Neuburg Siliceous Earth
for medium solid epoxy anti-corrosion coatings

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Status Quo

- Anti-corrosion coatings provide an important contribution to maintaining the value of economic assets.
- In such coatings, primers often contain zinc phosphate as an active anti-corrosion pigment as well as fillers like barite, talc etc., which are used to further improve properties.
- Ever stricter legal limitations as well as economical reasons are causing an increasing trend towards turning away from the use of high zinc phosphate additions → less heavy metals for “Green Coatings”.
- The lower the concentration of zinc phosphate is, the more important the filler type becomes.
- The surface roughness of the substrate should be considered as an additional challenge.

Objective

- Are functional, high quality fillers able to compensate for the loss of anti-corrosion properties due to low concentrations of a standard zinc phosphate?

- This investigation provides an approach based on a
  - ✓ medium-solid epoxy-polyamide primer
  - ✓ control formulation PVC 34 %, containing
    - 7.5 % standard zinc phosphate
    - 19.0 % talc
    - 7.8 % barium sulphate

Base Formulation

	Anti-Corrosion Primer, 2P Epoxy-Polyamide, medium-solid		parts by weight
A	Epikote 1001 x 75	Solid epoxy resin based on Bisphenol A, 75% in xylene, EEW 633	23.8
	Bentone 34 paste	Rheological additive, 10% in xylene / ethanol 87:3	4.3
	Xylene	Solvent	6.5
	Ethylglycol	Solvent	4.7
	MIBK	Solvent, Methylisobutylketone	6.6
	Nusa 57	Wetting and dispersing additive	0.4
	BYK-354	Leveling additive	0.8
	Sachtleben RD3	Pigment, Titanium dioxide	5.9
	Zinc phosphate	Anti-corrosion pigment	7.5
	Talc	Filler, hydrated magnesium silicate	19.0
	Blanc fixe	Filler, barium sulfate ppt	7.8
B	Versamid 115 x70	Polyamide resin, 70% in xylene, HEW 283	12.7
Total			100.0
Solids content w/w		[%]	68.0
Pigment volume concentration (PVC)		[%]	33.8
Volatile organic compounds (VOC)		[g/l]	430

Variation Formulation

Substitution of anti-corrosion pigment by filler, PVC constant

	Control [pbw]	Reduced [pbw]	Without [pbw]
Zinc phosphate	7.5	2.5	0
Talc	19.0	22.8	24.7

Substitution of filler type, PVC constant

Natural mixture of quartz, mica, chlorite	22.8	24.7
Neuburg Siliceous Earth grades	21.3	23.1

Addition of 1 pbw of (3-Aminopropyl)triethoxysilane to part B (Versamid) as indicated

Variation Substrates

Blasted and Non-blasted challenge for adhesion

Blasted

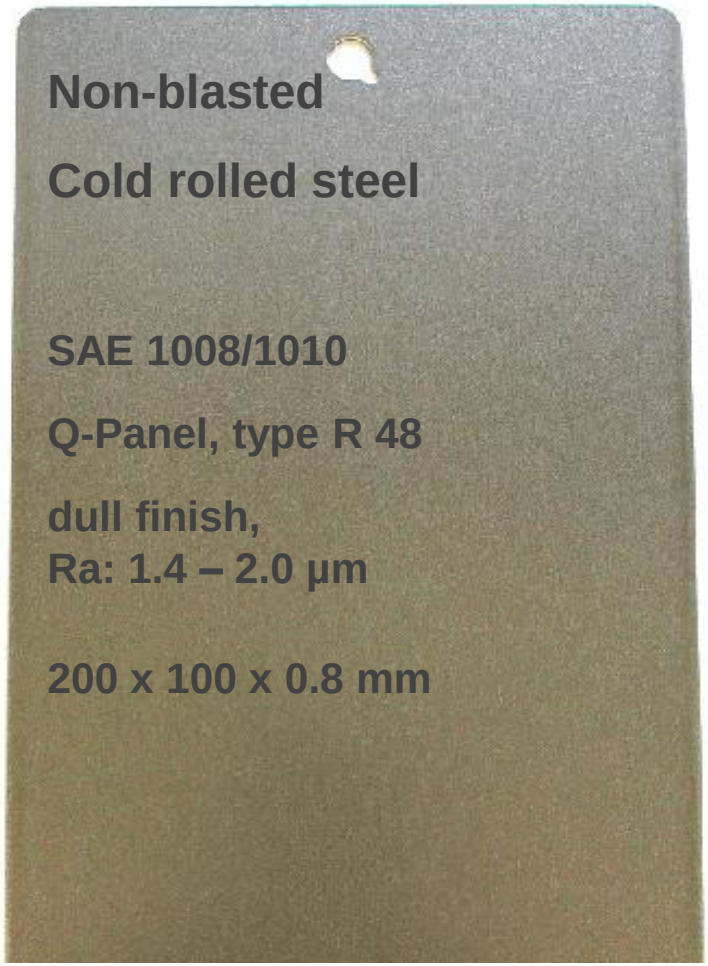
Cold rolled steel

non-alloyed steel

SA 2 1/2, blasted medium (G)

according to ISO 8503-1

150 x 100 x 2 mm



Filler Characteristics

		Talc	Natural Mixture of Quartz, Mica, Chlorite	Neuburg Siliceous Earth	
				Aktisil PF 777	Aktisil AM
Morphology		lamellar	corpuscular / lamellar		
Density	[g/cm³]	2.8	2.8	2.6	2.6
Particle size d_{50}	[µm]	6.8	8.0	2.2	2.2
Particle size d_{97}	[µm]	18	27	10	10
Oil absorption	[g/100g]	45	43	35	45
Specific surface area BET	[m²/g]	5	5	8	9
Surface treatment		---	---	alkyl functionalized hydrophobic	amino functionalized

Preparation

Mixing and Dispersing	Premixing with dissolver, grinding in an agitator bead mill at dissolver, 8 m/s for 15 min
Application	Spraying by air pressure, single-layered, dry film thickness: ~ 80 µm
Conditioning	Drying / Hardening conditions before corrosion tests: 14 days at 23 °C / 50 % relative humidity

Corrosion Protection Testing

Unscribed area:

Adhesion (Cross-cut)	DIN EN ISO 2409
Blistering	DIN EN ISO 4628-2
Under-film corrosion	ASTM D 610

Scribe:

Blistering	DIN EN ISO 4628-2
Corrosion intensity	
Delamination	DIN EN ISO 4628-8
Rust Creep	DIN EN ISO 4628-8
Pitting (max. depth of scribe)	

Corrosion Protection
Performance Evaluation

	Criterion	rating number	10	9	8	7	6	5	4	3	2	1	0
	Unscribed Area												
1	Adhesion Cross-cut	[GT]	0	0-1	1	1-2	2	2-3	3	3-4	4	4-5	5
2	Blistering	Quantity Size	- -	1 1	2 2	2-3 2-3	3 3	3-4 3-4	4 4	4-5 4-5	5 5	>5 >5	compl. Delam.
3	Under-film corrosion	[%]	0	0,03	0,1	0,3	1	3	10	16	33	50	100
	Scribed												
4	Blistering	Quantity Size	- -	1 1	2 2	2-3 2-3	3 3	3-4 3-4	4 4	4-5 4-5	5 5	>5 >5	compl. Delam.
5	Delamination	[mm]	0	2	4	7	10	13	16	20	25	30	40
6	Rust creep	[mm]	0	0,5	1	2	3	5	7	9	12	15	20
7	Pitting	[mm]	0	0,1	0,2	0,3	0,4	0,5	0,6	0,7	0,8	0,9	1,0
8	Corrosion intensity	-	very low		low		moderate			high		very high	
	1 - 8 cumulative rating 80 max.		80	72	64	56	48	40	32	24	16	8	0
			very good										very bad

General Properties

- **Producibility**
- **Rheology**
- **Leveling**
- **Hardness**

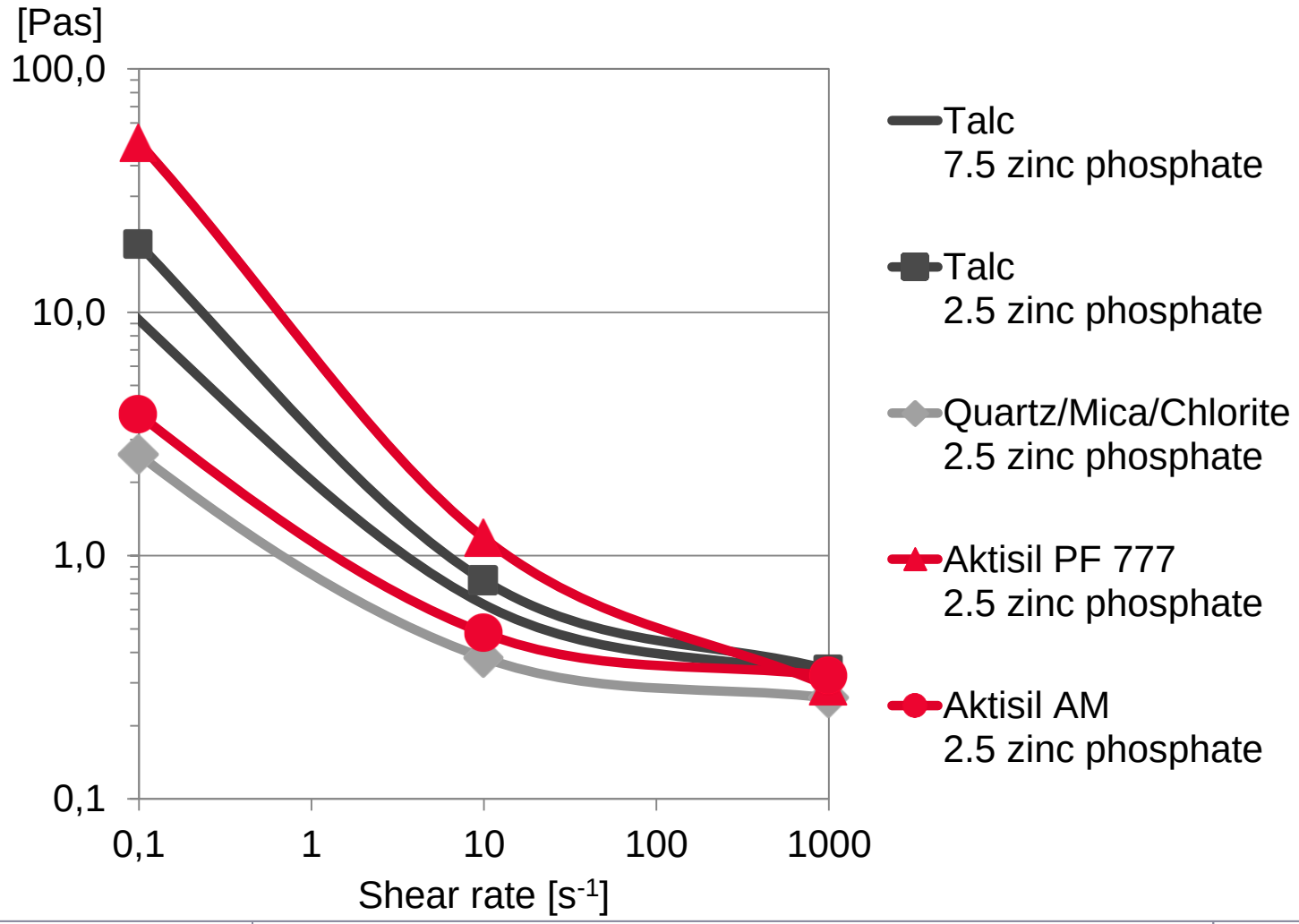
Producibility

Part A

		Control 7.5	2.5 pbw respectively 0 pbw zinc phosphate			
		Talc	Talc	Quartz/ Mica/ Chlorite	Aktisil PF 777	Aktisil AM
Ease of filler incorporation						
Fineness of grind	[µm]	5	5	10	5	5

Rheology

Viscosity curve, Part A+B, DIN 53019



Leveling

Dry film

Talc
7.5 zinc phosphate

Talc
2.5 zinc phosphate

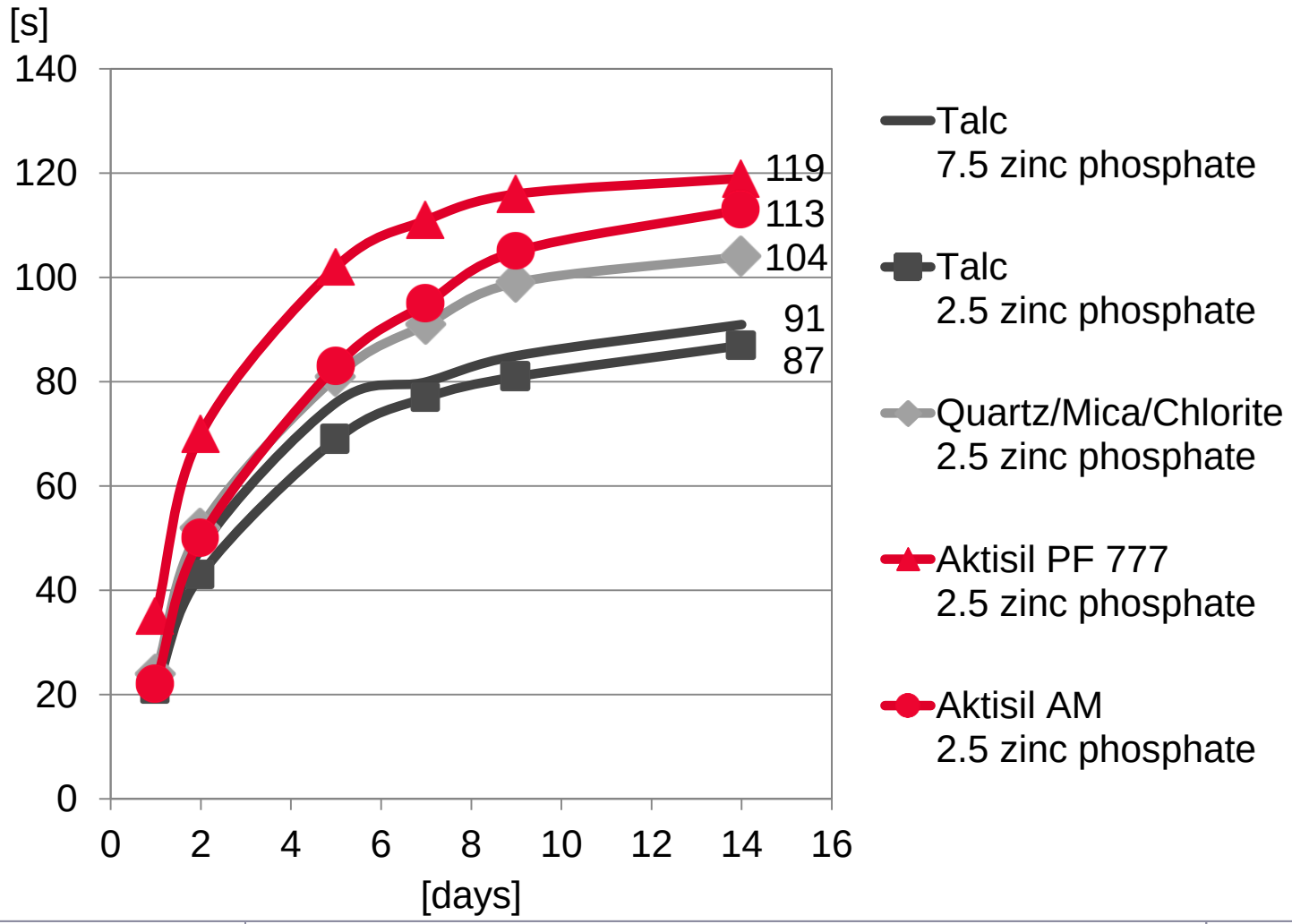
Quartz/Mica/Chlorite
2.5 zinc phosphate

Aktisil PF 777
2.5 zinc phosphate

Aktisil AM
2.5 zinc phosphate

Hardness

Koenig Pendulum 23 °C, DIN EN ISO 1522



Corrosion protection

Overview

(for selection of partial result please use hyperlinks)

Blasted steel

- Reduced loading of zinc phosphate
- Without zinc phosphate

Non-blasted steel

- Reduced loading of zinc phosphate
- Reduced loading of zinc phosphate and addition of amino silane

Summary

Blasted Steel

Reduced loading of zinc phosphate

Adhesion prior exposure tests

Humidity test 1000 h, DIN EN ISO 6270-2 CH

Evaluation on scribed and unscribed panels

Salt pray test 1000 h, DIN EN ISO 9227 NSS

Photos: unscribed area

↓

stripped

Photos: scribe

↓

delaminated coating peeled off

Evaluation

Adhesion
prior Exposure Tests

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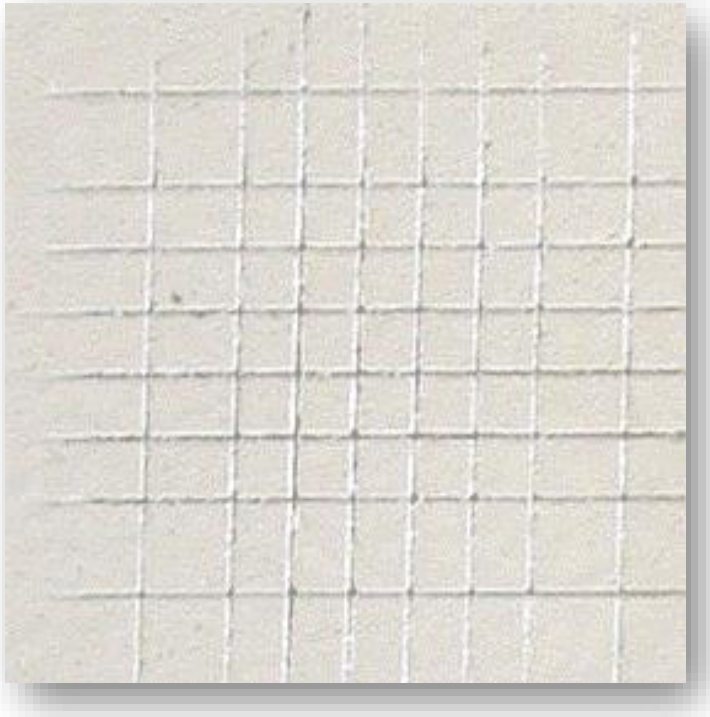
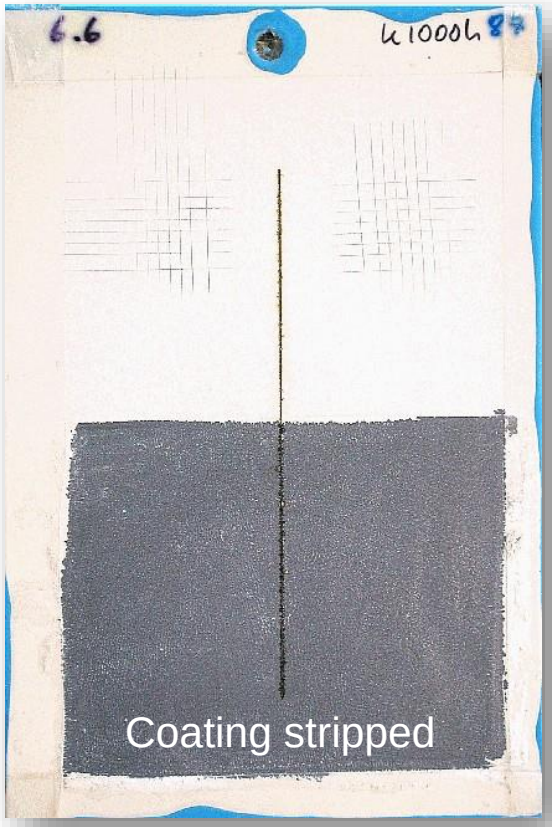
Cross-cut test, 2 mm, with tape tear off, DIN EN ISO 2409



All Formulations:

GT 0
→ Excellent result

Humidity Test



All formulations:

Unscribed area:

Adhesion	GT 0
Blistering	0
Under-film corrosion	0

Scribe:

Blistering	0
Delamination	0
Rust creep	< 0.5 mm
Pitting	< 0.1 mm
Corrosion intensity	very low

→ excellent result
(80 of 80 max.)

Salt Spray Test
Unscribed Area

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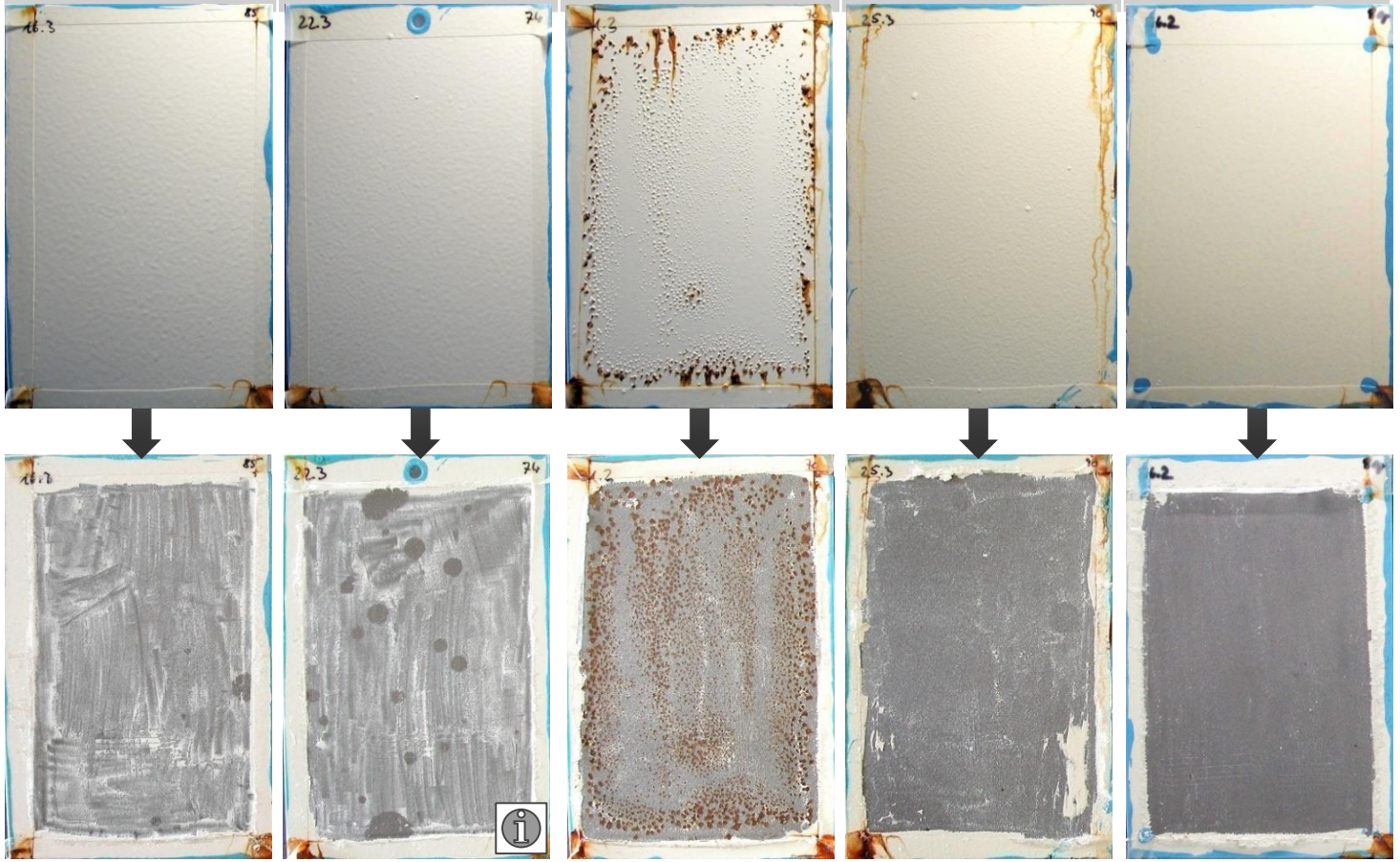
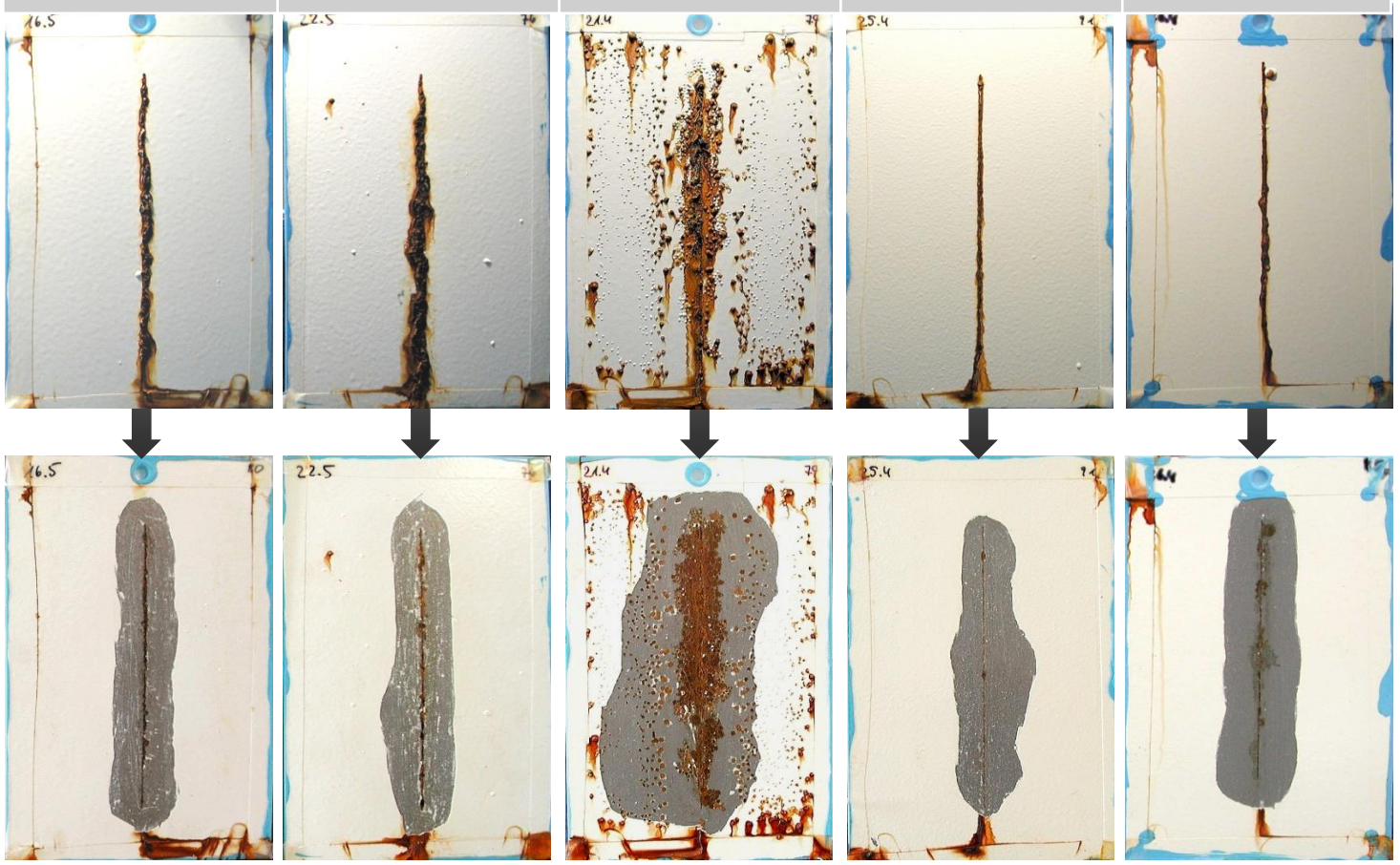
Control 7.5	2.5 pbw zinc phosphate			
Talc	Talc	Quartz/Mica/ Chlorite	Aktisil PF 777	Aktisil AM



Salt Spray Test
Scribe

Control 7.5	2.5 pbw zinc phosphate			
Talc	Talc	Quartz/Mica/ Chlorite	Aktisil PF 777	Aktisil AM



Salt Spray Test
Pitting

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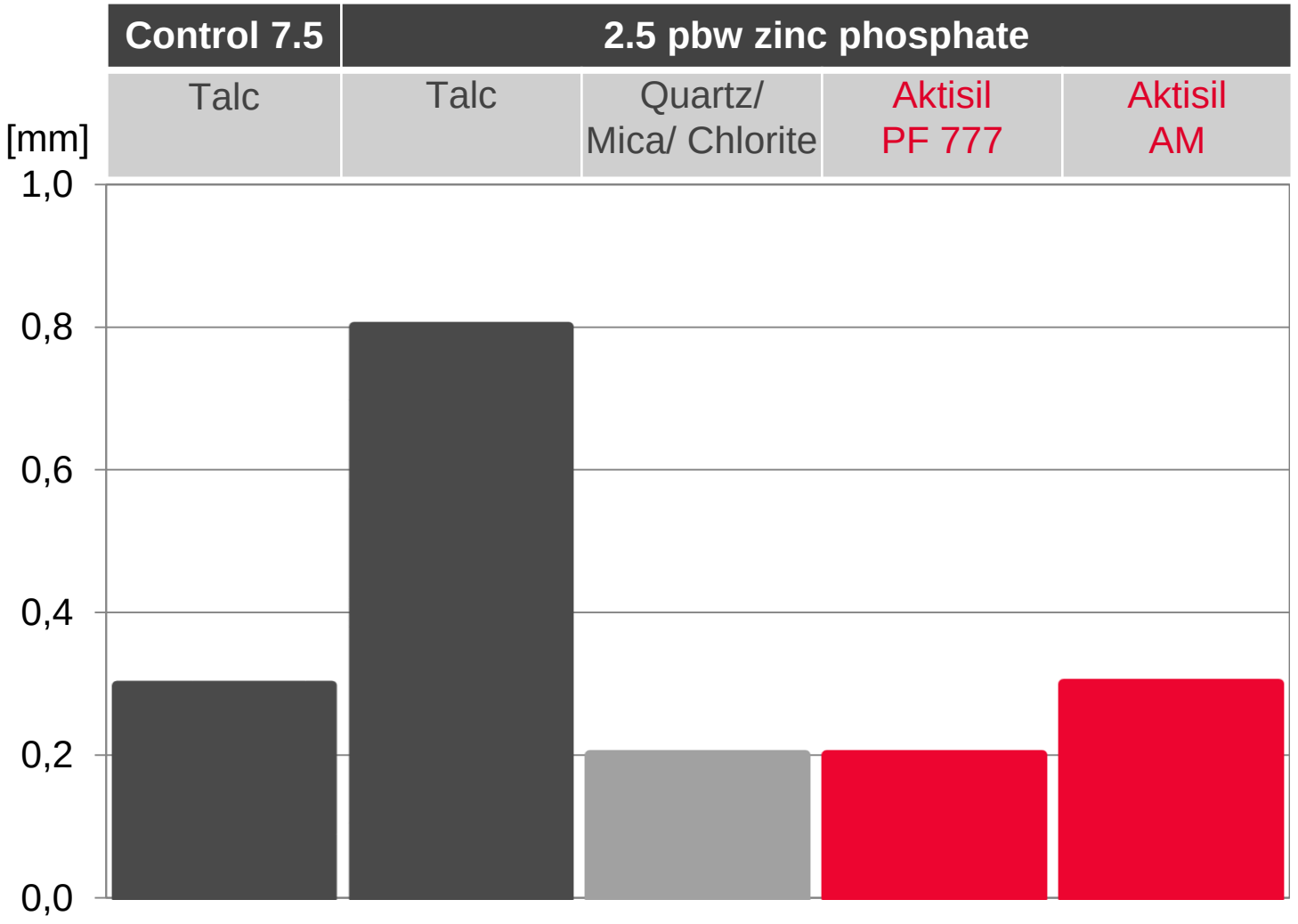
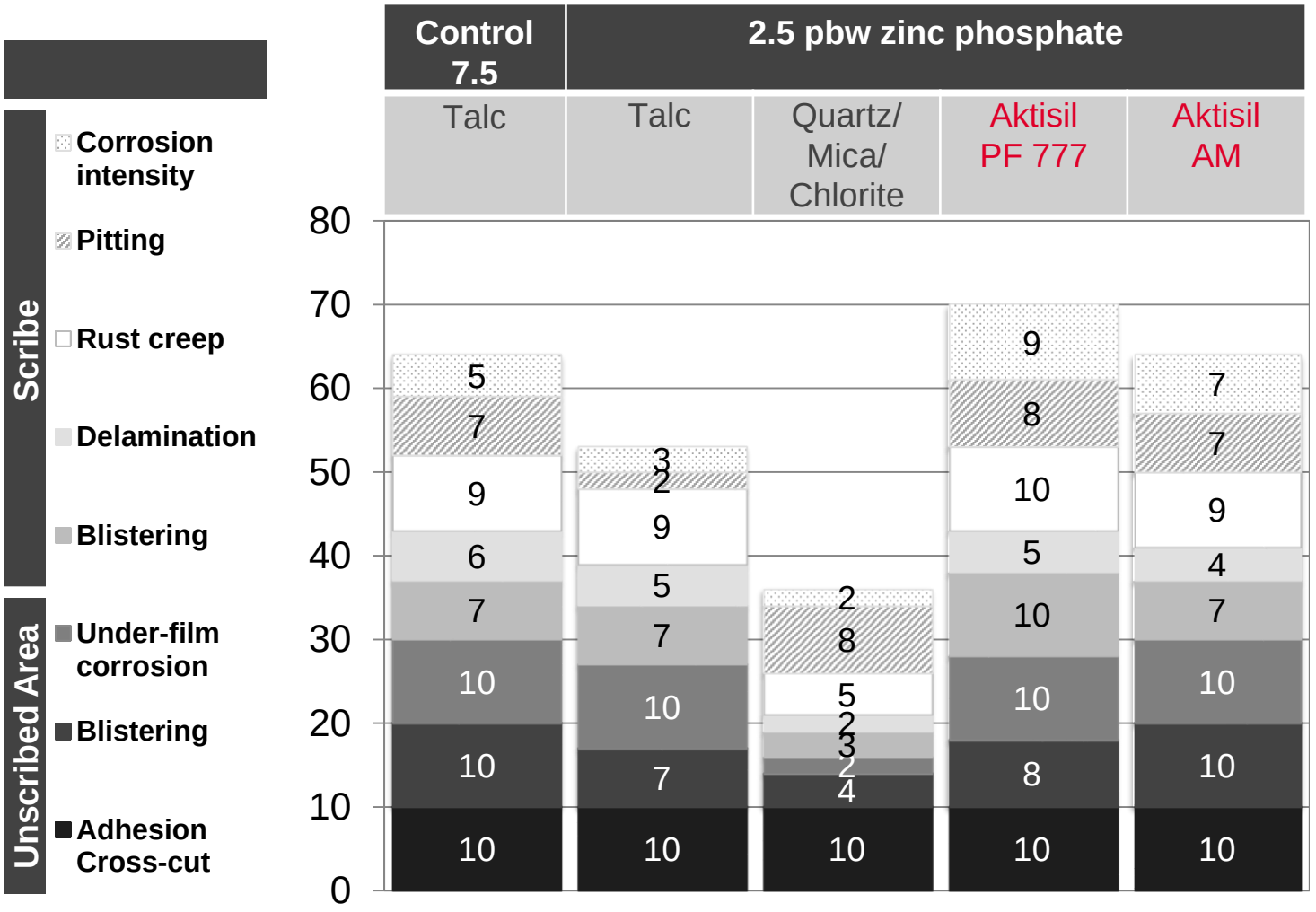
Maximum depth of scribe



Salt Spray Test
Summary Table

		Control 7.5	2.5 pbw zinc phosphate			
		Talc	Talc	Quartz/ Mica/ Chlorite	Aktisil PF 777	Aktisil AM
Unscribed Area						
Adhesion Cross-cut	[GT]	0	0	0	0	0
Blistering	Quantity Size	0 -	1 S3	4-5 S3	1 S2	0 -
Under-film corrosion	[%]	0	0	25	0	0
Scribe						
Blistering	Quantity Size	1 S4	1 S3	5 S3	0 -	1 S4
Delamination	[mm]	11	12	23	12	15
Rust creep	[mm]	0.5	0.7	5.5	0.1	0.4
Pitting max.	[mm]	0.3	0.8	0.2	0.2	0.3
Corrosion intensity	-	moderate	high	very high	very low	low

Salt Spray Test
Rating 80 max.



Corrosion Protection
Total Rating 160 max.

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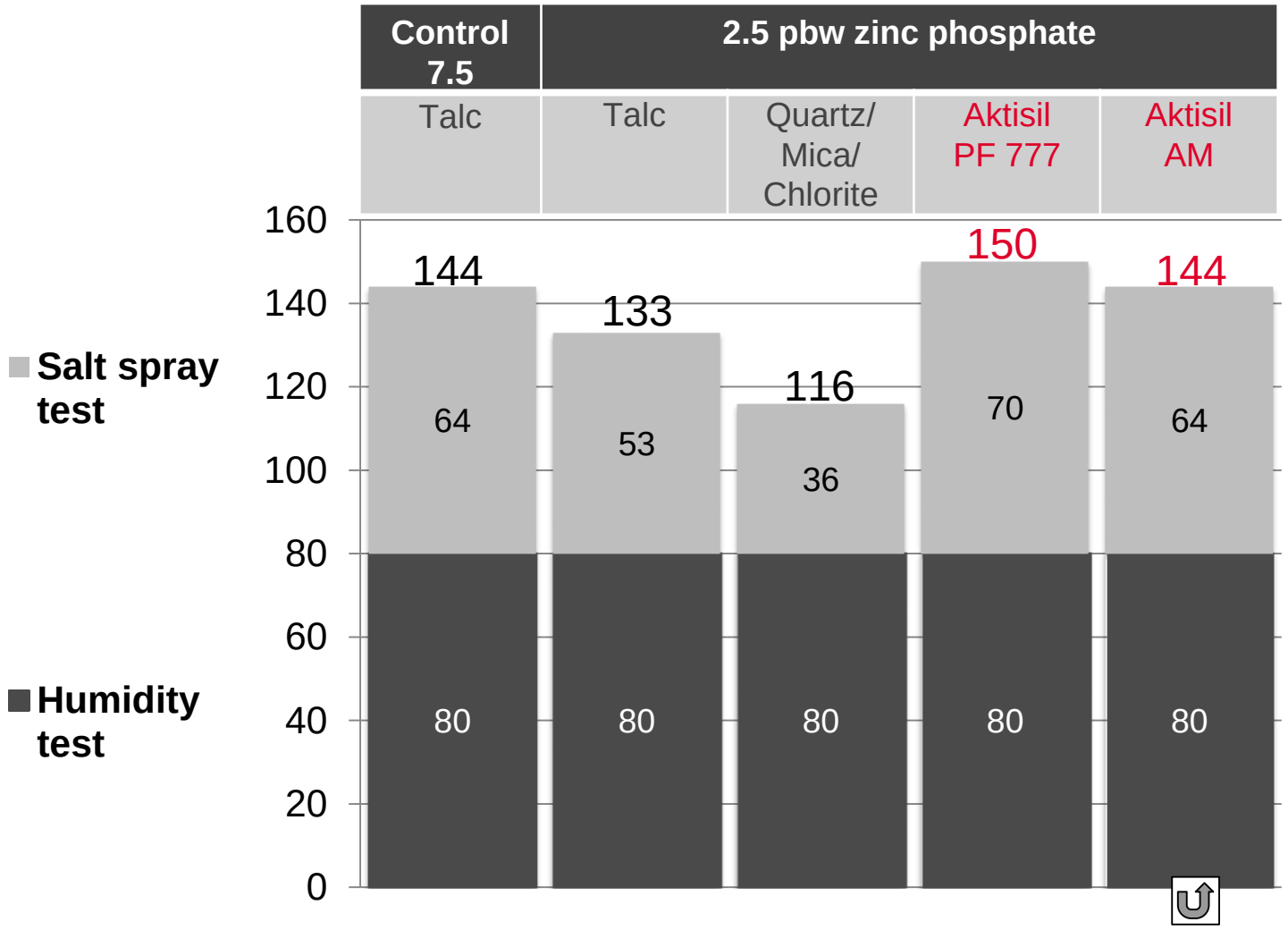

Blasted Steel

Without zinc phosphate

Adhesion prior exposure tests

Humidity test 1000 h, DIN EN ISO 6270-2 CH

Evaluation on scribed and unscribed panels

Salt pray test 1000 h, DIN EN ISO 9227 NSS

Photos: unscribed area

↓

stripped

Photos: scribe

↓

delaminated coating peeled off

Evaluation

Adhesion
prior Exposure Tests

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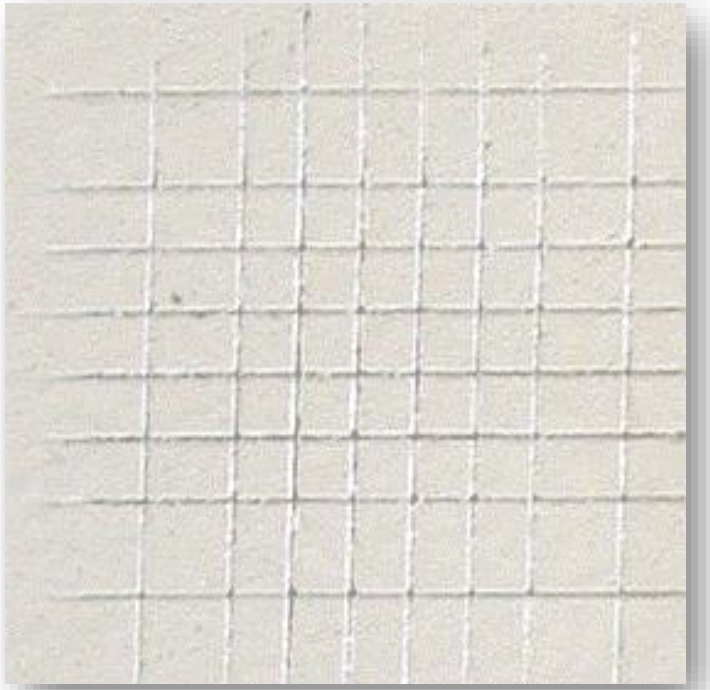
Cross-cut test, 2 mm, with tape tear off, DIN EN ISO 2409

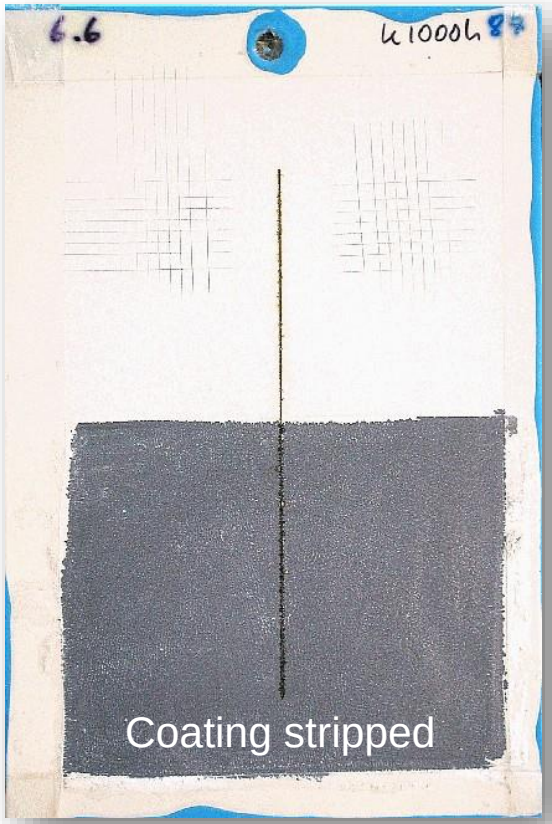


All Formulations:

GT 0

→ Excellent result

Humidity Test



All formulations:

Unscribed area:

Adhesion	GT 0
Blistering	0
Under-film corrosion	0

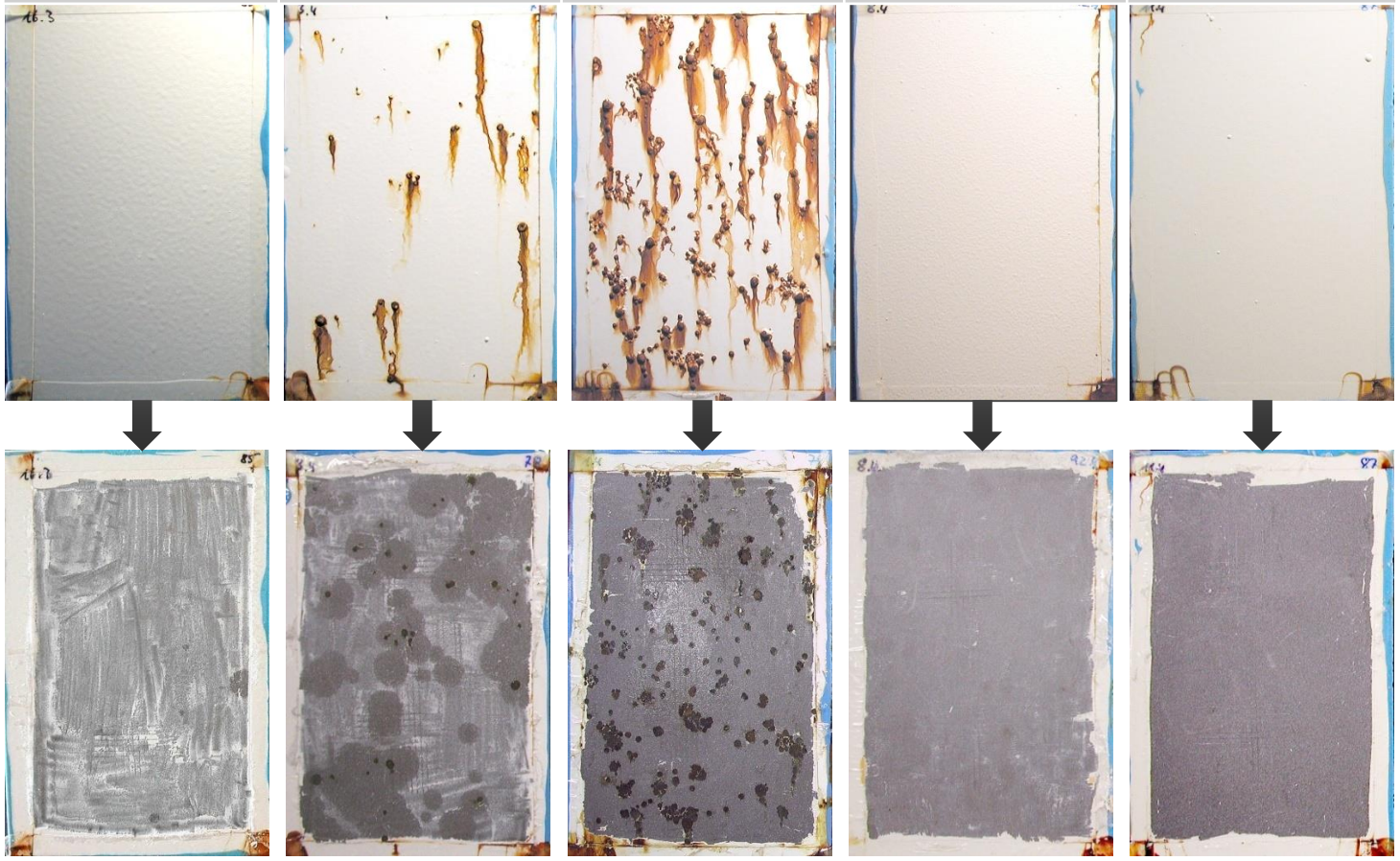
Scribe:

Blistering	0
Delamination	0
Rust creep	< 0.5 mm
Pitting	< 0.1 mm
Corrosion intensity	very low

→ excellent result
(80 of 80 max.)

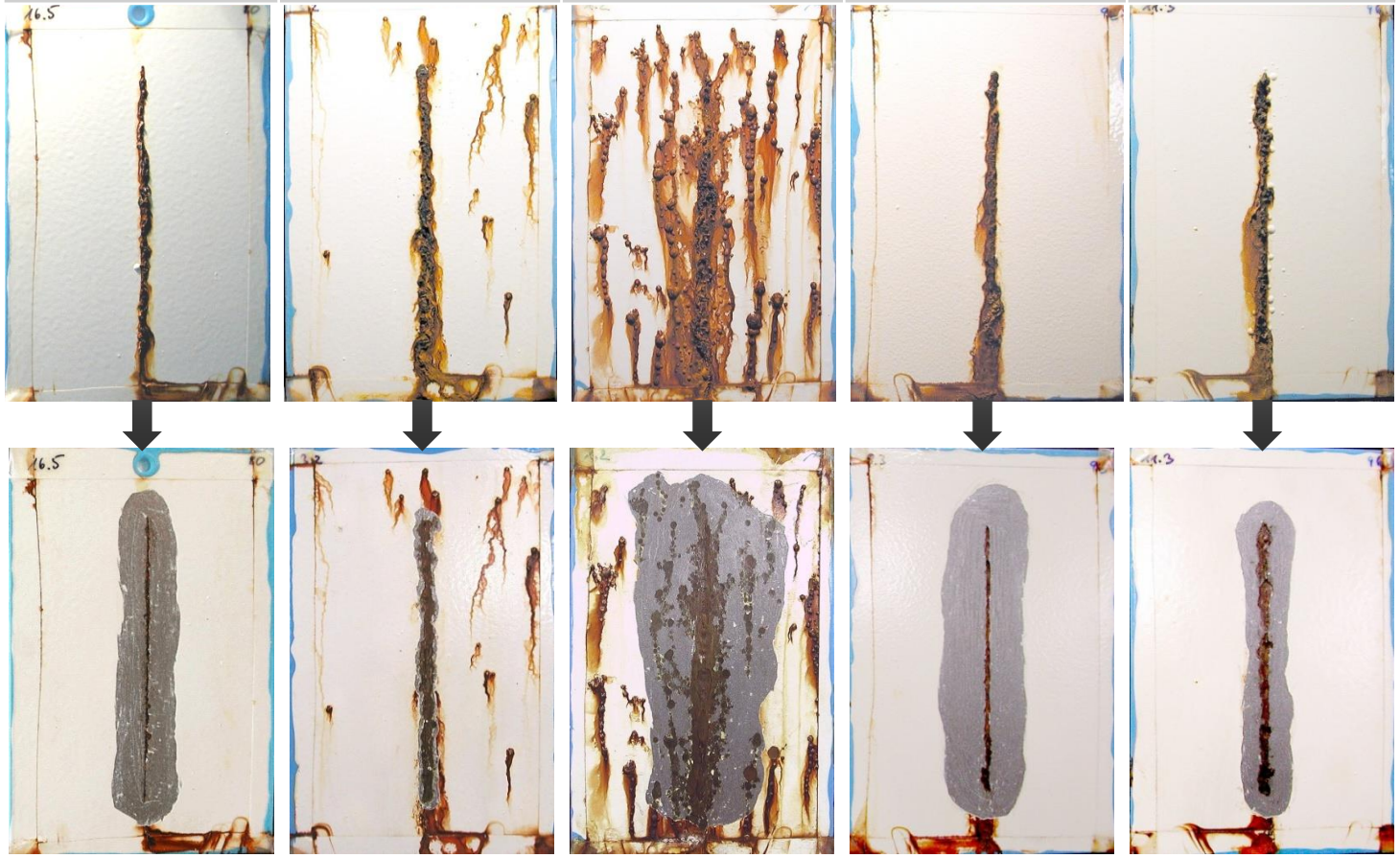
Salt Spray Test
Unscribed Area

Control 7.5	0 pbw zinc phosphate			
Talc	Talc	Quartz/Mica/ Chlorite	Aktisil PF 777	Aktisil AM



Salt Spray Test
Scribe

Control 7.5	0 pbw zinc phosphate			
Talc	Talc	Quartz/Mica/ Chlorite	Aktisil PF 777	Aktisil AM



Salt Spray Test
Pitting

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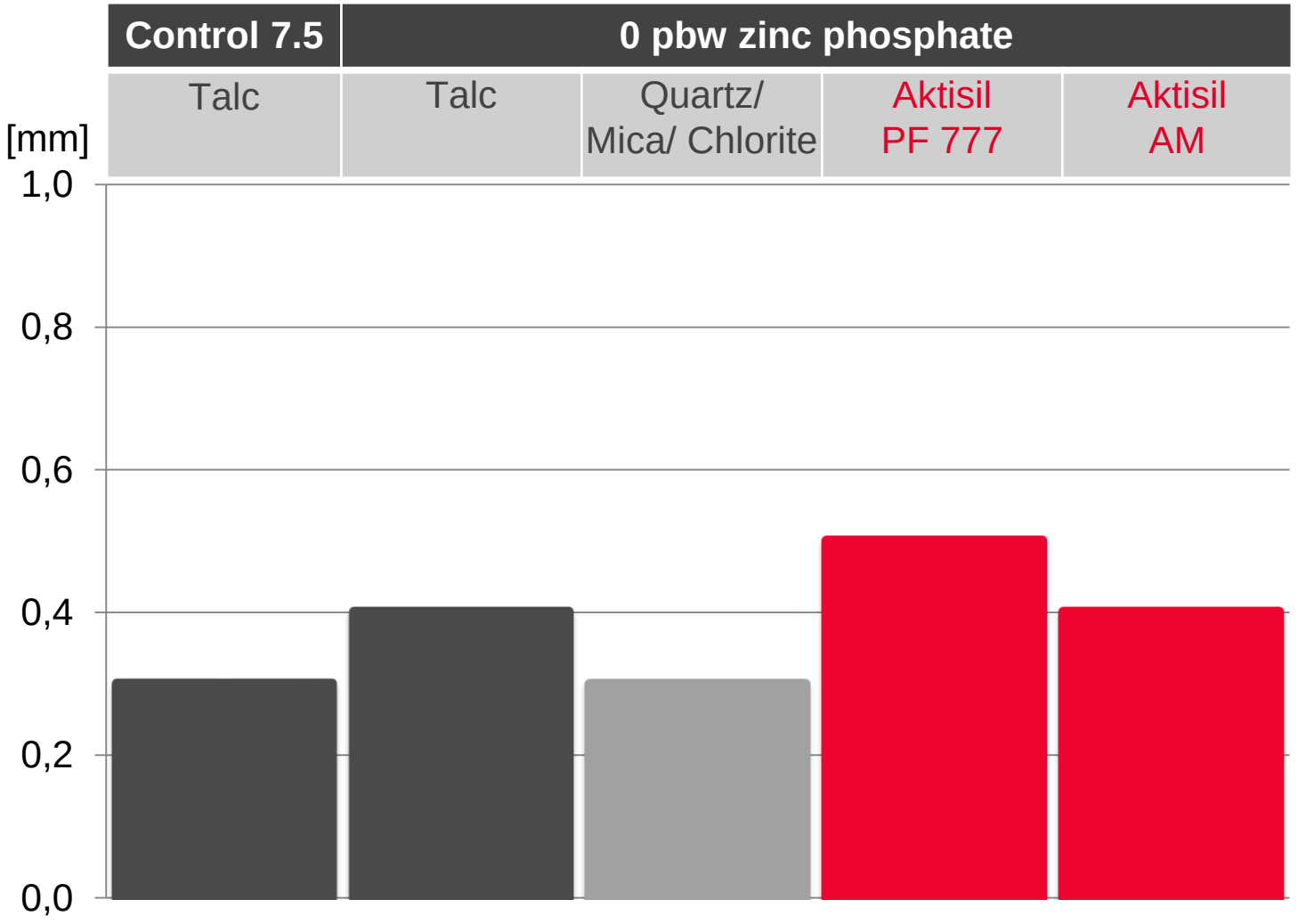
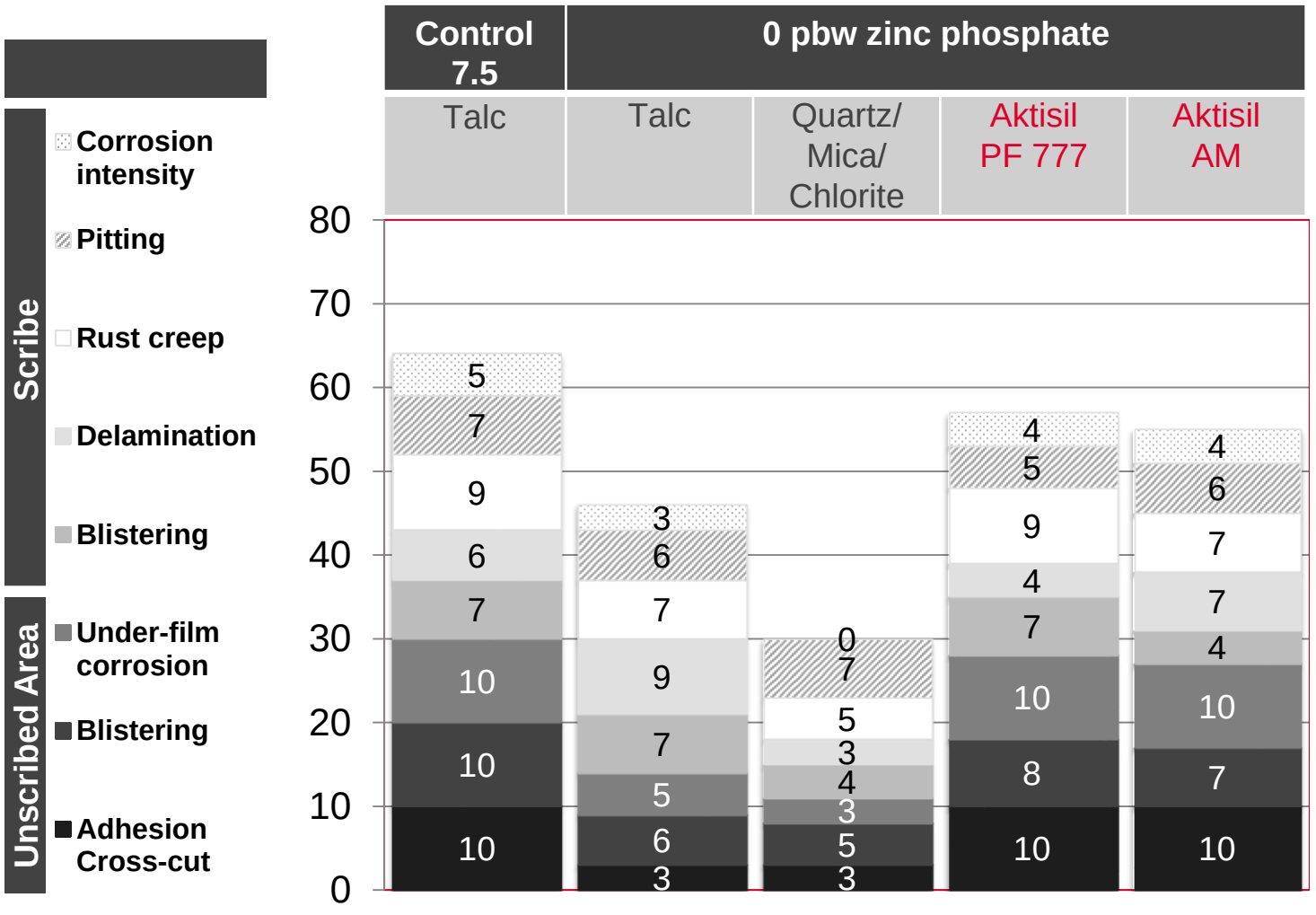
Maximum depth of scribe



Salt Spray Test
Summary Table

		Control 7.5	0 pbw zinc phosphate			
		Talc	Talc	Quartz/ Mica/ Chlorite	Aktisil PF 777	Aktisil AM
Unscribed Area						
Adhesion Cross-cut	[GT]	0	0/5	0/5	0	0
Blistering	Quantity Size	0 -	2 S3-4	3 S4	1 S2	1 S3
Under-film corrosion	[%]	0	2	15	0	0
Scribe						
Blistering	Quantity Size	1 S4	1 S4	4 S4	1 S4	4 S4
Delamination	[mm]	11	3	21	16	8
Rust creep	[mm]	0.5	2.0	5.5	0.7	1.7
Pitting max.	[mm]	0.3	0.4	0.4	0.5	0.4
Corrosion intensity	-	moderate	high	very high	moderate	moderate

Salt Spray Test
Rating 80 max.



Corrosion Protection
Total Rating 160 max.

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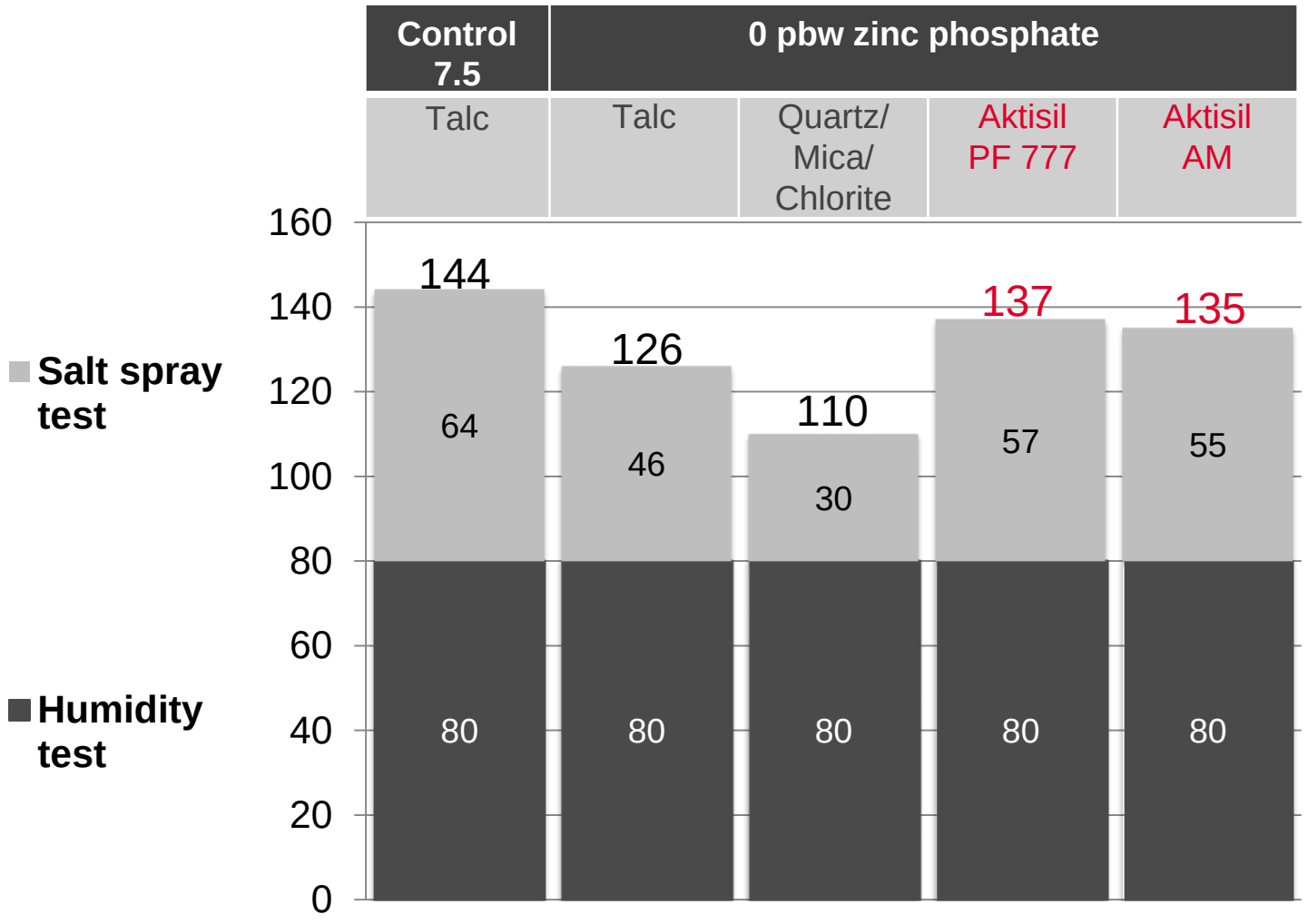


Conclusion
Blasted Steel

Adhesion prior exposure tests

- excellent for all formulations

Humidity test 1000 h

- excellent anti-corrosion properties for all formulations

Salt spray test 1000 h

- reduced loading of zinc phosphate:
  - » generally unaffected very good adhesion
  - » blistering, increased pitting and rust intensity at scribe using talc
  - » the natural mixture of quartz, mica and chlorite fails totally due to strong blistering, intense corrosion even at unscribed area and underneath the primer along with very high delamination and rust creep at scribe
- without zinc phosphate:
  - » further degradation of performance with competitive fillers in particularly with respect to adhesion, rust perforation and rust creep at scribe

Conclusion
Blasted Steel

Salt spray test 1000 h

- Aktisil PF 777 and Aktisil AM perform in
  - » almost non or no blistering
  - » no rust perforations and no corrosion underneath the coating
  - » generally reduced corrosion at scribe, in particular Aktisil PF 777 with extraordinary low pitting, rust creep and corrosion intensity
  - » even without zinc phosphate good protection at scribe, excellent result at unscribed area with better adhesion compared to competitive fillers

✓ zinc phosphate loading can be reduced without losing total performance if the right filler is chosen

Non-blasted Steel

Reduced loading of zinc phosphate

Adhesion prior exposure tests

Humidity test 1000 h, DIN EN ISO 6270-2 CH
Salt spray test 1000 h, DIN EN ISO 9227 NSS

Photos: unscribed area
↓
stripped

Photos: scribe
↓
delaminated coating peeled off

Evaluation

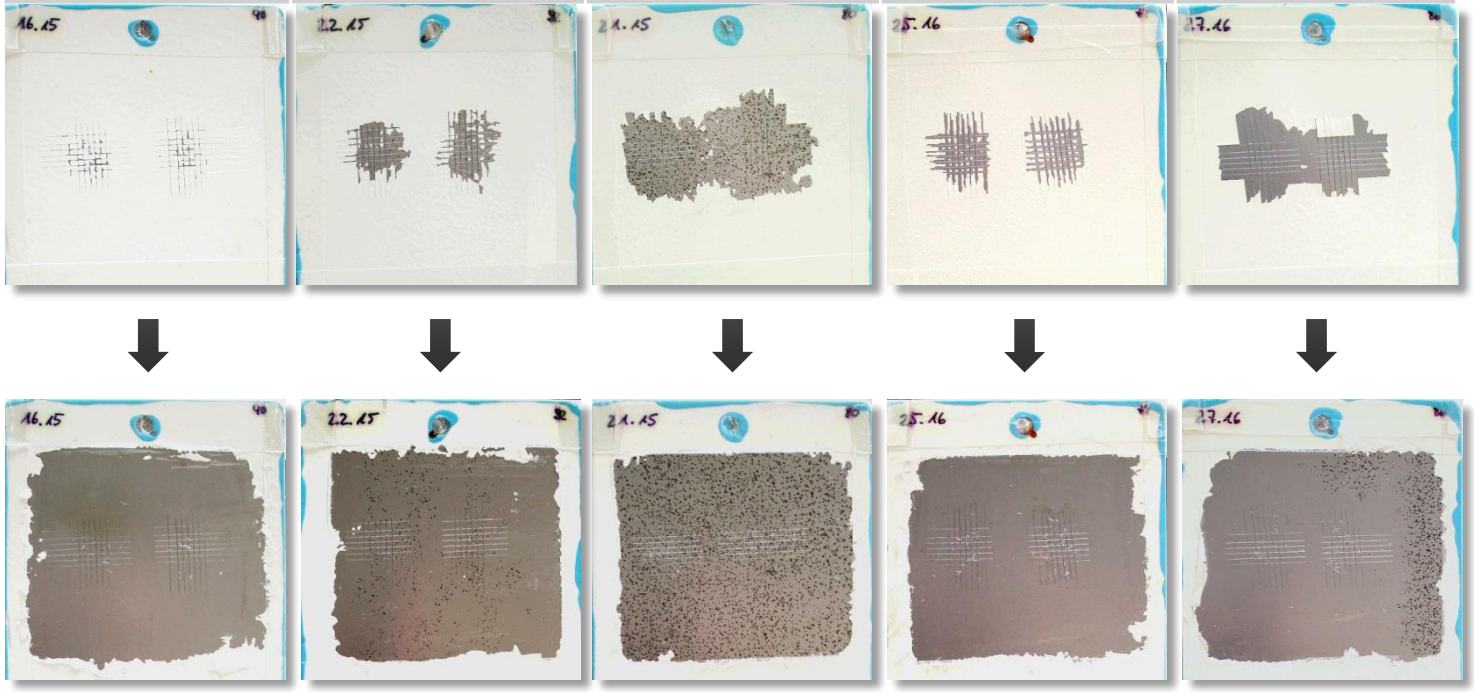
Adhesion
prior Exposure Tests

Cross-cut test, 2 mm, with tape tear off, DIN EN ISO 2409

		Control 7.5	2.5 pbw zinc phosphate			
		Talc	Talc	Quartz/ Mica/ Chlorite	Aktisil PF 777	Aktisil AM
Cross-cut	[GT]	0	0	0	0	1

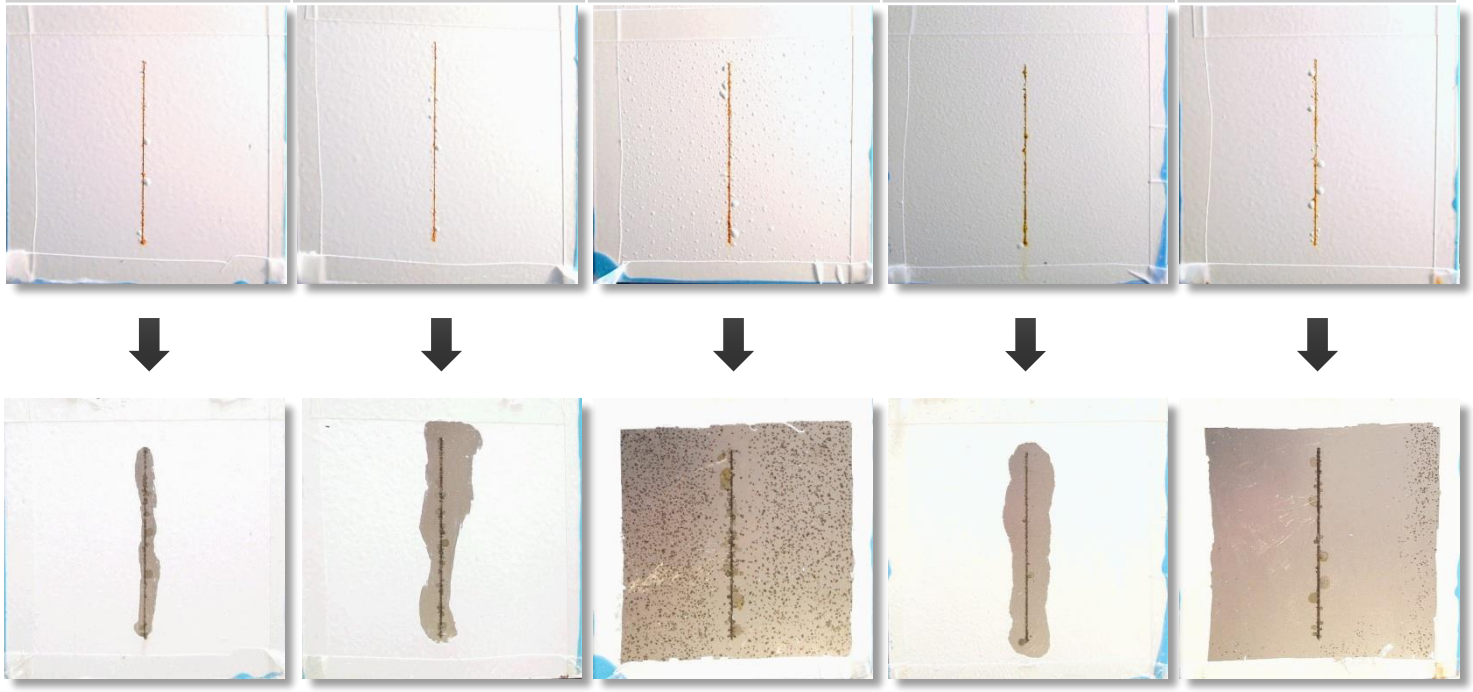
Humidity Test
Unscribed Area

Control 7.5	2.5 pbw zinc phosphate			
Talc	Talc	Quartz/Mica/ Chlorite	Aktisil PF 777	Aktisil AM



Humidity Test Scribe

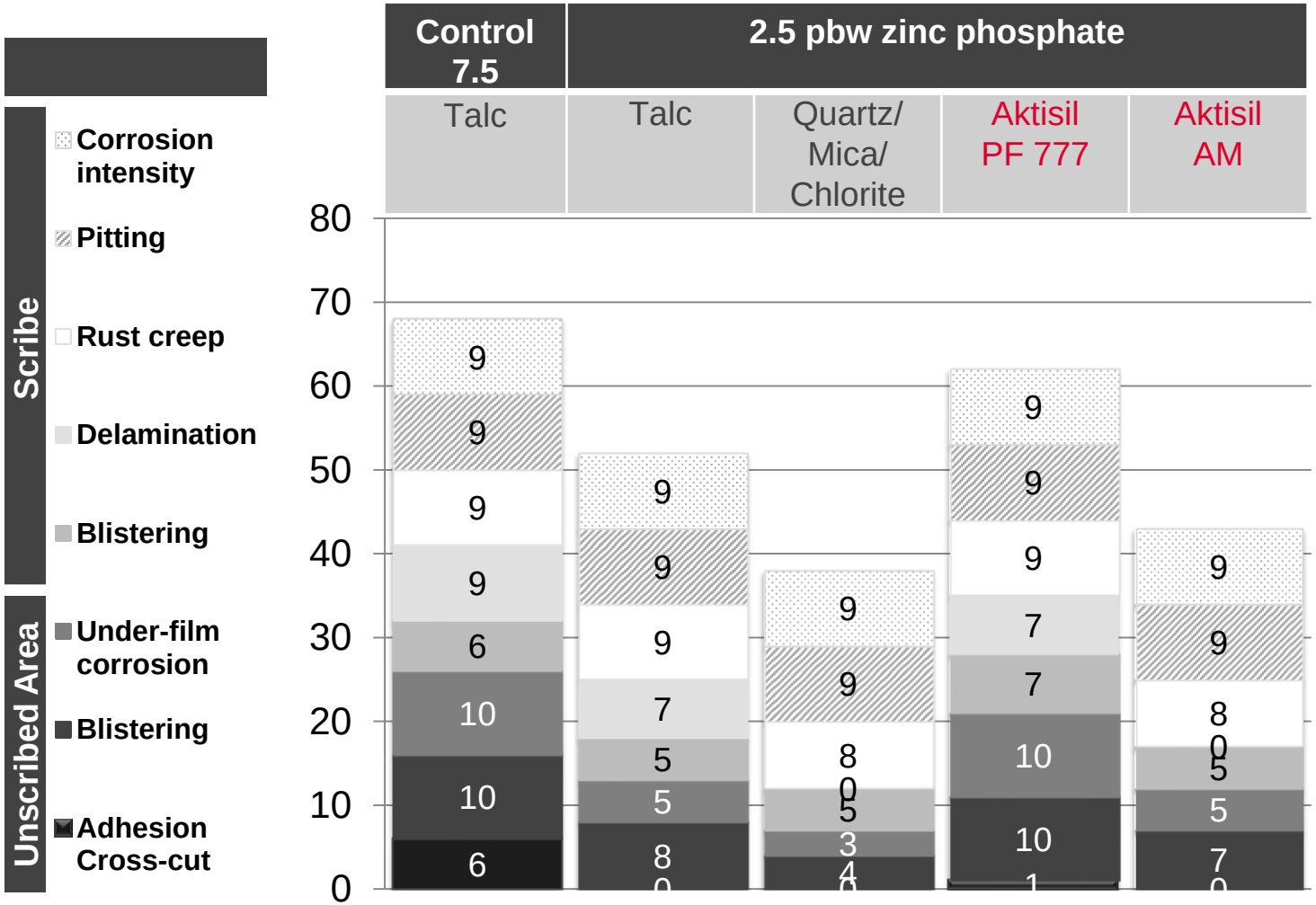
Control 7.5	2.5 pbw zinc phosphate			
Talc	Talc	Quartz/Mica/ Chlorite	Aktisil PF 777	Aktisil AM



Humidity Test Summary Table

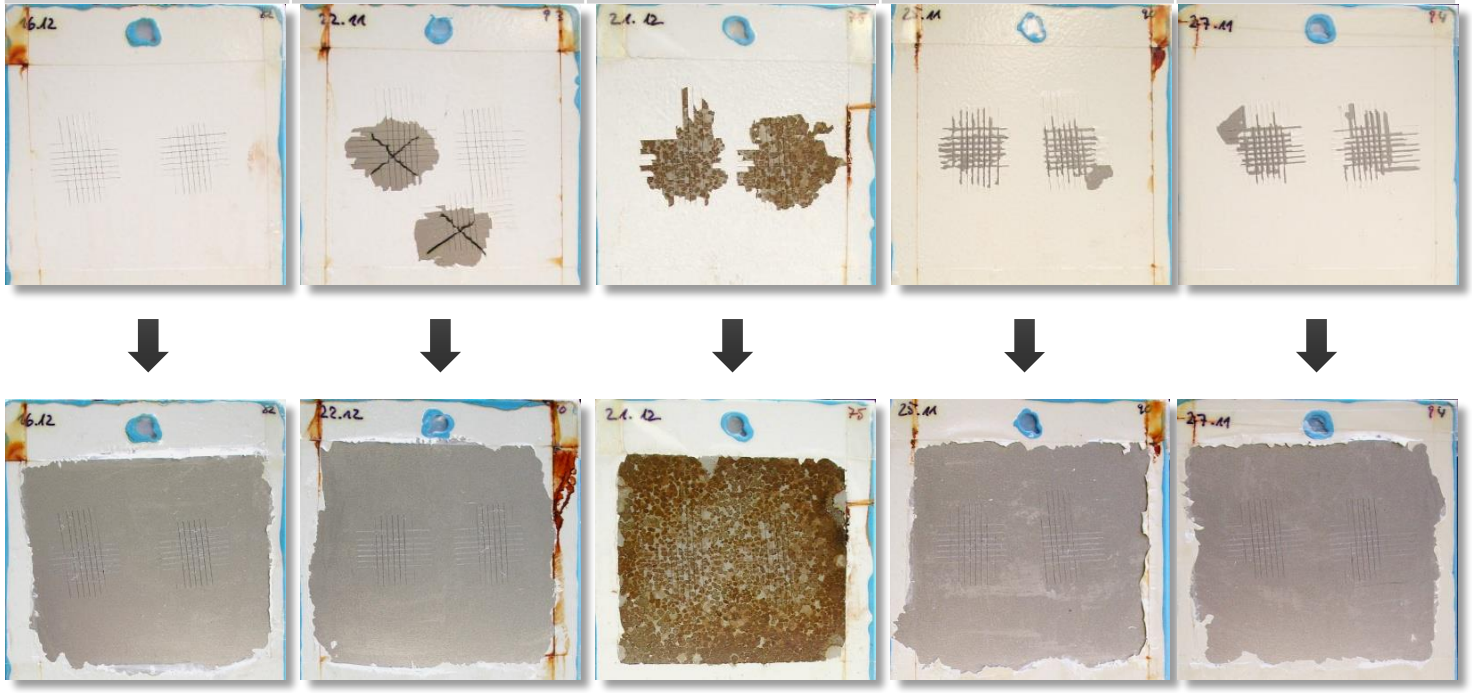
		Control 7.5	2.5 pbw zinc phosphate			
		Talc	Talc	Quartz/ Mica/ Chlorite	Aktisil PF 777	Aktisil AM
Unscribed Area						
Adhesion Cross-cut	[GT]	2	5	5	4-5	5
Blistering	Quantity Size	0 -	2-3 2	4-5 3	0 -	2-3 2
Under-film corrosion	[%]	-	4	20	-	4
Scribe						
Blistering	Quantity Size	2 4	2-3 3	3 4	1 3-4	3 4
Delamination	[mm]	3	7	> 40	7	> 40
Rust creep	[mm]	0.4	0.6	0.8	0.4	0.8
Pitting max.	[mm]	0.1	0.1	0.1	0.1	0.1
Corrosion intensity	-	very low	very low	very low	very low	very low

Humidity Test
Rating 80 max.



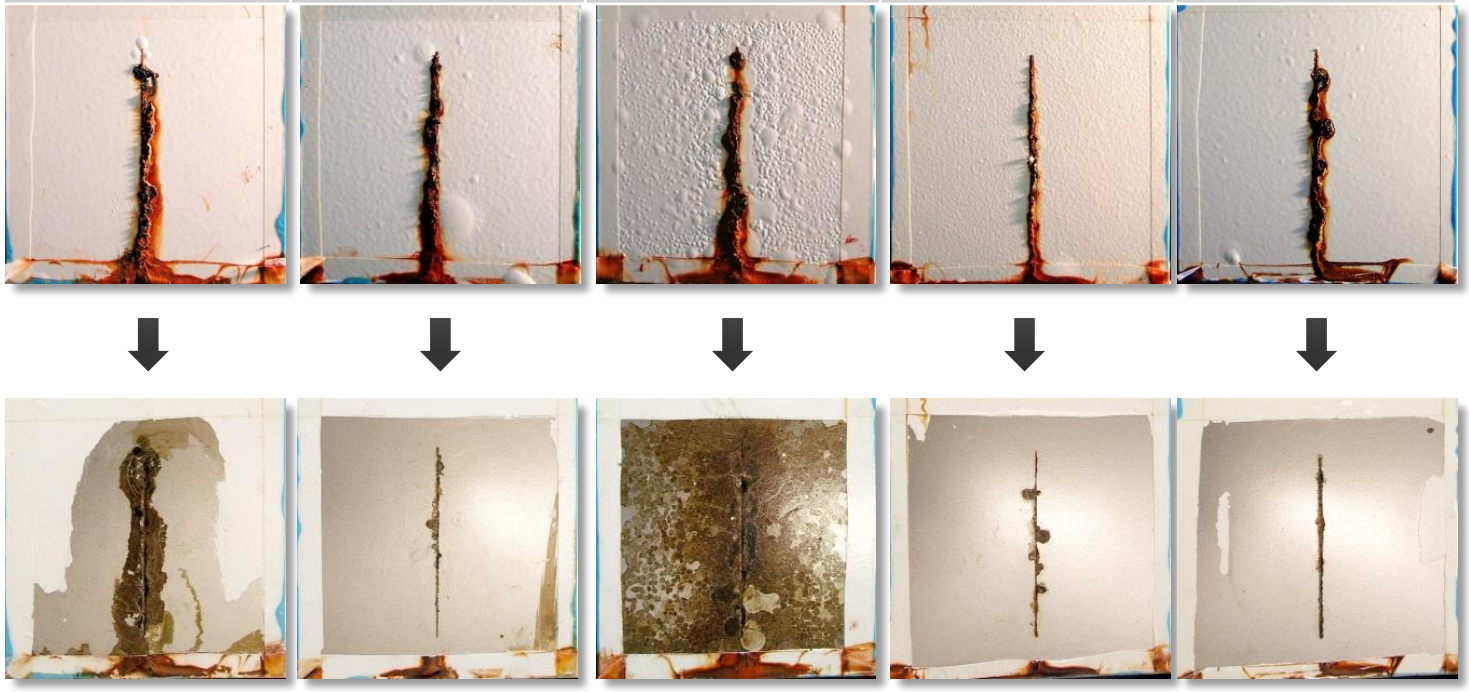
Salt Spray Test
Unscribed Area

Control 7.5	2.5 pbw zinc phosphate			
Talc	Talc	Quartz/Mica/ Chlorite	Aktisil PF 777	Aktisil AM



Salt Spray Test
Scribe

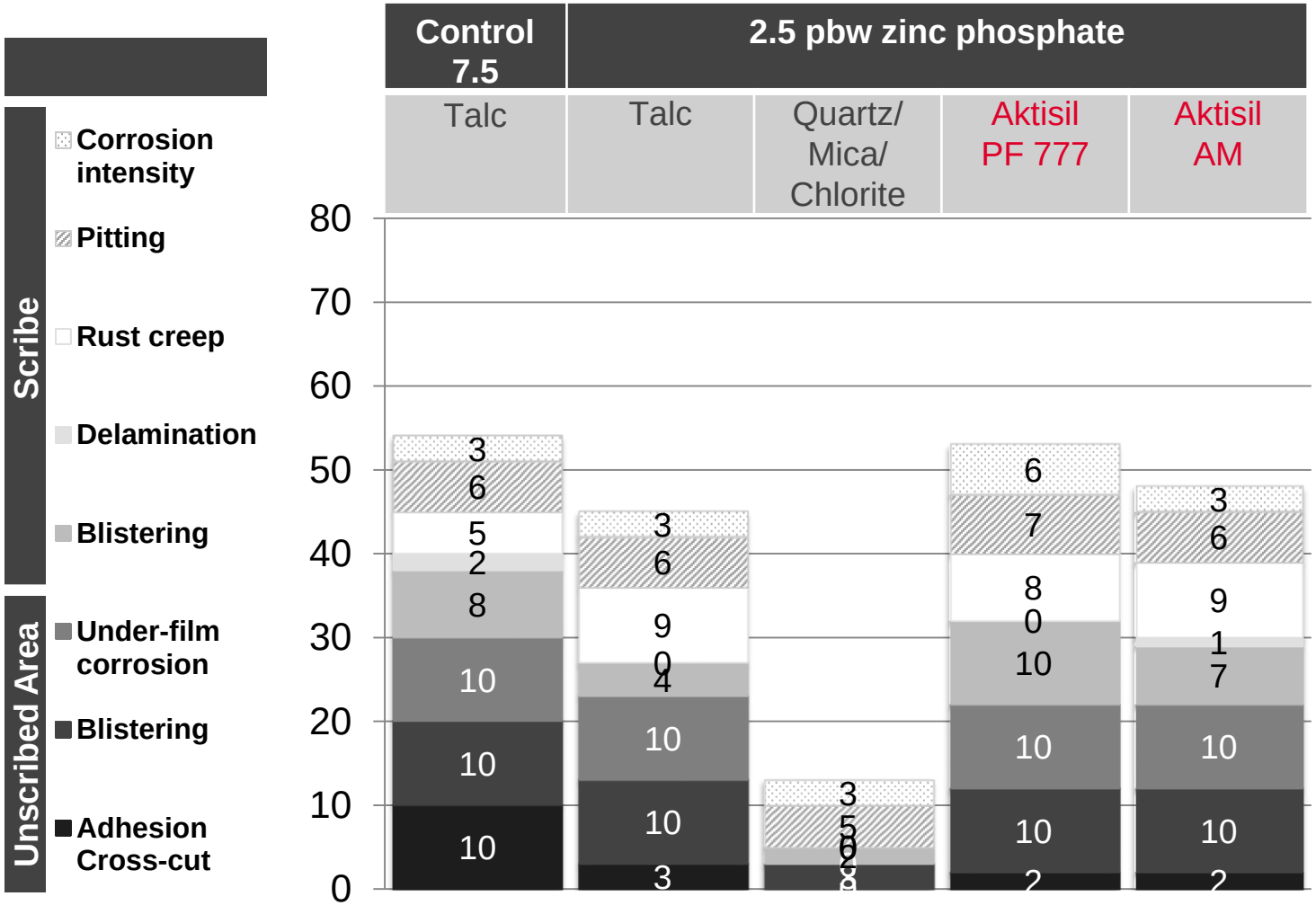
Control 7.5	2.5 pbw zinc phosphate			
Talc	Talc	Quartz/Mica/ Chlorite	Aktisil PF 777	Aktisil AM



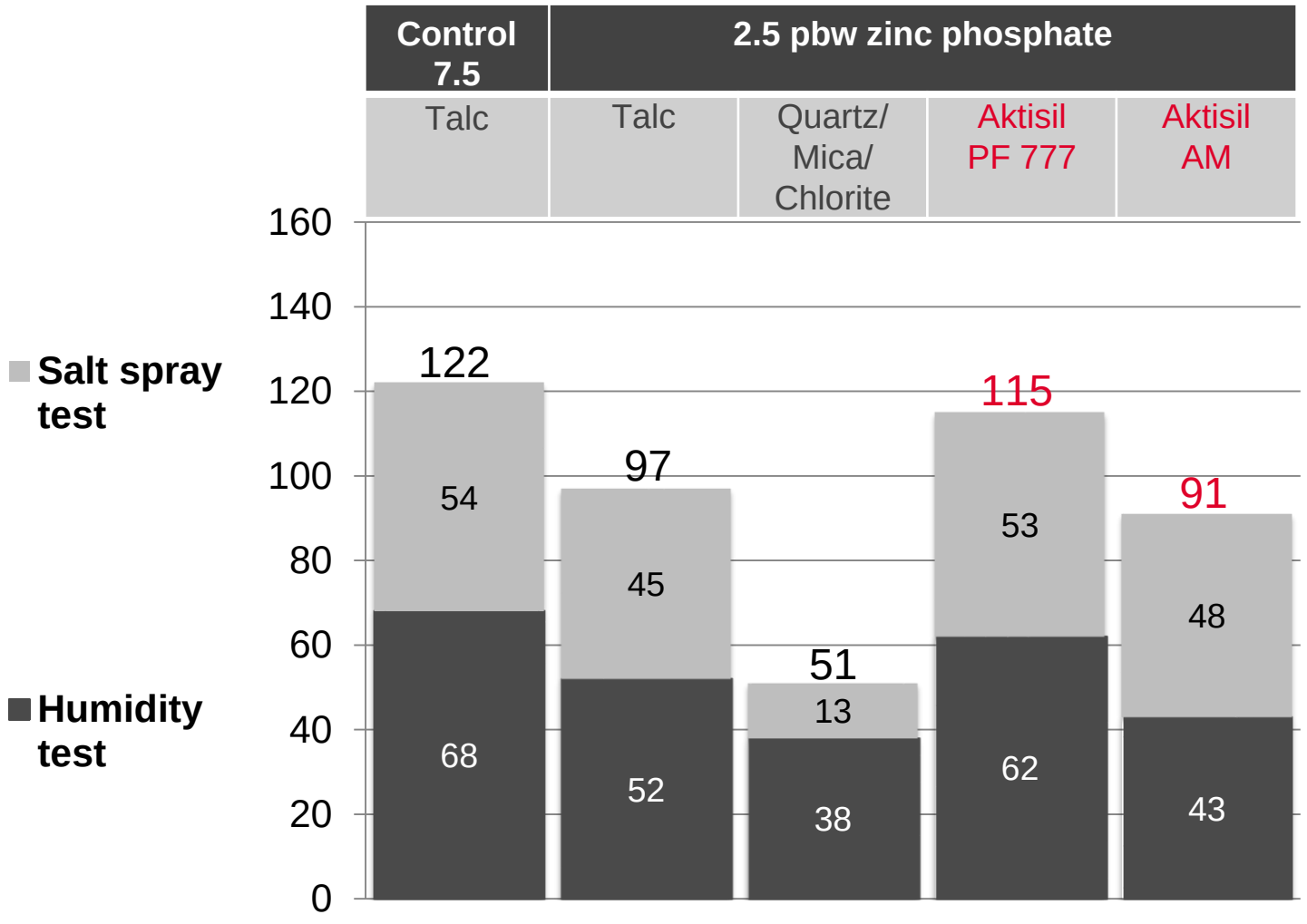
Salt Spray Test
Summary Table

		Control 7.5	2.5 pbw zinc phosphate			
		Talc	Talc	Quartz/ Mica/ Chlorite	Aktisil PF 777	Aktisil AM
Unscribed Area						
Adhesion Cross-cut	[GT]	0	0/5	5	4	4
Blistering	Quantity Size	0 -	0 -	5 3-4	0 -	0 -
Under-film corrosion	[%]	0	0	70	0	0
Scribe						
Blistering	Quantity Size	2 2	3 5	5 5	0 -	1 3
Delamination	[mm]	25	> 40	> 40	> 40	30
Rust creep	[mm]	4	0.5	21	1	0.7
Pitting max.	[mm]	0.4	0.5	0.4	0.3	0.4
Corrosion intensity	-	high	high	high	moderate	high

Salt Spray Test
Rating 80 max.



Corrosion Protection
Total Rating 160 max.



Conclusion
Non-blasted Steel

Adhesion prior exposure tests

- very good for all formulations, Aktisil AM good

Humidity test 1000 h and alt spray test 1000 h

- reduced loading of zinc phosphate generally results in
  - » significantly decreased adhesion after exposure, expecially after humidity test
  - » blistering and under-film corrosion in humidity test
  - » high delamination rate and increased blistering at scribe especially in salt spray test
  - » using the natural mixture of quartz, mica and chlorite, it fails totally
  - » Aktisil PF 777 performs in high resistance against blistering on surface and at scribe, a rust-free steel surface and moderate corrosion intensity at scribe
  - » Aktisil AM comes out lower on a level comparable to talc

– total performance worsens reducing zinc phosphate loading

Addition of Amino Silane

- 1 part by weight of amino silane dosed to hardener (Polyamidoamine, Versamid 115).
- This variant was carried out only with reduced loading of zinc phosphate for talc, Aktisil PF 777 and Aktisil AM.

Adhesion prior exposure tests

Humidity test 1000 h, DIN EN ISO 6270-2 CH
Salt spray test 1000 h, DIN EN ISO 9227 NSS

Photos: unscribed area
↓
stripped

Photos: scribe
↓
delaminated coating peeled off

Evaluation

Adhesion
prior Exposure Tests

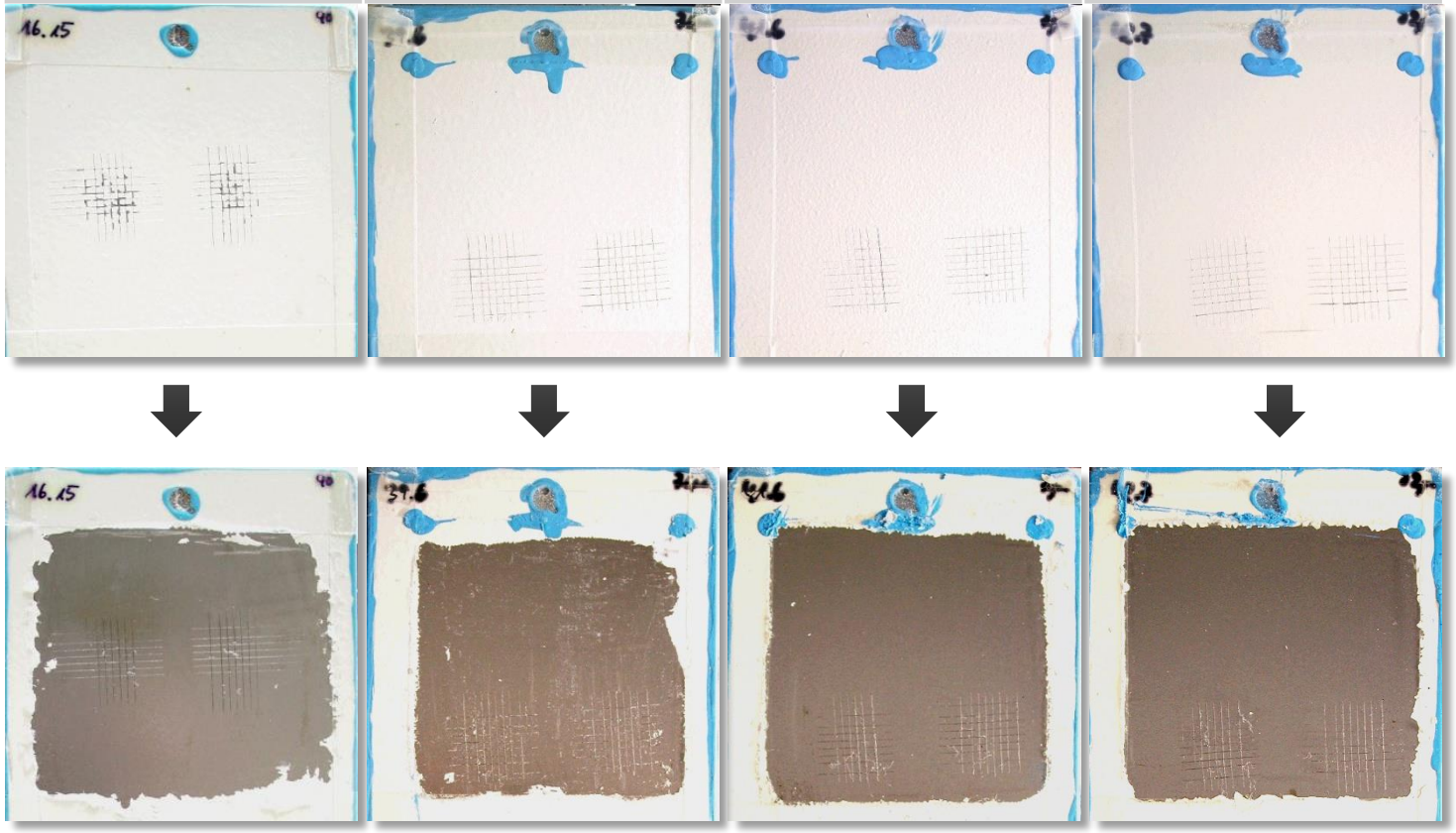
Cross-cut test, 2 mm, with tape tear off, DIN EN ISO 2409

		Control 7.5	2.5 pbw zinc phosphate + silane		
		Talc	Talc	Aktisil PF 777	Aktisil AM
Cross-cut	[GT]	0	0	0	0

Humidity Test
Unscribed Area

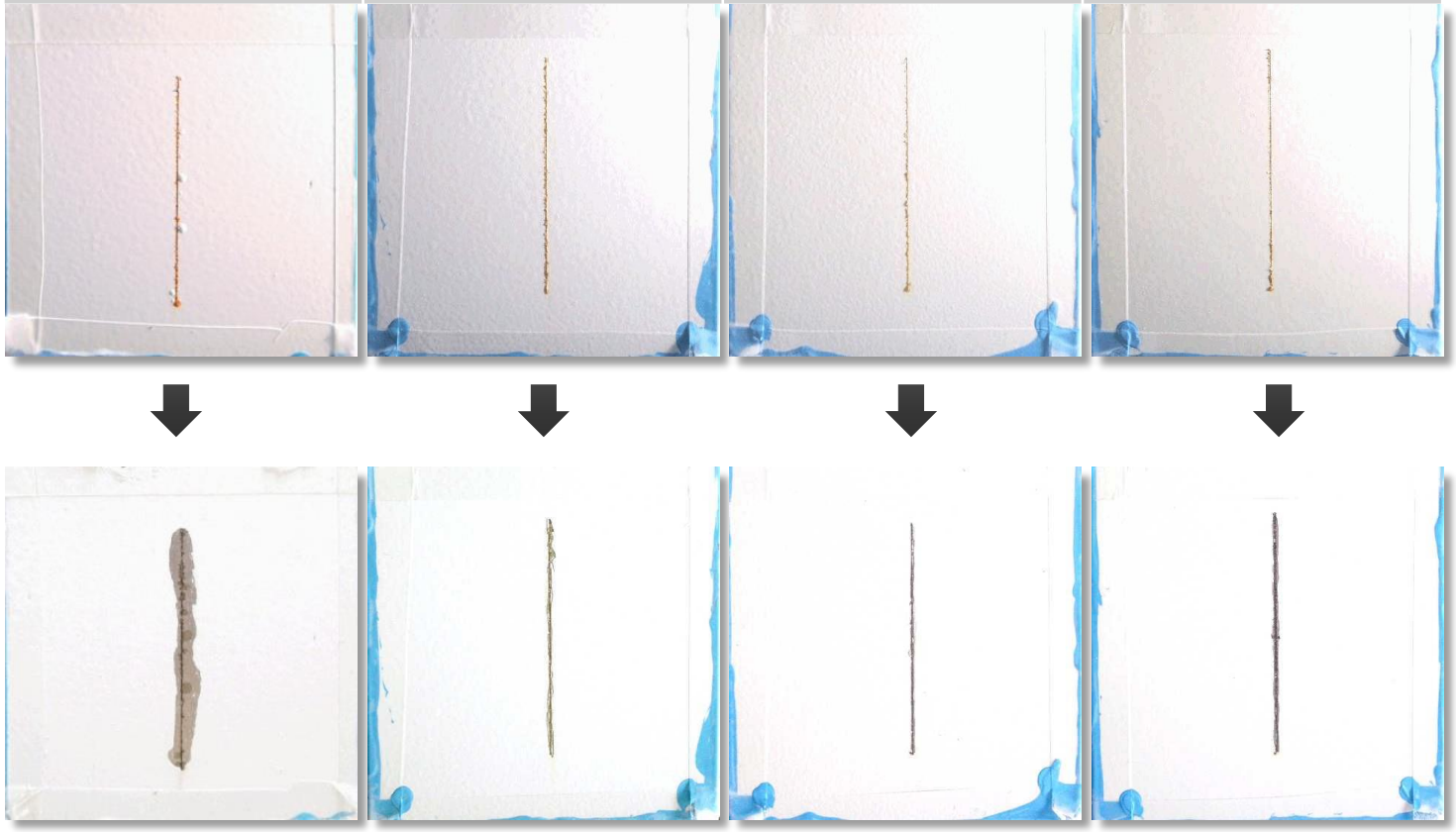
HOFFMANN MINERAL

Control 7.5	2.5 pbw zinc phosphate + silane		
Talc	Talc	Aktisil PF 777	Aktisil AM



Humidity Test Scribe

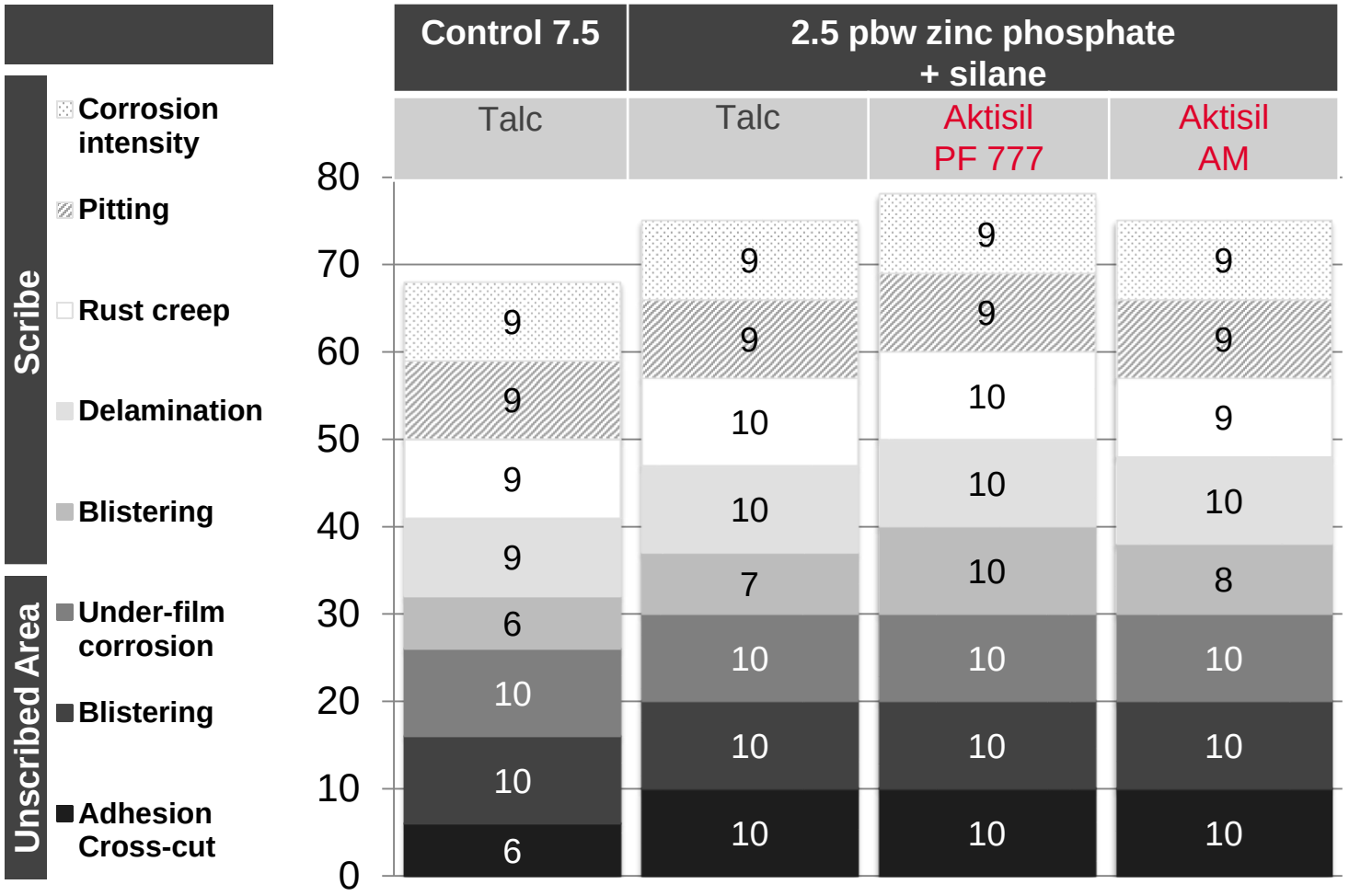
Control 7.5	2.5 pbw zinc phosphate		
Talc	Talc	Aktisil PF 777	Aktisil AM



Humidity Test
Summary Table

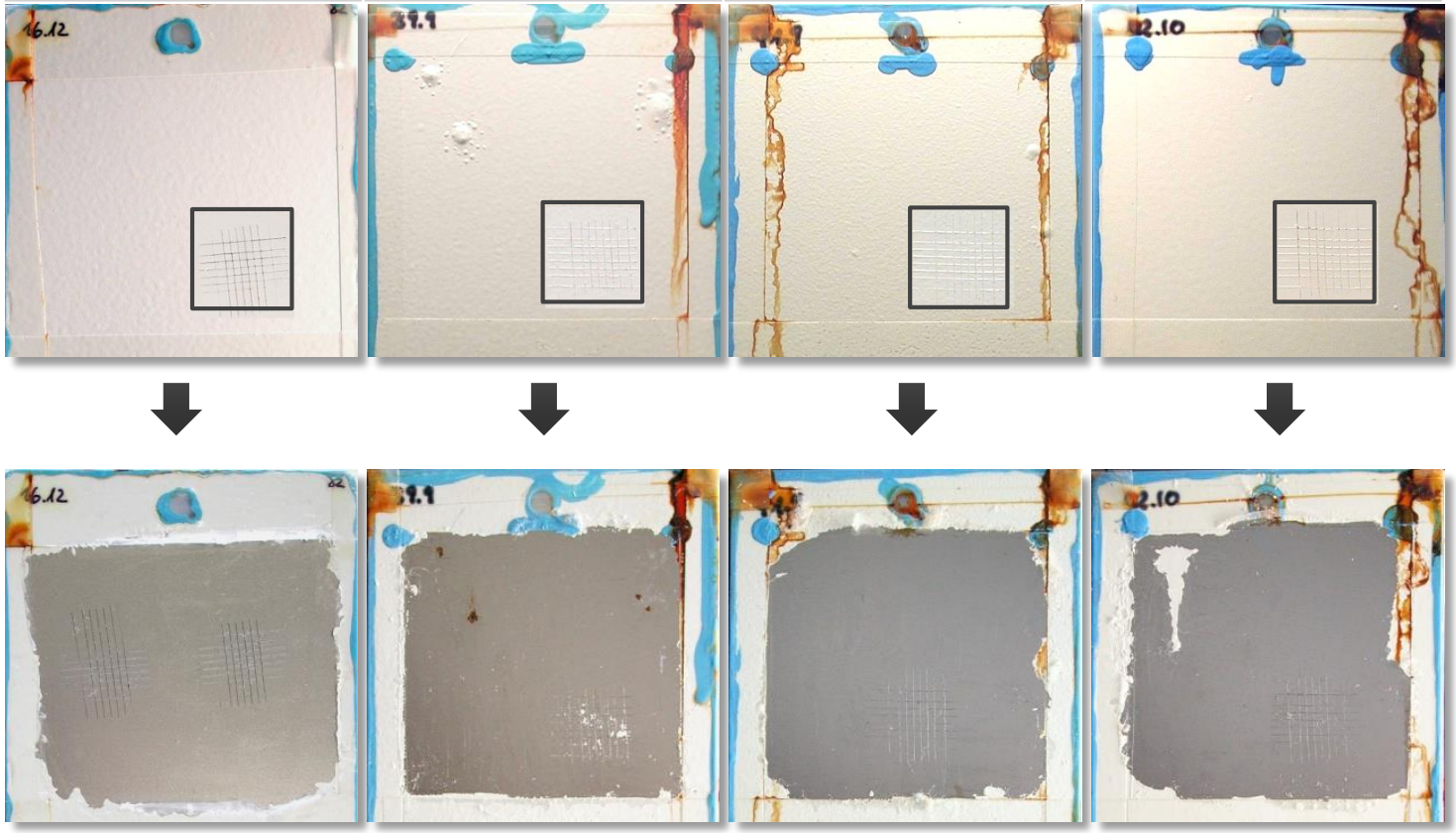
		Control 7.5	2.5 pbw zinc phosphate + silane		
		Talc	Talc	Aktisil PF 777	Aktisil AM
Unscribed Area					
Adhesion Cross-cut	[GT]	2	0	0	0
Blistering	Quantity Size	0 -	0 -	0 -	0 -
Under-film corrosion	[%]	0	0	0	0
Scribe					
Blistering	Quantity Size	2 4	1 3	0 -	1 2
Delamination	[mm]	3	0.5	0.5	0.8
Rust creep	[mm]	0.4	0.1	0.1	0.4
Pitting max.	[mm]	0.1	0.1	0.1	0.1
Corrosion intensity	-	very low	very low	very low	very low

Humidity Test
Rating 80 max.



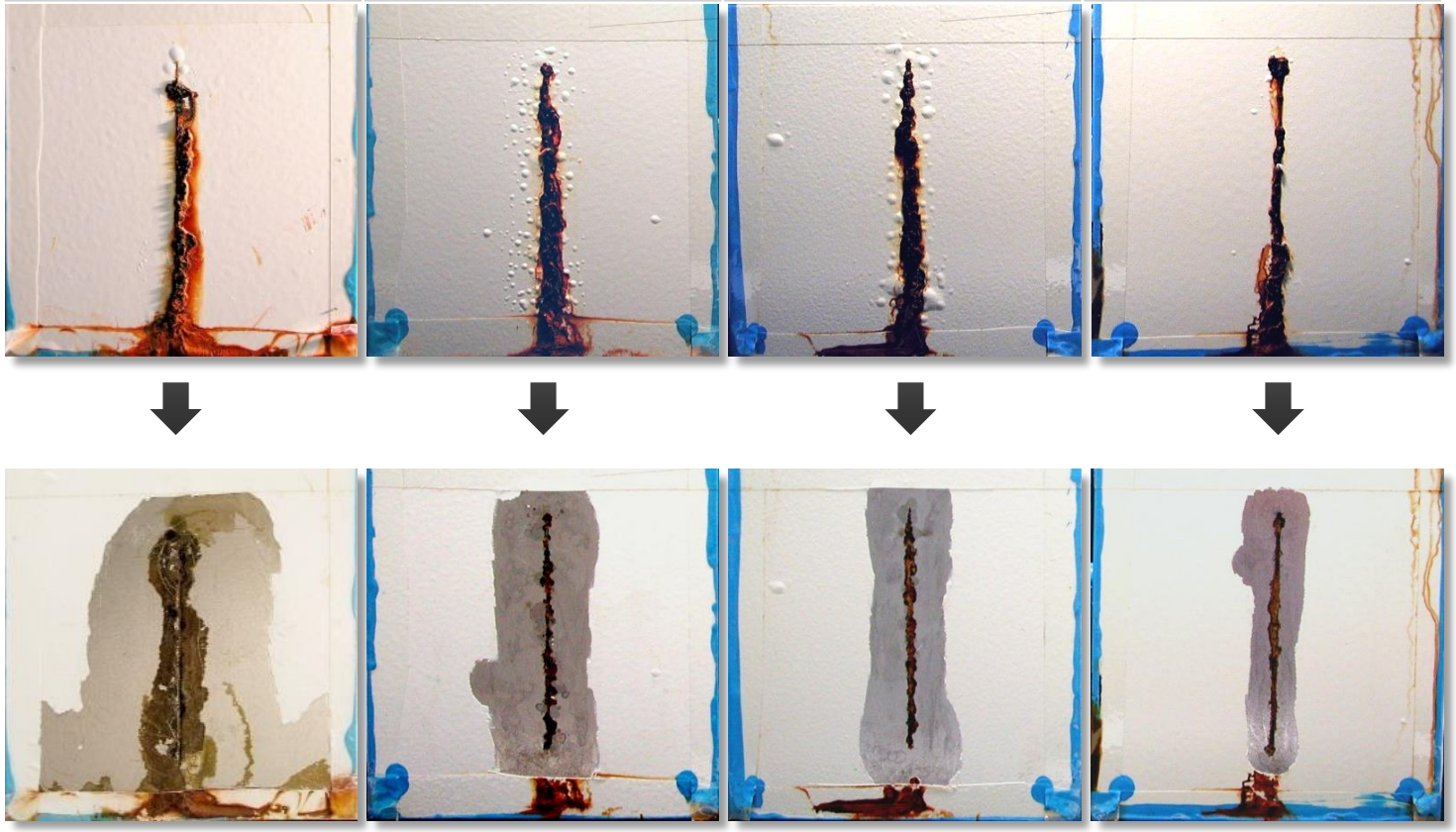
Salt Spray Test
Unscribed Area

Control 7.5	2.5 pbw zinc phosphate + silane		
Talc	Talc	Aktisil PF 777	Aktisil AM



Salt Spray Test
Scribe

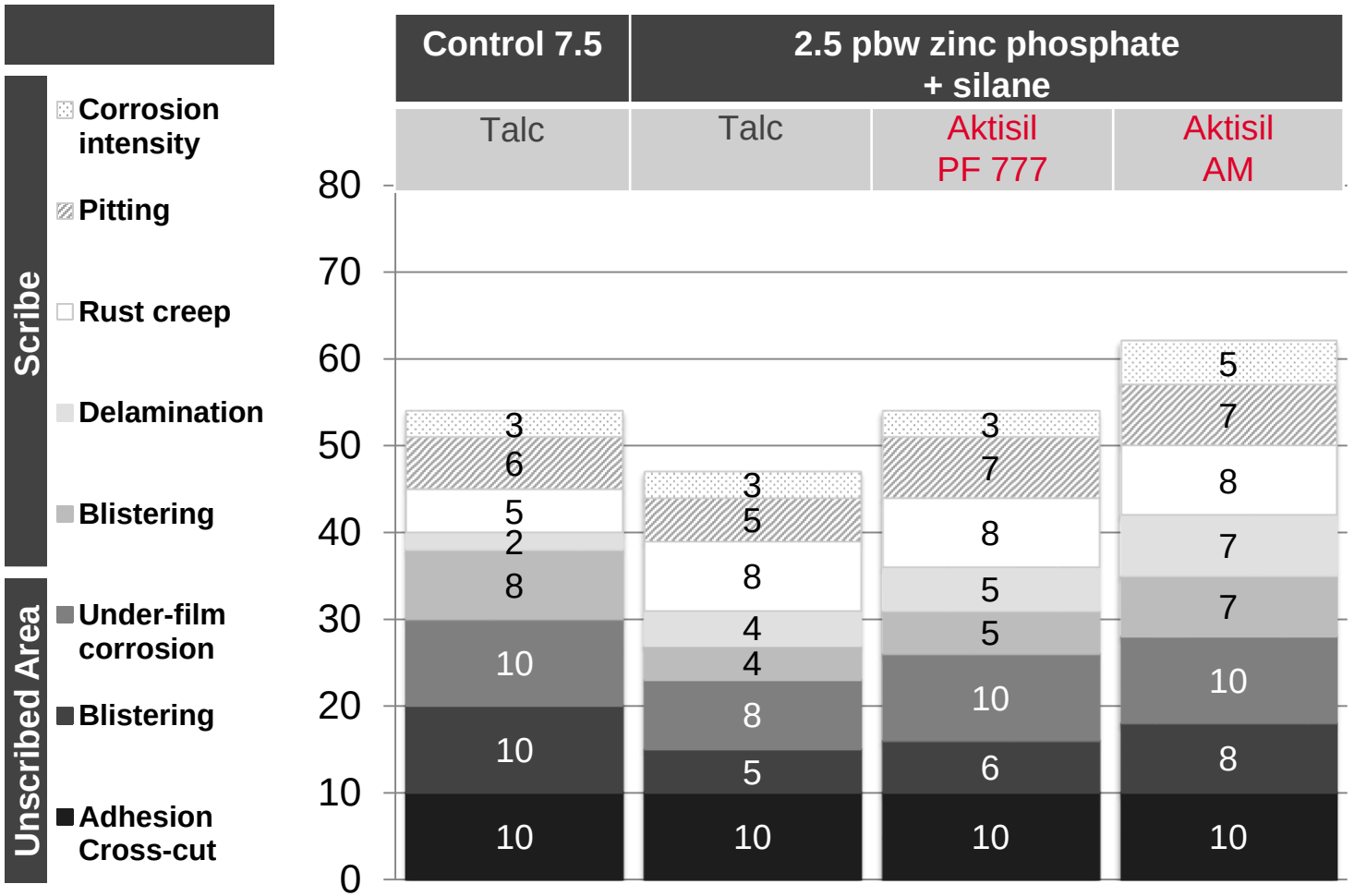
Control 7.5	2.5 pbw zinc phosphate + silane		
Talc	Talc	Aktisil PF 777	Aktisil AM



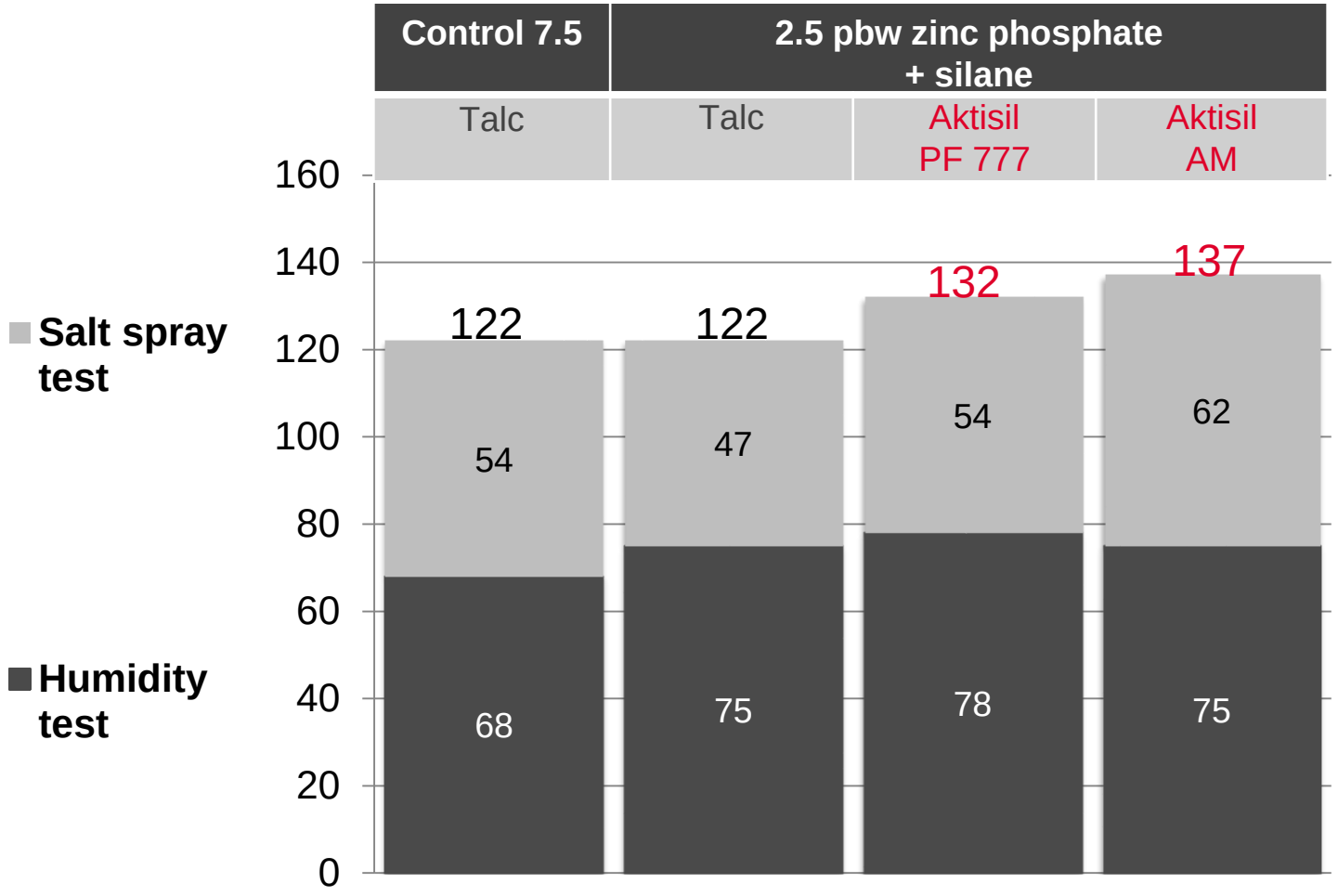
Salt Spray Test
Summary Table

		Control 7.5	2.5 pbw zinc phosphate + silane		
		Talc	Talc	Aktisil PF 777	Aktisil AM
Unscribed Area					
Adhesion Cross-cut	[GT]	0	0	0	0
Blistering	Quantity Size	0 -	1-2 5	1 4	0-1 2-3
Under-film corrosion	[%]	0	0.2	0	0
Scribe					
Blistering	Quantity Size	2 2	4 4	3 4	1 3
Delamination	[mm]	25	15	12	8
Rust creep	[mm]	4	1.3	1.3	1.3
Pitting max.	[mm]	0.4	0.5	0.3	0.3
Corrosion intensity	-	high	high	high	moderate

Salt Spray Test
Rating 80 max.



Corrosion Protection
Total Rating 160 max.



Conclusion
Addition of Amino Silane

Adhesion prior exposure tests

- excellent results for all formulations

Humidity test 1000 h and Salt spray test 1000 h

- Enhanced general performance:
  - » significantly promoted and excellent adhesion after exposure
  - » markedly reduced blistering on surface and at scribe as well as corrosion-free steel substrates in humidity test
  - » reduced delamination, in particular after humidity test
- In particular along with Aktisil AM outstanding performance at least comparable to controll formulation, regarding delamination and rust creep, even optimized efficiency.
- Optimum silane loading should be below 1 part by weight in a range of about 0.5 to 1 %.

✓ Zinc phosphate loading can be reduced without loosing total performance, if the right filler in combination with amino silane is chosen.

Summary

Surface modified Neuburg Siliceous Earth like

Aktisil PF 777 or Aktisil AM offers:

- High anti-corrosion performance already in single-layered coating
  - » on blasted steel with reduced loading or even without zinc phosphate.
  - » on non-blasted steel in combination with amino silane and reduced loading of zinc phosphate.
- Better performance compared to talc or a natural mixture of quartz, mica and chlorite.
- Combined with low dosage of zinc phosphate at least comparable to performance level of common standard systems with conventional fillers and high dosage of zinc phosphate.

Filler Recommendation

Aktisil PF 777

- hydrophobic
- rheological activity, suitable for thick film applications
- early hardness development
- highly protective against corrosion at scribe on blasted steel
- highly protective against blistering and corrosion at unscribed area

Aktisil AM

- good levelling
- high protection against corrosion at scribe on blasted steel
- high protection against blistering and corrosion at unscribed area
- combined with aminosilane beneficial synergetical effect in terms of excellent adhesion and good anti-corrosion performance on non-blasted steel

Starting Formulations

	[1], [2] preferably on blasted steel [3] preferably on non-blasted steel or adhesion-critical substrate		[1] pbw	[2] pbw	[3] pbw
A	**Epikote 1001 x 75**	Solid epoxy resin based on Bisphenol A, 75% in xylene, EEW 633	**23.8**	**23.8**	**23.8**
	Bentone 34 paste	Rheological additive, 10% in xylene/ethanol 87:3	**0 - 4.3**	**4.3**	**4.3**
	Xylene	Solvent	**6.5**	**6,5**	**6.5**
	Ethylglycol	Solvent	**4.7**	**4.7**	**4.7**
	MIBK	Solvent, Methylisobutylketone	**6.6**	**6.6**	**6.6**
	Nusa 57	Wetting and dispersion additive	**0.4**	**0.4**	**0.4**
	BYK-354	Leveling additive	**0.8**	**0.8**	**0.8**
	Sachtleben RD3	Titanium dioxide	**5.9**	**5.9**	**5.9**
	Zinc phosphate	Anti-corrosion pigment	**2.5**	**2.5**	**2.5**
	Aktisil PF 777	Neuburg Siliceous Earth	**21.3**	**-**	**-**
	Aktisil AM	Neuburg Siliceous Earth	**-**	**21.3**	**21.3**
	Blanc fixe	Barium sulfate ppt	**7.8**	**7.8**	**7.8**
B	**Versamid 115 x 70**	Polyamide resin, 70% in xylene, HEW 283	**12.7**	**12.7**	**12.7**
	Dynasylan AMEO	Amino silane	**-**	**-**	**< 1.0**
Total			**97.3**	**97.3**	**98.3**

Solid content w/w	[%]	**67.1**	**67.1**	**67.4**
Pigment volume concentration (PVC)	[%]	**33.8**	**33.8**	**33.8**
Volatile organic compounds (VOC)	[g/l]	**430**	**430**	**425**

We supply material for good ideas!

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