POWDER COATING SUMMIT
SEPTEMBER 8-9, 2022

Dispersion, Weathering and Performance of Inorganic Color Pigments

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MARKeting Manager

The Shepherd Color Company

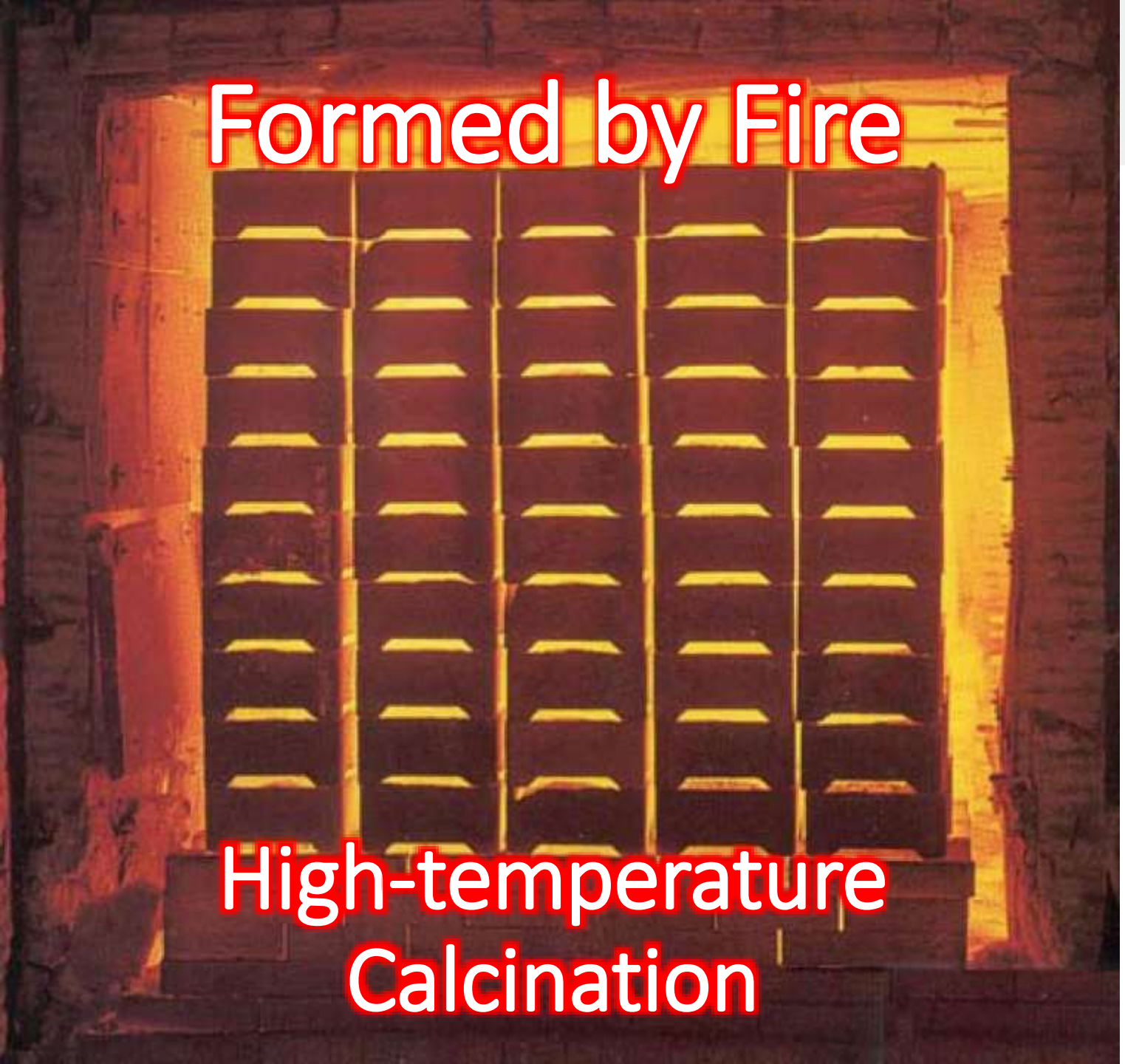
The Shepherd Color Company

- Leading global producer of Complex Inorganic Color Pigments (CICPs)
- Headquartered in Cincinnati, Ohio, with offices in Belgium, Australia, Japan, and China
- Produces thousands of tons of pigment annually

Shepherd

The Shepherd Color Company

We Brighten Lives



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- High-quality oxides and other raw materials are intimately blended
- Calcined at up to 1250°C/2300°F
- Chemically react to form new compounds
- Highly controlled particle size for color and other properties

Complex
Inorganic
Color
Pigment

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Pigment technology comparison

Organic Pigments

Higher chromaticity

Lower opacity

Lower heat stability

Simple Oxides

Lower heat stability

Less range of color

CICPs

Higher opacity

Higher heat stability

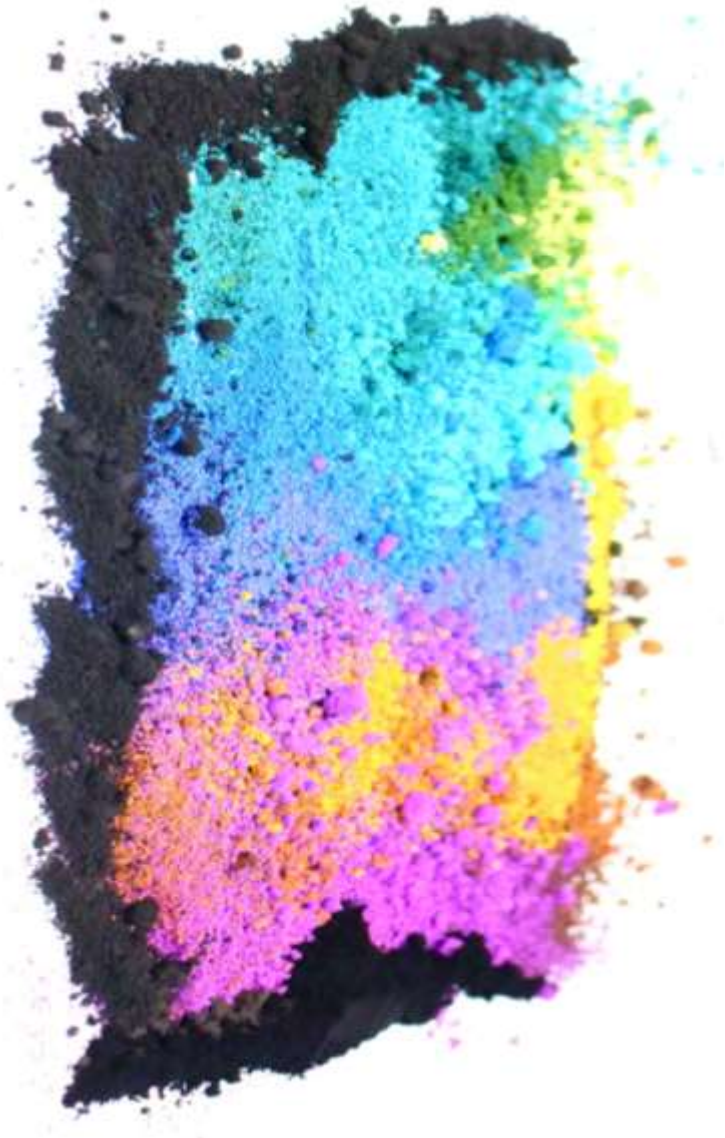
Higher stability

IR reflectivity

Low soluble iron

No red pigment – Low tint strength

CICPs



- Extreme inertness
  - Acids and bases
  - UV degradation
  - Solvents
  - High temperatures
- Broad regulatory approvals
- Expanding color envelope
- IR reflectivity
- Color and function

Topics

- Color pigment dispersion stability
  - Easily-dispersed versus standard
  - Organic versus inorganic
- Tinting of near-whites
  - Reduce yellow cast
  - Organic versus inorganic
- Long-term durability color
  - Fluoropolymer
  - Wide range of inorganic vs

Dispersion Stability Testing

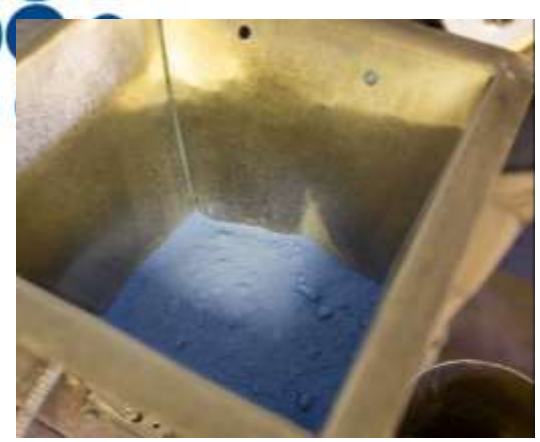
Standard Pigments

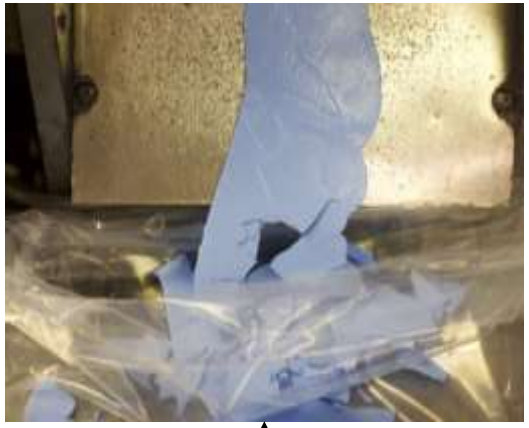
Vs

CICPs and Easily Dispersed CICPs

Pigment Dispersion Testing

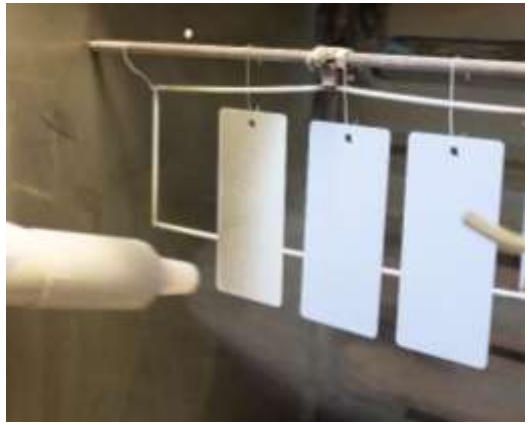
Extrude 1st Time





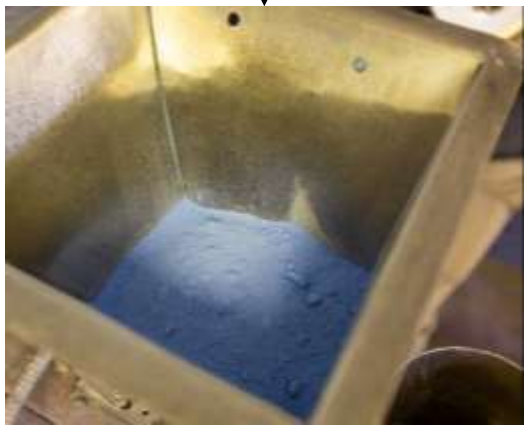
Powder

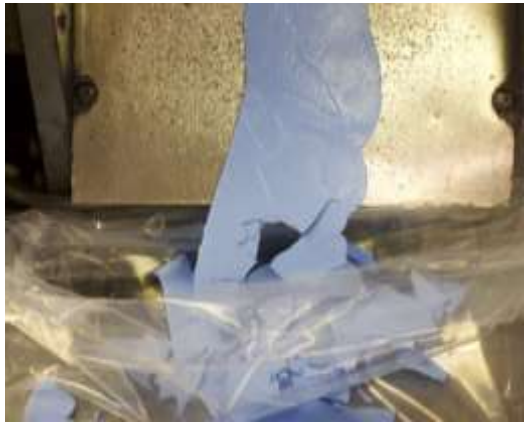
Spray Powder



Record Color Numbers

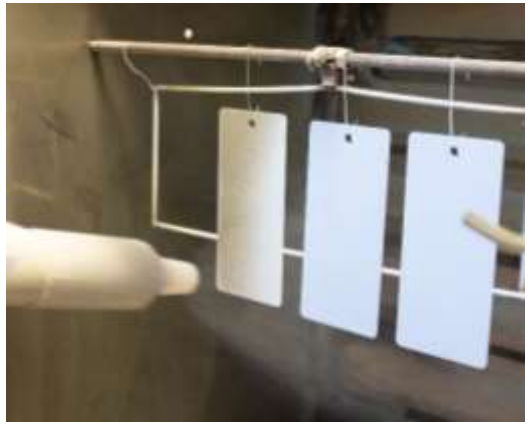
Extrude 2nd Time





Powder

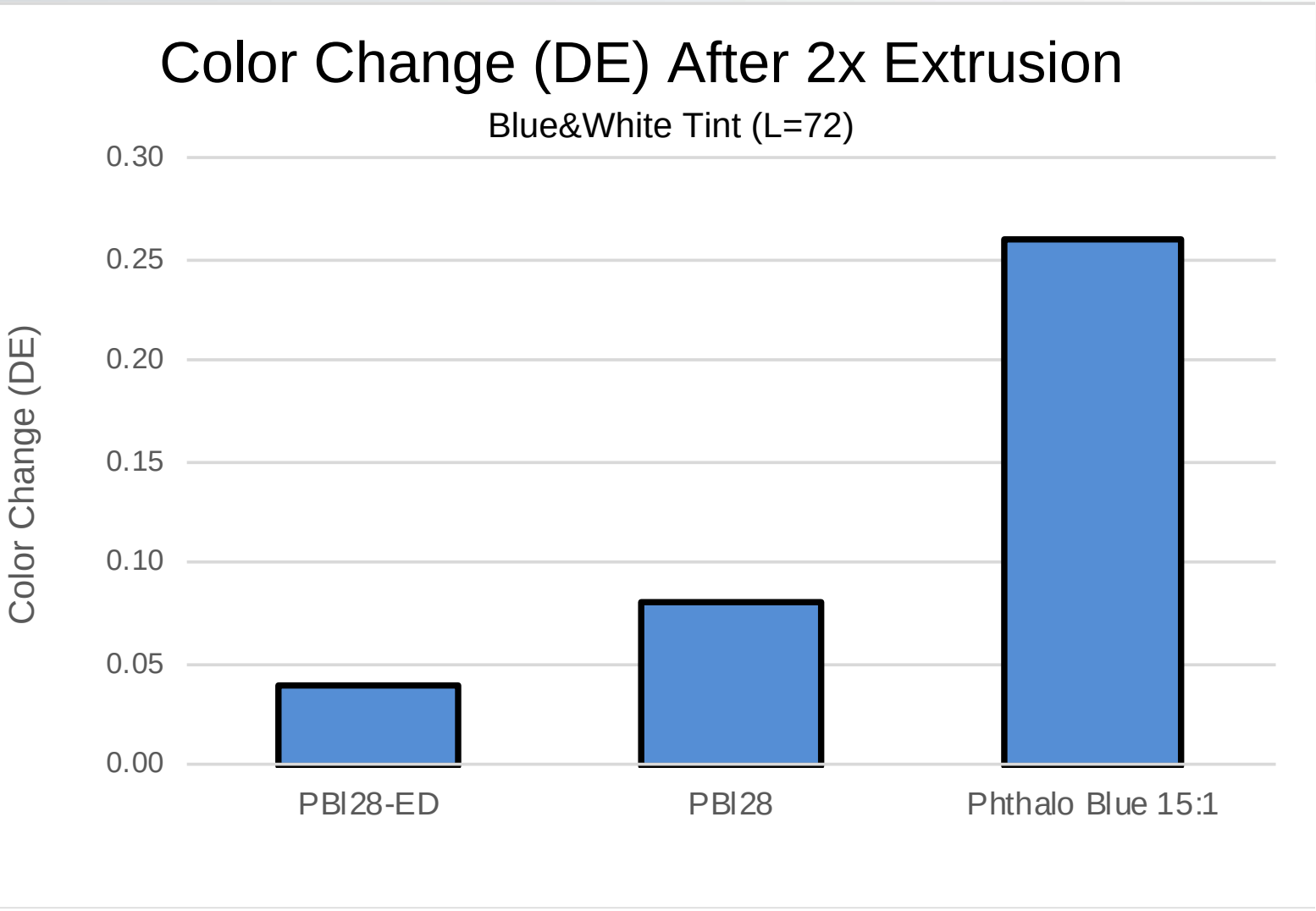
Spray Powder



Compare Color Numbers

Coatings Formulations

Blue	Polyester	438.03	438.03	438.03	438.03
	TGIC	32.97	32.97	32.97	32.97
	Flow agent	6.00	6.00	6.00	6.00
	Benzoin	3.00	3.00	3.00	3.00
	Barium Sulfate	60.00	60.00	60.00	60.00
	TiO2 PW6-High Durability	48.00	48.00	58.00	59.00
	PBl28-ED	12.00			
	PBl28		12.00		
	Phthalo Blue 15:1			2.00	1.00
		600.00	600.00	600.00	600.00

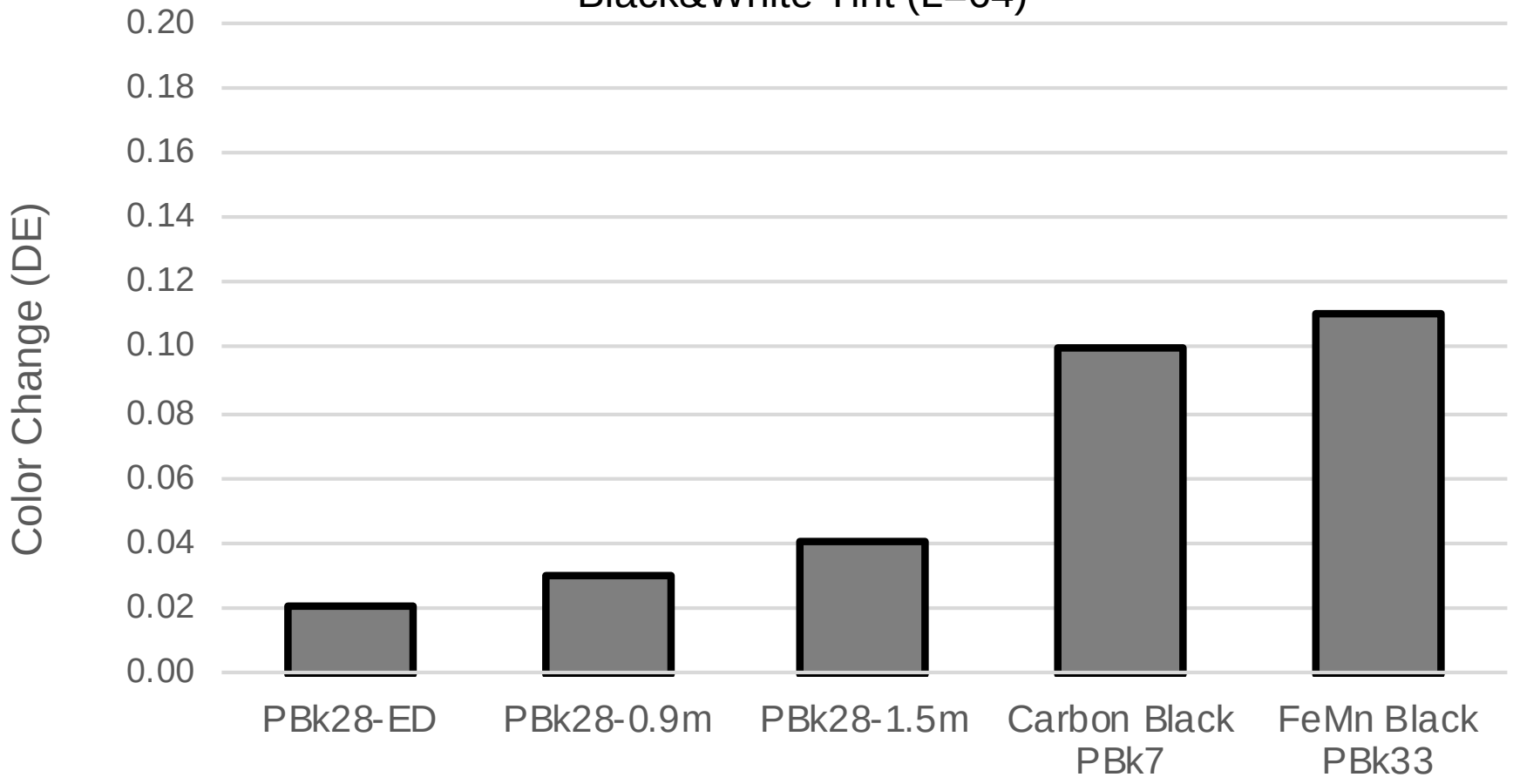


				L	a	b	C	h	DL	Da	Db	DC	Dh	DE
Double Extruded	6	PBl28-ED	4:1	75.09	-8.75	-25.19	26.67	250.84	-0.01	-0.03	-0.01	0.02	-0.03	0.04
	7	PBl28	4:1	75.16	-8.48	-25.09	26.48	251.32	-0.04	-0.02	-0.06	0.06	0.00	0.08
	9	Phthalo Blue 15:1	59:1	70.49	-16.59	-30.48	34.7	241.44	-0.02	-0.03	-0.26	0.24	0.10	0.26

Coatings Formulations

Black	Polyester	438.03	438.03	438.03	438.03	438.03
	TGIC	32.97	32.97	32.97	32.97	32.97
	Flow agent	6.00	6.00	6.00	6.00	6.00
	Benzoin	3.00	3.00	3.00	3.00	3.00
	Barium Sulfate	60.00	60.00	60.00	60.00	60.00
	TiO2 PW6-High Durability	54.00	54.00	54.00	59.00	54.00
	CuCrPBk28-ED	6.00				
	TiO2 PW6-High Durability		6.00			
	CuCr PBk28-1.5m			6.00		
	Carbon Black PBk7				1.00	
	FeMn Black PBk33					6.00
		600.00	600.00	600.00	600.00	600.00

Color Change (DE) After 2x Extrusion

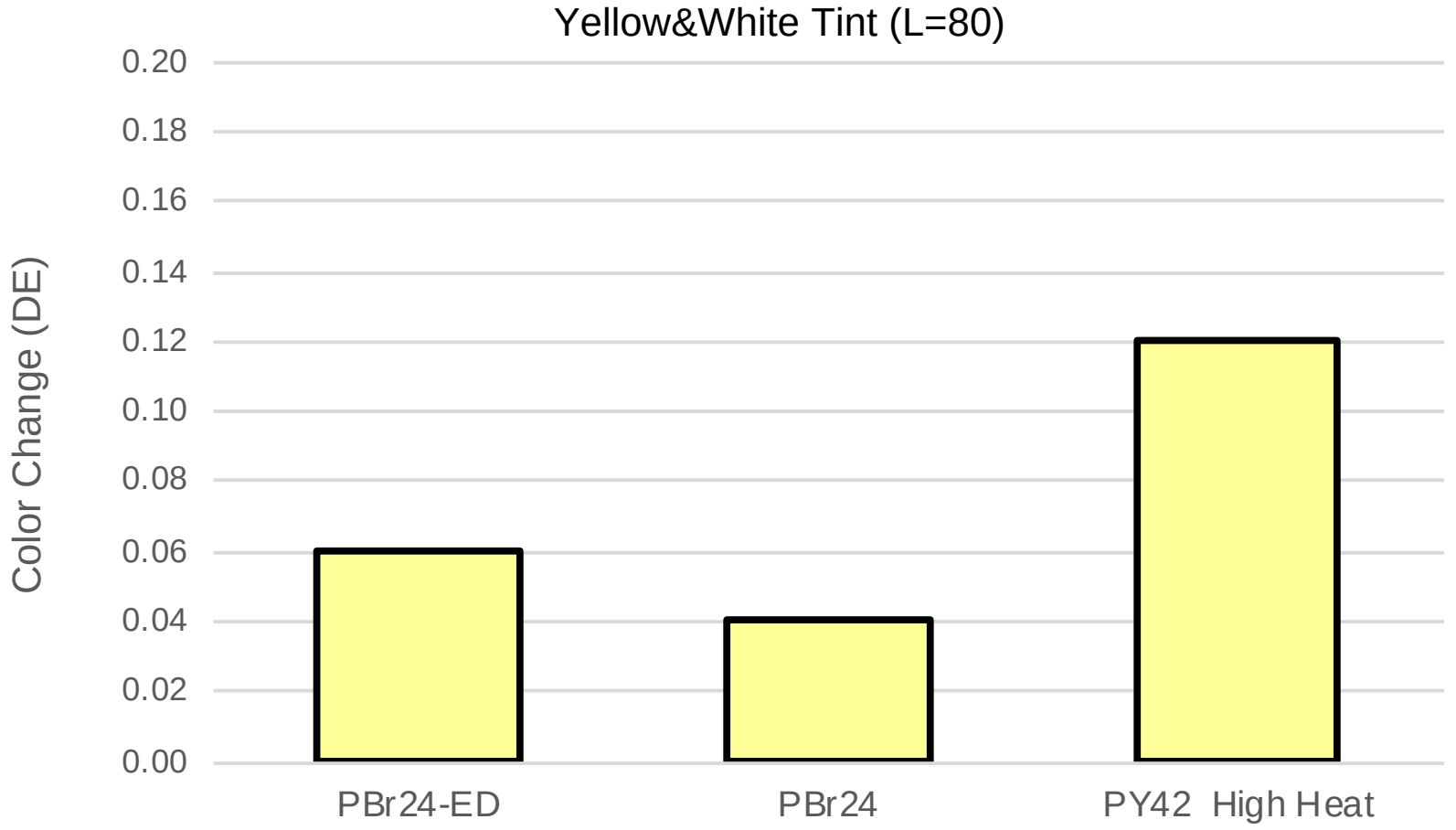


				L	a	b	C	h	DL	Da	Db	DC	Dh	DE
Double Extruded	1	PBk28-ED	9:1	65.74	-1.96	-3.83	4.30	242.89	0.01	0.01	0.01	-0.02	0.00	0.02
	2	PBk28-0.9m	9:1	65.35	-1.96	-3.81	4.29	242.84	-0.03	0.00	-0.01	0.01	0.00	0.03
	3	PBk28-1.5m	9:1	68.28	-1.81	-3.97	4.37	245.48	-0.04	-0.01	-0.01	0.01	-0.01	0.04
	4	Carbon Black PBk7	59:1	60.10	-1.07	-2.89	3.08	249.78	0.09	-0.01	0.01	-0.01	-0.01	0.10
	5	FeMn Black PBk33	9:1	68.74	-0.01	-2.42	2.42	269.67	-0.11	-0.04	0.00	0.00	-0.04	0.11

Coatings Formulations

Yellow	Polyester	438.03	438.03	438.03
	TGIC	32.97	32.97	32.97
	Flow agent	6.00	6.00	6.00
	Benzoin	3.00	3.00	3.00
	Barium Sulfate	30.00	30.00	30.00
	TiO2 PW6-High Durability	72.00	72.00	72.00
	CrSbTi PBr24-ED	18.00		
	CrSbTi PBr24		18.00	
	PY42 Heat Stable			18.00
		600.00	600.00	600.00

Color Change (DE) After 2x Extrusion

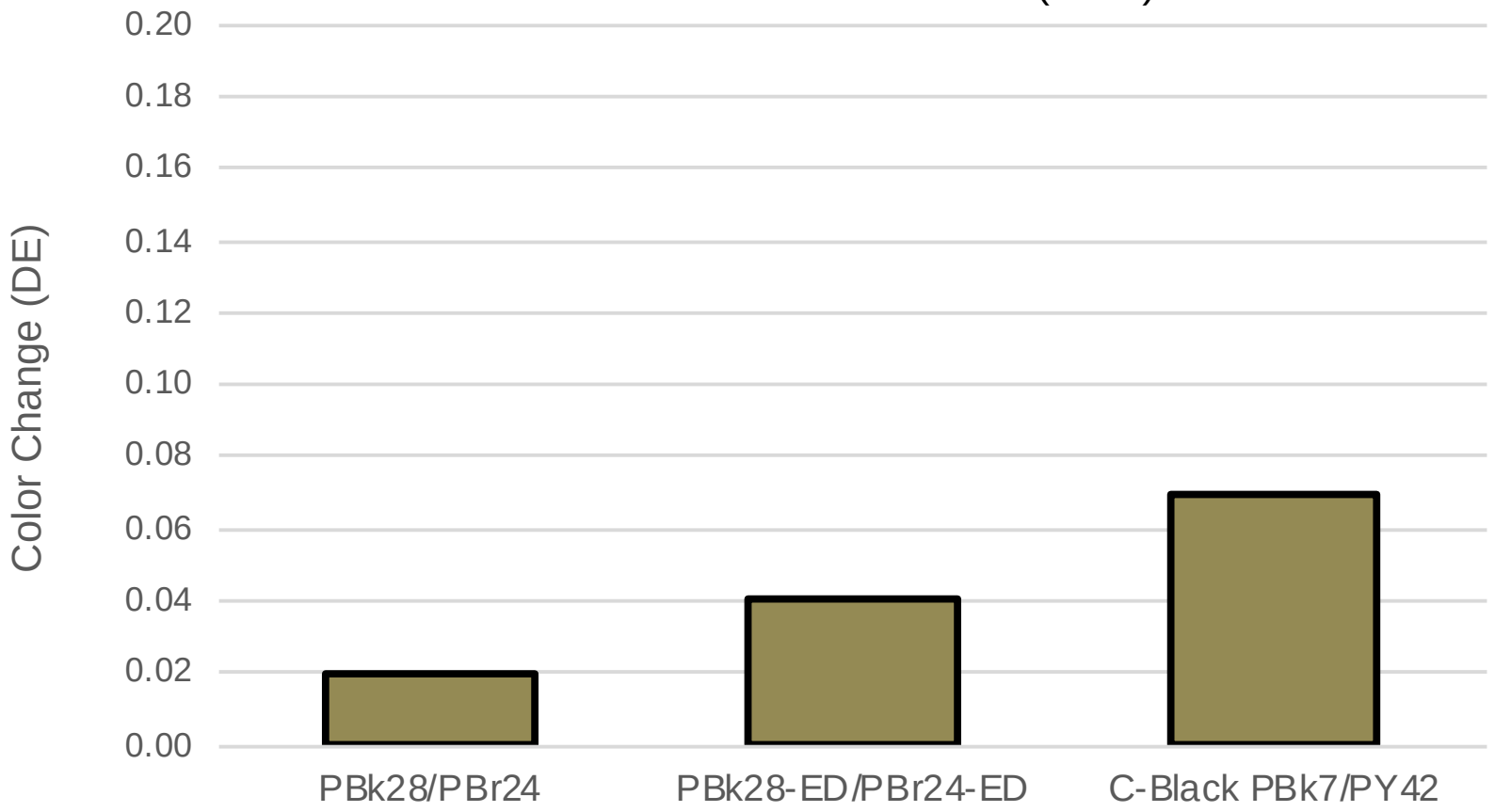


				L	a	b	C	h	DL	Da	Db	DC	Dh	DE
Double Extruded	10	PBr24-ED	4:1	84.33	6.51	33.01	33.65	78.85	0.00	0.05	0.03	0.04	-0.05	0.06
	11	PBr24	4:1	84.18	6.36	33.85	34.44	79.35	0.01	0.04	0.01	0.02	-0.03	0.04
	12	PY42 High Heat	4:1	80.27	8.87	34.58	35.7	75.61	-0.05	-0.07	-0.08	-0.09	0.04	0.12

Coatings Formulations

Tan	Polyester	421.29	421.29	421.29
	TGIC	31.71	31.71	31.71
	Flow agent	6.00	6.00	6.00
	Benzoin	3.00	3.00	3.00
	Barium Sulfate	60.00	60.00	60.00
	TiO2 PW6-High Durability	60.00	60.00	64.90
	CuCr PBk28-0.9m	6.00		
	CrSbTi PBr24	12.00		
	CuCr PBk28-ED		6.00	
	CrSbTi PBr24-ED		12.00	
	Carbon Black PBk7			1.10
	PY42 Heat Stable			12.00
		600.00	600.00	600.00

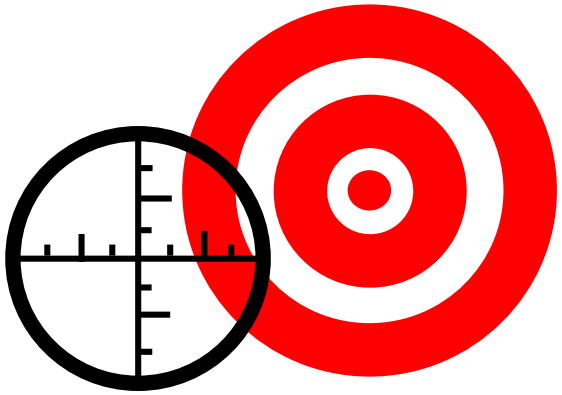
Color Change (DE) After 2x Extrusion



				L	a	b	C	h	DL	Da	Db	DC	Dh	DE
Double Extruded	13	PBk28/PBr24	10/1/2	65.77	-2.87	9.53	9.95	106.74	-0.02	0.00	0.00	0.00	0.00	0.02
	14	PBk28-ED/PBr24-ED	10/1/2	65.92	-2.72	9.17	9.57	106.49	0.04	0.00	-0.01	-0.01	0.00	0.04
	15	C-Black PBk7/PY42	59/1/11	58.69	-1.18	7.11	7.21	99.44	0.06	-0.01	-0.04	-0.03	0.02	0.07

Dispersion Testing Results

- CICPs in ED and standard form develop little to no color when extruded twice.
- Standard pigments continue to disperse and develop color
  - Carbon Black (PBk7)
  - Phthalo Blue (PBl15.1)
  - Iron Oxide Yellow (Heat Stable) (PY42)
- Stable color
  - Consistent color batch to batch
  - Ability to re-extrude to color correct without it being a ‘moving target’
  - How much of your color tolerance is ‘eaten’ by continued color development



With CICPs

How much of your color tolerance is taken up by the shift in color?

Trying to hit a drifting target.

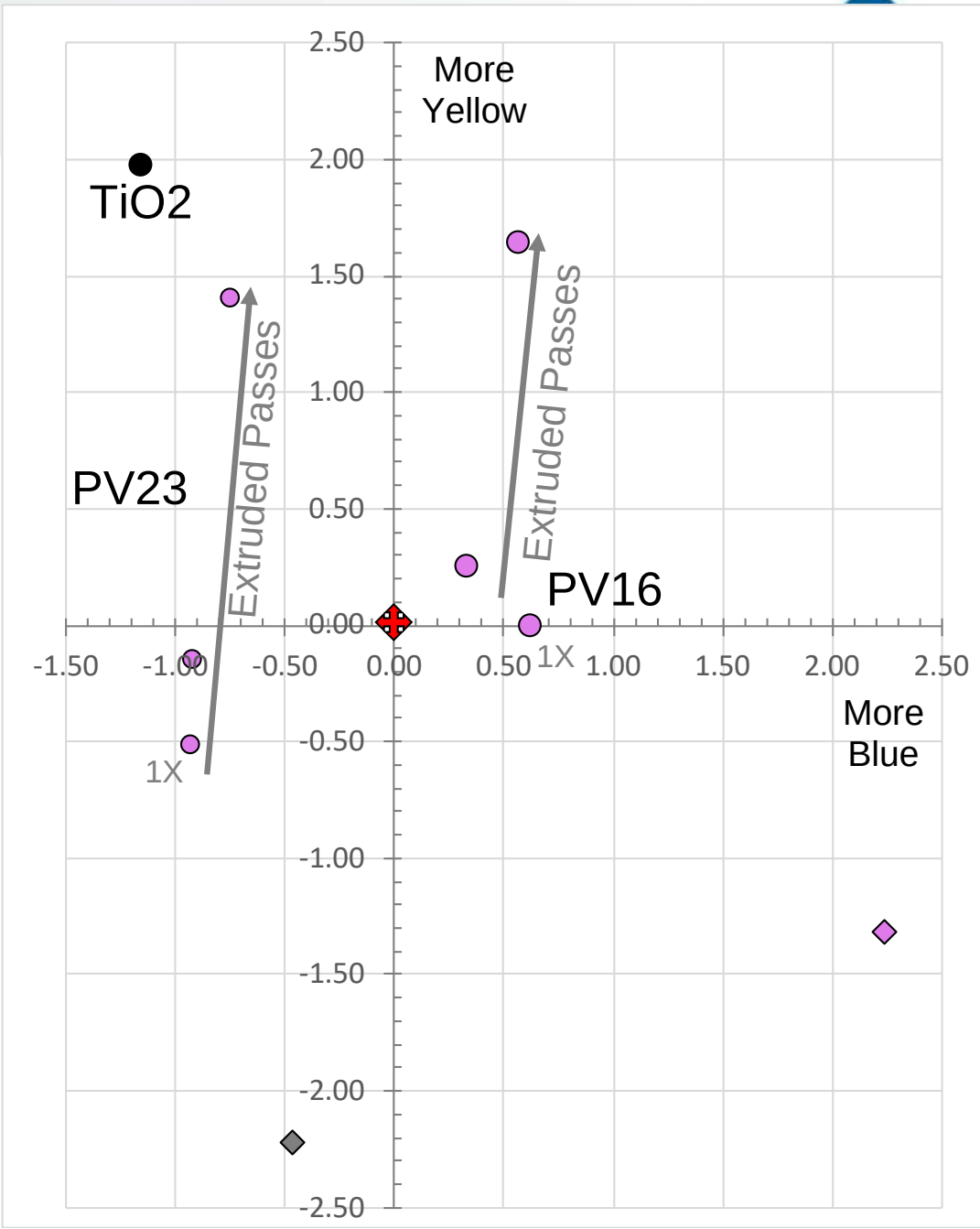
Toning of white colors

- Reduce yellow cast of resin in straight white pigmented coatings
- Organic and Inorganic violets
  - PV16 : Manganese ammonium pyrophosphate
  - PV23 : Carbazole violet
- Color tone and controllability

Violets toning white

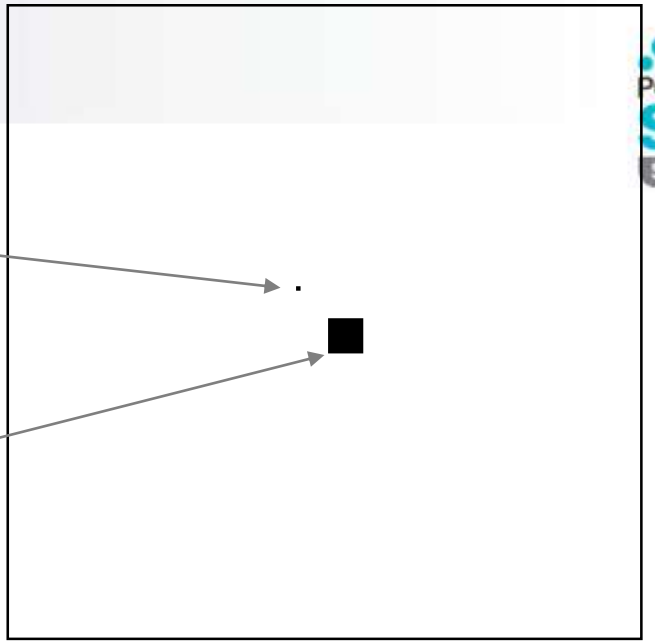
- HAA curing agent

	Times Extruded	L*	a*	b*
Control TiO2		96.67	-1.16	1.97
PV16 1/100	**1X**	93.42	2.24	-1.32
PV16 1/200	**1x**	94.57	0.62	0.00
PV16 1/200	**2x**	94.18	0.33	0.25
PV16 1/200	**3x**	94.13	0.57	1.64
PV23 1/6000	**1X**	93.79	-0.47	-2.22
PV23 1/12000	**1x**	94.86	-0.93	-0.52
PV23 1/12000	**2x**	94.73	-0.92	-0.15
PV23 1/12000	**3x**	94.48	-0.75	1.40



Violets toning whites

- PV23 Carbazole violet
  - More efficient
  - Less controllable
- PV16 Manganese violet
  - More controllable
  - No need for pre-mix
- Both:
  - Reduced L* value about 2 points
  - Stable color development over powered by resin yellowing with increased passes



Weathering Studies

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Standard Pigments

Vs

CICPs and Easily Dispersed CICPs

Historical Weathering Performance: Fluoropolymers and CICPs

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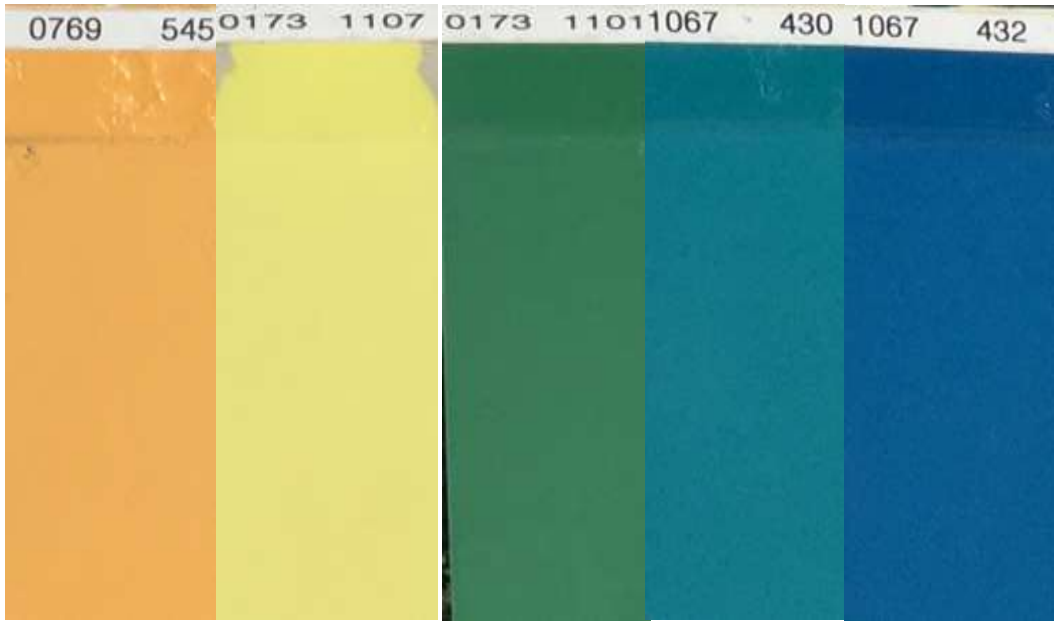
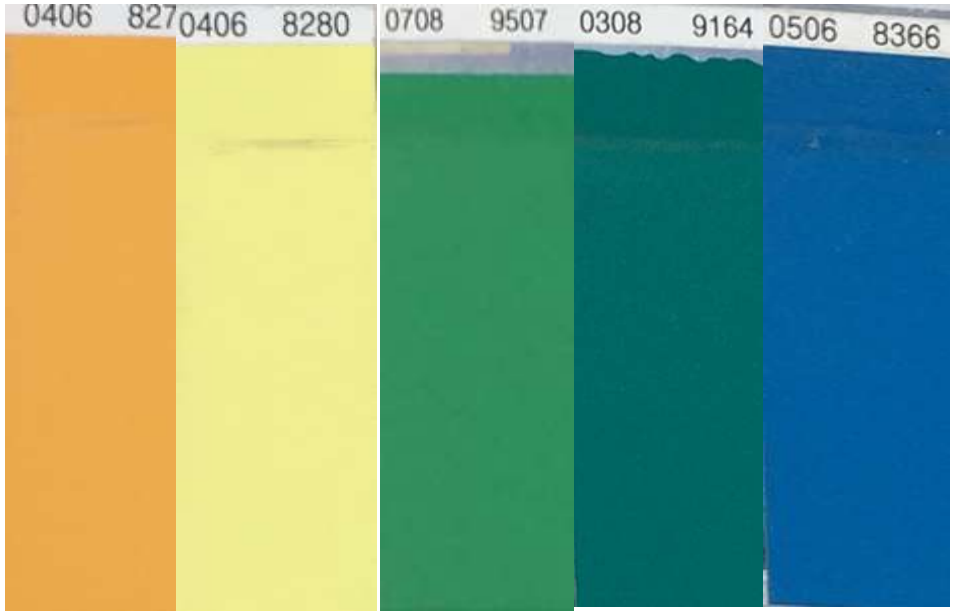
50 Year PVDF/Acrylic Coil Coating



Unexposed Portion

Exposed Portion

10 Year PVDF/Acrylic WB



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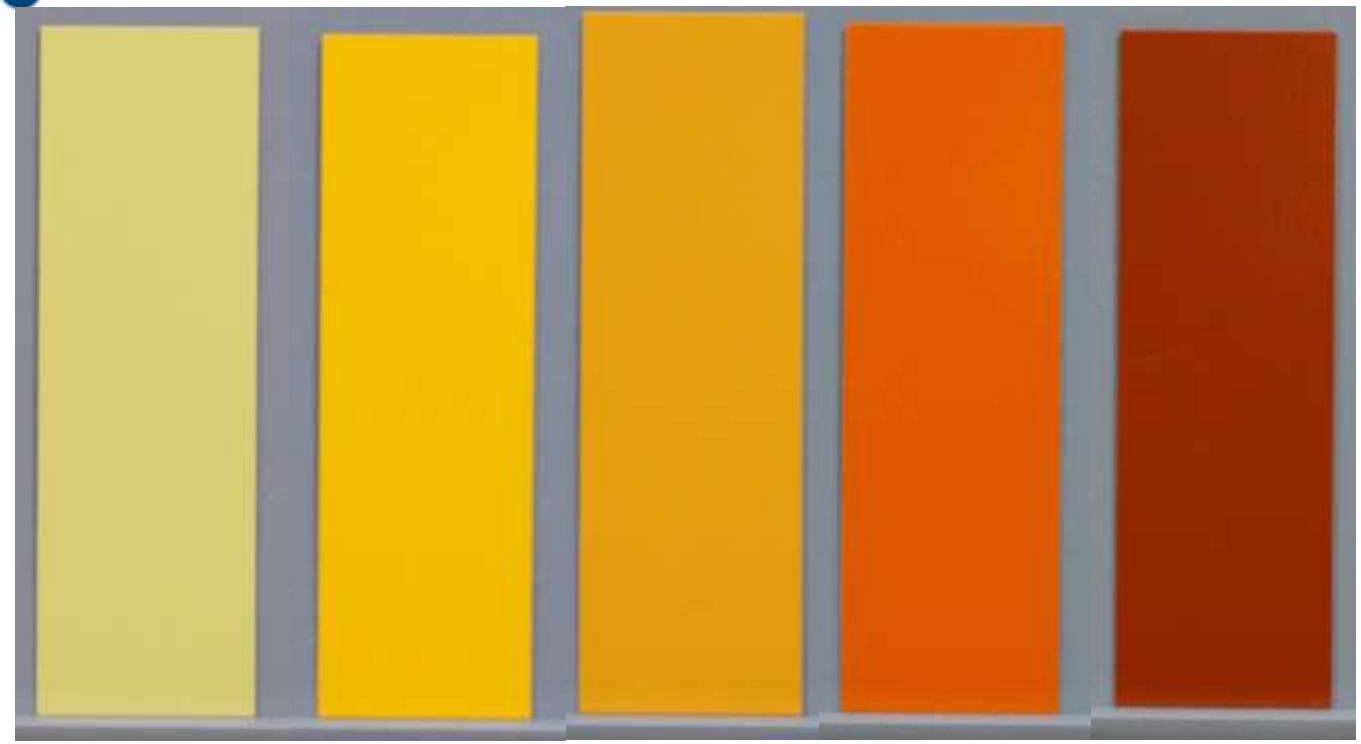
FEVE Testing

- 34 pigments
- 20 different pigment chemistries
- Masstone and 4:1 (white:color)

Testing methods

- QUV-A (ASTM G154-04)
- Emmaqua
- South Florida
- Ohio

Colors



Plus Tints!

Masstone Formulations

1:1 Stoich.

FEVE Resin	59.7%	59.7%	59.7%	59.7%	59.7%	59.7%	59.7%
Polyisocyanate	13.5%	13.5%	13.5%	13.5%	13.5%	13.5%	13.5%
Tin-based catalyst	0.4%	0.4%	0.4%	0.4%	0.4%	0.4%	0.4%
Flow & leveling	1.0%	1.0%	1.0%	1.0%	1.0%	1.0%	1.0%
Degassing agent	0.5%	0.5%	0.5%	0.5%	0.5%	0.5%	0.5%
Barium sulfate	17.5%	17.5%	17.5%	17.5%	10.0%	10.0%	10.0%
PBk28 (Copper Chromite)	7.5%						
PBr29 (IR Black Fe:Cr)		7.5%					
PBl36 (Cobalt Blue (GS))			7.5%				
PG50 (Titanate Green)				7.5%			
PY216 (RTZ Orange)					15.0%		
PBr24 (Buff titanate)						15.0%	
PY227 (NTP Yellow)							15.0%
	100.0%	100.0%	100.0%	100.0%	100.0%	100.0%	100.0%

Tint Formulations (4:1)

FEVE Resin	59.7%	59.7%	59.7%	59.7%	59.7%	59.7%	59.7%
Polyisocyanate	13.5%	13.5%	13.5%	13.5%	13.5%	13.5%	13.5%
Tin-based catalyst	0.4%	0.4%	0.4%	0.4%	0.4%	0.4%	0.4%
Flow & leveling	1.0%	1.0%	1.0%	1.0%	1.0%	1.0%	1.0%
Degassing agent	0.5%	0.5%	0.5%	0.5%	0.5%	0.5%	0.5%
Barium sulfate	10.0%	10.0%	10.0%	10.0%	0.0%	0.0%	0.0%
PW6 (Titanium Dioxide)	12.0%	12.0%	12.0%	12.0%	20.0%	20.0%	20.0%
PBk28 (Copper Chromite)	3.0%						
PBr29 (IR Black Fe:Cr)		3.0%					
PBl36 (Cobalt Blue (GS))			3.0%				
PG50 (Titanate Green)				3.0%			
PY216 (RTZ Orange)					5.0%		
PBr24 (Buff titanate)						5.0%	
PY227 (NTP Yellow)							5.0%
	100.0%	100.0%	100.0%	100.0%	100.0%	100.0%	100.0%

Processing

- 500g samples premixed in Vitamix blender
- Compounded in APV 19mm co-rotating twin screw extruder
  - Set temps.
    - Zone 1: 100C
    - Zone 2: 100C
  - 500rpm
  - 45-60% torque
- Pulverized with a Strand tabletop grinding mill
- Sieved through 140mesh (106micron screen)
- Baked 15 minutes (metal temp)
  - 200C in electric convection oven

QUV-A FEVE Testing

		Masstone Color Change (DE)						
		Hours of Exposure (QUV-A)						
Pigmentation	500	1000	1500	2000	2500	3000	3500	4000
IR Black PBr29	0.3	0.3	0.4	0.4	0.4	0.4	0.6	0.7
Std. Black PBk28	0.2	0.4	0.6	0.7	0.7	0.7	0.8	0.8
Cobalt Blue PBl36	0.9	0.9	1.1	1.0	1.2	1.1	1.4	1.5
Cobalt Green PG50	0.3	0.3	0.5	0.6	0.6	0.6	0.7	0.7
RTZ Orange PY216	0.5	0.9	0.8	0.8	0.8	1.1	1.0	1.2
Chrome Titanate PBr24	0.4	0.8	0.8	0.7	0.7	0.9	0.8	0.9
NTP Yellow PY227	0.6	1.1	1.0	1.0	1.0	1.3	1.3	1.4

		4:1 Tint Color Change (DE)						
		Hours of Exposure (QUV-A)						
Pigmentation	500	1000	1500	2000	2500	3000	3500	4000
IR Black PBr29	0.1	0.2	0.1	0.1	0.1	0.1	0.1	0.1
Std. Black PBk28	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Cobalt Blue PBl36	0.3	0.3	0.3	0.3	0.4	0.3	0.3	0.3
Cobalt Green PG50	0.2	0.2	0.3	0.2	0.4	0.3	0.3	0.3
RTZ Orange PY216	0.2	0.4	0.4	0.4	0.4	0.5	0.4	0.5
Chrome Titanate PBr24	0.2	0.3	0.4	0.4	0.4	0.5	0.5	0.5
NTP Yellow PY227	0.3	0.5	0.6	0.6	0.7	0.8	0.8	0.9

Emmaqua FEVE Testing

Emmaqua Weathering			
	Masstone Color Change (DE)		
	Emmaqua (MJ Exposure)		
Pigmentation	290	580	870
IR Black PBr29	0.7	0.6	1.1
Std. Black PBk28	0.3	0.4	0.4
Cobalt Blue PBl36	1.1	1.3	1.8
Cobalt Green PG0	0.4	0.5	0.7
RTZ Orange PY216	0.1	0.1	0.5
Chrome Titanate PBr24	0.4	0.4	0.2
NTP Yellow PY227	0.5	0.6	0.4

	4:1 Tint Color Change (DE)		
	Emmaqua (MJ Exposure)		
Pigmentation	290	580	870
IR Black PBr29	0.3	0.3	0.3
Std. Black PBk28	0.3	0.3	0.3
Cobalt Blue PBl36	0.5	0.5	0.6
Cobalt Green PG0	0.2	0.2	0.2
RTZ Orange PY216	0.2	0.3	0.2
Chrome Titanate PBr24	0.3	0.4	0.4
NTP Yellow PY227	0.6	0.9	0.9
290MJ/m2 roughly equivilent to 1 Year weathering			

South Florida weathering

Masstone Color Change (DE)

Pigmentation	South Florida (Months) 6	12	24
IR Black PBr29	0.4	0.4	0.9
Std. Black PBk28	0.1	0.3	0.6
Cobalt Blue PBl36	0.8	1.0	1.6
Cobalt Green PG50	0.5	0.4	0.7
RTZ Orange PY216	0.2	0.2	1.1
Chrome Titanate PBr24	0.1	0.4	0.2
NTP Yellow PY227	0.3	0.8	0.5

4:1 Tints Color Change (DE)

Pigmentation	South Florida (Months) 6	12	24
IR Black PBr29	0.1	0.1	0.1
Std. Black PBk28	0.1	0.1	0.1
Cobalt Blue PBl36	0.3	0.3	0.3
Cobalt Green PG50	0.2	0.3	0.3
RTZ Orange PY216	0.1	0.2	0.2
Chrome Titanate PBr24	0.1	0.5	0.6
NTP Yellow PY227	0.5	1.0	1.2

Weathering findings

- PBk26 (CuMnFe) didn’t weather as well as PBk28 (expected)
- PG50- Lithium modified versions weathering well (unexpected)
- Standard versus Easily-dispersed grades (hoped for)
  - DE within tenths in QUV-A, Emmaqua
- Looking to do more correlation work with more SF weathering

Topics

- Color pigment dispersion stability
  - Easily-dispersed versus standard
  - Organic versus inorganic

- Little difference between Std. &. ED
- Inorganics less shift than organics

- Tinting of near-whites
  - Reduce yellow cast
  - Organic versus inorganic

- Inorganic more controllable
- Inorganics less shift than organics

- Long-term durability color
  - Fluoropolymer
  - Wide range of inorganic vs

- Testing on-going
- Update next year
- Colors

Thanks!!!

Mark Ryan

Marketing Manager

The Shepherd Color Co.