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To Treat or Not to Treat—That is the Question: Options for the Conservation of Iron-Gall Ink on Paper

ABSTRACT

Treatment options for iron-gall ink corrosion on paper, including the phytate treatment, were compared and evaluated in a joint research project. Mounting papers from the late eighteenth century offered the chance to test and evaluate conservation methods on naturally aged inks and papers. Samples of newly produced ink on rag paper could be submitted to mechanical testing. The treated mounting papers and the new samples were artificially aged in order to evaluate the long-term effects of treatments. Evaluation was done by visual assessment, measurement of pH, color, scanning electron microscopy (SEM), tear resistance, tear index, folding endurance, micro x-ray-fluorescence analysis (μ-XRF) of iron intensities, x-ray absorption near edge structure (XANES), photometric measurement of cellulose degradation products, determination of carbonyl groups, and molar mass distribution.

The results have shown that the combined phytate treatment stabilizes ink corrosion on paper. No additional degradation caused by the phytate solution could be detected. However, methods of local reinforcement can result in migration of iron ions in non-inked areas in the process of aging.

1. INTRODUCTION

The conservation of iron-gall ink on paper and especially the treatment of the degradation caused by iron-gall inks have occupied conservators for centuries. Numerous treatments have been tried and discussed. The long-term effects of some of these treatments make conservators reluctant to treat manuscripts or drawings with iron-gall ink at all. Similar to Hamlet in his famous speech, conservators might be tormented by the question: "To treat or not to treat?" Like Hamlet they might first refrain from action and then, by the urgency of the case, engage themselves in a treatment that they can no longer control. Sometimes, like Hamlet says in his last words: "The rest is silence."

The present study aims to provide conservators with more evidence on the long-term effects of treatments in order to help in the decision-making process. In a comparative study the long-term effects of aqueous and nonaqueous treatments were investigated. Local treatments were compared with treatments of the whole sheet. The methods were intended to resemble real life situations; thus extreme conditions were avoided. Original mounting papers from the Lavater collection gave the opportunity to study naturally aged inks from the late eighteenth century. Using samples of new iron-gall ink on rag paper, treatment in a bath versus treatment on the suction table were compared. These samples could be used for destructive testing of mechanical strength. The study was a cooperative project among the Austrian State Archives, the Austrian National Library, the Academy of Fine Art, the Graz University of Technology, and the Vienna University of Technology. It was funded by the Austrian Federal Ministry of Economics and Labour. Additional analysis was carried out at the University of Natural Resources and Life Sciences, Vienna, and at the Federal Institute for Materials Research and Testing, Berlin.

The study aimed to answer the following questions:
- Does the treatment of iron-gall inks result in diffusion of iron ions within the paper?
- Is the color of the ink changed by the treatment?
- Does the phytate treatment stabilize iron-gall inks in the process of further aging?
- Does the phytate treatment diminish the mechanical stability of rag paper?
- How do methods of local reinforcement perform after aging?

2. OBJECTS AND SAMPLE PAPERS

2.1 Lavater Mounting Papers

The Lavater collection is part of the Pictures Archive of the Austrian National Library. Johann Caspar Lavater (1741–1801) lived and worked as a pastor and famous preacher in Zurich, Switzerland. He tried to prove scientifically that one could detect the character of a person by his outward appearance. He isolated parts of the face and developed a kind of alphabet of physiognomy. He was convinced that artistic presentations provided more evidence and therefore assembled a large collection of different portraits. Works by Daniel Chodowiecki, J. H. Fuessli, J. H. Lips, Wilhelm Tischbein, Angelika Kauffmann, and Domenico Tintoretto were collected by Lavater. Watercolors, drawings, and prints were mounted and put in a *passepartout* with a handwritten comment on their physiognomy (fig. 1). The mounted images are stored in specially manufactured boxes (Mraz 1999; Lachs 2001). In 911 portfolios Lavater had assembled 22,101 images that served him as a study collection for his research in physiognomy. Emperor Franz I bought the Lavater collection in 1828.

Sometimes a broad line drawn in iron-gall ink, seven to eight millimeters wide, was used to frame the mounted image. In the case of a group of etchings mounted on rag paper these lines had led to severe deterioration of the mounting paper (fig. 2). For twenty-two prints the mounting paper was so heavily deteriorated that the conservators, in consultation with the curators, decided to remove the prints and to remount them in the same style. The curators consented to the use of the original mounting papers for research.

The mounting papers consisted of rag paper and date from 1780. The main component of the ink is iron with minor amounts of potassium and calcium and traces of copper and zinc according to the XRF analysis (Jembrih-
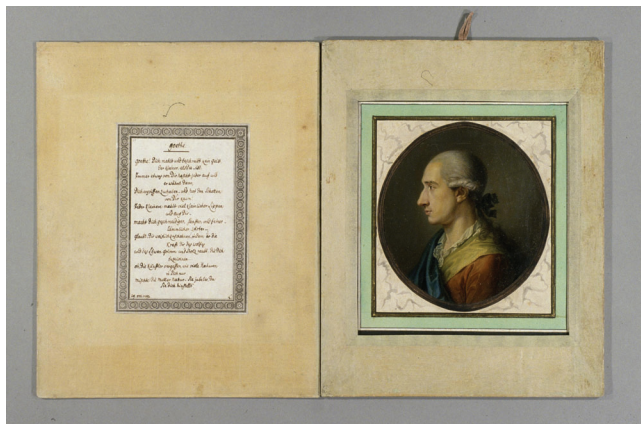
Simbuerger et al. 2004). The ink had been applied in a line seven to eight millimeters wide. The color of the ink ranged from dark brown to black. In some areas the ink was abraded, in others cups and cracks had formed on the thick ink deposits. The iron-gall ink had caused severe corrosion, "category four" according to Reissland (2000). Large parts of the inked lines had broken away. The parts of the rag paper without ink were in very good condition. The prints had been lined with wheat-starch paste to the rag paper support, as determined by a positive iodine-potassium iodide spot test (Schramm 1995). During unmounting the papers were briefly immersed in water. For the untreated control (paper T0) and the nonaqueous treatments, parts of the mounting paper that had fallen off due to ink corrosion were used. These parts were not immersed in water.

2.2 Austrian State Archives Rag Paper Samples

Lab-prepared iron-gall ink was applied to naturally aged rag paper to produce samples that could be used for destructive testing. These samples were used for comparisons of phytate treatment and deacidification in a bath and on the suction table. The results of the mechanical tests could be correlated with the experiments performed on the Lavater papers.

The naturally aged rag paper dates from 1706 and was produced in a paper mill in Wiener Neustadt, Lower Austria. The papers all belonged to the nonwritten sheets
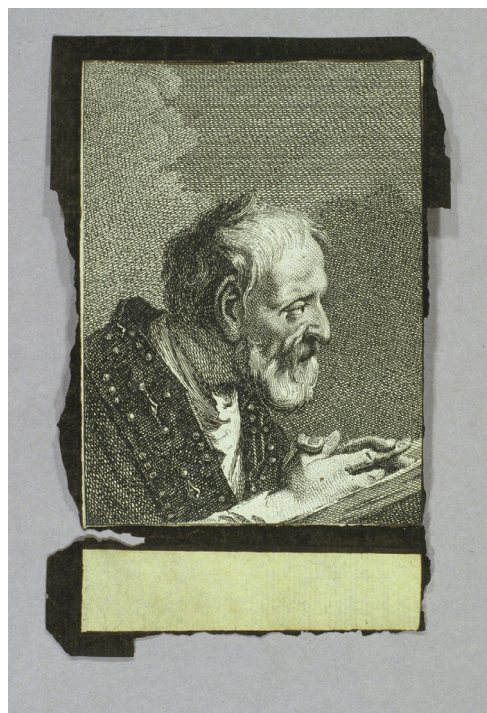

Fig. 1. Johann Daniel Bager. *Johann Wolfgang von Goethe,* 1772–1773. Oil on cardboard, 417 x 317 mm, Austrian National Library (POR Slg.Lav. 9845).

Fig. 2. Anonymous. *St. Peter.* Etching, 120 x 87 mm; mounting paper 160 x 102 mm, Austrian National Library (POR, Slg.Lav. 16737).

of the "Gestütbuch" (studhorse book) of Alois Harrach (Austrian State Archive, Oesta SR 170), which ensures that the individual sheets were stored and aged under the same conditions. New iron-gall ink was produced according to the recipe of Johan Neevel (Neevel 1995) using a molar ratio of 5.5:1 iron to tannin that roughly corresponds to the ratio occurring in historical ink recipes.[1] The ink was applied over the whole surface of the pre-wetted paper sheets (814 x 664 mm) by brush. The inked sheets were left to air dry on polyester web.

By artificial pre-aging, the condition of an aged ink on paper that is submitted to treatment should be simulated.

3. EXPERIMENTAL DESIGN

3.1 Treatments of Lavater Mounting Papers

The Lavater mounting papers were submitted to eight different treatments, described in table 1. They were compared with the untreated control after the treatment and after artificial heat aging. Treatments were chosen that are within the range of methods practiced at the conservation department of the Austrian National Library. Simmering and paper splitting were excluded. Treatments of local reinforcement were included that might be used to secure ink corrosion within a book (T1, T1G, T2). Because the ink corrosion was severe, all aqueous treatments were carried out on the suction table to avoid the migration of iron ions and degradation products. After aqueous treatment the papers were lined to reinforce the fragile margins.

T0 (34 x 107 mm): Untreated control.

T1 (34 x 107 mm) was sprayed twice from the front and once from the back with a 3% solution of methyl magnesium carbonate, MMC.[2] The paper was then lined with Japanese paper (*usumino* B5, 12 g, pure *kozo* fibers) and Klucel G 6% in ethanol. The paper was dried between polyester web, blotters, and felts under weight.

T1G (34 x 97 mm) was lined with a 10% solution of photographic gelatin (Rousselot, Type B, 285 Bloom) and Japanese paper. Drying was carried out as for T1.

T2 (36 x 97 mm) was lined with wheat-starch paste and Japanese paper. Drying was carried out as for T1.

T3 (137 x 103 mm) was humidified in a Gore-Tex sandwich for 30 minutes and put on the suction table with a polyester web support (Parafilm RT 30) on blotting paper. The sample was sprayed with deionized water (170 mL) using an airbrush. The blotting paper support was changed three times. The pressure on the suction table was 20 kPascal. After washing the paper T3 was dried between polyester web, blotting papers, and felts under weight. The blot-

ting paper was exchanged three times. After drying the slightly humidified paper was lined with diluted wheat-starch paste (1:4) and Japanese paper. Drying was carried out as described above.

T4 (164 x 105 mm): After humidification the sample was sprayed with a saturated solution of calcium bicarbonate (pH 5.94; 170 mL) on the suction table.[3] Drying and lining was carried out as for T3.

T5 (162 x 103 mm) was sprayed with a solution of calcium phytate (pH 6; 50 mL) and afterwards with a solution of calcium bicarbonate (100 mL).[4] Drying and lining was done as previously described.

T6 (169 x 109 mm) was sprayed with a 2:1 solution of calcium phytate and ethanol. Deacidification was carried out by spraying a 4:1 solution of calcium bicarbonate and ethanol. All other steps were done as for T5.

T7 (169 x 110 mm) was treated as T6, but on the suction table the inked area was resized with a 2% gelatin solution applied by brush. The drying and lining treatment was the same as for the other mounting papers.

3.2 Treatment of ASA Rag Paper Samples

The treatment of the inked rag paper samples from the Austrian State Archives (ASA) allowed a comparison between deacidification and the combined phytate treatment, and between treatment in a bath and on the suction table (table 2). The following procedures were carried out:

For testing the combined calcium phytate treatment in the bath (samples 4a, 4b, 5a, 5b and pre-aged samples 6a, 6b, 7a, 7b), the samples were rinsed in tap-water baths for 1.5 hours. The water was exchanged every 30 minutes. The samples were immersed in a calcium phytate solution (pH 5.3) for 30 minutes and afterwards in a calcium bicarbonate solution for 30 minutes. The papers were air dried on polyester web support.

Table 1. Treatments performed on the Lavater mounting papers

NUMBER	TREATMENT	RESIZING	LINING
T0	—	—	—
T1	Methyl magnesium carbonate (MMC)	—	Klucel G
T1G	—	—	Gelatin
T2	—	—	Wheat-starch paste
T3	Deionised Water	—	Wheat-starch paste
T4	Calcium bicarbonate	—	Wheat-starch paste
T5	Calcium phytate/ bicarbonate	—	Wheat-starch paste
T6	Calcium phytate/ bicarbonate + ethanol	—	Wheat-starch paste
T7	Calcium phytate/ bicarbonate + ethanol	Gelatin	Wheat-starch paste

Table 2. Aging and treatments of inked rag paper samples

Number	Pre-aging	Treatment	Post-aging
1a, 1b	—	—	—
2a, 2b	50 cycles		—
3a, 3b	50 cycles		50 cycles
4a, 4b	—	Calcium phytate/bicarbonate, bath	—
5a, 5b	—	Calcium phytate/bicarbonate, bath	50 cycles
6a, 6b	50 cycles	Calcium phytate/bicarbonate, bath	—
7a, 7b	50 cycles	Calcium phytate/bicarbonate, bath	50 cycles
8a, 8b	—	Calcium phytate/bicarbonate, suction table	—
9a, 9b	—	Calcium phytate/bicarbonate, suction table	50 cycles
10a, 10b	50 cycles	Calcium phytate/bicarbonate, suction table	—
11a, 11b	50 cycles	Calcium phytate/bicarbonate, suction table	50 cycles
12a, 12b	—	Calcium bicarbonate, bath	—
13a, 13b	—	Calcium bicarbonate, bath	50 cycles
14a, 14b	50 cycles	Calcium bicarbonate, bath	—
15a, 15b	50 cycles	Calcium bicarbonate, bath	50 cycles
16a, 16b	—	Calcium bicarbonate, suction table	—
17a, 17b	—	Calcium bicarbonate, suction table	50 cycles
18a, 18b	50 cycles	Calcium bicarbonate, suction table	—
19a, 19b	50 cycles	Calcium bicarbonate, suction table	50 cycles

For testing the combined calcium phytate treatment on the suction table (samples 8a, 8b, 9a, 9b and pre-aged samples 10a, 10b, 11a, 11b), the samples were sprayed (by air brush) with 100 mL calcium phytate solution (pH 5.3) on a blotter support on the suction table.

The calcium bicarbonate-bath samples (samples 12a, 12b, 13a, 13b, and pre-aged samples 10a, 10b, 11a, 11b) were rinsed in tap water baths for 1.5 hours, with a water exchange every 30 minutes. They were finally immersed in a saturated solution of calcium bicarbonate for 30 minutes and dried as described above.

For the calcium bicarbonate treatment on the suction table (samples 16a, 16b, 17a, 17b and pre-aged samples 18a, 18b, 19a, 19b), 180 mL calcium bicarbonate solution was sprayed on each sample sheet on the suction table.

3.3 Artificial Aging

The Lavater papers were artificially aged after treatment in a climate chamber (ESPEC- climate chamber PR2FP) at the Graz University of Technology using a dynamic aging system. The temperature was raised to 80°C and remained constant throughout the procedure. The relative humidity (RH) fluctuated between 30% and 90% during a time cycle

of 180 minutes (ÖNORM-Regel 11116). The artificial aging procedure comprised three hundred RH-cycles, which corresponds to 37.5 days.

A set of inked ASA rag paper samples was submitted before treatment to 50 RH-cycles, corresponding to 6.25 days, under the conditions described above. Pre-aging should result in ink corrosion and simulate an inked paper aged under natural conditions. After treatment post-aging also consisted of 50 RH-cycles. Pre- and post-aged samples were submitted to 100 RH-cycles altogether, corresponding to 12.5 days (table 2).

3.4 pH measurement

The surface pH on paper and inked areas on the Lavater papers were measured before treatment, after treatment, and after aging. A WTW pH-meter pH 537 was used with a surface electrode (Ingold Lot 403-M8-S//120). The electrode with one drop of deionised water was left on the surface for 2 minutes. Due to the fragility of the inked areas only one measurement per sample was carried out. A series of five to ten measurements would have resulted in severe damage to the original papers. Small changes in pH were interpreted with caution.

Cold extraction pH measurements were done on the inked ASA rag paper samples numbers 1 to 19 after treatment and aging, with ten replicas per sample. The measurements were conducted at the Graz University of Technology in accordance with ISO 6588-1 (2005–05).

3.5 Color Measurement

The color of paper and inked areas was measured on all Lavater papers before and after treatment and after aging. The measurements were taken with a Gretag SPM 50 spectrophotometer. Ten measurements were taken per sample and averaged. Color was recorded in the L★a★b★ mode.

The color of the inked ASA-rag paper samples was determined after treatment and after aging in accordance with ISO 5631 (2000–03).

3.6 Tear Resistance, Tear Index, and Folding Endurance of ASA Rag Paper Samples

Destructive testing of the mechanical strength, after treatment and after aging, was performed on the inked ASA rag paper samples. Tear resistance and tear index (Elmendorf) were measured in accordance with ISO 1974 (1990–02). Folding endurance was determined in the manner set out in the Zellcheming Merkblatt VI/1/66. All

measurements were performed at the Graz University of Technology.

3.7 Scanning Electron Microscopy (SEM)

SEM was carried out on the Lavater papers T0 to T7 after aging at the Graz University of Technology. Paper pieces (2 x 4 mm) were sputtered with gold and palladium (200 mTorr and 15 mA; three times 2 min.) and contacted from top and below with a coal tape adhering on both sides. The thickness of the coating was approximately 30 Å. All measurements were done on a JEOL JSM-T33A scanning electron microscope using an accelerating voltage of 15 kV.

3.8 Nondestructive Micro x-ray Fluorescence Analysis (μ-XRF) of Lavater Mounting Papers

In order to compare the different conservation treatments described above, analytical investigations using μ-XRF were carried out on papers T0 to T7 of the Lavater collection. All analyses were done using the portable μ-XRF instrument COPRA.[5] The technique is nondestructive. For the investigations the objects were placed vertically on a suction table built by Manfred Mayer in front of the instrument as shown in figure 11. The μ-XRF instrument used consists of a molybdenum x-ray tube, a silicon drift chamber detector, a polycapillary which focuses the primary x-ray beam on a spot of approximately 100 μm, an optical microscope for enlarging and focusing the measured area, and a CCD camera.[6]

The iron (Fe) migration from the ink area into the surrounding paper after the treatments as well as after artificial aging was studied. Intensities of the Fe-Kα-line were detected using line-scan measurements (measurements along chosen lines) that were carried out on the ink/paper border in 0.25 mm steps along a 4 mm long line. Thus two millimeters were analysed in the ink, two millimeters in the surrounding paper. Two-line scan analyses were performed on different positions in one object. As control points for the intensities of iron in the paper area, points at 10 mm distance from the inked area were always measured. To ensure that after treatments and after the artificial aging of the objects the same positions in the ink and in the paper would be measured as before, images were automatically taken with the CCD camera of the XRF instrument during the measurements of each position.

3.9 X-Ray Absorption Near Edge Structure (XANES) of Selected Lavater Papers

The objective of XANES investigations on four selected Lavater objects (T0, T1, T4, and T7), carried out four years after treatment and artificial aging, was to compare the effectiveness of the treatments applied. Analysis was carried out at the synchrotron accelerator BESSY II, Berlin, using the KMC-2 line equipment. The investiga-

tions were performed within the line-scan range of 7040–7300 eV (energy step 5 eV), and with a spot size of the beam of 250 x 600 μm^2. For signal detection a Si-PIN photodiode was used (Jembrih-Simbürger et al. 2006).

As already reported (Schreiner et al. 2003, Hofmann et al. 2004, and Jembrih-Simbürger et al. 2004) the iron-gall ink used for these objects contains the elements K, Ca, and Fe as well as traces of Cu and Zn. The aim of the XANES measurements was to determine the oxidation state of iron in the inked areas (Fe^{2+} and/or Fe^{3+}). It is well known that the paper degradation process largely results from two processes, the Fe^{2+}-catalyzed oxidation of the cellulose and the hydrolysis of the cellulose by sulfuric acid (Hahn 2002; Neevel 1999).

3.10 Water-Soluble Cellulose Degradation Products of Lavater Papers, Photometric Sugar Detection

Samples were taken from the Lavater papers T0 to T7 only after aging to measure the water-soluble cellulose degradation products. As a consequence of aging and other influences, monomer and oligomer sugars of the supporting material cellulose from paper can be formed, which are easily determined by a photometric method specially adapted for paper samples by Puchinger and Leichtfried following the publications of Scott et al. (1957), Schormüller (1967), and Stahl (1962).

The samples were prepared in the following way: 3 mL distilled water were added to 20–30 mg of the dyed textile and sonicated for 20 min. at room temperature. The solution was filtered through a 0.45 μm PTFE membrane. Constant water cooling was necessary, while 2 mL of a 0.2% solution of anthrone in sulfuric acid (H$_2$SO$_4$), having formed a lower layer, was added to 1 mL of the clear sample liquid. Then the solution was warmed to 100°C (15 min) with a heating block. After the extract had reached room temperature the extraction was measured with a photometer at 625 nm. For quantification a cellobiose standard curve was used (concentration range: 5–100 μg/mL).

3.11 Analysis of Molecular Weight Distribution and Carbonyl Group Content of Selected Lavater Papers

The analysis of the samples taken from four selected Lavater papers after aging (T0, T1, T4 and T7) was performed at the University of Natural Resources and Life Sciences, Vienna, using fluorescence labeling and the GPC multidetector set-up, following the CCOA method as described in Röhrling et al. (2002a), Röhrling et al. (2002b) and Potthast et al. (2003).

From each sample obtained, three different sub-samples were taken for analysis. Sub-sample "ink" was taken directly from the inked parts of the sample. To compare it to cellulose that has not suffered from the influence of iron-gall ink, another sub-sample "paper" was taken at

Tabel 3. Analysis of inked rag paper samples.

Number	Treatment	pH	L*	a*	b*	Tear Resistance (mN)	Tear Index (mNxm²/g)	Folding Endurance
1	control	3.5	32.78	3.04	-6.01	235.5	2.82	79.5
2	control pre-aged	3.4	27.28	1.31	1.16	28	2.82	1
3	control pre-, post-aged	3.5	34.24	1.77	5.97	29	0.42	1
4	Caphy, bath	6.6	26.71	4.32	-2.13	451.5	5.39	> 10.000
5	Caphy.b, pre-aged	5.8	30.67	2.82	8.47	238	2.96	218
6	Caphy.b, post-aged	6.5	27.6	1.66	1.09	30	0.39	1
7	Caphy.b, pre-, post-aged	6.1	29.1	2.06	3.25	36.5	0.47	1
8	Caphy. suction table	5.2	30.27	4.31	-3.31	276	3.48	296.5
9	Caphy.st, pre-aged	4.1	29.79	2.41	1.93	200	2.39	4.5
10	Caphy.st, post-aged	5.6	28.98	1.80	2.32	40.5	0.5	1
11	Caphy.st, pre-, post-aged	4.3	29.25	1.94	3.67	33	0.4	1
12	Cabicarbonate bath	6.7	31.33	4.50	-1.53	224.5	2.81	98.5
13	Caca.b, pre-aged	5.7	30.51	2.48	2.28	200	2.29	41
14	Caca.b, post-aged	6.9	29.34	1.99	2.26	34.5	0.5	1
15	Caca.b, pre-, post-aged	5.8	27.85	1.79	2.73	38	0.47	1
16	Caca, suction table	4.6	32.27	3.21	-4.34	221.5	2.63	149.5
17	Caca.st, pre-aged	4.1	29.88	1.52	0.31	97	1.22	8
18	Caca.st, post-aged	4.7	28.43	1.49	1.45	30.5	0.35	1
19	Caca.st, pre-, post-aged	3.8	28.81	1.49	3.68	30.5	0.37	1

Note: the values are an average of samples a and b.

least 1 cm away from the inked parts. As the influence of iron-gall ink is not strictly limited to the place where it was applied, a third sub-sample "border" was taken from the paper right next to the inked part. Therefore, twelve sub-samples from four differently treated samples have been analysed.

For analysis, 5 mg of sample material was used, except for all "ink" sub-samples, when preferably 10 mg were taken in order to improve the signal:noise ratio of the usually heavily degraded cellulose. Two sub-samples (T1paper and T1border) were only available in quantities of 2.5 and 3.5 mg respectively.

Only aged samples were analyzed for molecular weight distribution and carbonyl group content.

4. RESULTS AND DISCUSSION

4.1 Visual Assessment of Lavater Papers

The Lavater papers were visually assessed after aging. The lining paper was not trimmed off but used to hang the samples in the aging oven.

T0, untreated control: In the course of artificial aging, ink corrosion progressed and caused losses and diffusion of degradation products in non-inked areas (fig. 3).

T1, nonaqueous deacidification: The ink became darker after deacidification. During artificial aging the ink corrosion continued. Degradation products created a slight brown line at the border of the inked and non-inked areas. The Klucel lining is lifting and shows only limited adhesion (fig. 4).

T1G, lining with gelatin: After aging the adhesion was still good. The gelatin led to yellowing of the Japanese paper (fig. 5).

T2, lining with wheat-starch paste: During the lining process brown degradation products migrated into the rag paper and the polyester web support. After aging ink corrosion progressed. Degradation products caused a dark line in the non-inked paper (fig. 6).

T3, deionized water, lining with wheat-starch paste: After aging the ink border was brittle and showed losses of ink. On the back a diffusion of degradation products was visible.

Fig. 3. Paper T0, untreated control after aging.

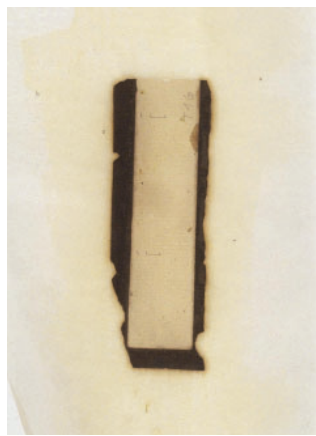Fig. 4. Paper T1, nonaqueous deacidification, lining with Klucel G, after aging.

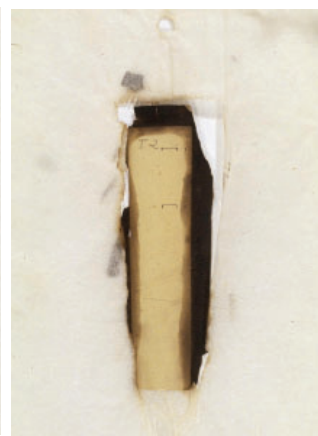Fig. 5. Paper T1G, lining with gelatin, after aging.

Fig. 6. Paper T2, lining with wheat-starch paste, after aging.

T4, calcium bicarbonate, lining with wheat-starch paste: Differences in wetting between degraded and sound paper resulted in tensions during treatment. Old tears in the ink parts widened and new tears appeared. There was a loss of a small area during treatment. During aging ink corrosion continued; the lower part broke off. Degradation products diffused into the paper (fig. 7).

T5, calcium phytate/calcium bicarbonate, lining with wheat-starch paste: Tensions created by the differences in water absorption between degraded and sound paper caused tears in the ink lines during treatment. After aging, adhesion of the lining was good. The condition of ink and paper appeared to be stable.

T6, phytate treatment with the addition of ethanol, lining with wheat-starch paste: Ethanol resulted in better wetting and less tension during treatment on the suction table. Some existing tears in the ink nevertheless broadened. One fragment became loose. After aging the sample appeared to be stable with regard to corrosion and lining.

T7, phytate treatment with addition of ethanol and gelatin sizing, lining with wheat-starch paste: After aging T7 is in good condition. There were no losses and no visible migration. The further progressing of paper degradation seemed to have been stopped (fig. 8).

A visual assessment of the inked ASA rag paper sample was not possible due to destructive mechanical testing of these papers.

4.2 pH Measurements

The surface pH of the untreated Lavater paper areas ranged between 4.6 and 6.5. After nonaqueous deacidification with MMC it was raised to 9.6 and only slightly lowered after aging. After deacidification with calcium bicarbonate and the combined phytate treatment, the pH was raised to values between 6.2 and 7. After aging the pH lowered by 0.5 to 1 points.

The surface pH of the inked areas on the Lavater papers ranged from 4 to 5.8 before treatment. Nonaqueous deacidification with MMC resulted in a rise to 7.9 at the highest and to a maximal difference of 2.1 after aging. The surface pH of the aqueous deacidified samples was significantly lower after aging. The pH values of all aged samples were within a similar range: 2.2 to 3.6. With regard to the inaccuracy of the surface measurements there were no significant differences among the different treatments. Nonaqueous deacidification caused the strongest differences in surface pH.

The cold extraction pH of the inked ASA rag paper samples (table 3) was 3.5 for the untreated control. Further aging did not result in further lowering of the pH. On the rag paper samples the pH of inked areas was raised after treatment to as high as 6.9. Treatment in a bath resulted in a higher pH than treatment on the suction table, with a difference of 0.5 to 2.2 points. There were no significant

Fig. 7. Paper T4, deacidification with calcium bicarbonate, lining with wheat-starch paste, after aging.

Fig. 8. Paper T7, combined calcium phytate/calcium bicarbonate treatment, lining with wheat-starch paste, after aging.

differences in pH between the combined calcium phytate/calcium bicarbonate and the calcium bicarbonate treatment alone. After one hundred cycles of aging (pre- and after aging) the pH of the calcium phytate sample was higher than the pH of the calcium bicarbonate sample, 6.1 to 5.8 respectively. The pH of the untreated control, aged for the same amount of time, was 3.5.

4.3 Color Measurements

After treatment nonaqueous deacidification resulted in the most pronounced color difference on the paper areas of the Lavater papers. The paper became darker, redder, and more yellow. Aging of the papers led to a decrease in lightness and to higher red and yellow values. After aging no significant differences between the treatments could be observed on paper areas.

After nonaqueous deacidification the ink on the Lavater papers was significantly darker (L★ -6.50). Aqueous treatments caused slightly lighter samples (+2). Aging led to a further increase in lightness. The a★-value was not significantly changed after treatment and after aging. After nonaqueous deacidification the b★-value was lower and remained so after aging. The MMC solution did not result in yellowing of the ink. T2, T6, and T7 have lower b★ values than the untreated control. T6 showed the fewest changes in color.

Nonaqueous deacidification resulted in the strongest color changes on the Lavater papers. The combined calcium phytate treatment with the addition of ethanol, T6, caused the smallest color changes.

The color measurements of the ASA rag paper samples are summarised in table 3. On the inked ASA rag paper samples the lightness of inked paper was lower after treatment in comparison to the untreated control. There are no significant differences due to the treatment variations. After one hundred RH-cycles of aging the treated inked paper was darker than the untreated aged control. On the treated samples the a★-value was higher than on the untreated control. On the aged samples there were no significant differences in the degree of redness between the control and treated samples after one hundred cycles. The b★-value of the untreated control and the treated inked papers increased after aging (one hundred cycles). After one hundred cycles the b★-value of the treated samples was lower than that of the untreated control. No significant differences were to be found among the treatments.

4.4 Tear Resistance, Tear Index, Folding Endurance of ASA Rag Paper Samples

The physical testing results can be found in table 3. The tear resistance of the phytate-treated inked rag paper was highest (451). After one hundred cycles of aging the samples bathed in calcium phytate (36.5) and calcium bicarbonate (38) have a higher tear resistance than the control sample (29). The tear index of the phytate-treated paper was also the highest. After one hundred aging cycles there were no significant differences between treated samples and the control. Folding endurance of the phytate treated sample was by far the highest, over 10,000 compared to 79.5 of the untreated control. After pre- and post aging the folding endurance of all samples was 1. The effect of aging on the mechanical properties of paper seemed to overrule the effects of the different treatments. The phytate treatment did not lead to lower mechanical strength of the paper.

4.5 Scanning Electron Microscopy (SEM) of Aged Lavater Papers

The fiber net was strongly fibrillated on the papers that were not washed: T0, T1, T1G, and T2. On T1 fine sediment could be found in inked areas. On T2G (gelatin) and T2 (starch) larger sediment could be observed locally. On T4, deacidification with calcium bicarbonate has resulted in the coating of fibers. The strongest visual differences were caused by the calcium phytate/calcium carbonate treatment: crystalline and needle-like formations were formed on the fiber surfaces (figs. 9–10).

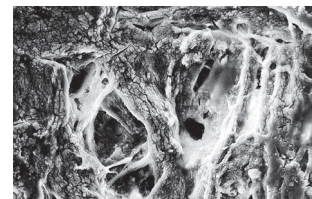Fig. 9. Paper T0, SEM image of paper surface, 1000x magnification.

Fig. 10. Paper T6, SEM image of paper surface, 1000x magnification.

4.6 Micro-XRF of Lavater Papers

The results of the μ-XRF investigations for the mounting papers T7, T0, T2, and T4 are summarized in figures 11-15. The diagrams present line scans for each paper. On the Y-axis the intensity of Fe-Kα-line is shown for each measurement, whereas the X-axis gives the distance of the measuring points in the paper area from the ink border (e.g. measuring point "0 mm" is the first point in the ink area, whereas the point "1.5 mm" shows a distance from the ink area of 1.5 mm). For further interpretation, diagrams are shown in detail until the first point in the ink area.

In general it could be shown that by applying calcium phytate nearly the same values for the x-ray intensities of iron (Fe-Kα-line) were found in the paper objects directly after the treatment as after their subsequent artificial aging (paper T7, fig. 12). This leads to the conclusion that the migration of the iron ions can be stopped by such a treatment. Furthermore, by applying an aqueous phytate treatment some iron ions have been removed.
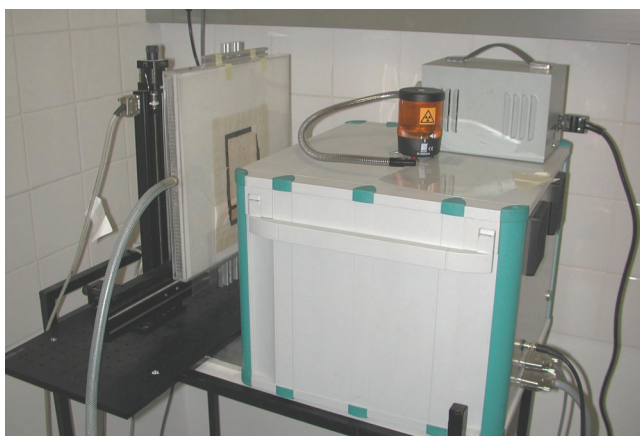
Fig. 11. Experimental μ-XRF set-up showing a Lavater paper analyzed in front of the transportable COPRA instrument.

Fig. 12. Line scan on Lavater object T7: comparison of the intensities of the Fe-Kα-line of the untreated (line T7_Fe), phytate-treated (line T7bh_Fe), and subsequently artificially aged sample (line T7alt_Fe).

In comparison to the phytate treatment, the untreated paper (T0, fig.13) showed an increased intensity of iron, i.e. by about 50%, after artificial aging near the paper/ink interface.

In figure 14 the results obtained for the paper T2 (lining using wheat-starch paste) are presented. A decrease in iron intensities after the treatment (line T2 bh_Fe) could be explained by the removal of iron ions during treatment. Artificial aging resulted in a strong increase in iron intensities in the non-inked area of the paper near the border of the inked area.

As depicted for paper T4 (deacidified using calcium bicarbonate solution on the suction table) in figure 15, iron ions were removed during the treatment. This is shown by a decrease in iron intensities after the treatment (line T4 bh_Fe) compared to the paper (line T4_Fe) before treatment. Subsequent artificial aging resulted in an increase in the intensity of the iron ions in the paper area within a distance of approximately 1.75 mm from the paper/ink border. This means that iron ions migrated from the ink area into the non inked area around paper/ink border during artificial aging, but iron intensities were not higher than the values of the untreated sample, as was the case for paper T2.

4.7 XANES of the Lavater Papers

The results of the measurements on papers T0, T1, T4, and T7 are summarized and compared in figure 16. It could be shown that for paper T7, treated with calcium phytate and deacidified with the calcium bicarbonate solution, mainly Fe^{3+} (absorption edge of Fe^{3+} at 7126 eV) was detected (Kanngießer et al. 2004). This can be explained by the fact that Fe^{2+} ions form a complex with (calcium) phytate and are subsequently oxidized to Fe^{3+} ions which stay bound to phytate. Due to its limited water solubility the formed complex (mixed phytate salts of Fe^{3+} and calcium)
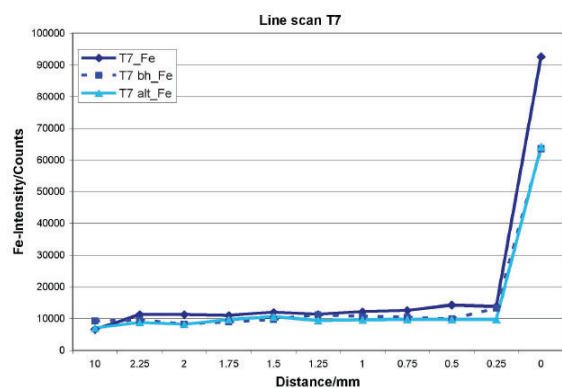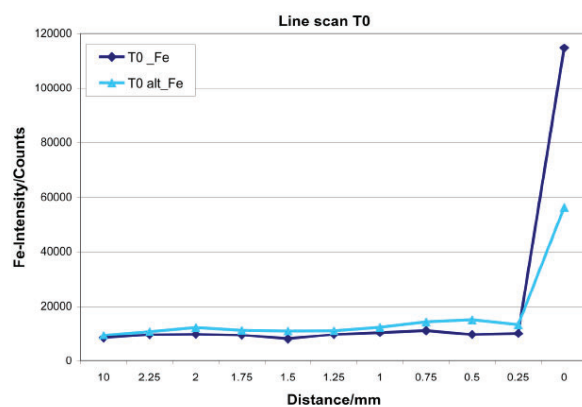

Fig. 13. Line scan on Lavater object T0 (control sample): comparison of the intensities of the Fe-Kα-line of the untreated (line T0_Fe) but artificially aged sample (line T0alt_Fe).
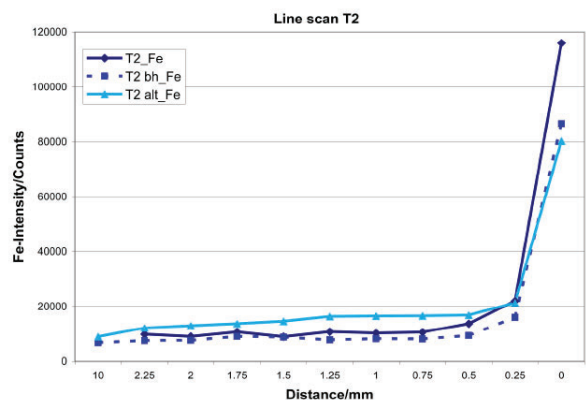

Fig. 14. Line scan on Lavater object T2: comparison of the intensities of the Fe-Kα-line of the untreated (line T2_Fe), treated (lining with wheat starch paste, line T2bh_Fe), and subsequently artificially aged sample (line T2alt_Fe).
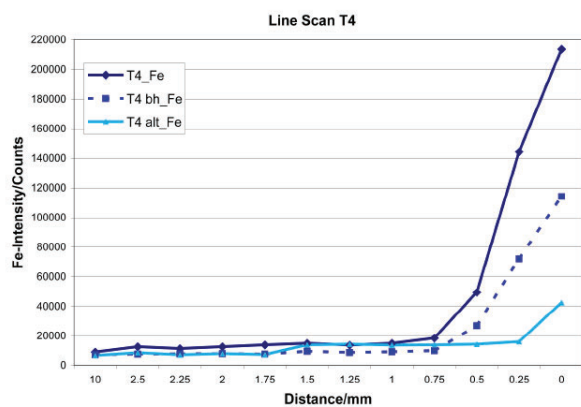
Fig. 15. Line scan on Lavater object T4: comparison of the intensities of the Fe-Kα-line of the untreated (line T4_Fe), calcium bicarbonate treated (line T4bh_Fe) and subsequently artificially aged sample (line T4alt_Fe).
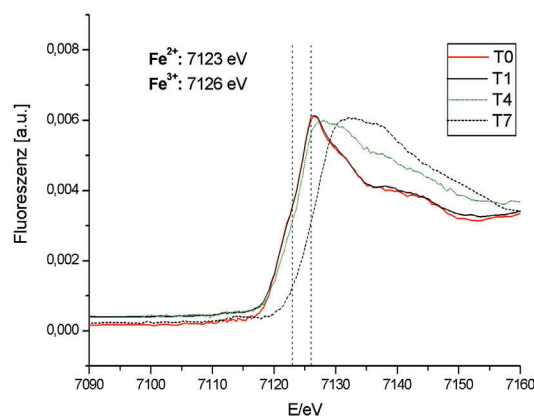

Fig. 16. Fe-K-XANES spectra of the control sample T0 and the treated and subsequently aged samples T1 (MMC), T4 (calcium bicarbonate), and T7 (calcium phytate).

precipitates on the paper fibres without showing catalytic activity (Neevel 1995).

In comparison to the phytate treated paper, the untreated aged paper T0, as well as the paper treated with methyl magnesium carbonate (MMC) T1, clearly showed an absorption edge at 7123 eV, attributed to Fe^{2+} ions.

For paper T4, deacidified using calcium bicarbonate solution, the absorption edge slightly shifted from 7123 eV to 7126 eV. This means that both oxidation states of iron, Fe^{2+} and Fe^{3+}, are present in the inked area.

Concerning the results discussed above, four years after the phytate treatment was applied to paper T7, the ink corrosion seems to be stabilised and stopped. In comparison, for the other objects analysed the degradation process still seemed to be going on due to the presence of Fe^{2+} as determined by XANES.

4.8 Photometric Detection of Carbohydrate Monomers and Oligomers Found in Aged Lavater Papers

The values (μg/mg) are the sum of monomer and oligomer sugars in total calculated on cellobiose; they are summarized in table 4. In the papers T0, T1, and T2 the highest amount of water-soluble cellulose degradation products was measured. In T1G, lined with gelatin, and all aqueous-treated samples significantly lower values of degradation products were detected. The inked piece of paper T2, lined with starch, contains by far the greatest amounts of these substances. Sample T7, treated with phytate, sized with gelatin and lined with starch, has the lowest quantities of degradation products. All other papers have similar values.

4.9 Analysis of Molecular Weight Distribution and Carbonyl Group Content of Selected Lavater Papers

Chain scission is an important parameter when evaluating the condition of cellulose and therefore of paper. The degree to which cellulose molecule chains are still intact can be monitored by measuring the molecular weight distribution (MWD) and calculating the average molecular weight parameters (Mw).

Table 5 gives a summary of all analyzed samples. In figure 17 (molecular weight distribution) the MWDs of all four paper sub-samples are shown. The best result for MWD and Mw was obtained for sample T7 (Mw=415.3 kg/mol). Sample T1 had the worst result when it comes to the shape of the MWD and Mw-graph (Mw=194.8 kg/mol). T0 and T4 showed moderate chain scission. Their Mws were 379.7 kg/mol and 315.7 kg/mol respectively. At

Table 4. Water-soluble cellulose degradation products of aged Lavater papers

NUMBER	PAPER (μg/mg)	INKED PAPER (μg/mg)
T0	31.8	39.9
T1	15.5	37.3
T1G	1.9	27.2
T2	27.9	100
T3	4.4	51.3
T4	5.2	44.6
T5	4.1	39.6
T6	5	30.1
T7	2.5	21.9

Table 5. Overview of results from carbonyl group and molecular weight determination

SAMPLE	Mw (kg/mol)	Mn (kg/mol)	REG calc.*	C=O (μmol/g)
T0paper	379.7	120.2	8.32	8.7
T0border	145.5	35.51	28.16	25.22
T0ink	60.05	12.55	79.68	155.06
T1paper	194.8	59.56	16.79	11.22
T1border	189.3	26.8	37.31	45.42
T1ink	evaluation not applicable			129.99
T4paper	315.7	60.88	16.43	23.34
T4border	243.9	38.43	26.02	34.74
T4ink	48.07	12.13	82.44	181.6
T7paper	415.3	67.18	14.89	14.74
T7border	evaluation not applicable			30.04
T7ink	54.97	11.97	83.54	99.73

*REG calc.: reducing end groups calculated using Mn

Fig. 17. Molecular weight distribution. Molecular weight distribution of all investigated samples at sub-sample "paper." T0paper and T7paper show a very similar distribution, but T7paper has lost its low-molecular shoulder. The same is true for T4paper, but the sample is more degraded. Sample T1 is the most degraded, but still has parts of the low-molecular shoulder though significantly shifted toward lower molecular weights.
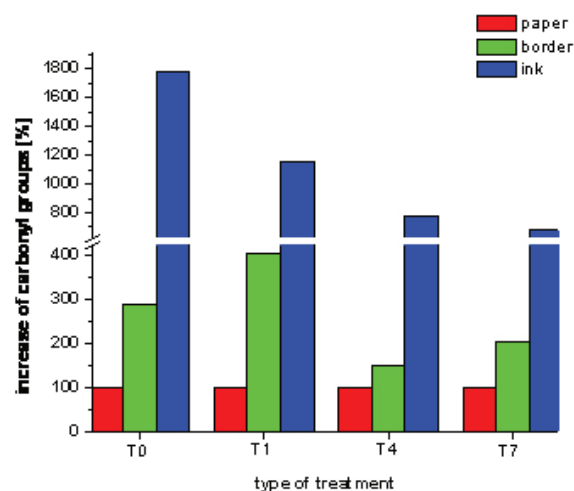
Fig. 18. Relative increase of carbonyl group content. Comparison of relative carbonyl group increase for all four samples on all sub-samples. In order to equal out uneven historic sample material, the sub-sample "paper" was set at 100% while the two other sub-samples "border" and "ink" were expressed in relative increase compared to "paper." All three treatments will reduce the development of carbonyl groups, T4 and T7 being the most efficient ones.

first glance it is surprising not to find the worst results for the untreated sample T0, but rather for sample T1 (non-aqueous deacidification).

To understand these results, the carbonyl group content has to be considered. Comparing the carbonyl group increase among the samples is not easy, as historic papers tend to be heterogeneous. In figure 18 (relative increase in carbonyl group content) the increase in carbonyl group content is shown in relation to the "paper" sub-sample; absolute values can be found in table 5. In the sub-sample T0paper, a carbonyl group content of 8.7 μmol/g was determined, and a content of 8.3 μmol/g of reducing end groups was calculated. This means that in the untreated paper sample T0paper mainly hydrolytic degradation took place, as the number of reducing end groups equals the total number of carbonyl groups detected. No further carbonyl groups are placed on the cellulose backbone. The picture changes when looking at the T0ink sub-sample. Here a carbonyl group content of 155.1 μmol/g was measured, while the theoretical amount of reducing end groups was calculated to be 79.7 μmol/g only. In this case, reducing end groups only contribute roughly 50% of the total amount of carbonyl groups detected, suggesting that the rest of them are situated along the cellulose chain and were introduced due to oxidation. In combination with a relatively high Mw of 379.7 kg/mol, it can be concluded that sub-sample T0ink mainly suffers from oxidation.

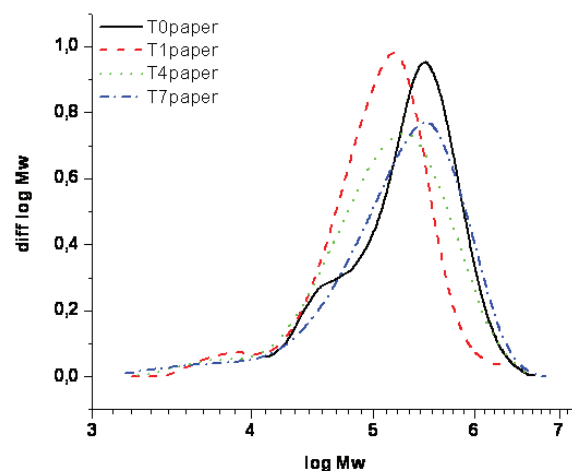Applying the same thoughts to the other samples, the results show clearly that treatment of paper T7 (calcium phytate/calcium hydrogen carbonate) yielded the best results, which is in good agreement with earlier results on the phytate treatment (Henniges et al. 2007). This sample had the highest remaining Mw and the lowest carbonyl group content, which was determined to be 99.7 μmol/g for T7ink. Calculated reducing end groups are 83.5 μmol/g. This indicates that oxidation was not completely stopped, but significantly inhibited as compared to sam-

ple T0. Obviously hydrolytic and oxidative chain scission were stopped due to the combined treatment of deacidification and complexing transition metal ions, just as further oxidation was reduced. In the T7paper sub-sample, almost identical values for measured carbonyl group content and calculated reducing end group content suggest that even though the value might be higher than for the T0 sample, this difference is probably not due to the treatment, since neither oxidation nor hydrolytic chain scission can be proved, but rather due to inherent differences in the sample paper. The effect of a gelatin sizing has not been analyzed within this project, but was addressed earlier (Potthast et al. 2007). No beneficial effect on iron-gall ink was detected in connection with gelatin sizing.

Both treatments without chelating agents are deacidification treatments: T1 is nonaqueous and T4 is aqueous. While T4 yielded better results for Mw (315.7 kg/mol versus 194.8 kg/mol), the nonaqueous treatment T1 yielded fewer carbonyl groups (11.2 μmol/g versus 23.3 μmol/g in the "paper" sub-sample and 130 μmol/g versus 181.6 μmol/g in the "ink" sub-sample). In sample T4 neither acid hydrolysis nor ß-elimination can be expected as the treatment solution was neither acidic nor alkaline enough to extensively catalyze these reactions. Thus, molecular weight is mainly retained as proven by the analysis. On the other hand, there is no means of stopping ongoing oxidation; therefore carbonyl group content increased. Considering the differences between the total measured amount of carbonyl groups and the calculated amount of reducing end groups underlines the assumption of ongoing oxidation. In treatment of T1 a highly alkaline solution was used which makes ß-elimination possible. When ß-elimination occurs, the cellulose chain is cleaved next to positions where a carbonyl group is present, resulting in severe loss of Mw as observed in sample T1.

Sub-samples T1ink and T7border could not be evaluated for molecular weight distribution.

The carbonyl group content on an untreated Lavater sample shows a very high increase in the inked area, above 100 μmol (fig. 19). When analyzing a slice of paper which covers up to 1 mm distance from the ink-paper border, about 40 μmol/g carbonyl group content is still obtained. The paper slice that is up to 2 mm distant from the ink application has a value of less than 15 μmol/g carbonyl group content. Here the influence of iron ions is of minor importance. These findings correspond to the XRF-analysis where higher iron intensities are found in untreated and treated Lavater papers within a distance of approximately 1 mm from the ink application.

5. CONCLUSION

Comparison and analysis of the Lavater mounting papers has shown that the combined calcium phytate/calcium bicarbonate treatment stabilizes corrosion caused by iron inks with a low copper content. Hydrolytic and oxidative chain scissions are stopped by deacidification and the complexing of transition metal ions after artificial aging. The phytate treatment inhibits additional oxidation of cellulose. The mechanical properties of paper are not negatively affected by the phytate treatment. After humid artificial aging there is no significant migration of iron ions into non-inked paper areas. The addition of ethanol to the phytate solution results in better penetration which diminishes tensions during treatment and enhances complexing.

Gelatin sizing seems to have a positive effect on paper considering the low amount of cellulose degradation products in aged paper, but it has no complexing effect on the iron-gall ink. After aging the degradation of paper is not prevented by the gelatin treatment.

On the Lavater papers washing and deacidification alone did not stop ink corrosion. As a method of local reinforcement wheat-starch paste showed the worst results. During artificial aging under humid conditions iron ions can migrate in papers lined with Klucel G and gelatin. Gelatin shows the best adhesion in local reinforcement. Nonaqueous deacidification does not prevent ink corrosion. Deacidification with methyl magnesium carbonate can cause a color change of the ink. A highly alkaline treatment can lead to chain scission decreasing the molecular weight of the cellulose.

The phytate treatment is an aqueous treatment with all the risks associated with the use of water. Even when per-
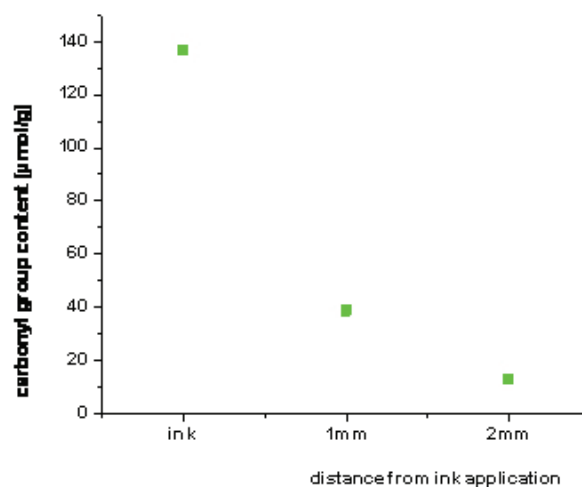

Fig. 19. Carbonyl group content on untreated, aged original sample material. On paper with ink application the carbonyl group content increases to very high values above 100 μmol. When analyzing a slice of paper that covers up to 1 mm distance from the ink application, about 40 μmol/g carbonyl group content is still obtained. The paper slice that is up to 2 mm distant from ink application has a value of less than 15 μmol/g carbonyl group content. Here, the influence of iron ions is of minor importance.

formed with the utmost care on the suction table and with the addition of ethanol, severely degraded papers can be mechanically damaged. Papers rendered extremely fragile as a result of the catalytic activity of iron ions usually cannot endure such mechanical stress.

This brings us back to the initial question: to treat or not to treat? According to the reported results, the phytate treatment is a good option for objects with iron-gall ink corrosion for stopping further degradation, provided that the objects can endure an aqueous treatment and that the changes brought about by the treatment are acceptable.

MATERIALS

Ecospray
Arts et Conservation
 75 rue Rateau,
 Urba Parc, Bat. B, 93120
 La Corneuve, France
 artetco@aol.com

Japanese paper *Usumino* B5, pure *kozo* paper, 12 g/m^2
Japico Feinpapier
 Rasmussengasse 2, 1210
 Vienna, Austria
 www.japico.at

Klucel G, hydroxypropyl cellulose ether
Gabi Kleindorfer
 Aster Str. 9,
 Kapfing, 84186
 Vilsheim, Germany
 www.gmw-gabikleindorfer.de

Photographic gelatin Rousselot demi dure, Type B, viscosity 6.99 cp (3%, 40°C), 285 Bloom
Rousselot SAS
 10 avenue de l'Arche, 92419
 Courbevoie Cedex, France
 www.rousselot.com

NOTES

1. Iron-gall ink: 400 mL gum arabic solution (78.5 g/L), 49.2 g tannin (95% Aldrich), and 42 g iron sulfate (FeSO$_4$ x 7H$_2$O) were diluted to 1000 mL with distilled water.

2. Methyl magnesium carbonate solution (MMC): 8 g of magnesium, 100 mL methanol absolute. After the magnesium dissolves, carbon dioxide gas is added until the solution is clear (1-2 hours).

3. Calcium bicarbonate solution: 1 g calcium carbonate, 1000 mL deionized water. Carbon dioxide gas is added until the solution is clear; pH 5.94.

4. Calcium phytate solution: 1.7 mL phytin acid (Aldrich), 0.33 g calcium carbonate, 500 mL deionized water. Some drops of concentrated ammonia were added until a pH of 6 was reached.

5. The μ-XRF instrument was built within the EU-project COPRA (A Compact Roentgen Analyzer), project no. SMT4-CT98-2237: Prof. Dr. Koen Janssens, Guido Vittiglio, University of Antwerp, Belgium; Dr. J. Heckel, Dr. P. Klinger, SpectroAnalytics, Kleve, Germany; Prof. Dr. M. Schreiner, Academy of Fine Arts Vienna, Austria.

6. The components of the μ-XRF instrument COPRA are: Mo x-ray tube (Oxford Instruments, type XTF5011), high voltage generator (Spellman, XRM 50P50), polycapillary (x-ray Optical Systems Inc., Albany, Model 629-01), silicon drift chamber detector (Röntec XFlash LT Serie), optical microscope (Opto, Zoom 100D), and a CCD camera (JAI-2040).

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