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Photograph: Detail from 'Bridge No. 2' from the series *Rust Never Sleeps*, John Moore, 1996

BETTER THAN PARALOID B-72? TESTING POLIGEN® WAXES AS COATINGS FOR METAL OBJECTS

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Abstract

This paper presents results from a comparative test of six products for coating metal objects. Poligen® ES 91009, Poligen® ES 91012, and Poligen® ES 91018 were tested and compared to Paraloid® B-72, Paraloid® B-44 and Cosmoloid® H80. Tests were undertaken on copper alloy and ferrous coupons, and ferrous nails, which were coated by brushing, immersion or impregnation. The coupons and objects were subjected to natural and accelerated aging under different climatic conditions (humidity and polluted atmospheres) and within museum galleries. Corrosion monitoring, as well as computer-assisted optical inspection techniques, were used to evaluate coating qualities. Results to date indicate that Poligen® ES 91009, Poligen® ES 91012, Paraloid® B-72 and Paraloid® B-44 offer comparable surface protection when applied to different ferrous metals, whereas Poligen® ES 91018 and Cosmoloid® H80 are inferior. On copper alloys, Paraloid® B-72, Paraloid® B-44 and Cosmoloid® H80 show better results than Poligen® ES 91018.

Keywords: protective coatings, iron, copper alloys, Poligen®, Paraloid®, Cosmoloid®

Introduction

The research project PROMET ('Protection of Metals', 2004-2008) tested different coating materials to find the best product for historical iron and copper alloy objects in museums around the Mediterranean. Seven promising coatings were tested on bare and corroded ferrous and copper alloy coupons, and on real objects (Argyropoulos et al 2007, Cano et al 2007, Siatou et al 2007, Argyropoulos 2008, Degrigny 2008). It was concluded that the recently developed polyethylene wax Poligen® ES 91009 is more suitable for protecting iron than traditional Paraloid® B-72.

The research project discussed in this paper was initiated to verify results from PROMET, and began in 2008. The study is a joint project between the State Academy of Art and Design in Stuttgart, Germany, and the Deutsches Bergbau-Museum Bochum in Bochum. Poligen® ES products from BASF are recently developed water dispersed polyethylene coating products for industrial application on metal, based on the copolymer wax BASF Luwax® EAS 5 (2004). Poligen® ES 91009 has caused a lot of interest in the conservation community because it is solvent-free and can be used without health risks. To confirm its suitability as a coating, Poligen® ES 91009 was tested with a different methodology to that used in the PROMET project (see Table 2, 3 and 4).

Other interesting products, Poligen® ES 91012 and Poligen® ES 91018, were included in this study, and compared to Paraloid® B72, Paraloid® B-44 and Cosmoloid® H80, commonly used in conservation. All coatings, besides Poligen® ES 91009 and Poligen® ES 91012, which cannot be used for copper alloys because of their ingredients, were tested on iron and copper alloy coupons and archaeological iron nails by accelerated aging.

In addition to testing the protective capabilities of these coatings, further technical information on the Poligen® ES products, including constituents, pH, volatile emissions and reversibility, are presented. This information is collected from technical data or is the result of analysis by FTIR, pH-test, Oddy-test or personal experience of the authors.

Coating materials

Poligen® ES

Polyethylene waxes are co-polymerized from polyethylene and methacrylic acid. According to the BASF data and the acid number, the ratio between methacrylic acid and polyethylene is 1: 8-9 (BASF

Luwax® EAS 5 2004, Csihony 2009). Water dispersibility is achieved by neutralizing the acid group with either an organic amine (Poligen® ES 91009), ammonia (Poligen® ES 91012) or potassium hydroxide (Poligen® ES 91018) (Schmidt 2007). The commercially available Poligen® products contain approximately 20%-25% copolymer and have a pH of approximately 8.5-9.5. These products can be diluted with water unless the pH drops below 8.5, in which case they will segregate. The minimum temperature at which film formation occurs is 10°C. Drying time at room temperature is over 24 hours, however, at 60°C products dry in one hour (BASF Lugalvan® FDC 2007).

Fourier transform infrared spectroscopy (FTIR) was carried out to analyze the ingredients of the Poligen® ES products to verify the composition. A variety of techniques were applied to ensure accuracy. Attenuated total reflectance (ATR) was performed in-situ on clean steel coupons ("DC04B"), coated with the products described. Further analysis was undertaken with FTIR in transmission mode; a) with pellets of a mixture of potassium bromide (KBr) and dried Poligen® ES, and b) on a Poligen® ES film (for methodology see Günzler and Böck 1983, Gottwald and Wachter 1997). All spectra established the presence of a polyethylene/methacrylic acid copolymer (see Figure 1). The neutralizing compounds could not be identified.

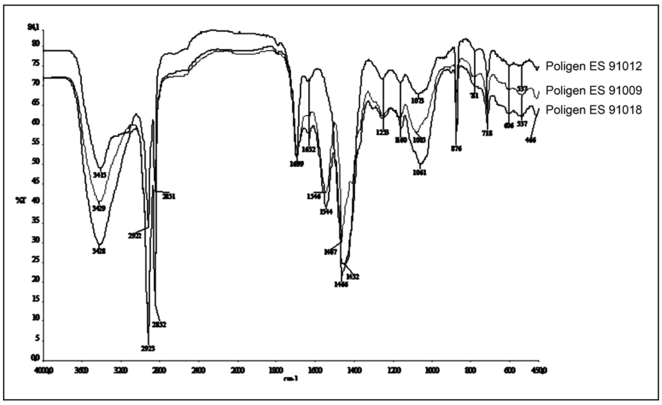

Figure 1. Poligen® IR spectra in transmission with KBr pellet.

The pH of all Poligen® ES films, measured according to the methodology of Down et al (1996), stayed slightly alkaline during testing for 74 days with only slight variation. The Oddy-test, performed according to the methodology described in Lee and Thickett (1996), showed reactions for volatile emissions from Poligen® ES 91009 with copper and from Poligen® ES 91018 with lead. With regard to reversibility, the coatings are only soluble in aqueous sodium hydroxide (20% w/w, pH 14, BASF Lugalvan® FDC 2007). The greatest solubility is given for Poligen® ES 91018 and the least for Poligen® ES 91012 (Csihony 2009).

The application of Poligen® ES coatings is often difficult as films tend to crack during drying if they are applied too thickly. For brush application, the quantity of material applied is easily controlled, but it is important to use a very fine synthetic brush, otherwise bubbles appear on the surface that do not disappear during drying. For application to rough surfaces, like archaeological iron, an application using small dabs of the brush is best.

For application by immersion, all Poligen® ES coatings must be diluted in water to a concentration of 75% (w/w), otherwise layers are too thick and cracks appear. Even then, when applied to rough surfaces, excess coating must be removed with a brush or swiped with a tissue paper after immersion to avoid a thick coating.

Other coating materials

Paraloid® B-72, Paraloid® B-44, and Cosmoloid® H80 are well described in the literature (Johnson 1984, Down et al 1996, Horie 2002, CAMEO 2009). For testing purposes, these Paraloid® formulations were applied as a 10% solution in ethyl acetate (w/w). In the PROMET study, these products were diluted to a 15% solution in acetone (w/v), but the authors observed that both Paraloids dried too fast and produced streaky coatings. Cosmoloid® H80 was used as a 10% dispersion (w/w) in the mineral spirit Shellsol® T, which is free of aromatic compounds but highly flammable above 60°C.

Testing Methodology

Specimens

For the aging tests, three coupons of each metal, coating material and application method were assessed. The coupons consisted of ferrous metal and copper alloys. The ferrous metals selected for the study were: uncorroded, low alloyed steel coupons 10 x 5 cm in size ("DC04B" with 0.08% carbon, 0.03%, phosphorus, 0.03% sulfur, 0.40% manganese); artificially corroded iron coupons 10 x 5 cm in size ("DC04B", prepared by defined accelerated weathering until the surface was completely covered with thick brown and orange corrosion layers); historic iron specimens from the preindustrial period before 1850, 3.5 x 3.5 cm in size with less than 0.5% carbon and a clean surface free of corrosion[1]; and Roman iron nails approximately 3-10 cm long with an iron core, one group desalinated in sodium hydroxide, the other group desalinated in alkaline sulphite. Copper coupons for the study consisted of un-corroded bronze 4 x 9 cm in size (8% tin,); brass 4 x 9 cm in size (20% zinc); and gunmetal 4.5 x 10.5 cm in size (7% tin, 4% zinc, and 7% lead). The number of specimens that could be included in this study was limited due to the size of the testing chambers.

Before coating, the artificially corroded iron coupons were sandblasted with aluminium oxide to remove loose corrosion layers, then all metal coupons were cleaned and degreased in acetone and mineral spirits in an ultrasonic bath, dried at 105°C and stored in a dessicator. The coupons were handled with latex or nitrile gloves at all times.

Coating techniques

Ferrous coupons were coated with each protective material described previously. The copper alloys coupons were only coated with Poligen® ES 91018, Paraloid® B-72, Paraloid® B-44 and Cosmoloid® H80 due to corrosion of copper with amine and ammonia, which are present in Poligen® ES 91009 and Poligen® ES 91012. All coupons were coated by brushing or immersed in baths. The archaeological nails were impregnated under vacuum. The nails desalinated in sodium hydroxide were only tested with the Poligen® ES products.

Film thickness measurements

For evaluation of the average film thickness, each coupon was weighed before and after coating. Thickness was roughly calculated by the given density of the products. To obtain more precise results, additional tests were carried out. Because of low film thickness of 1-2 µm, most methods for calculating thickness are too imprecise (as can be seen from DIN EN ISO 2808). Tests with a magnetic inductive system and tactile measurements failed; only microscopic analysis and reflection measurements showed precise results. As an example, Table 1 compares the average film thickness of coatings applied to new steel and bronze coupons, obtained from weight measurements, microscopic analysis and reflection measurements. To measure the average film thickness, three coupons were weighed. For the microscopic analysis, only four coupons could be measured, and for the reflection measurement, the wax Cosmoloid® H80 could not be evaluated because of its soft wax constitution. All results vary considerably. However, a thickness of 1-2 µm is an average thickness achieved for these films.

	Average mass difference (µm)		**Microscopic analysis (Confocal-Microscope)** (µm)		**Reflection measurement** (µm)
Material	**steel**	**bronze**	**steel**	**bronze**	**steel**
Po09	1.9	-	3	0.8	2.1
Po12	0.9	-	-	-	1.2
Po18	2.1	1.2	-	-	2.4
PB72	1.3	1.2	3	0.6	1
PB44	2.9	3.2	-	-	1.3
CH80	0.6	0.3	-	-	-

Table 1. Film thickness in µm of brushed coatings on bare metal coupons. (**Po09** = Poligen® ES 91009; **Po12** = Poligen® ES 91012; **Po18** = Poligen® ES 91018; **PB72** = Paraloid® B72; **PB44** = Paraloid® B44; **CH80** = Cosmoloid® H80)

Testing the coatings

The coupons were subjected to accelerated and natural aging in three different climatic chambers and in two different environments in the Deutsches Bergbau-Museum Bochum; (a) an exhibition hall, and b) an underground mine that is part of the museum. The terms of the tests are reported in Table 3. Accelerated aging was performed in chambers that simulated different environmental conditions: a) condensation effects: relative humidity (RH) ranging from > 100% RH (condensation period) to 35% RH (drying period); b) high humidity effects: relative humidity cycling between 95% RH and 35% RH, and c) a chamber that simulated an industrial environment with high levels of sulphur dioxide (SO_2). Chamber

Aging tests	**Tested specimens**
Alternating condensation atmosphere (16hrs. @ 35 °C, 90% RH +8hrs. @ 23 °C, 55% RH)	steel artificial corroded steel natural corroded steel copper gunmetal artificial corroded red brass natural corroded red brass
Museum gallery	natural corroded steel natural corroded red brass historical iron objects

Table 2. PROMET aging tests of the coatings (Siatou et al 2007: 3, Degriny et al 2007: 34, Degriny 2008: 182).

Aging tests	**Tested specimens**
Alternating condensation - dry atmosphere (8hrs. @ 40°C, 100% RH + 16hrs. @ 20 °C, 35%RH) (DIN 50017: 1982)	bare steel, corroded steel historic iron bare bronze and brass (brushed and immersed)
Varying humidity atmosphere (8hrs. @ 40°C, 95% RH + 16hrs. @ 20 °C, 35% RH)	bare steel corroded steel archaeological nails bare gunmetal (brushed and immersed)
SO_2-climate (acid rain simulation) (4 min. wetting/ 1 min. stop/ 3 min. wetting/ 12 min. SO_2-exposure (4 mg/m^3)/ 15 min. of warming till 60 °C/ 30 min. cooling till 20 °C)	corroded steel (brushed and immersed)
Museum gallery with 22-27 °C and 30-60 % RH	bare steel and corroded steel (brushed only)
Mining gallery with 13-14 °C and 60-100 % RH	bare steel and corroded steel, (brushed only)
Airproof chamber with silica gel	Each coating and metal with 1 specimen

Table 3. Authors' aging tests of the coatings.

parameters and aging conditions were adopted from several former research studies dealing with the efficacy of transparent coating materials[2].

Two uncoated specimens of each metal were kept as a reference in the aging tests. Furthermore, one coated and uncoated specimen for each metal and type of coating was stored in a desiccator (room temperature and under 30% RH). Tests with cycling condensation humidity and temperature ran for 77 days, and the SO_2 test for approximately 68 days (1500 cycles of 65 min each). Natural aging was performed for 12 months and was continued after a first inspection, because the coatings did not show enough differences. This test is still ongoing at the time of writing.

For documentation purposes, all coupons were scanned by a flat bed scanner with high definition[3] before, during and after the aging tests. The archaeological nails were photographed and all specimens were documented in writing.

Evaluation of results

Three different methodologies were applied to evaluate the results of the aging tests.

The appearance of corrosion products over time was monitored as an indicator of layer protectiveness. The length of time that elapsed until the surface of the iron or copper alloy coupon was corroded was recorded. This point was defined as having been reached when approximately 90% of the surface was covered by corrosion products. In some cases, a coupon was entirely corroded after 20 days, while others showed little corrosion over the same time period, and were still not fully corroded after 40 days.

Computer-assisted visual evaluation of data was carried out with the application of an image analysis system[4]. Freshly formed corrosion was defined by its particular color on images of scanned coupons, and the image analysis system was then used to calculate the percentage of fresh corrosion on the surface. Finally, visual monitoring (performed by one person to ensure consistency) was carried out for all samples. All these evaluations were then rated according to their performance (see Table 4 and 5). SEM did not provide applicable results, because the coating was not visible.

Results

Good surface protection was achieved for brush application of Poligen® ES 91009, Poligen® ES 91012, Paraloid® B-72 and Paraloid® B-44 onto a ferrous surface (see Table 5 and Figure 2 and 3) with a few exceptions. One specimen of corroded steel coated with Poligen® ES 91009 failed in the SO_2 test due to intensive formation of new corrosion products, and one specimen of bare steel coated with Paraloid® B-44 failed in the condensation test. Poligen® ES 91018 and Cosmoloid® H80 failed twice and Cosmoloid® H80 performed well only once (see Table 5). Uncoated reference specimens performed better than expected, because not much more corrosion formed on the reference coupons than on the coated specimens.

If applied by immersion, the ferrous coupons with Poligen® ES 91009 and Poligen® ES 91012 performed quite well, while those immersed in Paraloid® B-72

PROMET evaluation methods	Authors' methods	Advantage / Disadvantage
Visual inspection, macro and micro	Same	Recording and rating effects / subjective
Weight change	Only for measuring the film thickness before aging	Reason for weight change during aging still not finally clarified (preliminary interpretation: weight loss because of loss of corrosion products, weight increase because of water absorption by the coating; both effects will appear at the same time)
Film thickness measurements	-	Imprecise, coatings too thin to measure correctly with applied techniques
Colorimeter	-	Can show changes within the coating / influence on protection abilities?
SEM	Same	Details like cracks in coating, filiform corrosion, etc. observable / coating not visible
Electrochemical with polarisationresistance (Rp) and Impedancespectroscopy (EIS)	-	Results difficult to interpret
-	Time monitoring of corrosion	Which coating does fail first, which last / quite rough results
-	Image analysis	Percentage of new corrosion can be determined / on precorroded surface difficult because of similar corrosion colors

Table 4 . Evaluations methods used by PROMET (Siatou et al 2007: 3 f., Degriny et al 2007: 34, Argyropoulos et al 2007: 11 f., Cano et al 2007: 122) compared to the authors' methods.

and Paraloid® B-44 showed good or satisfactory results (see Table 5 and Figure 4). Poligen® ES 91018 and Cosmoloid® H80 performed satisfactory but failed on bare steel. Cosmoloid® H80 gave a very good result when it was applied to corroded steel in the humidity test (see Table 5).

The archaeological iron nails desalinated with sodium hydroxide showed the best results when coated with Poligen® ES 91009. Those desalinated with alkaline-sulphite had good results with Paraloid® B72, and satisfactory with all other coatings besides Poligen® ES 91018 and Cosmoloid® H80 (see Table 5).

Figure 2. Bare steel coupons with coatings applied by brush; Coupon A, coated with Poligen ES® 91009, shows less corrosion (*left*); Coupon B, coated with Poligen ES® 91018, shows much more corrosion (*right*).

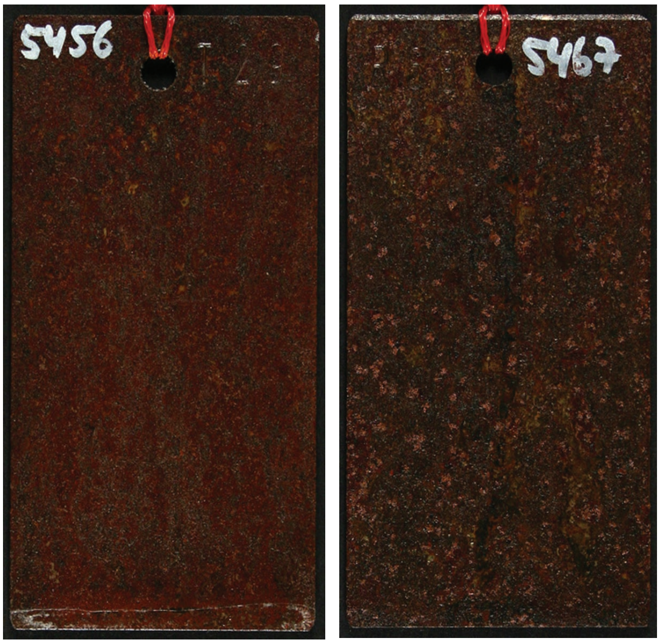

Figure 3. Corroded steel coupons with coatings applied by immersion: Coupon A, coated with Poligen ES® 91012, shows less corrosion (*left*); Coupon B, coated with Paraloid® B-44, shows much more corrosion (*right*).

Suitability	**Bare steel** (humid-test)	**Bare steel** (condensation -test)	**Corroded steel** (humid-test)	**Corroded steel** (so_2-test)	**Corroded steel** (condensation-test)	**Historic Iron** (condensation -test)	**Arch. Nails** (humid-test)
Brushed							**NaOH**
Very good						Po09	
Good	PB72, Po09, Po12, PB44	Po 12, PB 72	Po12, CH80, Po09, PB72		Po 09, Po 12	Po12, PB44, PB72	Po09
Satisfactory	without coating	Po 09, CH 80	PB44, Po18, without coating	Po12, Po18, PB72, PB44, CH80	Po 18, PB 72, PB 44, without coating	CH80, without coating	Po12, Po18
Failed	Po18, CH80	Po 18, PB 44, without coating		Po09, without coating	CH 80	Po18	without coating
Immersed							**Alkali-Sulphite**
Very good			Po09, Po12, CH80	Po09			
Good	Po09, PB72, PB44, Po12		PB44	Po12, Po18			PB72
Satisfactory	CH80		PB72, Po18	PB72, PB44			Po09, Po12, PB44, without coating
Failed	Po18			CH80, without coating			Po18, CH80

Table 5: Evaluation of the protectiveness of coatings on ferrous materials.

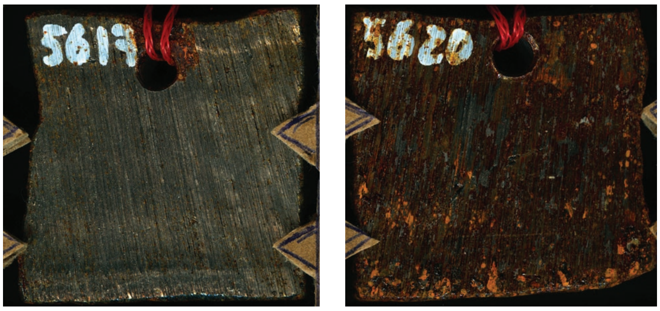

Figure 4. Historic iron coupons with coatings applied by brush; Coupon A, coated with Poligen® ES 91012, shows less corrosion (*left*); Coupon B, coated with Poligen ES® 91018, shows much more corrosion (*right*).

Suitability	Bronze (condensation -test)	Brass (condensation -test)	Gunmetal (humid-test)
Brushed			
Very good	PB44	PB44, PB72	CH80, without coating
Good	PB72, Po18	Po18	PB72, PB44
Satisfactory	CH80, without coating	CH80, without coating	
Failed			Po18
Immersed			
Very good			
Good	PB44	PB72	PB44, PB72, CH80
Satisfactory	CH80, PB72	CH80, PB44	
Failed	Po18	Po18	Po18

Table 6. Evaluation of the protectiveness of coatings on copper alloys.

Figure 5. Brass coupons coated by immersion: Coupon A, coated with Paraloid® B-72, shows less corrosion (*left*); Coupon B, coated with Poligen ES® 91018, shows much more corrosion (*right*).

On copper alloys, Paraloid® B-72 and Paraloid® B-44 performed comparatively well (see Table 6 and Figure 5). Poligen® ES 91018 behaved fairly well when brushed on bronze and brass, but otherwise it failed. The results of Cosmoloid® H80 were mostly just 'satisfactory'.

Conclusion

In this study, Poligen® ES 91009, Poligen® ES 91012, Paraloid® B-72 and Paraloid® B-44 applied to different ferrous metal coupons offered good protection of the metal surface during the accelerated aging tests. Poligen® ES 91018 and Cosmoloid® H80 displayed less ability to protect iron. On copper alloys, Poligen® ES is not suitable because of its basic ingredients of amine and ammonia, which corrode copper and its lack of reversibility. The coatings were soluble in sodium hydroxide, however, sodium hydroxide is corrosive to copper. Paraloid® B-72 and Paraloid® B-44 both performed well, however, Cosmoloid® H80 only performed satisfactorily.

For iron coupons, results indicate that both Poligen® ES 91009 and Paraloid® B-72 offer comparably good protection of iron. This result is in contrast to that determined in the PROMET project, which found that Poligen® ES 91009 provided better protection of iron than Paraloid® B-72. The result obtained here would seem to suggest there is no need to replace Paraloid® B-72 as a coating for iron objects, especially with regard to its well-proven reversibility and familiarity as a conservation material. At the time of writing, the long-term tests in the galleries of the Deutsches Bergbau-Museum Bochum are ongoing and results are not yet available. Results from PROMET's long-term testing of coatings on historic metal objects housed in museum conditions showed similar results for Poligen ES® 91009 and Paraloid® B-72 in Athens, Greece, whereas in Malta with abundant salt aerosols, Poligen ES® 91009 performed better than Paraloid® B-72. The comparison between the long-term effectiveness of Poligen® ES and Paraloid® B-72 is still to be determined and requires further testing.

Further research on the reversibility of Poligen® ES products is needed before these products can be recommended for routine application. Based on the results of this study, Poligen® ES 91009 and Poligen® ES 91012 should only be recommended for non-composite ferrous objects, that is, those with no organic or copper alloy component. Archaeological iron, which still contains chloride, will corrode during the application of Poligen® ES because of its water content, so it is not suitable for iron objects that have not been desalinated. A further disadvantage of Poligen® ES is the difficulties of handling during application, which can cause the appearance of bubbles and cracks in the coating.

Acknowledgements

We would like to thank BASF, colleagues at the Deutsches Bergbau-Museum Bochum, Wieland Werke, Landesdenkmalamt Baden-Württemberg, and Michael Stratmann for support and for providing the metal specimens in this study.

Endnotes

[1] The historic iron was from square bar steel, taken from an historic Gate. For the test it was cut into small slices where the bare cut side was the tested area.

[2] See for example, a German project financed by the Deutsche Bundesstiftung Umwelt 'Transparenter Korrosionsschutz für Industriedenkmaeler aus Eisen und Stahl' and the European project 'Comparison of conservation materials and strategies for sustainable exploitation of immovable industrial heritage made of iron and steel (CONSIST)'

[3] 48 Bit HDR colour, 1200 dpi, tif-data

[4] Picture software from Olympus, usually used for microscopic picture evaluation.

Materials

Poligen® ES 91009, Poligen® ES 91012 and Poligen® ES 91018:
BTC Speciality Chemical Distribution GmbH (BASF)
Maarweg 163 / 165
D-50825 Köln, Germany
Tel: ++49-221-954640

Paraloid® B-72, Paraloid® B-44, and Cosmoloid® H80; acetone, ethyl acetate, Shellsol® T, mineral spirits:
Kremer Pigmente GmbH & Co. KG
Hauptstr. 41 – 47
D-88317 Aichstetten, Germany
Tel: ++49-7565-91120

Sodium hydroxide:
Carl Roth GmbH + Co. KG
Schoemperlenstr. 3-5
D-76185 Karlsruhe, Germany
Tel: ++49-721-56060

DC 04 B steel coupons:
Franz Krüppel GmbH & Co.KG
Höffgashofweg 17-19
D-47607 Krefeld, Germany
Tel: ++49-2151-316070

Historic iron:
Schloss Borbeck, Essen
Michael Stratmann
Nierenhofer Str. 10 a
D-45257 Essen, Germany
Tel: ++49-201-8486173

Archaeological nails:
Regierungspräsidium Stuttgart, Landesamt für Denkmalpflege
Berliner Str. 12
D-73728 Esslingen a.N.
Tel: ++49-711-90445109

Bronze and brass:
Wieland Werke
Graf-Arco-Str. 36
D-89079 Ulm, Germany
Tel: ++49-731-9440

Gunmetal:
ALLMESON GmbH
Ottostr. 9-11
D-63150 Heusenstamm, Germany
Tel: ++49-6104-405980

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