TECHNICAL SPECIFICATION

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Effectiveness of paper deacidification processes

Efficacité des procédés de désacidification du papier

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 46, *Information and documentation*, Subcommittee SC 10, *Requirements for document storage and conditions for preservation*.

Introduction

Archives, libraries and similar institutions store written and printed documents which they are obliged to retain on a permanent basis for cultural reasons and, in some cases, in order to meet legal requirements.

Often, the condition of these documents is endangered for a number of reasons. One of these is related to the manufacturing process used for more modern types of paper.

In the industrial age, paper-making processes underwent significant changes. One of the processes affected was sizing, which, in industrial processes, was achieved by mixing additives into the fibre suspension before shaping the sheets. These additives included acidic substances like aluminium sulfate. The reaction of the sizing agent eventually leads to formation of free acids. The acids act as a catalyst for the hydrolysis of cellulose, making the material brittle. Climatic influences aggravate this process, air pollution and cellulose degradation processes are a further source of acid in paper.

Another factor for paper stability is the raw material itself. For centuries, paper was made of textile fibres like linen, hemp or cotton rags which rather deliver stable, long-chain cellulose. The search for a more abundant raw material led to the invention to produce pulp out of wood by a grinding process. The resulting ground wood paper still contains most of the lignin and hemicelluloses, in addition to cellulose. The low pulp purity and the mechanical process causing a partial cutting of fibres lead to a much weaker paper. Compared to the older rag papers, ground wood paper is also less stable on the long run.

The problem of paper degradation due to acid has developed into a tremendous problem for archives and libraries. In addition to the processes for deacidifying single sheets, such processes having been used in conservation for a long time, the past few decades have seen new developments in technical processes which can be used on a large scale to retard the further decay of cultural assets as bound volumes and single sheets ("mass deacidification").

The aim of deacidification is to appreciably improve the life expectancy of paper. This is achieved by adding an alkaline substance to neutralize existing acid and slow down future acidic degradation for at least some time (buffering, alkaline reserve). Deacidification cannot improve the actual physical properties of the paper, but in combination with proper storage, it can slow down further decay.

Without validated analytical methods, it is not possible to assess whether a paper has been deacidified, or to what degree deacidification has been successful. This Technical Specification compiles the suitable measurements.

Effectiveness of paper deacidification processes

1 Scope

This Technical Specification defines test methods and minimum requirements for paper deacidification processes regarding their effectiveness and consistency.

It is applicable for all large scale processes which offer deacidification of acid documents made of printed or hand-written paper.

Possible negative side effects of deacidification processes on the treated objects are not the subject of this Technical Specification. However, some general recommendations for how to cope with these side effects are given in Annex A.

It is not specified either, which types of paper objects can be treated by large scale deacidification methods. Whatever currently available deacidification method is used, some objects might be excluded from treatment to avoid mechanical damage to paper and bindings or other unwanted side effects. The provider of the deacidification treatment should inform the customer about the limitations of the chosen method.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 535, *Paper and board — Determination of water absorptiveness — Cobb method*

ISO 536, *Paper and board — Determination of grammage*

ISO 776, *Pulps — Determination of acid-insoluble ash*

ISO 5351:2010, *Pulps — Determination of limiting viscosity number in cupri-ethylenediamine (CED) solution*

ISO 5626, *Paper — Determination of folding endurance*

ISO 5630-5:2008, *Paper and board — Accelerated ageing — Part 5: Exposure to elevated temperature at 100 degrees C*

ISO 6588-1, *Paper, board and pulps — Determination of pH of aqueous extracts — Part 1: Cold extraction*

ISO 9184-1, *Paper, board and pulps — Fibre furnish analysis — Part 1: General method*

ISO 9184-4, *Paper, board and pulps — Fibre furnish analysis — Part 4: Graff "C" staining test*

ISO 10716, *Paper and board — Determination of alkali reserve*

3 Terms and definitions

For the purpose of this document, the following terms and definitions apply.

3.1
accelerated ageing
artificially induced ageing under laboratory condition by increasing temperature and sometimes changing humidity or exposure to light in order to accelerate chemical reactions in paper like hydrolysis or oxidation to simulate processes usually occurring under natural condition but at a much slower speed

3.2
alkaline reserve
compound like calcium or magnesium carbonate neutralizing acids in paper

3.3
average degree of polymerisation
average number of anhydroglucose units (monomers of cellulose) in the cellulose macromolecule

3.4
batch process
deacidification process for a definite quantity of documents

3.5
continuous process
deacidification process for an indefinite quantity of documents

3.6
deacidification
neutralization of the organic and inorganic acids in the paper and deposit of an alkaline reserve as buffer against any subsequent acidic activity on paper

3.7
extract pH
value obtained in a water extract after the paper has been extracted under defined condition.

Note 1 to entry: Value measured with a glass electrode immersed in a definite quantity of water in which paper is dispersed in small pieces.

3.8
folding endurance
common logarithm of the number of double folds required to cause rupture in a strip of paper

3.9
mass deacidification
process of paper deacidification on a large scale

3.10
process validation
securing an operation according to preset parameters determined at processed objects

3.11
routine monitoring
monitoring carried out at regular intervals during normal operations

3.12
side effects
any unintended consequence caused by the execution of a treatment process

3.13
test paper
paper with characteristics defined in this Technical Specification, which is deacidified together with original documents and then analysed

3.14
uniformity of deacidification
homogeneous distribution of the alkaline reserve and pH across the entire sheet and within whole book blocks

4 Principle

Specified uniform test papers are treated together with customer's documents in a deacidification process. Afterwards, the test papers are examined using standardized test methods. The test papers are acidic and similar in their properties to common paper qualities produced in the period from around 1870 onwards. The usage of such papers ensures reliable results and allows comparing different batches, deacidification methods and treatment plants.

NOTE It is to be emphasized that successful tests according to this Technical Specification cannot guarantee that all documents treated in the process are deacidified to the same degree as the test papers. The result of a deacidification treatment strongly depends on the properties of the treated object, such as porosity, thickness, sizing, coating and acidity of the paper, etc. Therefore, it is impossible to guarantee that certain pH levels and alkaline reserve amounts are achieved in each object by the deacidification treatment. A passing of the tests means, however, that there is a high percentage of successfully treated objects.

5 Requirements

5.1 General

This Technical Specification defines test methods for "process validation" (initial testing) and "routine monitoring". Process validation is used to prove that a technique fulfils its defined purpose. Routine monitoring is used to check that the effectiveness determined by process validation is being achieved in the course of the actual work. Routine monitoring, therefore, is based on process validation.

For "process validation", extended test procedures should be carried out before and after accelerated ageing of the samples, including measurements of pH value, alkaline reserve, uniformity of deacidification and degree of polymerisation.

For "routine monitoring", alkaline reserve of the test papers is examined.

5.2 Sampling

5.2.1 Material

Both process validation and routine monitoring are performed using samples of test paper, some of which are deliberately not subjected to the deacidification process serving as a reference.

Table 1 — Test paper

	Test paper (ground wood-free)	According to ISO standard
Fibrous material	Fully bleached sulphite pulp with hemicelluloses	ISO 9184-1, ISO 9184-4
Kaolin filler	12 %–15 % kaolin	ISO 776
Grammage	80 g/m^2	ISO 536
Surface finish	none	none
Sizing	approximately Cobb 60' 20 g/m^2	ISO 535
Type of sizing	Alum rosin sizing $Al_2(SO_4)_3$	none
Surface sizing	none	none
Extract pH	approximately 5	ISO 6588-1
Optical brighteners	none	none
Acidity, given as negative alkaline reserve	approximately −0,3 % $MgCO_3$	[a]

[a] Since no ISO standard is available, the German technical specification Zellcheming ZM IV/58/80 "Prüfung von Papier, Karton und Pappe…" can be applied. See Reference [2].

5.2.2 Procedure

All samples should be examined within four weeks after treatment has been completed (including post treatment measures).

Before the paper is examined, any loose residues occurring as a side effect of the deacidification process should be removed by brushing.

5.3 Process validation

5.3.1 Frequency of sampling

A complete process validation is required every four years and, additionally, following

— changes to the process technology,

— changes of chemical components or their supplier, or

— changes of the test paper for routine monitoring.

The process validation is valid for all treatment devices of a production site that use the same process and technology.

5.3.2 Sample quantities and preparation of samples

The process validation is performed using identical test papers (i.e. same production batch). A quantity of 32 (+4, if folding endurance is included) test sheets, size A5 or larger, is needed for the necessary testing (see Table 2) of one treated sample set. Four treated sample sets are necessary for the process validation. The untreated sample set included, the sum of test sheets for one complete process validation is therefore 148 (+20, if folding endurance is included).

Table 2 — Tested qualities and numbers of test sheets needed

Tested quality	untreated test paper (one untreated sample set)		treated test paper (one treated sample set)	
	unaged	aged	unaged	aged
pH value (cold extraction)	4	4	4	4
Alkaline reserve	4	4	4	4
Uniformity of deacidification			12	—
Cellulose DP	2	2	2	2
Folding endurance (optional)	(2)	(2)	(2)	(2)

For batch processes, the test papers should be placed into bound volumes which are thicker than 3 cm and feature a size of at least A5. For the first sample set, 32 (+4) test papers are placed evenly throughout the bound volume starting from page number 10.

The test papers should be centred vertically and placed as close to the spine as possible. The test papers should not extend outside the book block. The second sample set is prepared the same way, but placed in a different position in the treatment chamber. The third and fourth sample set should be treated on another day, and if applicable, in another treatment device. The positions of the samples in the chamber should be documented adequately.

NOTE Service providers can supply a constructional drawing of the deacidification device with the report and mark the positions of the books containing the test papers.

For continuous processes, the test papers of one sample set should be treated alternating with sheets of original items. After further treatment of 100 sheets of original items, the second sample set should be treated to the same pattern as the first. The third and fourth sample sets should be treated according to the first two sample sets, but on a different day, and, if applicable, in another device.

5.3.3 Test methods and minimum requirements

5.3.3.1 Accelerated ageing

Perform accelerated ageing of 10 (+2) test sheets of each of the four sample sets and 10 (+2) untreated test sheets as described in ISO 5630-5:2008, Clause 4 to 9.2.

Test tubes selected for this study shall be perfectly gas-tight and large enough to accommodate paper strips pre-cut for further measurements. It is required to perform aging for all samples simultaneously, in the same laboratory oven, using one type of a glass tube for all samples.

NOTE To ensure perfect airtightness of testing tubes, the following steps could be taken:

— original tube caps supplied with glass tubes could be exchanged for caps made of material with higher resistance to mechanical and thermal stresses (e.g. polyphenylsiloxane);

— sealing material – PTFE or silicone gaskets and o-rings should be avoided, fluoroelastomers are advisable (e.g. Viton);

— tightening of the tube with the use of the dynamometric wrench equipped with a tube cap holder, to ensure good repeatability of obtained sealing.

5.3.3.2 pH value

The pH value has to be measured in an aqueous extract as described in ISO 6588-1.

The average results and the average and the relative standard deviations should be given for treated paper with and without ageing, and the results should be expressed to two significant digits.

The measured pH of the paper following deacidification has to be higher than 6,5 (before accelerated ageing).

NOTE 1 The pH value of an aged sample will normally be lower compared to those of the non-aged sample. For a given paper, ageing after deacidification should only lead to a small reduction of its pH value. It is possible that the pH value measured after accelerated ageing will level out at around 6,5, even though an alkaline reserve is still present. This is particularly true of the pH value on the paper surface which is usually one unit lower than the pH value of the cold extract. Under these conditions, however, this kind of paper can still be described as being neutral.

The pH value discussed here applies solely to the described test papers. If original papers are examined as well, special agreements on an acceptable final pH value should be reached with the customer, as the achieved pH value depends very much on the original composition of the paper.

NOTE 2 In addition to this measurement of aqueous extract pH value, measurements of surface pH value are sometimes performed. The surface pH measurement is a faster method compared to extraction pH measurement to judge the pH value of a paper. If applied correctly (see Reference [3]), surface pH measurements also allow on-site measurements of original books and documents in libraries and archives and can also be used to follow the stability of deacidification on a longer timescale. However, surface pH measurement has its limits. It works well for acidic to neutral papers and also gives reasonable data until about pH 9. Usually, surface pH measurement has been successfully used with immersion treatments. Surface pH measurement may fail to give reliable results when larger amounts of alkaline reserve deposits are present at the paper surface and the solubility limit is reached.

5.3.3.3 Alkaline reserve

Determine the quantity of alkaline reserve of each of the four sample sets as described in ISO 10716. For determination of the dry matter content, it is in deviation to ISO 10716 sufficient for the purpose of this Technical Specification to weigh about 1 g to the nearest 0,001 g.

