

# Coatings formulation against corrosion: Smart use of Nubirox anticorrosive pigments

**UL Prospector Webinar** 

14<sup>th</sup> February, 2018



#### **Agenda**

#### Anticorrosive pigments:

- What is corrosion? Mechanism of corrosion protection
- Anticorrosive pigments:
  - Historical evolution
  - Nubirox Zinc based and Non Zinc based pigments:
    - Inhibition mechanism
    - Physical properties
    - Pigment surface treatments

#### Anticorrosive coating formulation:

- Formulation parameters
- Examples of coating formulation:
  - Replacement of conventional Zinc phosphate in a SB Alkyd primer
  - Improving corrosion protection of WB DTM coatings



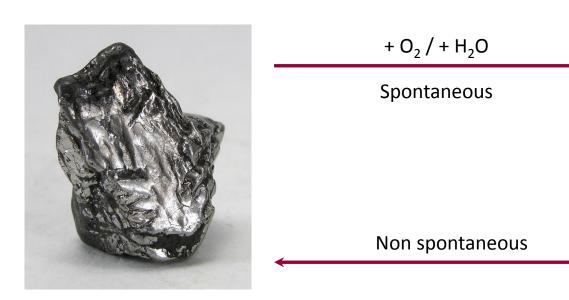




#### **Corrosion**

#### WHAT IS CORROSION?

Corrosion is a gradual **spontaneous** process as a result of a chemical reaction with the environment that damages the original metal





Entropy: Order → Disorder



#### **Corrosion**

#### **COST OF CORROSION**

The annual cost of steel corrosion, estimated to be \$2.5 trillion globally, which is equivalent to 3,4% of the global GDP (2013)

Source: "NACE International assessment of the global cost of corrosion" 2016



#### **Corrosion**

#### **HOW DOES CORROSION OCURR?**















#### **Corrosion mechanism**

Fe 
$$\rightarrow$$
 Fe<sup>2+</sup> + 2e<sup>-</sup>  
O<sub>2</sub> + 2 H<sub>2</sub>O<sub>2</sub> + 4 e<sup>-</sup>  $\rightarrow$  4 OH<sup>-</sup>



. . .



#### **Protection**

## HOW DO WE SLOW DOWN CORROSION?

Corrosion cannot be 100% avoided, but we can slow the process down.





#### **Protection**

#### **HOW DO WE SLOW DOWN CORROSION?**

#### Barrier protection

 Coating: Impermeability of the paint film, film thickness, pigment selection (e.g., type, morphology, size...)

#### Sacrificial protection

Cathodic protection: Zinc Rich Coatings (Zinc Dust).
 Zinc works as sacrificial anode and it is corroded instead of the steel.

#### Inhibitive protection

- Corrosion inhibitors: Inhibiting the rate of corrosion either chemically and/or electro-chemically (inorganic & organic)
  - Cathodic corrosion inhibitors
  - Anodic corrosion inhibitors
  - pH control



**Nubirox anticorrosive pigments** 



#### **Anticorrosive pigments evolution**

## TRADITIONAL ANTICORROSIVE PIGMENTS

**Chromate based pigments** 



Zinc Chromate Zinc Tetraoxychromate Strontium Chromate Barium Chromate Red lead



### ZINC BASED PIGMENTS

Zinc Phosphate

Nubirox N2 Nubirox SP (special particle)



- Modified Zinc Phosphates
- Modified with molybdate and organic surface treatment

Nubirox 102
Nubirox 106 (special particle)

-Modified with iron phosphateNubirox 213 (special particle)

NON ZINC BASED PIGMENTS

Calcium strontium phosphosilicate

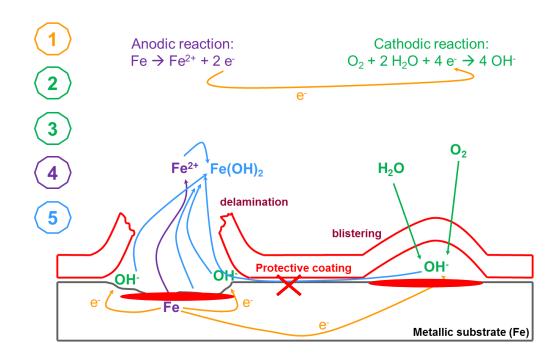
**Nubirox 301** 

**Nubirox 302** 

Modified with organic surface treatment



#### **Anticorrosive pigments mechanism**



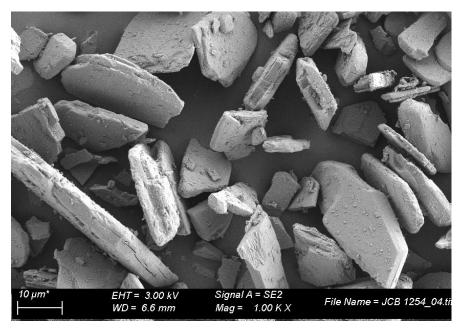
- Zinc phosphate (Nubirox N2, SP, 102, 106 & 213):
  - Direct anodic passivation: Zn phosphate complexes
  - Cathodic inhibition: basic Zn oxides
- Calcium Strontium phosphosilicate (Nubirox 301 & 302):
  - Direct anodic passivation: Ca, Sr, Fe phosphate complexes
  - Cathodic inhibition: basic Ca, Sr oxides

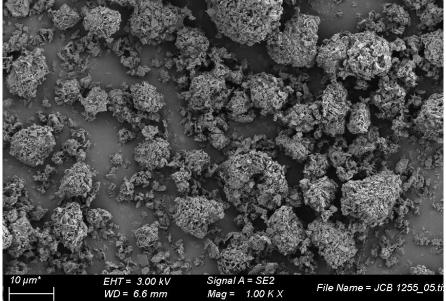


Zinc phosphate: same chemistry but different physics

## Conventional Zinc Phosphate Nubirox N2

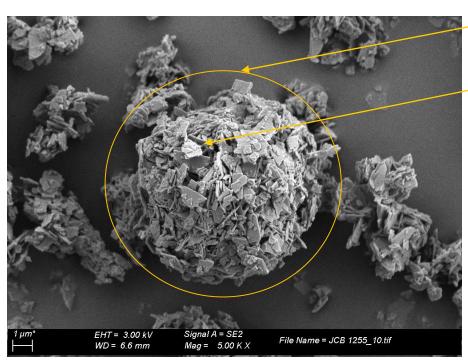
## Special particle Zinc Phosphate Nubirox SP







Nubirox SP: Special particle Zinc Phosphate

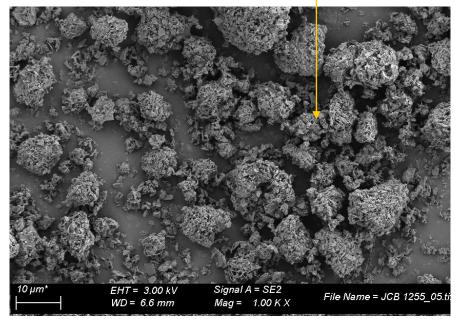


Microscope pictures (SEM)

Aggregated lamellar crystals forming a spherical agglomerate (4-10 μm)

Lamellar primary crystals (<1µm)

Agglomerated spherical particles (>8 μm)





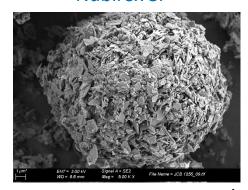
**Zinc phosphate**: same chemistry but different physics

**Conventional Zinc Phosphate** Nubirox N2



Particle size:  $8-12 \mu m$ 

#### **Special particle Zinc Phosphate Nubirox SP**



<1μm (aggregate 4-10 μm)



**Specific surface area** (BET):

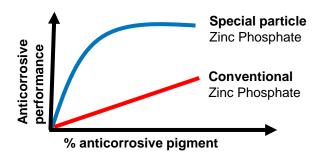
 $1 \text{ m}^2/\text{gr}$ 

 $8-15 \text{ m}^2/\text{gr}$ 

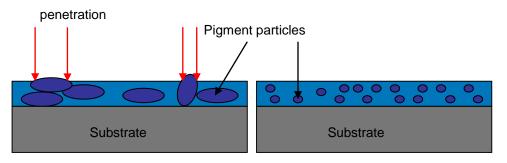
Oil absorption: ~ 50g/100g ~21g/100g



- Zinc phosphate: same chemistry but different physics and performance
  - effectiveness



performance in thin coatings





Solvent based system

CPV=35

10%
anticorrosive
pigment

Application on
satndardized cold
rolled steel panels S46 (Q-Panel)
Dry film
thickness~20µ

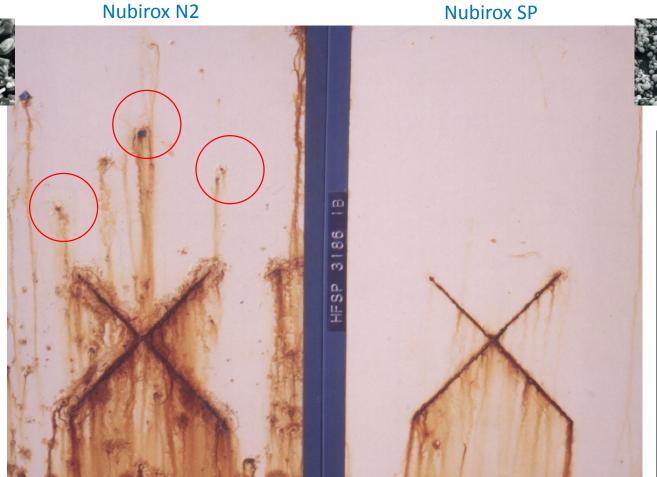
300 hours of
exposure in Salt
Spray (ASTM B-

Long oil air drying alkyd resin

#### **Anticorrosive pigments physical properties**

Zinc phosphate: performance in thin coatings

Conventional Zinc Phosphate Special particle Zinc Phosphate



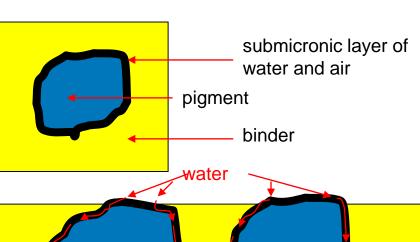
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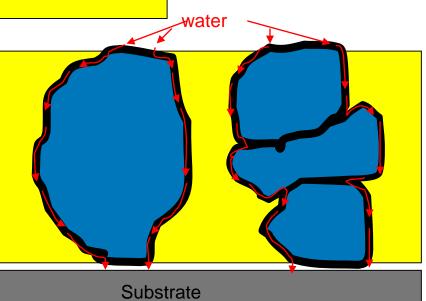


#### **Zinc based Nubirox**

- Nubirox 100 series: Nubirox 102 & Nubirox 106
  - ► Modification with molybdate and organic surface treatement

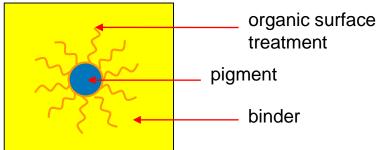
#### Particles not well wetted

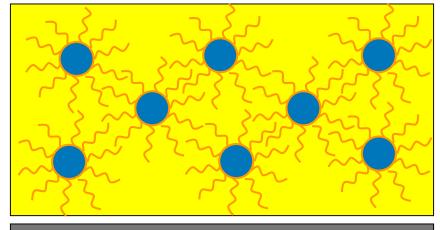




#### Particles with organic surface treatment

Nubirox 102 & Nubirox 106







#### **Zinc based Nubirox**

#### **Nubirox 106**

- Based on special particle Zinc Phosphate
- High pigment effectiveness at low loading level in many SB and WB paint systems

5% dry film volume (5.7% t.f.w.)

8% dry film volume (9,3% t.f.w.)

Without anticorrosive pigment



**Nubirox 106** 





**Conventional Zinc Phosphate** 

Water based system

Epoxy resin: **Beckopox** EP384W/53WAMP Hardener:

**Beckopox** EH623W/80WA

49% solids in volume CPV/CPVC=0.6

Standardized blasted hot rolled steel panels F130S1 (Espancolor) DFT~140µ

**890 hours** of exposure in Salt Spray (ASTM B-117).

FORMULA EPPAA 18951

FORMULA EPPAA 18953

FORMULA EPPAA 18952



#### **Zinc based Nubirox**

#### **Nubirox 213**

- Modification with Iron Phosphate
- Specially suitable for alkyd and epoxy primers used to protect steel substrates

Without anticorrosive pigment

(10.7% t.f.w.)

9.5% dry film volume (10.0% t.f.w.)

**Conventional Zinc Phosphate** 

9.5% dry film volume







#### Solvent based system

Epoxy resin: Uneresin 5471X75 Hardener: Unedur 5415X70

40% solids in volume CPV/CPVC=0.7

Standardized blasted hot rolled steel panels F130S1 (Espancolor) DFT~60µ

**741 hours** of exposure in Salt Spray (ASTM B-117).

FORMULA EPPA 12878

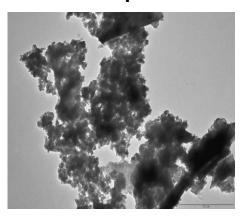


#### **Non Zinc based Nubirox**

#### Nubirox 300 series

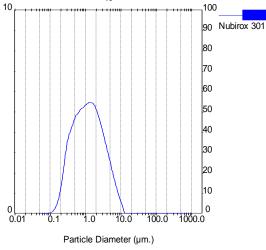
▶ Nubirox 301 & Nubirox 302 based on Calcium Strontium Phosphosilicate

#### **Particle shape**



SEM (scanning electron microscope)

#### Particle size distribution



Malvern Mastersizer S Ver 2.19

 $D(v,0.5)=1.15\mu$ 

#### **Specific surface area**

	Specific surface area (m2/g)
Zinc Phosphate	1
Nubirox 301	21

#### **Particle characterization:**

Elemental particles  $<1\mu$  forming aggregates and agglomerates up to  $<10\mu$ 

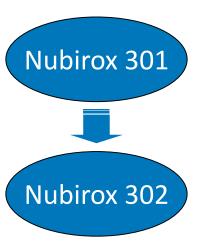
- More active surface (allows lower pigment dosage)
- Lower effect on gloss (DTM applications)



#### **Non Zinc based Nubirox**

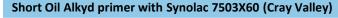
#### Nubirox 302

Organic surface treatment to improve binder compatibility and performance



Its alkalinity makes it reactive with acidic binders, like short oil alkyds, but not with main long oil alkyds (usually with low acid values).

Its organic treatment protects the pigment from reacting with the acid binder.

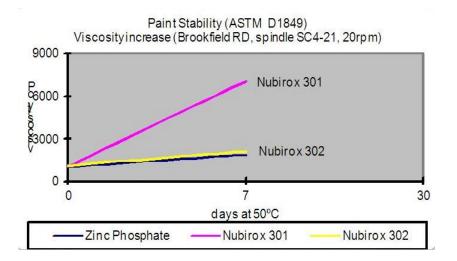


50% solids in volume

PVC/CPVC=0.7

6% Active pigment in volume of dry film

Viscosity measurements in Brookfield RV (spindle SC4-21, 20 rpm)





#### **Agenda**

#### Anticorrosive pigments:

- What is corrosion? Mechanism of corrosion protection
- Nubirox anticorrosive pigments
  - Zinc based Nubirox: mechanism, particle shape and modifications
  - Non-Zinc based Nubirox: mechanism, particle and modifications



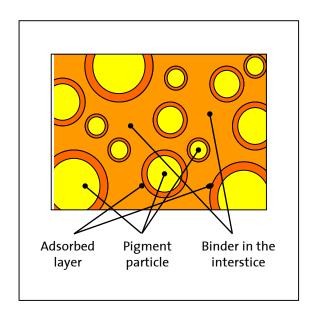
#### Anticorrosive coating formulation:

- Formulation parameters
- Examples of coating formulation:
  - Replacement of conventional Zinc phosphate in a SB Alkyd primer
  - Improving corrosion protection of WB DTM coatings





- PVC "Pigment volume concentration"
  - ▶ A paint film is a volume: formulation parameters must be fixed in volume units.



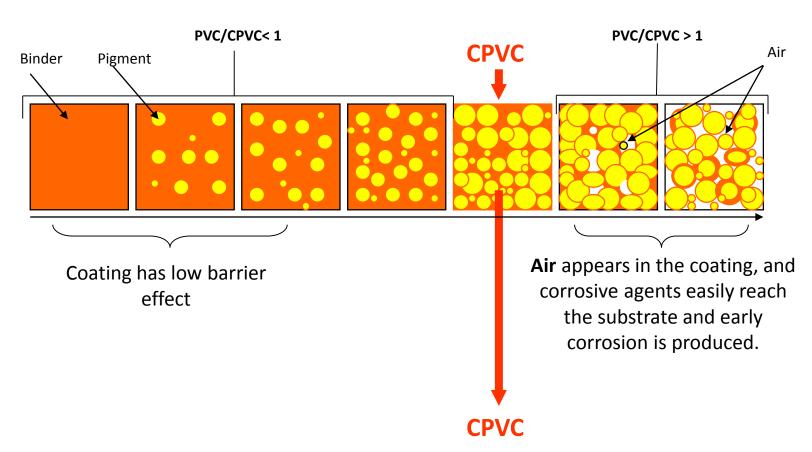
$$PVC = \frac{V_{pigment}}{V_{total dry film}} = \frac{V_{pigment}}{V_{pigment} + V_{binder}}$$

$$V_{pigment} = V_{color\ pigment} + V_{anticorrosive\ pigment} + V_{fillers}$$

$$V_{binder} = V_{adsorbed\ binder} + V_{free\ binder}$$



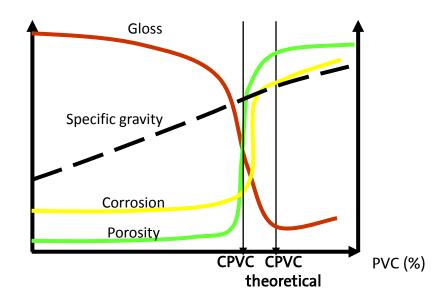
CPVC "Critical pigment volume concentration"



**CPVC** is just the **PVC** where there is just enough binder to wet all the pigments and fill the voids between particles.



#### PVC/CPVC ratio



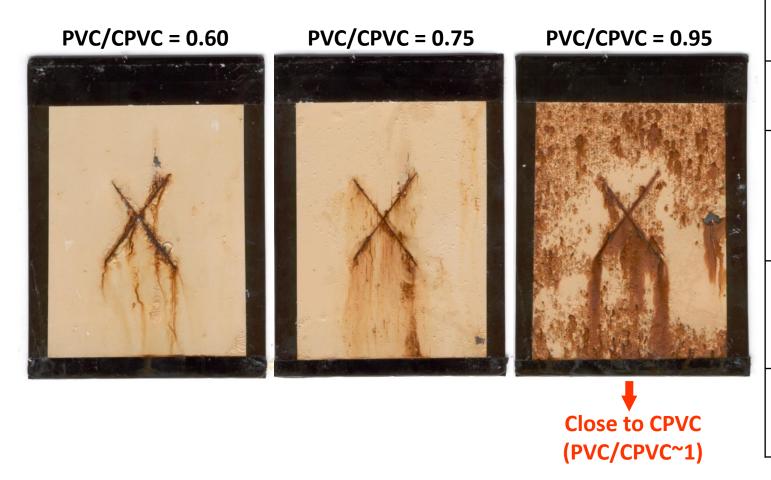
- **CPVC**: Characteristic of each pigment package and binder. Experimental.
- ▶ Theoretical CPVC: Calculated as the CPV for 100 grams of pigment and the grams of oil according to the oil absorption.

$$CPVC_{\text{theoretical}} = \frac{\frac{100 \, g_{\text{pigment}}}{\rho_{\text{pigment}}(g/l)}}{\left(\frac{100 \, g_{\text{pigment}}}{\rho_{\text{pigment}}(g/l)}\right) + \left(\frac{g_{\text{oiladsorved}}}{\rho_{\text{oil}}(0.93g/l)}\right)}$$

**100**  $g_{pigment}$ : grams of pigment for oil absorption value  $\rho_{pigment}$ : pigment specific gravity  $g_{oil\,absorbed}$ : grams of oil (oil absorption value)  $\rho_{oil}$ : specific gravity of linseed oil (0.93g/l)



Effect of PVC/CPVC ratio on performance



Solvent based system

Phenolic modified short oil alkyd

50% solids in volume

5% of anticorrosive pigment (Nubirox 213)

Standardized cold rolled steel panels S-46 (Q-Panel)

DFT ~40μ

**400 hour of exposure in Salt Spray** (ASTM B-117).



#### SB Alkyd primer:

Replacement of conventional Zinc Phosphate

#### Direct replacement

Replacement 1:1 in weight

#### ▶ Reformulation

Differences only due to anticorrosive pigment effectiveness





		Α
FORMULA	FUNCTION	% weight
Synolac 7503X60	Alkyd resin	21.81
Bentone 34 (10% in xylene)	Rheological additive	4.60
Calcium Naphtenate 4%	Wetting agent	0.41
Xylene	Solvent	10.98
Tioxide TR92	Titanium dioxide	7.56
Zinc Phosphate	Antic. Pigment	7.36
Nubirox 106	Antic. Pigment	-
Micral 2	Calcium carbonate	15.43
CBI-5 Talc	Micro talc	10.55
Synolac 7503X60	Alkyd resin	19.29
Cobalt Naphtenate 6%	Dryer	0.12
Synresol E-22	Anti-skinning agent	0.21
Dowanol PM	Solvent	1.68
Pigment Volume Concentration		36.1%
PVC/CPVC		0.70
Non-	Non-volatile contents, volume	
Non-volatile contents, weight		66.3%

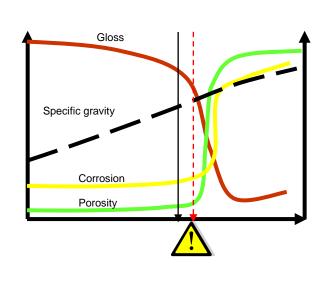
	Specific gravity (g/cm³)	Oil absorption (g oil/100g pig)
Zinc Phosphate	3.3	20
Nubirox 106	3.2	40

Anticorrosive pigment volume in dry film

6.0%

В
% weight
21.81
4.60
0.41
10.98
7.56
-
7.36
15.43
10.55
19.29
0.12
0.21
1.68
36.3%
0.74
50.0%
66.3%
6.2%
<b>^</b>

Weight replacement less free binder





		Α
FORMULA	FUNCTION	% weight
Synolac 7503X60	Alkyd resin	21.81
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Synresol E-22	Anti-skinning agent	0.21
Dowanol PM	Solvent	1.68
Pigment Volume Concentration		36.1%
PVC/CPVC		0.70
Non-volatile contents, volume		50.0%
Non-volatile contents, weight		66.3%
Anticorrosive pigment volume in dry film		6.0%

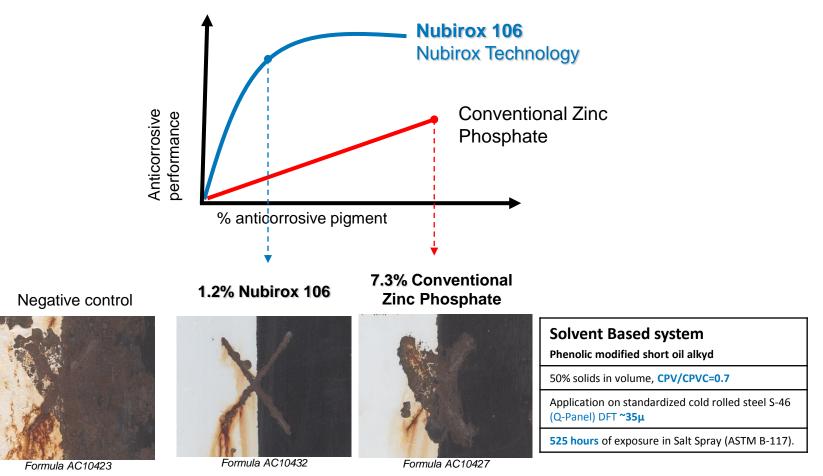
	Specific gravity (g/cm³)	Oil absorption (g oil/100g pig)
Zinc Phosphate	3.3	20
Nubirox 106	3.2	40

В		С	D
% weight		% weight	% weight
21.81	<b>^</b>	23.86	22.66
4.60		4.70	4.68
0.41		0.39	0.40
10.98		10.60	10.99
7.56		7.51	7.49
-		-	-
7.36		7.48	1.24
15.43	$\downarrow$	14.12	18.36
10.55	$\downarrow$	9.66	12.56
19.29	<b>^</b>	19.70	19.60
0.12		0.13	0.13
0.21		0.20	0.12
1.68		1.68	1.69
36.3%		33.6%	35.4%
0.74		0.70	0.70
50.0%		50.0%	50.0%
66.3%		65.6%	65.7%
6.2%		6.0%	1%
Weight		Reformulation at	Cost-effective
replacement		same PVC/CPVC	reformulation

less free binder



Nubirox 106: Pigment effectiveness at low loading level





#### WB Alkyd DTM for multisubstrate aplication:

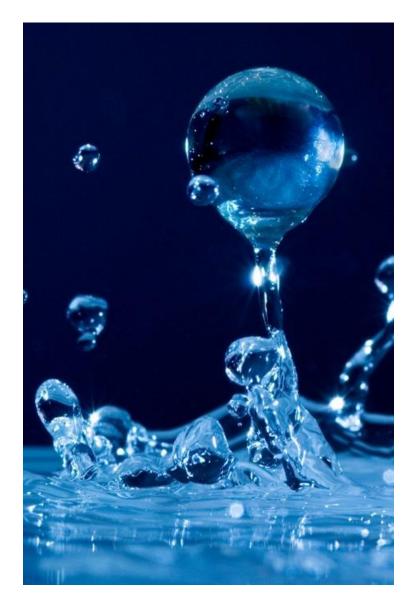
Addition of anticorrosive pigments

#### **▶** Direct replacement

Replacement in weight of color pigment or extender

#### ▶ Reformulation

Differences only due to anticorrosive pigment effectiveness





		Α
FORMULA	FUNCTION	% weight
Water		8,99
Byk 024	Defoamer	0,12
Disperbyk 190	Dispersing agent	1,52
Tioxide TR92	Titanium dioxide	28,77
Nubirox 302	Antic. Pigment	-
Nubirox 106	Antic. Pigment	-
Uradil AZ760	Medium oil alkyd emulsion	58,12
Additol VXW4940 (1:1 water)	ditol VXW4940 (1:1 water) Drier 1,28	
Nubirox FR-10	x FR-10 "Flash rust" inhibitor 0.50	
Byk 348	8 Levelling agent	
Acrysol RM-8W PU-thickener		0,47
Pigment Volume Concentration		19,3%
PVC/CPVC		0.36
Non-volatile contents, volume		49,7%
Non-volatile contents, weight		61,3%
Anticorrosive pigment volume in dry film		0%

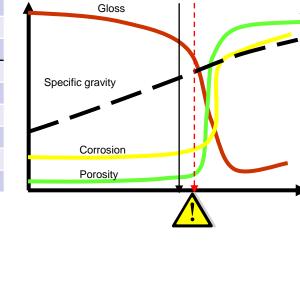
	Non-volatile contents, weight		
	Anticorrosive pigment volume in dry film		
	Specific gravity	Oil absorption	
	(g/cm³)	(g oil/100g pig)	
Titanium dioxide	4,1	21	
Nubirox 302	2,9	45	

% weight	
8,99	
0,12	
1,52	
24,77	
4,00	
-	
58,12	
1,28	
0.50	
0.23	
0,47	
20,2%	
0.40	
50,1%	
61,4%	
3,7%	
$\uparrow$	

TiO<sub>2</sub> Weight replacement

less free binder

В





		Α
FORMULA	FUNCTION	% weight
Water		8,99
Byk 024	Defoamer	0,12
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В
% weight
8,99
0,12
1,52
24,77
4,00
-
58,12
1,28
0.50
0.23
0,47
20,2%
0.40
50,1%
61,4%
3,7%
<b>^</b>
T`

TiO<sub>2</sub> Weight

replacement

less free binder

D

	C		D
	% weight		% weight
	8,90		9,56
	0,13		0,12
	1,38		1,43
/	21,77	$\forall$	23,07
	4,38		-
	-		3,87
\	60,84	<b>1</b>	60,04
	1,34		0,66
	0.53		0.52
	0.24		0.24
	0,49		0,49
	18,2%		18,8%
	0,36		0,36
	49,7%		49,7%
	60,1%		60,8%
	4,0%		4,0%
D	aformulation a	.+ D	oformulation





Water based system

Uradil AZ 760 is a medium oil alkyd emulsion based on soybean fatty acids

50% solids in volume CPV/CPVC≈ 0.36

Application on standardized galvanized Steel SG015 (Espancolor) Dry film thickness ~45µ

2800 hours of exposure in Salt Spray (ASTM B-117)

4% dry film volume

(3,9%total formula weight) **Nubirox 106** 

4% dry film volume (4,4%total formula weight)

**Nubirox 302** 

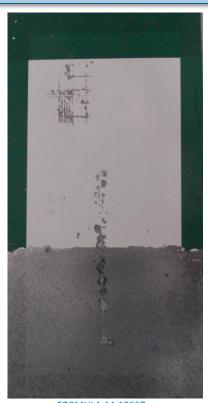
Without anticorrosive pigment





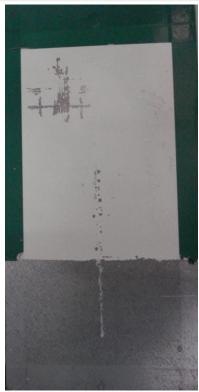
FORMULA AA 19666

Gloss (60º): 98 Gloss (20º): 93



FORMULA AA 19667

Gloss (60º): 93 Gloss (20º): 79



FORMULA AAA 19670

Gloss (60º): 96 Gloss (20º): 85



Without anticorrosive pigment

Water based system

Uradil AZ 760 is a medium oil alkyd emulsion based on soybean fatty acids

50% solids in volume CPV/CPVC≈ 0.36 Application on standardized cold rolled steel panels S-46 (Q-Panel) Dry film thickness ~45µ

500 hours of exposure in Salt Spray (ASTM B-117)

4% dry film volume

(3,9%total formula weight)

4% dry film volume (4,4%total formula weight)

**Nubirox 106** 

**Nubirox 302** 







FORMULA AA 19666

Gloss (60º): 98 Gloss (20º): 93

FORMULA AA 19667

Gloss (60º): 93 Gloss (20º): 79 FORMULA AAA 19670

Gloss (60º): 96 Gloss (20º): 85



Without anticorrosive pigment

Water based system

Uradil AZ 760 is a medium oil alkyd emulsion based on soybean fatty acids

50% solids in volume CPV/CPVC≈ 0.36 Application on standardized iron phosphated steel panels Bonderite 1000 (R-36-I from Q-Panel) Dry film thickness ~45µ

500 hours of exposure in Salt Spray (ASTM B-117)

4% dry film volume

(3,9%total formula weight)

4% dry film volume

(4,4%total formula weight)

**Nubirox 106** 

**Nubirox 302** 



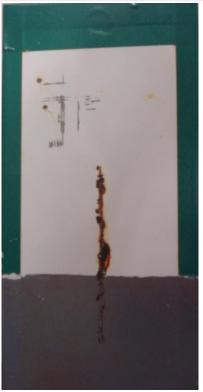
FORMULA AA 19666

Gloss (60º): 98 Gloss (20º): 93



FORMULA AA 19667

Gloss (60º): 93 Gloss (20º): 79



FORMULA AAA 19670

Gloss (60º): 96 Gloss (20º): 85



Nubirox 302: good gloss/performance balance

#### **Gloss**

Panel evaluation	Control	4% Nubirox 106	4% Nubirox 302
Gloss 60º	98	93 (-5%)	96 (-2%)
Gloss 20º	93	79 (-15%)	85 (-9%)

### Anticorrosive performance on Cold rolled steel

Panels exposed 500h in	Control	4%	4%
Salt Spray test		Nubirox 106	Nubirox 302
Rusting at the scribe (ASTM D1654)	5	6	7
	(3.5mm)	(3mm)	(2mm)
Rusting on the panel (ASTM D610)	8G	9G	9G
	(0.1%)	(0.03)	(0.03%)
Adhesion at the scribe (ASTM D1654)	5	7	9
	(15%)	(5%)	(1%)
Adhesion "cross cut" (ASTM B3359)	5B	5B	5B

### Anticorrosive performance on Galvanized steel

Panels exposed 2800h in Salt Spray test	Control	4% Nubirox 106	4% Nubirox 302
Rusting at the scribe (ASTM D1654)	5 (3.5mm)	7 (1.5mm)	9 (0.5mm)
Rusting on the panel (ASTM D610)	8G (0.1%)	7G (0.3%)	9 (0.03%)
Adhesion at the scribe (ASTM D1654)	2 (50%)	6 (10%)	9 (1%)
Adhesion "cross cut" (ASTM B3359)	ОВ	1B	1B

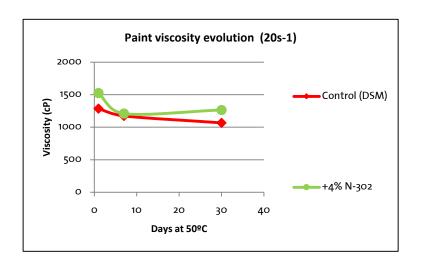
## Anticorrosive performance on Phosphated steel

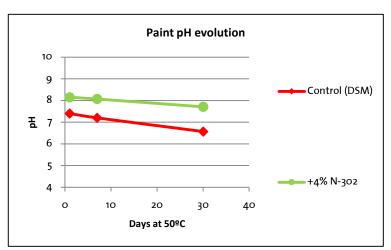
Panels exposed 500h in	Control	4%	4%
Salt Spray test		Nubirox 106	Nubirox 302
Rusting at the scribe (ASTM D1654)	4	7	7
	(6mm)	(1.5mm)	(1.5mm)
Rusting on the panel (ASTM D610)	7G	8G	9G
	(0.3%)	(0.1%)	(0.03%)
Adhesion at the scribe (ASTM D1654)	0	8	9
	(90%)	(2%)	(1%)
Adhesion "cross cut" (ASTM B3359)	4B	3B	1B



Nubirox 302: good paint stability

Package stability (ASTM D1849): Viscosity, pH and settling evolution after 7 and 30 days at 50°C.





	Settling (ASTM D-869)		
	initial 7 days at 50°C 30 days at 50°		30 days at 50ºC
Control	10	6	2
+4% N-302	10	8	6



#### **Conclusions**

#### Anticorrosive pigments:

- Not only is the chemistry important...
  also physical properties of pigment particles:
  - More active surface
  - Better performance in thin film systems
- ▶ Modifications improve performance of Zinc based pigments
- Non Zinc based anticorrosive pigments benefits



#### Anticorrosive coating formulation:

- ▶ Bear in mind formulation parameters
  - Adjust free binder volume to the desired properties
  - · Pigment replacement in weight could be dangerous
  - Benefits of cost efficient replacements







#### Thank you

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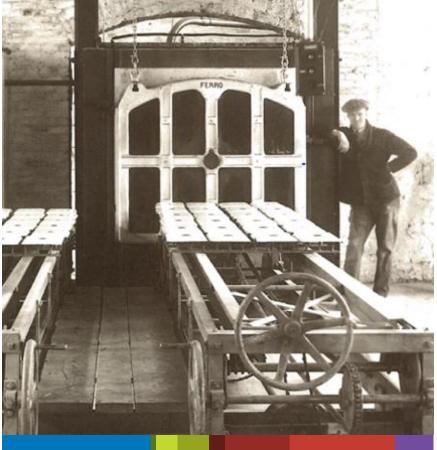
### **About Ferro Corporation**



#### **Ferro Overview**

- Founded 1919 as Ferro Enameling Company in Cleveland, Ohio USA
- Worldwide leader in production of glass enamels, porcelain enamels, ceramic tile coatings and high performance pigments
- Approximately 5,680 associates working in 27 countries
- 2016 sales of \$1.15 billion







#### **Diverse Customer Base, Singular Promise**

- We serve manufacturers in 100+ countries and in diverse markets.
- Our customers turn to us for innovative solutions and a commitment to delivering new possibilities for their products, businesses and customers.

**Building and Automotive Appliances** Construction Household Industrial **Electronics Furnishings Products** 



#### Ferro's Core Technologies

- Color and glass science
- Particle engineering
- Surface application technology
- Formulation











#### The Way We Work: Ferro Core Values

## **Customer Focus**

Our customers are why we exist; we build trusting relationships that are built on customer needs and a genuine interest in making customers successful.

## **Accountability for Performance**

We work to the highest performance standards, prioritizing safety, environmental stewardship, creating value for customers and Ferro shareholders.

## Innovative Thinking

We seek new ideas for technologies and business processes, always looking for ways to improve and better serve our customers.

**Teamwork and Collaboration** 

We are committed to a work environment of trust and respect, and work together to consistently deliver value to our customers and shareholders.



For more information on Ferro Pigments Business Unit, visit www.ferro.com



#### **Disclaimer for commercial products**

Ferro can certify only those parameters in the Technical Data Sheet for our products. Ferro is not responsible for any use, misuse, handling or storage conditions that may act to the detriment of the specifications of our products, or pose a risk to human health or the environment.

Due to the fact that there are a multitude of formulations, manufacturing processes and end uses, Ferro strongly recommends that customers conduct performance testing under their own testing program, and carry out appropriate verifications in order to determine the ultimate suitability of our products, and comply with existing laws and regulations.