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### Historical Perspective on ASTM Gel Standards

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<th>Guide</th>
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<td>ASTM D3351</td>
<td>ASTM D7310</td>
<td>ASTM D3596</td>
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<tr>
<td>Visual/projector</td>
<td>Optical Imaging</td>
<td>PVC – visual/light box</td>
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### Sample Prep
- **Film**
- Flat or tubular extrusion

### Gel Definition
- **“Plastic nodule not blended with matrix”**
- Excludes foreign particles
- **“Any entity large enough to be detected by an optical sensor”; polymer as well as foreign particles**

### Gel Size & Precision
- **400-800μm, > 800μm**
- **Counts per 1520 cm² (4 layers)**
- **Per agreement between supplier and customer**

### Reporting
- **Counts per 1520 cm² (≤100μm thickness)**
- **None**

### Practice
- **Issued 1992**

- Formulated with stabilizer & carbon black
- Compression molded sheet – conditions given

- Hard particle of resin which will not fuse when the plastic mass is subjected to set conditions of hot processing.

- Clear, unpigmented area.

- A. >500μm, ±1
- B. 400-500μm, ±5
- C. 300-400μm, ±12

- Counts per 232 cm² (500 μm thickness)
Evolution of an ASTM Measurement* Standards

- **General**
  - **Guide**
    - Def - a compendium of information or series of options that does not recommend a specific course of action.
    - **ASTM D7310** “Defect Detection and Rating of Plastic Films Using Optical Sensors”
  - **Practice**
    - Def - a definitive set of instructions for performing one or more specific operations that does not produce a test result.
    - E.g. application, assessment, cleaning, collection, decontamination, inspection, installation, preparation, sampling, screening, and training.
    - **ASTM D3596** “Determination of Gels (Fisheyes) In General-Purpose Poly(Vinyl Chloride) (PVC) Resins”
  - **Specific**
    - **Method**
      - Def - a definitive procedure that produces a specific test result.
      - Precision and bias is reported (method capability)
      - **ASTM D3351** “Gel Count of Plastic Film” (Withdrawn)

*Does not include Classification, Specification, Terminology,
Definitions — Clarification of “defect”

3.1.1 defect—for the purpose of this practice any entity in the film that is large enough to be detected by an optical sensor and is either polymeric in nature or caused by degradation, external contamination, undispersed additives or pigments, or similar sources. The defects can be classified into three groups:

3.1.1.1 Gels- A particle of plastic material in the film matrix not blended with the matrix and often acts as a miniature lens.

3.1.1.2 Contaminations- Any particle in or on the film matrix making the film significantly less transparent in transmission mode films than the matrix, or ....; in reflection mode (dirt, insects, oxidized additives or material, catalyst residues, solid particles, metallic particles, undispersed pigments or additives, etc.)

3.1.1.3 Structural defects- Visual deviations not caused by gels or contaminations, e.g. air bubbles, wrinkles, die lines, film holes shark skin, arrow heads.

Typical examples (pictures) are included in a new appendix.
3.1.2 parcel—a user defined smallest area of inspected film for statistical analysis to which a detected defect can be attributed. The statistical evaluation is based on number of parcels.

3.1.3 effective pixel size—distance between adjacent, individual pixels (picture elements) in the analyzed image. The effective pixel size of the optical system is determined by the physical pixel size of the sensor and a magnification factor.

3.1.4 resolution—representing the optical smallest pixel that is used for object size calculation.

3.1.5 Optical resolution—Smallest resolvable distance. = pixel size x 2.3 (area = 4 pixels)
Size Verification Procedure

Calibration object – A transparent plastic substrate printed with circular black points ranging in diameter from 100 to 3000 microns (certified by vendor or confirmed with optical microscopy).

- Place object on running film
- Capture image
- Compare measured diameters to certified values
- Determine resolution from known diameter and number of pixels
- Hardware calibration is performed by camera vendor
Workflow for New Material

1. Determine material properties, MI/MFR and density
   • optionally DSC to determine $T_m$ and degradation temperature

2. Estimate extrusion temperature as follows (linear across zones):
   • Die. $T_m + 20$-$30^\circ$C
   • Feed. $T_m - 10^\circ$C

3. Evaluate suitability considering:
   • Extruder pressure, temperature of melt, film thickness and visual quality

4. Adjust as necessary

5. Establish camera settings
   • Set gray value at 2/3 of range
   • Verify sizing accuracy using calibration object
   • Measure response on a sample over range of sensitivity values. Optimum setting is where the response is stable.

6. Further optimize extruder settings
### Reporting Requirements

<table>
<thead>
<tr>
<th>Standard Report</th>
<th>Evaluation Method</th>
<th>Detection System</th>
</tr>
</thead>
<tbody>
<tr>
<td>• Sample identification</td>
<td>• Extruder line</td>
<td>• Scanner Mode (Transmission or Reflection (and angles of camera and light source to the film for reflection))</td>
</tr>
<tr>
<td>• Date of sample</td>
<td>• Type (Lab or Production scale);</td>
<td>• Light source type, color, and orientation (diffuse or parallel)</td>
</tr>
<tr>
<td>• Method used</td>
<td>• Cast or Blown;</td>
<td>• Effective pixel size</td>
</tr>
<tr>
<td>• Film thickness</td>
<td>• Continuously or batch fed</td>
<td>• Any test settings that establish the criteria for defining the defect area</td>
</tr>
<tr>
<td>• Inspection area</td>
<td>• Operating conditions for the material</td>
<td>(e.g. Sensitivity, grey level, shape factor, use of any type of filters applied to eliminate detected defects)</td>
</tr>
<tr>
<td>• Density, if number of defects/ m² is reported the density needs to be reported for possible recalculation</td>
<td>• Description of the screw</td>
<td></td>
</tr>
<tr>
<td>• Defect observation, type (if different types are defined) and size.</td>
<td>• Description of the die</td>
<td></td>
</tr>
<tr>
<td>• Exclude any defects with a reported size below the size limit of detection established for the optical system, as defects of this size cannot be consistently detected by the system.</td>
<td>• Description of the take-of unit</td>
<td></td>
</tr>
<tr>
<td>• Statistical interpretation</td>
<td>• Description of the distance between die &amp; chill roll, the angle of the film coming the chill roll, position of air knife</td>
<td></td>
</tr>
<tr>
<td>• Identification of instrumentation used</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Variables Impacting Reported Gel Results

- **Continuous pellet feed from process**
- **Melt Extruder**
  - Extruder size and type
  - Number of zones
  - Screw geometry
  - Die design
  - Screen pack
  - Temperature profile
  - Extruder speed
  - Purging /cleaning practices
  - Pressure
  - Motor load
  - Output
  - Temperature at die
  - Residence time

- **Chill Rolls**
  - Sticking
  - Slippage
  - Wrinkles
  - Coating
  - Temperature
  - Speed
  - Die to chill roll distance

- **Air Knife**
  - Air flow
  - Distance to chill roll
  - Angle of orientation

- **Light**
  - Fluorescent
  - Halogen
  - LED
  - Color

- **Environment**
  - Ambient air quality (dust, fibers)

- **Camera**
  - Pixel resolution
  - Gain
  - Line vs. matrix
  - CCD vs. CMOS

- **Winder**
  - Speed
  - Rubber banding
  - Film thickness
  - Film tension

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*Hardware, Software/Operational, Dependent*
Protocol for First Round Robin

• Participation was anonymous

• Sample selection:
  — LDPE samples provided by six resin producers
  — Equipment vendors selected 1 sample unknown to participating companies

• Material information provided: MFR and density

• Sample size: 25 kg standard neutral bag

• Inspected area: 10 m² per test

• Defined size classes and reporting units (counts/m2)

• Equipment used was exactly described in a form created by equipment vendors

• Use local cleaning procedure prior to testing sample
No two equipment setups were identical. Differences in:

- Geometries of the screws, lengths of compression / metering are different
- Diameters are different 19/20/25/30
- Distance between die and chill roll vary (2 mm to 47 mm)
- Temperature profiles were different
- Chill roll temperatures varied from 25-55°C
- 2 companies with conflicting information related to die and screw
- Screw speeds
- Take off speed
Learnings from First Round Robin

- Non-uniform reporting
- Large variability in settings
- Vocabulary not fully clear
- Film thickness varied (no correction applied)
- 3 labs excluded due to not enough data to compare
- 1 major outlier. Note: Only 2 m² area inspected
- Others fall into 2 groups (based on Counts >100µm)
  - Driven by smallest gel class
  - Groups depend on smallest size considered
Data Analysis

![Graph showing data analysis with texts A and B highlighted.](image-url)
Consensus on most important factors
Based on RR data + shared experience

- Temperature of die and temperature profile of extruder
- Extruder torque and pressure
- Screw mixing elements
- Sensitivity + grey levels
- Chill roll (esp. small gels) + air knife settings
- Lighting source
Protocol for Second Round Robin

- Samples: LDPE, HDPE, PP
- Each provided by single supplier (2 x 25kg bags)
- 3 tests, each 10m² inspected area
- Defined thickness (50µm)
- Extrusion temperature profiles set by resin supplier
- Results reports as counts/m² in defined size classes

Data analysis by ASTM is in progress.
Summary of Changes

• Limited by inclusion of resin (lab extruder) as well as film (commercial extruder)
• Definitions: Updated and added additional terms
• Added example pictures of common defects
• Considerations for calibration and verification
• Proposed a workflow to establish test conditions for unfamiliar materials
• Elaborated on minimum reporting requirements
• Precision statement in progress based on round robin including 3 materials (LDPE, HDPE, and PP)
Thank You