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The Dutch Fe-Migration Mending Test

Exploring Further Areas of Use

The conservation of manuscripts and paper-based objects containing iron gall ink raises specific problems, even when the paper is in good condition. Iron gall inks are sensitive to water and some restoration treatments involving water cause small migrations of ink around the line. These brown halos, generally very subtle, are often acceptable. However, they are accompanied by iron migration that can be detrimental for the conservation of the support. Documents in good condition can be potentially damaged by inappropriate restoration treatments. This problem is all the more crucial as solubility tests performed by paper conservators are unreliable and do not allow identification of migrating compounds. An alternative tool has recently been proposed for treatment. It aims to test the act of treatment itself, which is applied to a paper impregnated with bathophenanthroline and stamped with an iron gall ink. Migrations of Fe(II) out of the ink line are visually assessed through the occurrence of pink halos. This test is

particularly sensitive. If no pink halo appears, the treatment process can be implemented with confidence on original objects.

As this test ensures a satisfactory procedure when repairing originals, it is tempting to use it more generally for any solvent-based treatment. To this end, several treatments were evaluated with this test and the results compared with the examination of original manuscripts subjected to the same treatments. This approach showed that this test is poorly reliable to evaluate alcohol-based treatments as bathophenanthroline and its derivatives are alcohol soluble. Regarding water-based treatments, the test is not suitable for assessing immersion treatments but gave convincing results when evaluating treatments using a moderate addition of water and where there is no possible extraction of soluble components. In particular, it allowed the determination of safe humidity levels to be applied.

Looking for an Alternative to Solubility Testing

When subjected to conservation treatments, such as flattening, mending, consolidation, infilling of missing parts, etc., iron gall ink-containing drawings and manuscripts are often exposed to various amounts of aqueous solutions, alcohol or alcohol-water mixtures (Reissland 1999a, 1999b; Reissland and Groot 1999). In many cases the use of these solvents causes treatment side effects (Reissland 2000). Indeed, some components of iron gall inks are sensitive to water and some conservation treatments have the effect of causing the migration of ink components (Jacobi et al 2011; Rouchon et al 2009a, 2007; Reissland 1999a, 1999b). Sometimes these migrations are visible as brown halos around and underneath ink lines, which are generally very subtle and therefore often accepted. However, they are accompanied by a migration of iron that is potentially detrimental for the long-term conservation of the support (Rouchon et al 2009a, 2009b, 2009c). Documents in relatively good condition can thus be damaged by the inappropriate application of a solvent. Moreover, tests regularly performed by conservators for assessing the solubility of iron gall inks are unreliable for predicting migration phenomena (Rouchon et al 2009a, 2009c). Solubility tests do not show migration of invisible components nor allow identification of migrating components.

An alternative tool has recently been proposed to assess the risk of migration of Fe(II) ions when an adhesive containing a certain percentage of water is applied to a document (Jacobi et al 2011). It consists of a paper impregnated with bathophenanthroline (an indicator for Fe(II) ions) and stamped with an iron gall ink inscription. This test does not aim to assess the sensitivity of ink but the treatment itself. The conservator can work on a test paper to achieve the most effective minimal amount of water to be used before treating an original. It gives satisfactory results in the case of water-based mending treatments. This

paper investigates the possibility of extending the application of this test to a larger range of conservation treatments and preservation measures.

The Dutch Mending Test (DMT)

The DMT consists of a sheet of filter paper (Whatman™ Grade 41: Sigma Aldrich) that is first impregnated with a solution of bathophenanthroline in alcohol ($1.6 \text{ g}\cdot\text{L}^{-1}$) then stamped with an iron gall ink (Fig 1). All experimental aspects dealing with the development of the DMT can be found elsewhere (Jacobi et al 2011).

Bathophenanthroline (Sigma Aldrich) is a colourless compound, which reacts with Fe(II), and to a lesser extent with Fe(III), to form several types of chelated species. The 1:3 Fe(II)-bathophenanthroline chelate is the best known: unlike the other chelates that are colourless, it has a characteristic fuchsia pink



1 The DMT. Front (top) and back (bottom). Small pink traces are visible on the verso indicating that bathophenanthroline reacts with available Fe(II) ions during the application of the ink.

colour which corresponds to a strong absorption at 533 nm (Cluley and Newman 1963; Clark 1962), thus allowing a spectrophotometric determination of Fe(II) over a wide pH range (pH 2-9). Bathophenanthroline paper strips (BPS) were specifically produced for use in conservation. They have been commercially available for several years and are widely used to identify the presence of Fe(II) in inks, pigments, stains and on textiles (Neevel and Reissland 2005; Vuori and Tse 2005).

The DMT was inspired by these bathophenanthroline test papers. It was first designed by Helen Wilson and her Dutch colleagues to test how long it takes for Fe(II) ions to migrate out of applied ink lines in different humidity conditions (Wilson 2007). Later, it was further developed to serve as a 'guinea pig' for a quick and effective assessment of the risk of Fe(II) ions migration during repair procedures. After being treated in a similar way as the originals, the DMT should remain unchanged. If this is the case, it can be concluded that the treatment did not cause a significant migration of Fe(II). On the contrary, if a pink halo appears around and on the verso of the ink line, a migration of Fe(II) is indicated, meaning that too much solvent was applied during treatment.

This test is highly sensitive for two reasons: firstly, the un-sized Whatman filter paper is very permeable to solvents. Secondly, the pink colour appears for Fe(II) at levels as low as $1 \mu\text{mol}\cdot\text{g}^{-1}$ (about 60 ppm) (Rouchon et al 2011; Vuori and Tse 2005). Due to this high sensitivity, it is reasonable to assume that a treatment that does not cause a pink halo will also pose no major risk of iron migration on original documents.

The use of this test enabled the formulation of aqueous-adhesive treatment protocols that minimize the risk of migration (Van Velzen and Jacobi 2011a, 2011b). In addition to this application, it is tempting to use this test to evaluate the side effects of any type of aqueous treatment, ranging from humidification to immersion. The DMT was therefore subjected to various aqueous treatments and the results compared with observations previously made on original documents (Rouchon et al 2009a, 2009b, 2007).

Precautions to be Taken

The DMT are handmade, so the ink applications vary in their visual appearance: the quantity of applied ink varies and, as Whatman filter paper is not sized, the ink migrates through the paper and becomes locally visible on the back of the sheet. Some areas in the close vicinity of the ink might already be coloured pink (Fig 1) because, at the moment of its application, the ink contained a certain amount of Fe(II) that reacts with the bathophenanthroline.

To overcome the difficulties associated with this differing aspect, the DMTs were systematically photographed before and after intervention in reproducible lighting conditions. In addition, the papers were systematically cut through the middle of the inscription in order to treat one part and keep the other as a control. By using these two approaches in a complementary way, migration of iron is clearly visible whether by comparison of before and after photographs or by examining the two parts re-joined (Fig 2).

Organic Solvent-Based Treatments

Bathophenanthroline and its derivatives (such as Fe(II)-bathophenanthroline compounds) are soluble in many organic solvents and particularly in alcohol and acetone. This property significantly limits the use of DMT in evaluating treatments with organic solvents. During immersion of DMT in alcohol, most of the bathophenanthroline goes into solution within ten minutes, which makes the test ineffective. For a short immersion time of about a few seconds, a faint pink halo sometimes appears, but in a very irreproducible manner. This pink halo may correspond to a migration of Fe(II) but also to a migration of the Fe(II)-bathophenanthroline compounds already present before immersion.

The assessment of the risk of migration caused by ethanol-based adhesives such as Klucel G raises similar difficulties: the DMT paper absorbs some ethanol when Klucel G in ethanol is applied to it. This enables the dissolution of bathophenanthroline and its derivatives. Thus the formation of a pink halo may not necessarily correspond to a migration of Fe(II) ions, but also to that of the coloured Fe(II) bathophenanthroline compounds that are already present in the stamped ink.

It would appear more appropriate to assess the risks of Fe(II) migration with ethanol by directly applying bathophenanthroline paper strips (BPS) to original documents in a fairly similar way to identifying iron gall inks (Neevel 2009; Neevel and Reissland 2005), but using ethanol instead of water. Fundamentally, the pink colour indicates the ability of Fe(II) ions to be dissolved by the solvent and to migrate from the original ink to the BPS. In this respect, the BPS is not only a test of identification, but can also be seen as a test of solubility. In this project the BPS test was applied to a large range of original manuscripts, mostly causing the appearance of a pink colour with water, but not with alcohol. With original inks, the risk of migration of Fe(II) is therefore greater with water than with ethanol, which is consistent with observations of original documents after treatment (Martin 2011). Although instructive, the BPS test should be considered with great care when evaluating the effects of solvents other than



2 DMT water immersion test. The upper and lower parts were kept separate as controls and not treated. The centre-right part was immersed for 5 minutes. The centre-left part was immersed for 1 hour.

water (e.g. ethanol or iso-propanol) on valuable objects. In particular, the contact should be applied to a very restricted spot, and on a thick line in order to limit the risk of bathophenanthroline migration from the BPS strip to the original paper during the test.

Immersion in Aqueous Solutions

Earlier studies performed on original documents immersed in distilled water showed that the risk of migration of iron around the ink lines and in the paper is greater with a short immersion time (Rouchon et al 2009a, 2007; Reissland 1999b). This is because it takes some time for water to enter the core of the paper and for soluble components to be dissolved and migrate. When the immersion lasts for a long time and the water is refreshed regularly, soluble components are transported into the solution. When immersion is short, soluble components migrate to the environment that is most accessible after removal from the bath, namely the paper itself.

When DMT is immersed in distilled water to simulate what could happen on an original document, a large pink halo forms regardless of the duration of the bath (Fig 2). A quick interpretation would be that the migration of Fe(II) is similar for 5 minutes and 1 hour of immersion but this appears inconsistent with observations of originals (Rouchon et al 2009a, 2007). A closer look reveals that the halo forms in the first few seconds of the bath, increases when the paper is moved and does not disappear afterwards. This behaviour obviously contradicts what happens with originals. It should be kept in mind that, unlike Fe(II) ions, bathophenanthroline and its derivatives are poorly soluble in water. Bathophenanthroline thus acts as a trap for Fe(II) ions, retaining them within the paper and impeding their removal. The presence of bathophenanthroline in the paper thus strongly interferes with Fe(II) migration phenomena. The pink colour that appears around the ink line is therefore not the same as the Fe(II) migration that would have taken place without bathophenanthroline.

This point can be demonstrated with test samples stamped



3 Additional water immersion test. The upper and lower parts were kept separate as controls and not treated. The centre-right part was immersed for one minute. The centre-left part was immersed for one hour. Bathophenanthroline was sprayed after immersion (not before).

with the same iron gall ink, but not impregnated with bathophenanthroline. These samples were used to test different kinds of immersion treatments (Figs 3, 4): one minute in water, one hour in water, and also the full calcium phytate treatment [1] (Rouchon 2011; Neevel 1995). After treatment these samples were sprayed with a solution of bathophenanthroline (1.6 g·L⁻¹ in 99.9% ethanol). The verso of untreated parts appeared pink, indicating the presence of some Fe(II) ions in the inks (Fig 4). The sample that was immersed for one minute in water showed a pinkish halo (Fig 3) characteristic of Fe(II) migration occurring after removal from the solution. However, no pink colour was perceptible on treated parts that were immersed for one hour in water (Fig 3), or treated with calcium phytate (Fig 4), indicating that the soluble Fe(II) was extracted or oxidised into Fe(III) during the treatments.

Exposure to High Levels of Humidity

Assessing the Risks Associated with Moisture

Observation of original manuscripts showed that the risk of formation of brown halos and iron migration are substantial beyond 90% RH and largely minimised below 85% RH (Rouchon et al 2013). The DMT confirmed these results and made it possible to determine more accurately the threshold limit. The test was repeated more than ten times at ambient temperature and 80% RH [2]. No pink halo was observed for periods as long as 14 days. The exposure duration was then limited to 24 hours and the relative humidity increased by using various salts (ISO 483 1999). The humidity threshold above which Fe(II) migrates is between 85% and 88% RH (Fig 5).

Given the limited precision of hygrometers, it is therefore reasonable not to exceed a relative humidity of 80%. This relatively low humidity level is not compatible with treatments requiring a substantial water supply, such as the removal of a secondary support. However it gives satisfactory results for flattening treatments.

Comparison of Different Humidification Chambers

Several methods are available to achieve a specific relative humidity. One uses a silica gel that corresponds to the desired humidity range. Some give satisfactory results in the range 70-90% RH (gel type M: Long Life for Art). Silica gel has the advantage of generating vapour without causing micro-droplets, but



4 Risk of Fe(II) migration related to the calcium phytate treatment. The upper part was kept separate as a control and the lower part underwent the full calcium phytate treatment. Bathophenanthroline was sprayed after immersion (not before).

needs to be periodically regenerated in a climatic chamber, which is not easily accessible for all conservation studios. Moreover, the humidity released by the silica gel must be verified with an accurate hygrometer, as it cannot be relied upon to remain below the desired value.

Another way to obtain humidity conditions below 100% is using closed containers filled with 50:50 water:ethanol mixtures. Several versions of this method have been developed in conservation studios (Fries 2006), using, for instance, cedar wood frames (Cedar wood humidity box: Alan Buchanan Designs). How humidity conditions could be monitored with this approach have been briefly tested (Vez 2013). Hermetic plastic containers of various dimensions were filled with the solution. Then a humidity recorder (hygrobutton: Proges Plus) was placed on an elevated grille held above the surface of the solution and the box was closed. These tests showed that the humidity increased more or less rapidly depending on the dimensions of the box and the amount of solution. The use of an ethanol and water mixture delays the increase of humidity when compared with the use of water only. Yet it does not prevent humidity rising up to 100%. A relative humidity below 100% can only be achieved if the chamber is not hermetic. This corresponds to a stationary system in which the increase of humidity is compensated by the release of water pressure. This kind of humidification technique should therefore be carefully monitored with reliable hygrometers.

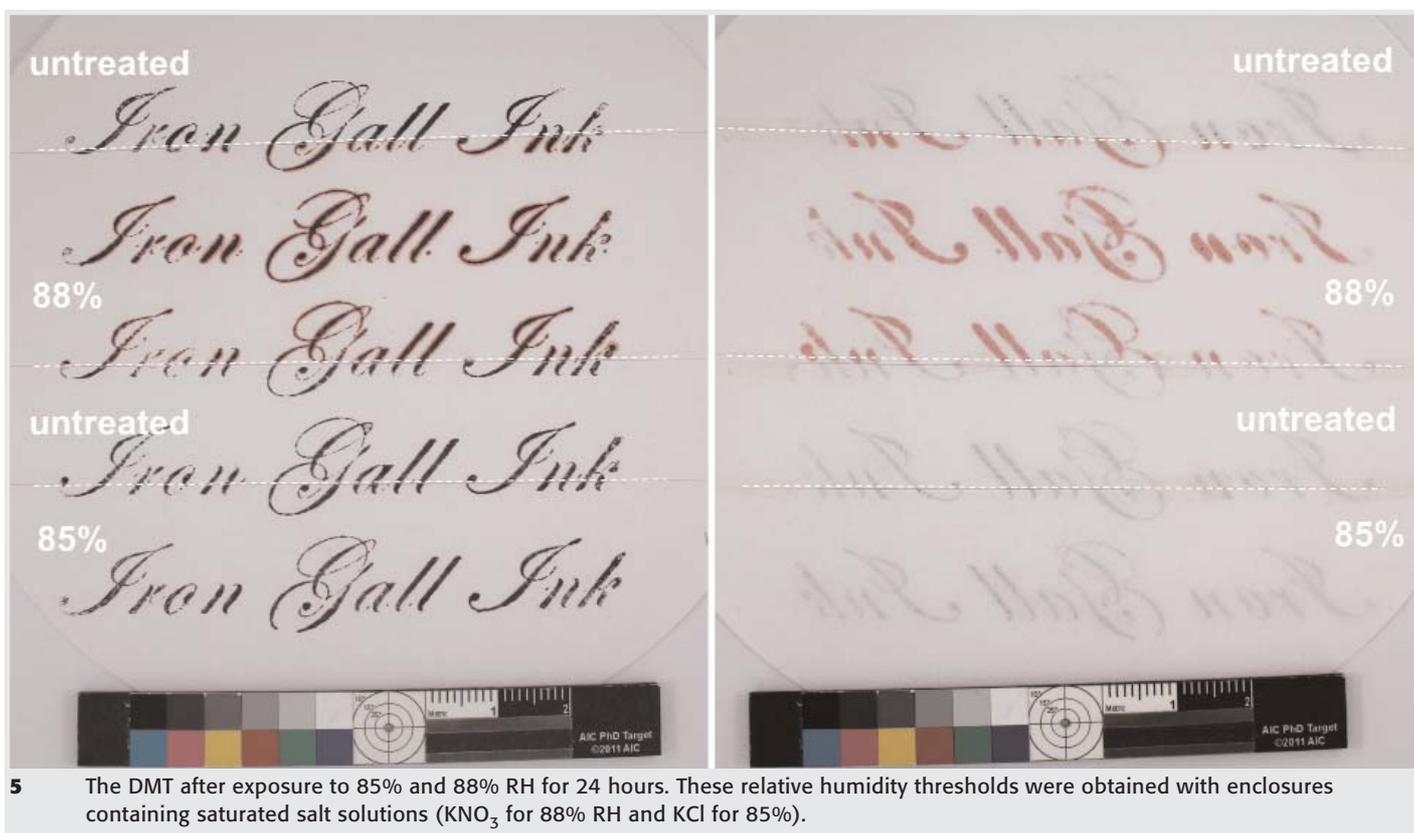
Saline atmosphere conditioning boxes (ISO 483 1999) may offer a safer alternative. The principle is identical as above, but water/alcohol solutions are replaced by saturated saline solutions (Fig 6). The chemical nature of the salt determines the relative humidity to be achieved when the enclosure reaches thermodynamic equilibrium, which usually takes several hours.

This method is obviously not rapid and thus not appealing when a large number of documents need to be treated but it presents several advantages. Firstly, it is not physically possible to exceed the desired relative humidity and secondly, the relative humidity is achieved with good accuracy over a large temperature range (for example, small boxes of this type are used to calibrate hygrometers). The use of a saline chamber has three main requirements: firstly, the solutions should be saturated, meaning that some undissolved crystals should remain in the solution; secondly, the chamber should have a restricted height with respect to its width for the desired moisture to be achieved evenly and within a reasonable time (4 hours is usually sufficient). Thirdly, the chamber should be cleaned regularly to prevent salt depositing on the walls (Fig 7).

These salt deposits are likely to occur over several weeks and their formation can be easily avoided. It however raises the question of whether similar deposits occur on the paper itself. Virgin Whatman No 1 was therefore exposed to various saline atmospheres for 48 hours and afterwards underwent elemental analysis [3]. These analyses, summarised in Tab 1, show that, according to the accuracy of the measurements, whatever the salt used, there is no major change in the composition of the paper over 48 hours of exposure. Saline atmosphere conditioning boxes also appear relatively safe: these boxes were recently used at 80% RH (Fig 8) to successfully flatten archival documents whose edges were folded or rolled (Meslet-Struyve 2013).

Migrations Related to Thermal Shocks

Archival storage buildings are maintained at a substantially cooler temperature than reading rooms and exhibition galleries. Thus many curators feel concerned about the potential impact



Tab 1 Elemental analysis performed on Whatman No 1 paper sheets placed in saline humidity chambers for 48 hours without protection.

| Salt | % RH | Na (ppm) | BR (ppm) | K (ppm) | S (ppm) | N (ppm) | Cl (ppm) |
|--|------|----------|----------|---------|---------|---------|----------|
| no. (virgin Whatman No 1) | - | 150 ± 30 | <40 | <50 | 30 ± 20 | 40 ± 10 | 270 ± 60 |
| Sodium bromide, NaBr | 59 | 150 ± 30 | <40 | - | - | - | - |
| Ammonium chloride, NH ₄ Cl | 79 | - | - | - | - | 20 ± 10 | 250 ± 60 |
| Potassium bromide, KBr | 81 | - | <40 | <50 | - | - | - |
| Ammonium sulphate, NH ₄ SO ₄ | 81 | - | - | - | 30 ± 10 | 35 ± 10 | - |
| Potassium chloride, KCl | 85 | - | - | <50 | - | - | 210 ± 60 |
| Potassium nitrate, KNO ₃ | 88 | - | - | <50 | - | 50 ± 10 | - |
| Potassium sulfate, K ₂ SO ₄ | 97 | - | - | <50 | 40 ± 20 | - | - |

of such temperature differences when these objects are transported from one place to another. There is no question here of addressing the risk of mechanical constraints that may apply to the weakest part of the bindings, or the risk of deformation-sensitive media such as parchment, but is simply focused on the issue of ink migration. Indeed, the fluctuations in temperature and humidity may cause additional water condensation in the paper and may thus induce migration of soluble compounds like Fe(II) ions.

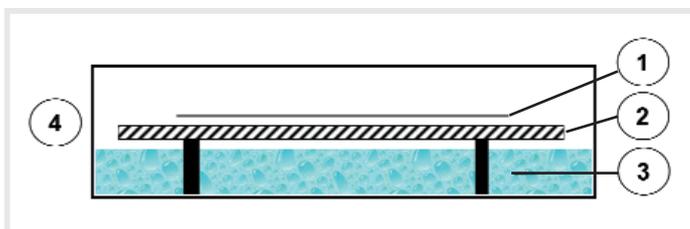
The DMT was used to assess this risk. Examples were placed with 50% RH pre-conditioned silica gel in sealed glass test tubes for 4 hours at 5°C in order to simulate the atmosphere of cold storage. They were then quickly moved to a climate chamber at 30°C and 80% RH to simulate a warm and steamy reading room. The temperature and humidity conditions were recorded (hygrobutton: Proges Plus) adjacent to the DMT. The transition from

one condition to another occurred in about 2 minutes. These tests were first carried out on loose DMT sheets to simulate individual documents. Tests were then duplicated on DMT sheets placed in the middle of a bundle of one hundred sheets of Whatman paper held on one side by a large clip in order to simulate the case of an archival bundle. No migration of Fe(II) was found in either case.

These observations suggest that the transition from a cold and dry storage to a warm and humid room does not pose a substantial risk of condensation when the relative humidity remains below 80%. However, these results should be taken cautiously: laboratory experience is no substitute for an on-site survey. To avoid ambiguities, it is strongly recommended to use the DMT in situ to check the safety of changing climatic conditions, either during the consultation or the loan of sensitive pieces.

Conclusion

The DMT has been designed for evaluating the risk of Fe(II) migration during a local repair using a water-based adhesive, and gives satisfactory results. Additional applications of this test were evaluated in this research. It appears to be unsuitable for assessing the risk of Fe(II) migration during an alcohol treatment and during a water immersion. Similarly, intermediate treatments in which soluble products can be extracted in a solvent (floating, vacuum table, etc.) cannot be reliably evaluated with this test. In all these cases, it is more appropriate to refer to the observation of original samples when assessing the migration risk. It could also be interesting to develop a variant of the



6 Diagram of a saline chamber. The document is laid on a non-woven polyester film (1) placed on a grille (2) above the surface of a saturated salt solution (3), the whole is placed in a large hermetic box (4) with a limited height.



7 Example of crystal growth of ammonium sulphate, (NH₄)₂SO₄, appearing on a glass plate after three weeks in an 80% RH saline chamber with no cleaning.



8 Overall view of an 80% RH saline chamber constructed for flattening documents.

DMT consisting of Whatman paper stamped with an iron gall ink on which a spray of bathophenanthroline/alcohol solution is applied after treatment.

Nevertheless, there is a wide range of application for the DMT in dealing with the impact of humidification. The test appears appropriate for the evaluation of the different humidification treatments currently used in conservation studios (spraying, misting, exposure to water vapour, etc.).

In another context, the DMT can additionally be used as a 'snitch' to assess the quality of an environment and the impact of changes in humidity and temperature when the environment is not perfectly controlled. This is particularly true for objects that travel between different buildings for a consultation or a loan. In this respect, the DMT could also be helpful in monitoring the risk of ink corrosion in tropical climates.

Endnotes

- [1] The calcium phytate treatment works in three stages. The first corresponds to a washing with water, the second to an immersion in a solution of calcium phytate, and the third to a de-acidification using calcium carbonate. These three steps have each been implemented for a period of 15 minutes, for a total immersion time of 45 minutes that is theoretically sufficient to remove most of the soluble compounds.
- [2] Several techniques have been tested: (a) silica gel preconditioned at 80% RH in a climate chamber, (b) saturated saline chamber made with ammonium sulphate and (c) saturated saline chamber made with ammonium chloride.
- [3] These analyses were performed by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES) at the Institut des Sciences Analytiques, Service Central d'Analyse (CNRS UMR 5280-ISA) after dissolving the papers in concentrated acids.

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Suppliers

Alan Buchanan Designs Ltd, 3 Arlington Cottages, Sutton Lane North, W4 4HB London, GB, Tel +44-208-9959780, www.alanbuchanandesigns.com (Cedar Wood Humidity Box).

Long Life for Art, Christoph Waller, Hauptstr. 47, 79356 Eichstetten, Germany, Tel +49-7663-608990, www.cwaller.de (Silica Gel type M).

Practice-in-Conservation, Afrikanerplein 15, 1091 PN Amsterdam, The Netherlands, Tel +31-644-659303, www.practice-in-conservation.com (DMT, Dutch Fe-Migration Mending Test).

Proges Plus, 2 rue de la République, 59780 Willems, France, Tel +33-320-646363, www.proges.com (Hygrobutton).

Sigma-Aldrich Chemie S.a.r.l., L'Isle d'Abeau Chesnes, 38297 Saint-Quentin Fallavier, France, Tel +33-800-211408, www.sigmaaldrich.com (WhatmanTM, Grade 41, quantitative ashless filter paper, Cat. No. 1441-090, Ø 9 cm; bathophenanthroline)

German Title and Abstract

Der Niederländische Fe-Migrations Test für Risschliessungen: Die Suche nach weiteren Anwendungsmöglichkeiten

Die Restaurierung von Handschriften und anderen Objekten mit Eisengallustinte ist mit spezifischen Problemen verbunden, selbst wenn das Papier in gutem Zustand ist. Eisengallustinten sind wasserempfindlich und einige wässrige Restaurierungsbehandlungen können eine geringe Tintenmigration verursachen. Die um die Linien entstehenden braunen Halos sind in der Regel jedoch subtil und werden daher oft akzeptiert. Jedoch tritt hierbei auch eine Wanderung von Eisenionen auf, was langfristig schädlich für die Erhaltung des Papierträgers sein kann. Dokumente in gutem Zustand können so durch unsachgemäße Restaurierungsbehandlungen beschädigt werden. Dieses Problem ist um so dringender, da die von Papierrestauratoren angewendeten Tests auf Wasserlöslichkeit meist unzuverlässig sind und keine Identifikation migrierender Komponenten erlauben. Deshalb wurde kürzlich eine alternative Testmethode auf den Markt gebracht. Diese ermöglicht es, eine vorgesehene Risschliessung erst auf einem Indikatorpapier auszuführen. Das Testpapier ist mit Bathophenanthroline imprägniert und mit Eisengallustinte bestempelt. Migrationen von Fe(II) Ionen aus den Tintenlinien werden optisch durch das Auftreten von pink gefärbten Halos sichtbar. Dieser Test ist besonders empfindlich. Wenn kein Halo erscheint, kann die Behandlung auch auf einem Original ausgeführt werden.

Da dieser Test zufriedenstellende Ergebnisse für das Risschliessen an Originalen erzielt, war es interessant, um seine Anwendungsmöglichkeit auch für andere Behandlungen zu testen. Mehrere Behandlungen wurden auf den Indikator-Testpapieren und an Originalhandschriften ausgeführt. Die Ergebnisse wurden verglichen. Es zeigte sich, dass dieser Test keine zuverlässigen Ergebnisse für alkohol-basierte Behandlungen erlaubt, da Bathophenanthroline und seine Derivate alkohollöslich sind. In Bezug auf wässrige Behandlungen ist der Test ungeeignet für die Beurteilung von Badbehandlungen, erreichte aber überzeugende Ergebnisse bei der Bewertung von Behandlungen mit einer moderaten Zugabe von Wasser, insofern keine löslichen Bestandteile extrahiert wurden. Vor allem ist der Test geeignet, um das Risiko verschiedener Luftfeuchtigkeitsbereiche zu bestimmen.

Authors

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