QUALITY ASSURANCE & TESTING OF POWDER COATINGS

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ABSTRACT

Quality of powder coatings has been defined as: powder which conforms to agreed upon performance standards.

Quality products can be assured by using the three C's: cooperation, communication and coordination.

The finished powder should be checked upon delivery, sprayed and evaluated as a cured film.

It is important to remember that maintaining a dialog between supplier and consumer is essential to achieving quality powder coatings.

INTRODUCTION

What is Quality?

Quality can be defined as: conforming to agreed upon performance standards.

Quality products can be assured by:

Cooperation
Communication
Coordination

Cooperation:

Joint action towards a common goal.

Do not have hidden agendas.

Let your supplier work with you to achieve success.

Communication:

Convey knowledge of; tell's one's thoughts.

Define the performance properties required to meet your needs. (Be honest.)

Understand . . . every coating's formulation is a compromise. To get certain properties optimized, others will be decreased.

What is <u>really</u> important for the success of the product?

Coordination:

Work together in a harmonious reciprocal manner.

Requirements must be stated clearly so that they can not be misunderstood.

Measurements are then taken continually to determine conformance to those requirements.

Non-conformance detected is the absence of quality.

Quality is measured by the **cost of non-conformance** -- the cost of doing things wrong.

Besides the ASTM, DIN and ISO references covered in this paper, other resources are available for standardized testing and evaluation of powder coating materials:

The PCI, Powder Coatings Institute has published recommended practices and technical briefs covering various aspects of powder coating. Copies are available from the PCI.

Twenty-one Technical Briefs have been published thus far by the PCI.

Quality of Powder Coatings Depends On:

Control of incoming raw materials

Quality control of the powder coating manufacturing process

Quality control testing of the finished powder coating

Control of the cured powder coating film performance properties

This paper will concentrate on Quality control testing of finished powder.

All of the above parameters influence the ultimate performance of powder coatings; therefore, it is important to recognize their interactions.

DISCUSSION:

Final characteristics of powder coatings are dependent on the resin selected and the curing agent type.

Very little can be done to modify the performance or appearance properties of powder coatings once the manufacturing process has been completed. Liquid paints can be tweaked after manufacture.

Powder coatings are ready to use as delivered. Once a powder coating is manufactured, it's cured film properties can no longer be modified. Color adjustment and curing behavior cannot be modified after powder coating manufacture.

Removal of particulate contamination in finished powder coatings is highly unlikely, since sieving cannot differentiate between powder and contaminates of the same size. The powder melts and flows on the substrate, but solid contaminates do not. These particulates show up as dirt or gel in the powder coating.

Since the powder coatings manufacture step is somewhere between the supplier of resins, curing agents and the end user of powder coatings, it is important to remember this position requires good communication and understanding in both directions.

A powder coatings manufacturer must have requirements for their raw materials used in the manufacture of powder coatings. Since the resins and curing agents determine most of the finished powder performance capability, control of the key incoming raw materials is quite important.

Quality of resins and curing agents may be assured by defining:

- softening temperature (glass transition temperature, Tg)
- equivalent weight
- acid value
- hydroxyl value
- molecular weight distribution
- viscosity
- color
- cleanliness

Controlling the Powder Coating Manufacturing Process:

Process Steps	Quality Assurance Tests
Premixing	on premix (melt-mixed on lab extruder)
	color control
Extrusion	**Extrusion**
feed rate	from each batch (extrudate ground, sprayed on test panels and cured)
screw(s) speed	
temperatures (extrudate, screw(s), barrel)	
electrical powder (load)	
	surface appearance
	gloss
	color control
	impact test
	on powder
	particle size
	gel time
	curing schedule
	DSC (if necessary)
Grinding/Sieving/post-blending	Powder
feed rate	particle size analysis
separator speed	fluidity
rotor speed	
rotor amperage	Spray-the powder
separator speed	gloss
air flow (pressure)	color
	impact resistance

The above steps need to be under control to help assure powder coatings meet the end-users requirements.

SOME IMPORTANT POWDER COATING TESTS

1. Fluidizing characteristics
2. Sprayability characteristics
3. Color match
4. Surface appearance
5. Charging characteristics
6. Physical performance
7. Functional performance
8. Density
9. Particle size distribution
10. Storage stability
11. Gel time
12. Plate flow
13. Cure

POWDER QUALITY

Standard:

Unbroken boxes, sealed containers, dry containers.

Non-Conformance:

Broken boxes, torn bags, punctured containers, rain/water soaked boxes.

Action:

Place claim with carrier

Also, communicate problem of non-conformance with powder supplier.

Obtain cooperation to prevent the quality problem in the future.

CHECKING FLUIDIZATION CONFORMANCE

Standard:

Free flowing dry powder which meets an agreed upon fluidization range.

Non-Conformance:

Lumping, spurting, poor expansion of powder (does not fall within agreed fluidization range).

Action:

Communicate deviation in fluidization to powder supplier.

Coordinate remedial actions with powder supplier.

Work together to prevent similar quality problems in the future.

(This includes troubleshooting the entire powder application system; not just the powder).

INSPECTING POWDER AS RECEIVED

Various tests have been devised to indicate the quality of powder as received.

Standard Practices For Testing Polymeric Powders And Powder Coatings were developed by the ASTM subcommittee D 01.51 and were designated D 3451-76.

Three tests currently used by most powder coating manufactures are:

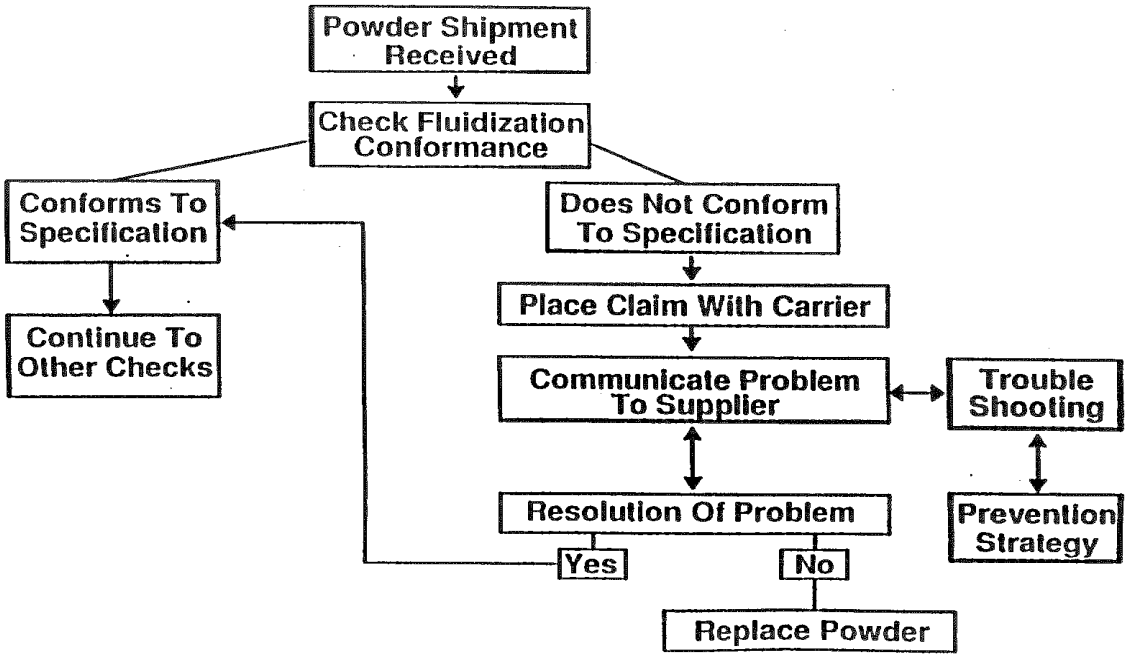
Gel Time

Alpine Sieve Analysis

Plate Flow

Consumers of powder coatings can agree with powder manufacturers on performance standards for gel time, Alpine sieve analysis, and plate flow.

POWDER COATINGS APPLICATION
EVALUATION OF FINISHED POWDER
FLUIDIZATION CHECK



INSPECTING POWDER AS RECEIVED

These tests can be described as follows:

ASTM D 3451-76 14 Gel Time

Interval required at a given temperature for a powder to be transformed from a dry solid to a gel-like state.

Provides reactivity information.

Apparatus:

Thermo Electric Company (Cure Plate)
1948 Columbus Road
Cleveland, OH 44113
(216) 241-6762

ASTM D 3451-76 8.2 Vacuum Sieve Analysis (Alpine Sieve Analysis)

Provides particle size distribution information. Sieving is accomplished by aerodynamics so there is no reduction in particle size and absolutely no wear on the sieves.

Apparatus:

Hosokawa Micron Powder Systems
10 Chatham Road
Summit, NJ 07901
(908) 273-6360

ASTM D 3451-76 17 Inclined Plate Flow Test (Plate Flow)

Consists of a metal rack to hold test panels on which pellets of compressed powder are melted, while inclined 65% from the horizontal.

Provides reactivity and flow information.

INCLINED PLATE FLOW

The inclined plate flow test is a useful indicator of the degree of flow that may occur during the cure cycle of coated parts. This characteristic contributes to surface appearance, however, it should not be used as the sole factor for judgment. The test is recommended as an "in house" tool only to control the described conditions. Oven drafts, angle of repose and pill variations significantly affect results making interlab reproducibility somewhat difficult to correlate.

This test is designed to be beneficial to the raw material suppliers, powder coating manufacturers and end users.

INCLINED PLATE FLOW TEST

Scope

To determine the flow characteristics of coating powders.

Apparatus

1. Constant-Temperature Convection Electric Oven: Thermostatically controlled to maintain a recommended temperature as agreed upon between the purchaser and the seller.

2. Metal Plate Rack Assembly: That fits into inner oven chamber and is capable of being maintained in both a horizontal position and at a 65 degree angle by means of an exterior lever.

3. Glass Panel: Of appropriate size. (1)

4. Balance: Sensitive to 0.01 grams.

5. Steel Pellet Mold Press (2) and knockout rod to make pellets approximately 0.25 inch (6mm) thick by 0.50 inch (12.7mm) diameter.

6. Stop Watch.

7. Steel Rule.

Procedure

1. Place the glass panel to hold the pellet(s) on the metal rack assembly in the oven in a horizontal position. Close the oven door and allow the assembly to remain in the oven to preheat for a minimum of 10 minutes.

2. The pellet weight should be one-half the specific gravity of the test powder measured in grams. Press to 0.25 inch (6mm) thickness and push the pellet out of the mold with the knockout. Place the pellet on the glass panel and allow to remain flat for 30 seconds.

3. Tilt the rack assembly holding the panel to 65 degrees from the horizontal without opening the oven door and allowing the oven to cool.

4. After 15 minutes, remove the panel from the oven and allow to cool to room temperature. Measure the amount of flow as the total length viewed from the backside of the panel.

Report

1. Weight of sample.

2. Type and thickness of glass plate.

3. Oven temperature.

4. Flow in millimeters.

(1) Type and thickness of glass as agreed upon by customer and supplier.

(2) Parr Calorimeter Pellet Press, Cam and Cover type, stainless steel die with standard cavity for making 1/2 inch thickness obtainable from Fischer Scientific Company, Pittsburgh, PA has been found suitable. An equivalent may be used.

Note: A control pellet if possible of known reactivity and flow, should be stored in sealed polyethylene bags prior to use and likewise, pressed pellets should be stored in a desiccator or sealed bags to prevent moisture absorption.

TESTING THE CURED POWDER COATING

One of the first things a consumer of powder coatings should do is spray the powder.

Suitable test panels and parts should be sprayed and baked.

An enormous amount of information can be obtained from testing the cured powder coating.

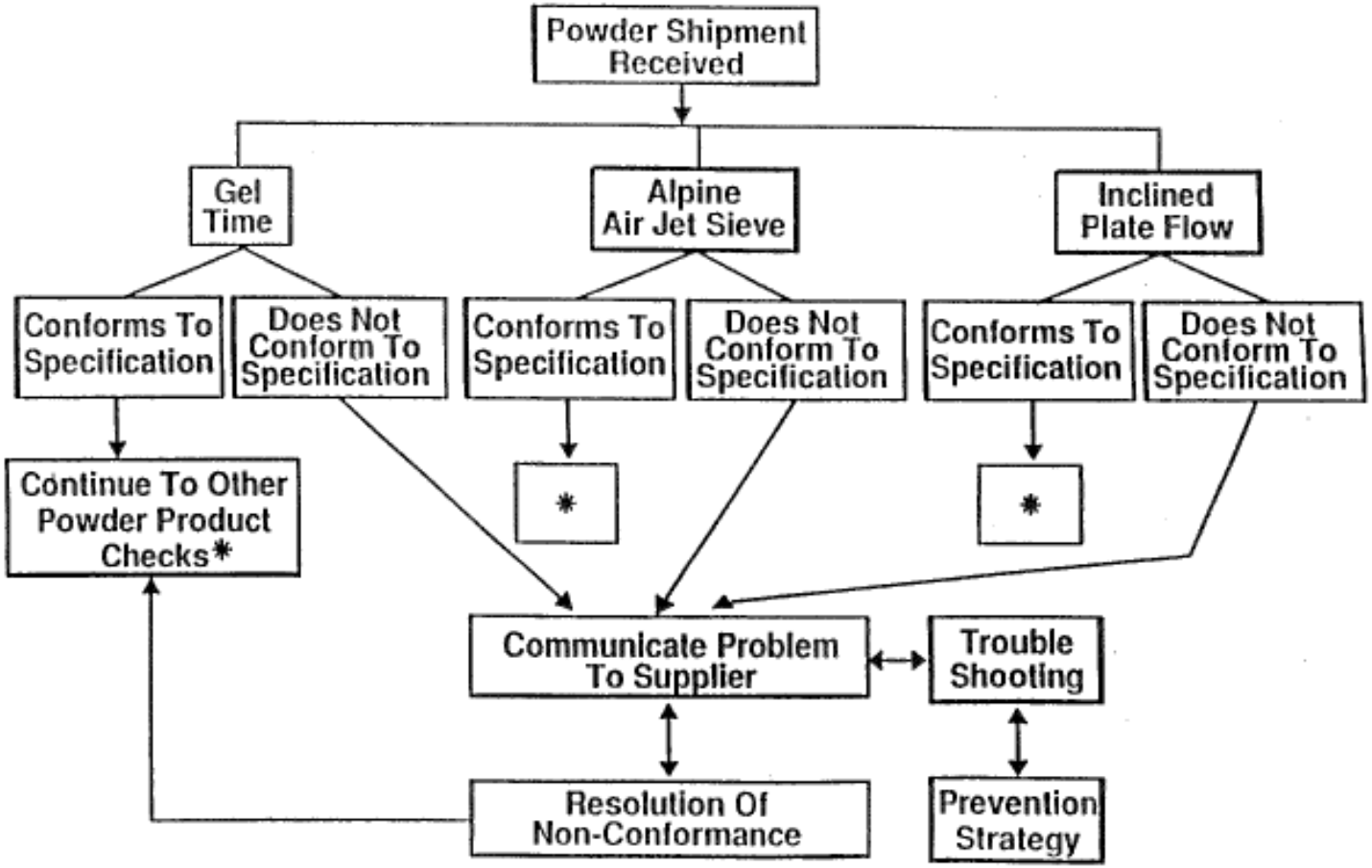
Obvious things, like the general appearance, flow and visual color can be compared to standards.

ASTM procedures for other performance properties can be used to more adequately define the coatings performance needed.

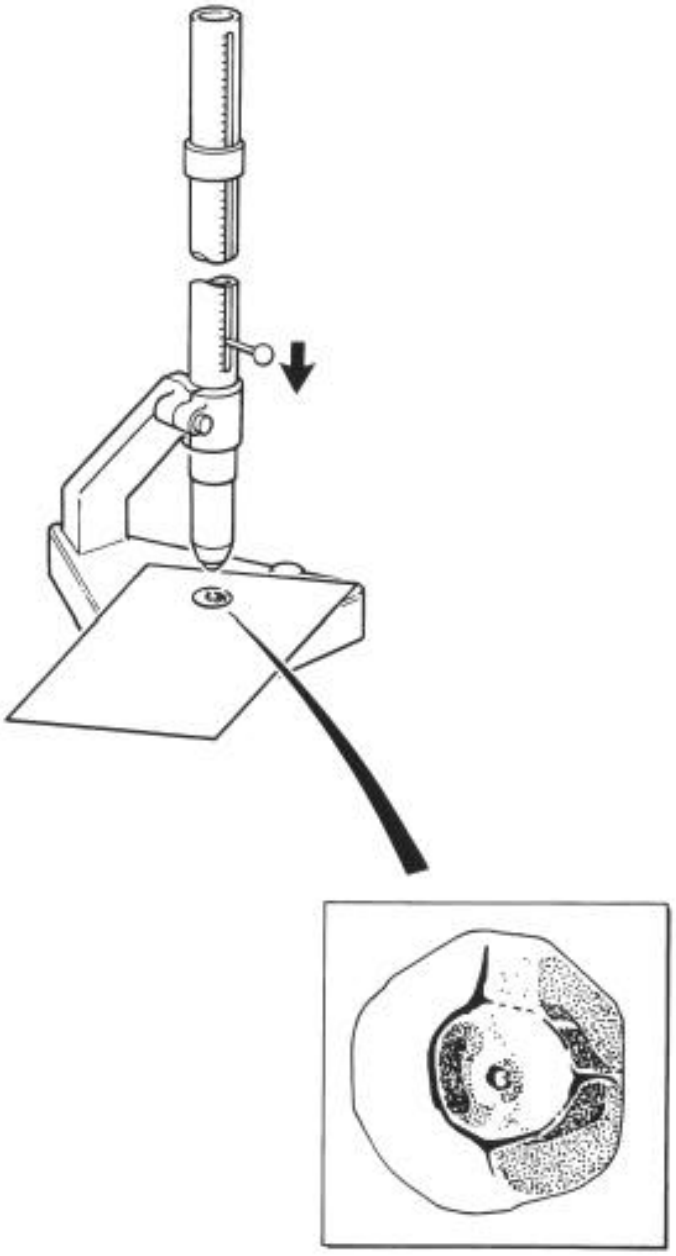
D 523	Gloss
D 2794-69	Impact Resistance F/R
D 522-60	Flexibility (Mandrel)
D 1186	Film Thickness
D 3359	Crosshatch Adhesion
D 3363	Pencil Hardness
D 1044	Taber Abrasion

Define the powder coating's performance properties needed to obtain an acceptable coated product is extremely important. Emphasis should be placed on specification of critical performance areas - not just saying "optimize everything". (For example, if flexibility and impact resistance are very important, then conformance to these areas will be critical to obtaining product acceptance).

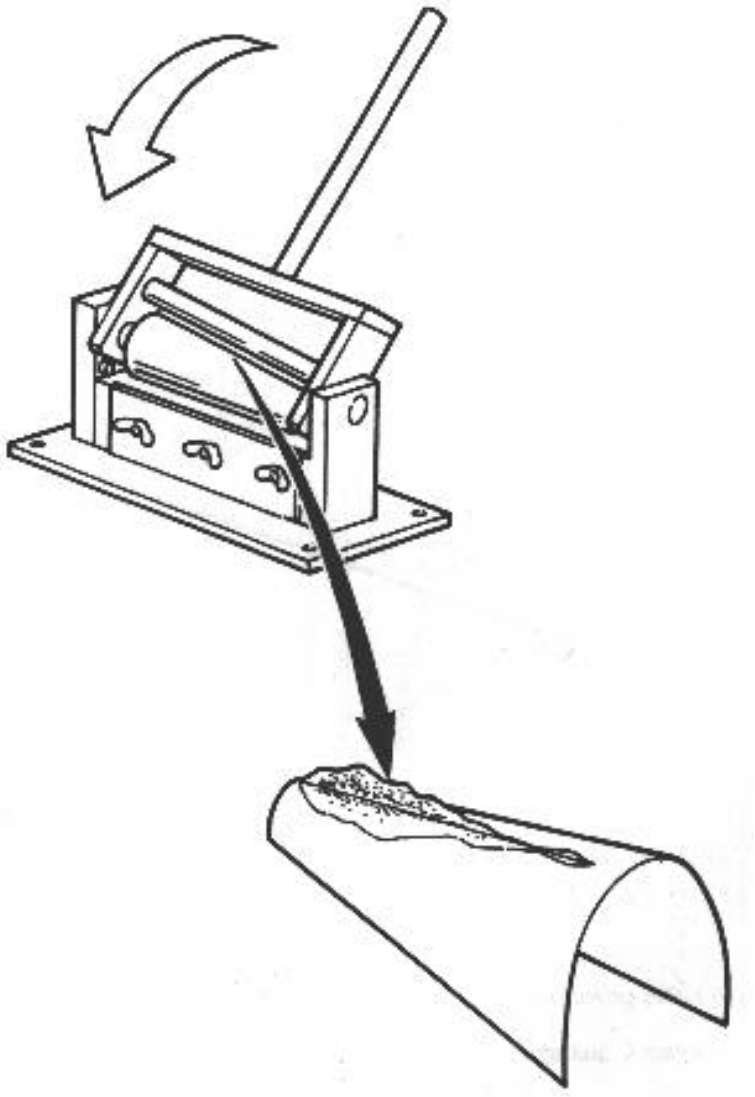
TESTING POWDER AS RECEIVED



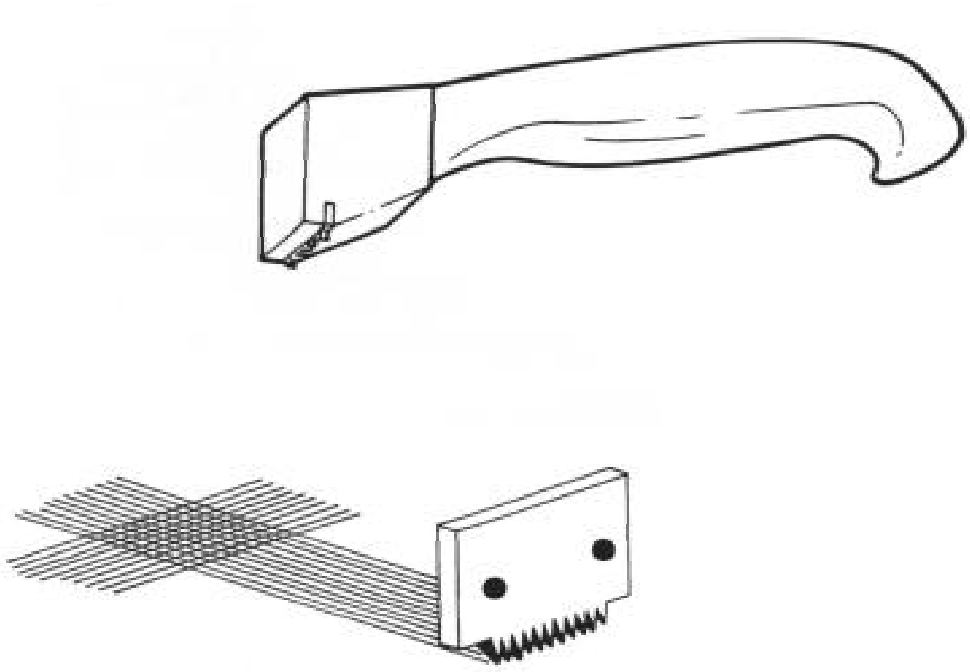
Impact Resistance



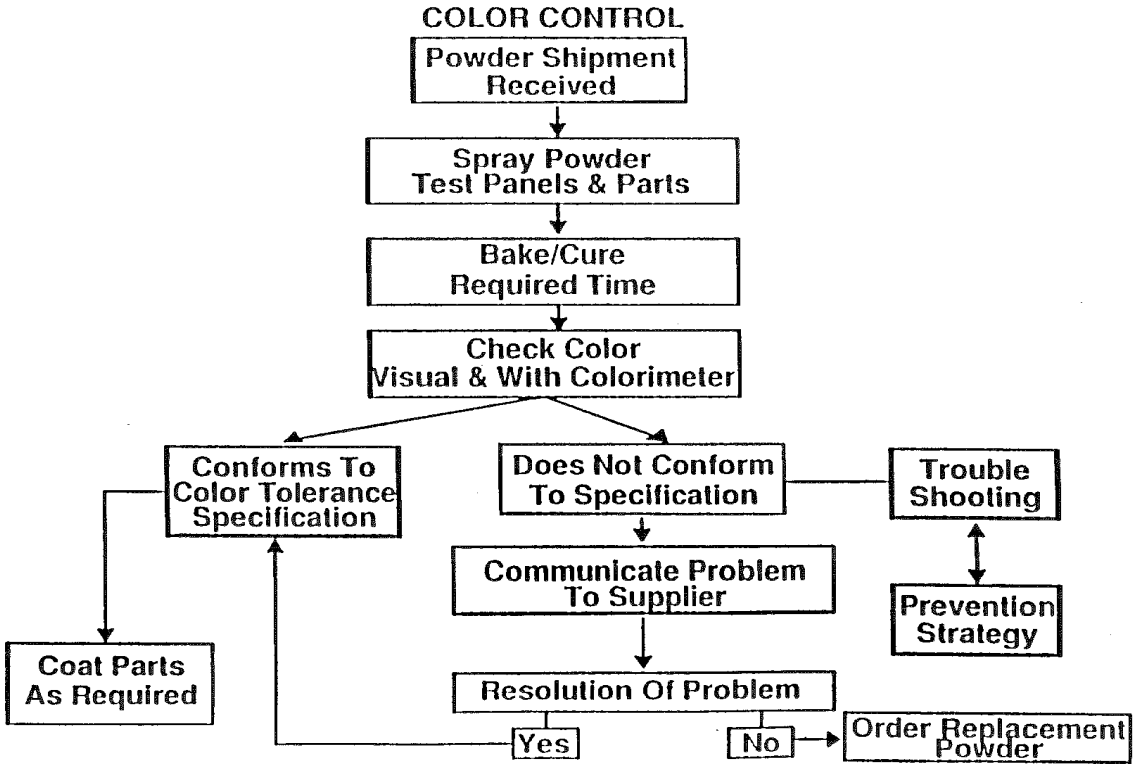
Conical mandrel flexibility test



Cross hatch adhesion test



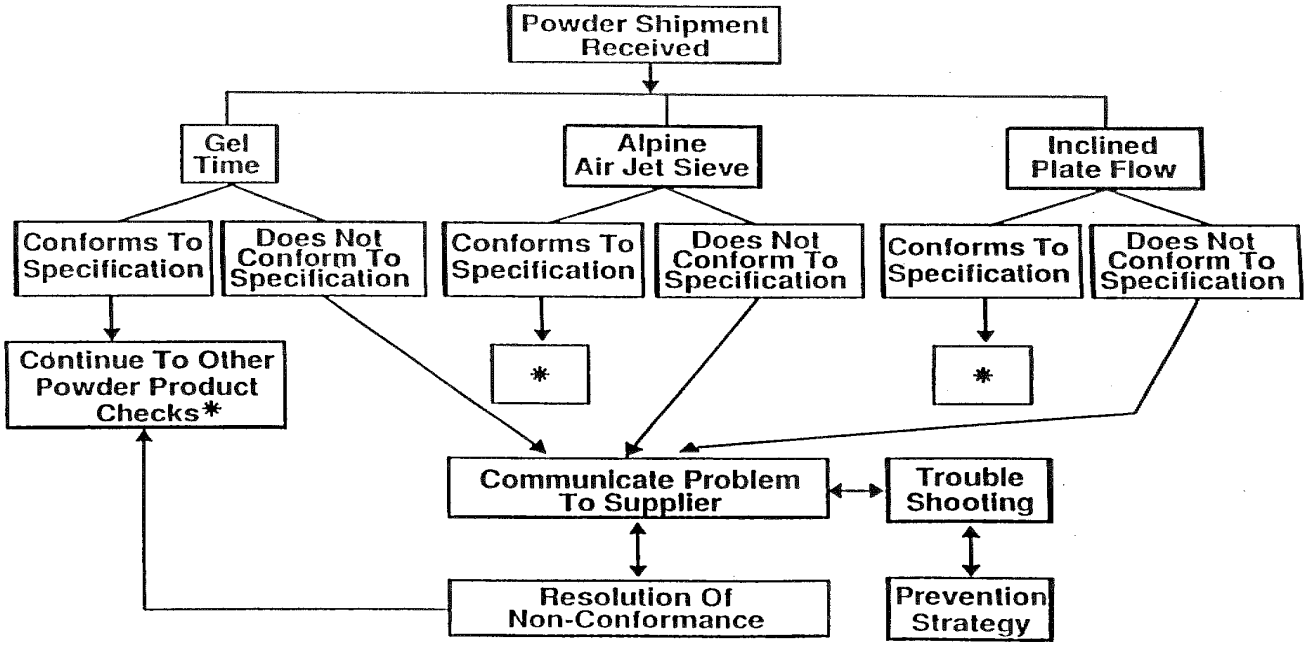
TESTING THE CURED POWDER COATING



INTERNATIONALLY RECOGNIZED TEST METHODS

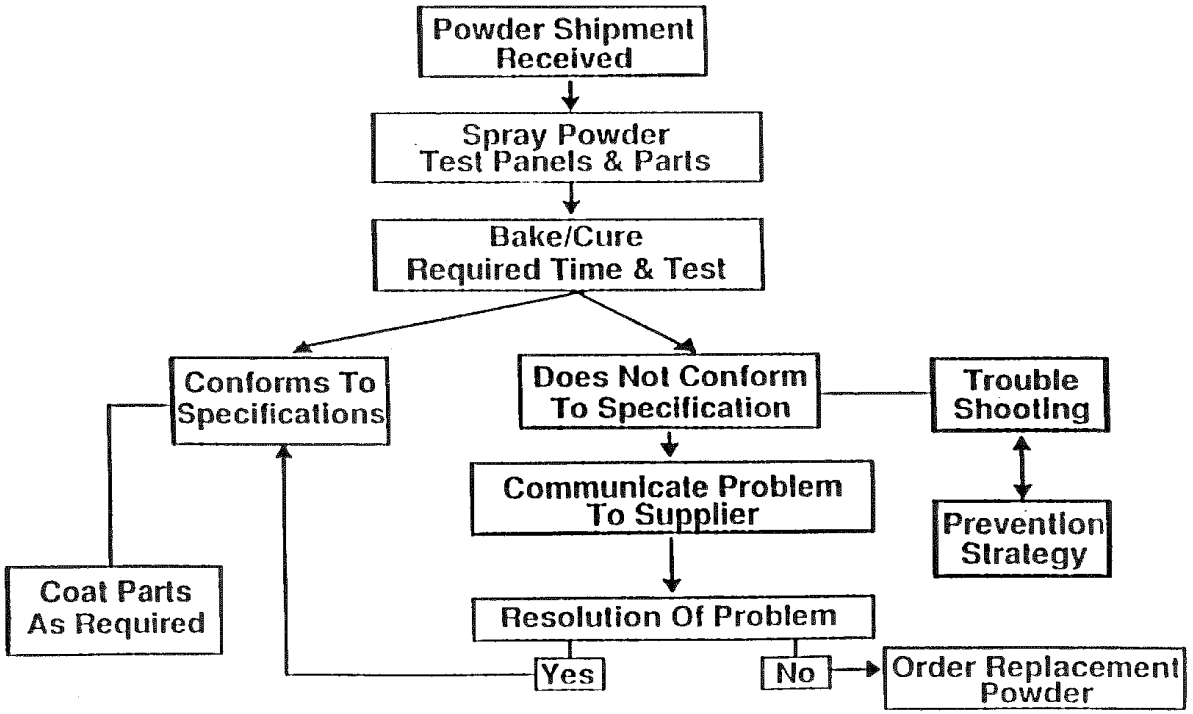
Test Methods	Standards
• Film thickness	ISO 2808; B.S. 3900D5
• Gloss	ISO 2813; ASTM D523; DIN 67530
• Flow (orange-peel)	
• Color: Visual	ISO 3668
• Colorimetry	ISO 7724
• Adhesion (cross-cut test)	ISO 2409; ASTM D3002
• Impact resistance	ISO 6272; ASTM D2794
• Cylindrical mandrel bend test	ISO 1519; ASTM D1737; DIN 53152; NFT 30040
Conical mandrel bend test	ISO 6860; ASTM D552; NFT 30078
• Persoz pendulum hardness	ISO 1552; NFT 30016
• König pendulum hardness	ISO 3711; DIN 53157
• Buchholz indentation hardness	ISO 2815; DIN 53153
• Scratch resistance	ISO 1518; ASTM 2793
• Erichesen cupping test	ISO 1520; DIN 50102; B.S. 3900; NFT 30019
• Pencil hardness	ASTM D3363
• Taber abrasion resistance	ASTM D821; DIN 53774; ANF T 30015
• Heat resistance	- - - - - - - - - -
• Humidity resistance test	ISO 6270; DIN 50017; B.S. 3900 F2
• Kesternich sulphur test	ISO 3231; DIN 50018; B.S. 3900 F8
• Salt spray test	ISO 9227; DIN 50021;
Acetic acid salt spray test	ISO 3769; B.S. 6496 C15
• Mortar resistance	ASTM C207; B.S. 6496 C15
• Chemical resistance	- - - - - - - - - - - -
• Detergent resistance	- - - - - - - -- - - --

TESTING POWDER AS RECEIVED



TESTING THE CURED POWDER COATING

PHYSICAL PROPERTY EVALUATION



TESTING THE CURED POWDER COATING

The Powder Coatings Institute, PCI, is actively working to provide industry recognized standard methods for powder coatings where there are no American Society for Testing and Materials, ASTM, methods.

Included, for reference purposes, are two methods that are being reviewed by the PCI. Eventually, they will be available as industry recognized standards.

POWDER COATINGS PROCEDURE (Solvent Cure Test)

I. **Scope**

1.1 This method describes a procedure for the determination of cure in powder coatings.

1.1.1 Reagent (A) of the procedure is recommended for epoxy powder coatings, or other powder coatings which exhibit a high degree of solvent resistance and are virtually unaffected by strong solvents such as methyl ethyl ketone (MEK).

1.1.2 Reagent (B) of the procedure is recommended for other powder coatings; including hybrids (epoxy cured polyesters), polyester urethanes, TGIC (triglycidyl isocyanurate) cured polyesters etc. This procedure uses a blend of MEK and xylene (1) in a ratio suitable to discern cure.

1.2 This test method can be tailored to more clearly define "cure" in a particular powder coating formulation by either increasing or decreasing the concentration of MEK in the solvent blend.

II. **Summary**

A quick, reliable method for determining cure of thermoset powder coatings was needed. A solvent cure test has been devised which will differentiate between uncured and cured powder coating products. Strong solvents such as MEK (methyl ethyl ketone) are capable of dissolving insufficiently cured powder coating products. Little or no dissolving of cured powder coatings is possible.

Polyester powder coatings products may soften and lose gloss if subjected to pure MEK. To avoid questions about the degree of cure of powder coatings susceptible to attack by pure MEK, a blend of MEK and xylene is recommended.

Typically, an absorbent soft cloth or soft paper towel is folded into a 3" by 3" pad approx. 1/2" thick, saturated with the appropriate solvent blend, attached to the ball end of the ball peen hammer and rubbed back and forth across an area on the suspect panel.

Degree of cure is determined by the number of double rubs attained before softening and loss of gloss occurs.

MEK and xylene are flammable solvents; proper handling procedures must be used. Be sure to consult Material Safety Data Sheets and supplier Technical Data Sheets.

III. **Apparatus**

3.1 Reagents: MEK and xylene.

3.3 Containers (squeeze bottles) to hold mixed or single reagents.

3.4 A 2-pound ball peen hammer.

3.5 Soft cloth or soft paper towel folded into a 3" by 3" pad approximately 1/2" thick.

3.6 Length of copper wire to attach pad to ball end of a 2-pound ball peen hammer.

3.7 Powder coated panel - comparison should be made on the same coating film thickness.

IV. **Procedure**

4.1 Mix MEK and xylene in ratios of:

Regeant A: 100% MEK - 1 container.

Regeant B: 10% MEK/90% xylene by volume - 1 container.

Mixing of other ratios can be used to more clearly discern "cure" in certain instances.

4.2 Fill squeeze bottles with reagent blends. Be sure to properly label each container.

4.3 Fold a soft cloth, such as cheese cloth or cotton, into a 3" by 3" square approximately 1/2" thick.

4.4 Attach the 3" by 3" pad to the ball end of a 2-pound ball peen hammer with No. 18 copper wire, for example.

4.5 Saturate pad with appropriate solvent blend.

4.5.1 Caution: Provide adequate ventilation, consistent with accepted lab practice, to prevent solvent vapors from accumulating to dangerous levels.

4.6 Use Reagent (A) for coatings with strong chemical resistance. Use Reagent (b) for coatings with mild chemical resistance.

4.7 Stroke/slide pad on the test panel while looking for obvious signs of powder coating failure.

V. **Results**

5.1 Interpretation of Results

5.1.1 Results from this test should always be compared with known cured panels representing the same system under evaluation.

5.2 Examples of **degree of apparent cure:**

	Rating
Dissolving to bare metal	No Cure (usually less than 25 double rubs)
Some film removal & softening	Partial Cure (100 double rubs)
No effect on film appearance or gloss	Cured Coating (100 double rubs)

5.3 Report number of double rubs to bare metal or failure if applicable.

VI. **Comments**

6.1 Results from the cure test should always be compared with known cured panels representing the same system under evaluation.

6.2 Interpretation of "cure" is becoming more difficult, as lower hydroxyl polyesters, lower crosslink density products are introduced into the market.

TEST METHODS BEING REVIEWED BY PCI (Powder Coatings Institute)

POWDER COATINGS PROCEDURE (Contrast Ratio)

1.0 **Scope**

1.1 A method for comparing the hiding powder of similar coatings.

2.0 **Apparatus**

2.1 Black/white T-12G panels (Leneta Co., Ho-Ho-Kus, NJ).

2.2 Electrostatic spray equipment.

2.3 Film thickness gauge with 0.05 mil accuracy.

2.4 Forced air oven.

2.5 Spectrophotometer or colorimeter capable of providing the reflectivity value (Y).

3.0 **Procedure**

3.1 Calibrate thickness gauge to the T-12G panels. Note and record the difference in thickness of the existing black and white films on the panel.

3.2 Coat the T-12G panel calibrated in Section 3.1 as directed by the specification for the products being tested, and cure accordingly.

3.3 Find areas on the coated black and the coated white sections that are EXACTLY the same film thickness after correcting for the thickness of the black film and the white film already on the panel.

3.4 Determine the reflectivity (Y) value of each area pin-pointed in Section 3.3. (The test areas must be precisely the same test film thickness.)

4.0 **Calculation**

$$100 \times \frac{\text{Y value over Black area @ X mils}}{\text{Y value over White area @ X mils}} = Z$$

Z = Contrast Ratio

COLOR CONTROL

Standard

Conform to the color tolerance specification. This means that meaningful, accurate color standards are being used by both the powder coating producer and the powder coating consumer.

The powder coating consumer and the powder coating producer must reach agreement on the allowable amount of color deviation.

Non-Conformance

Color does not meet the range agreed upon.

Action

1. **Communication**

   Check that both the powder producer and the powder consumer are indeed using the same color standard.

2. **Cooperation**

   Perform troubleshooting analysis to resolve the cause of the non-conformance.

3. **Coordination**

   Review entire product specification to make sure that there are no conflicting requirements which may affect color.

4. **Coordination**

   Resolve problem; replace powder of necessary.

REFERENCES

Bodnar, Erwin, Powder Coatings - A Technology of the Future (3)
Quality Assurance: Testing Methods and Procedures Euro Coat 11/1993

Data Color International (609) 924-2189
5 Princess Road
Lawenceville, NJ 08648
(Color Measurement Systems)

DeFelsko Corporation..... (315) 393-4450
802 Proctor Ave
Ogdenburg, NY 13669
(film thickness measurement)

Hosokawa Micron Powder Systems (908) 273-6360
10 Chatham Road
Summit, NJ 07901
(Air Jet Sieve and ACM Grinders)

Liberto, Nick, *Setting Up a Powder Coating Test Lab: Why?, How?* Powder Coating, April, 1994

Macbeth (914) 565-7660
405 Little Britton Road
New Windsor, 12553
(Color Measurement Systems)

Powder Coating The Complete Finisher's Handbook,1994, pages 225-240

The Powder Coatings Institute (703) 684-1770
2121 Eisenhower Ave. FAX (703) 684-1771
Suite 401
Alexandria, VA 22314

Q Panel Company (216) 835-8700
26200 First Street
Cleveland, Ohio 44145
(Test Panels and Accelerated Weathering Test Equipment)

Quality Is Free
Philip B. Crosby
(Quality Guide/Reference)

Thermo Electric Corporation (216) 241-6762
1948 Columbus Road
Cleveland, Ohio 44113
(Cure Plate)