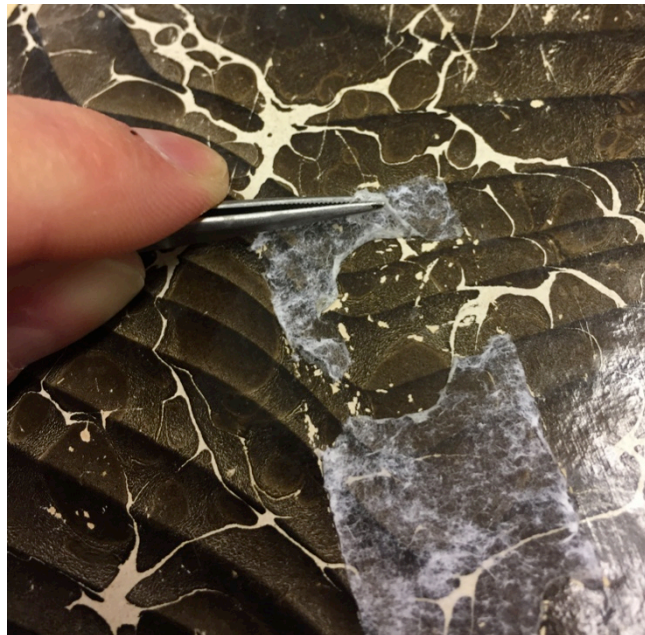




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# REMOVAL OF PRESSURE-SENSITIVE LABELS FOR LIBRARY APPLICATION

A Study of the Use of Agar as a Solvent Gel



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Removal of Pressure-Sensitive Labels for Library Application:  
A Study of the Use of Agar as a Solvent Gel

## **ABSTRACT**

The purpose of this study was to investigate pressure-sensitive labels for library application, techniques, and considerations for their removal from paper substrates. The aim was to use information from the literature to design a method for the removal of pressure-sensitive labels from paper covered half bindings using agar, commonly known as agar-agar, as a solvent gel.

The study is divided into two parts: a literature study with the aim to compile information relevant to the purpose of the study, and a case study for the removal of barcodes from three books made available by the Humanities Library of Gothenburg University Library. To analyse the barcodes, stereo microscopy, Attenuated Total Reflectance – Fourier Transform Infrared (ATR-FTIR) spectroscopy, and spot tests were conducted. Agar was subsequently tested and evaluated as a solvent gel in a practical removal.

Analysis of the barcodes on the studied books showed that they are built-up of three layers with a pressure-sensitive adhesive closest matching that of a poly(methyl acrylate) emulsion. A 2% agar gel was divided into three batches and mixed with 20% ethanol, 20% 1:1 acetone:ethanol, and 29% ethyl acetate. While all of the removals were considered somewhat effective, the ethyl acetate agar gel gave the best results with the least damage to the surface layer of the paper. Based on the evaluation it could be concluded that agar shows great promise as a solvent gel.

The study showed that issues to take into consideration can be roughly divided into three aspects: 1) the purpose of the label, and historic circumstances related to the label; 2) whether the label obscures or contains information crucial to the interpretation of the object as an entity; and 3) damages caused, or potential damages that might arise from its continued adherence to the substrate.

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## **Appendix 1: Materials and suppliers**

# 1. Introduction

Labelling has almost always been an aspect in the history of the book. With the early book being regarded as a symbol of status, the markings of provenance through ex-libris and other markings added to the binding of the book proclaimed wealth. The 1800s saw the emergence of fast-growing libraries, and the open shelving system and constant movement of the books through patrons required proper labelling and declarations of provenance. In the 1970s the invention and implementation of electronic systems for libraries made it possible to electronically link material to patrons through barcodes and later through Radio-Frequency Identification (RFID)-tags (Evans 1983). Ex-libris<sup>1</sup>, labels declaring shelf position or classification, colour-coded labels, barcodes, and RFID-tags are some of the countless labels used frequently in the working routines of many libraries. Since the labelling system is meant to provide the libraries with a security aspect, one of the main conditions is that the labels attached need to “remain attached permanently and can be removed only with difficulty” (Library of Congress 2016, p. 1).

Many of the larger libraries in Sweden divide their collections. Material before 1850 is considered a part of the heritage collections, while material after 1850 is considered a part of the ‘new’ collections. The general idea is that heritage collections do not get labelled, while the newer collections are. This delimitation contributes to a very static view of the collections, which is not necessarily the truth. Parts of the newer collections may still be of great value and even considered rare, and as such, in need of a status change. For conservator-restorers working with library material, the act of labelling constitutes a compromise between the assignment to preserve the material as it is, and the library’s duty to provide and make their collections available. New labelling systems through barcodes and RFID-tags are usually regarded as high quality and safe, but few resources are invested to evaluate labelling material and their sustainability in regards to the library materials and their needs. Over time, problems with pressure-sensitive labels become apparent, and it will mostly affect the new material that is today almost always down prioritised, but that might constitute the heritage of tomorrow.

A few of the most observed problems include: drying of the adhesive which leads to loss of the label; adhesive creeping out on the sides of the label and adhering adjacent pages leading to loss of material when pulled apart; and, discolourations, such as yellowing of the adhesive, and transparency in the paper substrate (Lloyd 2010). Problems arising from the deterioration of inherent properties in pressure-sensitive labels and their composition as well as the need for a status change might evoke the question of label removal, a question that has been sparsely researched in the field of paper conservation.

The interest in pressure-sensitive labels and their removal arose from the author’s own interest in the compromise between preservation and availability of library material, and the grey zone that labels comprise. During the author’s in-program conservation internship at the National Library of Denmark in the winter of 2016-2017, some of the damage caused by labels were made apparent. A literature search revealed a lack of information about pressure-sensitive labels, and their removal aimed at conservator-restorers. This study was conducted as a way for the author to develop a higher understanding of pressure-sensitive labels and their removal both theoretically and practically.

## 1.1. Purpose, aim, and question formulation

The purpose of this study is to present information regarding pressure-sensitive labels and how to remove them in an attempt to broaden the existing information on pressure-sensitive tapes and adhesives available to conservator-restorers. This would help provide libraries, archives, and other institutions and individuals working with collection care and conservation with information and simple means of developing methods on how to work with similar problems. The aim of this study is to

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<sup>1</sup> A bookplate, usually a print or a pasted label, declaring provenance of the book.

implement the researched information in a case study where the author attempts removal of self-adhesive barcodes from paper-covered library books using agar, commonly known as agar-agar, as a solvent gel. The inspiration for working with agar as a solvent gel was obtained from the article 'The use of agar as a solvent gel in objects conservation' (Scott 2012).

## 1.2. Definition of problems and issues

- a) What kind of pressure-sensitive adhesive is used on the studied material and how are the labels built up?
- b) What are the advantages and disadvantages of working with agar as a solvent gel in the removal of pressure-sensitive labels in paper conservation?
- c) What are some of the standpoints that need to be taken into consideration in the process of removal of pressure-sensitive labels for library application?

## 1.3. Methods and materials

This study was conducted in two parts: a literature study, and a case study. The first part was conducted as a literature study, with a focus to research pressure-sensitive labels, tapes and adhesives, as well as relevant conservation literature. The second part was carried out as a case study. The aim of the case study was to implement the information gained from the literature study and design a method for the removal of barcodes from three paper covered half bindings<sup>2</sup> using agar as a solvent gel. Stereo microscopy, Attenuated Total Reflectance – Fourier Transform Infrared (ATR-FTIR) spectroscopy, and spot tests were conducted to analyse material composition of the barcodes.

Due to the many advantages of agar, it was chosen as a technique for removal in the case study. The choice was based on the author's interest to test and evaluate the material, but also based on the author's presumption that the removal carried out in the case study would require a gentle technique such as a poultice or a gel. Limits of current facilities and material also played a role in the decision of technique. Potential risks connected to the use of agar will be acknowledged and discussed further by the author.

## 1.4. Restrictions

This study was limited to looking at pressure-sensitive labels for library application. This includes later attachments of labels to library material such as barcodes, RFID-tags, and other mark-up labels declaring provenance and shelf location, excluding original label attachments. The delimitation focuses the study on labels produced and attached in the second part of the 1900s.

The study aims to provide the reader with overall information about pressure-sensitive labels and their removal, but for the case study carried out, a uniform material including only one type of pressure-sensitive label and paper substrate was tested to provide a more qualified basis for evaluation. The study was also restricted to testing agar as a technique for removal. More extensive testing of techniques for removal, as well as testing material, should be able to provide a more comprehensive evaluation and information about pressure-sensitive labels, but due to limitations in time, this will not be possible to carry out. The author would also have wished to carry out a more extensive chemical analysis of agar to examine its potential and suitability as a poultice material in paper conservation.

## 1.5. Ethical considerations

Removal of pressure-sensitive tapes and labels from paper substrates always poses a great risk for the object. The endeavour in conservation, to do the least possible impact on an object, often clashes with

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<sup>2</sup> A type of hardback bookbinding where the spine and corners are bound in one material, for example cloth or leather, and the sides of the cover is bound in another material, for example paper or cloth.

an aim for removal of foreign material. In the removal of pressure-sensitive products from paper, these two aspects need to be carefully considered and evaluated. Even with the best techniques and intentions, it is often far from possible to carry out the procedure without affecting the object, whether it is the loss of surface sheen or surface layer, bleeding of soluble media, staining, or change in paper grain. It is therefore important to consider if a removal is really necessary before initiating treatment.

Due to the condition of the objects used in the case study, and an initial assessment of risks accompanied by the removal of the barcodes, a complete risk-free removal was estimated almost non-existent. Potential risks and damages that may befall the objects were discussed with the loaning institution and subsequent treatments for removal were accepted. The practical work was carried out with great respect to the objects, and the author based the practical considerations and performances, as much as possible, on information gained during conservation training, as well as the following literature study. Based on these notions, the removal of barcodes conducted in the case study followed mainly two of the notions set up in the preamble to the American Institute for Conservation of Historic and Artistic Works (AIC) code of ethics.

IV. The conservation professional shall practice within the limits of personal competence and education as well as within the limits of the available facilities.

V. While circumstances may limit the resources allocated to a particular situation, the quality of work that the conservation professional performs shall not be compromised.

*(AIC Code of Ethics and Guidelines for Practice 1994)*

Further considerations regarding books objects and labelling are discussed in section 2.3. 'Considerations before treatment' and 5.2. 'Ethical aspects'.

## 1.6. Definitions

In this section a few of the most commonly used terms in the study are presented.

Pressure-sensitive product (PSP)	Products with pressure-sensitive traits, for such as tapes, labels, and self-adhesive films.
Pressure-sensitive adhesive (PSA)	The author has chosen to follow the definition used by Benedek: "Pressure-sensitive adhesives are adhesives that form films exhibiting permanent tack, and display an adhesion which do not strongly depend on the substrate." (Benedek 2004, p. 1)
Pressure-sensitive label	Label exhibiting pressure-sensitive traits.
Pressure-sensitive tape	Tape exhibiting pressure-sensitive traits.
Self-adhesive label	Used synonymous to pressure-sensitive label, but the term acknowledges the fact that most modern pressure-sensitive labels do not require pressure to adhere to a substrate.
Elastomer	An elastic polymer. The elastomer makes up the majority of a PSA.
Tack	The inherent stickiness and bond-formation ability in an adhesive
Cold flow	The movement of an adhesive at room temperature, causing the adhesive to penetrate a porous substrate as far as it allows.

Sol

The semi-solid colloidal solution of agar. Precursor to the formation of rigid gel.

## 2. Literature study

The focus of the literature study is to provide a broad introduction to pressure-sensitive labels, and various conservation strategies for their removal from paper substrates. In the following section, the previous research that forms a framework for the literature study is presented.

### 2.1. Previous research

Pressure-sensitive tapes and their removal constitute one of the major problematic areas within the field of paper conservation; hence a lot of research and development of techniques for removal has been conducted to deal with the problem. Due to the age of many objects and their repairs in question for conservation, a focus in case studies and other object related studies has been on the removal of protein-based and rubber-based adhesives, and not on modern pressure-sensitive adhesives. Two of the more inclusive and prominent articles in the field are ‘Pressure-sensitive tape and techniques for its removal from paper’ (Smith, Jones, Page & Dirda 1984) and ‘A closer look at pressure-sensitive adhesive tapes: update on conservation strategies’ (O’Loughlin & Stiber 1992). These quite similar articles cover a short history of pressure-sensitive tapes, deterioration, and techniques and strategies for their removal from paper. The focus is on pressure-sensitive tapes, and the discussions are kept wide, and left somewhat open with regard to potential treatments and choices. These articles serve as a base for building up a general framework regarding pressure-sensitive labels and how to address the problem as a conservator-restorer.

The *Paper Conservation Catalog* serves as a practical reference guide to the various fields within paper conservation. The chapter ‘Hinge, tape, and adhesive removal’ (O’Loughlin & Stiber 1994) introduces a comprehensive overview of the removal of adhesives. O’Loughlin and Stiber discuss material compositions of natural gums and adhesives as well as synthetic polymers, solvents, techniques for removal, and also bring up some of the ethical considerations when it comes to removal. The chapter is written as a checklist, and some of the topics are more thoroughly covered than others. For a more practical approach to the removal of pressure-sensitive labels it serves its purpose, but for closer examination into materials, the reader needs to conduct complementary research.

Complementary research into the field of pressure-sensitive labels was carried out outside the field of conservation and in to that of pressure-sensitive technology. For a more thorough insight on material composition and build-up of pressure-sensitive labels and adhesive two books were used: *Development and Manufacture of Pressure-Sensitive Products* (Benedek 1999) and *Handbook of Pressure Sensitive Adhesive Technology* (Satas 1989b).

Inspiration for this study came from the article ‘Label removal from deteriorated leather-bound books’ (O’Hern & Pearlstein 2013). This study is one of few within the paper conservation field that addresses the problem of pressure-sensitive labels on books. The aim of the study is to test and evaluate different techniques for removal of pressure-sensitive labels from leather-bound books. Although the substrate is leather, many of the techniques used are based on techniques commonly used for tape and adhesive removal in paper conservation, and the analytical and practical conduct of the authors will be used as inspiration for this study.

Inspiration for working with agar as a solvent gel poultice came from two articles: ‘The use of agar as a solvent gel in objects conservation’ (Scott 2012) and ‘Analyse of agarose, carbopol, and laponite gel poultices in paper conservation’ (Warda, Brückle, Bezúr & Kushel 2007).

In the following sections pressure-sensitive labels, considerations before treatment, choice of solvents, and techniques for removal of pressure-sensitive labels are discussed. The objective is not to cover the

entire field, but rather to give a general overview of the material and how the conservator-restorer can work with objects in regard to the removal of pressure-sensitive labels.

## 2.2. Pressure-sensitive labels

Pressure-sensitive products (PSPs) comprise a broad industrial field including all products with pressure-sensitive traits. PSPs are generally divided into three sub-categories containing pressure-sensitive tapes, pressure-sensitive labels, and miscellaneous PSPs such as protective films (Benedek 1999). Today most modern PSAs do not require applied pressure to adhere to a surface, and, as such, pressure-sensitive labels and other PSPs are sometimes referred to as self-adhesive. The terms are used somewhat synonymously, but the term pressure-sensitive has gained more recognition and will thus be used throughout the study.

The following sections present a short history of the development of pressure-sensitive labels and adhesives, as well as a generalisation of some of the properties that apply to pressure-sensitive labels, tapes and adhesives. Knowledge of history and the application of labels, their compositions as well as different stages of deterioration enable the conservator-restorer to formulate a hypothesis regarding the identification of a pressure-sensitive label and its adhesive. This could help narrow down options when it comes to selecting appropriate treatments for removal.

### 2.2.1. History and application

The first use of PSAs was for medical tapes and dressings, and in 1845 Horace H. Day patented a soft adhering cloth bandage, coated with a composition of natural rubber and tackifier resin. Natural rubbers were the preferred elastomer used for the early PSAs and to get a product with desirable properties, natural resins, waxes, and fillers were mixed to produce a heterogeneous, yet tacky product. In 1855 Stanton Avery developed and introduced the self-adhesive label and in 1935-1936 industrial pressure-sensitive labels were introduced to the market. The development of pressure-sensitive labels in the 19<sup>th</sup> century caused a split in the PSP industry, causing the manufacture of tapes and labels to take two separate routes. This separation continued up until late 20<sup>th</sup> century when the industries, due to merges and conglomeration, reunited once again. (Benedek 1999; Satas & Satas 1989)

Natural rubbers continued to rule the PSA industry up until World War II when a shortage of resources, and rising costs forced the industry to look for alternative solutions. Rubber had been synthesised as 1,3 butadiene in the early 20<sup>th</sup> century, but with inferior properties as a PSA, in comparison with natural rubbers. In 1918 and 1939 respectively, a copolymer of styrene and butadiene (S/B) and polyisobutylene were developed and introduced as elastomers. This marked the introduction of various synthetic rubbers to the PSA market. (Hickman 1989; O'Loughlin et al. 1992)

Acrylic acids were synthesised as early as 1843, but their utilisation as PSAs came over 100 years later. The first United States patent for the use of polyacrylics in the manufacture of PSAs was obtained in 1959 by the Minnesota Mining and Manufacturing company (3M). By the 1960s, acrylics and vinyl acetate/acrylic copolymers had started to expand onto the PSA market and together with the synthetic rubbers, natural rubbers had been almost altogether replaced. (O'Loughlin et al. 1992; Satas 1989a)

Pressure-sensitive labels have a wide field of application. Labels in general have an intended function to identify a product, or mediate information, and as such they find application everywhere in everyday life (Satas et al. 1989). For library application, pressure-sensitive labels are used as mark-ups for books and other library material. Early uses include water-soluble gummed or protein-based labels such as ex-libris or other markings declaring provenance. The introduction and implementation of computer technology at libraries in the 1970s marked a new era for labelling within book collections. Barcodes that saw frequent uses in commercial fields reached the libraries, enabling the formation of a computer systemised link between patron and the borrowed objects (Evans 1983). At the beginning of



the 21<sup>st</sup> century, systems such as the already established RFID were implemented into library routines as a way, among other things, to make self-checkout systems easier. These later pressure-sensitive label additions include both natural and synthetic polymer-based labels used to mark shelving, provenance, as well as barcodes, RFID-tags, and other labels meant for analogue or digital identification. Labels used for library application often differ from labels used for commercial application. In commercial application the labels do not usually have the requirement of strength in application, and some ephemerality can therefore be tolerated. This is not the case for labels used for library application, where the time frames for availability and accessibility are much longer. The requirements for shear force are therefore much higher, resulting in a pressure-sensitive label that will not be able to be removed without causing substantial damage to the substrate (O'Hern et al. 2013).

## 2.2.2. Build-up and composition

Pressure-sensitive labels have a laminate multiweb construction, that is, labels are manufactured as sheet-like constructions that require another sheet, a so called release liner, to protect the adhesive layer and prevent the adhesive from bonding to adjacent surfaces. Due to the need for a separate release liner and the use of labels as information carriers, pressure-sensitive labels constitute one of the most complex build-ups of the PSPs. The general pressure-sensitive label is built-up, and requires a minimum of three layers to function, but is mostly more complex than that. These layers consist of a carrier or a backing, an elastomer constituting the adhesive, and a release liner (see Fig. 1a) (Benedek 1999). For labels used for library application, or where the printed information on the carrier needs to be protected to withstand abrasion, a fourth layer is usually added. This layer usually consists of a plastic laminate top layer or coating of the carrier (see Fig. 1b).

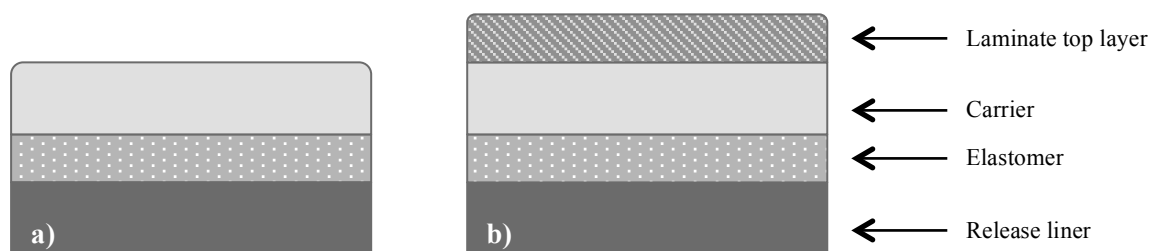


Fig. 1: Schematic build-up of a) simple pressure-sensitive label and b) pressure-sensitive label with laminate top layer.

### 2.2.2.1. Carriers

The carrier usually acts as the face-stock of the label with a primary function as information carrier. As such, the carrier must possess mechanical resistance and dimensional stability enough to be printed and mechanically 'die cutted' i.e. shape-cutted during manufacture. Materials used for carrier stock depend on both economical factors, as well as end-use, and range from paper stock to plastic films, laminates, cloth, and metal foils. (Benedek 1999)

### 2.2.2.2. Elastomers

The primary function of a PSA is to have high enough tack for the adhesive to successfully bond to a designated surface. If the label is of a removable kind, like removable decals and sticky notes, the adhesive bond should be weak enough for the label to be able to be peeled of without leaving residue adhesive behind on the surface. For a permanent label for library application, the bond should be strong enough to resist peel and shear forces. Depending on the end-use of the label, the pressure-sensitive adhesive formulae can be modified accordingly.

PSAs can be roughly divided into two major groups: rubber-based adhesives and acrylic-based adhesives. Rubber-based adhesives include natural rubber as well as a wide variety of synthetic rubbers. Elastomers mainly used as PSAs are natural or reclaimed rubber, styrene-butadiene rubber (SBR), polyisobutylene/butyl rubber, higher polyvinyl alkylethers, Buna *N* (butadiene-acrylonitrile

copolymer), higher polyacrylate esters, styrene-butadiene-styrene (S-B-S), styrene-isoprene-styrene (S-I-S) block copolymers (Kendall 1989), monomers of alkyl acrylates, and 4-17 carbon methacrylates. (O'Loughlin et al. 1994)

### 2.2.2.3. Additives

As mentioned above, the compounding of an elastomer with additives such as plasticisers, tackifiers, antioxidants, fillers and curing agents can be done to improve the properties of a PSA. For a list of commonly used additives see Table 1. The compounding of additives with an elastomer is most prominent in rubber-based adhesives where the elastomer displays insufficient inherent properties as an adhesive, and poor resistance to heat and light degradation. Composition and degradation of rubber-based adhesives are somewhat dependent on each other. The low inherent tack constitutes a need for additives such as tackifiers and plasticisers. Poor heat and light resistance of rosins and other derivatives used as tackifiers and plasticisers cause yellowing. To counteract this problem fillers and antioxidants that help mask discolourations and stabilise the material are added.

PSAs based on synthetic acrylic polymers have the advantage over rubber-based PSAs due to their inherent tack, which reduces the need to compound the adhesive with tackifiers and plasticisers. The adhesive formulae can therefore be made out of homogenous co-monomers leading to saturation and pre-crosslinking of the polymer. If a product does not display enough strength, additional cross-linking sites can be compounded, with the addition of polar comonomers, or with metallic ions. To create a more economical product or to change the properties of acrylic-based PSAs, tackifiers such as rosin esters, polystyrenes, or other materials are sometimes added. Plasticisers such as phosphate, phthalate, sulphonamide, and butyl benzyl phthalate may also be added. (O'Loughlin et al. 1992)

**Table 1:** List of additives commonly used in PSAs to enhance properties. List compiled from information from O'Loughlin and Stieber (1992, pp. 282-284) and Kendall (1989, pp. 219-239).

<b>Tackifiers</b>	<b>Plasticisers</b>	<b>Fillers</b>	<b>Antioxidants</b>
Gum rosin	Mineral oil	CaCO <sub>3</sub>	Used mostly in rubber-based adhesives to stabilise against heat and light degradation.
Rosin esters	Liquid polybutene	TiO <sub>2</sub>	
Other rosin derivatives	Liquid polyacrylates	ZnO	
Polyterpene resin	Phthalate	Clay	
Coumarone-indene resin	Butyl benzyl phthalate	Pigments	
Oil-soluble phenolic resin	Lanolin	Aluminium hydrates	
Petroleum hydrocarbon resin	Phosphate		
Polystyrene	Sulphonamide		

### 2.2.3. Ageing and degradation of pressure-sensitive adhesives

Pressure-sensitive tapes and labels have been used widely within archive, museum, and library collections. The material has many advantages when it comes to keeping broken pieces and tears together as well as fast application and the ability to retain information, for example inventory numbers or barcodes linking to catalogue information. But often there has been a failure to take notice of the different rates of deterioration. Even though labels and tapes have been used on most material groups available in collections, organic materials such as paper are most susceptible to the degradation of adhesives (O'Loughlin 2001).

The ageing and deterioration of rubber-based PSAs has been thoroughly investigated, and described as a three-stage process in a conference paper by Feller and Enck (1982), but the information presented below has been gathered from secondary sources. The first stage of the deterioration process is the oxidative induction period. During this stage very little chemical alterations take place and the adhesive is somewhat easy to remove. The second stage is characterised by increasing oxidation and chain-scissions of the rubber polymer. This causes the adhesive to go sticky and oily, leading to adhesive migration and subsequent translucency in the paper adherend. The adhesive can still be removed in this stage, but with increasing difficulty. In the third stage, the PSA residues start to cross-

link causing both adhesive and carrier to yellow and go hard and brittle (see Fig. 2). Brittleness causes loss of adhesive properties, and delamination between adhesive residues and carrier will occur. Rubber-based PSAs in the third stage of deterioration require increasingly polar solvents to be removed, often with high associated risks for the object under treatment. Common oxidation products in the degradation process of rubber-based PSA's are carbon dioxide (CO<sub>2</sub>), water, formic acid, and formaldehyde. The cross-linked material has also been shown to contain small amounts of peroxides, which in turn lead to acid hydrolysis of the substrate and adjacent papers. (O'Loughlin et al. 1992; Smith et al. 1984)

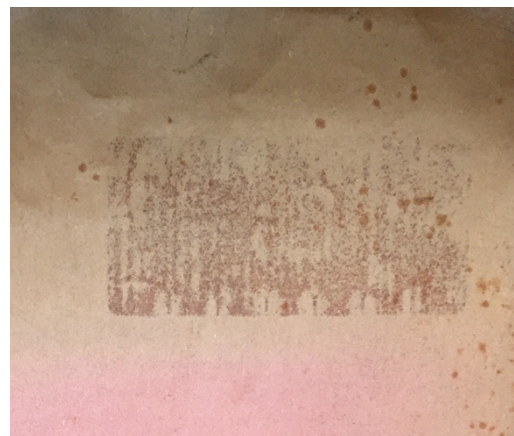
As mentioned above, the inherent properties of acrylic polymers enable them to be created from homogenous co-monomers leading to saturation and pre-crosslinking of the polymer. This makes acrylic-based PSAs resistant to ageing and discolouration, but in turn also causes them to be more resistant towards solvents commonly used in paper conservation. As a result, most acrylic-based PSAs can only be swollen by solvents, or scraped off mechanically. (O'Loughlin et al. 1992)

A common trait for all PSAs is that they must exhibit cohesive elasticity when bonding to a substrate, but still exhibit enough stability to resist creep or flow at room temperature or under applied stress (Dahlquist 1989, p. 97). Failure in doing so results in 'cold flow' i.e. the movement of adhesive at room temperature; a property causing the adhesive to penetrate a porous substrate as far as it allows. This together with the addition of polar comonomers or metallic ion cross-linking sites, leads to an increase in adhesive–adherend strength over time, which makes acrylic-based PSAs increasingly difficult to remove with age. Cold flow can also cause translucency of the paper substrate (see Fig. 3). Even though most acrylic polymers are inherently stable against degradation, compounding with additives may enhance certain properties desirable by end-users or manufacturers, however, this can also give rise to problems with discolourations of rosins, migration and volatilisation of plasticisers leading to possible bleeding of adjacent ink. (O'Loughlin et al. 1992; Smith et al. 1984)

As seen above all pressure-sensitive adhesives, no matter how inherently stable they are presumed to be, suffer from degradation that will affect the paper substrate. Rubber-based adhesives, both natural and synthetic ones, tend to discolour by yellowing, seep into the substrate, and go brittle causing loss of the carrier. Acrylic-based adhesives have a tendency to be subjected to cold flow and subsequent translucency of the substrate. The longer an adhesive is left on the substrate the harder it will be to remove, and as such it is important to try to remove it as soon as possible. Before a removal is attempted, it is important to consider some circumstances regarding the object and the attached pressure-sensitive label in a risk assessment. Some considerations before treatment are discussed in section 2.3.



**Fig. 2:** Damage caused by old rubber-based pressure-sensitive label. New smaller label showing signs of poor adhesive–adherend bond.



**Fig. 3:** Acrylic-based adhesive from the pressure-sensitive label on the other side has caused translucency in the substrate.

## 2.3. Considerations before treatment

The reasons why a label is attached to an object can vary widely, and since removal is a risk in itself, it is important to take time to consider a few aspects before attempting a removal. An important factor in conservation is to be able to maintain the original appearance of an object as far as possible. In light of the associated risks in removal, the conservator-restorer has to be able to carry out a risk analysis in regards to both the object and the attached label.

One of the first things to consider is the purpose of the label. Pressure-sensitive labels may be attached to objects for varying purposes and might be a part of the original historic appearance of the object such as ex-librises and other labels declaring provenance or shelf location. Older label attachments can provide interesting and relevant information about older classification systems used, or the shift in ownerships through ex-librises. As such they should as far as possible be regarded as an integrated part of the history of the object. Another factor to consider is whether the label obscures information. Text relevant to the object, early notations, designs, and pictures may be obscured with the attachments of pressure-sensitive labels. In these cases there is a great risk associated with removal. Pressure-sensitive labels placed on top of media might cause bleeding of some inks through contact between the PSA and the media (O'Loughlin et al. 1992).

Assessing a removal includes the evaluation of the condition of the object as well as the label. As noted earlier, the degradation of adhesives causes damage such as discolouration and degradation of the paper substrate. It is therefore necessary for the conservator-restorer to put the object and the label in context to observed damage or damage that is considered likely to occur with prolonged contact between the object and the label. These damages include further discolorations, translucency, and further degradation of the object, but also a diminishing adhesion leading to adhesive failure. The condition of the paper substrate will also affect the decision whether a removal is considered possible. A porous, soft paper or a brittle degraded paper may be at higher risk at being damaged through delamination during a removal than a good quality paper or a highly calendered paper (O'Loughlin et al. 1992).

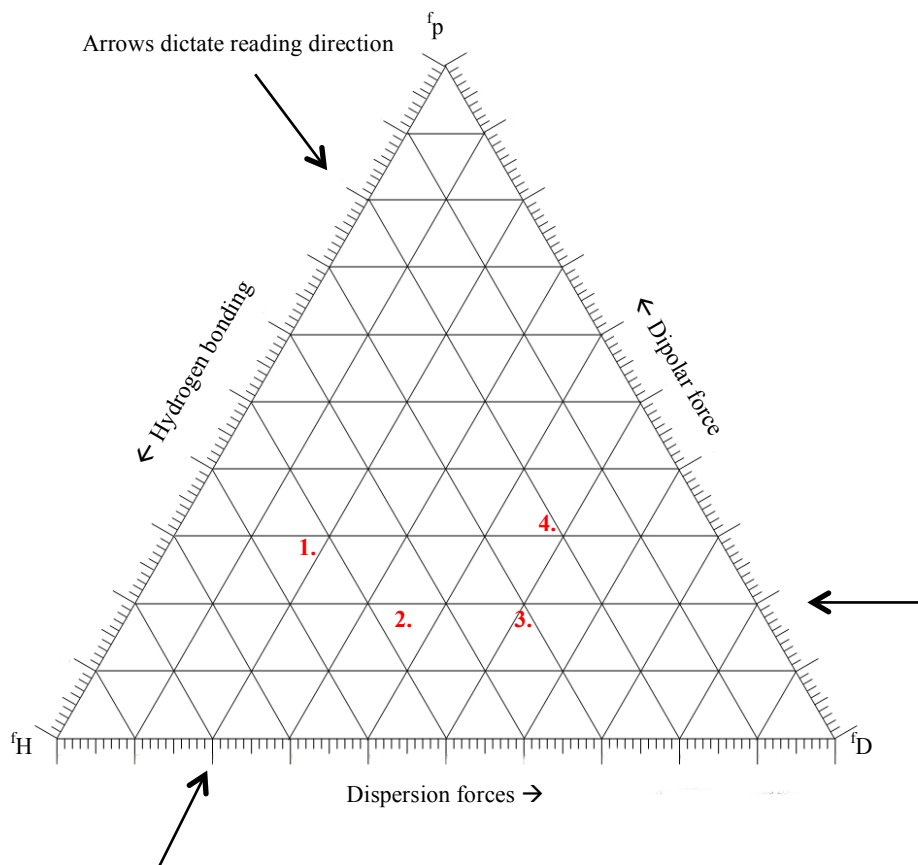
A problem with pressure-sensitive labels for library application is that they have generally been attached in great numbers on what is regarded as new material (1850-). The placing can at times be seen as unguided, and without thought. But the value for the new material lies almost solely in the information that can be gained from studying the material, and, as such, it has been wrongfully bereft of other fundamental values such as economical, cultural, emotional, and even age value. In the decision-making regarding removal of pressure-sensitive labels, the four latter values often play a crucial part which opens up for conservation of material with a heritage status, while new material gets set aside, and prioritised lower. This behaviour towards the new material can generally be explained by what is known as the rubbish theory, a theory created by Michael Thompson. The rubbish theory divides objects in three different stages of their lives. In the 'transient phase' objects are somewhat new and can be acquired easily, but their value is starting to decline. After a sharp decline the object will enter the 'rubbish phase'. Here the value is lost until it once again increases in a 'durable phase'. The new material at libraries often falls under the categories of transient or rubbish, which in turn has led to, careless handling when it comes to routines regarding labelling. Today there is often failure to notice that there is a high potential that the material that is today regarded as new, transient, or rubbish, will in the near future enter a durable phase where the careless handling today will be disproved. (Ashley-Smith 1999)

Considerations and risk assessment before treatment builds up a framework of how to handle and work with the object, and the upcoming treatment for removal of pressure-sensitive labels. But before initiating the removal, a decision regarding choice of solvent must be made. The following section will focus on some of the strategies that can be used for the selection and adaptation of solvent for the removal of pressure-sensitive adhesives.

## 2.4. Choice of solvents

Choice of a suitable solvent is often founded on experience and trial and error-based working methods. A conservator-restorer can greatly benefit from formulating and testing a hypothesis regarding the identification of the adhesive to be removed. Formulating a hypothesis, or even being able to identify the basic material and chemical composition of an adhesive, enables the conservator-restorer to make a more informed choice of solvent, based on the premises of ‘like dissolves like’.

Another method commonly used to enable a more systematic testing is the use of Teas chart. Teas chart is a triangular chart where solvents are plotted based on their molecular interaction: polar forces, hydrogen bonding, and dispersion forces (see Fig. 4). The chart can also be used to calculate the properties that mixed solvents in various concentrations will adapt. (Smith et al. 1984)



**Fig. 4:** Teas chart. The triangular chart makes it possible to calculate the molecular interaction according to the hydrogen bonding ( $f_H$ ), dispersion forces ( $f_D$ ), and dipolar forces ( $f_p$ ). Marks 1-4 represent solvents to be used in the case study. 1. Water, 2. Ethanol, 3. Ethyl acetate, 4. Acetone.

In the case of large molecular structures such as polymers commonly employed in PSAs, the premise of like dissolves like lacks some of its usefulness. The choice of a polar solvent for the removal of a polar polymer adhesive will not be sufficient to predict the effect and outcome of a treatment. The problem with polymer structures is that they are often composed of large, long-chain molecules, therefore, their solubility parameters cannot be sharply defined (Horie 1987). Closer attention must be paid to the intermolecular forces of the solvents in comparison to the adhesive to be removed. For the removal of larger polymer structures, the solvation energy in a specific solvent must be very high. The introduction, and compounding of metallic ions or polar comonomers into PSAs may also alter the solubility parameters of a PSA. This means that a solvent that could be utilised to cause a pure PSA to

become soluble, might after the introduction of above mentioned additives only cause a slight swelling (O'Loughlin et al. 1994)

Choice of solvent must also suit the object to be treated. Paper objects impregnated with resins or rosin size have an inherent weakness against polar solvents. Introduction of polar solvents may cause staining by lateral movement of degradation products. This problem mostly affects poor quality groundwood paper. Similarly, short fibred papers are at risk of mechanical damage due to solvents breaking up the fibres further, resulting in extreme brittleness of the paper object (O'Loughlin et al. 1992). It is also important to take health issues into consideration in the choice of solvent. Some solvents have hazardous effects, and, as such, a fume hood should be used to extract volatile gases, something that may obstruct the treatment.

Deciding upon a solvent might also depend largely on the technique to be used for removal. In the following section, some of the more common techniques for removal are briefly presented.

## 2.5. Techniques for removal

The techniques that will be discussed below include mechanical removal, immersion, direct local application, suction table, and poultices. More comprehensive focus is placed on agar gel that will be used in this case study. These only make up a selection of techniques that can be used for removal, but comprise some of the most commonly used.

Conducting a removal might be desirable, but the risk of skinning, or damaging the paper structure poses a great threat to the integrity of the object. In these cases it is always good to conduct a thorough discussion with curatorial staff, owner, or colleagues to assess if the removal is worth the risk (O'Loughlin et al. 1994). Care should also be taken to perform all required pre-treatment testing to ensure the stability of the object in regards to choice of solvents and treatment. Pre-treatment testing can consist of spot tests as well as more elaborate chemical analytical tests. The aim is to choose a treatment that best suits the need of the object. The theoretical framework and practical execution of pre-treatment testing will be discussed further in the case study.

Mechanical removal is a dry technique and as such it is deemed preferable over aqueous techniques that might give rise to solute induced damage such as bleeding or tidelines on the object. A dry mechanical technique is usually regarded as a first step in almost all treatments for the removal of a PSP. Initial removal of the carrier or other surface coatings and layers leaves the adhesive layer intact and accessible for further treatment, either dry or aqueous. Mechanical techniques include all techniques that cause a collision between the adhesive and the tool with the purpose to relax contact between the adhesive and the substrate, and cause a removal (Moncrieff & Weaver 1992). For mechanical removal various tools such as rubbers and gums, Teflon or bamboo spatulas, scalpels, and other hand tools can be used to try to lift and scrape of the carrier or adhesive layers. Rubbers and gums can be used to try to pick-up or abrade the adhesive further. A concern with mechanical techniques is the possibility of causing mechanical damage to the object. Sanding or rubbers can cause abrasion of the paper fibres or other surface layer while scraping can cause delamination of the surface of the object. In cases where the adhesive is thought to cause damage to the object either by degradation and discolouring or by risking adhering to other objects, the risks of mechanical damage can be deemed acceptable. (O'Loughlin et al. 1994; Smith et al. 1984)

Direct local application combines the use of solvents with mechanical removal. Using brushes, pipettes, cotton swabs, and other tools the conservator-restorer can apply the solvent directly onto the adhesive, or at the edges and observe the effects. This makes it possible to mechanically work the removal in stages, using spatulas and scalpels to lift and scrap off the adhesive. The technique enables the conservator-restorer to more closely observe the reactions between adhesive and solvent, but due to the local application, there is a risk of lateral movements of degradation product and pigments causing tidelines. (O'Loughlin et al. 1994)

Removal of adhesives on a suction table is conducted in a similar manner to direct local application with the addition of applied suction wicking the solvent down, into the adhesive, and enabling a removal of soluble particles and degradation products. The technique limits the possibility of solvent spread, which makes the technique more ideal to employ when working with objects with solvent-soluble media. Just as with direct local application, a disadvantage is the possibility of causing lateral movements of degradation products in the paper, giving rise to tidelines or bright halos at the treated area. The advantage of working on the suction table is that it eliminates the need for mechanical removal. (Smith et al. 1984)

Objects with no solvent-soluble media, usually single sheet objects, can be treated through immersion in a solvent bath. As such the technique is an aqueous one that acts upon the chemical interaction between the adhesive and the solvent, to swell or cause the adhesive to go into solute. The technique is fast and eliminates the risks associated with mechanical abrasion and tidelines caused by uneven wetting. Since the technique requires great amounts of solvent it is recommended to use it when many objects with similar PSPs need to be removed, to make it cost efficient. It is also recommended to place the immersion bath in a fume hood, to avoid unnecessary health risks. In immersion treatment the object is placed on a support and immersed in the solvent bath. After a while the carrier, if it has not previously been removed, can be lifted or scraped off. Upon removal from the bath, a cotton swab can be used to swab the adhesive area to make sure all adhesive has been removed. (O'Loughlin et al. 1994; Smith et al. 1984)

Another gentle way to remove PSAs and stains is through poulticing. Poultices comprise a way for indirect application of a solvent onto the substrate and the adhesive as a way to reduce the possibility of staining and formation of tidelines (O'Loughlin et al. 1994). A poultice consists of a two-way system including a solvent and an absorbent that prevents the solvent from evaporating too quickly, while causing a slow diffusion of solvent into the adhesive. The general thought is that the poultice should serve as an absorbent, which upon drying should pick up the adhesive and enable easy removal. Common materials used as an absorbent consists of clays, such as Fuller's earth, siliceous materials, such as diatomaceous earth, and cellulosic materials such as cellulose powder or blotters. Adhesives and gels can also be used as poultices, but with a reduced effect as an absorbent. Many of the common gels include tylose, methylcellulose, starch, agarose, and agar. (Smith et al. 1984) The use of adhesives and gels as poultices usually requires the addition of mechanical processing to lift and scrape off the swollen and solubilised adhesive. A concern regarding poultices is the risk of formation of deposits from the poultice material onto the object. Clays and fibres might get embedded into the porous surface of the paper object while more wet systems like gels might dry out and adhere to the surface.

### **2.5.1. Agar gel**

Agar is in itself an entirely non-toxic, biodegradable material that can be used as a poultice material in conservation practices. Due to its ability to form a rigid gel the problem of deposits and adhesion that many of the other poulticing materials have in common can be overlooked almost completely. In the following sections, agar gel will be examined and put into a paper conservation context.

#### **2.5.1.1. History and application**

Agars are classified as phycocolloids, gelling substances extracted from red algae (Rhodophyta phylum). The Agar polysaccharide accumulates in the cell walls of agarophyte algae species such as *Gelidium*, *Gracilaria*, and *Pterocladia*, and make up the polysaccharide reserve of the algae. The low molecular agar embeds the cellulosic fibres of the algae and through an entirely natural internal enzymatic process it polymerises and desulfates to form higher molecular agarose. Because of this the agarose content varies both with season and species of the algae (Armisen & Galatas 2009).

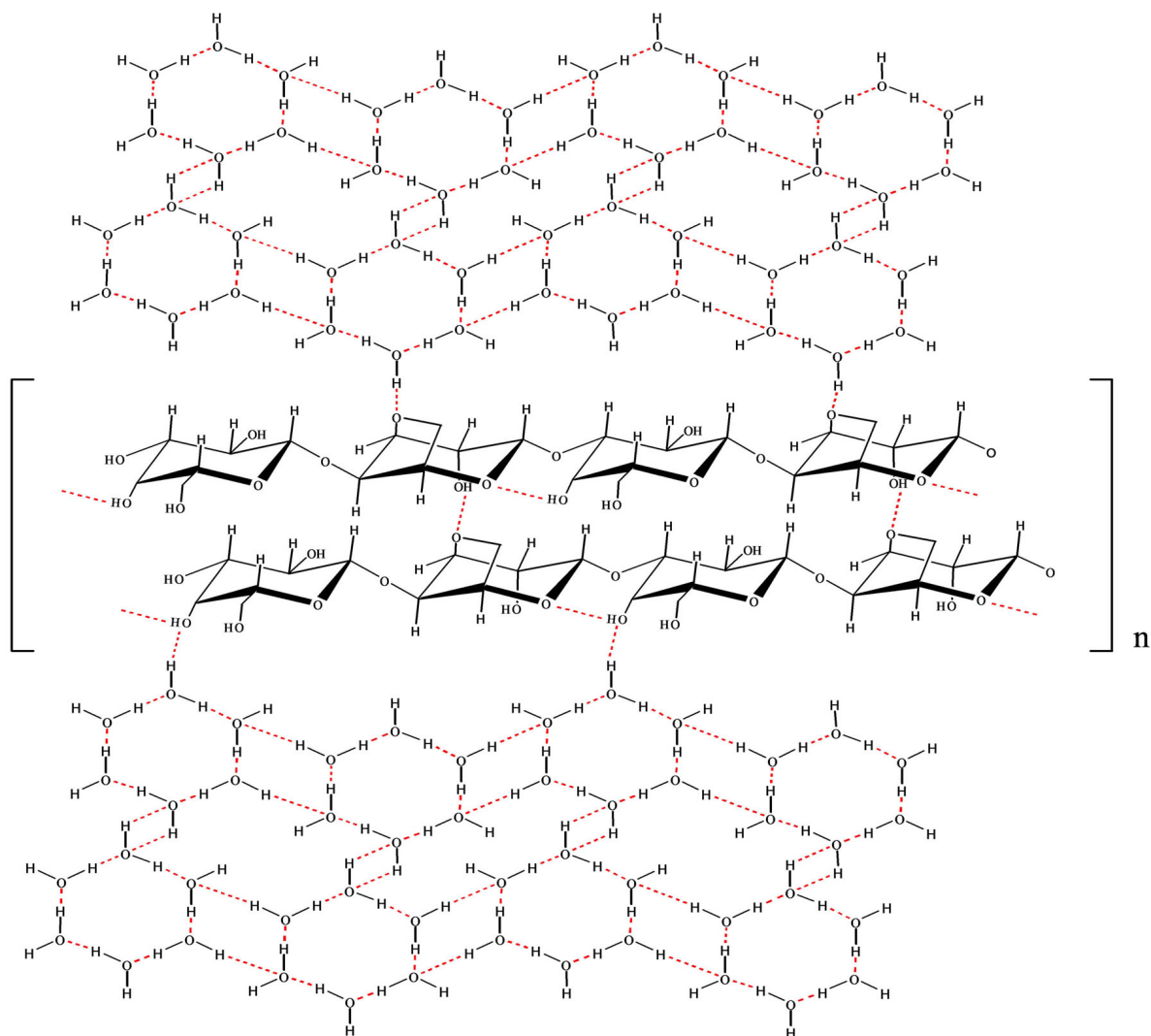
Agars are generally divided into 'natural agars' and 'industrial agars'. The first category includes agars prepared by artisans as an ingredient for food, while the second category includes manufactured food grade agars, and agars used for microbiology and biotechnology. Industrial agars are produced to



create a product that lives up to physio-chemical and bacteriological specifications, and in accordance to sanitary standards the agar is dehydrated to remove organic and inorganic salts, oligomers, and proteins. Dehydration via syneresis i.e. the extraction of a liquid from a gel, also enables the removal of soluble impurities present in the agar. (Armisen et al. 2009) Agars can be purchased as powder, flakes, and tablets, or as natural agar sheets and are insoluble in cold water.

### 2.5.1.2. Chemical composition and gelation

Agars are built-up of a mix of two different types of polysaccharides: agarose and agaropectin. Agarose is a linear polysaccharide with repeating units of the disaccharide agarbiose, and is an alternating copolymer of 1,3-linked  $\beta$ -D-galactopyranose and 1,4-linked 3,6-anhydro- $\alpha$ -L-galactopyranose (Warda et al. 2007). Agaropectins are polysaccharides consisting of a mix of highly charged galactans i.e. polymerised galactose, with substituted groups consisting of 5-8 % sulphate (Armisen et al. 2009), pyruvate, methyl, or glycuronate. The high density of hydroxyl groups (-OH) is a common trait in all galactans and gives rise to their hydrophilic character and ability to form strong hydrogen networks (Pierre, Delattre, Laroche & Michaud 2015).



**Fig. 5:** Gel formation of agarose molecules in the presence of water. Dotted lines represent the tetrahedrally directed hydrogen bonds. © CC BY (Tako, Tamaki, Teruya & Takeda 2014, p. 283).

The gelation properties of agar are dependent on the occurrence of agarose and are made up entirely of tetrahedrally directed hydrogen bonds forming between the water molecule and hydrogen of the



agarose polysaccharide molecule (see Fig. 5). A minimum concentration of 0,1 – 1,0% weight/volume (w/v) agarose is needed for the bond formation to take place and with pure agarose, bond formation occurs already at room temperature (Tako et al. 2014) while agar requires heating up to >85°C. After dispersion and subsequent heating of the agar powder in water, the agarose polymer chains uncoil and start to form double helices that agglomerate into three-dimensional networks with large pores (Warda et al. 2007).

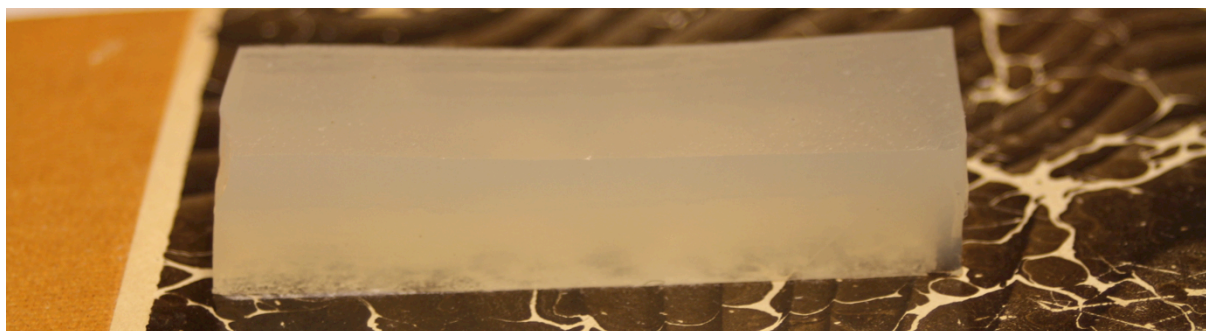
Another property of agar gel is its reversibility, giving rise to the term ‘reversible hydrocolloid gel’ or ‘sol-gel’ due to their semi-solid state (Scott 2012). When reheated, the rigid gel will melt and upon cooling, it will reform as a rigid gel, a procedure that can be repeated indefinitely (Armisen et al. 2009). From here on, the warm semi-solid colloidal solution will be referred to as sol, while the cooled rigid form will be referred to as gel.

### **2.5.1.3. Application of agar and agarose in paper conservation**

The use of agar and agarose gels in paper conservation literature has received very limited attention. It has been used with enzymes as an alternative to gellan gum for surface cleaning of water sensitive media (van Dyke 2003), or plain as a poultice material in the removal of water-soluble adhesives (O’Loughlin et al. 1994). It has also been proposed and evaluated as a medium for localized bleaching treatments (Burgess 1988) and for its ability to hold solvents, chelating agents, and surfactants (Scott 2012). In conservation practices it is most common to work with highly purified agarose that contains little or no sulphate. The advantage of pure agarose is the increase in gelation properties and its transparency, which make observations of potentially water sensitive media easier. The negative aspect of agarose is the high cost in comparison to agar and other materials used for poulticing. Almost all research in the conservation field is concentrated on agarose and as a result, much of the following information will revolve around agarose. But since agar has the same basic properties as agarose, this was considered acceptable, and that the information gained would be applicable regardless.

Agar is prepared by dispersing the dry powder or flakes in cold deionised water and heating to >85°C. During heating, the agar starts to network with the water molecules to form the sol, that upon cooling below 35°C will set as a rigid gel. As mentioned above, the agarose forms polymer–hydrogen bond networks at a molecular level, which give rise to an inherent porosity that is directly linked to the agar concentration. This makes agars easy to use and manipulate in regard to each individual treatment. Changes in concentration affect viscosity, absorption, and dispersion. Lower concentrations permit a higher degree of water diffusion into the object, while higher concentrations allow a lower degree of water diffusion based on capillary forces. (Scott 2012)

Criticism against agar and agarose as a poultice material often focuses on the gel’s ability to hold and diffuse a solution. Warda et al. (2007) tried a 1 % agarose gel for the removal of adhesives on paper and found the gel to be too wet. To retard the diffusion of water into the object, the gel had to be blotted before use. Iannuccelli & Sotgiu (2010) compared the diffusion rates of water into paper from agar gel and gellan gum and found the diffusion of water from agar gel to be substantially higher than that of gellan gum causing a weight increase of 1-33% in the paper substrate in 10 minutes (see Table 2). They also criticise the slightly higher opacity of agarose in comparison with gellan gum, claiming that this property makes agar unsuitable for treatments on works of art on paper (see Fig. 6). (Iannuccelli et al. 2010)



**Fig. 6:** Picture displaying the slight opaque appearance of agar gel.

Warda et al. (2007) also performed an overall evaluation of agarose where they analysed the colour stability and ageing resistance of the dry powder, as well as samples that had been in contact with agarose gel. The samples were also examined to spot potential gel deposits formed during treatment. The general conclusion was positive, showing no signs of discolouration of the samples that had been in contact with the gel, and no traces of deposited residue. (Warda et al. 2007)

**Table 2:** Percentage “of increase in weight in samples of Whatman paper (cat. no. 1001090) immersed in 200 ml of water and placed in contact with [...] Agar agar at increasing concentrations, for 10 minute and 18 hours respectively. Readings carried out with Wunder scales at 26°C and RH equal to 55%” (Iannuccelli et al. 2010). Table based on information provided by Iannuccelli & Sotgiu (2010). W indicates weight.

<b>W/Agar</b>	<b>10 minutes</b>	<b>18 hours</b>
W/H <sub>2</sub> O	242%	270%
W/Agar 1%	177%	206%
W/Agar 2%	150%	193%
W/Agar 3%	126%	175%
W/Agar 4%	125%	163%

Van Dyke (2003) applied an 1,2% agarose gel as a enzyme poultice for the removal of gelatin adhesive from an Indian miniature painting dating approximately 1675. During treatment, van Dyke observed the amber coloured adhesive being pulled up and into the agarose gel block (van Dyke 2003). This interesting property in agar gel depends highly upon its networked mesh structure that allows soluble macromolecules to pass through it, something that enables the absorption of large polymer structures (Armisen et al. 2009).

Because the gel has a gelling memory, it will essentially mould itself after the storage container (Armisen et al. 2009). Uneven wetting of the treated object might therefore be a result of this property. The problem can be mitigated by the application of slight pressure on the gel to improve its contact surface with the object. Using the gel in its sol state is also a way to mitigate this problem. When used as a sol the colloidal mix can be brushed onto an uneven surface and left to gel. The gel can then be removed by peeling force and might prove helpful in the removal of surface dirt on porous surfaces. When used as a sol, it is important to remember that the colloidal mix is still warm and care should be taken to ensure that the object to be treated can withstand the localised temperature change as well as the application of the gel in sol form.

### 3. Case study

The following case study has been conducted as a way for the author to put the information gained in the literature study into context. It was also carried out as a way to test and evaluate agar as a solvent gel. The case study is divided into four parts: an introduction to the material, a pre-treatment testing, a summary of the results gained in the pre-treatment testing, and the experimental. Results from the experimental will be presented in Chapter 4.

The case study focuses on barcode labels adhered to three library books on loan from the Humanities Library, of Gothenburg University Library. For the characterisation of the barcodes, stereo microscopy, ATR-FTIR, and spot tests were chosen as analysis methods. ATR-FTIR is an invasive, but non-destructive method of analysis. It was chosen as a suitable method for identification of the adhesive mass since all the samples could withstand the pressure needed to get a reliable result without causing harm to the objects. The aim of the pre-treatment testing was to answer the first formulated research question of this study: what kind of pressure-sensitive adhesive is used on the studied material and how are the labels built up? In the experimental, agar was used as a solvent gel in the attempted removal of the barcodes from the books. The experimental aimed to test the second research question: what are the advantages and disadvantages of working with agar as a solvent gel in the removal of pressure-sensitive labels in paper conservation?

To evaluate the result of the pressure-sensitive label removal, photographic documentation was conducted before and after treatment. Photodocumentation was carried out using a Canon EOS 100D, digital camera.

#### 3.1. Material

The Humanities Library, a part of Gothenburg University Library, provided the books used in this study. The books form part of a periodical suite, *Samlaren: tidskrift / utgifven af Svenska litteratursällskapets arbetsutskott*, 1883 – 1911. For a complete list see Table 3. They are edition half cloth bindings with brownish marbled paper covers and spine and corners in tan buckram cloth (see Fig. 7).

The books are available on open shelves, for in house use only. They were originally barcoded on the endpaper, but with the introduction of technology that enabled patron self-scanning, new barcodes had to be placed on the outer covers to enable easy machine reading. All barcodes have now been replaced by RFID-tags making the barcodes somewhat obsolete. The outer cover barcode still corresponds to catalogue data of the object and as such it can be seen as a back-up to the RFID-tag. The pressure-sensitive labels to be analysed and removed in this study are the barcodes on the outer front cover.

**Table 3:** List of book objects used in the case study.

Title	Year	Volume	Measurements
<i>Samlaren</i> : tidskrift / utgifven af Svenska litteratursällskapets arbetsutskott	1883 - 1885	4 - 6	24,5 × 16,7 × 3,5 cm
<i>Samlaren</i> : tidskrift / utgifven af Svenska litteratursällskapets arbetsutskott	1892 - 1893	13 - 14	24,5 × 17,8 × 3,3 cm
<i>Samlaren</i> : tidskrift / utgifven af Svenska litteratursällskapets arbetsutskott	1911	32	24,5 × 17,6 × 1,7 cm

In addition to the books, the author obtained three unused barcodes still attached to the release lining. With their exact resemblance to the barcodes used on the object, they were considered a valuable asset in the analysis, making it possible to subject the unused barcodes to the tests and in that way reducing

the amount of tests performed on the objects themselves. The barcodes measure  $2 \times 9$  cm and have a plastic laminate top layer and an adhesive that is clear and tacky at room temperature.

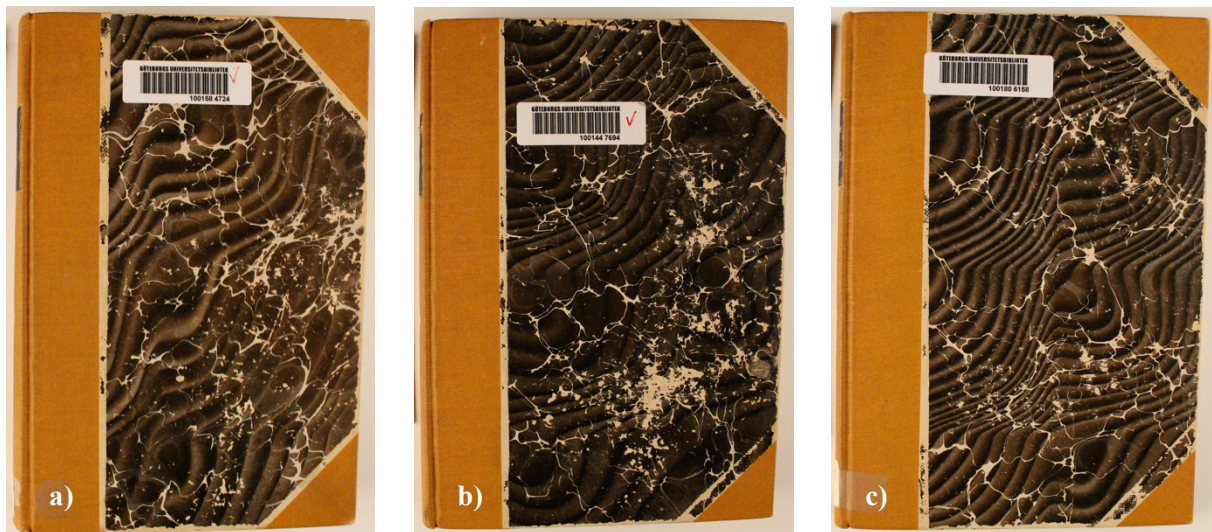


Fig. 7: Book objects used in the case study. a) 1883-1885, vol. 4-6, b) 1892-1893, vol. 13-14, and c) 1911, vol. 32

## 3.2. Pre-treatment testing

Pre-treatment testing poses an important step in the line of conservation treatment. The pre-treatment testing aims to help the conservator-restorer to analyse objects, materials, their composition and their reactions to different conservation techniques and solutes before treatment. This enables the conservator-restorer to formulate a hypothesis regarding the object and the treatment to be carried out while distinguishing potentially damaging treatments from safe ones in a controlled way, without putting the whole object at risk. Pre-treatment tests conducted were: stereo microscopy, analysis via ATR-FTIR, and spot tests.

### 3.2.1. Stereo microscopy

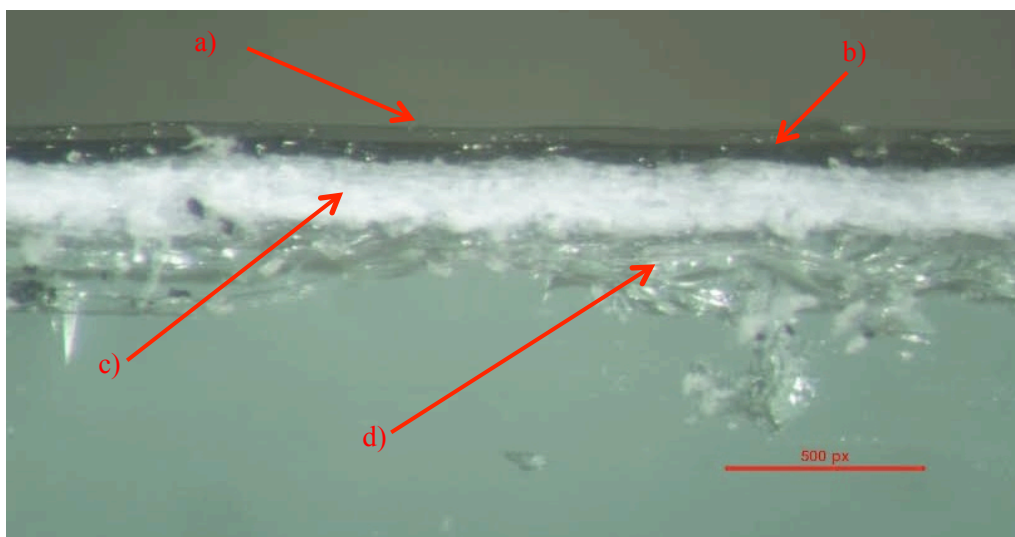
Microscopy constitutes an easy way to perform ocular analysis of an object or a material. The magnification enables the conservator-restorer to see things that are barely noticeable to the naked eye. In this study, stereo microscopy was used as a way to analyse the material build-up of the barcodes and for that, a cross-section was considered the most effective means of obtaining that information. Microscopy cross-sections of the barcodes were carried out on a stereo microscope at  $80\times$  magnification. Images for documentation were taken using a Nikon DS-Fi2 microscope camera-head.

#### 3.2.1.1. Performed

The cross-section was prepared by adhering a piece of one of the unused barcode samples on to a microscopy glass slide so that the label overlapped the edge of the slide. Using a razor blade, the label was cut along the side of the microscopy slide to ensure an even cross-section.

#### 3.2.1.2. Results

The microscopy cross-section revealed that the barcodes are built-up of three layers, with an additional printing layer. The barcodes consist of a clear plastic laminate top layer, a paper carrier that holds the printed information of the barcode, and a pressure-sensitive adhesive layer (see Fig. 8).



**Fig. 8:** Cross-section of pressure-sensitive label seen under 80× magnification, showing a) the plastic laminate top layer, b) ink, c) paper carrier, and d) pressure-sensitive adhesive.

### 3.2.2. ATR-FTIR

ATR-FTIR is an invasive, non-destructive mid-infrared, analytical method, commonly used to analyse materials such as liquids, polymers, powders, and pellets. The method is based upon the internal reflectance of a crystal with a high refractive index, generally diamond, germanium, or zinc selenide. The infrared beam reflects into the crystal and under the right conditions, the outgoing and the ingoing beam will occupy the same volume, leaving the beam nowhere to go but up. The refracted beam, called the evanescent beam or ‘hot spot’ will generally penetrate the sample somewhere between less than 0,5 micrometres ( $\mu\text{m}$ ) up to 10  $\mu\text{m}$  (Smith 2010).

To gather the spectrum of a sample, the sample is brought into close contact with the crystal and the ‘hot spots’ with the help of applied pressure. In regions where the sample absorbs energy, the evanescent beam will be attenuated and the attenuated energy of each beam is then passed back into the infrared beam and into the detector generating an infrared spectrum (see Fig. 8) (Smith 2010).

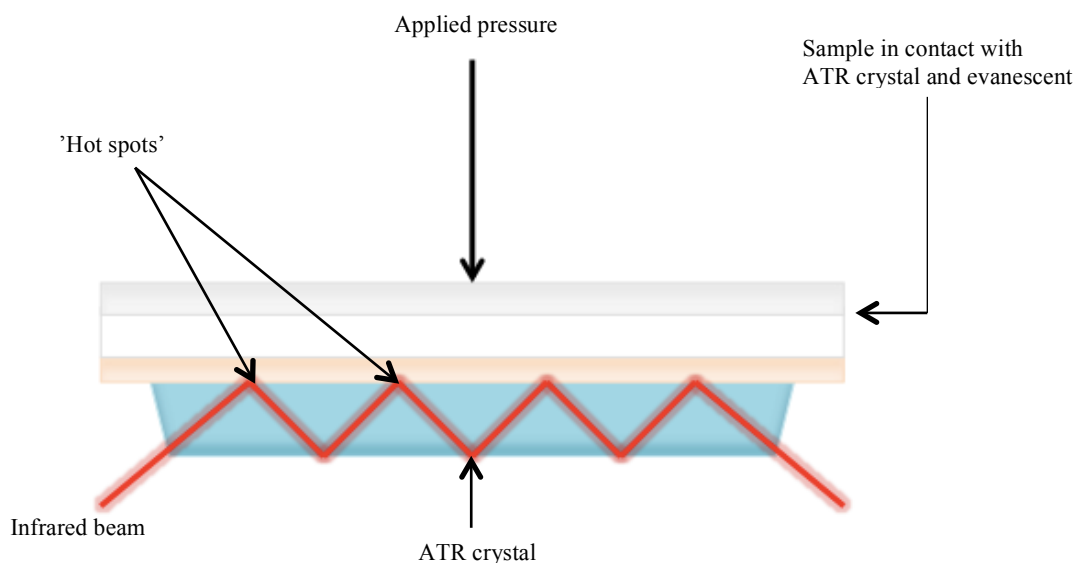
The ATR-FTIR spectra can be roughly divided into two regions: the ‘fingerprint region’, and the ‘functional group region’. The fingerprint region occupies the wavelengths between approximately 700 – 1200  $\text{cm}^{-1}$  and corresponds to the resonance within the molecules. For identification of an unknown sample, the fingerprint region will provide information to help aid in the identification. Comparing spectra with close resemblance within this region provides strong evidence towards a possible match. The functional group region occupies the wavelengths from approximately 1500  $\text{cm}^{-1}$  – 4000  $\text{cm}^{-1}$  and often shows larger peaks belonging to the functional groups that make up the majority of the molecules (Skoog, Holler & Nieman 1998).

Low or no sample preparation time makes ATR-FTIR easy and fast to use and enables the gathering of large amount of data in a short amount of time. Since the method is invasive it greatly limits its uses on conservation objects, but it has proven useful for the analysis of, for example, unknown repair materials such as adhesives, paper, and tape that can be removed from the object, or for the analysis of new materials to be used in the conservation practice to ensure high standards. Even though ATR-FTIR is easy to use, the analysis of obtained spectra and the connection of peaks with molecules for the exact identification of an unknown material require experience.

Since most PSAs are complex compositions, a plain ATR-FTIR analysis of the adhesive will not be able to clearly account for the presence of eventual fillers, plasticisers, antioxidants, or tackifiers, nor give a precise identification of the polymer. To accomplish such a complex analysis, the PSA need to



be treated to extract compositions other than the elastomer (Kendall 1989). In this study, the goal of the ATR-FTIR is to give a general identification of the elastomer and for this purpose the ATR-FTIR is adequate.



**Fig. 9:** Schematic picture of the IR-pathway through the ATR crystal and its contact with the sample through the evanescent wave or 'hot spots'.

### 3.2.2.1. Performed

An ATR-FTIR analysis was conducted to aid in the identification of the pressure-sensitive adhesive. Knowledge of the material components would help with the selection of solvents for the removal as well as give an understanding of how the adhesive would interact with its paper substrate chemically.

A  $1 \times 2$  cm piece of one of the unused barcodes, the reference, R, was cut and removed from the label release lining. Acetone was dropped onto the adhesive layer using a micropipette and the adhesive was subsequently scraped off using a steel scalpel. Using a bamboo spatula, the removed adhesive was placed on the ATR-FTIR crystal. This was done to eliminate the possibility of getting mixed results due to the evanescent wave reaching beyond the adhesive layer resulting in a spectrum containing peaks from the different material layers in the label. An additional piece of the unused barcode, R1, was then cut the same way as R and analysed, adhesive side directly on the ATR-crystal to see if the R and R1 spectra would differ.

A sample, S, was also taken from one of the objects. S had been placed with a degree of overlap on the buckram cloth resulting in low adhesion between the label adhesive and the cloth, which enabled easy sampling. S was cut in a  $0,3 \times 2$  cm piece and tested following the same procedure as R1.

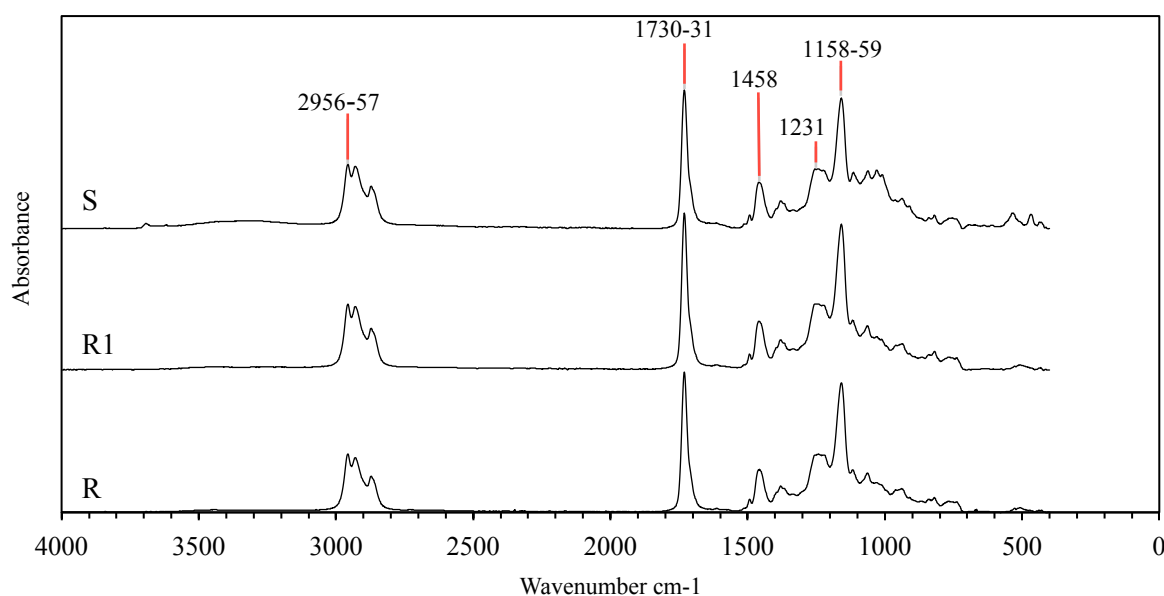
### 3.2.2.2. Results

Results from the ATR-FTIR analysis of R, R1 as well as the used sample S were compared to the OPUS 7.0 in-program reference library, the 'Infrared & Raman Users Group' (IRUG) spectral database (Infrared & Raman Users Group 2017) and a correlation chart published in 'Applications of Infrared Spectroscopy' (Skoog et al. 1998). The spectra (see Fig. 10) show characteristic peaks showing correspondence to an acrylic polymer (Gorassini, Adami, Calvini & Giacomello 2016), and a comparison of various spectra of synthetic resins in the IRUG database indicate an acrylic emulsion, possibly a poly(methyl acrylate), or a methyl methacrylate (MMA) (see Fig. 11) while the in-program reference library matched the spectra with poly(methyl methacrylate).



**Fig. 11:** From left to right: Molecular structures of poly(methyl acrylate) and methyl methacrylate (MMA). Figure created using ChemDraw Direct (Perkin Elmer 2016).

Absorbance at 2956 respectively 2957  $\text{cm}^{-1}$  indicates carbon-hydrogen (C-H) stretching of methylene groups ( $\text{CH}_2$ ) and methyl groups ( $\text{CH}_3$ ). Peaks at 1730 respectively 1731  $\text{cm}^{-1}$  indicate a double bond stretching of carbon=oxygen (C=O) of ester groups and at 1458  $\text{cm}^{-1}$  bending of C-H. According to Gorassini et al. (2016) the broader peaks at approximately 1231  $\text{cm}^{-1}$ , attributed to C-O stretching, and the strong peaks at 1158 respectively 1159  $\text{cm}^{-1}$ , attributed to stretching vibrations of C-C(=O)-O-groups can be used to identify acrylic compositions, in their case a commercial brand acrylic tape.



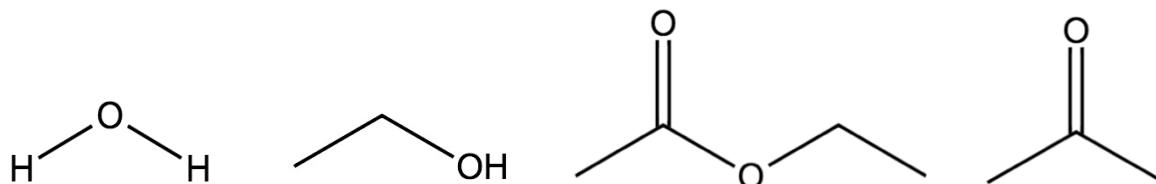
**Fig. 10:** Comparison of ATR-FTIR spectra of adhesive from R scraped reference sample, R1 reference sample, and S sample taken from one of the objects. To create a stacked spectra, the absorbance of each spectra was divided by itself, adding +1 for R1 and +2 for S to create different absorbance levels.

### 3.2.3. Spot tests and solvent choice

To determine solubility or swelling of the adhesive, and to counteract the occurrence of damages to the objects while carrying out the removal, spot tests were conducted on both the adhesive of the unused barcode and the marbled surface layer. When working with paper objects with potential water-soluble media, or objects with an elevated risk of structural damage from a wet treatment, a spot test is usually carried out, using all solutions considered for the treatment. It is important to remember that even though a spot test with the chosen solution gives a positive result, it only gives an approximate indication of how the object will react to a full treatment (Baker et al. 1990).

Solvents chosen for the case study and subsequent spot tests were: water (H<sub>2</sub>O), ethanol (C<sub>2</sub>H<sub>5</sub>OH), ethyl acetate (CH<sub>3</sub>COOC<sub>2</sub>H<sub>5</sub>), and acetone (CH<sub>3</sub>(CO)CO<sub>3</sub>) (see Fig. 12). The choice was based on parameters drawn up by the author. Parameters dictated were that they had to be:

- a) Safe to work with and within the author's level of experience to handle i.e. not being carcinogenic or health hazardous in a high extent and requiring minimal work in a fume hood or other setting that might obstruct the treatment.
- b) Assessed to be able to correspond to the non-polar end of a poly(methyl acrylate) molecule on the premises of like dissolves like.
- c) Polar enough to be able to be mixed into the agar gel.



**Fig. 12:** Molecular structures showing, from left to right, water, ethanol, ethyl acetate, and acetone. Figures created using ChemDraw Direct (Perkin Elmer 2016).

Since unused reference labels were available, it was possible to carry out a pre-treatment testing to determine how the PSA would react with the introduction of various solvents. This made it possible to test and eliminate solvents with no effect on the PSA before testing it on the surface layer of the objects and thus decreasing the amount of different solvents in contact with the objects.

#### **3.2.3.1. Performed**

The pre-treatment testing is divided into two sections: a) spot tests conducted on the pressure-sensitive adhesive of the unused label, and b) pre-treatment testing on the marbled surface layer of the book covers.

- a) Pressure-sensitive adhesive: 10 microliters (μl) of each solute to be tested, water, ethanol, ethyl acetate, and acetone, was taken with micropipette and dropped on to the pressure-sensitive adhesive of one of the unused reference labels. A steel scalpel was used to try to scrape the pressure-sensitive adhesive. The effect of the solutes and the mechanical processing was observed through a stereo microscope. Spot tests for ethyl acetate was carried out in a fume hood due to safety reasons.
- b) Marbled surface layer: The procedures for the spot tests on the marbled surface layer followed the procedures set up by Smith et al. (1984) for the testing of solvents on inks and colours. Spot tests were carried out on the marbled inside linings of the front cover where potential damage due to the spot tests would not cause distraction. The parameters were selected by the author from a list compiled in the 'Spot Tests' chapter of the *Paper Conservation Catalog* (Baker et al. 1990). Parameters evaluated were:
  1. Bleeding or feathering of marbling colorants
  2. Loss of surface sheen
  3. Loss of surface layer
  4. Staining
  5. Changes in surface texture



10 µl of each solvent to be tested was taken with micropipette and dropped on to the marbled surface layer of the books. The spot was subsequently blotted with a piece of blotter paper and both blotter and surface layer were observed through stereo microscope. The procedure was then repeated three times to ensure the stability of the marbled surface layer. Spot tests for ethyl acetate were carried out in a fume hood due to safety reasons.

### 3.2.3.2. Results

Results of the spot tests are presented in Table 4: pressure-sensitive adhesive, and Table 5: marbled surface layer.

**Table 4:** Information concerning solutions used for spot tests on the PSA and observations regarding swelling and solubility. Marking - indicates no observed reaction while ✓ indicates an observed reaction.

<b>a) Pressure-sensitive adhesive</b>			
<b>Solution</b>	<b>Swelling</b>	<b>Solubility</b>	<b>Observations</b>
Water	-	-	No reaction. Water resists to enter due to high surface tension on the PSA surface.
Ethanol	✓	-	Adhesive can be scraped from carrier.
Ethyl acetate	✓	-	Delamination of adhesive from paper carrier.
Acetone	✓	-	Delamination of adhesive from paper carrier.

**Table 5:** Performed spot tests on marbled paper surface and observed effects. Marking - indicates no observed reaction.

<b>b) Marbled surface layer</b>				
<b>Solution</b>	<b>Drops</b>	<b>Time/drop</b>	<b>Bleeding</b>	<b>Observations</b>
Water	3	30 seconds	-	No absorption of water into the surface layer.
Ethanol	3	20 seconds	-	Fast absorption. Loss of surface layer when blotted.
Ethyl acetate	3	20 seconds	-	Fast absorption. No observed damages.
Acetone	3	10 seconds	-	Fast absorption. No observed damages.
<b>Comments</b>				
Stereo microscopical observations of the blotter used for the ethanol spot test displayed minimal losses of surface layer, but whether this was because of the solvent or due to structural instabilities of the surface layer could not be determined. The spot test was repeated a second time with no observed damage. Other than this, no changes of the parameters in section 3.2.3.1. could be detected.				

## 3.3. Summary of pre-treatment testing

A pre-treatment testing was conducted to characterise the barcodes, the PSA, and to test the solubility of the PSA and the marbled surface layer. Methods used were stereo microscopy, ATR-FTIR, and spot tests.

The results of the cross-section stereo microscopy analysis showed that the barcode is built-up in three layers consisting of a clear plastic top layer, a printed-paper carrier, and a clear PSA. This indicated that it would be possible to remove the plastic top layer and a mist of the paper carrier of the barcode during treatment. A reduction in layers and material would help provide a higher degree of contact between the agar gel and the PSA as well as increase the diffusion of solvent into the PSA, thus increasing the probability of a successful removal.

The ATR-FTIR analysis did not give a clear match, but indicated a poly(methyl acrylate) emulsion. Identification of the adhesive enabled the identification of the molecules belonging to poly(methyl acrylate) and MMA, showing a slight non-polar property. This made it possible to try to tailor the choice of solvents on the premise of like dissolves like, by looking at molecule structures of suitable solvents and their respective solvation energies of solutes.

The choice of solvents was water, ethanol, ethyl acetate, and acetone. The solvents were used in two subsequent spot tests carried out on the PSA of one of the unused barcodes to determine swelling and solubility, and on the marbled surface layer of the objects to get an indication of possible damages that the solvents might cause. For the spot test on the PSA all solvents except water showed a swelling, making them eligible for the solvent gel experiment. None of the solvents tested on the marbled surface layer showed any substantial observable damage. This resulted in the conclusion that ethanol, ethyl acetate, and acetone would be used in the solvent gel experiment.

### 3.4. Experimental methods: agar as a solvent gel

In the following section, the preparation and the techniques used in the case study will be presented. Each technique was tried once per object and label.

#### 3.4.1. Preparation of agar as a solvent gel

Agar gel was prepared in three different batches for the three solvents to be used in the study, a) ethanol, b) 1:1 acetone:ethanol, and c) ethyl acetate. The solvents were chosen due to their low health risks and their somewhat polar nature, making them hydrophilic enough to be mixed with the colloidal gel mixture.

A 2% w/v agar gel was prepared by dispersion of 2 gram (g) agar powder in 80 millilitres (ml) deionised cold water, with the later addition of 20 ml solvent. The dispersion was heated to 90 °C on a hot plate while stirring continuously. After heating, the sol was removed from the hot plate and left to cool down to a temperature that allowed the different solvents to be mixed in. Due to the combustible nature of many solvents it is important to allow the sol to cool down well below the solvents boiling point before mixing it in.

1. 20 ml w/v ethanol was added to the sol around 20°C below boiling point ( $\approx 78^\circ\text{C}$ ) and mixed with a glass-stirring rod to achieve homogeneity. Enough sol to form a 1.5 cm thick gel was then poured into a wide glass container and sealed with plastic film.
2. Since acetone has been proven in previous studies to be immiscible with the colloidal mixture (Scott 2012), an equal amount of ethanol was added to improve miscibility. 20 ml v/v 1:1 acetone:ethanol was added to the sol around 15°C below the boiling point of Acetone (56.05°C) and mixed with a glass stirring rod to achieve homogeneity. Enough sol to form a 1.5 cm thick gel was then poured into a wide glass container and sealed with plastic film.
3. 20 ml v/v ethyl acetate was added to the sol around 30°C below boiling point (77.1°C) and mixed with a glass stirring-rod to achieve homogeneity. The solvent proved to be harder to mix with the colloidal mix and thus needed to be stirred extensively to achieve homogeneity. Enough sol to form a 1.5 cm thick gel was then poured into a wide glass container and sealed with plastic film.

The sealed containers were initially left to cool at room temperature and were later moved into a refrigerator. For additional information regarding preparation, see Table 6.

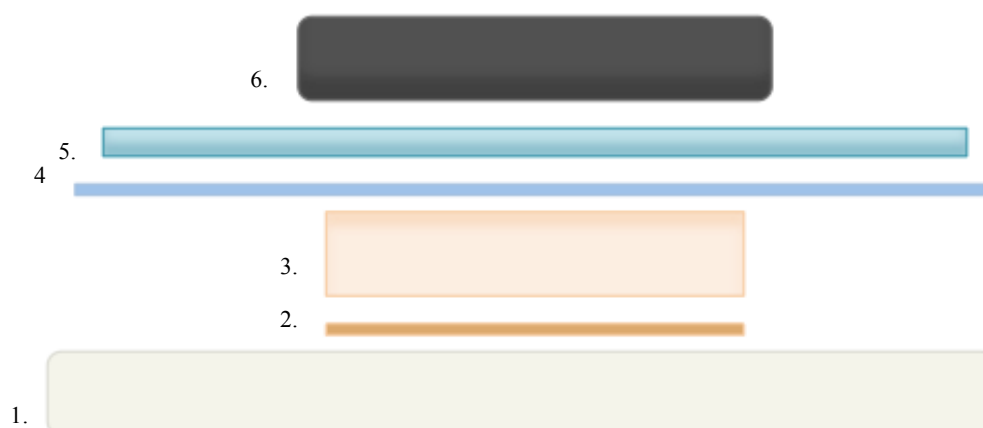
**Table 6:** Miscibility, eventual colour change, and other observations from the preparation of the solvent gels. Marking – indicates no observable reaction.

ID	Solvent	Miscibility	Colour change	Observations
1	Ethanol 20%	Miscible	-	
2	Acetone:Ethanol 1:1 20%	Miscible	-	
3	Ethyl acetate 20%	Does not mix as readily as ethanol.	Turns the colloid mixture slightly opaque.	The gel reaches saturation faster and some of the solvent does not diffuse into the gel.

### 3.4.2. Removal using agar as a solvent gel

Before application of the solvent agar gel, the plastic top layer of the barcode was mechanically removed using a scalpel to lift it. The paper carrier was left almost undamaged and could be thinned using a blunt scalpel to scrape away the paper fibres. This left the barcode with minimal amount of paper fibres still attached before application of the solvent agar gel.

Using a scalpel, the rigid gel was cut to match the measurements of the barcode ( $9 \times 2$  cm). The solvent agar gel was subsequently placed on the adhesive and paper fibre residue of the barcode. To minimise the risk of uneven wetting of the label, the gel was covered with a plastic film, a glass slide, and finally a light weight (see Fig. 13).



**Fig. 13:** Poultice with agar gel. 1. Object, 2. Adhesive to be removed, 3. Agar gel, 4. Melinex, 5. Glass or Plexiglas, and 6. light weight.

The gel and its effects on the object and the pressure-sensitive adhesive were monitored at 5-minute intervals. At these intervals the pressure-sensitive adhesive was mechanically worked with a steel spatula and tweezers. After solvent evaporation from the adhesive, the gel was once again applied and the procedure was repeated. For treatment with ethanol and acetone:ethanol a ventilator was sufficient to extract volatile gases from the solvents while the removal using ethyl acetate was conducted inside a fume hood.

After treatment with the solvent agar gel, the spot that had been in contact with the gel was cleaned using a cotton swab lightly wetted with deionised water to ensure removal of potential agar or solvent residues. The treated spot was then covered with non-woven polyester fabric and a blotter, secured with a weight and left to dry up for approximately 2 hours.

## 4. Results

In the following chapter, the results from the experimental are presented. The results are presented in Tables 7-9.

**Table 7:** Technique, amount of time/treatment, and other observations regarding the ethanol agar gel treatment and subsequent adhesive removal.

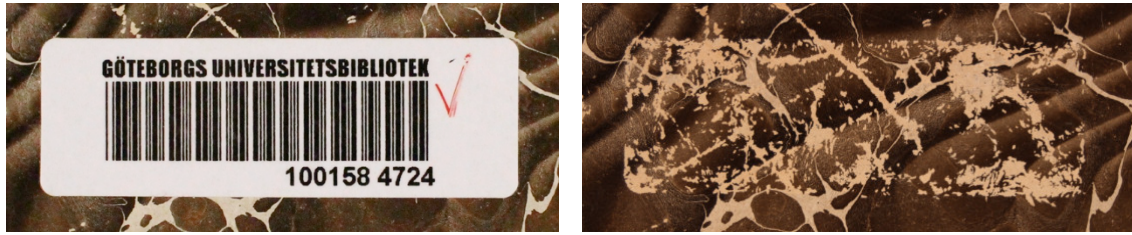
<b>1. Ethanol</b>			
<b>Agar</b>	<b>Solvent</b>	<b>Technique</b>	<b>Time per treatment</b>
2%	20%	Applied with pressure directly on adhesive	3 hours
<b>Observations</b>			
The ethanol treatment of one barcode took 3 hours to carry out. This included gel application and wait time for solvent diffusion into the substrate, but excluded preparation. Even with applied pressure, the gel caused uneven wetting of the label, but the overall wettability was considered high and the five-minute intervals more than enough to cause a slight swelling of the adhesive. Fast evaporation rates of the solvent resulted in short time frames for the mechanical removal. Removal caused a high degree of abrasion on the surface layer (see Fig. 14).			

**Table 8:** Technique, amount of time/treatment, and other observations regarding the acetone:ethanol agar gel treatment and subsequent adhesive removal.

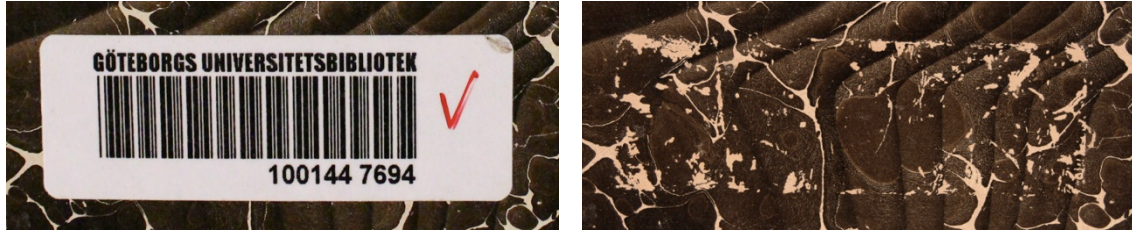
<b>2. Acetone:ethanol 1:1</b>			
<b>Agar</b>	<b>Solvent</b>	<b>Technique</b>	<b>Time per treatment</b>
2%	1:1 20%	Applied with pressure directly on adhesive	2.45 hours
<b>Observations</b>			
The acetone:ethanol treatment of one barcode took 2.45 hours to carry out. This included gel application and wait time, but excluded preparation. Even with applied pressure, the gel caused uneven wetting of the label, but the overall wettability was considered high and the five-minute intervals more than enough to cause a swelling of the adhesive. Fast evaporation rates of the solvent resulted in short time frames for the mechanical removal. Removal caused abrasion of the surface layer (see Fig. 15)			

**Table 9:** Technique, amount of time/treatment, and other observations regarding the ethyl acetate agar gel treatment and subsequent adhesive removal.

<b>3. Ethyl acetate</b>			
<b>Agar</b>	<b>Solvent</b>	<b>Technique</b>	<b>Time per treatment</b>
2%	20%	Applied with pressure directly on adhesive	1.15 hours
<b>Observations</b>			
The ethyl acetate treatment of one barcode took 1.15 hours to carry out. This included gel application and wait time, but excluded preparation. Even with applied pressure, the gel caused uneven wetting of the label, but the overall wettability was considered high and the five-minute intervals more than enough to cause high swelling of the adhesive. Working in a fume hood obstructed the removal and caused an increase in evaporation rates of the solvent, which resulted in short time frames for the mechanical removal. Removal caused minor damages to the surface layer (see Fig. 16).			



**Fig. 14:** 1883-1885, vol. 4-6. From left to right. Before and after removal with 20% ethanol solvent agar gel.



**Fig. 15:** 1892-1893, vol. 13-14. From left to right. Before and after removal with 20% 1:1 acetone:ethanol solvent agar gel.



**Fig. 16:** 1911, vol. 32. From left to right. Before and after removal with 20% ethyl acetate solvent agar gel.

## 5. Discussion

The literature study regarding conservation, and the removal of pressure-sensitive labels was largely based on literature with a focus on pressure-sensitive tapes. To complement the practical considerations, the author consulted literature with a focus on pressure-sensitive technology. This showed that the differences between tapes and labels were fewer than initially anticipated. What separates pressure-sensitive labels for library application from tapes are their build-up and application. With a similar history in terms of chemical composition of the PSA, most techniques for pressure-sensitive tape removal, choice of solvents, priorities, and considerations can be adapted for the removal of pressure-sensitive labels as well. One important factor that will affect the removal is the notion that many modern rubber-based, and acrylic-based pressure-sensitive labels for library application are manufactured with a high resistance to shear force. This property will greatly affect the adhesive–adherend bond, making the removal of pressure-sensitive labels for library application increasingly difficult.

Pressure-sensitive labels can be used for a variety of purposes, but for library application they have mostly been used to declare provenance through ex-libris, to indicate shelf location, or to electronically link material to patrons through barcodes and RFID-tags. Standpoints for the conservator-restorer to take into consideration before a potential removal seem to revolve around the context of the object in relation to the attached pressure-sensitive label. When regarding pressure-sensitive labels, the conservator-restorer should consider factors such as: whether it can be seen as an historic attachment or not; if it has caused damage; or if there is a risk that it might cause damage in the future; and, whether it obscures information vital to the interpretation of the object. For the object, values related to a heritage status often play a crucial role in the decision-making of whether or not to remove an attached pressure-sensitive label. When object and pressure-sensitive label are considered as separate, the decision-making can seem straightforward and simple, but in reality it is a complex procedure that cannot be easily systematised. The divide between new material, heritage material, and other collection divisions in libraries can be seen as a mechanism to simplify the decision-making by not prioritising a large part of the collections eligible for conservation. A heritage collection will, for example, have a high priority for conservation, while new collections will have a lower priority. It is understandable that these delimitations in decision-making must be made when it comes to large collections, but it is regrettable that many objects fall outside the parameters of conservation.

For objects that pass the initial considerations, and as a result, prove eligible for removal, other standpoints will guide the practical treatment for removal. The choice of solvents can generally be considered in a way, similar to all practical paper conservation. Spot tests, trial and error testing, experience and the use of systems such as Teas chart can be used to procure a suitable solvent that will cause the PSA to swell or go into solution, but yet prove to be sensitive to the object.

The removal of pressure-sensitive labels from library material can be an increasingly difficult problem to tackle due to the complex structure of the objects opposed to flat objects. Pressure-sensitive labels on book objects are commonly placed on the inside or outside covers, or even on end papers, thus greatly limiting the techniques that can be used for removal. Common techniques like immersion or working at the suction table, can in general be ruled out. Complete immersion risks causing solvation to other adhesive components in the book structure, and it would be a wrong to immerse an entire object for the removal of one label. The structure and thick boards used as covers also prevent the use of suction to pull the solvent through, bringing along degradation products and adhesive particles. This limits the choice of techniques available. In the removal of pressure-sensitive labels for library application, direct local application, and poulticing in combination with mechanical removal seems to comprise the best techniques since they can be used on substrates with varying thickness, with the advantage that they are localised. Direct local application can be hazardous to the object if the application is not conducted with care, but a carefully and well-planned direct application, either by applying solvent at the edge of the label or directly on top of the adhesive-mass can prove to be quite

effective, and decreases the time spent on the removal. Poulticing comprises a controlled way of causing solvent diffusion into the adhesive mass and depending on poultice material and adhesive component, the possibility of causing absorption of solvated adhesive mass up into the poultice limits the possibility of lateral movements of solvated degradation products.

The information gained from the stereo microscopy observation correlated with information from the literature study. Three clearly separated layers consisting of an adhesive, a paper carrier, and a plastic top laminate, could be detected. As mentioned above, a typical approach to a removal of tape and labels consists of the initial removal of the carrier. Having a carrier made out of paper greatly facilitated this approach, making it possible to delaminate the barcode by removing the plastic top layer without causing damage to the adhesive layer or the marbled surface layer of the books. After having scraped off the majority of the carrier, the adhesive layer was exposed for solvent treatment. Information about the build-up could have been gained directly during treatment, but with an increased risk of damaging the objects. The positive aspect was that the stereo microscopy gave this information prior to treatment, and as a result, eliminated the need to test the barcodes directly on the objects. If the carriers had been constructed out of plastic, this would have altered the entire approach.

Having a reference material, like the unused barcodes from the case study, greatly facilitated the initial understanding of the studied material. This meant that all destructive tests, like the stereo microscopy cross-section, adhesive spot tests, and to some extent ATR-FTIR could be performed on reference material, as a result, limiting the physical and chemical impact on the objects. It also enabled the author to gain a higher understanding of the pressure-sensitive labels, their build-up and composition, before initiating a removal. Even though the performed agar gel experiment to some extent required a trial and error-based execution, the author had a fairly good idea of which solvent that would work the best. One of the problems, regarding the ATR-FTIR analysis, was the lack of a complete in-software reference library. A demonstration library was available, but it did not seem to be extensive enough to give a complete identification. This forced the author to search for complementing information in databases like IRUG (Infrared & Raman Users Group 2017) and in articles (Gorassini et al. 2016). A problem was also that the author had too little experience in the interpretation of ATR-FTIR spectra. This might have caused some misinterpretations or simply caused loss of information relevant to the study, for example the presence of potential fillers.

The initial thought after the performed pre-treatment tests was that an increasingly non-polar solvent would cause the highest degree of swelling or solvation of the non-polar end of the poly(methyl acrylate) or MMA adhesive used on the barcodes. Based on this conclusion, acetone that has the highest non-polar content should have acted as the best solvent for the removal, but in the spot test of the PSA, no difference in swelling could be observed between acetone and ethyl acetate. Regardless of the result of the spot tests, ethyl acetate was the solvent that performed the best in the experimental. Based on this result, a few hypotheses could be formulated. The ATR-FTIR identification of the adhesive did not give an exact match, and further analysis would have been needed to isolate the presence of potential additives. This means that poly(methyl acrylate) or MMA could be a part of a larger molecular structures giving rise to a more complex picture regarding solvation energies, something that was not taken into consideration during analysis and subsequent treatment. A general problem regarding large polymer structures used for PSAs is that their solubility parameters cannot be sharply defined thus making it harder to pin point a solvent that would cause the polymer to go into solution (Horie 1987). As noted earlier, polar cross-linking sites might be added to acrylic-based adhesives to strengthen the adhesive-adherend bond. The presence of polar cross-linking sites may also lead to alterations in solvation energies causing a slight polar shift. These hypotheses could all lead to a PSA molecular structure with a solvation energy more favourable in ethyl acetate than acetone giving the ethyl acetate agar gel the advantage as removal material.

Initiating a removal of a modern acrylic polymer posed an interesting problem to the author. In most pressure-sensitive tape literature, there is a focus on the removal of rubber-based and protein-based

adhesives. The removal of acrylic polymers is often mentioned fleetingly, resulting in a lack of case studies and other literature with regards to their removal. This left the author with little knowledge on how the material would react during a removal. Even with the pre-treatment testing the author experienced a factor of trial and error during the process of removal and this enabled the author to try to connect information from the literature study while the problems were arising. Further discussions regarding the agar and the removal will be discussed below.

## 5.1. Agar as a solvent gel

Both the literature study and the case study proved that agar comprised a versatile, biodegradable, and non-toxic material for conservation, and for the removal of pressure-sensitive labels. One of its greatest advantages lies in the possibilities of customisation through change in concentration. A change in concentration will affect the gels ability to diffuse and absorb solutions and particles. The network structure based on hydrogen bond formations also opens up for the possibility of adding bleaching agents, enzymes, chelating agents, and solvents. However, the choice of solvents is restricted by polarity, dictating that the solvent needs to be hydrophilic enough to interact with the agarose to form hydrogen bonds. In the performed case study, this posed a slight problem. The only solvent that was considered polar enough to be mixed in with the sol was ethanol, while the slight shift in non-polarity in ethyl acetate and acetone could prove to be problematic. In a previous study conducted by Scott (2012), acetone had been tested and proved immiscible by itself, a problem that could be counteracted with the addition of ethanol, as a result, this was adapted into the case study. Since no information was found regarding the miscibility of ethyl acetate, this mixture proved to be a small experiment in itself. Most likely due to the non-polar shift, the ethyl acetate proved hard to mix with the colloid mixture and the 20% added, caused over-saturation of the sol. This left a solvent layer on top of the sealed gel and caused pits to form in the top layer of the rigid gel during cooling. With its rigid form the gel proved to be easy to cut in the desired shapes to match that of the label to be removed. It was rigid enough to handle without causing it to break and enabling easy application and removal. Being a physical gel, the possibilities of it leaving residues was also non existing, something that has been previously proved by Warda et al. (2007).

In previous studies (Iannuccelli et al. 2010; Warda et al. 2007), the high diffusion of solution into the paper substrate has been criticised for being too fast and uncontrolled. This problem was only detected in the ethyl acetate solvent gel, which was over-saturated to begin with. An explanation for this could be the increase in concentration causing a slower solvent diffusion. In the study by Warda et al. (2007), they used a 1% gel, which in opposition to the 2% gel used in this study, would have caused a 27% weight increase in the paper substrate in 10 minutes due to diffusion (see Table 2). This shows that the problem of high diffusion can be mitigated by an increase in agar concentration. In the performed study, 2% proved to give a good diffusion, but for working with more sensitive material, a higher concentration could probably be used. A problem with diffusion in the case study was connected to the gelling memory of agar. Uneven solvent diffusion of the scraped label fibres and adhesive was a recurring result of all three removals. Using a weight, the problem was tried to be mitigated, but could not be completely avoided. The problem shows one of the disadvantages of the gelling memory of agar that enables it to mould itself according to the container, in which it is kept, during agglomeration and subsequent gel formation (Armisen et al. 2009). In the preparation, circular glass containers were used to hold the sol during gel formation, and the author failed to take into notice the slight convex appearance of the bottom of the glass containers. This shows the importance in choosing an even container. In this case, the uneven wettability did not pose a major problem since multiple gel applications had to be performed in order to complete the removal. This enabled the author to work with the removal more systematically, wetting and removing one piece at a time.

As stated in section 2.5.1.3. 'Application of agar and agarose in paper conservation' another critique against agar is connected to the slightly opaque nature of the gel. While this might be an important aspect when working with works of art on paper and water sensitive media, this property did not pose



an obstacle when working with objects with a proven water and solvent stability. Because the gel could easily be manipulated into a shape matching the adhesive label, the solvent gel never touched the surface layer directly, which counteracted any possible damage to the surface layer that the gel or solvents might have caused. The close intervals of observation would also have helped counteract a possible occurrence of bleeding or other damages caused by the gel or solvents.

The fast evaporation rate of the solvents used in the study made the poulticing technique cumbersome and slow. The solvents helped to cause a swelling of the pressure-sensitive adhesive, which made it possible to mechanically scrape and lift the pressure-sensitive adhesive. But parallel to the solvent evaporation over time, the adhesive got increasingly tougher and the initial tack of the adhesive returned and allowed the adhesive to regain its bond with the substrate. This clearly shows the inherent properties of polyacrylic adhesives, which allow them to be increasingly resistant to solvents and remain tacky in room temperature.

Since most available conservation literature focuses on agarose, the author struggled to find information that could explain the full extent of the advantages of agarose in comparison to agar, except with regard to translucency/opacity. With an economical advantage far greater than that of agarose it is hard to see why agar has been rejected as a material for conservation. One of the factors that may explain this is related to presence of agarpectin. Due to the lack of industrial application of the agarpectins present in agar, little research has been conducted to evaluate their role and effect in agar gel (Armisen et al. 2009). The presence of sulphate, pyruvate, methyl, or glycuronate could be troublesome for the use of agar in conservation, if they prove to be water, or solvent soluble. The use of deionised water could minimise the risks to some extent due to the fact that the ions that might cause the sulphate to go into solution can be filtered out. Most industrial agars are today dehydrated through syneresis, a process that contributes to the removal of soluble impurities, which would further eliminate the possibilities of having soluble impurities diffuse into the substrate, making agar in theory a safe choice for uses in conservation.

## 5.2. Ethical aspects

Due to the homogeneity and saturation of the polymers making up most modern acrylic-based PSAs, they are seen as stable and resistant to ageing and degradation. In comparison with rubber-based PSAs they compose a more suitable labelling material for cultural heritage material. But all PSPs, including acrylic-based ones, have an expected performance life and shelf life, and in comparison to the expected life span of most cultural heritage objects, PSPs are ephemeral at best. The potential damage they may cause to an object or a collection may in many cases overshadow the positive aspects. These potential threats are often discovered when the damage is already done, causing, in many cases, irreversible damage such as discolorations, translucency, or bleeding of pigments and inks due to migration of plasticisers.

Many books and library objects, which fall under the previously mentioned category of new material, lack most of the basic values often discussed in conservation practices. For these books and objects the fundamental value consists of their informational value, a value closely corresponding with availability. The informational value requires the patron – object interaction to be truly meaningful. But as such they often lack other values like economical, cultural, emotional value, and with the strict delimitation between heritage and new material, they are also generally bereft of their age value. Material from the late 1800s and early 1900s are still either in their ‘transient phase’, meaning they are still generally available and are common objects, while some have gone even further and entered the ‘rubbish phase’ where they have ceased to be desirable. This loss in value reflects clearly onto the view of both patrons and professionals facing this new material. Excessive labelling and unconsidered placing of labels on covers, over designs and pictures, or even over information all comes back to the lacking view of the objects as valuable. Labelling also symbolises accessibility of the object, which is a good thing, but poorly executed labelling can in turn lead to a relaxed view on the objects, causing mishandling. For the sake of the objects and collections, a higher consciousness is needed regarding

labelling in context to objects. Guidelines for labelling, and tests to determine sustainability regarding the ageing and material build-up of pressure-sensitive labels should be adapted to all institutions working with cultural heritage material with the need for labelling. Guidelines and good communications with manufactures can to a high degree ensure the development and purchase of a labelling material that meets the guideline standards.

### 5.3. Conclusions

Cross-sectional analysis showed that the studied pressure-sensitive labels are built up of three layers: adhesive, a printed carrier or backing, and a clear plastic laminate top layer or surface coating. ATR-FTIR analysis and comparison of the adhesive of both the labels used as a reference material, as well as the sample taken from one of the labels on the objects, showed that all spectra were largely similar. The closest match in the in-program reference library was with poly(methyl methacrylate), a hard clear plastic, and in the IRUG-database with a methyl containing polyacrylate emulsions. After analysis, the adhesive was characterised as a methyl containing poly acrylate emulsion, possibly poly(methyl acrylate), or MMA.

Regarding the agar, the study showed that it is an economical, and extremely versatile poultice material that is, in itself, biodegradable and non-toxic. Some of its advantages comes from its nature as a physical gel, causing it to form molecular networks that can retain, diffuse, and absorb solutions and particles. Changes in concentration enable the conservator-restorer to customise the agar gel to cause a diffusion-rate suitable for the object to be treated and with the possibility of adding bleaching agents, enzymes, chelating agents, and solvents it can be used for a great number of purposes. Since agar requires hydrogen bonds to agglomerate, it needs some amount of water. This limits the amount of solvent that can be added to the mixture making the solvent gel gentler than, for example, direct local application and other solvent treatments. Its rigid form makes the gel easy to handle during application and removal, and prevents residual deposits on the substrate.

Criticisms mentioned in the literature often include high diffusion-rates and a shift towards opacity, although none of these problems were seen as substantial in the performed case study. Problems were detected with uneven wettability due to, at places, low contact between the gel and the substrate, and the rigidity of the gel counteracted the author's attempt to mitigate the problem with a weight. The chemical composition of agar also limits the solvents that can be added to the mixture. Polar, hydrophilic solvents have an advantage, but might not always pose the best choice in regard to the object to be treated. A question that yet remains to be answered is the suitability of agar as a solvent gel in conservation due to the presence of agarose and its impurities. Theoretically these impurities should be minimal due to dehydration during industrial grade processing, and the use of deionised water should help prevent solubilisation of remaining sulphate ions. Because of the fast evaporation rate of the solvents and the return of initial adhesive tack parallel to evaporation, the gel had to be consequently reapplied to introduce more solvent and moisture into the adhesive system to enable further mechanical processing. This caused the procedure to be time-consuming and not nearly effective enough, in regards to acrylic-based adhesives.

The placing of labels and barcodes on hard covers greatly limits the techniques that can be used for their removal. Common techniques such as solvent immersion and solvent treatment on a vacuum table are not always possible. If the label to be removed is a fairly modern one, contact with manufacturer should be established. Good communication with the manufacturers or technicians working in the pressure-sensitive trade can enable access to more specified product information, including chemical composition. The considerations regarding a removal often constitute a complex process that requires individual judgements and considerations, which almost always come down to the object and the pressure-sensitive label to be removed. The study showed that considerations, which the conservator-restorer needs to take into account, can be divided into three general aspects:

1. The purpose of the label and historic circumstances related to the label,

2. Whether the label obscures or contains information crucial to the interpretation of the object as an entity, and
3. damage caused, or potential damage that might arise from the continued adhering to the substrate.

Lastly, an important question to keep in mind, when assessing the risk of a procedure, is if the removal is really required and why?

#### 5.4. Further research

Since agar consists of agarose, a sulphated polysaccharide, there will always be a small amount, around 3 – 10%, of sulphate and other minor functional groups present in the gel. For direct application on an object, these sulphates could potentially cause degradation of the paper. To establish agar as a more economical alternative to agarose as a poultice material, further research would need to be carried out to study the potential effects of the agarose. This could be done by a more chemical analysis of the interaction of the agarose with various solvents and cellulose fibres, but also through accelerated ageing of paper samples after being exposed to agar gel.

A more extensive study of pressure-sensitive labels for library application and their adhesives would also be of great interest. The ATR-FTIR proved to be an effective method for analysing the pressure-sensitive adhesive and the author would have liked to be able to continue to map modern PSAs used for library labels both by sampling in collections, and through manufacturers and vendors. Further studies could also include a higher focus on polymers used as elastomers, chemical analyses to try to isolate potential fillers, but also accelerated ageing to try to connect more to the actual problems caused by pressure-sensitive labels in library collections.

## 6. Summary

The use of pressure-sensitive labels in library collections often constitutes a compromise between the assignment of the conservation professional to preserve the material as it is, and the library's duty to make the material available. It is often failed to take into notice the ephemeral nature of pressure-sensitive labels, and especially their adhesives. Common damage on the material includes yellowing, brittleness, adhesive creep, 'cold flow', and translucency in the substrate. The purpose of this study is to present information regarding pressure-sensitive labels and how to remove them in an attempt to broaden the existing information on pressure-sensitive tapes and adhesives available to conservator-restorers. The aim of the study is to implement information in a case study, where the author attempts the removal of self-adhesive barcodes from paper covered half bindings, using agar, commonly known as agar-agar, as a solvent gel.

Two methods have been used to conduct the study: a literature study and a case study. The literature study aimed to investigate pressure-sensitive labels for library application, techniques, and considerations for their removal from paper substrates; while, the case study was conducted to test and evaluate the use of agar as a solvent gel for the removal of barcodes from paper covered library books.

For the case study, three books were made available by the Humanities Library of Gothenburg University Library. The books are a part of a periodical suite from the late 1800s, early 1900s and are comprised of edition half-cloth bindings labelled with barcodes that are no longer in use. In addition to the books, unused barcodes of the same kind were obtained. Two research questions were formulated for the case study: what kind of pressure-sensitive adhesive is used on the studied material and how are the labels built up? And, what are the advantages and disadvantages of working with agar as a solvent gel in the removal of pressure-sensitive labels in paper conservation?

To analyse and characterise the studied material, a pre-treatment testing was conducted. The pre-treatment-testing included stereo microscopy, ATR-FTIR, and spot tests conducted on both the PSA as well as on the surface layer of the books. From the barcode cross-sections studied in stereo microscopy, it could be concluded that the barcodes were built up of three layers consisting of a clear adhesive, a paper carrier, and a plastic top layer laminate. A build-up that opened up for delamination of the plastic top layer before attempted removal by solvent gel, thus enabling solvent diffusion through the paper carrier. Analysis of the PSA was conducted on the unused barcodes as well as on a sample taken from the objects. The results were analysed through an in-software reference database, as well as through the IRUG database and it could be concluded that the adhesive was composed of a methyl containing polyacrylate, possibly poly(methyl acrylate), or MMA.

For the removal, a 2% concentration agar was used. The preparation was split in three batches to obtain different solvent gels: 20% ethanol, 20% 1:1 acetone:ethanol, and 20% ethyl acetate. Literature as well as the performed case study showed that agar comprises a versatile material for conservation. Through change in concentration, properties such as retention, diffusion, and absorption can be modified, and the possibilities to add bleaching agents, enzymes, chelating agents, and solvents makes it applicable not only in adhesive removal, but in various paper conservation treatments. Properties such as opacity and high diffusion often mentioned in criticism against agar did not pose a problem in the study. The ethyl acetate solvent gel enabled a clean removal, but the technique was considered inadequate due to the fast evaporation rates of the solvents used and the time spent on carrying out the removal.

To connect the information gained in the literature study and the case study a third research question was formulated: what are some of the standpoints that need to be taken into consideration in the process of removal of pressure-sensitive labels for library application? The study showed that considerations which the conservation-restorer need to take into account can be divided into three general aspects: the purpose of the label and historic circumstances related to the label, whether the

label obscures or contains information crucial to the interpretation of the object as an entity; and, damage caused, or potential damage that might arise from the continued adhering to the substrate.

After the study was completed, it was clear that pressure-sensitive labels do not substantially differ from pressure-sensitive tapes. The build-up and their applications may differ slightly, but the adhesive-induced problems are the same, and priorities and guidelines previously compiled by O'Loughlin and Stiber (1994; 1992) and Smith et al. (1984) still apply to some extent. The advantages of pressure-sensitive labels for library application are many and this decreases the possibilities of their complete removal from use within collections. Developing routines such as material tests, policies and guidelines adapted for handling and application of labels could substantially help prevent potential damage that might arise from adhesives and the placing of labels on material, whether it is considered heritage or new material.

## 7. Sammanfattning

Användningen av tryckkänsliga, eller självhäftande, etiketter i bibliotekssamlingar utgör ofta en kompromiss mellan konservatorns uppdrag att bevara materialet så som det är och bibliotekets plikt att göra materialet tillgängligt. I denna kompromiss är det lätt att glömma den efemära natur som präglar tryckkänsliga etiketter och speciellt deras adhesiv. Vanliga skador utgörs av gulning och skörhet av adhesivet, att adhesivet kryper, 'cold flow' och att underlaget blir genomskinligt. Syftet med denna studie är att presentera information om tryckkänsliga etiketter och hur man avlägsnar dessa i ett försök att bidra till det redan existerande fältet av konserveringslitteratur om tejp och adhesiv. Målet är sedan att implementera informationen i en fallstudie där författaren kommer att försöka avlägsna självhäftande streckkoder från biblioteksböcker med hjälp av agar, allmänt känt som agar-agar, som en lösningsmedelsbaserad gel.

För att utföra studien användes två metoder, en litteraturstudie och en fallstudie. Målet med litteraturstudien var att undersöka tryckkänsliga biblioteketiketter samt tekniker och ställningstagande för deras avlägsnande från papper, medan fallstudien ämnade testa och utvärdera användningen av agar som lösningsmedelsbaserad gel vid avlägsnandet av självhäftande streckkoder från biblioteksböcker.

Humanistiska biblioteket, en del av Göteborgs universitetsbibliotek, lånade ut tre böcker som användes i fallstudien. Böckerna är en del av en periodikasvit från slutet av 1800-talet - början av 1900-talet och består av halvklotband som märkts upp med streckkoder under sent 1900-tal. Efter implementeringen av RFID-etiketter i bibliotekets samlingar, är streckkoderna inte längre i bruk. Utöver böckerna fick författaren även tillgång till oanvända streckkoder av samma typ som de som använts på objekten. För att gå vidare i fallstudien formulerades två frågeställningar: vilken typ av adhesiv består de tryckkänsliga etiketterna av och hur är dessa uppbyggda? Och vilka är fördelarna respektive nackdelarna med att arbeta med agar som en lösningsmedelsbaserad gel i avlägsnandet av tryckkänsliga etiketter?

En förstudie utfördes för att analysera och identifiera materialet. Förstudien inkluderade analyser genom stereomikroskop, ATR-FTIR och spottester utförda både på adhesivet såväl som på böckernas ytskikt. Utifrån observationer gjorda i stereomikroskop på streckkoder i genomskärning kunde slutsatsen dras att dessa var uppbyggda i tre skikt som utgörs av ett genomskinligt adhesiv, ett pappersskikt och ett topplaminat av plast. Identifieringen öppnade upp för möjligheten att delaminera topplaminatet innan avlägsnandet av streckkoderna med hjälp av agar påbörjades och på så sätt möjliggöra diffusion av lösningsmedel genom pappersskiktet ner till adhesivet. Analys av adhesivet genom ATR-FTIR utfördes, på såväl de oanvända streckkoderna, som ett prov taget från en av böckerna. Analysresultatet analyserades i ett referensbibliotek tillgängligt i mjukvaran, samt ställdes mot spektra tillgängliga genom IRUG. Den närmsta matchningen visade att adhesivet består av en poly akrylat med högt metylinnehåll, troligtvis poly(metyl akrylat), eller MMA.

För att utföra avlägsnandet av streckkoderna användes en 2% agar gel som i tre omgångar blandades med olika lösningsmedel: 20% etanol, 20% 1:1 aceton:etanol och 20% etylacetat. Den studerade litteraturen såväl som fallstudien visade att agar utgör ett mångsidigt material för konservering. Förändringar i koncentration påverkar gelens egenskaper såsom dess förmåga att bibehålla en vätska samt dess förmåga till diffusion och absorption. Det är också möjligt att blanda in olika medel som blekningsmedel, enzymer, kelatbildare och lösningsmedel vilket gör gelen användbar inte bara för borttagning av adhesiv utan för flertalet områden inom papperskonservering. Egenskaper såsom opacitet och hög nivå av diffusion som ofta kritiserats i tidigare studier utgjorde inget problem i fallstudien. Den agar gel som blandats med etylacetat möjliggjorde ett rent avlägsnande med minimalt med skador på ytskiktet, men på grund av den snabba avdunstningen av lösningsmedel, den ständiga översikten och lågsamma utföranden, bedömdes tekniken vara inadekvat för fallstudien.

För att binda ihop informationen från litteraturstudien formulerades en tredje frågeställning: vilka är några av de faktorer som man behöver ta ställning till vid avlägsnandet av tryckkänsliga biblioteketiketter? Studien visade att de ställningstaganden som en konservator behöver ta hänsyn till kan delas in i tre generella aspekter: syftet med etiketten och dess historiska omständigheter, huruvida etiketter täcker eller bidrar till information som är relevant för tolkningen av objektet, och nuvarande skador eller skador som kan tänkas uppstå vid ett fortsatt vidhäftande till underlaget.

Efter att studien slutförts var det tydligt att tryckkänsliga etiketter inte skiljer sig substantiellt från tejp. Deras komposition och applikation skiljer sig minimalt, men de problem som härrör från adhesiven är de samma och som sådant kan prioriteringsordningar och riktlinjer som tidigare ställts samman av bland annat O'Loughlin och Stiber (1994; 1992) och Smith, Jones, Page och Dirda (1984) användas. Fördelarna med tryckkänsliga biblioteketiketter är många, något som bidrar till deras fortsatta behov inom biblioteksverksamheterna. Dock kan förändrade arbetsrutiner vad gäller materialtester, riktlinjer samt policys för hantering och applicering bidra till att motverka eventuella skador åsamkade av tryckkänsliga etiketter på biblioteksmaterial. Något som kan förbättra förhållandena för materialet, oavsett om det bedöms vara kulturarvsmaterial eller nytt material.

## Tables and figures

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Photos taken by Alexandra Tengelin. All illustrations made by Alexandra Tengelin if nothing else indicated.

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- Fig. 4, p. 10 Teas chart. The triangular chart makes it possible to calculate the molecular interaction according to the hydrogen bonding ( $f^H$ ), dispersion forces ( $f^D$ ), and dipolar forces ( $f^p$ ). Marks 1-4 represent solvents to be used in the case study. 1. Water, 2. Ethanol, 3. Ethyl acetate, 4. Acetone.
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- Fig. 16, p. 27 1911, vol. 32. From left to right. Before and after removal with 20% ethyl acetate solvent agar gel.

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# Appendix 1: Materials and manufacturers

## **Agar powder**

VWR International bvba  
Geldenaaksebaan 464  
B-3001 Leuven Belgium

## **Ethanol**

VWR International S.A.S  
ZI de VAUGEREAU  
F-45250 Briare  
FRANCE

## **Acetone**

VWR International S.A.S  
201 Rue Carnot  
F-94126 Fontenay-sous-bois  
FRANCE

## **Ethyl acetate**

VWR International S.A.S  
201 Rue Carnot  
F-94126 Fontenay-sous-bois  
FRANCE