METALS

Protection of organic remains in alkaline iron desalination

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ABSTRACT

The department for conservation of archaeological objects at the Swiss National Museum often faces the challenge of conserving and desalinating archaeological iron finds connected with organic remains. Due to the high pH of the desalination solutions, organic remains must be protected throughout the process. In order to verify and establish either existing or new protection methods, a series of tests were performed. Samples of suitable consolidants were treated in an alkaline desalination bath with common parameters. Afterwards, the resistance and permeability of the films were analyzed visually with FTIR, Oddy tests, and pH measurements. None of the protective films proved to remain impermeable in the alkaline solution throughout the whole desalination process but an additional cyclododecane layer extended the time of protection significantly.

INTRODUCTION

The department for conservation of archaeological objects at the Swiss National Museum (SNM) frequently encounters organic remains of clothing, sword sheaths, bags, belts, and ribbons preserved in connection with archaeological iron. These remains made of textile, leather, fur, feathers, and other rarely preserved materials offer extraordinary archaeological information (Rast-Eicher 2016). In most cases, they cannot be separated from the iron as they are preserved, in various stages of mineralization, within the adhering corrosion products. As a general practice, the SNM's department of archaeological conservation desalinates all iron finds to reduce the risk of active corrosion during long-term storage. However, due to the high pH of the desalination solutions, organic remains must be protected throughout the process.

BACKGROUND

The mineralization process for organic remains is a complex and heterogeneous phenomenon (Gillard et al. 1994). The form and degree of mineralization varies for each object, and it is therefore assumed that non-mineralized organic structures are likely to be present within mineralized remains (Janaway 1989, Keepax 1989). It is also known that these structures are susceptible to alkaline hydrolysis (Florian 1987, Selwyn et al. 1993). As a result, due to the high pH of desalination baths, it is necessary to protect organic remains during this treatment phase. All organic remains in contact with metals should also be documented by a specialist before desalination (Rast-Eicher 2016).

Desalination in heated alkaline sulfite has been developed and used at the SNM for more than 22 years (Schmidt-Ott and Oswald 2006). Experience has shown that Paraloid B-44 seems to resist alkaline solutions and temperatures up to 50°C and it has consequently been used at the SNM to protect organic remains during desalination. There are, however, limitations to this method. A significant issue when using Paraloid B-44 is that it can only be applied on dry surfaces. Since it is not possible to dry only part of an object, the whole artifact must be dried before desalination, which could result in damaging both the organics and the metal (Guilminot et al. 2012). Another drawback is that it is difficult to apply a thick layer of Paraloid B-44 without air inclusions and then remove excessive Paraloid

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Figure 1. Zoom of a sword sheath made of leather: (top) before desalination; (middle) surface consolidated with Paraloid B-44 and cyclododecane; (bottom) surface after desalination and mechanical cleaning

off organic remains after desalination. For this reason, an additional layer of cyclododecane was added on top of the Paraloid B-44 as part of the SNM protocol. The cyclododecane provides further protection and reduces the thickness of the Paraloid B-44 layer. However, the low sublimation temperature of cyclododecane prevents the baths from being heated. This research aims either to confirm that Paraloid B-44 and cyclododecane are the best way to protect organic remains during desalination, or to find the most appropriate alternative. A water-based consolidant would be ideal as it would make it possible to consolidate the organic remains prior to the drying phase (Figure 1).

PROCEDURE

Choice of consolidants

To achieve the maximum protection of organic remains, the products applied have to provide thorough consolidation of the fibers. The chosen consolidant is not likely to be removed after treatment to prevent crumbling of the organic remains. As a result, it is not necessary for the chosen consolidants to be completely reversible, although this is an ideal that should be strived for within conservation (ECCO 2003). Consequently, the selected products have to be chemically inert, provide long-term stability and be free of harmful substances. They should also be readily available, easy to apply, and non-acidic to prevent further corrosion of the metal. A high glass-transition temperature is preferable as it is best to heat baths during desalination (Schmidt-Ott and Oswald 2006). Following these criteria, a literature review was conducted (Down 2009 and 2015, Horie 2010) to narrow down the list of suitable products for further testing. Table 1 presents the list of eight products originally chosen for these trials. It was decided that the three Paraloid acrylic resins would be tested while diluted in acetone, in methyl ethyl ketone (MEK) or in toluene. The SNM purchases solvents with similar levels of purity from three main providers: Merck, Roth, and Sigma. Acetone, MEK, and toluene from each provider were tested within these trials to identify possible differences between the solvents.

Trade name	Composition		
Paraloid B-44	methyl methacrylate copolymer		
Paraloid B-67	isobutyl methacrylate polymer		
Paraloid B-72	ethyl methacrylate polymer		
Dispersion K 9	pure acrylic dispersion		
Primal AC 35	acrylic latex		
Plextol B 500	aqueous dispersion based on methyl methacrylate and ethyl acrylate		
Plextol D 498	aqueous dispersion based on methyl methacrylate and ethyl acrylate		
Lascaux 498 HV	aqueous dispersion with methyl methacrylate and butyl acrylate		

Table 1		List	of	tested	products
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pH measurement and Oddy test

In order to confirm that the products listed in Table 1 and the selected solvents met our criteria, pH measurements were taken and Oddy tests were conducted. This combination of tests is standard at the SNM to

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determine if products intended for direct or close contact with the artifact are inert, as well as to assess their long-term stability.

pH measurements were sometimes complex to achieve due to the fact that some of the products and solvents are not soluble in water. This section discusses the protocols used to get around this difficulty and measure pH. All measurements were done with an 827 pH lab Fa. Metrohm pH-meter with a pH electrode 6.0224.100 (Biotrode) Fa. Metrohm. The samples were measured at $21^{\circ}C \pm 1^{\circ}C$. The pH meter and pH electrode were calibrated with two buffers (pH 4.00 and 7.0) each day that measurements were taken.

The Paraloid pH was tested in two ways. First, the Paraloid pellets were tested as follows: two grams of pellets were stirred with 50 ml of ultrapure water (UPW¹) for 24 hours. After 24 hours, the pH of these solutions was measured and compared to the pH of the UPW. In parallel, 20% w/w solutions of Paraloid in each solvent cited above were prepared, applied with a brush on glass, and allowed to dry creating "dry films." Two grams of dry films were then scraped away and stirred with 50 ml of UPW for 24 hours. The pH of these solutions was then measured and compared to the pH of the UPW.

The pH of the acrylic dispersions was also measured in two ways: in aqueous solutions and as "dry films." For the aqueous solutions, the dispersions were diluted in UPW to a 10% solution (w/w). The pH of these solutions was measured and compared to the pH of the UPW. The pH measurements for the dispersion films were performed using the same protocol as the one for the Paraloid dry films.

Regarding the solvents, all of them, except toluene, were diluted to a 10% aqueous solution (w/w) with pH-adjusted UPW (as UPW is slightly acidic, its pH was raised to 7 with the addition of a 0.001N NaOH solution). The pH of these solutions was measured and compared to the pH-adjusted UPW.

The following procedure was used for toluene as it is insoluble in water: 5 ml of toluene was stirred with 50 ml of pH-adjusted UPW for 24 hours; the pH of these "solutions" was measured, after 24 hours, and compared to the pH of the adjusted UPW.

In 1973, the Oddy test was developed at the British Museum in order to indicate the presence of volatile harmful compounds under artificial aging conditions for materials around museum objects (Oddy 1973). At the SNM, a variation of the optimized "three-in-one" protocol from Robinett and Thickett (2003) is followed. Oddy tests on all the products listed in Table 1 as well as all the solvents previously mentioned were performed and the results were used to classify each as either of "permanent use," "temporary use" or "unsuitable for use" (Lee and Thickett 1996).

Acrylate "dry films" were tested after drying for one month to detect potential remains of harmful chemicals. Since solvents can contain substances that may cause corrosion of materials, they too were tested separately. For this purpose, 1-ml samples of acetone, MEK, and toluene were reduced by evaporation to 0.1 ml and then submitted to an Oddy test.

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Figure 2. Selection of products and prepared glass slides



Figure 3. Paraloid B-72 in acetone samples on glass slides: the lower row is covered with cyclododecane

Behavior of the chosen consolidants in alkaline solution

Of the 19 solutions originally selected (i.e., products listed in Table 1 and combinations of Paraloid with solvents), only six were considered inert and stable after pH measurements and Oddy tests. They are presented in the results section. These six products were further evaluated in alkaline solution as described in this section (Figure 2).

Solutions of these six consolidants (20% w/w for Paraloid and 10% w/w for acrylic dispersions) were applied to glass slides. Before applying the solutions, one side of each slide was sanded with an air abrasive apparatus to create a more adherent surface. For each of these six products, a group of 10 slides was prepared (60 samples in total). From these 10 samples per group, five were just covered with the consolidant and five with the consolidant plus an additional coating of cyclododecane. For each subgroup of five samples, two were used as controls and three were tested in alkaline solutions. Once applied on both sides of the slides, the samples were allowed to dry for one month (Figure 3).

For each group of three samples, a bath of 200 ml of concentrated alkaline sulfite solution (20 g/l NaOH and 63 g/l Na₂SO₃) was prepared (Schmidt-Ott and Oswald 2006). The baths were not heated because of the low glass temperature transition of some of the consolidants and because of the sublimation temperature of cyclododecane. The duration of the baths was 12 weeks with the solution being renewed every three weeks. This reproduced the average duration and methodology of a desalination treatment at the SNM. After treatment, the samples were rinsed with UPW until a neutral pH was reached and then dried in a vacuum-oven, also according to the SNM protocol. The cyclododecane evaporated during the drying process.

The samples were photographed before and after the bath and changes such as the formation of cracks, detachment, and whitening were documented.

The surface pH of the samples after desalination was measured with a flat-membrane electrode (827 pH lab Fa. Metrohm pH meter and a pH electrode 6.0256.100). Three drops of UPW were placed on the surface of the samples and the pH was measured.

In addition, two grams of the scraped "dry films" (see preparation in previous section) were treated for 12 weeks in the alkaline solution so that later Oddy tests on the six consolidants could be conducted after desalination.

Lastly, the surfaces of the samples located on glass slides were examined with Fourier transform infrared (FTIR) spectroscopy (FTS 3500 GX Excalibur Series with a UMA-500 microscope). Analyses were also performed before and after desalination in order to identify any changes in the molecular structure or composition of the consolidants after treatment.

Efficiency of the protection layers

After testing the behavior of the six products in alkaline solution (see previous section), the consolidant options for organic remains during desalination treatment were narrowed down to only five solutions. New tests were set up with these five options to assess their impermeability in alkaline solution baths.

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Figure 4. Paraloid B-72 in acetone samples on carbon steel with pH indicator strip: the lower row is covered with cyclododecane

Strips of pH paper were placed on modern and naturally corroded carbon steel coupons and then covered with the chosen products. For each consolidant, six samples were prepared and half of them were covered with cyclododecane after drying for one month (Figure 4).

The procedure for the alkaline sulfite bath was the same as the one described in the previous section.

Contact of the pH paper with the alkaline solution is indicated by discoloration. Therefore, during the bath, the samples were examined regularly and photos were taken. After 12 weeks, the samples were rinsed and dried as described in the previous section. Visual changes after the bath, such as detachment and whitening, were photographed.

RESULTS

pH measurement and Oddy test

The pH measurements are summarized in Table 2. The pH of a material in contact with organic remains should lie in the range of 5.5 to 8.5 (Lee and Thickett 1996). The tolerance range therefore lies within \pm 1.5 of a neutral pH.

The pH of pure Paraloid B-44 and B-67 pellets is close to neutral. Paraloid B-72 had an acidic pH. The dried films of the 20% w/w Paraloid solutions also provided a pH value close to neutral. This renders the three Paraloid samples suitable to be in contact with organic remains.

All pH values for the dispersion solutions slightly exceeded the tolerance range and tested alkaline. The pH of the Primal AC 35 dry film was considered too alkaline and excluded from further testing. Plextol B 500, Lascaux 498 HV, and Dispersion K 9 also tested alkaline, but less than Primal AC 35. The dry film of Plextol D 498 tested acidic.

All Oddy test results are summarized in Table 3. Regarding the Paraloid samples, the tests performed on pure pellets showed that Paraloid B-44 and B-72 tested suitable for permanent use. Paraloid B-67 tested suitable for temporary use. The Oddy tests on the solvents showed that MEK is unsuitable for use. Of the Paraloid samples, the ones diluted in MEK also tested unsuitable for use, confirming this result. The solutions made with acetone and toluene tested suitable for permanent use. This outcome, in combination with the pH results, demonstrates that Paraloid B-44 and B-72 are suitable for use on organic remains with either acetone or toluene, but not with MEK.

Regarding the dispersions, Dispersion K 9 and Primal AC 35 tested suitable for permanent use. Lascaux 498 HV tested suitable for temporary use. Plextol B 500 and Plextol D 498 tested unsuitable for use. This result, combined with the pH measurements, narrows down the suitable dispersions to Lascaux 498 HV and Dispersion K 9.

Behavior in alkaline solution

In light of the pH and Oddy test results, only the following six options were tested in alkaline baths: Paraloid B-44 (20% w/w in acetone and toluene), Paraloid B-72 (20% w/w in acetone and toluene), Dispersion K 9, and Lascaux 498 HV.

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Figure 5. Deformation, thin cracks, and partial loss of adhesion observed on the consolidation film made with Lascaux 498 HV

Compared to the other consolidants, the film produced with Lascaux 498 HV showed clear deformations and the formation of cracks (Figure 5). Also, all films covered with cyclododecane turned opaque. Observation under the microscope showed that the protection layer of Paraloid B-72 in combination with cyclododecane developed fine cracks on the surface.

The pH measured on the surface of the films before and after desalination showed no significant changes on the samples without cyclododecane. The samples covered with cyclododecane were more alkaline after desalination. As these samples were rinsed before the removal of the cyclododecane layer, they were rinsed again and the pH measurement was repeated after the cyclododecane was removed. The values were then lower and closer to the initial pH. It seems that the additional cyclododecane layer rendered the alkaline solution more difficult to rinse.

Table 2. Results of pH measurements

Sample	pH value compared to the water used
	+/- 0.10: neutral +/- 0.11 - 0.50: slightly acid/alkaline +/- > 0.50: acid/alkaline
<u> </u>	Solvents
Acetone (Merck)	-0.18 (slightly acid)
Acetone (Roth)	-0.05 (neutral)
MEK (Merck)	-2.26 (acid)
MEK (Roth)	-2.29 (acid)
Toluene (Roth) Toluene (Fluka)	+0.20 (slightly alkaline) +0.19 (slightly alkaline)
Para	aloid pellets
Paraloid B-44 pellets	-0.10 (neutral)
Paraloid B-67 pellets	+0.09 (neutral)
Paraloid B 72 pellets	-0.79 (acid)
Paraloid films di	ried from 20% solutions
Paraloid B-44 in acetone (Roth)	+0.31 (slightly alkaline)
Paraloid B-44 in MEK (Sigma)	+0.26 (slightly alkaline)
Paraloid B-44 in MEK (Merck)	+0.03 (neutral)
Paraloid B-44 in toluene (Roth)	+0.12 (slightly alkaline)
Paraloid B-44 in toluene (Sigma)	+0.01 (neutral)
Paraloid B-67 in MEK (Sigma)	+0.79 (alkaline)
Paraloid B-72 in acetone (Merck)	+0.17 (slightly alkaline)
Paraloid B-72 in acetone (Roth)	+0.07 (neutral)
Paraloid B-72 in MEK (Sigma)	+0.62 (alkaline)
Paraloid B-72 in MEK (Merck)	+0.03 (neutral)
Paraloid B-72 in toluene (Roth)	+0.06 (neutral)
Paraloid B-72 in toluene (Sigma)	+0.06 (neutral)
Acrylic dispersions: aqueou	us solution compared to dried film
Dispersion K 9, aqueous solution	+2.88 (alkaline)
Dispersion K 9, dry film	+0.88 (slightly alkaline)
Primal AC 35, aqueous solution	+2.28 (alkaline)
Primal AC 35, dry film	+.2.47 (alkaline)
Plextol B 500, aqueous solution	+2.97 (alkaline)
Plextol B 500, dry film	+1.16 (slightly alkaline)
Plextol D 498, aqueous solution	+2.52 (alkaline)
Plextol D 498, dry film	-0.65 (slightly acid)
Lascaux 498 HV, aqueous solution	+2.32 (alkaline)
Lascaux 498 HV, dry film	+1.15 (slightly alkaline)

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The results of the Oddy tests for all of the selected consolidants showed that they were suitable for permanent use after the desalination bath (Table 3).

The FTIR examination showed that the chain lengths of Lascaux 498 HV molecules were slightly shortened after the baths, but no significant changes could be observed for any of the other consolidants (Figure 6).

Efficiency of the protective layers

During the first week in the desalination bath, all the pH indicator strips located on the samples without cyclododecane changed color, meaning that the alkaline solution had penetrated the protection layer.

For the samples with cyclododecane, the pH indicator coated with the Paraloid B-44 in acetone samples presented slight discoloration after the first week and was completely discolored after two weeks. The indicator strips coated with Paraloid B-44 in toluene were largely discolored after the first week and completely discolored after two weeks. The pH indicator

Sample	Result	
Pure solvents		
Acetone (Merck)	Т	
Acetone (Roth)	Т	
MEK (Sigma)	U	
MEK (Merck)	U	
MEK (Roth)	U	
Toluene (Sigma)	Т	
Toluene (Roth)	Т	
Selected consolidation media for test ser	ies	
Paraloid B-44 pellets	Р	
Paraloid B-44 in acetone (Merck, Roth)	Р	
Paraloid B-44 in acetone (Roth), after desalination		
Paraloid B-44 in toluene (Merck, Roth)		
Paraloid B-44 in toluene (Roth), after desalination		
Paraloid B 72 pellets		
Paraloid B-72 in acetone (Merck, Roth)		
Paraloid B-72 in acetone (Roth), after desalination	Р	
Paraloid B-72 in toluene (Merck, Roth)	Р	
Paraloid B-72 in toluene (Roth), after desalination	Р	
Dispersion K 9		
Dispersion K 9, after desalination		
Lascaux 498 HV		
Lascaux 498 HV, after desalination		
Discarded consolidation media, not tested in desa	lination bath	
Paraloid B-67 pellets	Т	
Paraloid B-67 in MEK (Sigma, Merck)	U	
Paraloid B-44 in MEK (Sigma, Merck)	U	
Paraloid B-72 in MEK (Sigma, Merck)	U	
Primal AC 35		
Plextol B 500	U	
Plextol D 498		

Table 3. Results of Oddy tests

T = temporary use

U = unsuitable for use

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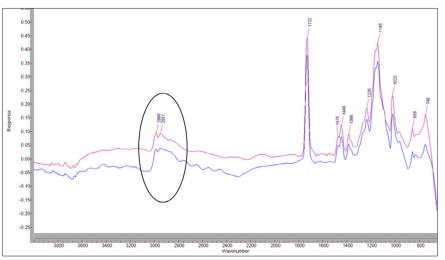


Figure 6. FTIR analyses of Paraloid B-72 before (blue) and after (magenta) soaking in concentrated alkaline solution for 12 weeks showing no significant changes in the molecular chain length

strips related to the Paraloid B-72 in acetone samples showed initial discoloration after the first week and were completely discolored after six weeks. The pH strips coated with Paraloid B-72 in toluene also showed color changes after the first weeks. After 12 weeks, one out of three pH indicators coated with the Paraloid B-72 was still unchanged. Dispersion K 9 achieved the best results. The pH indicator strips also showed some discoloration after the first week but only about 50% of the surface was discolored at the end of the desalination bath (Table 4).

Table 4.	Duration of impermeability in alkaline desalination
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Sample	First signs of discoloration	Complete discoloration
	No additional cyclododecane la	yer
Paraloid B-44 in acetone (Roth)	During first 2 days	During first week
Paraloid B-44 in toluene (Roth)	During first 2 days	During first week
Paraloid B-72 in acetone (Roth)	During first 2 days	During first week
Paraloid B-72 in toluene (Roth)	During first 2 days	During first week
Dispersion K 9	During first 2 days	During first week
	With additional cyclododecane la	ayer
Paraloid B-44 in acetone (Roth)	During first week	During second week
Paraloid B-44 in toluene (Roth)	During first week	During second week
Paraloid B-72 in acetone (Roth)	During first week	After 5 weeks
Paraloid B-72 in toluene (Roth)	During first week	After 12 weeks about 70% of surfac
Dispersion K 9	During first week	After 12 weeks about 50% of surfac

After drying the samples, their physical appearance was examined visually. All the samples with Paraloid B-44 and B-72 showed a slight detachment of the layers from the carbon steel coupons, whereas Dispersion K 9 showed no such detachment. Again, Paraloid B-72 covered with cyclododecane showed fine cracks at the surface and the consolidant film turned opaque.

DISCUSSION

The pH values and Oddy tests revealed that the solvents used have an important impact on whether a chemical is inert or not. This finding was not expected before the study but is certainly of interest to the conservation community.

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The addition of a cyclododecane layer on top of the other product gave the solution higher impermeability. One has to keep in mind that rinsing these samples was more time consuming than rinsing samples without cyclododecane. Considering that all films covered with cyclododecane turned opaque, there seems to be an interaction between the cyclododecane and the consolidant. However, FTIR results revealed no significant changes and further testing is required. One should also remember that the Paraloid B-72 film in toluene with cyclododecane protection showed fine surface cracks.

The use of the concentrated solution of alkaline sulfite was intentional as it is the most aggressive desalination solution used in conservation laboratories. It would be interesting to perform similar tests with lower concentrations as it has been demonstrated that they are efficient (Schmidt-Ott and Oswald 2006).

The baths were not heated due to the low glass temperature transition of some of the products and because of the low melting point of cyclododecane.

For the application of the protective layers on top of the pH indicator strips, it would be interesting to test different thicknesses of layers. It would also be interesting to verify the impermeability of the protection layers with actual samples of organic materials.

CONCLUSION

Complete protection of organic remains in alkaline solutions could not be obtained during these trials. Nonetheless, several acrylic resins used in conservation proved to be resistant to a 12-week treatment in alkaline solution. Dispersion K 9 showed the best results of all the products tested. As an aqueous dispersion, it offers new possibilities, especially to consolidate wet and humid finds. Paraloid B-72 in toluene showed the highest level of impermeability of the solvent-based products. The tests revealed that the cyclododecane layers improve protection during desalination. Also, the choice of solvent has an impact on both the chemical stability and the permeability of the protection layer.

These tests have shown that the current method used at the SNM of protecting organics during desalination can be further optimized. Currently, the consolidation of organic remains is performed with a combination of Paraloid B-44 and cyclododecane. The combination of Dispersion K 9 and cyclododecane will be tested soon on actual artifacts. Although the visual appearance of Paraloid B-72 in toluene in combination with cyclododecane was not entirely satisfying, further testing on artifacts should be conducted as it still showed better impermeability than the Paraloid B-44 currently used.

The general practice of desalinating archaeological iron finds in connection with organic remains has to be refined until a reliable solution has been found. Due to the complexity of the parameters, additional testing is of great interest. Further product options should also be considered outside the conservation field.

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NOTES

¹ The SNM uses an RO/DI water purification system to produce ultrapure water.

MATERIALS LIST

Acetone, EMSURE® Reag. pH Eur Merck KGaA www.merckgroup.com

Acetone, 99.5% for synthesis Carl Roth GmbH & Co. KG www.carlroth.com

Dispersion K 9 (pure acrylic dispersion) Kremer Pigmente GmbH & Co KG www.kremer-pigmente.de

Lascaux Acrylkleber 498 HV (aqueous dispersion with methyl methacrylate and butyl acrylate) Lascaux Colours & Restauro www.lascaux.ch

Paraloid B-44 (methyl methacrylate copolymer) The Dow Chemical Company, distributed by Rhom and Haas Europe Trading www.dow.com

Paraloid B-67 (isobutyl methacrylate polymer) Kremer Pigmente GmbH & Co KG www.kremer-pigmente.de

Paraloid B-72 (ethyl methacrylate polymer) Kremer Pigmente GmbH & Co KG www.kremer-pigmente.de

Plextol B 500 (aqueous dispersion based on methyl methacrylate and ethyl acrylate) Lascaux Colours & Restauro www.lascaux.ch

Plextol D 498 (aqueous dispersion based on methyl methacrylate and ethyl acrylate) Lascaux Colours & Restauro www.lascaux.ch

Primal AC 35 (acrylic latex) Kremer Pigmente GmbH & Co KG www.kremer-pigmente.de

Methyl ethyl ketone, EMPLURA® Merck KGaA www.merckgroup.com

Methyl ethyl ketone, puriss 99.5% p.a. Sigma-Aldrich Co. LLC. www.sigmaaldrich.com

Methyl ethyl ketone, Rotipura 99.5% p.a. Carl Roth GmbH & Co. KG www.carlroth.com

Toluene, Fluka puriss Sigma-Aldrich Co. LLC. www.sigmaaldrich.com

Toluene, 99.5% for synthesis Carl Roth GmbH & Co. KG www.carlroth.com

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