Technical Note on Treatment Options for Iron Gall Ink on Paper with a Focus on Calcium Phytate

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This technical note presents treatment options for works on paper inscribed with iron gall ink. The note consists of a flow chart with accompanying text that summarizes key points regarding the evaluation, examination and treatment options available for iron gall ink on paper. It is presented here as a précis of treatment considerations and is intended to be used as a practical laboratory tool.

La présente note technique indique les mesures de traitement des œuvres sur papier contenant de l'encre ferro-gallique. La note comporte un diagramme et un texte explicatif qui résume les principaux points des mesures d'évaluation, d'examen et de traitement disponibles pour les œuvres sur papier contenant de l'encre ferro-gallique. Elle constitue un précis des considérations de traitement et devrait être utilisée comme outil de laboratoire pratique.

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Introduction

Treatment protocol for works on paper inscribed with iron gall ink has evolved over the last 15 years. Research has shown that the calcium phytate/calcium bicarbonate method, first suggested for treating ink-corroded manuscripts by Neevel in 1995, is an effective aqueous method to prolong the lifetime of ink corroded objects.^{1,2,3} The treatment has become a standard treatment option for works with iron gall ink in paper conservation labs and studios.

Even so, the examination, evaluation and treatment of works with iron gall ink involve a complex set of procedures. Several very comprehensive treatment protocols have been published that are extremely useful to conservators for detailed description of each treatment step, as well as information pertaining to the risks and benefits of each option.^{4,5,6} This technical note provides a succinct summary of possible examination and treatment steps in the form of a visual map or outline⁷ (see following page) with explanatory text and relevant references below. The purpose of this note is to help experienced paper conservators when treating works with iron gall ink. It is not a substitute for comprehensive reading or training in the treatment of objects with iron gall ink.

Preventive Measures

The use of an aqueous treatment for iron gall ink will have side effects, either visible or invisible, which may affect the long term preservation of the object.^{8,9} Furthermore, the conservator should be cognizant that some information that is inherent in the material aspect of the artifact such as the ink and paper will be altered as a result of these treatments.¹⁰ Therefore, preventive measures (careful handling, interleaving, copying and/or cool dry storage), local repair, mechanical stabilization and minimal intervention are the preferred alternatives to any aqueous or non-aqueous treatment of works on paper inscribed with iron gall ink.

Artifact Examination

The decision to treat is undertaken after an examination of the artifact in conjunction with a thorough evaluation of the risks and benefits of any treatment. The conservator must consider if the associated risks outweigh that of no treatment or the problem of inherent vice posed by the iron gall ink. Even if a treatment is deemed beneficial, its viability is determined by the following factors:

- the presence of historical elements that cannot be removed
- its format and/or condition of the artifact
- the presence of elements such as signatures, notations, stamps or seals
- the availability of expertise, time and equipment to carry out the treatment

These factors help to determine the feasibility as well as the nature and extent of any interventions.

Diagnostic Procedures

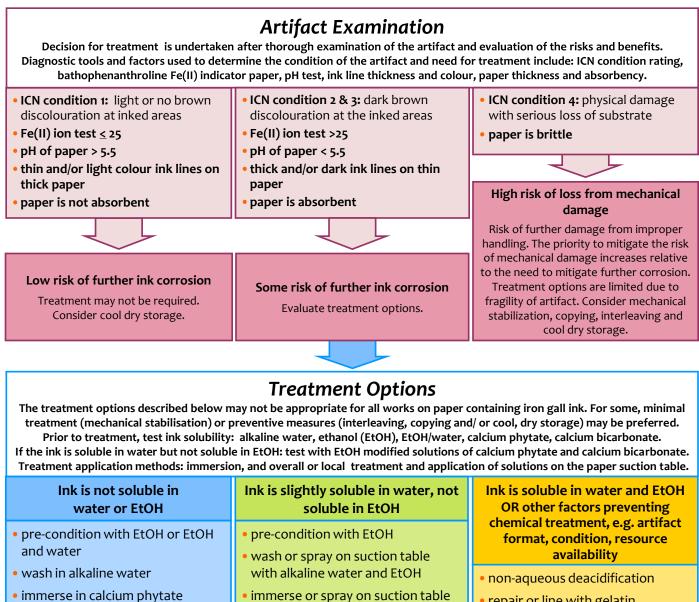
An important part of the decision whether to treat an object is based on results from the examination and evaluation of the ink and paper substrate to estimate the risk of further ink corrosion. The diagnostic tools and factors used to evaluate the condition of the artifact and the ink include:

- ICN condition rating for paper objects with iron gall ink based on visible progress of degradation published in 2000^{11,12,13}
- Bathophenanthroline Fe(II) indicator paper to detect the presence of iron(II) ions^{11,14,15}

The test is an indicator of potential risk of oxidation catalyzed by corrosive free Fe(II) ions. Inks that test very positive,¹⁶ indicating a lot of free Fe(II) ions, are potentially

Treatment Options for Iron Gall Ink on Paper

This flow chart is a summary of treatment options for iron gall ink on paper with a focus on calcium phytate. It highlights key treatment steps and presents treatment tips. It provides experienced paper conservators with a visual map or outline of examination and treatment steps to consider when treating works with iron gall ink. It is not-a substitute for literature review and training in the treatment of objects with iron gall ink. Refer to the conservation literature for detailed description of treatment protocols.



- immerse in calcium bicarbonate
- partially dry on suction table and finish drying in a blotter stack or dry only in a blotter stack
- size with gelatin
- repair or line with gelatin remoistenable tissue or solvent reactivated tissue

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- immerse or spray on suction table with EtOH diluted calcium phytate
- immerse or spray on suction table with EtOH diluted calcium bicarbonate
- partially dry on suction table and finish drying in a blotter stack or dry only in a blotter stack
- repair or line with gelatin remoistenable tissue or solvent reactivated tissue

 repair or line with gelatin remoistenable tissue or solvent reactivated tissue

Treatment Tips

To prevent lateral migration of soluble acids and iron ions, **AVOID** humidification or local wetting of objects with iron gall ink.

Fe(II) ion indicator paper should be used to monitor completeness of phytate treatments.

Highly acidic or alkaline conditions can cause colour change in iron gall inks. To avoid changes in ink colour, the pH of treatment solutions, and of the artifact after treatment, should be kept between pH 5.0 – 8.5. more corrosive than inks that test mildly positive. The risk of corrosion is greater for papers that are thin and/or highly absorbent.

- pH of the inked area and the paper¹⁷
- quantity of ink on the paper (ink line thickness)¹⁷
- thickness and absorbency of the paper¹⁷

Treatment Options

Solubility Spot Tests

After an examination of the ink and paper where diagnostic procedures indicate there is risk of further ink corrosion and treatment is deemed beneficial, spot tests for ink solubility should be carried out to determine the appropriate treatment procedure. Solubility tests are not standardized and paper conservators use various methods and techniques.^{4,5,6,18}

Initially, water followed by ethanol, ethanol/water solutions, and finally solutions of calcium phytate and calcium bicarbonate should be tested. If the ink is soluble in water but not in ethanol, then water and ethanol and ethanol modified solutions of calcium phytate and calcium bicarbonate should be tested. Results from these tests will determine the various treatment options.

Treatment Solutions

If the ink is not soluble in water or ethanol, then washing in alkaline water¹⁹ followed by immersion in calcium phytate and calcium bicarbonate is an option.

If the ink is slightly soluble in water but not ethanol, then ethanol and alkaline water mixtures can be used to wash the object followed by immersion in ethanol modified calcium phytate and ethanol modified calcium bicarbonate.

The effectiveness of ethanol modified calcium phytate treatments in protecting paper with iron gall inks was laboratory tested at the Canadian Conservation Institute. The results showed that ethanol modified calcium phytate is a beneficial treatment for works on paper inscribed with soluble iron gall ink. When used, it must be followed by deacidification such as ethanol modified calcium carbonate in order to achieve full protection from iron gall ink corrosion. The results also show that repeated applications of ethanol diluted calcium phytate can accumulate more phytate, thereby offering more protection against the recurrence of Fe(II) ions.^{20,21}

If the ink is soluble in water and ethanol, then aqueous treatments are not an option.

Treatment Application Methods

Treatment application methods to consider include immersion as well as treatment on the paper suction table (either applied overall or locally). Local application of calcium phytate and other treatment solutions, with an ultrasonic mist or by brush, may be an option when there are isolated areas of iron gall ink to treat.

Surface Sizing

If the ink is not soluble in water, the object can be sized with gelatin by applying the size with a soft brush. Research has shown that gelatin size may offer some protection against iron gall ink corrosion by binding transition metal ions and inhibiting their migration when they are exposed to high humidity conditions. Gelatin may also protect the paper and the ink against degradation caused by oxidation.²²

Drying

To avoid local tensions during drying, works on paper with iron gall ink must be dried as homogeneously as possible. One option is to pre-dry the object on the suction table and then transfer it to a blotter stack or to place it between natural felts under pressure. Spraying the object with ethanol on the suction table will facilitate an even and fast drying process.

Non-aqueous Interventions

When the ink is soluble, or if resources are unavailable or the artifact's format and/or condition prevent aqueous treatment, it is still possible to consider non-aqueous interventions as well as interleaving.

For example, it is possible to consider non-aqueous deacidification through the use of a product such as the Bookkeeper® Spray System and magnesium ethoxide,²³ and/or the use of local mending of ink corroded areas with remoistenable tissue. There is active research into the use of various antioxidants in non-aqueous solvents for treatment of inks and pigments containing iron and copper.²³ Their use on original material may be considered when the benefits of treatment are supported by further research.²⁴ Recent publications have shown that gelatin, re-activated with 25:75 water:ethanol, can be used to prepare remoistenable tissue for repair.^{25,26}

Any local repairs on iron gall ink documents should be executed with as little moisture as possible as too much humidity may cause the iron(II) ions and acids in the ink to migrate to the surrounding paper.²⁷ Using repair tissues which can be reactivated with solvent, such as those coated with Klucel-G, can lower the risk of ink migration.²⁸

Interleaving with paper impregnated with a deacidification agent and an antioxidant is an option that has been studied and shown to have merit. This should be considered for works where chemical treatments are not a viable option. Both the benefits and the risks are expected to be lower compared to chemical intervention.^{29,30}

Conclusion

This technical note is a practical lab tool for experienced paper conservators treating works on paper with iron gall ink. It summarizes treatment options, highlights key treatment steps and directs the user to detailed information and treatment guidelines in the conservation literature through the references. It is not a substitute for the complex procedures involved in the examination, evaluation and treatment of works on paper with iron gall ink. Neither does it replace a comprehensive literature review or training in the treatment of objects with iron gall ink.

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