to the participants. These are the killers.

Did you know the procedure used to cool a sample from a process such as injection molding, as an example, can affect its long term color stability? This can occur before and/or during long term storage. The environmental conditions of the storage environment can affect long term color stability.

If the surface of the stored sample is not completely protected from scratches, smudges and or other defacements the sample is no longer valid for visual and/or measurement evaluations!

A many laboratories store standards and samples in freezers. This is OK so far. But many do not temperature stabilize the sample before an evaluation. This will bias any subsequent testing. This includes removing any surface moisture that may develop as a sample warms to room temperature.

One of the most insidious problems that many times go un-noticed is the wrapping or protection given samples as they go into storage. One very convenient method used by numerous operations is to put the sample in a manila folder then put the sample containing folder into a file drawer cabinet where the cabinet environment is not considered. This may not be a good idea. Is the folder used pH neutral? The composition of folders and/or papers can be highly acidic or alkaline. Either variety may affect the stability of the standards and/or samples stored in these papers. The last one for this message is that many manila folders contain some form of sulfur in the paper. Does any reader remember sulfide staining of polymers and their additives? Maybe this is affecting your samples.

This and many other possibilities may lead to changes in samples during storage. The major puzzle for storage is, be sure you know and understand the storage parameters. If the parameters are not understood, stored samples may not be stable during the storage cycle.

Enough for this mischief for now! As usual, the guilty shall remain anonymous to protect them. Look for the next Color Mischief #8 article soon.
The celebratory SPE CAD RETEC® 2012 was considered a wonderful success by all reports. At the beginning of October, 475 plastics coloring industry professionals gathered in Louisville, KY for an opportunity to learn, share and network. This year’s conference was the 50th anniversary of the first SPE plastics coloring conference and the importance of this milestone was celebrated throughout the conference. The conference kicked off with a Sunday evening reception at Glassworks, an art glass studio, and included a demonstration of the artistry.

In addition to the usual technical sessions, this year, there was an interactive panel discussion where the audience provided their answers to questions which were then addressed by three panelists, Dwight Morgan (Techmer PM/Accel Color), Craig Sibol (BASF) and Larry Nitardy (ComAssist). While the concept was new to this group, the audience responded quickly.

The coordination of the conference was team effort lead by Sandra Davis (DuPont Titanium Technologies), with support from Betty Puckerin (Ampacet), Jeff Drusda (Silberline), Johnny Suthers (retired), Bruce Mulholland (Ticona), Sharyl Reid (A. Schulman), Sharon Ehr (Uniform Color), Tracy Phillips (Uniform Color), Brian West (Techmer PM), Howard Kennedy (Dominion Colour), and Mark Tyler (Ticona).

Right: Bob Charvat receives an honorary plaque for his many contributions to the Color and Appearance Division over the last 50 years.

Invitation to Attend Our CAD Board Meetings

The Color and Appearance Division regularly holds Board of Director (BOD) meetings at the ANTEC® and the CAD RETEC®. In addition, a Summer BOD meeting is typically held about 6 weeks prior to the next CAD RETEC®.

The Summer meeting is scheduled in various locations. A Winter BOD meeting is held in January. The Winter meeting is typically held at a site of a future RETEC®.

Any SPE CAD members who wish to attend are welcome at these meetings. If interested in attending the next Board meeting, please contact the Division Chairperson for more information.
SOCIETY OF PLASTICS ENGINEERS
ENDOWMENT SCHOLARSHIP PROGRAM
FOR THE 2013 – 2014 SCHOOL YEAR

The Endowment Scholarship Program offered by the Color & Appearance Division of the Society of Plastics Engineers awards up to five scholarships each year to students who have demonstrated or expressed an interest in the coloring of plastics industry. The students must be majoring in or taking courses that would be beneficial to a career in this industry. This would include, but is not limited to, plastics engineering, polymer science, coloring of plastics, chemistry, physics, chemical engineering, mechanical engineering, industrial design and industrial engineering. All applicants must be in good standing with their colleges. Financial need is considered for most scholarships.

Undergraduate and graduate scholarships range up to $4,000 annually. Scholarships are awarded for one year only, but applicants may apply for a re-award for each year they are enrolled in school.

Scholarship Eligibility
1. Applicants for these scholarships must be full-time undergraduate students in either a four-year college or a two-year technical program or enrolled in a graduate program.
2. All applicants must be graduates of public or private high schools.

Scholarship Criteria
1. Applicants must have a demonstrated or expressed interest in the coloring of plastics industry.
2. Applicants must be majoring in or taking courses that would be beneficial to a career in the coloring of plastics industry.
3. An applicant must be in good academic standing with his or her school.
4. Preference is given to student members of SPE and also to students who have a parent(s) as a member of the Color & Appearance Division of the SPE.
5. Financial need of an applicant will be considered for most scholarships.

Application Procedure
To be considered for a scholarship from the Color & Appearance Division Endowment Scholarship Program, applicants must complete an application available at WWW.SPECAD.org and return it to the address specified on the application by June 15, 2013. All applications submitted must include:
1. A completed application form.
2. Three recommendation letters: two from a teacher or school official and one from an employer or non-relative.
3. A high school and/or college transcript for the last two years.
4. An essay by the student (500 words or less) telling why the applicant is applying for the scholarship, the applicant’s qualifications, and the applicant’s educational and career goals in the coloring of plastics industry.

Dear Members:

Just a reminder that you can view past and current BOARD MINUTES on the SPECAD website.

We do not typically publish the minutes in the electronic versions of our newsletter, but they are always available for our members to view from our website. The site is is not completely current at this time, as we are in the process of giving it a makeover and new launch in January.

Click here for the link to view:  http://www.specad.org/index.php?navid=28
The beautiful Inner Harbor District is the perfect venue to present your topic to the premier technical conference devoted to the color and appearance of plastics.

Contacts:        Paul Bykowski Brenda Mullins
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Portage, IN                         Cumming GA
219-331-6981                     678-237-2975
paulbykowski@hotmail.com brenda.mullins@clariant.com
On-Line Plastics and Coloring of Plastics at Terra State Community College

A very distinctive feature of the Terra plastics program is its ability to provide “distance learning” (online) courses to students who may reside too far from the Fremont, Ohio campus to participate in full time day or evening classroom activities. This distance learning program has successfully served students globally, as well as locally, for several years.

We all know people within the industry (technicians, sales staff, new hires, etc.) that have no color education to speak of. One aspect of Terra's program that can benefit many of the newer, or under-educated, members of our industry is its internet based, three course certificate program. It is a relatively low cost, no travel, flexible program that the employee can complete anywhere, on their schedule. The three courses provide solid background knowledge for anyone working in the many segments of the coloring of plastics industry. The three courses are:

- **Introduction to Color**
  - Introductory course on color theory
  - Basic background knowledge for anyone working with color

- **Colorants for Plastics**
  - The study of colorant types and their incorporation into polymer materials
  - More in depth treatment than in Intro Class

- **Introduction to Plastics**
  - Introductory course on plastics
  - Polymer types, properties and processing

Courses are an excellent opportunity for newer color matchers, quality control technicians, production technicians, and others to learn more about the coloring of plastics. These courses are also good for people with industry experience, since many of them have learned on-the-job. This is a good opportunity for them to learn the theory behind what they do every day. Students completing this certificate can expect benefits including:

- Quickier color matches
- Better understanding of pigments and their use
- Prevention color problems
- Solve color problems quicker
- Quickier batch corrections in production
- Better understanding of color at processors
- Cost savings

For more information, contact Jamie Przybylski, Program Professor at 419.539.2459 or toll free 866.AT.TERRA, ext. 2459 or email jprzybylski@terra.edu

**Distance Learning Courses Offered**

**Section VI  PET 1100 Introduction to Plastics** (3 Credits)

- Fees: $400 Ohio students/$600 out-of-state
- Books: approximately $200
- Offered Fall 2012: (August 20—December 14)
- Offered Spring 2013: (January 14—May 9)

**Section VI  PET 1240 Introduction to Color** (3 Credits)

- Fees: $400 Ohio students/$600 out-of-state
- Books: approximately $200
- Offered Fall 2012: (August 20—December 14)
- Offered Spring 2013: (January 14—December 14)

**Section VI  PET 2320 Colorants for Plastics** (4 Credits)

- Fees: $500 Ohio students/$790 out-of-state
- Books: approximately $150
- Offered Spring 2013: (January 14—May 9)

Click here for more information.

Plastics News Article highlighting Terra!
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The Filter Pressure Test DIN EN 13900-5 for Pigment Masterbatch Dispersions

Author: Roger Reinicker, BASF Corporation, Newport DE 19804

Abstract DIN EN13900-5 is used to measure the degree of dispersion of pigments or other additives contained in a polymeric matrix. If the preparation of the pigment masterbatch is well defined and, in addition, emphasizes pigment wetting over pigment shear and aggressive pigment development, it is also useful for giving neat or powder pigments a pass/fail on dispersion quality. While the DIN or EN standard test has been around for about five years, it has not been universally adopted by some segments of industry in the NAFTA region. This paper will examine the history and development of the test and compare it to more traditional tests used in the US masterbatch industry, including some correlation data between the new test and older methods. Suggestions for potential improvements will also be offered.

Introduction

The proper use of pigments in plastics necessitates achieving a quality dispersion of the particulates, thus to achieve a number of goals:

- Reproducible color results
- Good economics
- Good physical appearance (lack of specks, streaks, and other defects)
- Efficiency in spinning of fine fibers or blowing of films
- Physical integrity of those plastics which may fail key performance criteria if there are point defects in the matrix.

For example, some plastic constructions may be pressurized, others need to have good impact strength.

In order to achieve success for the latter two items, those who manufacture and use pigments have instituted various tests to insure that the pigments can be properly dispersed or are indeed properly dispersed in masterbatches and concentrates. Among these tests are the pressure test, also know as the screen test or filter value test.

Pressure Test History and Development

Prior to the middle of the last decade there was no standard for this test; companies developed their own tests based upon internal criteria or customer demands. All pressure tests utilize the basic principle from fluid mechanics of the modified Bernoulli equation which predicts for incompressible fluids a rise in head loss or pressure drop with decreasing area of flow. There has existed an ASTM filter test for plastics, but the author is not aware of any specific use by the masterbatch industry. ASTM D3218-07 section 17 describes a test for insuring that polypropylene resins used in monofilament spinning are clean enough for efficient use by predicting filter life. The main thrust of this test being polypropylene resins and not particulate dispersions, and also the large quantities of material required, made this test not really applicable for particle or pigment dispersions. Filter tests that were developed locally in the USA often employed a simple setup of a single screw extruder (often 1” diameter), screen pack, and breaker plate. As in ASTM D3218, use of a melt pump was optional.

**TABLE 1** Some details of ASTM D3218 section 17 filter test

<table>
<thead>
<tr>
<th>reason for use</th>
<th>screening molten PP resins</th>
</tr>
</thead>
<tbody>
<tr>
<td>extruder</td>
<td>1 inch to 2.5 inch (25 - 64 mm)</td>
</tr>
<tr>
<td>melt or metering pump</td>
<td>optional - any type</td>
</tr>
<tr>
<td>screens, composition</td>
<td>304 or 316 stainless steel</td>
</tr>
<tr>
<td>screens, diameter</td>
<td>2,235 inches (56.7 mm)</td>
</tr>
<tr>
<td>screens, mesh, in order</td>
<td>325/50, 325/50, 200X1200 DTW/50, 325/50</td>
</tr>
<tr>
<td>breaker plate (screen holder)</td>
<td>specified 31 hole, 0.125 inch diameter in concentric circles</td>
</tr>
<tr>
<td>volume of material</td>
<td>0.25 x rate/hr plus 30 pounds</td>
</tr>
<tr>
<td>temperature 2</td>
<td>65°C</td>
</tr>
<tr>
<td>throughput</td>
<td>not specified</td>
</tr>
<tr>
<td>max pressure</td>
<td>5000 psig or 34.5Mpa</td>
</tr>
<tr>
<td>procedure</td>
<td>purge 15 minutes, then run 30 pounds</td>
</tr>
<tr>
<td>measure of rate of filter increase</td>
<td>megapascals per kg or psi per pound</td>
</tr>
</tbody>
</table>
Screen packs often used a 325 square mesh screen (44 micron) as the critical screen to collect oversize particles, but later the 165x1400 Dutch twill weave became popular. Temperatures, quantity of material used, loading of the pigment, specifications, etc. varied widely. Around 2004, the DIN EN13900-51 test became available, beginning in Europe but based on existing tests currently done then in Europe, but also in the USA. It must be noted from the start that the standard says absolutely nothing about the creation of the pigment concentrate itself that undergoes test. In the examples it refers to 40% loaded dispersions, but not how to make them; this is beyond the scope of the standard. It was written especially for chromatic and achromatic pigments in thermoplastic carriers that have application in extrusion and meltspinning. What the standard does offer is a method for testing such concentrates that has a reasonable chance of reproducibility from testing lab to testing lab.

**Screen Types**

The standard specifies three different types of screen packs. The screen pack is the critical piece of the entire apparatus. The draft standard does not indicate a preference for any of the screen types nor does it suggest a specification on the rise in pressure over the course of the test. A sealing ring is required for the screen. Typically this is an aluminum border that serves as a sealing surface top and bottom and prevents leakage of polymer around the screen. The practice in the industry of using loose individual screens without an encompassing border should be discouraged, as well as the practice of using an automatic screen changer which then implies that the only sealing force is the pressure generated during the test.

Screens, in increasing order of fineness:

- Screen-pack type 1: a two layer screen pack with the key screen being a 615/108
- Screen-pack type 2: a two layer screen pack with the key screen being 615/132
- Screen-pack type 3: a three layer screen pack with the key screen being 165/1400

Table 2 shows the screen types and wire diameters. Screens must conform to “ISO 9044: Industrial woven wire cloth—Technical requirements and testing”. Table 3 gives other important parameters.

**TABLE 2**

<table>
<thead>
<tr>
<th>TYPE</th>
<th>CONSTRUCTION &amp; APERTURE WIDTH</th>
<th>WIRE 1</th>
<th>WIRE 2</th>
<th>ABSOLUTE RETENTION MICRONS</th>
<th>NOMINAL RETENTION MICRONS</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>TYPE 1</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>screen 1</td>
<td>reverse plain Dutch weave 615/108</td>
<td>0.42 mm</td>
<td>0.14 mm</td>
<td>35 – 38</td>
<td>25</td>
</tr>
<tr>
<td>screen 2</td>
<td>square mesh plain weave 0.63 mm</td>
<td>0.4 mm</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>TYPE 2</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>screen 1</td>
<td>reverse plain Dutch weave 615/132</td>
<td>0.42 mm</td>
<td>0.13 mm</td>
<td>23 – 26</td>
<td>20</td>
</tr>
<tr>
<td>screen 2</td>
<td>square mesh plain weave 0.63 mm</td>
<td>0.4 mm</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>TYPE 3</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>screen 1</td>
<td>twilled Dutch weave 165/1400</td>
<td>0.071 mm</td>
<td>0.040 mm</td>
<td>16 – 18</td>
<td>10</td>
</tr>
<tr>
<td>screen 2</td>
<td>square mesh plain weave 0.25 mm</td>
<td>0.16 mm</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>screen 3</td>
<td>square mesh plain weave 0.63 mm</td>
<td>0.40 mm</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

* estimates


continued on page 14
TABLE 3

<table>
<thead>
<tr>
<th>PARAMETER</th>
<th>TEST STANDARD</th>
<th>ENGLISH UNIT</th>
</tr>
</thead>
<tbody>
<tr>
<td>single screw extruder diameter (D)</td>
<td>19mm to 30 mm</td>
<td>0.75 inch to 1.18 inch</td>
</tr>
<tr>
<td>length of screw (L)</td>
<td>20L/D to 30L/D</td>
<td></td>
</tr>
<tr>
<td>pressure at melt pump</td>
<td>30 to 60 bar</td>
<td>435 to 870 psig</td>
</tr>
<tr>
<td>pressure maximum, achromatic colorants</td>
<td>350 bar 5075 psig</td>
<td></td>
</tr>
<tr>
<td>pressure maximum, chromatic colorants</td>
<td>100 bar 1450 psig</td>
<td></td>
</tr>
<tr>
<td>outside filter diameter</td>
<td>33.8 mm 1.33 inch</td>
<td></td>
</tr>
<tr>
<td>exposed filter area</td>
<td>8.97 cm2 1.39 in2</td>
<td></td>
</tr>
<tr>
<td>throughput rate</td>
<td>50 to 60 cm3/min*</td>
<td>3.05 to 3.66 in3/min*</td>
</tr>
<tr>
<td>screen wire diameter</td>
<td>0.042 mm</td>
<td>1.7 mil</td>
</tr>
<tr>
<td>screen wire diameter</td>
<td>0.071 mm</td>
<td>2.8 mil</td>
</tr>
<tr>
<td>screen wire diameter</td>
<td>0.13 mm</td>
<td>5.1 mil</td>
</tr>
<tr>
<td>screen wire diameter</td>
<td>0.14 mm</td>
<td>5.5 mil</td>
</tr>
<tr>
<td>screen wire diameter</td>
<td>0.16 mm</td>
<td>6.3 mil</td>
</tr>
<tr>
<td>screen wire diameter</td>
<td>0.40 mm</td>
<td>15.7 mil</td>
</tr>
</tbody>
</table>

* The throughput rate is probably better understood in the context of the amount of polypropylene in grams per minute that this volume represents, roughly 40 to 55 grams per minute.

Extrusion apparatus
The apparatus for running a filter test is shown in Figure 1. The typical one-inch diameter single screw extruder with a 24/1 or 30/1 L/D is perfectly acceptable for running the test. The standard however requires a melt (or gear) pump with a feedback control loop to the extruder drive to maintain a constant pressure of the melt at the gear pump. This feedback loop insures a constant volumetric flow through the pump and through the screen. If the melt pump is eliminated from the system, a uniform volumetric flow rate is unlikely to be maintained during the test since single screw extruders pump less at constant rpm as pressure at the end of the barrel increases and in addition, the mass flow becomes pulsating. Systems with feedback loops to melt pumps are common on laboratory fiber spinning lines, and it is likely that such lines could easily be adapted to perform the test with a minimum of cost and re-tooling.

Breaker plate
A specific breaker plate design with nineteen holes is prescribed. The breaker plate also has an influence on the degree of backpressure in the system, which in turn affects the shear given to the pigment under process. Hence, deviating from the exact breaker plate design given in the draft standard is not a good idea, just as is deviating from the described screen packs must be avoided.

Test materials
DIN EN13900-5 does not dictate the type of polymer to be used in the test, either as the “basic test polymer” or as the carrier for the pigment under test. Polypropylene is the most common carrier in our experience, and this is the polymer that was used for development of the standard. In the industry, polypropylene is sometimes used as the pigment concentrate carrier for final application in other polymers, and in BASF’s laboratory nylon concentrates are frequently pressure tested in polypropylene. Appropriate processing temperatures must be selected in the case of mismatched polymers. In our laboratory, 230°C (446°F) is a convenient processing temperature for polypropylene and 265°C (509°F) for nylon 6 concentrates. It is prudent to specify that the basic test polymer undergo its own filter value test perhaps with the finest screen available (165 x 1400) and to place a very low specification on the result. It is not difficult to find polypropylene that has a very low filter value. For other polymers, the development of char must be carefully monitored and appropriate stabilization selected to prevent char formation.

continued on page 15
Under the standard, the pure pigment content and total throughput weight is set for two different mixtures.

**Mixture one** is set for 2.5% pure pigment, total mixture weight including the basic test polymer is 200 grams, and the recommendation is that this is for color (chromatic) pigments.

**Mixture two** is set at 8.0% pure pigment, total mixture weight including test polymer is 1000 grams, and the recommendation is that this is for black and white pigments. The larger volume for blacks (assume carbon black) and whites (assume titanium dioxide) is appropriate for several reasons: higher density for the white, the need for blacks and whites in some applications to have very long runs (for example in film), and finally the nature of some carbon blacks to suffer from undispersible grit in very low concentrations.

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Given the volumetric flow rate, this implies that mixture one will run only about four minutes through the screen and mixture two about twenty minutes. This seems adequate for the blacks and whites but somewhat short of the usual processing time and amount of pigment processed for organic chromatic pigments in the heretofore-practiced methods in the United States. Our lab, for example, has been running about fifty grams of pigment in our version of this test for over twenty years. Larger mixture amounts are certainly allowed by the standard if agreed upon between interested parties, but in no case should the total pigment processed be less than 5.0 grams.

**Interpretation of results**

Running the test involves placing a new screen for each test in the apparatus that has been pre-heated to an appropriate temperature for the polymer, purging with the test polymer, adding and processing the test mixture (mixture 1 or mixture 2) until it runs out, then purging again with the basic test polymer. This will generate a pressure response over time for the pressure transducer marked PT in Figure 2. In the hypothetical chart, TS and TP are the points where, respectively, the test mixture and the purge mixture are added. It is highly desirable that the pressure trace be plotted on a recorder chart or digitally, although this is not required by the standard. Figure 3 is a photomicrograph of a test screen with captured pigment particles.

Note that the pressure trace may show a pressure decrease as the basic test polymer is replaced with the test mixture; this response is typical for a well dispersed chromatic pigment in an olefin carrier under test in polypropylene since these olefin masterbatches frequently contain a low melting component such as a wax. As the test mixture runs through the screen, oversize particles are caught on the screen, reducing the available flow area for the polymer. Since volume flow rate is kept constant by the melt pump, a pressure rise occurs at PT (again due to the Bernoulli relationship). After the test mixture has run out, purge material of 100 grams of the basic test polymer is run through the screen after which the test is complete. The pressure response of the purge material is usually a decline as some of the particles caught on the screen become broken down with time and pass through the screen thus slightly increasing the area available for polymer flow.

The test result is indicated in the figure as Pmax - Ps. Again the standard sets no specification on what constitutes a good or bad result.

**Test report**

The standard requires that the complete test report for a given concentrate contain a reference to DIN/EN13900-5, details about the basic test polymer, the mixture tested, type of screen pack used, melt temperature, and the volume throughput, as well as of course the final result (the pressure rise in bar per gram of pigment) and any notes about deviations from the standard method. This completeness cannot be underemphasized; given the latitude of polymers, mixtures, and screen types allowed by the standard, a given test value, (for example, 5 bar/gram), is meaningless without proper understanding of the other parameters.

continued on page 16
Advantages of the EN13900-5 test

The new protocol has several good features and some advantages over locally developed tests:

- The test is short, running only five minutes with color versus many industry tests that run 30 minutes or more.
- The test consumes much smaller amounts of concentrate or pigment allowing better efficiency when dealing with developmental samples.
- The test employs single screw technology already utilized for this test by much of the industry.
- A melt pump is required to insure constant flow through the screen. Tests that utilize single screws without melt pumps lose flow rate as the pressure rises, and also have differential shear throughout the test.
- Critical parameters such as filter screen configuration and breaker plate are defined.
- The screen is bordered and locked into place to insure no bypass of material.
- The test allows for three different screen types and all three are defined.

Industry Response

So some seven years after introduction, has this standard test been accepted in the US industry, and how has it fared against existing tests? A very unscientific survey conducted by the author found that there has been some adoption of the new test, especially for newly created dispersions and export situations, but also a continued use of older tests, and some suspicion that the new test is not satisfactory for some applications.

Overall, the test seems popular with pigment producers, particularly those who produce in Europe but also may produce at locations across the globe. This makes good sense in that uniformity of testing within a company allows useful comparisons of production capability. The chief drawback of employing DIN/EN 13900-5 in this manner is that the test does not specify how the pigment is to be dispersed prior to the test, hence there also needs to be a strong emphasis on uniformity of the primary dispersion step. Here is where a comparison of intercompany pigment test results will present a challenge, since different companies may claim good pigment dispersion based on a good number from the test without mentioning the pigment dispersion protocol. The subject test is not a test for pigments; the test is for a pigment dispersion. Let the buyer beware.

For concentrate houses, use of the 13900-5 test makes even better sense. Let their dispersion process be whatever it is, proprietary or fully disclosed, but a number from the standardized filter value test should allow inter-company comparisons that make sense. Surprisingly, this is where adoption of the test has lagged.

US producers continue to rely on a variety of pressure or filter tests. Some of this is understandable, since specifications developed between masterbatch supplier and masterbatch user can be long-standing and, once implemented and accepted, difficult to displace. Also, the new test may require capital investment from the one or two machinery vendors that have specialized in machines matching the requirements of the DIN/EN test. Some masterbatch producers have found it beneficial to adopt certain aspects of the test, particularly the melt pump at the end of the extruder before the screen pack. This melt pump isolates pressure build from the extruder and allows the extruder to operate under uniform conditions throughout the test. Without this isolation, as mentioned, shear conditions would vary as the pressure increased.

Another reason is mistrust of the new test since for some situations such as organic pigment dispersions, it calls for as little as 5 grams of pigment to pass through the screen. Locally developed tests in the US often called for 10 to 20 times as much pigment (there are exceptions). Strictly uniform sampling then is a necessity for this test with such a small amount of sample. The DIN/EN test does allow for larger quantities, however, and the author feels that a rise in bar/gram in a defined test is a much better specification than a pressure rise in psig that lacks suitable reference and definition.
Comparison between one local test and DIN EN13900-5

A number of commercial olefin based dispersions were obtained and run in two different filter value tests in the BASF plastics laboratory in Newport, DE, in order to establish a correlation between the older inhouse pressure test and the new DIN/EN13900-5 filter value test. The "local" in-house test, used for more than 20 years, employed a single screw extruder and either a 325-mesh screen for less critical applications, or a 165 x 1400 screen for more critical fiber applications. The test used 1750 grams of nominal 12 MFI polypropylene polymer at 3% pigment loading and ran about 35 minutes at 50°F. The same dispersions were then run on a Dr. Collin filter value machine dedicated to the DIN/EN procedures. Several variations of the DIN/EN test were run, employing three types of screens and also, for black and two inorganic pigments, a higher loading of 8.0% and a longer run of 1000 grams of material versus 200 grams for the usual test.

The sixteen test results are listed in Table 4. three tests were run on the 325 mesh screen and 13 on the finer 165 x 1400 screen (left side of table). Under the existing in-house test, a rise of 800 psig or less over a 35-minute period was acceptable for the 165 x 1400 screen (rates a pass if <26.7 psig/min) and a rise of 75 psig was acceptable for the 325-mesh screen (rates a pass if <2.5 psig/min). Somewhat more than half of these commercial dispersions did not pass this long-established test. The correlation obtained with the DIN/EN test (right side of table), primarily the 108 x 615 Type 1 screen (absolute retention 32 microns, nominal 25 microns), was fair with some notable exceptions. It appears that a value of 1.0 bar/gram or less on the Type 1 screen is adequate comparing against the prior in-house test for the chromatic organic pigments. This is in agreement with the developers of the DIN/EN test who suggested that such a value obtained on the Type 1 screen was indicative of a dispersion that was satisfactory for most critical applications.

Correlation: Using 1.0 bar/g as the pass/fail criterion for the new test, it is possible to make a test to test comparison. There were no false negatives comparing the DIN/EN test to the local test, but there were six false positives, meaning that the new test passed six dispersions that would have been rejected by the older local test. Does this lend some credence to those suspicions of the new test for not employing sufficient pigment passing through the screen?

What could be the reasons for the false positives? Machine to machine comparisons need to be understood. The older machine, in use for some time, had a worn screw and barrel, ran at lower viscosity (higher temperature). The newer machine presumably had tighter tolerances, ran at higher viscosity (lower temperature) meaning more shear was imparted to the pigment dispersion. See Table 4 for some other test comparisons. It is expected then that marginal dispersions might appear "improved" on the DIN/EN test since the lower temperature, higher viscosities, and presumably tighter tolerances would lead to more pigment aggregate breakdown. Further, the PY34 tests were suspect since polymer degradation was noted during the local test, possibly due to inadequate polymer stabilization at the higher temperature.

### Table 4 Correlation between local test and the DIN/EN test using commercial dispersions

<table>
<thead>
<tr>
<th>Pigment in the conc.</th>
<th>Pressure Rise in psig</th>
<th>Time, min</th>
<th>165X1400 psi/min</th>
<th>325 mesh psi/min</th>
<th>P/F Status</th>
<th>ISO/DIN EN13900-5 Status</th>
<th>correlation</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 PY93</td>
<td>40</td>
<td>35</td>
<td>1</td>
<td>0</td>
<td>Pass</td>
<td>bar/1</td>
<td>Type 1, 2.5%</td>
</tr>
<tr>
<td>2 PY95</td>
<td>10</td>
<td>35</td>
<td>0</td>
<td>0</td>
<td>Pass</td>
<td>bar/1</td>
<td>Type 1, 2.5%</td>
</tr>
<tr>
<td>3 PR144</td>
<td>35</td>
<td>35</td>
<td>1</td>
<td>0</td>
<td>Pass</td>
<td>bar/1</td>
<td>Type 2, 2.5%</td>
</tr>
<tr>
<td>4 PR214</td>
<td>0</td>
<td>0</td>
<td>0.0</td>
<td>0.0</td>
<td>Pass</td>
<td>bar/1</td>
<td>Type 3, 2.5%</td>
</tr>
<tr>
<td>5 PR202</td>
<td>840</td>
<td>7.5</td>
<td>117</td>
<td>0</td>
<td>Fail</td>
<td>bar/1</td>
<td>OK</td>
</tr>
<tr>
<td>6 PV19</td>
<td>830</td>
<td>3</td>
<td>277</td>
<td>8.0</td>
<td>Fail</td>
<td>bar/1</td>
<td>OK</td>
</tr>
<tr>
<td>7 PV19</td>
<td>120</td>
<td>15</td>
<td>15</td>
<td>8.0</td>
<td>Fail</td>
<td>bar/1</td>
<td>OK</td>
</tr>
<tr>
<td>8 PV23</td>
<td>120</td>
<td>35</td>
<td>3</td>
<td>0</td>
<td>Pass</td>
<td>bar/1</td>
<td>OK</td>
</tr>
<tr>
<td>9 PB15.1</td>
<td>150</td>
<td>25</td>
<td>6.0</td>
<td>6.0</td>
<td>Fail</td>
<td>bar/1</td>
<td>OK</td>
</tr>
<tr>
<td>10 PB15.1</td>
<td>830</td>
<td>16</td>
<td>52</td>
<td>0</td>
<td>Fail</td>
<td>bar/1</td>
<td>NG</td>
</tr>
<tr>
<td>11 PB15.1</td>
<td>980</td>
<td>16</td>
<td>133</td>
<td>2.0</td>
<td>Fail</td>
<td>bar/1</td>
<td>OK</td>
</tr>
<tr>
<td>12 PG7</td>
<td>830</td>
<td>23.25</td>
<td>36</td>
<td>1.45</td>
<td>Fail</td>
<td>bar/1</td>
<td>OK</td>
</tr>
<tr>
<td>13 PB17</td>
<td>860</td>
<td>25.2</td>
<td>34</td>
<td>0.45</td>
<td>Fail</td>
<td>bar/1</td>
<td>NG</td>
</tr>
<tr>
<td>14 PW6</td>
<td>840</td>
<td>30.3</td>
<td>28</td>
<td>0.09</td>
<td>Fail</td>
<td>bar/1</td>
<td>NG</td>
</tr>
<tr>
<td>15 PY34</td>
<td>800</td>
<td>11</td>
<td>73</td>
<td>0.27</td>
<td>Fail*</td>
<td>bar/1</td>
<td>NG</td>
</tr>
<tr>
<td>16 PY34</td>
<td>800</td>
<td>6</td>
<td>133</td>
<td>0.31</td>
<td>Fail</td>
<td>bar/1</td>
<td>NG</td>
</tr>
</tbody>
</table>

* Both the 325 mesh and the 165 x 1400 mesh screens contained within a pack of several screens with appropriate coarser backing screens to prevent deformity under high pressures.
Given this experience, it seems that all the factors that influence dispersion come into play when comparing tests done on two different testing devices. To say that one test does not correlate with another without first understanding the details of both tests is to avoid the due diligence required to establish a correlation. Therefore in principle, it should be possible to correlate older tests to newer ones, and provide benefit to customers by reporting, uniformly, FPV results in bar per gram of pigment on standardized equipment.

Still, there are areas where the DIN/EN13900-5 test would have to be severely modified to be useful. Some of these situations approach the conditions of the ASTM test mentioned at the beginning of this paper. For situations of creating efficiency in very long runs (e.g., one or two weeks of continuous production) on fiber or film lines, it really is necessary to run quite large quantities of polymer through the machine to look at very dilute concentrations of additives or particulates, or polymers that may produce gels. The latitude to do so, however, is available within the test standard.

Conclusions

The following quote is taken from a European publication soon after the test was established, the authors were from BASF AG, Ciba Specialty Chemicals, and Dr. Collin GmbH.

“The new standard allows a global harmonization of filter test methods and creates a common denominator for partners in business transactions. Used as part of quality control of colorants and concentrates, suppliers of well-dispersed concentrates can now use this method to establish a leading edge over competitors.

The general availability of a normative standard will significantly affect the manufacture of testing equipment and increase the existing pressure on the dispersion quality of colorants and concentrates.

Moreover, The EN13900-5 provides an ideal foundation for establishing filter tests for other applications such as tests for additives, fillers, and polymer materials.”

The mechanics of the DIN/EN test are robust. It is the author’s opinion that older pressure tests can be adopted to use the newer equipment with a melt pump for probable advantages in reproducibility, and certainly to aid in uniform communication and understanding within the industry. In the data gathered for this paper, a filter pressure value of 1.0 bar/gram correlated roughly with a long-established pass/fail in a local dispersion test.

continued on page 19

3 Dr. Volker Königstein, Hans-Peter Meyer, and Dr.-Ing Günter Müllerferli, Kunststoffe plast Europe 10/2004
FIGURE 1: Filter Value Apparatus

FIGURE 2: Fiber Value Pressure Trace

FIGURE 3:
Test screen with pigment particles.
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